Structural Factors that Govern the Synthesis Efficiency of Organoplatinum Poly(dendrimer)s

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Abstract

This thesis described the research work on the synthesis of poly(dendrimer)s using an outer-sphere–outer-sphere connection strategy. Two G1–G3 series of dendritic macromonomers having short branches (**73–75**) and long branches (**76–78**) containing two surfaces (functionalized acetylene surface groups) were prepared in high purity and copolymerized with three different type of platinum linkers **65**, **152** and **156** to form organoplatinum poly(dendrimer)s. The structural effects of the dendritic macromonomers and platinum linkers on the polymerization efficiency were investigated.

Chapter 1 gives a brief introduction about dendrimers, their synthesis, characterization methods and applications. Chapter 2 summarizes the current research progress on the various dendritic nanostructures, namely, dendronized polymers, dendrimer-linear polymer hybrids, poly(dendrimer)s and poly(dendrimer) networks. In Chapter 3, we reported the detailed synthesis of the two series of dendritic macromonomers (S-Gn and L-Gn, n = 1–3) with different branch lengths (C–O for S-Gn and C–C–C–O for L-Gn) and hence varying structural flexibilities. The structures of these dendritic compounds were characterized by ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy, gel permeation chromatography (GPC), mass spectrometry (MS) and elemental analysis. The polymerization reactions with *trans*-Pt-linker **65** was firstly conducted to obtain organoplatinum poly(dendrimer)s Pt-S/L-Gn **144–149**. It was found that the structurally more rigid dendritic mamcromonomers (S-Gn series) have a higher degree of polymerization (DP) than the structurally more flexible one (L-Gn series) of the same generation. This was attributed to the formation of cyclic oligomers in the latter series. In

Chapter 4, we investigated the polymerization behavior of the two series of dendritic macromonomers towards two other Pt-linkers of different geometry, namely, *cis*-Pt-linker **152** and *long-trans*-Pt-linker **156** to obtain poly(dendrimer)s *cis*-Pt-S/L-Gn **157–162** and *long-trans*-Pt-S/L-Gn **163–168** respectively. It was revealed that only oligomers and/or cyclic oligomers were formed in both S-Gn and L-Gn series in the former case. In contrast, poly(dendrimer)s *long-trans*-Pt-S/L-Gn **163–168** were obtained in high DP values and the formation of oligomers and/or cyclic oligomers was greatly suppressed. A model was proposed to account for the different polymerization outcomes based on the different steric and structural environment of the two reacting partners. A conclusion of this work and an outlook of the project were given in Chapter 5.

摘要

本論文敘述了運用球外層之間的相互連接策略來合成聚樹枝狀化合物的研究工作。兩系列擁有短分枝(**73–75**)和長分枝(**76–78**)的高純度樹枝狀巨單體(第一代、第二代和第三代)被合成，它們擁有兩個乙炔官能基在其表面，與三種鉑絡合物(**65**, **152** 和 **156**)進行共聚反應而生成有機鉑聚樹枝狀化合物，當中樹枝狀巨單體和鉑絡合物的結構對聚合效益的影響已被研究。

第一章簡略地介紹了樹枝狀化合物的合成、表征方法和應用。第二章總結了現時樹枝狀納米結構的研究進展，包括樹枝狀聚合物、樹枝狀化合物-線性聚合物雜交物、聚樹枝狀化合物和樹枝狀聚合物網。在第三章中，我們詳細地報道了兩系列樹枝狀巨單體(S-Gn 和 L-Gn, n = 1–3)的合成方法。由於它們擁有不同長度的分枝(S-Gn: C–O 和 L-Gn: C–C–C–O)，所以它們在結構上也擁有不同的剛性。這些樹枝狀化合物的結構已通過核磁共振氫譜、碳譜、凝膠滲透色譜、質譜和元素分析進行了表征。我們首先以反式-鉑絡合物 **65** 進行共聚反應，從而生成了有機鉑聚樹枝狀化合物—鉑-S/L-Gn **144–149**。其中發現了在同一世代中，結構上剛性較強的樹枝狀巨單體(S-Gn

系列)比結構上剛性較弱的樹枝狀巨單體(L-Gn 系列)有較高的聚合度。這是由於後者會生成環狀寡聚物之緣故。在第四章中，我們探討了兩系列樹枝狀巨單體與另外兩種鉑絡合物(包括順式-鉑絡合物 **152** 和長-反式-鉑絡合物 **156**)的聚合情況，繼而分別生成了順式-鉑-S/L-Gn **157–162** 和長-反式-鉑-S/L-Gn **163–168**。無論是 S-Gn 或 L-Gn 系列，與順式-鉑絡合物 **152** 進行共聚反應時，都只會生成寡聚物和/或環狀寡聚物。然而，獲得的長-反式-鉑-S/L-Gn 聚樹枝狀化合物 **163–168** 卻擁有高聚合度，而只生成痕量寡聚物和/或環狀寡聚物。基於兩個反應物不同的空間和結構環境，我們提出了一個模型去說明不同的聚合結果。第五章總結了這個研究計劃及其展望。

Abbreviations and Acronyms

AFM	atomic force microscopy
Ar	aromatic
a.u.	arbitrary unit(s)
Bn	benzyl
brs	broad singlet (spectral)
°C	degree Celsius
calcd	calculated
δ	chemical shift in parts per million
d	day(s); doublet (spectral)
DIBAL-H	diisobutylaluminium hydride
DMF	*N*,*N*-dimethylformamide
DP	degree of polymerization
EI	electron impact
equiv.	equivalent
ESI	electrospray ionization
Et	ethyl
EtOAc	ethyl acetate
EtOH	ethanol
Et_2O	diethyl ether
FAB	fast atom bombardment
g	gram(s)
GPC	gel permeation chromatography
h	hour(s)
HRMS	high-resolution mass spectrometry
Hz	hertz
IR	infra-red
J	coupling constant
L	liter(s)
LAH	lithium aluminium hydride
LLS	laser light scattering
μ	micro
m	multiplet (spectral); milli
M	molar (moles per liter); mega
M^+	parent molecular ion
MALDI-TOF	Matrix-assisted laser desorption/ionization time-of flight
Me	methyl
MHz	megahertz
min	minute(s)
mM	millimole(s) per liter
mmol	millimole(s)
M_n	number-average molecular weight
mol	mole(s)
mp	melting point
MS	mass spectrometry
M_w	weight-average molecular weight
m/z	mass-to-charge ratio
NMR	nuclear magnetic resonance
PDI	polydispersity index
ppm	part(s) per million
q	quartet (spectral)
quin	quintet (spectral)
R_f	retention factor
R_h	hydrodynamic radius

rt	room temperature
s	singlet (spectral)
SEM	scanning electron microscopy
STM	scanning tunneling microscopy
t	triplet (spectral)
THF	tetrahydrofuran
TLC	thin layer chromatography
TMS	trimethylsilyl
UV	ultraviolet
vis	visible
v/v	volume-to-volume ratio

Publications Originated from the Work of This Thesis

1. Cheung, S.-Y.; Chow, H.-F. "Organoplatinum Poly(dendrimer)s", *Abstract of "12th International Conference on Polymers and Organic Chemistry 2006 (POC'06)"*, **2006**, Okazaki, Japan, p.P-43-a.
2. Cheung, S.-Y.; Chow, H.-F.; Ngai, T.; Wei, X. "Synthesis of Organometallic Poly(dendrimer)s by Macromonomer Polymerization: Effect of Dendrimer Size and Structural Rigidity on the Polymerization Efficiency", *Chem. Eur. J.* **2009,** *15*, 2278.
3. Cheung, S.-Y.; Chow, H.-F. "Synthesis of Organoplatinum Poly(dendrimer)s: Pronounced Effect of Size and Geometry of Small Organoplatinum Linkers on the Copolymerization Efficiency with Bifunctional Dendritic Macromonomers", *Chem. Eur. J.* **2009,** *15*, 8874.

Chapter 1

Introduction to Dendrimers

In the past two decades, a kind of special molecule—"dendrimer" not only played an important role in polymer chemistry, but also in nanotechnology. They also possess many applications in different fields (these will be discussed in Section 1.4) according to their functional structures. The synthesis and properties of dendrimers have already been summarized in several excellent reviews and monographs.[1]

This thesis describes the use of dendritic units as the basic building blocks towards the construction of more complex macromolecular systems. Here we will first summarize some fundamental concepts about dendrimer chemistry, and in the next chapter we will review the use of dendritic units to build up higher order macromolecular systems.

1.1. Dendrimer

Dendrimers are hyperbranched, fractal-like macromolecules of defined three-dimensional size, shape and topology. Basically, a dendrimer can be mainly divided into two parts—the core and the dendrons (Greek: dendron means tree) (Figure 1).

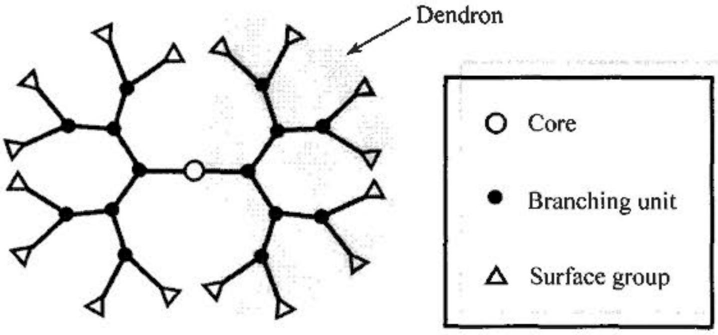

Figure 1. *Schematic representation of a third-generation (G3) dendrimer.*

A sector-shaped dendron consists of branching units and surface groups. Surface groups play an important role in the dendrimer properties such as solubility. The number of concentric layers between the core and the surface groups is referred to the dendrimer generation (Gx). Therefore dendrimers with high generation tend to adopt spherical structure because of steric hindrance between their surface groups.

1.2. Synthetic Approaches

In contrast to linear polymers and hyperbranched polymers, dendrimers are synthesized by stepwise and controlled procedures, therefore they have perfect architectures and are called "monodisperse". By convention, "monodisperse" means that the polydispersity index (PDI = M_w/M_n) of a dendrimer is smaller than 1.05. Due to this structural homogeneity, the structural-property relationship of dendrimers, in contrast to that of polymers, can be precisely rationalized. Needless to say, the synthetic works are much more challenging.

The synthetic approaches of dendrimers are divided into two main categories—divergent and convergent methods. There were also some other synthetic methods for dendrimers, but the principles are still based on these two approaches.

1.2.1. Divergent Methods

The divergent synthetic method was developed at the early stage during the development stage of dendrimer chemistry. The contributors of this method include Vögtle,[2] Denkewalter,[3] Tomalia,[4] and Newkome.[5]

According to the divergent method, dendrimers grow from the core, through the branching units and finally ends in the surface groups (Figure 2). Firstly, the core **1** is coupled to the protected branching units **2** and the dendrimer starts to grow. After deprotection, the surface groups are functionalized and readily react with the same protected branching units **2** again. By repeating this iterative cycle, the dendrimer grows larger and larger to form higher-generation dendrimers. The dendrimer growth can be terminated by anchoring the dendrimer with the desired surface groups **3**. Although this method is the most efficient procedure for preparing high-generation dendrimers, incomplete reaction of all the surface groups is a potential problem due to the sterically crowded environment. Therefore the anchoring reaction between the dendrimer and the surface groups must be highly efficient, otherwise it will create structural defects on the dendrimers. In addition, selective conversion of only one or several surface groups cannot be achieved, therefore specific surface-functionalized dendrimers[6a, 6b] cannot be prepared by this method.

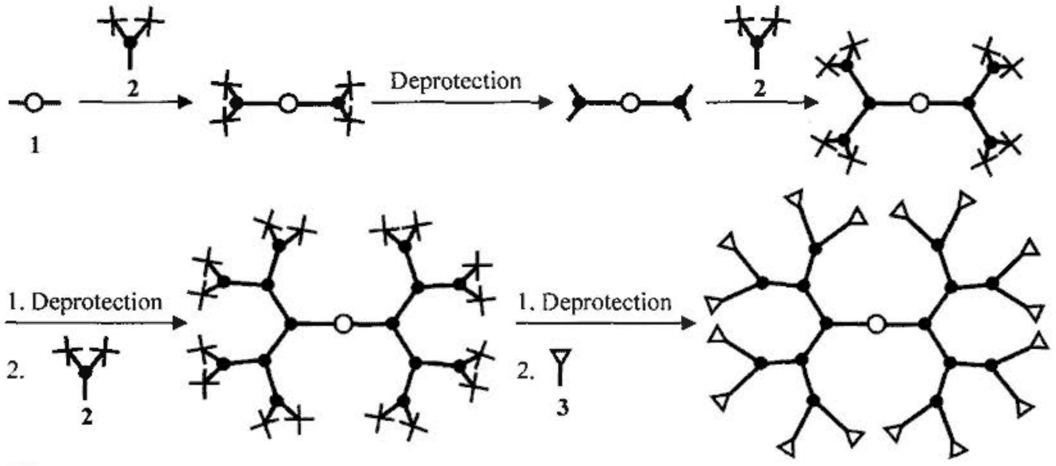

Figure 2. *Divergent synthesis of a G3 dendrimer.*

1.2.2. Convergent Method

This method was developed by Fréchet[6] and Miller[7] independently. In contrast to the divergent method, this synthetic pathway starts from the surface groups (Figure 3). The surface groups **3** are firstly fixed onto a focal point-protected branching unit **4** to form a protected G1 dendron **5**. After the functionalization (deprotection) of the focal point, the G1 dendron **6** is readily react with the branching unit **4** again.

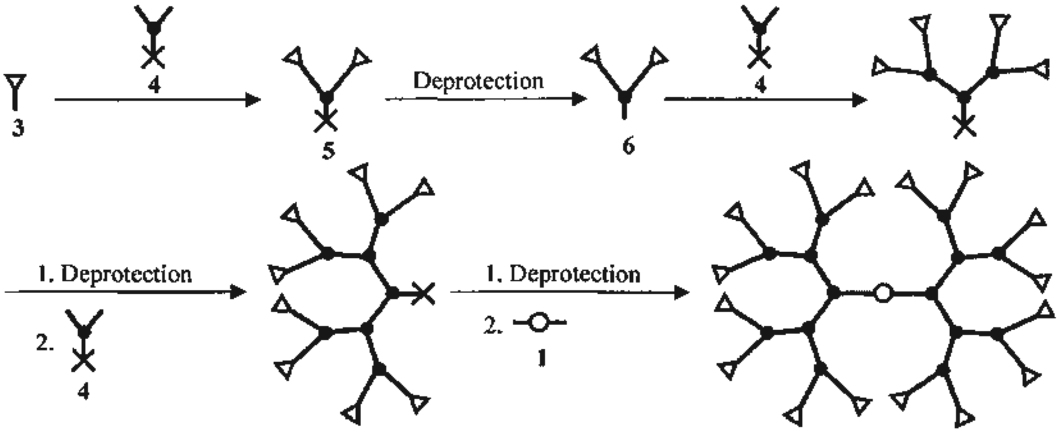

Figure 3. Convergent synthesis of a G3 dendrimer.

Higher-generation dendrons (e.g. G3) can be prepared by repeating the iterative cycle. The final step is anchoring of the dendrons onto the core 1. This method is more time-consuming when compared with the divergent method, but the number of side reactions is lowered and the resulting dendrimers would have more perfect structures or they are monodispersed. Moreover the surface groups can be functionalized specifically by the convergent method. This is very important because the required dendrimers in this project are specific surface-functionalized dendrimers that contain only two specific functional surface groups (Figure 4). By this method, layer-block dendrimers, segment-block dendrimer[6e] and specific surface-functionalized dendrimers can now be easily prepared (Figure 4).

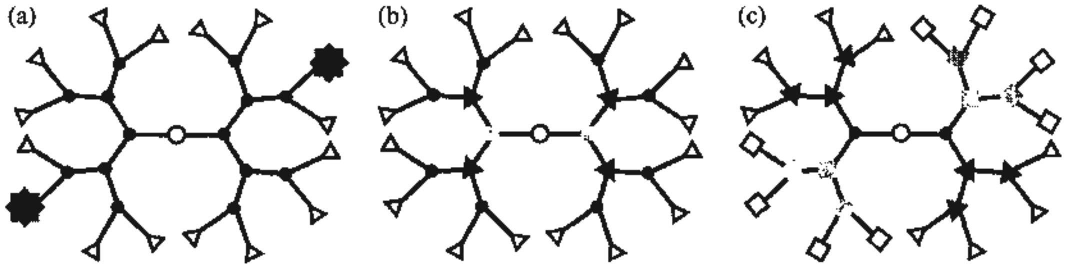

Figure 4. A schematic diagram for (a) specific surface-functionalized dendrimers, (b) layer-block dendrimers and (c) segment-block dendrimers.

1.2.3. Miscellaneous Methods

Divergent and convergent methods have their own advantages and disadvantages, and stepwise synthesis and purification are involved in both methods. In order to facilitate the preparation of high-generation dendrimers with narrow molecular weight distribution in large quantity, some other synthetic methods had been developed including double-stage hypercore convergent growth,[8] double exponential growth,[9] branched monomer approach,[10] orthogonal coupling strategy[11] and solid support synthesis.[12] Details of such methods can be found in the references cited therein.

1.3. Applications

Since dendrimer molecules adopt spherical structures that cannot be found in linear polymers and they can also be functionalized at different positions including surface groups,[13] branching units[14] and the central core[15] by stepwise synthetic procedures, they have many properties that are different from linear polymers and therefore have a wide range of applications in different fields such as catalysis,[1j, 14, 15a, 16] medicinal chemistry,[17] light harvesting/emitting materials,[15b, 18] electrochemistry[18a, 19] and molecular recognitions.[20]

1.3.1. Catalysis

The catalytic sites can be anchored either on the surface groups or the interior parts of dendrimers. In the former case, it provides multiple catalytic sites with high local density at the dendrimer periphery.[16c] For dendrimers with catalytic center located at the core, their dendritic sectors encapsulate the reactants and provide higher reaction selectivity, and sometimes enhance the reactivity due to microenvironment effect.[14, 16d, 16f] Dendrimers can also be used as supports[16e] and serve as hosts to encapsulate metal nanoparticles[16b] for recoverable catalysts.

1.3.2. Medicinal chemistry

The shape of dendrimers is generally spherical and they are easily functionalized to interact with cell membranes, therefore dendrimers are one of the suitable candidates for gene delivery purpose.[17f] By anchoring targeting drugs onto dendrimers, they can also deliver drugs owing to their encapsulating ability.[17d, 17i–17k] In addition, they can be used as magnetic resonance imaging (MRI) contrast agents.[17a]

1.3.3. Light harvesting/emitting materials

Energy-donating and energy-accepting functionalities can be decorated on specific positions of dendrimers by stepwise synthetic procedures and the spacial distance and extent of electronic conjugation between them can also be adjusted, therefore providing tunable light-harvesting properties.[18f] Some dendrimers also have liquid crystalline properties[18m] and light-emitting properties[18k] that can be used in organic light-emitting diodes (OLED).[18n]

1.3.4. Electrochemistry

Dendrimers containing metal centers are good candidates for simulating biological redox processes since the sterically hindered dendrons mimic metalloproteins surrounding a redox active site in the biological system.[19b] In the case of dendrimers with multiple metal accepting ligating sites, they can also act as molecular sensors.[19c]

1.3.5. Molecular recognitions

Dendrimers have defined size and shape that make them suitable for use as hosts in molecular recognition. They use their interior parts or peripheries to bind with guest molecules by non-covalent interactions such as hydrogen bonds, metal-ligand coordination, hydrophobic interaction and electrostatic interactions.[20a]

Chapter 2

Using Dendritic Molecules as Building Blocks

2.1. Using dendritic molecules as building blocks

In the last chapter, the structures, synthesis, properties and applications of dendrimers have already been introduced. In this chapter, the author will focus on how to use dendritic molecules, including dendrons and demdrimers, as building blocks to obtain ordered macromolecular architectures.

Dendritic molecules can be linked up either randomly or in highly ordered manner. This chapter will only focus on the "ordered build-up" approach, and the "random build-up" of dendritic molecules will not be mentioned here.[4a, 21]

Basically, there are four types of macromolecular architectures that are constructed from dendritic molecules: 1) dendrimer-linear polymer hybrids, 2) dendron-dendrimer connectivity, 3) dendron-dendron connectivity and 4) poly(dendrimer)s. In the following sections, the formations of these four macromolecular architectures will be reviewed.

2.2. Dendrimer-linear polymer hybrids

In the dendrimer-linear polymer hybrids, dendrons or dendrimers are connected to linear polymers. These architectures can be constructed by either connecting linear polymers to the focal point of dendrons or linking linear polymers to the periphery of dendrimers (Figure 5).

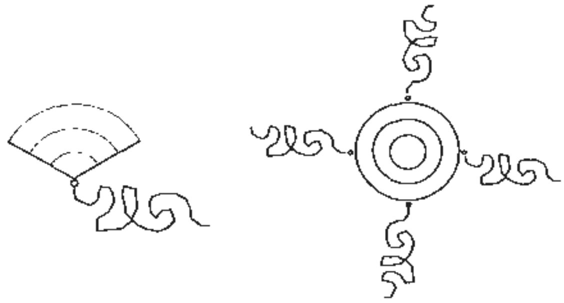

Figure 5. *The structures of dendrimer-linear polymer*

The first example of dendrimer-linear polymer hybrid was reported by Fréchet.[22] The hybrid 7 (Figure 6) was prepared by connecting polyethylene glycols (PEGs) with two G4-Fréchet-type dendrons by Williamson-ether synthesis.

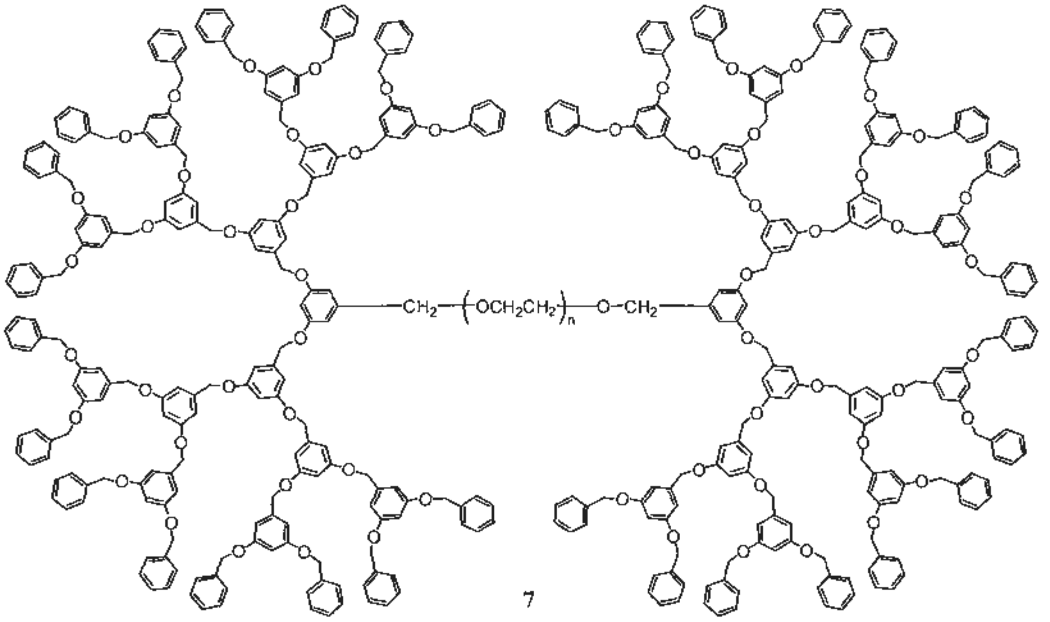

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Figure 6. *Dendrimer-linear polymer hybrid 7 reported by Fréchet.*

Conformational changes of hybrid 7 in different solvents (THF, chloroform and methanol) were found as the “dendron part” and the “polymer part” of the hybrid would possess different solubilities in various solvents. In THF, dendrons are more soluble than the PEG chain, therefore the PEG chain would “hide” between two sterically hindered dendrons. On the other hand, the more hydrophobic dendrons were surrounded by the expanded PEG chain in the more polar MeOH solution.

The solvent dependent conformation property was also investigated by Mackay and coworkers.[23] Dendrimer hybrid **8** was prepared by living free radical polymerization (Figure 7). It was found that the size of the dendron would expand in benzene, with the polystyrene (PS) chain wandered within the expanded dendron and led to less exposure to the solvent. On the contrary, the size of the dendron became relatively smaller in chloroform solution and the polystyrene chain became exposed to the solvent.

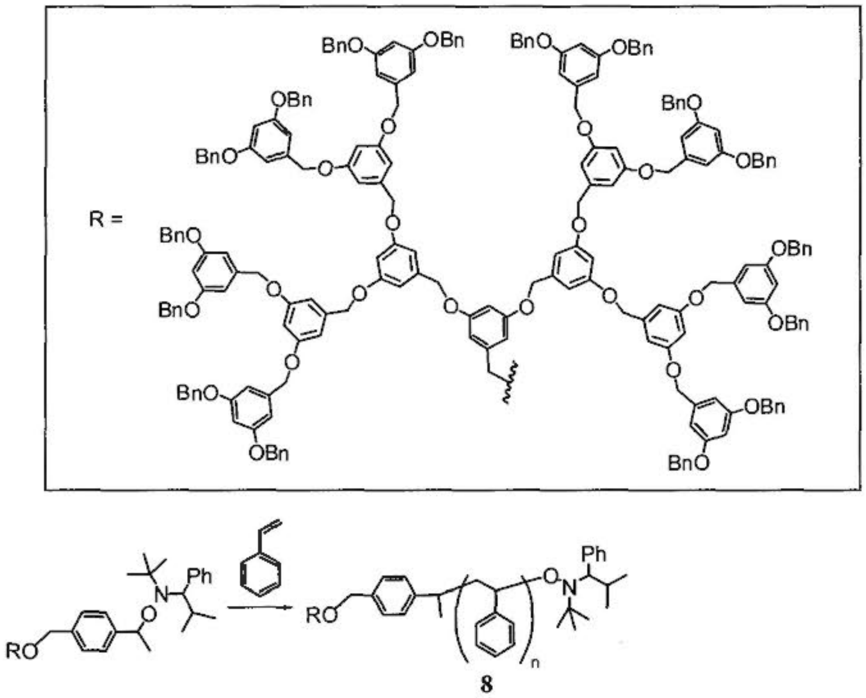

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Figure 7. *Synthesis of dendrimer-linear polymer hybrid 8 by living radical polymerization.*

Apart from the conformational change, dendrimer-linear polymer hybrids also exhibit aggregation property that was first reported by Meijer.[24] A series of dendrimer-linear polymer hybrids **9** with different generations (G1–G5) were synthesized and their aggregation properties were investigated (Figure 8). Surprisingly, these hybrids showed generation-dependent aggregation. It formed vesicular structures for G3 **9a**, micellar rods for G4 **9b** and spherical micelles for G5 **9c** while the G1 and G2 macromolecules showed inverted micellar behavior.

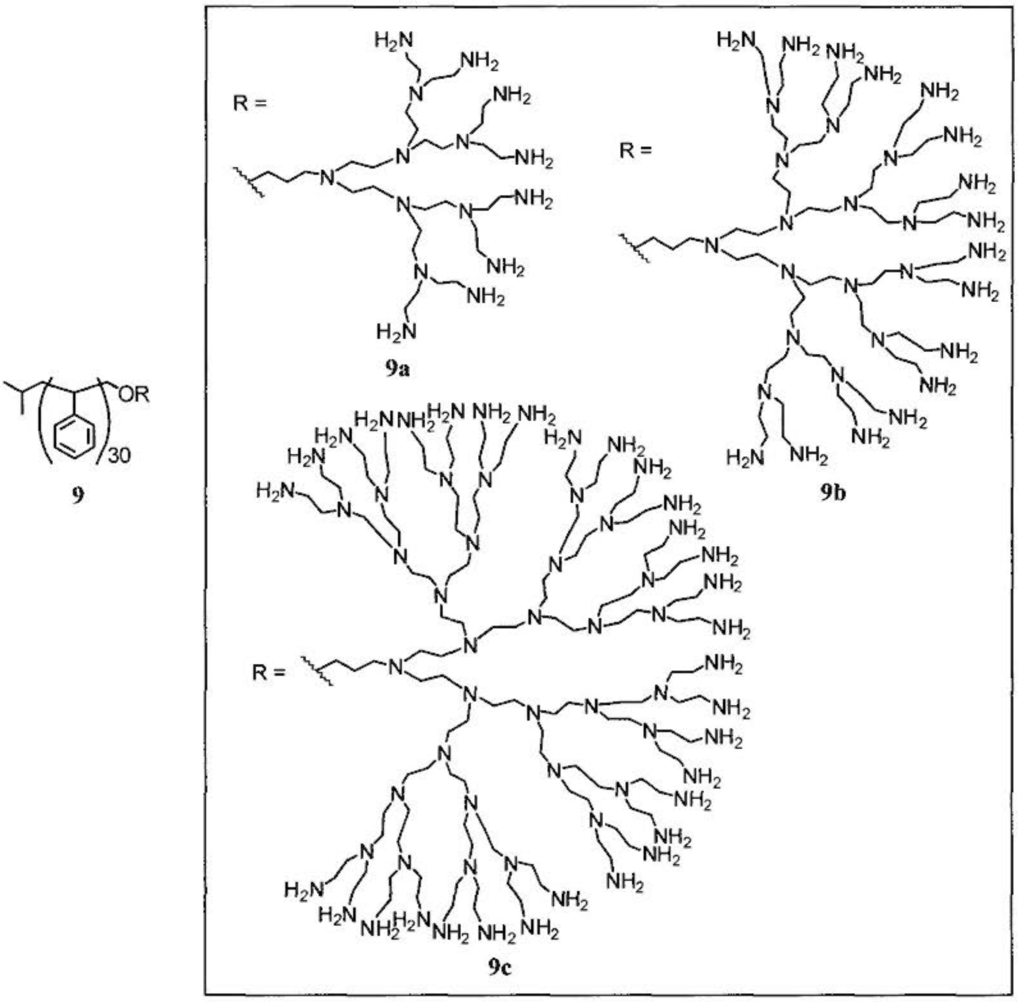

Figure 8. *Meijer's dendrimer-polymer hybrids 9 that exhibit generation-dependent aggregation property.*

Similar property was also reported by Wiesner and coworkers (Figure 9).[25] They found that the G1 hybrid **10a** aggregated to form cylindrical architectures while micellar structures were found for the G2 hybrid **10b**. This result was attributed to the different shapes of the hybrids (**10a** and **10b**) as they are bearing dendrons of different sizes.

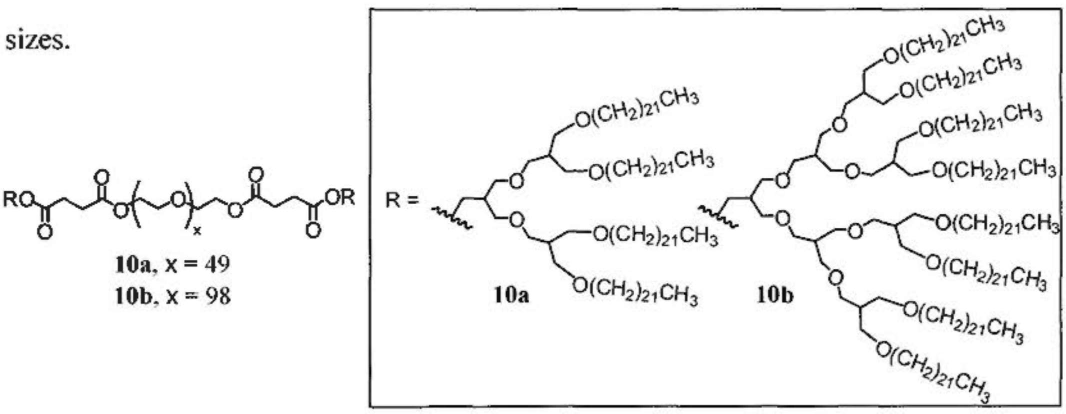

Figure 9. *Generation-dependent aggregation of hybrid 10 was reported by Wiesner.*

There were many other examples about the synthesis and properties of dendrimer-linear polymer hybrids.[26] Recently, Hawker and coworkers[27] prepared a new class of cross-linked star polymer by linking dendrimer-linear polymer hybrid **11** (G1–G5) with different polymer chain lengths together by atom transfer radical polymerization (ATRP) (Figure 10). The effects of the dendron size and the polystyrene chain length on the degree of polymerization were studied. It was found that smaller dendrons or shorter polymer chains could increase the polymerization efficiency and generate star polymers with more "arms".

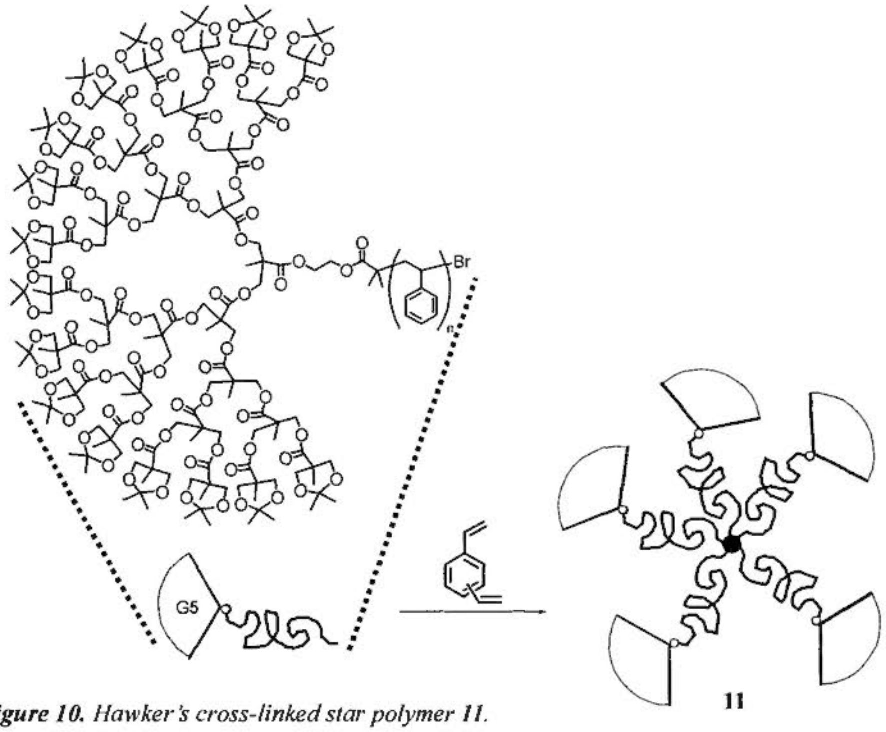

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Figure 10. *Hawker's cross-linked star polymer 11.*

The above are some dendrimer-linear polymer hybrids in which the polymer chains are attached to the focal point of dendrons. The example of which the polymer chains were attached to the periphery of dendrimers was reported by Fréchet (Figure 11).[17e, 28] For this type of dendrimer-linear polymer hybrids, their solubilities could be tuned by attaching PEG polymer chains of different lengths to periphery of the dendrimer. This increases the scope of applications to the biological system.[29] Several biologically active tetracycline units were covalently linked to the dendrimer and the hybrid **12** was water soluble and its *in vitro* and *in vivo* biological properties were also studied.

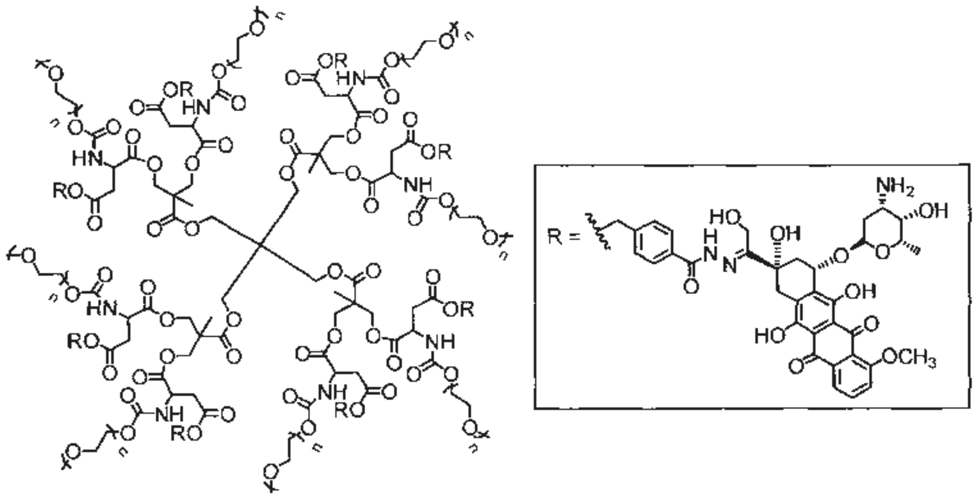

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Figure 11. *Fréchet's dendrimer-linear polymer hybrid 12.*

2.3. Dendron-dendrimer connectivity

Dendrons and dendrimers can be connected together deliberately to form higher order architectures and this type of connection is also called outer-sphere–inner-sphere connectivity.[19] It is constructed by linking the inner focal points of the dendrons to the outer surfaces of the dendrimers.

Newkome and coworkers[30] connected one dendrimer molecule **13** with four dendron molecules **14** by the metal-ligand interactions between ruthenium ions and terpyridine units (Figure 12). The hybrid **15** was called "dendritic methane" and its electrochemical properties were studied by cyclic voltammetry (CV).

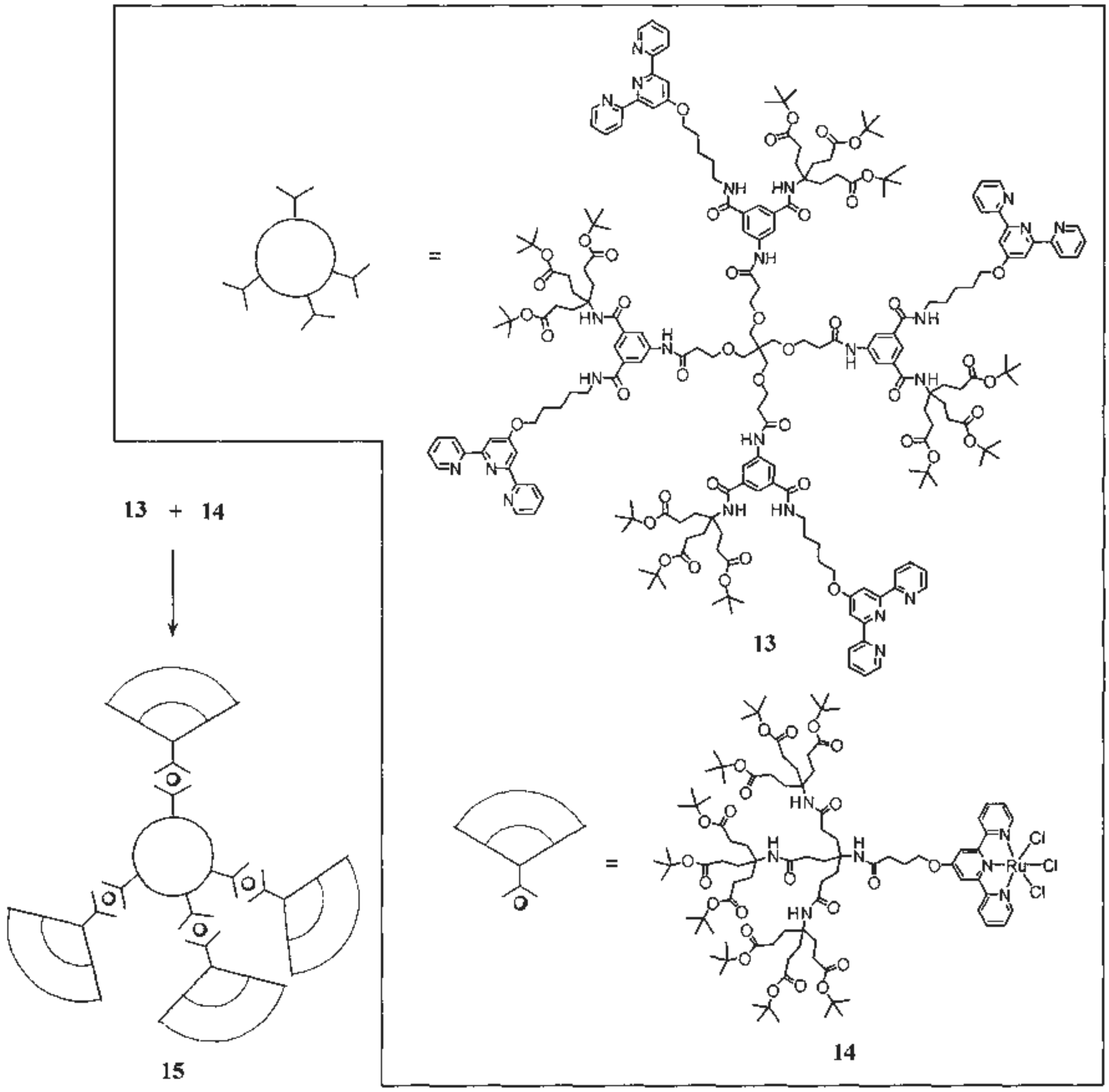


Figure 12. Newkome's dendron-dendrimer connectivity 15.

Another example was reported by Reinhoudt and coworkers (Figure 13).[31] The connectivity was created by mixing three equivalent of **16** with one equivalent of **17** through the metal-ligand interaction between palladium (II) and nitrile groups. This resulted in a layer-block dendritic structure **18** that consisted of many SCS Pd ^{II} pincer units and the structure was characterized by NMR spectroscopy and MALDI-TOF mass spectrometry.

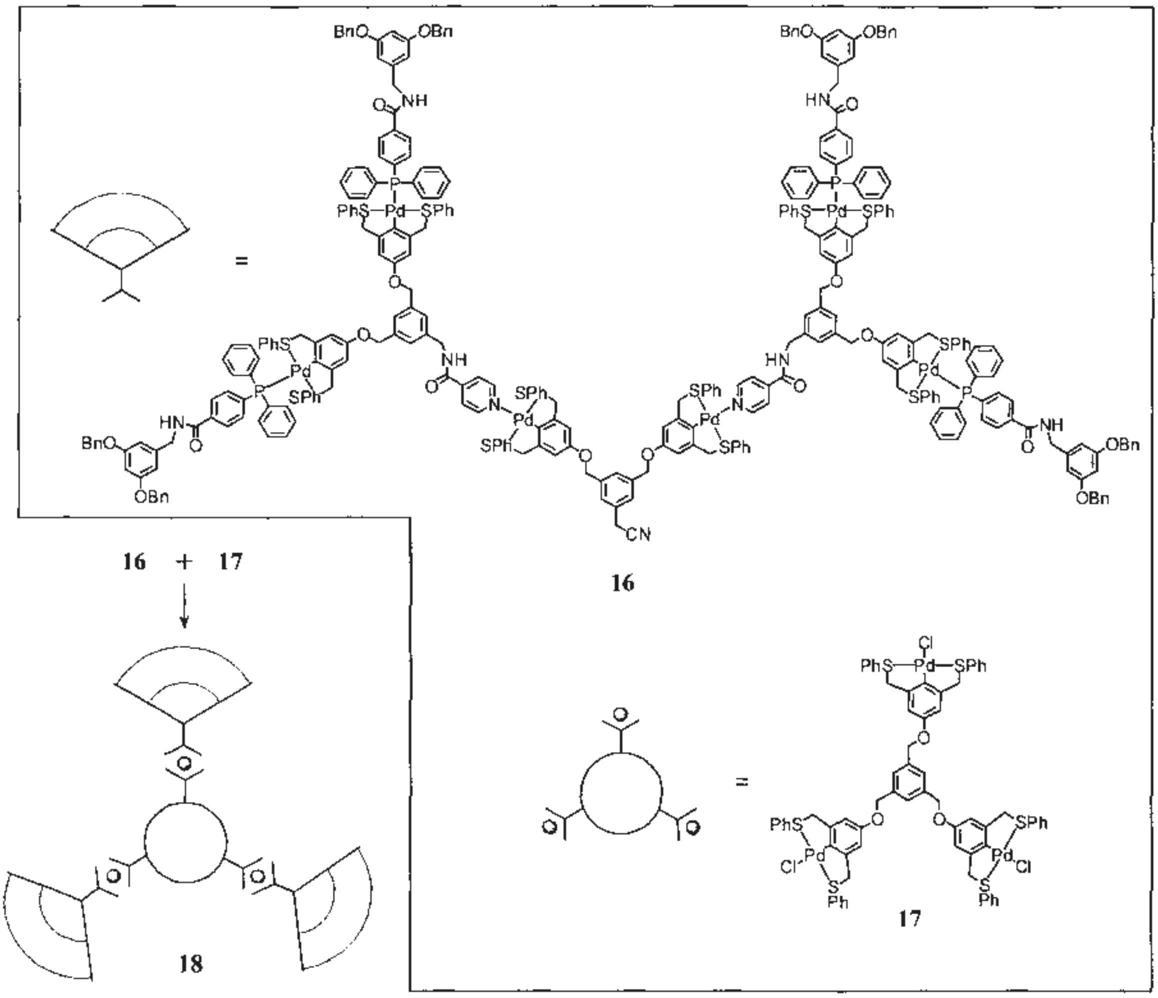


Figure 13. Reinhoudt's dendron-dendrimer connectivity 18.

2.4. Dendron-dendron connectivity

This is also known as inner-sphere–inner-sphere connection.[19] Dendrons are connected together by linking their inner focal points to form ordered macromolecular structures. Generally, dendrons can either be connected together "at a point" or "along a rod" to create two different architectures. In the former case, "dendrimer-like" structures (Figure 14a) would be constructed and polymeric structures (Figure 14b) would be obtained in the latter case. The latter type of polymeric structures is also called "dendronized polymers".

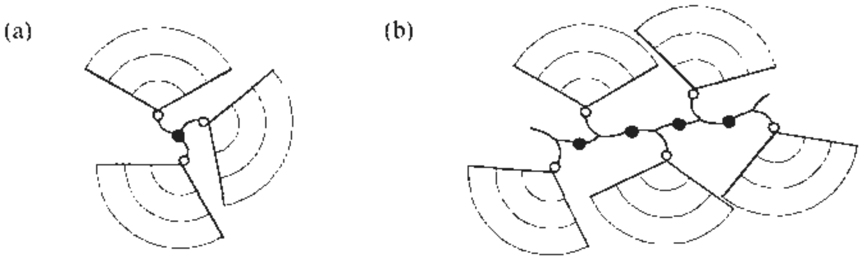


Figure 14. Connecting dendrons in different manners would form (a) "dendrimer-like" structures or (b) dendronized polymers.

2.4.1. "Dendrimer-like" structures constructed by connecting dendrons "at a point"

The earliest example of this architecture was reported by Newkome and coworkers.[32] They prepared two different dendrons and both of them bear terpyridine units at the focal points. In the presence of ruthenium ions, the dendrons were connected together by metal-ligand interactions to form the "dendrimer-like" structure **19** (Figure 15).

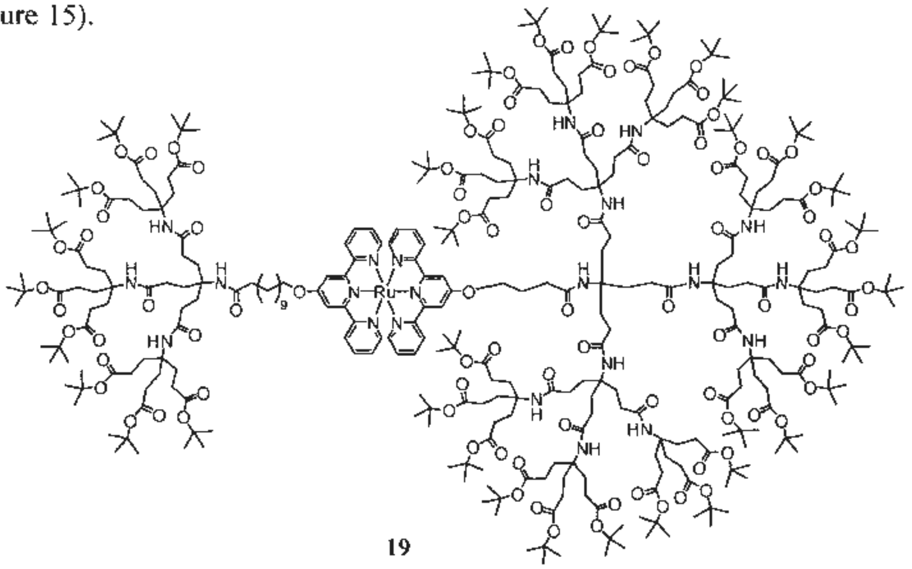
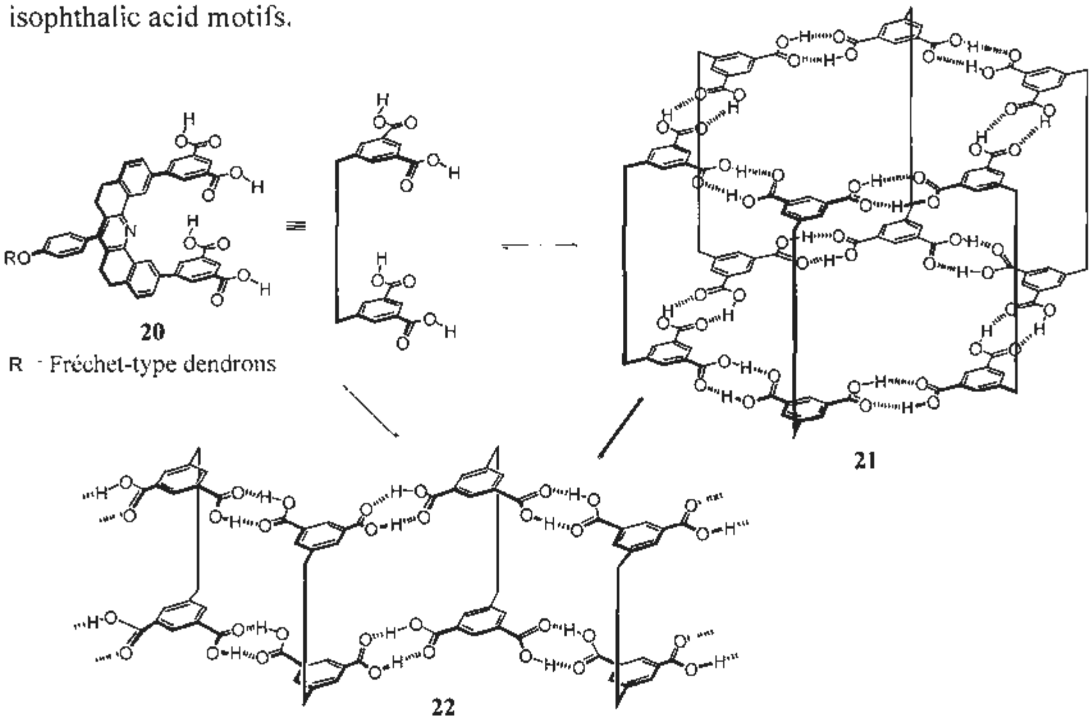

19

Figure 15. Newkome's "dendrimer-like" structure 19.

Later Zimmerman and coworkers[33] have synthesized a macromonomer **20** bearing Fréchet-type dendrons and two isophthalic acid moieties. This dendronized macromonomer formed hydrogen bonds with each other and resulted in the formation of cyclic hexameric architecture **21** and/or linear dendronized polymers **22** (Figure 16). Compound **21** was the major product due to the well-preorganization of the isophthalic acid motifs.


20

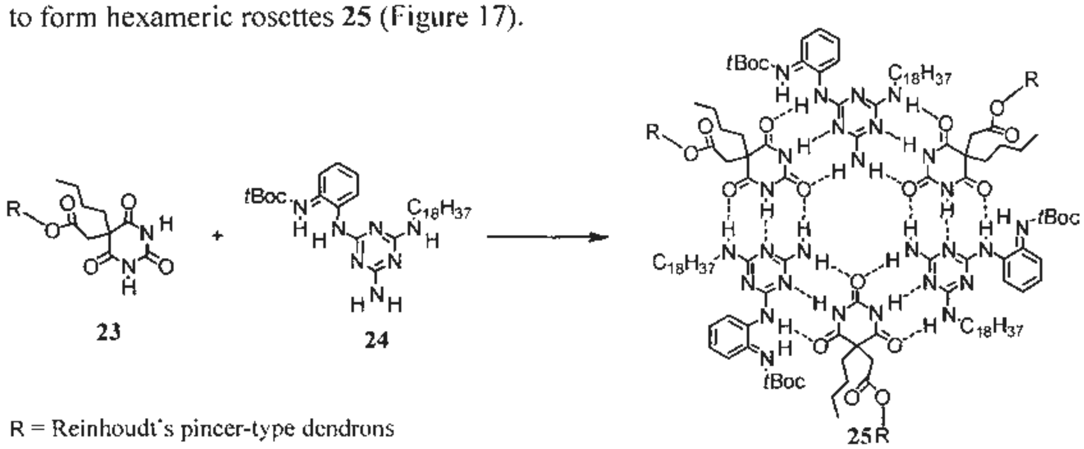
R = Fréchet-type dendrons

21

22

Figure 16. *Zimmerman's dedronized cyclic hexamer 21.*

Another architecture constructed by hydrogen bonds was reported by Reinhoudt and coworkers.[34] They prepared pincer-type dendrons with barbituric acid units **23**. In the presence of melamine **24**, the dendrons bound with it through hydrogen bonding to form hexameric rosettes **25** (Figure 17).


23

24

R = Reinhoudt's pincer-type dendrons

25

Figure 17. *Reinhoudt's hexameric rosettes 25.*

Stoddart and coworkers[35] constructed "dendrimer like" architectures by using host-guest complexation chemistry. They have prepared two types of dendrons with different focal points—crown ether **26** and dialkyl ammonium ion **27**. These two dendrons self-assembled in dichloromethane to form the dendrimer-like structure **28** (Figure 18). The rotaxane system was also used for linking dendrons together by Gibson and coworkers.[36]

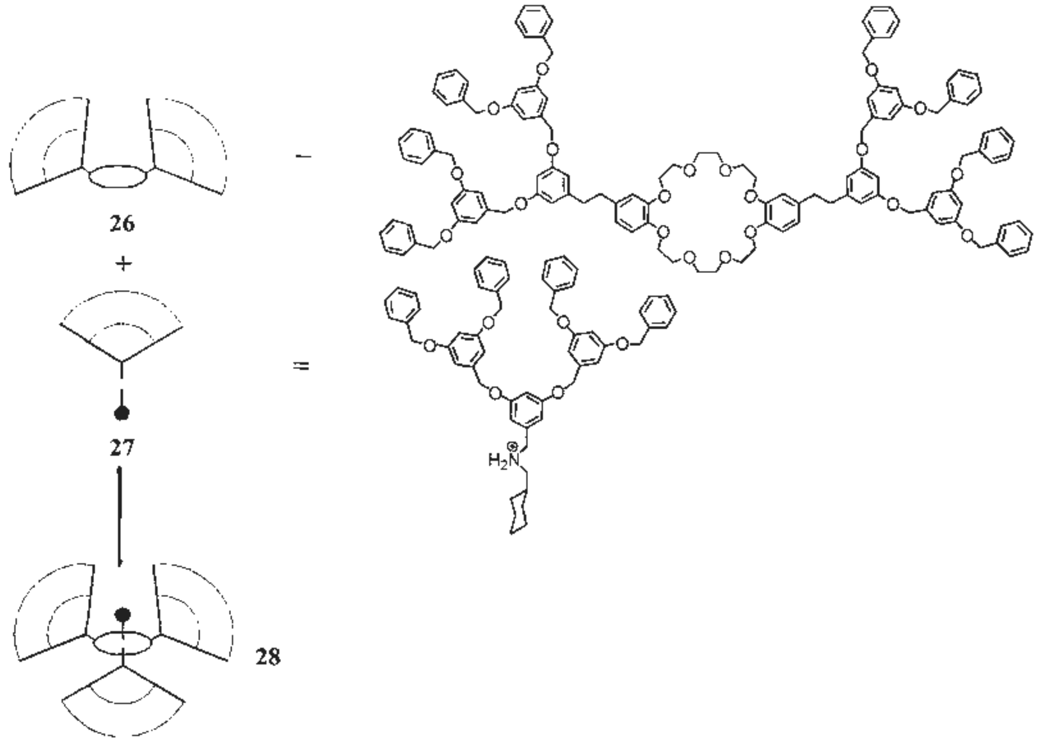


Figure 18. *Stoddart's "dendrime-like" architecture 28.*

Dendronized cyclic dimer,[37a] trimer[37b] and hexamer[37a] were prepared by Stang and coworkers by using metal-ligand interactions. They synthesized dendrons **29** with two pyridine units. In the presence of a platinum complex **30**, dendronized cyclic trimer **31** was formed through the interactions between platinum centers and pyridine units (Figure 19). By changing the platinum complex of different geometry, dendronized dimer and hexamer could also be formed.

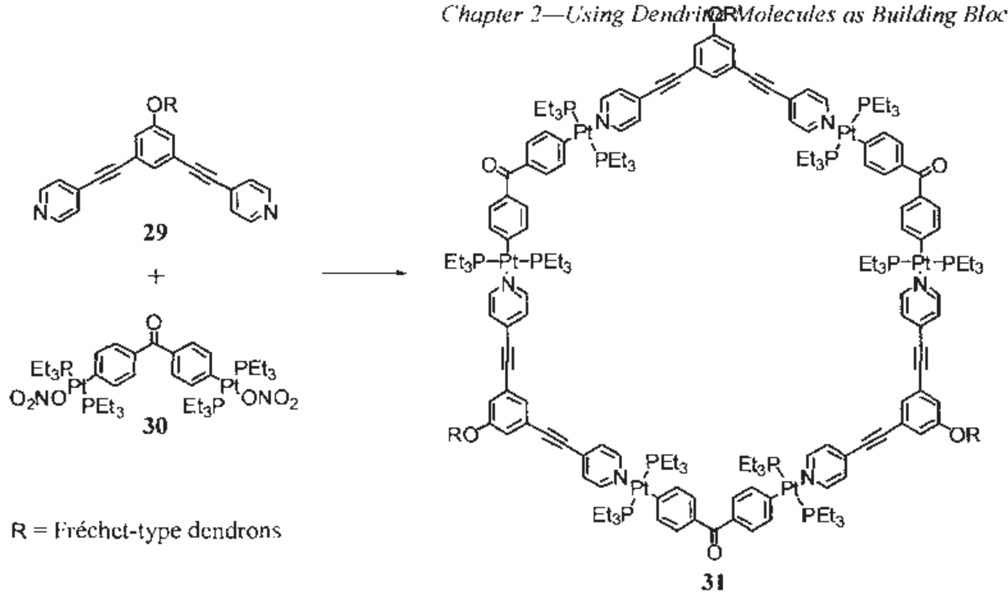


Figure 19. Stang's dendronized trimer 31 constructed by metal-ligand interactions.

2.4.2. Dendronized Polymers

In the past ten years, dendronized polymers[38] have played an important role in both polymer chemistry and dendrimer chemistry since they have unique properties that cannot be found in traditional polymers. First, the polymer backbone can be strengthened in the presence of sterically hindered dendron side chains (Figure 20). The conformation of polymer chains would change from a random coil (Figure 20a) to a rod-like structures (Figure 20d) with increasing dendron generation. Second, the polymer backbone can be shielded by the surrounding dendrons. For example, Aida

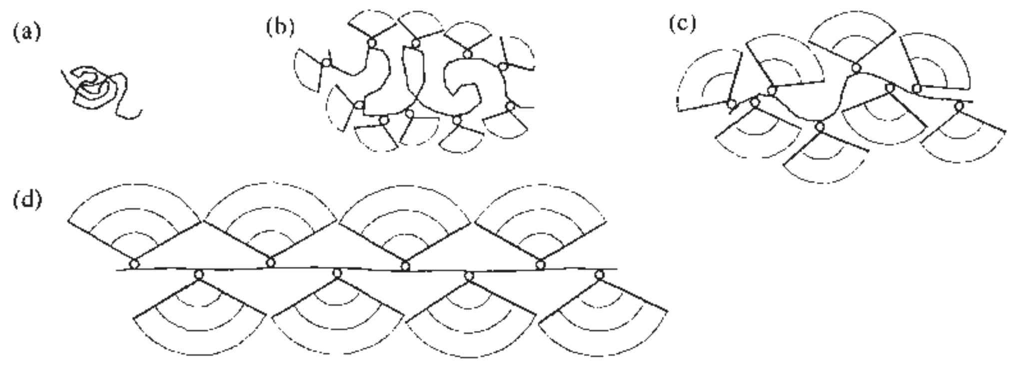


Figure 20. The conformation of dendronized polymers bearing (a) no dendrons, (b) G1 dendrons, (c) G2 dendrons and (d) G3 dendrons.

and coworkers[39] showed that dendronized poly(phenyleneethynylene) with G3 dendron side chain were discrete conjugated wires. Third, dendrons act as solubilizing groups to make the polymer chains dissolve in solvents, in which the polymer backbone is not soluble originally. Due to these special properties of dendronized polymers, they have applications in various areas such as optoelectronics,[40] surface patterning[41] and bioscience.[42]

There are three methods available for the preparation of dendronized polymers—graft-to, graft from and macromonmer approaches (Figure 21).

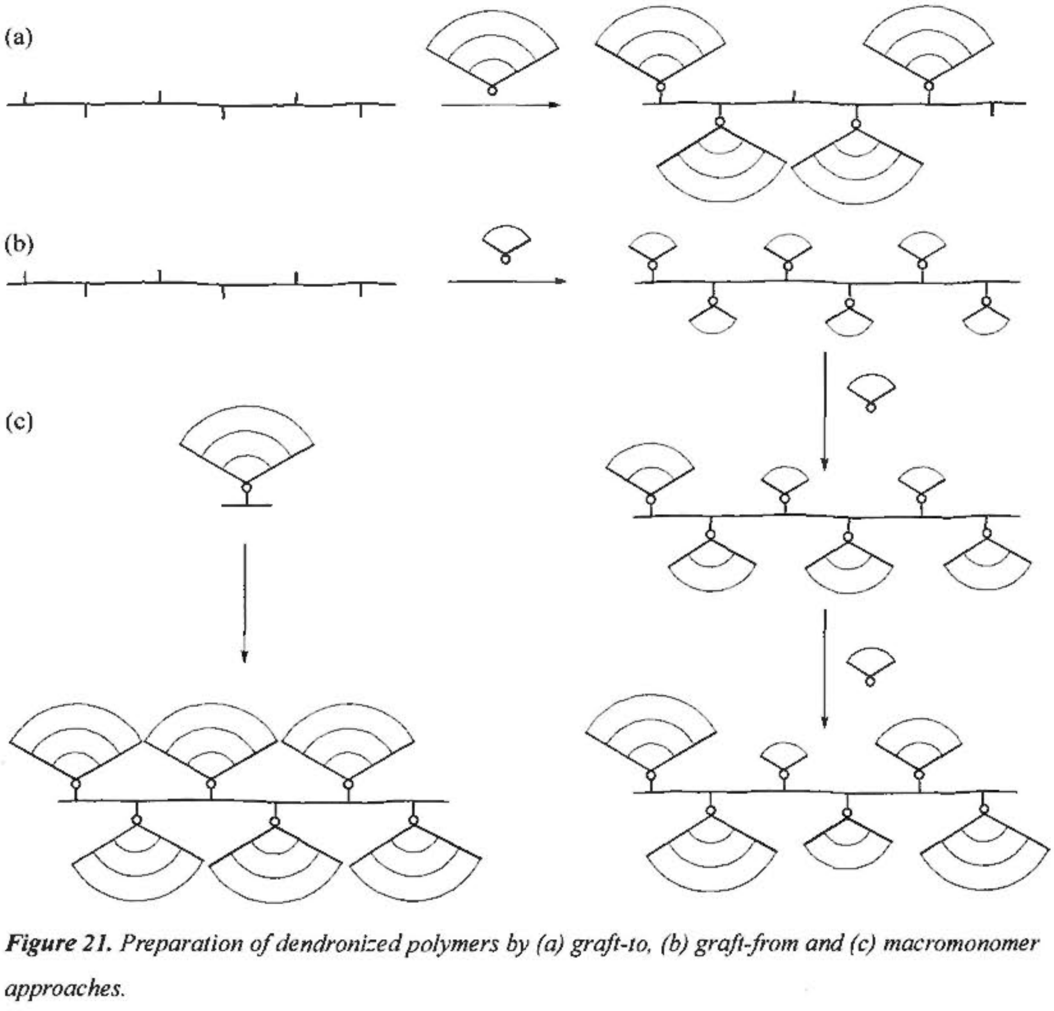


Figure 21. *Preparation of dendronized polymers by (a) graft-to, (b) graft-from and (c) macromonomer approaches.*

2.4.2.1. Graft-to approach

This is also known as "convergent growth". According to this approach, preformed dendrons of desired generation are coupled to the preformed polymer (Figure 21a). This strategy is more efficient than graft-from approach (See Section 2.4.2.2) but the post-polymerization reaction between preformed dendrons and the polymer may not be complete. Since the original polymer chain is a random coil, some of the reactive sites along the backbone may be buried and dendrons cannot be "grafted". Unfortunately, it is impossible to remove those polymer chains with such structural defects.

One of the earliest examples of dendronized polymers prepared by this approach was reported by Percec and coworkers.[43] The oligoether dendrons with an olefin unit at the focal points **32** were grafted to a poly(hydrogen methylsiloxane) chain **33** by the hydrosilylation reaction to form the dendronized polymer **34** (Figure 22). The resulting polymer has liquid crystalline property and characterized by GPC and optical polarized microscopy.

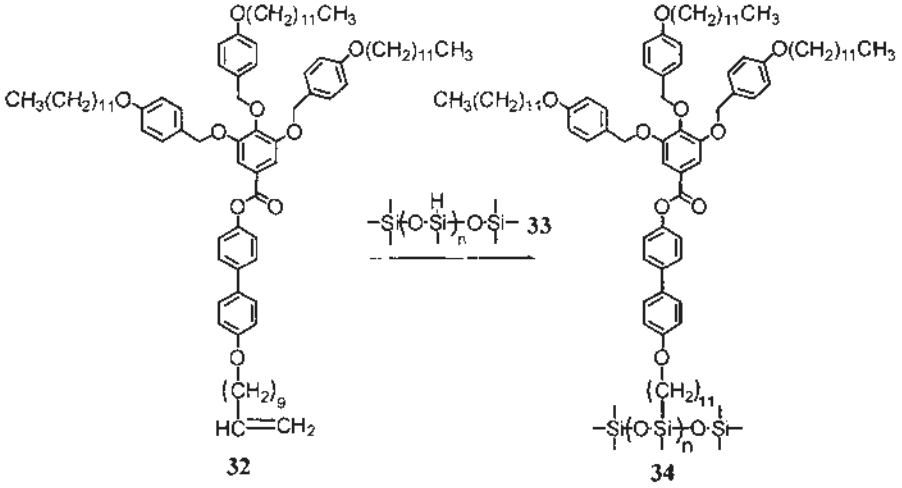


Figure 22. Percec's dendronized polymers 34 with liquid crystalline property.

Later Schlüter and coworkers[44] synthesized dendronized polymer **36** by reacting polymer **35** with Fréchet-type dendrons bearing an isocyanate unit at the focal point.

They also grafted dendrons (G1–G3) to a preformed polymer **37** to form a dendronized polymer **38** by Williamson ether synthesis (Figure 23). The percentage coverages of both G1 and G2 dendrons were 100%, but the percentage sharply decreased to 70% in the case of G3 dendrons due to its bulkiness.

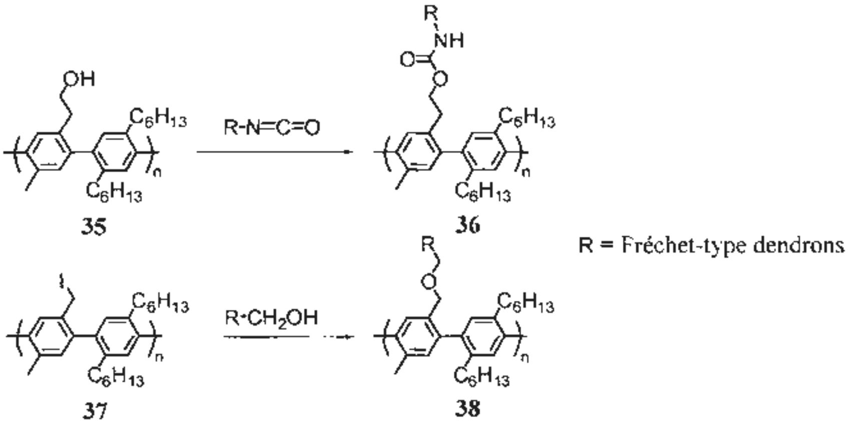


Figure 23. Schlüter's polymers 36 and 38 prepared by graft-to approach.

In order to graft high-generation dendrons to the preformed polymer, reactions with high efficiency must be applied. "Click reaction"[45] is the most suitable reaction. Fréchet and Hawker[46] made good use of Cu(I)-catalyzed Huisgen [2 + 3] dipolar cycloaddition reaction by coupling azide-containing dendrons **39** with alkyne-containing polymer **40** to generate the dendronized polymer **41** (Figure 24). In this case, the percentage coverage of G1 and G2 dendrons were quantitative, and it was >98% for G3 dendrons. Furthermore the rigid morphology of G3 dendronized polymers was observed by AFM.

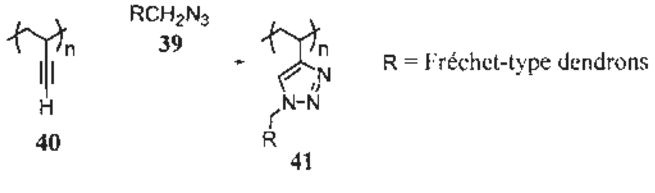


Figure 24. Fréchet's polymers 41 prepared by graft-to approach.

Apart from the above examples, there are other examples reported by Roy,[47] Ronda[48] and Bilibin.[49] Leung et al.[50] also reported that a reversible graft-to procedure of dendrons through acid–base reactions.

2.4.2.2. Graft-from approach

This strategy is also called the "divergent route". G1 dendrons are firstly attached to the preformed polymer, and then higher generations are obtained by successive attachment of G1 dendrons (Figure 21b). For preparing the high-generation dendronized polymer (e.g. G3), in contrast to graft-to route, the post-polymerization reaction is more efficient because the smaller G1 dendrons are grafted to the preformed polymer rather than large G3 dendrons. Unfortunately, G1 dendrons may not be attached to the polymer completely during the generation growth. This leads to the structural defects. Hence, a G3 dendronized polymer may contain small amount of G1 or G2 dendrons. In order to minimize this problem, very efficient coupling methods are needed.

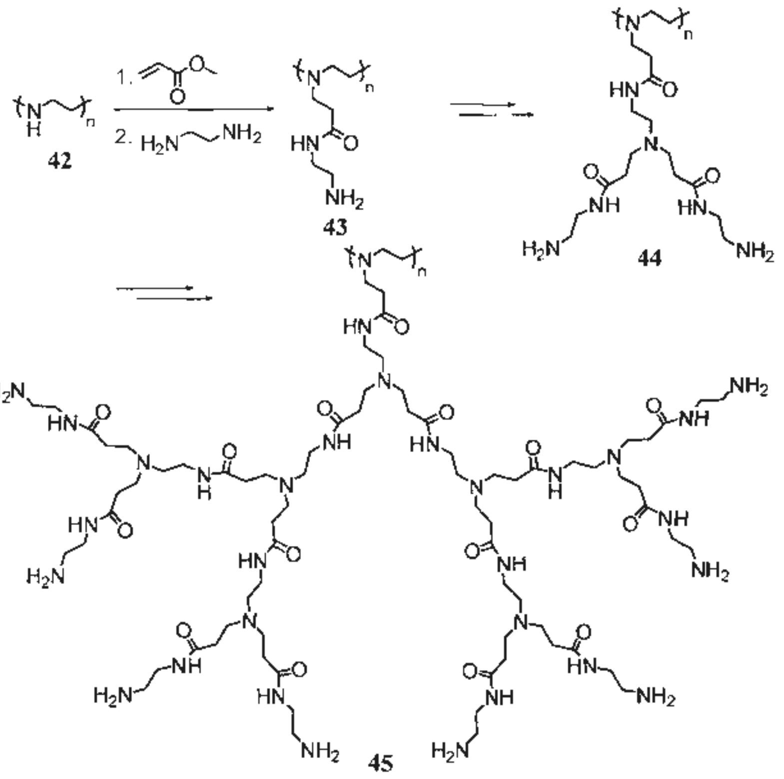


Figure 25. *Tomalia's dendronized polymers 43–45 prepared by graft-from route.*

Tomalia and coworkers[1a, 51] showed the first example on the graft-from approach (Figure 25). They used a linear poly(ethylene imine) **42** as the preformed polymer. After adding methyl acrylate and ethylene diamine, the G0 dendronized polymer **43** was afforded. Through the same reactions, the G1 **44** and G2 dendronized polymers **45** were obtained. Although the degree of coverage was not reported, the rod-shaped features of the resulting polymer were observed by TEM.

Later Kim and coworkers[52] functionalized poly(siloxane)s **46** by catalytic hydrosilylation and alkenylation to form G1 dendronized polymer **47**. After performing the same reaction sequence, G2 polymer **48** was also prepared (Figure 26). The polymers were characterized by NMR and GPC.

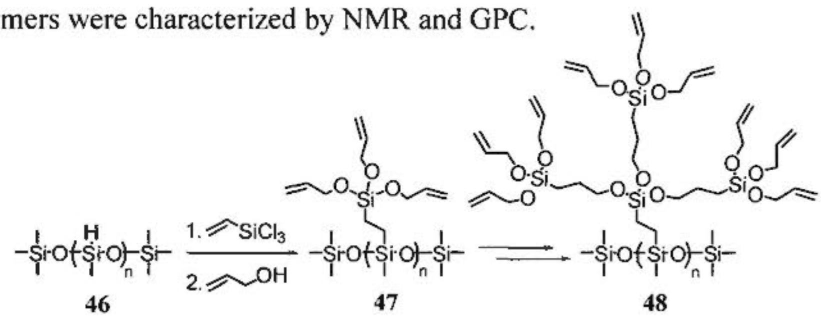


Figure 26. Kim's dendronized poly(siloxane) ***47*** *and* ***48****.*

Another example of graft-from route was reported by Fréchet and coworkers.[53] Poly(4-hydroxystyrene) **49** was firstly esterified to obtain the protected G1 polymer **50** (Figure 27). Then catalytic hydrogenolysis and esterification were performed again to afford the G2 polymer **51**. Similarly, G3 **52** and G4 (not shown in Figure 27) could

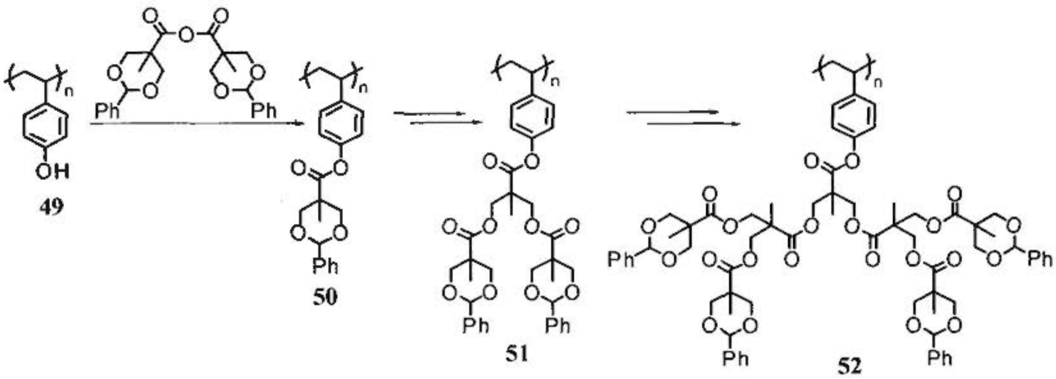


Figure 27. Fréchet's dendronized poly(4-hydroxystyrene) ***50–52****.*

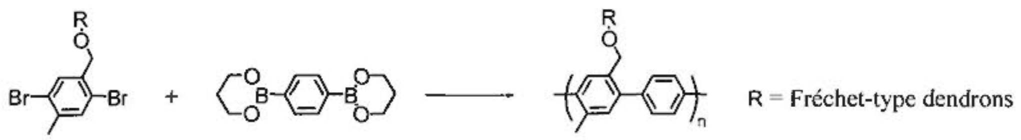
be easily synthesized. By using NMR spectroscopy, MALDI-TOF mass spectrometry and GPC, the authors claimed that the "grafting reactions" were quantitative up to the G3 polymer. The coupling reaction might not proceed to completion during the preparation of the G4 polymer.

Other literatures related to graft-from route were also reported by Hay[54] and Méry.[55] Fréchet and Hawker[56] also prepared dendronized polymers through graft-from route *via* "click chemistry".

2.4.2.3. Macromonomer approach

In this strategy, the desired-generation dendrons with polymerizable groups at their focal points are connected together by polymerization reactions (Figure 21c). Since this approach does not involve post-polymerization, the degree of dendron attachment must be quantitative. On the other hand, the chain length of the resulting polymer differs in various cases because no polymer chain is preformed. Due to the steric hindrance of high-generation dendrons and the flexibility of the polymerizable groups, oligomers and cyclic oligomers[57] may be formed respectively instead of the desired polymer.

Schlüter and coworkers[58] prepared dendronized macromonomer **53** and copolymerized with boronic acid **54** by Suzuki coupling reaction[59] to form desired polymer **55** (Figure 28). The difficulty of this method is to ensure equal stoichiometry of two monomers **53** and **54**, therefore weighing the exact amount of the smaller monomer **54** is very crucial. This method could be applied to a G4 dendronized polymer and the yield was quantitative. The degree of polymerization (DP_n) was 25 and the polydispersity index (PDI) was 5.0 by GPC measurement.



53 **54** **55**

Figure 28. *Schlüter's dendronized polymer 55 prepared by Suzuki coupling.*

Apart from Suzuki coupling,[60] there were other coupling reactions commonly used in this approach including addition of isocyanate,[61] acyl substitution,[62] Williamson ether synthesis[63] and Yamamoto coupling.[64]

Free radical polymerization was also reported by Schlüter and coworkers.[65] They firstly synthesized macromonomers with G1 and G2 Fréchet-type dendrons **56-G1** and **56-G2**. By radical polymerization, polymer **57-G1** was afforded (Figure 29a). The reaction failed in the case of the G2 polymer **57-G2** due to the sterical hindrance between the large G2 dendrons.

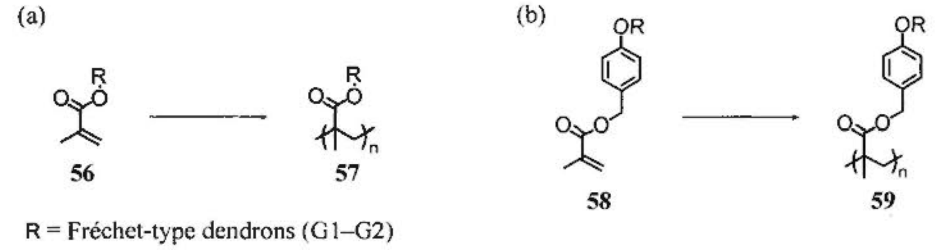


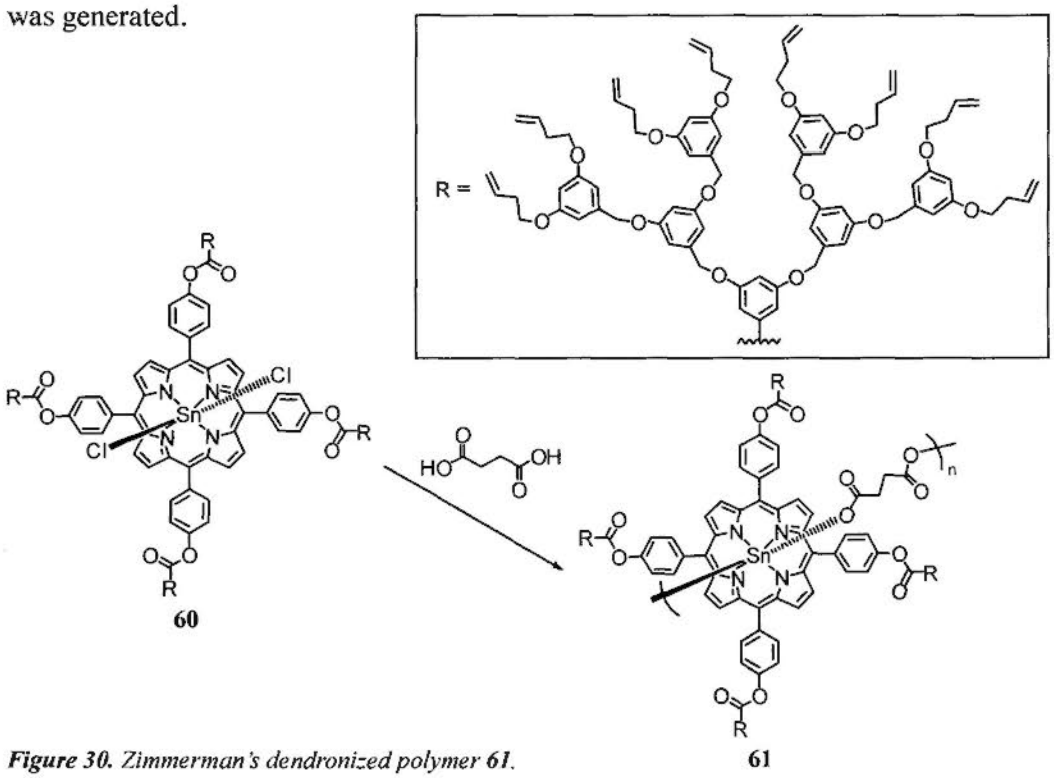
Figure 29. *Free radical polymerization of dendronized macromonomers.*

In a subsequent study, they tried to add a spacer between the dendron and the backbone in order to tackle this sterically crowded problem. Therefore compounds **58-G1** and **58-G2** were prepared and subjected to the polymerization reaction again (Figure 29b). Fortunately, the desired polymers **59-G1** and **59-G2** were formed and characterized by GPC with poly(styrene) standards. The DP_n value were 544 and 90 for G1 and G2 respectively, and the PDI value was 2.2 in both cases.

In addition to the above reports, similar examples had also been published by Schlüter,[66] Ritter,[67] Xi[68] and Chessa.[69]

Instead of dendrons, dendrimers could also be used as macromonomers for polymerization. It involved "dendrimer-dendrimer connectivity", and dendrimers were linked together by connection *via* the central cores. Therefore the resulting architectures formed are very different from poly(dendrimer)s (See Section 2.5) which are prepared from surface-group connection.

The first example was reported by Zimmerman and coworkers.[70] They first prepared a dendrimer with a tin-porphyrin central core **60** and then connect them together by adding a linker—butanedioic acid to obtain the dendronized polymer **61** (Figure 30). Then the outer surface of polymer **61** already containing many olefin functionalities was covalently linked by performing ring-closing metathesis reaction. After that, all the ester linkages were cleaved by hydrolysis and a hollow nanotube was generated.

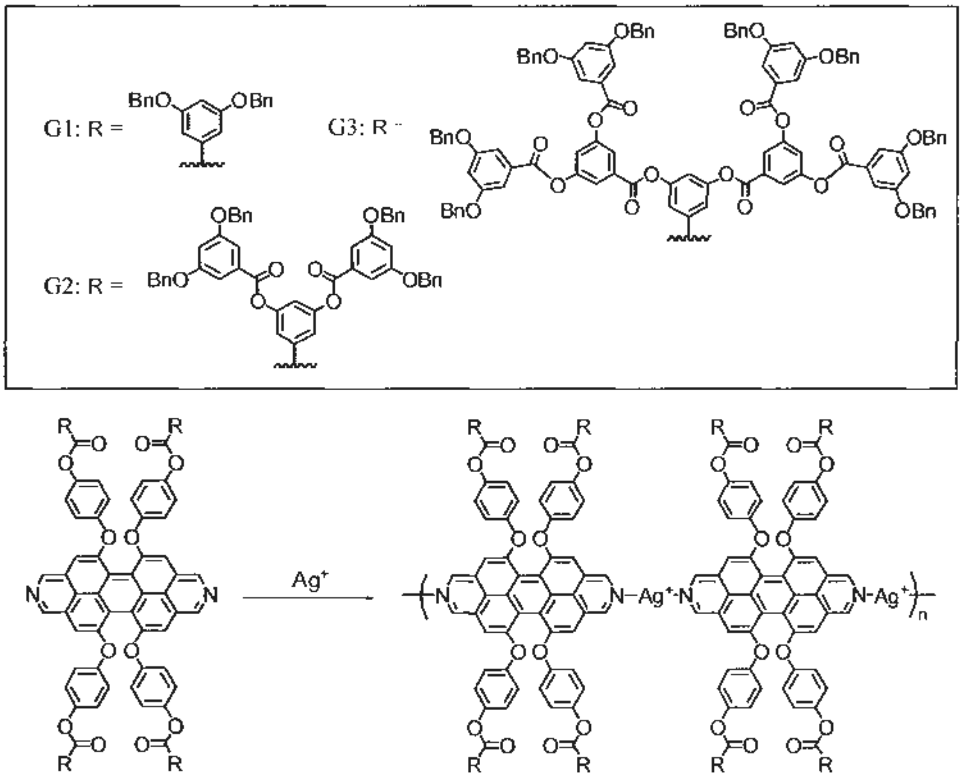


60

61

Figure 30. *Zimmerman's dendronized polymer 61.*

Würthner and coworkers[71] reported another example. Dendrimers (G1–G3) **62** were firstly synthesized and polymerized by adding silver ions. Polymers **63** were formed through metal-ligand interactions between dendrimer cores (Figure 31). By using ^{1}H DOSY NMR spectroscopy, it was found that the G3 polymer, in contrast to G1 and G2 polymers, could not be formed because the diffusion coefficients were almost the same for compound **63-G3** and its 1:1 stoichiometric mixture with Ag^+ ion. Owing to the steric hindrance of the dendritic wedges, G3 monomer could not undergo polymerization by complexation with Ag^+ ions. This explanation was supported by molecular modeling studies. In addition, AFM images also showed G1 and G2 polymers existed as rod-like structures.



62 **63**

Figure 31. *Würthner's dendronized polymer 63 mediated by metal-ligand interactions.*

2.5. Poly(dendrimer)s

It is also known as outer-sphere–outer-sphere connectivity.[19] Poly(dendrimer)s are constructed by linking specific surface-functionalized dendrimers together (Figure

32). In this case, the reactive sites (polymerizable groups) are located on the periphery of the monomer so that they are more accessible. On the contrary, the reactive sites are buried by sterically hindered dendrons in the case of dendronized polymers. As a consequence, poly(dendrimer)s may have a better polymerization result.

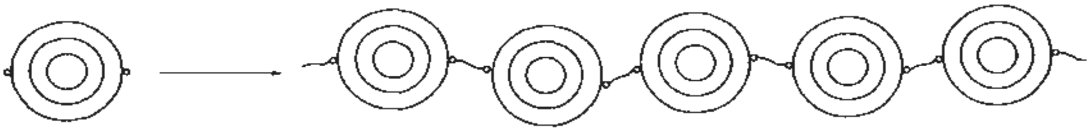
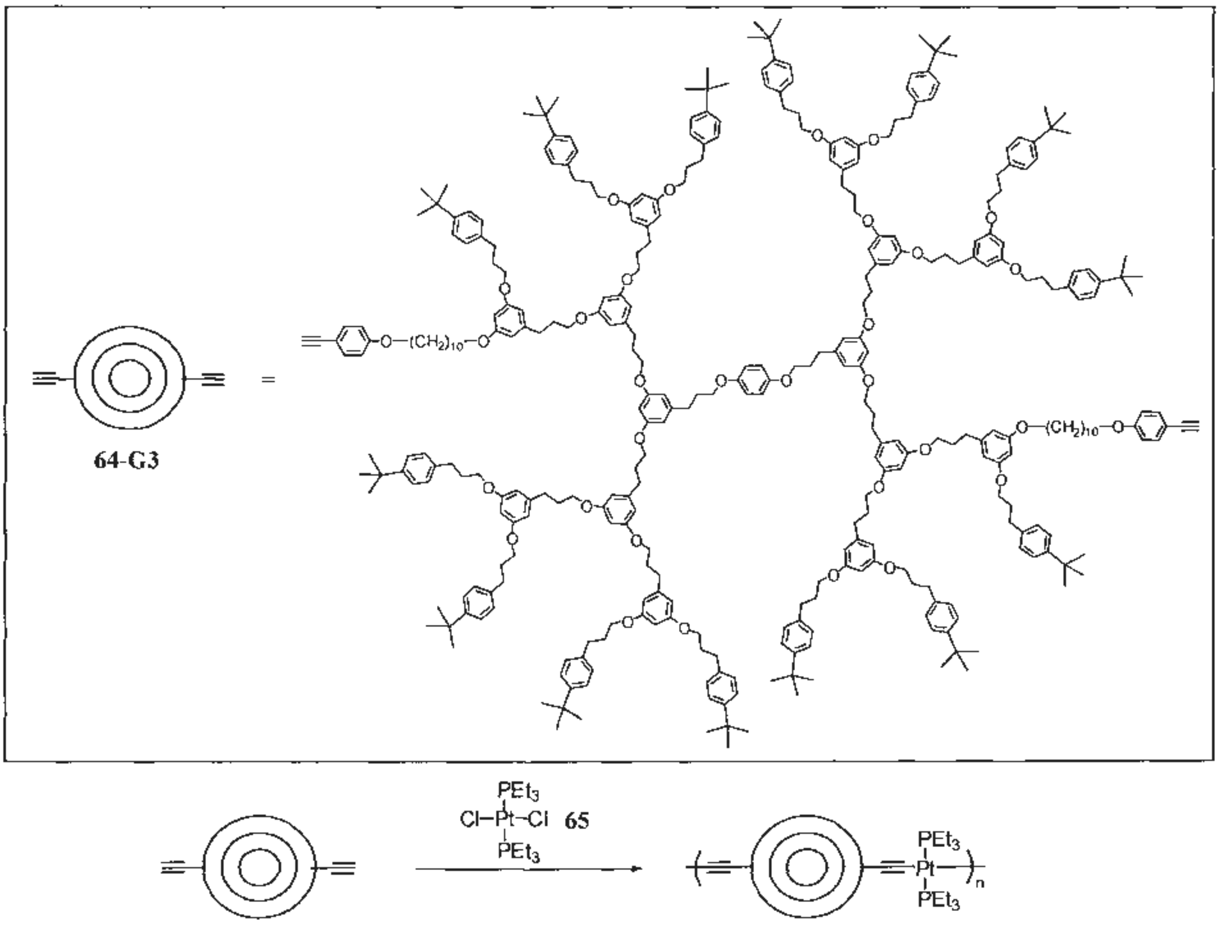


Figure 32. *Construction of a poly(dendrimer).*

Although the preparation of poly(dendrimer)s has this perceived advantage, synthetic investigations about them were scarce. The concept was firstly reported by Thayumanavan and coworkers.[72] It was disappointing to note that the polymerization was not put into practice.



64-G3

64-G3

66-G3

Figure 33. *Poly(dendrimer)s 66 prepared in our laboratory.*

The first real example using this outer-sphere–outer-shpere approach was reported by our group.[94] Dendrimers (G1–G3) **64** with two reactive acetylenes on the periphery were synthesized. Then they were copolymerized with *trans*-[$Pt(PEt_3)_2Cl_2$] **65** to form poly(dendrimer)s **66** or so-called "dendritic necklaces". (Figure 33). The resulting polymers were characterized by NMR spectroscopy, GPC, laser light scattering (LLS), and visualized by STM. From the LLS results, the DP values of G1, G2 and G3 poly(dendrimer)s were 880, 90 and 30 respectively.

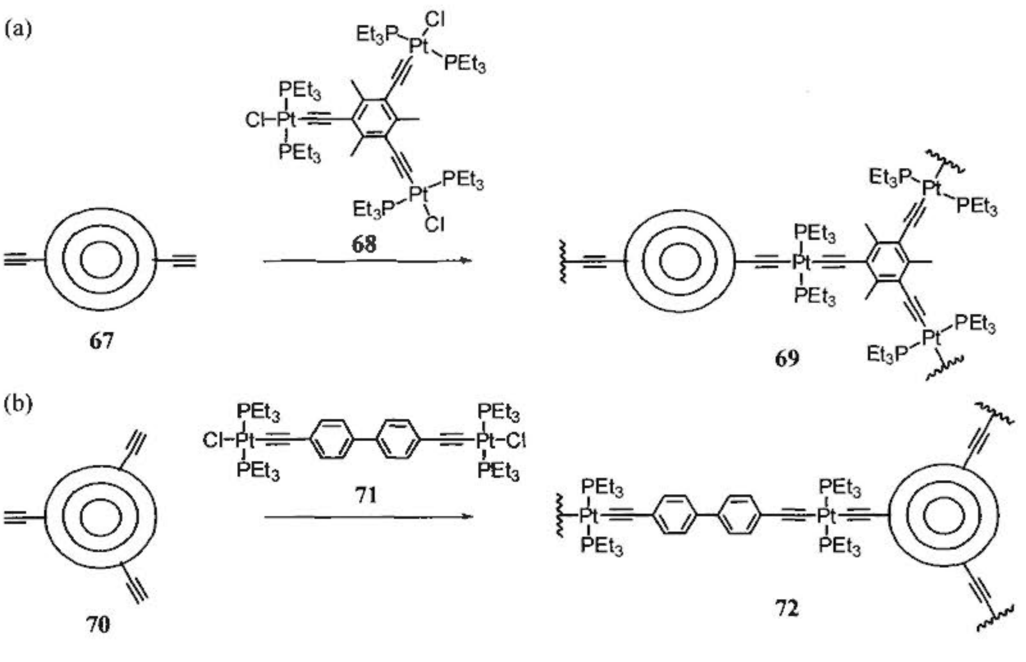


Figure 34. *Poly(dendrimer) networks **69** and **72** prepared by (a) a bi-functional dendrimer **67** and a tri-functional linker **68**; (b) a tri-functional dendrimer **70** and a bi-functional linker **71** respectively.*

Similar poly(dendrimer) networks were also prepared by our group.[74] One of the attempts was to link bi-functional dendrimers (G1–G3) **67** and a tri-functional linker **68** together to form a poly(dendrimer) network **69** (Figure 34a). In practice, linear poly(dendrimer)s instead of poly(dendrimer) networks were formed due to the close proximity of the three reactive platinum center on compound **68**. On the other hand, another type of insoluble poly(dendrimer) networks **72** were obtained by connecting a tri-functional dendrimer (G0–G2) **70** with a bi-functional linker **71** (Figure 34b). The different copolymerization behavior between these two protocols was explained in terms of steric crowding around the branching points.

The formation of dendronized polymers and poly(dendrimer)s provides a new route for constructing ordered nanoscopic polymeric materials of which the properties are different from traditional polymers. Many people pay attention on dendronized polymers, on the contrary, poly(dendrimer)s are seldom investigated. Moreover in order to study the properties of poly(dendrimer)s, high polymerization efficiencies must be ensured. Therefore it is necessary to conduct a research project on studying how the structural elements, dendrimer beads and linkers, would affect the polymerization efficiencies. This thesis aims to address these issue with the goal to develop efficient polymerization methods for such poly(dendrimer)s.

Chapter 3

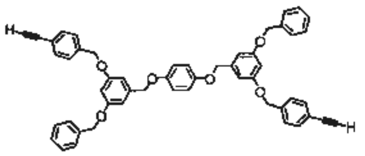
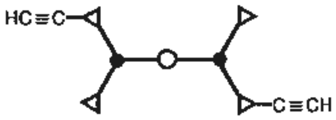
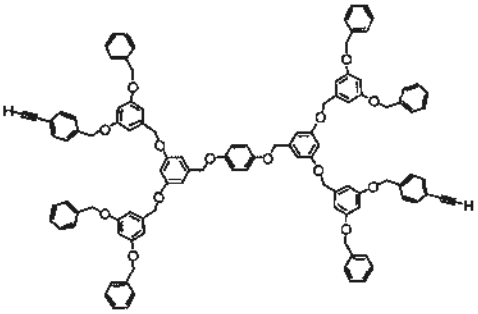
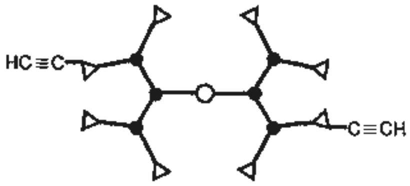
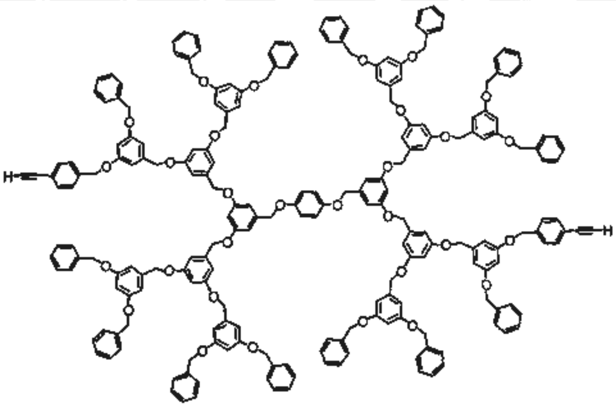
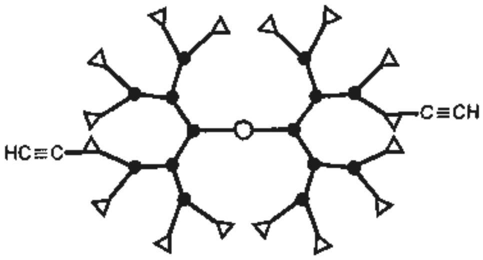
Objectives, Synthesis and Characterization of Poly(dendrimer)s

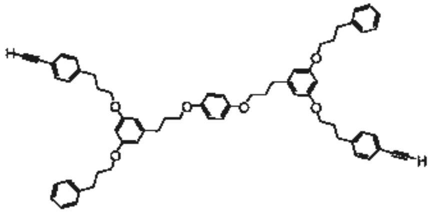
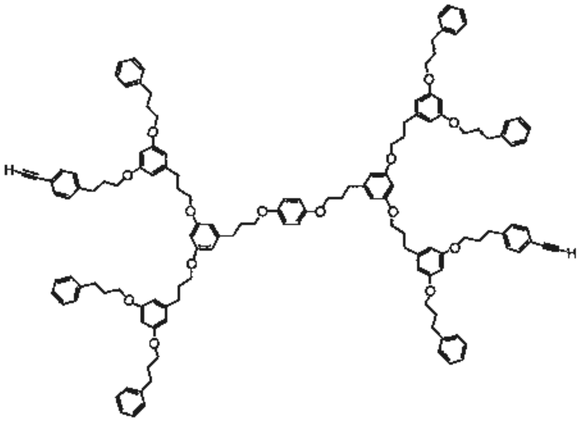
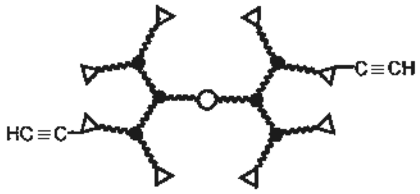
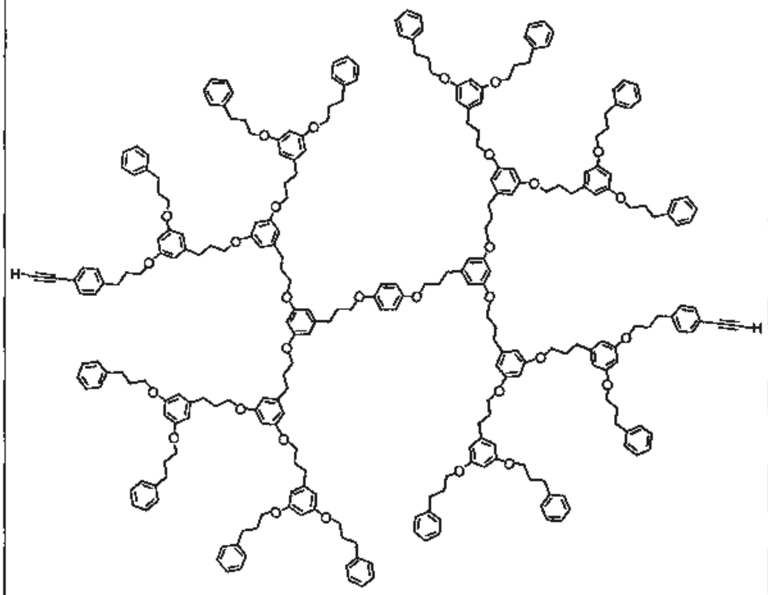
3.1. Objectives

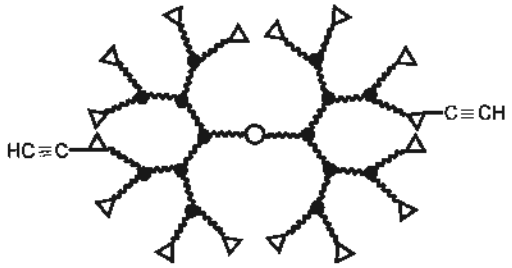
As mentioned in the last chapter (See Section 2.5), our research group has successfully synthesized and characterized a novel class of dendritic macromolecules—organoplatinum poly(dendrimer)s (G1–G3). This offers a new entry for constructing nanoscopic-sized materials with controlled architecture. Poly(dendrimer)s are constructed by surface bifunctional dendritic monomers—dendrimer beads and a platinum linker, therefore the author believe that not only the size and the structural flexibility of dendrimer beads, but also the length and geometry of the platinum linkers would have some influences on the polymerization outcome. By investigating these factors, one should be able to exert better control on the construction of poly(dendrimer)s. In the mean time, the effect of the platinum linkers will be discussed in the chapter 4. This chapter will focus on how the size and structural flexibility of dendrimer beads affect the polymerization.

3.2. Design of dendritic macromonomers

Two series of surface bifunctional dendrimer beads (S-Gn *vs* L-Gn, where n = 1–3) **73–78** with different structural flexibilities were firstly synthesized (Table 1). The differences in structural flexibility are controlled by constructing the dendrimers with different branching units, i.e. a two atom C–O branch for S-Gn (S denotes shorter linker) and a four atom C–C–C–O branch for L-Gn (L denotes longer linker).

S-Gn Seires		
HC≡C–[S-G1]–C≡CH **73**	**Structure**	
	Schematic **Diagram**	
HC≡C–[S-G2]–C≡CH **74**	**Structure**	
	Schematic **Diagram**	
HC≡C–[S-G3]–C≡CH **75**	**Structure**	
	Schematic **Diagram**	

L-Gn Seires		
$HC\equiv C$–[L-G1]–$C\equiv CH$ **76**	**Structure**	
	Schematic **Diagram**	
$HC\equiv C$–[L-G2]–$C\equiv CH$ **77**	**Structure**	
	Schematic **Diagram**	
$HC\equiv C$–[L-G3]–$C\equiv CH$ **78**	**Structure**	

	Schematic Diagram	

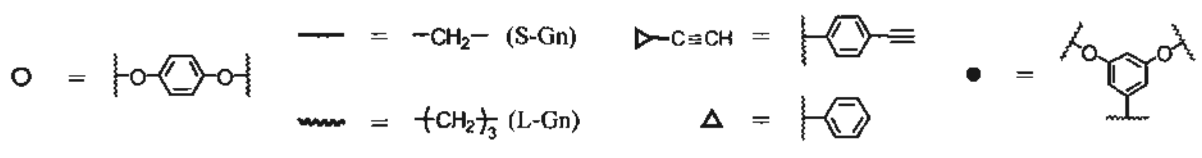


Table 1. Structures and schematic diagrams of dendritic macromonomers 73–78.

Due to the extra degree of rotation freedom of the 4 atom units, therefore the L-Gn series has a more flexible dendritic skeleton than the S-Gn series. In addition, the former series also possess a larger hydrodynamic radius as compared to the latter series of the same generation. All the dendrimer beads (dendritic macromonomers) contain two terminal alkyne groups (polymerizable groups) on the surface of the dendrimer for the copolymerization reaction with a platinum linker—*trans*-$[Pt(PEt_3)_2Cl_2]$ **65** (Figure 35).[75] In order to study the effect of structural flexibility on the polymerization efficiencies, the results obtained from dendrimer beads of the same generation among different series would be compared, i.e. **73** *vs* **76**, **74** *vs* **77** and **75** *vs* **78**. It should be noted that the purity of the dendritic beads is highly crucial to the success of the copolymerization. If there is a small amount of dendrimer bead that is mono-functionalized, it would become a chain stopper and decrease the degree of polymerization (DP) value. Theoretically, the presence of 1% of mono-functionalized dendrimer would make the maximum DP value become 200. On the other hand, if a small amount of tri-functionalized dendrimer bead is formed during the synthesis, poly(dendrimer) networks would be formed instead of poly(dendrimer)s. Unfortunately, the existence of 1% of mono-functional or

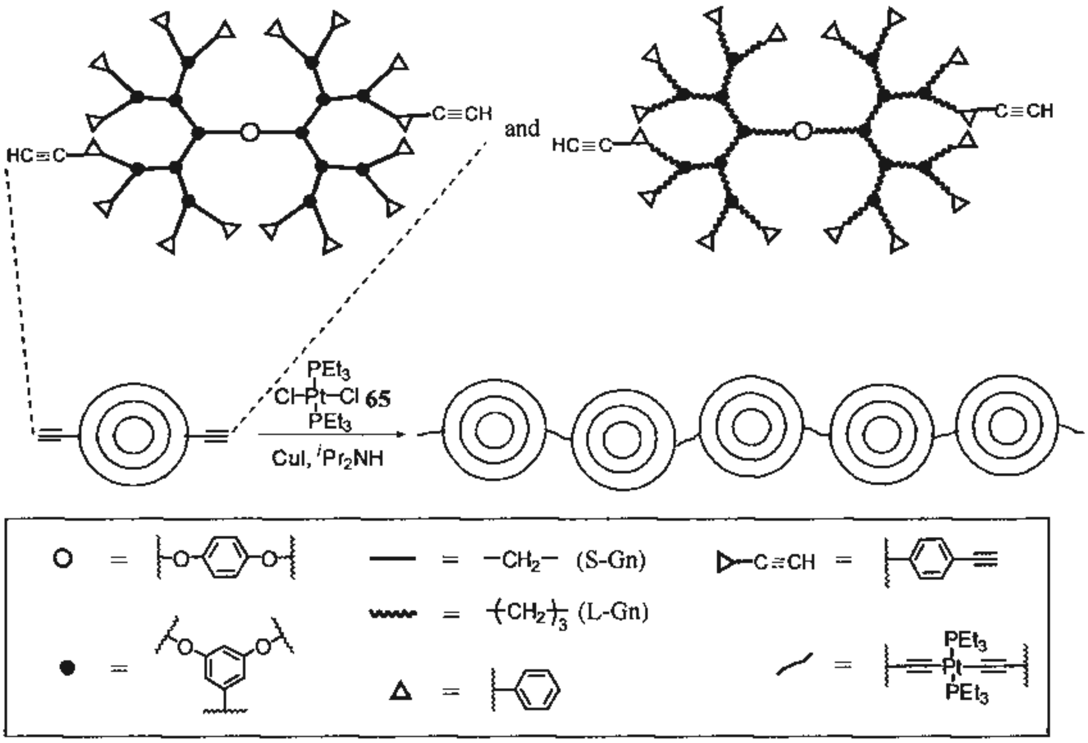


Figure 35. Schematic structure of the G3 dendritic beads (S-G3 and L-G3) and their copolymerizations with platinum linkers to form poly(dendrimer)s.

tri-functional impurities is not detectable by ^{1}H NMR spectroscopy and/or gel permeation chromatography (GPC) easily, therefore a well-designed synthetic route is needed to guarantee the production of the bi-functional macromonomers in extremely pure form. As a result, this makes the synthetic work become more challenging.

When comparing with the dendritic macromonomers used in our group before (compound **64-G3** in Figure 33 in chapter 2), two structural modifications are introduced in order to fulfill the objectives in this project. Firstly, the polymerizable acetylene surface groups were protruded by a 10-carbon-length spacer in our original work. Hence the polymerizable groups were stretched out from the dendritic surface with the aim to increase the polymerization efficiency. As this "long arm" system affected the polymerization result significantly, it was not employed in this project. On the contrary, the two terminal alkyne groups were directly anchored on the surface of the dendrimer in order to make sure that the polymerization result is only affected by the structural flexibility of the dendrimer bead itself. Secondly, the inert surface groups in this project are simply benzyl groups instead of the original *p-tert*-butylbenzyl groups. Although the *p-tert*-butylbenzyl groups could increase the solubility of dendrons and dendrimers, their steric hindrance lower the accessibility of the neighboring terminal alkyne groups during polymerizations. Unfortunately, the poor solubility of dendrons and dendrimers makes the purification procedures become more complicated and time-consuming, as will be discussed in Section 3.3.2.

3.3. Synthesis

Our initial goal was to prepare the target dendrimer beads with two terminal alkyne surface groups (Figure 35). As the chemical tolerance of the alkyne groups is very low, therefore they should be anchored to the dendrimer at a later stage of the synthetic pathway. This could be accomplished by a Sonogashira coupling[76] between TMSC≡CH with an aryl iodide surface functionalized dendrimer followed by removal of the TMS protective group (Figure 36). In the following sections, the synthesis of the S-Gn dendrimers will be discussed first, followed by that of the L-Gn dendrimers.

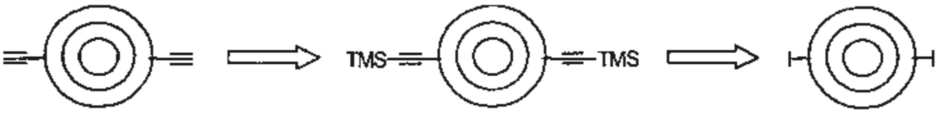


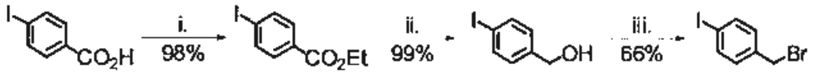
Figure 36. Retrosynthesis of dendrimer beads with two terminal alkyne surface groups.

3.3.1. Synthesis of HC≡C–[S-Gn]–C≡CH dendrimers

The synthesis of HC≡C–[S-Gn]–C≡CH dendrimers (n = 1–3) could be divided into five separate processes: a) synthesis of the functionalized surface group, b) preparation of the branching unit, c) growth of the dendrons, d) coupling of the dendrons to the central core and e) chemical transformations of functionalized surface group.

a) Synthesis of the functionalized surface group

4-Iodobenzoic acid **79** was used as the starting material. It was firstly subjected to esterificaction to obtain the corresponding ethyl ester **80**[77] in 98% yield. After that, the ethyl ester was reduced to the corresponding alcohol **81**[78] by diisobutylaluminium hydride (DIBAL-H) and then followed by bromination (PPh_3, CBr_4), 4-iodobenzyl bromide **82**[79] was obtained as the functionalized surface group in an overall 64% yield (Scheme 1).

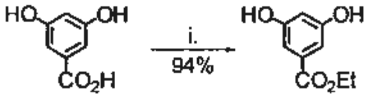


79 **80** **81** **82**

Scheme 1. Reagents and conditions: i. EtOH, conc. H_2SO_4, reflux, 24 h; ii. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; iii. PPh_3, CBr_4, THF, 25 °C, 2 h.

b) Preparation of the branching unit

In the S-Gn series, the preparation of the branching unit involved only one step. 3,5-Dihydroxybenzoic acid **83** was dissolved in ethanol and refluxed for 24 h in the presence of concentrated sulfuric acid to afford ethyl 3,5-dihydroxybenzoate **84**[80] in 94% yield (Scheme 2).



83 **84**

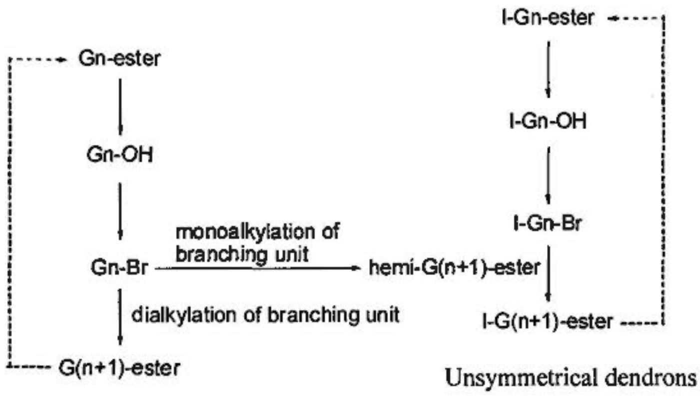
Scheme 2. Reagents and conditions: i. EtOH, conc. H_2SO_4, reflux, 24 h.

c) Growth of the dendrons

Since the target dendrimers contain two different types of surface functionalities, hence both symmetrical and unsymmetrical dendrons were involved in the synthetic pathway. To simplify our description, some notations were used to represent dendrons and dendrimers. The notation S/L-Gn-X and Y-S/L-Gn-X are used to designate symmetrical S/L-dendrons and unsymmetrical surface Y-monofunctionalized

S/L-dendrons, both of generation n with an X focal point group, respectively. For hemi-substituted S/L-dendrons (i.e. dendrons that possess only one branch at the innermost branching juncture), the notation hemi-S/L-Gn-X will be used. Finally, the notation Y–[S/L-Gn]–Y is employed to represent S/L-dendrimers, instead of dendrons, containing two surface-functionalized Y substituents.

The growth of a symmetrical dendron was simply done by following the cycle on the left in Figure 37. The focal point of a Gn-ester was firstly transformed through an alcohol to a bromide and then the branching unit was doubly alkylated with S/L-Gn-Br. Finally G(n+1)-ester was obtained and it could be further grown by repeating this cycle. On the other hand, the growth of an unsymmetrical dendron was more complicated due to the involvement of hemi-Gn-ester that was prepared by a single *O*-alkylation of the branching unit with a symmetrical dendron Gn-Br, followed by the second *O*-alkylation with an unsymmetrical dendron I-S/L-Gn-Br. The growth of unsymmetrical dendrons was shown in the cycle on the right in Figure 37.



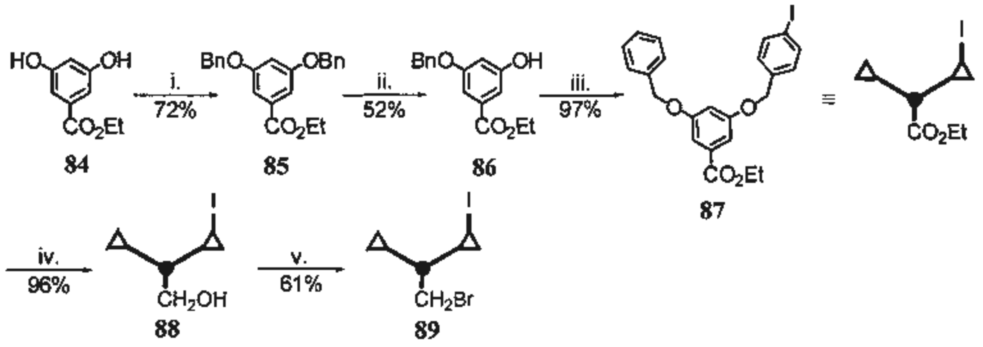
Unsymmetrical dendrons

Symmetrical dendrons

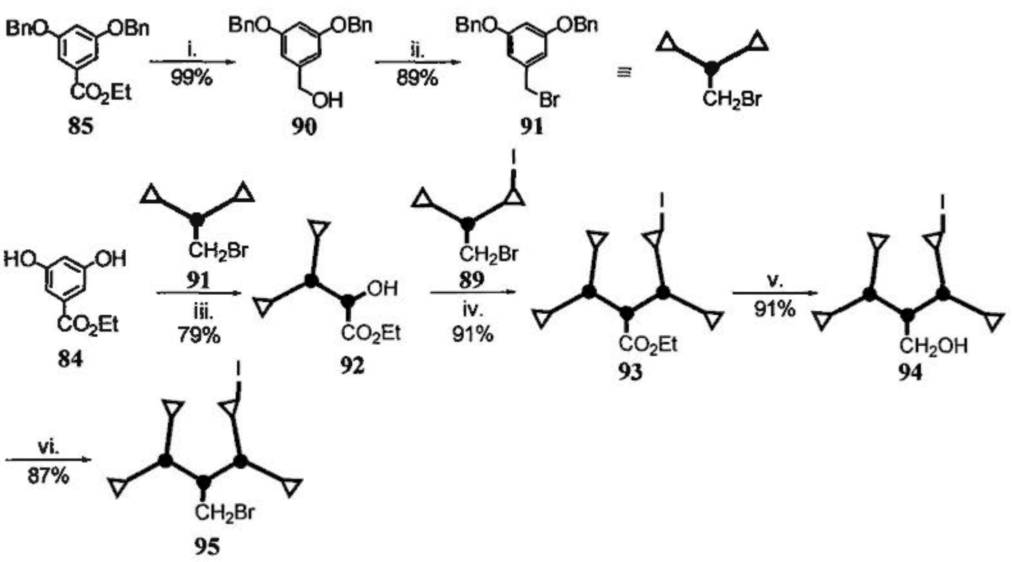
Figure 37. *Schematic diagram representing the growth of S-Gn dendrons.*

For the synthesis of I-S-G1-Br (Scheme 3), the shorter brancher **84** firstly underwent di-*O*-benzylation by reacting with benzyl bromide to obtain compound **85** (S-G1-ester)[81] in 72% yield followed by partial hydrogenolysis (H_2, Pd-C) to afford compound **86** (hemi-S-G1-ester)[82] in 52% yield. In this mono-debenzylation step, the fully deprotected ethyl 3,5-dihydroxybenzoate **84** and the unreacted starting material **85** could be cleanly removed by flash column chromatography owing to the large differences in their R_f values. Hemi-S-G1-ester **86** was then reacted with 4-iodobenzyl bromide **82** in the presence of K_2CO_3 and 18-crown-6 in acetone under reflux for 12 h to generate the unsymmetrical dendron **87** (I-S-G1-ester) in 97% yield.

It should be emphasized that if there was a small amount of compounds **84** and **85** remained in this step, a doubly surface-functionalized dendron and a dendron with no surface functionality would be formed. If the synthesis was carried on this mixture, an inseparable multi-functional cross-linkers and a polymer chain stopper would be generated and this could adversely affect the polymerization results. Therefore the complete removal of compounds **84** and **85** from compound **86** (hemi-S-G1-ester) was extremely important. After that, I-S-G1-ester **87** was subjected to diisobutylaluminium hydride (DIBAL-H) reduction and followed by bromination (PPh_3, CBr_4) to afford the unsymmetrical bromide **89** (I-S-G1-Br) in an overall 59% yield.



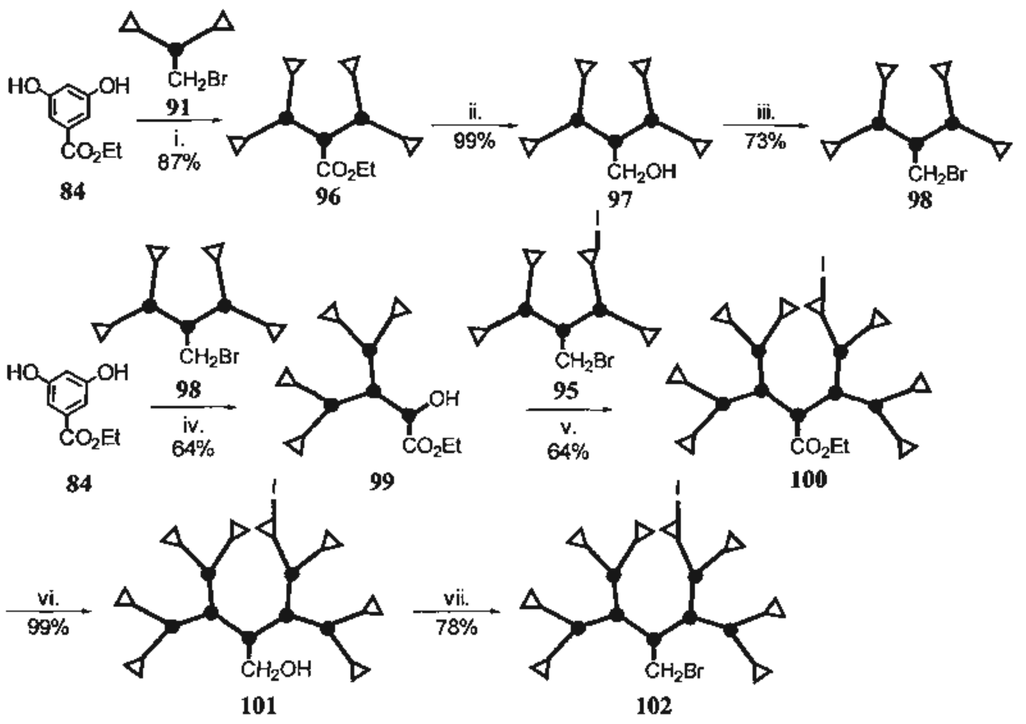
Scheme 3. Reagents and conditions: i. BnBr (2.2 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; ii. H_2, Pd-C, EtOAc/EtOH (1/1), 25 °C, 2 h; iii. 4-iodobenzyl bromide 82 (1.1 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; iv. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; v. PPh_3, CBr_4, THF, 25 °C, 2 h.



Scheme 4. Reagents and conditions: i. $LiAlH_4$, THF, 0 °C→25 °C, 2 h; 2) H_3O^+; ii. PPh_3, CBr_4, THF, 25 °C, 2 h; iii. 91 (0.25 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; iv. 89 (1.1 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; v. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; vi. PPh_3, CBr_4, THF, 25 °C, 2 h.

The same strategy was applied to the synthesis of the G2 dendrons (Scheme 4). First, compound **85** (S-G1-ester) underwent reduction by lithium aluminium hydride ($LiAlH_4$) and bromination (PPh_3, CBr_4) to yield S-G1-Br **91**[6d] in overall 82% yield. Then the branching unit **84** was mono-*O*-alkylated with S-G1-Br **91** (0.25 equiv.) to afford hemi-S-G2-ester **92** in 79% yield.[83] No *C*-alkylated product was formed, but a small amount (~ 10%) of di-*O*-alkylated product and unreacted starting material **84** was found. Fortunately, they were separable from hemi-S-G2-ester **92** by flash column chromatography. Subsequently, hemi-S-G2-ester **92** was subjected to the second *O*-alkylation with I-S-G1-Br **89** to obtain I-S-G2-ester **83** under Williamson conditions in 91% yield. Finally the focal point functionality was transformed from ethyl ester to the corresponding alcohol I-S-G2-OH **94** by DIBAL-H reduction in 91% yield and the corresponding bromide I-S-G2-Br **95** was obtained finally by PPh_3/CBr_4 bromination in 87% yield.

Preparation of G3 dendrons was shown in Scheme 5. Firstly the branching unit **84** underwent di-*O*-alkylation with S-G1-Br **91** (2.2 equiv.) to provide the

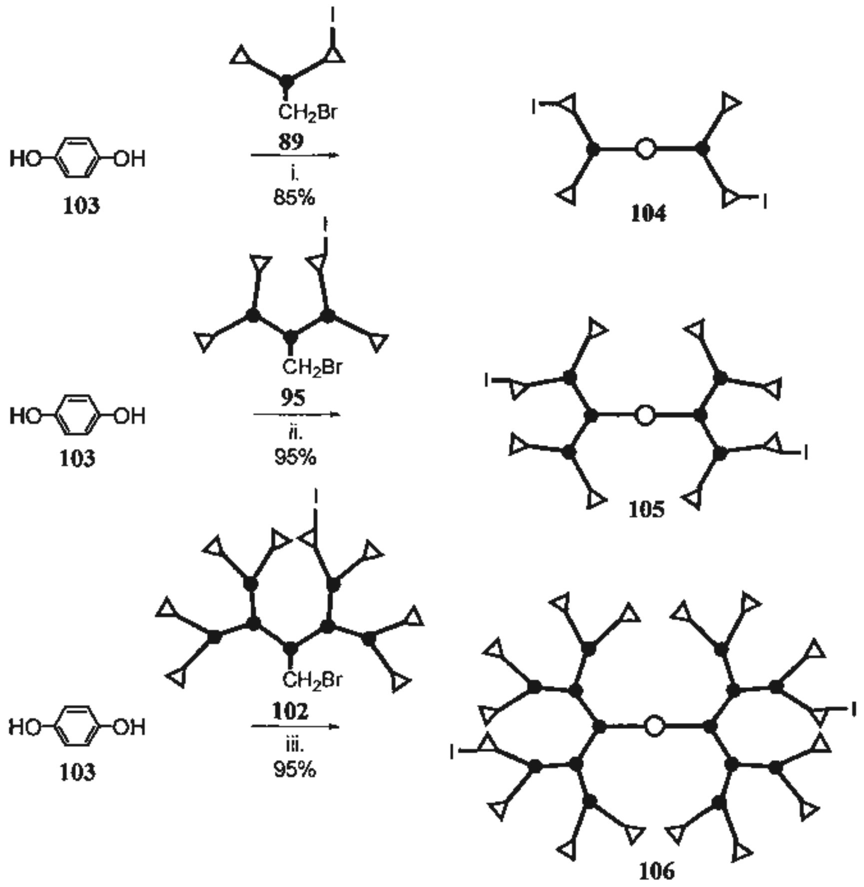


Scheme 5. *Reagents and conditions: i. 91 (2.2 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; ii. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; iii. PPh_3, CBr_4, THF, 25 °C, 2 h; iv. 98 (0.25 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; v. 95 (1.1 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 60 h; vi. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; vii. PPh_3, CBr_4, THF, 25 °C, 2 h.*

symmetrical G2 dendron S-G2-ester **96**[84] in 87%. It was then converted into S-G2-Br **98**[6d] by reduction (DIBAL-H) and bromination (PPh_3, CBr_4) in 72% overall yield. S-G2-Br **98** (0.25 equiv.) was then acted as an alkylating agent for the mono-*O*-alkylation of the same branching unit **84** to obtain hemi-S-G3-ester **99** in 64% yield.[83] Again, the di-*O*-alkylation product and the unreacted branching unit were removed by flash column chromatography. Hemi-S-G3-ester **99** was then further *O*-alkylated with the unsymmetrical dendron I-S-G2-Br **95** to afford I-S-G3-ester **100** under Williamson conditions in 64% yield. Finally, the ethyl ester was converted into the corresponding bromide I-S-G3-Br **102** in overall 77% yield.

d) Coupling of the dendrons to the central core

With I-S-Gn-Br (n = 1–3) in hand, coupling of hydroquinone **103** with the various I-S-Gn-Br **89, 95** and **102** (2.2 equiv.) produced the corresponding dendrimer

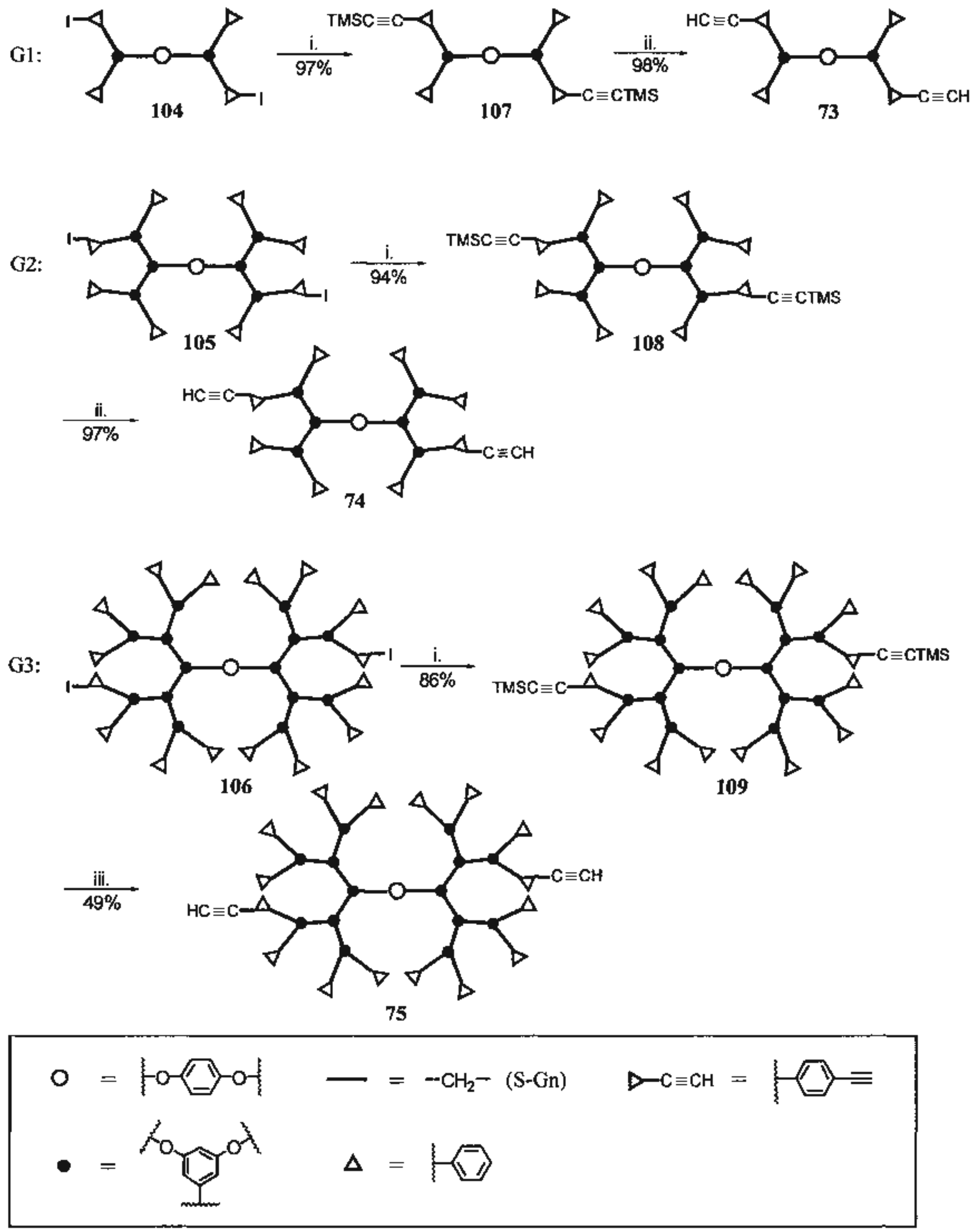


Scheme 6. Reagents and conditions: i. 89 (2.2 equiv.), Cs_2CO_3, DMF, dibenzo-24-crown-8, 25 °C, 12 h; ii. 95 (2.2 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 3 d; iii. 102 (2.2 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 3 d.

I–[S-Gn]–I **104–106** respectively, in 85–95% yield (Scheme 6). It should be mentioned that the conditions for preparing I–[S-G2]–I **105** and I–[S-G3]–I **106** were slightly different from the case of I–[S-G1]–I **104**. For the G1 analog, Cs_2CO_3 was found to be a better base, but many difficultly separable side products were formed. For the G2 and G3 analogs, instead, K_2CO_3/acetone was found to be a better choice as the desired products could be obtained in high yields (95%) although it took 72 h to complete the reaction.

e) Chemical transformations of functionalized surface group

The final operation of completing the synthesis of the dendrimer beads was to convert the two functionalized surface groups from iodide to terminal alkyne (Scheme



Scheme 7. Reagents and conditions: i. TMSC≡CH, CuI, $Pd(PPh_3)_2Cl_2$, PPh_3, Et_3N, toluene, 100 °C in sealed tube, 3 d; ii. K_2CO_3, MeOH/THF (1/1), 25 °C, 2 h; iii. TBAF, THF, 25 °C, 15 min.

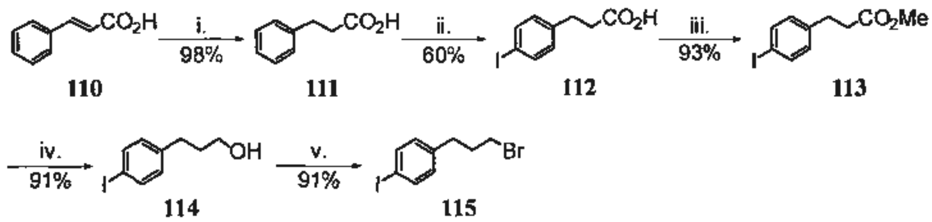
7). Firstly, I–[S-Gn]–I (n = 1–3) **104–106** were subjected to Sonogashira coupling reaction with trimethylsilyl acetylene (TMSC≡CH) at 100 °C for 3 days to give TMSC≡C–[S-Gn]–C≡CTMS (n = 1–3) **107–109**, respectively in 86%–97% yields. Then the TMS groups were removed by alkaline cleavage (for S-G1 and S-G2) and tetrabutylammonium fluoride (TBAF) (for S-G3) to afford the target compounds HC≡C–[S-Gn]–C≡CH (n = 1–3) **73–75**, respectively in 49–98% yield. The TMS groups in TMSC≡C–[S-G3]–C≡CTMS **109** could also be removed by K_2CO_3. Unfortunately, a mixture of HC≡C–[S-G3]–C≡CH **75** together with 10% of some oligomeric Hay homocoupling products[85] were found according to ^1H NMR spectroscopy and GPC analysis. It was suspected that there was trace amount of copper salts left over from the previous Sonogashira coupling and led to the formation of oligomeric Hay homocoupling products. This side reaction could be suppressed by using TBAF as the deprotecting agent for 15 minutes but some homocoupling products could still be found if the reaction time was allowed to stay longer than 15 minutes.

3.3.2. Synthesis of HC≡C–[L-Gn]–C≡CH dendrimers

Our next goal was to prepare target compounds belonging to the HC≡C–[L-Gn]–C≡CH dendrimers (n = 1–3). In the L-Gn series, the preparation of the functionalized surface group and the branching unit involved more steps but the reactions governing the growth of dendrons were similar to those of the S-Gn series.

a) Synthesis of the functionalized surface group

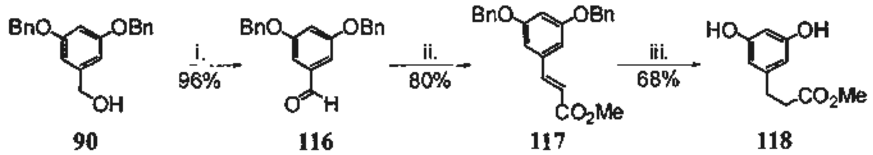
Starting from cinnamic acid **110** (Scheme 8), the double bond was hydrogenated in the presence of Pd-C to give dihydrocinnamic acid **111**[86] in 98% yield, subsequent mono-iodination of the phenyl ring then obtained 3-(4-iodophenyl)propanoic acid **112**[87] in 60% yield. The carboxylic acid group was converted to the corresponding bromide **115** through esterification, reduction (DIBAL-H) and bromination (PPh_3, CBr_4) to afford the desired functional surface unit 1-bromo-3-(4-iodophenyl)propane **115**[88] in overall 77% yield.



Scheme 8. Reagents and conditions: i. H_2, Pd-C, EtOAc/EtOH (1/1), 25 °C, 12 h; ii. H_5IO_6, I_2, conc. H_2SO_4, HOAc, 70 °C, 12 h; iii. MeOH, conc. H_2SO_4, reflux, 12 h; iv. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; v. PPh_3, CBr_4, THF, 25 °C, 2 h.

b) Preparation of the branching unit

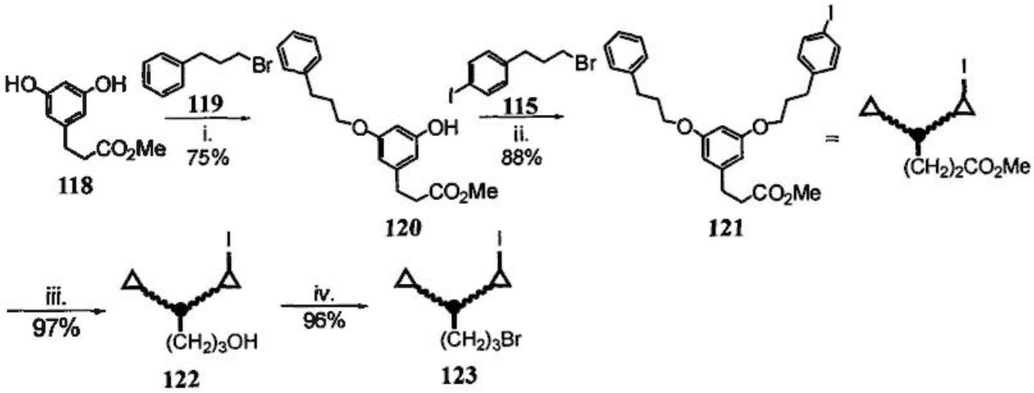
For the synthesis of the branching unit in L-Gn series (Scheme 9), compound **90** was firstly oxidized to the corresponding aldehyde **116**[89] in 96% yield and then reacted with trimethylphosphonoacetate under Horner-Wadsworth-Emmons conditions[90] to afford the α,β-unsaturated ester **117** in 80% yield.[91] Debenzylation and hydrogenation were carried out simultaneously under hydrogen atmosphere in the presence of Pd-C to give the branching unit—methyl 3-(3,5-dihydroxyphenyl)propanoate **118**[91] in 68% yield.



Scheme 9. Reagents and conditions: i. PCC, silica gel, CH_2Cl_2, 25 °C, 2 h; ii. NaH, trimethylphosphonoacetate, THF, reflux, 12 h; iii. H_2, Pd-C, EtOAc/EtOH (1/1), 25 °C, 24 h.

c) Growth of the dendrons

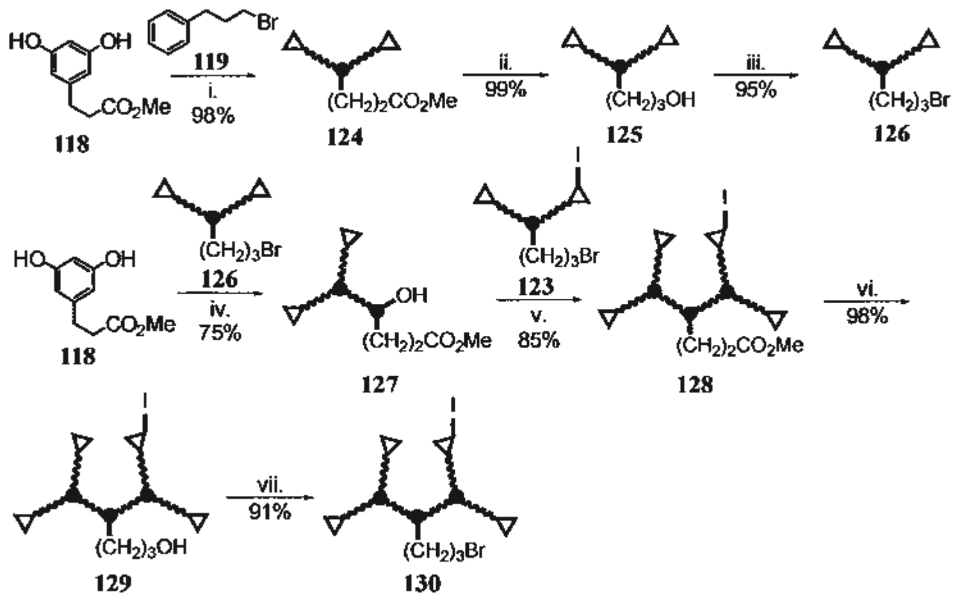
As the branching unit in the L-Gn series was lengthened by two carbon atoms, two differences were noted. Firstly, the dendrons and dendrimers in the L-Gn series had better solubility as compared to the S-Gn series. Secondly, some C-alkylation products were formed during the Williamson ether synthesis and this slightly increased the difficulties in the purification process.



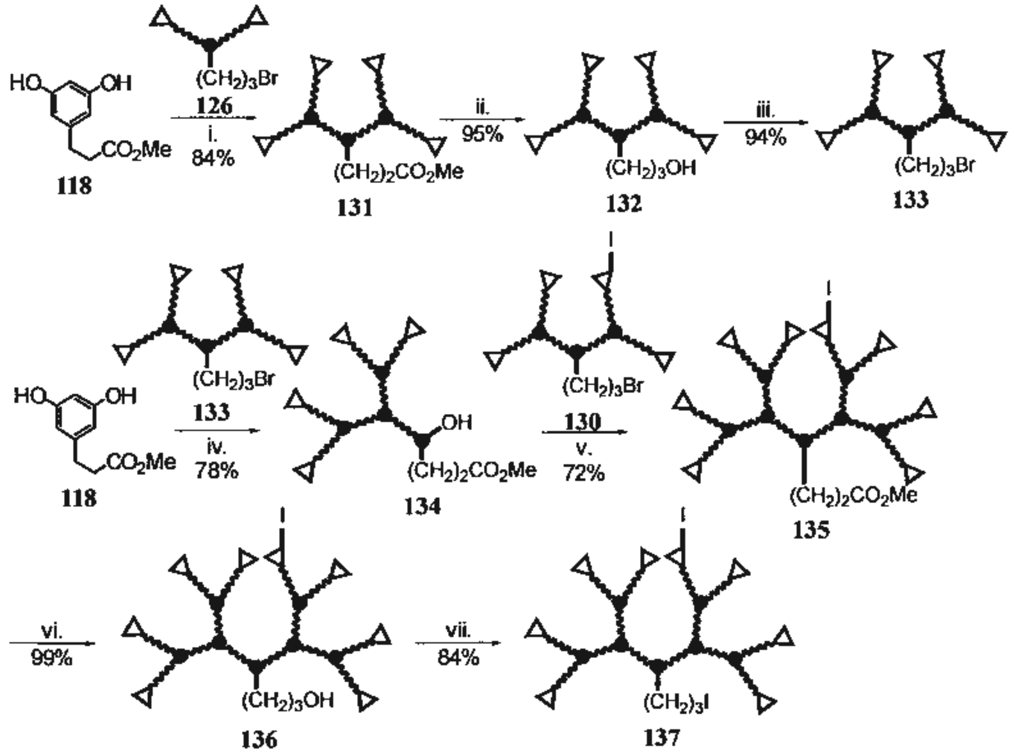
Scheme 10. Reagents and conditions: i. 119 (0.25 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; ii. 115 (1.1 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; iii. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; iv. PPh_3, CBr_4, THF, 25 °C, 2 h.

For the synthesis of L-G1 dendrons (Scheme 10), the branching unit **118** firstly underwent mono-*O*-alkylation with 1-bromo-3-phenylpropane **119** (0.25 equiv.) under Williamson conditions to obtain hemi-L-G1-ester **120** in 75%.[83] Di-*O*-alkylation product (13%) and *C*, *O*-alkylation product (ca. 5%) were also found. Fortunately, these side products could be removed by flash column chromatography. Hemi-L-G1-ester **120** was then *O*-alkylated with the functionalized surface group **115** to provide the unsymmetrical dendron I-L-G1-ester **121** in 88% yield. I-L-G1-ester **121** was then converted to the corresponding bromide I-L-G1-Br **123** by DIBAL-H reduction and bromination (PPh_3, CBr_4) in 93% overall yield in two steps.

En route to L-G2 dendrons, di-*O*-alkylation of the branching unit **118** with the bromide **119** gave L-G1-ester **124** in 98% yield (Scheme 11). Reduction of the ester group followed by bromination of the resulting alcohol **125**, L-G1-Br **126** was afforded in 94% overall yield. Then the branching unit **118** was mono-*O*-alkylated with L-G1-Br **126** (0.25 equiv.) to produce hemi-L-G2-ester **127** in 75% yield.[83] It was then subjected to the second *O*-alkylation with I-L-G1-Br **123** followed by DIBAL-H reduction and bromination (PPh_3, CBr_4) to afford the desired unsymmetrical dendron I-L-G2-Br **130** in 76% overall yield.



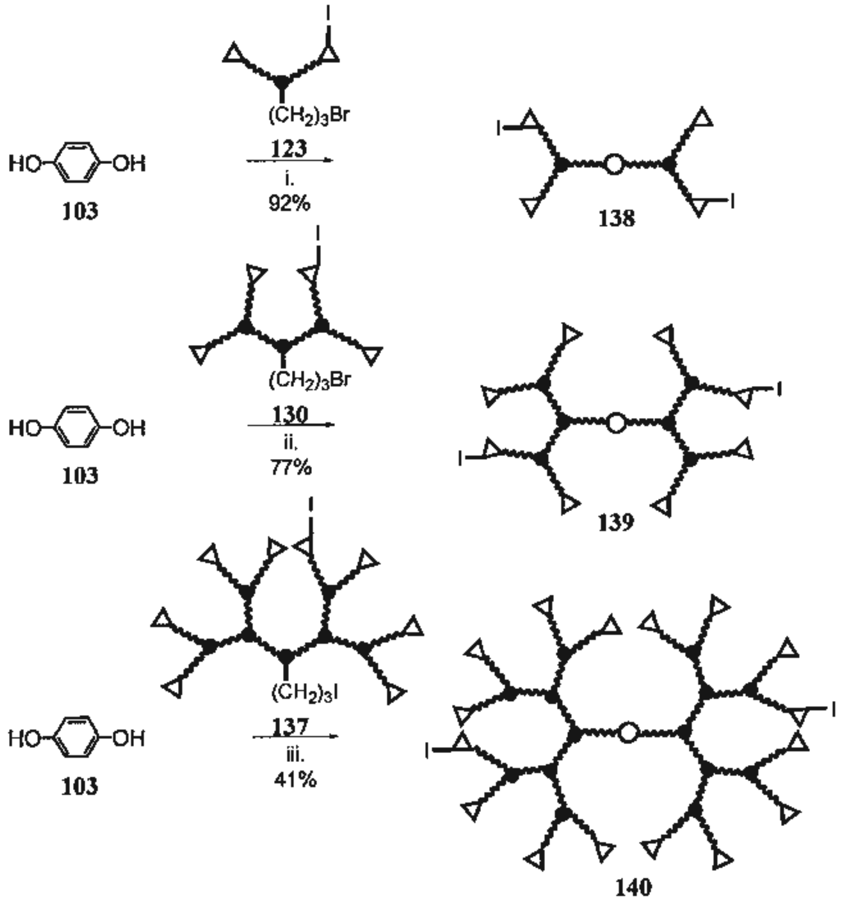
Scheme 11. *Reagents and conditions: i.* ***119*** *(2.2 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; ii. $LiAlH_4$, THF, 0 °C→25 °C, 2 h; 2) H_3O^+; iii. PPh_3, CBr_4, THF, 25 °C, 2 h; iv.* ***126*** *(0.25 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; v.* ***123*** *(1.1 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; vi. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; vii. PPh_3, CBr_4, THF, 25 °C, 2 h.*



Scheme 12. *Reagents and conditions: i.* ***126*** *(2.2 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; ii. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; iii. PPh_3, CBr_4, THF, 25 °C, 2 h; iv.* ***133*** *(0.25 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; v.* ***130*** *(1.1 equiv.), K_2CO_3, acetone, 18-crown-6, reflux, 12 h; vi. 1) DIBAL-H/hexane, toluene, −60 °C→0 °C, 2 h; 2) H_3O^+; vii. PPh_3, NIS, CH_2Cl_2, 0 °C→25 °C, 12 h.*

For the preparation of L-G3-dendrons (Scheme 12), the branching unit **118** was firstly di-*O*-alkylated with L-G1-Br **126** followed by DIBAL-H reduction and bromination (PPh_3, CBr_4) to give L-G2-Br **133** in 75% overall yield. The branching unit **118** was then mono-*O*-alkylated with L-G2-Br **133** (0.25 equiv.) to yield hemi-L-G3-ester **134** in 78% yield.[83] It was further *O*-alkylated with I-L-G2-Br **130** followed by DIBAL-H reduction and iodination [PPh_3, *N*-iodosuccinamide (NIS)] this time to obtain the desired product I-L-G3-I **137** in 60% overall yield.

d) Coupling of the dendrons to the central core

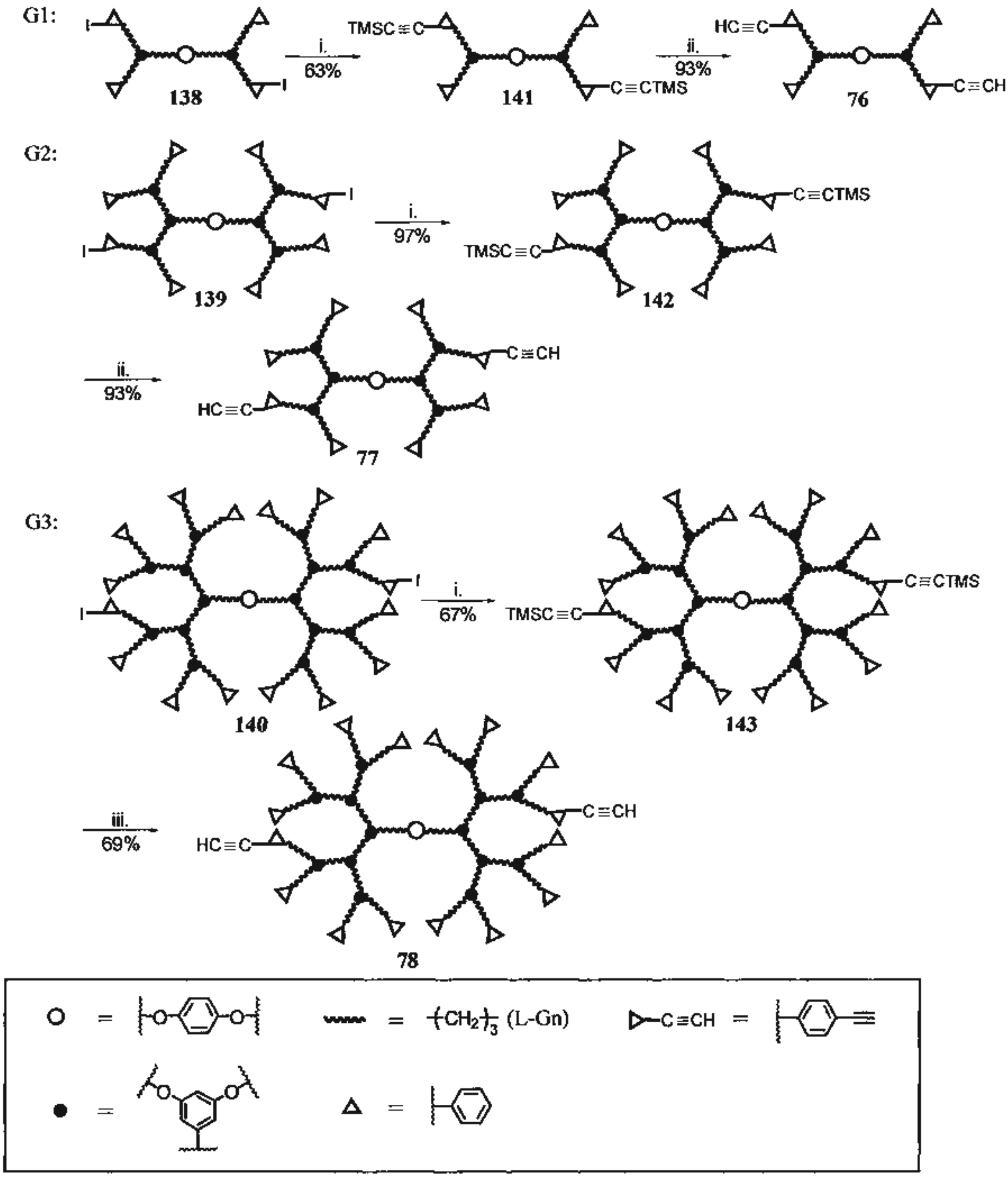


Scheme 13. *Reagents and conditions: i.* ***123*** *(2.2 equiv.),* K_2CO_3*, acetone, 18-crown-6, reflux, 3 d; ii.* ***130*** *(2.2 equiv.),* K_2CO_3*, acetone, 18-crown-6, reflux, 3 d; iii.* ***137*** *(2.2 equiv.),* K_2CO_3*, acetone, 18-crown-6, reflux, 3 d.*

I-L-G1-Br **123**, I-L-G2-Br **130** and I-L-G3-I **137** were anchored to the hydroquinone core **103** by Williamson ether synthesis to afford dendrimers I–[L-Gn]–I (n = 1–3) **138–140**, respectively in 41–92% (Scheme 13). As the generation increased, the coupling reaction yield decreased owing to the increasing steric hindrance. Comparing with benzyl bromides used in the S-Gn series, alkyl bromides used in the L-Gn series were less reactive and the coupling reaction proceeded much slower. Another side reaction, namely, hydrolysis of the halide became more significant but fortunately the hydrolyzed products I-L-Gn-OH (n = 1–3) **122**, **129** and **136** were separable from the target compounds. Due to the extremely poor reactivity of the I-L-G3-Br, the coupling reaction was incomplete even performed under higher concentrations and for longer reaction time. As a result, the corresponding alkyl iodide (I-L-G3-I) **137** was used as the alkylating agent. Although I-L-G3-I **137** was more reactive, the reaction yield was still 41% and a large amount of the hydrolyzed product I-L-G3-OH **136** was still formed.

e) Chemical transformations of functionalized surface group

Similar to the S-Gn series, the two surface iodide groups of dendrimers were converted to the corresponding bifunctional alkyne in two steps (Scheme 14). Thus, Sonogashira coupling reactions were performed on the dendrimers I–[L-Gn]–I (n = 1–3) **138–140** to obtain TMSC≡C–[L-Gn]–C≡CTMS (n = 1–3) **141–143**, respectively in 63–97% yield. Then the TMS groups were removed by alkaline hydrolysis (for G1 and G2) or TBAF (for G3) to afford the target compounds HC≡C–[L-Gn]–C≡CH (n = 1–3) **76–78** in 69–93% yield. Hence, the six dendrimer beads with two surface-functionalized terminal alkyne groups were prepared. In the next section, their copolymerizations with the linear *trans*-[Pt(PEt_3)$_2$$Cl_2$] platinum linker **65** will be discussed.

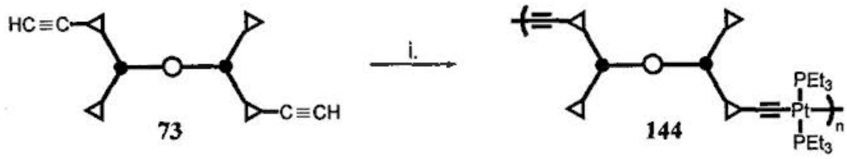


***Scheme 14.** Reagents and conditions: i. TMSC≡CH, CuI, Pd(PPh$_3$)$_2$Cl$_2$, PPh$_3$, Et$_3$N, toluene, 100 °C in sealed tube, 3 d; ii. K$_2$CO$_3$, MeOH/THF (1/1), 25 °C, 2 h; iii. TBAF, THF, 25 °C, 15 min.*

3.3.3. Formation of organoplatinum poly(dendrimer)s

Before carrying out the copolymerizations, the reaction conditions such as monomer concentrations, reaction temperature and solvent had to be optimized. The dendritic monomers studied in this project were more soluble in chloroform ($CHCl_3$) than in dichloromethane (CH_2Cl_2) and as solubility was one of the major factors in determining the polymerization results. To prevent the precipitation of the poly(dendrimer)s, therefore the solvent was changed from $CH_2Cl_2/{}^{i}Pr_2NH$ (*v*/*v* = 1/1) to $CHCl_3/{}^{i}Pr_2NH$ (*v*/*v* = 1/1).

The concentration optimization process was then carried out by performing the 1:1 copolymerization reactions between monomer HC≡C–[S-G1]–C≡CH **73** and the platinum linker—*trans*-$[Pt(PEt_3)_2Cl_2]$ **65** in the presence of CuI in $CHCl_3/{}^{i}Pr_2NH$ (*v*/*v* = 1/1) at 40 °C under three different monomer concentrations of 4.4, 8.7 and 17 mM. Three reactions were carried out inside sealed tubes to prevent the solvent evaporation and a change of concentration (Scheme 15). Since this is a 1:1 copolymerization reaction, the stoichiometric ratio between the dendritic macromonomer and the platinum linker must be ensured. The excess amount of either the dendritic monomer or the platinum linker would become a polymer chain stopper to lower the DP value. Therefore it was extremely important for weighing exact amounts of reagents in the weighing procedure.



***Scheme 15.** Reagents and conditions: i. trans-$[Pt(PEt_3)_2Cl_2]$ 65 (1.0 equiv.), CuI, $CHCl_3/{}^{i}Pr_2NH$ (v/v = 1/1), 40 °C in sealed tube, 2 d.*

After the polymerization reactions, the solvent was evaporated and the crude reaction mixtures were redissolved in minimum amount of $CHCl_3$ followed by passing through a short pad of alumina in order to get rid of the remaining copper salt. The obtained crude products Pt-S-G1 **144** under the different monomer concentrations (4.4, 8.7 and 17 mM) were subjected to GPC analysis (Figure 38). It was found that many oligomeric species were formed at the lowest concentration of the monomer (4.4 mM) and therefore this concentration was not a good concentration for this polymerization reaction. On the contrary, the amount of low molecular weight oligomers was much less when the polymerizations were carried out at 8.7 and 17 mM. However, as the G3 monomers possessed higher molecular weights and would lead to a highly viscous solution if the polymerization reaction was performed at 17 mM. The high viscosity of the solution would lower the stirring efficiency, and therefore all copolymerization reactions were conducted in $CHCl_3/{}^iPr_2NH$ (*v*/*v* = 1/1) at 40 °C at 8.7 mM in sealed tubes.

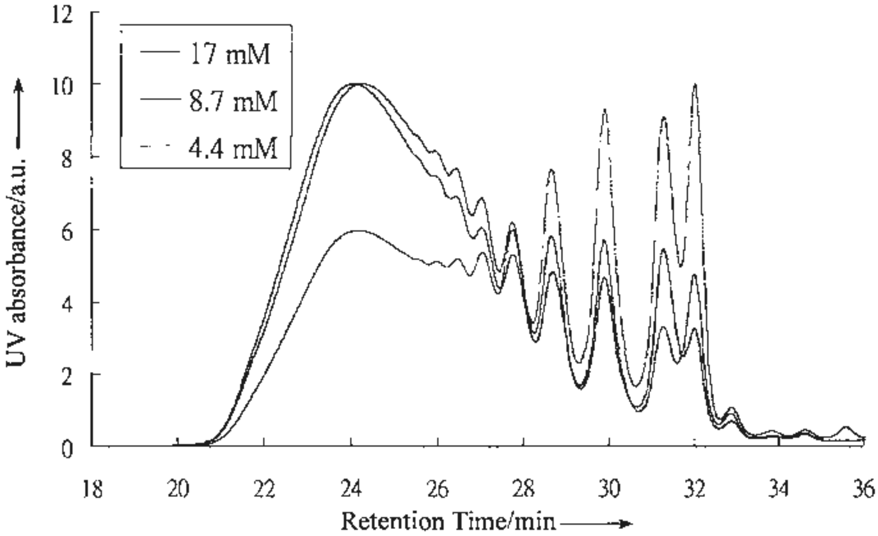
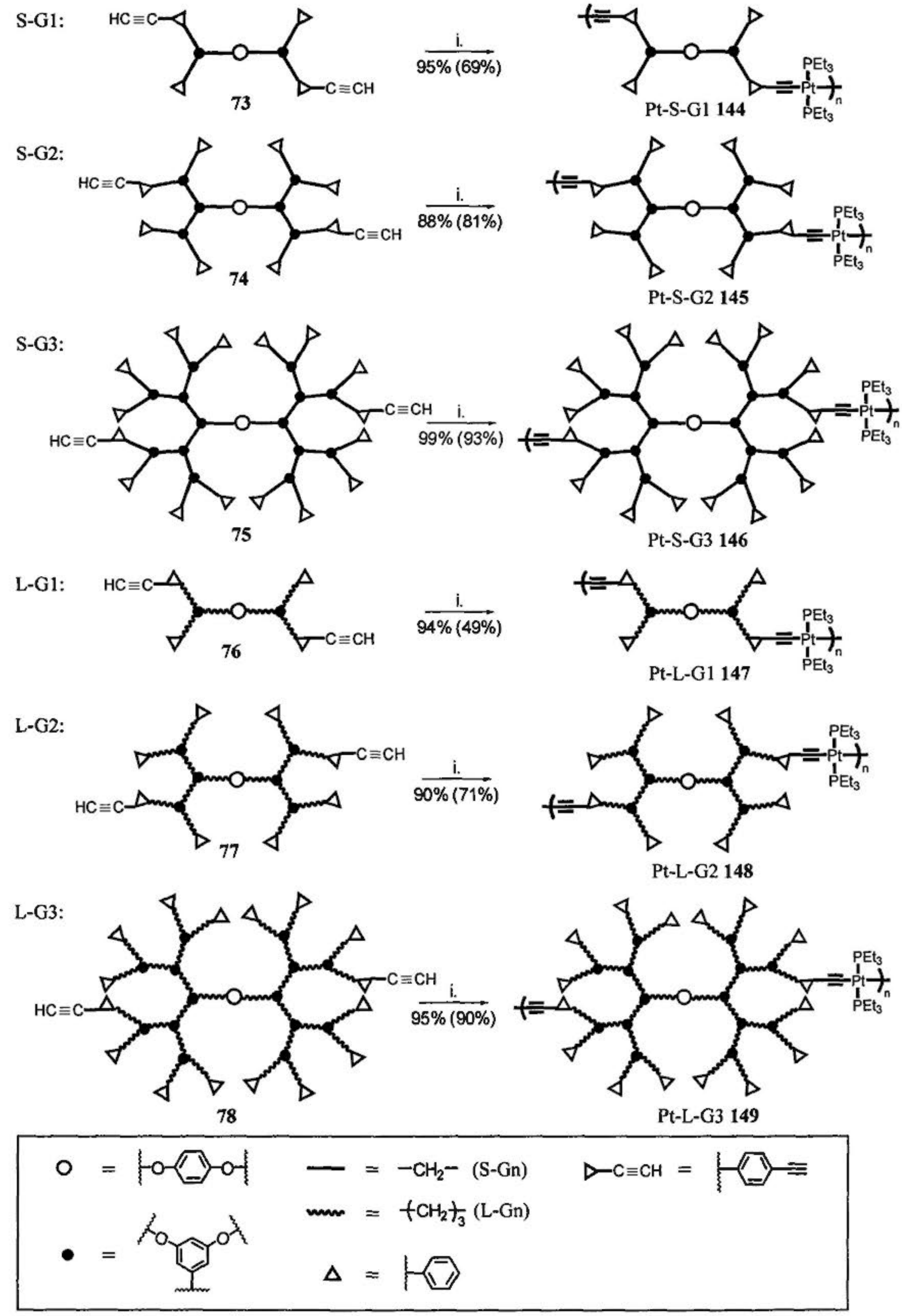


Figure 38. *Copolymerizations of HC≡C–[S-G1]–C≡CH* ***73*** *with trans-[Pt(PEt$_3$)$_2$Cl$_2$] at different concentrations.*

In order to study the effect of structural flexibility of dendritic macromonomers (dendrimer beads) on the polymerization results, the polymerization reactions were conducted side by side (S-Gn *vs* L-Gn) for each generation (G1–G3) under identical reaction conditions (Scheme 16). Totally, three "pairs" of reactions were carried out and the yields of the crude products, after passing through a short pad of alumina, Pt-S-G1 **144**, Pt-L-G1 **147**, Pt-S-G2 **145**, Pt-L-G2 **148**, Pt-S-G3 **146** and Pt-L-G3 **149** were 95%, 94%, 88%, 90%, 99% and 95% respectively. A small sample of all these



Scheme 16. *Reagents and conditions: i. trans-[Pt(PEt$_3$)$_2$Cl$_2$] **65** (1.0 equiv.), CuI, CHCl$_3$/^{i}Pr$_2$NH (v/v = 1/1), 40 °C in sealed tube, 2 d.*

products was taken out for the GPC analysis. Hence the GPC analysis is truly reflected the polymerization results and not those of the precipitated fractions. They were then dissolved in minimum amount of $CHCl_3$ and purified by precipitation in methanol (MeOH). The reaction yields after precipitation were shown in Scheme 16 (in blanket) and the purified products were characterized by ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectroscopy.

Although the synthesis of dendrimers involves many sequential steps, the synthetic strategy used in this project was quite efficient. For the preparation of dendritic macromonomers, HC≡C–[S/L-Gn]–C≡CH (where n = 1–3) **73–78**, most of the steps proceeded in >80% yields, some of the yields were even higher than 90%. The minor concern was the requirement of excess branching units in their mono-alkylation reactions. Fortunately, the branching unit of S-Gn series was easily accessible from commercial materials while the branching unit of L-Gn series could be recycled by column chromatography after the reaction. Typical in most dendrimer synthesis, reaction yields generally decreased for both S-Gn and L-Gn series upon increasing generation due to the increased steric hindrance round the reactive sites. For macropolymerization reactions, the product yields ranged from 88–99%, suggesting that the polymerization reactions between dendrimers with two acetylenic groups and the platinum linker were very efficient.

3.4. Characterization of dendritic macromonomers and poly(dendrimer)s

The structures of dendrons and dendrimers were characterized by ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry (MS), elemental analysis, high resolution MS and GPC analysis. Furthermore all the poly(dendrimer)s were characterized by ^{1}H, ^{13}C and ^{31}P NMR spectroscopies, GPC analysis, and laser light scattering (LLS).

3.4.1. ^{1}H NMR spectroscopy

3.4.1.1. The S-Gn Series

For the molecular structures of all dendrons in the S-Gn series, the aromatic proton signals of inert surface groups (benzyl groups) were located at δ 7.3–7.5. The functional surface groups (*p*-iodobenzyl groups) were characterized by two doublet signals located at δ 7.2 and δ 7.7 due to the aromatic protons. In addition, the aromatic proton signals and the benzylic proton signals of the branching units were found at δ 6.4–6.7 and δ 4.8–5.0 (as a sharp singlet), respectively. On the other hand, all the ethyl esters were characterized by two sets of signals—a triplet signal located at δ 1.4 (J = 7.2 Hz) and a quartet signal at δ 4.4 (J = 7.2 Hz) that were corresponding to the proton signals of $CO_2CH_2C\mathit{H}_3$ and $CO_2C\mathit{H}_2$ respectively. For the dendritic alcohols, they were characterized by a signal (singlet or doublet ($J \approx 5.4$ Hz)) located at δ 4.6 that corresponded to the proton signals of $C\mathit{H}_2OH$. Moreover the proton signals of the hydroxyl groups (OH) appeared as a broad singlet or a triplet ($J \approx 5.1$ Hz) at δ 1.7–2.0. Furthermore the proton signals of CH_2Br of dendritic bromides were found at δ 4.4 as a singlet. For the hemi-S-Gn-esters, the phenolic proton signals (ArO*H*) appeared as a broad singlet at δ 5.07 for hemi-S-G1-ester **86** and δ 5.37 for hemi-S-G2-ester **92** and hemi-S-G3-ester **99**.

After the dendrons were anchored to the core, the resulting dendrimers I–[S-Gn]–I were characterized by a singlet located at δ 6.9 that was corresponding to the aromatic proton signals of the hydroquinone core. En route to the transformation to TMSC≡C–[S-Gn]–C≡CTMS, the proton signals of TMS groups were found at δ 0.2 as a sharp singlet.

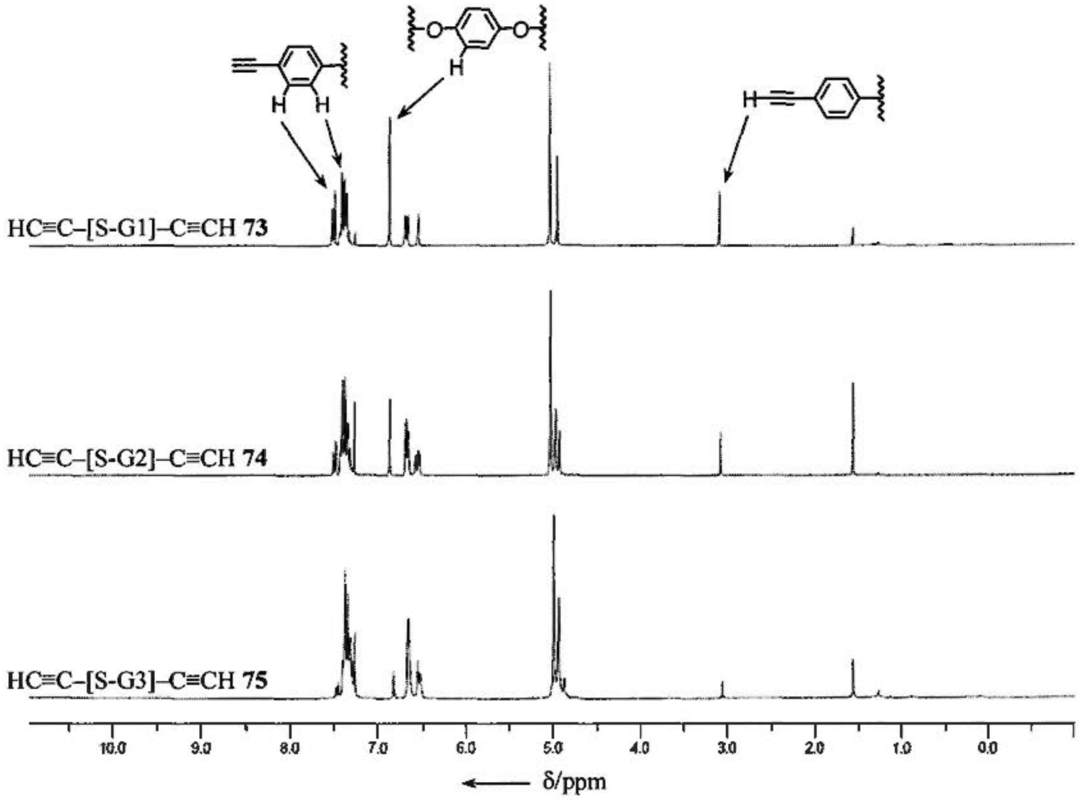


Figure 39. *The stacked* ^1H *NMR (300 MHz,* $CDCl_3$*) spectra of HC≡C–[S-Gn]–C≡CH 73–75.*

For the dendritic macromonomers HC≡C–[S-Gn]–C≡CH (n = 1–3) **73–75**, the ^{1}H NMR spectra showed singlet signals at δ 3.0 and δ 6.9 that were due to the acetylenic protons (C≡CH) and the aromatic protons of hydroquinone core, respectively (Figure 39). Besides one part of the aromatic AB system signal due to the aromatic protons of the functional surface groups was found at δ 7.5 and the other part was merged with other aromatic signals.

After copolymerization reactions, the resulting organoplatinum poly(dendrimer)s Pt-S-Gn **144**, **145** and **146** were characterized by ^{1}H NMR spectroscopy (Figure 40). Their spectra illustrated the reactions were complete because of the disappearance of the acetylenic proton signals at δ 3.0. It was found that the aromatic signals *ortho* to the functional 4-ethynylphenyl surface moieties were upfield shifted. In addition, two sets of signals at δ 1.0–1.4 and δ 2.0–2.4 due to ethylphosphine ligands (PCH_2CH_3

and PCH_2 respectively) attaching to the platinum complex were present.

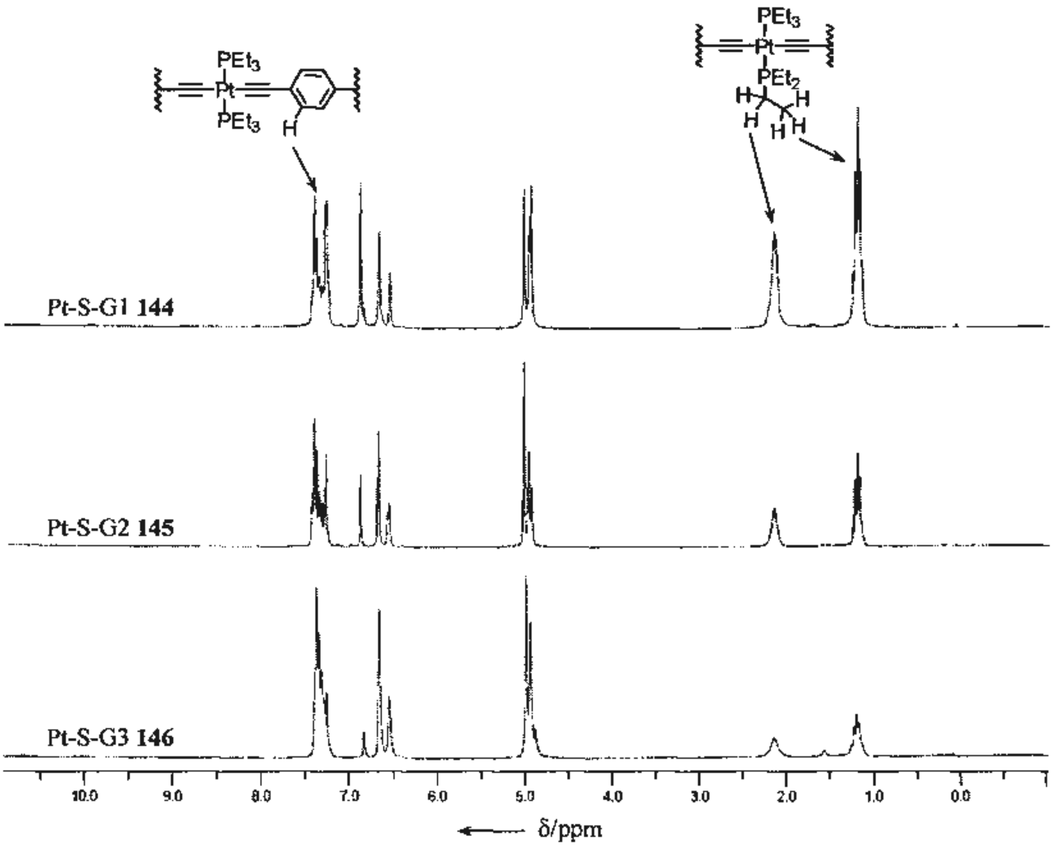


Figure 40. *The stacked ^1H NMR (300 MHz, $CDCl_3$) spectra of poly(dendrimer)s Pt-S-Gn* ***144, 145*** *and* ***146.***

3.4.1.2. The L-Gn Series

For all the dendrons in the L-Gn series, the aromatic proton signals due to inert peripheries were found at δ 7.1–7.4, whereas the aromatic AB system signals due to the aromatic protons of the functional surfaces were found at δ 7.0 and δ 7.6. For the elongated branching units, the aromatic proton signals at δ 6.2–6.4 and the benzylic proton signals at δ 3.8–4.0 were found. Furthermore, the aliphatic proton signals also appeared at δ 1.9–2.2 and δ 2.6–2.8.

All the esters were characterized by a sharp singlet located at δ 3.7 due to the protons of the methyl ester (CO_2CH_3). Moreover the structures of dendritic alcohols were characterized by a signal at δ 1.3–1.4 [broad singlet or triplet ($J \approx 5.1$ Hz)] due to the hydroxyl groups and a signal at δ 3.7 [triplet or quartet ($J \approx 6$ Hz)] due to the methylene protons adjacent to the OH group (CH_2OH). On the other hand, the characterization signal of the dendritic bromides appeared at δ 3.4 [triplet ($J \approx 6.6$ Hz)] due to the methylene protons attaching to the bromine atom (CH_2Br). In the case of I-L-G3-I, the proton signal of the corresponding methylene protons (CH_2I) was found at δ 3.2 as a triplet ($J = 6.9$ Hz). Furthermore the phenolic proton signals of the hemi-L-Gn-esters (n = 1–3) were located at δ 4.9–5.4 as a broad singlet.

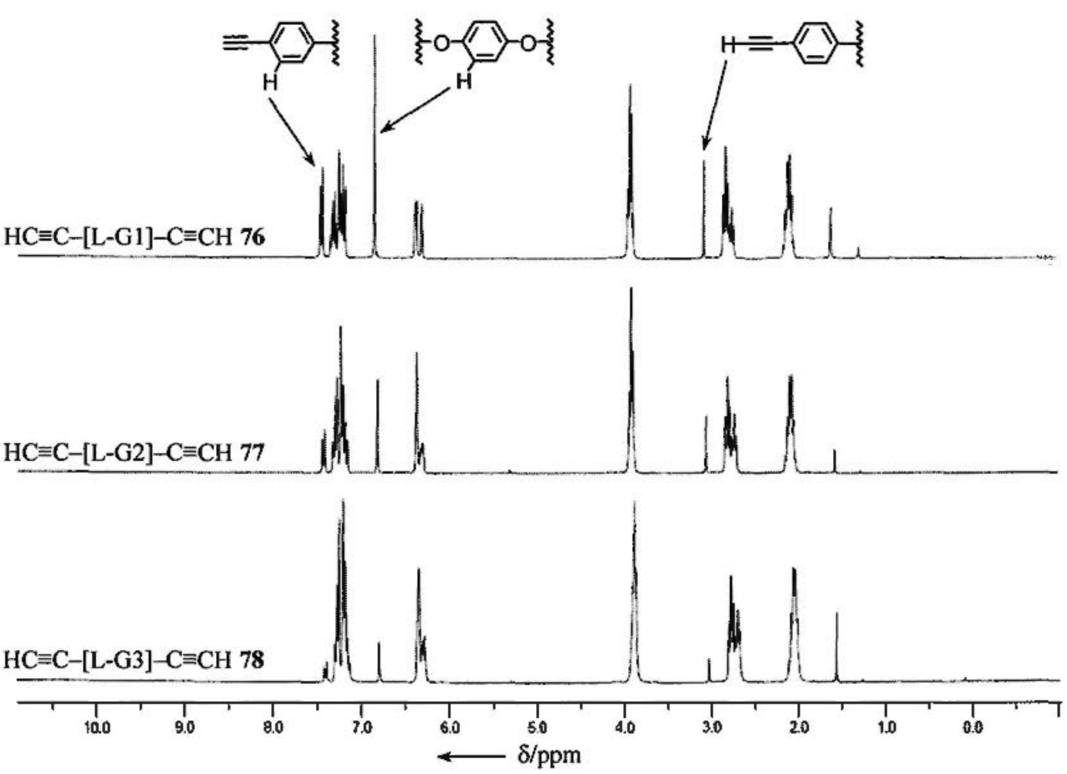


***Figure 41.** The stacked ^1H NMR (300 MHz, $CDCl_3$) spectra of HC≡C–[L-G1]–C≡CH 76–78.*

After coupling to the central cores, the dendrimers were characterized by the appearance of an aromatic proton signal at δ 6.8 due to the hydroquinone core protons. On the other hand, a sharp singlet was observed at δ 0.2 for the dendrimers with $C \equiv CSi(CH_3)_3$ groups. For the dendritic macromonomers HC≡C–[L-Gn]–C≡CH (n = 1–3) **76–78**, their spectra exhibited the aromatic signals *ortho* to the functional 4-ethynylphenyl surface moieties at δ 7.4, the aromatic signals of central core at δ 6.8

and the acetylenic proton signals at δ 3.0 (Figure 41).

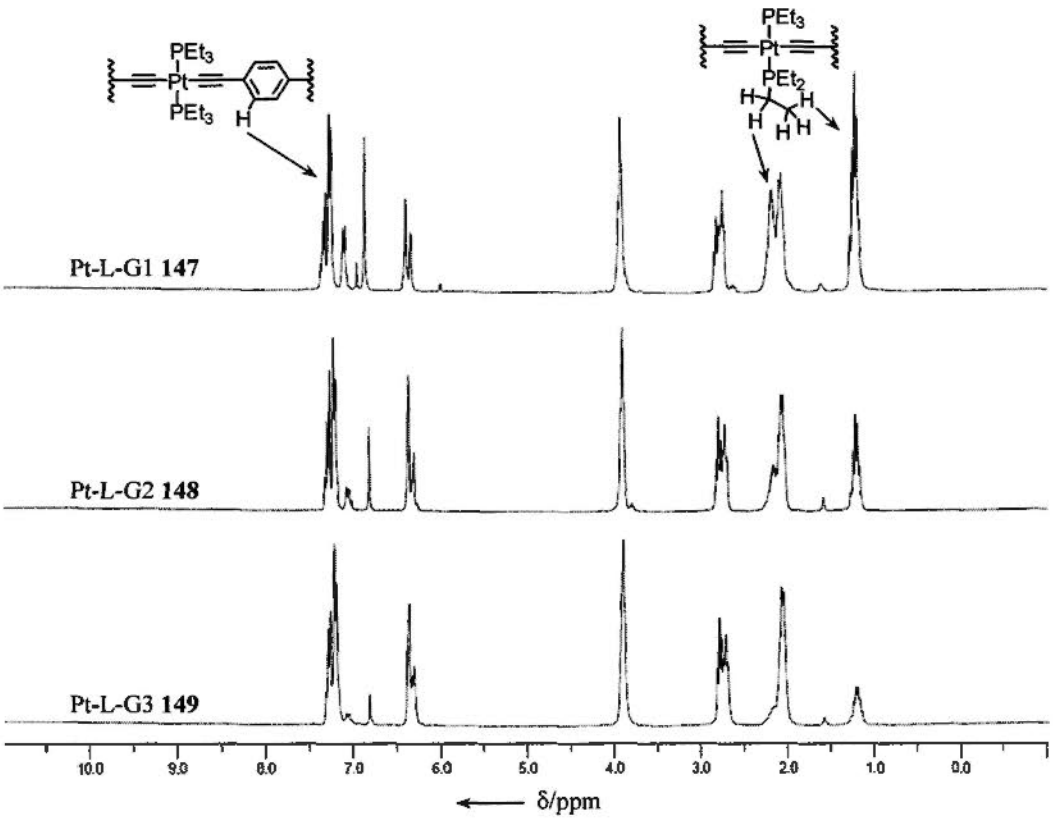


Figure 42. The stacked ^{1}H NMR (300 MHz, $CDCl_3$) spectra of poly(dendrimer)s Pt-L-Gn 147, 148 and 149.

Similar to the S-Gn series, the spectra of the Pt-L-Gn (n = 1–3) **147**, **148** and **149** poly(dendrimer)s (Figure 42) showed an upfield shift of the aromatic signals *ortho* to the functional 4-ethynylphenyl surface moieties, the disappearance of the acetylenic proton signals at δ 3.0 and the appearance of the signals of ethylphosphine ligands at δ 1.1–1.4 (PCH_2CH_3) and δ 2.1–2.4 (PCH_2).

3.4.2. ^{13}C NMR spectroscopy

3.4.2.1. The S-Gn Series

The ^{13}C NMR spectral features of the synthesized compounds were consistent with the proposed structures. For all dendrons and dendrimers, the aromatic carbon signals of the surface groups, branching units and central core were located at δ

100–160 and the benzylic carbon signals were found at δ 69–71. The dendritic esters, alcohols and bromides were characterized by the carbon signals at δ 166, δ 65 and δ 34 due to the carbonyl carbon (C=O), the carbon atom attaching to the hydroxyl group (CH_2OH) and the carbon atom bearing bromine atoms (CH_2Br) respectively. For the compounds with iodo surface groups, the signals of the aromatic carbon atom attached to the iodine atom directly was found at δ 94.

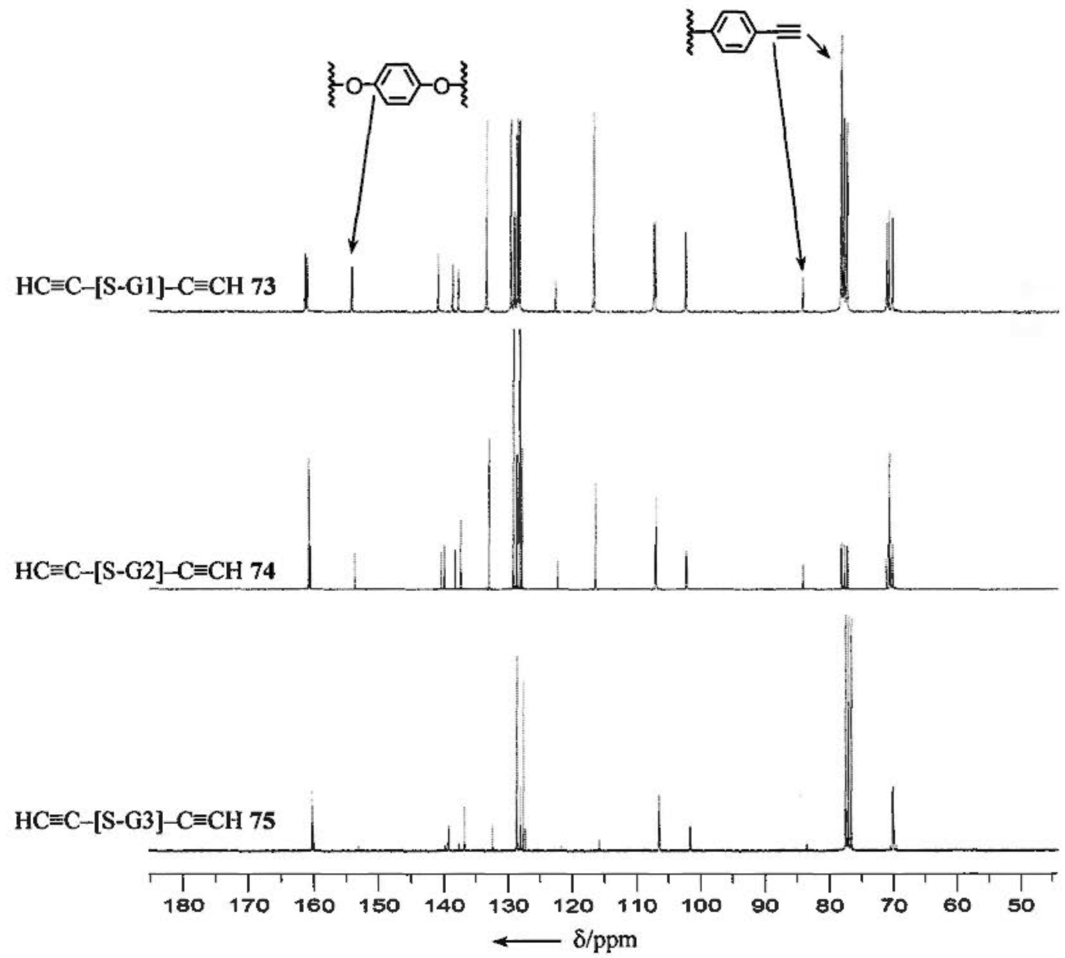


Figure 43. *The stacked ^{13}C NMR (75.5 MHz, $CDCl_3$) spectra of HC≡C–[S-Gn]–C≡CH 73–75.*

After coupling to the central core, the dendrimers were characterized by the signals at δ 153 due to the aromatic carbon of the hydroquinone core moiety. On the other hand, the acetylenic carbon signals were found at δ 95 and δ 105 for the TMSC≡C groups and at δ 77 and δ 84 for the HC≡C functionality. The ^{13}C NMR spectra of dendritic macromonomers HC≡C–[S-Gn]–C≡CH (n = 1–3) **73–75** showed that their molecular structures containing the hydroquinone core and the alkyne surface groups (Figure 43).

After copolymerization reactions, the terminal alkynes were attached to the platinum and therefore the carbon signals were downfield shifted to δ 108 [triplet (J_{C-P} = 14.3 Hz), C≡*C*Pt] and δ 109 (*C*≡CPt), but the corresponding ^{13}C signals of G3 dendrimer were very weak to be observed. Moreover the carbon signals of ethylphosphine ligands were also found at δ 8.4 (PCH_2CH_3) and δ 16.4 [quintet like (J ≈ 18 Hz), PCH_2] (Figure 44).

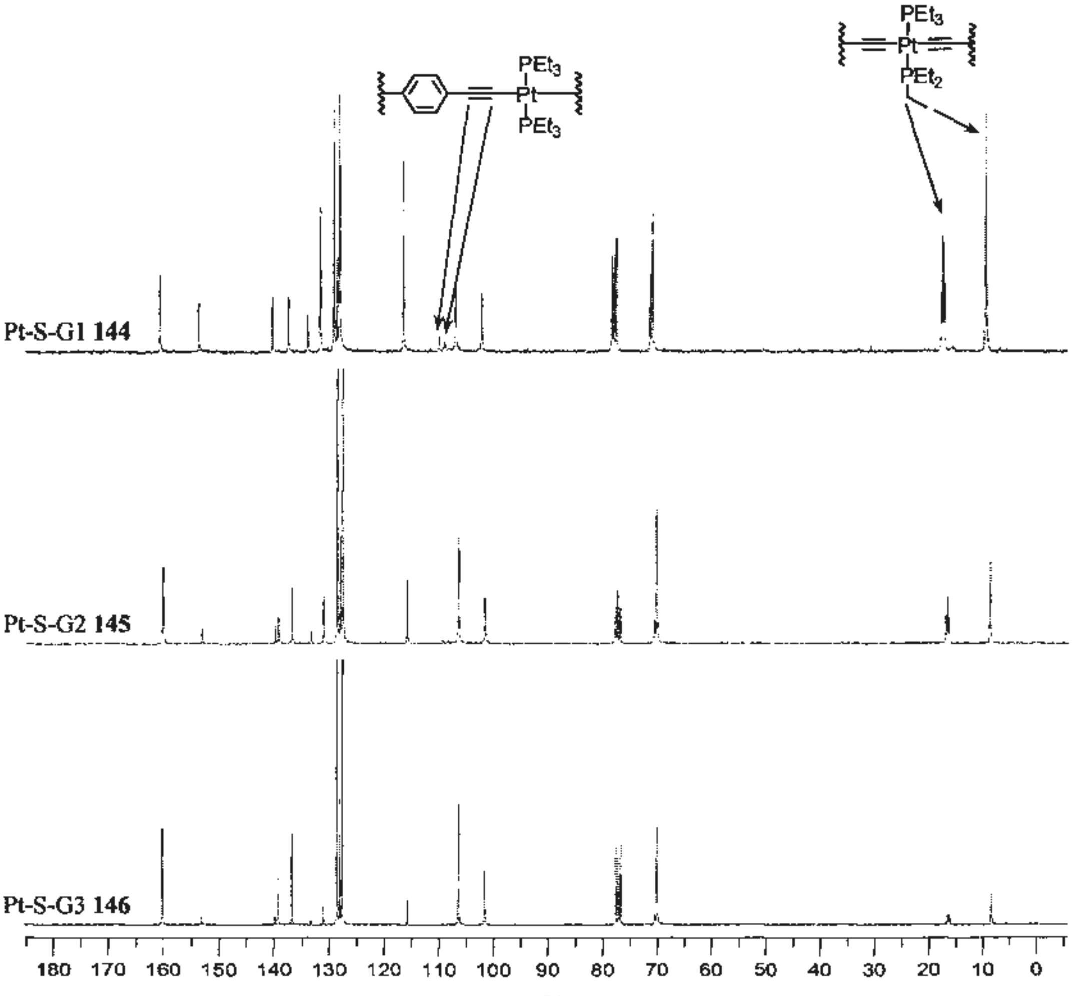


Figure 44. *The stacked ^{13}C NMR (75.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s Pt-S-Gn* ***144, 145*** *and* ***146.***

3.4.2.2. The L-Gn Series

The ^{13}C NMR spectra of compounds in S-Gn series were already investigated, similar trends were also observed for the L-Gn series, except for the presence of the aliphatic carbon signals at δ 30–33. Furthermore the signal of the aromatic carbon attached to the iodine atom was now found at δ 91 instead of δ 94. For the dendritic macromonomers HC≡C–[L-Gn]–C≡CH (n = 1–3) **76–78**, their ^{13}C spectra showed that the signals of central core were found at δ 153 and the characteristic alkyne signals (C≡C) were observed at δ 77 and δ 84, but the signal located at δ 77 was very weak for G2 and G3 dendrimers (Figure 45).

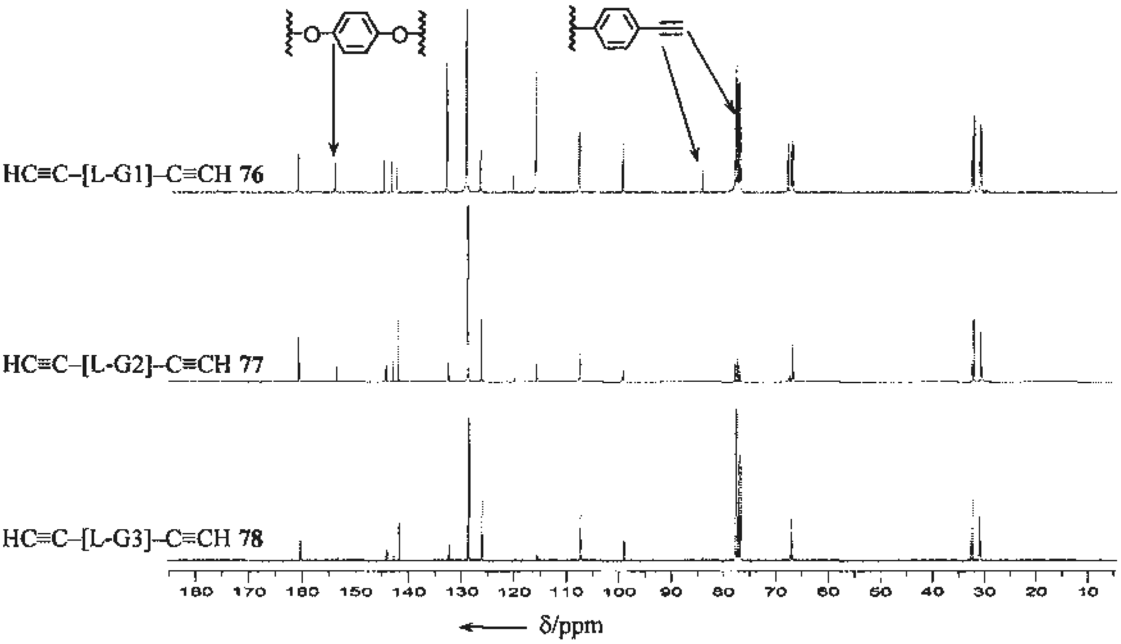


Figure 45. The stacked ^{13}C NMR (75.5 MHz, $CDCl_3$) spectra of HC≡C–[L-Gn]–C≡CH 76–78.

Similar to Pt-S-Gn, the poly(dendrimer)s Pt-L-Gn (n = 1–3) **147–149** would also be characterized by the acetylenic carbon signals at δ 107 (C≡*C*Pt) and δ 109 (*C*≡CPt), and the signals due to ethylphosphine ligands located at δ 8.4 (PCH_2CH_3) and δ 16 [quintet like ($J_{C\text{-}P}$ ≈ 18 Hz), PCH_2] (Figure 46). However the C≡*C*Pt signal of Pt-L-G3 **149** was too weak to be observed.

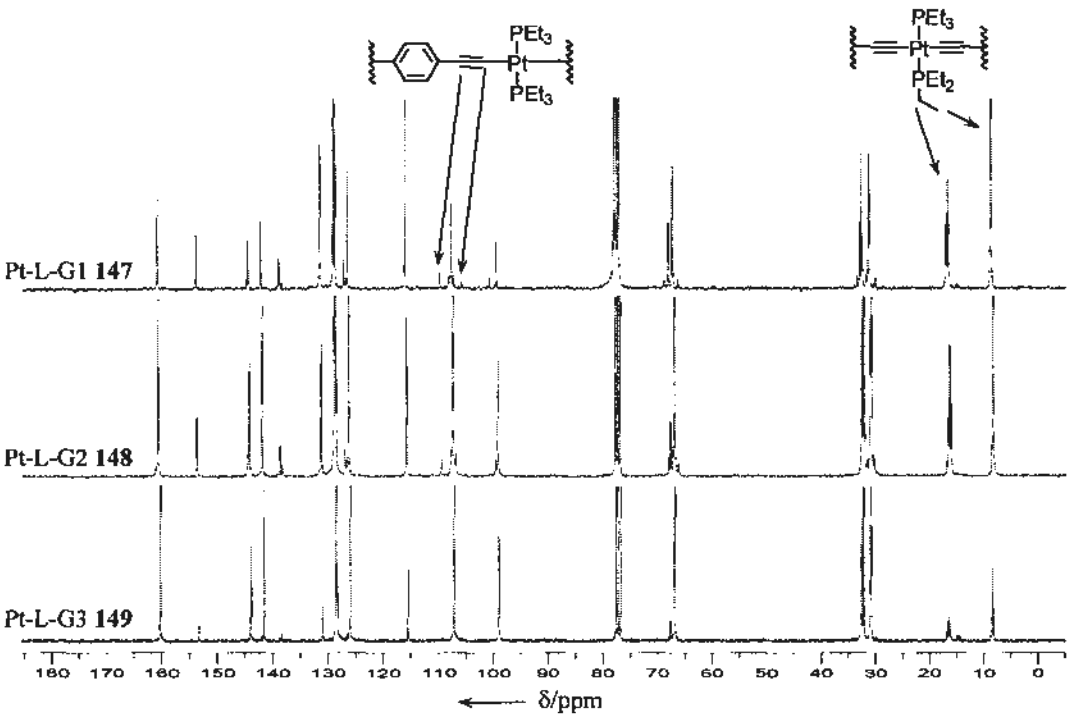


Figure 46. The stacked ^{13}C NMR (75.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s Pt-L-Gn 147, 148 and 149.

3.4.3. ^{31}P NMR spectroscopy

All the poly(dendrimer)s were characterized by ^{31}P NMR spectroscopy due to the presence of ethylphosphine ligands of the platinum linker. The spectra of both Pt-S-Gn (Figure 47) and Pt-L-Gn (Figure 48) exhibited one major ^{31}P signal located at δ 11.1 with two ^{195}Pt satellite signals ($^{1}J_{Pt-P} \approx 2360$ Hz) of one sixth intensity. Moreover two minor peaks were also observed at δ 8.6 and δ 14.9 for the G3 polymers and this was probably due to signals arising from the oligomers because of the low efficiency of polymerization for the G3 monomers.

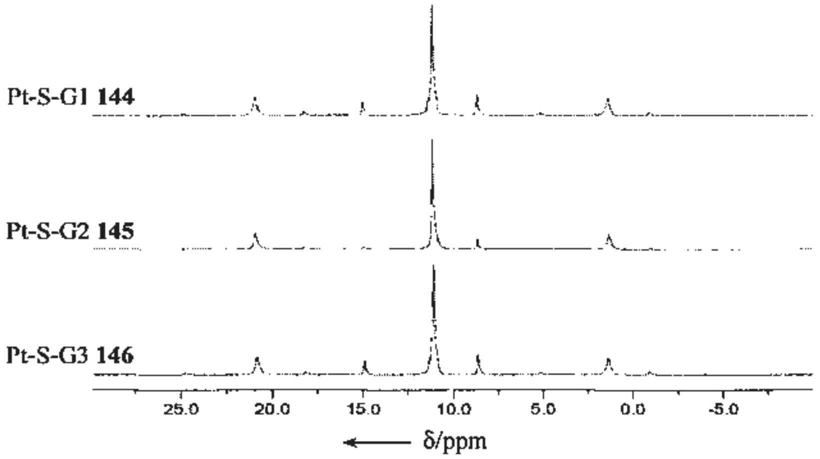


Figure 47. *The stacked ^{31}P NMR (121.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s Pt-S-Gn **144**, **145** and **146**.*

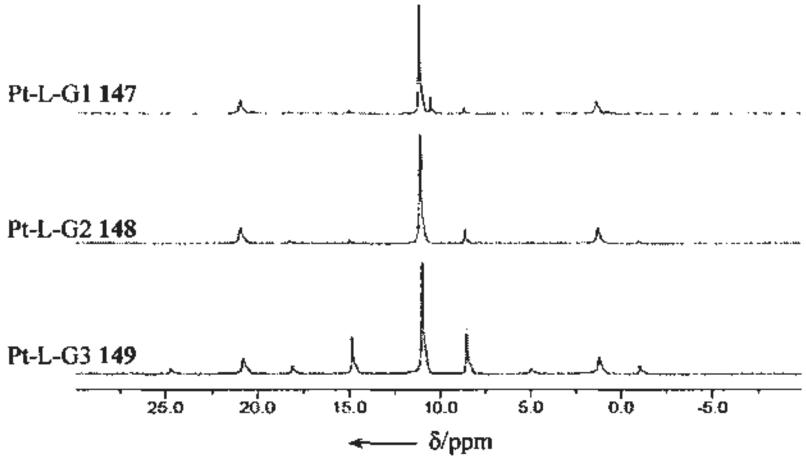


Figure 48. *The stacked ^{31}P NMR (121.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s Pt-L-Gn **147**, **148** and **149**.*

3.4.4. GPC analysis

S-Gn Series	**Theoretical MW (g mol^{-1})**	M_n **(g mol^{-1})**	M_w **(g mol^{-1})**	**PDI = M_w/M_n**
S-Gn Dendrons				
hemi-S-G1-ester **86**	272.30	311	313	1.01
S-G1-ester **85**	362.42	337	339	1.01
S-G1-OH **90**	320.38	334	337	1.01
S-G1-Br **91**	383.28	301	303	1.01
I-S-G1-ester **87**	488.31	375	379	1.01
I-S-G1-OH **88**	446.28	370	374	1.01
I-S-G1-Br **89**	509.17	335	338	1.01
hemi-S-G2-ester **92**	484.54	567	572	1.01
S-G2-ester **96**	786.91	874	883	1.01
S-G2-OH **97**	744.87	873	883	1.01
S-G2-Br **98**	807.77	834	841	1.01
I-S-G2-ester **93**	912.80	921	930	1.01
I-S-G2-OH **94**	870.77	910	925	1.02
I-S-G2-Br **95**	933.66	873	887	1.02
hemi-S-G3-ester **99**	909.03	1093	1114	1.02
I-S-G3-ester **100**	1761.78	1966	1990	1.01
I-S-G3-OH **101**	1719.74	1969	1988	1.01
I-S-G3-Br **102**	1782.64	1902	1958	1.03
S-Gn Dendrmers				
I–[S-G1]–I **104**	966.64	886	893	1.01
TMSC≡C–[S-G1]–C≡CTMS **107**	907.25	1402	1415	1.01
HC≡C–[S-G1]–C≡CH **73**	762.89	1025	1035	1.01
I–[S-G2]–I **105**	1815.61	1991	2019	1.01
TMSC≡C–[S-G2]–C≡CTMS **108**	1756.22	2433	2458	1.01
HC≡C–[S-G2]–C≡CH **74**	1611.86	2146	2166	1.01
I–[S-G3]–I **106**	3513.56	3807	3843	1.01
TMSC≡C–[S-G3]–C≡CTMS **109**	3454.18	4172	4296	1.03
HC≡C–[S-G3]–C≡CH **75**	3309.81	3499	3598	1.03

Table 2. *GPC analysis of S-Gn dendrons and dendrimers.*

L-Gn Series	**Theoretical MW (g mol^{-1})**	**M_n (g mol^{-1})**	**M_w (g mol^{-1})**	**PDI = M_w/M_n**
L-Gn Dendrons				
hemi-L-G1-ester **120**	314.38	382	385	1.01
L-G1-ester **124**	432.55	462	467	1.01
L-G1-OH **125**	404.54	492	496	1.01
L-G1-Br **126**	467.44	444	448	1.01
I-L-G1-ester **121**	558.45	500	505	1.01
I-L-G1-OH **122**	530.44	525	529	1.01
I-L-G1-Br **123**	593.33	477	481	1.01
hemi-L-G2-ester **127**	582.73	746	756	1.01
L-G2-ester **131**	969.25	1227	1245	1.01
L-G2-OH **132**	941.24	1223	1246	1.02
L-G2-Br **133**	1004.14	1195	1205	1.01
I-L-G2-ester **128**	1095.15	1247	1266	1.02
I-L-G2-OH **129**	1067.14	1285	1298	1.01
I-L-G2-Br **130**	1130.03	1250	1262	1.01
hemi-L-G2-ester **134**	1119.43	1508	1530	1.01
I-L-G3-ester **135**	2168.55	2569	2674	1.04
I-L-G3-OH **136**	2140.54	2617	2720	1.04
I-L-G3-I **137**	2250.44	2706	2740	1.01
L-Gn Dendrimers				
I–[L-G1]–I **138**	1134.96	1238	1248	1.01
TMSC≡C–[L-G1]–C≡CTMS **141**	1075.57	1787	1808	1.01
HC≡C–[L-G1]–C≡CH **76**	931.21	1390	1404	1.01
I–[L-G2]–I **139**	2208.36	2784	2814	1.01
TMSC≡C–[L-G2]–C≡CTMS **142**	2148.97	3272	3329	1.02
HCC–[L-G2]–CCH **77**	2004.61	2941	2987	1.02
I–[L-G3]–I **140**	4355.16	5416	5484	1.01
TMSC≡C–[L-G3]–C≡CTMS **143**	4295.77	5839	5966	1.02
HC≡C–[L-G3]–C≡CH **78**	4151.41	4924	5029	1.01

Table 3. *GPC analysis of L-Gn dendrons and dendrimers.*

All the dendrons, dendrimers and poly(dendrimer)s were characterized by GPC analysis using polystyrene as the standards. The GPC studies on the dendrons and dendrimers were aimed at checking the purity of the compounds that could be reflected by the polydispersity index (PDI). Generally, when the PDI value was smaller than or equals to 1.03, the dendrons or dendrimers could be considered as monodispersed and with high homogeneity. The determined PDI values of dendrons and dendrimers were reported in Table 2 (S-Gn series) and Table 3 (L-Gn series).

As nearly all the measured PDI values are ≤ 1.03, the dendrons and dendrimers synthesized were monodispersed. Besides the relative hydrodynamic volume of dendrons (all hemi-Gn-esters were excluded) and dendrimers could be compared by the GPC cluster plot of retention time versus theoretical molecular weight (MW) (Figure 49). There were several findings observed from the graphs. First, the hydrodynamic volumes of dendrimers were larger than that of dendrons of the same generation (Figure 49a *vs* Figure 49b). Second, the hydrodynamic volumes of dendrons (or dendrimers) having the same generation number, irrespective of the focal point and surface functional groups, were very close to each other. Third, the size of dendrons (or dendrimers) increased as the generation increased. Fourth, the L-Gn dendrons (or dendrimers) had a shorter retention time than the corresponding S-Gn dendrons (or dendrimers) and therefore compounds in the L-Gn series had a slightly larger hydrodynamic volume.

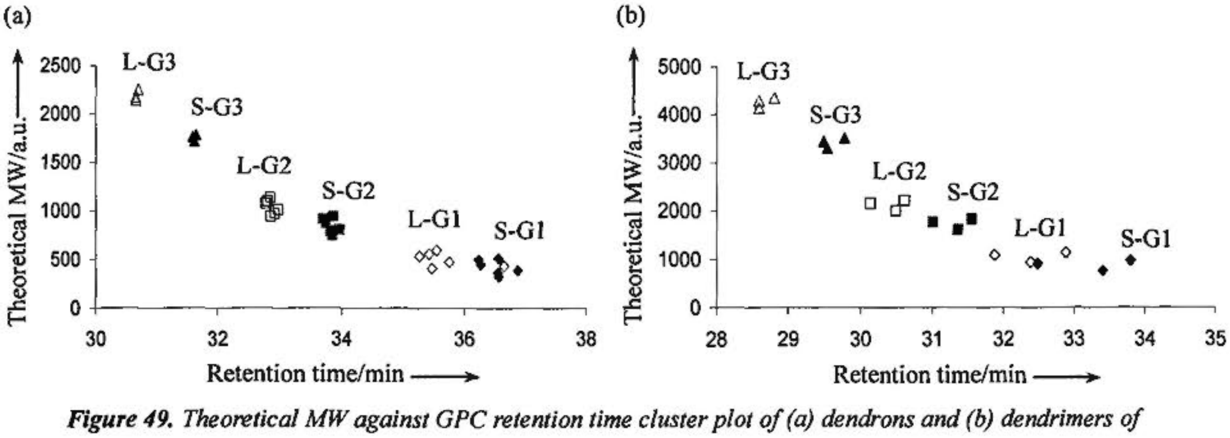


Figure 49. *Theoretical MW against GPC retention time cluster plot of (a) dendrons and (b) dendrimers of various generations.*

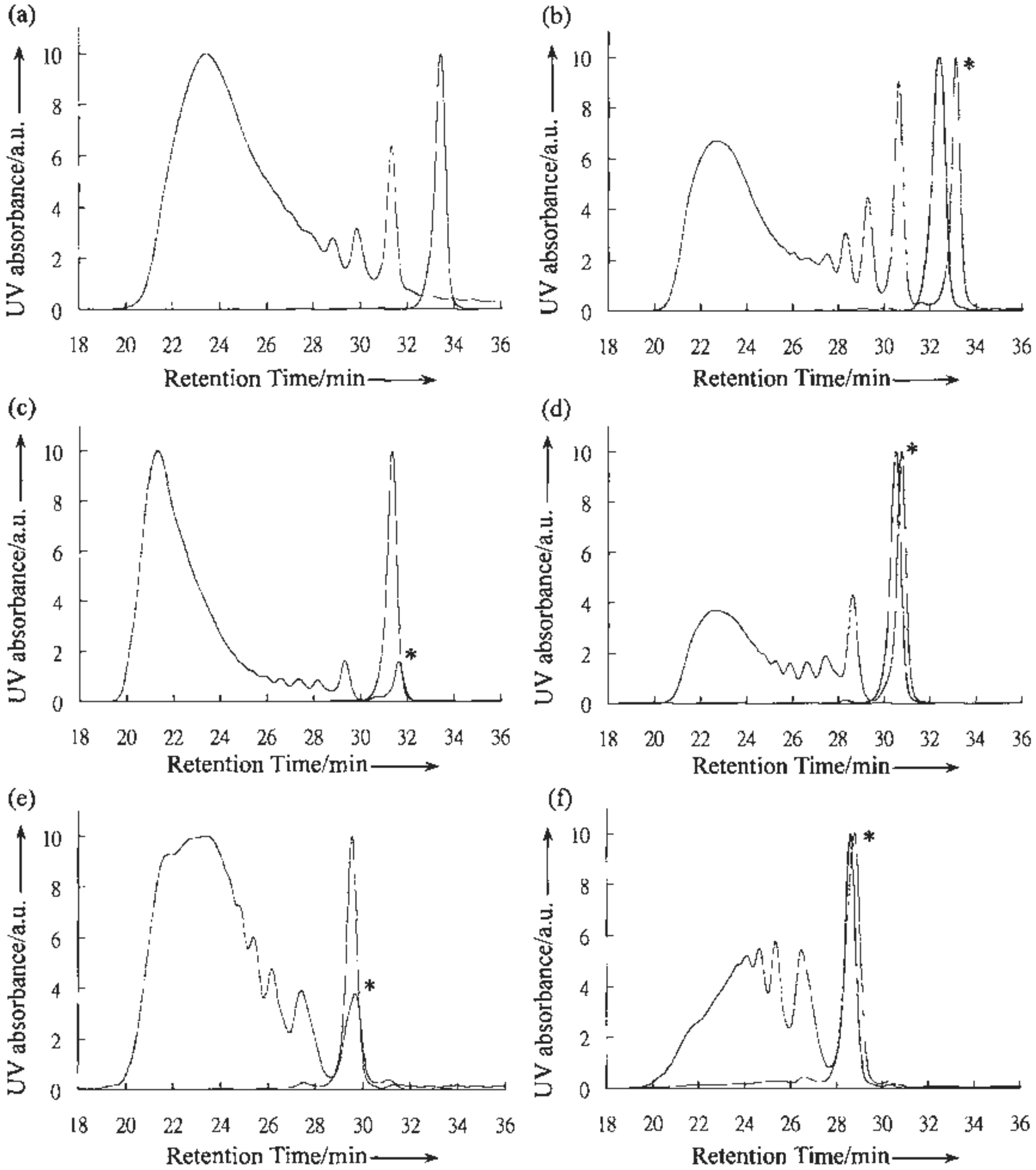


*Figure 50. Stacked GPC chromatograms of poly(dendrimer)s (blue lines) with their corresponding dendritic monomers (red lines) for (a) Pt-S-G1 **144**, (b) Pt-L-G1 **147**, (c) Pt-S-G2 **145**, (d) Pt-L-G2 **148**, (e) Pt-S-G3 **146** and (f) Pt-L-G3 **149**. The peaks labeled with an asterisk are the cyclic monomer peaks.*

Poly(dendrimer)s	$M_w \times 10^3$	DP	PDI	% of LMW oligomers[a]
Pt-S-G1 **144**	40	34	1.9	1
Pt-L-G1 **147**	33	39	1.9	2
Pt-S-G2 **145**	122	60	2.3	<0.2
Pt-L-G2 **148**	55	22	1.6	4
Pt-S-G3 **146**	79	21	1.8	3
Pt-L-G3 **149**	86	19	1.6	14

***Table 4.** GPC data of poly(dendrimer)s **144–149** in THF at 40 °C.* [a] *Weight% up to pentamer.*

The molecular weights and distributions of all the poly(dendrimer)s **144–149** were analyzed by GPC in order to investigate how the polymerization results (DP values) were affected by the various dendritic macromonomers with different structural flexibility (S-Gn *vs* L-Gn). The GPC chromatograms of poly(dendrimer)s **144–149** (blue lines) were plotted together with those of their corresponding dendritic macromonomers (red lines) (Figure 50). The weight-average molecular weights (M_w) and the degree of polymerization (DP) values were also calculated from the chromatograms (Table 4). Since polystyrenes were used as the standards for the GPC analysis, the calculated M_w values were known to be underestimated[92] by a factor of 1.5–14 for poly(dendrimer)s.[73] However, the relative polymerization efficiencies of the two series (S-Gn *vs* L-Gn) could still be compared. As the GPC measurements were carried out in highly diluted solutions in THF at elevated temperature (40 °C), polymer aggregation should be avoided and hence the calculated M_w and DP values should reflect the polymer chains and not their aggregates.

There were several interesting findings noted. First, the polymerization efficiency of dendrimers within the same series was dependent on the dendrimer generation. The DP values of G3 polymers was smaller than that of G1 and G2 polymers because the linking of individual G3 dendritic macromonomers was inhibited by the sterically congested environment and the lower functional surface density of the acetylenic groups (decreased from 1/8 of the G3 dendrimers to 1/2 of the G1 dendrimers). Second, the DP values of the poly(dendrimer)s were very similar to those obtained from our previous results involving C≡CH surface groups extended by a 10-C spacer on the surface (See Figure 33 in Chapter 2).[73] It was therefore suggested that dendrimers with the "longer arms" did not have any obvious advantage on increasing the polymerization efficiency. Third, significant amounts (0.2–14% by weight, calculated up to the pentamer) of low molecular weight (LMW) oligomers were found. These oligomers were suspected as the partially reacted open-chain oligomers with free acetylene units or Pt-Cl end group initially. However, Figures 50b–50f showed the presence of a peak (labeled with an asterisk) that had a longer retention time than that of the corresponding dendritic macromonomer. Hence, this must be a compound that had a smaller hydrodynamic volume than the dendritic monomer. This species was then separated from the rest of the polymeric mixture by preparative GPC and later confirmed as the cyclic monomer by mass spectrometry (MS) (See Section 3.4.5). Fourth, the Pt-S-Gn polymers contained fewer amounts of LMW oligomers than the Pt-L-Gn polymers. This result was more significant for the G3 compounds, i.e. Pt-S-G3 **146** only contained 3% of LMW oligomers, and in contrast, Pt-L-G3 **149** contained 14%. As a result, for the polymerization of dendritic macromonomers (dendrimer beads) with two surface functionalities at a distant, the structural flexibility was an important factor in affecting the polymerization efficiency. Dendrimers with a higher structural flexibility were more prone to undergo cyclization rather than linear propagation. This was a significant finding as this had not been reported before.

3.4.5. Mass spectrometry (MS)

All the dendrons and dendrimers were characterized by FAB, EI, ESI or MALDI-TOF MS depending on their molecular masses. However, there were two significant uses of ESI and MALDI-TOF MS in this project. The first one was to confirm the absence of de-iodination in the DIBAL-H reduction reactions. As an example, I-S-G3-ester **100** was firstly reduced by DIBAL-H to the corresponding alcohol I-S-G3-OH **101** and then converted into I-S-G3-Br **102**. The iodo surface group could be potentially removed during DIBAL-H reduction, then a small amount of S-G3-Br could be formed in addition to I-S-G3-Br **102** and a monofunctional dendrimer chain stopper could be generated. As mentioned before, the polymerization efficiency would be greatly affected when small amounts of monofuctional dendrimers were present. Therefore the absence of de-iodination must be confirmed. Fortunately, in the MS spectrum of I-S-G3-Br **102**, the major signals were the molecular peaks due to $[M]^+$, $[M+H]^+$, $[M+Na]^+$ or $[M+K]^+$ of the dendron having the iodo functional surface groups (Figure 51). No peak due to the corresponding de-iodinated dendron was observed.

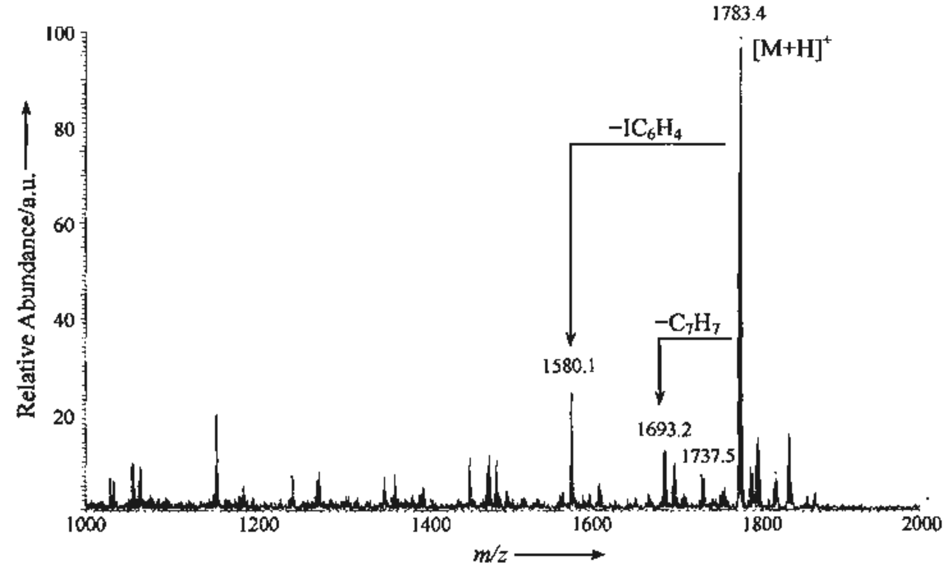


Figure 51. The ESI MS of I-S-G3-Br 102.

Second, the aforementioned cyclic monomers formed during the polymerization reactions were also characterized by MALDI-TOF MS (Figure 52). The major peak signals at *m/z* 1361.6254, 2433.2043 and 4581.4136 were due the cyclic monomers of Pt-L-G1, Pt-L-G2 and Pt-L-G3 respectively. These results were consistent with their theoretical molecular masses of 1361.6269 $[M+H]^+$, 2433.2053 $[M]^+$ and 4581.3901 $[M+H]^+$, respectively.

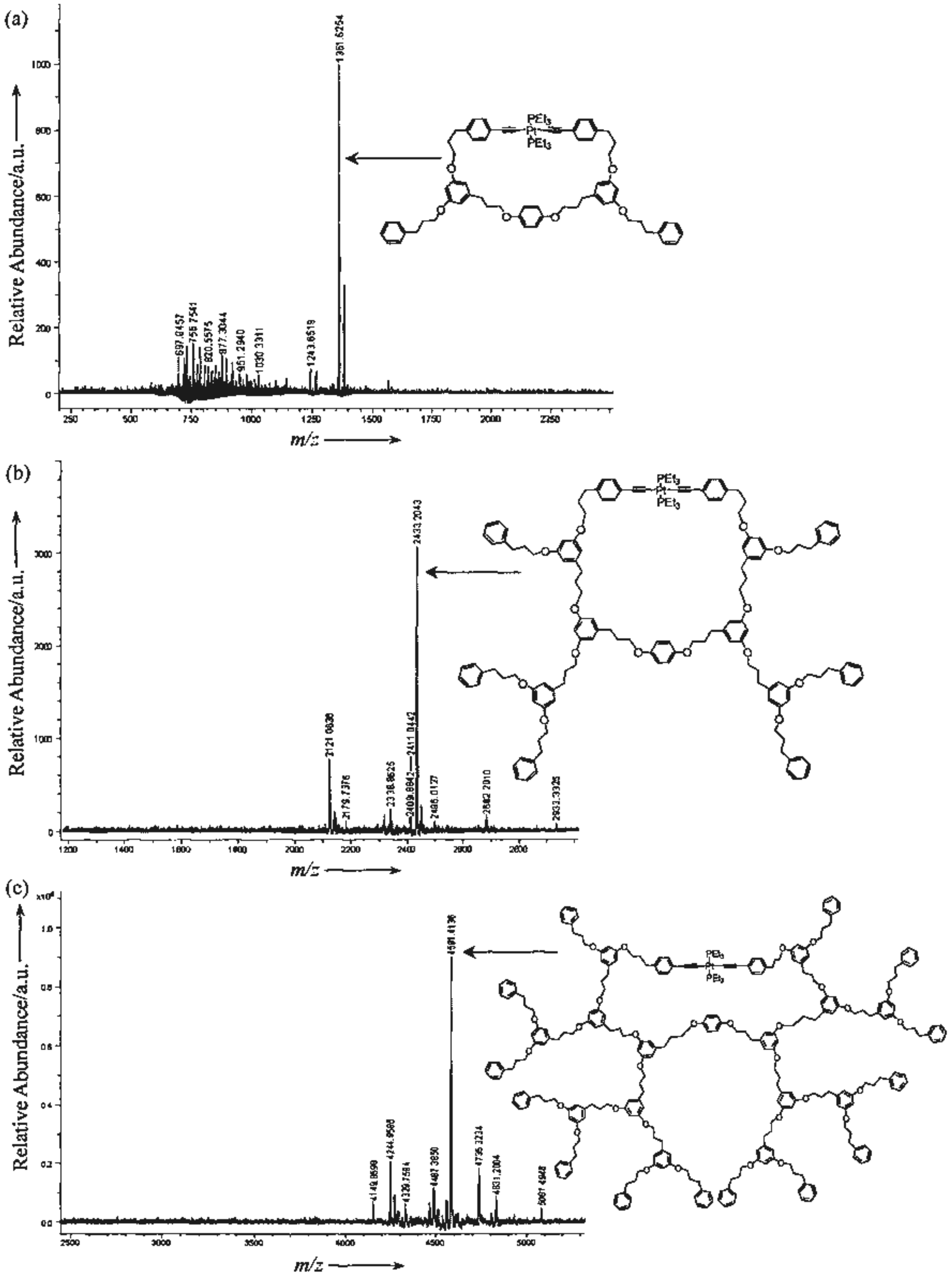


Figure 52. MS spectra and structures of cyclic monomers for (a) Pt-L-G1, (b) Pt-L-G2 and (c) Pt-L-G3.

3.4.6. Laser light scattering (LLS)

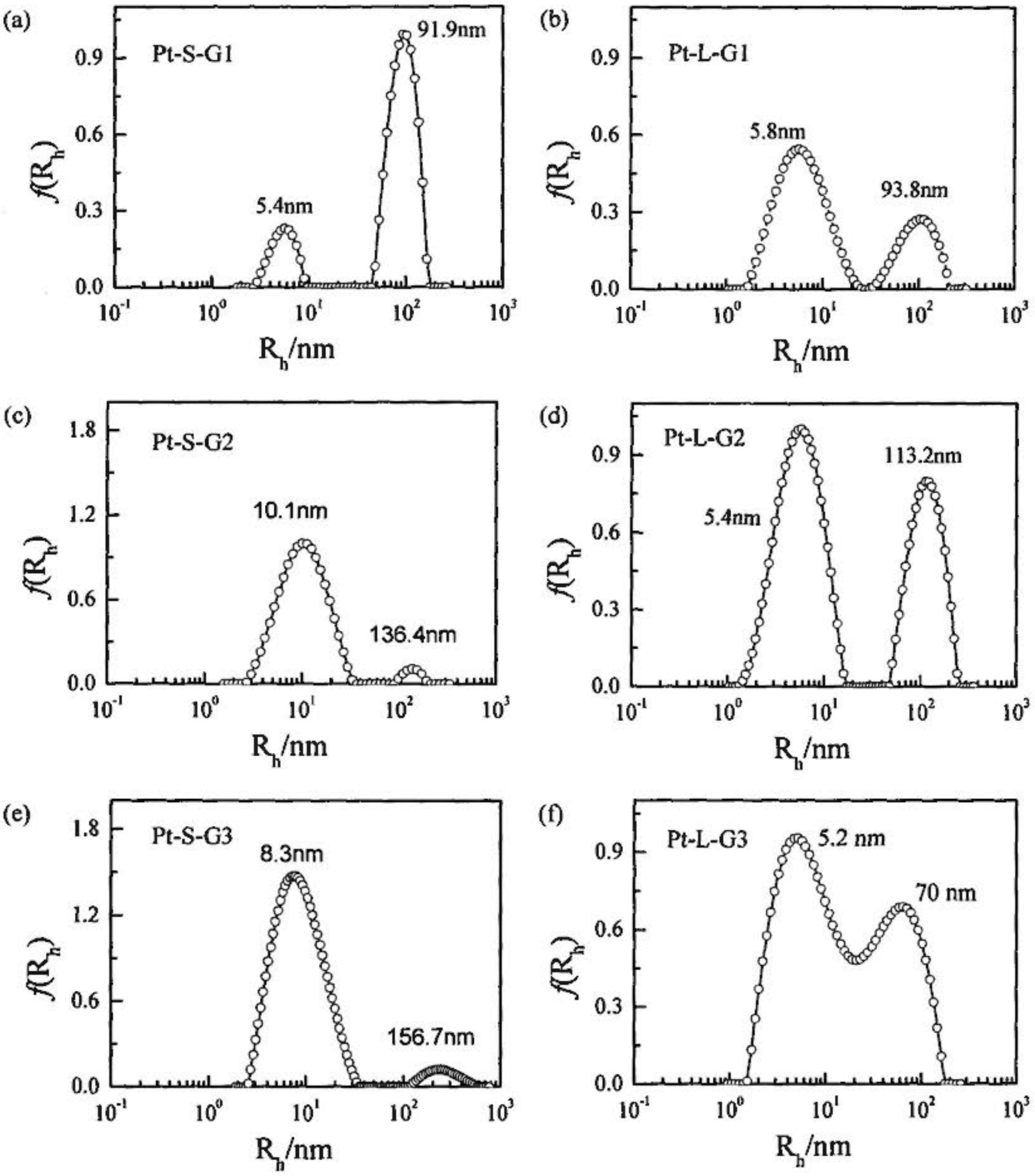


Figure 53. *The hydrodynamic radius distribution $f(R_h)$ by dynamic LLS of (a) Pt-S-G1* ***144*** *at 5.6×10^{-4} g mL^{-1}, (b) Pt-L-G1* ***147*** *at 5.8×10^{-4} g mL^{-1}, (c) Pt-S-G2* ***145*** *at 1.0×10^{-3} g mL^{-1}, (d) Pt-L-G2* ***148*** *at 4.67×10^{-3} g mL^{-1}, (e) Pt-S-G3* ***146*** *at 8.4×10^{-4} g mL^{-1} and (f) Pt-L-G3* ***149*** *at 6.0×10^{-4} g mL^{-1}.*

All the poly(dendrimer)s **144–149** were investigated by LLS in order to determine their absolute molecular weights. The measurements were carried out in THF at 25 °C and the hydrodynamic radii (R_h) were determined by dynamic LLS (Figure 53 and Table 5). The presence of two peaks indicated that the samples contained two different species. The peak with a smaller R_h value was due to individual polymers and the one with a larger R_h value was due to the polymer aggregates. In this case, the M_w values obtained were just the average M_w values of these two species, therefore the absolute M_w values of poly(dendrimer)s could not be determined. However, the intensity of the aggregate peaks for Pt-S-G2 (Figure 53c) and Pt-S-G3 (Figure 53e) were much lower than that of the non-aggregate one. As a result, these two determined values could be considered as the M_w values of the corresponding non-aggregated poly(dendrimer)s (Pt-S-G2 and Pt-S-G3) and these values matched very well with the results obtained from GPC analyses (Table 4).

Poly(dendrimer)s	R_h	M_w (g mol^{-1})
Pt-S-G1 **144**	5.4 nm, 92 nm	24.0×10^4
Pt-L-G1 **147**	5.8 nm, 94 nm	8.8×10^4
Pt-S-G2 **145**	10.1 nm, 136 nm	7.1×10^4 [a]
Pt-L-G2 **148**	5.4 nm, 113 nm	2.5×10^4
Pt-S-G3 **146**	8.3 nm, 156 nm	6.4×10^4 [a]
Pt-L-G3 **149**	5.2 nm, 70 nm	3.9×10^4

Table 5. *The hydrodynamic radius (R_h) and M_w of the mixture of poly(dendrimer)s **144–149** and their aggregates.* [a] *M_w values could be considered due to the non-aggregate poly(dendrimer)s.*

3.5. Summary

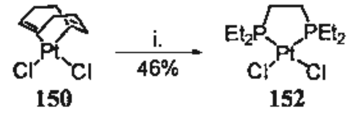
Two series of dendrimers (G1–G3) with different structural flexibilities (S-Gn *vs* L-Gn) were synthesized, then they acted as monomers and copolymerized with a platinum linker—*trans*-[$Pt(PEt_3)_2Cl_2$] **65** to form the corresponding poly(dendrimer)s. The polymerization efficiency was highly dependent on the structural flexibility of the dendritic macromonomers. Dendrimers with higher structural flexibility (L-Gn series), as compared to the more rigid one (S-Gn series), were more prone to undergo cyclization and thus led to a decrease of the polymerization efficiency. In the next chapter, we will focus on how the length and the geometry of the platinum linkers would affect the polymerization results.

Chapter 4

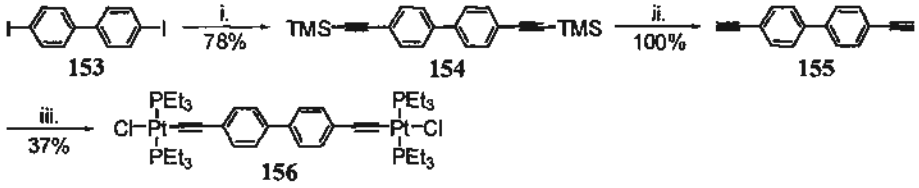
Synthesis of Poly(dendrimer)s by Using Different Platinum Linkers

In the last chapter, the effect of the structural flexibility of dendritic macromonomers on polymerization efficiencies for the formation of poly(dendrimer)s was already investigated. Apart from this factor, the other key factors that can potentially affect the polymerization efficiencies are the length and geometry of the platinum linker. In this chapter, this study by conducting the copolymerizations with two different types of linkers will be disclosed. The first was a *cis*-platinum **152**, and the second was an elongated rod-liked platinum linker **156**.

4.1. Synthesis of platinum linkers



Scheme 17. . *Reagents and conditions: i.* $Et_2PCH_2CH_2PEt_2$ *151,* CH_2Cl_2*, −10 °C, 10 min.*

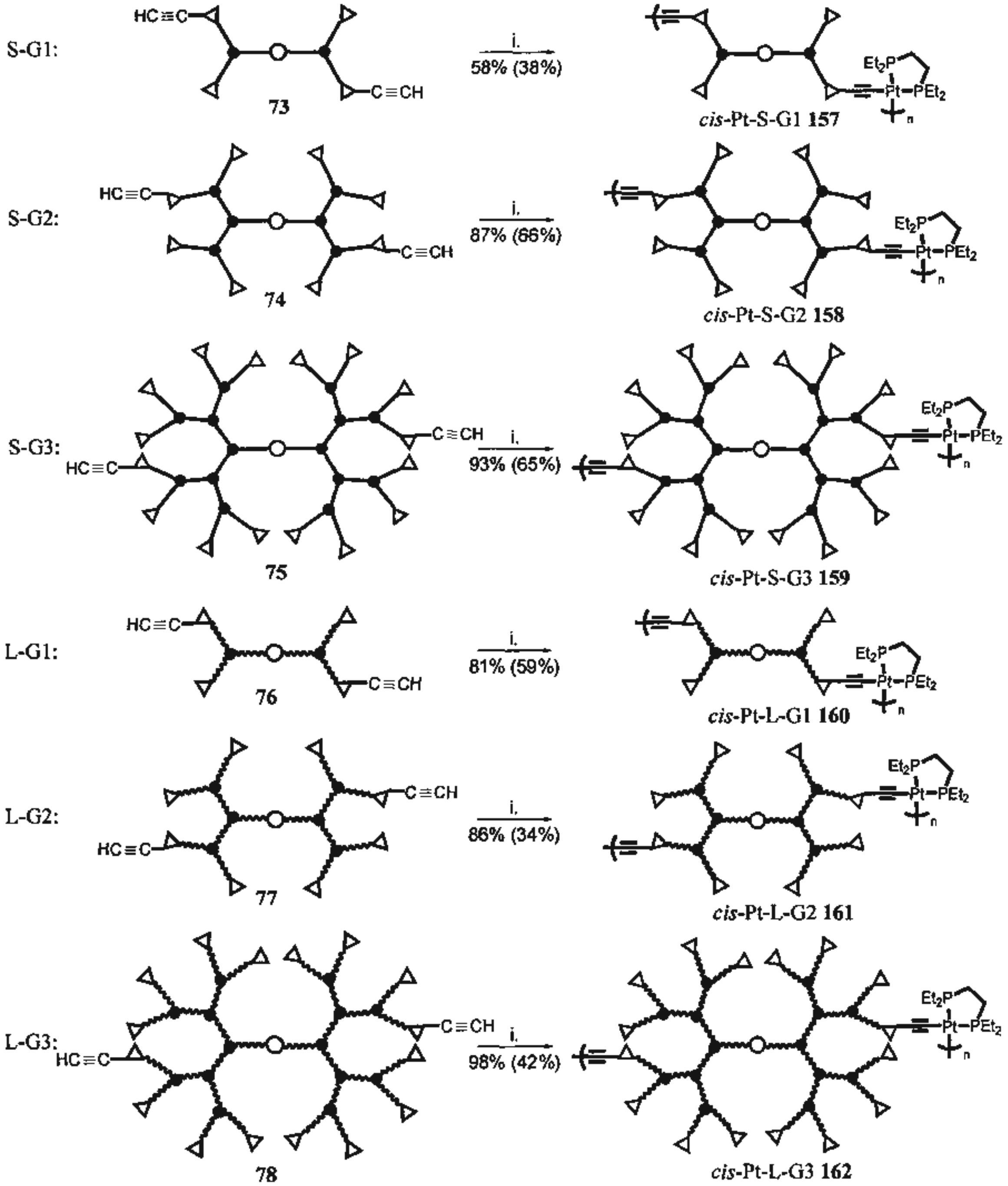


Scheme 18. . *Reagents and conditions: i. TMSC≡CH,* $Pd(PPh_3)_2Cl_2$*, CuI,* Et_3N*, THF, 25 °C, 12 h; ii.* K_2CO_3*, THF/MeOH (v/v = 1/1), 25 °C, 2 h; iii. trans-*$[Pt(PEt_3)_2Cl_2]$ *65 (2.2 equiv.), CuCl,* $^iPr_2NH$*, toluene, 100 °C, 5 h.*

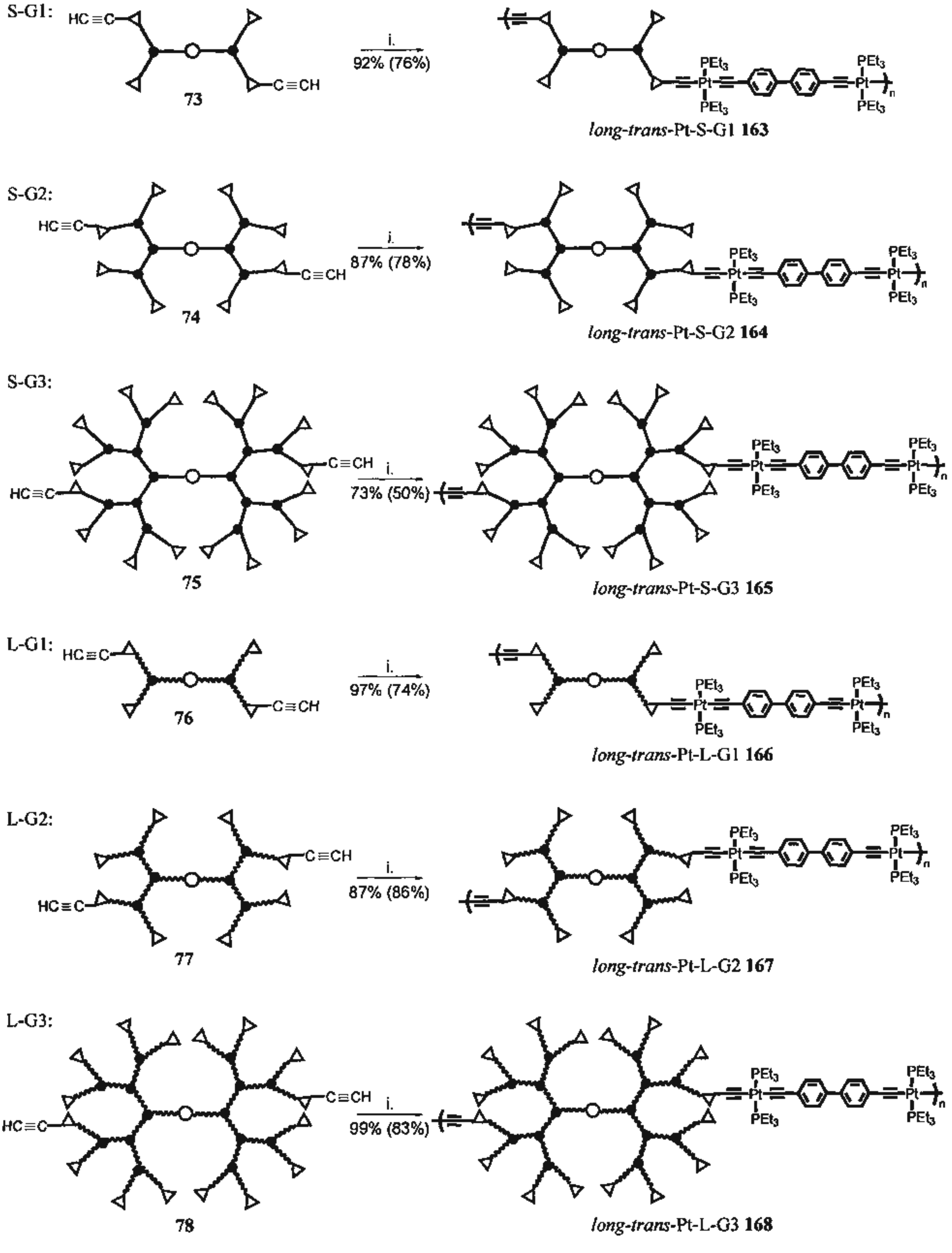
Dichloro(1,5-cyclooctadiene)platinum(II) **150** was subjected to ligand exchange reaction with a bidentate phosphine ligand—$Et_2PCH_2CH_2PEt_2$ **151** to afford the *cis*-platinum linker **152** in 46% yield (Scheme 17).[93] On the other hand, 4,4'-diiodobiphenyl **153** was coupled to trimethylsilylacetylene (TMSC≡CH) under

Sonogashira reaction conditions, followed by removal of the TMS group and ligand exchange with *trans*-[Pt(PEt$_3$)$_2$Cl$_2$] **65** to obtain the *long-trans*-platinum linker **160** in overall 29% yield (Scheme 18).[74]

4.2. Preparation of poly(dendrimer)s by using different platinum linkers



Scheme 19. *Reagents and conditions: i. cis-platinum linker 152 (1.0 equiv.), CuI, CHCl$_3$/^{i}Pr$_2$NH (v/v = 1/1). 40 °C in sealed tube. 2 d.*



Scheme 20. *Reagents and conditions: i. long-trans-platinum linker 156 (1.0 equiv.), CuI, $CHCl_3$/$^iPr_2NH$ (v/v = 1/1), 40 °C in sealed tube, 2 d.*

The dendritic macromonomers (HC≡C–[S-Gn]–C≡CH **73–75**, and HC≡C–[L-Gn]–C≡CH **76–78** where n = 1–3) were then subjected to the polymerization reactions with the two platinum linkers **152** (Scheme 19) and **156** (Scheme 20). In order to investigate the structural effect (length and geometry) of the platinum linker on the formation of poly(dendrimer)s, all reaction conditions were identical to those involving *trans*-$[Pt(PEt_3)_2Cl_2]$ **65** (Scheme 16 in chapter 3). The concentration of dendritic macromonomers used in the polymerization was 8.7 mM and the reactions proceeded in $CHCl_3/^{i}Pr_2NH$ (*v*/*v* = 1/1) at 40 °C for 2 days in the presence of CuI. After polymerization, the reaction mixtures were passed through a short pad of alumina to remove the copper salt and then purified by precipitation in methanol. GPC analyses were performed before precipitation in order to reflect the full molecular weight distribution. The precipitated poly(dendrimer)s were then characterized by NMR spectroscopy.

For the polymerization reactions with *cis*-platinum linker **152**, *cis*-Pt-S-Gn **157–159** and *cis*-Pt-L-Gn **160–162** (n = 1–3) were obtained in 58–98% yields and the yields after precipitation were 34–66%. Similarly, *long-trans*-Pt-S-Gn **163–165** and *long-trans*-Pt-L-Gn **166–168** (n = 1–3) were afforded in 73–99% (50–86% after precipitation) by copolymerizing with *long-trans*-platinum linker **156**.

4.3. Characterization

The poly(dendrimer)s **157–168** were characterized by ^{1}H, ^{13}C and ^{31}P NMR spectroscopy and the polymerization efficiencies were studied by GPC analysis. Since the poly(dendrimer)s tended to form aggregates in relatively concentrated solutions (See Section 3.4.6), their absolute molecular weights were therefore not determined by laser light scattering technique.

4.3.1. ^{1}H NMR spectroscopy

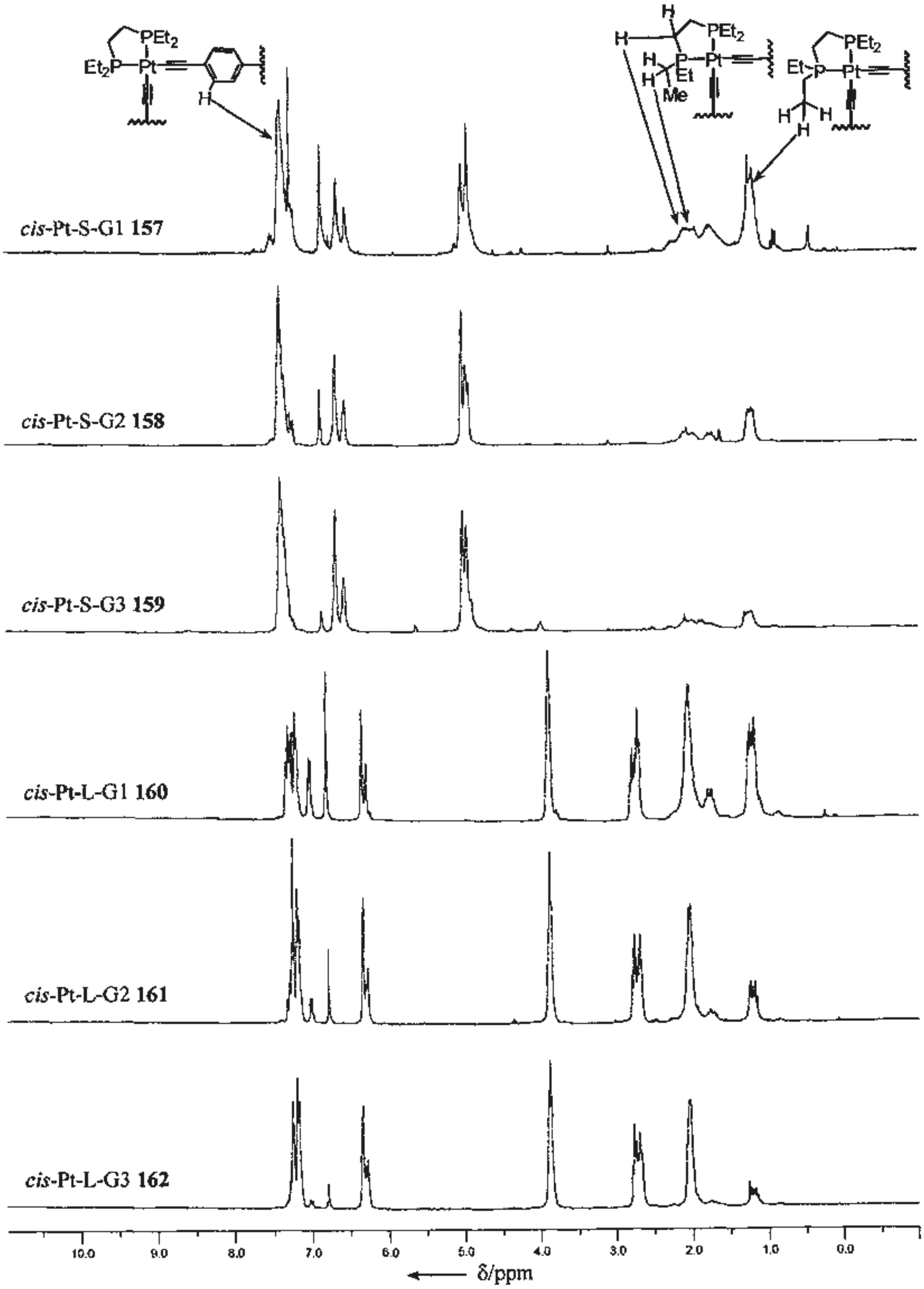


Figure 54. *The stacked ^{1}H NMR (300 MHz, CDCl$_3$) spectra of poly(dendrimer)s cis-Pt-S-Gn **157–159** and cis-Pt-L-Gn **160–162**.*

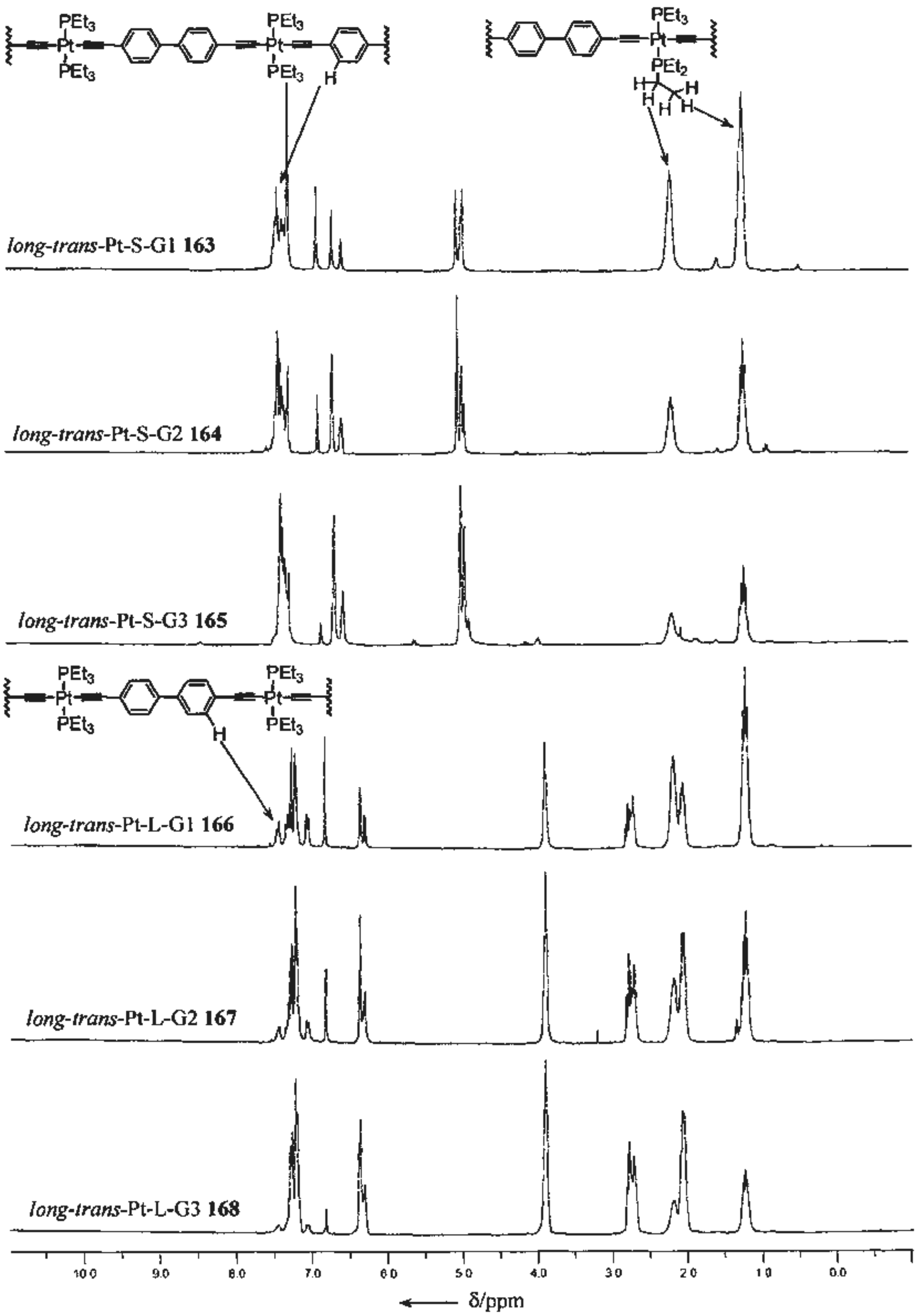


Figure 55. *The stacked ^{1}H NMR (300 MHz, $CDCl_3$) spectra of poly(dendrimer)s long-trans-Pt-S-Gn 163–165 and long-trans-Pt-L-Gn 166–168.*

The ^{1}H NMR spectra of *cis*-Pt-S/L-Gn (n = 1–3) **157–162** showed the presence of both the dendritic and the *cis*-platinum linker moieties (Figure 54). The upfield shifting of the signal due to the aromatic protons that are *ortho* to the acetylenic unit proved the anchoring of the *cis*-platinum moiety to the dendritic macromonomer. Moreover the proton signals of the phosphine ligands located at δ 1.0–1.5 (PCH_2CH_3) and δ 1.5–2.4 (PCH_2CH_3 and PCH_2CH_2P) were also observed.

For the poly(dendrimer)s *long-trans*-Pt-S/L-Gn (n = 1–3) **163–168**, the aromatic signal corresponding to the functional surface of dendritic macromonomers was upfield shifted after copolymerization (Figure 55). The proton signals of the ethylphosphine ligand were found at δ 1.1–1.4 (PCH_2CH_3) and δ 2.0–2.3 (PCH_2). For the L-Gn compounds **166–168**, one of the aromatic proton signals due to the biphenyl moiety appeared as a doublet at δ ≈ 7.4.

4.3.2. ^{13}C NMR spectroscopy

From the ^{13}C NMR spectra of *cis*-Pt-S/L-Gn (n = 1–3) **157–162** (Figure 56), the carbon signals of the bidentate phosphine ligand were observed at δ ≈ 8.8 (triplet like, $J \approx 10$ Hz, PCH_2CH_3), δ ≈ 18.6 (PCH_2CH_3) and δ ≈ 24 (PCH_2CH_2P). However, the signal at δ ≈ 24 was too weak to be observed for *cis*-Pt-L-G3 **162**. The $C{\equiv}CPt$ signal was found as a doublet[94] at δ 111 except for *cis*-Pt-S-G3 **159** and *cis*-Pt-L-G3 **162** as the signal was again too weak to be observed again. Unfortunately, another acetylenic carbon signal ($C{\equiv}CPt$) could not be identified clearly as it was masked by a large signal at δ ≈ 107.

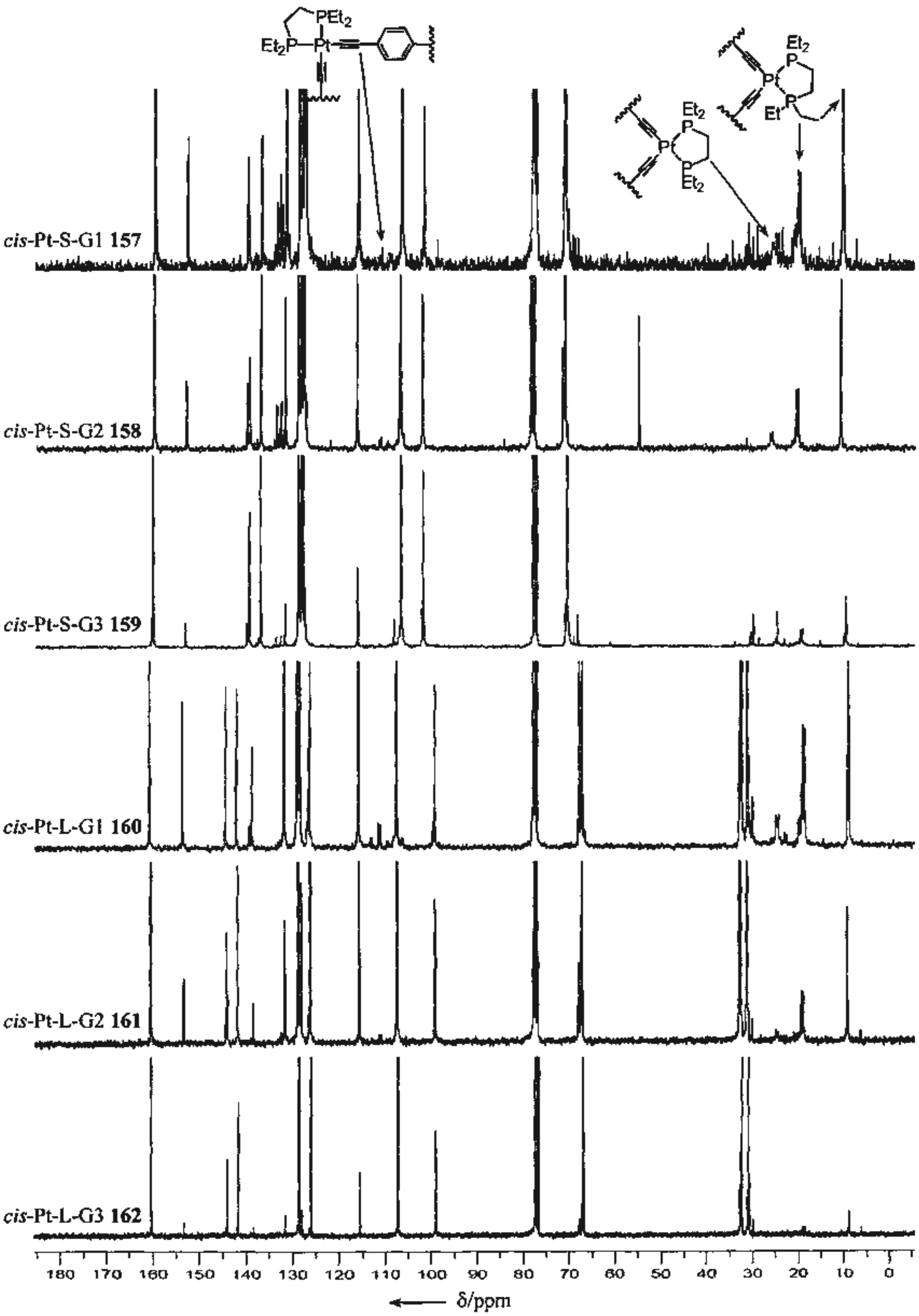


***Figure 56.** The stacked ^{13}C NMR (75.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s cis-Pt-S-Gn **157–159** and cis-Pt-L-Gn **160–162**.*

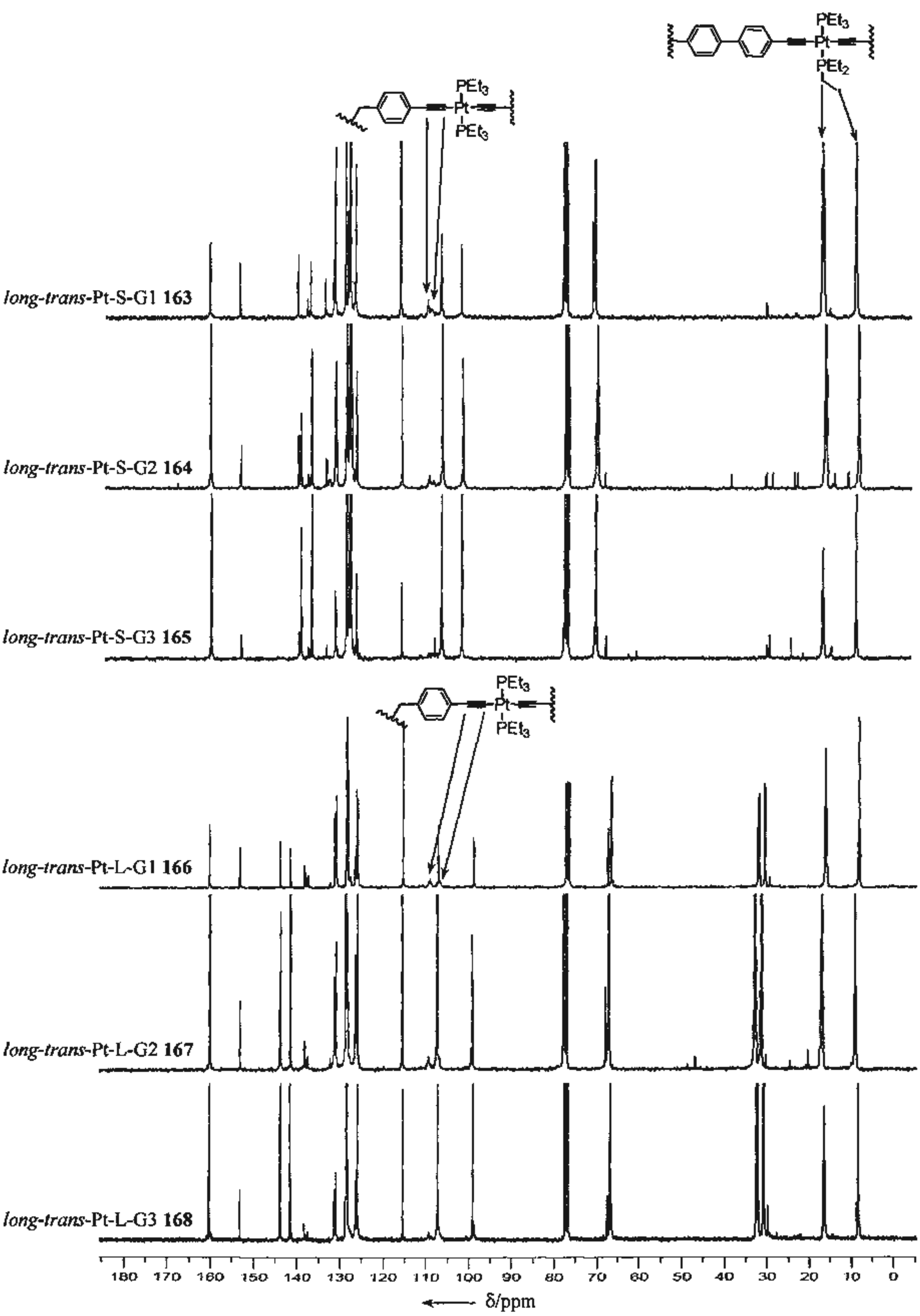


***Figure 57.** The stacked ^{13}C NMR (75.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s long-trans-Pt-S-Gn* ***163–165** and long-trans-Pt-L-Gn **166–168**.*

The ^{13}C NMR spectra of poly(dendrimer)s *long-trans*-Pt-S-Gn (n = 1–3) **163–165** (Figure 57) showed that the two acetylenic carbon signals were located at δ 108 (C≡*C*Pt) and δ 109 (*C*≡CPt), but the latter signal was not observable for *long-trans*-Pt-S-G3 **165**. In the case of L-Gn compounds **166–168**, the signal due to the acetylenic carbon attached to the platinum center (C≡*C*Pt) was found at δ 107, but it was also too weak to be observed for *long-trans*-Pt-L-G3 **168**. In addition, the carbon signals of the ethylphosphine located at δ 8.5 (PCH_2CH_3) and δ 16 (quintet like, $J \approx 18$ Hz, PCH_2) were also observed.

4.3.3. ^{31}P NMR spectroscopy

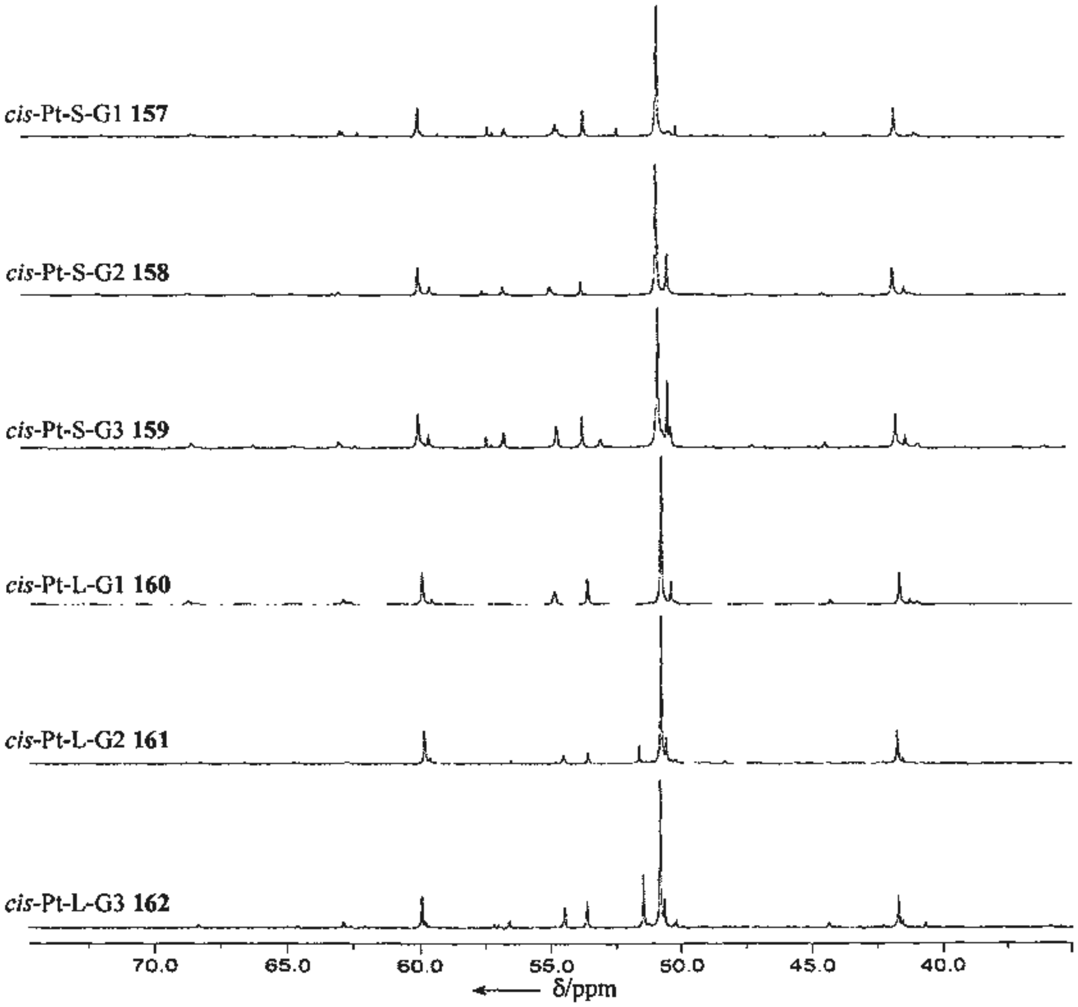


***Figure 58.** The stacked ^{31}P NMR (121.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s cis-Pt-S-Gn 157–159 and cis-Pt-L-Gn 160–162.*

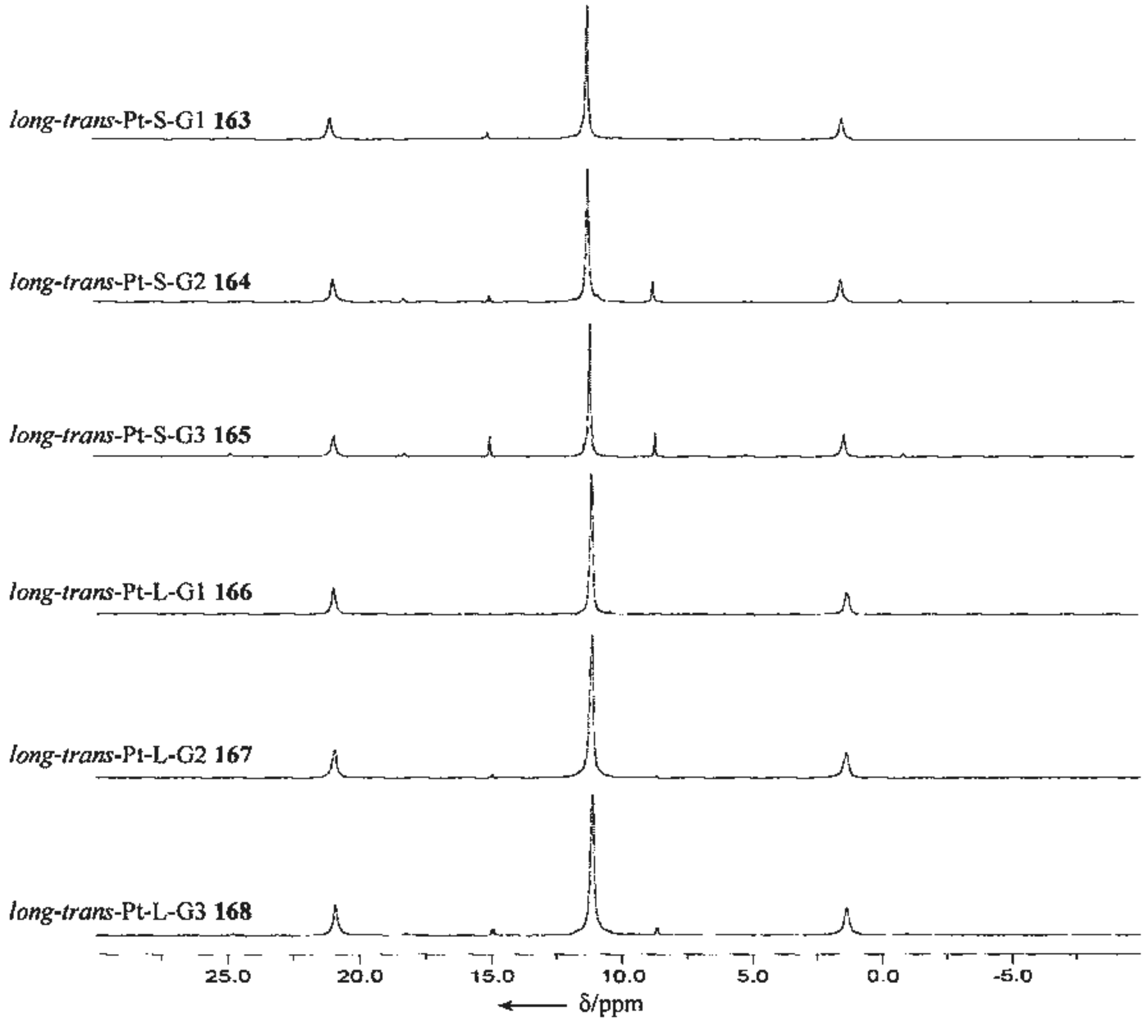


Figure 59. *The stacked ^{31}P NMR (121.5 MHz, $CDCl_3$) spectra of poly(dendrimer)s long-trans-Pt-S-Gn **163–165** and long-trans-Pt-L-Gn **166–168**.*

The ^{31}P NMR spectra of poly(dendrimer)s *cis*-Pt-S/L-Gn **157–162** showed the presence of many peaks (Figure 58), therefore the products obtained were highly heterogeneous. As the bidentate phosphine ligand adopt a *cis*-geometry, all the signals were downfield shifted as compared to those of poly(dendrimer)s Pt-S/L-Gn **144–149** containing *trans*-platinum linkers (See Section 3.4.3). Generally, a major peak located at δ 51 with two ^{195}Pt satellite signals ($^{1}J_{Pt-P}$ = 2210 Hz)[94] of one sixth intensity was observed. However two sets of minor peaks located at δ 50 ($^{1}J_{Pt-P} \approx$ 2220 Hz) and δ 54 ($^{1}J_{Pt-P}$ = 2250 Hz) were also found. For the *cis*-Pt-S-Gn compounds **157–159**, an extra minor peak at δ 57 ($^1J_{Pt-P}$ ≈ 2300 Hz) together with a peak at δ 55 were observed. The latter peak had a relatively larger coupling constant ($^1J_{Pt-P}$ ≈ 3360 Hz) and this signal was probably due to the Cl–Pt–P end-group. Again, the ^{31}P NMR results suggested that the degree of polymerization values of *cis*-Pt-S/L-Gn **157–162** were very low and further evidence could also been obtained from GPC and MS analyses (See Section 4.3.4).

In sharp contrast, the ^{31}P NMR spectra of *long-trans*-Pt-S/L-Gn **163–168** showed a major peak with ^{195}Pt satellites was located at δ 11 ($^1J_{Pt-P}$ = 2370 Hz) (Figure 59), suggesting that the samples possessed fair higher DP values. In some cases, peaks located at δ 8.7 ($^1J_{Pt-P}$ ≈ 2330 Hz) and δ 15 ($^1J_{Pt-P}$ = 2390 Hz) were also found.

4.3.4. GPC analysis

GPC analyses were performed in diluted THF solutions at elevated temperature 40 °C to prevent the poly(dendrimer)s from aggregation and polystyrenes were used as the calibration standards. For the polymerization reactions by using the *cis*-platinum linker **152**, the efficiencies were very low. The DP values of the poly(dendrimer)s **157–162** were around 2–3, and the PDI values were ranged from 1.3–2.1 (Figure 60 and Table 6). The existence of a peak possessing a higher retention time than the dendritic macromonomer (labeled with an asterisk) in the GPC chromatograms revealed the presence of cyclic monomer species. However the slowest running fractions were obtained in much higher proportion and they were isolated by preparative GPC and further analyzed by MALDI-TOF mass spectrometry.

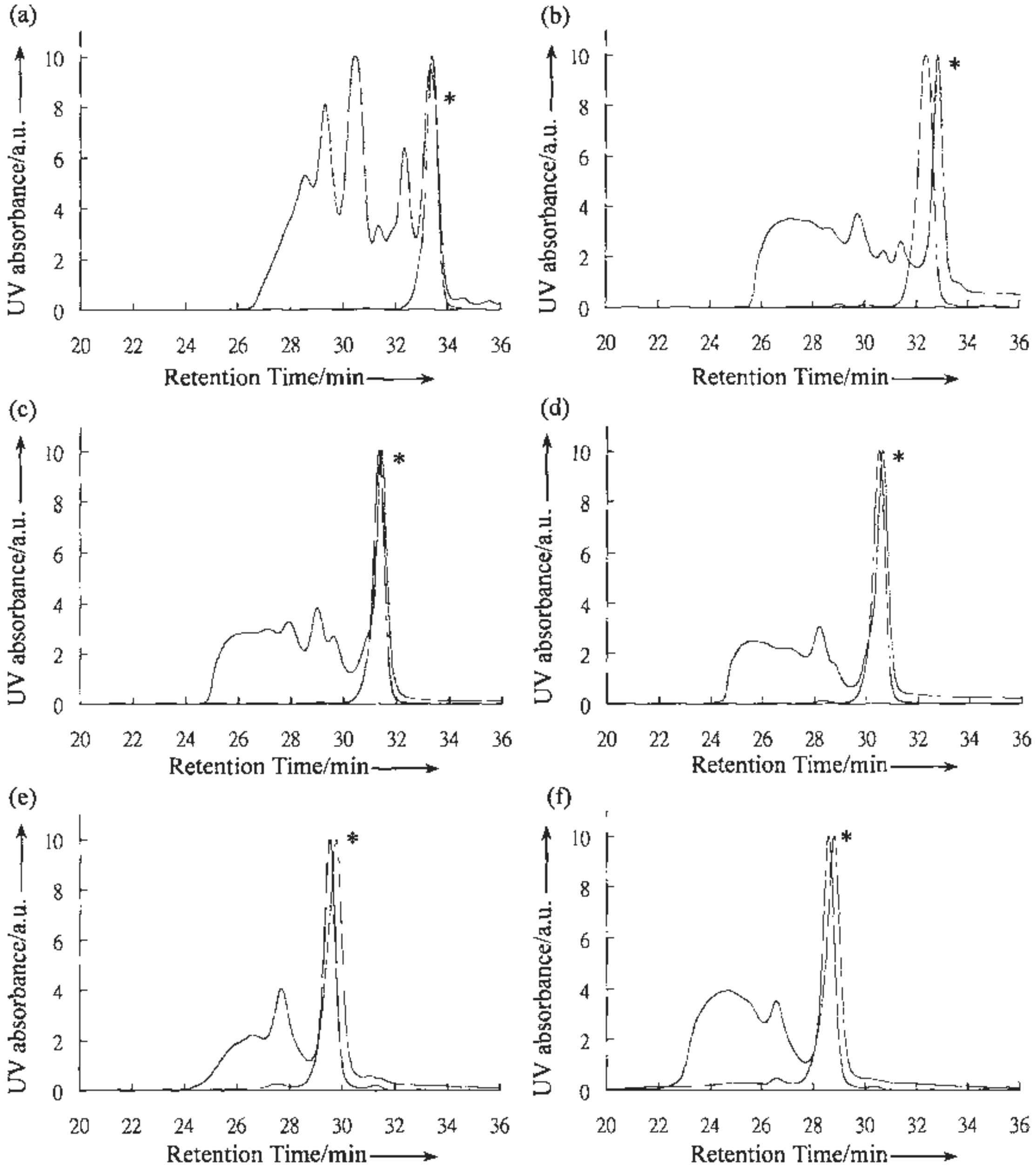


Figure 60. *Stacked GPC chromatograms of poly(dendrimer)s (blue lines) with their corresponding dendritic monomers (red lines) for (a) cis-Pt-S-G1* ***157****, (b) cis-Pt-L-G1* ***160****, (c) cis-Pt-S-G2* ***158****, (d) cis-Pt-L-G2* ***161****, (e) cis-Pt-S-G3* ***159*** *and (f) cis-Pt-L-G3* ***162****.*

Compound	$M_w \times 10^3$	DP	PDI
cis-Pt-S-G1 **157**	3	3 (34)[a]	1.6
cis-Pt-L-G1 **160**	4	3 (39)[a]	2.1
cis-Pt-S-G2 **158**	6	3 (60)[a]	1.5
cis-Pt-L-G2 **161**	7	3 (22)[a]	1.6
cis-Pt-S-G3 **159**	6	2 (21)[a]	1.3
cis-Pt-L-G3 **162**	13	3 (19)[a]	1.6

Table 6. *GPC data of poly(dendrimer)s cis-Pt-S/L-Gn* ***157–162*** *in THF at 40 °C.* [a] *DP values of poly(dendrimer)s Pt-S/L-Gn* ***144–149*** *obtained from the short trans-[Pt(PEt$_3$)$_2$Cl$_2$] linker* ***65****.*

The slowest running GPC fractions from *cis*-Pt-S/L-Gn **157–162** were the mixture of unreacted dendritic macromonomer, 1:1 platinum-dendritic monomer adduct, cyclic monomer, and/or the fragmentation products. Taking *cis*-Pt-S-G2 **158** as an example, the mass spectrum showed the presence of 1:1 platinum-dendritic monomer adduct, cyclic monomer and the platinum-dendritic monomer adduct with *iso*-propylamine as the ligand (Figure 61). When compared the DP values to those by using *trans*-[Pt(PEt$_3$)$_2$Cl$_2$] linker **65**, it was found that only oligomers/cyclic oligomers were formed when using the *cis*-platinum linker **152**. The formation of dendritic cyclic oligomers by using non-linear platinum linkers *via* inner-sphere–inner-sphere connectivity had been reported before (Figure 19 in Chapter 2).[37] As the placement of two dendritic macromonomers at a 90° relationship at the platinum metal center would creat severe steric hindrance, the polymerization process was prohibited and only oligomers or cyclic oligomers were formed.

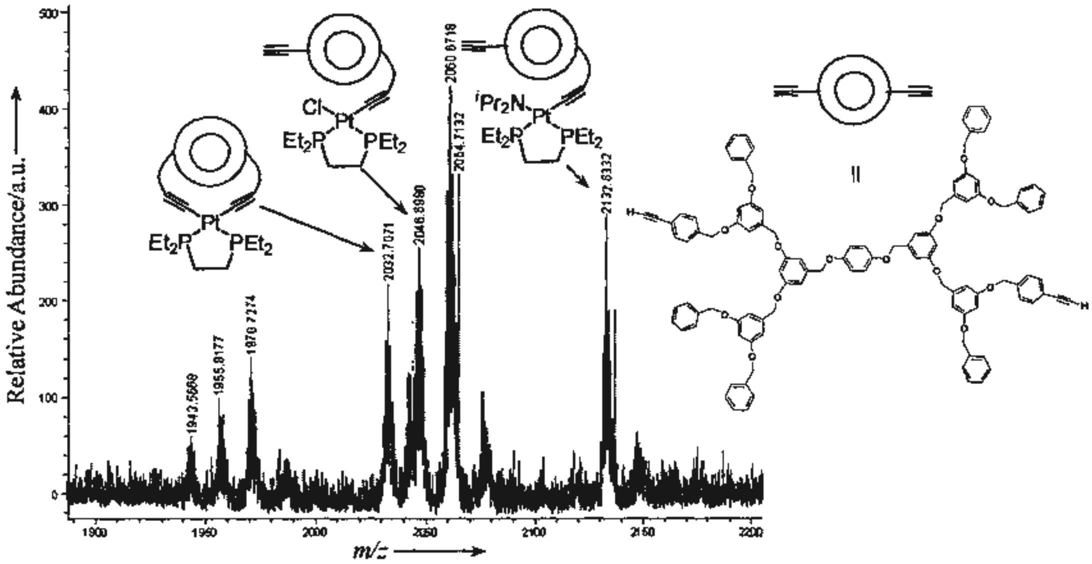


Figure 61. The MALDI-TOF mass spectrum of cis-Pt-S-G2 158.

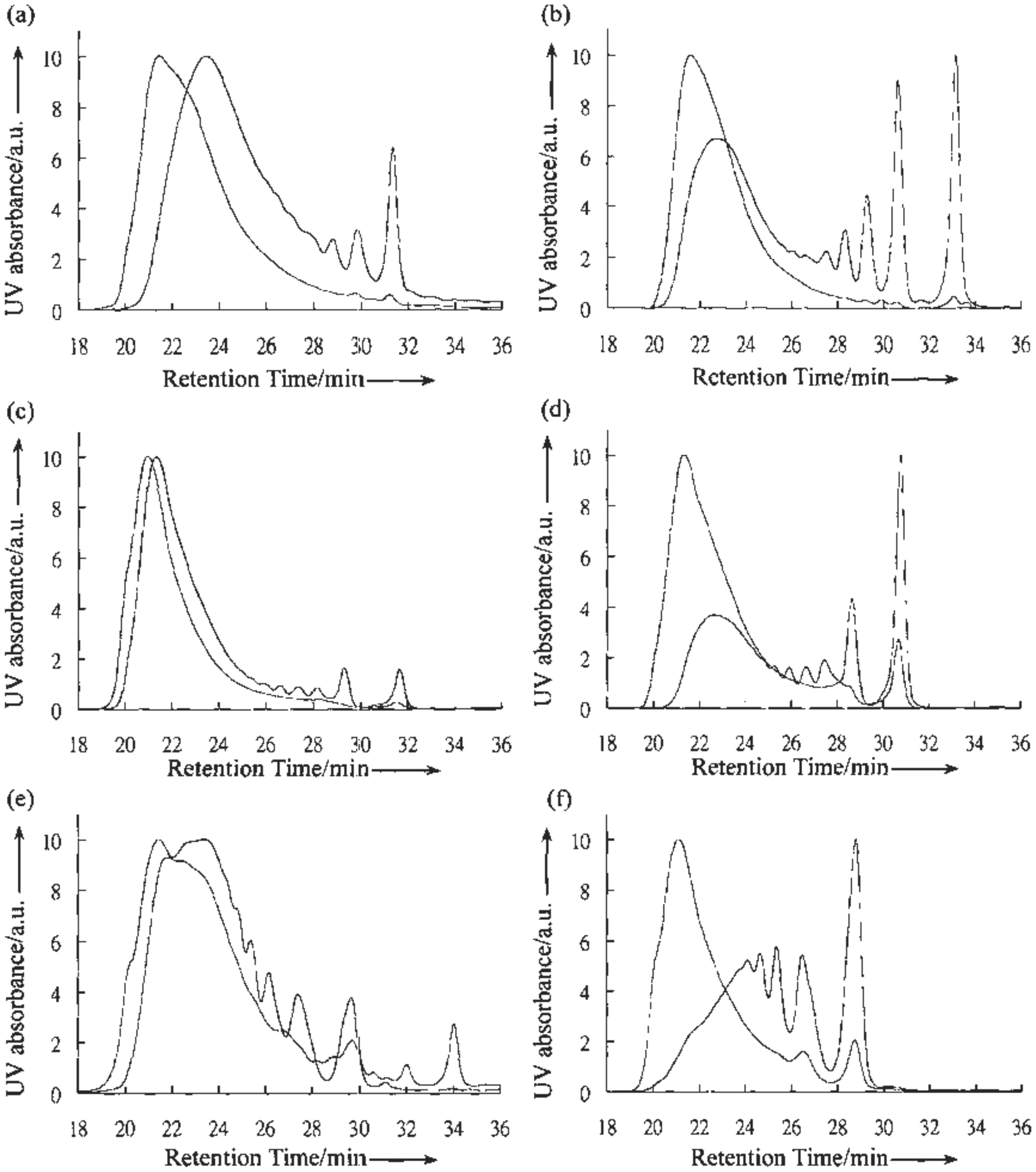


***Figure 62.** Stacked GPC chromatograms of poly(dendrimer)s (a) long-trans-Pt-S-G1 **163**, (b) long-trans-Pt-L-G1 **166**, (c) long-trans-Pt-S-G2 **164**, (d) long-trans-Pt-L-G2 **167**, (e) long-trans-Pt-S-G3 **165** and (f) long-trans-Pt-L-G3 **168** (blue lines) with their corresponding Pt-S/L-Gn seires (red lines).*

Compound	$M_w \times 10^3$	**DP**	**PDI**
long-tans-Pt-S-G1 **163**	107	59 (34)[a]	2.5
long-tans-Pt-L-G1 **166**	91	46 (39)[a]	2.0
long-trans-Pt-S-G2 **164**	191	72 (60)[a]	2.3
long-trans-Pt-L-G2 **167**	127	41 (22)[a]	2.0
long-trans-Pt-S-G3 **165**	148	34 (21)[a]	2.2
long-trans-Pt-L-G3 **168**	188	36 (19)[a]	1.9

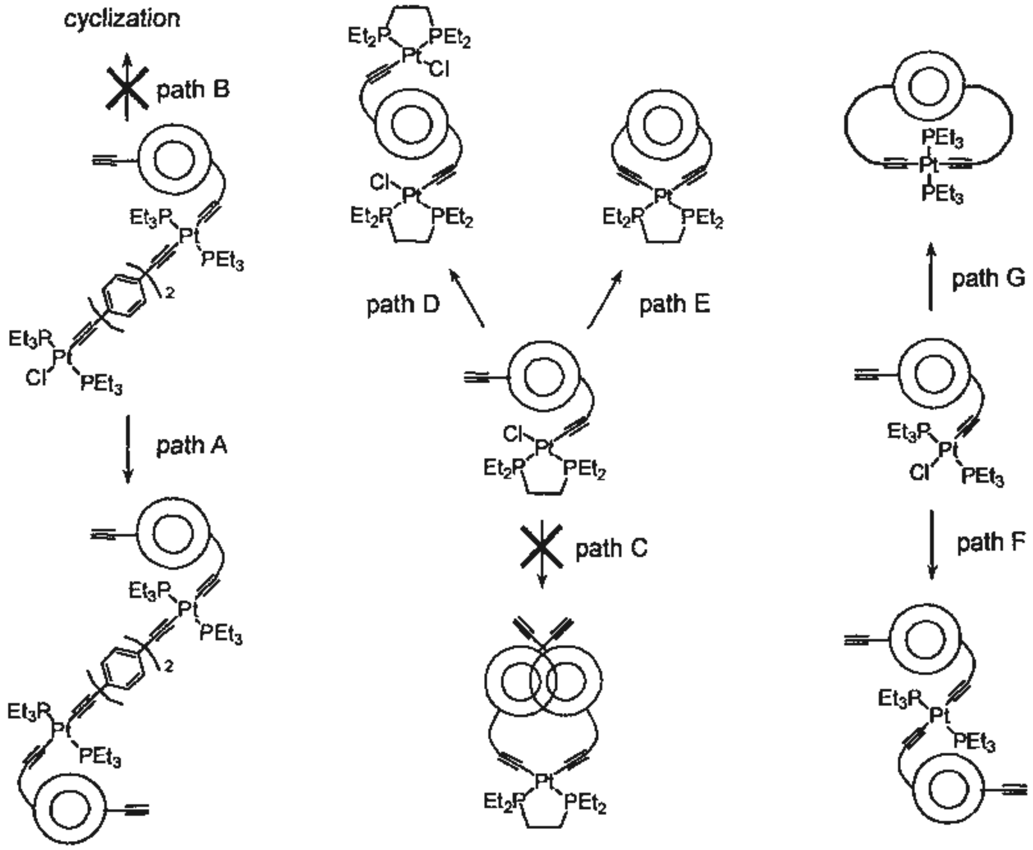
***Table 7.** GPC data of poly(dendrimer)s long-trans-Pt-S/L-Gn **163–168** in THF at 40 °C.* [a] *DP values of corresponding Pt-S/L-Gn **144–149** by using the short trans-[Pt(PEt$_3$)$_2$Cl$_2$] linker **65**.*

In contrast, by using the *long-trans*-platinum linker **156** to copolymerize with dendritic macromonomers, the polymerization reactions proceeded efficiently. There were several interesting findings. First, from the GPC chromatograms (Figure 62), the formation of cyclic oligomers was highly suppressed when compared to those by using the short or *trans*-platinum linker **65**. Second, the DP and PDI values ranged from 34–72 and 1.9–2.5 respectively, and all the DP values were higher than those of the corresponding Pt-S/L-Gn compounds **144–149** (Table 7). This showed that the polymerization efficiencies were enhanced by increasing the length of the platinum linker. Third, the DP values of *long-trans*-Pt-S-Gn (n = 1–2) generally were higher than those of *long-trans*-Pt-L-Gn (n = 1–2) of the same generation, and in the case of G3, the DP values were comparable. This revealed that the structural flexibility of dendritic macromonomers still exerted some effects on the polymerization efficiency. The dendritic macromonomers with higher structural rigidity (S-Gn series) tended to have higher DP values (See Section 3.4.4). Fourth, the polymerization efficiency involving the higher generation dendritic macromonomers, especially in the case of HC≡C–[L-G3]–C≡CH **78**, was significantly increased (Figure 62f). To conclude, by using the *long-trans*-platinum linker **156**, all the dendritic macromonomers would form organoplatinum poly(dendrimer)s efficiently. Although the DP values determined by GPC were in the order of 10^2, one has to bear in mind that these values could be underestimated by a factor of 1.5–14.[73]

4.4. Proposed model for the polymerization reactions

Based on the results obtained from the above experiments (Chapter 3 and Chapter 4), a reaction model for the formation of organoplatinum poly(dendirmer)s is proposed. This will explain how the structural flexibility of dendritic macromonomers

(S-Gn *vs* L-Gn) and the length and geometry of platinum linkers (*trans*-[Pt(PEt$_3$)$_2$Cl$_2$] **65** *vs* *cis*-platinum linker **152** *vs* *long-trans*-platinum linker **156**) can affect the polymerization efficiencies. For the three different platinum linkers, addition of the first dendritic macromonomer should proceed smoothly, but the addition of second dendritic macromonomer became more difficult because of the sterically hindered environment now around the platinum centers (Figure 63). In the case of the *long-trans*-platinum linker **156**, the steric hindrance was the smallest due to the large spatial separation between the two Pt–Cl groups and thus the second addition of dendritic macromonomer was highly favorable. As a result, the dendritic macromonomers possessing different structural flexibility (S-Gn and L-Gn) would both undergo polymerization (path A) while the cyclization process (path B) was



long-trans-Pt-linker **156** *cis*-Pt-linker **152** *trans*-Pt-linker **65**

Figure 63. Polymerization vs cyclization of dendritic macromonomers with platinum linkers.

greatly suppressed. When performing copolymerization reactions by using the *cis*-platinum linker **152**, the Pt–Cl group was hindered by the anchored dendritic macromonomer as they were at a 90° relationship. Therefore the addition of second dendritic macromonomer to such a sterically hindered environment would be prohibited (path C). Nonetheless, both the addition of another *cis*-platinum linker **152** (path D) and cyclization (path E) were feasible. This results in the formation of only low molecular weight oligomers (including cyclic oligomers), regardless of the structural flexibility of dendritic macromonomers. For the *trans*-Pt-linker **65**, the steric congestion around the platinum center should be higher than the case of using *long-trans*-platinum linker **156** but lower than that of using *cis*-platinum linker **152**. Hence, addition of the second dendritic macromonomer to the *trans*-platinum linker **65** was not strongly deterred, therefore the polymerization process could still happen (path F), but the reaction rate must be slower when compared to the corresponding process involving the *long-trans*-platinum linker **156**. As a result, the free acetyleic moiety of the anchored denritic monomer might have enough time to react with the same platinum center and form a cyclic monomer (path G). In this situation, the structural flexibility of dendritic macromonomers (S-Gn *vs* L-Gn) played an important role in controlling whether the reaction should proceed *via* polymerization (path F) or cyclization (path G). As expected, the second acetylenic moiety of the L-Gn dendritic macromonomers possessed a higher degree of freedom and hence a higher opportunity to anchor to the platinum center to form cyclic monomers. Therefore the copolymerization reactions involving the *trans*-platinum linker **65** was determined by dendritic macromonomers with different structural flexibilities.

Chapter 5

Conclusion and Outlook

Organoplatinum poly(dendrimer)s were successfully synthesized by copolymerizing various dendritic macromonomers (dendrimer beads) possessing two polymerizable groups on the periphery with different platinum linkers. The effect of structural flexibility of dendritic macromonomers and the length and geometry of platinum linkers on the polymerization reaction was studied. Poly(dendrimer)s with high DP values (34–72) were obtained by using *long-trans*-platinum linker **156**. For the connection between dendritic macromonomers and *cis*-platinum linker **152**, only low molecular weight (LMW) oligomers, including cyclic oligomers were afforded (DP = 2–3). When the polymerization was performed *via trans*-platinum linker **65**, the structural flexibility of dendritic macromonomers also played an important role in determining the polymerization efficiency. Dendritic monomers possessing higher structural flexibility (L-Gn series) preferred to undergo cyclization than the S-Gn series of the corresponding generation. Based on the experimental results, a working model was proposed to rationalize the various observations. The author believe that this model could also be used to predict propagation *vs* cyclization pathways for other polymerization reactions involving dendritic macromolecules. This work also showed that the outer-shpere–outer-shpere connection was a viable strategy for the preparation of nano-sized materials with controlled architectures. Moreover multi-block poly(dendrimer)s can also be constructed by using dendritic macromonomers and linkers with different properties *via* similar polymerization methods.

Chapter 6

Experimental Procedures

(a). General Information.

All ^{1}H NMR (300 MHz), ^{13}C NMR (75.5 MHz) and ^{31}P NMR (121.5 MHz) spectra were recorded on a Bruker Avance DPX 300 spectrometer at 298 K in $CDCl_3$ unless otherwise stated. Chemical shifts were reported as parts per million (ppm) in δ scale. Coupling constant (J) were reported in hertz (Hz). NMR signals were calibrated using the residual $CHCl_3$ proton signal at $\delta = 7.26$ ppm for ^{1}H NMR and at $\delta = 77.16$ ppm for ^{13}C NMR. For ^{31}P NMR, PPh_3 was used as the external standard at $\delta = -5.4$ ppm. Mass spectra analyses using electrospray ionization (ESI), electron ionization (EI) or fast atom bombardment (FAB) method were obtained on a ThermoFinnigan MAT 95 XL mass spectrometer, and matrix assisted laser desorption/ionization time of flight (MALDI-TOF) spectrometry analyses were performed on a MALDI Autoflex mass spectrometer. The reported molecular mass to charge ratio (m/z), unless otherwise specified, are monoisotopic mass. Elemental analyses were carried out at MEDAC Ltd., Surrey, United Kingdom. Melting points were measured on an Electrothermal IA9100 digital melting point apparatus. Unless otherwise stated, all chemicals were purchased commercially and used without purification. Tetrahydrofuran (THF) and dichloromethane (CH_2Cl_2) were freshly distilled prior to use over sodium-benzophenone and calcium hydride (CaH_2) respectively. *N*,*N*-Dimethylformamide (DMF), chloroform ($CHCl_3$), triethylamine (Et_3N)

and diisopropylamine (*i*-Pr_2NH) were distilled and dried with 4 Å molecular sieve prior to use. All reactions were carried out under a nitrogen atomsphere unless otherwise stated.

(b). General Procedures.

General Procedure A: Preparation of Esters.

A mixture of the carboxylic acid and 98% H_2SO_4 (5–10 mL) in ethanol or methanol was heated to reflux for 24 h. After the solvent was evaporated *in vacuo*, the residue was treated with saturated $NaHCO_3$ solution until pH = 8 and extracted with Et_2O (3 ×). The combined extracts were washed with brine, dried ($MgSO_4$), filtered and evaporated *in vacuo* to give the target compound.

General Procedure B: Preparation of Alcohols by DIBAL-H Reduction.

A mixture of the ester (1.0 equiv.) and DIBAL-H solution (1.0 M in hexane or toluene, 2.5–3.0 equiv.) in toluene was stirred at −60 → −10 °C for 2 h. The reaction mixture was acidified with 1 M HCl until pH = 3 and extracted with Et_2O (3 ×). The combined extracts were washed with brine, dried ($MgSO_4$), filtered and evaporated *in vacuo* to give the target compound.

General Procedure C: Preparation of Alkyl/Benzyl Bromides.

A mixture of the alcohol (1.0 equiv.), PPh_3 (1.25–2.0 equiv.) and CBr_4 (1.25–1.5 equiv.) in THF was stirred at 25 °C for 2 h. The reaction mixture was filtered and washed with Et_2O. The combined filtrates were dried *in vacuo* and the residue was purified by flash column chromatography to give the target compound.

General Procedure D: Synthesis of Di-*O*-alkylated Esters. A mixture of the phenol (1.0 equiv.), alkyl bromide (1.1–2.2 equiv.), K_2CO_3 (2.0–3.0 equiv.) and 18-crown-6 (2.0 mg) in acetone was heated to reflux for 12 h. The reaction mixture was filtered, dried *in* *vacuo* and purified by either recrystallization or flash column chromatography to give the target compound.

General Procedure E: Preparation of Alcohols by LAH Reduction.

A mixture of the ester (1.0 equiv.) and LAH (1.0 equiv.) in THF was stirred at 0 → 25 °C for 2 h. The reaction mixture was acidified with 1 M HCl until pH = 3 and extracted with Et_2O (3 ×). The combined extracts were washed with brine, dried ($MgSO_4$), filtered and evaporated *in vacuo* to give the target compound.

General Procedure F: Synthesis of Mono-*O*-alkylated Esters. A mixture of the phenol (1.0 equiv.), alkyl bromide (0.25 equiv.), K_2CO_3 (1.5 equiv.) and 18-crown-6 (2 mg) in acetone was heated to reflux for 12 h. The reaction mixture was filtered, dried *in vacuo* and purified either by recrystallization or flash column chromatography to give the target compound.

General Procedure G: Synthesis of Arylacetylenes by Sonogashira Coupling. A mixture of the I–[S/L-Gn]–I (1.0 equiv.), $(PPh_3)_2PdCl_2$ (0.5 equiv.), PPh_3 (0.5 equiv.), CuI (0.5 equiv.), trimethylsilylacetylene (15 equiv.), Et_3N (15 equiv.) in toluene was frozen in a sealed tube by liquid N_2 and degassed with N_2 (3 ×). The mixture was allowed to warm to 25 °C and then heated at 100 °C for 3 d. After the reaction was completed, Et_2O was added and the reaction mixture was filtered. The filtrate was dried *in vacuo* and purified by flash column chromatography to give the target compound.

General Procedure H: Desilylation Reactions by K_2CO_3. A mixture of the TMSC≡C–[S/L-Gn]–C≡CTMS (1.0 equiv.) and K_2CO_3 (5.0 equiv.) in THF/MeOH (v/v = 1/1) was stirred at 25 °C for 2 h. The reaction mixture was filtered and washed with CH_2Cl_2. The combined filtrates were dried *in vacuo* and purified by flash column chromatography to give the target compound.

General Procedure I: Desilylation Reactions by TBAF. A mixture of the TMSC≡C–[S/L-Gn]–C≡CTMS (1.0 equiv.) and TBAF solution (1.0 M in THF, 3.0 equiv.) in THF was stirred at 25 °C for 15 min. The reaction mixture was filtered and washed with CH_2Cl_2. The combined filtrates were dried *in vacuo* and purified by flash column chromatography to give the target compound.

General Procedure J: Synthesis of Poly(dendrimer)s Pt-S/L-Gn. A mixture of the HC≡C–[S/L-Gn]–C≡CH (1.0 equiv., concentration = 8.7 mM), *trans*-$[Pt(PEt_3)_2Cl_2]$ **65** (1.0 equiv.) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1) was frozen in a sealed tube by liquid N_2 and degassed with N_2 (3 ×). The mixture was allowed to warm to 25 °C followed by the addition of CuI (0.5 equiv.). The mixture was then heated at 40 °C for 2 d. The solvent was concentrated *in vacuo* and the residue dissolved in minimum amount of $CHCl_3$, filtered through a short pad of alumina. A sample of the aliquot was then taken out for GPC analysis. The residue compounds was purified by precipitation in MeOH to obtain the target poly(dendrimer)s.

General Procedure K: Synthesis of Poly(dendrimer)s *cis*-Pt-S/L-Gn. A mixture of the HC≡C–[S/L-Gn]–C≡CH (1.0 equiv., concentration = 8.7 mM), *cis*-platinum linker **152** (1.0 equiv.) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1) was frozen in a sealed tube by liquid N_2 and degassed with N_2 (3 ×). The mixture was allowed to warm to 25 °C followed by the addition of CuI (0.5 equiv.). The mixture was then heated at 40 °C for 2 d. The solvent was concentrated *in vacuo* and the residue dissolved in minimum amount of $CHCl_3$, filtered through a short pad of alumina. A sample of the aliquot was then taken out for

GPC analysis. The residue compounds was purified by precipitation in MeOH to obtain the target poly(dendrimer)s.

General Procedure L: Synthesis of Poly(dendrimer)s *long-trans*-Pt-S/L-Gn. A mixture of the HC≡C–[S/L-Gn]–C≡CH (1.0 equiv., concentration = 8.7 mM), *long-trans*-platinum linker **156** (1.0 equiv.) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1) was frozen in a sealed tube by liquid N_2 and degassed with N_2 (3 ×). The mixture was allowed to warm to 25 °C followed by the addition of CuI (0.5 equiv.). The mixture was then heated at 40 °C for 2 d. The solvent was concentrated *in vacuo* and the residue dissolved in minimum amount of $CHCl_3$, filtered through a short pad of alumina. A sample of the aliquot was then taken out for GPC analysis. The residue compounds was purified by precipitation in MeOH to obtain the target poly(dendrimer)s.

(c). Preparation of Dendrimers.

(i). Shorter Branch Series (S-Gn Series)

Ethyl 4-iodobenzoate (80).[77] This compound was prepared by General Procedure A. Starting from 4-iodobenzoic acid **79** (30.0 g, 0.12 mol) and 98% H_2SO_4 (5 mL) in ethanol (1.5 L), the product (32.8 g, 98%) was obtained as a pale yellow liquid. R_f = 0.79 (hexane/EtOAc = 1/4); ^1H NMR: δ = 7.79 (d, *J* = 8.7 Hz, 2 H; Ar*H*), 7.74 (d, *J* = 8.7 Hz, 2 H; Ar*H*), 4.36 (q, *J* = 7.2 Hz, 2 H; CO_2CH_2), 1.38 (t, *J* = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR: δ = 165.5, 137.3, 130.7, 129.6, 100.5, 60.9, 14.2.

4-Iodobenzyl alcohol (81).[78a] This compound was prepared by General Procedure B. Starting from ethyl 4-iodobenzoate **80** (31.0 g, 0.11 mol) and DIBAL-H solution (1.0 M in hexane, 340 mL, 0.34 mol) in toluene (100 mL), the product (36.1 g, 99%) was obtained as a white solid. M.p. 70.5–73.7 °C (lit. M.p. 61.0–66.5 °C;[78b] 65–67 °C[78c]); R_f= 0.38 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.68 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 7.10 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 4.62 (d, *J* = 6.0 Hz, 2 H; C*H*$_2$OH), 1.92 (t, *J* = 6.0 Hz, 1 H; OH); ^{13}C NMR: δ = 140.2, 137.4, 128.7, 93.0, 64.0.

4-Iodobenzyl bromide (82).[79] This compound was synthesized by General Procedure C. Starting from 4-iodobenzyl alcohol **81** (25.1 g, 0.11 mol), PPh_3 (56.2 g, 0.21 mol) and CBr_4 (53.3 g, 0.16 mol) in THF (300 mL), the product (20.9 g, 66 %) was obtained as a white solid. M.p. 78.6–80.6 °C (lit. M.p. 77–78.5 °C;[79] 78–79 °C[95]); R_f = 0.79 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.68 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 7.14 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 4.42 (s, 2 H; CH_2Br); ^{13}C NMR: δ = 138.1, 137.5, 131.0, 94.3, 32.6.

Ethyl 3,5-dihydroxybenzoate (84).[80] This compound was synthesized by General Procedure A. Starting from 3,5-dihydroxybenzoic acid **83** (100 g, 0.65 mol) and 98% H_2SO_4 (10 mL) in EtOH (1.7 L), the product (112 g, 94%) was obtained as a white solid. M.p. 126.4–128.2 °C (lit. M.p. 124–126 °C[80]); R_f = 0.71 (hexane/EtOAc = 1/4). ^{1}H NMR (DMSO-d_6): δ = 9.62 (s, 2 H; ArO*H*), 6.82 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.43 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 4.24 (q, *J* = 7.2 Hz, 2 H; CO_2CH_2), 1.28 (t, *J* = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR (DMSO-d_6) (one aromatic C signal is missing due to signal overlapping): δ = 166.0, 158.8, 131.9, 107.4, 60.7, 14.3.

S-G1-ester (85).[81] This compound was synthesized by General Procedure D. Starting from ethyl 3,5-dihydroxybenzoate **84** (80.0 g, 0.44 mol), BnBr (110 mL, 0.92 mol), K_2CO_3 (182 g, 1.32 mol) and 18-crown-6 (2 mg) in acetone (1.0 L), the product (115.0 g, 72%) was obtained as a white solid. M.p. 68.2–69.4 °C (lit. M.p. 68–68.5 °C[81]); R_f = 0.74 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.33–7.48 (m, 10 H; Ar*H*), 7.31 (d, *J* = 2.4 Hz, 2 H;

Ar*H*), 6.81 (t, *J* = 2.4 Hz, 1 H; Ar*H*), 5.08 (s, 2 H; PhCH_2), 4.37 (q, *J* = 7.2 Hz, 2 H; CO_2CH_2), 1.39 (t, *J* = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR: δ = 166.3, 159.8, 136.6, 132.5, 128.7, 128.2, 127.6, 108.4, 107.0, 70.3, 61.2, 14.4.

Hemi-S-G1-ester (86).[82] A mixture of S-G1-ester **85** (30.0 g, 82.8 mmol), 10% Pd-C (3.0 g) and K_2CO_3 (3.0 g) was stirred in EtOH/EtOAc (*v*/*v* = 1/1, 500 mL) under H_2 for 2 h. The reaction mixture was filtered, dried *in vacuo* and purified by flash column chromatography (eluent: hexane/EtOAc = 4/1 to 2/1) to give the target compound (11.8 g, 52%) as a white solid. M.p. 90.7–92.5 °C; R_f = 0.44 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.30–7.48 (m, 5 H; Ar*H*), 7.26–7.30 (m, 1 H; Ar*H*), 7.20–7.25 (m, 1 H; Ar*H*), 6.71 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 5.84 (brs, 1 H; ArO*H*), 5.07 (s, 2 H; PhCH_2O), 4.37 (q, *J* = 7.2 Hz, 2 H; CO_2CH_2), 1.39 (t, *J* = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR: δ = 167.3, 159.9, 157.3, 136.4, 132.0, 128.6, 128.1, 127.6, 109.7, 108.0, 107.5, 70.2, 61.7, 14.1.

I-S-G1-ester (87). This compound was prepared by General Procedure D. Starting from hemi-S-G1-ester **86** (12.0 g, 44.2 mmol), 4-iodobenzyl bromide **82** (14.4 g, 48.6 mmol), K_2CO_3 (12.2 g, 88.4 mmol) and 18-crown-6 (2 mg) in acetone (250 mL), the product (20.8 g, 97%) was obtained as a white solid after flash column chromatography (eluent: hexane/EtOAc = 12/1). M.p. 83.5–86.4 °C; R_f = 0.52 (hexane/EtOAc = 4/1); ^{1}H NMR: δ = 7.72 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.33–7.47 (m, 5 H; Ar*H*), 7.29–7.33 (m, 1 H; Ar*H*), 7.26–7.29 (m, 1 H; Ar*H*), 7.17 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.77 (t, *J* = 2.4 Hz, 1 H; Ar*H*), 5.07 (s, 2 H; PhCH_2O), 5.01 (s, 2 H; ArCH_2O), 4.37 (q, *J* = 7.2 Hz, 2 H; CO_2CH_2), 1.39 (t, *J* = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR: δ = 166.1, 159.8, 159.5, 137.7, 136.5, 136.2, 132.5, 129.3, 128.6, 128.1, 127.6, 108.6, 108.3, 107.0, 93.7, 70.3, 69.5, 61.2, 14.4; MS (EI): *m/z* (%):488 (37) [M^+]; HRMS (EI) calcd for $C_{23}H_{21}IO_4^+$: 488.0479; found:

488.0474; elemental analysis calcd (%) for $C_{23}H_{21}IO_4$: C 56.57, H 4.33; found: C 56.60, H 3.99, N <0.10.

I-S-G1-OH (88). This compound was prepared by General Procedure B. Starting from I-S-G1-ester **87** (19.8 g, 40.5 mmol) and DIBAL-H solution (1.0 M in hexane, 102 mL, 102 mmol) in toluene (150 mL), the product (17.4 g, 96%) was obtained as a white solid. M.p. 75.1–77.9 °C; $R_f = 0.36$ (hexane/EtOAc = 2/1). ^{1}H NMR: $\delta = 7.70$ (d, $J = 8.1$ Hz, 2 H; Ar*H*), 7.28–7.48 (m, 5 H; Ar*H*), 7.15 (d, $J = 8.4$ Hz, 2 H; Ar*H*), 6.63 (s, 1 H; Ar*H*), 6.59 (s, 1 H; Ar*H*), 6.52 (t, $J = 2.4$ Hz, 1 H; Ar*H*), 5.03 (s, 2 H; PhC*H*$_2$O), 4.96 (s, 2 H; ArC*H*$_2$O), 4.61 (d, $J = 4.5$ Hz, 2 H; C*H*$_2$OH), 1.99 (t, $J = 4.8$ Hz, 1 H; OH); ^{13}C NMR : $\delta = 160.2$, 159.9, 143.6, 137.7, 136.8, 136.6, 129.3, 128.7, 128.1, 127.6, 105.9, 105.8, 101.4, 93.6, 70.2, 69.4, 65.2; MS (EI): *m/z* (%): 466 (25) [M^+]; HRMS (EI) calcd for $C_{21}H_{19}IO_3^+$: 446.0373; found: 446.0375; elemental analysis calcd (%) for $C_{21}H_{19}IO_3$: C 56.52, H 4.29; found: C 56.70, H 4.34, N <0.10.

I-S-G1-Br (89). This compound was prepared by General Procedure C. Starting from I-S-G1-OH **88** (17.4 g, 39.0 mmol), PPh_3 (19.4 g, 58.5 mmol) and CBr_4 (20.5 g, 78.0 mmol) in THF (280 mL), the product (12.1 g, 61%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 20/1). $R_f = 0.38$ (hexane/EtOAc = 20/1); ^{1}H NMR: $\delta = 7.72$ (d, $J = 8.4$ Hz, 2 H; Ar*H*), 7.29–7.49 (m, 5 H; Ar*H*), 7.17 (d, $J = 8.4$ Hz, 2 H; Ar*H*), 6.66 (t, $J = 1.5$ Hz, 1 H; Ar*H*), 6.63 (t, $J = 1.5$ Hz, 1 H; Ar*H*), 6.53 (t, $J = 2.4$ Hz, 1 H; Ar*H*), 5.04 (s, 2 H; PhC*H*$_2$O), 4.97 (s, 2 H; ArC*H*$_2$O), 4.42 (s, 2 H; CH_2Br); ^{13}C NMR: $\delta = 160.2$, 159.9, 140.0, 137.8, 136.6, 136.4, 129.4, 128.7, 128.2, 127.7, 108.4, 108.2, 102.3, 93.7, 70.3, 69.5, 33.6; MS (EI): *m/z* (%): 508 (4) [$M^+(^{79}Br)$];

HRMS (EI) calcd for $C_{21}H_{18}BrIO_2^+$: 507.9529; found: 507.9531; elemental analysis calcd (%) for $C_{21}H_{18}BrIO_2$: C 49.54, H 3.56; found: C 49.84, H 3.58, N <0.10.

S-G1-OH (90).[96] This compound was synthesized by General Procedure E. Starting from S-G1-ester **85** (38.0 g, 0.11 mol) and LAH (4.00 g, 0.11 mol) in THF (300 mL), the product (33.2 g, 99%) was obtained as a white solid. M.p. 80.6–82.4 °C (lit. M.p. 80–81 °C[96]); R_f = 0.34 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.30–7.53 (m, 10 H; Ar*H*), 6.64 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.57 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 5.04 (s, 4 H; PhC*H*$_2$O), 4.62 (q, *J* = 5.7 Hz, 2 H; C*H*$_2$OH), 1.95 (t, *J* = 6.0 Hz, 1 H; OH); ^{13}C NMR: δ = 160.0, 143.6, 136.8, 128.6, 128.0, 127.5, 105.6, 101.2, 69.9, 64.9.

S-G1-Br (91).[6d] This compound was synthesized by General Procedure C. Starting from S-G1-OH **90** (17.0 g, 53.1 mmol), PPh_3 (27.8 g, 106 mmol) and CBr_4 (26.4 g, 79.6 mmol) in THF (280 mL), the product (18.0 g, 89%) was obtained as a white solid after flash column chromatography (eluent: hexane/EtOAc = 15/1). M.p. 85.5–86.9 °C (lit. M.p. 77–79 °C[97]); R_f = 0.80 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.28–7.47 (m, 10 H; Ar*H*), 6.65 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.56 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 5.04 (s, 4 H; PhC*H*$_2$O), 4.42 (s, 2 H; CH_2Br); ^{13}C NMR: δ = 160.0, 139.8, 136.6, 128.6, 128.0, 127.6, 108.1, 102.1, 70.0, 33.7.

Hemi-S-G2-ester (92). This compound was prepared by General Procedure F. Starting from ethyl 3,5-dihydroxybenzoate **84** (8.00 g, 44.0 mmol), S-G1-Br **91** (4.21 g, 11.0 mmol), K_2CO_3 (9.10 g, 65.9 mmol) and 18-crown-6 (2 mg) in acetone (200 mL), the product (4.18 g, 79%)[83] was obtained as a white solid after flash column chromatography (eluent: hexane/EtOAc = 5/2 to 2/1). M.p. 99.2–101.5 °C; R_f = 0.29 (hexane/EtOAc = 5/2); ^{1}H NMR: δ = 7.28–7.47 (m, 10 H; Ar*H*), 7.21–7.26 (m, 1 H; Ar*H*), 7.13–7.21 (m, 1

H; Ar*H*), 6.67 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.64 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 6.58 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 5.37 (brs, 1 H; ArO*H*), 5.04 (s, 4 H; PhC*H*$_2$O), 5.00 (s 2 H; ArC*H*$_2$O), 4.36 (q, *J* = 7.2 Hz, 2 H; CO$_2$CH$_2$), 1.38 (t, *J* = 7.2 Hz, 3 H; CH$_2$C*H*$_3$); ^{13}C NMR: δ = 166.9, 160.2, 159.9, 157.2, 139.0, 136.8, 132.3, 128.7, 128.1, 127.7, 109.7, 108.2, 107.4, 106.5, 101.8, 70.21, 70.17, 61.6, 14.3; MS (FAB): *m/z* (%): 485 (25) [M + H$^+$]; HRMS (FAB) calcd for $C_{30}H_{28}O_6 + H^+$: 485.1959; found: 485.1955; elemental analysis calcd (%) for $C_{30}H_{28}O_6$: C 74.36, H 5.82; found: C 74.12, H 5.79.

I-S-G2-ester (93). This compound was prepared by General Procedure D. Starting from hemi-S-G2-ester **92** (2.68 g, 5.52 mmol), I-S-G1-Br **89** (3.09 g, 6.08 mmol), K$_2$CO$_3$ (1.53 g, 11.0 mmol) and 18-crown-6 (2 mg) in acetone (125 mL), the product (4.61 g, 91%) was obtained as a white foam after flash column chromatography (eluent: hexane/EtOAc = 5/1 to 5/2). R_f = 0.58 (hexane/EtOAc = 5/2); ^{1}H NMR: δ = 7.70 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.27–7.48 (m, 17 H; Ar*H*), 7.16 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.77 (t, *J* = 2.4 Hz, 1 H; Ar*H*), 6.67–6.72 (m, 3 H; Ar*H*), 6.66 (t, *J* = 1.8 Hz, 1 H; Ar*H*), 6.59 (t, *J* = 2.4 Hz, 1 H; Ar*H*), 6.55 (t, *J* = 2.4 Hz, 1 H; Ar*H*), 5.04 (s, 6 H; PhC*H*$_2$O), 5.014 (s, 2 H; ArC*H*$_2$O), 5.009 (s, 2 H; ArC*H*$_2$O), 4.98 (s, 2 H; ArC*H*$_2$O), 4.38 (q, *J* = 7.2 Hz, 2 H; CO$_2$CH$_2$), 1.40 (t, *J* = 7.2 Hz, 3 H; CH$_2$C*H*$_3$); ^{13}C NMR: δ = 166.3, 160.2, 159.9, 159.71, 159.67, 139.1, 139.0, 137.7, 136.80, 136.75, 136.5, 132.5, 129.4, 128.7, 128.1, 127.6, 108.5, 106.9, 106.6, 106.5, 106.4, 101.7, 93.6, 70.2, 70.1, 69.4, 61.3, 14.4; MS (ESI): *m/z* (%): 935 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{51}H_{45}IO_8 + Na^+$: 935.2051; found: 935.2061; elemental analysis calcd (%) for $C_{51}H_{45}IO_8$: C 67.11, H 4.97; found: C 67.47, H 5.05.

I-S-G2-OH (94). This compound was prepared by General Procedure B. Starting from I-S-G2-ester **93** (4.61 g, 5.05 mmol) and DIBAL-H solution (1.0 M in hexane, 15.1 mL, 15.1 mmol) in toluene (60 mL), the product (4.02 g, 91%) was obtained as a white foam. R_f = 0.18 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.70 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.29–7.47 (m, 15 H; Ar*H*), 7.15 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.67–6.73 (m, 3 H; Ar*H*), 6.65 (brs, 1 H; Ar*H*), 6.56–6.63 (m, 3 H; Ar*H*), 6.49–6.56 (m, 2 H; Ar*H*), 5.03 (s, 6 H; PhC*H*$_2$O), 4.97 (s, 2 H; ArC*H*$_2$O), 4.96 (s, 4 H; ArC*H*$_2$O), 4.62 (d, *J* = 5.4 Hz, 2 H; C*H*$_2$OH), 1.80 (t, *J* = 5.1 Hz, 1 H; OH); ^{13}C NMR: δ = 160.2, 160.12, 160.09, 159.9, 143.6, 139.5, 139.4, 137.7, 136.84, 136.79, 136.6, 129.4, 128.7, 128.1, 127.7, 106.6, 106.5, 106.4, 105.8, 101.7, 101.6, 101.4, 93.6, 70.2, 70.0, 69.9, 69.4, 65.3; MS (ESI): *m/z* (%): 893 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{49}H_{43}IO_7$ + Na$^+$: 893.1946; found: 893.1954; elemental analysis calcd (%) for $C_{49}H_{43}IO_7$: C 67.59, H 4.98; found: C 67.94, H 5.13.

I-S-G2-Br (95). This compound was prepared by General Procedure C. Starting from I-S-G2-OH **94** (3.26 g, 3.74 mmol), PPh_3 (1.23 g, 4.68 mmol) and CBr_4 (1.55 g, 4.68 mmol) in THF (20 mL), the product (3.27 g, 87%) was obtained as a white foam by flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/1). R_f = 0.44 (hexane/CH_2Cl_2 = 1/1); ^{1}H NMR: δ = 7.70 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.27–7.49 (m, 15 H; Ar*H*), 7.15 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.66–6.73 (m, 3 H; Ar*H*), 6.61–6.66 (m, 3 H; Ar*H*), 6.59 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 6.55 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 6.52 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 5.04 (s, 6 H; PhC*H*$_2$O), 4.974 (s, 4 H; ArC*H*$_2$O), 4.966 (s, 2 H; ArC*H*$_2$O), 4.41 (s, 2 H; CH_2Br); ^{13}C NMR: δ = 160.3, 160.1, 160.04, 160.00, 139.9, 139.3, 139.2, 137.8, 136.9, 136.8, 136.6, 129.4, 128.7, 128.2, 127.7, 108.3, 106.6, 106.5, 106.4, 102.3, 101.8, 93.6, 70.3, 70.2, 70.1, 69.5, 33.7; MS (ESI): *m/z* (%): 933 (95) [M(^{79}Br) + H$^+$]; HRMS (ESI) calcd for $C_{49}H_{42}BrIO_6$ + H$^+$: 933.1282; found: 933.1271; elemental analysis calcd (%) for $C_{49}H_{42}BrIO_6$: C 63.03, H 4.53; found: C 63.03, H 4.37, N <0.10.

S-G2-ester (96).[84] This compound was prepared by General Procedure D. Starting from ethyl 3,5-dihydroxybenzoate **84** (2.35 g, 12.9 mmol), S-G1-Br **91** (10.9 g, 28.4 mmol), K_2CO_3 (5.35 g, 38.7 mmol) and 18-crown-6 (2 mg) in acetone (100 mL), the product (8.86 g, 87%) was obtained as a white solid after flash column chromatography (eluent: hexane/CH_2Cl_2 = 3/2 to CH_2Cl_2). M.p. 121.4–124.7 °C; R_f = 0.29 (hexane/CH_2Cl_2 = 1/1); ^{1}H NMR: δ = 7.30–7.46 (m, 20 H; Ar*H*), 7.29 (d, J = 2.1 Hz, 2 H; Ar*H*), 6.76 (t, J = 2.1 Hz, 1 H; Ar*H*), 6.69 (d, J = 2.1 Hz, 4 H; Ar*H*), 6.58 (t, J = 2.1 Hz, 2 H; Ar*H*), 5.04 (s, 8 H; PhC*H*$_2$O), 5.01 (s, 4 H; ArC*H*$_2$O), 4.37 (q, J = 7.2 Hz, 2 H; CO_2CH_2), 1.39 (t, J = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR: δ = 166.3, 160.2, 159.7, 139.0, 136.8, 132.5, 128.6, 128.1, 127.6, 108.5, 106.9, 106.5, 101.7, 70.1, 61.2, 14.4.

S-G2-OH (97).[6d] This compound was prepared by General Procedure B. Starting from S-G2-ester **96** (8.86 g, 11.3 mmol) and DIBAL-H solution (1.0 M in hexane, 28.0 mL, 28.0 mmol) in toluene (100 mL), the product (8.38 g, 99%) was obtained as a colorless foam. R_f = 0.58 (hexane/EtOAc = 1/1); ^{1}H NMR: δ = 7.30–7.49 (m, 20 H; Ar*H*), 6.70 (d, J = 2.4 Hz, 4 H; Ar*H*), 6.62 (d, J = 2.1 Hz, 2 H; Ar*H*), 6.60 (t, J = 2.4 Hz, 2 H; Ar*H*), 6.54 (t, J = 2.4 Hz, 1 H; Ar*H*), 5.04 (s, 8 H; PhC*H*$_2$O), 4.98 (s, 4 H; ArC*H*$_2$O), 4.62 (s, 2 H; C*H*$_2$OH), 1.77 (brs, 1 H; OH); ^{13}C NMR: δ = 160.0, 159.8, 143.6, 139.3, 136.7, 128.4, 127.9, 127.5, 106.3, 105.4, 101.4, 101.1, 69.8, 69.6, 64.7.

S-G2-Br (98).[6d] This compound was prepared by General Procedure C. Starting from S-G2-OH **97** (8.37 g, 11.2 mmol), PPh_3 (4.42 g, 16.8 mmol) and CBr_4 (5.59 g, 16.8 mmol) in THF (150 mL), the product (6.61 g, 73%) was obtained as a white solid after passing through a short pad of silica gel (eluent: hexane/EtOAc = 1/1) followed by recrystallization (hexane/EtOAc). M.p. 130.8–131.6 °C (lit. M.p. 129–130.5 °C[6d]); R_f =

0.67 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.28–7.48 (m, 20 H; Ar*H*), 6.67 (d, *J* = 2.1 Hz, 4 H; Ar*H*), 6.62 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.58 (t, *J* = 2.4 Hz, 2 H; Ar*H*), 6.52 (d, *J* = 2.4 Hz, 1 H; Ar*H*), 5.04 (s, 8 H; PhCH_2O), 4.97 (s, 4 H; ArCH_2O), 4.41 (s, 2 H; CH_2Br); ^{13}C NMR: δ = 160.3, 160.1, 139.9, 139.2, 136.9, 128.7, 128.2, 127.7, 108.3, 106.5, 102.4, 101.8, 70.3, 70.2, 33.7.

Hemi-S-G3-ester (99). This compound was prepared by General Procedure F. Starting from ethyl 3,5-dihydroxybenzoate **84** (3.05 g, 16.7 mmol), S-G2-Br **98** (3.38 g, 4.19 mmol), K_2CO_3 (3.47 g, 25.1 mmol) and 18-crown-6 (2 mg) in acetone (100 mL), the product (2.44 g, 64%)[83] was obtained as a white solid after flash column chromatography (eluent: CH_2Cl_2 to CH_2Cl_2/Et_2O = 20/1). M.p. 121.4–123.7 °C; R_f = 0.69 (CH_2Cl_2); ^{1}H NMR: δ = 7.27–7.46 (m, 20 H; Ar*H*), 7.19–7.25 (m, 1 H; Ar*H*), 7.09–7.18 (m, 1 H; Ar*H*), 6.66–6.71 (m, 4 H; Ar*H*), 6.64 (d, *J* = 1.8 Hz, 2 H; Ar*H*), 6.61 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 6.57 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 6.54 (t, *J* = 1.8 Hz, 1 H; Ar*H*), 5.37 (brs, 1 H; ArO*H*), 5.03 (s, 8 H; PhCH_2O), 4.99 (s, 2 H; ArCH_2O), 4.97 (s, 4 H; ArCH_2O), 4.34 (q, *J* = 6.9 Hz, 2 H; CO_2CH_2), 1.37 (t, *J* = 6.9 Hz, 3 H; CH$_2$CH_3); ^{13}C NMR: δ = 166.4, 160.3, 160.2, 159.9, 156.8, 139.4, 139.1, 136.9, 132.7, 128.7, 128.2, 127.7, 109.6, 108.3, 107.3, 106.5, 101.9, 101.8, 70.3, 70.1, 61.4, 14.4; MS (ESI): *m/z* (%): 931 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{58}H_{52}O_{10}$ + Na$^+$: 931.3453; found: 931.3462; elemental analysis calcd (%) for $C_{58}H_{52}O_{10}$: C 76.63, H 5.77; found: C 76.63, H 6.05.

I-S-G3-ester (100). This compound was prepared by General Procedure D. Starting from hemi-S-G3-ester **99** (2.42 g, 2.67 mmol), I-S-G2-Br **95** (2.74 g, 2.93 mmol), K_2CO_3 (0.55 g, 4.00 mmol) and 18-crown-6 (2 mg) in acetone (30 mL) for 60 h, the product (4.67 g, 99%) was obtained as a white foam after flash column chromatography (eluent:

hexane/CH_2Cl_2 = 2/3 to CH_2Cl_2). R_f = 0.53 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.67 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 7.27–7.45 (m, 37 H; Ar*H*), 7.13 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.76–6.81 (m, 1 H; Ar*H*), 6.61–6.73 (m, 12 H; Ar*H*), 6.50–6.60 (m, 6 H; Ar*H*), 5.02 (s, 14 H; PhCH_2O), 4.99 (s, 4 H; ArCH_2O), 4.97 (s, 8 H; ArCH_2O), 4.95 (s, 2 H; ArCH_2O), 4.35 (q, *J* = 7.2 Hz, 2 H; CO_2CH_2), 1.38 (t, *J* = 7.2 Hz, 3 H; CH_2CH_3); ^{13}C NMR: δ = 166.3, 106.3, 160.2, 160.1, 159.9, 159.8, 139.4, 139.3, 139.01, 138.98, 137.7, 136.9, 136.8, 136.6, 132.6, 129.4, 128.7, 128.1, 127.6, 108.5, 107.0, 106.6, 106.5, 106.4, 101.8, 101.7, 93.6, 70.2, 70.1, 70.0, 69.4, 61.3, 14.4; MS (ESI): *m/z* (%): 1784 (85) [M + Na^+]; HRMS (ESI) calcd for $C_{107}H_{93}IO_{16}$ + Na^+: 1783.5401; found: 1783.5381; elemental analysis calcd (%) for $C_{107}H_{93}IO_{16}$: C 72.95, H 5.32; found: C 72.79, H 5.04, N <0.10.

I-S-G3-OH (101). This compound was prepared by General Procedure B. Starting from I-S-G3-ester **100** (4.15 g, 2.35 mmol) and DIBAL-H solution (1.0 M in hexane, 7.10 mL, 7.10 mmol) in toluene (20 mL), the product (4.03 g, 99%) was obtained as a white foam. R_f = 0.65 (CH_2Cl_2); ^{1}H NMR: δ = 7.67 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.27–7.47 (m, 35 H; Ar*H*), 7.12 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.61–6.73 (m, 12 H; Ar*H*), 6.59 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.57 (t, *J* = 2.4 Hz, 3 H; Ar*H*), 6.49–6.55 (m, 4 H; Ar*H*), 5.01 (s, 14 H; PhCH_2O), 4.96 (s, 12 H; ArCH_2O), 4.94 (s, 2 H; ArCH_2O), 4.58 (s, 2 H; CH_2OH), 1.70 (brs, 1 H; OH); ^{13}C NMR: δ = 160.1, 160.02, 159.98, 159.8, 143.7, 139.39, 139.35, 139.2, 137.6, 136.8, 136.7, 136.5, 129.3, 128.6, 128.0, 127.6, 106.5, 106.4, 106.3, 105.6, 101.6, 101.2, 93.5, 70.0, 69.9, 69.8, 69.2, 65.0; MS (ESI): *m/z* (%): 1742 (80) [M + Na^+]; HRMS (ESI) calcd for $C_{105}H_{91}IO_{15}$ + Na^+: 1741.5295; found: 1741.5308; elemental analysis calcd (%) for $C_{105}H_{91}IO_{15}$: C 73.33, H 5.33; found: C 73.52, H 5.36, N <0.10.

I-S-G3-Br (102). This compound was prepared by General Procedure C. Starting from I-S-G3-OH **101** (1.51 g, 0.876 mmol), PPh_3 (0.29 g, 1.09 mmol) and CBr_4 (0.36 g, 1.09 mmol) in THF (5 mL), the product (1.21 g, 78%) was obtained as a white foam after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/1 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 100/1). R_f = 0.63 (hexane/CH_2Cl_2 = 1/2); ^1H NMR: δ = 7.67 (d, J = 8.4 Hz, 2 H; Ar*H*), 7.27–7.47 (m, 35 H; Ar*H*), 7.13 (d, J = 8.1 Hz, 2 H; Ar*H*), 6.66–6.72 (m, 7 H; Ar*H*), 6.60–6.66 (m, 7 H; Ar*H*), 6.57 (t, J = 2.1 Hz, 3 H; Ar*H*), 6.49–6.56 (m, 4 H; Ar*H*), 5.02 (s, 14 H; PhC*H*$_2$O), 4.96 (s, 8 H; ArC*H*$_2$O), 4.95 (s, 6 H; ArC*H*$_2$O), 4.38 (s, 2 H; CH_2Br); ^{13}C NMR: δ = 160.15, 160.06, 159.9, 159.8, 139.8, 139.3, 139.2, 139.1, 137.6, 136.8, 136.5, 129.3, 128.6, 128.0, 127.6, 108.2, 106.45, 106.39, 106.3, 102.1, 101.6, 93.5, 70.0, 69.9, 69.3, 33.7; MS (ESI): *m/z* (%): 1781 (55) [M(^{79}Br) + H$^+$]; HRMS (ESI) calcd for $C_{105}H_{90}BrIO_{14}$ + H$^+$: 1781.4631; Found: 1781.4657; elemental analysis calcd for $C_{105}H_{90}BrIO_{14}$: C 70.75, H 5.09; found: C 70.71, H 5.08, N <0.10.

I–[S-G1]–I (104). A mixture of hydroquinone **103** (0.803 g, 7.29 mmol), I-S-G1-Br **89** (8.17 g, 16.0 mmol), Cs_2CO_3 (8.32 g, 25.5 mmol) and dibenzo-24-crown-8 (5 mg) in DMF (40 mL) was stirred under N_2 at 25 °C for 12 h. The reaction mixture was filtered and washed with CH_2Cl_2. The combined filtrates were dried *in vacuo* and purified by flash column chromatography (eluent: hexane/$CHCl_3$ = 1/1) to give the target compound (5.98 g, 85%) as a white solid. M.p. 136.5–137.6 °C; R_f = 0.30 (hexane/$CHCl_3$ = 1/1); ^1H NMR: δ = 7.71 (d, J = 8.4 Hz, 4 H; Ar*H*), 7.29–7.48 (m, 10 H; Ar*H*), 7.16 (d, J = 8.4 Hz, 4 H; Ar*H*), 6.88 (s, 4 H; core-Ar*H*), 6.70 (s, 2 H; Ar*H*), 6.66 (s, 2 H; Ar*H*), 6.54 (t, J = 2.1 Hz, 2 H; Ar*H*), 5.04 (s, 4 H; ArC*H*$_2$O), 4.98 (s, 4 H; ArC*H*$_2$O), 4.96 (s, 4 H; ArC*H*$_2$O); ^{13}C NMR: δ = 160.2, 159.9, 153.1, 139.9, 137.7, 136.8, 136.6, 129.4, 128.7, 128.1, 127.6, 115.9, 106.5, 106.3, 101.5, 93.6, 70.5, 70.1, 69.4; MS (EI): *m/z* (%): 966 (<1) [M^+]; HRMS (EI) calcd for $C_{48}H_{40}I_2O_6^+$: 966.0909; found: 966.0913.

I–[S-G2]–I (105). A mixture of hydroquinone **103** (0.16 g, 1.46 mmol), I-S-G2-Br **95** (3.00 g, 3.21 mmol), K_2CO_3 (0.61 g, 4.38 mmol) and 18-crown-6 (2 mg) in acetone (80 mL) was heated to reflux for 3 d. The mixture was filtered and washed with CH_2Cl_2. The combined filtrates were evaporated *in vacuo* and the residue was chromatographed on silica gel to give the product (2.52 g, 95%) as a white foam (eluent: hexane/CH_2Cl_2 = 1/2 to CH_2Cl_2). R_f = 0.33 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.68 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 7.27–7.47 (m, 30 H; Ar*H*), 7.14 (d, *J* = 8.4 Hz, 4 H; Ar*H*), 6.86 (s, 4 H; core-Ar*H*), 6.66–6.71 (m, 6 H; Ar*H*), 6.60–6.66 (m, 6 H; Ar*H*), 6.57 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 6.53 (t, *J* = 2.1 Hz, 4 H; Ar*H*), 5.03 (s, 12 H; PhC*H*$_2$O), 4.97 (s, 4 H; ArC*H*$_2$O), 4.96 (s, 8 H; ArC*H*$_2$O), 4.92 (s, 4 H; ArC*H*$_2$O); ^{13}C NMR: δ = 160.1, 160.0, 159.9, 159.8, 153.0 ,139.8, 139.3, 139.2, 137.5, 136.73, 136.68, 136.4, 129.3, 128.5, 128.0, 127.5, 115.7, 106.4, 106.3, 101.5, 93.5, 70.3, 69.9, 69.83, 69.77, 69.2; MS (ESI): *m/z* (%): 1816 (100) [M + H^+]; HRMS (ESI) calcd for $C_{104}H_{88}I_2O_{14}$ + H^+: 1815.4336; found: 1815.4327; elemental analysis calcd (%) for $C_{104}H_{88}I_2O_{14}$: C 68.80, H 4.89; found: C 68.76, H 4.56, N <0.10.

I–[S-G3]–I (106). A mixture of hydroquinone **103** (13.6 mg, 0.124 mmol), I-S-G3-Br **102** (484 mg, 0.272 mmol), K_2CO_3 (51.2 mg, 0.371 mmol) and 18-crown-6 (2 mg) in acetone (10 mL) was heated to reflux for 3 d. The mixture was filtered and washed with CH_2Cl_2. The combined filtrates were concentrated *in vacuo* and the residue was chromatographed on silica gel to give the product (412 mg, 95%) as a colorless oil (eluent: hexane/CH_2Cl_2 = 1/1 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 100/1). R_f = 0.52 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.67 (d, *J* = 8.1 Hz, 4 H; ArH), 7.27–7.48 (m, 70 H;

Ar*H*), 7.12 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 6.85 (s, 4 H; core-Ar*H*), 6.61–6.74 (m, 28 H; Ar*H*), 6.49–6.61 (m, 14 H; Ar*H*), 5.01 (s, 28 H; PhCH_2O), 4.95 (s, 24 H; ArCH_2O), 4.93 (s, 4 H; ArCH_2O), 4.89 (s, 4 H; ArCH_2O); ^{13}C NMR: δ = 160.2, 160.14, 160.11, 160.09, 159.9, 153.2, 139.9, 139.41, 139.39, 139.36, 139.3, 137.7, 136.9, 136.8, 136.6, 129.4, 128.7, 128.1, 128.0, 127.6, 115.9, 106.6, 106.5, 106.4, 101.7, 93.6, 70.5, 70.1, 70.0, 69.4; HRMS (MALDI-TOF) calcd for $C_{216}H_{184}I_2O_{30}$ + Na$^+$: 3536.0921; found: 3536.1139; elemental analysis calcd (%) for $C_{216}H_{184}I_2O_{30}$: C 73.84, H 5.28; found: C 73.64, H 5.21, N <0.10.

TMSC≡C–[S-G1]–C≡CTMS (107). This compound was prepared by General Procedure G. Starting from I–[S-G1]–I **104** (610 mg, 0.63 mmol), $(PPh_3)_2PdCl_2$ (220 mg, 0.32 mmol), PPh_3 (83 mg, 0.32 mmol), CuI (60 mg, 0.32 mmol), trimethylsilylacetylene (1.34 mL, 0.95 mmol) and Et_3N (1.32 mL, 0.95 mmol) in toluene (20 mL), the product (556 mg, 97%) was obtained as a pale yellow foam after flash column chromatography (eluent: hexane/EtOAc = 8/1); R_f = 0.24 (hexane/EtOAc = 8/1); ^{1}H NMR: δ = 7.47 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 7.28–7.44 (m, 14 H; Ar*H*), 6.87 (s, 4 H; core-Ar*H*), 6.68 (s, 2 H; Ar*H*), 6.65 (s, 2 H; Ar*H*), 6.53 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 5.03 (s, 4 H; ArCH_2O), 5.02 (s, 4 H; ArCH_2O), 4.95 (s, 4 H; ArCH_2O), 0.25 (s, 18 H; Si(CH$_3$)$_3$); ^{13}C NMR: δ = 160.2, 160.0, 153.2, 139.9, 137.4, 136.8, 132.3, 128.7, 128.1, 127.7, 127.2, 122.8, 115.9, 106.5, 106.4, 104.9, 101.6, 94.6, 70.6, 70.2, 69.7, 0.1; MS (ESI): *m/z* (%): 929 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{58}H_{58}O_6Si_2$ + Na$^+$: 929.3664; found: 929.3680; elemental analysis calcd (%) for $C_{58}H_{58}O_6Si_2$: C 76.78, H 6.44; found: C 76.43, H 6.11, N <0.10.

HC≡C–[S-G1]–C≡CH (73). This compound was prepared by General Procedure H. Starting from TMSC≡C–[S-G1]–C≡CTMS **107** (320 mg, 0.35 mmol) and K_2CO_3 (240 mg, 1.76 mmol) in THF/MeOH (v/v = 1/1, 20 mL), the product (265 mg, 98%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/1 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 100/1). R_f = 0.53 (hexane/CH_2Cl_2 = 1/4); ^{1}H NMR: δ = 7.51 (d, J = 8.1 Hz, 4 H; Ar*H*), 7.29–7.46 (m, 14 H; Ar*H*), 6.87 (s, 4 H; core-Ar*H*), 6.69 (s, 2 H; Ar*H*), 6.66 (s, 2 H; Ar*H*), 6.55 (t, J = 2.1 Hz, 2 H; Ar*H*), 5.04 (s, 8 H; PhCH_2O + ArCH_2O), 4.95 (s, 4 H; ArCH_2O), 3.09 (s, 2 H; C≡CH); ^{13}C NMR: δ = 160.3, 160.0, 153.1, 140.0, 137.7, 136.8, 132.5, 128.7, 128.2, 127.7, 127.4, 121.8, 115.9, 106.5, 106.4, 101.6, 83.5, 77.6, 70.6, 70.2, 69.6; MS (ESI): *m/z* (%): 763 (100) [M + H^+]; HRMS (ESI) calcd for $C_{52}H_{42}O_6$ + H^+: 763.3054; found: 763.3060; elemental analysis calcd (%) for $C_{52}H_{42}O_6$: C 81.87, H 5.55; found: C 82.25, H 5.63, N <0.10.

TMSC≡C–[S-G2]–C≡CTMS (108). This compound was prepared by General Procedure G. Starting from I–[S-G2]–I **105** (408 mg, 0.22 mmol), $(PPh_3)_2PdCl_2$ (79 mg, 0.11 mmol), PPh_3 (29 mg, 0.11 mmol), CuI (21 mg, 0.11 mmol), trimethylsilylacetylene (0.48 mL, 3.37 mmol) and Et_3N (0.47 mL, 3.37 mmol) in toluene (10 mL), the product (369 mg, 94%) was obtained as a pale yellow foam after flash column chromatography (eluent: hexane/CH_2Cl_2 = 4/5 to CH_2Cl_2). R_f = 0.54 (hexane/CH_2Cl_2 = 2/3); ^{1}H NMR: δ = 7.47 (d, J = 7.8 Hz, 4 H; Ar*H*), 7.27–7.45 (m, 34 H; Ar*H*), 6.88 (s, 4 H; core-Ar*H*), 6.68–6.72 (m, 6 H; Ar*H*), 6.61–6.68 (m, 6 H; Ar*H*), 6.58 (t, J = 2.1 Hz, 2 H; Ar*H*), 6.50–6.56 (m, 4 H; Ar*H*), 5.03 (s, 12 H; PhCH_2O), 5.02 (s, 4 H; ArCH_2O), 4.98 (s, 4 H; ArCH_2O), 4.97 (s, 4 H; ArCH_2O), 4.93 (s, 4 H; ArCH_2O), 0.27 (s, 18 H; $Si(CH_3)_3$); ^{13}C NMR: δ = 160.1, 160.0, 159.9, 153.1, 139.8, 139.3, 137.3, 136.8, 132.1, 128.6, 128.0, 127.6, 127.2, 122.7, 115.8, 106.4, 105.0, 101.6, 94.5, 70.4, 70.0, 69.9, 69.5, 0.1; MS (ESI): *m/z* (%): 1756 (75) [M + H^+]; HRMS (ESI) calcd for $C_{114}H_{106}O_{14}Si_2$ + H^+: 1755.7194; found: 1755.7201;

elemental analysis calcd (%) for $C_{114}H_{106}O_{14}Si_2$: C 77.96, H 6.08; found: C 77.69, H 5.96, N <0.10.

HC≡C–[S-G2]–C≡CH (74). This compound was prepared by General Procedure H. Starting from TMSC≡C–[S-G2]–C≡CTMS **108** (701 mg, 0.40 mmol) and K_2CO_3 (280 mg, 2.00 mmol) in THF/MeOH (v/v = 1/1, 10 mL), the product (623 mg, 97%) was obtained as a white foam after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/2 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 100/1). R_f = 0.29 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.49 (d, *J* = 8.4, 4 H; Ar*H*), 7.27–7.45 (m, 34 H; Ar*H*), 6.86 (s, 4 H; core-Ar*H*), 6.67–6.71 (m, 6 H; Ar*H*), 6.61–6.67 (m, 6 H; Ar*H*), 6.57 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 6.49–6.56 (m, 4 H; Ar*H*), 5.02 (s, 16 H; PhC*H*$_2$O + ArC*H*$_2$O), 4.97 (s, 8 H; ArC*H*$_2$O), 4.92 (s, 4 H; ArC*H*$_2$O), 3.07 (s, 2 H; C≡CH); ^{13}C NMR: δ = 160.2, 160.12, 160.08, 160.0, 153.2, 139.9, 139.4, 139.3, 137.7, 136.9, 136.8, 132.4, 128.7, 128.1, 127.7, 127.3, 121.8, 115.9, 106.6, 106.5, 106.4, 101.69, 101.66, 101.6, 83.5, 77.6, 70.6, 70.2, 70.04, 69.98, 69.6; MS (ESI): *m/z* (%): 1612 (100) [M + H$^+$]; HRMS (MALDI-TOF) calcd for $C_{108}H_{90}O_{14}$ + Na$^+$: 1634.6257; found: 1634.6292; elemental analysis calcd (%) for $C_{108}H_{90}O_{14}$: C 80.48, H 5.63; found: C 80.10, H 5.30, N <0.10.

TMSC≡C–[S-G3]–C≡CTMS (109). This compound was prepared by General Procedure G. Starting from HC≡C–[S-G3]–C≡CH **106** (346 mg, 0.098 mmol), $(PPh_3)_2PdCl_2$ (79 mg, 0.049 mmol), PPh_3 (29 mg, 0.049 mmol), CuI (21 mg, 0.049 mmol), trimethylsilylacetylene (0.48 mL, 1.48 mmol) and Et_3N (0.47 mL, 1.48 mmol) in toluene (3.5 mL), the product (294 mg, 86%) was obtained as a pale yellow foam after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/2 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 100/1). R_f = 0.46 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.45 (d, *J* = 8.4 Hz, 4 H; ArH), 7.27–7.42 (m, 74 H; Ar*H*), 6.83 (s, 4 H; core-Ar*H*), 6.59–6.71 (m, 28 H; Ar*H*), 6.55 (t, *J* = 2.1 Hz, 8 H; Ar*H*), 6.48–6.54 (m, 6 H; Ar*H*), 4.99 (s, 28 H; PhC*H*$_2$O), 4.98 (s, 4 H; ArC*H*$_2$O), 4.94 (s, 24 H; ArC*H*$_2$O), 4.88 (s, 4 H; ArC*H*$_2$O), 0.25 (s, 18 H; Si(CH$_3$)$_3$); ^{13}C NMR: δ = 160.2, 160.14, 160.05, 160.0, 159.9, 153.1, 139.8, 139.32, 139.26, 137.3, 136.82, 136.79, 136.76, 132.1, 128.6, 128.0, 127.6, 127.2, 122.7, 115.8, 106.4, 105.0, 101.6, 94.5, 70.4, 70.0, 69.9, 69.5, 0.0; HRMS (MALDI-TOF) calcd for $C_{226}H_{202}O_{30}Si_2$ + Na$^+$: 3476.3774; found: 3476.3867; elemental analysis calcd (%) for $C_{226}H_{202}O_{30}Si_2$: C 78.58, H 5.89; found: C 78.37, H 5.83, N <0.10.

HC≡C–[S-G3]–C≡CH (75). This compound was prepared by General Procedure I. Starting from TMSC≡C–[S-G3]–C≡CTMS **109** (357 mg, 10.3 mmol) and TBAF (1.0 M in THF, 0.3 mL) in THF (10 mL), the product (169 mg, 49%) was obtained as a pale yellow foam after flash column chromatography (eluent: hexane/CH$_2$Cl$_2$ = 1/1 to CH$_2$Cl$_2$ to CH$_2$Cl$_2$/Et$_2$O = 50/1). R_f = 0.39 (hexane/CH$_2$Cl$_2$ = 1/3); ^{1}H NMR: δ = 7.46 (d, *J* = 8.4 Hz, 4 H; Ar*H*), 7.27–7.43 (m, 74 H; Ar*H*), 6.82 (s, 4 H; core-Ar*H*), 6.59–6.73 (m, 28 H; Ar*H*), 6.46–6.59 (m, 14 H; Ar*H*), 4.99 (s, 32 H; PhC*H*$_2$O + ArC*H*$_2$O), 4.94 (s, 24 H; ArC*H*$_2$O), 4.87 (s, 4 H; ArC*H*$_2$O), 3.06 (s, 2 H; C≡CH); ^{13}C NMR (one of the C≡C signal was too weak to be observed): δ = 160.3, 160.2, 160.1, 160.0, 153.2, 139.9, 139.4, 139.3, 137.7, 136.9, 136.8, 132.4, 128.7, 128.1, 127.7, 127.4, 121.8, 115.9, 106.5, 101.7, 83.5, 70.6, 70.2, 70.1, 69.6; HRMS (MALDI-TOF) calcd for $C_{220}H_{186}O_{30}$ + Na$^+$: 3332.2988; found: 3332.2790; elemental analysis calcd for $C_{220}H_{186}O_{30}$: C 79.83, H 5.66; found: C 79.77, H 5.66.

(ii). Longer Branch Series (L-Gn Series)

3-Phenylpropanoic acid (111).[86] A mixture of cinnamic acid **110** (30.0 g, 202 mmol) and 10% Pd-C (3.0 g) was stirred in EtOH/EtOAc (*v*/*v* = 1/1, 500 mL) under H_2 at 1 atm for 24 h. The reaction mixture was filtered, dried *in vacuo* to give the target compound (29.9 g, 98%) as a white solid. M.p. 45.4–47.3 °C (lit. M.p. 46–48 °C[86]); R_f = 0.79 (hexane/EtOAc = 1/2); ^{1}H NMR: δ = 11.72 (brs, 1 H; CO_2H), 7.10–7.36 (m, 5 H; Ar*H*), 2.98 (t, *J* = 7.5 Hz, 2 H; C*H*$_2$CH$_2$CO$_2$), 2.70 (t, *J* = 7.2 Hz, 2 H; CH$_2$CO$_2$); ^{13}C NMR: δ = 179.8, 140.2, 128.6, 128.3, 126.4, 35.7, 30.6.

3-(4'-Iodophenyl)propanoic acid (112).[87] A mixture of 3-phenylpropanoic acid **111** (3.00 g, 20.0 mmol), H_5IO_6 (0.98 g, 4.29 mmol), iodine (2.03 g, 7.99 mmol) and 98% H_2SO_4 (0.6 mL) in water (4 mL) and acetic acid (20 mL) was heated to 65–70 °C for 12 h. The reaction mixture was quenched with water (50 mL). The crude product was then filtered, re-dissolved in Et_2O (30 mL), washed with saturated $Na_2S_2O_5$ solution (2 × 30 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo* and recrystallized (heptane) to give the target compound (3.31 g, 60%) as a white crystal. M.p. 139.7–141.7 °C (lit. M.p. 140–142 °C[98]); R_f = 0.74 (hexane/EtOAc = 1/2); ^{1}H NMR: δ = 11.40 (brs, 1 H; CO_2H), 7.61 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.97 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 2.90 (t, *J* = 7.5 Hz, 2 H; C*H*$_2$CH$_2$CO$_2$), 2.66 (t, *J* = 7.8 Hz, 2 H; CH$_2$CO$_2$); ^{13}C NMR: δ = 179.2, 139.8, 137.7, 130.5, 91.7, 35.4, 30.1.

Methyl 3-(4'-iodophenyl)propanoate (113).[99] This compound was prepared according to General Procedure A. Starting from 3-(4'-iodophenyl)propanoic acid **112** (20.1 g, 75.4 mmol) and 98% H_2SO_4 (5 mL) in methanol (350 mL), the product (20.4 g, 93%) was obtained as a white solid. M.p. 47.6–49.5 °C (lit. M.p. 47 °C[99]); R_f = 0.67 (hexane/EtOAc

= 2/1); ^{1}H NMR: δ = 7.60 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.95 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 3.66 (s, 3 H; CO_2CH_3), 2.89 (t, *J* = 7.8 Hz, 2 H; C*H*$_2$CH$_2$CO$_2$), 2.60 (t, *J* = 8.1 Hz, 2 H; CH_2CO_2); ^{13}C NMR: δ = 172.6, 139.9, 137.3, 130.3, 91.4, 51.5, 35.1, 30.2.

3-(4'-Iodophenyl)propan-1-ol (114).[100] This compound was prepared by General Procedure B. Starting from methyl 3-(4'-iodophenyl)propanoate **113** (20.4 g, 70.3 mmol) and DIBAL-H solution (1.0 M in hexane, 176 mL, 176 mmol) in toluene (100 mL), the product (16.8 g, 91%) was obtained as a white solid. M.p. 38.3–39.9 °C; R_f = 0.26 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.60 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.95 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 3.66 (t, *J* = 6.3 Hz, 2 H; C*H*$_2$OH), 2.65 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 1.76–1.95 (m, 2 H; C*H*$_2$CH$_2$OH), 1.42 (brs, 1 H; OH); ^{13}C NMR: δ = 141.5, 137.4, 130.6, 90.9, 61.8, 33.9, 31.5.

1-Bromo-3-(4'-iodophenyl)propane (115).[88] This compound was prepared by General Procedure C. Starting from 3-(4'-iodophenyl)propan-1-ol **114** (16.8 g, 64.2 mmol), PPh_3 (21.0 g, 80.2 mmol) and CBr_4 (26.6 g, 80.2 mmol) in THF (100 mL), the product (19.1 g, 91%) was obtained as a pale pink oil after flash chromatography (eluent: hexane). R_f = 0.61 (hexane); ^{1}H NMR: δ = 7.61 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.96 (d, *J* = 8.1 Hz, 2 H; Ar*H*), .38 (t, *J* = 6.6 Hz, 2 H; CH_2Br), 2.73 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 2.01–2.25 (m, 2 H; C*H*$_2$CH$_2$Br); ^{13}C NMR: δ = 140.2, 137.6, 130.8, 91.4, 33.9, 33.5, 32.9.

3,5-Dibenzyloxybenzaldehyde (116).[89] A mixture of PCC (74.8 g, 0.35 mol) and silica Gel (74.8 g) was added to a stirred solution of S-G1-OH **90** (74.1 g, 0.23 mol) in CH_2Cl_2 (600 mL) at 0 °C and then allowed to warm to 25 °C over a period of 2 h. The solution was filtered by silica gel and dried *in vacuo* to give the target compound (70.6 g, 96%) as a pale orange solid. This product was used in the next reaction without further purification. M.p. 81.2–83.1 °C (lit. M.p. 77.5–79 °C;[89a] 80 °C[89b]); R_f = 0.39 (hexane/EtOAc = 5/1); ^{1}H NMR: δ = 9.91 (s, 1 H; CHO), 7.29–7.61 (m, 10 H; Ar*H*), 7.12 (s, 2 H; Ar*H*), 6.88 (s, 1 H; Ar*H*), 5.10 (s, 4 H; PhCH_2); ^{13}C NMR: δ = 191.9, 160.5, 138.5, 136.3, 128.8, 128.3, 127.7, 108.7, 108.4, 70.4.

Methyl 3-(3',5'-dibenzyloxyphenyl)prop-2*E*-enoate (117).[91] To a suspension of 60% NaH (12.0 g, 0.29 mol) in THF (300 mL), trimethylphosphonoacetate (47.0 mL, 0.29 mol) was added at 0 °C and allowed to stirred for 30 min, then 3,5-dibenzyloxybenzaldehyde **116** (70.6 g, 0.22 mol) in THF (200 mL) was added and the mixture was heated to reflux for 12 h. The reaction mixture was dried *in vacuo*, acidified to pH = 3 and extracted with Et_2O (3 × 400 mL). The combined extracts were washed with brine, dried ($MgSO_4$), filtered, evaporated *in vacuo* and purified by flash chromatography (eluent: hexane/EtOAc = 8/1) to give the target compound (66.5 g, 80%) as a white solid. M.p. 84.2–85.2 °C (lit. M.p. 68–70 °C;[91] 86 °C[89b]); R_f = 0.37 (hexane/EtOAc = 5/1); ^{1}H NMR: δ = 7.60 (d, *J* = 15.9 Hz, 1 H; C*H*=CHCO$_2$), 7.29–7.49 (m, 10 H; Ar*H*), 6.76 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.66 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 6.39 (d, *J* = 15.9 Hz, 1 H; CH=C*H*CO$_2$), 5.05 (s, 4 H; PhCH_2O), 3.81 (s, 3 H; CO_2CH_3); ^{13}C NMR: δ = 167.0, 160.0, 144.6, 136.5, 136.1, 128.5, 127.9, 127.4, 118.2, 107.0, 104.0, 69.9, 51.5.

Methyl 3-(3',5'-dihydroxyphenyl)propanoate (118).[91] A mixture of methyl 3-(3',5'-dibenzyloxyphenyl)prop-2*E*-enoate **117** (15.4 g, 41.0 mmol), 10% Pd-C (1.5 g) and K_2CO_3 (1.5 g) was stirred in EtOH/EtOAc (*v*/*v* = 1/1, 400 mL) under H_2 at 1 atm for 24 h. The reaction mixture was filtered, dried *in vacuo* and purified by flash chromatography (eluent: hexane/EtOAc = 2/1 to 1/1) to give the target compound (5.45 g, 68%) as a white solid. M.p. 112.0–113.5 °C (lit. M.p. 110–112 °C[91]); R_f = 0.33 (hexane/EtOAc = 1/1); ^{1}H

NMR (DMSO-d_6): δ = 9.10 (s, 2 H; ArO*H*), 6.04 (s, 3 H; Ar*H*), 3.58 (s, 3 H; CO_2CH_3), 2.65 (t, *J* = 6.6 Hz, 2 H; C*H*$_2$CH$_2$CO$_2$), 2.53 (t, *J* = 6.6 Hz, 2 H; CH_2CO_2); ^{13}C NMR (DMSO-d_6): δ = 173.1, 158.6, 142.9, 106.6, 100.8, 51.5, 35.1, 30.7.

Hemi-L-G1-ester (120). This compound was prepared by General Procedure F. Starting from methyl 3-(3',5'-dihydroxyphenyl)propanoate **118** (69.1 g, 0.35 mol), 1-bromo-3-phenylpropane **119** (13.4 mL, 88.1 mmol), K_2CO_3 (73.1 g, 0.53 mol) and 18-crown-6 (2 mg) in acetone (600 mL), the product (20.7 g, 75%)[83] was obtained as a pale yellow oil after flash chromatography (eluent: hexane/EtOAc = 3/1 to 1/5). R_f = 0.38 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.27–7.34 (m, 2 H; Ar*H*), 7.14–7.25 (m, 3 H; Ar*H*), 6.32 (s, 1 H; Ar*H*), 6.27 (s, 1 H; Ar*H*), 6.25 (d, *J* = 1.8 Hz, 1 H; Ar*H*), 5.40 (brs, 1 H; ArO*H*), 3.91 (t, *J* = 6.3 Hz, 2 H, ArOC*H*$_2$), 3.68 (s, 3 H; CO_2CH_3), 2.85 (t, *J* = 7.5 Hz, 2 H; C*H*$_2$CH$_2$CO$_2$), 2.79 (t, *J* = 7.5 Hz, 2 H; PhC*H*$_2$), 2.61 (t, *J* = 7.8 Hz, 2 H; CH_2CO_2), 1.99–2.16 (m, 2 H; PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 174.3, 160.3, 157.2, 142.8, 141.5, 128.5, 128.4, 125.9, 107.9, 106.9, 100.1, 66.9, 51.9, 35.5, 32.1, 30.9, 30.8; MS (ESI): *m/z* (%): 337 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{19}H_{22}O_4 + Na^+$: 337.1410; found: 337.1410; elemental analysis calcd (%) for $C_{19}H_{22}O_4$: C 72.59, H 7.05; found: C 72.09, H 7.15.

I-L-G1-ester (121). This compound was prepared by General Procedure D. Starting from hemi-L-G1-ester **120** (18.0 g, 57.3 mmol), 1-bromo-3-(4'-iodophenyl)propane **115** (20.5 g, 63.0 mmol), K_2CO_3 (15.8 g, 115 mmol) and 18-crown-6 (2 mg) in acetone (100 mL), the product (28.1 g, 88%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 8/1). R_f = 0.26 (hexane/EtOAc = 8/1); ^{1}H NMR: δ = 7.61 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.27–7.40 (m, 2 H; Ar*H*), 7.07–7.25 (m, 3 H; Ar*H*), 6.97 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.30–6.39 (m, 2 H; Ar*H*), 6.28 (t, *J* = 2.1 Hz, 1 H;

Ar*H*), 3.93 (t, *J* = 6.3 Hz, 2 H; ArOC*H*$_2$), 3.90 (t, *J* = 6.0 Hz, 2 H; ArOC*H*$_2$), 3.68 (s, 3 H; CO$_2$CH$_3$), 2.88 (t, *J* = 7.2 Hz, 2 H; C*H*$_2$CH$_2$CO$_2$), 2.81 (t, *J* = 7.2 Hz, 2 H; PhC*H*$_2$), 2.75 (t, *J* = 7.8 Hz, 2 H; ArC*H*$_2$), 2.62 (t, *J* = 7.2 Hz, 2 H; CH$_2$CO$_2$), 1.86–2.22 (m, 4 H; PhCH$_2$C*H*$_2$ + ArCH$_2$C*H*$_2$); ^{13}C NMR: δ = 173.1, 160.2, 160.0, 142.7, 141.4, 141.0, 137.3, 130.6, 128.4, 128.3, 125.8, 106.8, 106.7, 99.1, 91.0, 66.7, 66.4, 51.5, 35.4, 32.1, 31.6, 31.1, 30.7, 30.5; MS (ESI): *m/z* (%): 581 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{28}H_{31}IO_4$ + Na$^+$: 581.1159; found: 581.1162; elemental analysis calcd (%) for $C_{28}H_{31}IO_4$: C 60.22, H 5.59; found: C 60.02, H 5.59, N <0.10.

I-L-G1-OH (122). This compound was prepared by General Procedure B. Starting from I-L-G1-ester **121** (26.9 g, 48.2 mmol) and DIBAL-H solution (1.0 M in hexane, 120 mL, 120 mmol) in toluene (100 mL), the product (24.9 g, 97%) was obtained as a colorless oil. *R*$_f$ = 0.13 (hexane/EtOAc = 3/1); ^{1}H NMR: δ = 7.61 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.27–7.37 (m, 2 H; Ar*H*), 7.16–7.25 (m, 3 H; Ar*H*), 6.97 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.32–6.40 (m, 2 H; Ar*H*), 6.29 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 3.94 (t, *J* = 6.3 Hz, 2 H; ArOC*H*$_2$), 3.92 (t, *J* = 6.0 Hz, 2 H; ArOC*H*$_2$), 3.68 (t, *J* = 5.7 Hz, 2 H; C*H*$_2$OH), 2.82 (t, *J* = 7.2 Hz, 2 H; PhC*H*$_2$), 2.76 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 2.65 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 1.97–2.22 (m, 4 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$), 1.76–1.97 (m, 2 H; C*H*$_2$CH$_2$OH), 1.40 (brs, 1 H; O*H*); ^{13}C NMR: δ = 160.1, 159.9, 144.1, 141.4, 141.0, 137.3, 130.6, 128.4, 128.3, 125.8, 107.1, 107.0, 98.7, 91.0, 66.7, 66.5, 61.9, 33.9, 32.3, 32.0, 31.6, 30.7, 30.5; MS (ESI): *m/z* (%): 531 (100) [M + H$^+$]; HRMS (ESI) calcd for $C_{27}H_{31}IO_3$ + H$^+$: 531.1391; found: 531.1398; elemental analysis calcd (%) for $C_{27}H_{31}IO_3$: C 61.14, H 5.89; found: C 60.92, H 6.06, N <0.1.

I-L-G1-Br (123). This compound was prepared by General Procedure C. Starting from I-L-G1-OH **122** (23.7 g, 44.7 mmol), PPh_3 (14.7 g, 55.9 mmol) and CBr_4 (18.5 g, 55.9 mmol) in THF (80 mL), the product (25.5 g, 96%) was obtained as a colorless oil after flash chromatography (eluent: hexane/EtOAc = 20/1). $R_f = 0.29$ (hexane/EtOAc = 20/1); ^{1}H NMR: δ = 7.61 (d, J = 8.4 Hz, 2 H; Ar*H*), 7.27–7.36 (m, 2 H; Ar*H*), 7.15–7.25 (m, 3 H; Ar*H*), 6.97 (d, J = 8.1 Hz, 2 H; Ar*H*), 6.31–6.38 (m, 2 H; Ar*H*), 6.29 (t, J = 2.1 Hz, 1 H; Ar*H*), 3.94 (t, J = 6.3 Hz, 2 H; $ArOCH_2$), 3.91 (t, J = 6.3 Hz, 2 H; $ArOCH_2$), 3.40 (t, J = 6.6 Hz, 2 H; CH_2Br), 2.81 (t, J = 7.5 Hz, 2 H; $PhCH_2$), 2.63–2.78 (m, 4 H; $ArCH_2$), 1.96–2.25 (m, 6 H; $ArCH_2CH_2$ + $PhCH_2CH_2$); ^{13}C NMR: δ = 160.2, 160.1, 142.8, 141.4, 141.1, 137.4, 130.7, 128.5, 128.4, 125.9, 107.2, 107.1, 99.1, 91.1, 66.8, 66.5, 34.2, 33.9, 33.2, 32.1, 31.7, 30.8, 30.6; MS (ESI): *m/z* (%): 593 (100) [M(^{79}Br) + H$^+$]; HRMS (ESI) calcd for $C_{27}H_{30}BrIO_2 + H^+$: 593.0547; found: 593.0545; elemental analysis calcd (%) for $C_{27}H_{30}BrIO_2$: C 54.66, H 5.10; found: C 54.26, H 5.12.

L-G1-ester (124). This compound was prepared according to General Procedure D. Starting from methyl 3-(3’,5’-dihydroxyphenyl)propanoate **118** (27.0 g, 13.8 mmol), 1-bromo-3-phenylpropane **119** (46.0 mL, 30.3 mmol), K_2CO_3 (57.1 g, 41.3 mmol) and 18-crown-6 (2 mg) in acetone (400 mL), the product (58.5 g, 98%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 8/1). $R_f = 0.28$ (hexane/EtOAc = 8/1); ^{1}H NMR: δ = 7.27–7.36 (m, 4 H; Ar*H*), 7.15–7.25 (m, 6 H; Ar*H*), 6.35 (d, J = 2.1 Hz, 2 H; Ar*H*), 6.31 (t, J = 2.1 Hz, 1 H; Ar*H*), 3.93 (t, J = 6.3 Hz, 4 H; $ArOCH_2$), 3.68 (s, 3 H; CO_2CH_3), 2.88 (t, J = 7.5 Hz, 2 H; $CH_2CH_2CO_2$), 2.81 (t, J = 7.5 Hz, 4 H; $PhCH_2$), 2.62 (t, J = 7.5 Hz, 2 H; CH_2CO_2), 2.01–2.19 (m, 4 H; $PhCH_2CH_2$); ^{13}C NMR: δ = 173.2, 160.2, 142.7, 141.5, 128.5, 128.4, 125.9, 106.8, 99.2, 66.7, 51.5, 35.5, 32.1, 31.2, 30.8; MS (ESI): *m/z* (%): 455 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{28}H_{32}O_4$ + Na$^+$: 455.2193; found: 455.2198; elemental analysis calcd (%) for $C_{28}H_{32}O_4$: C 77.75, H 7.46; found: C 77.72, H 7.43.

L-G1-OH (125). This compound was prepared according to General Procedure E. Starting from L-G1-ester **124** (58.6 g, 135 mmol) and LAH (5.14 g, 135 mmol) in THF (250 mL), the product (54.0 g, 99%) was obtained as a colorless oil. R_f = 0.23 (hexane/EtOAc = 3/1); ^{1}H NMR: δ = 7.12–7.37 (m, 10 H; Ar*H*), 6.37 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 6.31 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 3.95 (t, *J* = 6.3 Hz, 4 H; ArOC*H*$_2$), 3.69 (t, *J* = 6.0 Hz, 2 H; C*H*$_2$OH), 2.82 (t, *J* = 7.5 Hz, 4 H; PhC*H*$_2$), 2.65 (t, *J* = 7.5 Hz, 2 H; ArC*H*$_2$), 1.97–2.23 (m, 4 H; PhCH$_2$C*H*$_2$), 1.78–1.97 (m, 2 H; C*H*$_2$CH$_2$OH), 1.39 (brs, 1 H; OH); ^{13}C NMR: δ = 160.0, 144.1, 141.2, 128.24, 128.16, 125.7, 106.9, 98.6, 66.5, 61.5, 33.8, 32.2, 31.9, 30.6; MS (ESI): *m/z* (%): 427 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{27}H_{32}O_3$ + Na$^+$: 427.2244; found: 427.2241; elemental analysis calcd (%) for $C_{27}H_{32}O_3$: C 80.16, H 7.97; found: C 80.40, H 8.15.

L-G1-Br (126). This compound was prepared according to General Procedure C. Starting from L-G1-OH **125** (51.5 g, 127 mmol), PPh_3 (41.7 g, 159 mmol) and CBr_4 (52.8 g, 159 mmol) in THF (150 mL), the product (56.6 g, 95%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 20/1). R_f = 0.24 (hexane/EtOAc = 6/1); ^{1}H NMR: δ = 7.12–7.40 (m, 10 H; Ar*H*), 6.34 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.31 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 3.94 (t, *J* = 6.3 Hz, 4 H; ArOC*H*$_2$), 3.40 (t, *J* = 6.6 Hz, 2 H; CH$_2$Br), 2.81 (t, *J* = 7.8 Hz, 4 H; PhC*H*$_2$), 2.70 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 1.98–2.26 (m, 6 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 160.3, 142.7, 141.5, 128.5, 128.4, 125.9, 107.2, 99.1, 66.8, 34.2, 33.9, 33.2, 32.1, 30.8; MS (ESI): *m/z* (%): 489 (100) [M(^{79}Br) + Na$^+$];

HRMS (ESI) calcd for $C_{27}H_{31}BrO_2 + Na^+$: 489.1400; found: 489.1398; elemental analysis calcd (%) for $C_{27}H_{31}BrO_2$: C 69.38, H 6.68; found: C 69.30, H 6.74.

Hemi-L-G2-ester (127). This compound was prepared by General Procedure F. Starting from methyl 3-(3',5'-dihydroxyphenyl)propanoate **118** (32.0 g, 16.3 mmol), L-G1-Br **126** (19.1 g, 40.8 mmol), K_2CO_3 (33.8 g, 24.5 mmol) and 18-crown-6 (2 mg) in acetone (150 mL), the product (17.9 g, 75%)[83] was obtained as a pale pink oil after flash column chromatography (eluent: hexane/EtOAc = 3/1 to 1/5). $R_f = 0.35$ (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.27–7.35 (m, 4 H; Ar*H*), 7.14–7.25 (m, 6 H; Ar*H*), 6.37 (d, *J* = 2.1 Hz, 2 H; Ar*H*), 6.32–6.35 (m, 1 H; Ar*H*), 6.31 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 6.20–6.29 (m, 2 H; Ar*H*), 5.24 (brs, 1 H; ArO*H*), 3.84–3.98 (m, 6 H; ArOC*H*$_2$), 3.68 (s, 3 H; CO_2CH_3), 2.75–2.92 (m, 6 H; ArC*H*$_2$ + PhC*H*$_2$), 2.72 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 2.61 (t, *J* = 7.5 Hz, 2 H; C*H*$_2$CO$_2$), 1.99–2.17 (m, 6 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 173.9, 160.3, 160.1, 157.2, 143.8, 142.8, 141.5, 128.44, 128.35, 125.9, 107.8, 107.2, 106.8, 100.0, 99.1, 66.8, 51.7, 35.3, 32.3, 32.1, 30.9, 30.8, 30.5; MS (ESI): *m/z* (%): 605 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{37}H_{42}O_6 + Na^+$: 605.2874; found: 605.2868; elemental analysis calcd (%) for $C_{37}H_{42}O_6$: C 76.26, H 7.26; found: C 76.53, H 7.65.

I-L-G2-ester (128). This compound was prepared by General Procedure D. Starting from hemi-L-G2-ester **127** (17.3 g, 29.6 mmol), I-L-G1-Br **123** (19.3 g, 32.6 mmol), K_2CO_3 (8.19 g, 59.3 mmol) and 18-crown-6 (2 mg) in acetone (150 mL), the product (27.6 g, 85%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 4/1). $R_f = 0.35$ (hexane/EtOAc = 4/1); ^{1}H NMR: δ = 7.60 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 7.25–7.37 (m, 6 H; Ar*H*), 7.10–7.25 (m, 9 H; Ar*H*), 6.96 (d, *J* = 7.8 Hz, 2 H; Ar*H*), 6.20–6.49 (m, 9 H; Ar*H*), 3.81–4.00 (m, 12 H; ArOC*H*$_2$), 3.67 (s, 3 H; CO_2CH_3), 2.66–2.94 (m, 14 H; ArC*H*$_2$ + PhC*H*$_2$), 2.61 (t, *J* = 7.8 Hz, 2 H; C*H*$_2$CO$_2$), 1.93–2.20 (m, 12 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 172.9, 160.14, 160.06, 159.9, 143.6, 143.5, 142.6, 141.3, 141.0, 137.2, 130.5, 128.3, 128.2, 125.8, 106.9, 106.7, 99.1, 98.8, 90.9, 66.5, 66.3, 51.3, 35.3, 32.3, 32.0, 31.5, 31.0, 30.7, 30.4; MS (ESI): *m/z* (%): 1095 (100) [M + H$^+$]; HRMS (ESI) calcd for $C_{64}H_{71}IO_8$ + H$^+$: 1095.4266; found: 1095.4263.

I-L-G2-OH (129). This compound was prepared by General Procedure B. Starting from I-L-G2-ester **128** (26.1 g, 23.8 mmol) and DIBAL-H (1.0 M in hexane, 60.0 mL, 60.0 mmol) in toluene (100 mL), the product (24.9 g, 98%) was obtained as a colorless oil. *R*$_f$ = 0.34 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.60 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.26–7.37 (m, 6 H; Ar*H*), 7.10–7.24 (m, 9 H; Ar*H*), 6.96 (d, *J* = 8.4 Hz, 2 H; ArH), 6.24–6.41 (m, 9 H; Ar*H*), 3.79–4.04 (m, 12 H; ArOC*H*$_2$), 3.66 (q, *J* = 6.3 Hz, 2 H; C*H*$_2$OH), 2.67–2.87 (m, 12 H; ArC*H*$_2$ + PhC*H*$_2$), 2.63 (t, *J* = 7.2 Hz, 2 H; ArC*H*$_2$), 1.95–2.19 (m, 12 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$), 1.79–1.95 (m, 2 H; C*H*$_2$CH$_2$OH), 1.27 (t, *J* = 5.4 Hz, 1 H; OH); ^{13}C NMR: δ = 160.19, 160.17, 160.16, 160.0, 144.2, 143.8, 143.7, 141.48, 141.47, 141.1, 137.4, 130.6, 128.5, 128.4, 125.9, 107.2, 107.14, 107.10, 98.94, 98.90, 91.0, 66.8, 66.5, 62.1, 34.0, 32.42, 32.37, 32.1, 31.6, 30.8, 30.6 ppm; MS (ESI): *m/z* (%): 1068 (100) [M + H$^+$]; HRMS (ESI) calcd for $C_{63}H_{71}IO_7$ + H$^+$: 1067.4317; found: 1067.4330; elemental analysis calcd (%) for $C_{63}H_{71}IO_7$: C 70.91, H 6.71; found: C 71.02, H 6.82.

I-L-G2-Br (130). This compound was prepared by General Procedure C. Starting from I-L-G2-OH **129** (21.7 g, 20.3 mmol), PPh$_3$ (6.65 g, 25.4 mmol) and CBr$_4$ (8.41 g, 25.4 mmol) in THF (100 mL), the product (20.8 g, 91%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 6/1). *R*$_f$ = 0.35 (hexane/EtOAc = 6/1); ^{1}H NMR: δ = 7.61 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.27–7.41 (m, 6 H; Ar*H*), 7.10–7.25

(m, 9 H; Ar*H*), 6.97 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.25–6.42 (m, 9 H; Ar*H*), 3.84–4.00 (m, 12 H; ArOC*H*$_2$), 3.39 (t, *J* = 6.6 Hz, 2 H; CH$_2$Br), 2.62–2.89 (m, 14 H; ArC*H*$_2$ + PhC*H*$_2$), 1.97–2.24 (m, 14 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 160.21, 160.20, 160.12, 160.11, 160.0, 143.61, 143.55, 142.6, 141.3, 141.0, 137.2, 130.5, 128.4, 128.3, 125.8, 107.0, 99.1, 98.9, 91.0, 66.6, 66.3, 34.1, 33.8, 33.1, 32.3, 32.0, 31.5, 30.7, 30.52, 30.47; MS (ESI): *m/z* (%): 1129 (90) [M(^{79}Br) + H$^+$]; HRMS (ESI) calcd for $C_{63}H_{70}BrIO_6$ + H$^+$: 1129.3473; found: 1129.3473; elemental analysis calcd (%) for $C_{63}H_{70}BrIO_6$: C 66.96, H 6.24; found: C 66.87, H 6.31.

L-G2-ester (131). This compound was prepared by General Procedure D. Starting from methyl 3-(3',5'-dihydroxyphenyl)propanoate **118** (3.82 g, 19.5 mmol), L-G-Br **126** (20.0 g, 42.8 mmol), K_2CO_3 (8.07 g, 58.4 mmol) and 18-crown-6 (2 mg) in acetone (150 mL), the product (15.8 g, 84%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/CH$_2$Cl$_2$ = 3/2 to CH$_2$Cl$_2$). *R*$_f$ = 0.32 (hexane/EtOAc = 4/1); ^{1}H NMR: δ = 7.27–7.35 (m, 8 H; Ar*H*), 7.15–7.25 (m, 12 H; Ar*H*), 6.31–6.40 (m, 7 H; Ar*H*), 6.30 (t, *J* = 2.4 Hz, 2 H; Ar*H*), 3.81–4.00 (m, 12 H; ArOC*H*$_2$), 3.67 (s, 3 H; CO$_2$CH$_3$), 2.66–2.92 (m, 14 H; ArC*H*$_2$ + PhC*H*$_2$), 2.61 (t, *J* = 8.1 Hz, 2 H; CH$_2$CO$_2$), 1.98–2.17 (m, 12 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 172.8, 160.2, 160.1, 143.5, 142.6, 141.3, 128.3, 128.2, 125.7, 106.9, 106.6, 99.1, 98.8, 66.5, 51.2, 35.2, 32.2, 31.9, 31.0, 30.7, 30.4; MS (ESI): *m/z* (%): 991 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{64}H_{72}O_8$ + Na$^+$: 991.5119; found: 991.5127.

L-G2-OH (132). This compound was prepared by General Procedure B. Starting from L-G2-ester **131** (17.5 g, 18.1 mmol) and DIBAL-H (1.0 M in hexane, 45.2 mL, 45.2 mmol) in toluene (100 mL), the product (16.2 g, 95%) was obtained as a colorless oil. *R*$_f$ = 0.26

(hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.26–7.38 (m, 8 H; Ar*H*), 7.14–7.25 (m, 12 H; Ar*H*), 6.26–6.42 (m, 9 H; Ar*H*), 3.84–4.02 (m, 12 H; ArOC*H*$_2$), 3.66 (q, *J* = 6.0 Hz, 2 H; C*H*$_2$OH), 2.80 (t, *J* = 7.2 Hz, 8 H; PhC*H*$_2$), 2.73 (t, *J* = 7.2 Hz, 4 H; ArC*H*$_2$), 2.63 (t, *J* = 7.5 Hz, 2 H; ArC*H*$_2$), 1.98–2.20 (m, 12 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$), 1.80–1.95 (m, 2 H; C*H*$_2$CH$_2$OH), 1.29 (t, *J* = 5.1 Hz, 1 H; OH); ^{13}C NMR: δ = 160.21, 160.17, 144.2, 143.8, 141.5, 128.5, 128.4, 125.9, 107.14, 107.07, 99.0, 98.9, 66.8, 62.0, 34.0, 32.41, 32.37, 32.1, 30.8, 30.6; MS (ESI): *m/z* (%): 963 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{63}H_{72}O_7$ + Na$^+$: 963.5170; found: 963.5180; elemental analysis calcd (%) for $C_{63}H_{72}O_7$: C 80.39, H 7.71; found: C 80.43, H 7.83.

L-G2-Br (133). This compound was prepared by General Procedure C. Starting from L-G2-OH **132** (16.2 g, 17.2 mmol), PPh$_3$ (5.65 g, 21.5 mmol) and CBr$_4$ (7.14 g, 21.5 mmol) in THF (50 mL), the product (16.3 g, 94%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 8/1). *R*$_f$ = 0.32 (hexane/EtOAc = 8/1); ^{1}H NMR: δ = 7.26–7.36 (m, 8 H; Ar*H*), 7.15–7.25 (m, 12 H; Ar*H*), 6.27–6.41 (m, 9 H; Ar*H*), 3.85–4.00 (m, 12 H; ArOC*H*$_2$), 3.39 (t, *J* = 6.3 Hz, 2 H; CH$_2$Br), 2.63–2.86 (m, 14 H; ArC*H*$_2$ + PhC*H*$_2$), 1.97–2.24 (m, 14 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 160.3, 160.2, 143.8, 142.8, 141.5, 128.5, 128.4, 125.9, 107.2, 99.2, 99.0, 66.9, 66.8, 34.3, 34.0, 33.2, 32.5, 32.2, 30.9, 30.6; MS (ESI): *m/z* (%): 1003 (75) [M(^{79}Br) + H$^+$]; HRMS (ESI) calcd for $C_{63}H_{71}BrO_6$ + H$^+$: 1003.4507; found: 1003.4503; elemental analysis calcd (%) for $C_{63}H_{71}BrO_6$: C 75.36, H 7.13; found: C 75.43, H 7.28.

Hemi-L-G3-ester (134). This compound was prepared by General Procedure F. Starting from methyl 3-(3’,5’-dihydroxyphenyl)propanoate **118** (10.9 g, 55.5 mmol), L-G2-Br **133** (13.9 g, 13.9 mmol), K$_2$CO$_3$ (11.5 g, 83.2 mmol) and 18-crown-6 (2 mg) in acetone (100 mL), the product (12.2 g, 78%)[83] was obtained as a colorless oil after flash column chromatography (eluent: hexane/EtOAc = 3/1 to 1/5, then CH_2Cl_2 to CH_2Cl_2/EtOAc = 120/1). R_f = 0.32 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.19–7.29 (m, 8 H; Ar*H*), 7.09–7.19 (m, 12 H; Ar*H*), 6.22–6.35 (m, 10 H; Ar*H*), 6.20 (brs, 1 H; Ar*H*), 6.15 (t, *J* = 2.1 Hz, 1 H; Ar*H*), 4.90 (brs, 1 H; ArO*H*), 3.76–3.93 (m, 14 H; ArOC*H*$_2$), 3.61 (s, 3 H; CO_2CH_3), 2.59–2.85 (m, 16 H; ArC*H*$_2$ + PhC*H*$_2$), 2.54 (t, *J* = 8.1 Hz, 2 H; C*H*$_2$CO$_2$), 1.91–2.11 (m, 14 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 173.6, 160.2, 160.1, 160.0, 157.2, 143.7, 142.7, 141.3, 128.3, 128.2, 125.8, 107.6, 107.0, 106.7, 99.8, 98.9, 66.6, 51.5, 35.2, 32.3, 31.9, 30.7, 30.5; MS (ESI): *m/z* (%): 1142 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{73}H_{82}O_{10}$ + Na$^+$: 1141.5800; found: 1141.5801.

I-L-G3-ester (135). This compound was prepared by General Procedure D. Starting from hemi-L-G3-ester **134** (9.94 g, 8.88 mmol), I-L-G2-Br **130** (11.0 g, 9.77 mmol), K_2CO_3 (2.45 g, 17.8 mmol) and 18-crown-6 (2 mg) in acetone (60 mL), the product (13.9 g, 72%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/CH_2Cl_2 = 2/3 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 50/1). R_f = 0.56 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.58 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 7.26–7.33 (m, 14 H; Ar*H*), 7.10–7.24 (m, 21 H; Ar*H*), 6.94 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.23–6.41 (m, 21 H; Ar*H*), 3.81–3.97 (m, 28 H; ArOC*H*$_2$), 3.65 (s, 3 H; CO_2CH_3), 2.64–2.90 (m, 30 H; ArC*H*$_2$ + PhC*H*$_2$), 2.59 (t, *J* = 7.5 Hz, 2 H; C*H*$_2$CO$_2$), 1.94–2.15 (m, 28 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 173.3, 160.32, 160.26, 160.1, 143.9, 142.9, 141.6, 141.2, 137.5, 130.7, 128.6, 128.5, 128.2, 126.0, 107.2, 106.9, 99.4, 99.0, 91.0, 66.9, 66.6, 51.7, 35.6, 32.5, 32.2, 31.7, 31.3, 30.9, 30.7; MS (ESI): *m/z* (%): 2191 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{136}H_{151}IO_{16}$ + Na$^+$: 2190.9973; found: 2190.9956.

I-L-G3-OH (136). This compound was prepared by General Procedure B. Starting from I-L-G3-ester **135** (12.9 g, 5.96 mmol) and DIBAL-H (1.0 M in hexane, 18.0 mL, 18.0 mmol) in toluene (20 mL), the product (12.7 g, 99%) was obtained as a colorless oil. R_f = 0.26 (hexane/CH_2Cl_2 = 1/4); ^{1}H NMR: δ = 7.59 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.26–7.35 (m, 14 H; Ar*H*), 7.10–7.23 (m, 21 H; Ar*H*), 6.95 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 6.24–6.40 (m, 21 H; Ar*H*), 3.80–3.99 (m, 28 H; ArOCH_2), 3.64 (q, *J* = 6.0 Hz, 2 H; CH_2OH), 2.65–2.84 (m, 28 H; ArCH_2 + PhCH_2), 2.61 (t, *J* = 7.2 Hz, 2 H; ArCH_2), 1.94–2.15 (m, 28 H; ArCH_2CH_2 + PhCH_2CH_2), 1.78–1.91 (m, 2 H; C$H_2$$CH_2$OH), 1.29 (t, *J* = 5.1 Hz, 1 H; OH); ^{13}C NMR: δ = 160.30, 160.27, 160.2, 160.1, 144.2, 143.89, 143.85, 141.58, 141.56, 141.2, 137.5, 130.7, 128.6, 128.5, 126.0, 107.2, 99.1, 99.0, 91.0, 66.92, 66.86, 66.6, 62.2, 34.0, 32.5, 32.4, 32.2, 31.7, 30.9, 30.8, 30.7; MS (ESI): *m/z* (%): 2163 (100) [M + Na^+]; HRMS (ESI) calcd for $C_{135}H_{151}IO_{15}$ + Na^+: 2163.0023; found: 2163.0035; elemental analysis calcd (%) for $C_{135}H_{151}IO_{15}$: C 75.75, H 7.11; found: C 75.86, H 7.40.

I-L-G3-I (137). *N*-Iodosuccinimide (0.86 g, 3.80 mmol) was added to a solution of I-L-G3-OH **136** (6.50 g, 3.04 mmol) and PPh_3 (1.00 g, 3.80 mmol) in CH_2Cl_2 (20 mL) at 0 °C. The mixture was then stirred at 25 °C for 12 h. The reaction mixture was filtered, dried *in vacuo* and purified by flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/1 to CH_2Cl_2) to give the target compound (5.72 g, 84%) as a colorless oil. R_f = 0.47 (hexane/CH_2Cl_2 = 1/4); ^{1}H NMR: δ = 7.59 (d, *J* = 8.4 Hz, 2 H; Ar*H*), 7.26–7.35 (m, 14 H; Ar*H*), 7.10–7.24 (m, 21 H; Ar*H*), 6.95 (d, *J* = 8.1 Hz, 2 H; Ar*H*), 6.23–6.48 (m, 21 H; Ar*H*), 3.81–4.03 (m, 28 H; ArOCH_2), 3.15 (t, *J* = 6.9 Hz, 2 H; CH_2I), 2.66–2.84 (m, 28 H; ArCH_2 + PhCH_2), 2.63 (t, *J* = 7.5 Hz, 2 H; ArCH_2), 1.94–2.19 (m, 30 H; ArCH_2CH_2 + PhCH_2CH_2); ^{13}C NMR (some overlapping peaks): δ = 160.2, 160.15, 160.10, 160.0, 143.7, 143.6, 142.5, 141.4, 141.0, 137.2, 130.5, 128.7, 128.4, 128.3, 128.0, 125.8, 107.5, 107.0, 106.2, 98.9, 91.0, 67.4, 67.2, 66.6, 66.3, 36.4, 34.6, 32.4, 32.0, 31.5, 30.7, 30.6, 29.39, 29.37, 6.4; HRMS (MALDI-TOF) calcd for $C_{135}H_{150}I_2O_{14}$ + Na^+: 2272.9041; found: 2272.9048; elemental analysis calcd (%) for $C_{135}H_{150}I_2O_{14}$: C 72.05, H 6.72; found: C 72.42, H 6.51.

I–[L-G1]–I (138). A mixture of hydroquinone **103** (216 mg, 1.97 mmol), I-L-G1-Br **123** (2.57 g, 4.32 mmol), K_2CO_3 (815 mg, 5.90 mmol) and 18-crown-6 (2 mg) in acetone (50 mL) was heated to reflux for 3 d. The mixture was filtered and washed with CH_2Cl_2. The combined filtrate was concentrated *in vacuo* and the residue was chromatographed on silica gel to give the product (2.05 g, 92%) as a colorless oil (eluent: hexane/EtOAc = 10/1). R_f = 0.30 (hexane/EtOAc = 10/1); ^{1}H NMR: δ = 7.62 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 7.27–7.39 (m, 4 H; Ar*H*), 7.13–7.25 (m, 6 H; Ar*H*), 6.98 (d, *J* = 8.4 Hz, 4 H; Ar*H*), 6.84 (s, 4 H; core-Ar*H*), 6.38 (s, 2 H; Ar*H*), 6.36 (s, 2 H; Ar*H*), 6.30 (s, 2 H; Ar*H*), 3.83–4.00 (m, 12 H; ArOCH_2), 2.68–2.88 (m, 12 H; ArCH_2 + PhCH_2), 1.95–2.20 (m, 12 H; ArCH$_2$CH_2 + PhCH$_2$CH_2); ^{13}C NMR: δ = 160.2, 160.1, 153.2, 143.9, 141.5, 141.2, 137.4, 130.7, 128.5, 128.4, 125.9, 115.4, 107.2, 107.1, 98.9, 91.1, 67.4, 66.8, 66.5, 32.5, 32.2, 31.7, 30.9, 30.7, 30.6; MS (ESI): *m/z* (%): 1135 (100) [M + H^+]; HRMS (ESI) calcd for $C_{60}H_{64}I_2O_6$ + H^+: 1135.2865; found: 1135.2875; elemental analysis calcd (%) for $C_{60}H_{64}I_2O_6$: C 63.50, H 5.68; found: C 63.58, H 5.83.

I–[L-G2]–I (139). A mixture of hydroquinone **103** (111 mg, 1.01 mmol), I-L-G2-Br **130** (2.50 g, 2.21 mmol), K_2CO_3 (0.42 g, 3.02 mmol) and 18-crown-6 (2 mg) in acetone (15 mL) was heated to reflux for 3 d. The mixture was filtered and washed with CH_2Cl_2. The combined filtrates were concentrated *in vacuo* and the residue was chromatographed on silica gel to give the product (1.73 g, 77%) as a colorless oil (eluent: hexane/CH_2Cl_2 = 1/1 to CH_2Cl_2). $R_f = 0.50$ (hexane/CH_2Cl_2 = 2/3); ^{1}H NMR: $\delta = 7.57$ (d, $J = 8.1$ Hz, 4 H; Ar*H*), 7.27–7.33 (m, 12 H; Ar*H*), 7.08–7.27 (m, 18 H; Ar*H*), 6.92 (d, $J = 7.8$ Hz, 4 H; Ar*H*), 6.79 (s, 4 H; core-Ar*H*), 6.21–6.40 (m, 18 H; Ar*H*), 3.77–3.96 (m, 28 H; ArOCH_2), 2.61–2.83 (m, 28 H; ArCH_2 + PhCH_2), 1.91–2.16 (m, 28 H; ArCH_2CH_2 + PhCH_2CH_2); ^{13}C NMR: δ = 160.2, 160.13, 160.11, 160.0, 153.0, 143.7, 143.64, 143.59, 141.34, 141.33, 141.0, 137.2, 130.5, 128.4, 128.3, 125.8, 115.3, 107.0, 98.9, 91.0, 67.2, 66.6, 66.3, 32.3, 32.0, 31.5, 30.7, 30.5, 30.4; MS (ESI): *m/z* (%): 2208 (65) [M + H^+]; HRMS (ESI) calcd for $C_{132}H_{144}I_2O_{14}$ + H^+: 2207.8718; found: 2207.8739; elemental analysis calcd (%) for $C_{132}H_{144}I_2O_{14}$: C 71.79, H 6.57; found: C 71.55, H 6.75, N <0.10.

I–[L-G3]–I (140). A mixture of hydroquinone **103** (66.7 mg, 0.61 mmol), I-L-G3-I **137** (3.00 g, 1.33 mmol), K_2CO_3 (0.25 g, 1.82 mmol), and 18-crown-6 (2 mg) in acetone (50 mL) was heated to reflux for 3 d. The mixture was filtered and washed with CH_2Cl_2. The combined filtrates were evaporated *in vacuo* to give the product (1.07 g, 41%) as a colorless oil after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/2 to CH_2Cl_2). $R_f = 0.47$ (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: $\delta = 7.57$ (d, $J = 8.4$ Hz, 4 H; Ar*H*), 7.10–7.36 (m, 70 H; Ar*H*), 6.93 (d, $J = 8.1$ Hz, 4 H; Ar*H*), 6.80 (s, 4 H; core-Ar*H*), 6.19–6.48 (m, 42 H; Ar*H*), 3.73–4.02 (m, 60 H; ArOCH_2), 2.59–2.86 (m, 60 H; ArCH_2 + PhCH_2), 1.91–2.16 (m, 60 H; ArCH_2CH_2 + PhCH_2CH_2); ^{13}C NMR: δ = 160.4, 160.31, 160.30, 160.28, 160.2, 153.3, 144.1, 144.01, 143.98, 143.9, 141.7, 141.6, 141.3, 137.5, 130.8, 128.6, 128.5, 126.0, 115.5, 107.3, 107.2, 99.0, 91.1, 67.8, 67.1, 67.0, 66.9, 66.6, 32.7, 32.6, 32.3, 31.8, 31.0, 30.9, 30.8; HRMS (MALDI-TOF) calcd for $C_{276}H_{304}I_2O_{30}$ + Na^+: 4378.0344;

found: 4378.0749; elemental analysis calcd (%) for $C_{276}H_{304}I_2O_{30}$: C 76.12, H 7.04; found: C 76.12, H 6.97.

TMSC≡C–[L-G1]–C≡CTMS (141). This compound was prepared by General Procedure G. Starting from I–[L-G1]–I **138** (59.5 mg, 0.52 mmol), $(PPh_3)_2PdCl_2$ (180 mg, 0.26 mmol), PPh_3 (69 mg, 0.26 mmol), CuI (50 mg, 0.26 mmol), trimethylsilylacetylene (1.11 mL, 7.86 mmol) and Et_3N (1.10 mL, 7.86 mmol) in toluene (20 mL), the product (357 mg, 63%) was obtained as a pale yellow oil after flash column chromatography (eluent: hexane/EtOAc = 8/1). R_f = 0.28 (hexane/EtOAc = 8/1); ^{1}H NMR: δ = 7.39 (d, *J* = 8.4 Hz, 4 H; Ar*H*), 7.26–7.34 (m, 4 H; Ar*H*), 7.16–7.25 (m, 6 H; Ar*H*), 7.14 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 6.82 (s, 4 H; core-Ar*H*), 6.30–6.39 (m, 4 H; Ar*H*), 6.27 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 3.79–3.97 (m, 12 H; $ArOCH_2$), 2.63–2.86 (m, 12 H; $ArCH_2$ + $PhCH_2$), 1.94–2.16 (m, 12 H; $ArCH_2CH_2$ + $PhCH_2CH_2$), 0.24 (s, 18 H; $Si(CH_3)_3$); ^{13}C NMR: δ = 160.24, 160.16, 153.2, 143.9, 142.3, 141.6, 132.1, 128.6, 128.50, 128.47, 126.0, 120.7, 115.5, 107.24, 107.16, 105.3, 99.0, 93.6, 67.4, 66.8, 66.6, 32.5, 32.2, 32.1, 30.9, 30.8, 30.6, 0.1; MS (ESI): *m/z* (%) 1097 (100) [M + Na$^+$]; HRMS (ESI) calcd for $C_{70}H_{82}O_6Si_2$ + Na$^+$: 1097.5542; found: 1097.5532; elemental analysis calcd (%) for $C_{70}H_{82}O_6Si_2$: C 78.17, H 7.68; found: C 77.83, H 7.61.

HC≡C–[L-G1]–C≡CH (76). This compound was synthesized by General Procedure H. Starting from TMSC≡C–[L-G1]–C≡CTMS **141** (301 mg, 0.28 mmol) and K_2CO_3 (190 mg, 1.40 mmol) in THF/MeOH (v/v = 1/1, 20 mL), the product (242 mg, 93%) was obtained as a pale yellow oil after flash chromatography (eluent: hexane/EtOAc = 6/1). R_f = 0.32 (hexane/ EtOAc = 5/1); ^{1}H NMR: δ = 7.43 (d, *J* = 8.4 Hz, 4 H; Ar*H*), 7.27–7.35 (m, 4 H; Ar*H*), 7.10–7.25 (m, 10 H; Ar*H*), 6.83 (s, 4 H; core-Ar*H*), 6.32–6.43 (m, 4 H;

Ar*H*), 6.29 (t, *J* = 2.1 Hz, 2 H; Ar*H*), 3.79–4.02 (m, 12 H; ArOC*H*$_2$), 3.05 (s, 2 H; C≡CH), 2.66–2.86 (m, 12 H; ArC*H*$_2$ + PhC*H*$_2$), 1.95–2.23 (m, 12 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 160.3, 160.2, 153.3, 144.0, 142.7, 141.7, 132.3, 128.68, 128.66, 128.5, 126.0, 119.7, 115.5, 107.3, 107.2, 99.0, 83.8, 76.9, 67.6, 66.9, 66.7, 32.6, 32.3, 32.2, 31.0, 30.8, 30.7; MS (ESI): *m/z* (%): 931 (100) [M + H$^+$]; HRMS (ESI) calcd for $C_{64}H_{66}O_6$ + Na$^+$: 931.4932; found: 931.4947; elemental analysis calcd for $C_{64}H_{66}O_6$: C 82.55, H 7.14; found: C 82.74, H 7.28, N <0.10.

TMSC≡C–[L-G2]–C≡CTMS (142). This compound was synthesized by General Procedure G. Starting from I–[L-G2]–I **139** (1.16 g, 0.52 mmol), $(PPh_3)_2PdCl_2$ (180 mg, 0.26 mmol), PPh$_3$ (69 mg, 0.26 mmol), CuI (50 mg, 0.26 mmol), trimethylsilylacetylene (1.11 mL, 7.86 mmol) and Et$_3$N (1.10 mL, 7.86 mmol) in toluene (20 mL), the product (1.09 g, 97%) was obtained as a pale yellow oil after flash column chromatography (eluent: hexane/EtOAc = 4/1). *R*$_f$ = 0.45 (hexane/EtOAc = 4/1); ^{1}H NMR: δ = 7.40 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 7.17–7.36 (m, 30 H; Ar*H*), 7.15 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 6.82 (m, 4 H; core-ArH), 6.23–6.47 (m, 18 H; Ar*H*), 3.77–4.02 (m, 28 H; ArOC*H*$_2$), 2.62–2.90 (m, 28 H; ArC*H*$_2$ + PhC*H*$_2$), 1.93–2.22 (m, 28 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$), 0.26 (s, 18 H; Si(CH$_3$)$_3$); ^{13}C NMR: δ = 160.2, 160.1, 160.0, 153.1, 143.7, 143.62, 143.59, 142.1, 141.3, 131.9, 128.4, 128.3, 125.8, 120.6, 115.2, 107.0, 105.4, 98.9, 93.5, 67.1, 66.5, 66.4, 32.3, 32.0, 31.9, 30.7, 30.5, 30.4, 0.0; HRMS (MALDI-TOF) calcd for $C_{142}H_{162}O_{14}Si_2$ + Na$^+$: 2171.1427; found: 2171.1419; elemental analysis calcd (%) for $C_{142}H_{162}O_{14}Si_2$: C 79.37, H 7.60; found: C 79.20, H 7.94.

HC≡C–[L-G2]–C≡CH 77. This compound was prepared by General Procedure H. Starting from TMSC≡C–[L-G2]–C≡CTMS **142** (903 mg, 0.42 mmol) and K$_2$CO$_3$ (290 mg, 2.10 mmol) in THF/MeOH (v/v = 1/1, 10 mL), the product (787 mg, 93%) was obtained as a colorless oil after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/2 to CH_2Cl_2). R_f = 0.41 (hexane/CH_2Cl_2 = 1/2); ^{1}H NMR: δ = 7.42 (d, J = 8.1 Hz, 4 H; Ar*H*), 7.27–7.37 (m, 12 H; Ar*H*), 7.18–7.25 (m, 18 H; Ar*H*), 7.16 (d, J = 7.8 Hz, 4 H; Ar*H*), 6.81 (s, 4 H; core-Ar*H*), 6.25–6.42 (m, 18 H; Ar*H*), 3.79–3.99 (m, 28 H; $ArOCH_2$), 3.04 (s, 2 H; C≡CH), 2.66–2.85 (m, 28 H; $ArCH_2$ + $PhCH_2$), 1.94–2.20 (m, 28 H; $ArCH_2CH_2$ + $PhCH_2CH_2$); ^{13}C NMR (one of the C≡C signal was too weak to be observed): δ = 160.2, 160.1, 160.0, 153.1, 143.8, 143.7, 143.6, 142.4, 141.4, 132.1, 128.43, 128.39, 128.3, 125.8, 119.6, 115.3, 107.0, 98.9, 83.7, 67.2, 66.6, 66.4, 32.3, 32.0, 31.9, 30.7, 30.6, 30.4; HRMS (MALDI-TOF) calcd for $C_{136}H_{146}O_{14}$ + Na^+: 2027.0639; found: 2027.0640; elemental analysis calcd (%) for $C_{136}H_{146}O_{14}$: C 81.49, H 7.34; found: C 81.83, H 7.71.

TMSC≡C–[L-G3]–C≡CTMS (143). This compound was prepared by General Procedure G. Starting from I–[L-G3]–I **140** (1.07 g, 0.25 mmol), $(PPh_3)_2PdCl_2$ (86 mg, 0.12 mmol), PPh_3 (32 mg, 0.12 mmol), CuI (23 mg, 0.12 mmol), trimethylsilylacetylene (0.52 mL, 3.69 mmol) and Et_3N (0.51 mL, 3.69 mmol) in toluene (9 mL), the product (709 mg, 67%) was obtained as a pale yellow oil after flash column chromatography (eluent: hexane/EtOAc = 4/1 to 1/2). R_f = 0.68 (hexane/EtOAc = 2/1); ^{1}H NMR: δ = 7.37 (d, J = 8.1 Hz, 4 H; Ar*H*), 7.14–7.33 (m, 70 H; Ar*H*), 7.11 (d, J = 8.1 Hz, 4 H; Ar*H*), 6.79 (m, 4 H; core-Ar*H*), 6.20–6.42 (m, 42 H; Ar*H*), 3.75–4.01 (m, 60 H; $ArOCH_2$), 2.62–2.83 (m, 60 H; $ArCH_2$ + $PhCH_2$), 1.94–2.15 (m, 60 H; $ArCH_2CH_2$ + $PhCH_2CH_2$), 0.23 (s, 18 H; $Si(CH_3)_3$); ^{13}C NMR: δ = 160.2, 160.13, 160.05, 153.1, 143.6, 142.1, 141.4, 131.9, 131.8, 128.4, 128.3, 128.0, 125.8, 120.6, 115.3, 107.0, 105.3, 98.9, 93.5, 66.6, 66.4, 32.4, 32.0,

30.8, 30.6, 30.4, 0.0; HRMS (MALDI-TOF) calcd for $C_{286}H_{322}O_{30}Si_2$ + Na^+: 4318.3195; found: 4318.2975; elemental analysis calcd (%) for $C_{286}H_{322}O_{30}Si_2$: C 79.96, H 7.55; found: C 79.74, H 7.79.

HC≡C–[L-G23]–C≡CH (78). This compound was prepared by General Procedure I. Starting from TMSC≡C–[L-G3]–C≡CTMS **143** (435 mg, 10.1 mmol) and TBAF (1.0 M in THF, 0.3 mL) in THF (10 mL), the product (289 mg, 69%) was obtained as a pale brown oil after flash column chromatography (eluent: hexane/CH_2Cl_2 = 1/1 to CH_2Cl_2 to CH_2Cl_2/Et_2O = 50/1). $R_f = 0.45$ (hexane/CH_2Cl_2 = 1/5); ^1H NMR: $\delta = 7.41$ (d, $J = 8.1$ Hz, 4 H; Ar*H*), 7.23–7.35 (m, 28 H; Ar*H*), 7.07–7.23 (m, 46 H; Ar*H*), 6.80 (s, 4 H; core-Ar*H*), 6.22–6.44 (m, 42 H; Ar*H*), 3.74–4.00 (m, 60 H; ArOC*H*$_2$), 3.03 (s, 2 H; C≡CH), 2.61–2.85 (m, 60 H; ArC*H*$_2$ + PhC*H*$_2$), 1.90–2.17 (m, 60 H; ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$); ^{13}C NMR: δ = 160.4, 160.3, 160.2, 153.3, 144.1, 144.04, 144.00, 143.97, 142.7, 141.7, 132.3, 128.6, 128.5, 126.0, 119.7, 115.5, 107.2, 99.0, 83.8, 76.9, 67.8, 67.0, 66.9, 66.7, 32.7, 32.6, 32.3, 31.0, 30.8, 30.7; HRMS (MALDI-TOF) calcd for $C_{280}H_{306}O_{30}$ + Na^+: 4174.2411; found: 4174.2310; elemental analysis calcd (%) for $C_{280}H_{306}O_{30}$: C 81.01, H 7.43; found: C 81.02, H 7.15.

(d). Preparation of Poly(dendrimer)s Pt-S/L-Gn.

Pt-S-G1 (144). This compound was prepared by General Procedure J. Starting from HC≡C–[S-G1]–C≡CH **73** (100 mg, 0.131 mmol), *trans*-[Pt(PEt$_3$)$_2$Cl$_2$] **65** (65.8 mg, 0.131 mmol) and CuI (12.5 mg, 0.066 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 15 mL), the product (148 mg, 95%; 107 mg, 69% after precipitation) was obtained as a yellow solid. ^1H NMR: $\delta = 7.15$–7.55 (m, 18 H; Ar*H*), 6.89 (s, 4 H; core-Ar*H*), 6.67 (s, 4 H; Ar*H*), 6.55 (s, 2 H;

Ar*H*), 5.03 (s, 4 H; ArCH_2O), 4.97 (s, 4 H; ArCH_2O), 4.95 (s, 4 H; ArCH_2O), 1.97–2.31 (m, 12 H; PCH_2), 1.03–1.37 (m, 18 H; PCH_2CH_3); ^{13}C NMR: δ = 160.23, 160.16, 153.1, 139.8, 136.8, 133.3, 131.1, 128.6, 128.1, 127.6, 127.5, 115.8, 109.3 (*C*≡CPt), 108.4 (t, J_{C-P} = 14.3 Hz, C≡*C*Pt), 106.4, 106.3, 101.5, 70.6, 70.1, 16.4 (quintet like, J_{C-P} = 17.5 Hz), 8.4; ^{31}P NMR (major peak): δ = 11.1 (J_{Pt-P} = 2370 Hz); (minor peaks): δ = 14.9 (J_{Pt-P} = 2390 Hz), 8.6 (J_{Pt-P} = 2320 Hz).

Pt-S-G2 (145). This compound was prepared by General Procedure J. Starting from HC≡C–[S-G2]–C≡CH **74** (211 mg, 0.131 mmol), *trans*-[$Pt(PEt_3)_2Cl_2$] **65** (65.8 mg, 0.131 mmol), CuI (12.5 mg, 0.066 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 15 mL), the product (236 mg, 88%; 218 mg, 81% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.17–7.51 (m, 38 H; Ar*H*), 6.87 (s, 4 H; core-Ar*H*), 6.67 (s, 12 H; Ar*H*); 6.49–6.60 (m, 6 H; Ar*H*), 5.02 (s, 12 H; ArCH_2O), 4.96 (s, 12 H; ArCH_2O), 4.93 (s, 4 H; ArCH_2O), 1.97–2.31 (m, 12 H; PCH_2), 1.08–1.32 (m, 18 H; PCH_2CH_3); ^{13}C NMR: δ = 160.2, 160.1, 153.1, 139.8, 139.3, 139.2, 136.8, 133.3, 131.0, 128.6, 128.0, 127.6, 127.5, 115.8, 109.3 (*C*≡CPt), 108.4 (t, J_{C-P} = 14.3 Hz, C≡*C*Pt), 106.4, 101.6, 70.5, 70.1, 16.4 (quintet like, J_{C-P} = 17.5 Hz), 8.4; ^{31}P NMR: (major peak): δ = 11.2 (J_{Pt-P} = 2370 Hz); (minor peak): δ = 8.7 (J_{Pt-P} = 2320 Hz).

Pt-S-G3 (146). This compound was prepared by General Procedure J. Starting from HC≡C–[S-G3]–C≡CH **75** (155 mg, 0.047 mmol), *trans*-[$Pt(PEt_3)_2Cl_2$] **65** (23.5 mg, 0.047 mmol), CuI (4.5 mg, 0.023 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 5.4 mL), the product (173 mg, 99%; 162 mg, 93% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.15–7.47 (m, 78 H; Ar*H*), 6.84 (s, 4 H; core-Ar*H*), 6.66 (brs, 28 H; Ar*H*), 6.55 (brs, 14 H; Ar*H*), 4.99 (s, 32 H; ArCH_2O), 4.94 (s, 24 H; ArCH_2O), 4.88 (s, 4 H; ArCH_2O), 1.95–2.39 (m, 12 H; PCH_2), 1.05–1.38 (m, 18 H; PCH_2CH_3); ^{13}C NMR: $\delta = 160.2$, 160.1, 153.1, 139.8, 139.31, 139.26, 139.2, 136.8, 133.3, 131.1, 128.6, 128.1, 127.6, 127.5, 115.8, 109.4 (C≡CPt), 108.4 (C≡CPt, weak signal), 106.4, 101.6, 70.5, 70.1, 70.0, 16.4 (quintet like, $J_{C-P} = 17.7$ Hz), 8.4; ^{31}P NMR: (major peak): $\delta = 11.1$ ($J_{Pt-P} = 2370$ Hz); (minor peaks): $\delta = 14.9$ ($J_{Pt-P} = 2380$ Hz), 8.6 ($J_{Pt-P} = 2320$ Hz); HRMS (MALDI-TOF) calcd for cyclic monomer $C_{232}H_{214}O_{30}P_2Pt^+$: 3738.4401; found: 3738.4399.

Pt-L-G1 (147). This compound was prepared by General Procedure J. Starting from HC≡C–[L-G1]–C≡CH **76** (122 mg, 0.131 mmol), *trans*-[Pt(PEt_3)$_2$$Cl_2$] **65** (65.8 mg, 0.131 mmol), CuI (12.5 mg, 0.066 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 15 mL), the product (168 mg, 94%; 88 mg, 49% after precipitation) was obtained as a yellow solid. ^{1}H NMR: $\delta = 7.12$–7.35 (m, 14 H; Ar*H*), 6.98–7.12 (m, 4 H; Ar*H*), 6.90 (s, 4 H, core-Ar*H*), 6.23–6.42 (m, 6 H; Ar*H*), 3.73–4.02 (m, 12 H; $ArOCH_2$); 2.53–2.94 (m, 12 H; $ArCH_2$ + $PhCH_2$), 2.11–2.43 (m, 12 H; PCH_2), 1.88–2.11 (m, 12 H; $ArCH_2CH_2$ + $PhCH_2CH_2$), 1.07–1.37 (m, 18 H; PCH_2CH_3); ^{13}C NMR: $\delta = 160.3$, 160.1, 153.3, 144.2, 144.0, 141.7, 138.4, 131.0, 128.7, 128.5, 128.3, 126.6, 126.0, 115.5, 109.2 (C≡CPt), 107.7, 107.3, 107.2, 106.8 (t, $J_{C-P} = 14.3$ Hz, C≡CPt), 99.0, 67.7, 66.9, 65.9, 32.6, 32.3, 32.1, 31.5, 31.3, 31.0, 30.9, 16.4 (quintet like, $J_{C-P} = 17.5$ Hz), 8.5; ^{31}P NMR: (major peak): $\delta = 11.0$ ($J_{Pt-P} = 2380$ Hz); (minor peaks): $\delta = 14.8$ (t, $J_{Pt-P} = 2390$ Hz), 10.4 ($J_{Pt-P} = 2370$ Hz), 8.5 ($J_{Pt-P} = 2330$ Hz); HRMS (MALDI-TOF) calcd for cyclic monomer $C_{76}H_{94}O_6P_2Pt + H^+$: 1361.6269; found: 1361.6254.

Pt-L-G2 (148). This compound was prepared by General Procedure J. Starting from HC≡C–[L-G2]–C≡CH **77** (263 mg, 0.131 mmol), *trans*-[Pt(PEt_3)$_2$$Cl_2$] **65** (65.8 mg, 0.131 mmol), CuI (12.5 mg, 0.066 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 15 mL), the product

(286 mg, 90%; 227 mg, 71% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.11–7.37 (m, 34 H; Ar*H*), 6.97–7.11 (m, 4 H; Ar*H*), 6.81 (s, 4 H; core-Ar*H*), 6.21–6.43 (m, 18 H; Ar*H*), 3.72–4.05 (m, 28 H; ArOCH$_2$), 2.59–2.93 (m, 28 H; ArCH_2 + PhCH_2), 2.12–2.33 (m, 12 H; PCH$_2$), 1.94–2.12 (m, 28 H; ArCH$_2$CH_2 + PhCH$_2$CH_2), 1.08–1.34 (m, 18 H; PCH$_2$CH_3); ^{13}C NMR: δ = 160.2, 153.2, 144.0, 143.9, 143.8, 141.5, 138.3, 130.9, 128.5, 128.4, 128.2, 126.5, 125.9, 115.4, 109.2 (*C*≡CPt), 107.4, 107.1, 106.8 (t, $J_{\text{C–P}}$ = 16.3 Hz, C≡*C*Pt), 99.4, 98.9, 67.7, 67.5, 66.8, 66.0, 32.7, 32.5, 32.2, 32.0, 31.4, 30.9, 30.7, 16.3 (quintet like, $J_{\text{C–P}}$ = 17.5 Hz), 8.4; ^{31}P NMR: (major peak): δ = 11.1 ($J_{\text{Pt–P}}$ = 2380 Hz); (minor peaks): δ = 15.0 ($J_{\text{Pt–P}}$ = 2390 Hz), 8.7 ($J_{\text{Pt–P}}$ = 2330 Hz); HRMS (MALDI-TOF) calcd for cyclic monomer $C_{148}H_{174}O_{14}P_2Pt^+$: 2433.2053; found: 2433.2043.

Pt-L-G3 (149). This compound was prepared by General Procedure J. Starting from HC≡C–[L-G3]–C≡CH **78** (194 mg, 0.047 mmol), *trans*-[Pt(PEt$_3$)$_2$Cl$_2$] **65** (23.5 mg, 0.047 mmol), CuI (4.5 mg, 0.023 mmol) in CHCl$_3$/*i*-Pr$_2$NH (v/v = 1/1, 5.4 mL), the product (204 mg, 95%; 193 mg, 90% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 6.97–7.36 (m, 78 H; Ar*H*), 6.82 (s, 4 H; core-Ar*H*), 6.17–6.46 (m, 42 H; Ar*H*), 3.73–4.03 (m, 60 H; ArOCH_2), 2.57–2.92 (m, 60 H; ArCH_2 + PhCH_2), 1.88–2.33 (m, 72 H; PCH$_2$ + ArCH$_2$CH_2 + PhCH$_2$CH_2), 1.06–1.40 (m, 18 H; PCH$_2$CH_3); ^{13}C NMR (C≡CPt signal is too weak to be observed): δ = 160.3, 160.2, 153.2, 144.0, 143.94, 143.88, 141.6, 138.4, 130.9, 128.6, 128.5, 128.2, 126.0, 115.4, 109.2 (*C*≡CPt), 107.2, 99.0, 67.6, 66.93, 66.86, 32.5, 32.2, 32.0, 30.9, 30.8, 30.7, 16.4 (quintet like, $J_{\text{C–P}}$ = 17.7 Hz), 14.8,[101] 14.6,[101] 14.3,[101] 8.45, 8.4,[101] 8.1;[101] ^{31}P NMR: (major peak): δ = 11.0 ($J_{\text{Pt–P}}$ = 2380

Hz); (minor peaks): δ = 14.9 (J_{Pt-P} = 2390 Hz), 8.6 (J_{Pt-P} = 2330 Hz); HRMS (MALDI-TOF) calcd for cyclic monomer $C_{292}H_{334}O_{30}P_2Pt + H^+$: 4581.3901; found: 4581.4136.

(e). Preparation of Platinum Linkers.

***cis*-Platinum linker (152).**[93] A solution of dichloro(1,5-cyclooctadiene)platinum(II) **150** (62.5 mg, 0.17 mmol) in CH_2Cl_2 (7.5 mL) was cooled at –10 °C, then a solution of 1,2-bis(diethylphosphino)ethane **151** (39 μL, 0.17 mmol) in CH_2Cl_2 (1.25 mL) was added dropwise over a period of 2 min and the reaction mixture was allowed to stirred at –10 °C for another 10 min. Then the reaction mixture was dried *in vacuo*, CH_2Cl_2 (*c.a.* 35 mL) was added and the solution was filtered. The filtrate was collected and the solvent was evaporated until the solution became cloudy. Then Et_2O was added and some solid was precipitated, filtered and washed by Et_2O to give the target compound (36.6 mg, 46%) as a pale yellow solid. M.p. >295 °C dec.; ^{1}H NMR: δ = 1.90–2.44 (m, 8 H; PCH_2CH_3), 1.66–1.90 (m, 4 H; PCH_2CH_2P), 1.24 (dt, J_{P-H} = 18.6 Hz and J_{H-H} = 7.5 Hz, 12 H; PCH_2CH_3); ^{13}C NMR: δ = 23.5 (dd, J_{C-P} = 38.0 Hz and J_{C-P} = 7.3 Hz; PCH_2CH_2P), 18.9 (dt, J_{C-P} = 38.9 Hz and J_{Pt-C} = 33.6 Hz; PCH_2CH_3), 8.9 (td, J_{Pt-C} = 28.4 Hz and J_{C-P} = 3.2 Hz; PCH_2CH_3); ^{31}P NMR: δ = 57.1 (J_{Pt-P} = 3540 Hz).

4,4'-Bis(trimethylsilylethynyl)biphenyl (154).[76b] A mixture of 4,4'-diiodobiphenyl **153** (1.00 g, 2.46 mmol), $(PPh_3)_2PdCl_2$ (34.5 mg, 0.049 mmol), CuI (46.9 mg, 0.25 mmol), trimethylsilylacetylene (1.0 mL, 7.39 mmol) and Et_3N (1.0 mL, 7.39 mmol) in THF (20 mL) was stirred at 25 °C for 12 h. Hexane (*c.a.* 20 mL) was added into the reaction mixture and then the reaction mixture was filtered. The filtrate was collected, dried *in vacuo* and purified by flash chromatography (eluent: hexane) to give the target compound

(662 mg, 78%) as a white solid. M.p. 168.3–170.9 °C (lit. M.p. 166–167 °C[76b]); $R_f = 0.69$ (hexane); ^{1}H NMR: $\delta = 7.53$ (s, 8 H; Ar*H*), 0.26 (s, 18 H; $Si(CH_3)_3$); ^{13}C NMR: $\delta = 140.3$, 132.6, 126.8, 122.5, 105.0, 95.3, 0.1.

4,4'-Diethynylbiphenyl (155).[76b] A mixture of 4,4'-bis(trimethylsilylethynyl)biphenyl **154** (435 mg, 1.26 mmol) and K_2CO_3 (870 mg, 6.28 mmol) was stirred in THF/MeOH (v/v = 1/1, 20 mL) at 25 °C for 2 h. The reaction mixture was filtered, dried *in vacuo* and purified by flash chromatography (eluent: hexane/EtOAc = 30/1 to 5/1) to give the target compound (254 mg, 100%) as a white solid. M.p. 172.2–173.2 °C (lit. M.p. 169–170 °C[76b]); $R_f = 0.44$ (hexane/EtOAc = 20/1); ^{1}H NMR: $\delta = 7.51$–7.60 (m, 8H; Ar*H*), 3.14 (s, 2 H; C≡CH); ^{13}C NMR: $\delta = 140.6$, 132.8, 127.0, 121.6, 83.5, 78.3.

***long-trans*-Platinum linker (156).**[74] A mixture of 4,4'-diethynylbiphenyl **155** (175 mg, 0.87 mmol), *trans*-$[Pt(PEt_3)_2Cl_2]$ **65** (958 mg, 1.91 mmol), CuCl (21.5 mg, 0.22 mmol), *i*-Pr_2NH (5 mL) in toluene (5 mL) was frozen in a Schlenk tube by liquid N_2 and degassed with N_2 (3 ×). The mixture was allowed to warm to 25 °C and then heated at 100 °C for 5 h. The reaction mixture was then dried *in vacuo* and purified by flash column chromatography (eluent: hexane/EtOAc = 5/1) to give the target compound (364 mg, 37%) as a pale yellow solid. M.p. >220 °C dec. (lit. M.p. >230 °C dec.[74]); $R_f = 0.29$ (hexane/EtOAc = 5/1); ^{1}H NMR: $\delta = 7.44$ (d, $J = 8.1$ Hz, 4 H; biphenyl-Ar*H*), 7.29 (d, $J = 8.1$ Hz, 4 H; biphenyl-Ar*H*), 1.96–2.20 (m, 24 H; PCH_2), 1.13–1.31 (m, 36 H; PCH_2CH_3); ^{13}C NMR: $\delta = 137.4$, 131.1, 127.4, 126.1, 101.3, 83.3 (t, $J_{C-P} = 14.6$ Hz, C≡CPt), 14.4 (quintet like, $J_{C-P} = 17.1$ Hz), 7.9; ^{31}P NMR: $\delta = 14.9$ ($J_{Pt-P} = 2390$ Hz).

(f). Preparation of Poly(dendrimer)s *cis*-Pt-S/L-Gn.

***cis*-Pt-S-G1 (157).**[102] This compound was prepared by General Procedure K. Starting from HC≡C–[S-G1]–C≡CH **73** (100 mg, 0.131 mmol), *cis*-platinum linker **152** (61.9 mg, 0.131 mmol) and CuI (12.5 mg, 0.066 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 15 mL), the product (88.6 mg, 58%; 58.5 mg, 38% after precipitation) was obtained as a yellowish foam. ^{1}H NMR: δ = 7.10–7.57 (m, 18 H; Ar*H*), 6.86 (s, 4 H; core-Ar*H*), 6.65 (brs, 4 H; Ar*H*), 6.53 (brs, 2 H; Ar*H*), 4.65–5.22 (m, 12 H; ArCH_2O), 1.47–2.35 (m, 12 H; PCH_2CH_2P + PCH_2CH_3), 0.95–1.47 (m, 12 H; PCH_2CH_3); ^{13}C NMR (C≡*C*Pt signal could not be observed,[94] probably masked by a large signal at δ = 106.4): δ = 160.2, 153.1, 139.8, 136.9, 133.4, 132.8, 132.4, 131.6, 128.7, 128.1, 127.7, 127. 5, 127.3, 115.3, 111.0 (d, $^3J_{C-P\,(trans)}$ = 34.1 Hz, *C*≡CPt),[94] 106.4, 101.5, 70.6, 70.2, 24.4 (m, P*C*H_2*C*H_2P), 18.6 (dt, J_{C-P} = 35.1 Hz and J_{Pt-C} = 35.8 Hz; P*C*H_2CH_3), 8.8 (triplet like, J_{C-P} = 10.1 Hz); ^{31}P NMR (major peak): δ = 50.8 (J_{Pt-P} = 2210 Hz);[94] (minor peaks): δ = 57.2 (J_{Pt-P} = 2170 Hz), 54.6 (J_{Pt-P} = 3350 Hz),[103] 53.6 (J_{Pt-P} = 2250 Hz), 50.0 (J_{Pt-P} = 2220 Hz).

***cis*-Pt-S-G2 (158).**[102] This compound was prepared by General Procedure K. Starting from HC≡C–[S-G2]–C≡CH **74** (150 mg, 0.093 mmol), *cis*-platinum linker **152** (43.9 mg, 0.093 mmol), CuI (8.9 mg, 0.047 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 10.6 mL), the product (163 mg, 87%; 124 mg, 66% after precipitation) was obtained as a yellowish foam. ^{1}H NMR : δ = 7.17–7.53 (m, 38 H; Ar*H*), 6.78–6.92 (m, 4 H, core-Ar*H*), 6.46–6.77 (m, 18 H; Ar*H*), 4.81–5.10 (m, 28 H; ArCH_2O), 1.65–2.34 (m, 12 H; PCH_2CH_2P + PCH_2CH_3), 1.00–1.40 (m, 12 H; PCH_2CH_3); ^{13}C NMR (C≡*C*Pt signal could not be observed, [94] probably masked by a large signal at δ = 106.7): δ =160.1, 160.0, 153.1, 139.8, 139.3, 139.2, 136.8, 133.3, 132.7, 132.3, 131.5, 128.6, 128.0, 127.6, 127.2, 115.8, 110.8 (d, $^{3}J_{\mathrm{C-P\,(trans)}} = 33.7$ Hz, *C*≡CPt),[94] 106.7, 106.3, 101.5, 70.6, 70.1, 69.9, 69.5, 53.5, 24.2 (m, $\mathrm{P}C\mathrm{H_2}C\mathrm{H_2P}$), 18.5 (dt, $J_{\mathrm{C-P}} = 34.9$ Hz and $J_{\mathrm{Pt-C}} = 35.5$ Hz; $\mathrm{PCH_2CH_3}$), 8.7 (triplet like, $J_{\mathrm{C-P}} = 10.5$ Hz); ^{31}P NMR (major peak): δ = 50.8 ($J_{\mathrm{Pt-P}} = 2210$ Hz);[94] (minor peaks): δ = 56.7 ($J_{\mathrm{Pt-P}} = 2300$ Hz), 54.9 ($J_{\mathrm{Pt-P}} = 3360$ Hz),[103] 53.7 ($J_{\mathrm{Pt-P}} = 2250$ Hz), 50.4 ($J_{\mathrm{Pt-P}} = 2220$ Hz).

***cis*-Pt-S-G3 (159).**[102] This compound was prepared by General Procedure K. Starting from HC≡C–[S-G3]–C≡CH **75** (119 mg, 0.036 mmol), *cis*-platinum linker **152** (17.0 mg, 0.036 mmol), CuI (3.4 mg, 0.018 mmol) in $\mathrm{CHCl_3}$/*i*-$\mathrm{Pr_2NH}$ (v/v = 1/1, 4.1 mL), the product (124 mg, 93%; 86.6 mg, 65% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.15–7.51 (m, 78 H; Ar*H*), 6.84 (s, 4 H; core-Ar*H*), 6.67 (brs, 28 H; Ar*H*), 6.55 (brs, 14 H; Ar*H*), 4.69–5.09 (m, 60 H; $\mathrm{ArC}H_2\mathrm{O}$), 1.50–2.37 (m, 12 H; $\mathrm{PC}H_2\mathrm{C}H_2\mathrm{P}$ + $\mathrm{PC}H_2\mathrm{CH_3}$), 1.00–1.43 (m, 12 H; $\mathrm{PCH_2C}H_3$); ^{13}C NMR (*C*≡CPt signal was too weak to be observed[94] and C≡*C*Pt signal could not be observed,[94] probably masked by a large signal at δ = 106.4): δ = 160.2, 160.1, 153.1, 139.8, 139.3, 136.8, 133.3, 132.4, 131.5, 128.6, 128.1, 127.6, 127.3, 127.1, 115.8, 106.4, 101.6, 70.5, 70.1, 70.0, 24.3 (m, $\mathrm{P}C\mathrm{H_2}C\mathrm{H_2P}$), 18.6 (dt, $J_{\mathrm{C-P}} = 35.1$ Hz and $J_{\mathrm{Pt-C}} = 35.4$ Hz; $\mathrm{PCH_2CH_3}$), 8.8 (triplet like, $J_{\mathrm{C-P}} = 9.9$ Hz); ^{31}P NMR (major peak): δ = 50.8 ($J_{\mathrm{Pt-P}} = 2210$ Hz);[94] (minor peaks): δ = 56.6 ($J_{\mathrm{Pt-P}} = 2300$ Hz), 54.6 ($J_{\mathrm{Pt-P}} = 3350$ Hz),[103] 53.6 ($J_{\mathrm{Pt-P}} = 2250$ Hz), 50.4 ($J_{\mathrm{Pt-P}} = 2210$ Hz).

***cis*-Pt-L-G1 (160).**[102] This compound was prepared by General Procedure K. Starting from HC≡C–[L-G1]–C≡CH **76** (129 mg, 0.139 mmol), *cis*-platinum linker **152** (65.6 mg, 0.139 mmol), CuI (13.2 mg, 0.069 mmol) in $\mathrm{CHCl_3}$/*i*-$\mathrm{Pr_2NH}$ (v/v = 1/1, 15.9 mL), the product (150 mg, 81%; 110 mg, 59% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.11–7.47 (m, 14 H; Ar*H*), 7.03 (d, $J = 7.8$ Hz, 4 H; Ar*H*), 6.82 (s, 4 H;

core-Ar*H*), 6.21–6.42 (m, 6 H; Ar*H*), 3.74–4.03 (m, 12 H; ArOCH_2), 2.61–2.89 (m, 12 H; ArCH_2 + PhCH_2), 1.64–2.35 (m, 24 H; PCH_2CH_2P + PCH_2CH$_3$ + ArCH$_2$CH_2 + PhCH$_2$CH_2), 1.02–1.43 (m, 12 H; PCH$_2$CH_3); ^{13}C NMR (C≡*C*Pt signal could not be observed,[94] probably masked by a large signal at δ = 107.1): δ = 160.2, 153.2, 143.9, 141.6, 138.8, 138.3, 131.3, 128.5, 128.4, 127.9, 126.3, 125.9, 115.4, 110.8 (d, $^3J_{\text{C–P (trans)}}$ = 34.7 Hz, *C*≡CPt),[94] 107.1, 98.9, 67.5, 66.9, 66.8, 32.5, 32.2, 32.0, 30.9, 30.8, 29.7, 24.3 (m, P*C*H$_2$*C*H$_2$P), 18.6 (dt, $J_{\text{C–P}}$ = 34.9 Hz and $J_{\text{Pt–C}}$ = 32.5 Hz; P*C*H$_2$CH$_3$), 8.7 (triplet like, $J_{\text{C–P}}$ = 10.3 Hz); ^{31}P NMR (major peak): δ = 50.9 ($J_{\text{Pt–P}}$ = 2210 Hz);[94] (minor peaks): δ = 54.9 ($J_{\text{Pt–P}}$ = 3360 Hz),[103] 53.7 ($J_{\text{Pt–P}}$ = 2250 Hz), 50.5 ($J_{\text{Pt–P}}$ = 2220 Hz).

***cis*-Pt-L-G2 (161).**[102] This compound was prepared by General Procedure K. Starting from HC≡C–[L-G2]–C≡CH **77** (131 mg, 0.066 mmol), *cis*-platinum linker **152** (31.0 mg, 0.066 mmol), CuI (6.2 mg, 0.033 mmol) in CHCl$_3$/*i*-Pr$_2$NH (v/v = 1/1, 7.5 mL), the product (135 mg, 86%; 54 mg, 34% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.08–7.45 (m, 34 H; Ar*H*), 7.02 (d, J = 8.1 Hz, 4 H; Ar*H*), 6.80 (s, 4 H; core-Ar*H*), 6.20–6.40 (m, 18 H; Ar*H*), 3.77–3.99 (m, 28 H; ArOCH_2), 2.60–2.88 (m, 28 H; ArCH_2 + PhCH_2), 1.65–2.32 (m, 40 H; PCH_2CH_2P + PCH_2CH$_3$ + ArCH$_2$CH_2 + PhCH$_2$CH_2), 1.05–1.38 (m, 12 H; PCH$_2$CH_3); ^{13}C NMR (C≡*C*Pt signal could not be observed,[94] probably masked by a large signal at δ = 107.2): δ = 160.3, 153.3, 144.0, 143.9, 141.7, 138.4, 131.5, 128.6, 128.5, 127.9, 126.0, 115.5, 111.0 (d, $^3J_{\text{C–P (trans)}}$ = 35.3 Hz, *C*≡CPt),[94] 107.2, 99.0, 67.7, 66.9, 32.6, 32.3, 32.1, 31.0, 30.8, 24.4 (m, P*C*H$_2$*C*H$_2$P), 18.7 (dt, $J_{\text{C–P}}$ = 34.7 Hz and $J_{\text{Pt–C}}$ = 32.2 Hz; P*C*H$_2$CH$_3$), 8.8 (triplet like, $J_{\text{C–P}}$ = 10.3 Hz); ^{31}P NMR (major peak): δ = 50.8 ($J_{\text{Pt–P}}$ = 2210 Hz);[94] (minor peaks): δ = 54.6 ($J_{\text{Pt–P}}$ = 3360 Hz),[103] 53.6 ($J_{\text{Pt–P}}$ = 2250 Hz), 50.6 ($J_{\text{Pt–P}}$ = 2210 Hz).

***cis*-Pt-L-G3 (162).**[102] This compound was prepared by General Procedure K. Starting from HC≡C–[L-G3]–C≡CH **78** (154 mg, 0.037 mmol), *cis*-platinum linker **152** (17.5 mg, 0.037 mmol), CuI (3.5 mg, 0.019 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 4.2 mL), the product (165 mg, 98%; 71.3 mg, 42% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.08–7.36 (m, 74 H; Ar*H*), 7.02 (d, *J* = 7.8 Hz, 4 H; Ar*H*), 6.80 (s, 4 H; core-Ar*H*), 6.21–6.44 (m, 42 H; Ar*H*), 3.76–4.00 (m, 60 H; ArOC*H*$_2$), 2.55–2.87 (m, 60 H; ArC*H*$_2$ + PhC*H*$_2$), 1.58–2.34 (m, 72 H; PC*H*$_2$C*H*$_2$P + PC*H*$_2$CH$_3$ + ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$), 1.05–1.35 (m, 12 H; PCH$_2$C*H*$_3$); ^{13}C NMR (*C*≡CPt signal was too weak to be observed[94] and C≡*C*Pt signal could not be observed,[94] probably masked by a large signal at δ = 107.2): δ = 160.4, 160.3, 153.3, 144.03, 143.96, 141.7, 131.5, 128.6, 128.5, 127.9, 126.0, 115.5, 107.2, 99.0, 67.8, 67.0, 66.9, 32.6, 32.3, 31.0, 30.8, PCH$_2$CH$_2$P signal was too weak to be observed, 18.7 (d, J_{C-P} = 34.9 Hz), 8.8 (triplet like, J_{C-P} = 9.4 Hz); ^{31}P NMR (major peak): δ = 50.8 (J_{Pt-P} = 2210 Hz);[94] (minor peaks): δ = 54.5 (J_{Pt-P} = 3360 Hz),[103] 53.6 (J_{Pt-P} = 2250 Hz), 50.7 (J_{Pt-P} = 2210 Hz).

(g). Preparation of Poly(dendrimer)s *long-trans*-Pt-S/L-Gn.

***long-trans*-Pt-S-G1 (163).** This compound was prepared by General Procedure L. Starting from HC≡C–[S-G1]–C≡CH **73** (100 mg, 0.131 mmol), *long-trans*-platinum linker **156** (148 mg, 0.131 mmol) and CuI (12.5 mg, 0.066 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 15 mL), the product (220 mg, 92%; 182 mg, 76% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.16–7.58 (m, 26 H; Ar*H* + biphenyl-Ar*H*), 6.88 (s, 4 H; core-Ar*H*), 6.67 (s, 4 H; Ar*H*), 6.54 (s, 2 H; Ar*H*), 4.86–5.12 (m, 12 H; ArC*H*$_2$O), 2.00–2.32 (m, 24 H; PCH$_2$), 1.10–1.36 (m, 36 H; PCH$_2$C*H*$_3$); ^{13}C NMR: δ = 160.3, 160.2, 153.2, 139.8, 137.6, 136.9, 133.4, 131.3, 131.1, 128.7, 128.1, 127.7, 127.5, 126.4, 115.9, 109.4 (*C*≡CPt), 108.4 (C≡*C*Pt), 106.4, 101.6, 70.7, 70.2, 16.5 (quintet like, $J_{C\text{–}P}$ = 17.5 Hz), 8.5; ^{31}P NMR (major peak): δ = 11.1 ($J_{Pt\text{–}P}$ = 2370 Hz); (minor peaks): δ = 14.9 ($J_{Pt\text{-}P}$ = 2390 Hz).

***long-trans*-Pt-S-G2 (164).** This compound was prepared by General Procedure L. Starting from HC≡C–[S-G2]–C≡CH **74** (60.0 mg, 0.037 mmol), *long-trans*-platinum linker **156** (42.1 mg, 0.037 mmol), CuI (3.5 mg, 0.019 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 4.2 mL), the product (86.8 mg, 87%; 78.1 mg, 78% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.20–7.51 (m, 46 H; Ar*H* + biphenyl-Ar*H*), 6.88 (s, 4 H; core-Ar*H*), 6.67 (s, 12 H; Ar*H*), 6.50–6.60 (m, 6 H; Ar*H*), 4.88–5.07 (m, 28 H; ArCH_2O), 2.01–2.32 (m, 24 H; PCH_2), 1.09–1.36 (m, 36 H; PCH_2CH_3); ^{13}C NMR: δ = 160.2, 160.1, 153.1, 139.8, 139.3, 137.6, 136.8, 133.3, 131.3, 131.1, 128.6, 128.1, 127.6, 126.6, 126.3, 115.8, 109.3 (*C*≡CPt), 108.4 (C≡*C*Pt), 106.4, 101.6, 70.5, 70.1, 16.4 (quintet like, $J_{C\text{–}P}$ = 17.7 Hz), 8.4; ^{31}P NMR (major peak): δ = 11.2 ($J_{Pt\text{–}P}$ = 2370 Hz); (minor peaks): δ = 15.0 ($J_{Pt\text{–}P}$ = 2390 Hz), 8.7 ($J_{Pt\text{–}P}$ = 2320 Hz).

***long-trans*-Pt-S-G3 (165).** This compound was prepared by General Procedure L. Starting from HC≡C–[S-G3]–C≡CH **75** (119 mg, 0.036 mmol), *long-trans*-platinum linker **156** (40.8 mg, 0.036 mmol), CuI (3.4 mg, 0.018 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 4.1 mL), the product (115 mg, 73%; 78.7 mg, 50% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.16–7.50 (m, 86 H; Ar*H* + biphenyl-Ar*H*), 6.83 (s, 4 H; core-Ar*H*), 6.66 (brs, 28 H; Ar*H*), 6.55 (brs, 14 H; Ar*H*), 4.79–5.06 (m, 60 H; ArCH_2O), 1.99–2.34 (m, 24 H; PCH_2), 1.08–1.35 (m, 36 H; PCH_2CH_3); ^{13}C NMR (*C*≡CPt signal was too weak to be observed) : δ = 160.2, 160.1, 153.1, 139.9, 139.3, 137.6, 136.8, 133.3, 131.3, 131.1, 128.7, 128.1, 127.7, 126.4, 115.8, 108.0 (C≡*C*Pt), 106.4, 101.7, 70.6, 70.2, 70.1, 16.4 (quintet like, J_{C-P} = 17.7 Hz), 8.5; ^{31}P NMR (major peak): δ = 11.1 (J_{Pt-P} = 2370 Hz); (minor peaks): δ = 14.9 (J_{Pt-P} = 2390 Hz), 8.6 (J_{Pt-P} = 2320 Hz).

***long-trans*-Pt-L-G1 (166).** This compound was prepared by General Procedure L. Starting from HC≡C–[L-G1]–C≡CH **76** (111 mg, 0.119 mmol), *long-trans*-platinum linker **156** (134 mg, 0.119 mmol), CuI (11.3 mg, 0.059 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 13.6 mL), the product (229 mg, 97%; 174 mg, 74% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.44 (d, *J* = 7.5 Hz, 4 H; biphenyl-Ar*H*), 7.12–7.38 (m, 18 H; Ar*H* + biphenyl-Ar*H*), 7.05 (d, *J* = 8.1 Hz, 4 H; Ar*H*), 6.82 (s, 4 H; core-Ar*H*), 6.35 (s, 4 H; Ar*H*), 6.21–6.31 (m, 2 H; Ar*H*), 3.77–3.97 (m, 12 H; ArOCH_2), 2.57–2.85 (m, 12 H; ArCH_2 + PhCH_2), 2.11–2.32 (m, 24 H; PCH_2), 1.91–2.11 (m, 12 H; $ArCH_2CH_2$ + $PhCH_2CH_2$), 1.07–1.34 (m, 36 H; PCH_2CH_3); ^{13}C NMR: δ = 160.31, 160.28, 153.3, 144.0, 141.7, 138.4, 137.6, 131.3, 131.0, 128.6, 128.5, 128.3, 126.6, 126.4, 126.0, 115.5, 109.2 (*C*≡CPt), 107.3, 106.8 (C≡*C*Pt), 99.0, 67.6, 66.9, 32.6, 32.3, 32.1, 31.0, 30.9, 16.4 (quintet like, J_{C-P} = 17.5 Hz), 8.5; ^{31}P NMR (major peak): δ = 11.1 (J_{Pt-P} = 2370 Hz).

***long-trans*-Pt-L-G2 (167).** This compound was prepared by General Procedure L. Starting from HC≡C–[L-G2]–C≡CH **77** (131 mg, 0.066 mmol), *long-trans*-platinum linker **156** (74.2 mg, 0.066 mmol), CuI (6.2 mg, 0.033 mmol) in $CHCl_3$/*i*-Pr_2NH (v/v = 1/1, 7.5 mL), the product (175 mg, 87%; 174 mg, 86% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.45 (d, *J* = 8.1 Hz, 4 H; biphenyl-Ar*H*), 7.12–7.39 (m, 38 H; Ar*H* + biphenyl-Ar*H*), 7.06 (d, *J* = 7.5 Hz, 4 H; Ar*H*), 6.82 (s, 4 H; core-Ar*H*), 6.24–6.45 (m, 18 H; Ar*H*), 3.82–4.02 (m, 28 H; ArOCH_2), 2.62–2.89 (m, 28 H; ArCH_2 + PhCH_2), 1.92–2.34 (m, 52 H; PCH_2 + $ArCH_2CH_2$ + $PhCH_2CH_2$), 1.09–1.44 (m, 36 H;

PCH$_2$C*H*$_3$); ^{13}C NMR: δ = 160.3, 153.2, 144.0, 143.9, 141.6, 138.3, 137.5, 131.3, 130.9, 128.6, 128.5, 128.2, 126.6, 126.3, 126.0, 115.5, 109.2 (*C*≡CPt), 107.2, 106.8 (C≡CPt), 99.0, 67.6, 66.9, 32.5, 32.2, 32.0, 30.9, 30.7, 16.4 (quintet like, J_{C-P} = 17.6 Hz), 8.5; ^{31}P NMR (major peak): δ = 11.1 (J_{Pt-P} = 2370 Hz).

***long-trans*-Pt-L-G3 (168).** This compound was prepared by General Procedure L. Starting from HC≡C–[L-G3]–C≡CH **78** (154 mg, 0.037 mmol), *long-trans*-platinum linker **156** (41.9 mg, 0.037 mmol), CuI (3.5 mg, 0.019 mmol) in CHCl$_3$/*i*-Pr$_2$NH (v/v = 1/1, 4.2 mL), the product (191 mg, 99%; 160 mg, 83% after precipitation) was obtained as a yellow solid. ^{1}H NMR: δ = 7.12–7.53 (m, 82 H; Ar*H* + biphenyl-Ar*H*), 7.07 (d, *J* = 7.2 Hz, 4 H; Ar*H*), 6.82 (s, 4 H; core-Ar*H*), 6.25–6.47 (m, 42 H; Ar*H*), 3.79–4.04 (m, 60 H; ArOC*H*$_2$), 2.61–2.95 (m, 60 H; ArC*H*$_2$ + PhC*H*$_2$), 1.95–2.34 (m, 84 H; PCH$_2$ + ArCH$_2$C*H*$_2$ + PhCH$_2$C*H*$_2$), 1.08–1.41 (m, 36 H; PCH$_2$C*H*$_3$); ^{13}C NMR (C≡*C*Pt signal was too weak to be observed): δ = 160.31, 160.26, 153.2, 144.0, 143.94, 143.88, 141.6, 138.3, 137.5, 131.3, 130.9, 128.6, 128.5, 128.2, 126.3, 126.0, 115.4, 109.2 (*C*≡CPt), 107.2, 99.0, 67.6, 66.92, 66.85, 32.5, 32.2, 32.0, 30.9, 30.8, 30.7, 16.4 (quintet like, J_{C-P} = 17.7 Hz), 8.5; ^{31}P NMR (major peak): δ = 11.1 (J_{Pt-P} = 2370 Hz); (minor peaks): δ = 14.9 (J_{Pt-P} = 2390 Hz), 8.7 (J_{Pt-P} = 2330 Hz).

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Chapter 6:

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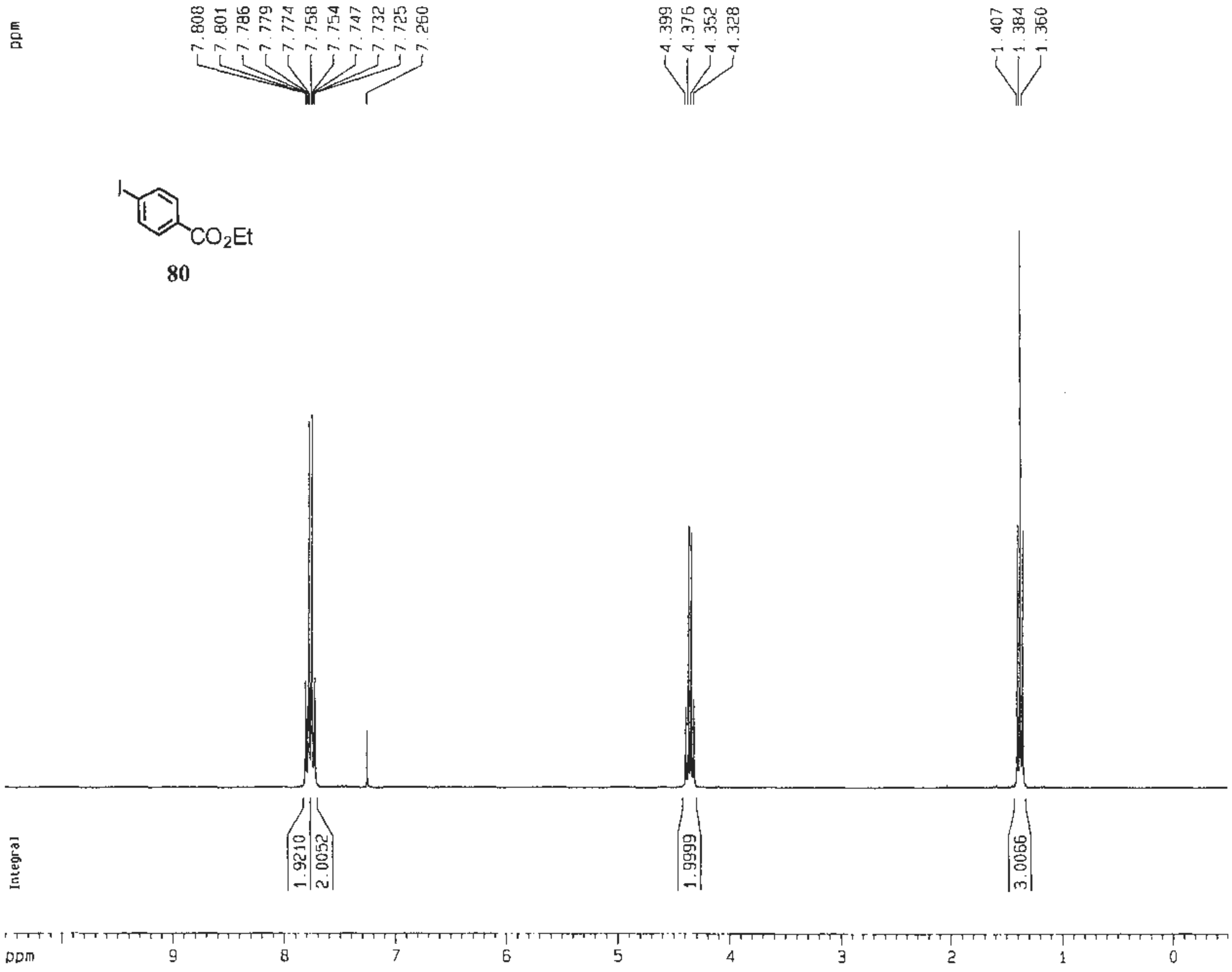
101. Minor signal peaks due to oligomeric species.

102. For all the *cis*-Pt-S/L-Gn compounds (n = 1, 2 and 3), they were the mixture of oligomers and cyclic oligomers even after precipitation, therefore the NMR data

were just taken for the recording purpose.

103. For ^{31}P NMR sepctroscopy, the signal at $\delta \approx 55$ ppm ($^{1}J_{\mathrm{Pt-P}} \approx 3360$ Hz) was probably due to the Cl–Pt–P end-group.

NMR Spectra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SOLVENT             CDCl3
NS                      8
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
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RG                  228.1
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MCWRK          0 01500000 sec

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WDW                    EM
SSB                     0
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GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22 00 cm
CY                  10.00 cm
F1P                10 500 ppm
F1                3151 36 Hz
F2P                -0.500 ppm
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PPMCM             0.50000 ppm/cm
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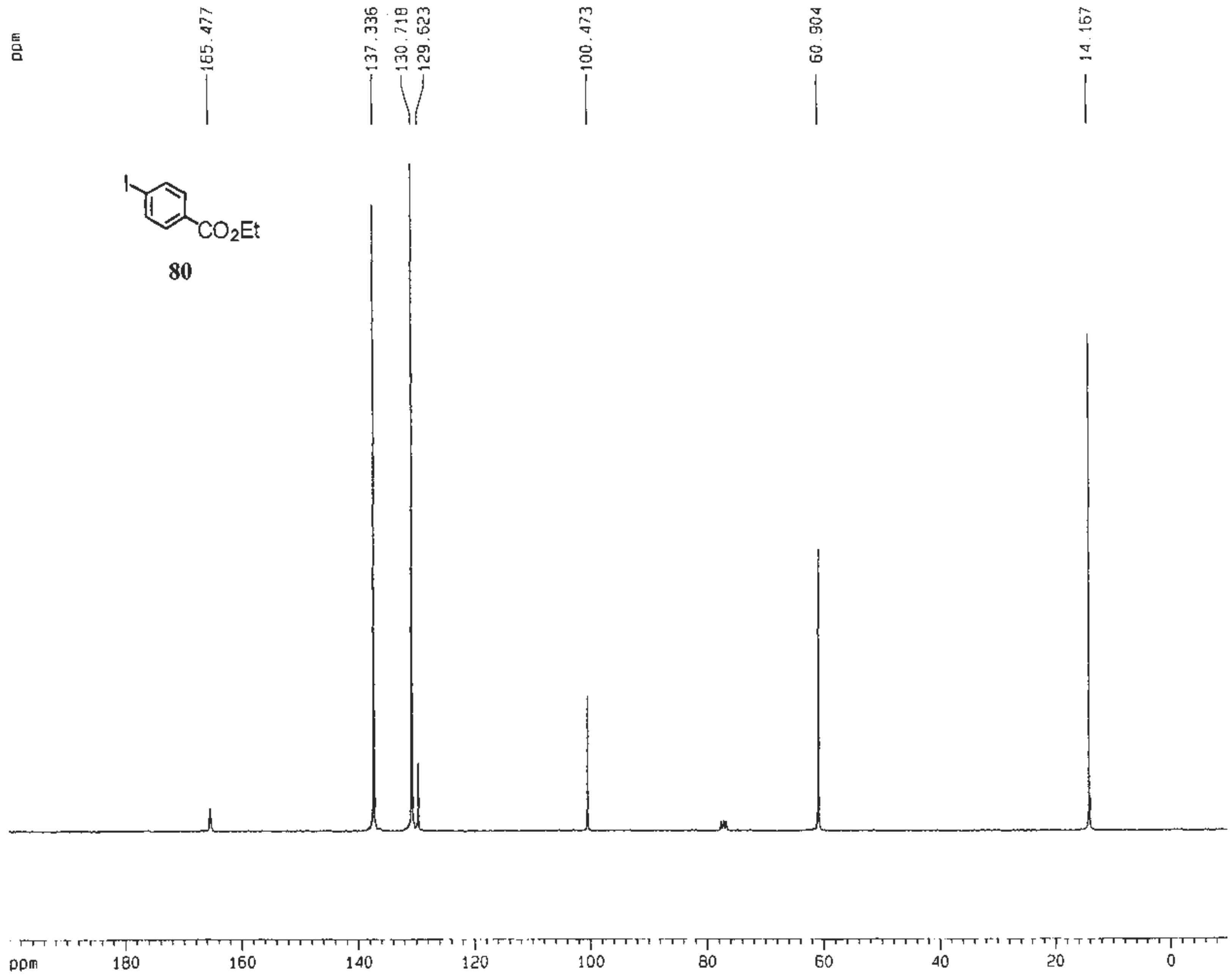
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NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19 00 dB
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1D NMR plot parameters
CX                22 00 cm
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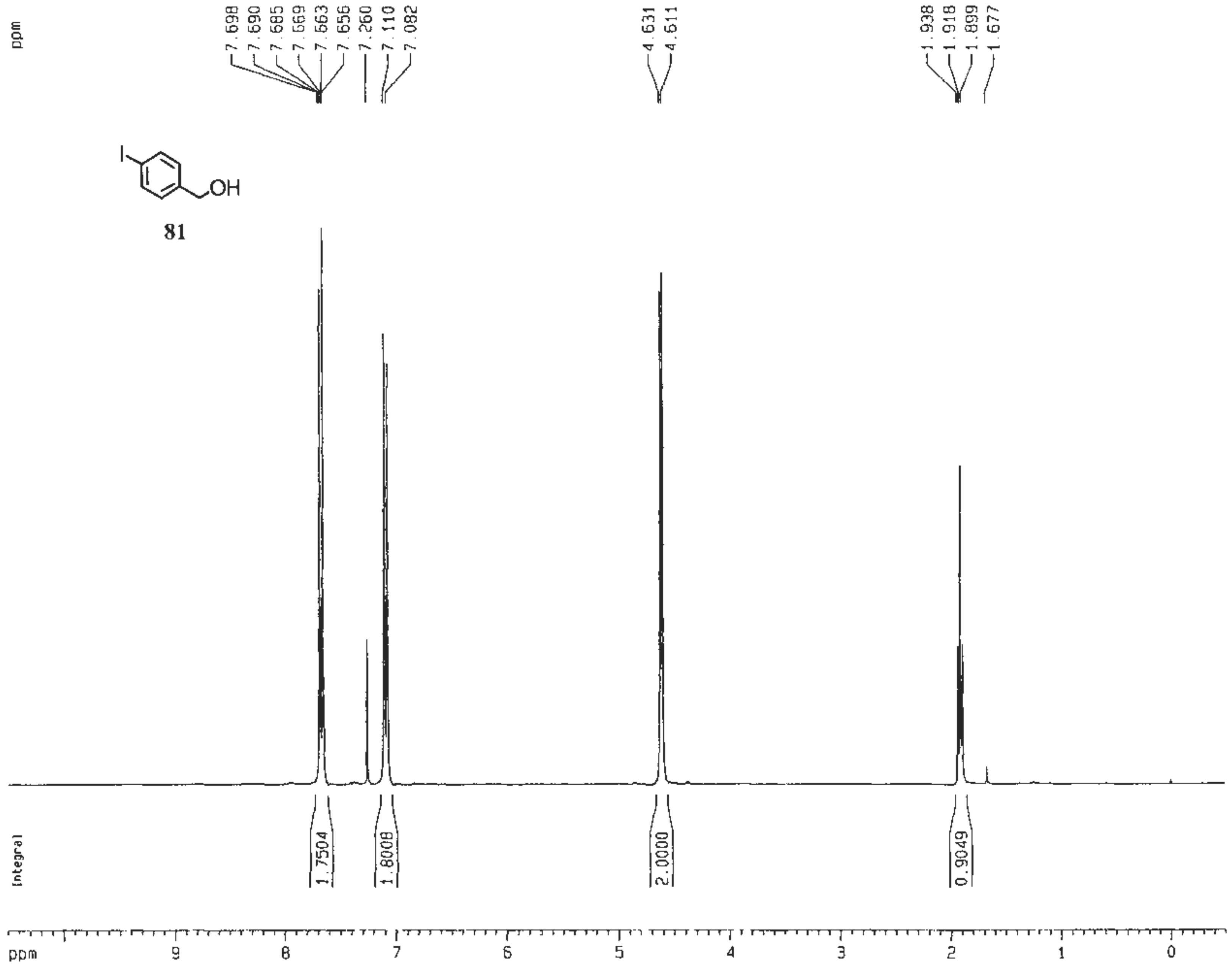

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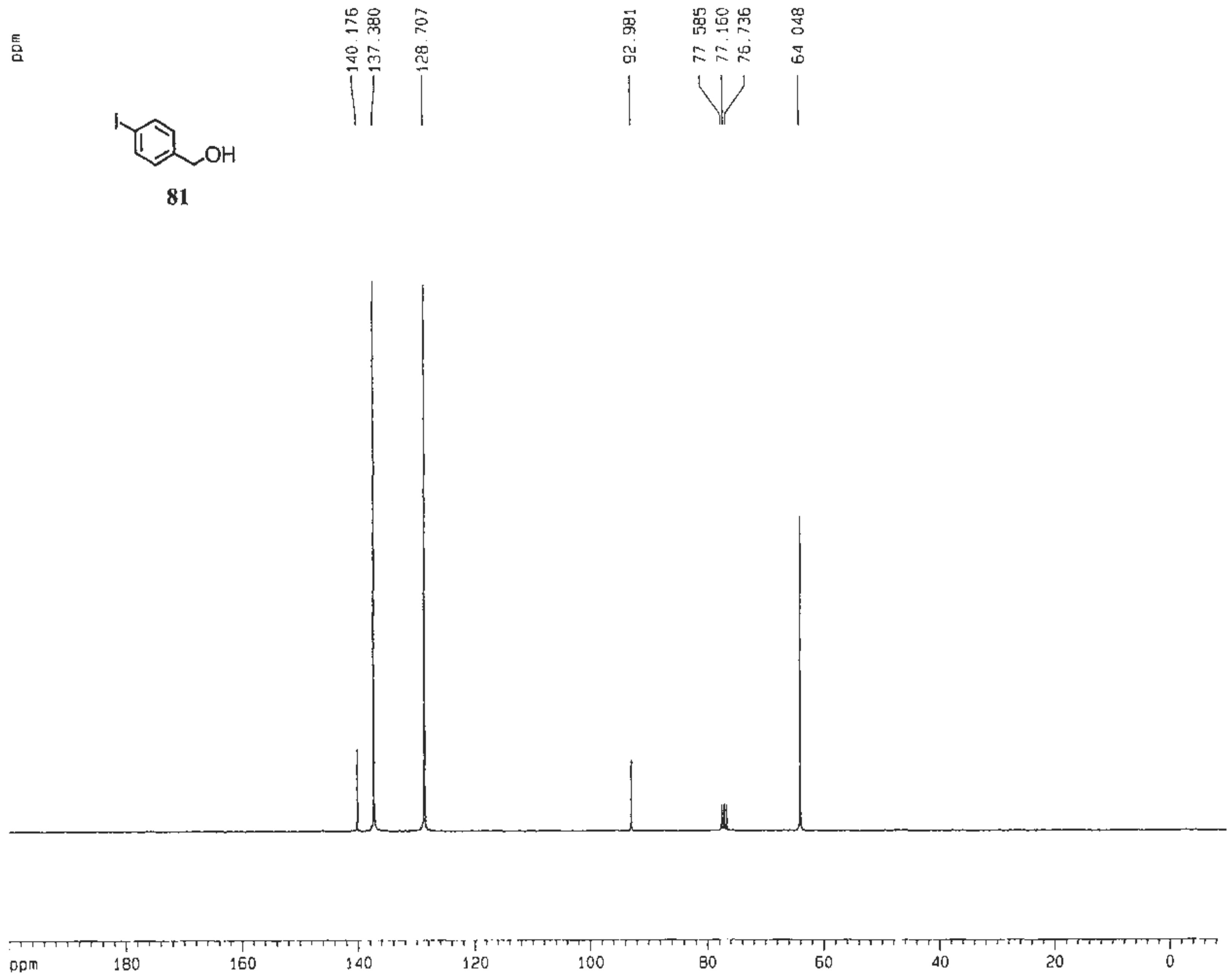

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DS                     0
SWH            22675 736 Hz
FIDRES          0.346004 Hz
AQ             1 4451188 sec
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DE                  6.00 usec
TE                   0 0 K
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MCWRK         0 01500000 sec

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SFO1         75.4745111 MHz

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PL12               19.00 dB
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GB                     0
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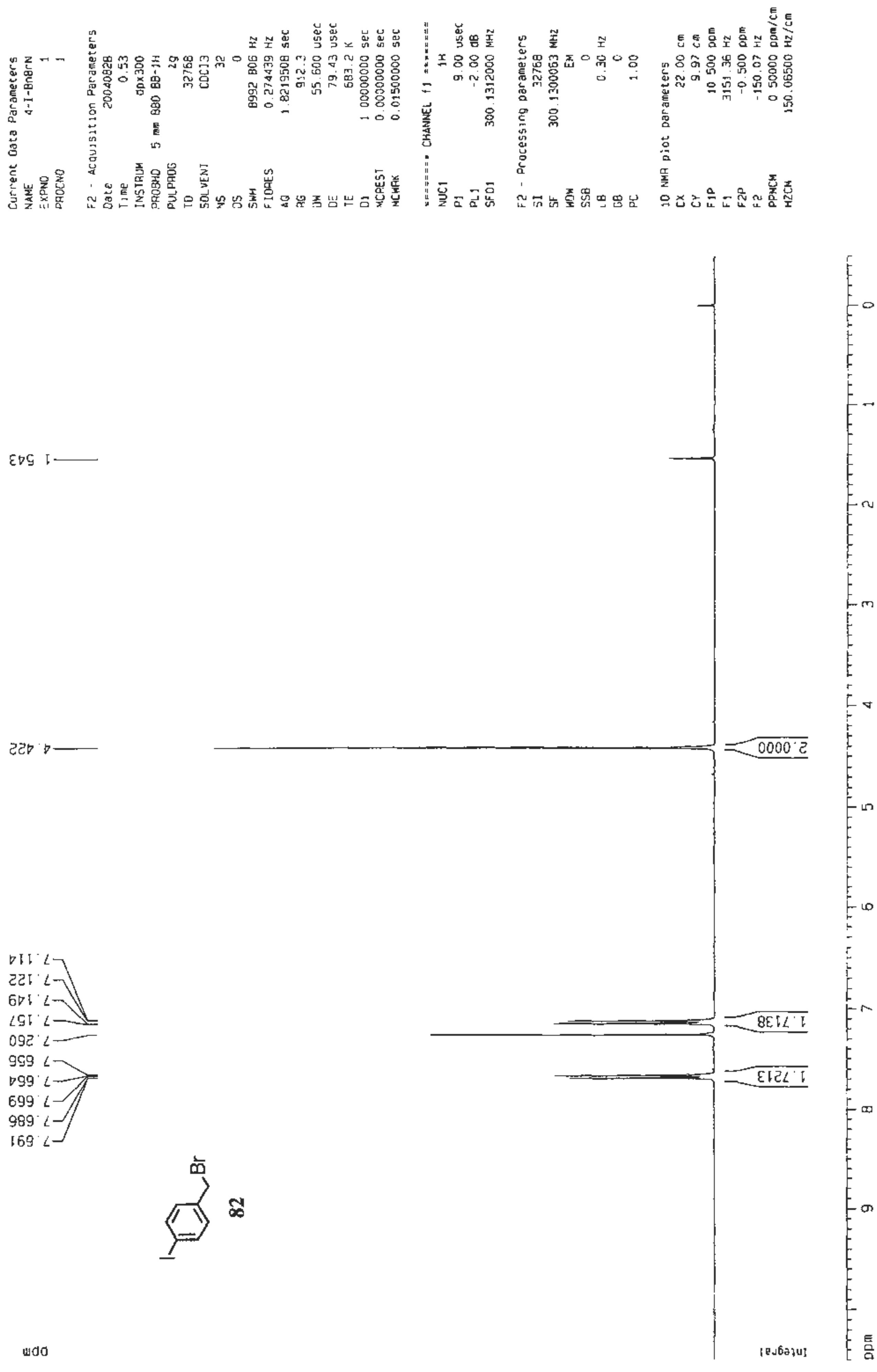

82

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DS                    0
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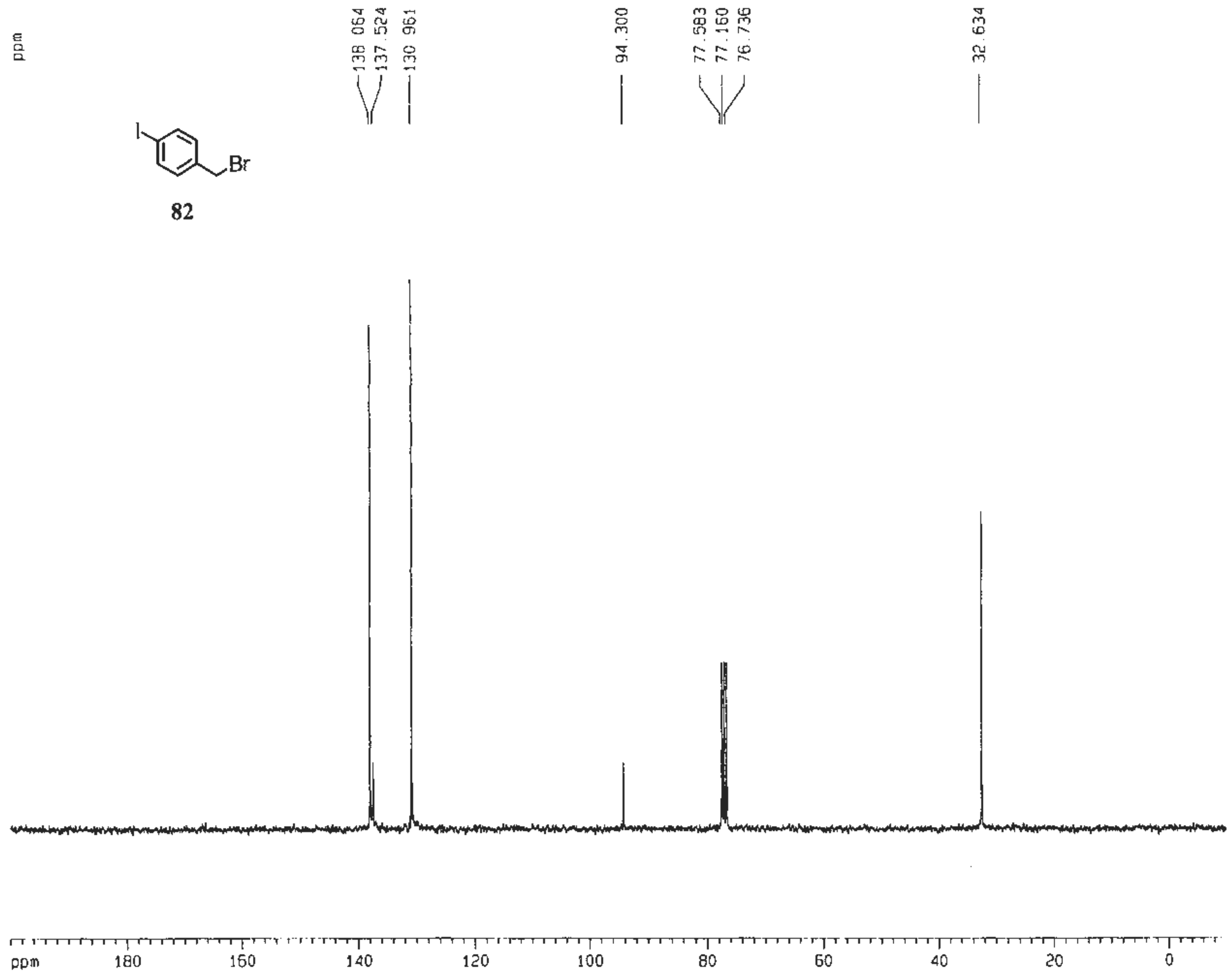
AQ            1.8219508 sec
RG                912.3
DW               55.600 usec
DE                79.43 usec
TE                683.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1312000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300063 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                 9.97 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



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```
Current Data Parameters
NAME          4-I-BnBrNC
EXPNO                  1
PROCNO                 1

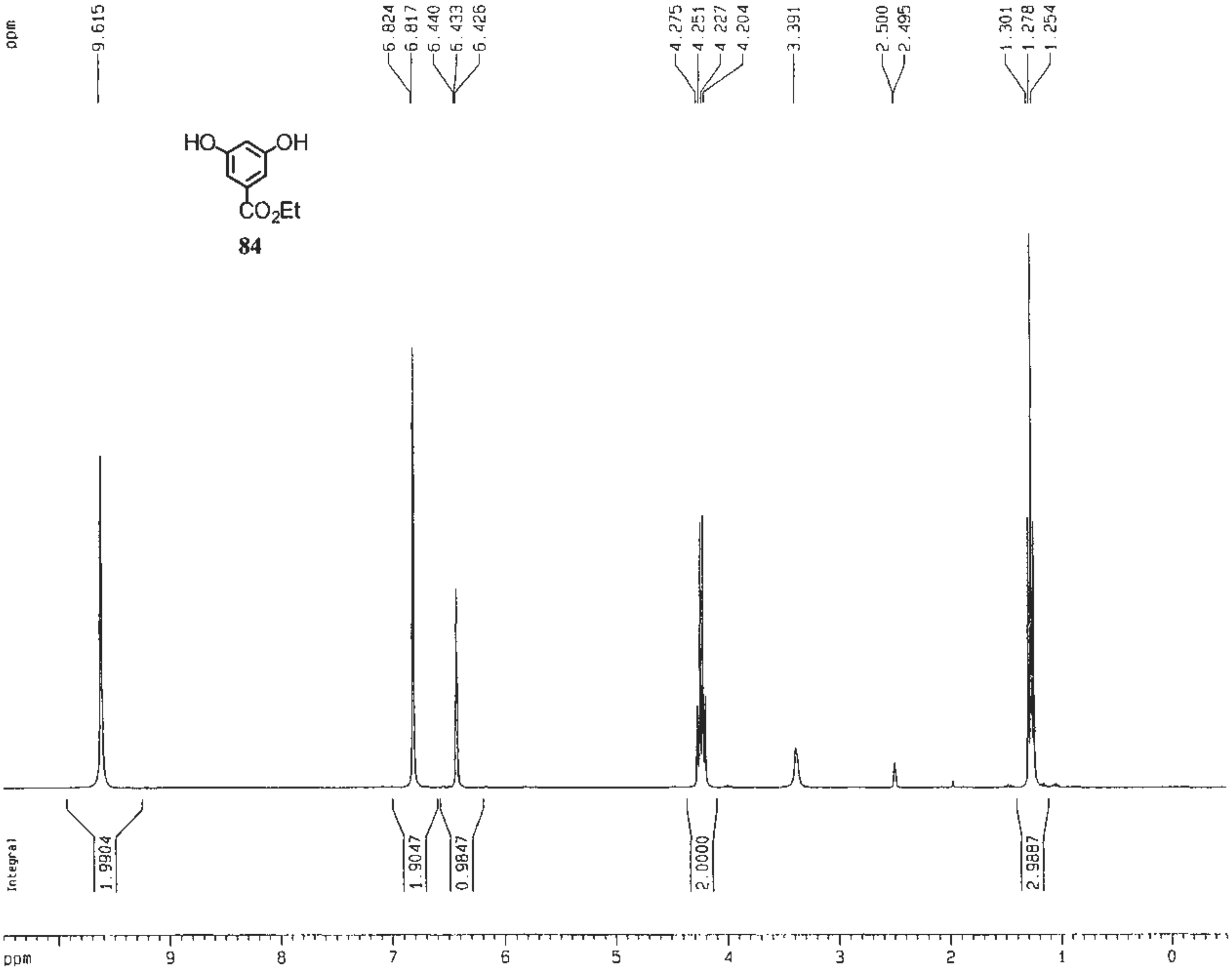
F2 - Acquisition Parameters
Date_           20051029
Time               13.47
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1 4451188 sec
RG                  8192
DW                22.050 usec
DE                  6 00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0 03000000 sec
MCREST        0 00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100 00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677411 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1 40

1D NMR plot parameters
CX                 22.00 cm
CY                  9.97 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754 68 Hz
PPMCM            9 54545 ppm/cm
HZCM         720 37384 Hz/cm
```



84

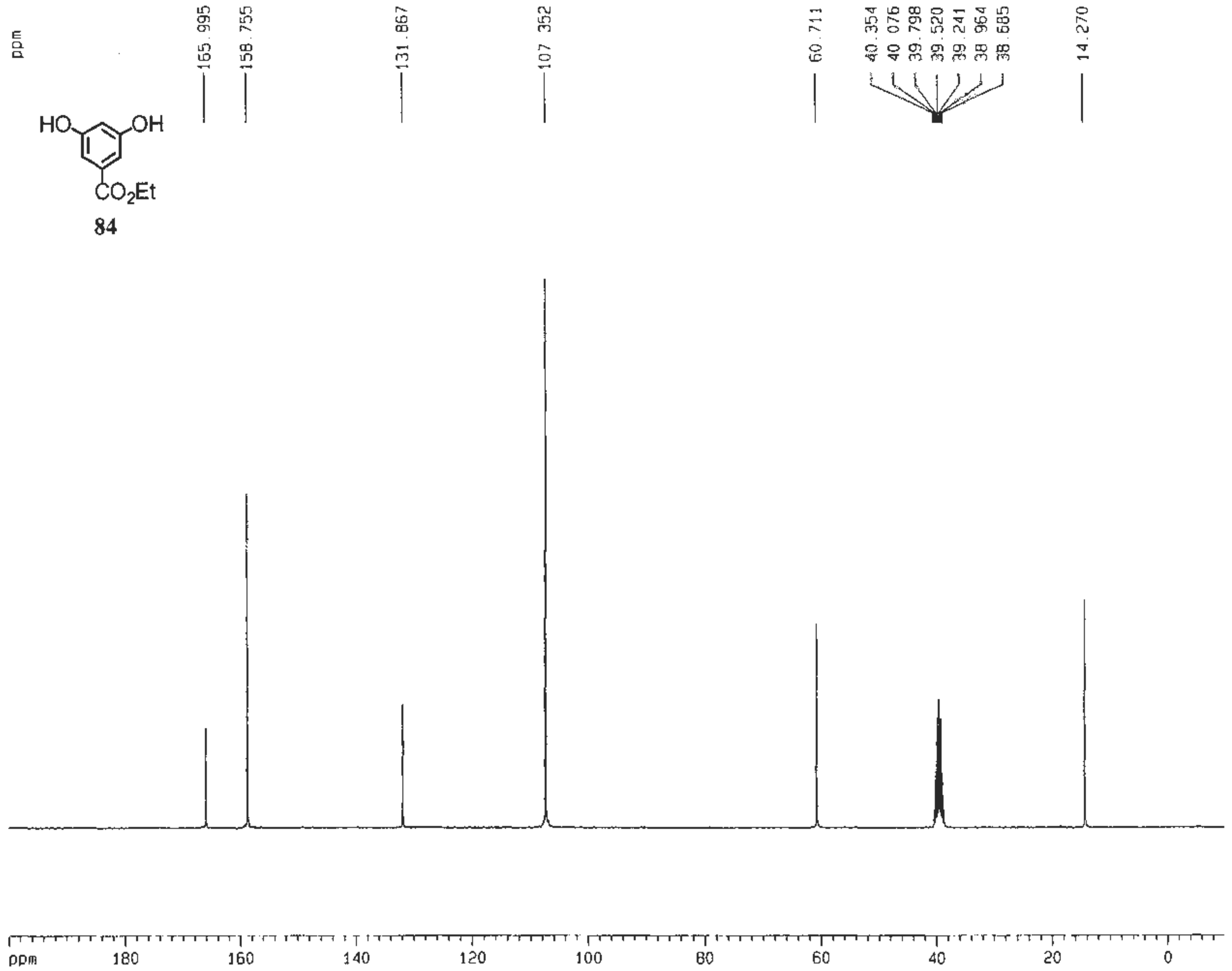
```
Current Data Parameters
NAME        Di-OH-ester
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20030623
Time              19.07
INSTRUM          dpx300
PROBHD    5 mm Dual 13
PULPROG              zg
TD                32768
SOLVENT           CDC13
NS                    4
DS                    0
SWH          8992.806 Hz
FIDRES       0.274439 Hz
AQ         1.8219508 sec
RG                  128
DW           55.600 usec
DE             6.00 usec
TE              300.0 K
D1        1.00000000 sec

============ CHANNEL f1 =======
NUC1                 1H
P1             4.50 usec
PL1            -2.00 dB
SFO1     300.1312000 MHz

F2 - Processing parameters
SI                32768
SF       300.1300015 MHz
WDW                  EM
SSB                   0
LB              0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX             22.00 cm
CY              9.99 cm
F1P          10.500 ppm
F1           3151.36 Hz
F2P          -0.500 ppm
F2           -150.07 Hz
PPMCM      0.50000 ppm/cm
HZCM     150.06500 Hz/cm
```



84

```
Current Data Parameters
NAME        01-OH-esterC
EXPNO                  1
PROCNO                 1

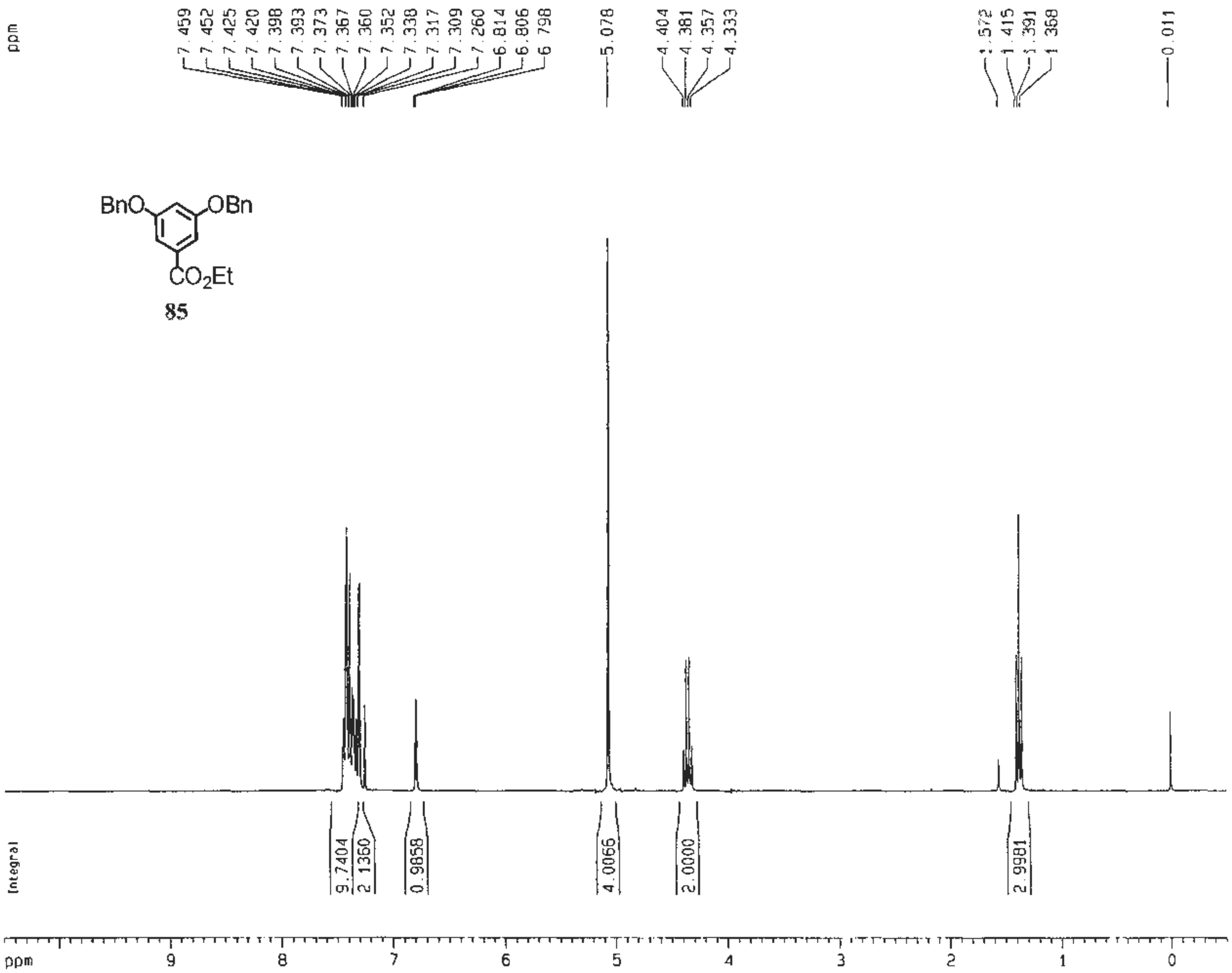
F2 - Acquisition Parameters
Date_           20050201
Time               19 11
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     0
SWH           22675 736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22 050 usec
DE                  6.00 usec
TE                   0 0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0 00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3 00 usec
PL1                -6 00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100 00 usec
PL2               120 00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677654 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                  9.97 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37408 Hz/cm
```



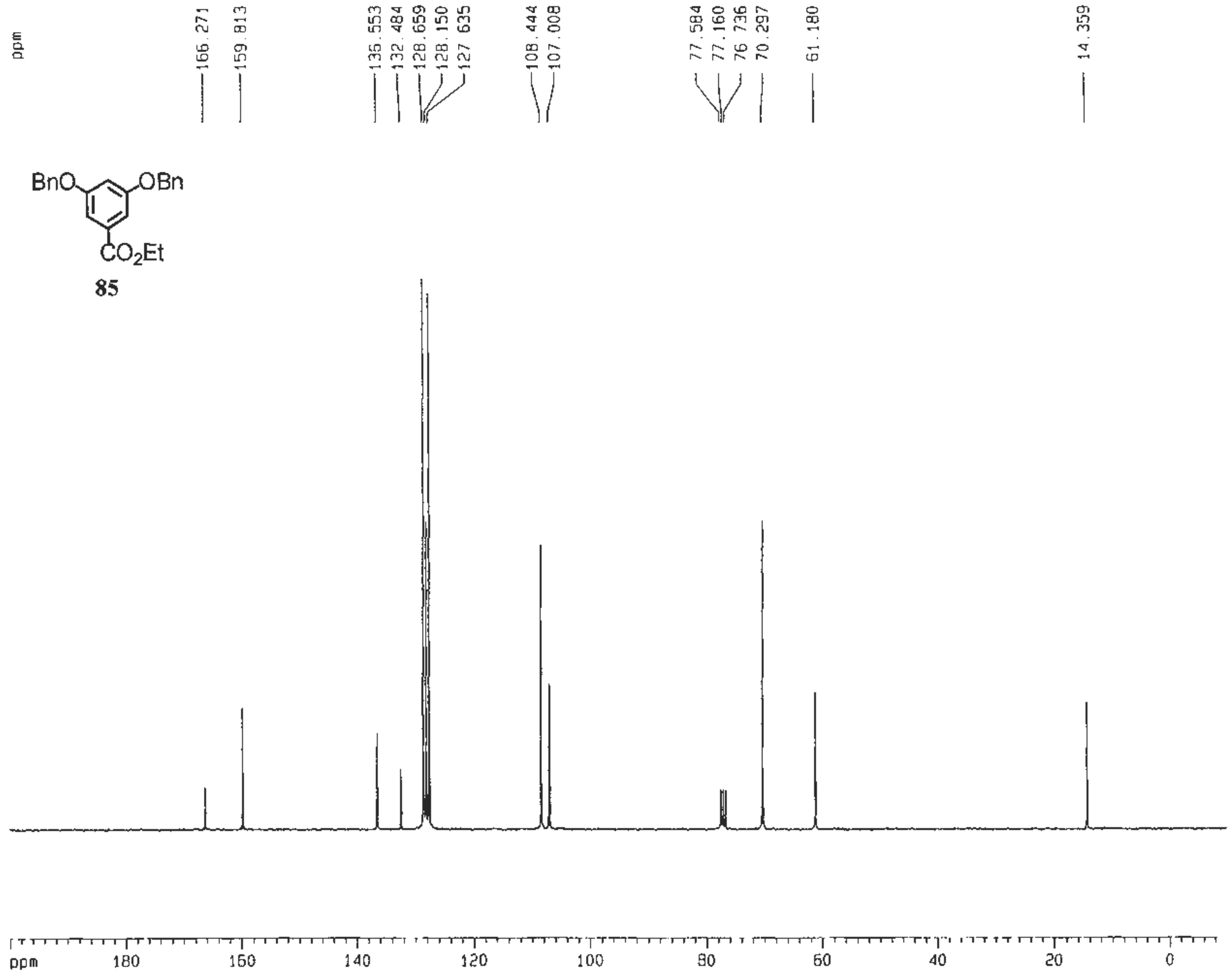
```
Current Data Parameters
NAME             G1 ester
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20030821
Time                17.38
INSTRUM            dpx300
PROBHD       5 mm Dual 13
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                      4
DS                      0
SWH              8992.806 Hz
FIDRES           0.274439 Hz
AQ              1 8219508 sec
RG                    181
DW                 55 600 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec

============ CHANNEL f1 ======
NUC1                   1H
P1                   4.50 usec
PL1                 -2.00 dB
SFO1          300.1312000 MHz

F2 - Processing parameters
SI                  32768
SF            300 1300060 MHz
WDW                    EM
SSB                     0
LB                   0 30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  10 00 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0 500 ppm
F2                -150.07 Hz
PPMCM             0 50000 ppm/cm
HZCM            150.06500 Hz/cm
```



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```
Current Data Parameters
NAME            G1-esterC
EXPNO                   1
PROCNO                  1

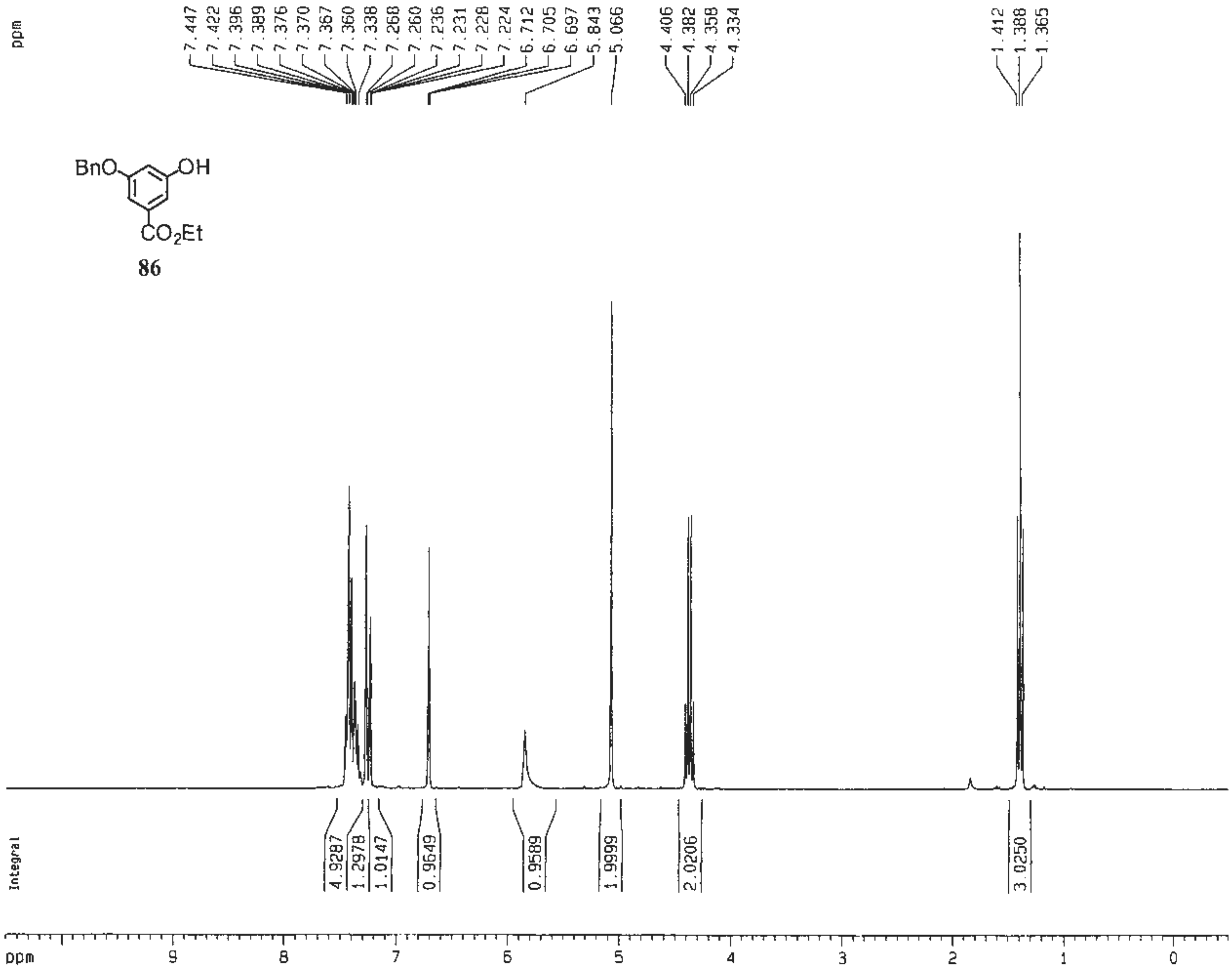
F2 - Acquisition Parameters
Date_            20050201
Time                19 35
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                    275
DS                      0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451168 sec
RG                   4096
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1           1 00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2         300 1315007 MHz

F2 - Processing parameters
SI                  65536
SF           75.4673927_MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                   9.96 cm
F1P               200.000 ppm
F1               15093.48 Hz
F2P               -10.000 ppm
F2                -754.67 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37048 Hz/cm
```



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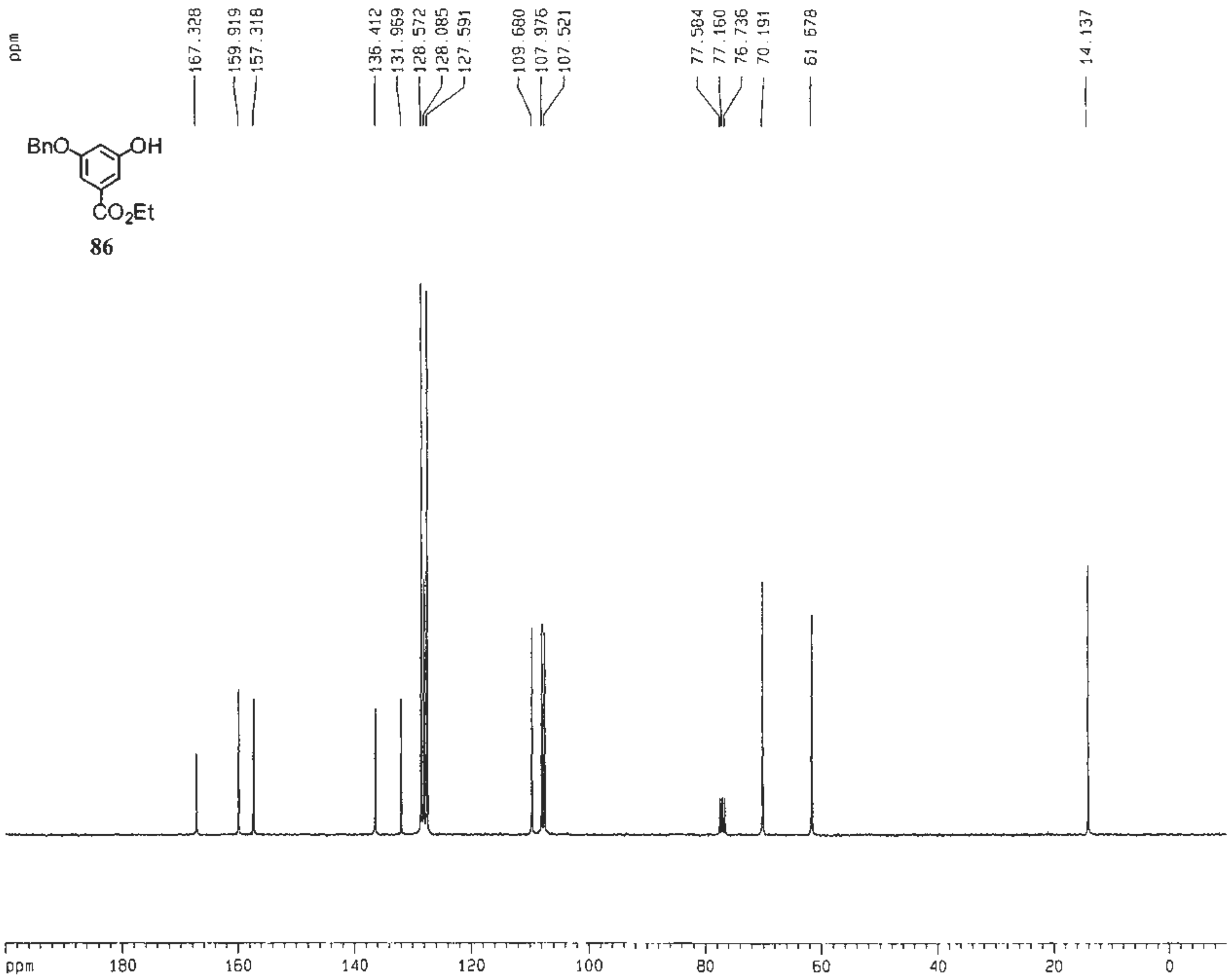
```
Current Data Parameters
NAME          GO-M-ester
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080429
Time               18.14
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    24
DS                     0
SWH             3601.657 Hz
FIDRES          0.219828 Hz
AQ             2.2745588 sec
RG                   362
DW               138.825 usec
DE                198.32 usec
TE                 295.2 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  5.00 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300036 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 10.00 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```



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```
Current Data Parameters
NAME          GO-M-esterC
EXPNO                   1
PROCNO                  1

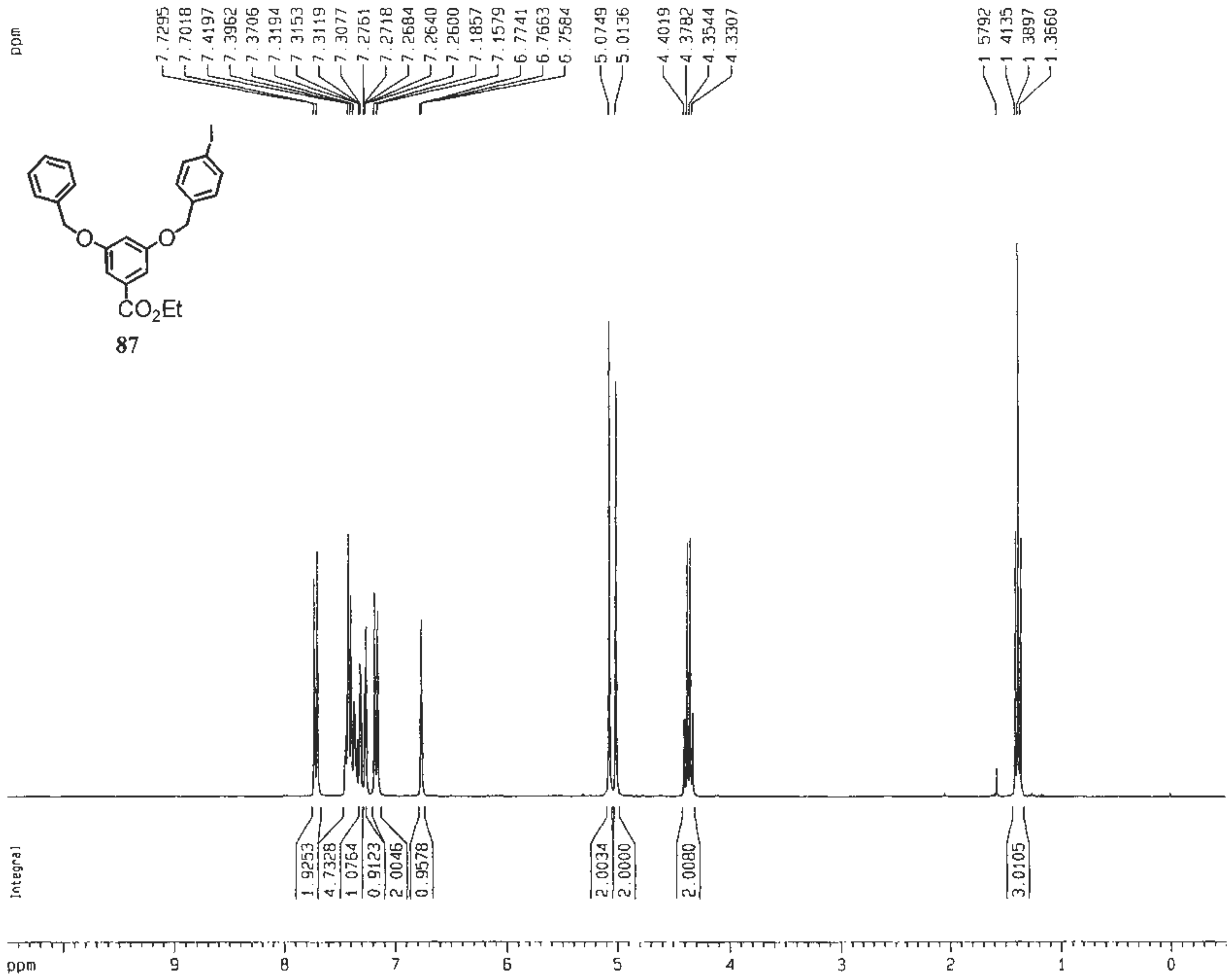
F2 - Acquisition Parameters
Date_            20060201
Time                19.33
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                    181
DS                      0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                    0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677555 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  10.01 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37402 Hz/cm
```



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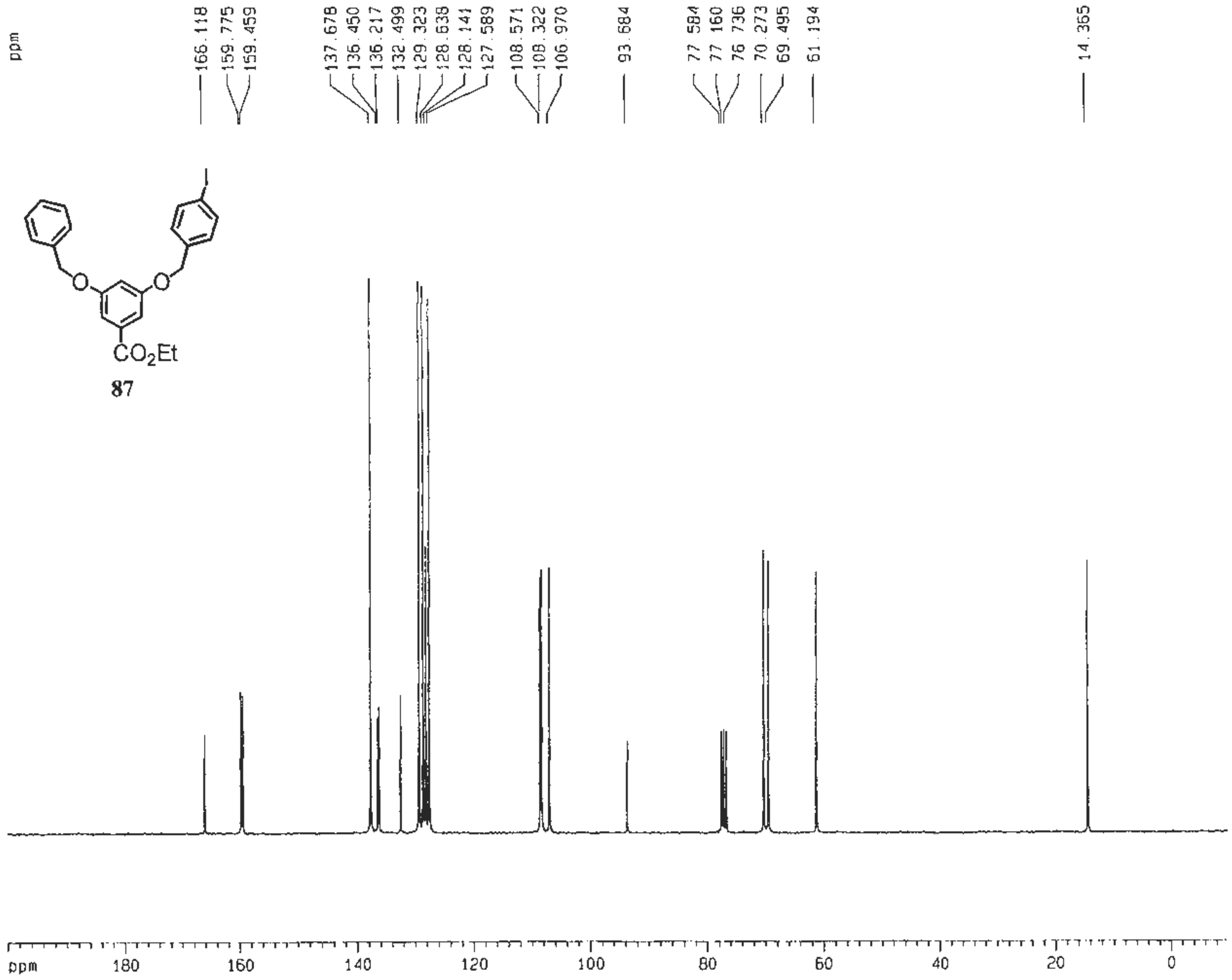
```
Current Data Parameters
NAME         I-G1-esterN
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20041104
Time                9.36
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             3601.657 Hz
FIDRES          0.219828 Hz
AQ             2.2745588 sec
RG                 143.7
DW               138.825 usec
DE                198.32 usec
TE                   0.0 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300061 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  9.96 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```



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```
Current Data Parameters
NAME        I-61-esterNC
EXPNO                  1
PROCNO                 1

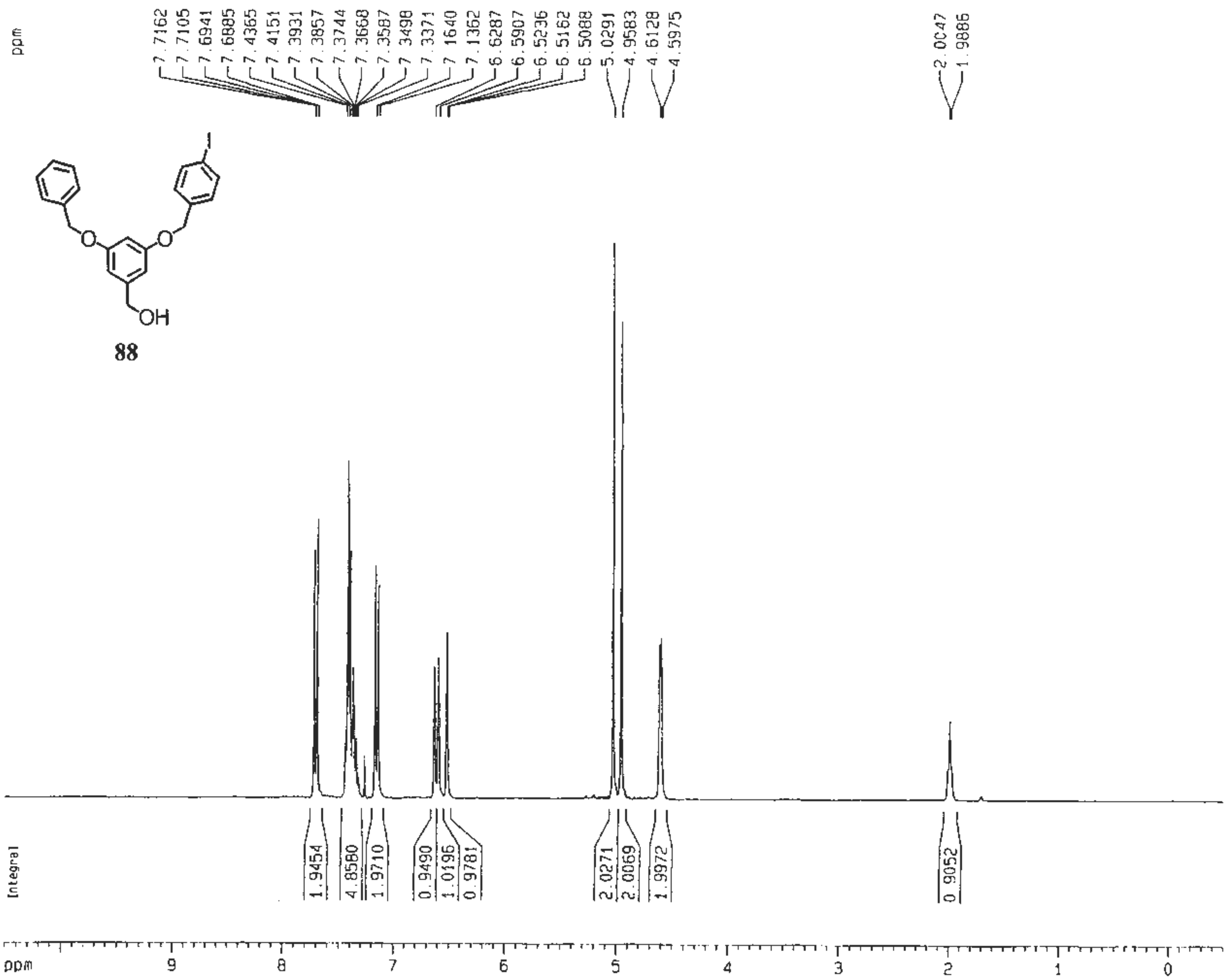
F2 - Acquisition Parameters
Date_           20041104
Time               16.52
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   800
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677535 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 10.00 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```



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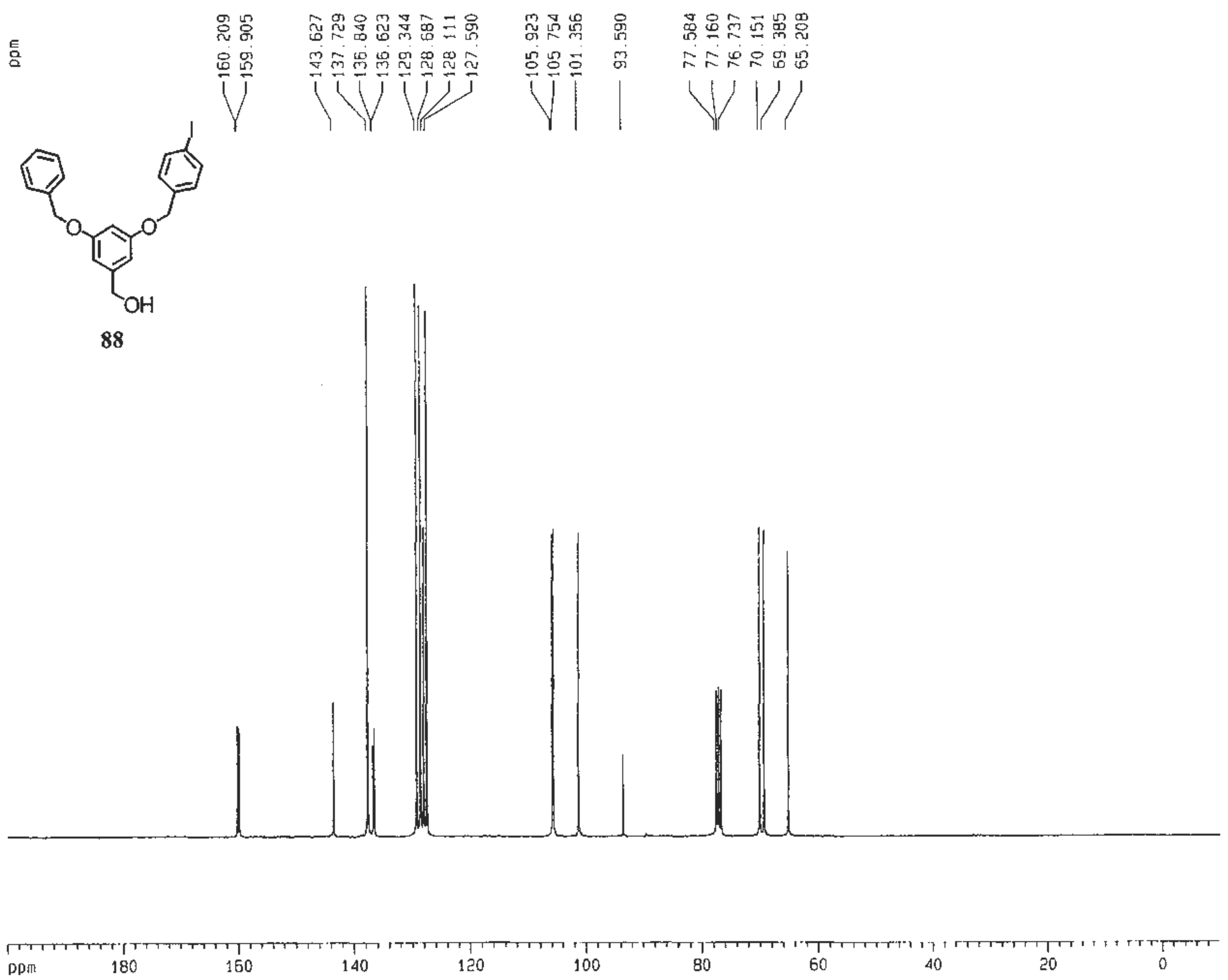
```
Current Data Parameters
NAME          I-G1-OHN
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20050104
Time             19 58
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                   8
DS                   0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AO           2 2745588 sec
RG                  57
DW             138.825 usec
DE              198 32 usec
TE                 0.0 K
D1          5 00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9 00 usec
PL1              -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300 1300059 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1 00

1D NMR plot parameters
CX               22.00 cm
CY               10.02 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150 07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



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```
Current Data Parameters
NAME           1-G1-OHNC
EXPNO                  1
PROCNO                 1

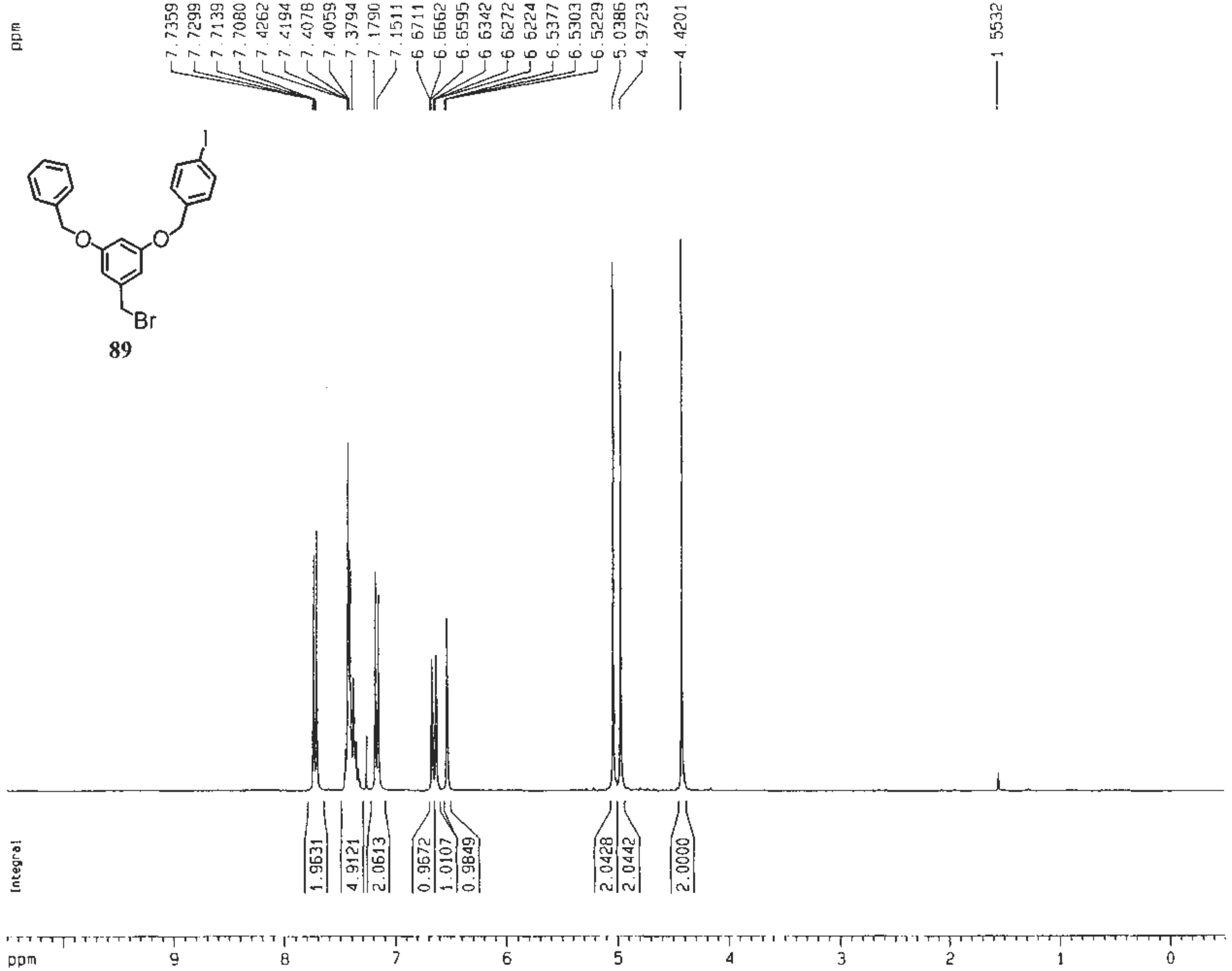
F2 - Acquisition Parameters
Date_           20050108
Time                1.43
INSTRUM           dpx300
PROBHD   5 mm 680 BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  8800
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                2896.3
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677458 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 10.00 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P               10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37384 Hz/cm
```



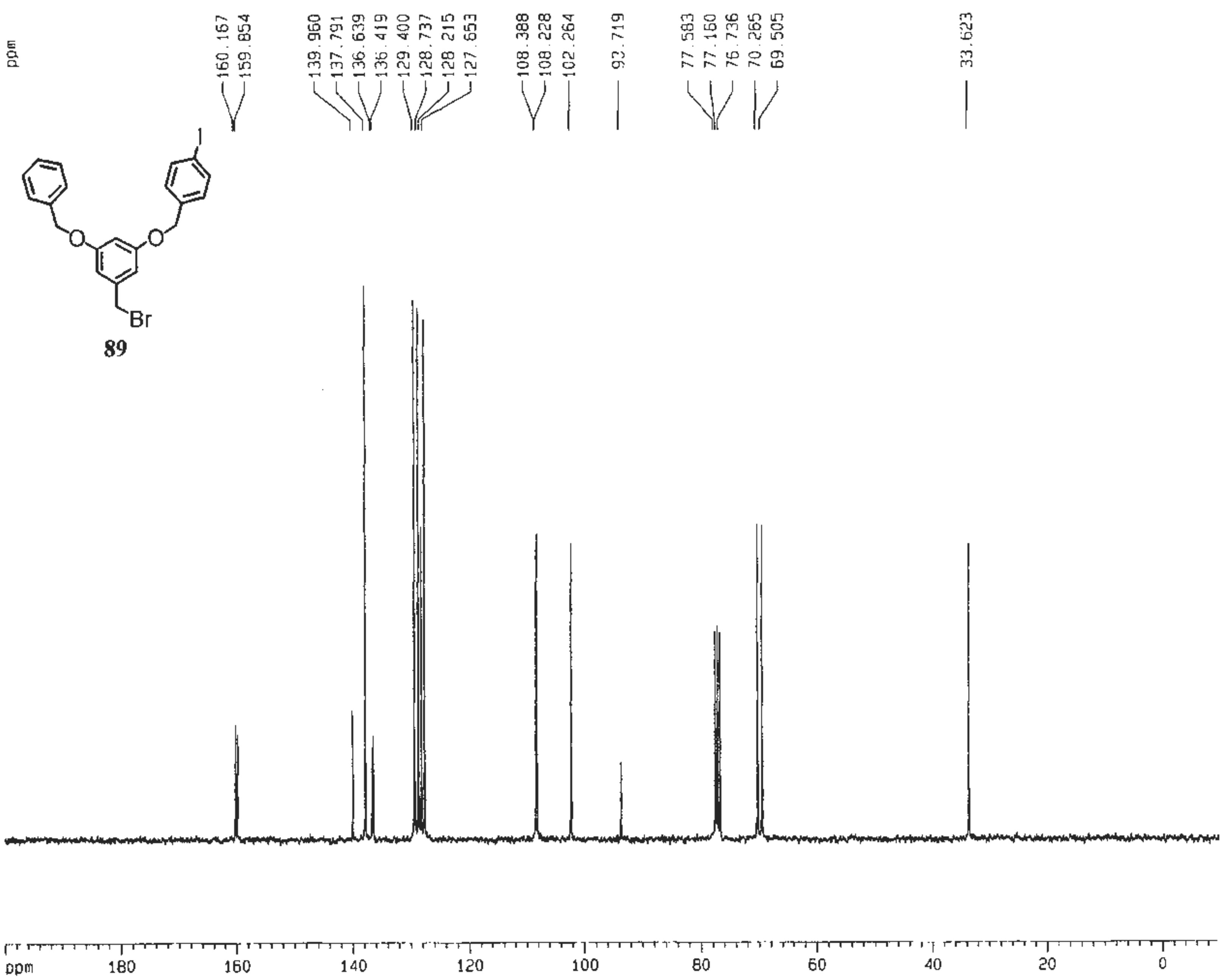
```
Current Data Parameters
NAME          I-G1-BrN
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20050105
Time             11.52
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                   8
DS                   0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AQ           2.2745588 sec
RG                  57
DW             138.825 usec
DE              198.32 usec
TE                 0.0 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1              -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300065 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY                9.95 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



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```
Current Data Parameters
NAME             I-G1-BrNC
EXPNO                    1
PROCNO                   1

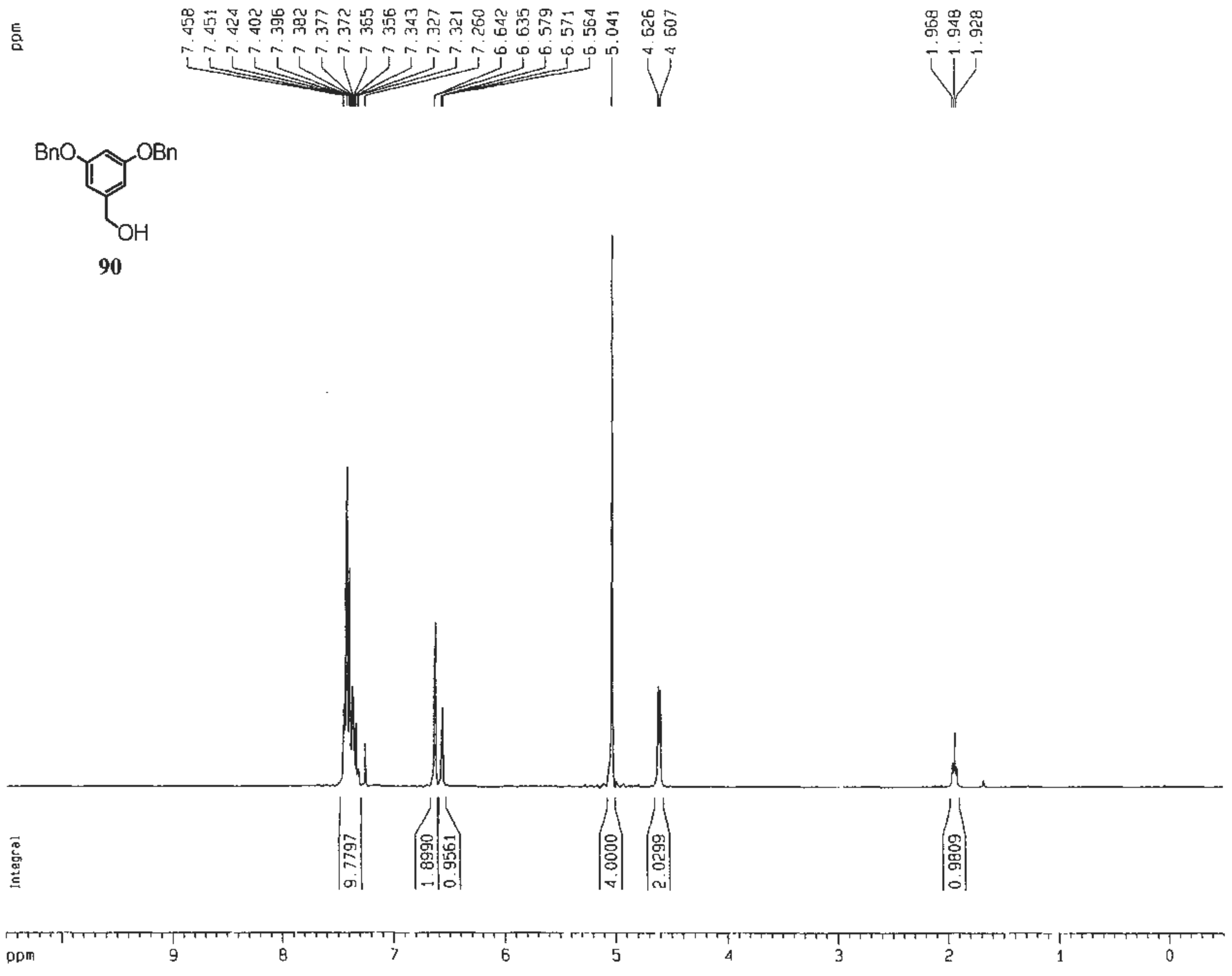
F2 - Acquisition Parameters
Date_             20050108
Time                  0.59
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     401
DS                       0
SWH           22675.736 Hz
FIDRES         0 346004 Hz
AQ           1 4451188 sec
RG                  1625.5
DW              22.050 usec
DE                6 00 usec
TE                   0.0 K
D1        1.00000000 sec
d11       0 03000000 sec
MCREST    0.00000000 sec
MCWRK     0 01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                3.00 usec
PL1               -6 00 dB
SF01        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2           100.00 usec
PL2               120 00 dB
PL12               19.00 dB
SF02       300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF          75 4677444 MHz
WDW                     EM
SSB                      0
LB                 3.00 Hz
GB                       0
PC                    1 40

1D NMR plot parameters
CX                22.00 cm
CY                10.03 cm
F1P            200.000 ppm
F1             15093.55 Hz
F2P            -10 000 ppm
F2              -754.68 Hz
PPMCM        9.54545 ppm/cm
HZCM        720 37384 Hz/cm
```



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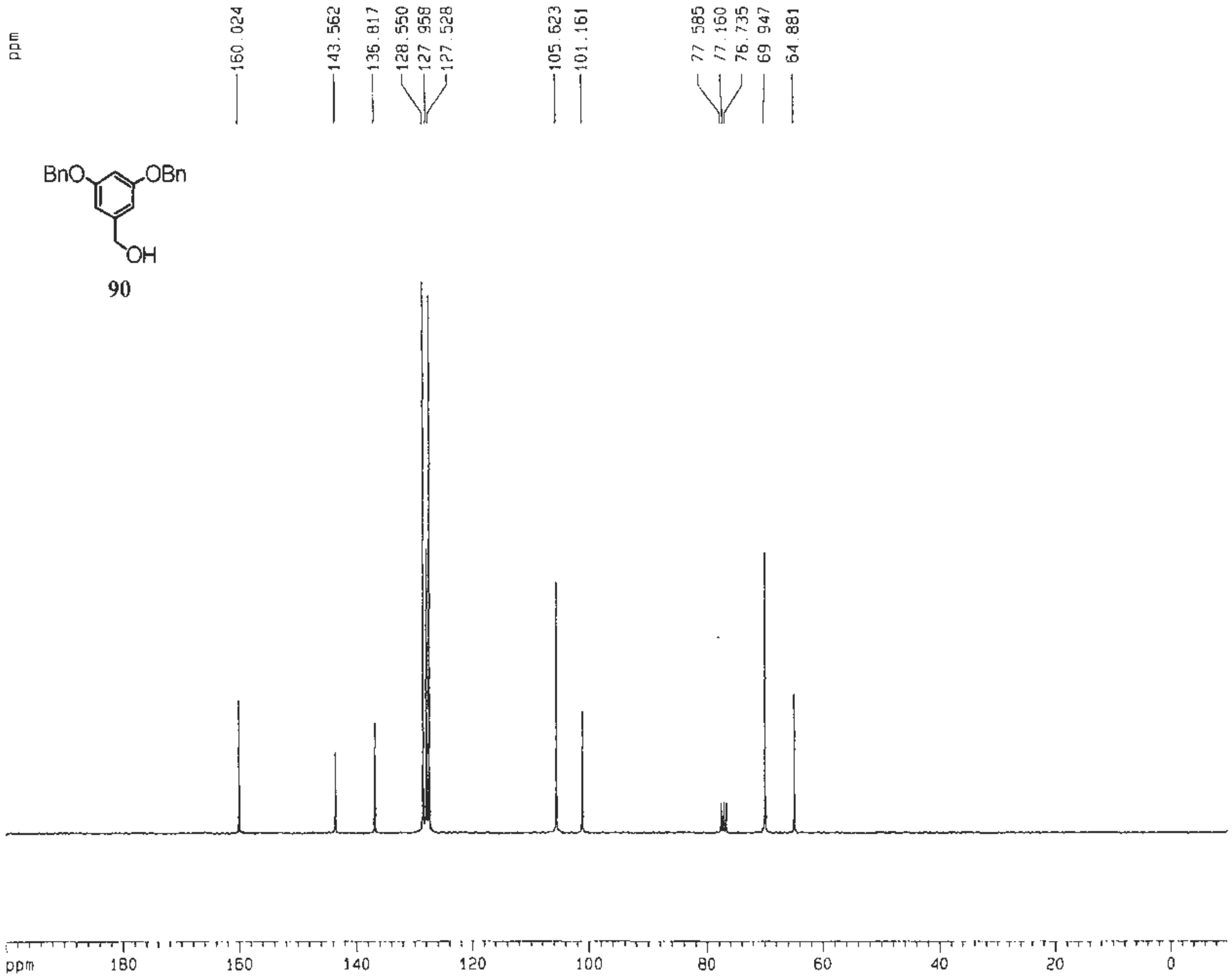
```
Current Data Parameters
NAME            G1-OH
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_        20040617
Time             9.48
INSTRUM        dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zg
TD              32768
SOLVENT         CDCl3
NS                  4
DS                  0
SWH          8992.806 Hz
FIDRES       0.274439 Hz
AQ          1.8219508 sec
RG               90.5
DW             55.600 usec
DE               6.00 usec
TE              297.2 K
D1         1.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1               4.50 usec
PL1             -2.00 dB
SFO1      300.1312000 MHz

F2 - Processing parameters
SI              32768
SF        300.1300066 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00

1D NMR plot parameters
CX              22.00 cm
CY               9.98 cm
F1P            10.500 ppm
F1            3151.36 Hz
F2P            -0.500 ppm
F2            -150.07 Hz
PPMCM         0.50000 ppm/cm
HZCM        150.06500 Hz/cm
```



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```
Current Data Parameters
NAME            G1-OHC
EXPNO                1
PROCNO               1

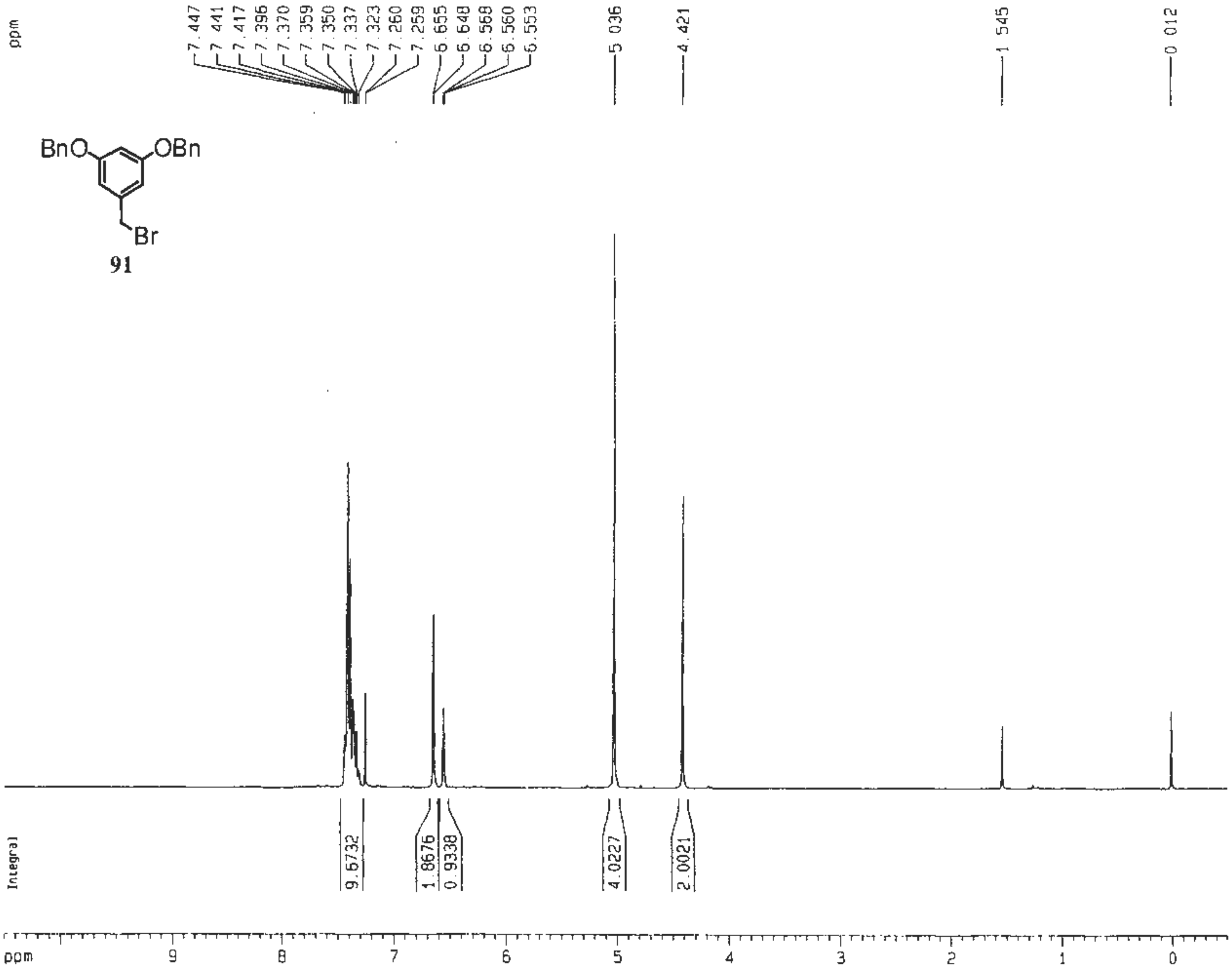
F2 - Acquisition Parameters
Date_         20061206
Time             19.33
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG           zgdc
TD               65536
SOLVENT          CDCl3
NS                 120
DS                   0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                8192
DW              22.050 usec
DE                6.00 usec
TE                 0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                3.00 usec
PL1              -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI               65536
SF          75.4677613 MHz
WDW                 EM
SSB                  0
LB                3.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX               22.00 cm
CY               10.00 cm
F1P            200.000 ppm
F1            15093.55 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM          9.54545 ppm/cm
HZCM         720.37402 Hz/cm
```



91

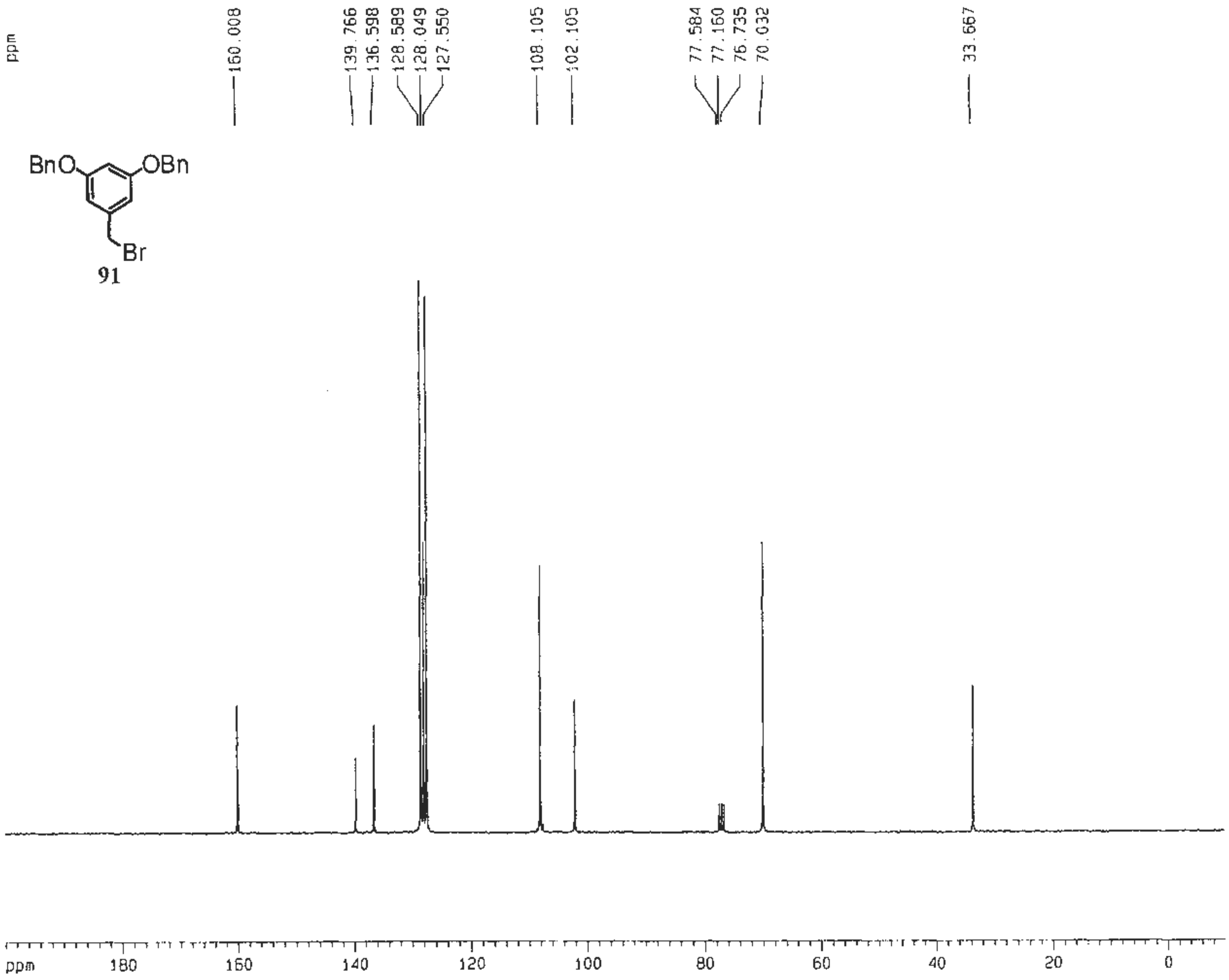
```
Current Data Parameters
NAME             G1-Br
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20030821
Time             17 48
INSTRUM         dpx300
PROBHD    5 mm Dual 13
PULPROG             zg
TD               32768
SOLVENT          CDCl3
NS                   4
DS                   0
SWH           8992.806 Hz
FIDRES        0.274439 Hz
AQ           1.8219508 sec
RG               228.1
DW              55.600 usec
DE                6.00 usec
TE               300.0 K
D1          1.00000000 sec

============ CHANNEL f1 ======
NUC1                1H
P1                4.50 usec
PL1              -2.00 dB
SFO1       300.1312000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300063 MHz
WDW                 EM
SSB                  0
LB                0 30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY                9.97 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0 500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



91

```
Current Data Parameters
NAME            G1-BrC
EXPNO                1
PROCNO               1

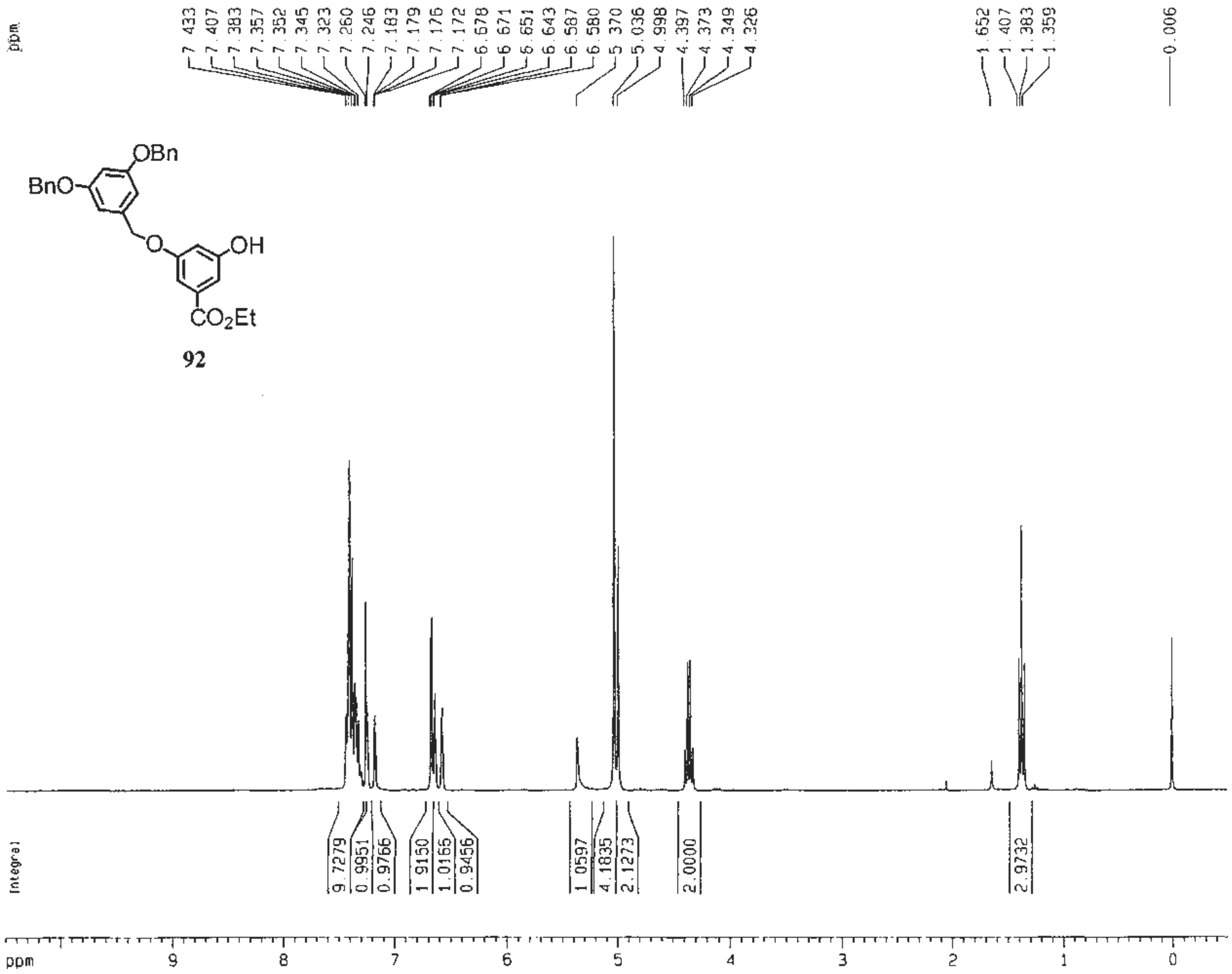
F2 - Acquisition Parameters
Date_         20061207
Time             13 33
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG          zgdc
TD               65536
SOLVENT          CDCl3
NS                 100
DS                   0
SWH         22675 736 Hz
FIDRES       0.346004 Hz
AQ          1 4451188 sec
RG                8192
DW              22.050 usec
DE                6 00 usec
TE                 0.0 K
D1          1.00000000 sec
d11         0 03000000 sec
MCREST      0 00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                3.00 usec
PL1              -6 00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2      300 1315007 MHz

F2 - Processing parameters
SI               65536
SF          75.4677656 MHz
WDW                 EM
SSB                  0
LB                3 00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX               22.00 cm
CY               10.00 cm
F1P            200 000 ppm
F1             15093 55 Hz
F2P            -10 000 ppm
F2             -754.68 Hz
PPMCM          9.54545 ppm/cm
HZCM         720 37408 Hz/cm
```



92

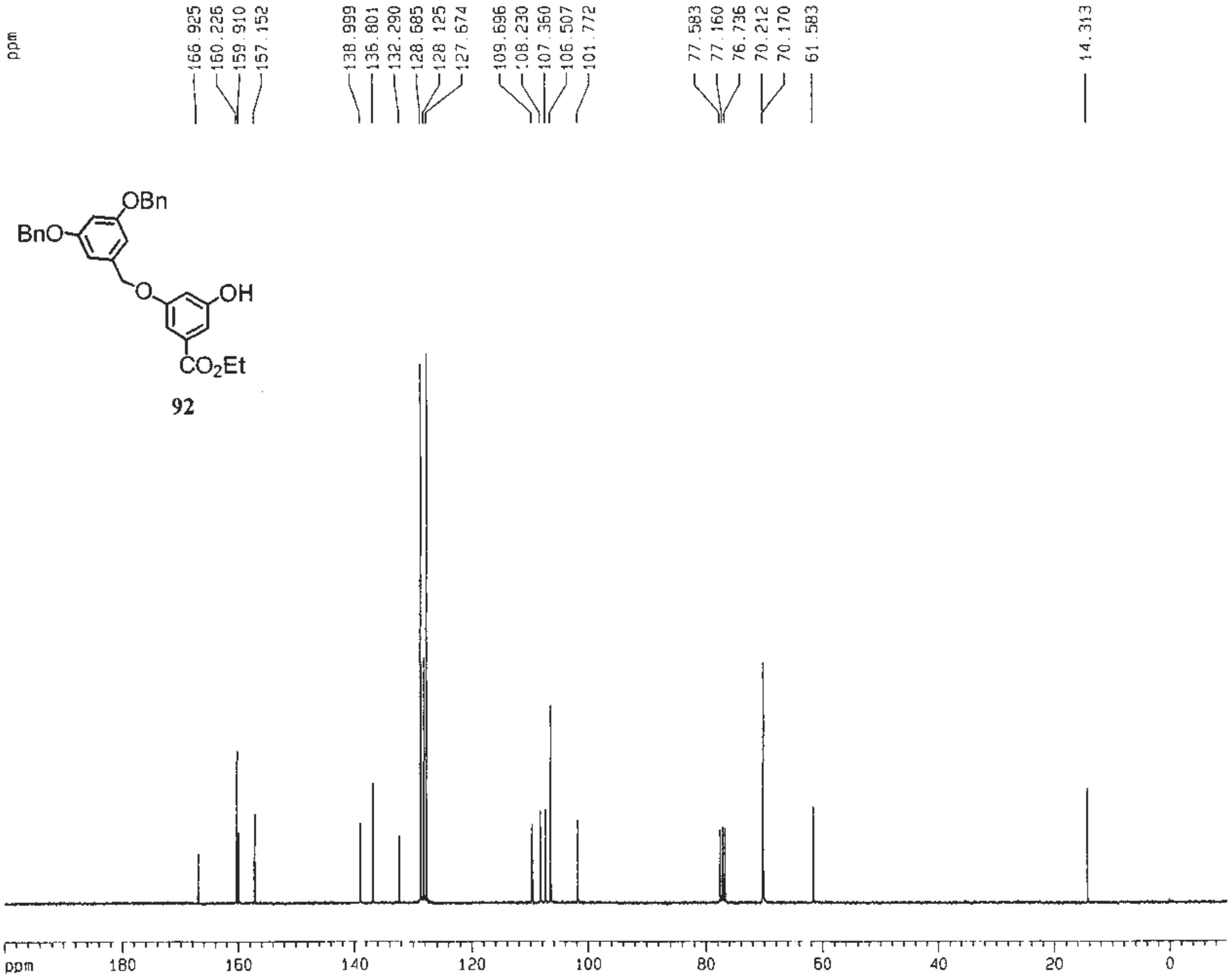
```
Current Data Parameters
NAME             G1-M-ester
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20030822
Time                  12.32
INSTRUM              dpx300
PROBHD      5 mm Dual 13
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                        4
DS                        0
SWH             8992.806 Hz
FIDRES          0 274439 Hz
AQ            1 8219508 sec
RG                      256
DW              55.600 usec
DE                6 00 usec
TE                  300.0 K
D1           1.00000000 sec

============ CHANNEL f1 ======
NUC1                     1H
P1                4.50 usec
PL1                -2.00 dB
SFO1         300 1312000 MHz

F2 - Processing parameters
SI                    32768
SF           300.1300060 MHz
WDW                      EM
SSB                       0
LB                  0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  9.92 cm
F1P               10 500 ppm
F1               3151 36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM         0.50000 ppm/cm
HZCM        150 06500 Hz/cm
```



92

```
Current Data Parameters
NAME         G1-M-esterC
EXPNO                  1
PROCNO                 1

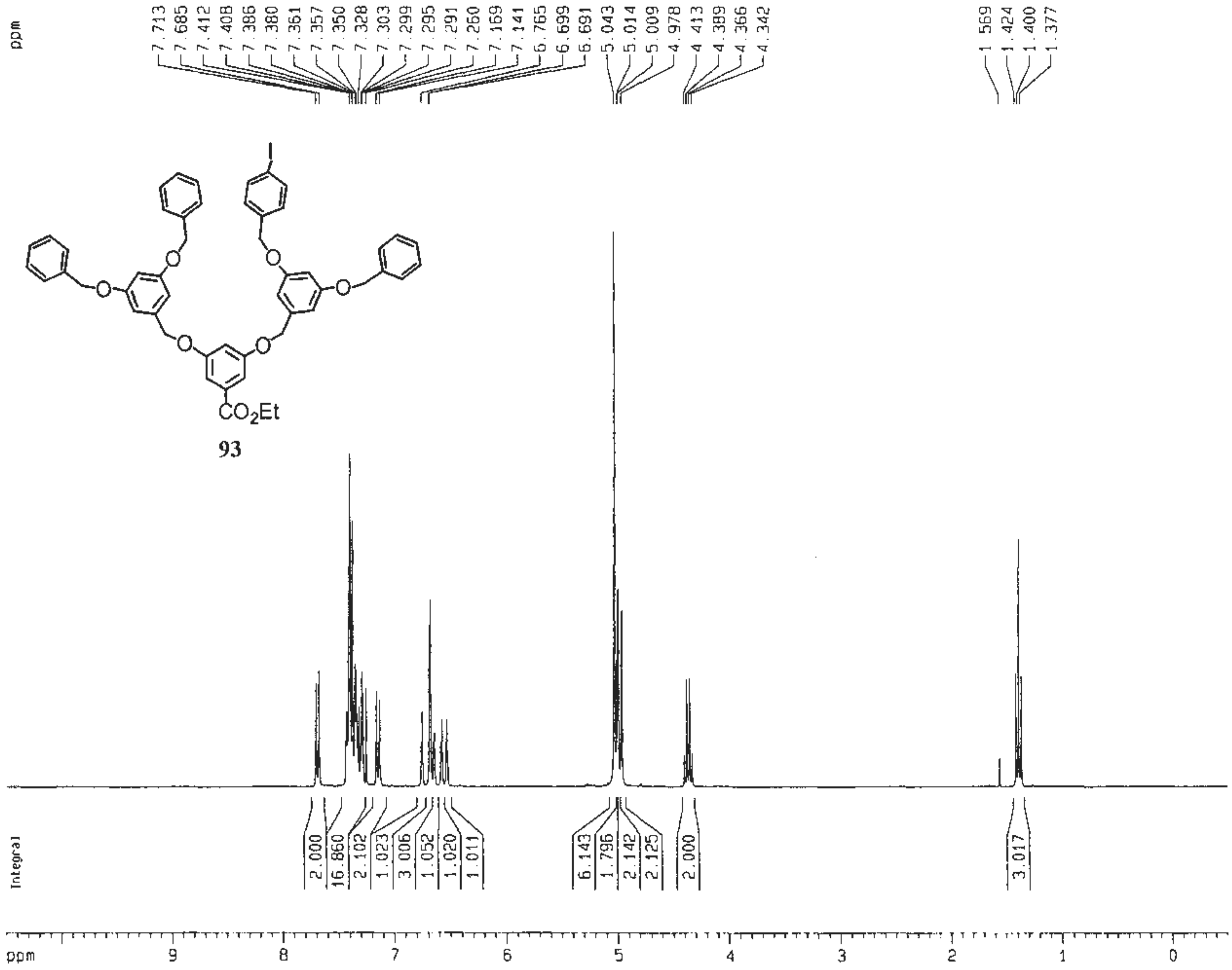
F2 - Acquisition Parameters
Date_           20030905
Time               16.32
INSTRUM           dpx300
PROBHD      5 mm Dual 13
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   381
DS                     0
SWH            22675 736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 300 0 K
D1            1 00000000 sec
d11           0.03000000 sec

============ CHANNEL f1 ======
NUC1                 13C
P1                  3 00 usec
PL1                -6 00 dB
SFO1         75.4745111 MHz

============ CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120 00 dB
PL12               19.00 dB
SFO2         300 1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677456 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                  9.99 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754 68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37384 Hz/cm
```



93

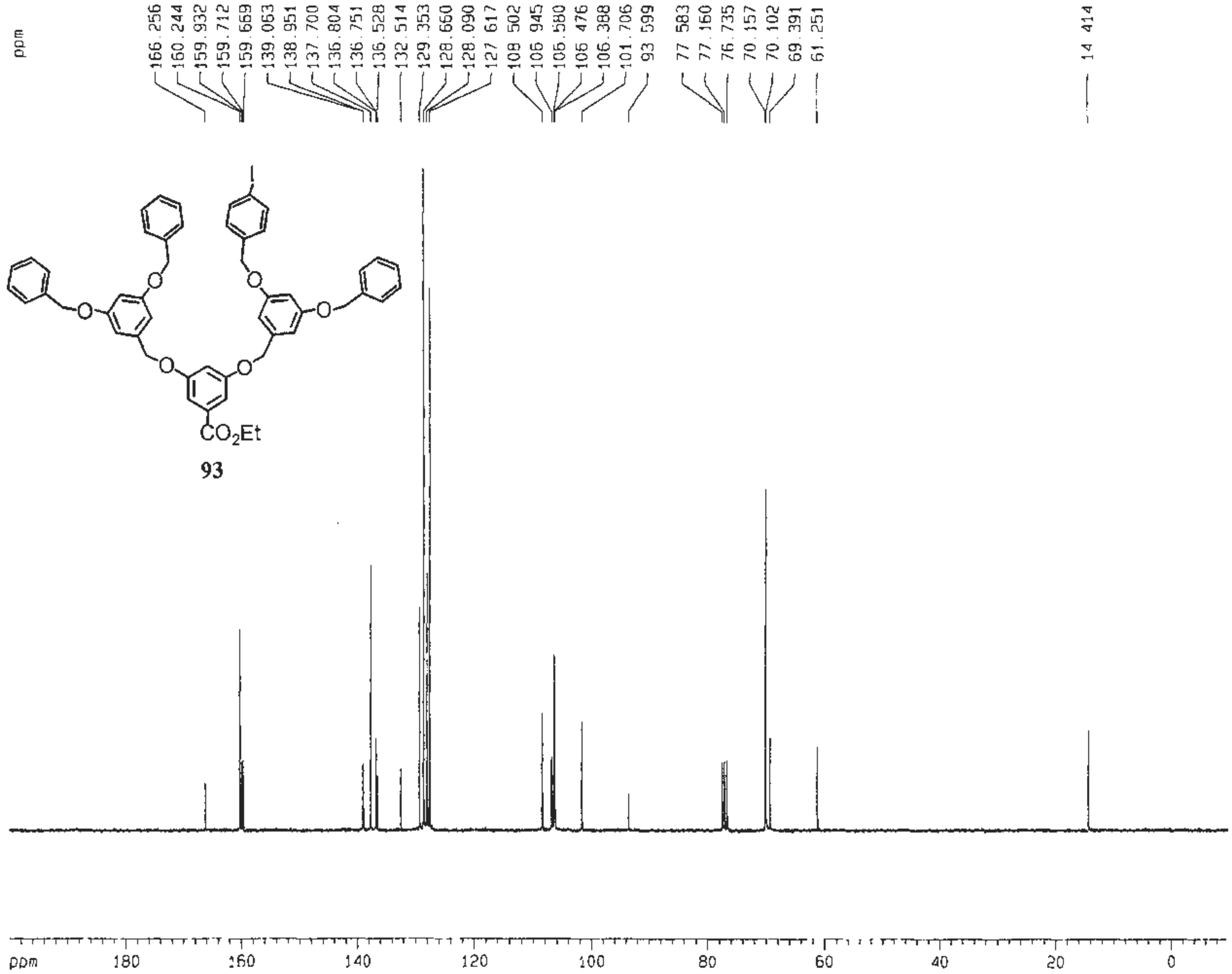
```
Current Data Parameters
NAME          I-G2-esterN
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20061220
Time                17.59
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              4202.563 Hz
FIDRES           0.256504 Hz
AQ              1.9493364 sec
RG                  143.7
DW                118.975 usec
DE                 169.96 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0 01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9 00 usec
PL1                 -2.00 dB
SFO1          300.1318000 MHz

F2 - Processing parameters
SI                  32768
SF            300 1300062 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1 00

1D NMR plot parameters
CX                  22.00 cm
CY                   9 97 cm
F1P                10 500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0 50000 ppm/cm
HZCM            150.06500 Hz/cm
```



93

```
Current Data Parameters
NAME       I-G2-esterNC
EXPNO                 1
PROCNO                1

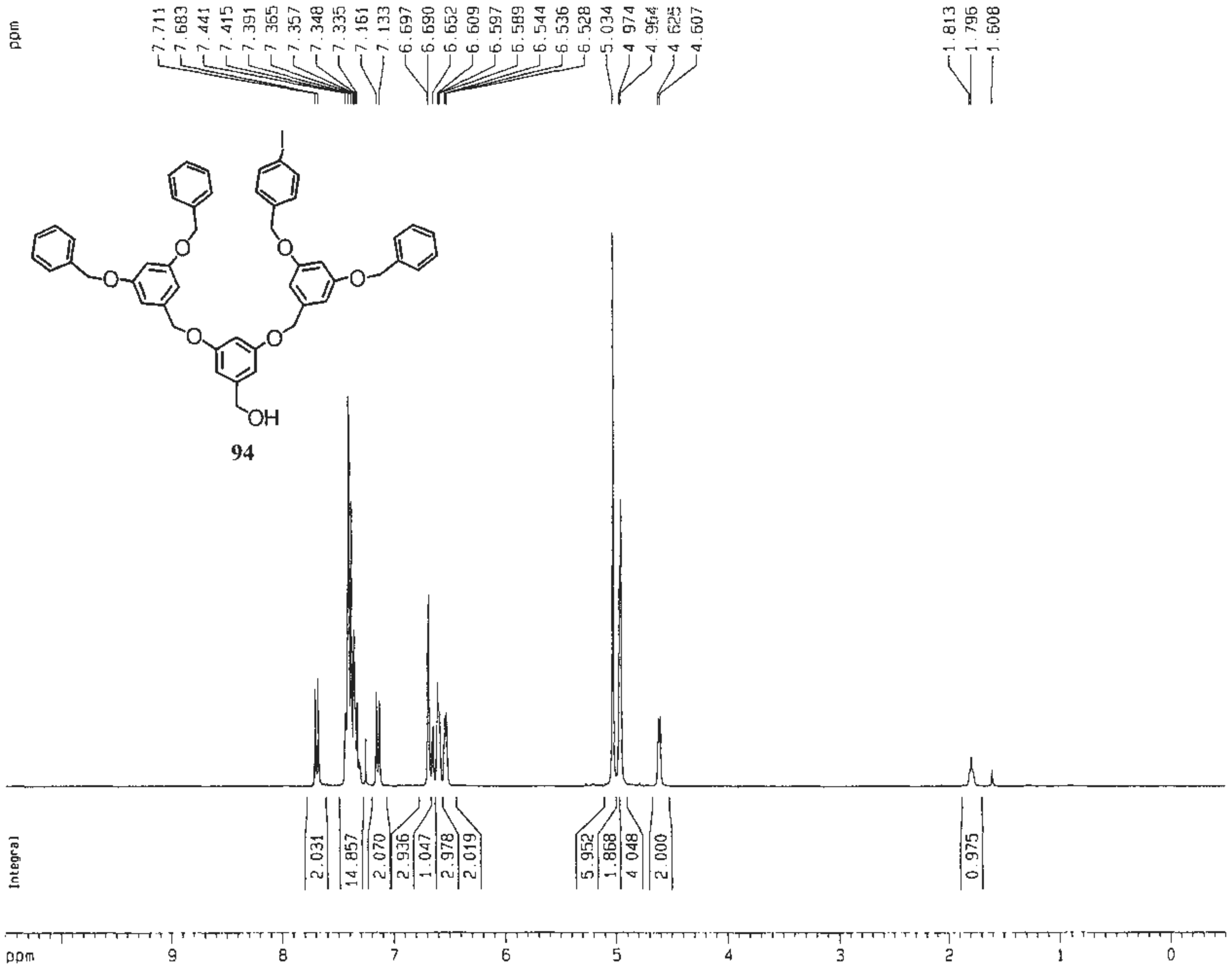
F2 - Acquisition Parameters
Date_          20061220
Time              18 03
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1          1 00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2       300 1315007 MHz

F2 - Processing parameters
SI                65536
SF           75 4677502 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                12.00 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9 54545 ppm/cm
HZCM          720.37390 Hz/cm
```



94

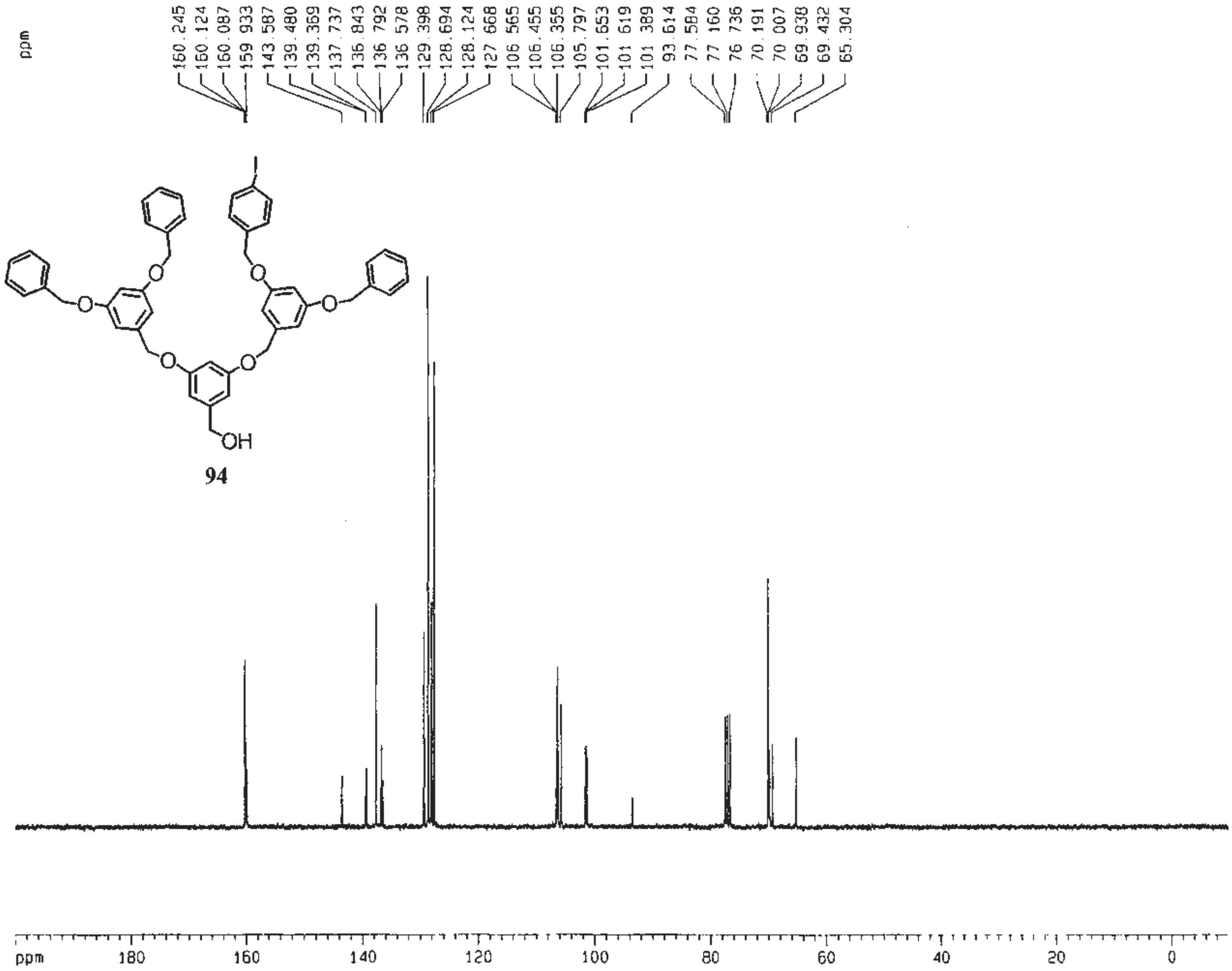
```
Current Data Parameters
NAME          I-G2-OHN
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20070106
Time             18 09
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                  32
DS                   0
SWH          4194.631 Hz
FIDRES       0.256020 Hz
AQ          1.9530228 sec
RG                90.5
DW             119.200 usec
DE               6.00 usec
TE                0.0 K
D1         5.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                4.50 usec
PL1              -2.00 dB
SFO1       300.1318000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300062 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY                9.95 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



94

```
Current Data Parameters
NAME          I-G2-OHNC
EXPNO                 1
PROCNO                1

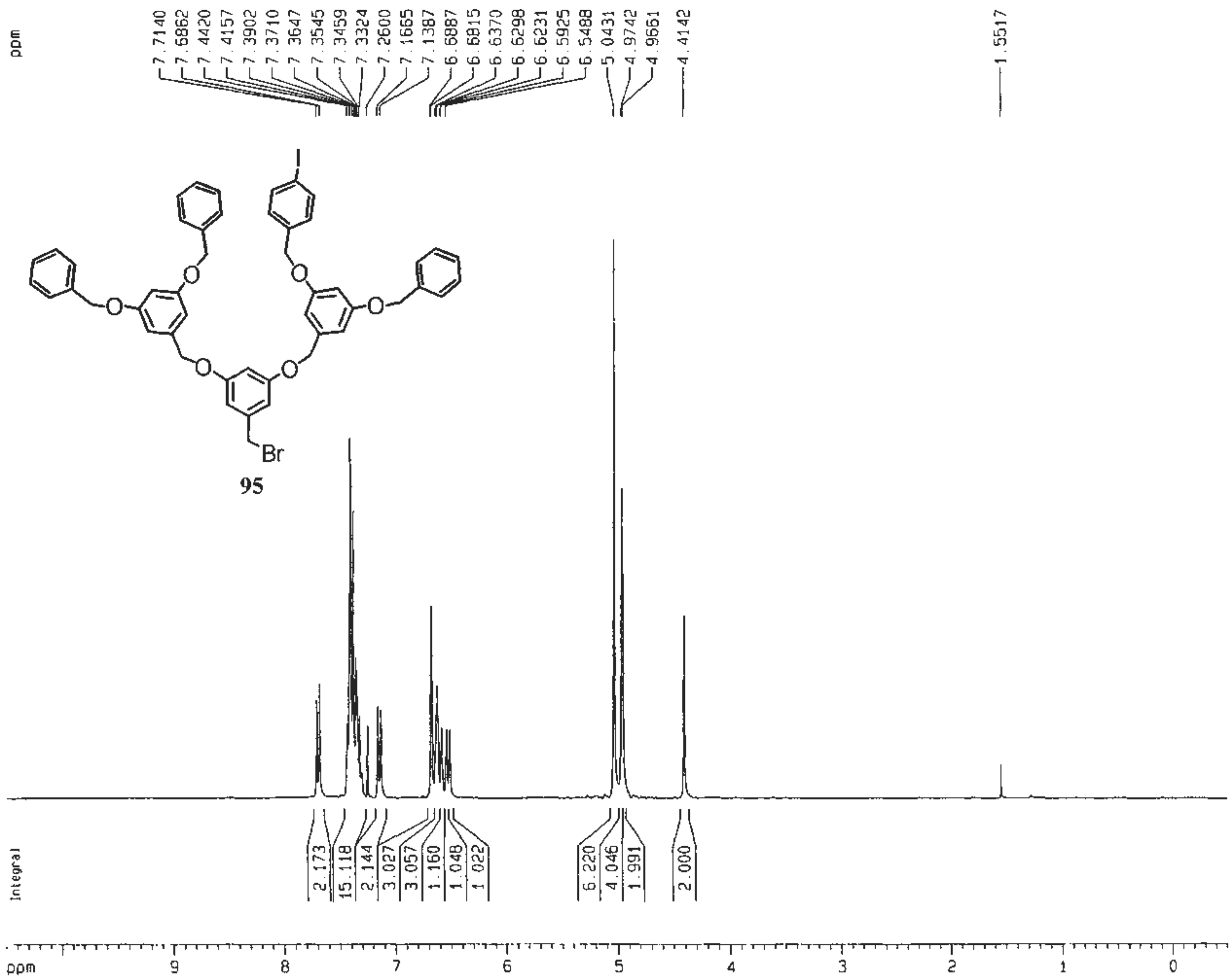
F2 - Acquisition Parameters
Date_          20061222
Time              23.38
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677453 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                10.02 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37384 Hz/cm
```



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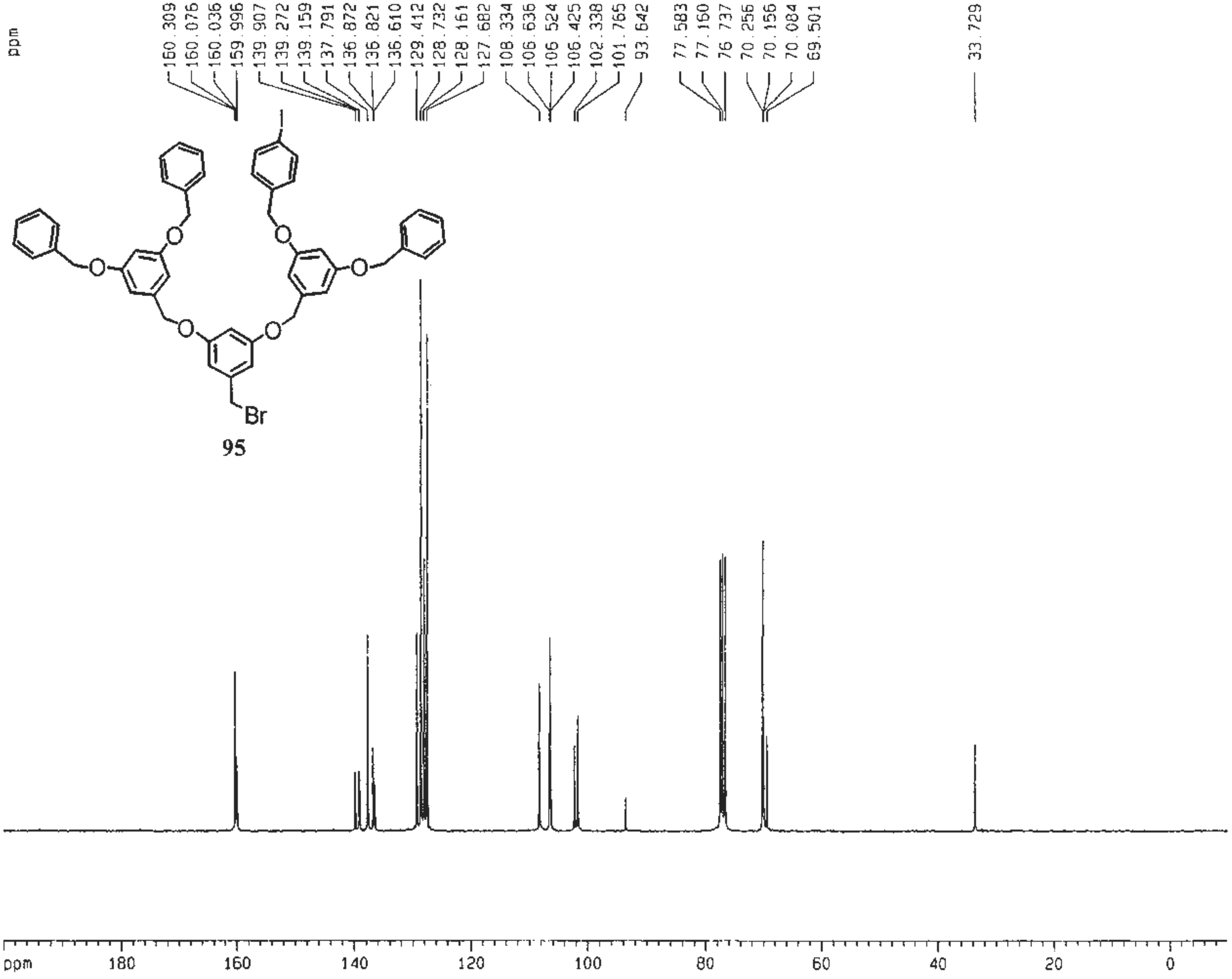
```
Current Data Parameters
NAME             I-G2-BrN
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20050110
Time                18.37
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                      8
DS                      0
SWH             3601.657 Hz
FIDRES          0.219828 Hz
AQ             2 2745588 sec
RG                  101.6
DW               138.825 usec
DE                198 32 usec
TE                    0.0 K
D1            5.00000000 sec
MCREST        0 00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300054 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  10.04 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150 06500 Hz/cm
```



95

```
Current Data Parameters
NAME          I-62-BrNC
EXPNO               1
PROCNO              1

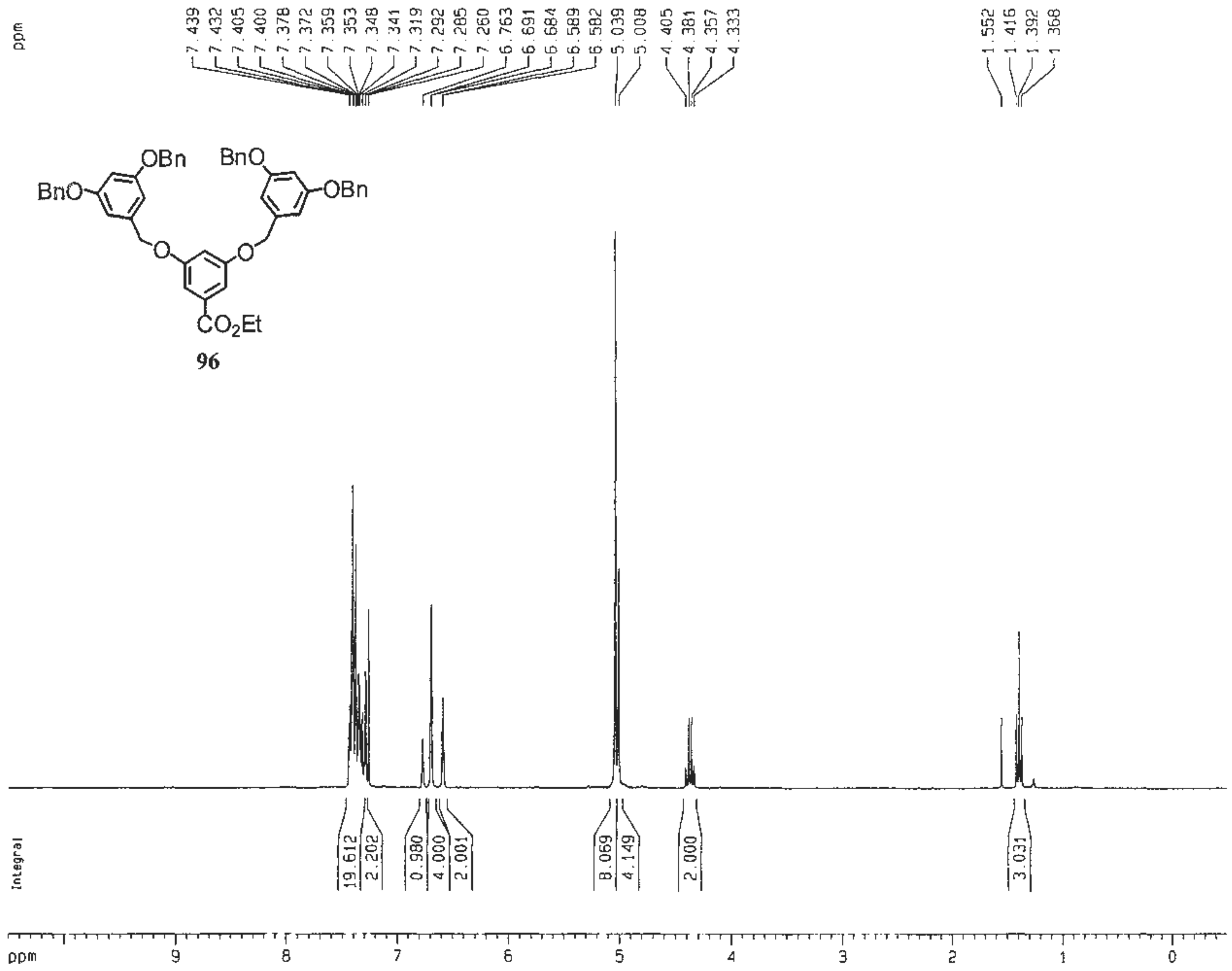
F2 - Acquisition Parameters
Date_         20050129
Time              0.04
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                11040
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SF01        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SF02       300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677411 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                 9.95 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37384 Hz/cm
```



96

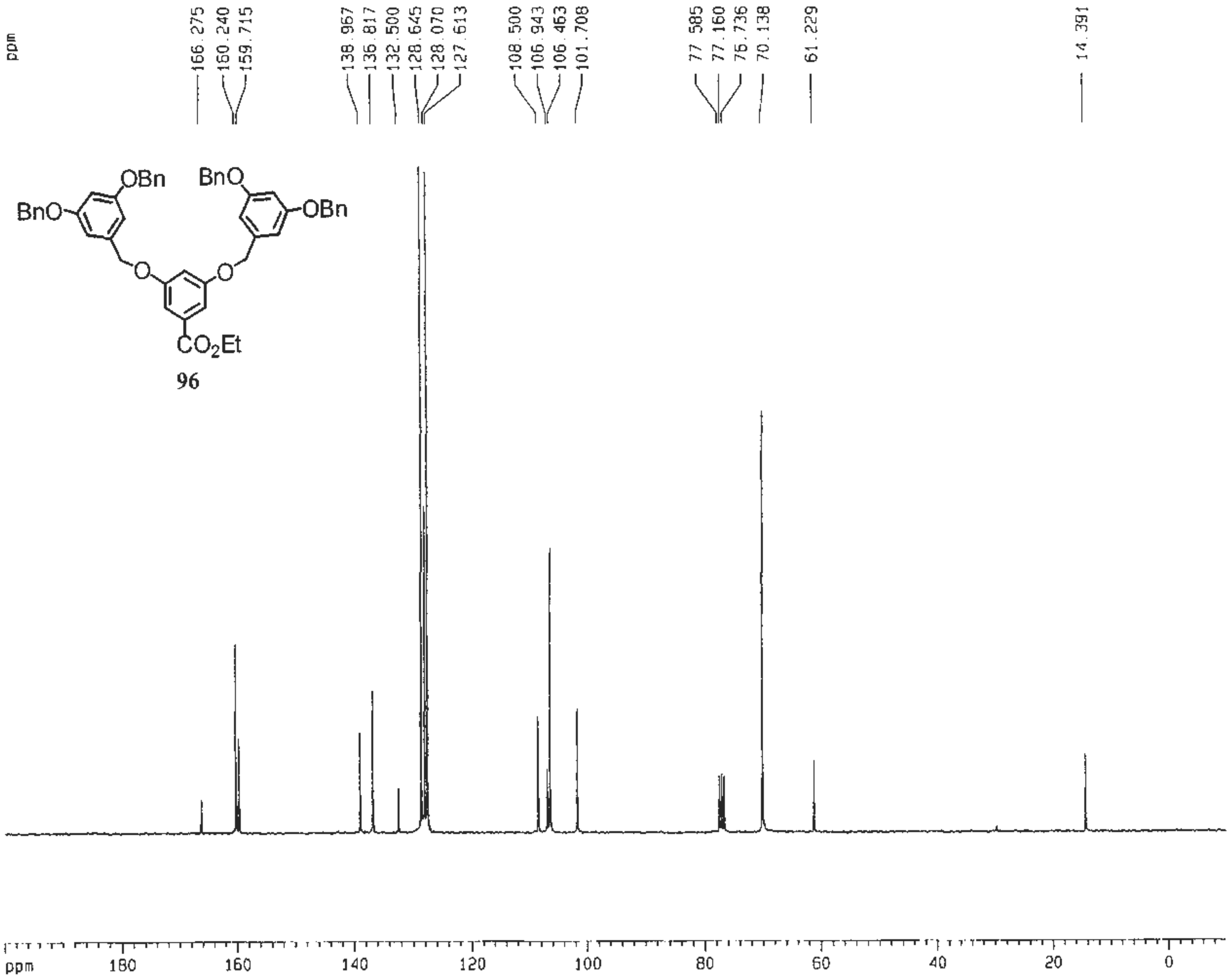
```
Current Data Parameters
NAME          G2-ester
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20060404
Time             15 04
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zg
TD               16384
SOLVENT          CDCl3
NS                   8
DS                   0
SWH          3601 657 Hz
FIDRES       0 219828 Hz
AQ          2.2745588 sec
RG                 256
DW             138.825 usec
DE              198.32 usec
TE                 0 0 K
D1         5.00000000 sec
MCREST     0.00000000 sec
MCWRK      0 01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1              -2 00 dB
SFO1      300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300064 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1 00

1D NMR plot parameters
CX               22.00 cm
CY               10.02 cm
F1P             10.500 ppm
F1             3151 36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



96

```
Current Data Parameters
NAME          G2-esterC
EXPNO                 1
PROCNO                1

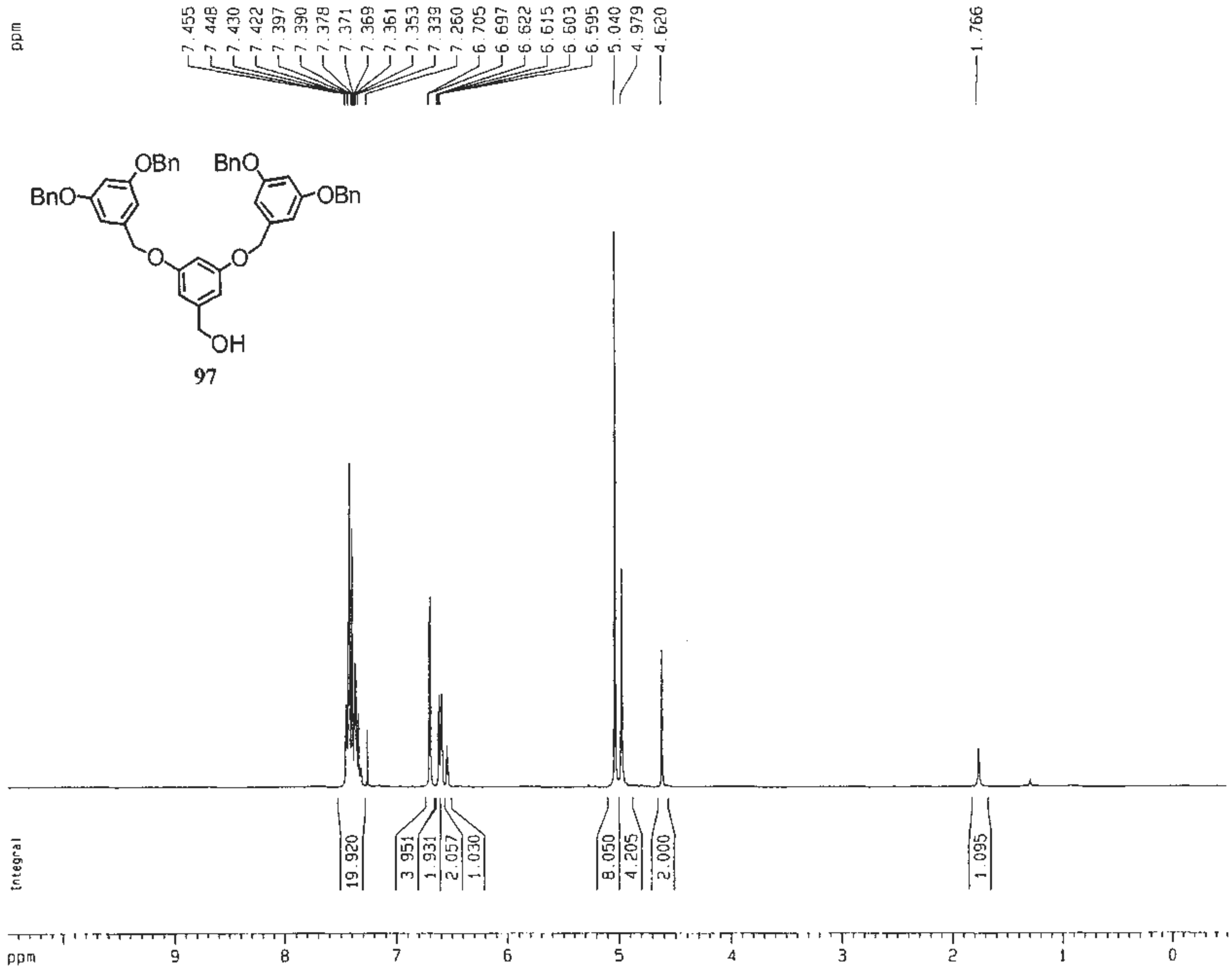
F2 - Acquisition Parameters
Date_          20060404
Time              16 18
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  381
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
HCREST      0.00000000 sec
HCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3 00 usec
PL1               -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100 00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677520 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                12.00 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10 000 ppm
F2              -754.68 Hz
PPMCM           9 54545 ppm/cm
HZCM          720.37390 Hz/cm
```



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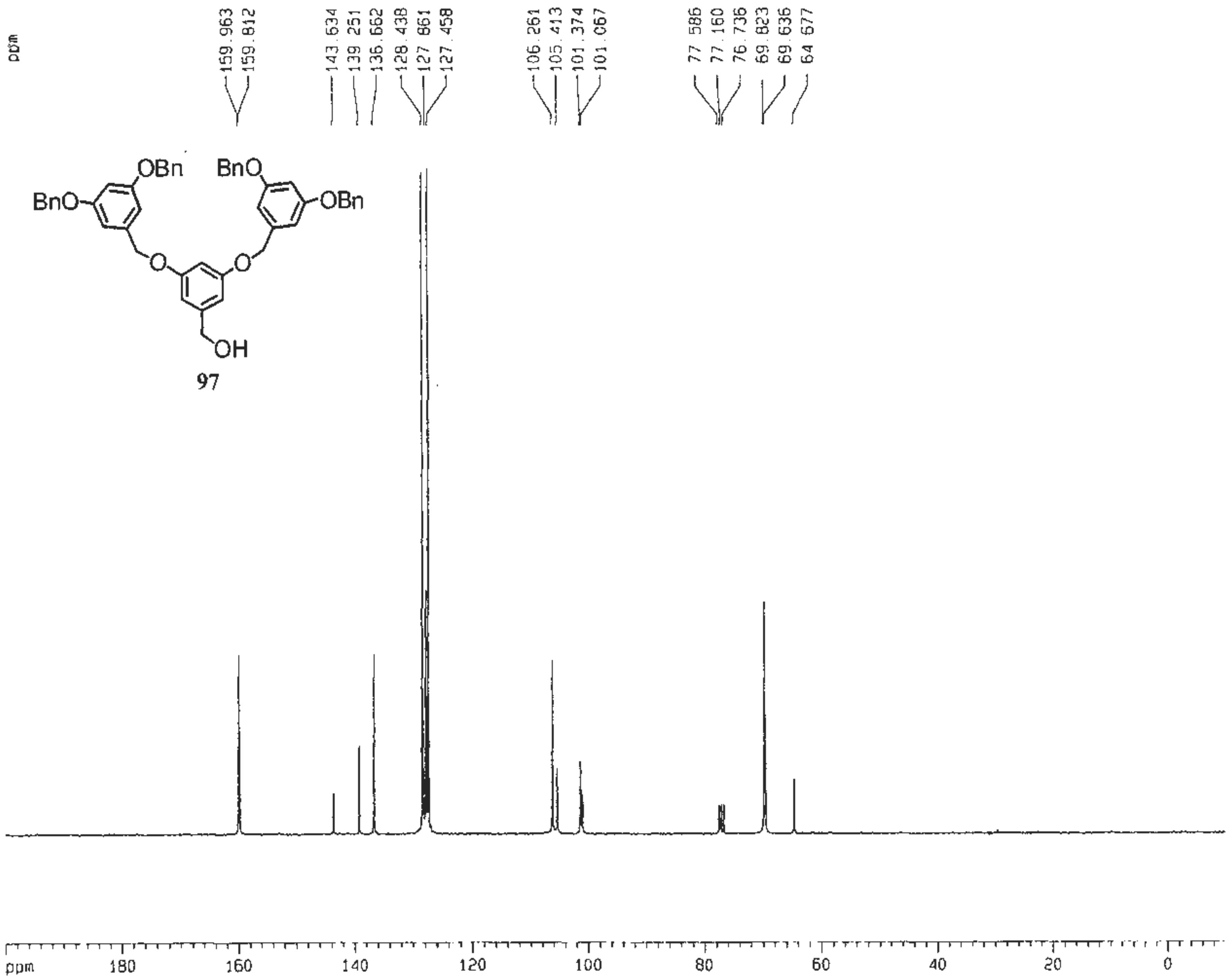
```
Current Data Parameters
NAME             G2-OH
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20060509
Time             16.43
INSTRUM         dpx300
PROBHD   5 mm BB0 BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           4202.563 Hz
FIDRES        0.256504 Hz
AQ           1.9493364 sec
RG                28.5
DW             118.975 usec
DE              169.96 usec
TE                 0.0 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWORK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1              -2.00 dB
SFO1       300.1318000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300063 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY                9.98 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



```
Current Data Parameters
NAME            G2-OHC
EXPNO                1
PROCNO               1

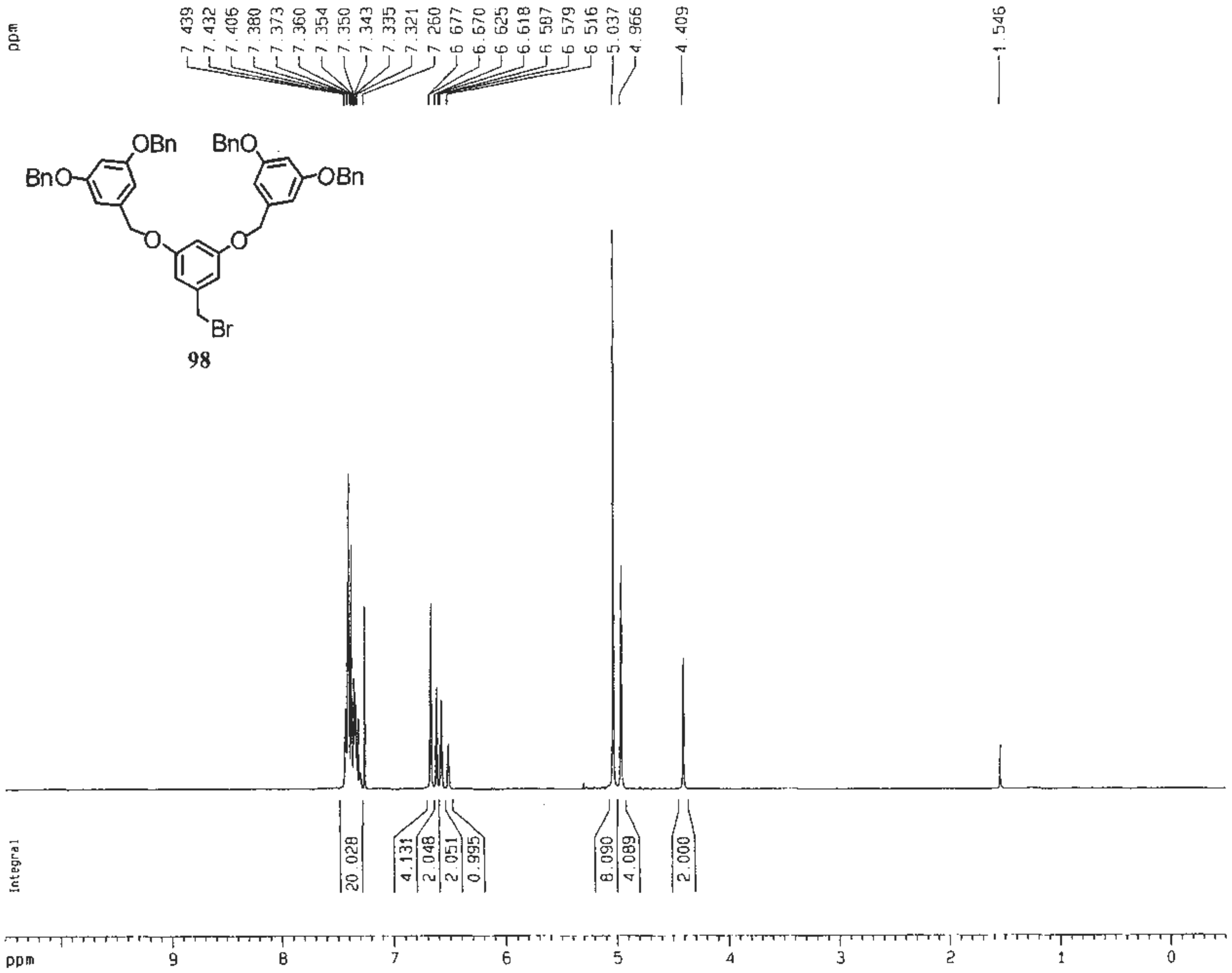
F2 - Acquisition Parameters
Date_         20060509
Time             16.02
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG           zgdc
TD               65536
SOLVENT          CDCl3
NS                 101
DS                   0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                8192
DW              22.050 usec
DE                6.00 usec
TE                 0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                3.00 usec
PL1              -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI               65536
SF          75.4677791 MHz
WDW                 EM
SSB                  0
LB                3.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX               22.00 cm
CY               11.97 cm
F1P            200.000 ppm
F1            15093.55 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM          9.54545 ppm/cm
HZCM         720.37421 Hz/cm
```



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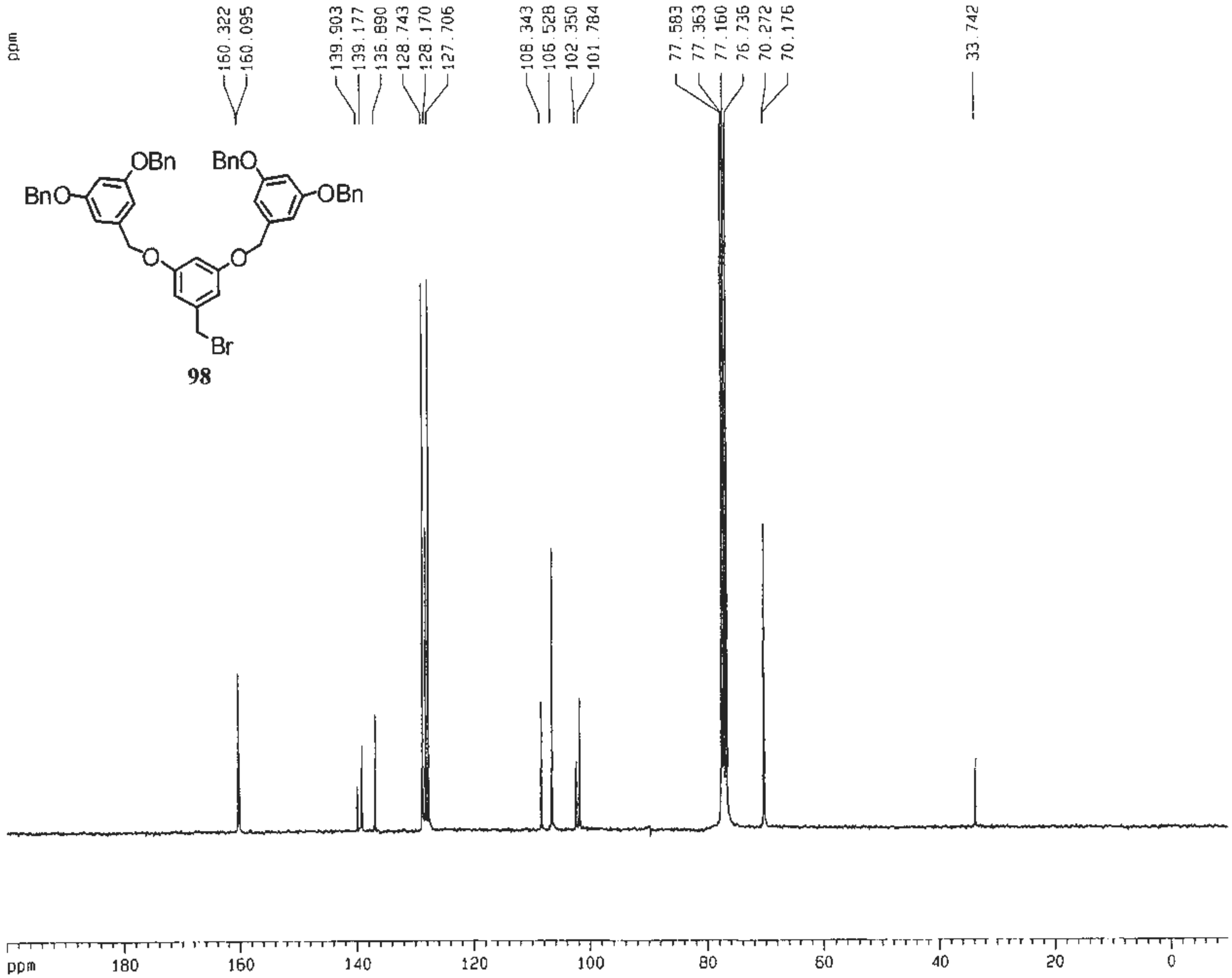
```
Current Data Parameters
NAME              62-Br
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20060510
Time              17.27
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            4202.563 Hz
FIDRES         0.256504 Hz
AQ            1.9493364 sec
RG                 28.5
DW              118.975 usec
DE               169.96 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1318000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300063 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.02 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



98

```
Current Data Parameters
NAME              G2-BrC
EXPNO                  1
PROCNO                 1

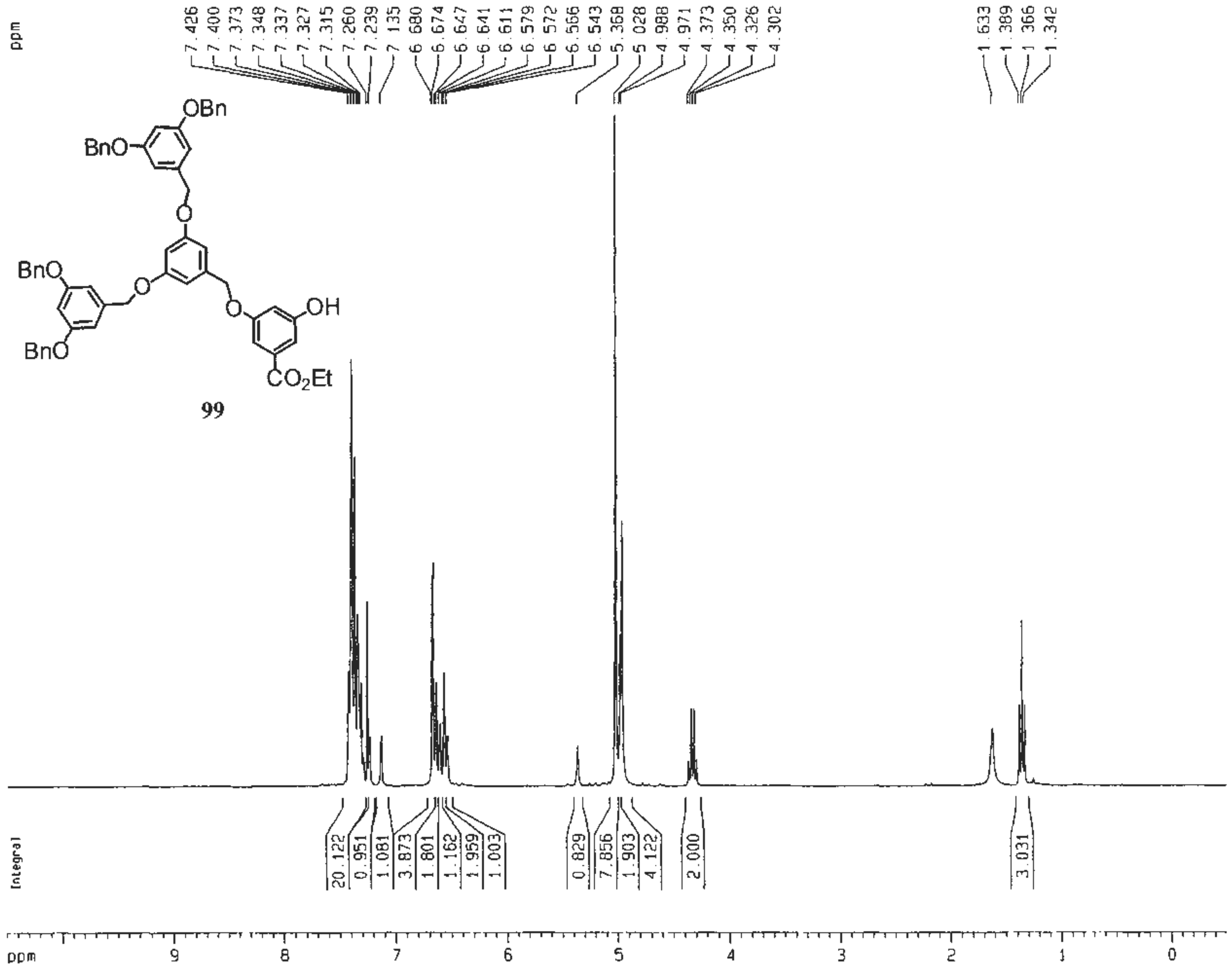
F2 - Acquisition Parameters
Date_           20060510
Time               21 04
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                 21000
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0 0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3 00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677389 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 19.85 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM          720.37378 Hz/cm
```



99

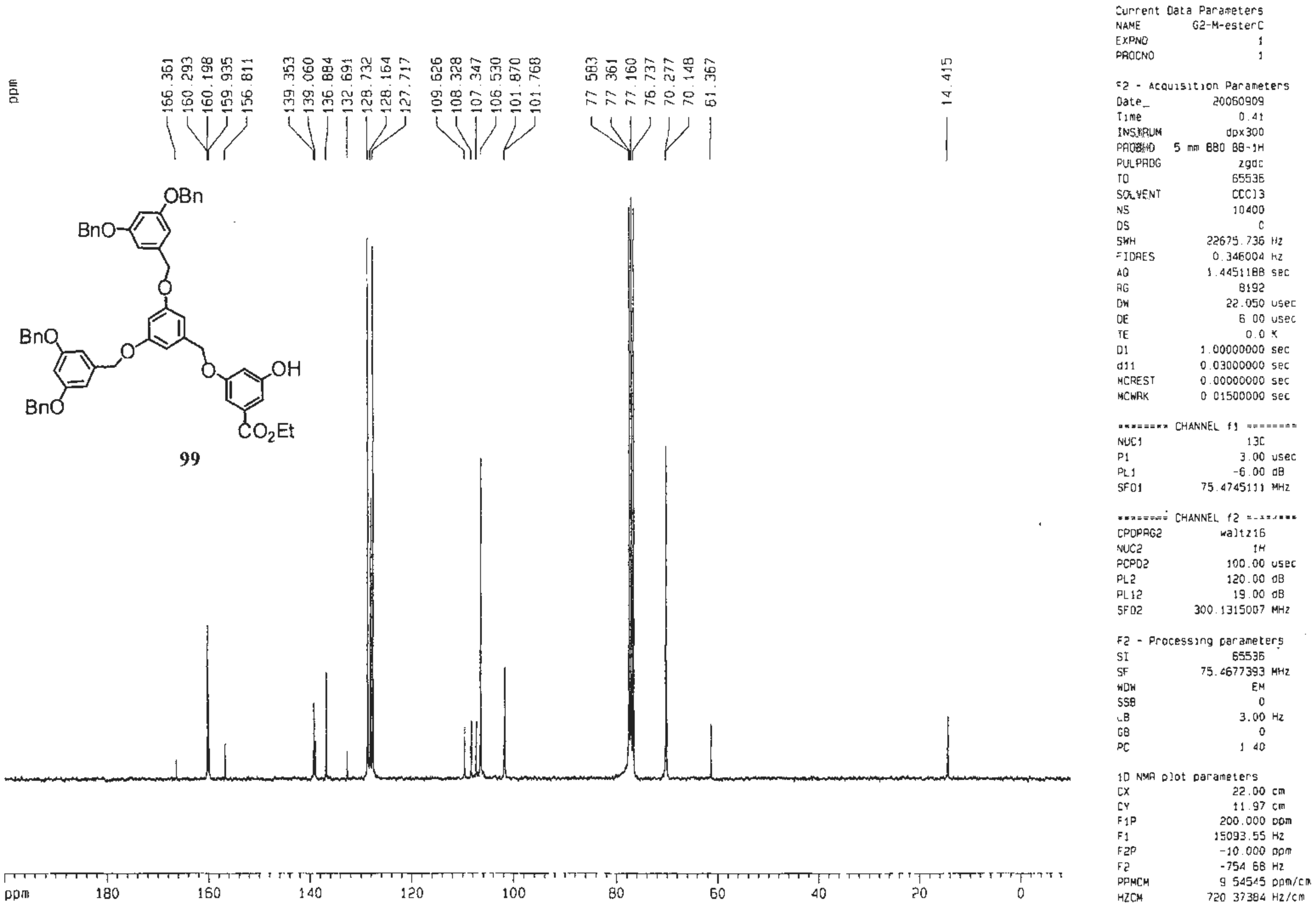
```
Current Data Parameters
NAME        G2-M-ester
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20060908
Time             11.22
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                  21
DS                   0
SWH           4194.631 Hz
FIDRES        0.256020 Hz
AQ           1.9530228 sec
RG               287.4
DW             119.200 usec
DE                6.00 usec
TE                 0.0 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWORK      0.01500000 sec

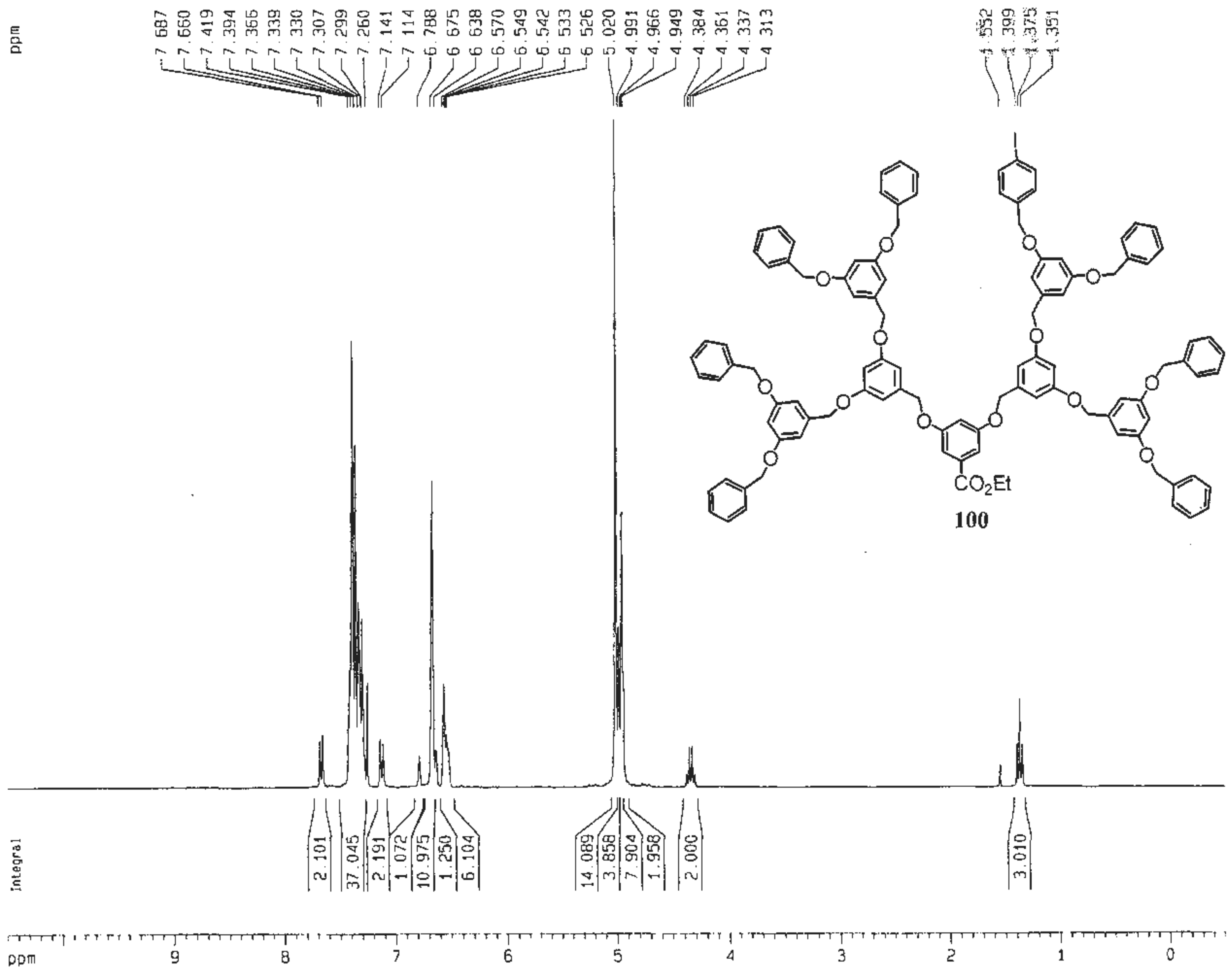
======== CHANNEL f1 ========
NUC1                1H
P1                4.50 usec
PL1              -2.00 dB
SFO1       300.1318000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300052 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY               12.03 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```


ppm
166.361
160.293
160.198
159.935
156.811
139.353
139.060
136.884
132.691
128.732
128.164
127.717
109.626
108.328
107.347
106.530
101.870
101.768
77.583
77.361
77.160
76.737
70.277
70.148
61.367
14.415
OBn
BnO
BnO
BnO
OH
CO2Et
Current Data Parameters
NAME G2-M-esterC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060909
Time 0.41
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 10400
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz
F2 - Processing parameters
SI 65536
SF 75.4677393 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
CY 11.97 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.54545 ppm/cm
HZCM 720.37384 Hz/cm
ppm
180
160
140
120
100
80
60
40
20
0

99



100

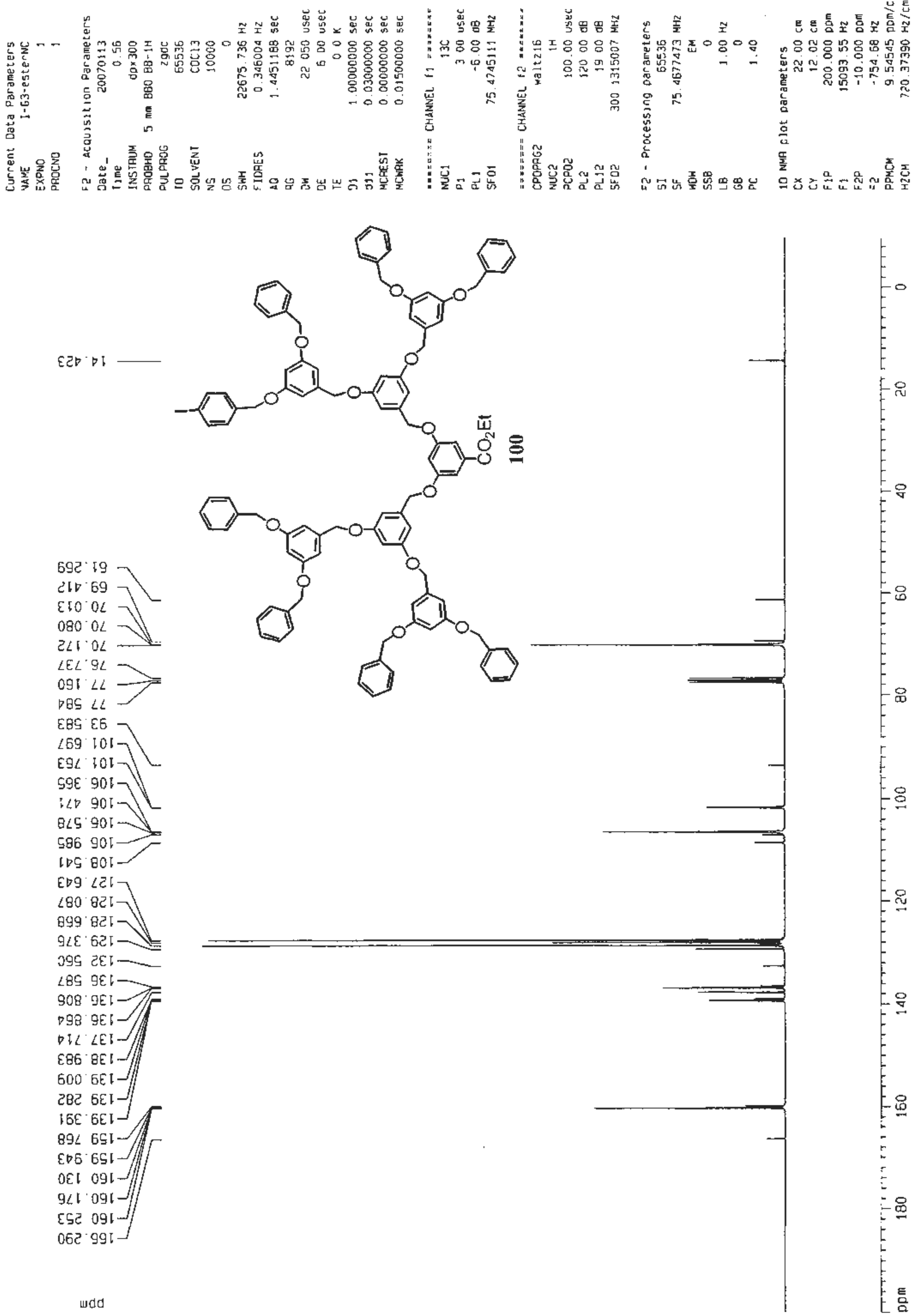
```
Current Data Parameters
NAME          I-63-esterN
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070110
Time                13.34
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              4194.631 Hz
FIDRES           0.256020 Hz
AQ              1.9530228 sec
RG                  203.2
DW                119.200 usec
DE                   6.00 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

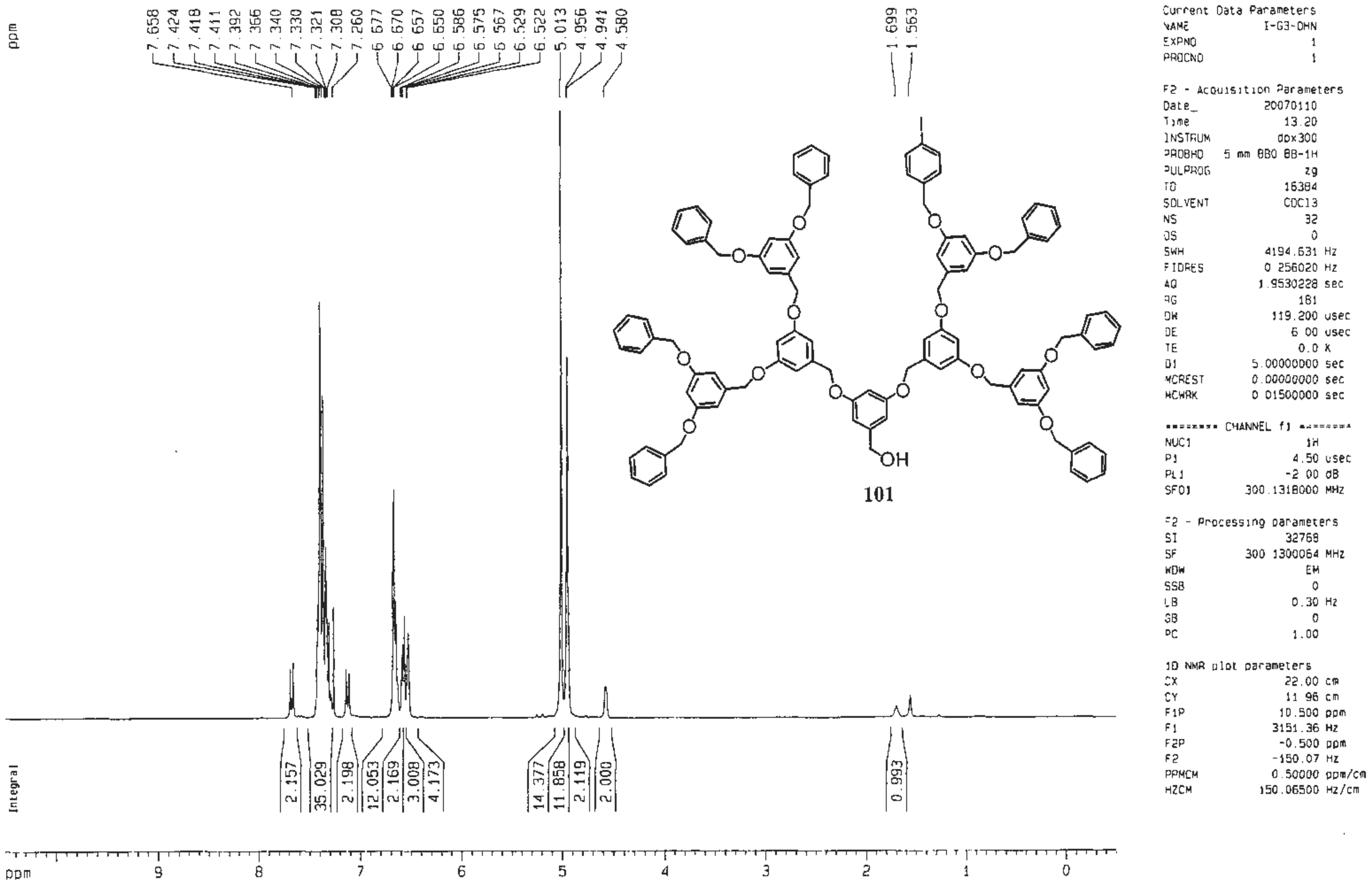
======== CHANNEL f1 ========
NUC1                   1H
P1                   4.50 usec
PL1                 -2.00 dB
SFO1          300.1318000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300065 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  11.99 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```

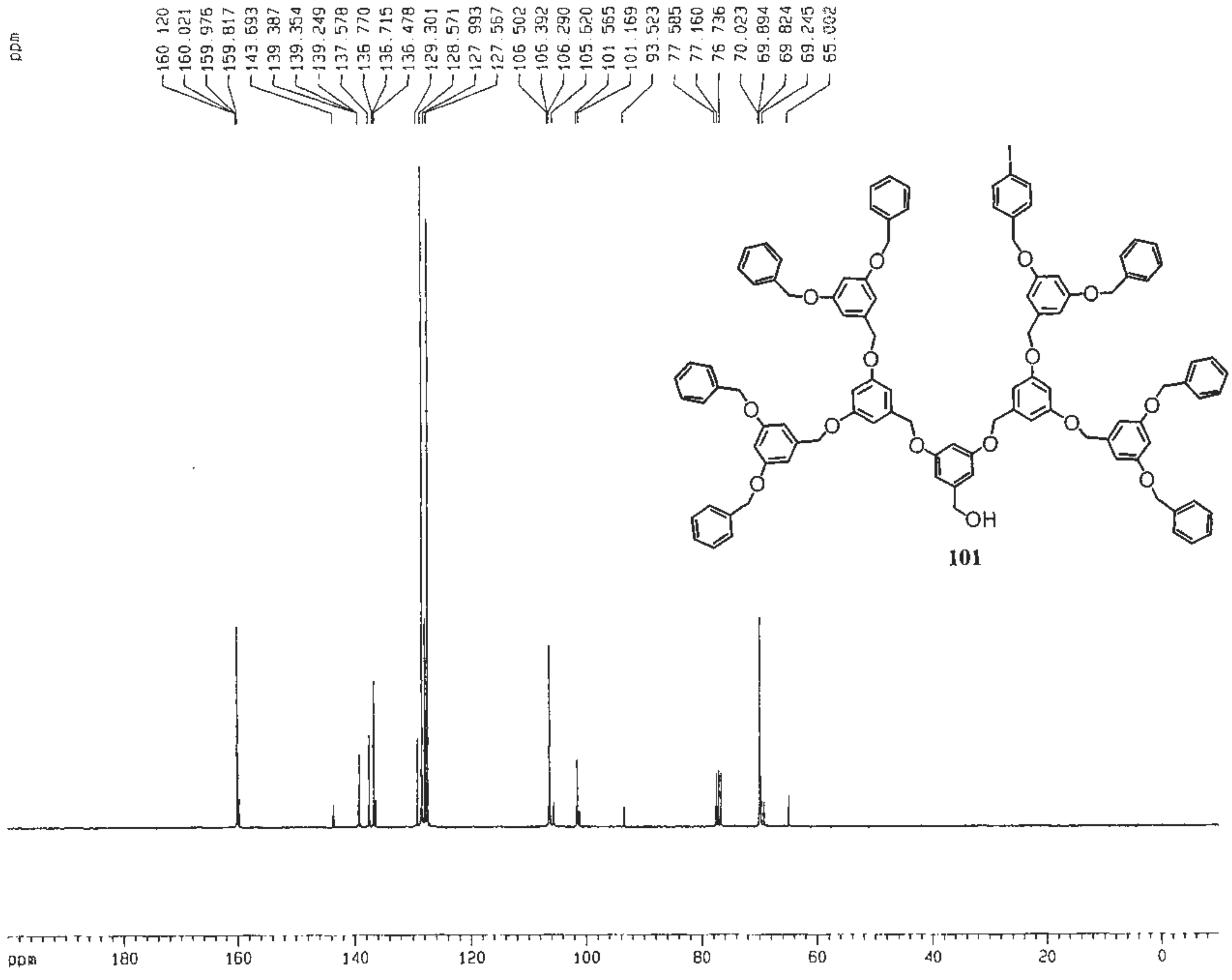

Current Data Parameters
NAME I-63-esterNC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070113
Time 0.56
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 10000
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz
F2 - Processing parameters
SI 65536
SF 75.4677473 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
CY 12.02 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.54545 ppm/cm
HZCM 720.37390 Hz/cm
14.423
61.269
69.412
70.013
70.080
70.172
76.737
77.160
77.584
93.583
101.697
101.763
106.365
106.471
106.578
106.985
108.541
127.643
128.087
128.668
129.376
132.560
136.587
136.806
136.864
137.714
138.983
139.009
139.282
139.391
159.768
159.943
160.130
160.176
160.253
166.290
ppm
O
CO2Et
0
20
40
60
80
100
120
140
160
180
ppm

100



ppm
7.658
7.424
7.418
7.411
7.392
7.366
7.340
7.330
7.321
7.308
7.260
6.677
6.670
6.657
6.650
6.586
6.575
6.567
6.529
6.522
5.013
4.956
4.941
4.580
1.699
1.563
OH
Integral
2.157
35.029
2.198
12.053
2.169
3.008
4.173
14.377
11.858
2.119
2.000
0.993
ppm
9
8
7
6
5
4
3
2
1
0
Current Data Parameters
NAME I-G3-OHN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070110
Time 13.20
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 4194.631 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 181
DW 119.200 usec
DE 6.00 usec
TE 0.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 4.50 usec
PL1 -2.00 dB
SFO1 300.1318000 MHz
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 11.96 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm

101



101

```
Current Data Parameters
NAME          1-G3-OHNC
EXPNO                 1
PROCNO                1

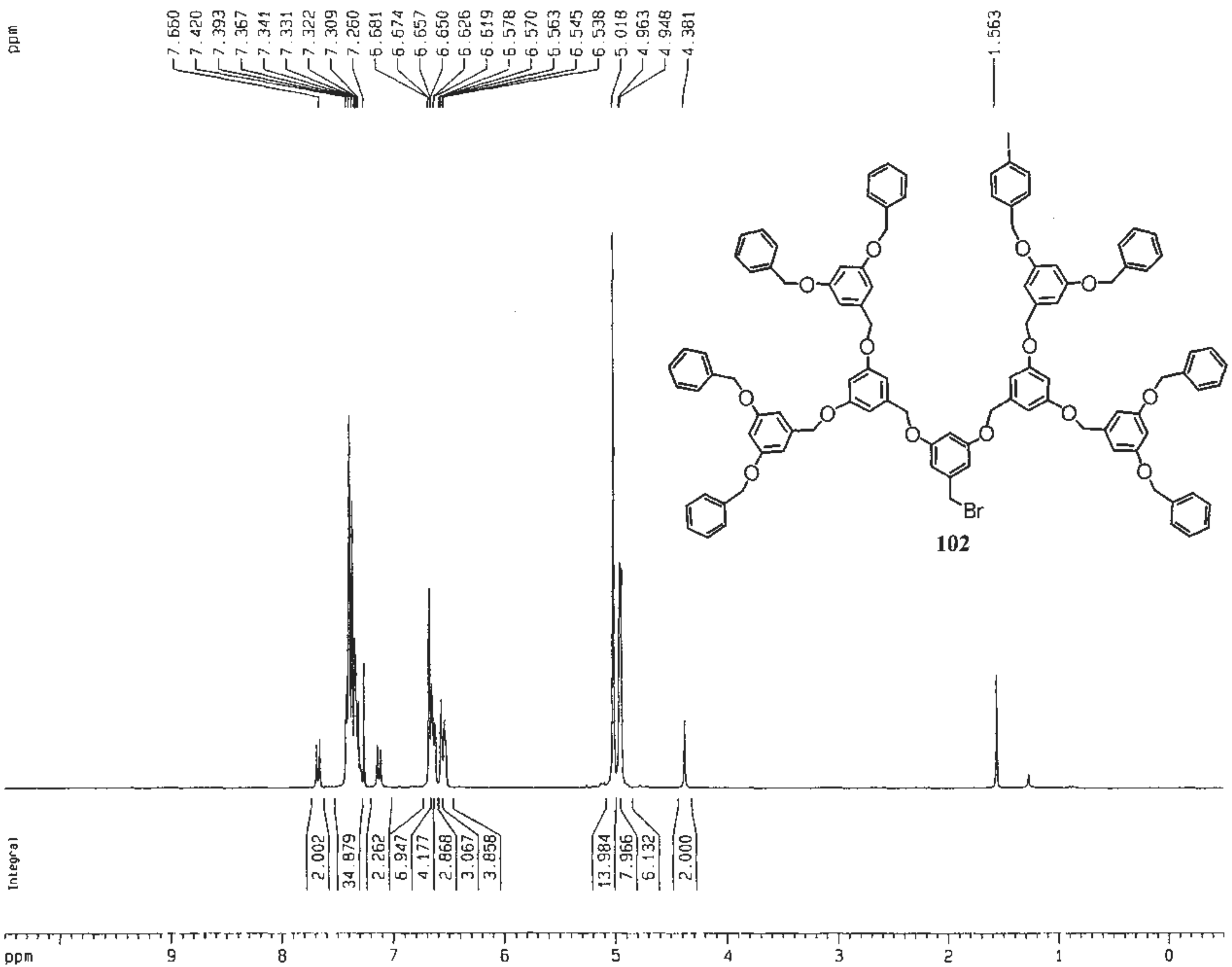
F2 - Acquisition Parameters
Date_          20070113
Time               0.33
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1 4451188 sec
RG                 8192
DW               22.050 usec
DE                 6 00 usec
TE                  0 0 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0 01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3 00 usec
PL1               -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75 4677613 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11 96 cm
F1P             200 000 ppm
F1             15093 55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37402 Hz/cm
```



102

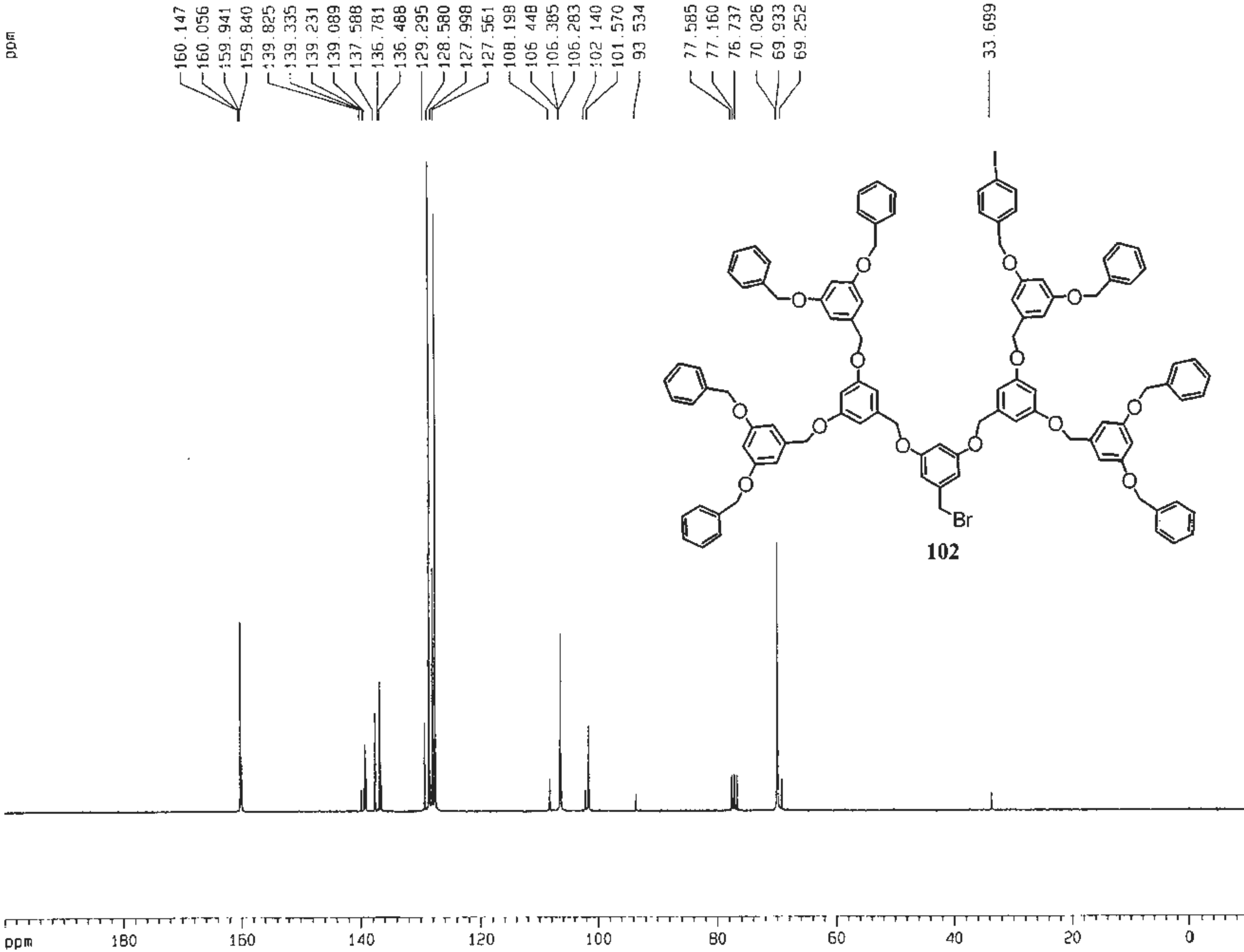
```
Current Data Parameters
NAME          I-G3-BrN
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20070119
Time              19.03
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                203.2
DW              138.825 usec
DE               198.32 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300063 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.01 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



102

```
Current Data Parameters
NAME          I-G3-BrNC
EXPNO                 1
PROCNO                1

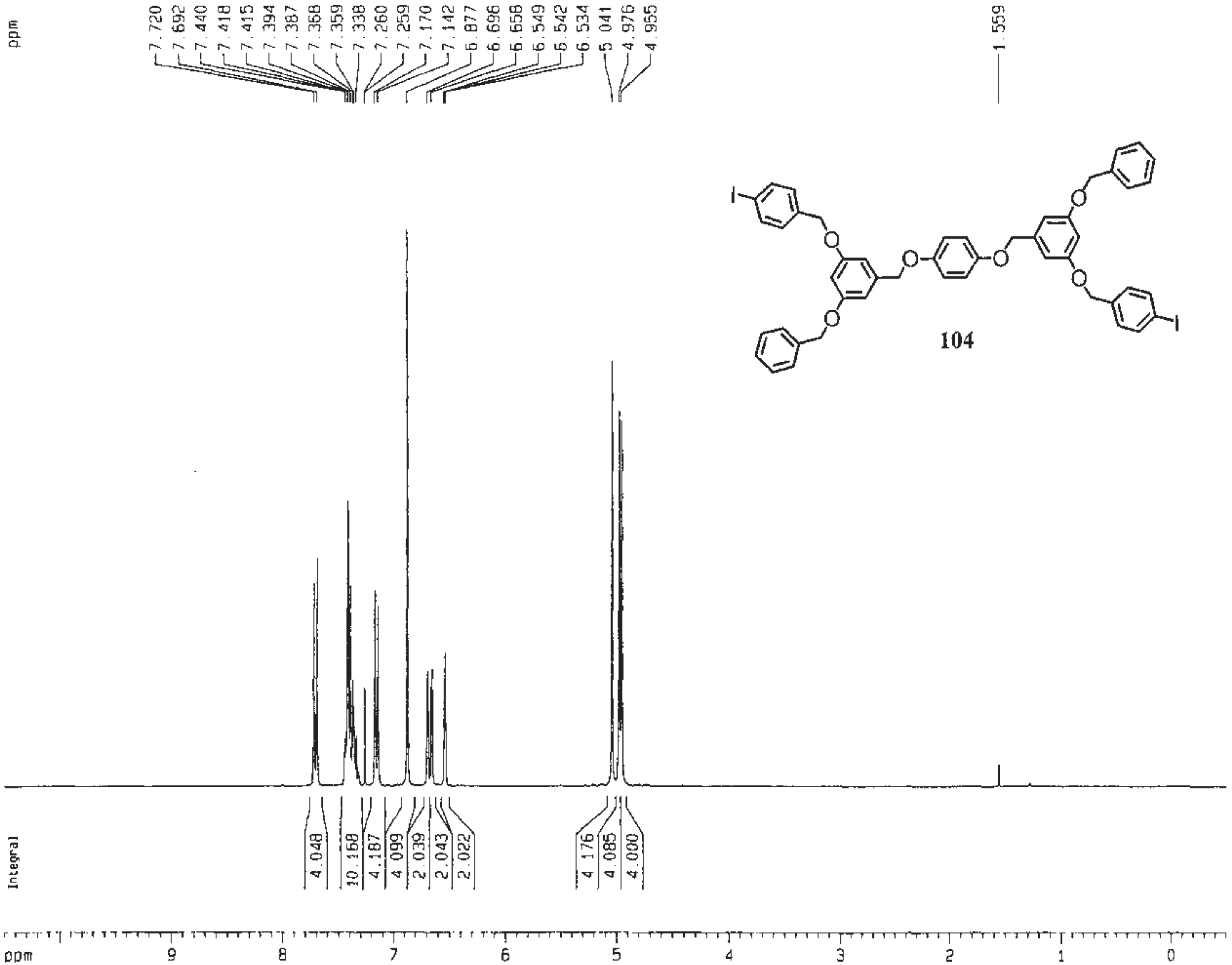
F2 - Acquisition Parameters
Date_          20070120
Time               0.50
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677619 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                23.00 cm
CY                11.99 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.13043 ppm/cm
HZCM          689.05347 Hz/cm
```



104

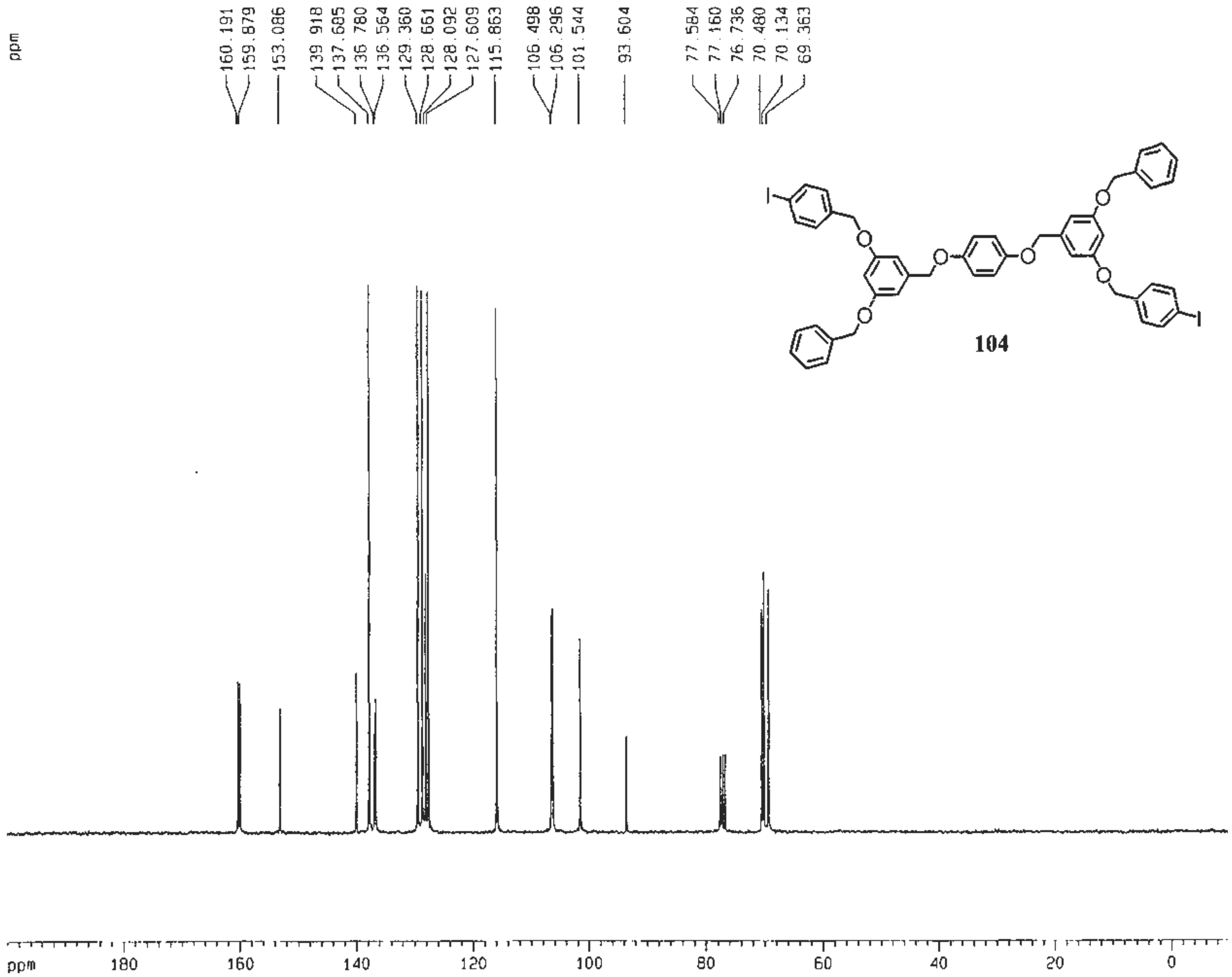
```
Current Data Parameters
NAME          I-G1-denN
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20060406
Time              12.32
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                  256
DW              138.825 usec
DE               198.32 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300060 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                 9.99 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



104

```
Current Data Parameters
NAME          I-G1-denNC
EXPNO                  1
PROCNO                 1

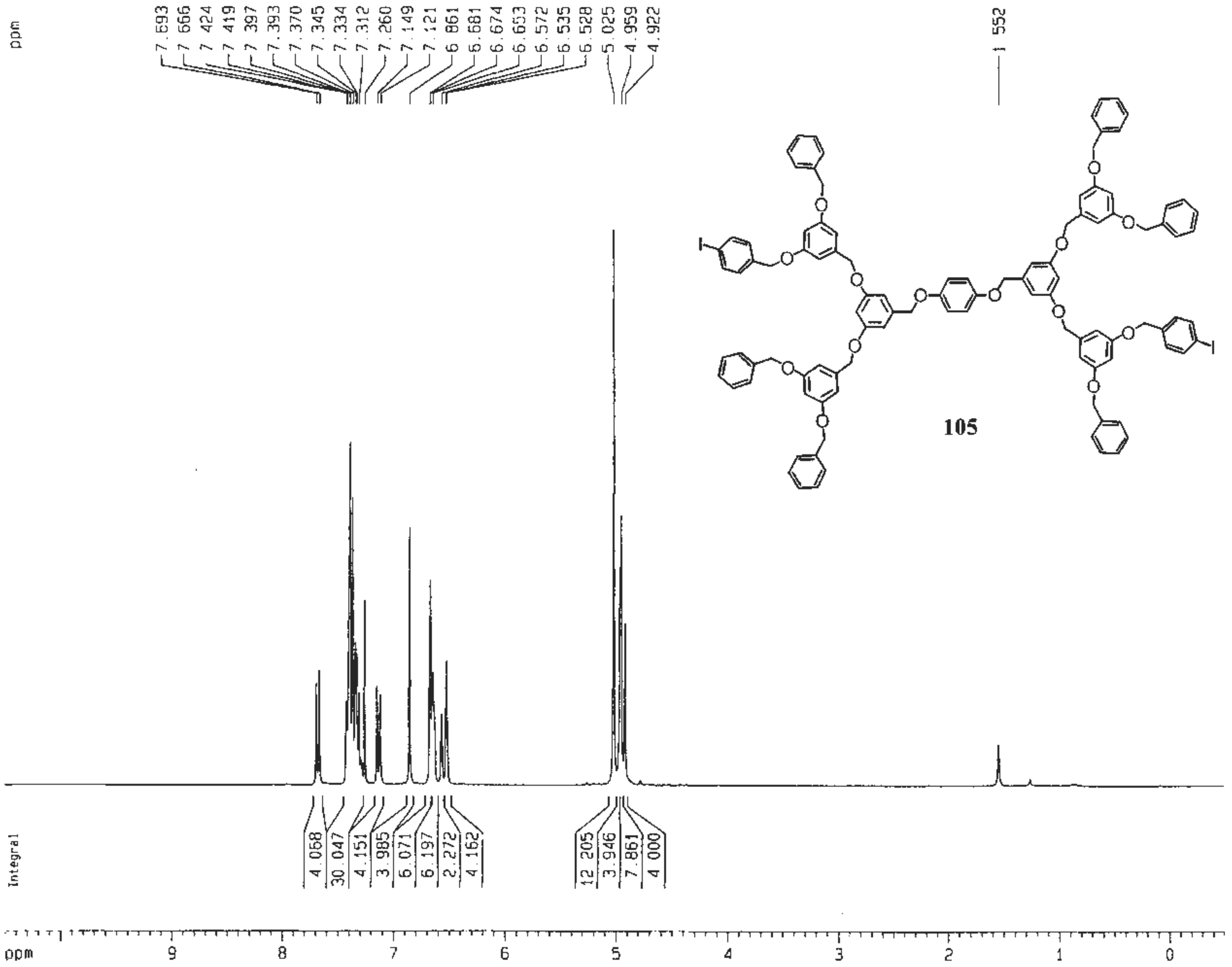
F2 - Acquisition Parameters
Date_           20060407
Time               22 26
INSTRUM           dpx300
PROBHD   5 mm B80 BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   445
DS                     0
SWH           22675 736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6 00 usec
TE                   0 0 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3 00 usec
PL1                -6.00 dB
SFO1          75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120 00 dB
PL12               19 00 dB
SFO2         300 1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677519 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22 00 cm
CY                  9.95 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754 68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```



105

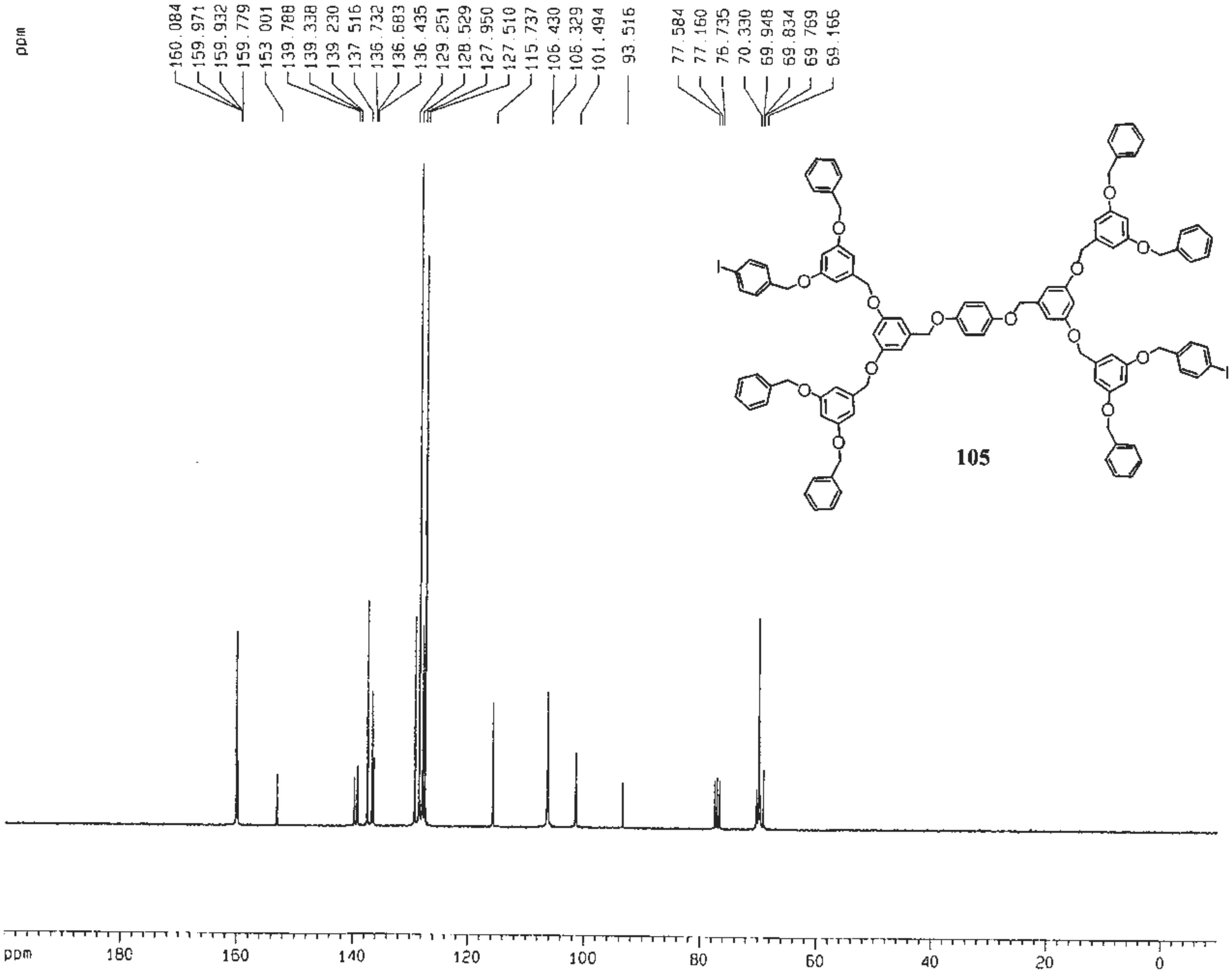
```
Current Data Parameters
NAME             I-62-denN
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070110
Time                 13 45
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                      32
DS                       0
SWH              4194.631 Hz
FIDRES           0.256020 Hz
AQ              1 9530228 sec
RG                     362
DW                 119.200 usec
DE                    6 00 usec
TE                     0 0 K
D1              5 00000000 sec
MCREST          0 00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    4.50 usec
PL1                  -2.00 dB
SFO1           300.1318000 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300062 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   10.00 cm
F1P                 10.500 ppm
F1                 3151.36 Hz
F2P                 -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.50000 ppm/cm
HZCM             150.06500 Hz/cm
```



105

```
Current Data Parameters
NAME          I-G2-denNC
EXPNO                  1
PROCNO                 1

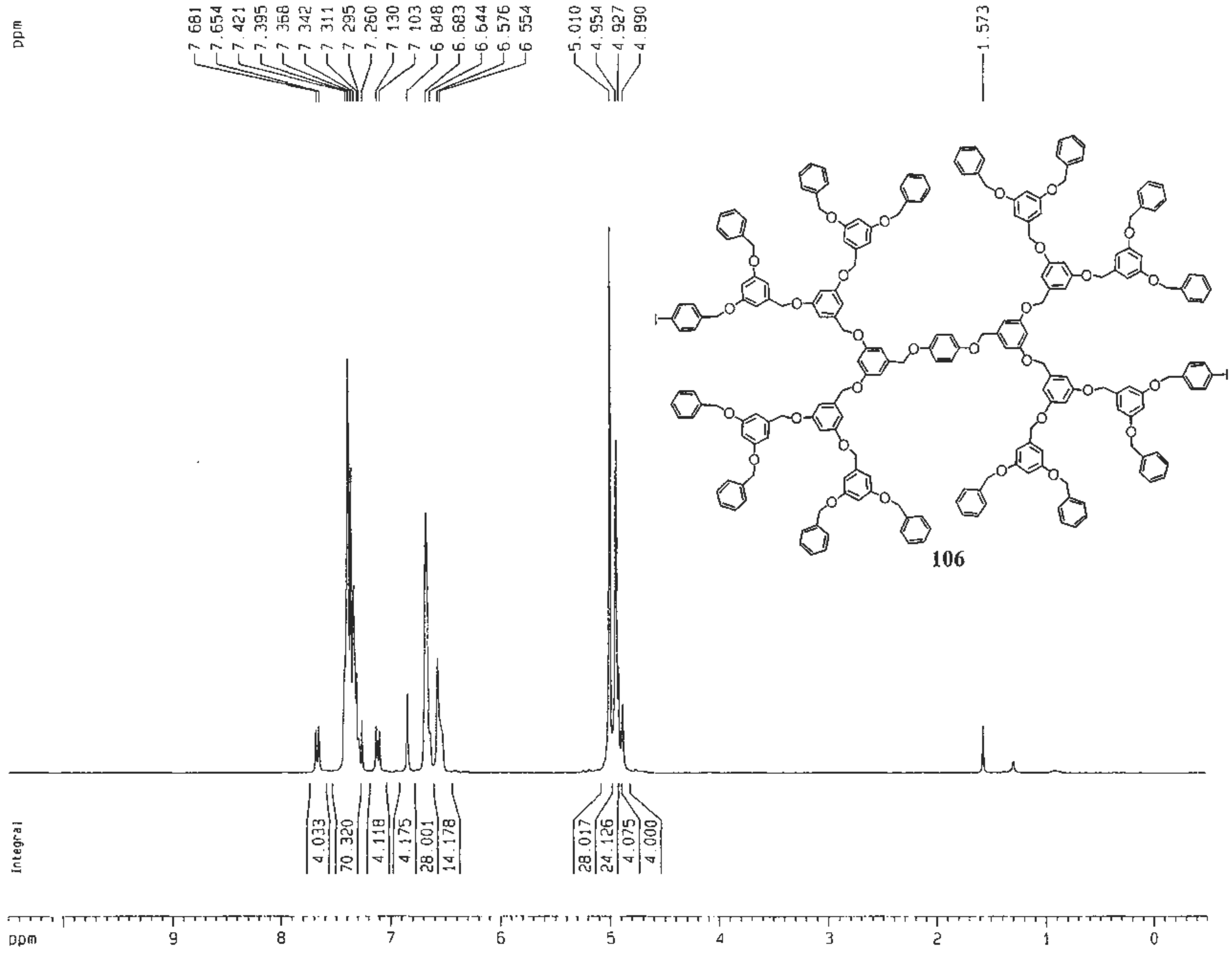
F2 - Acquisition Parameters
Date_           20070113
Time                0.14
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   352
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677706 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.99 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37415 Hz/cm
```



106

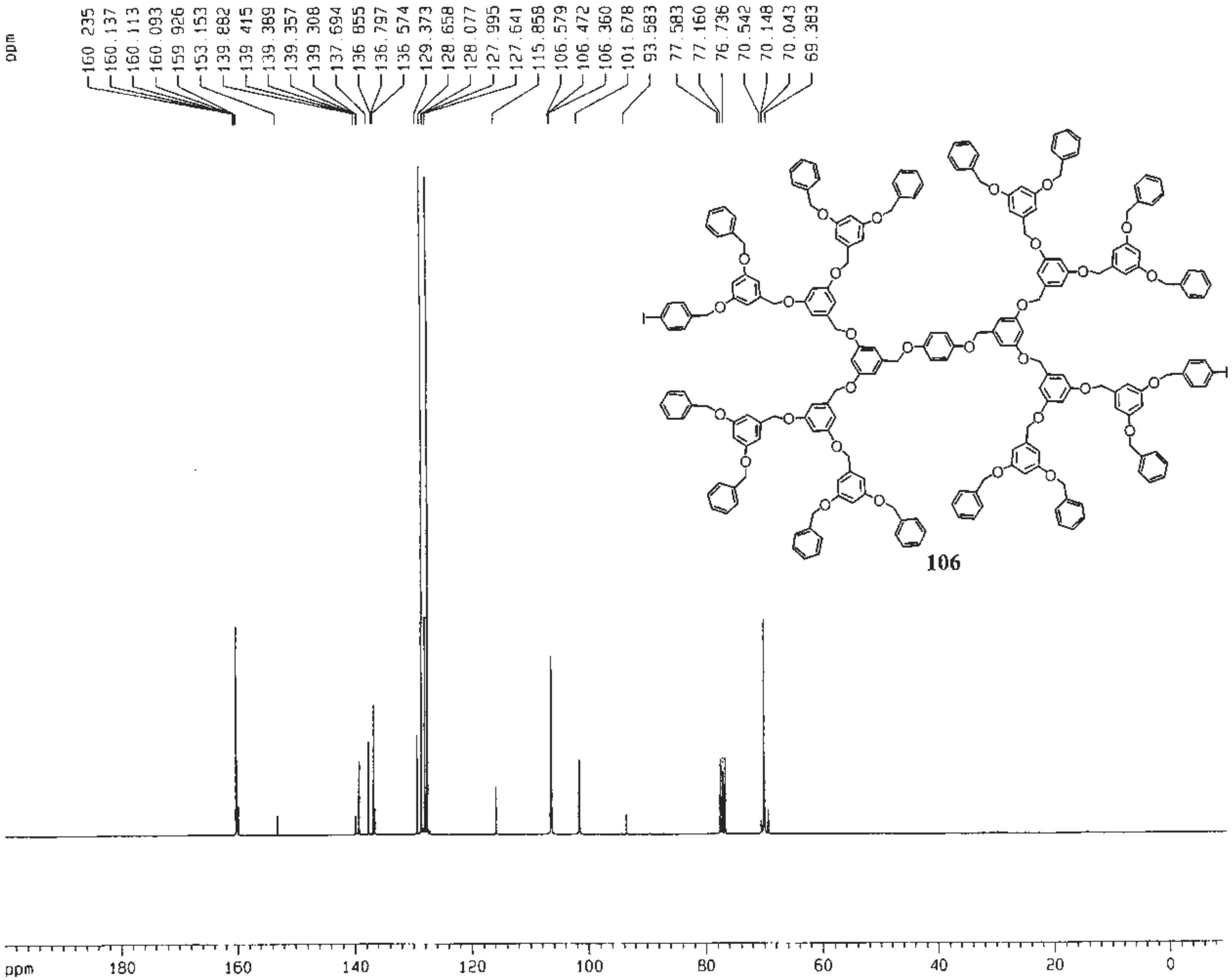
```
Current Data Parameters
NAME          I-G3-denN
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070213
Time              19.30
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   64
DS                    0
SWH            4202.563 Hz
FIDRES         0.256504 Hz
AQ            1.9493364 sec
RG                101.6
DW              118.975 usec
DE               169.96 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1        300.1316000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300061 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                 9.99 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



106

```
Current Data Parameters
NAME          I-G3-denNC
EXPNO                  1
PROCNO                 1

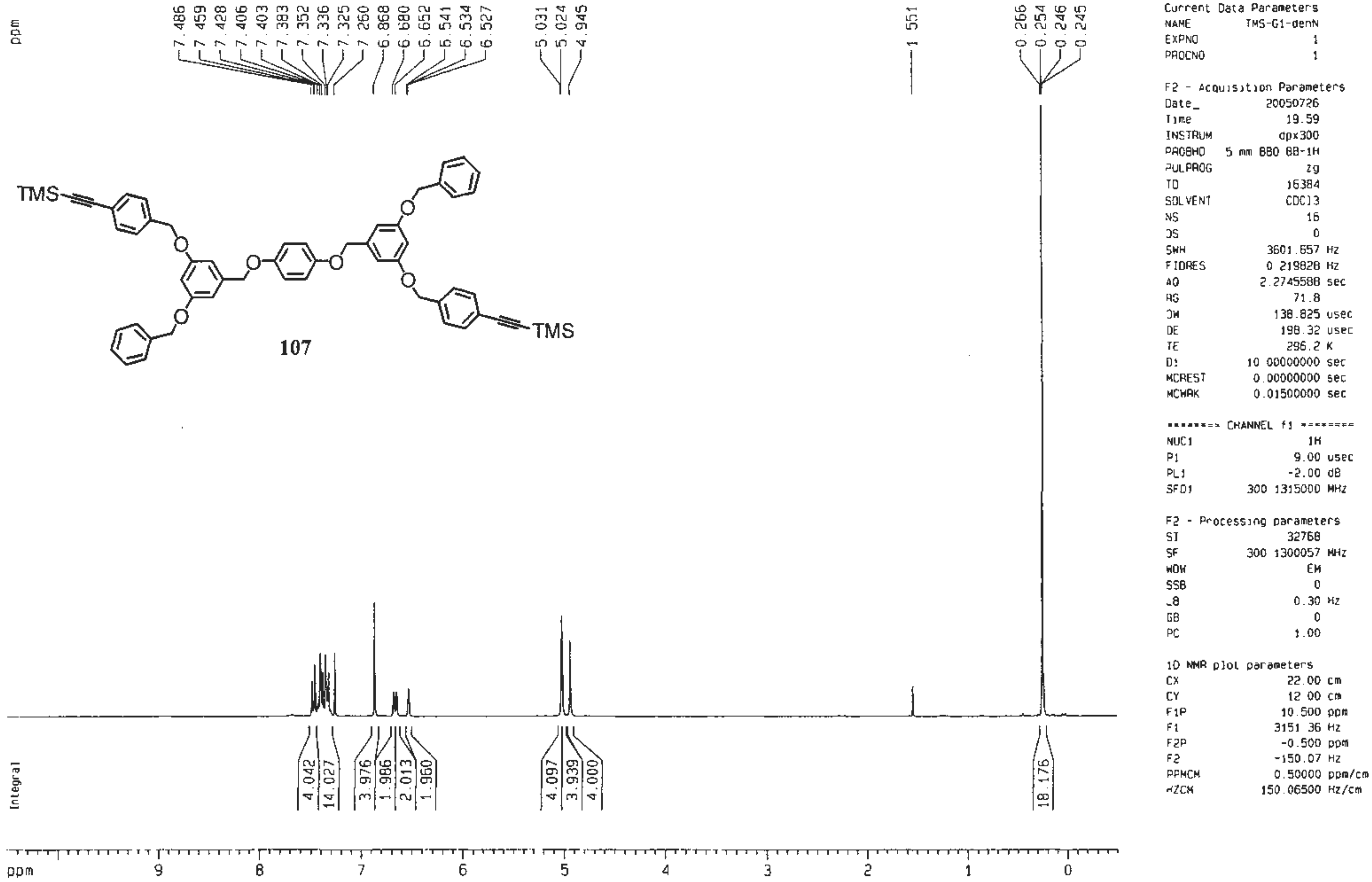
F2 - Acquisition Parameters
Date_           20070226
Time               21.58
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                 14600
DS                     0
SWH           22675 736 Hz
FIDRES         0.346004 Hz
AQ            1 4451188 sec
RG                  8192
DW                22 050 usec
DE                  6 00 usec
TE                   0.0 K
D1            1 00000000 sec
d11           0.03000000 sec
MCREST        0 00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3 00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19 00 dB
SFO2        300 1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677481 MHz
WDW                   EM
SSB                    0
LB                  0 50 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.98 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```

ppm
7.486
7.459
7.428
7.406
7.403
7.383
7.352
7.336
7.325
7.260
6.868
6.680
6.652
6.541
6.534
6.527
5.031
5.024
4.945
1.551
0.266
0.254
0.246
0.245
TMS
TMS
O
Integral
4.042
14.027
3.976
1.986
2.013
1.960
4.097
3.939
4.000
18.176
ppm
9
8
7
6
5
4
3
2
1
0
Current Data Parameters
NAME TMS-G1-denN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050726
Time 19.59
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 3601.657 Hz
FIDRES 0.219828 Hz
AQ 2.2745588 sec
RG 71.8
DW 138.825 usec
DE 198.32 usec
TE 296.2 K
D1 10.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -2.00 dB
SFO1 300.1315000 MHz
F2 - Processing parameters
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm

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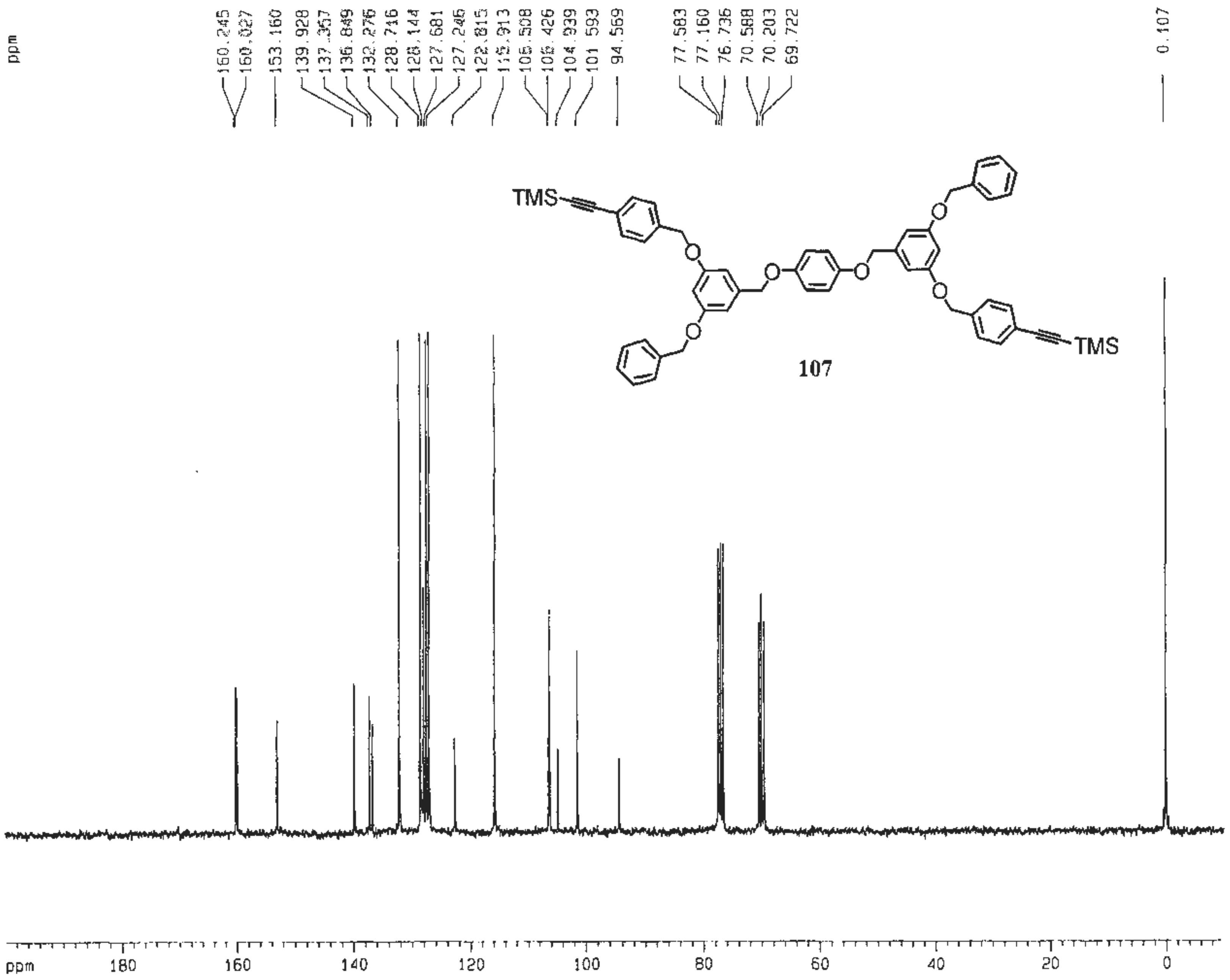

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```
Current Data Parameters
NAME        TMS-G1-denNC
EXPNO                  1
PROCNO                 1

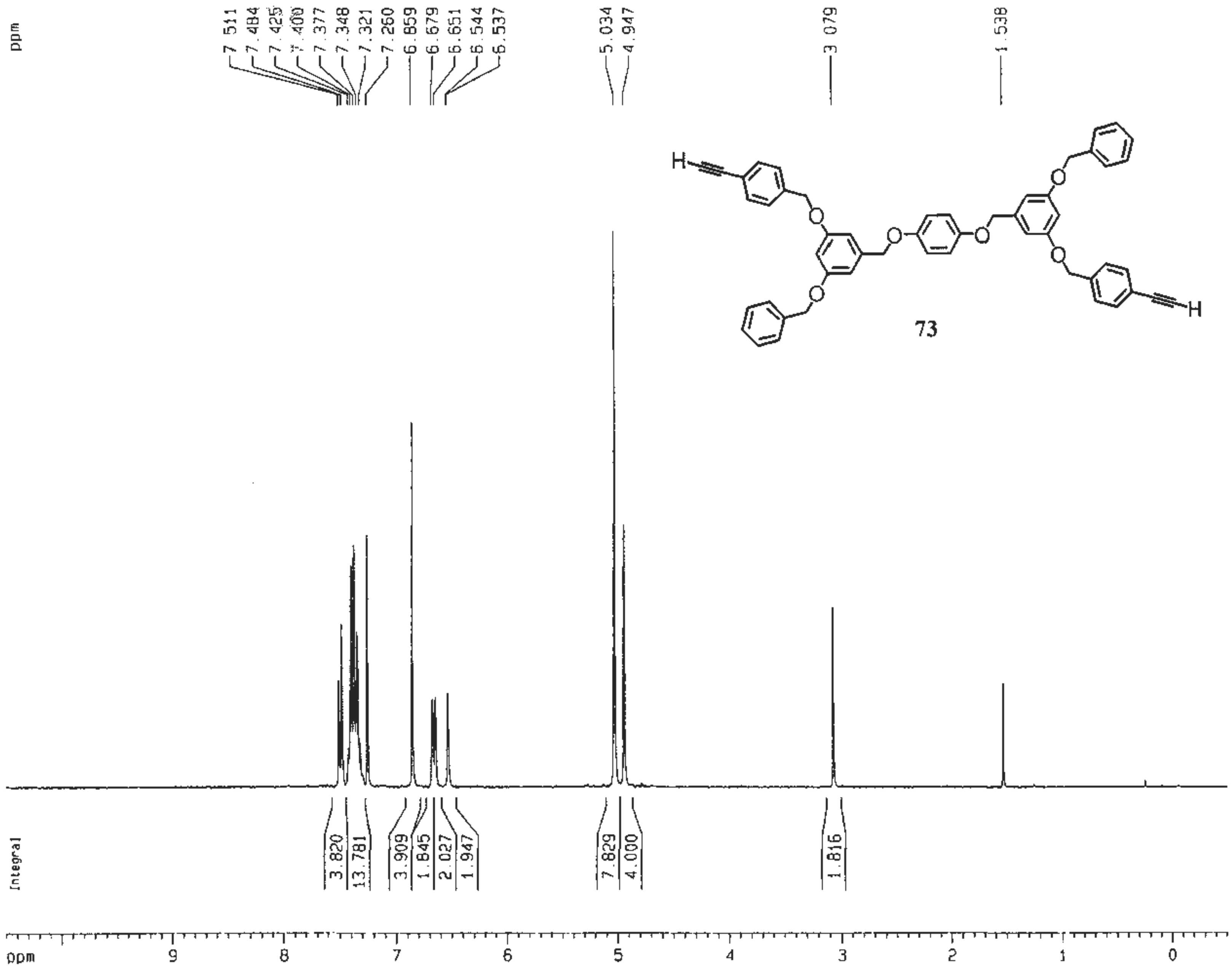
F2 - Acquisition Parameters
Date_           20050726
Time               20.35
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   558
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 297.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677431 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 10.02 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37384 Hz/cm
```



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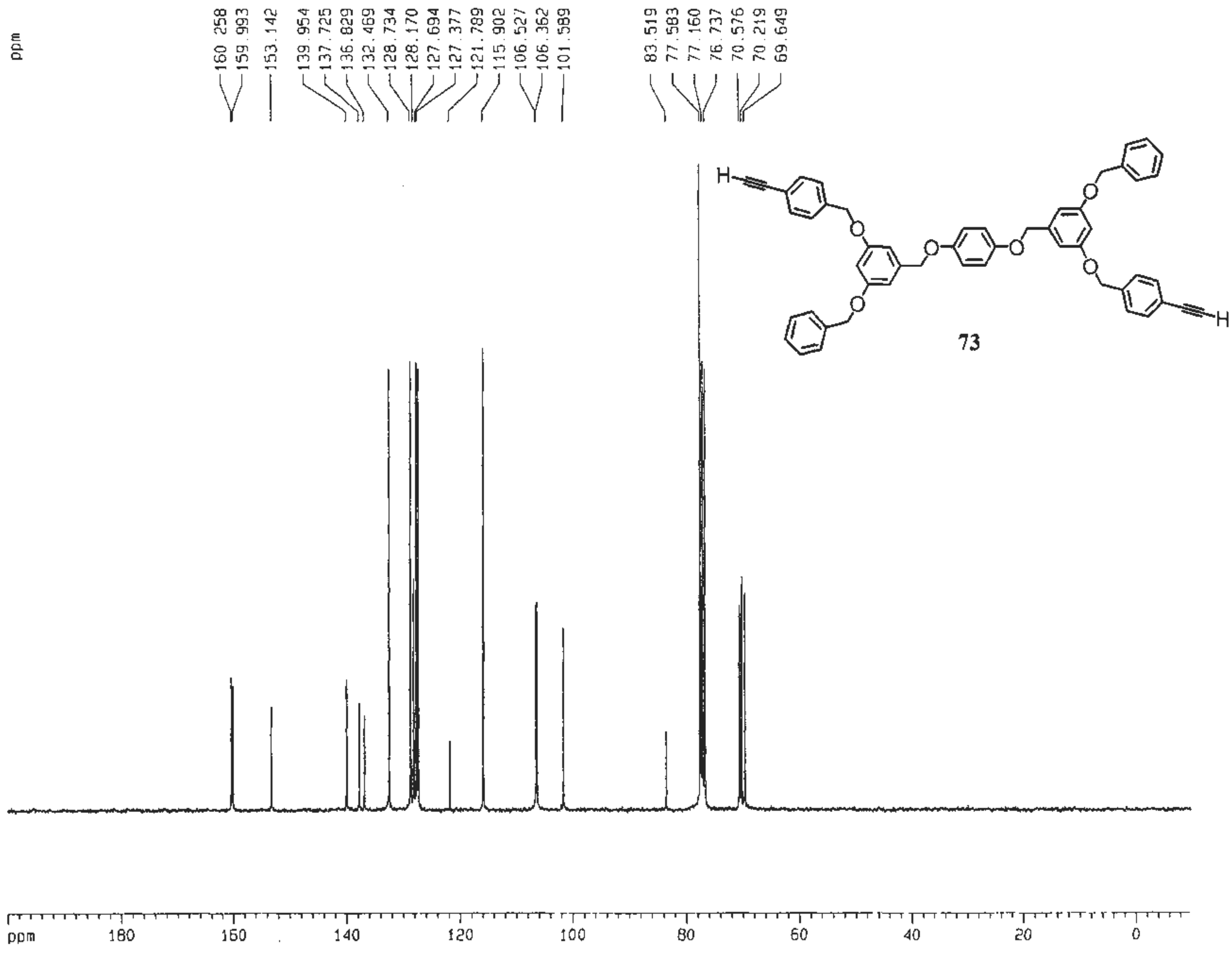
```
Current Data Parameters
NAME       HCC-G1-denN2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081101
Time              16.17
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   12
DS                    0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AQ           2 2745588 sec
RG                912.3
DW              138.825 usec
DE               198.32 usec
TE                298.2 K
D1           5.00000000 sec
MCREST       0 00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 4.50 usec
PL1               -2.00 dB
SF01  .    300.1315000 MHz

F2 - Processing parameters
SI                16384
SF          300.1300063 MHz
WDW                  EM
SSB                   0
LB                 0 30 Hz
GB                    0
PC                 1 00 `

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



73

```
Current Data Parameters
NAME        HCC-G1-denNC
EXPNO                  1
PROCNO                 1

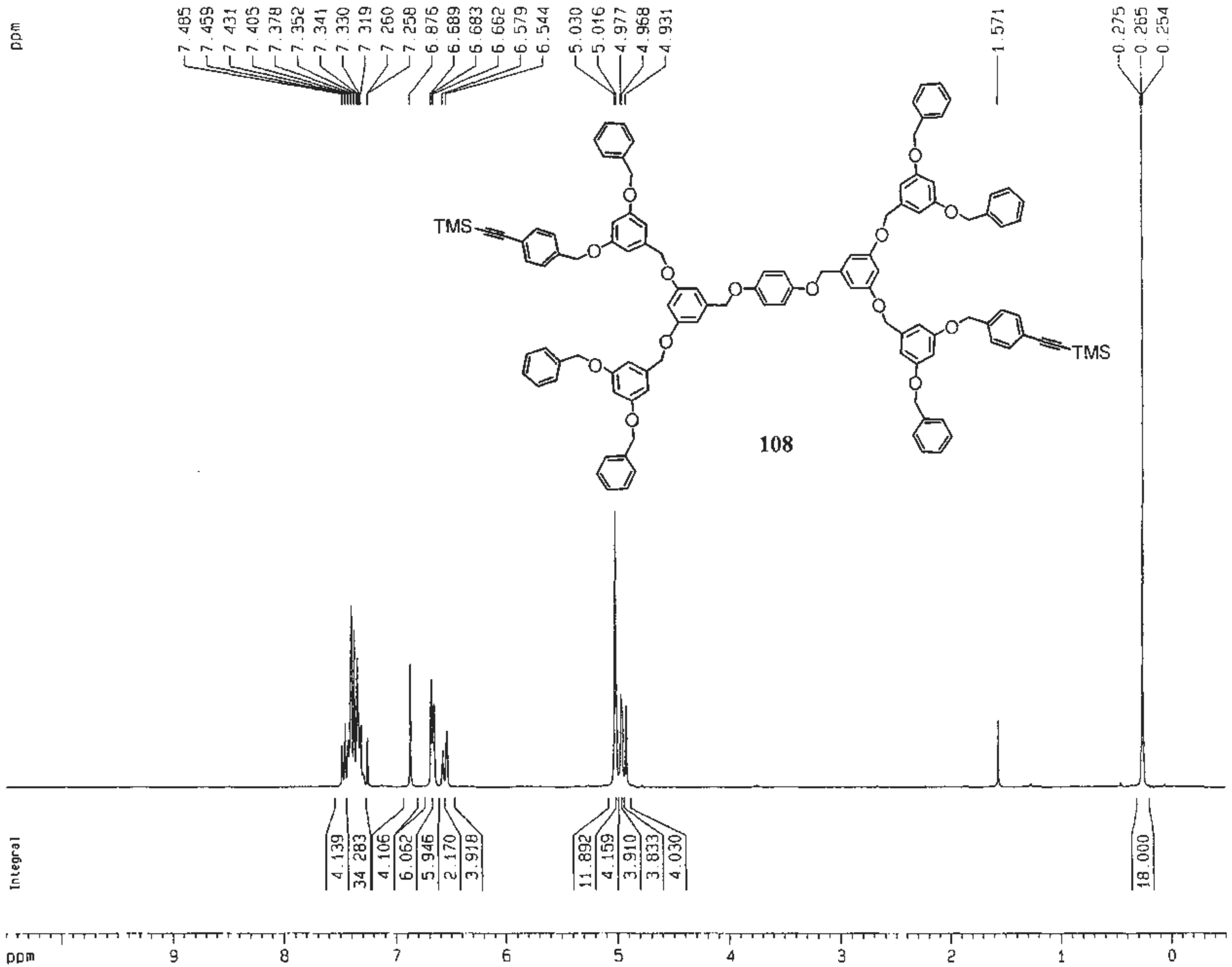
F2 - Acquisition Parameters
Date_           20080905
Time               18.54
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  4912
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 295.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677418 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.00 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37384 Hz/cm
```



108

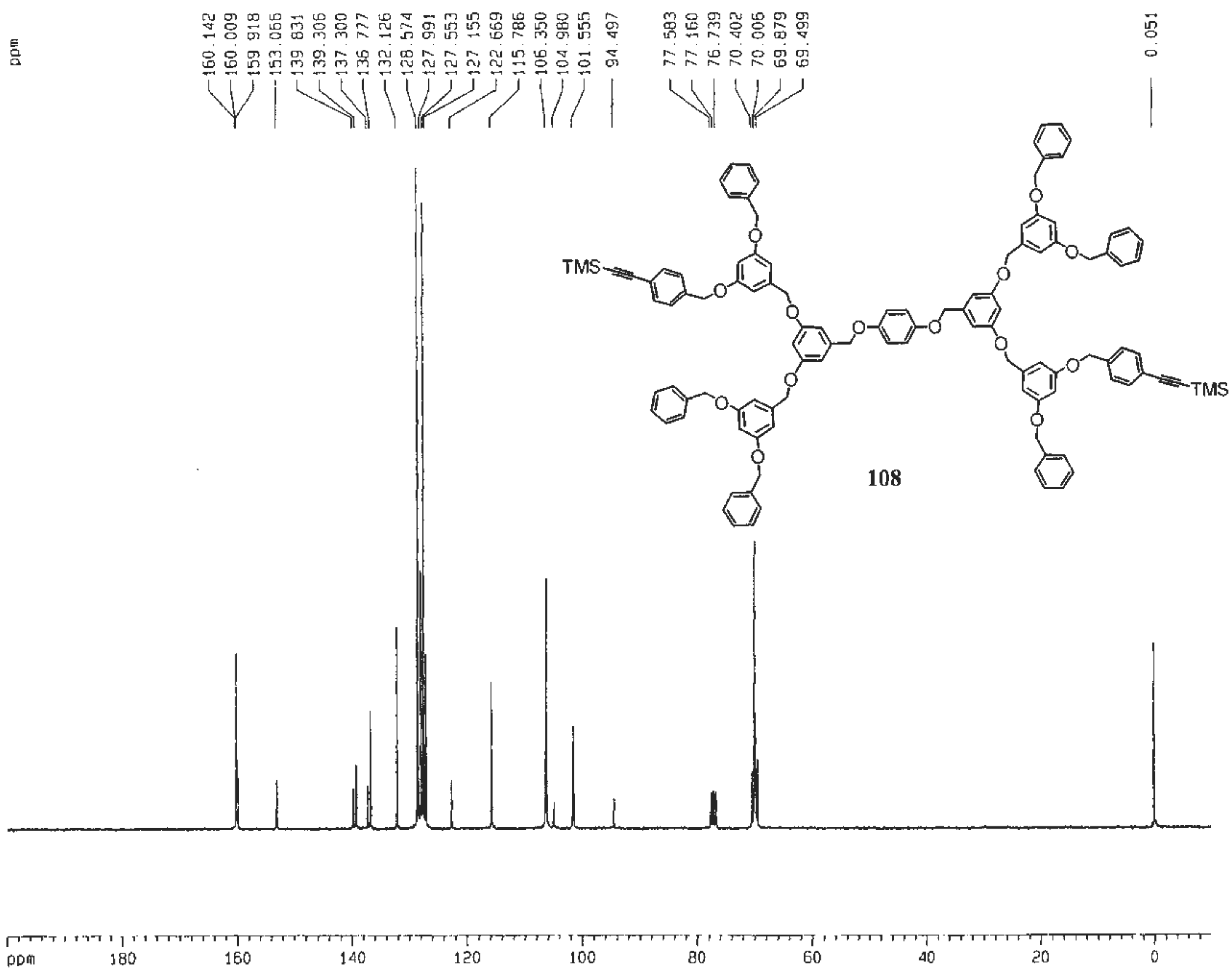
```
Current Data Parameters
NAME          TMS-G2-denN
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070120
Time                 0.12
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              4194.631 Hz
FIDRES           0.256020 Hz
AQ              1.9530228 sec
RG                  161.3
DW                119.200 usec
DE                   6.00 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   4.50 usec
PL1                 -2.00 dB
SFO1          300.1318000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300065 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  12.42 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



108

```
Current Data Parameters
NAME        TMS-G2-denNC
EXPNO                  1
PROCNO                 1

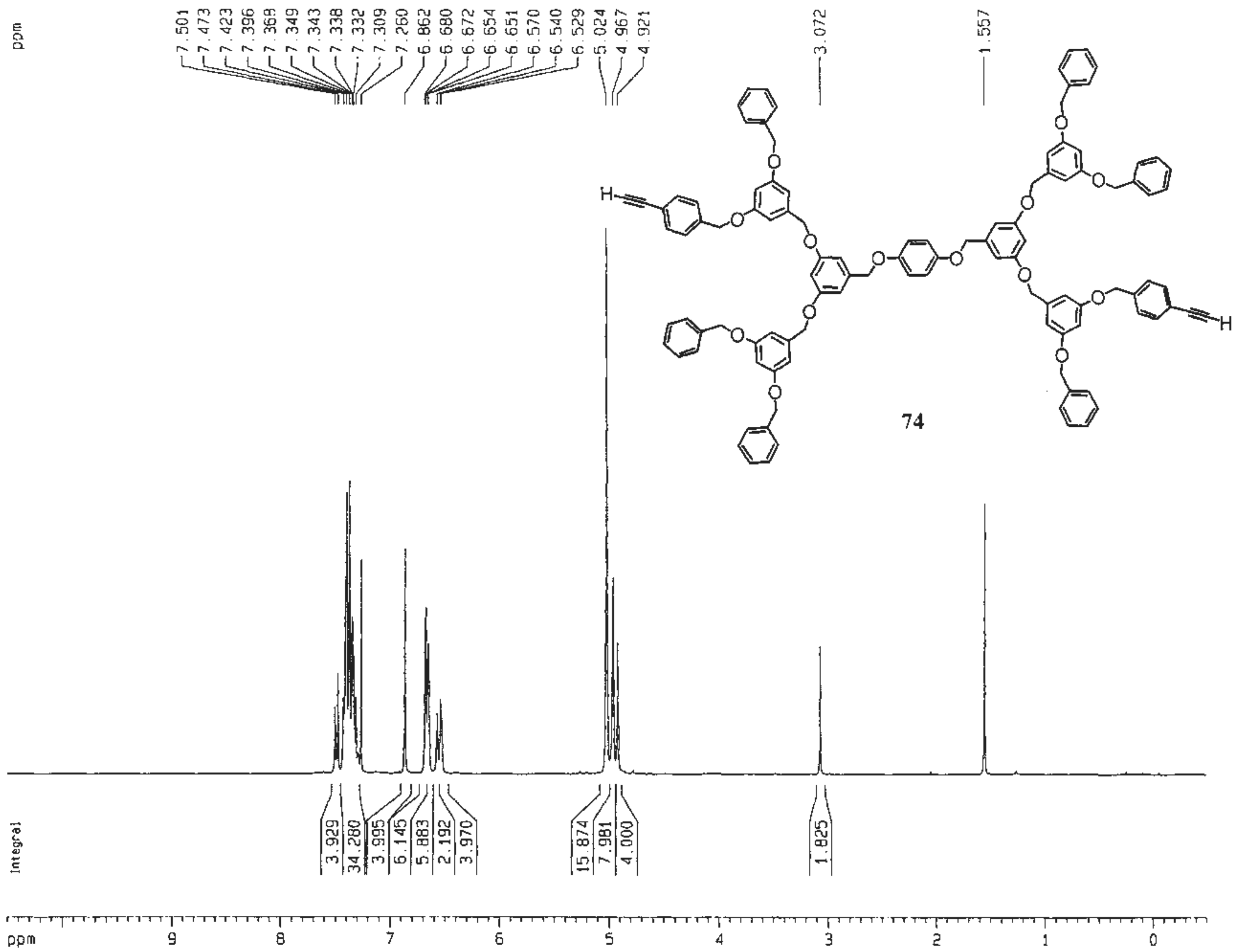
F2 - Acquisition Parameters
Date_           20070120
Time                0.26
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   600
DS                     0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                  8192
DW              22.050 usec
DE                6.00 usec
TE                 0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677631 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.01 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM          720.37408 Hz/cm
```



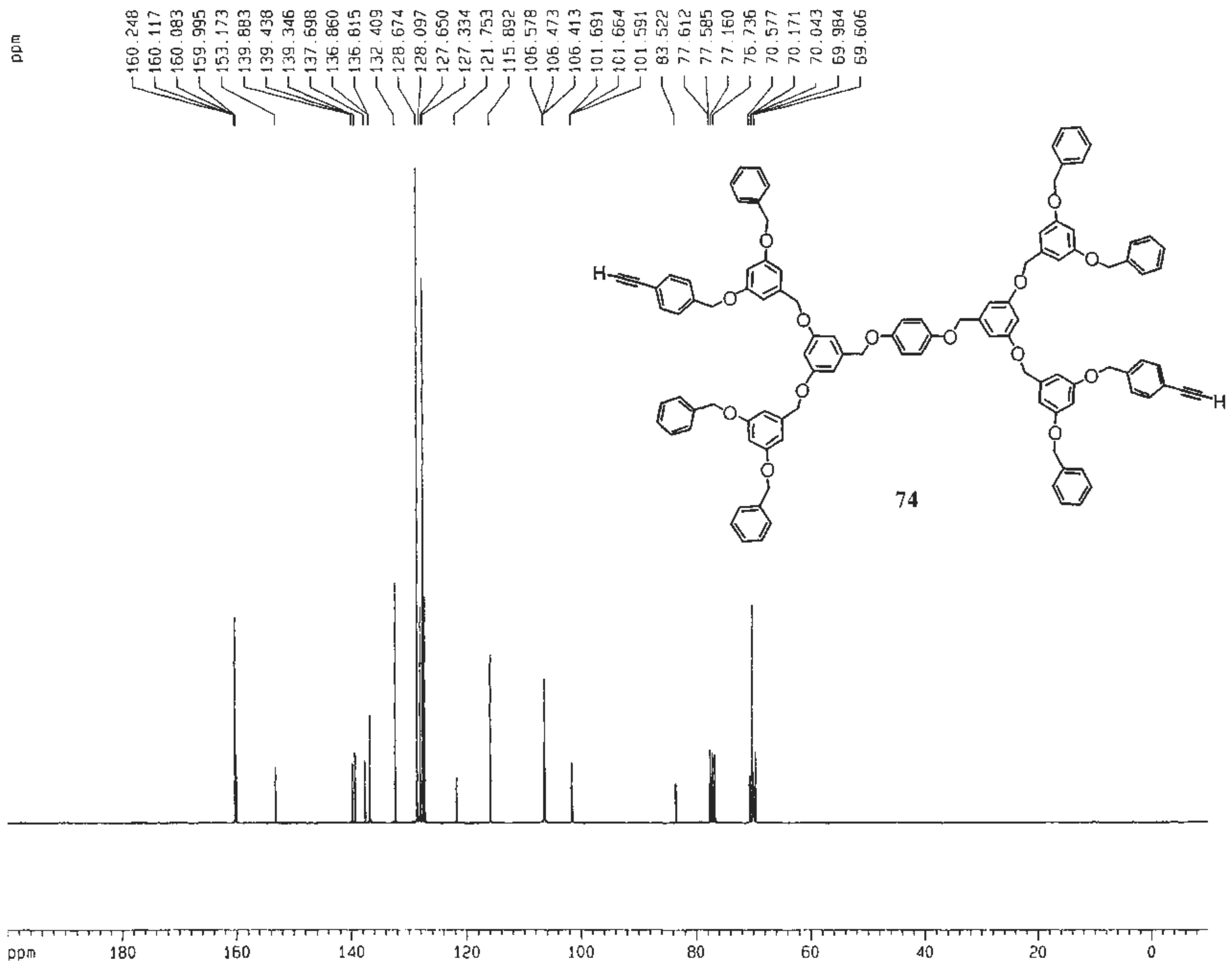
```
Current Data Parameters
NAME         HCC-G2-denN
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070210
Time                0.29
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            4202.563 Hz
FIDRES         0.128252 Hz
AQ            3 8986228 sec
RG                   362
DW              118.975 usec
DE               169.96 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0 01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  5 00 usec
PL1                -2.00 dB
SFO1   .    300.1318000 MHz

F2 - Processing parameters
SI                 32768
SF           300 1300065 MHz
WDW                   EM
SSB                    0
LB                  0 30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  9.99 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150 06500 Hz/cm
```



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```
Current Data Parameters
NAME        HCC-G2-denNC
EXPNO                  1
PROCNO                 1

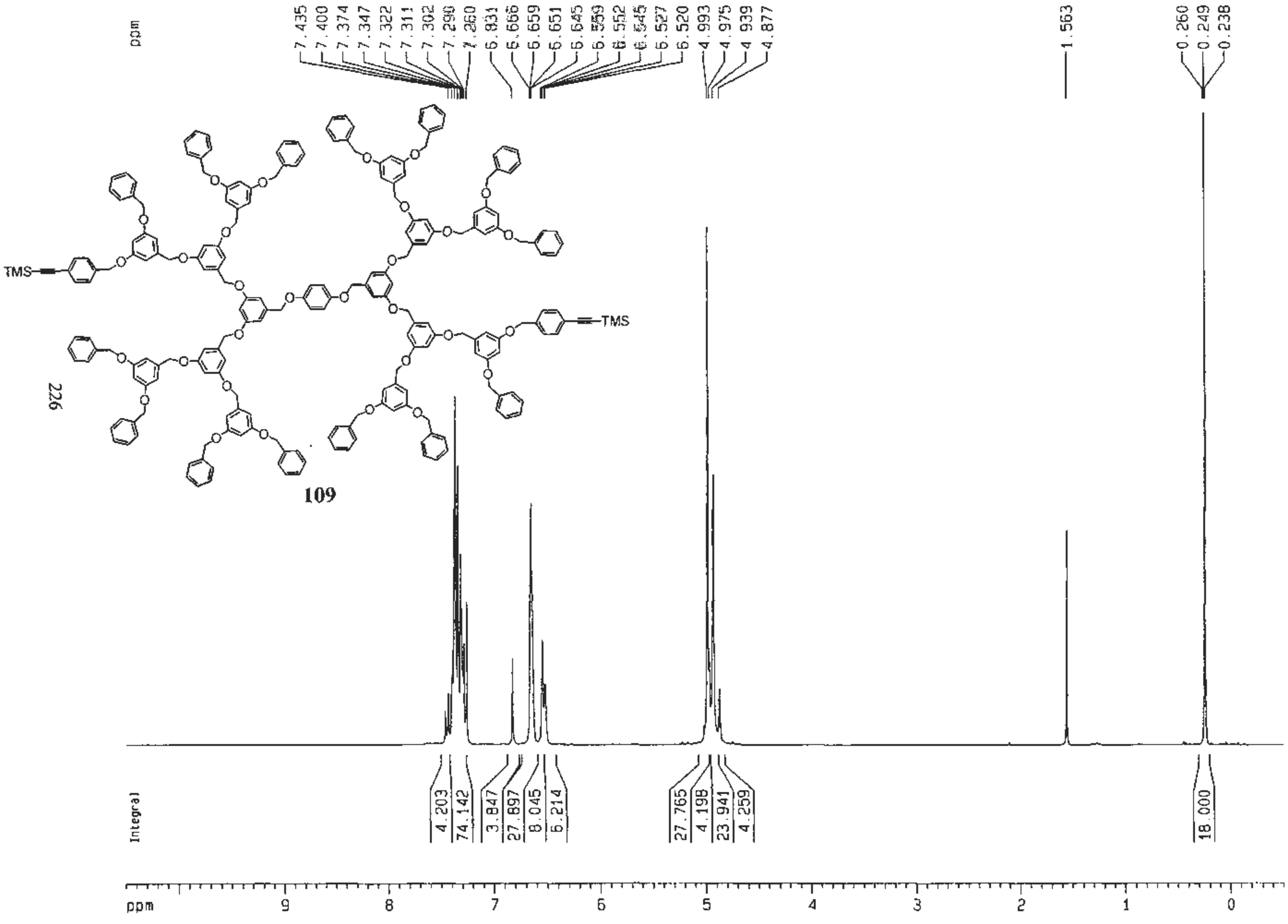
F2 - Acquisition Parameters
Date_           20070216
Time               22.09
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                 15000
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677480 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.97 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```



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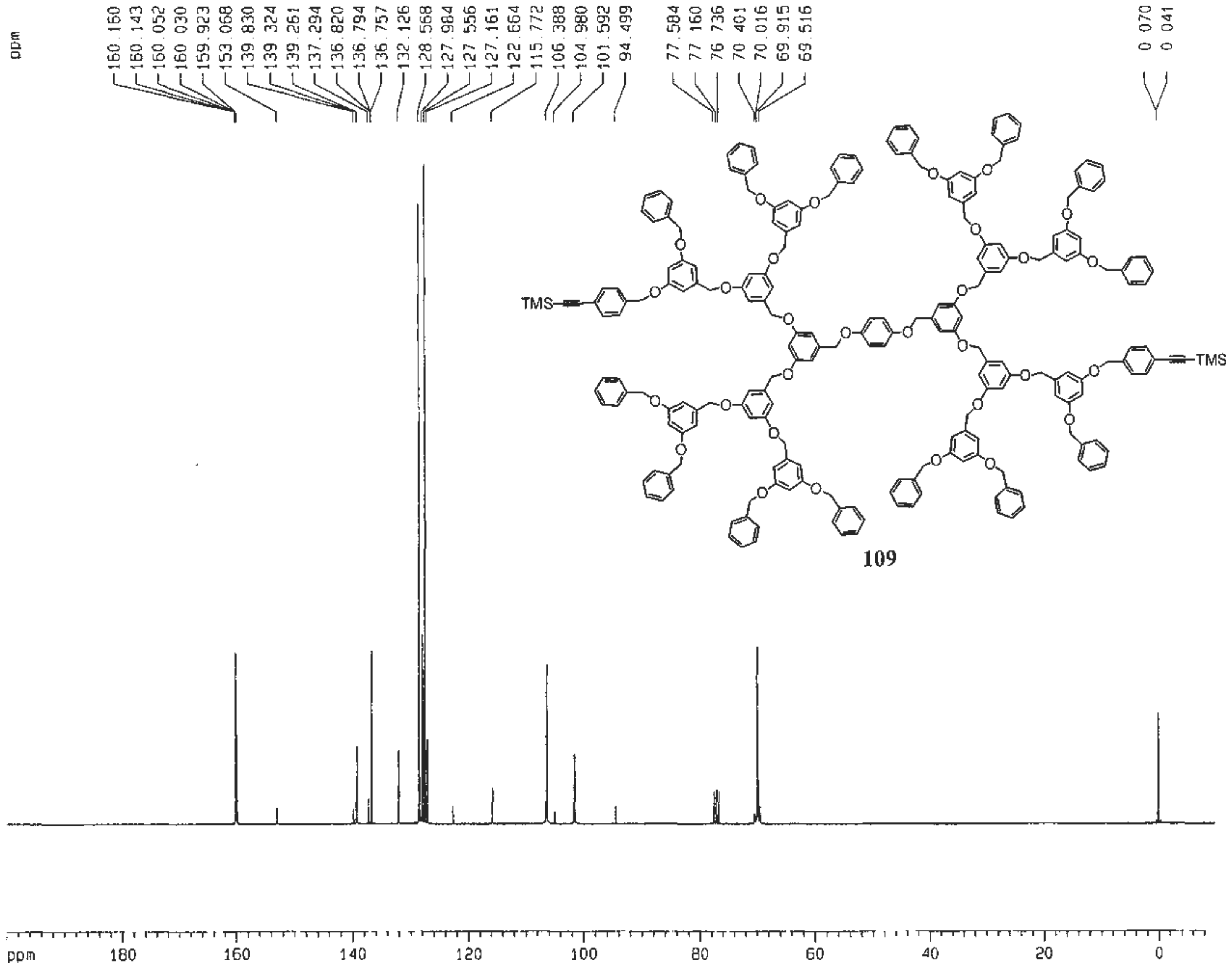
```
Current Data Parameters
NAME        TMS-G3-denN
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070208
Time              12.51
INSTRUM          dpx300
PROBHD   5 mm BB0 BB-1H
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    0
SWH            4202.563 Hz
FIDRES         0.128252 Hz
AQ            3.8986228 sec
RG                  256
DW              118.975 usec
DE               169.96 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1        300.1318000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300067 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                11.97 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



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```
Current Data Parameters
NAME        TMS-G3-denNC
EXPNO                  1
PROCNO                 1

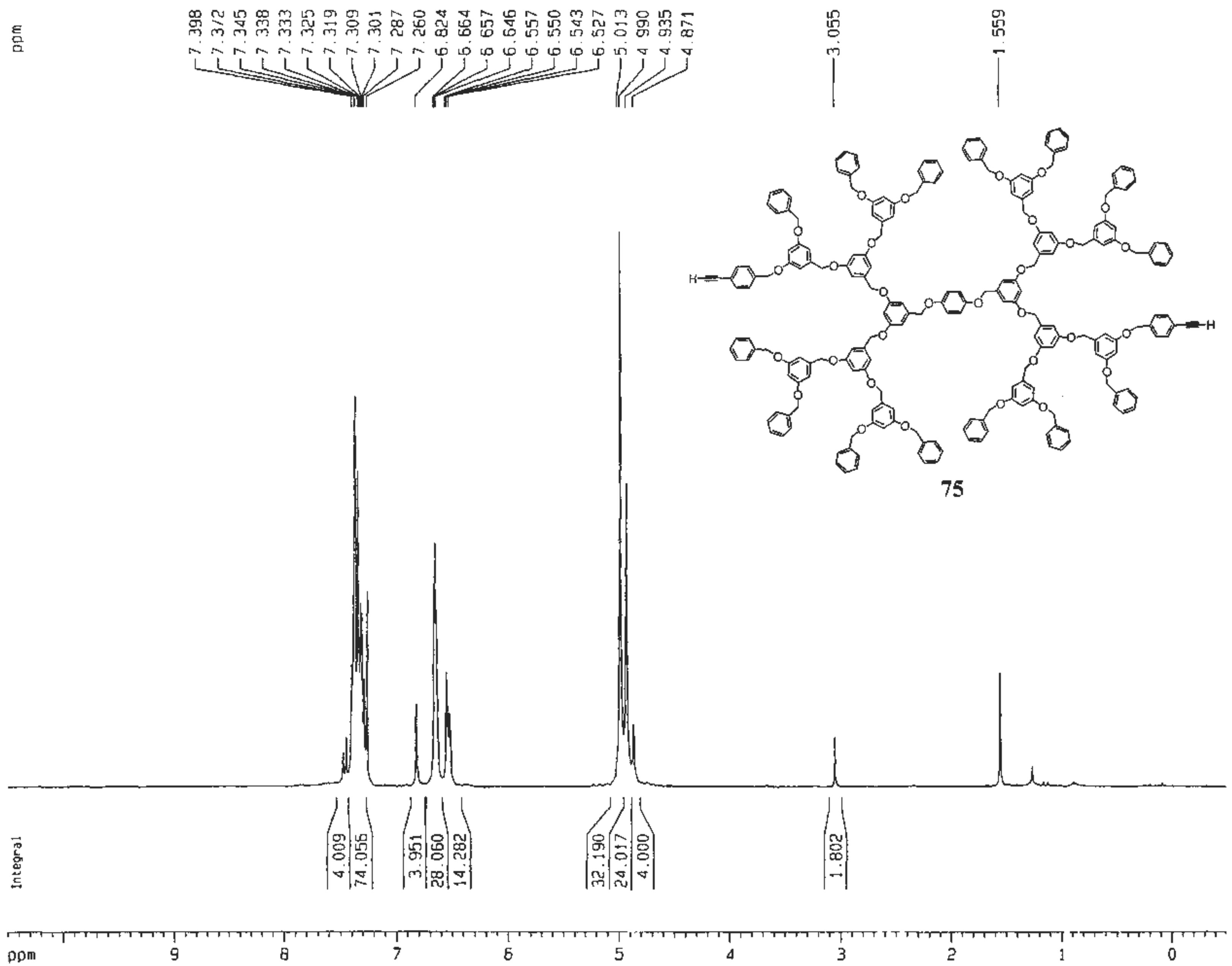
F2 - Acquisition Parameters
Date_           20070210
Time                1.06
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                 10000
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677618 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.99 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37402 Hz/cm
```



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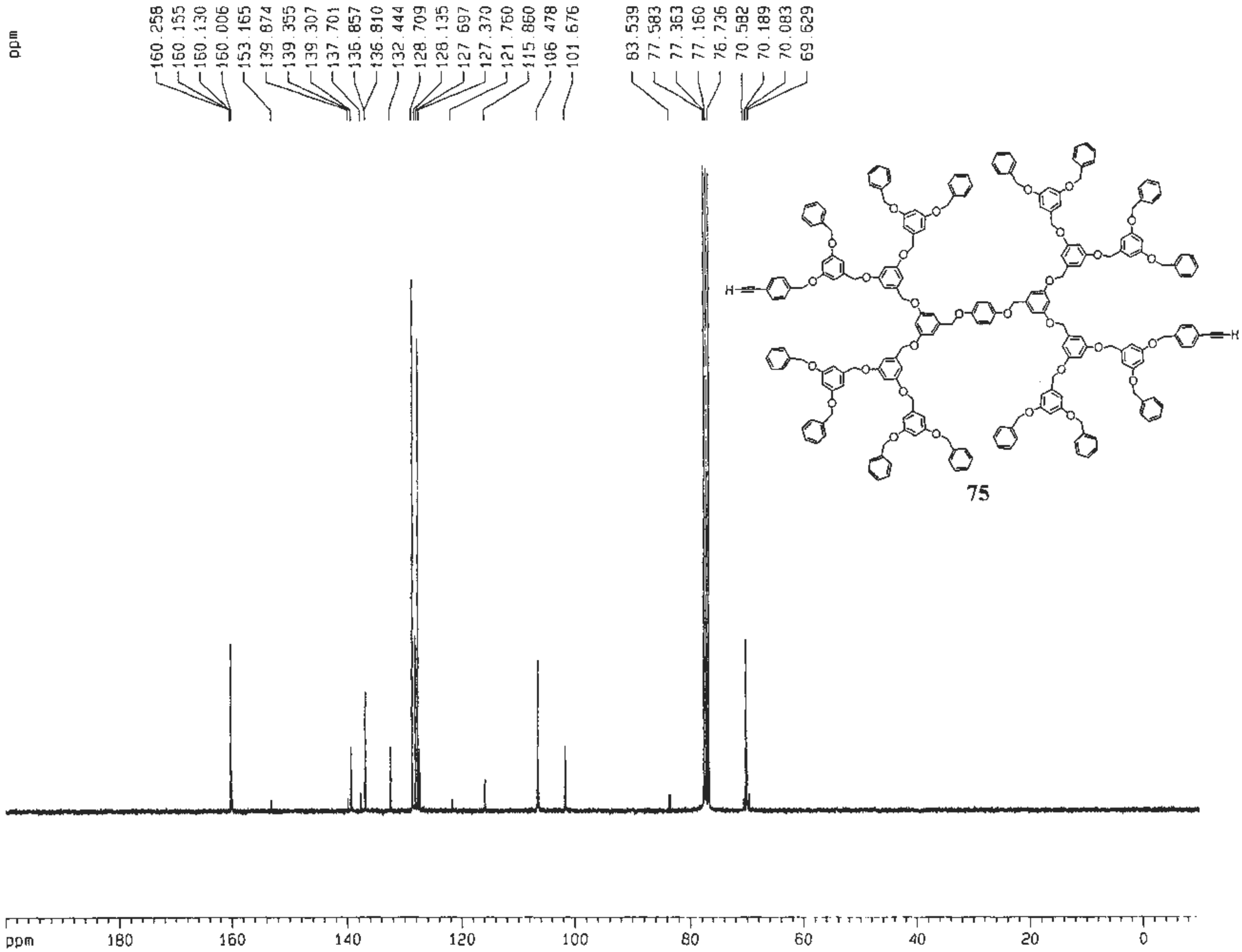
```
Current Data Parameters
NAME       HCC-G3-denN
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20080609
Time              0.35
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                  32
DS                   0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AQ           2.2745588 sec
RG                 256
DW             138.825 usec
DE              198.32 usec
TE               295.2 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                5.00 usec
PL1              -2.00 dB
SFO1     - 300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300063 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY               10.00 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



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```
Current Data Parameters
NAME         HCC-G3-denNC
EXPNO                   :
PROCNO                  1

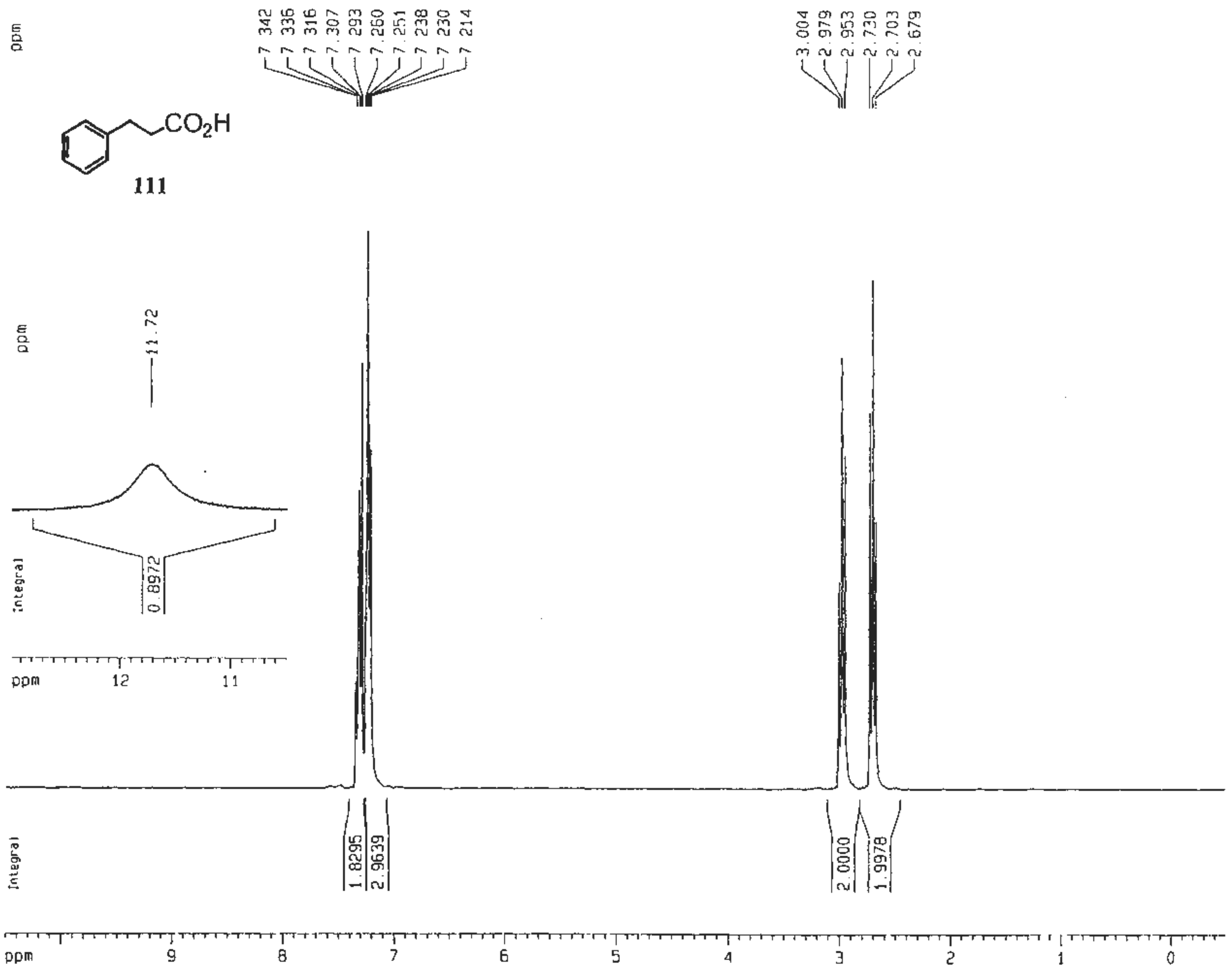
F2 - Acquisition Parameters
Date_            20080609
Time                 0 56
INSTRUM            dpx300
PROBHD   5 mm BBC BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   5800
DS                      0
SWH             22675.736 Hz
FIDRES           0.345004 Hz
AQ              1 4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                  295.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100 00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677405 MHz
WDW                    EM
SSB                     0
LB                   0 50 Hz
GB                      0
PC                   1 40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.09 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754 68 Hz
PPMCM             9.54545 ppm/cm
HZCM           720 37384 Hz/cm
```



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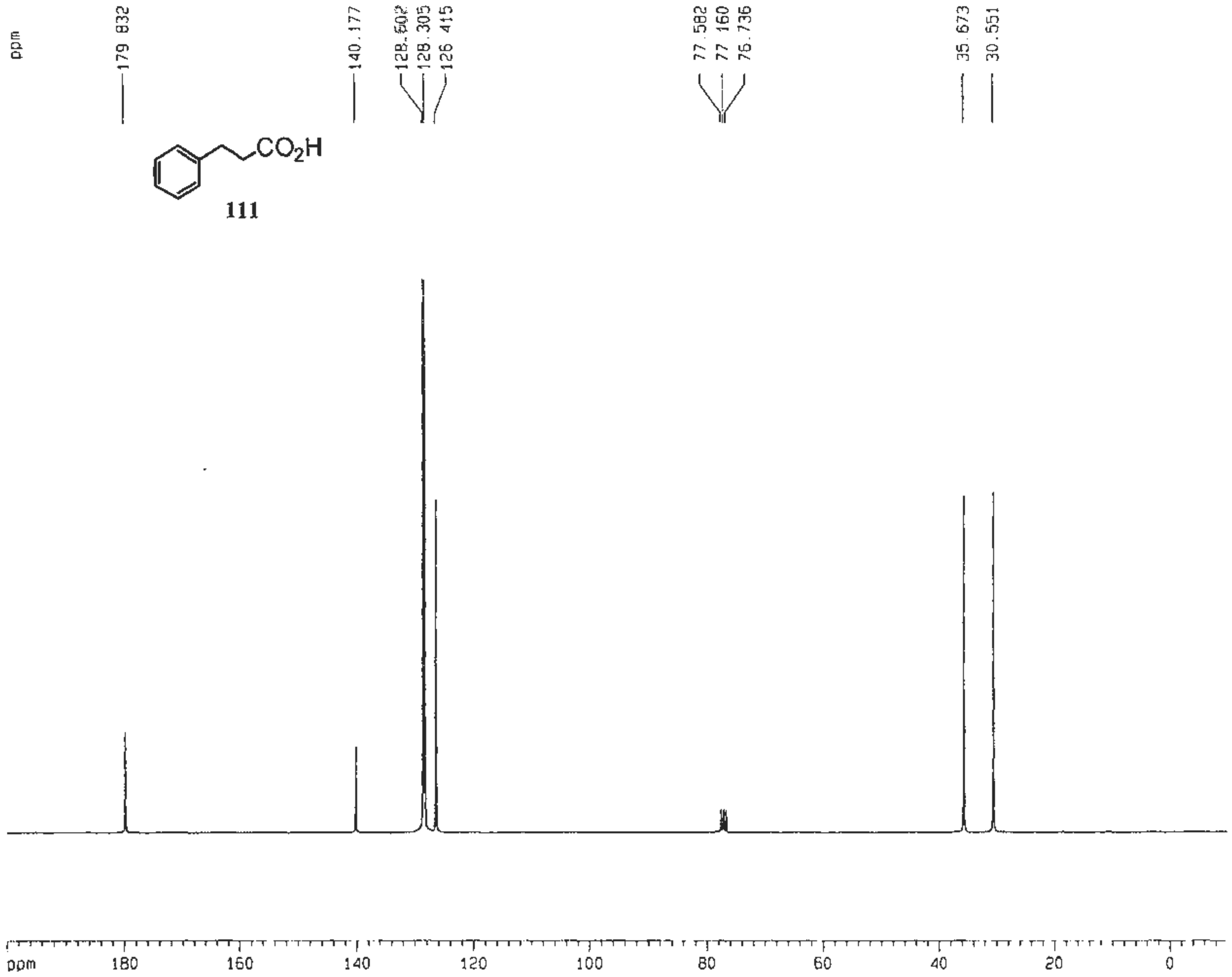
```
Current Data Parameters
NAME     Di-H-Cinnamicacid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20050702
Time               11.38
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                 16384
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             4202 563 Hz
FIDRES          0.256504 Hz
AQ             1 9493364 sec
RG                 143 7
DW               118 975 usec
DE                169 96 usec
TE                 296 2 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0 01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  5.00 usec
PL1                -2.00 dB
SFO1         300.1318000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300057 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 10.01 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150 07 Hz
PPMCM            0.50000 ppm/cm
HZCM         150 06500 Hz/cm
```



111

```
Current Data Parameters
NAME     Di-H-Cinnamic acidC
EXPNO                  1
PROCNO                 1

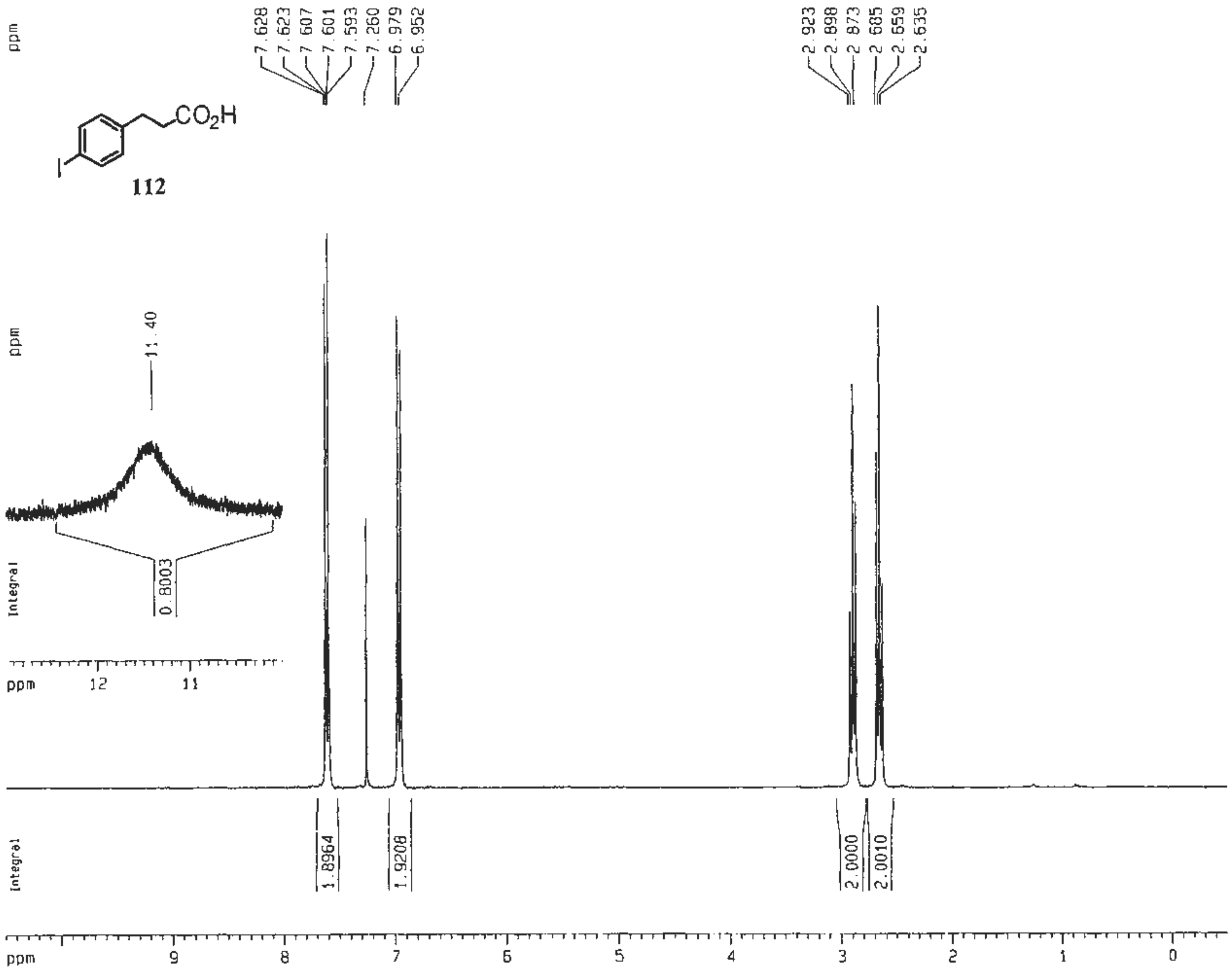
F2 - Acquisition Parameters
Date_           20050704
Time               13.45
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  1080
DS                     0
SWH           22675 736 Hz
FIDRES         0 346004 Hz
AQ            1 4451188 sec
RG                  8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1         1.00000000 sec
d11        0.03000000 sec
MCREST     0 00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100 00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF           75.4677581 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                  9 98 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10 000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37402 Hz/cm
```



112

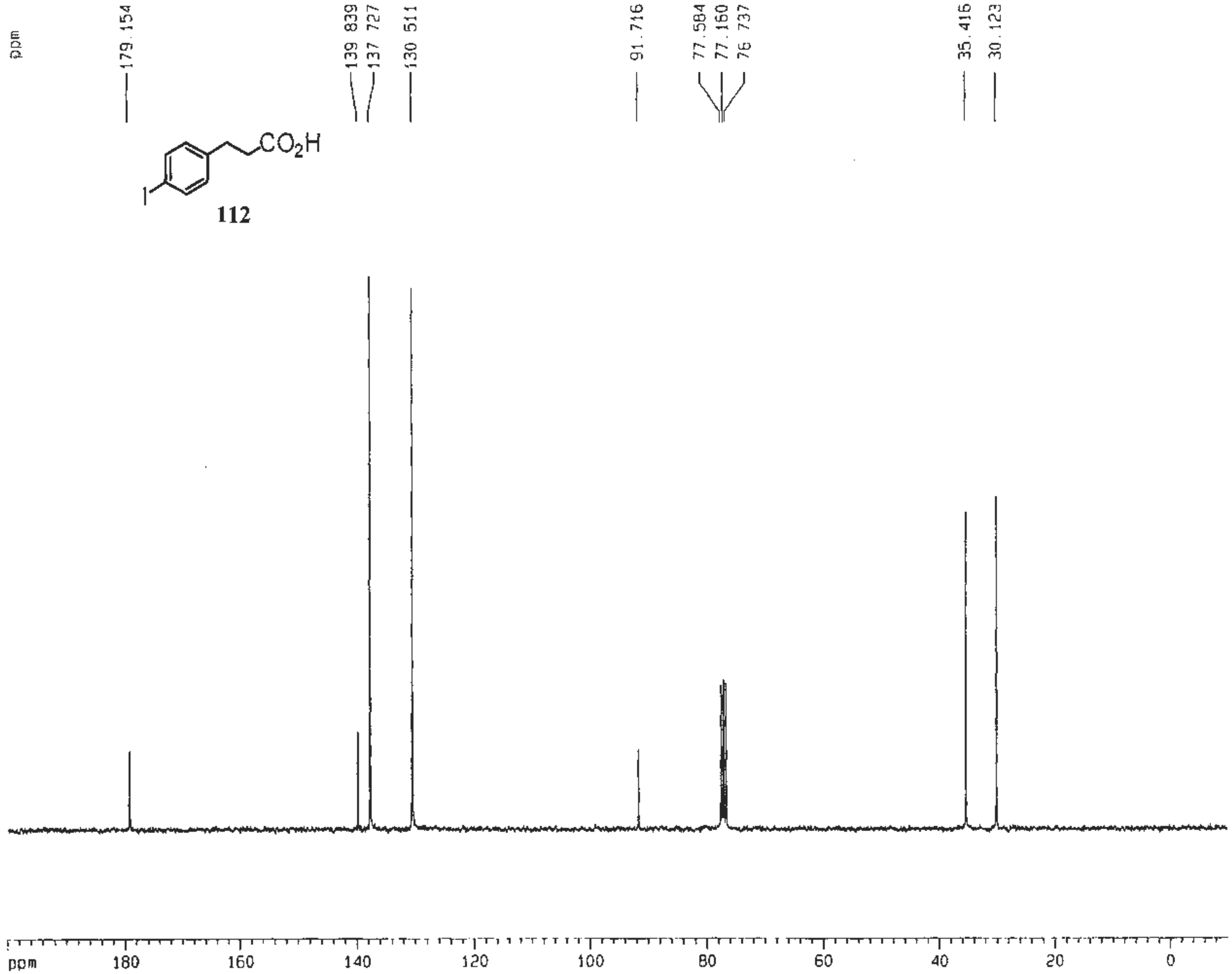
```
Current Data Parameters
NAME       I-Ph-(CH2)2COOH
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20060118
Time                 18 34
INSTRUM             dpx300
PROBHD    5 mm 880 88-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       0
SWH            4202.563 Hz
FIDRES         0.256504 Hz
AQ           1 9493364 sec
RG                     512
DW             118.975 usec
DE              169.96 usec
TE                   0 0 K
D1         5.00000000 sec
MCREST     0 00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1               5.00 usec
PL1                -2 00 dB
SFO1        300.1318000 MHz

F2 - Processing parameters
SI                   32768
SF          300.1300062 MHz
WDW                     EM
SSB                      0
LB                 0 30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                22 00 cm
CY                10.01 cm
F1P             10.500 ppm
F1              3151.36 Hz
F2P              0.500 ppm
F2              -150.07 Hz
PPMCM       0.50000 ppm/cm
HZCM      150.06500 Hz/cm
```



112

```
Current Data Parameters
NAME       ]-Pn-(CH2)2COOHC
EXPNO                    1
PROCNO                   1

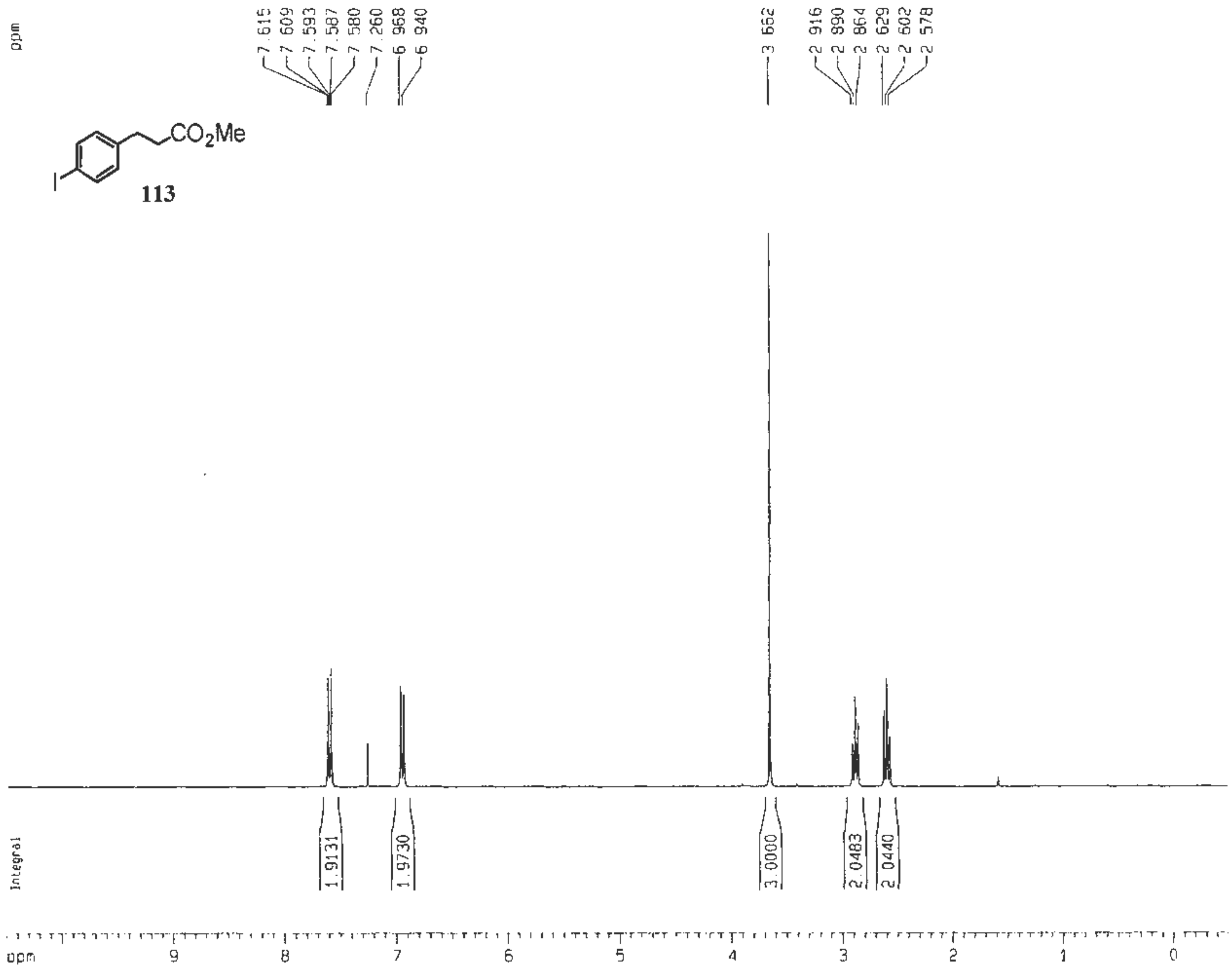
F2 - Acquisition Parameters
Date_             20050314
Time                 18 39
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDC13
NS                     401
DS                       0
SWH             22675 736 Hz
FIDRES           0.346004 Hz
AO              1 4451188 sec
RG                 1032:.3
DW                 22.050 usec
DE                   6 00 usec
TE                    0.0 K
D1             1.00000000 sec
d11            0 03000000 sec
MCRES1         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    3 00 usec
PL1                  -6.00 dB
SFO1           75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100 00 usec
PL2                 120 00 dB
PL12                 19 00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF              75.4677413 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1 40

1D NMR plot parameters
CX                   22.00 cm
CY                   10.00 cm
F1P                200.000 ppm
F1                15093.55 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM              9 54545 ppm/cm
HZCM             720 37384 Hz/cm
```



113

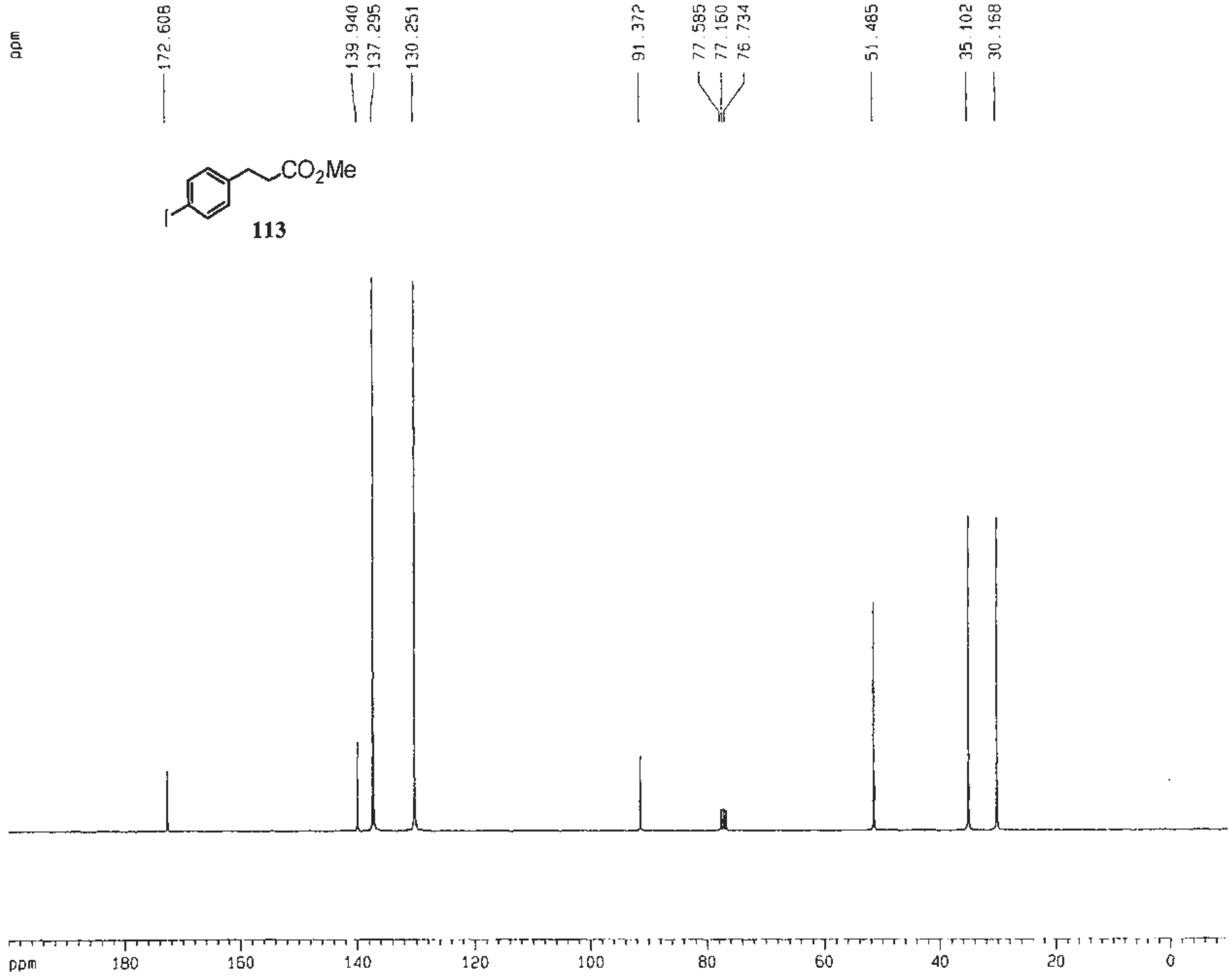
```
Current Data Parameters
NAME     I-Ph-(CH2)2CO2Me
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20050509
Time                21 32
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     10
DS                      0
SWH             3601 657 Hz
FIDRES          0 219828 Hz
AO             2.2745588 sec
RG                    256
DW               138.825 usec
DE                198 32 usec
TE                 296 2 K
D1            5 00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5 00 usec
PL1                 -2 00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF           300 1300060 MHz
WDW                    EM
SSB                     0
LB                   0 30 Hz
GB                      0
PC                   1 00

1D NMR plot parameters
CX                  22 00 cm
CY                  10 00 cm
F1P                10 500 ppm
F1                3151.36 Hz
F2P                -0 500 ppm
F2                -150.07 Hz
PPMCM             0 50000 ppm/cm
HZCM            150.06500 Hz/cm
```



113

```
Current Data Parameters
NAME     I-Ph-(CH2)2CO2MeC
EXPNO                    1
PROCNO                   1

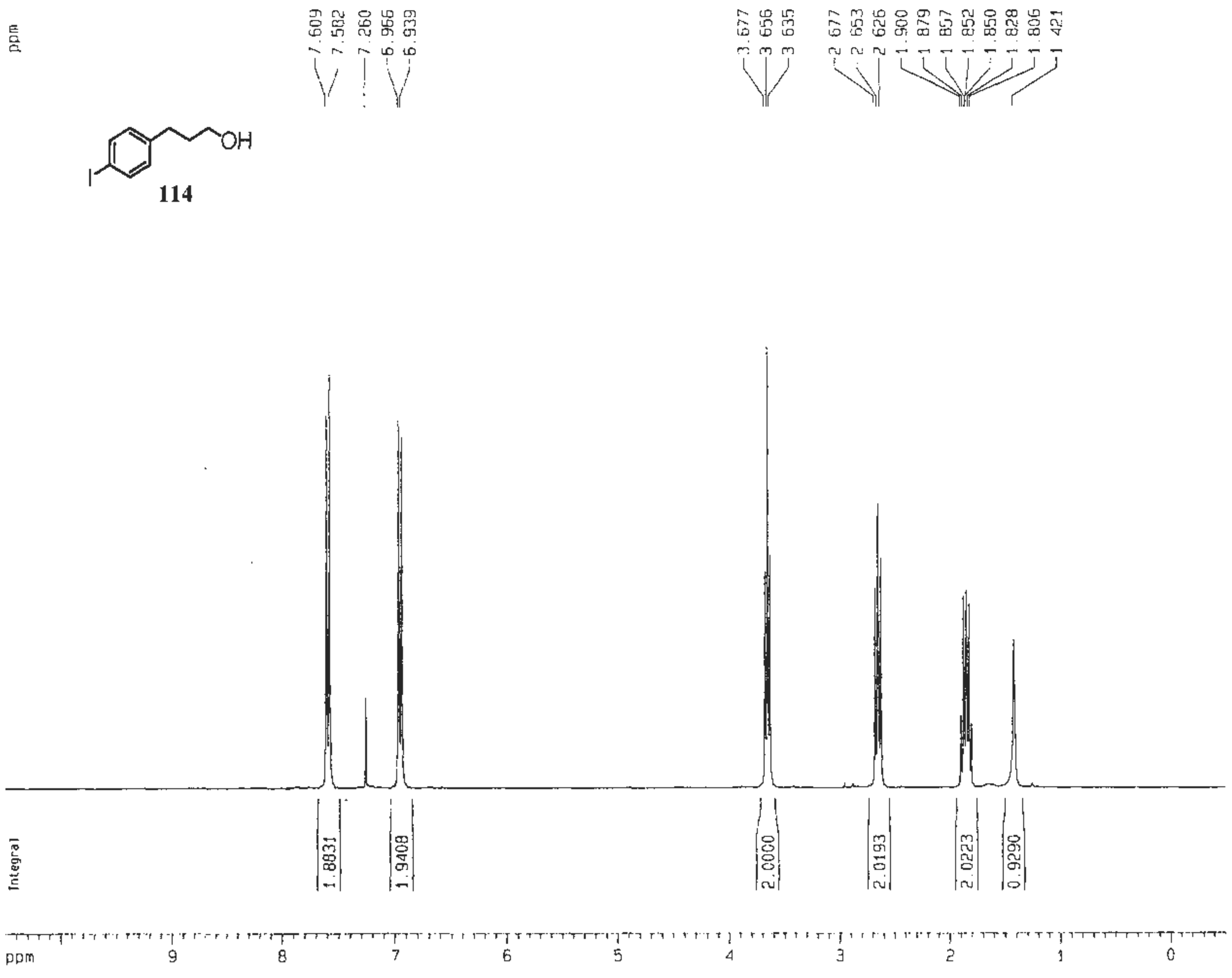
F2 - Acquisition Parameters
Date_             20050510
Time                 18.56
INSTRUM             dpx300
PROBHD   5 mm BBO BB 1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     400
DS                       0
SWH            22675 736 Hz
FIDRES          0 346004 Hz
AQ            1 4451188 sec
RG                    8192
DW              22.050 usec
DE                6.00 usec
TE                 296.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0 00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                3 00 usec
PL1                -6 00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2           100 00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300 1315007 MHz

F2 - Processing parameters
SI                   65536
SF           75 4677659 MHz
WDW                     EM
SSB                      0
LB                 3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                22.00 cm
CY                 9 99 cm
F1P            200 000 ppm
F1             15093.55 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM        9 54545 ppm/cm
HZCM        720 37408 Hz/cm
```



114

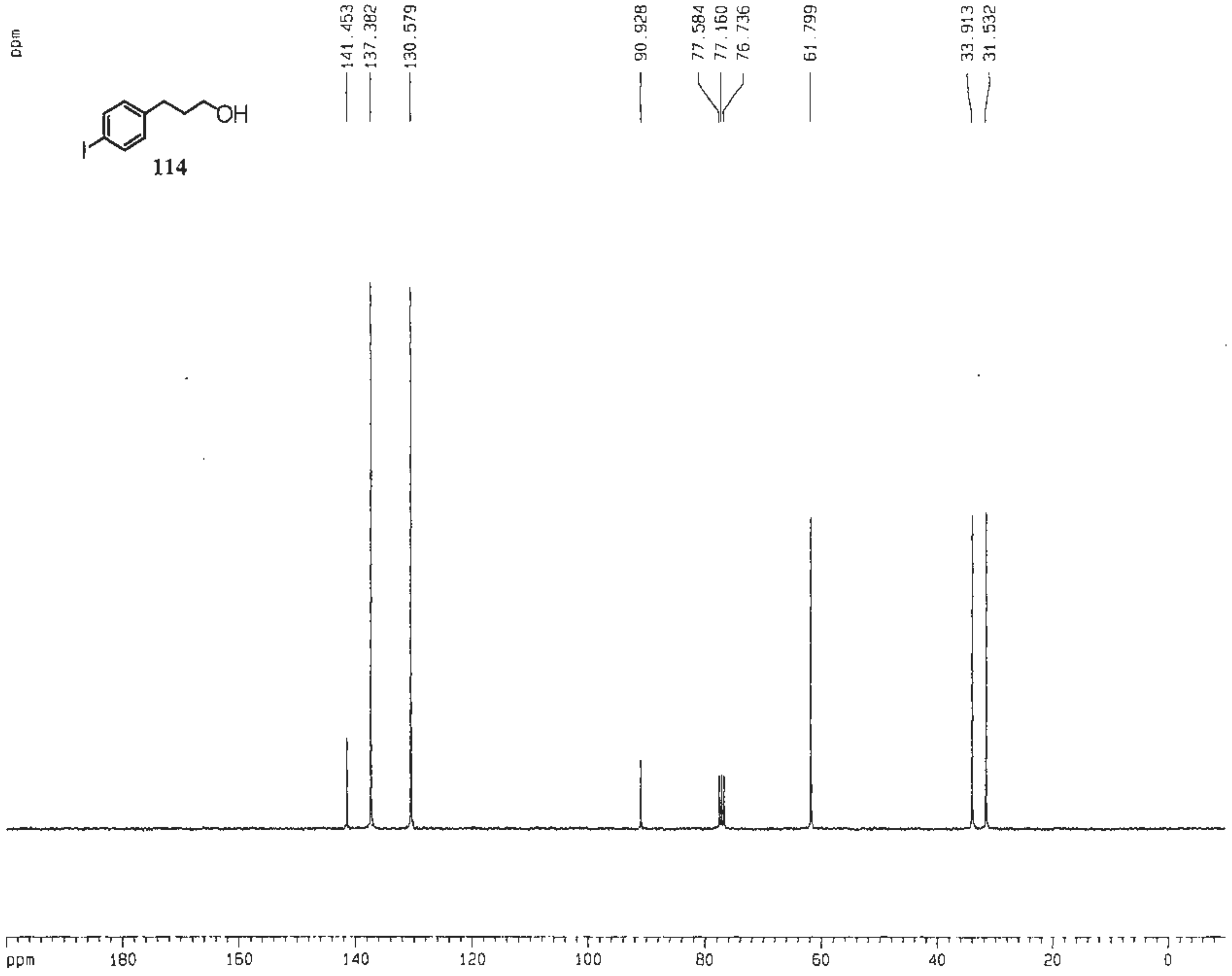
```
Current Data Parameters
NAME     I-Ph-(CH2)3-OH
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20050322
Time              18.52
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   16
DS                    0
SWH         3601.657 Hz
FIDRES      0.219828 Hz
AQ         2.2745588 sec
RG                101.6
DW         138.825 usec
DE          198.32 usec
TE                0.0 K
D1        5.00000000 sec
MCREST    0.00000000 sec
MCWAK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1            5.00 usec
PL1            -2.00 dB
SFO1     300.1315000 MHz

F2 - Processing parameters
SI                32768
SF      300.1300064 MHz
WDW                  EM
SSB                   0
LB              0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX             22.00 cm
CY              7.98 cm
F1P          10.500 ppm
F1           3151.36 Hz
F2P          -0.500 ppm
F2           -150.07 Hz
PPMCM    0.50000 ppm/cm
HZCM    150.06500 Hz/cm
```



```
Current Data Parameters
NAME     I-Ph-(CH2)3-OHC
EXPNO                  1
PROCNO                 1

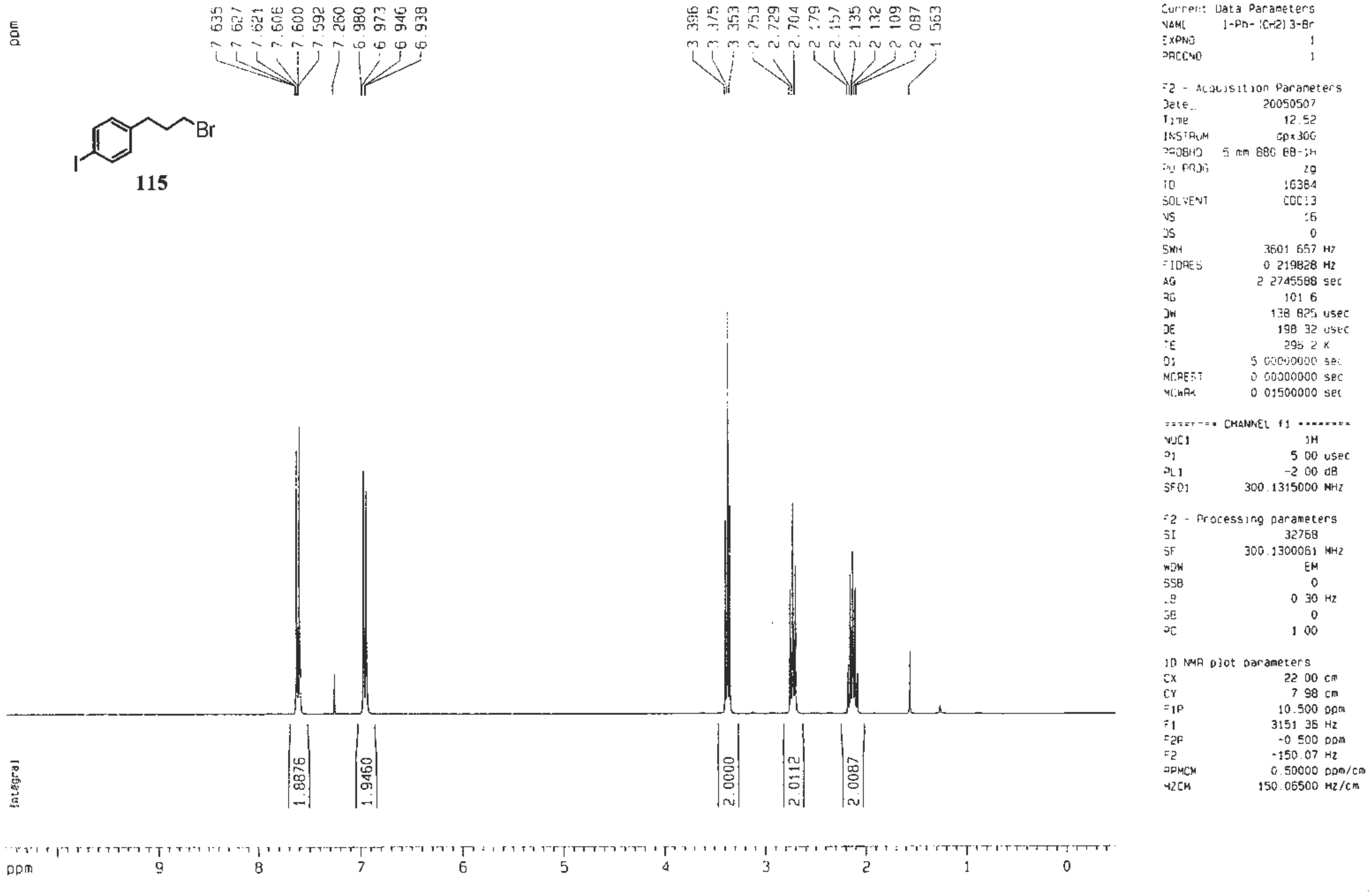
F2 - Acquisition Parameters
Date_           20050322
Time               20.38
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   183
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

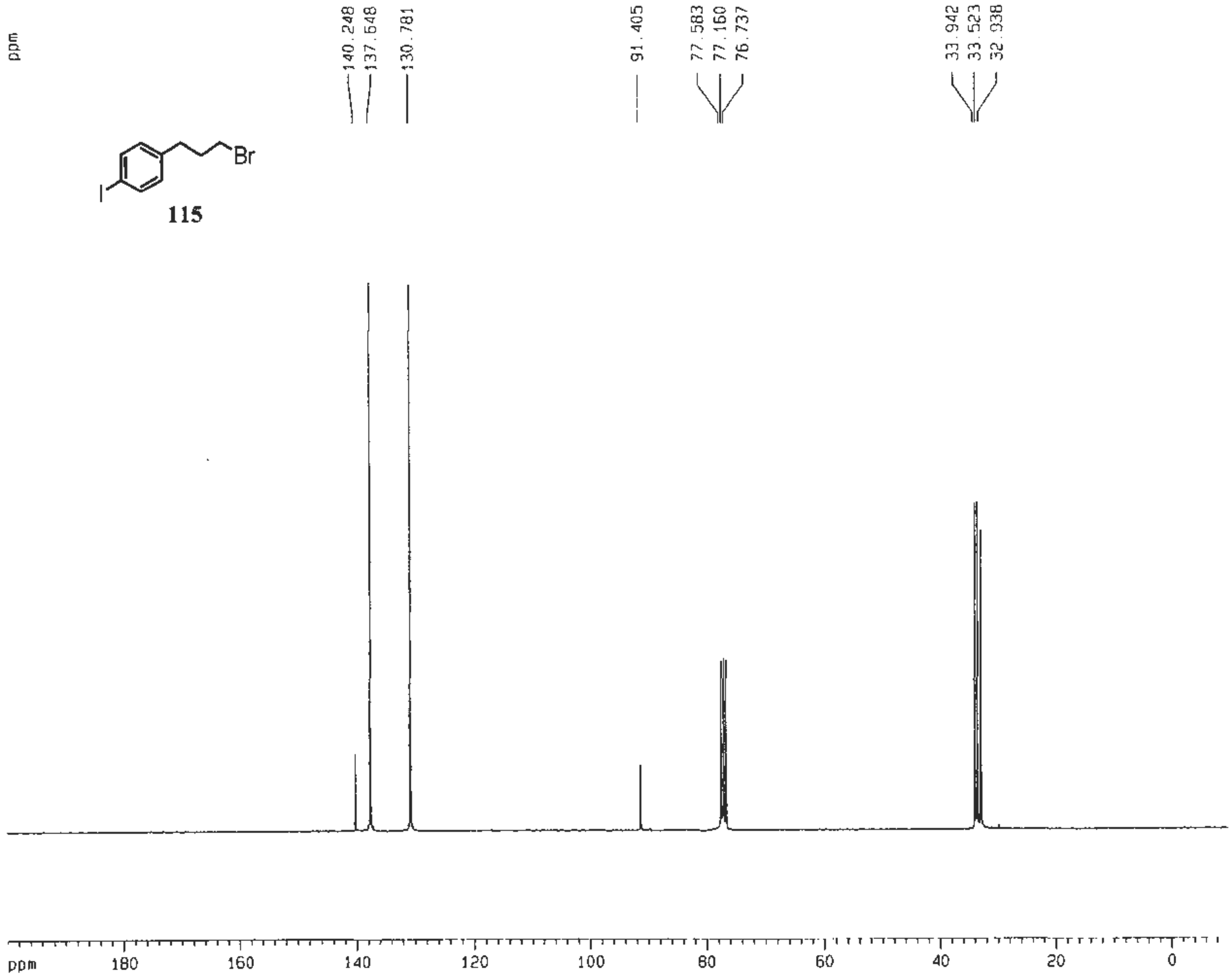
======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677522 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                  9.97 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```


115
Br
I
ppm
7.635
7.627
7.621
7.606
7.600
7.592
7.260
6.980
6.973
6.946
6.938
3.396
3.375
3.353
2.753
2.729
2.704
2.179
2.157
2.135
2.132
2.109
2.087
1.563
1.8876
1.9460
2.0000
2.0112
2.0087
Integral
ppm
9
8
7
6
5
4
3
2
1
0
Current Data Parameters
NAME 1-Ph-(CH2)3-Br
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050507
Time 12.52
INSTRUM spx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 3601.657 Hz
FIDRES 0.219828 Hz
AQ 2.2745588 sec
RG 101.6
DW 138.825 usec
DE 198.32 usec
TE 295.2 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SFO1 300.1315000 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 7.98 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm



115

```
Current Data Parameters
NAME      I-Ph-(CH2)3-BrC
EXPNO                   1
PROCNO                  1

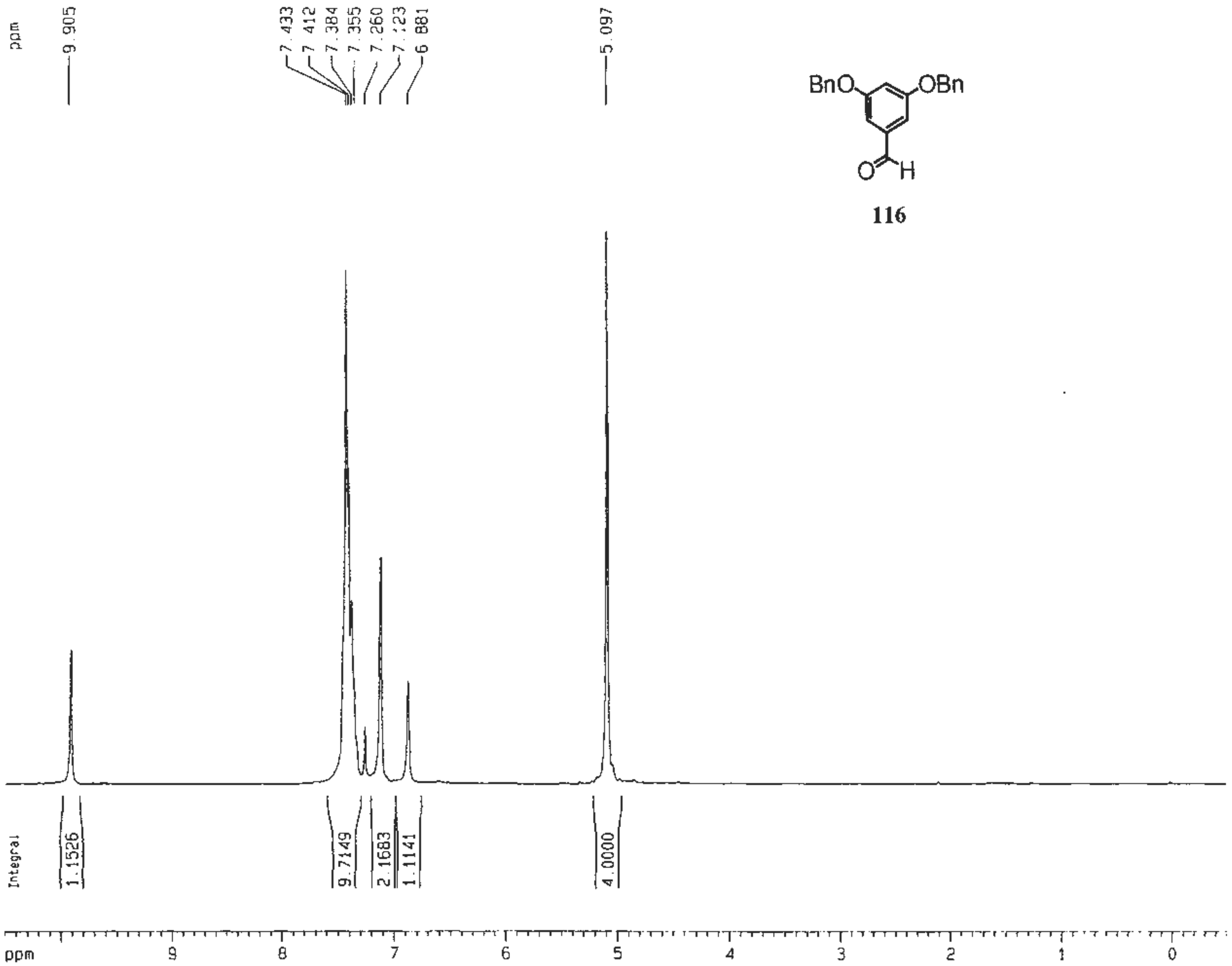
F2 - Acquisition Parameters
Date_            20050509
Time                23.58
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                  11887
DS                      0
SWH             22675.736 Hz
FIDRES           0 346004 Hz
AO              1.4451188 sec
RG                   8192
DW                 22 050 usec
DE                   6.00 usec
TE                  296.2 K
D1             1 00000000 sec
d11            0 03000000 sec
MCREST         0 00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120 00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75 4677429 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                   9 95 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10 000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37384 Hz/cm
```



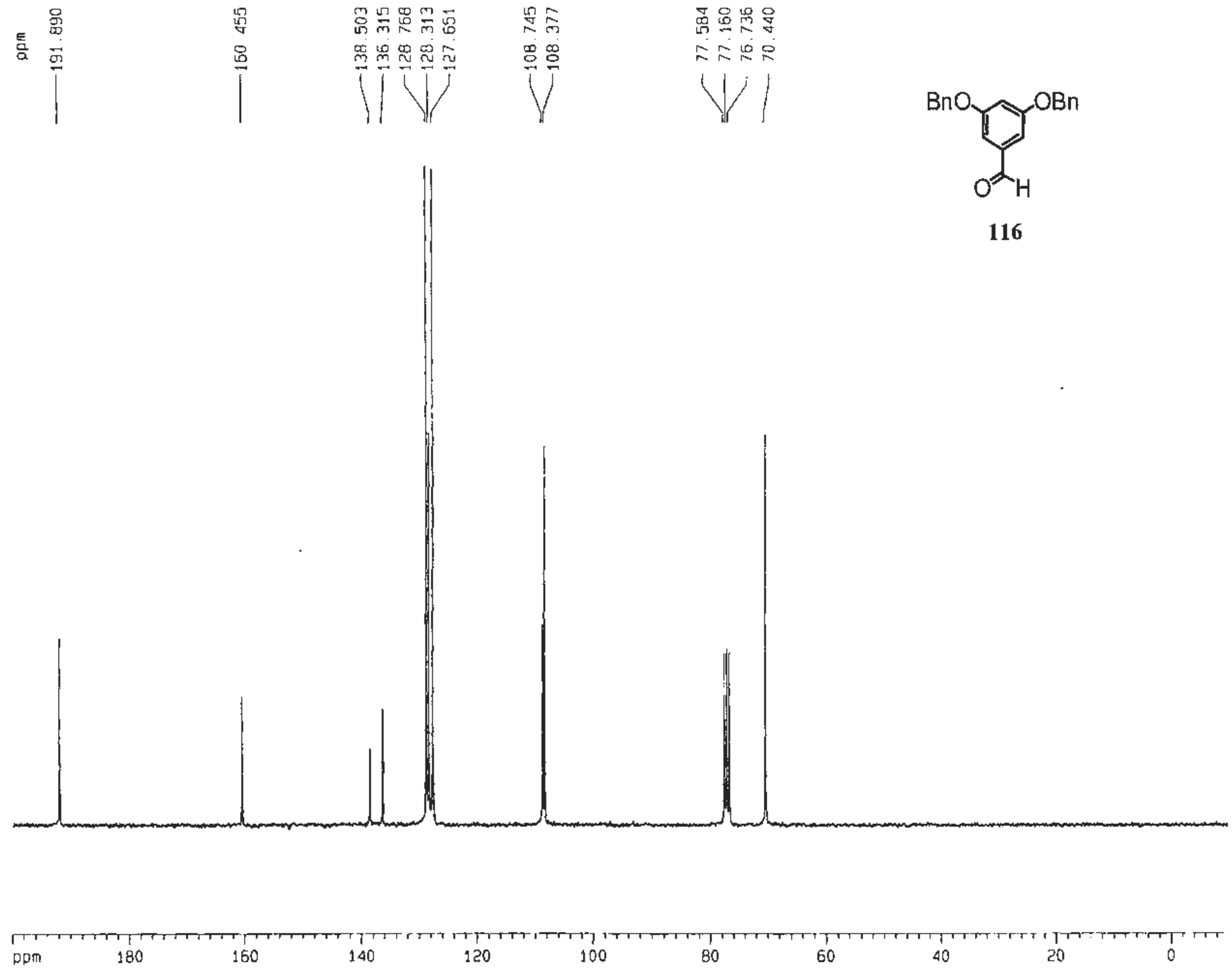
```
Current Data Parameters
NAME          Di-OBn-CHO
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20040625
Time               16 15
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH          8992 806 Hz
FIDRES       0 274439 Hz
AQ          1 8219508 sec
RG                 101 6
DW            55.600 usec
DE             79.43 usec
TE               296.2 K
D1         1.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1             9.00 usec
PL1              -2 00 dB
SF01      300.1312000 MHz

F2 - Processing parameters
SI                 32768
SF        300.1300063 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              22.00 cm
CY               9.99 cm
F1P           10.500 ppm
F1            3151 36 Hz
F2P           -0.500 ppm
F2           -150.07 Hz
PPMCM      0.50000 ppm/cm
HZCM      150 06500 Hz/cm
```



116

```
Current Data Parameters
NAME          Dj-OBn-CHOC
EXPNO                   1
PROCNO                  1

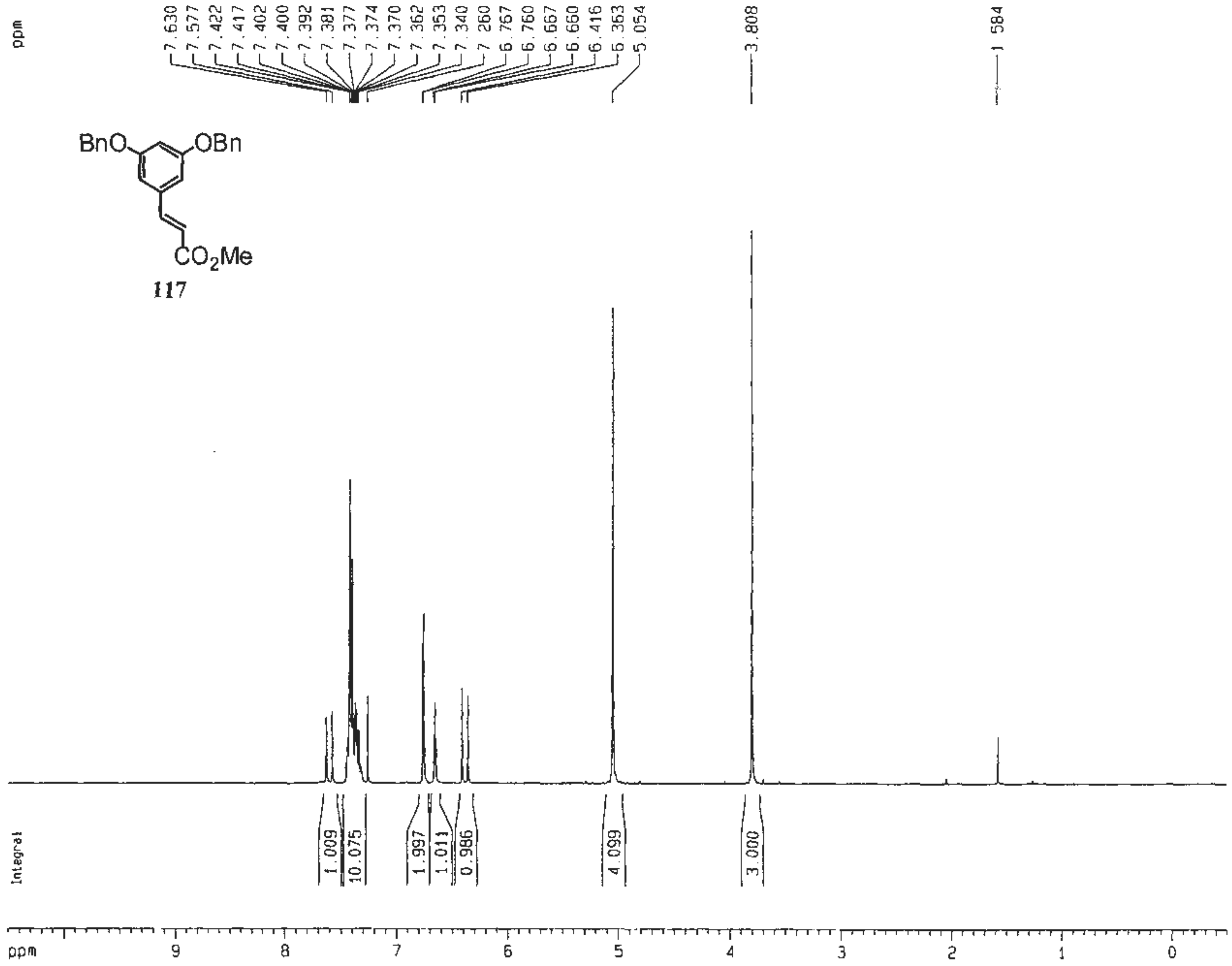
F2 - Acquisition Parameters
Date_            20050214
Time                17 22
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                    466
DS                      0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1 4451188 sec
RG                   3251
DW             22.050 usec
DE               6.00 usec
TE                298.2 K
D1         1.00000000 sec
d11        0.03000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1               3.00 usec
PL1               -6.00 dB
SFO1        75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2          100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2      300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF         75.4677461 MHz
WDW                    EM
SSB                     0
LB                3 00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX               22.00 cm
CY               11.95 cm
F1P           200.000 ppm
F1            15093.55 Hz
F2P           -10.000 ppm
F2             -754 68 Hz
PPMCM      9.54545 ppm/cm
HZCM      720.37384 Hz/cm
```


117

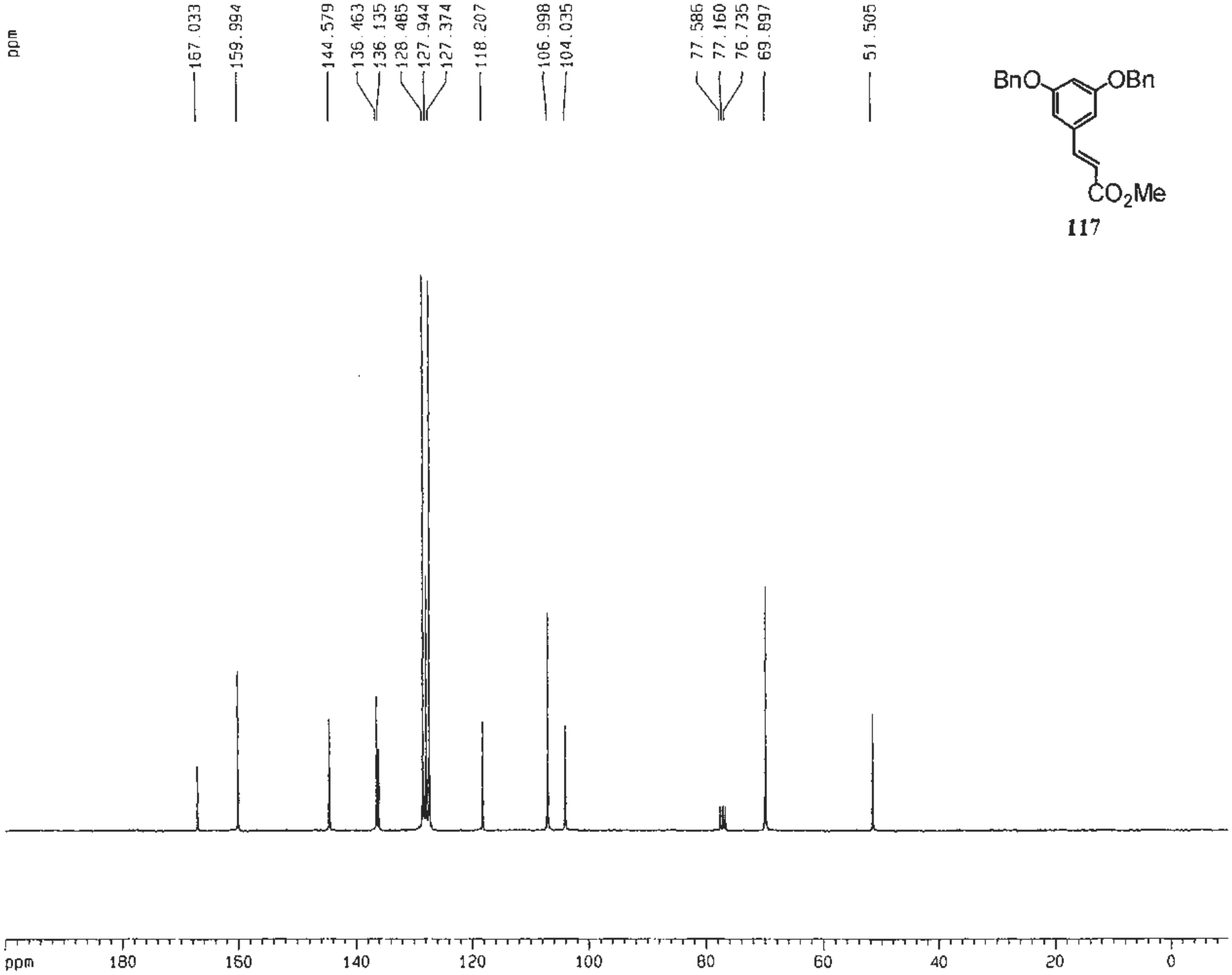
```
Current Data Parameters
NAME      D1-OBn-en-CO2Me
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20050322
Time                19 10
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              3601 657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                    128
DW                138.825 usec
DE                 198.32 usec
TE                    0 0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300061 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9 99 cm
F1P                10.500 ppm
F1                3151 36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



117

```
Current Data Parameters
NAME     Di-OBn-en-CO2MeC
EXPNO                   1
PROCNO                  1

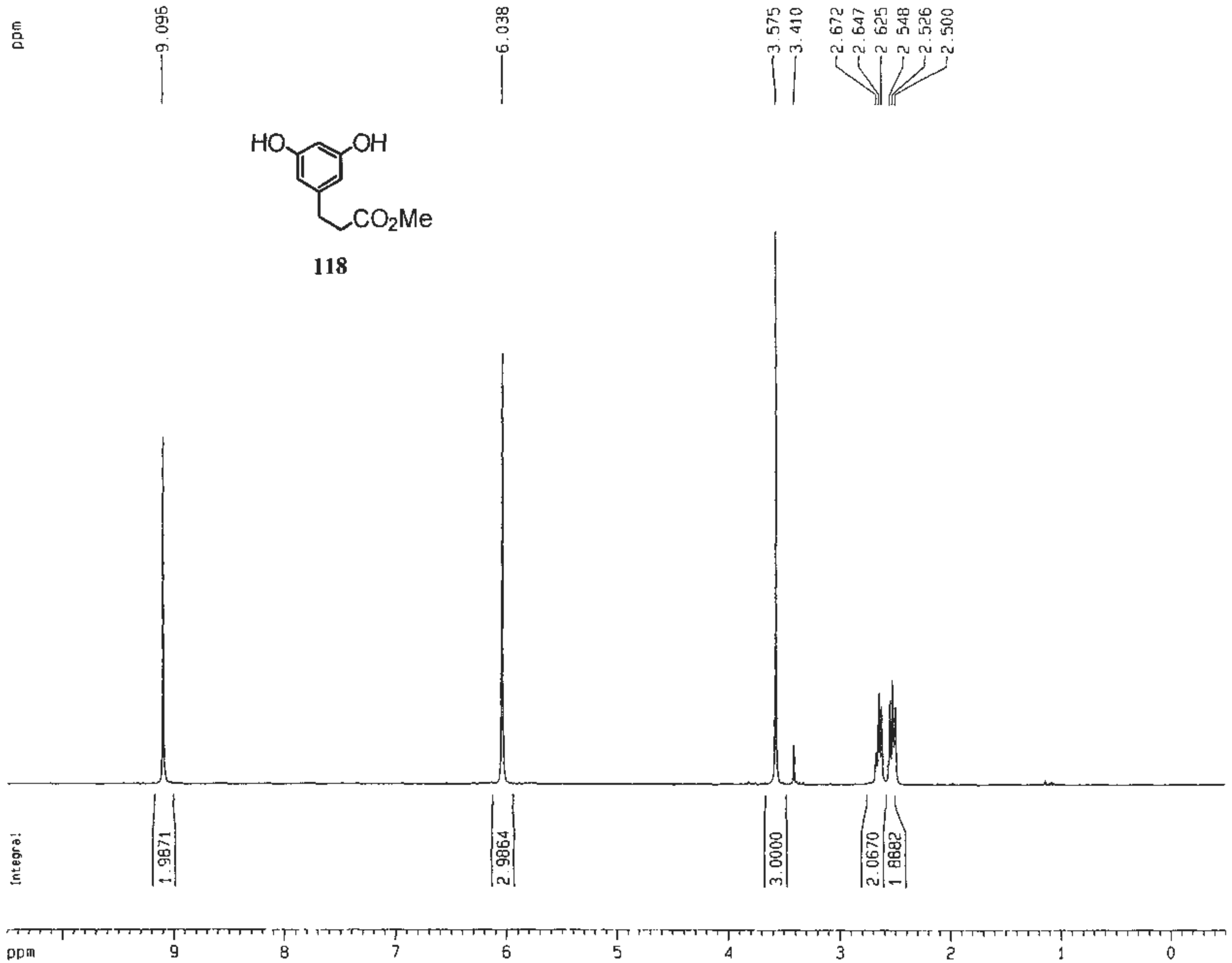
F2 - Acquisition Parameters
Date_           20050322
Time               20.51
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    89
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677696 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 10.02 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37408 Hz/cm
```



118

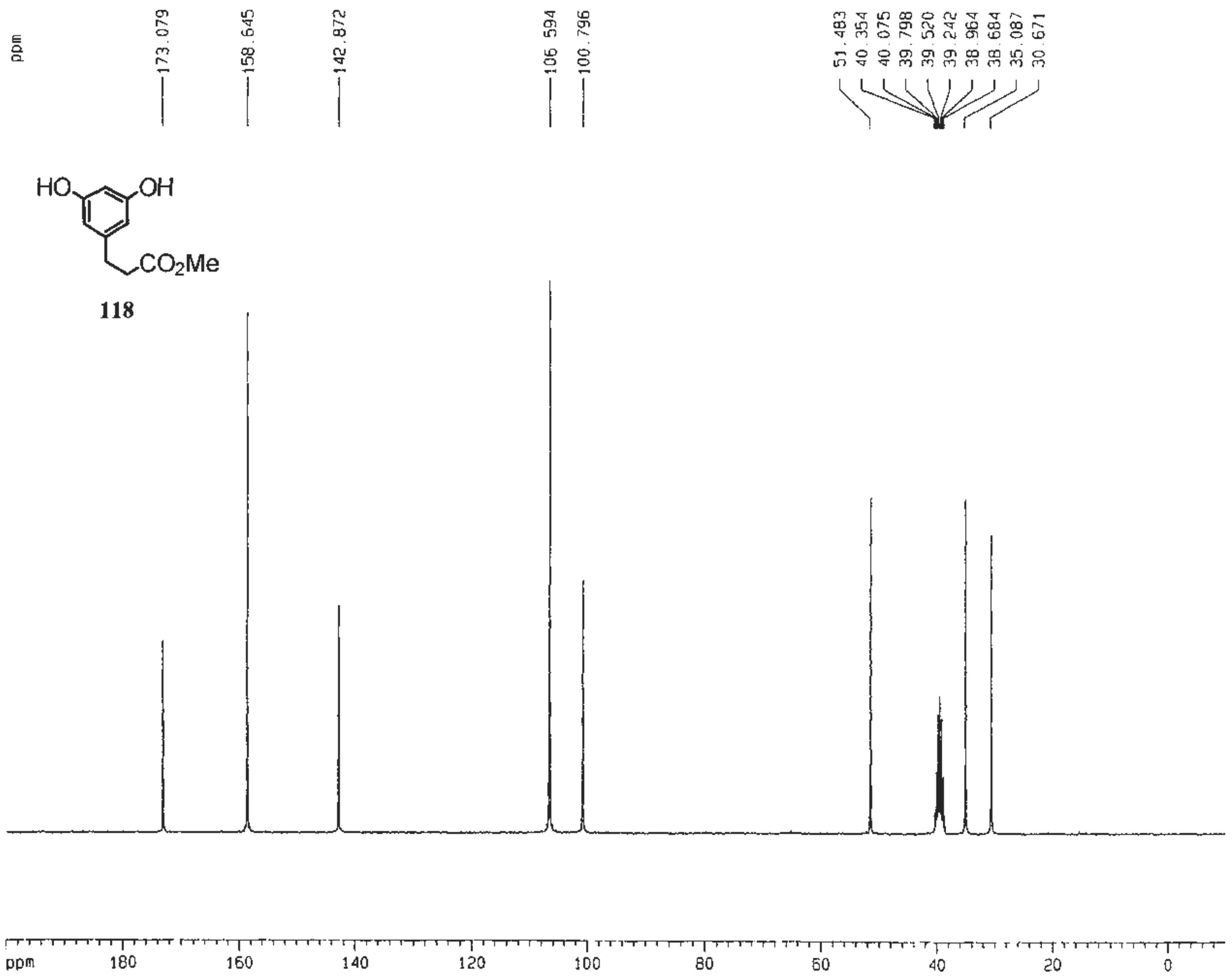
```
Current Data Parameters
NAME      D1-OH-(CH2)2CO2Me
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20060120
Time                17 12
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT              DMSO
NS                      4
DS                      0
SWH              3601 657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                    256
DW                138.825 usec
DE                 198.32 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2 00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300014 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22 00 cm
CY                   9.99 cm
F1P                10 500 ppm
F1                3151 36 Hz
F2P                -0.500 ppm
F2                -150 07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150 06500 Hz/cm
```



118

```
Current Data Parameters
NAME      Di-OH-(CH2)2CO2MeC
EXPNO                  1
PROCNO                 1

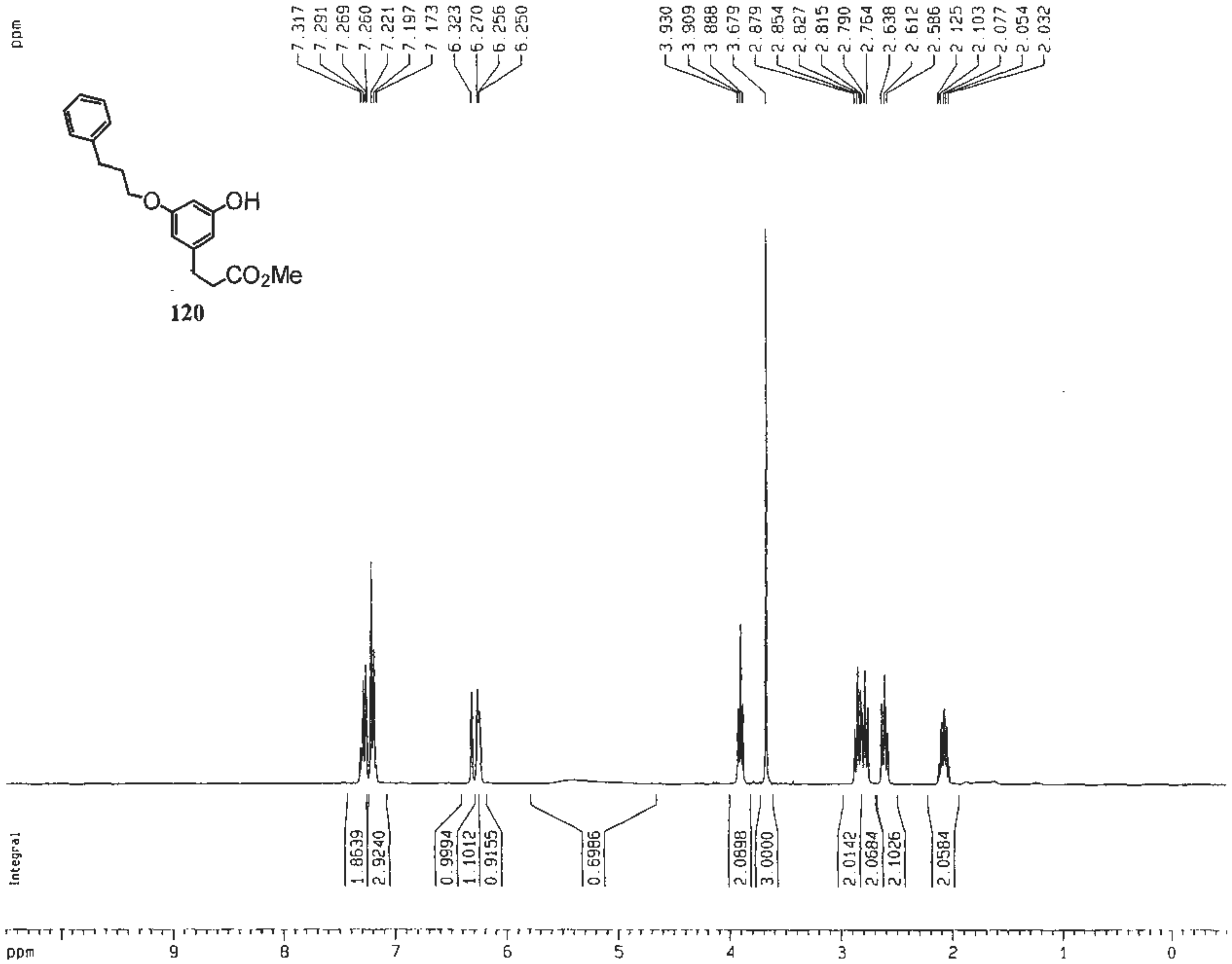
F2 - Acquisition Parameters
Date_           20060121
Time               14.41
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   449
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677566 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 10.01 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37402 Hz/cm
```



120

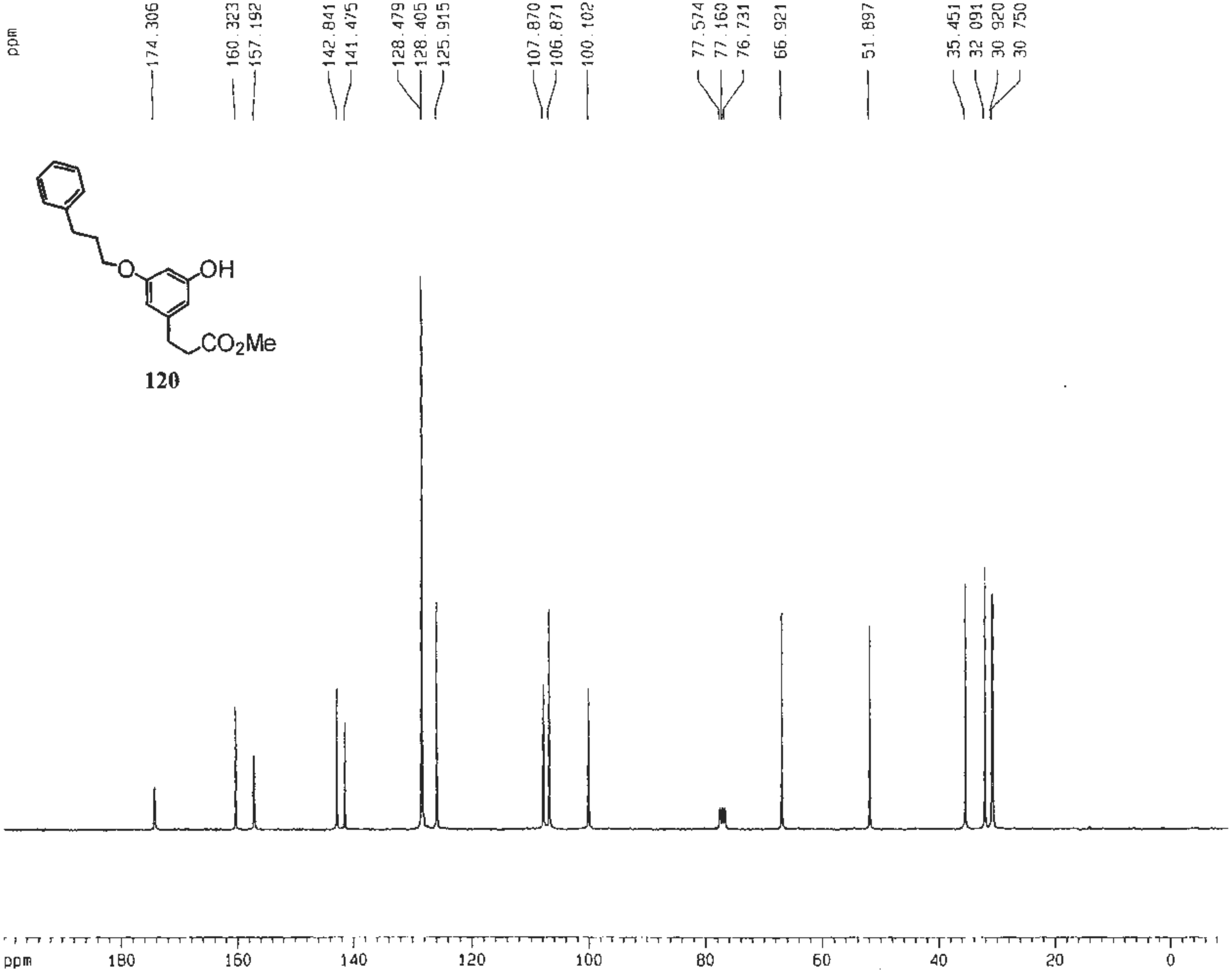
```
Current Data Parameters
NAME          GO'-M-ester
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071004
Time                12.47
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                  161.3
DW                138.825 usec
DE                 198.32 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0 01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300064 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  10 00 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



120

```
Current Data Parameters
NAME        G0'-M-esterC
EXPNO                  1
PROCNO                 1

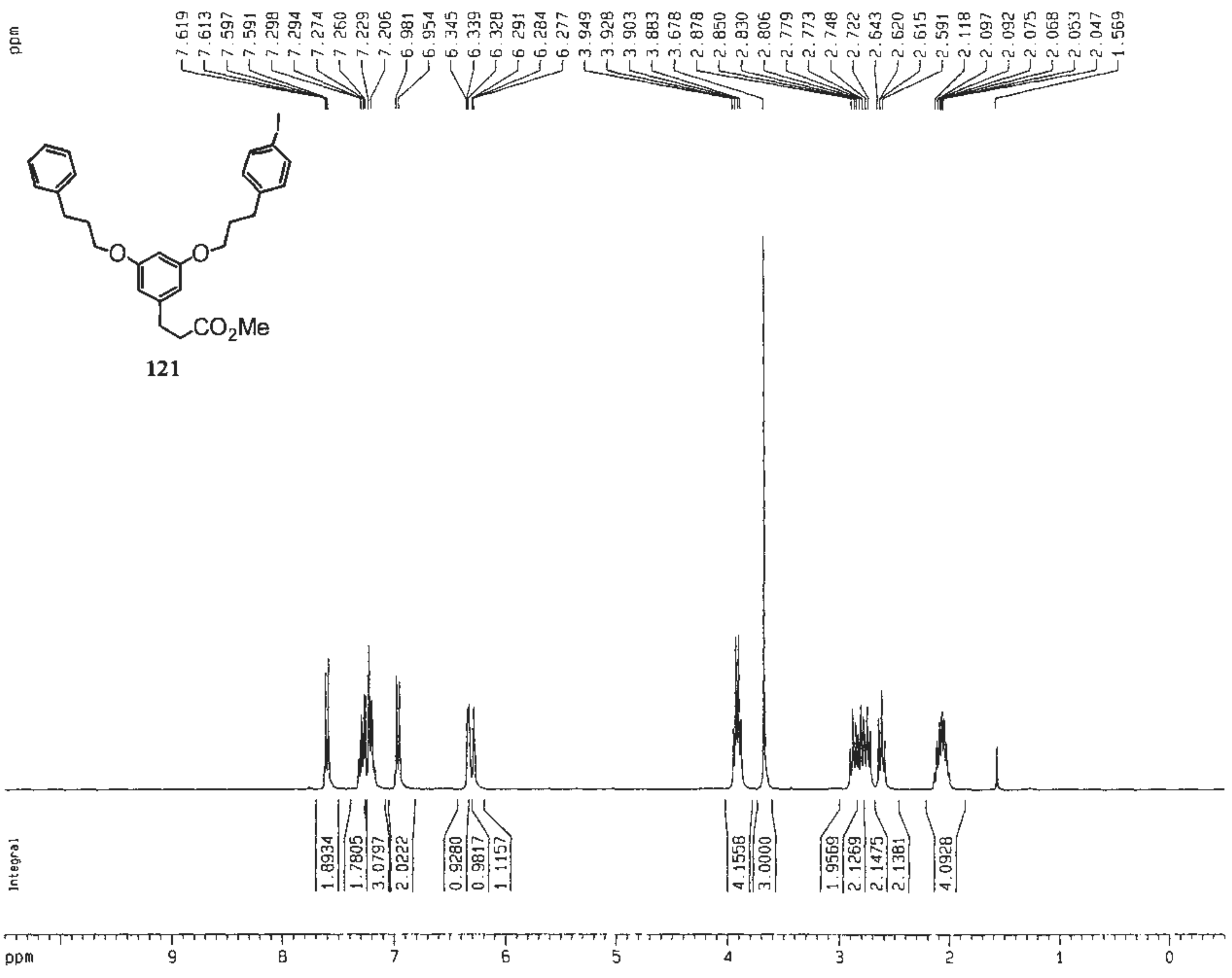
F2 - Acquisition Parameters
Date_           20071004
Time               12.20
INSTRUM           dpx300
PROBHD   5 mm 880 BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                   0.0 K
D1            1 00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120 00 dB
PL12               19.00 dB
SFO2         300 1315007 MHz

F2 - Processing parameters
SI                 65536
SF           75.4677572 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                  9.97 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37402 Hz/cm
```



121

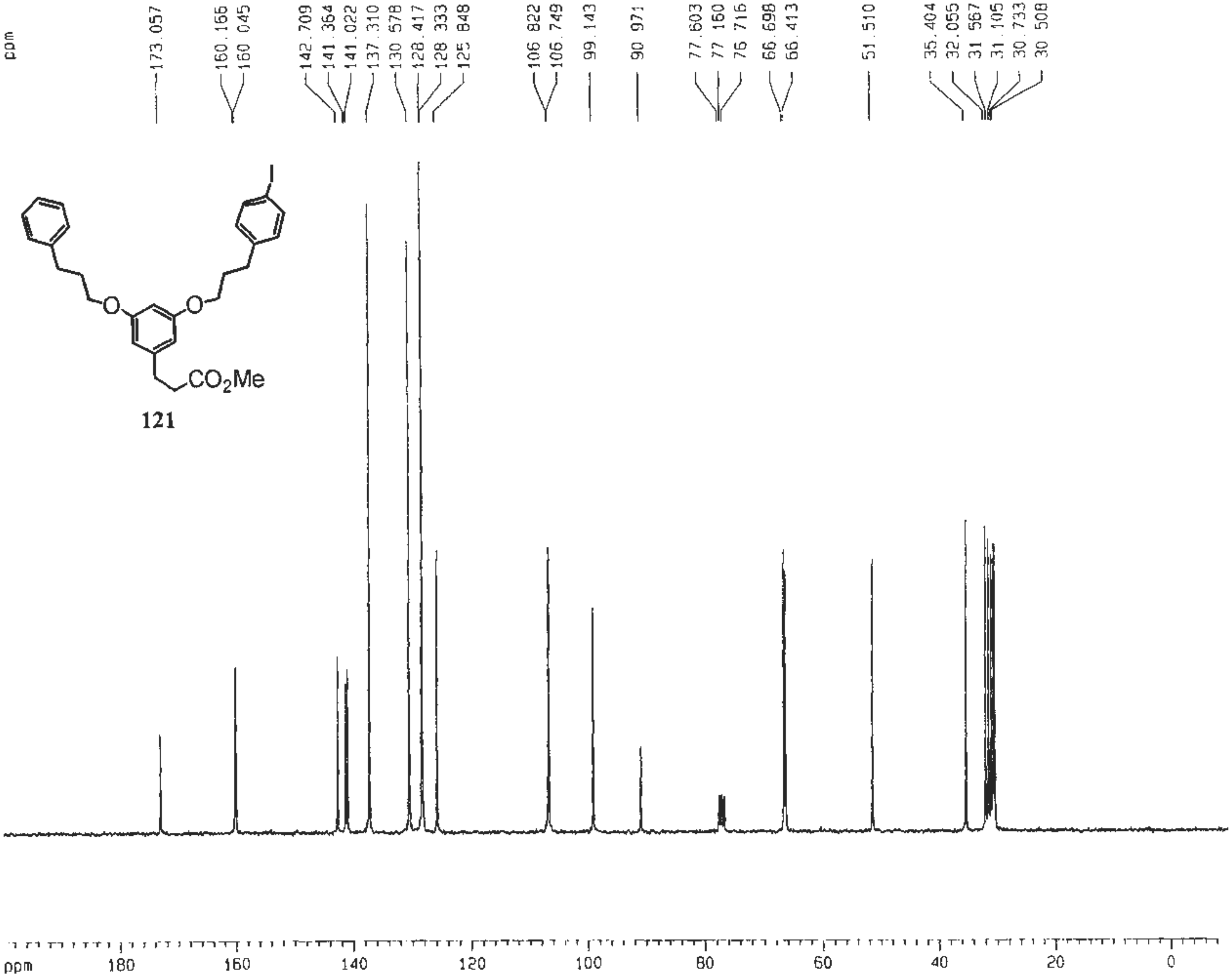
```
Current Data Parameters
NAME          I-G1'-ester
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071108
Time                12.29
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                    181
DW                138.825 usec
DE                 198.32 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF          300.1300061 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9.98 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



121

```
Current Data Parameters
NAME         I-GI'-esterC
EXPNO                   1
PROCNO                  1

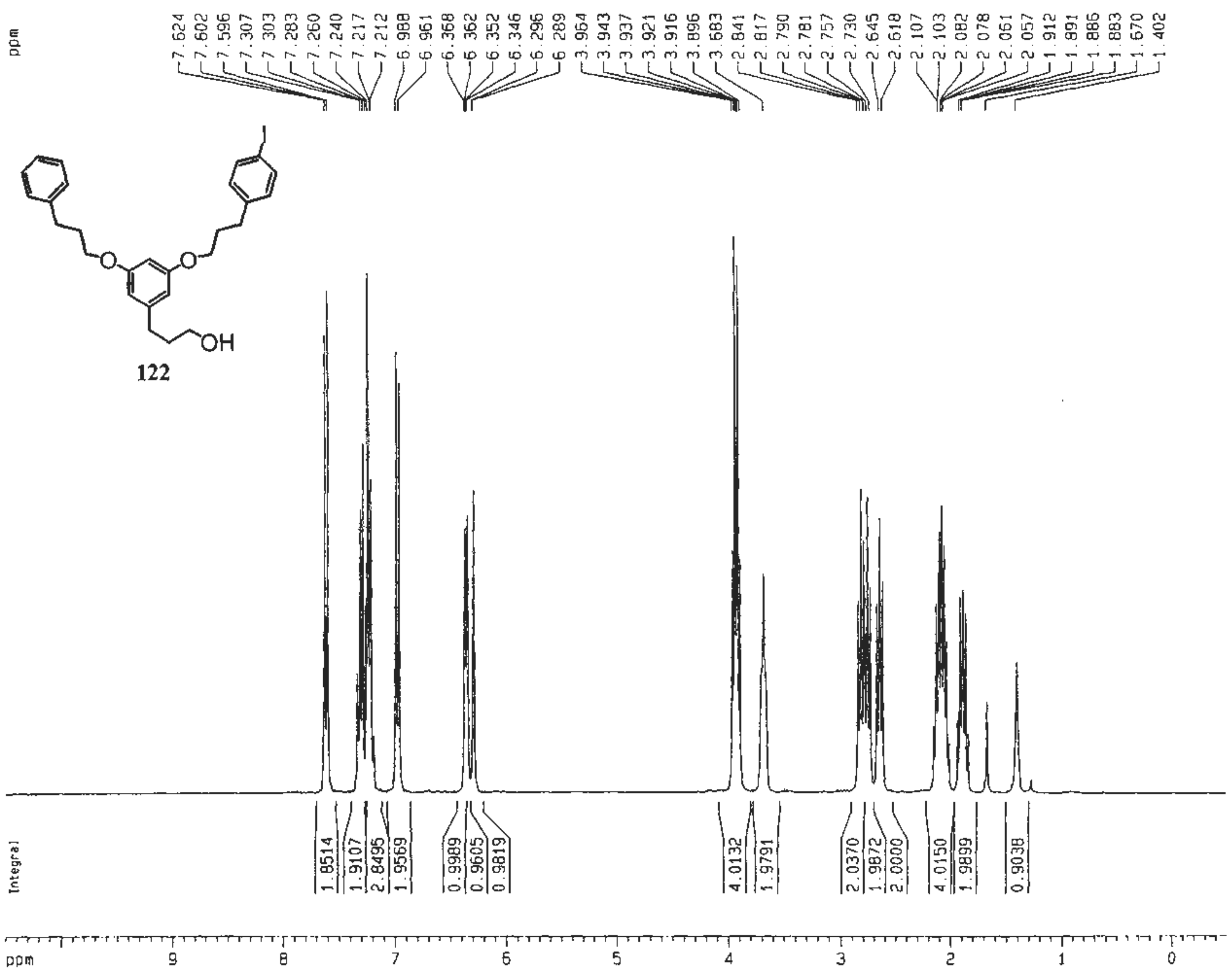
F2 - Acquisition Parameters
Date_            20070410
Time                19.58
INSTRUM            dpx300
PROBHD    5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   1000
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                    0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677669 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.01 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P                10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37408 Hz/cm
```



122

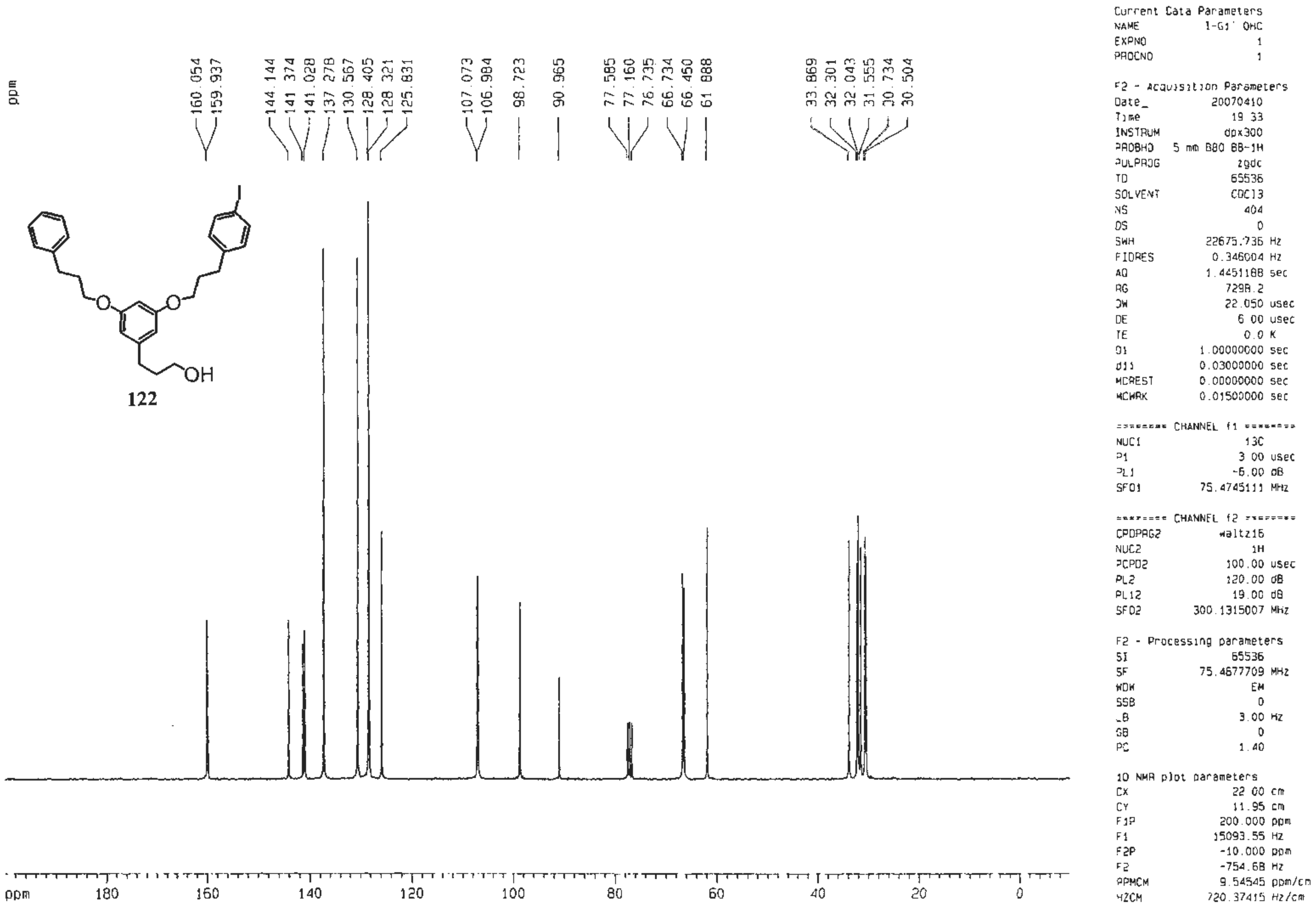
```
Current Data Parameters
NAME            I-G1'-OH
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date            20070410
Time               18.32
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             3601.657 Hz
FIDRES          0.219828 Hz
AQ             2.2745588 sec
RG                    64
DW               138.825 usec
DE                198.32 usec
TE                   0.0 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

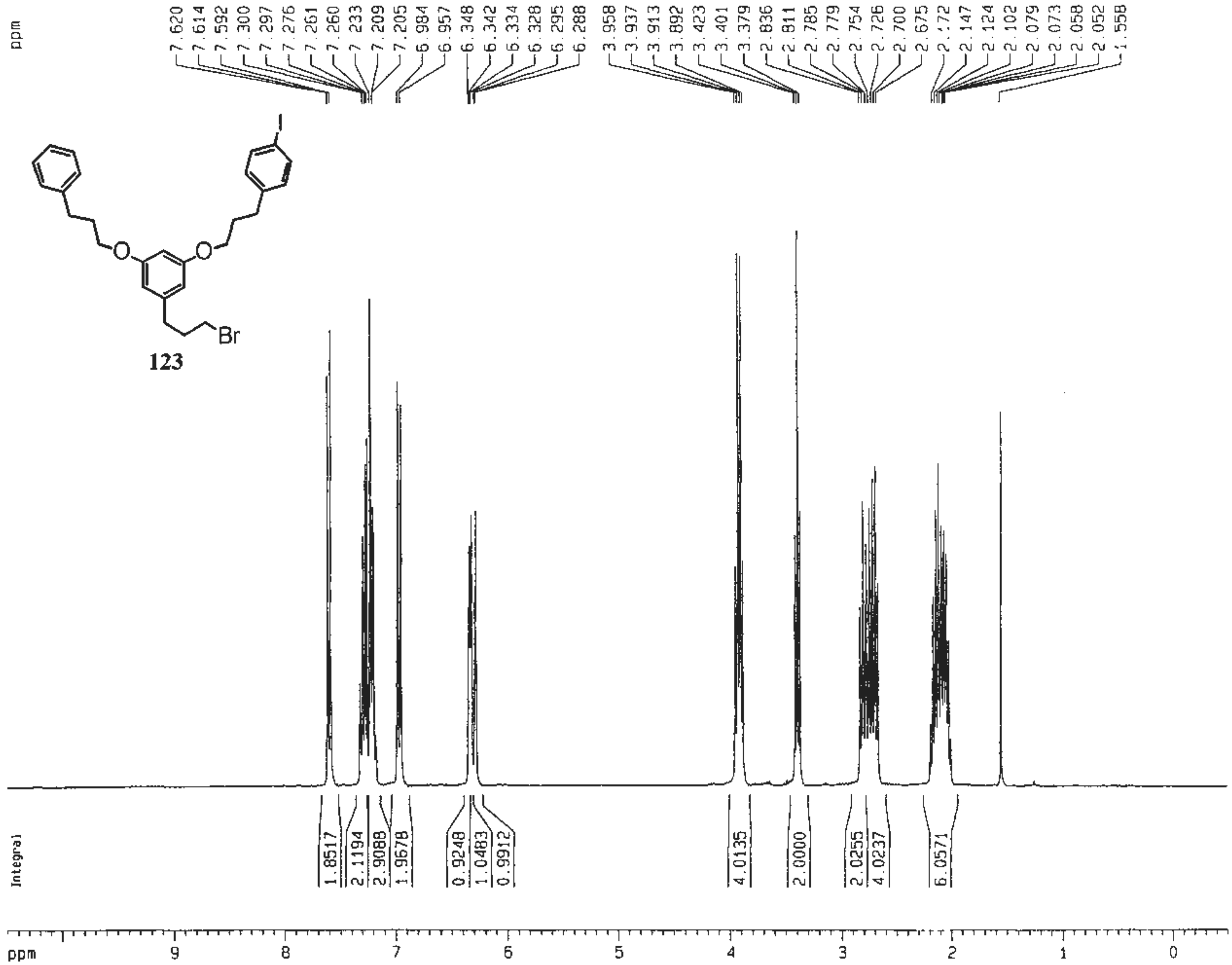
======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300065 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 10.03 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```


Current Data Parameters
NAME 1-G1' OHC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070410
Time 19 33
INSTRUM dpx300
PROBHD 5 mm B80 BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 404
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 7298.2
DW 22.050 usec
DE 6 00 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 3 00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz
F2 - Processing parameters
SI 65536
SF 75.4677709 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22 00 cm
CY 11.95 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.54545 ppm/cm
HZCM 720.37415 Hz/cm
ppm
160.054
159.937
144.144
141.374
141.028
137.278
130.567
128.405
128.321
125.831
107.073
106.984
98.723
90.965
77.585
77.160
76.735
66.734
66.450
61.888
33.869
32.301
32.043
31.555
30.734
30.504
OH
O
O
ppm
180
160
140
120
100
80
60
40
20
0

122



123

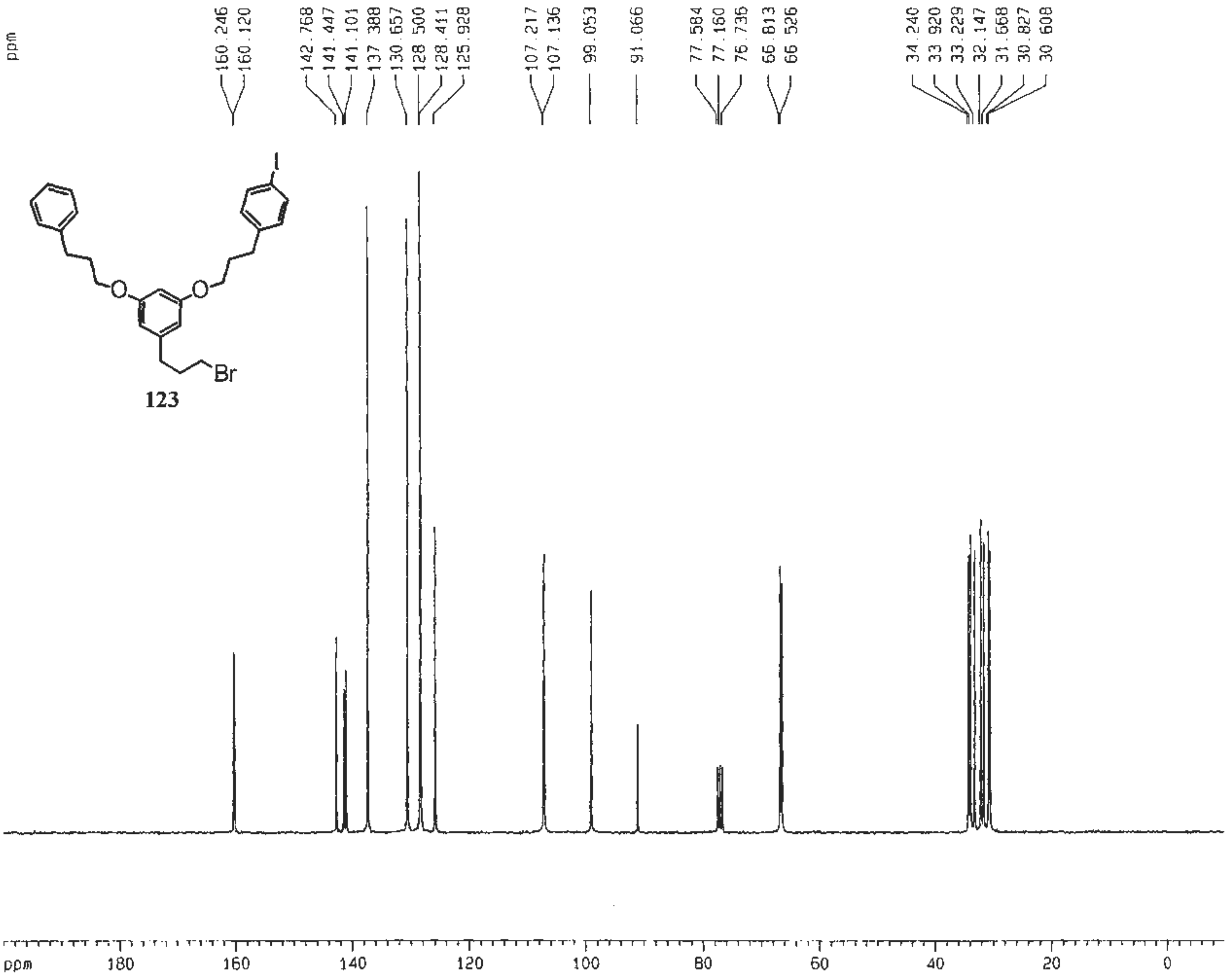
```
Current Data Parameters
NAME              I-G1'-Br
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070507
Time                 23.22
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                      32
DS                       0
SWH               3601.657 Hz
FIDRES            0.219828 Hz
AQ               2.2745588 sec
RG                     181
DW                 138.825 usec
DE                  198.32 usec
TE                     0.0 K
D1              5.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.00 usec
PL1                  -2.00 dB
SFO1           300.1315000 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300062 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                    9.98 cm
F1P                 10.500 ppm
F1                 3151.36 Hz
F2P                 -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.50000 ppm/cm
HZCM             150.06500 Hz/cm
```



123

```
Current Data Parameters
NAME          I-G1'-BrC
EXPNO                 1
PROCNO                1

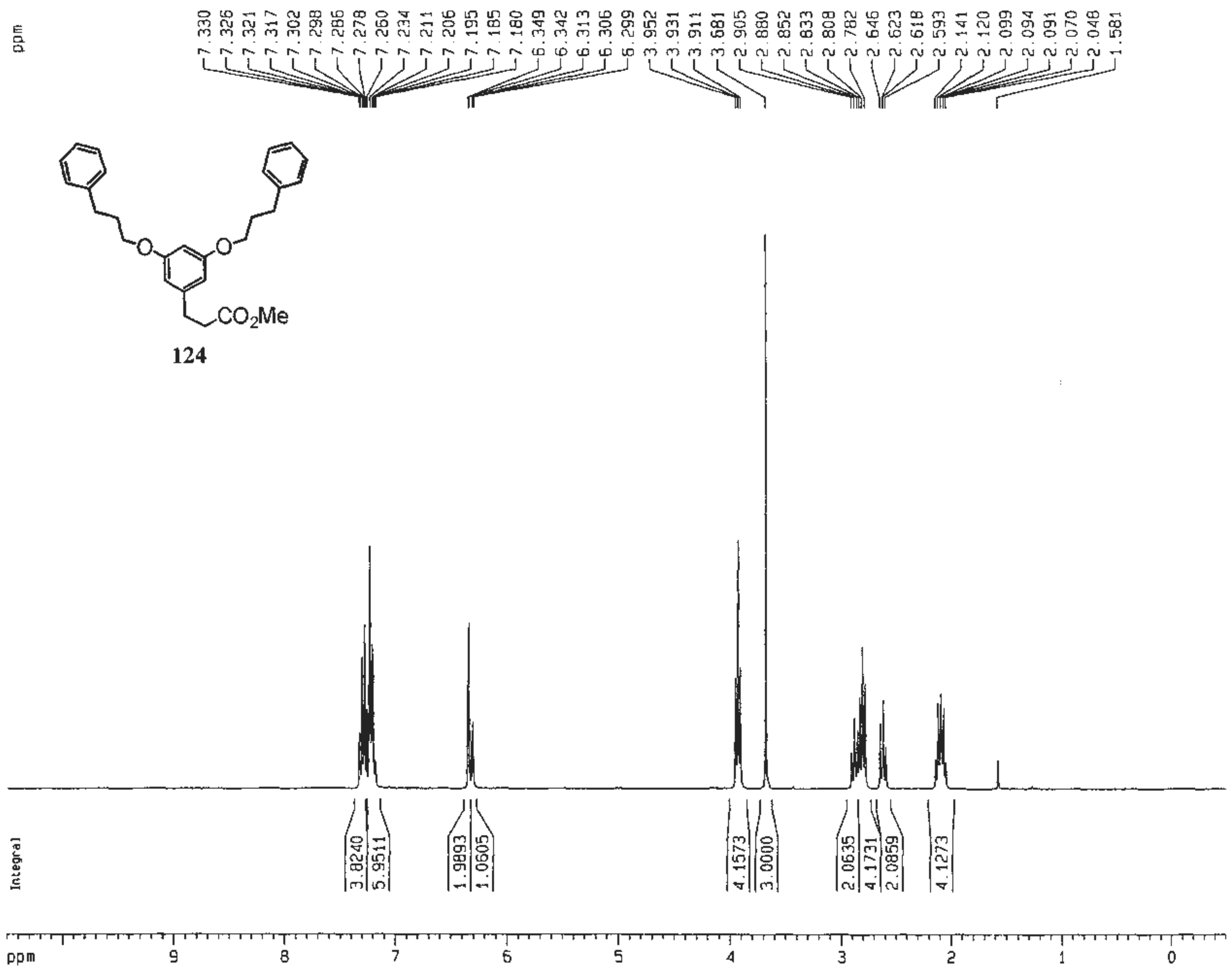
F2 - Acquisition Parameters
Date_          20070508
Time               1.52
INSTRUM          dpx300
PROBHD   5 mm BB0 BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          22675 736 Hz
FIDRES        0.346004 Hz
AQ           1 4451188 sec
RG                 8192
DW               22.050 usec
DE                6 00 usec
TE                 0.0 K
D1          1 00000000 sec
d11         0 03000000 sec
MCREST      0 00000000 sec
MCWRK       0 01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120 00 dB
PL12              19 00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75 4677635 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.98 cm
F1P             200.000 ppm
F1             15093 55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM        720 37408 Hz/cm
```



124

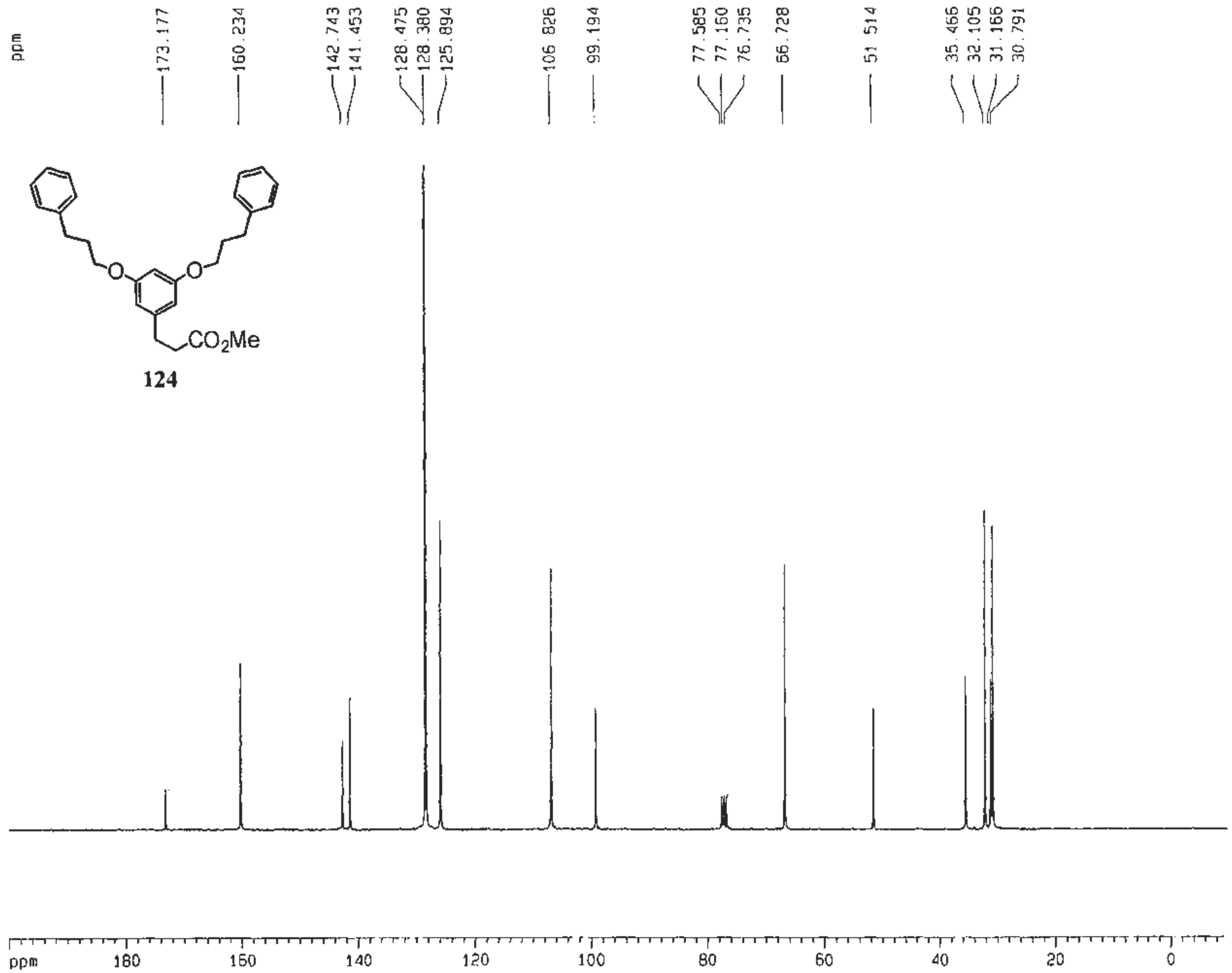
```
Current Data Parameters
NAME           61'-ester
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20050902
Time               15.01
INSTRUM           dpx300
PROBHD   5 mm BB0 BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDC13
NS                    16
DS                     0
SWH             3601.657 Hz
FIDRES          0.219828 Hz
AQ             2.2745588 sec
RG                  71.8
DW               138.825 usec
DE                198.32 usec
TE                 295.2 K
D1            5.00000000 sec
MCREST        0 00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300062 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  9.98 cm
F1P               10.500 ppm
F1               3151 36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```



124

```
Current Data Parameters
NAME          G1'-esterC
EXPNO                  1
PROCNO                 1

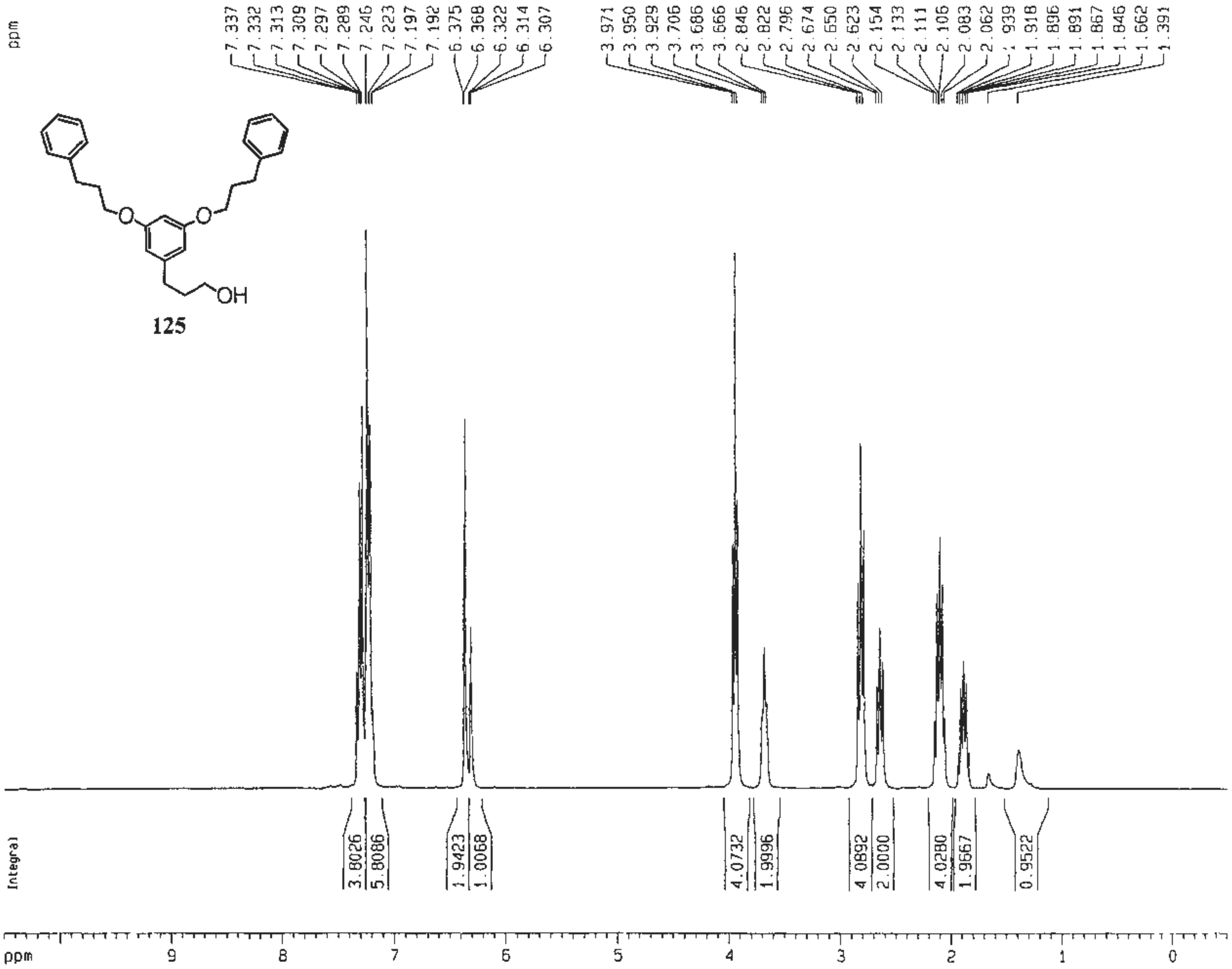
F2 - Acquisition Parameters
Date_           20070925
Time               15.39
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                  8192
DW              22.050 usec
DE                6.00 usec
TE                 0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 3 00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120 00 dB
PL12               19.00 dB
SFO2        300 1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677636 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1 40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.98 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720 37408 Hz/cm
```



125

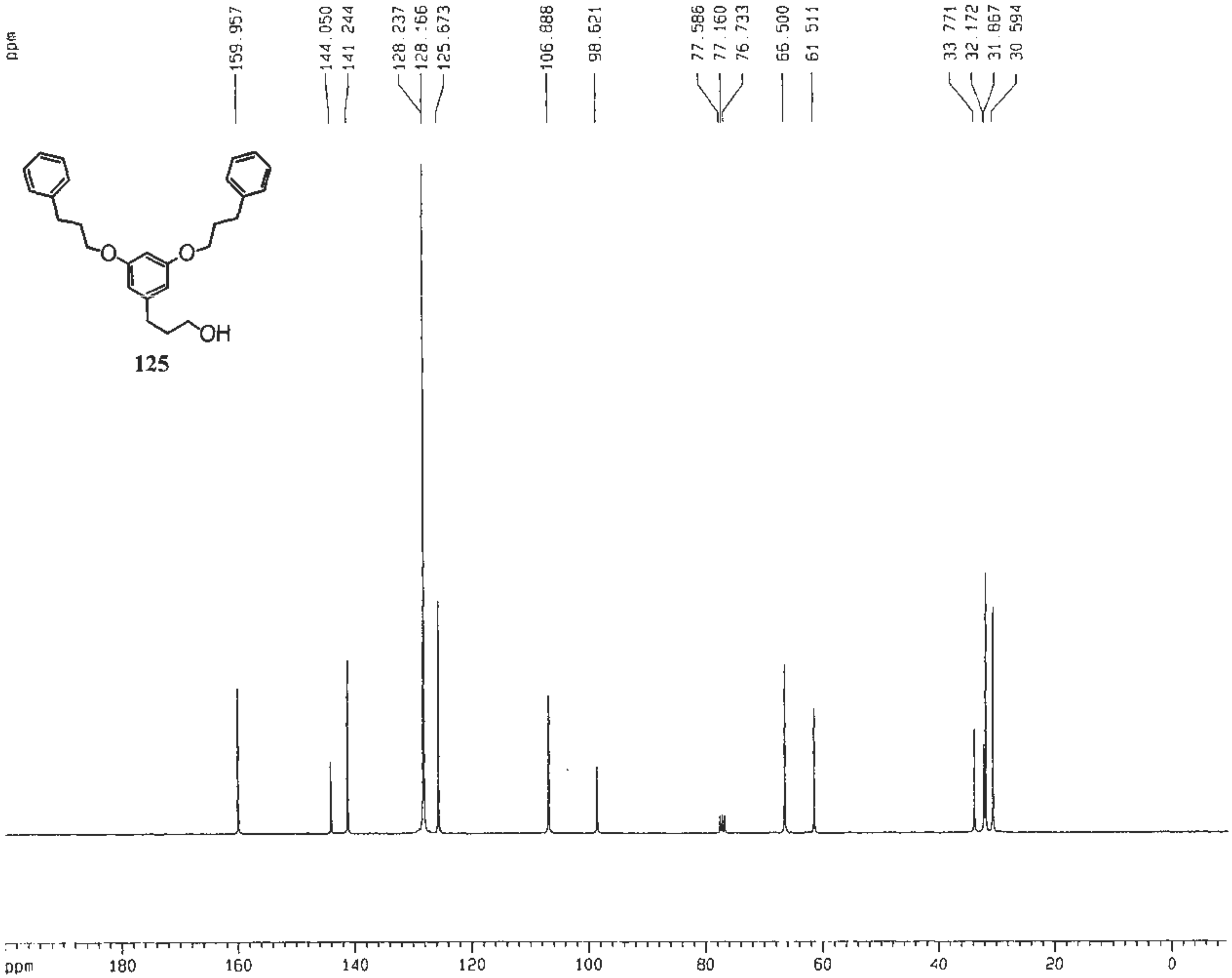
```
Current Data Parameters
NAME             G1'-OH
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070403
Time              17.54
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                 90.5
DW              138.825 usec
DE               198.32 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300059 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



```
Current Data Parameters
NAME            G1'-OHC
EXPNO                 1
PROCNO                1

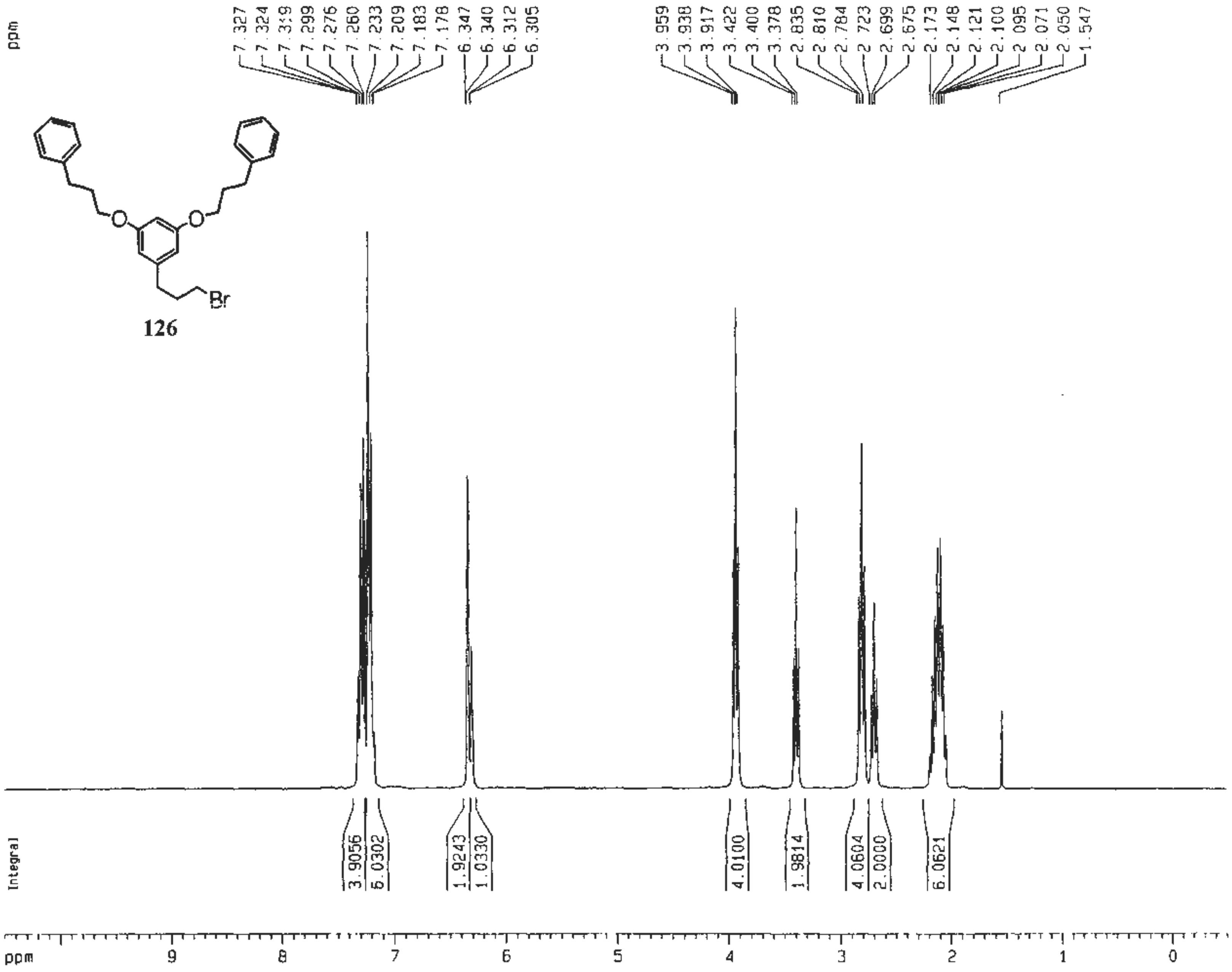
F2 - Acquisition Parameters
Date_          20070410
Time              18 43
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          22675 736 Hz
FIDRES        0 346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22 050 usec
DE                 6.00 usec
TE                  0 0 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0 00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3 00 usec
PL1               -6 00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2       300 1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677962 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1 40

1D NMR plot parameters
CX                22.00 cm
CY                12.00 cm
F1P             200.000 ppm
F1             15093.56 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720 37439 Hz/cm
```



126

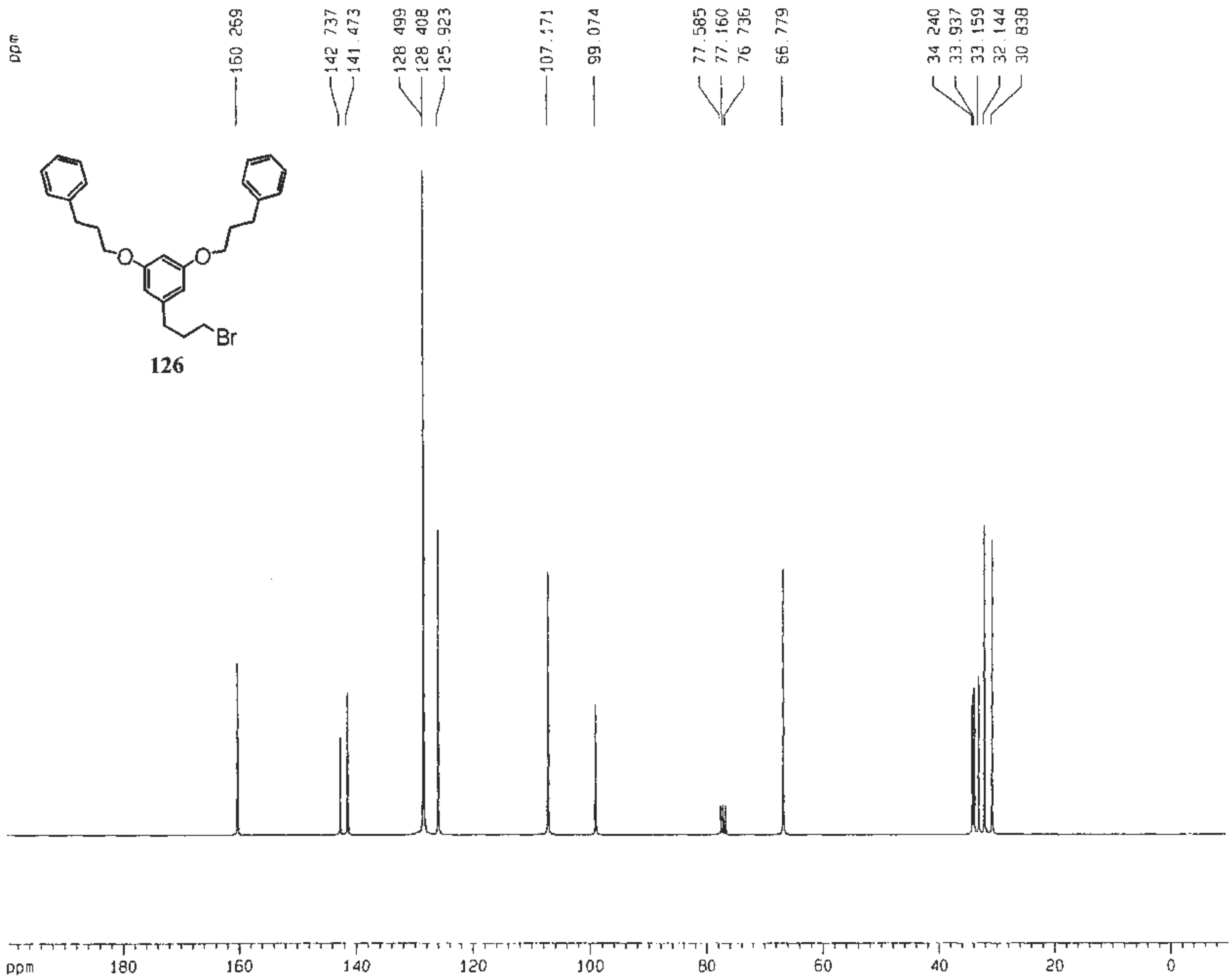
```
Current Data Parameters
NAME             G1'-Br
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070403
Time              18.05
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                228.1
DW              138.825 usec
DE               198.32 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWORK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300064 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                 9.98 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



126

```
Current Data Parameters
NAME              G1'-BrC
EXPNO                   1
PROCNO                  1

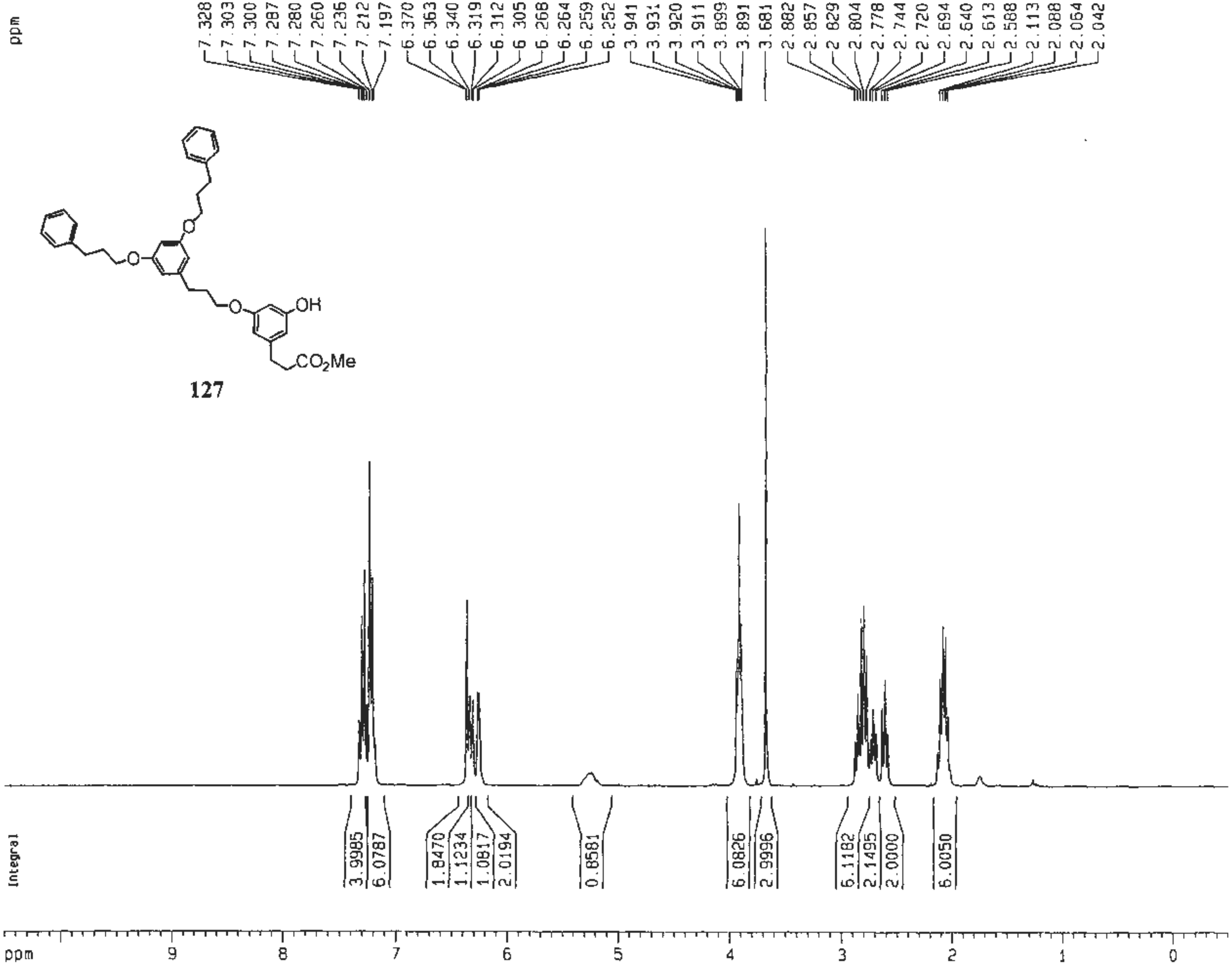
F2 - Acquisition Parameters
Date_            20070407
Time                 0 42
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                  16428
DS                      0
SWH            22675 736 Hz
FIDRES          0 346004 Hz
AQ             1 4451188 sec
RG                   8192
DW                 22 050 usec
DE                   6.00 usec
TE                    0.0 K
D1             1.00000000 sec
d11            0 03000000 sec
MCREST         0.00000000 sec
MCWRK          0 01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3 00 usec
PL1                 -6.00 dB
SFO1          75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100 00 usec
PL2                120 00 dB
PL12                19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF            75 4677682 MHz
WDW                    EM
SSB                     0
LB                   3 00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.00 cm
F1P               200 000 ppm
F1               15093 55 Hz
F2P               -10 000 ppm
F2                -754 68 Hz
PPMCM             9 54545 ppm/cm
HZCM            720 37408 Hz/cm
```


127

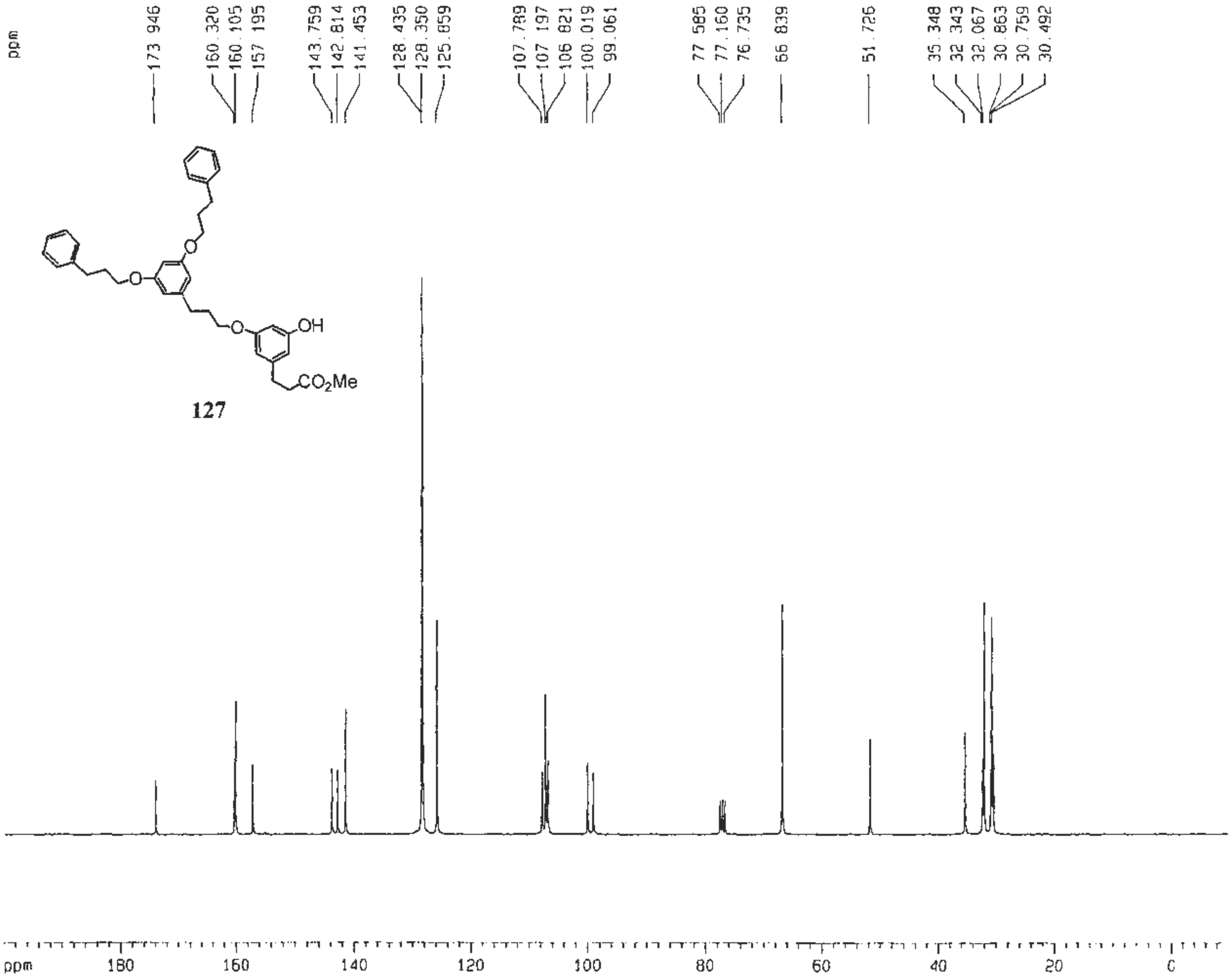
```
Current Data Parameters
NAME          G1'-M-ester
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080515
Time                21.18
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                    128
DW                138.825 usec
DE                 198.32 usec
TE                  294.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300059 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  10.00 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



127

```
Current Data Parameters
NAME          G1'-M-esterC
EXPNO                    1
PROCNO                   1

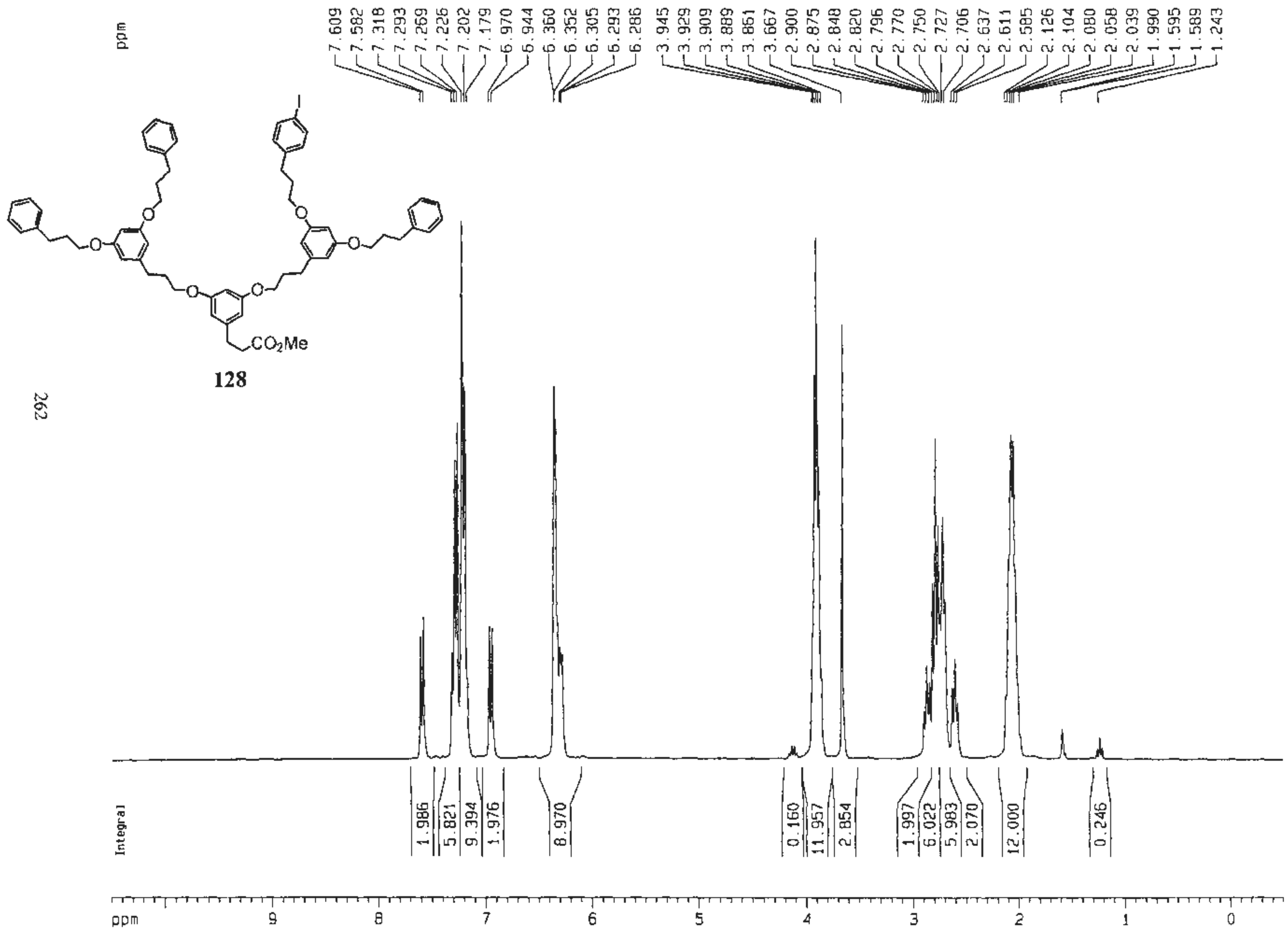
F2 - Acquisition Parameters
Date_             20071112
Time                 21.29
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                    1400
DS                       0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                    8192
DW                  22.050 usec
DE                    6.00 usec
TE                     0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    3.00 usec
PL1                  -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF              75.4677690 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   10.00 cm
F1P                200.000 ppm
F1                15093.55 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM              9.54545 ppm/cm
HZCM             720.37408 Hz/cm
```



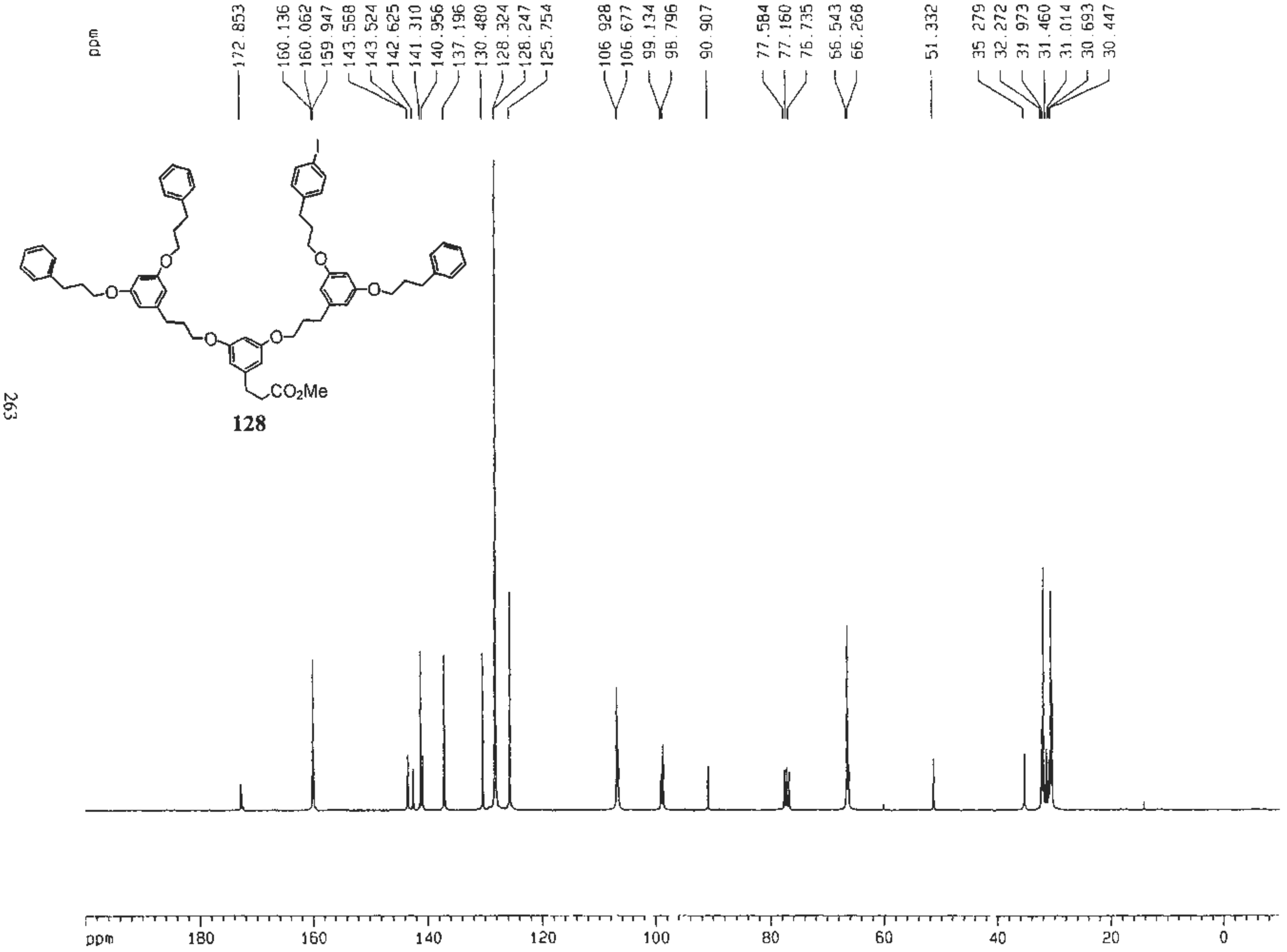
```
Current Data Parameters
NAME        I-G2'-ester
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080430
Time              16.47
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                 80.6
DW              138.825 usec
DE               198.32 usec
TE                295.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1    .  300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300136 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00 .

1D NMR plot parameters
CX                22.00 cm
CY                10.03 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



128

```
Current Data Parameters
NAME       J-G2'-esterC
EXPNO              1
PROCNO             1

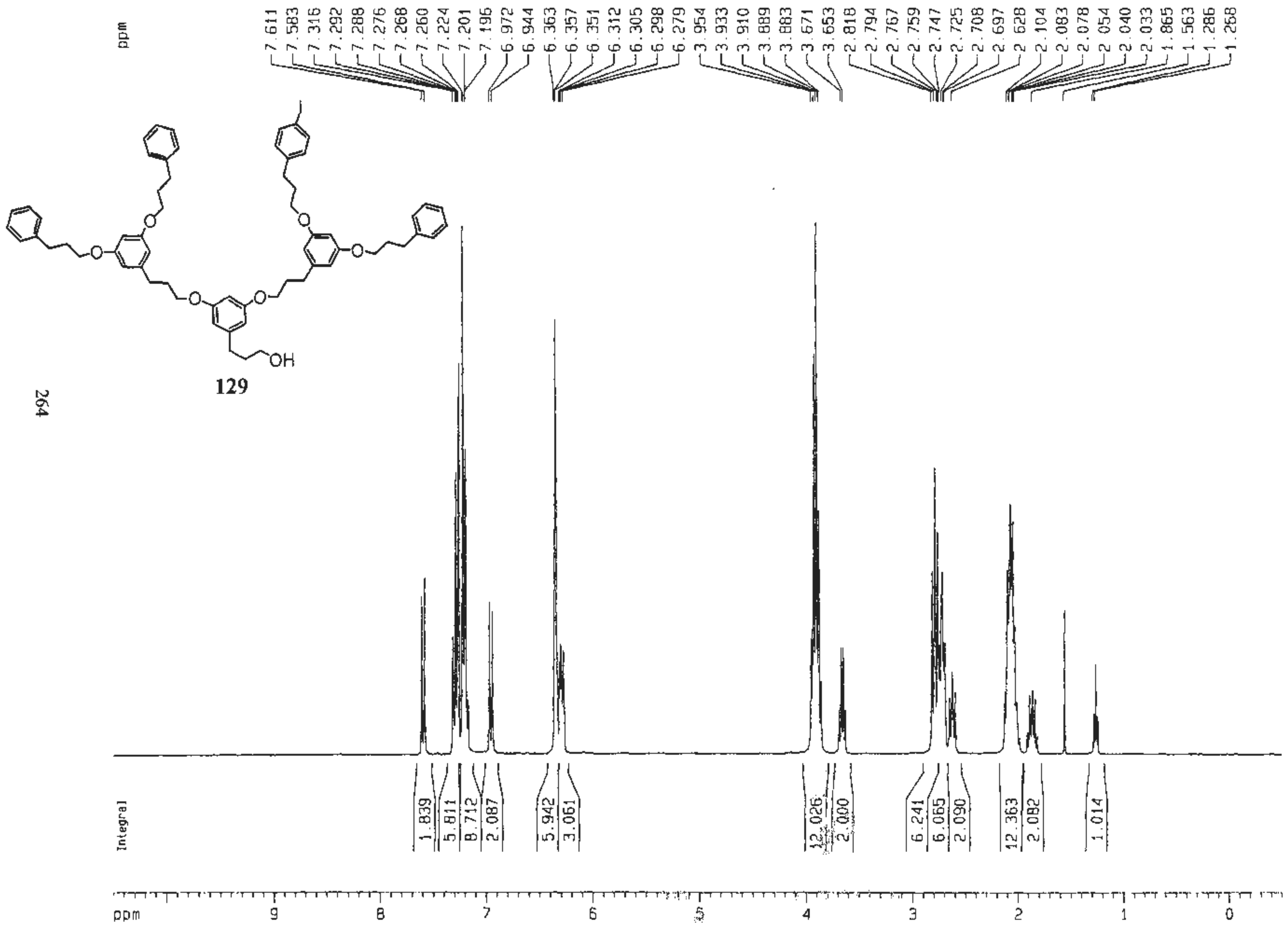
F2 - Acquisition Parameters
Date_          20080428
Time               5.40
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  700
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677840 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.97 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37421 Hz/cm
```



129

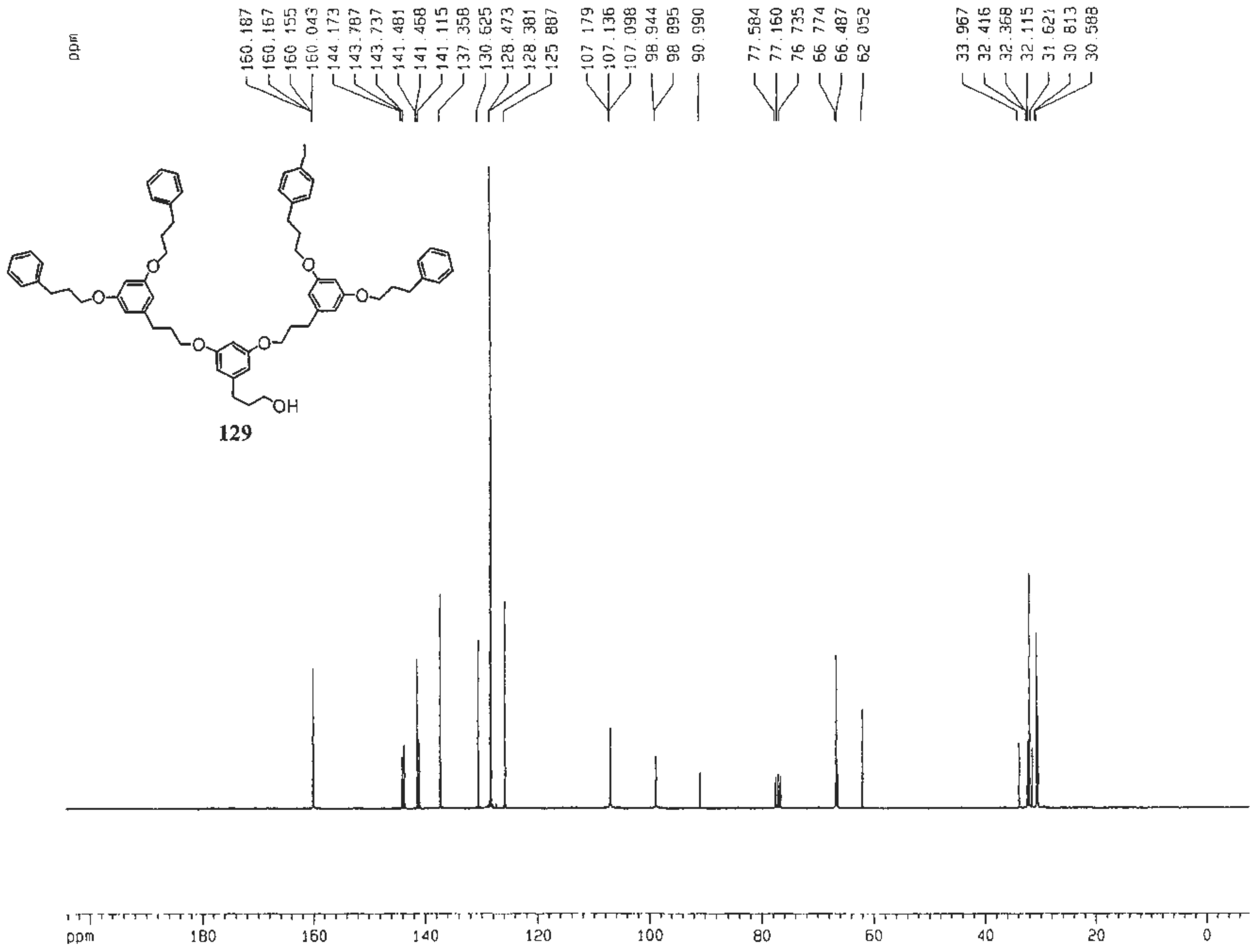
```
Current Data Parameters
NAME             I-G2'-OH
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071203
Time                22 06
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                  228.1
DW                138.825 usec
DE                 198 32 usec
TE                    0 0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300064 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9.98 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



129

```
Current Data Parameters
NAME          I-G2'-OHC
EXPNO                 1
PROCNO                1

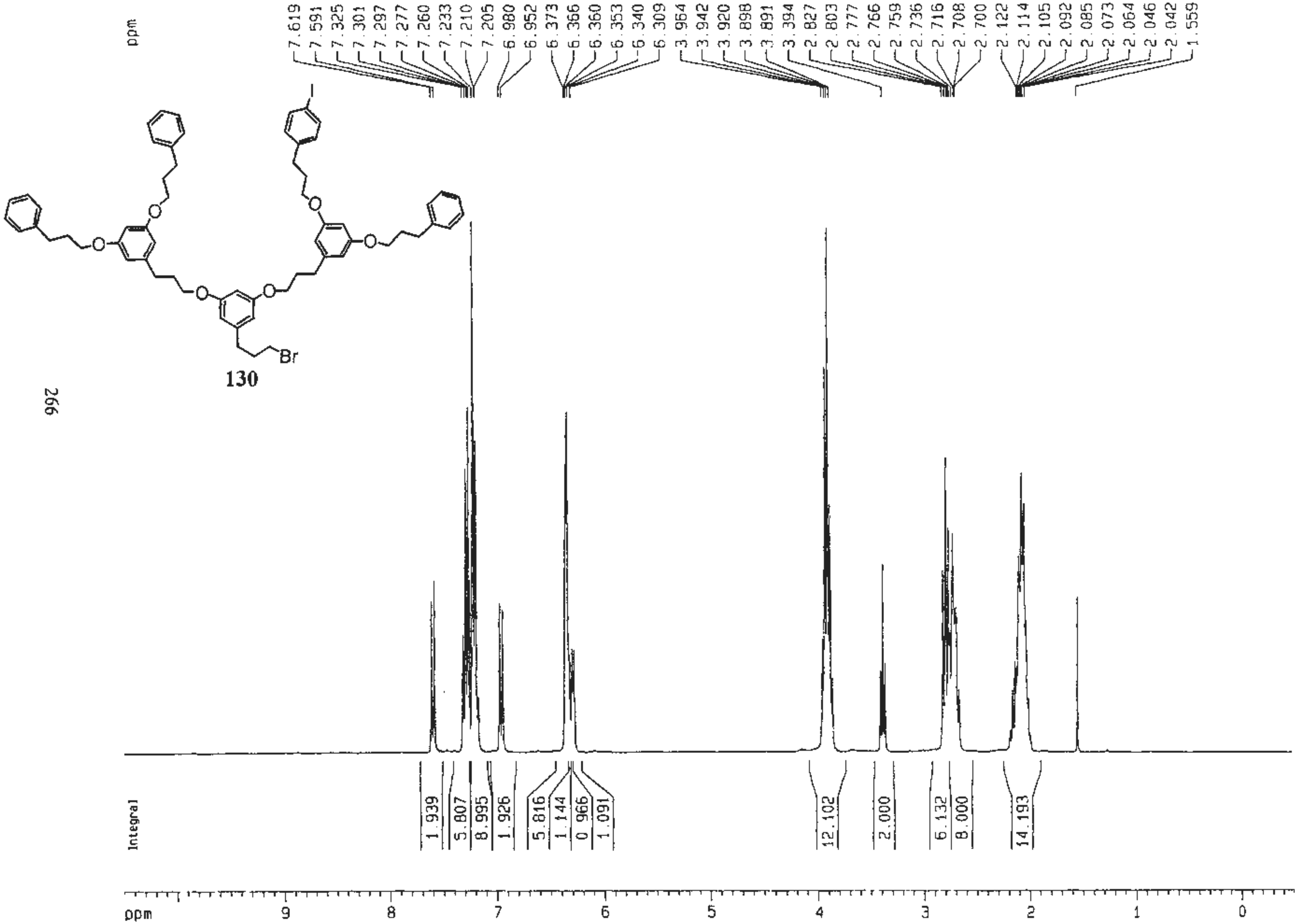
F2 - Acquisition Parameters
Date_          20070508
Time               2.17
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1 4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1           1.00000000 sec
d11          0 03000000 sec
MCREST       0.00000000 sec
MCWRK        0 01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1        75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120 00 dB
PL12              19 00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677669 MHz
WDW                  EM
SSB                   0
LB                 0 30 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.91 cm
F1P             204 375 ppm
F1             15423.76 Hz
F2P              -7 698 ppm
F2              -580.97 Hz
PPMCM           9.63972 ppm/cm
HZCM          727 48779 Hz/cm
```



130

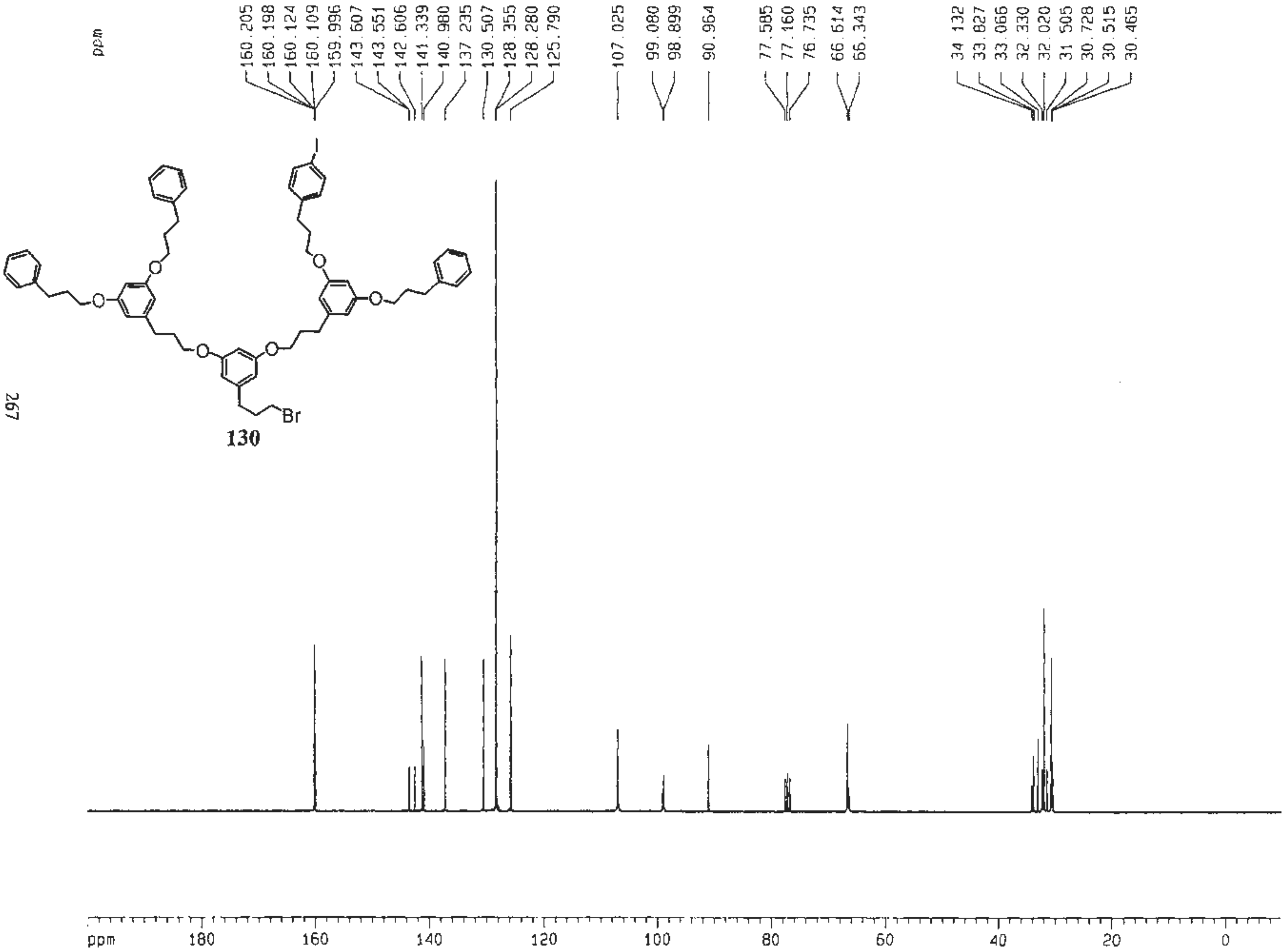
```
Current Data Parameters
NAME          I-G2'-Br
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20071206
Time             12.42
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                  32
DS                   0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AQ           2 2745588 sec
RG                 128
DW             138.825 usec
DE              198.32 usec
TE                 0.0 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1              -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300063 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22 00 cm
CY                9.94 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150 07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



130

```
Current Data Parameters
NAME            I-G2'-BrC
EXPNO                   1
PROCNO                  1

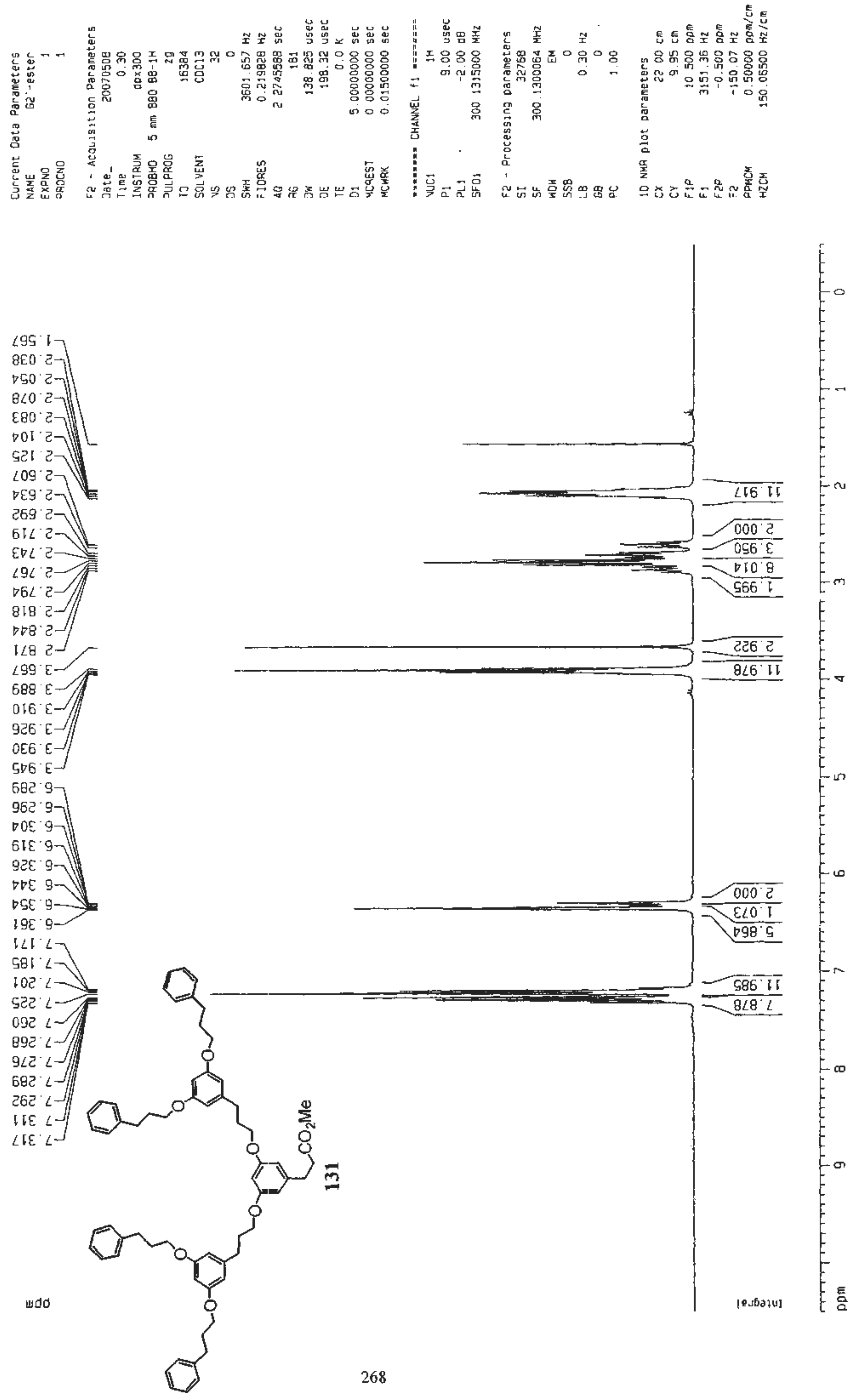
F2 - Acquisition Parameters
Date_            20071206
Time                12 56
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDC13
NS                    800
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                    0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1          75.4745111 MHz

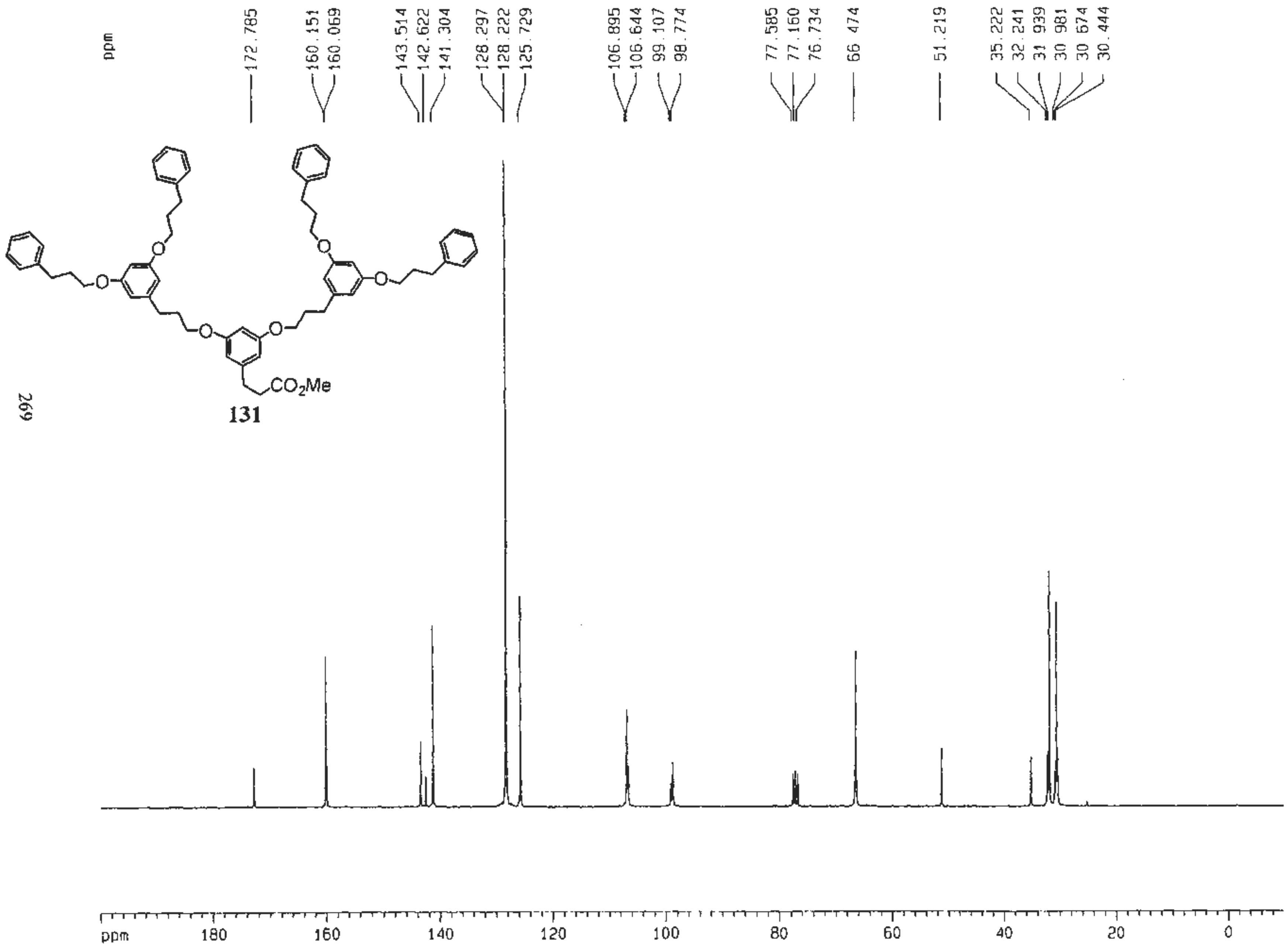
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters.
SI                  65536
SF             75.4677869 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.00 cm
F1P               200.000 ppm
F1               15093.56 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37427 Hz/cm
```


Current Data Parameters
NAME G2'-ester
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070508
Time 0.30
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 3601.657 Hz
FIDRES 0.219828 Hz
AQ 2.2745588 sec
RG 181
DW 138.825 usec
DE 198.32 usec
TE 0.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -2.00 dB
SFO1 300.1315000 MHz
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 9.95 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm
ppm
7.317
7.311
7.292
7.289
7.276
7.268
7.260
7.225
7.201
7.185
7.171
6.361
6.354
6.344
6.326
6.319
6.304
6.296
6.289
3.945
3.930
3.926
3.910
3.889
3.667
2.871
2.844
2.818
2.794
2.767
2.743
2.719
2.692
2.634
2.607
2.125
2.104
2.083
2.078
2.054
2.038
1.567
Integral
7.878
11.985
5.864
1.073
2.000
11.978
2.922
1.995
8.014
3.950
2.000
11.917
ppm
9
8
7
6
5
4
3
2
1
0
O
O
O
O
O
O
CO2Me

131



131

```
Current Data Parameters
NAME           G2'-esterC
EXPNO                   1
PROCNO                  1

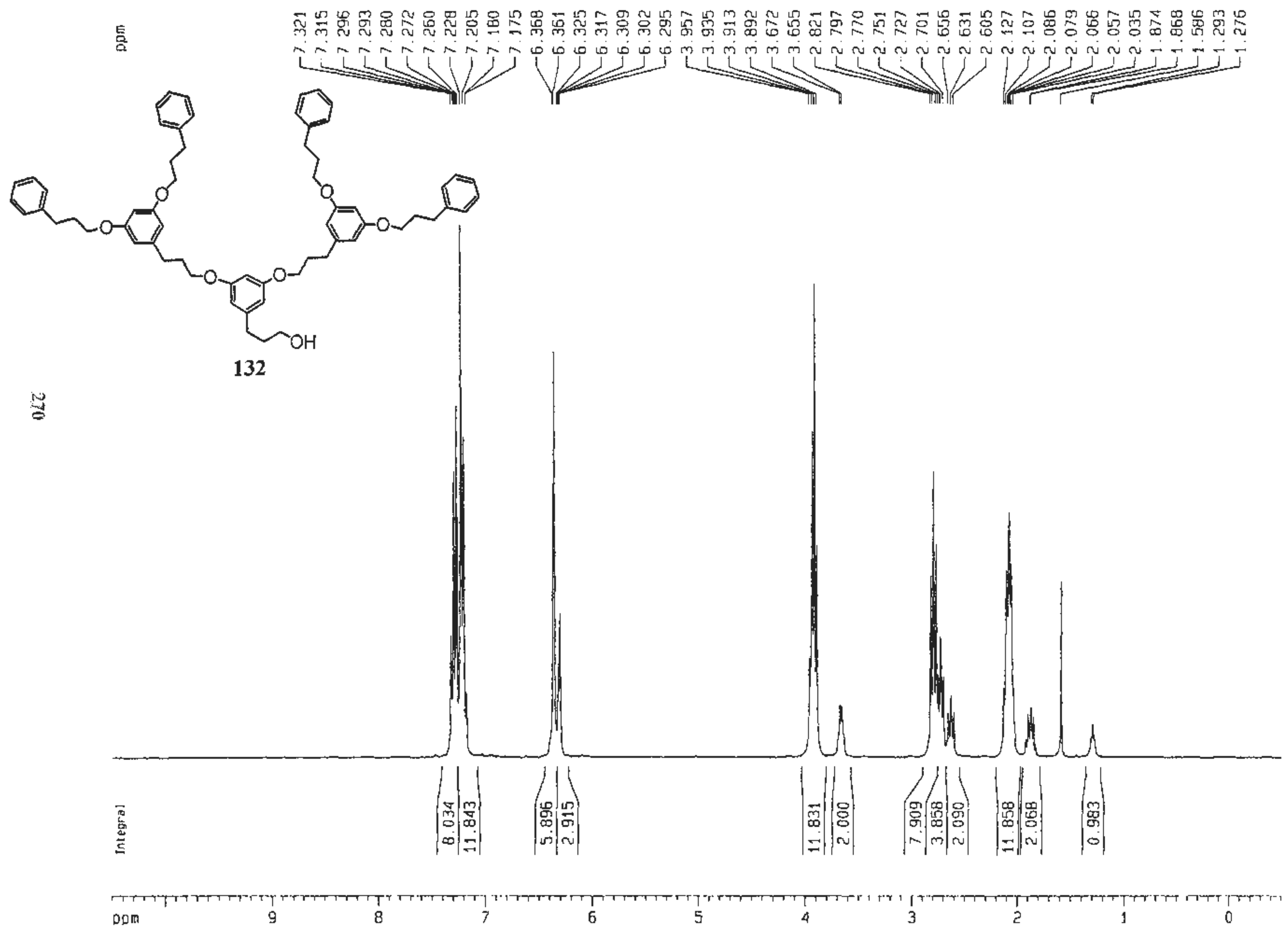
F2 - Acquisition Parameters
Date_            20080423
Time                16.51
INSTRUM            dpx300
PROBHD     5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                    274
DS                      0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                   8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677910 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.03 cm
F1P               200.000 ppm
F1               15093.56 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37427 Hz/cm
```



132

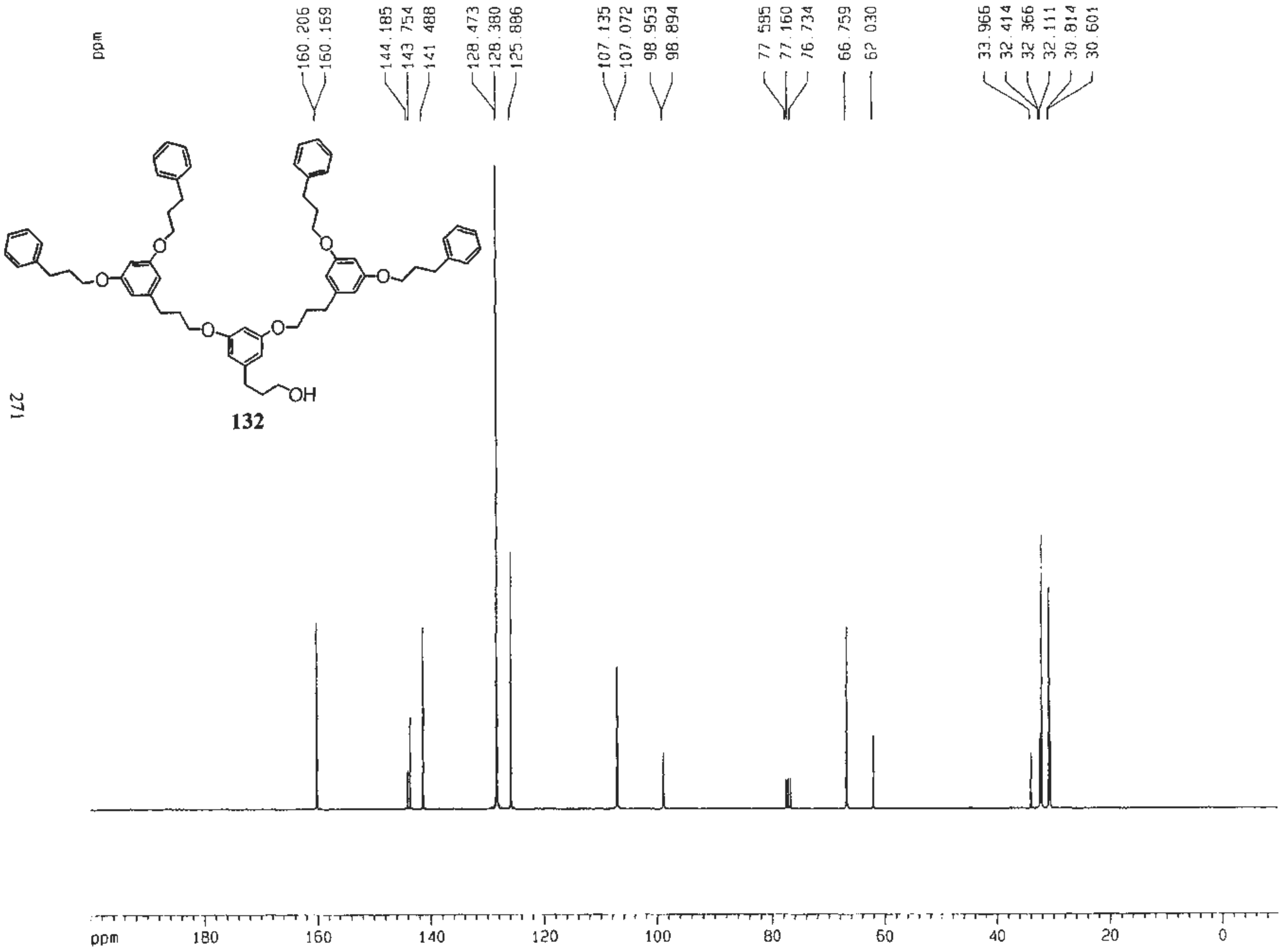
```
Current Data Parameters
NAME            G2'-OH
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20070508
Time              0.52
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               32768
SOLVENT          CDCl3
NS                  17
DS                   0
SWH           3601.657 Hz
FIDRES        0.109914 Hz
AQ           4.5490675 sec
RG                 128
DW             138.825 usec
DE              198 32 usec
TE                 0 0 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                5.00 usec
PL1              -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300065 MHz
WDW                 EM
SSB                  0
LB                0 30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY                9.98 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



132

```
Current Data Parameters
NAME            G2'-OHC
EXPNO                 1
PROCNO                1

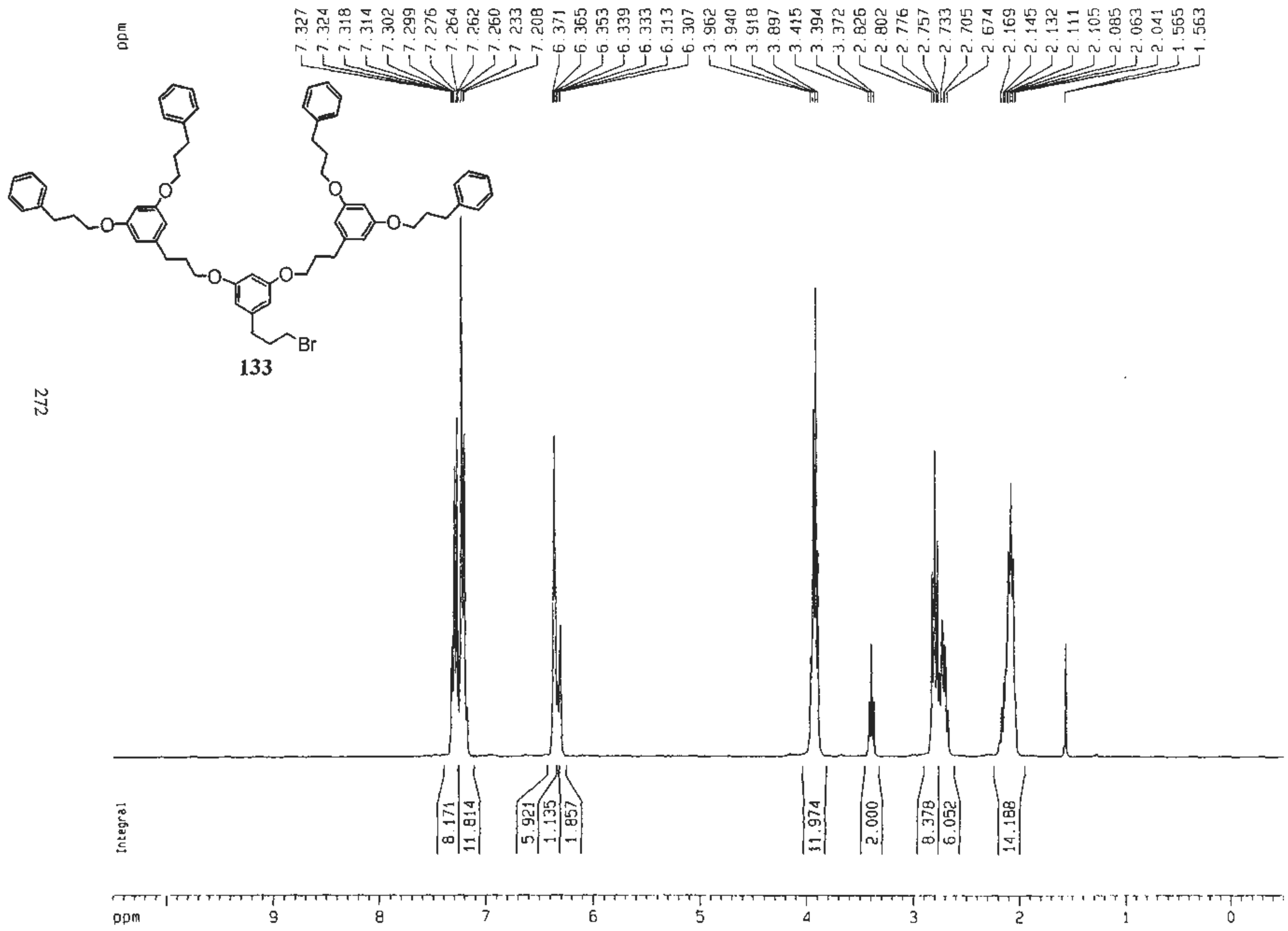
F2 - Acquisition Parameters
Date_          20070508
Time               2.40
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.95 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37408 Hz/cm
```



133

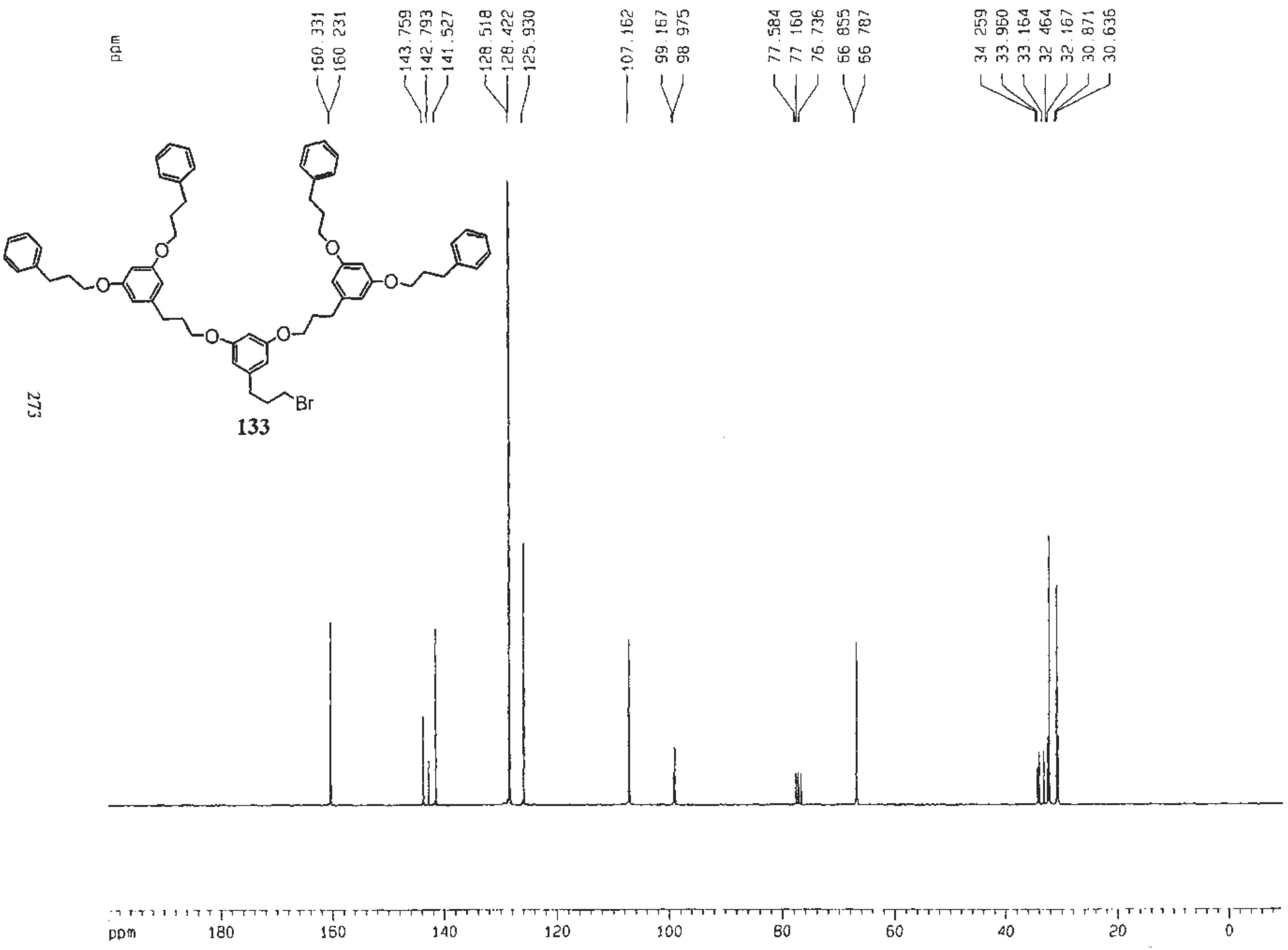
```
Current Data Parameters
NAME            G2'-Br
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20070508
Time              1.03
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zg
TD               16384
SOLVENT          CDCl3
NS                  32
DS                   0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AQ           2.2745588 sec
RG               161.3
DW             138.825 usec
DE              198.32 usec
TE                 0.0 K
D1          5.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1              -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI               32768
SF         300.1300057 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
CY               10.11 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM          0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



133

```
Current Data Parameters
NAME           G2'-BrC
EXPNO                1
PROCNO               1

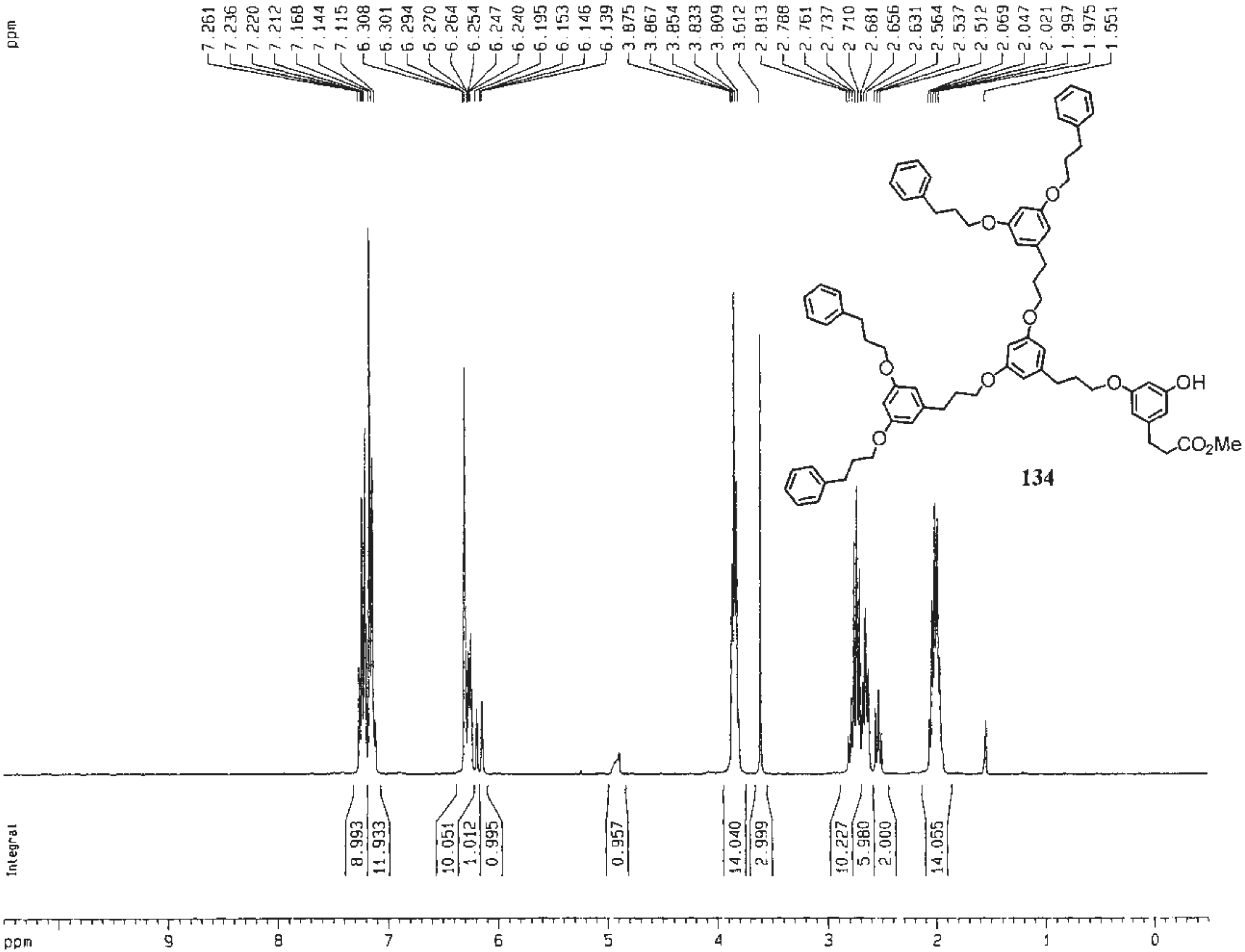
F2 - Acquisition Parameters
Date_         20070508
Time              2.57
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG           zgdc
TD               65536
SOLVENT          CDCl3
NS                 400
DS                   0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                8192
DW              22.050 usec
DE                6.00 usec
TE                 0.0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                3.00 usec
PL1              -5.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI               65536
SF          75.4677651 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX               22.00 cm
CY               12.01 cm
F1P            200.000 ppm
F1            15093.55 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM          9.54545 ppm/cm
HZCM         720.37408 Hz/cm
```



134

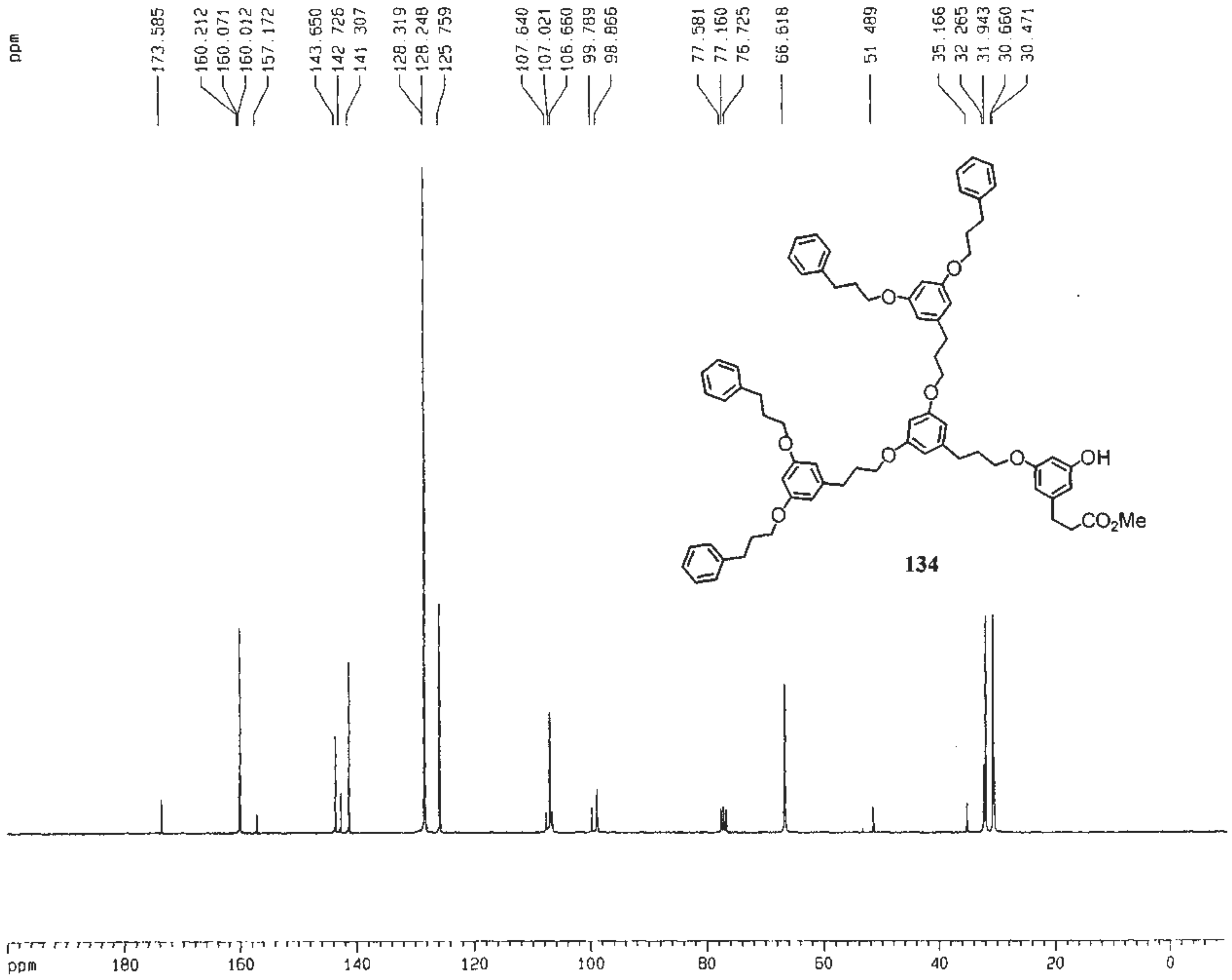
```
Current Data Parameters
NAME          G2'-M-ester
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080423
Time                16 46
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              3601.657 Hz
FIDRES           0 219828 Hz
AQ              2 2745588 sec
RG                  203 2
DW                138.825 usec
DE                 198.32 usec
TE                  296.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300240 MHz
WDW                    EM
SSB                     0
LB                   0 30 Hz
GB                      0
PC                   1 00

1D NMR plot parameters
CX                  22 00 cm
CY                   9 97 cm
F1P                10 500 ppm
F1                3151 37 Hz
F2P                -0.500 ppm
F2                -150 07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06502 Hz/cm
```



134

```
Current Data Parameters
NAME        G2'-M-esterC
EXPNO                  1
PROCNO                 1

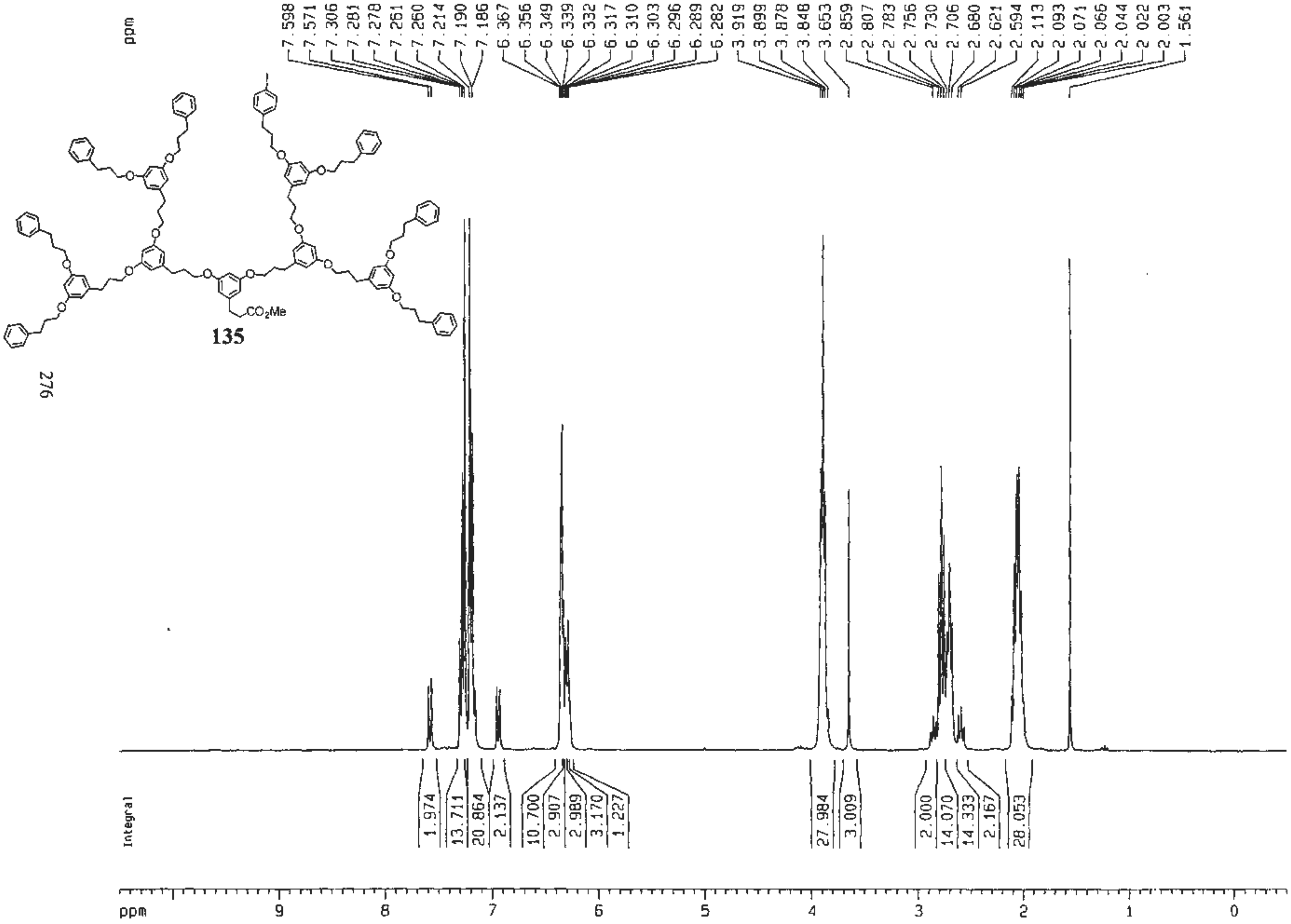
F2 - Acquisition Parameters
Date_           20080423
Time               16.20
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   465
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677862 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.00 cm
F1P              200.000 ppm
F1              15093.56 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37427 Hz/cm
```



135

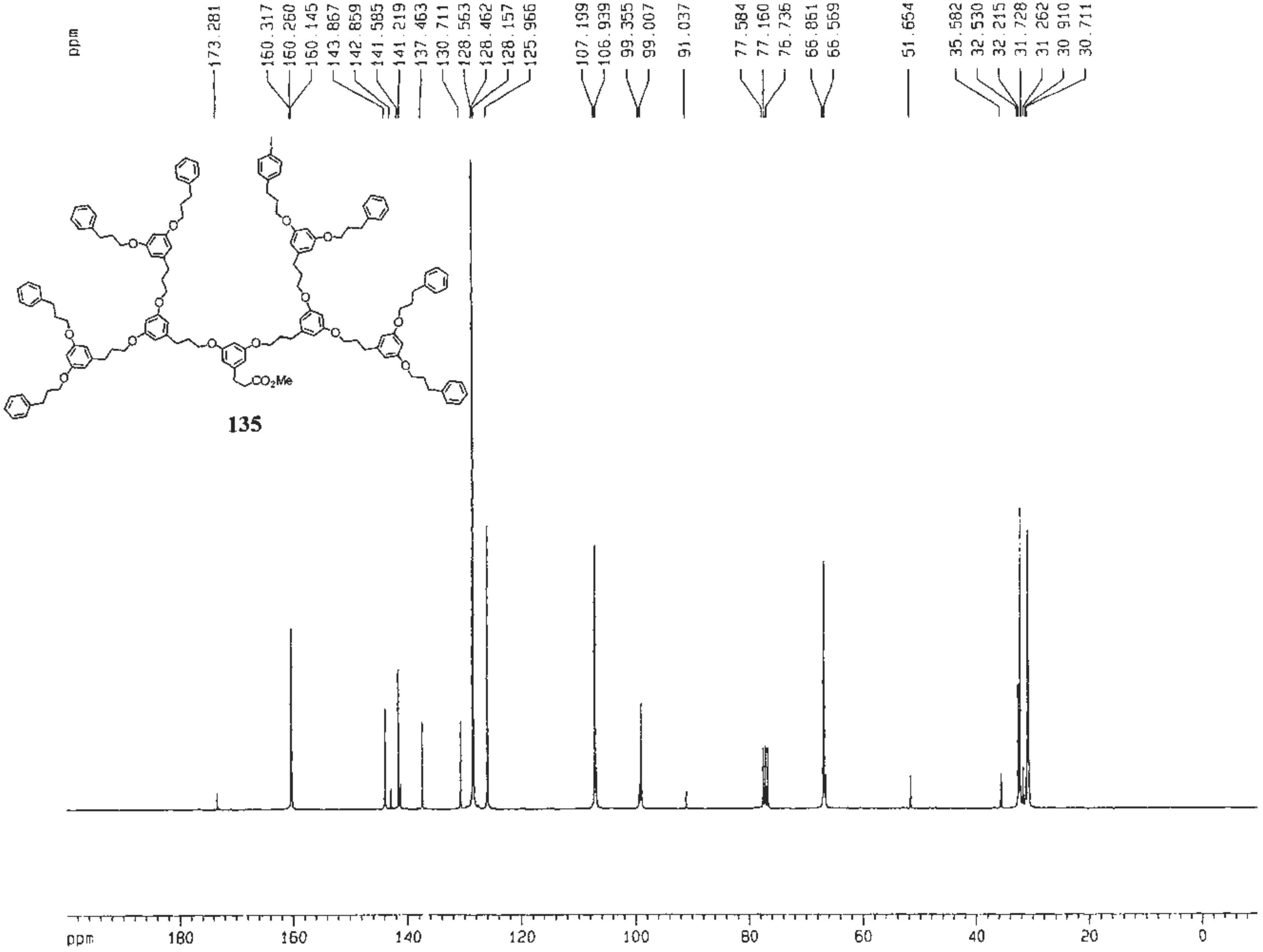
```
Current Data Parameters
NAME        I-G3'-ester
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070508
Time               1.18
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219826 Hz
AQ            2.2745588 sec
RG                287.4
DW              138.825 usec
DE               198.32 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300060 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                 9.98 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



135

```
Current Data Parameters
NAME          I-G3'-esterC
EXPNO                    1
PROCNO                   1

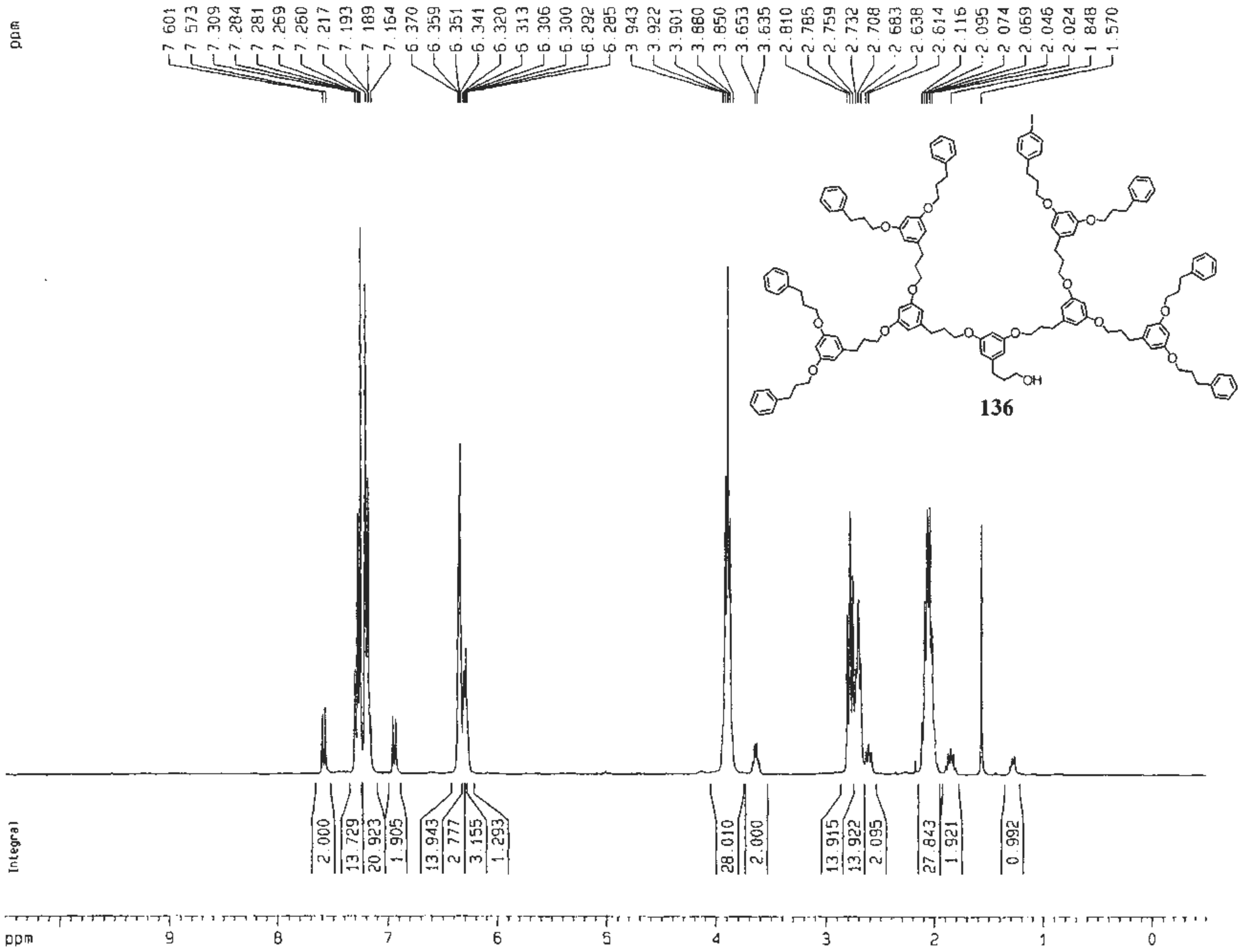
F2 - Acquisition Parameters
Date_             20070508
Time                  4.50
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                    5000
DS                       0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451168 sec
RG                    8192
DW                  22.050 usec
DE                    6.00 usec
TE                     0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    3.00 usec
PL1                  -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF              75.4677554 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   12.02 cm
F1P                200.000 ppm
F1                15093.55 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM              9.54545 ppm/cm
HZCM             720.37402 Hz/cm
```



136

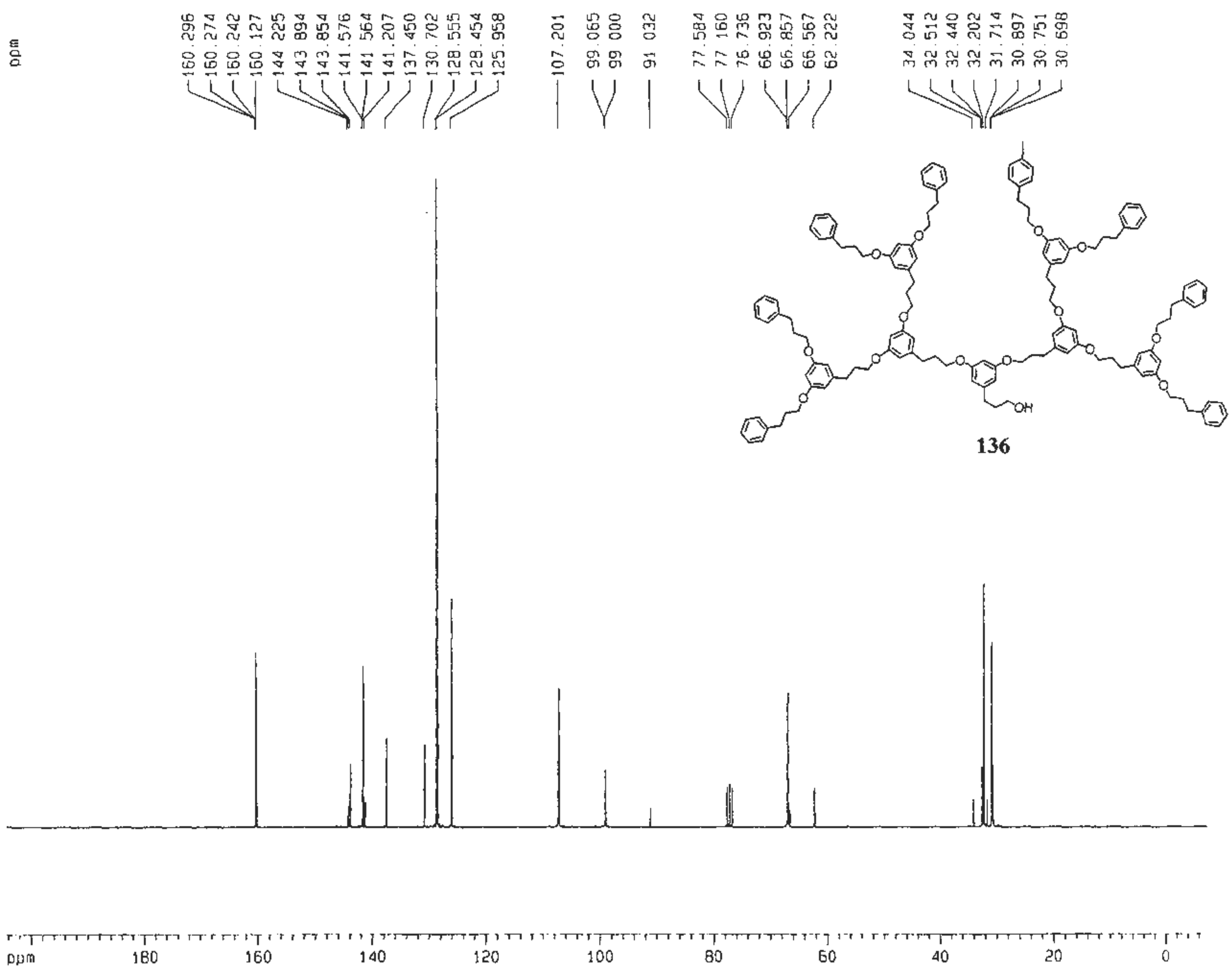
```
Current Data Parameters
NAME           I-G3'-OH
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070508
Time               1.35
INSTRUM          dpx300
PROBHD   5 mm BB0 BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                203.2
DW              138.825 usec
DE               198.32 usec
TE                  0.0 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300063 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



136

```
Current Data Parameters
NAME          I-G3'-OHC
EXPNO                 1
PROCNO                1

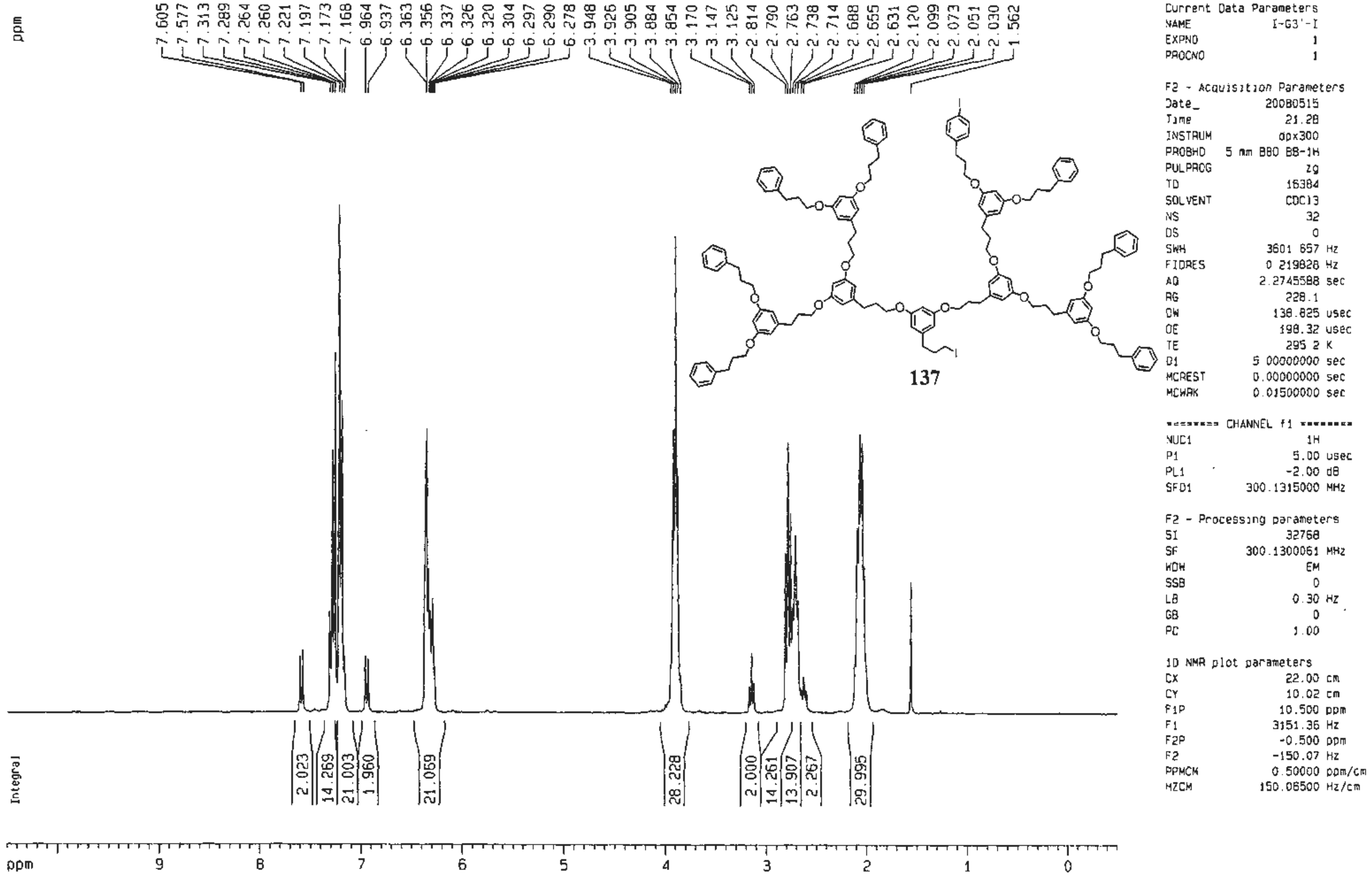
F2 - Acquisition Parameters
Date_          20070508
Time               3.23
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                 1600
DS                    0
SWH          22675 736 Hz
FIDRES        0 346004 Hz
AQ           1 4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0 0 K
D1           1 00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0 01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1        75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100 00 usec
PL2              120.00 dB
PL12              19 00 dB
SFO2      300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75 4677569 MHz
WDW                  EM
SSB                   0
LB                 0.40 Hz
GB                    0
PC                 1 40

1D NMR plot parameters
CX                22.00 cm
CY                12 02 cm
F1P             204 375 ppm
F1             15423.75 Hz
F2P              -7 698 ppm
F2              -580 97 Hz
PPMCM         9 63972 ppm/cm
HZCM        727.48773 Hz/cm
```



137

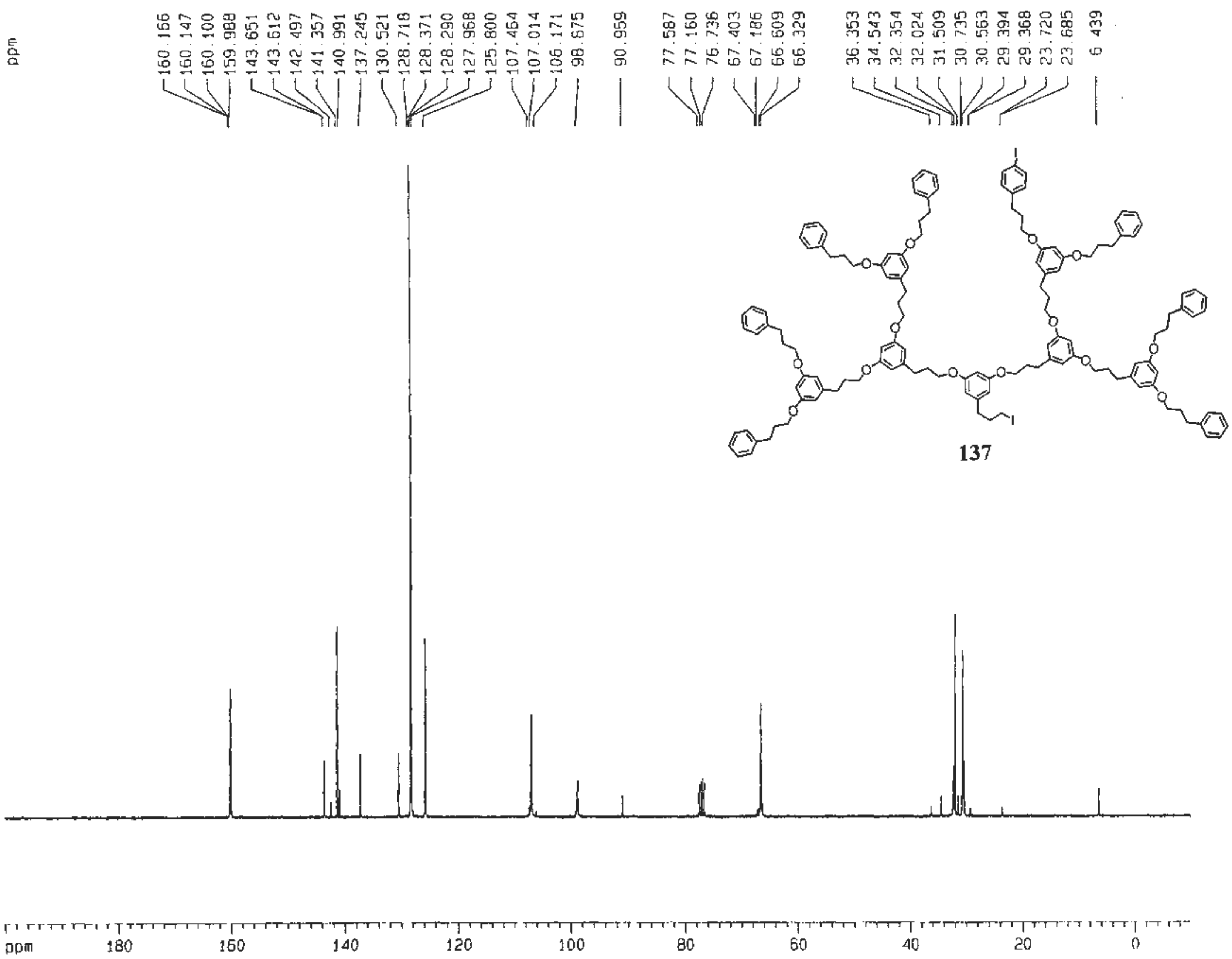
```
Current Data Parameters
NAME            I-G3'-I
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080515
Time              21.28
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH          3601.657 Hz
FIDRES       0.219828 Hz
AQ          2.2745588 sec
RG                228.1
DW           138.825 usec
DE            198.32 usec
TE              295.2 K
D1        5.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1              5.00 usec
PL1             -2.00 dB
SFD1     300.1315000 MHz

F2 - Processing parameters
SI                32768
SF       300.1300061 MHz
WDW                  EM
SSB                   0
LB              0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX             22.00 cm
CY             10.02 cm
F1P          10.500 ppm
F1           3151.36 Hz
F2P          -0.500 ppm
F2           -150.07 Hz
PPMCM     0.50000 ppm/cm
HZCM    150.06500 Hz/cm
```



137

```
Current Data Parameters
NAME          I-G3'-IC
EXPNO                1
PROCNO               1

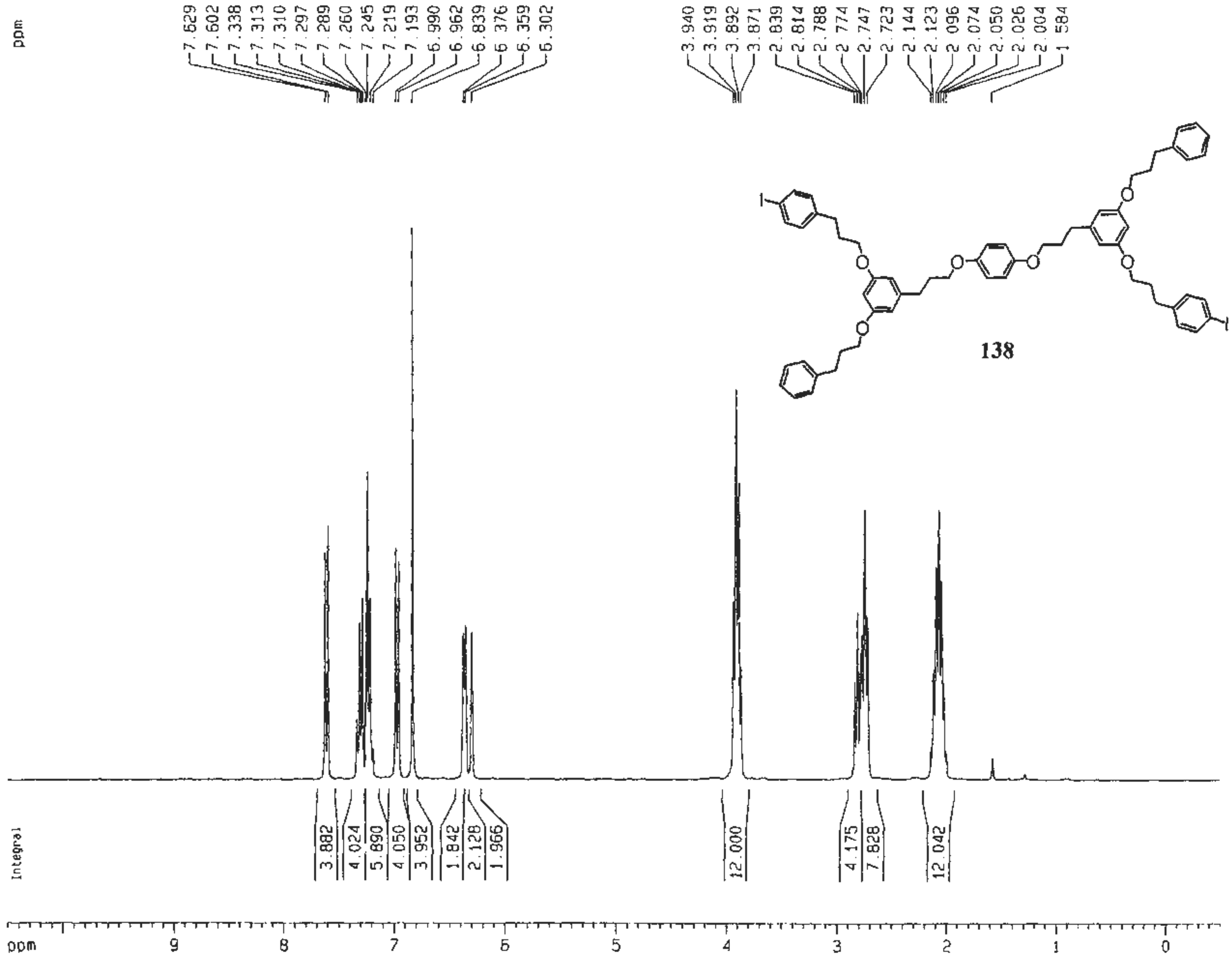
F2 - Acquisition Parameters
Date_         20080228
Time             12.23
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG           zgdc
TD               65536
SOLVENT          CDCl3
NS                1000
DS                   0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                8192
DW              22.050 usec
DE                6.00 usec
TE               298.2 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                3.00 usec
PL1              -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI               65536
SF          75.4677851 MHz
WDW                 EM
SSB                  0
LB                0.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX               22.00 cm
CY               12.02 cm
F1P            200.000 ppm
F1            15093.56 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM          9.54545 ppm/cm
HZCM         720.37427 Hz/cm
```



138

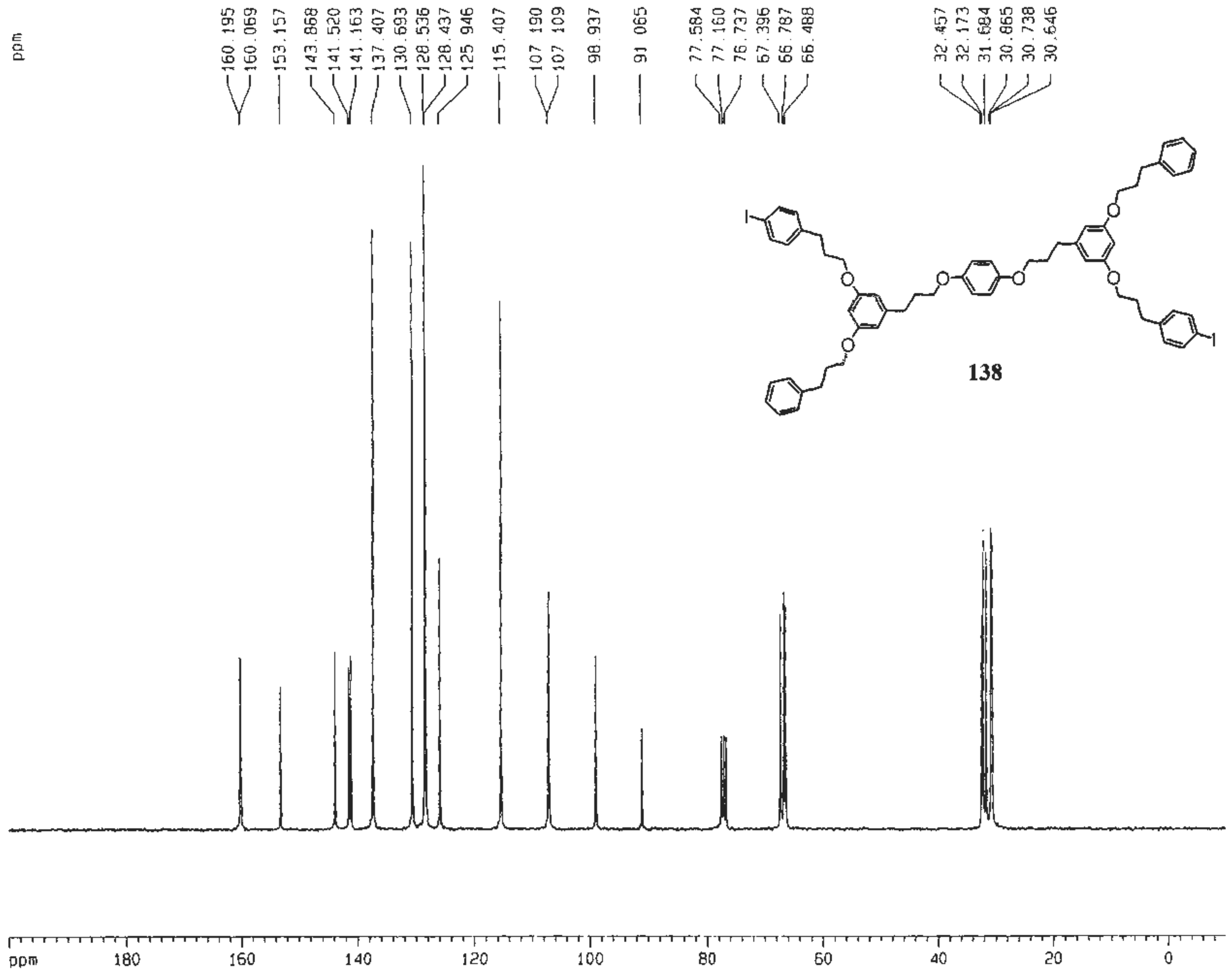
```
Current Data Parameters
NAME          I-G1`-den
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080429
Time              18.22
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                  114
DW              138.825 usec
DE               198.32 usec
TE                296.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300061 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                 9.98 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



138

```
Current Data Parameters
NAME          I-G1'-denC
EXPNO                  1
PROCNO                 1

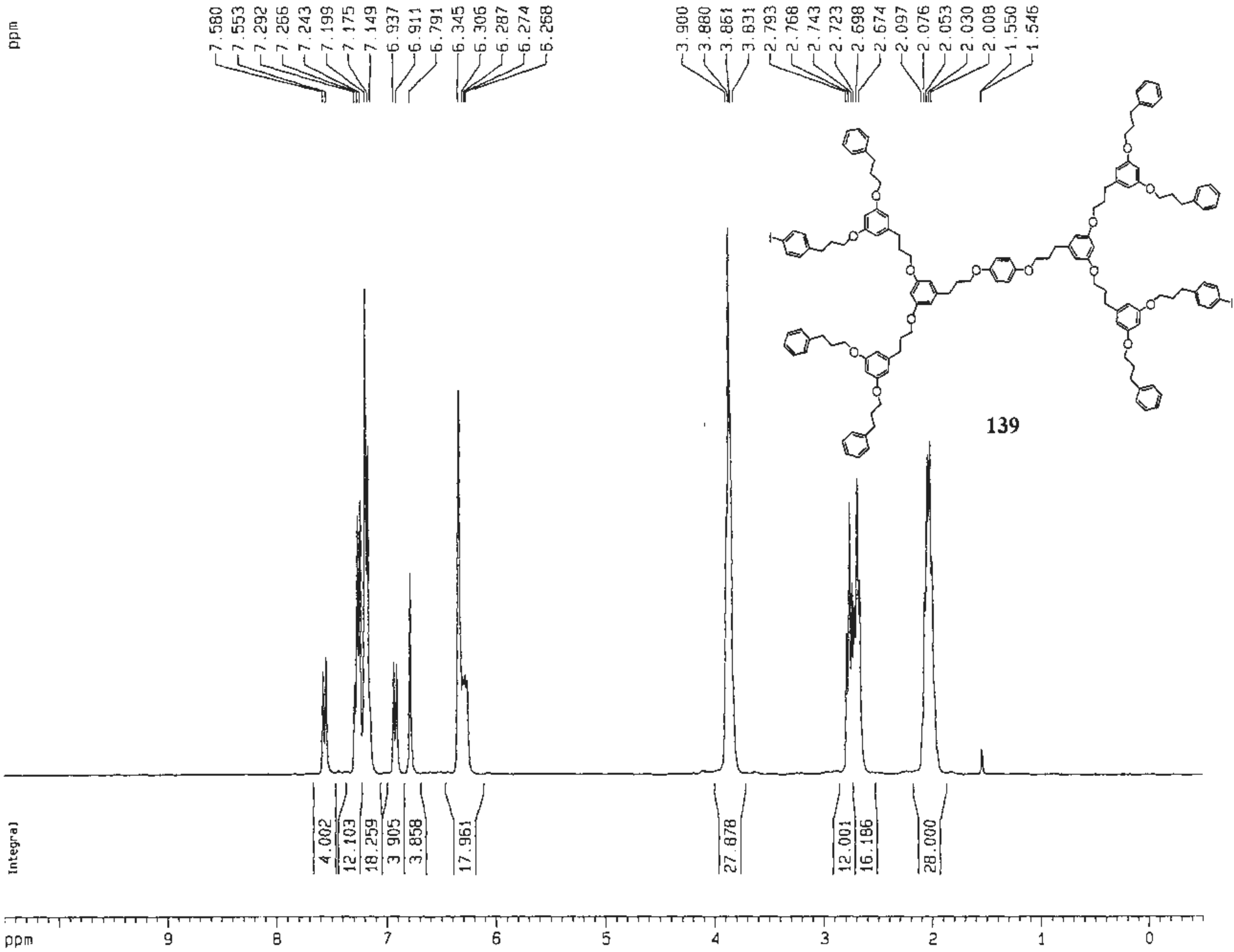
F2 - Acquisition Parameters
Date_           20080428
Time                4.08
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   924
DS                     0
SWH         22675.736 Hz
FIDRES       0.346004 Hz
AQ          1.4451188 sec
RG                  8192
DW            22.050 usec
DE              6.00 usec
TE               296.2 K
D1        1.00000000 sec
d11       0.03000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1              3.00 usec
PL1              -6.00 dB
SFO1       75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2         100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2      300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF         75.4677611 MHz
WDW                   EM
SSB                    0
LB               3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX              22.00 cm
CY              11.99 cm
F1P          200.000 ppm
F1           15093.55 Hz
F2P          -10.000 ppm
F2           -754.68 Hz
PPMCM     9.54545 ppm/cm
HZCM     720.37402 Hz/cm
```



139

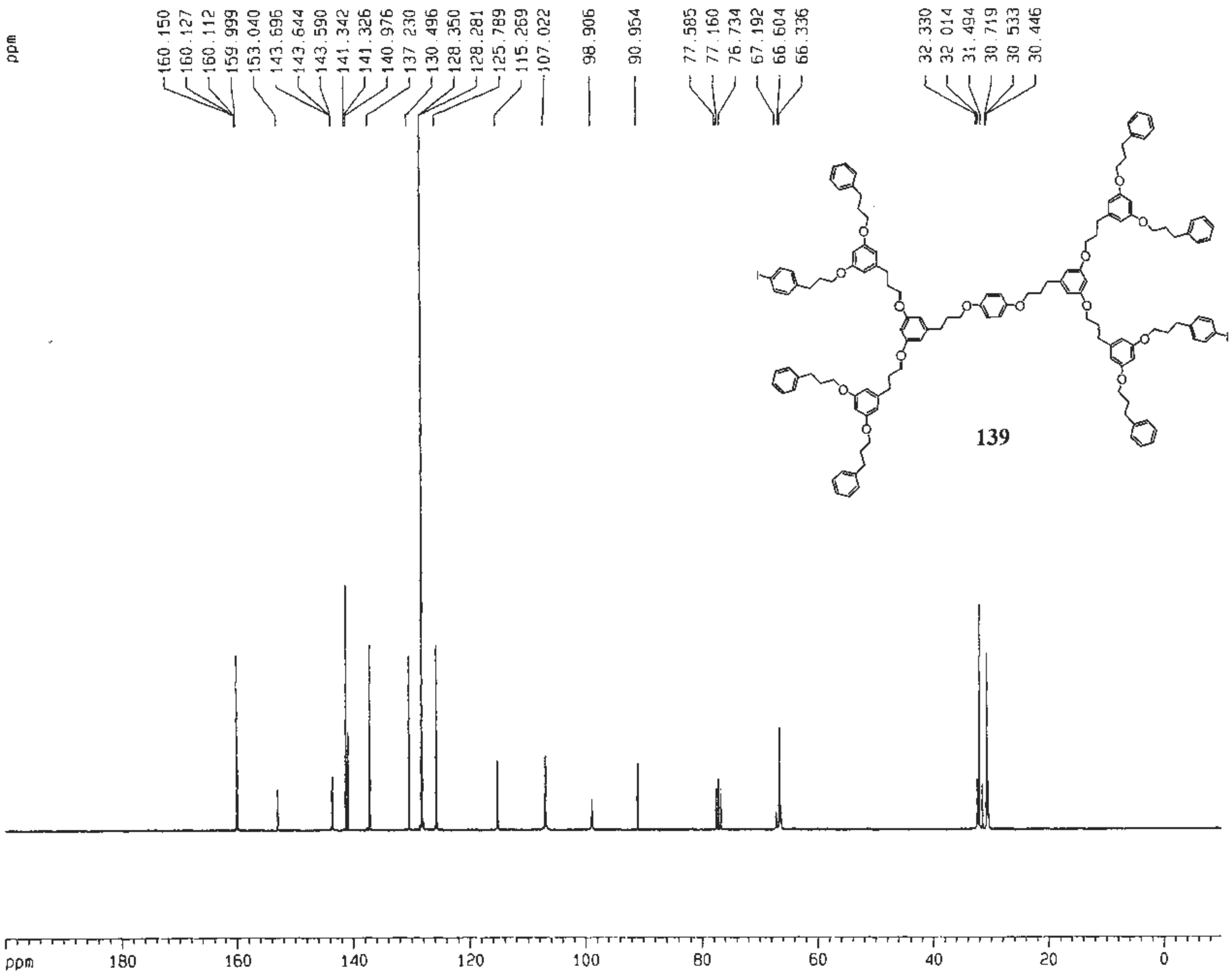
```
Current Data Parameters
NAME            I-G2'-oen
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080430
Time                16.36
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                   40.3
DW                138.825 usec
DE                 198.32 usec
TE                  294.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300266 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9.95 cm
F1P                10.500 ppm
F1                3151.37 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.05502 Hz/cm
```



139

```
Current Data Parameters
NAME            I-G2'-denC
EXPNO                    1
PROCNO                   1

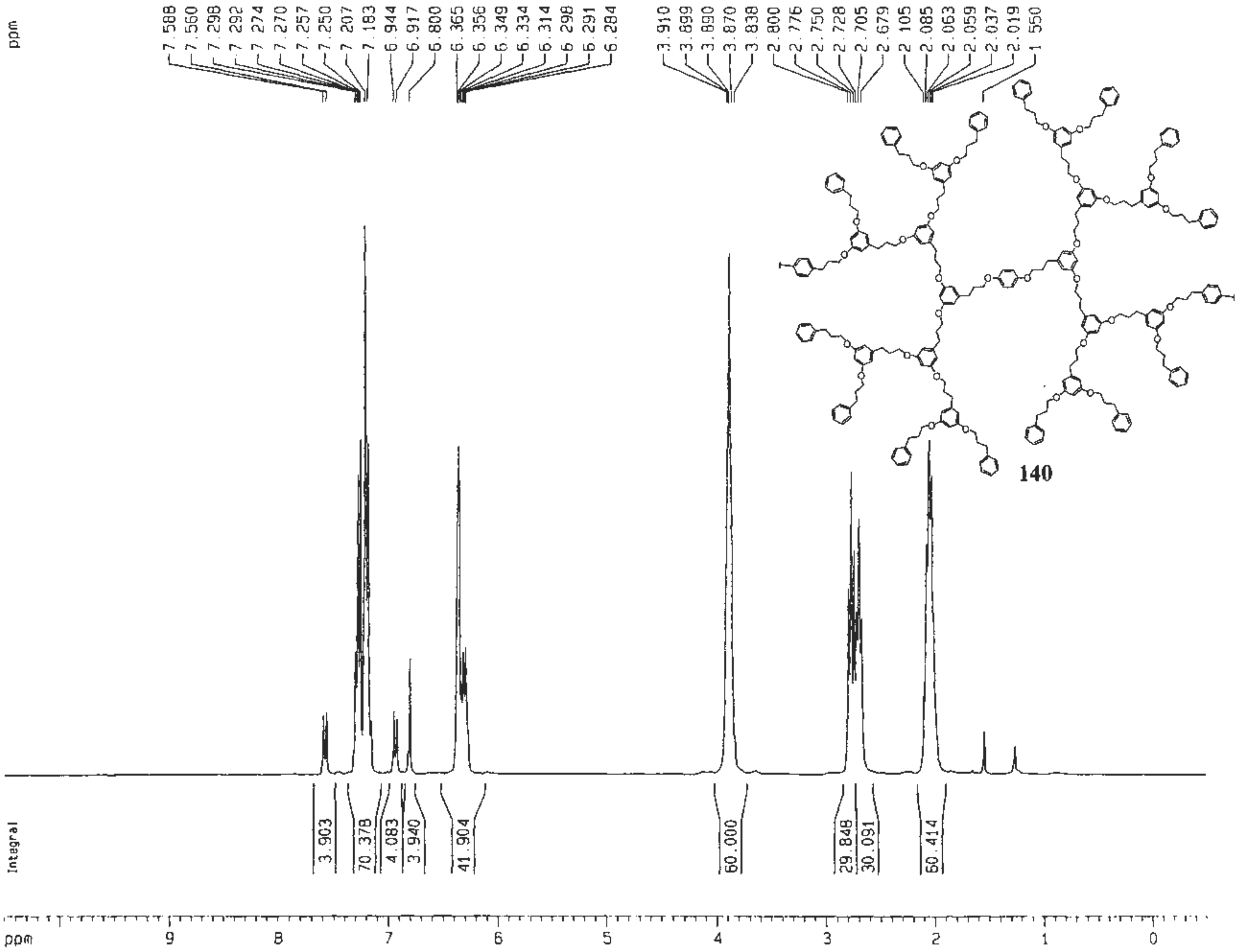
F2 - Acquisition Parameters
Date_             20071218
Time                 16.38
INSTRUM             dpx300
PROBHD   5 mm 880 BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                    1000
DS                       0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                    8192
DW                  22.050 usec
DE                    6.00 usec
TE                     0.0 K
D1              1.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    3.00 usec
PL1                  -6.00 dB
SFO1            75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2           300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF              75.4677898 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   14.40 cm
F1P                200.000 ppm
F1                15093.56 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM              9.54545 ppm/cm
HZCM             720.37427 Hz/cm
```



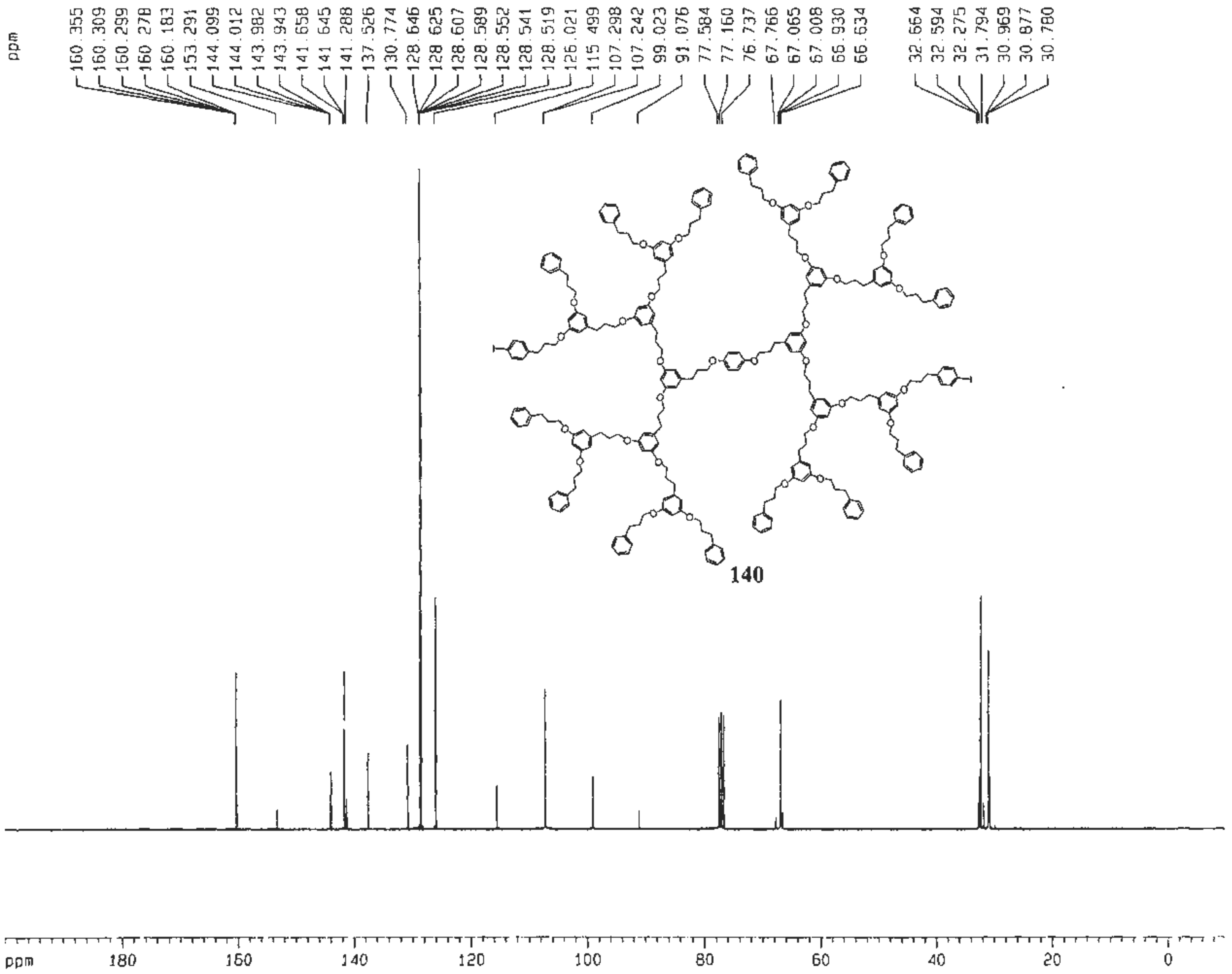
```
Current Data Parameters
NAME          I-G3'-den
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080122
Time              17.13
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                 45.3
DW              138.825 usec
DE               198.32 usec
TE                298.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300185 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



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```
Current Data Parameters
NAME             I-G3'-denC
EXPNO                     1
PROCNO                    1

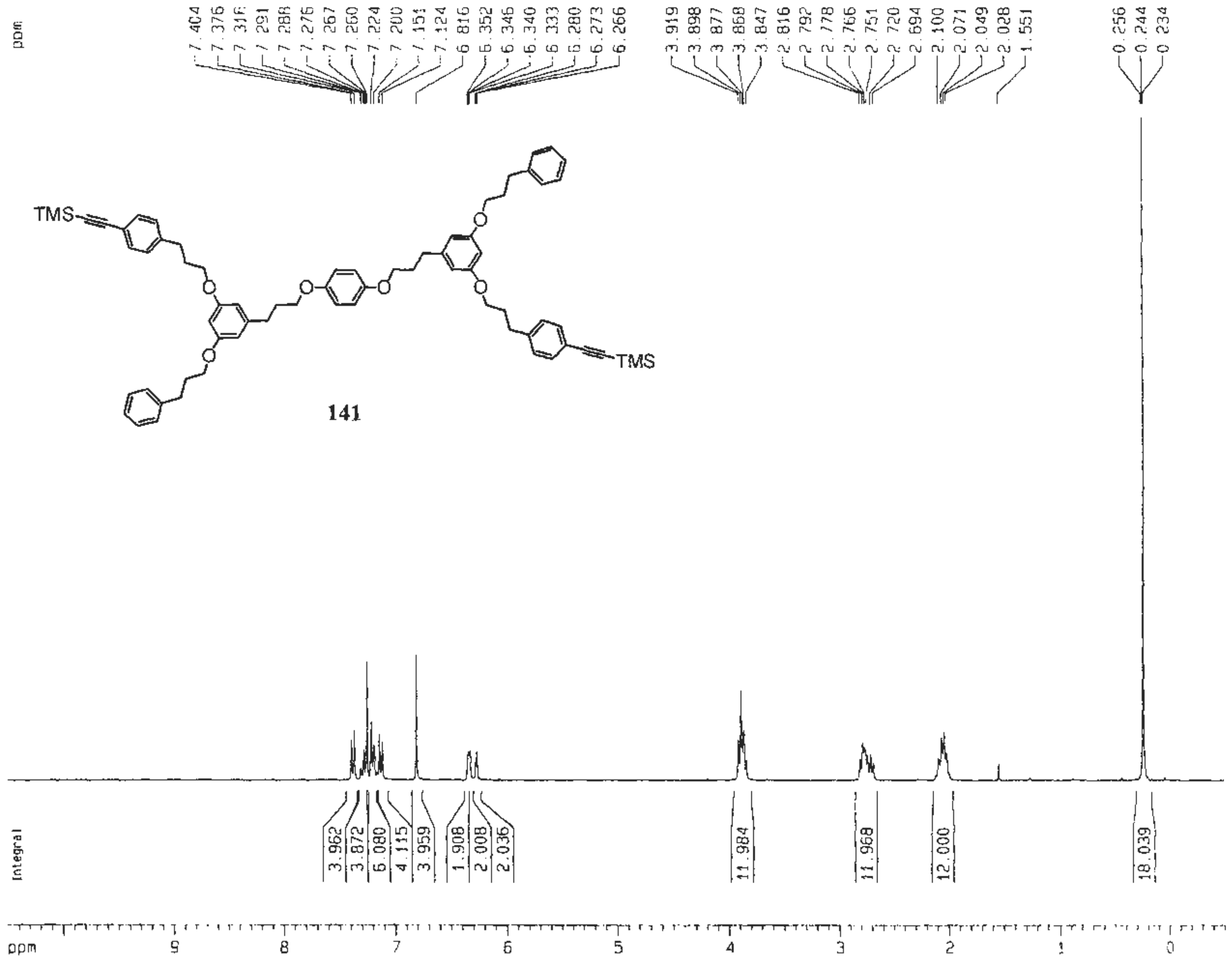
F2 - Acquisition Parameters
Date_              20080125
Time                  21.59
INSTRUM              dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                    15608
DS                        0
SWH              22675.736 Hz
FIDRES            0.346004 Hz
AQ               1.4451168 sec
RG                     8192
DW                   22.050 usec
DE                     6.00 usec
TE                    298.2 K
D1               1.00000000 sec
d11              0.03000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     3.00 usec
PL1                   -6.00 dB
SFO1             75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                  120.00 dB
PL12                  19.00 dB
SFO2            300.1315007 MHz

F2 - Processing parameters
SI                    65536
SF               75.4677448 MHz
WDW                      EM
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    22.00 cm
CY                    11.96 cm
F1P                 200.000 ppm
F1                 15093.55 Hz
F2P                 -10.000 ppm
F2                  -754.68 Hz
PPMCM               9.54545 ppm/cm
HZCM              720.37384 Hz/cm
```



141

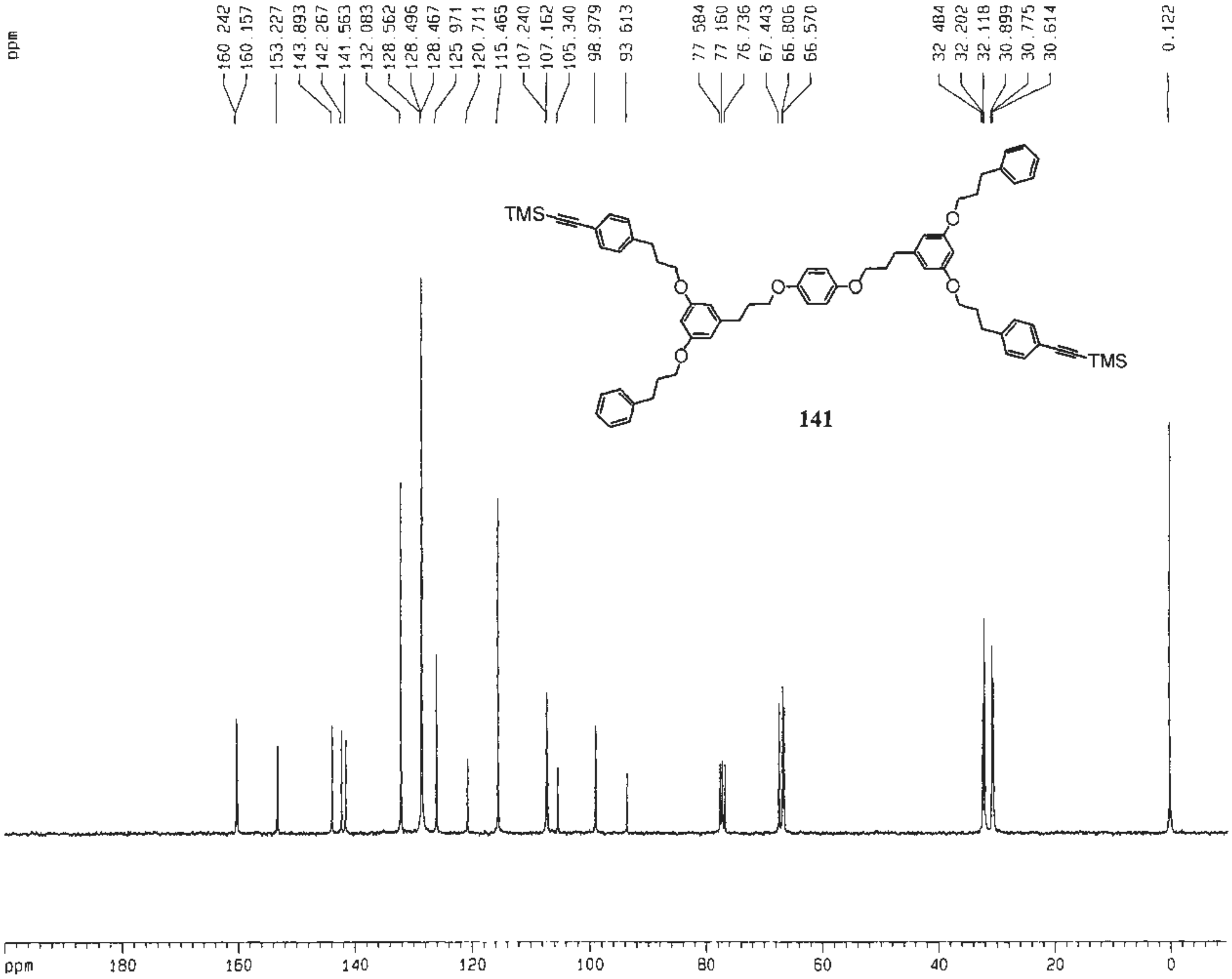
```
Current Data Parameters
NAME          TMS-G1'-den
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20060127
Time                 0.54
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                  16384
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                    512
DW                136.825 usec
DE                 198.32 usec
TE                    0.0 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300061 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  11.98 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



141

```
Current Data Parameters
NAME       TMS-G1'-denC
EXPNO                 1
PROCNO                1

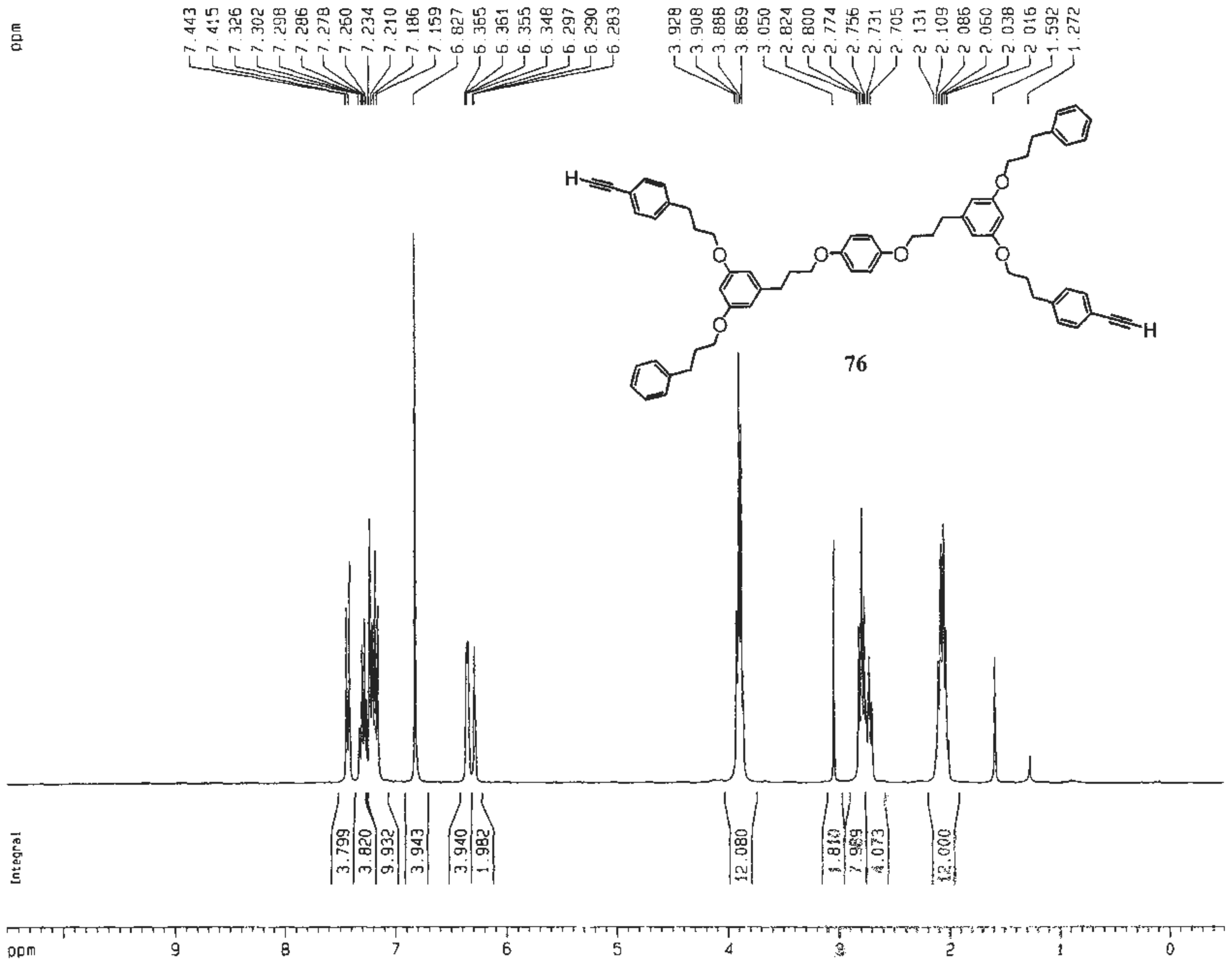
F2 - Acquisition Parameters
Date_          20060127
Time               1 30
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  161
DS                    0
SWH          22675.736 Hz
FIDRES        0 346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                  0 0 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0 00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100 00 usec
PL2              120 00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75 4677564 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                10.02 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754 68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37402 Hz/cm
```



76

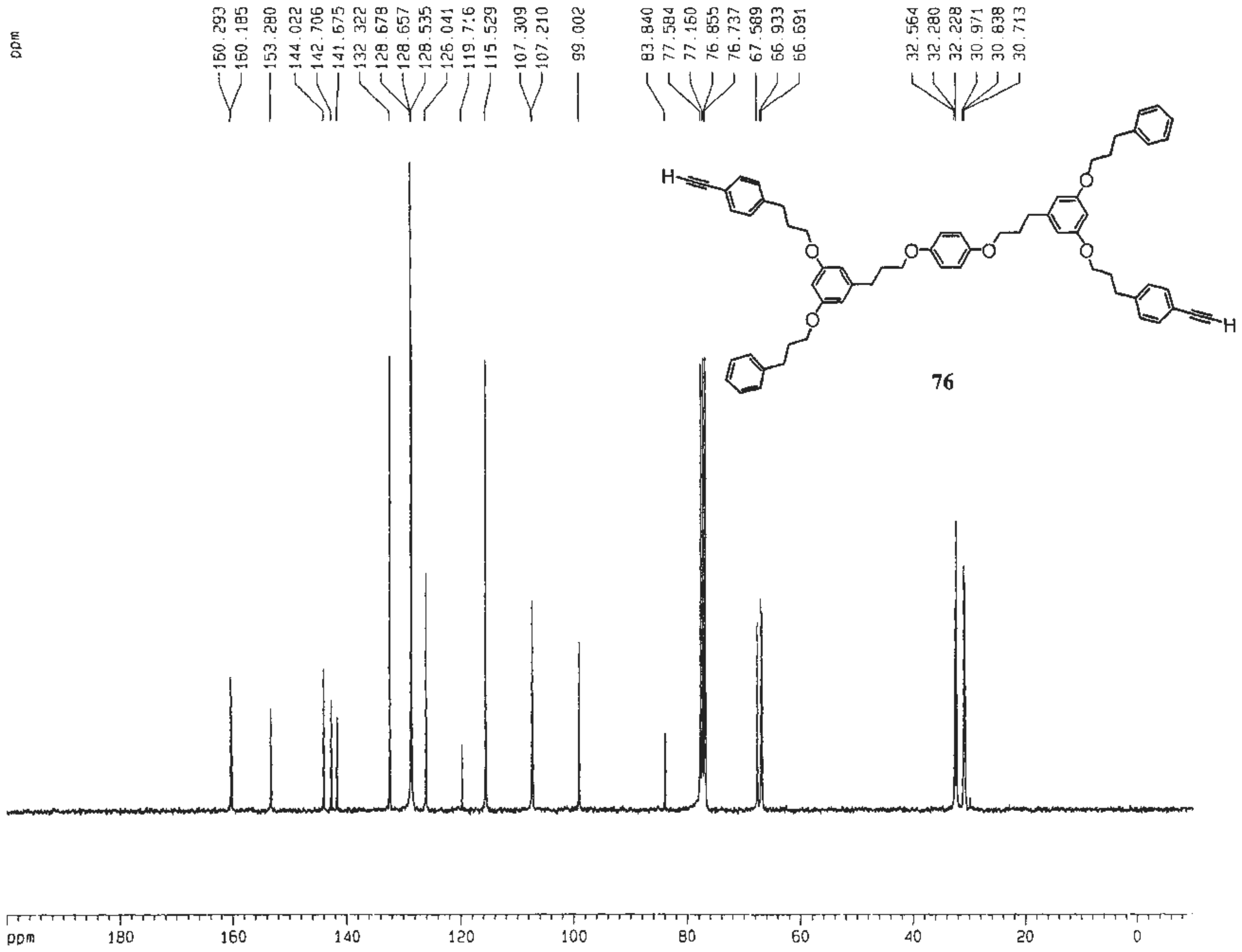
```
Current Data Parameters
NAME        HCC-G1'-den
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080829
Time              21.46
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH           3601 657 Hz
FIDRES        0 219828 Hz
AQ           2.2745588 sec
RG                  128
DW            138.825 usec
DE             198.32 usec
TE              295.2 K
D1         5.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1              9.00 usec
PL1            -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300061 MHz
WDW                  EM
SSB                   0
LB                 0 30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22 00 cm
CY                 9.95 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



76

```
Current Data Parameters
NAME        HCC-G1'-denC
EXPNO                  1
PROCNO                 1

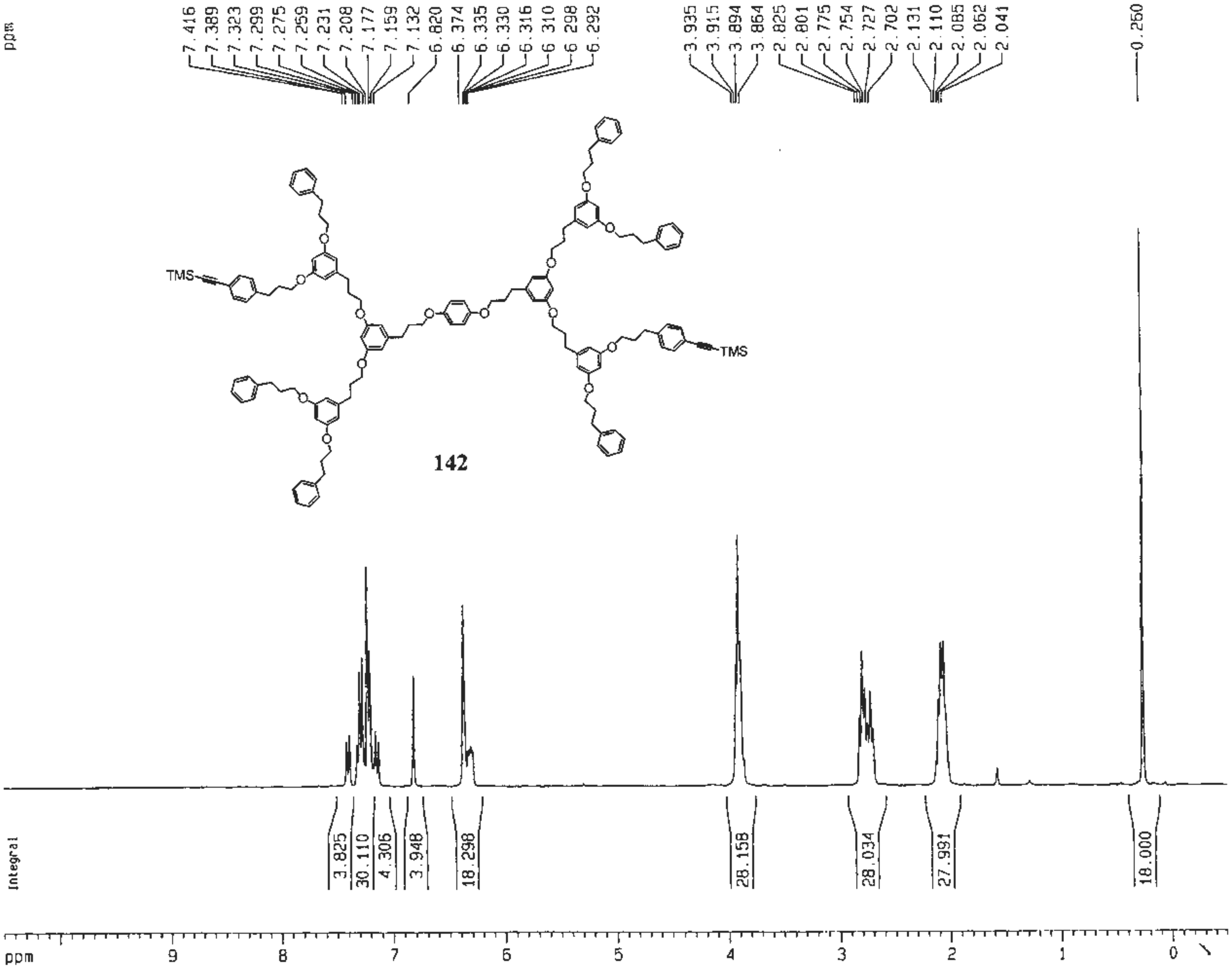
F2 - Acquisition Parameters
Date_          20080829
Time              19.21
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                 3000
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677419 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.97 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37384 Hz/cm
```



142

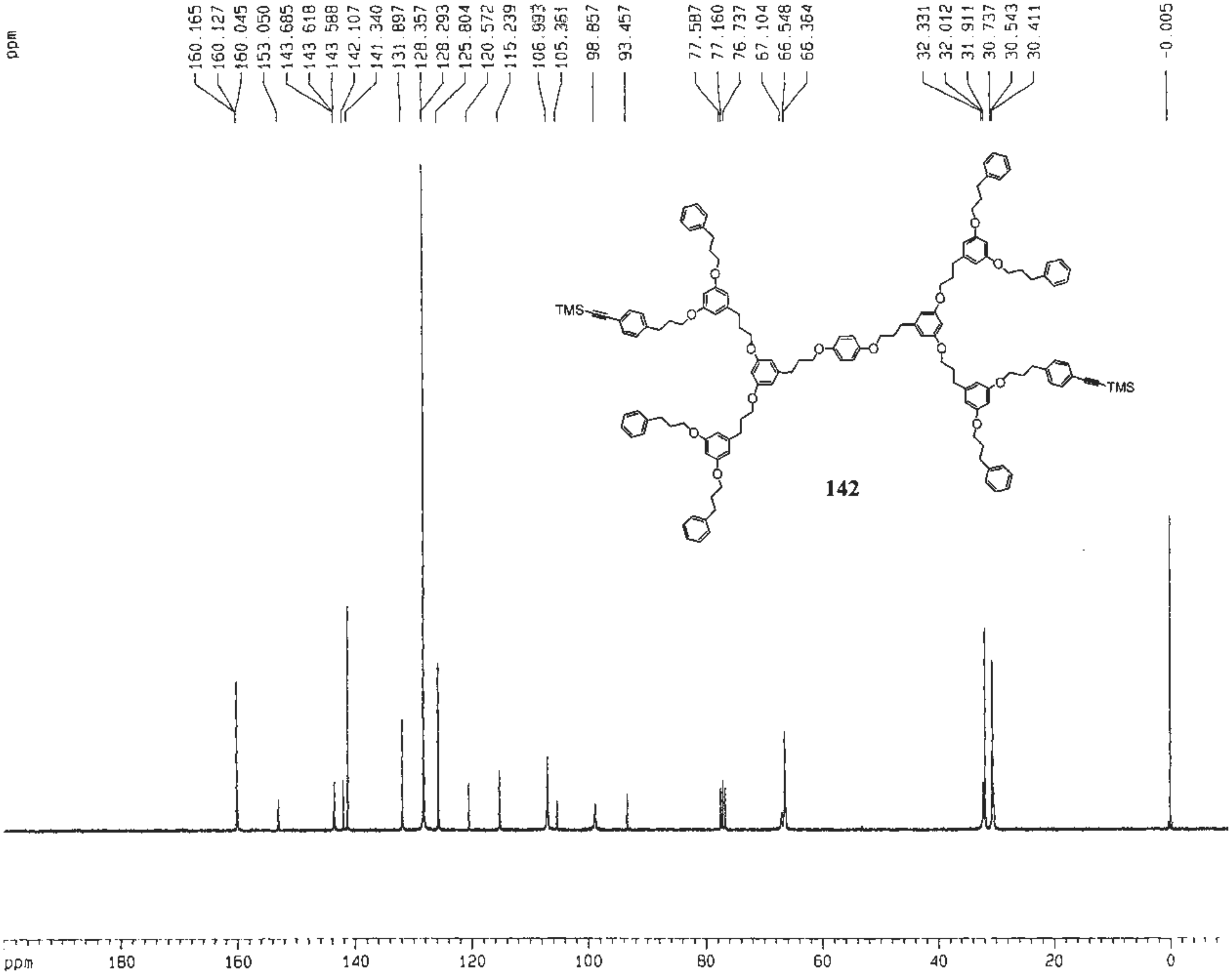
Current Data Parameters
NAME TMS-G2'-den
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080122
Time 17.24
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 3601.657 Hz
FIDRES 0.219828 Hz
AQ 2.2745588 sec
RG 64
DW 138.825 usec
DE 198.32 usec
TE 298.2 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -2.00 dB
SFO1 300.1315000 MHz

F2 - Processing parameters
SI 32768
SF 300.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 9.97 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm



142

```
Current Data Parameters
NAME        TMS-G2'-denC
EXPNO                  1
PROCNO                 1

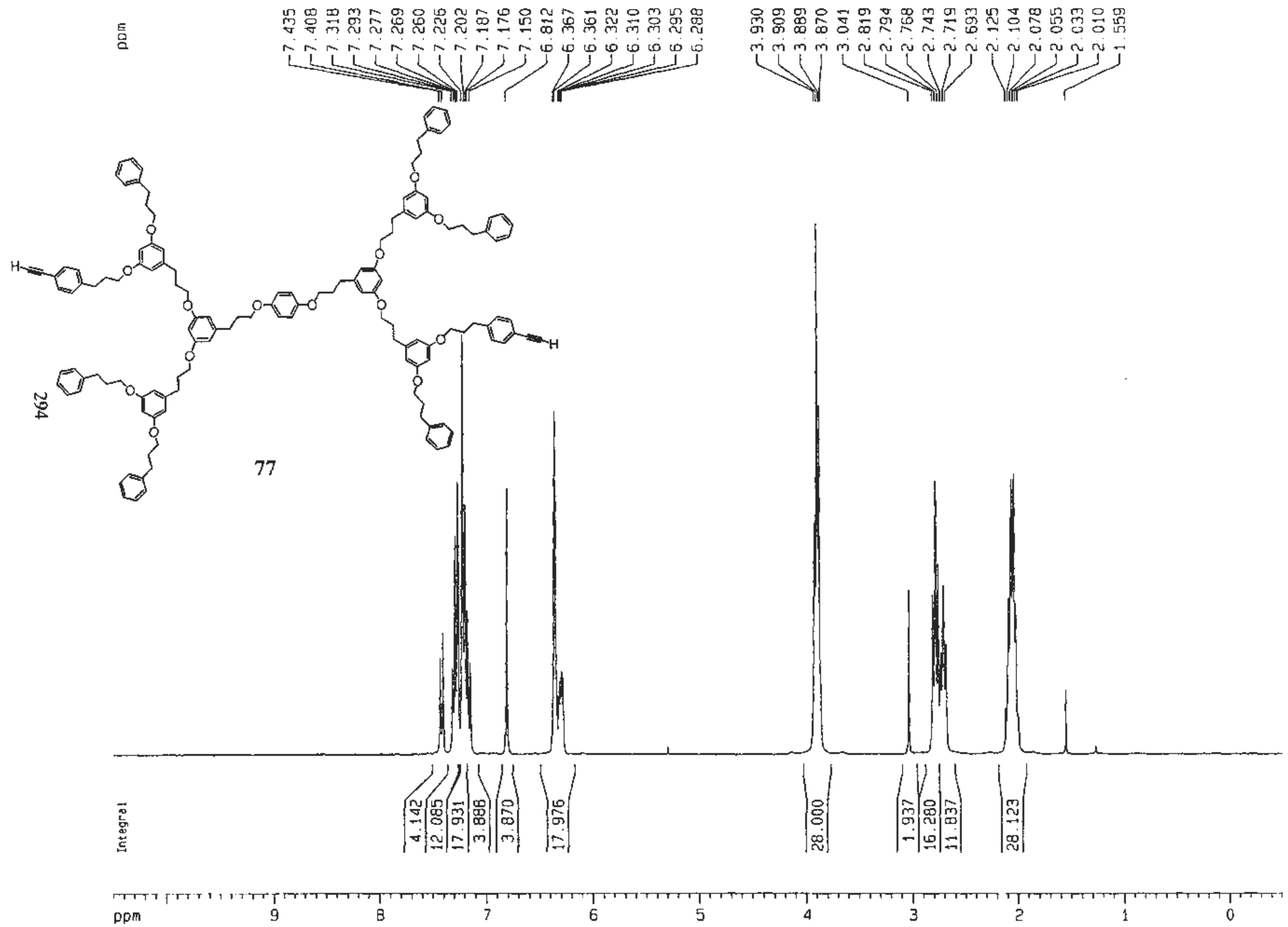
F2 - Acquisition Parameters
Date_           20080122
Time               17.33
INSTRUM           dpx300
PROBHD    5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   700
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AO            1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4676862 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.00 cm
F1P              200.000 ppm
F1              15093.54 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37335 Hz/cm
```



77

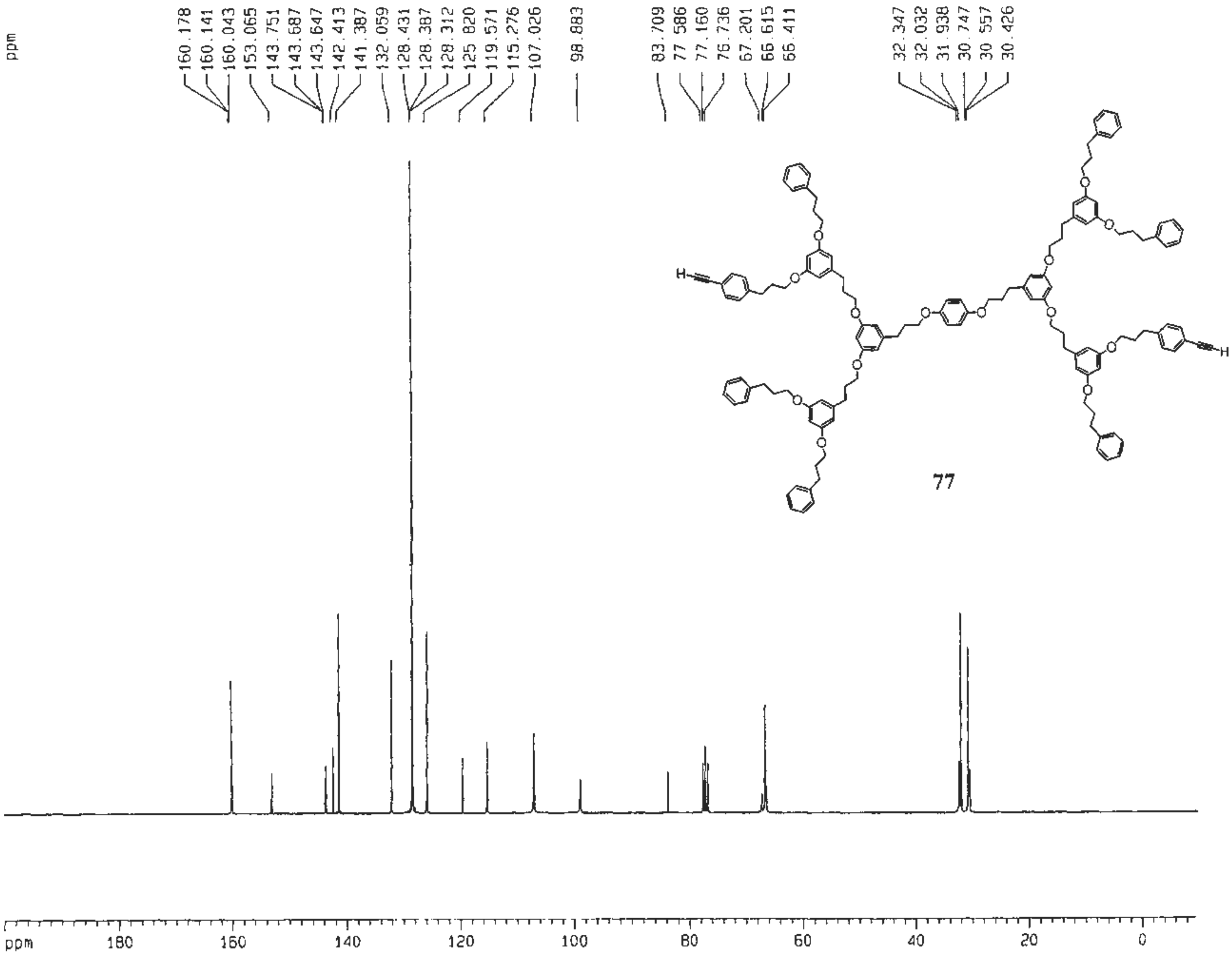
```
Current Data Parameters
NAME          HCC-G2'-den
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080214
Time                13.01
INSTRUM            dpx300
PROBHD   5 mm 880 BB-1H
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              4202.563 Hz
FIDRES           0.128252 Hz
AQ              3.8986228 sec
RG                    256
DW                118.975 usec
DE                 169.96 usec
TE                  297.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   5.00 usec
PL1                 -2.00 dB
SFO1          300.1318000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300063 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9.96 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



77

```
Current Data Parameters
NAME      HCC-G2'-denC
EXPNO               1
PROCNO              1

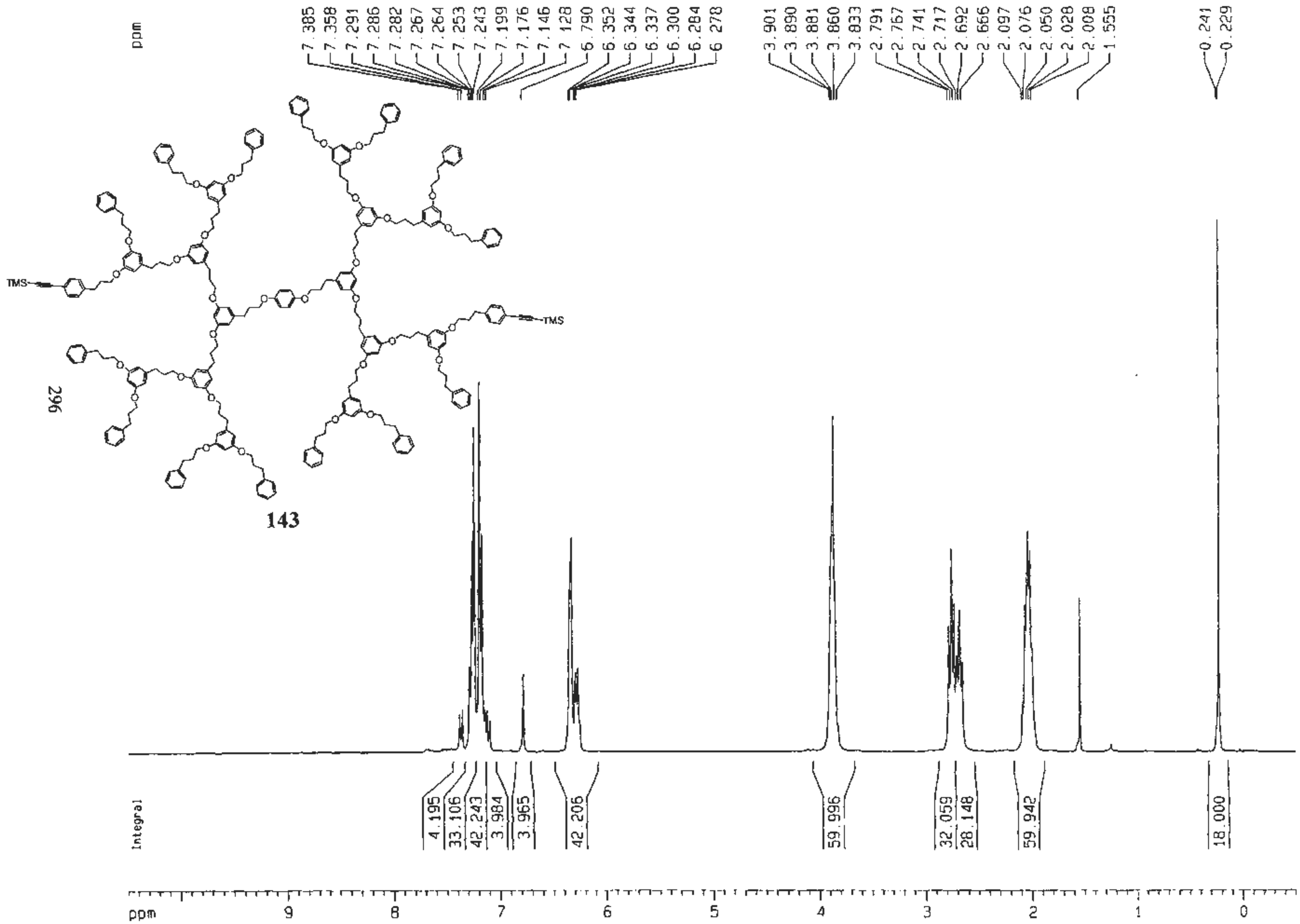
F2 - Acquisition Parameters
Date_        20080219
Time             5.42
INSTRUM        dpx300
PROBHD   5 mm BBO BB-1H
PULPROG          zgdc
TD              65536
SOLVENT         CDCl3
NS              10000
DS                  0
SWH        22675.736 Hz
FIDRES      0.346004 Hz
AQ         1.4451188 sec
RG               8192
DW             22.050 usec
DE               6.00 usec
TE              298.2 K
D1         1.00000000 sec
d11        0.03000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1              13C
P1               3.00 usec
PL1             -6.00 dB
SFO1      75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2       waltz16
NUC2               1H
PCPD2          100.00 usec
PL2            120.00 dB
PL12            19.00 dB
SFO2     300.1315007 MHz

F2 - Processing parameters
SI              65536
SF         75.4677855 MHz
WDW                EM
SSB                 0
LB               0.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX              22.00 cm
CY              12.01 cm
F1P           200.000 ppm
F1           15093.56 Hz
F2P           -10.000 ppm
F2            -754.68 Hz
PPMCM         9.54545 ppm/cm
HZCM        720.37427 Hz/cm
```



143

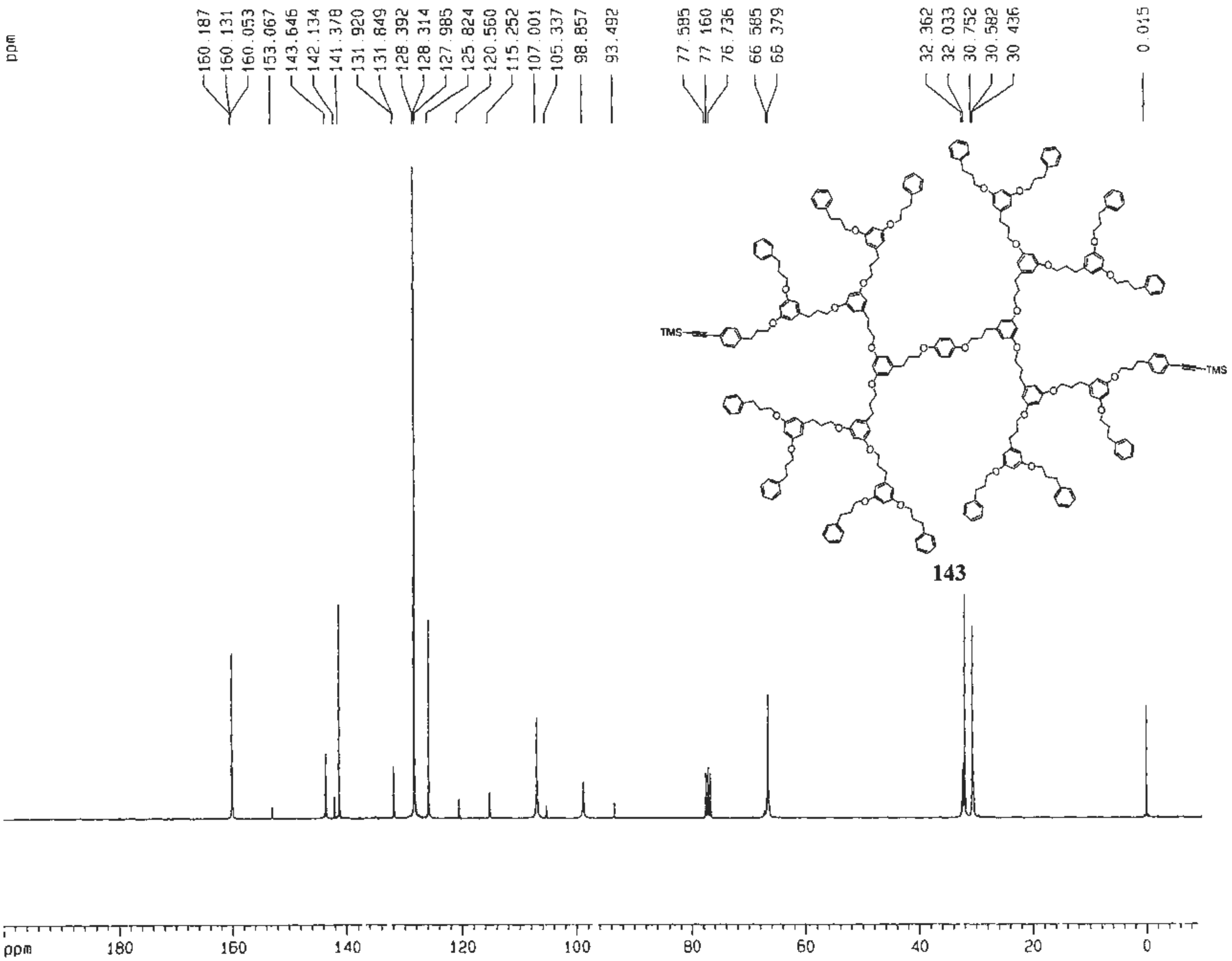
```
Current Data Parameters
NAME         TMS-G3'-den
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080329
Time               12.54
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           3601.657 Hz
FIDRES        0.219828 Hz
AQ           2.2745588 sec
RG                   362
DW           138.825 usec
DE            198.32 usec
TE               295.2 K
D1        5.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1              5.00 usec
PL1             -2.00 dB
SFO1      300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF        300.1300086 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              22.00 cm
CY              10.00 cm
F1P           10.500 ppm
F1            3151.36 Hz
F2P           -0.500 ppm
F2            -150.07 Hz
PPMCM      0.50000 ppm/cm
HZCM     150.06500 Hz/cm
```



143

```
Current Data Parameters
NAME        TMS-G3'-denC
EXPNO                  1
PROCNO                 1

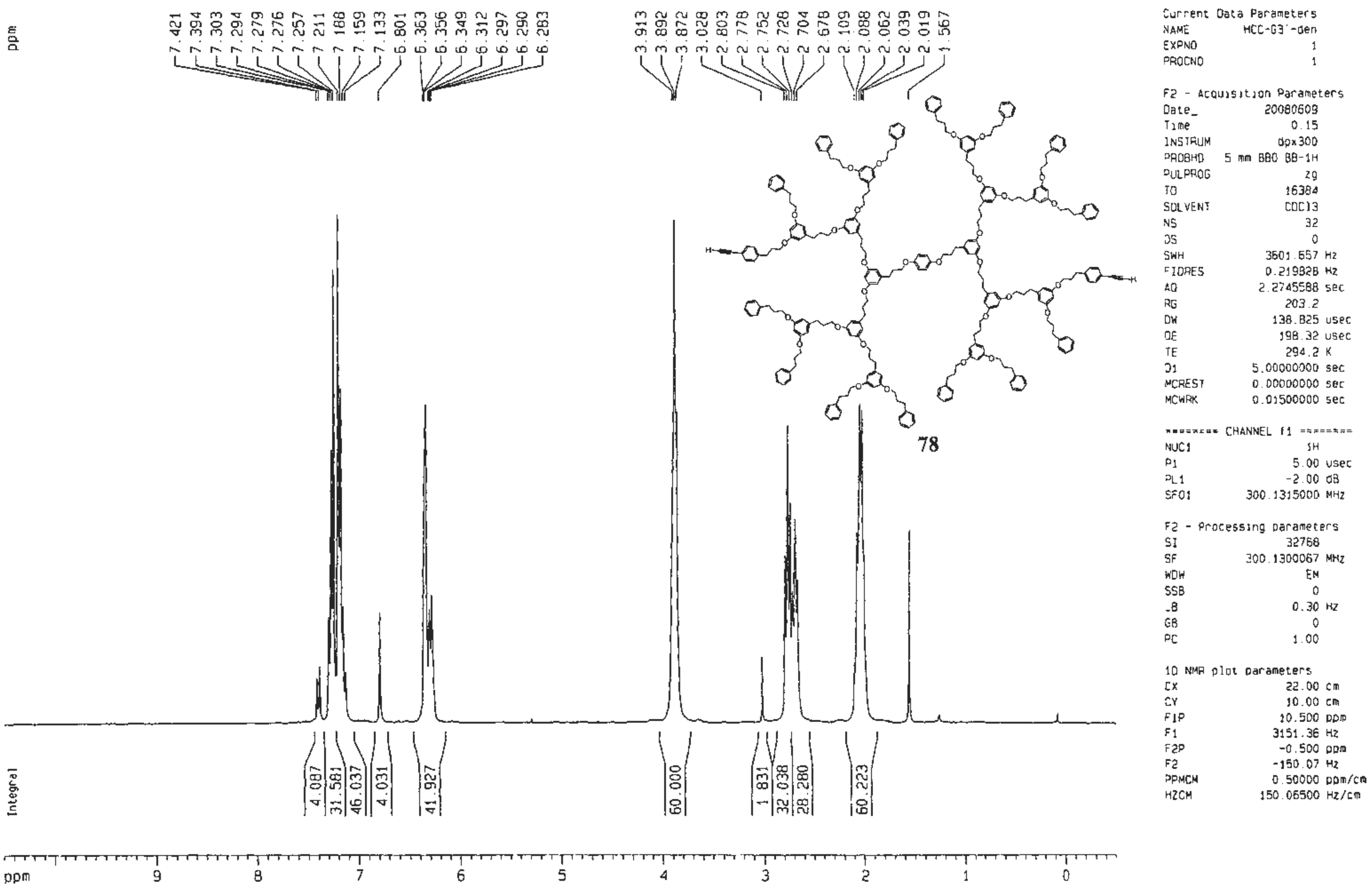
F2 - Acquisition Parameters
Date_           20080329
Time               19.23
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  3800
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451168 sec
RG                5160.6
DW                22.050 usec
DE                  6.00 usec
TE                 295.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677849 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.98 cm
F1P              200.000 ppm
F1              15093.56 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37427 Hz/cm
```



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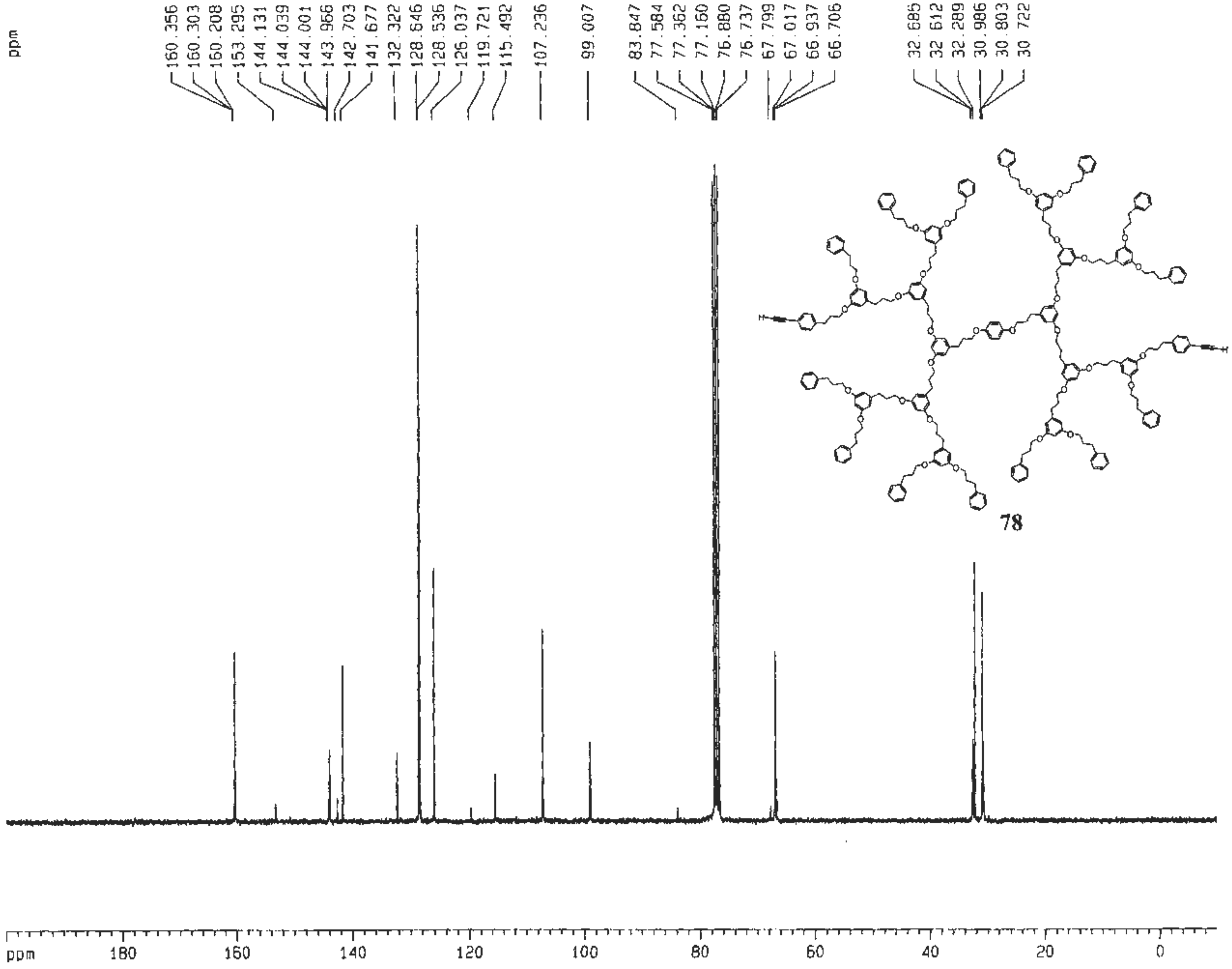
```
Current Data Parameters
NAME        HCC-G3'-den
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080609
Time               0.15
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                203.2
DW              138.825 usec
DE               198.32 usec
TE                294.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 5.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300067 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



78

```
Current Data Parameters
NAME          HCC-G3'-denC
EXPNO                    1
PROCNO                   1

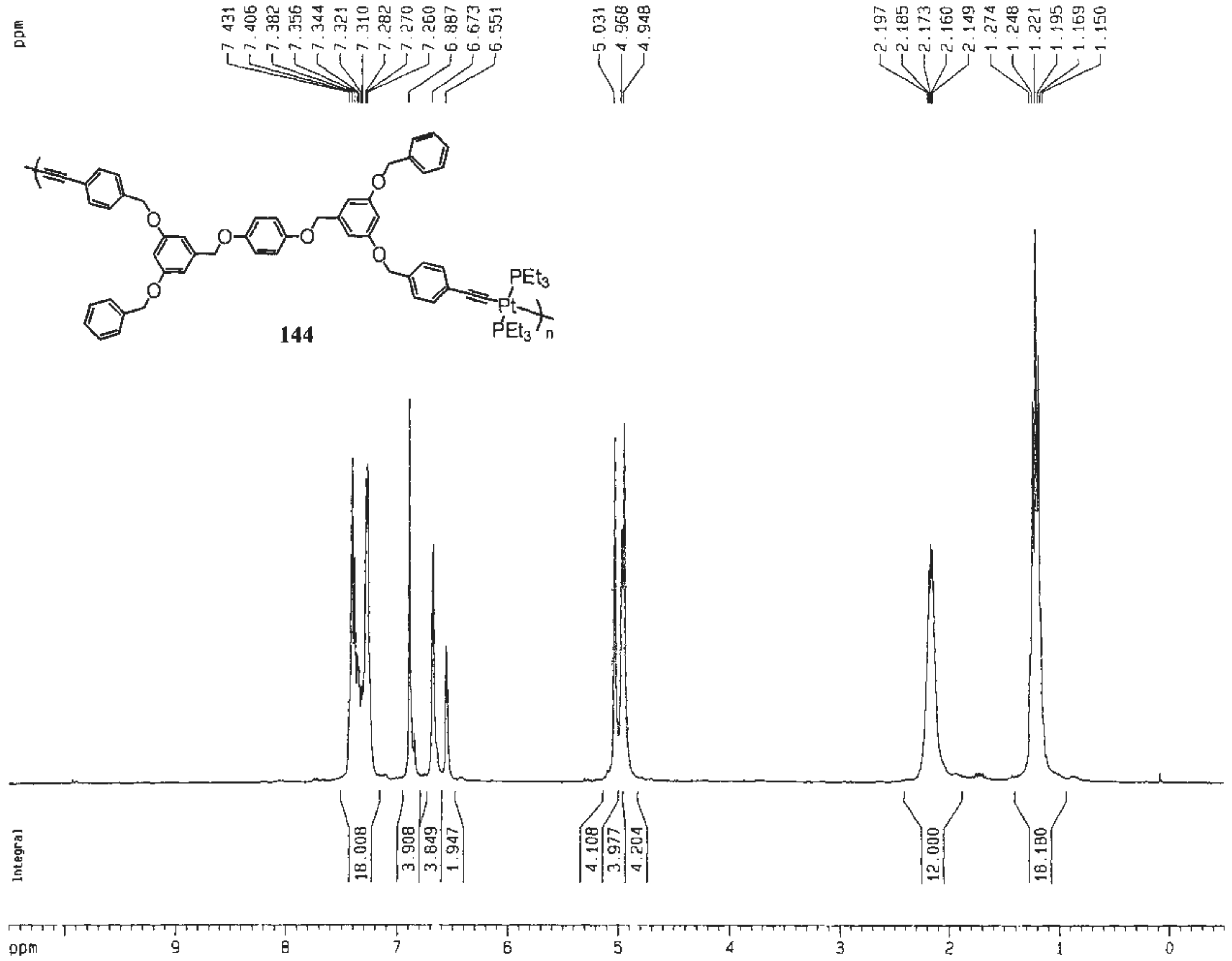
F2 - Acquisition Parameters
Date_             20080509
Time                  7.30
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgpg
TD                   65536
SOLVENT              CDC13
NS                    6080
DS                       0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                    8192
DW              22.050 usec
DE                6.00 usec
TE                 294.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                3.00 usec
PL1                -6.00 dB
SF01          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2           100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SF02         300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF            75.4677407 MHz
WDW                     EM
SSB                      0
LB                 1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.95 cm
F1P            200.000 ppm
F1            15093.55 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM       9.54545 ppm/cm
HZCM        720.37384 Hz/cm
```



144

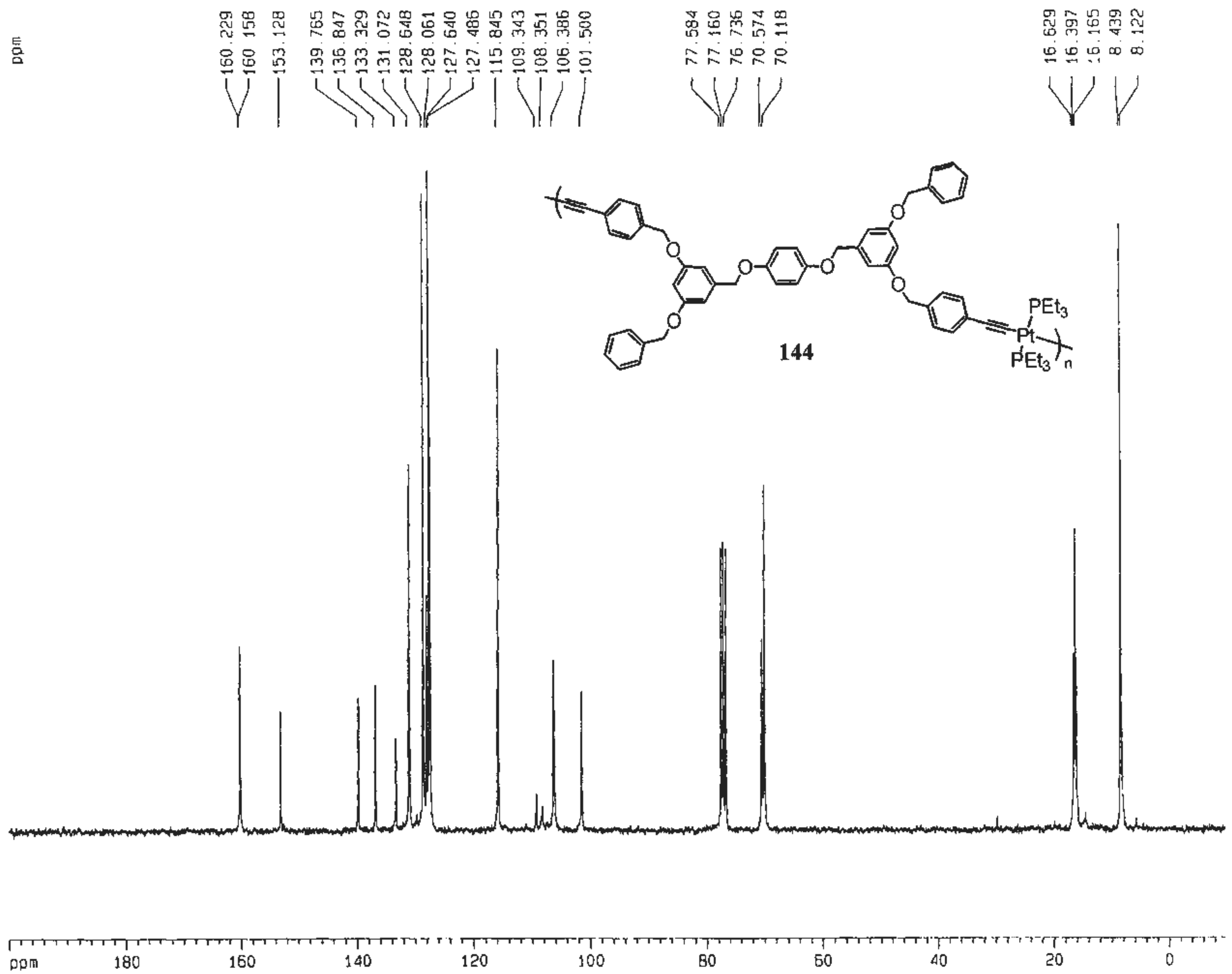
```
Current Data Parameters
NAME       Poly(Pt-G1)N
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080711
Time              19.52
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH          3601.657 Hz
FIDRES       0 219828 Hz
AQ         2.2745588 sec
RG                161.3
DW          138.825 usec
DE           198.32 usec
TE              295 2 K
D1       5.00000000 sec
MCREST   0.00000000 sec
MCWRK    0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1              9.00 usec
PL1            -2.00 dB
SFO1     300 1315000 MHz

F2 - Processing parameters
SI                32768
SF       300.1300064 MHz
WDW                  EM
SSB                   0
LB              0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX             22.00 cm
CY              9.97 cm
F1P          10.500 ppm
F1           3151.36 Hz
F2P          -0.500 ppm
F2           -150.07 Hz
PPMCM    0.50000 ppm/cm
HZCM   150.06500 Hz/cm
```



```
Current Data Parameters
NAME       Poly(Pt-G1)NC
EXPNO                  1
PROCNO                 1

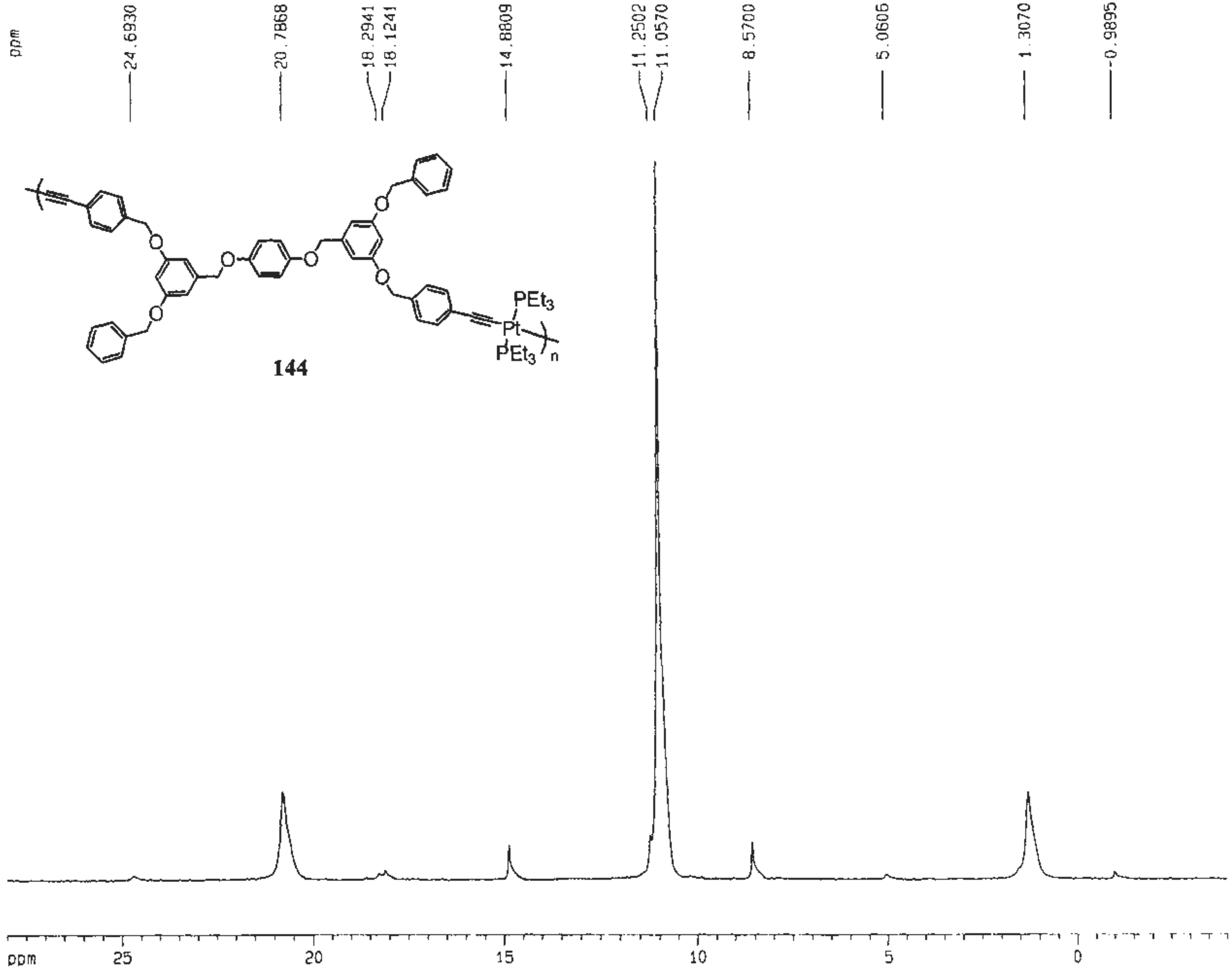
F2 - Acquisition Parameters
Date_          20080711
Time              20.37
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                 1201
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ           1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677497 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.99 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37390 Hz/cm
```



144

```
Current Data Parameters
NAME       Poly(Pt-G1)NP
EXPNO                  1
PROCNO                 1

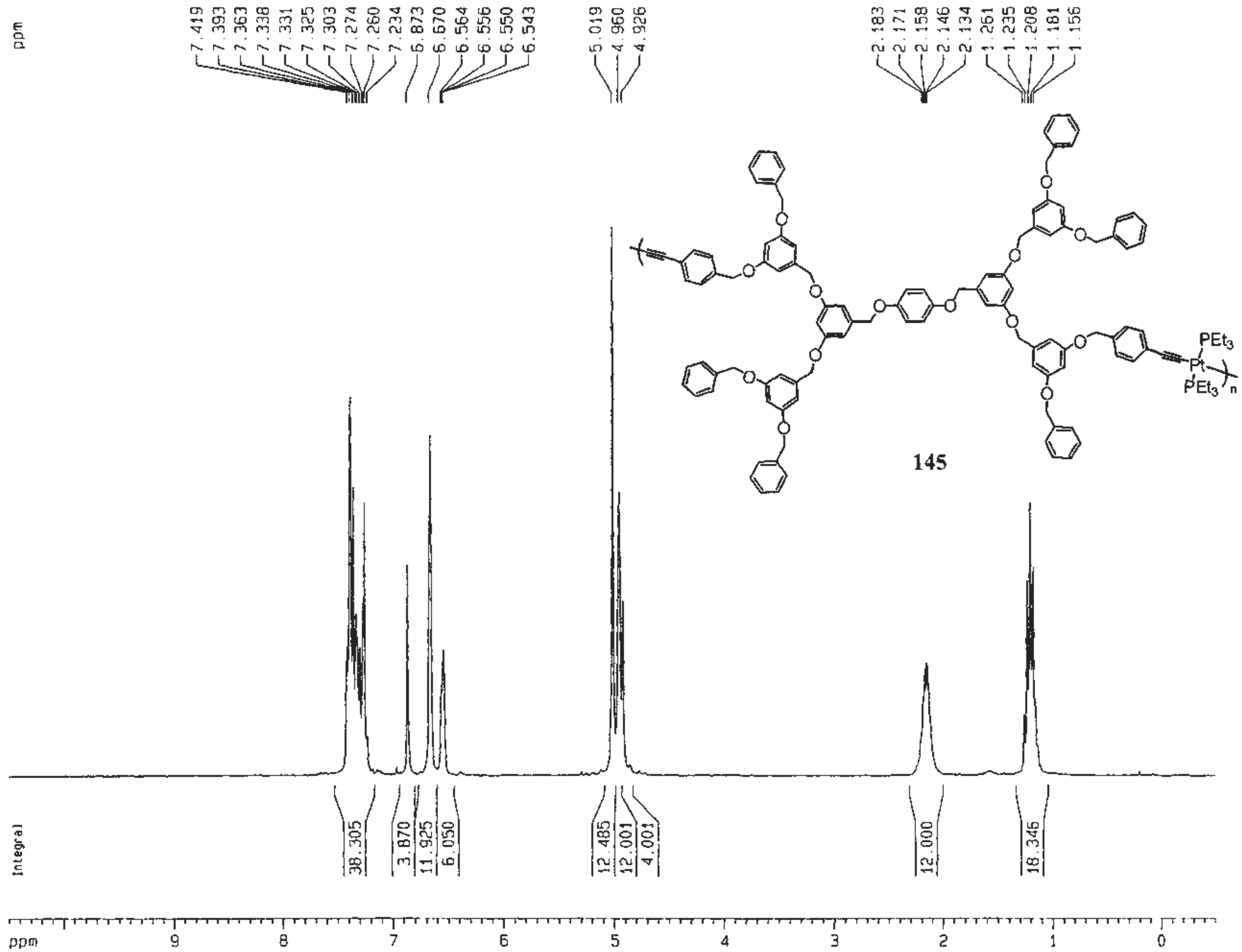
F2 - Acquisition Parameters
Date_           20080916
Time               22.41
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                7298.2
DW                13.275 usec
DE                  6.00 usec
TE                 294.2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 13.02 cm
F1P               28.000 ppm
F1               3401.86 Hz
F2P               -4.000 ppm
F2               -485.98 Hz
PPMCM            1.45455 ppm/cm
HZCM           176.71977 Hz/cm
```



145

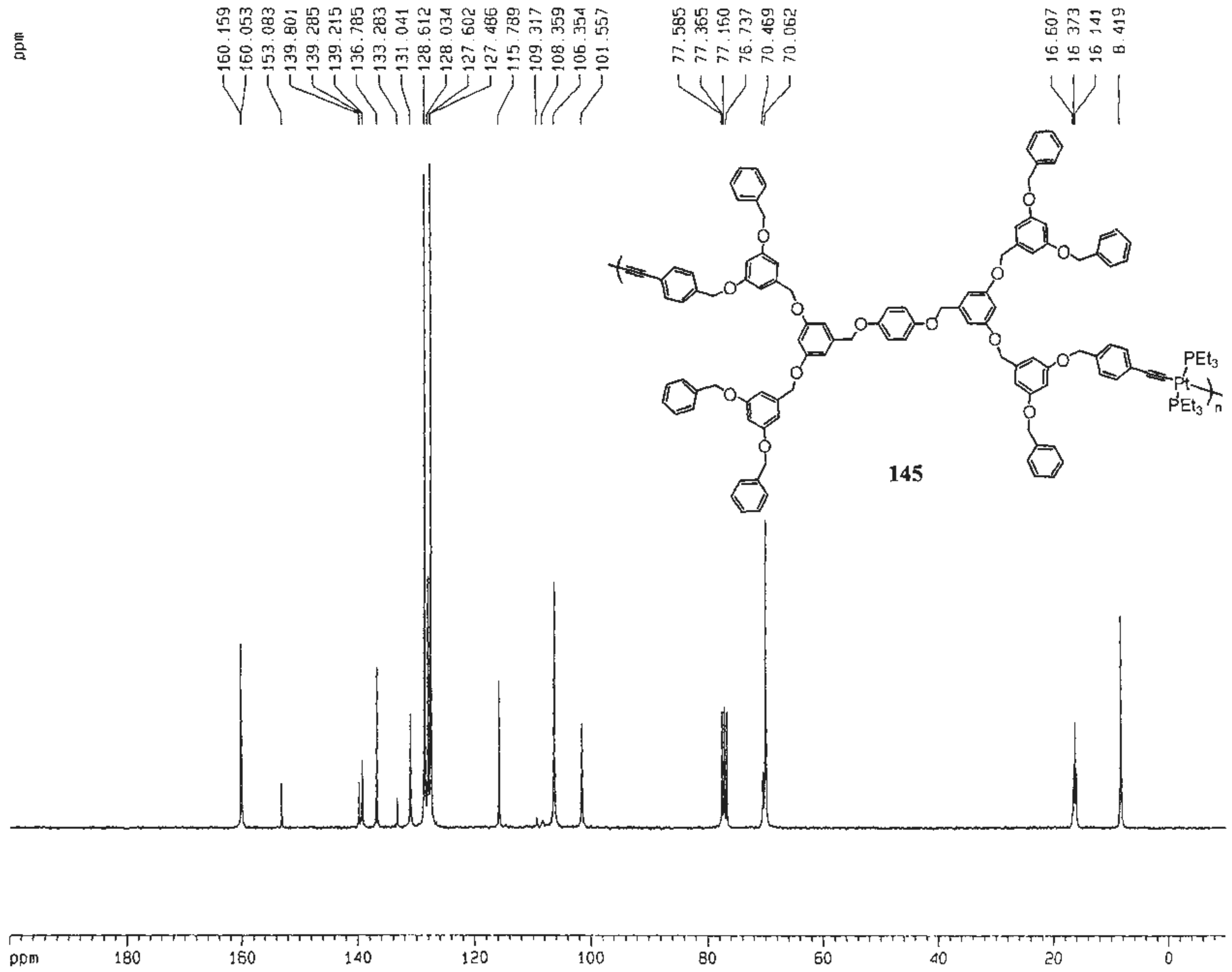
```
Current Data Parameters
NAME        Poly(Pt-G2)N
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080711
Time               19.05
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             3601.657 Hz
FIDRES          0.219828 Hz
AQ             2.2745588 sec
RG                   362
DW               138.825 usec
DE                198.32 usec
TE                 296.2 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300063 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  9.98 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```



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```
Current Data Parameters
NAME        Poly(Pt-G2)NC
EXPNO                   1
PROCNO                  1

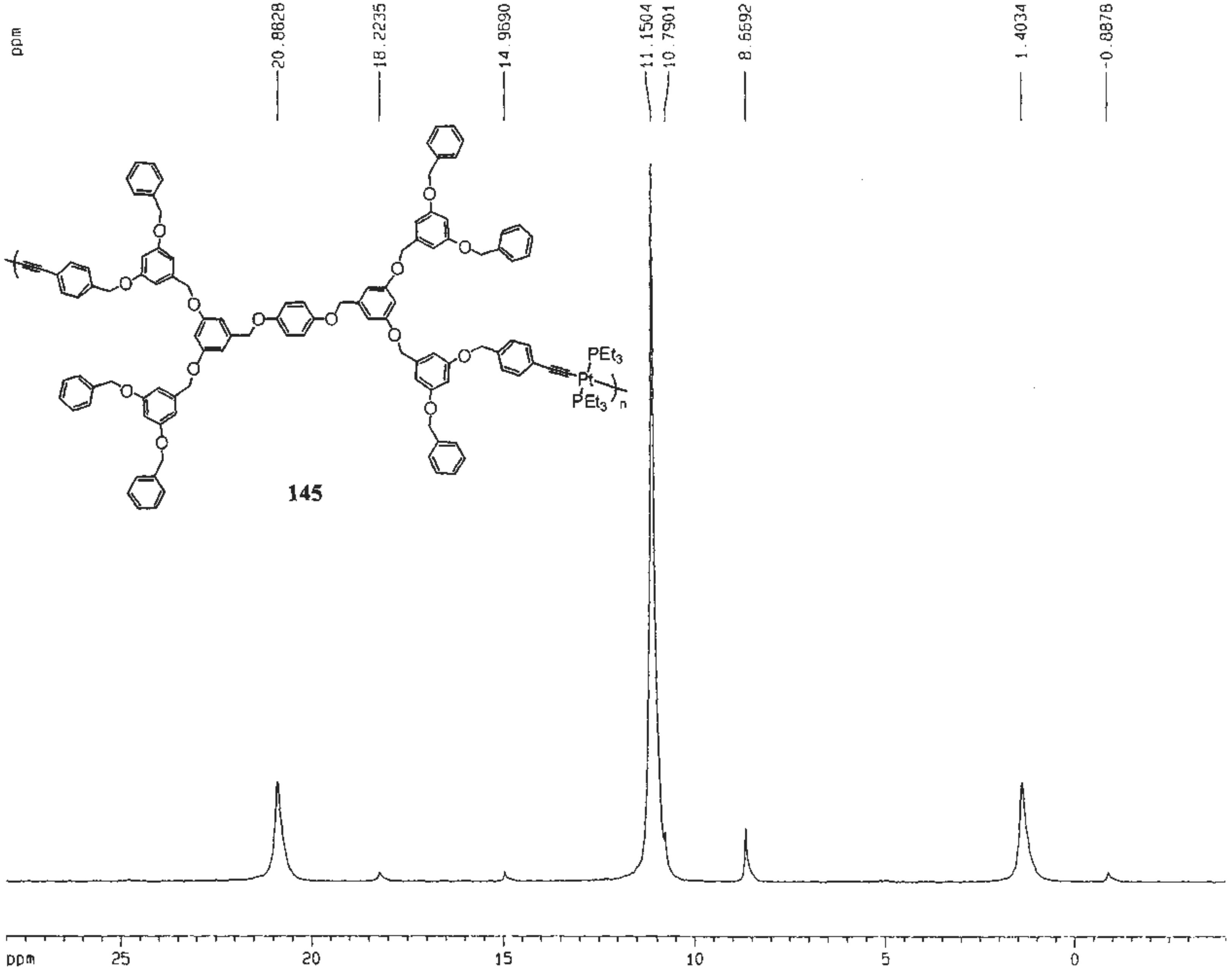
F2 - Acquisition Parameters
Date_            20080711
Time                21.35
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   1564
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                  295.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677566 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.00 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37402 Hz/cm
```



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```
Current Data Parameters
NAME        Poly(Pt-G2)NP
EXPNO                   1
PROCNO                  1

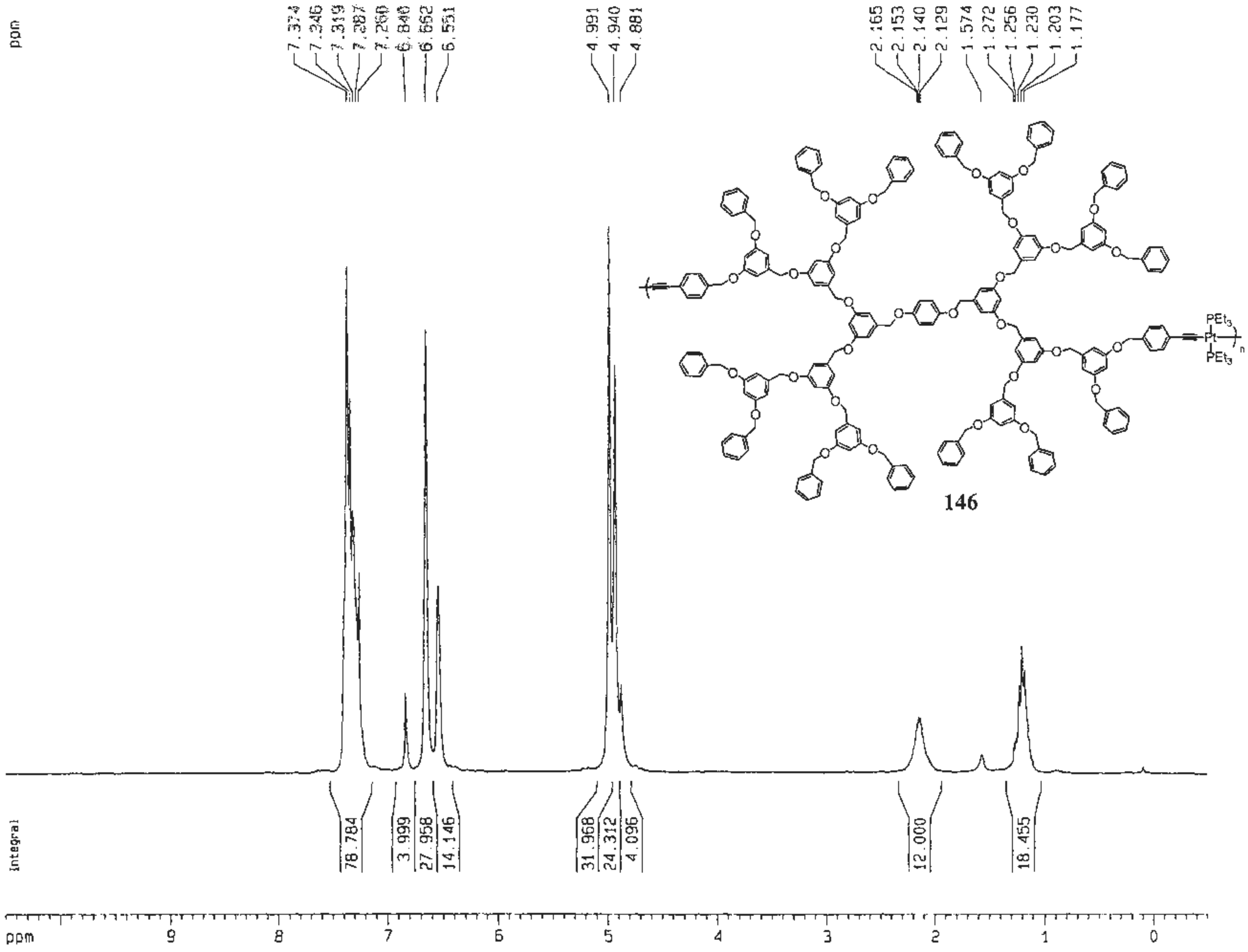
F2 - Acquisition Parameters
Date_            20080916
Time                22.24
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                    413
DS                      0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                 5160.6
DW                 13.275 usec
DE                   6.00 usec
TE                  296.2 K
D1             0.30000001 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  31P
P1                   3.00 usec
PL1                 -6.00 dB
SFO1         121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                  65536
SF           121.4948412 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  13.01 cm
F1P                28.000 ppm
F1                3401.86 Hz
F2P                -4.000 ppm
F2                -485.98 Hz
PPMCM             1.45455 ppm/cm
HZCM            176.71977 Hz/cm
```



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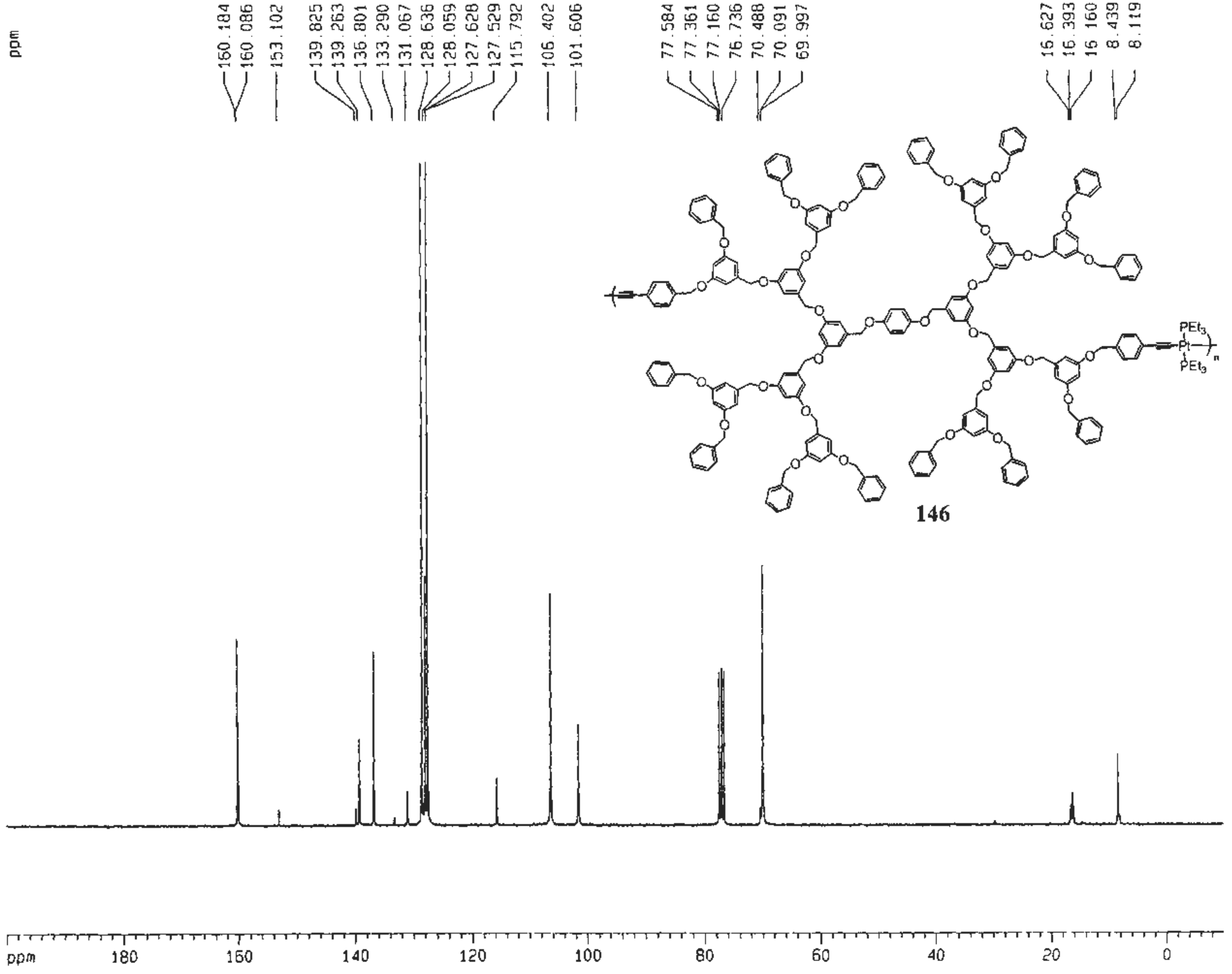
```
Current Data Parameters
NAME         Poly(Pt-G3)N
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080718
Time                21.43
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                    114
DW                138.825 usec
DE                 198.32 usec
TE                  296.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300063 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9.99 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(Pt-G3)NC
EXPNO                  1
PROCNO                 1

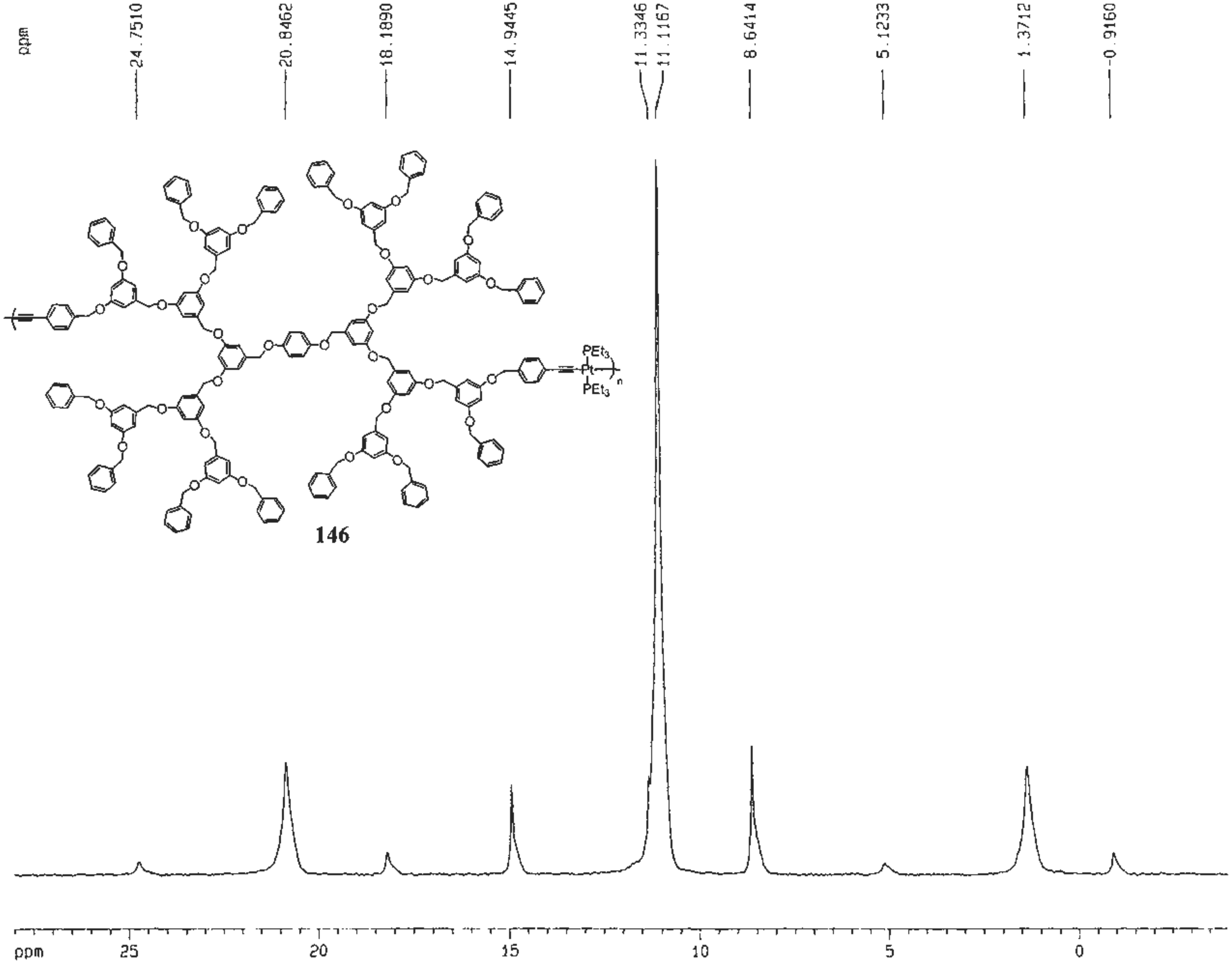
F2 - Acquisition Parameters
Date_           20080718
Time               23.14
INSTRUM           dpx300
PROBHD   5 mm B80 8B-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDC13
NS                  2764
DS                     0
SWH           22675 736 Hz
FIDRES         0.346004 Hz
AQ            1 4451188 sec
RG                7298.2
DW                22.050 usec
DE                  6.00 usec
TE                 296.2 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SF01        75 4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100 00 usec
PL2               120.00 dB
PL12               19.00 dB
SF02       300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677523 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.01 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9 54545 ppm/cm
HZCM           720.37390 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(Pt-G3)NP
EXPNO                  1
PROCNO                 1

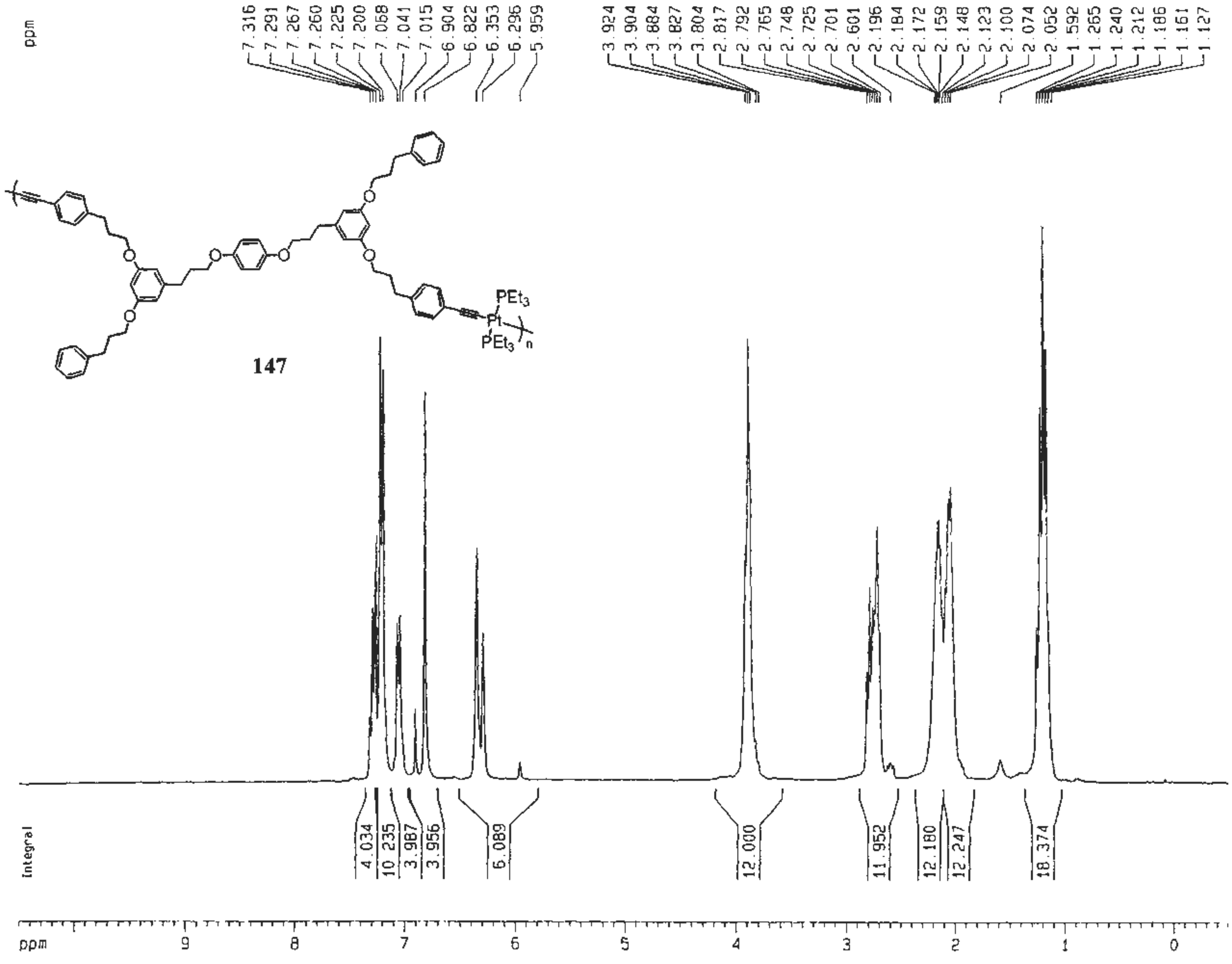
F2 - Acquisition Parameters
Date_           20080916
Time               22.03
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDC13
NS                   800
DS                     0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                7298.2
DW                13.275 usec
DE                  6.00 usec
TE                 296.2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121 4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3 00 Hz
GB                     0
PC                  1 40

1D NMR plot parameters
CX                 22.00 cm
CY                 13.01 cm
F1P               28.000 ppm
F1               3401.86 Hz
F2P               -4.000 ppm
F2               -485.98 Hz
PPMCM            1.45455 ppm/cm
HZCM           176.71976 Hz/cm
```



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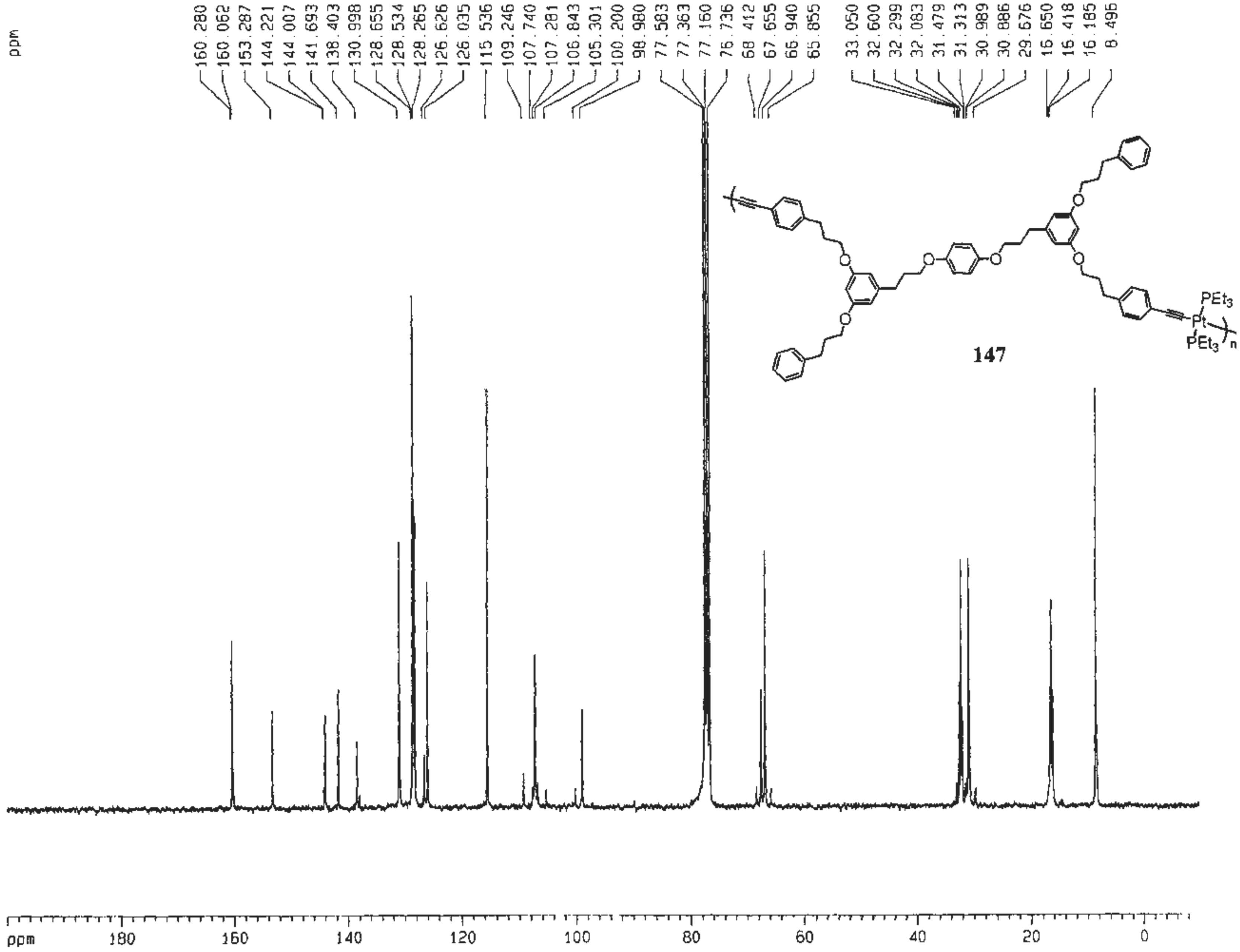
```
Current Data Parameters
NAME        Poly(Pt-G1')
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080711
Time              22.21
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                161.3
DW              138.825 usec
DE               198.32 usec
TE                296.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300065 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.01 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



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```
Current Data Parameters
NAME      Poly(Pt-G1')C
EXPNO                 1
PROCNO                1

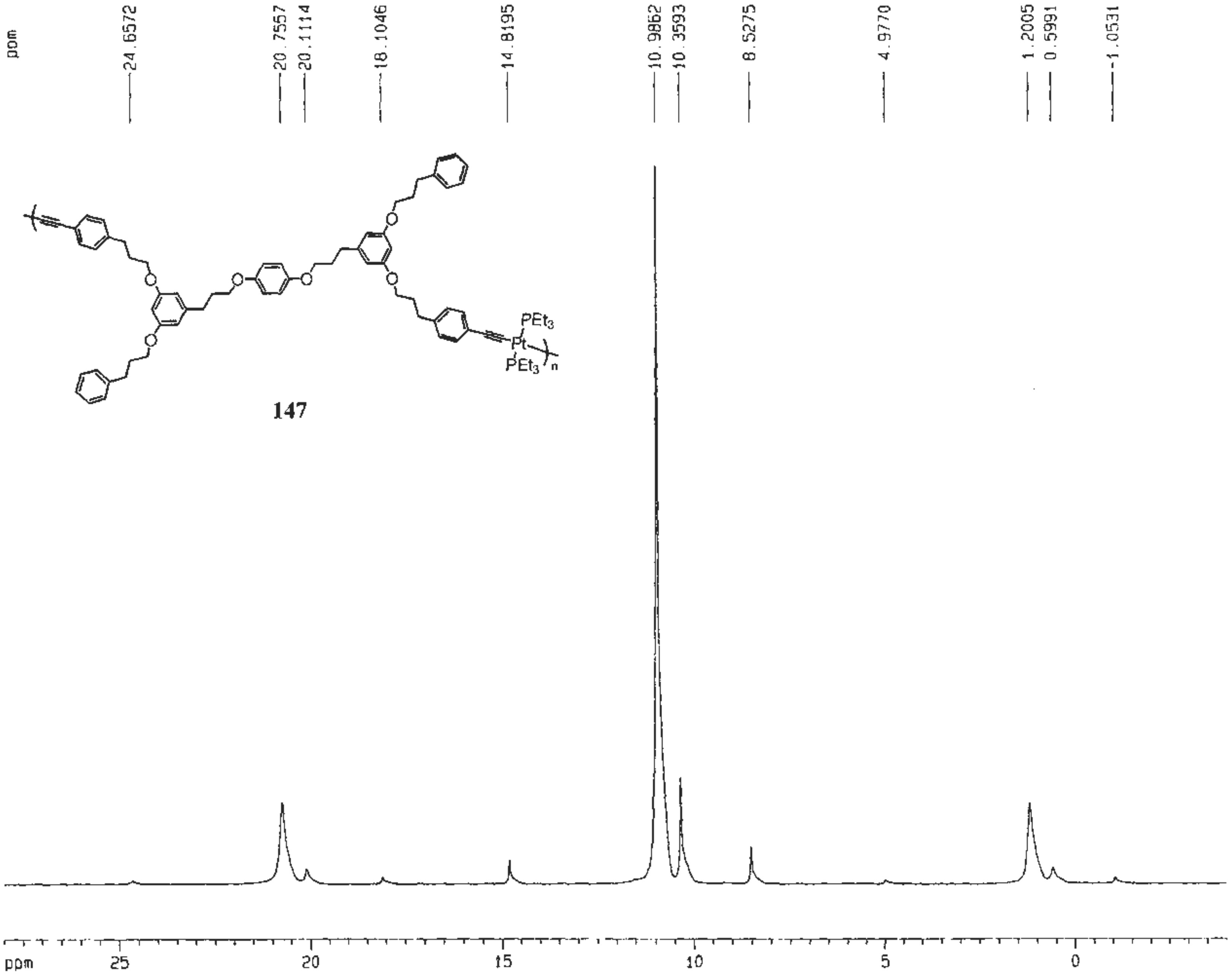
F2 - Acquisition Parameters
Date_          20080719
Time               1.44
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                14969
DS                    0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677408 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                22.18 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37384 Hz/cm
```



147

```
Current Data Parameters
NAME       Poly(Pt-G1')P
EXPNO                  :
PROCNO                 1

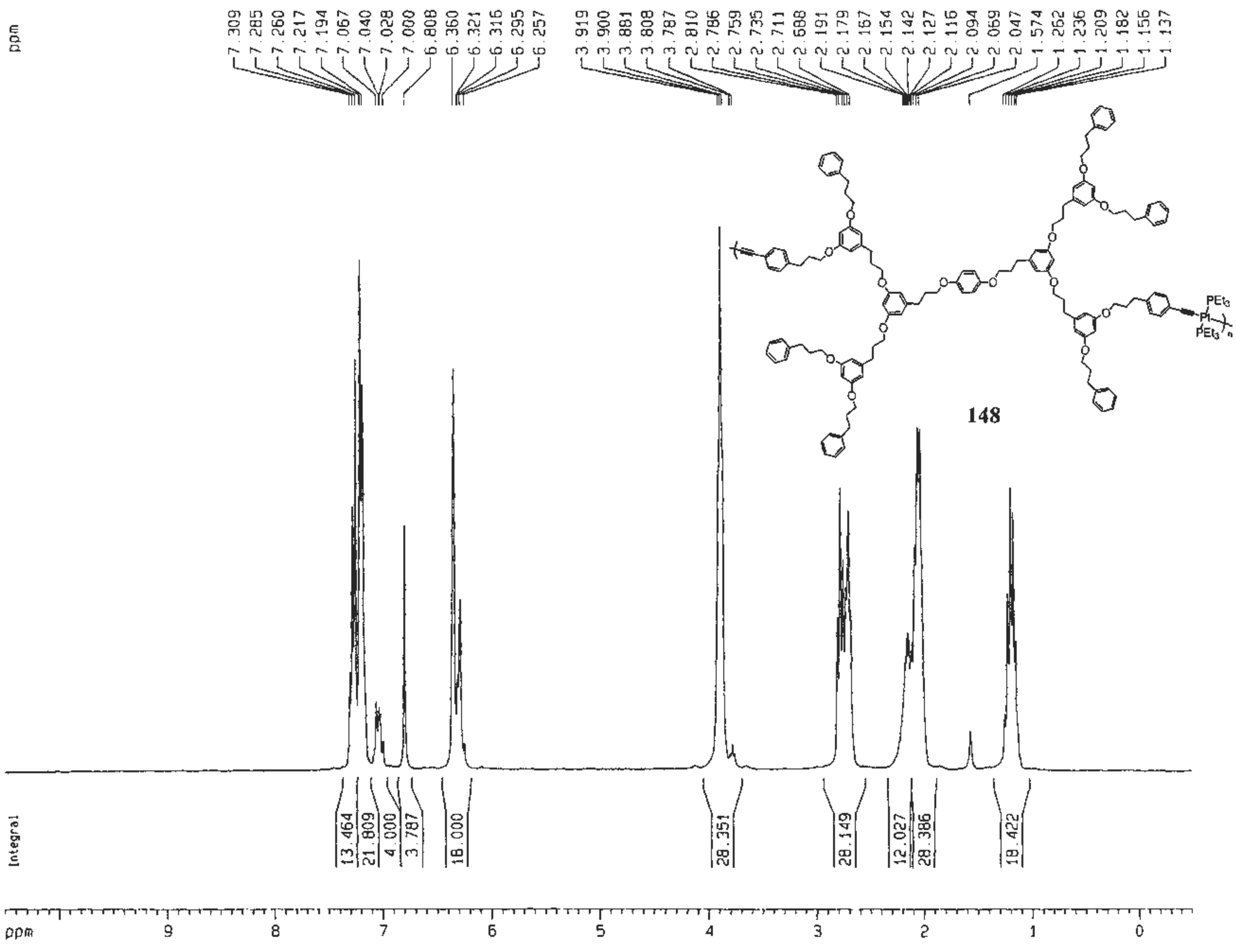
F2 - Acquisition Parameters
Date_           20080916
Time               23.55
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                 26000
DS                     0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG               10321.3
DW                13.275 usec
DE                  6.00 usec
TE                 295.2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121 4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100 00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.96 cm
F1P               28.000 ppm
F1               3401.86 Hz
F2P               -4.000 ppm
F2               -485.98 Hz
PPMCM            1.45455 ppm/cm
HZCM           176.71977 Hz/cm
```



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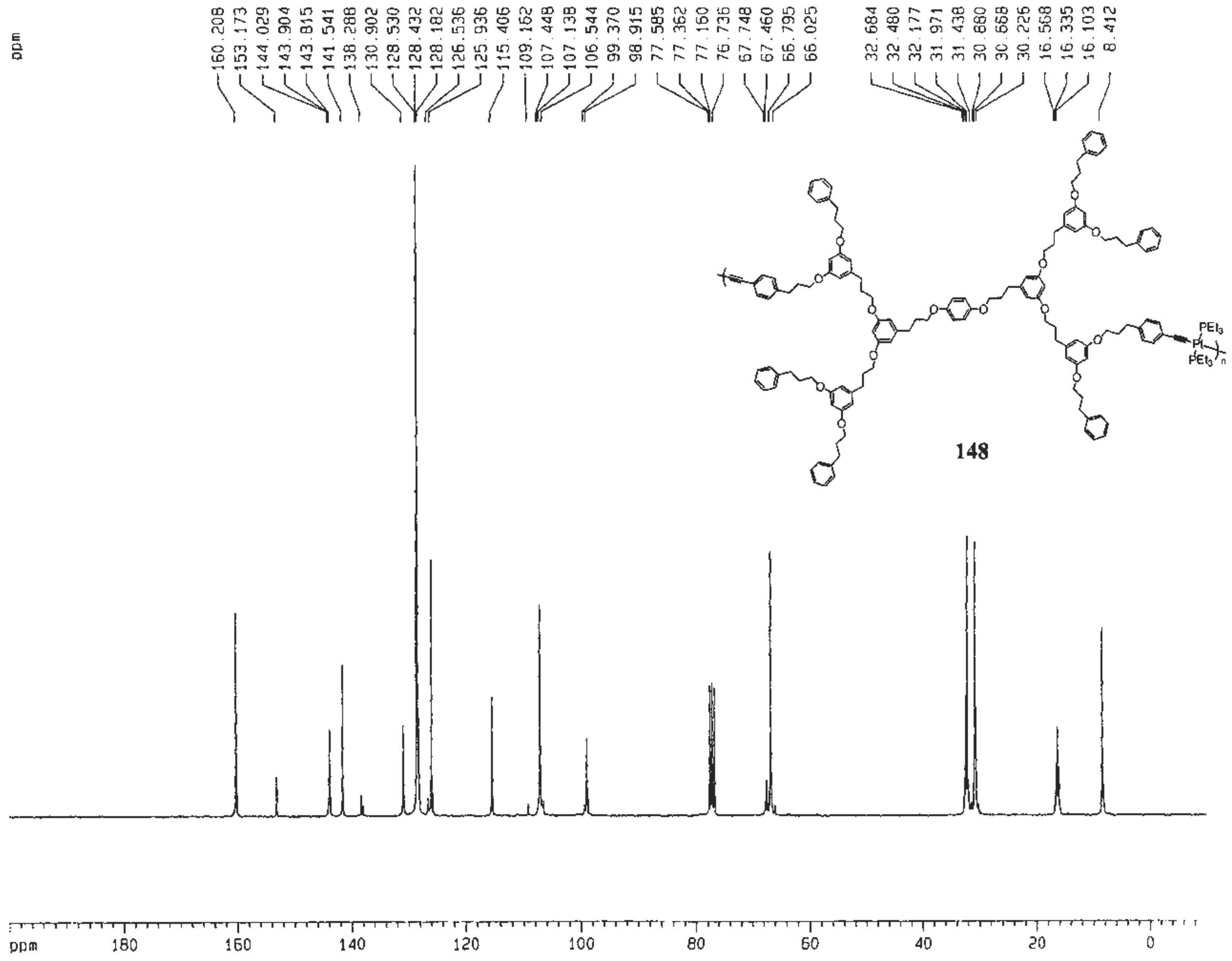
```
Current Data Parameters
NAME        Poly(Pt-G2')
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080711
Time               22.14
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    32
DS                     0
SWH          3501.657 Hz
FIDRES       0.219828 Hz
AQ         2.2745588 sec
RG                 161.3
DW         138.825 usec
DE          198.32 usec
TE               295.2 K
D1        5.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1             9.00 usec
PL1              -2.00 dB
SFO1      300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF        300.1300061 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              22.00 cm
CY              10.00 cm
F1P           10.500 ppm
F1            3151.36 Hz
F2P           -0.500 ppm
F2            -150.07 Hz
PPMCM      0.50000 ppm/cm
HZCM      150.06500 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(Pt-G2')C
EXPNO                  1
PROCNO                 1

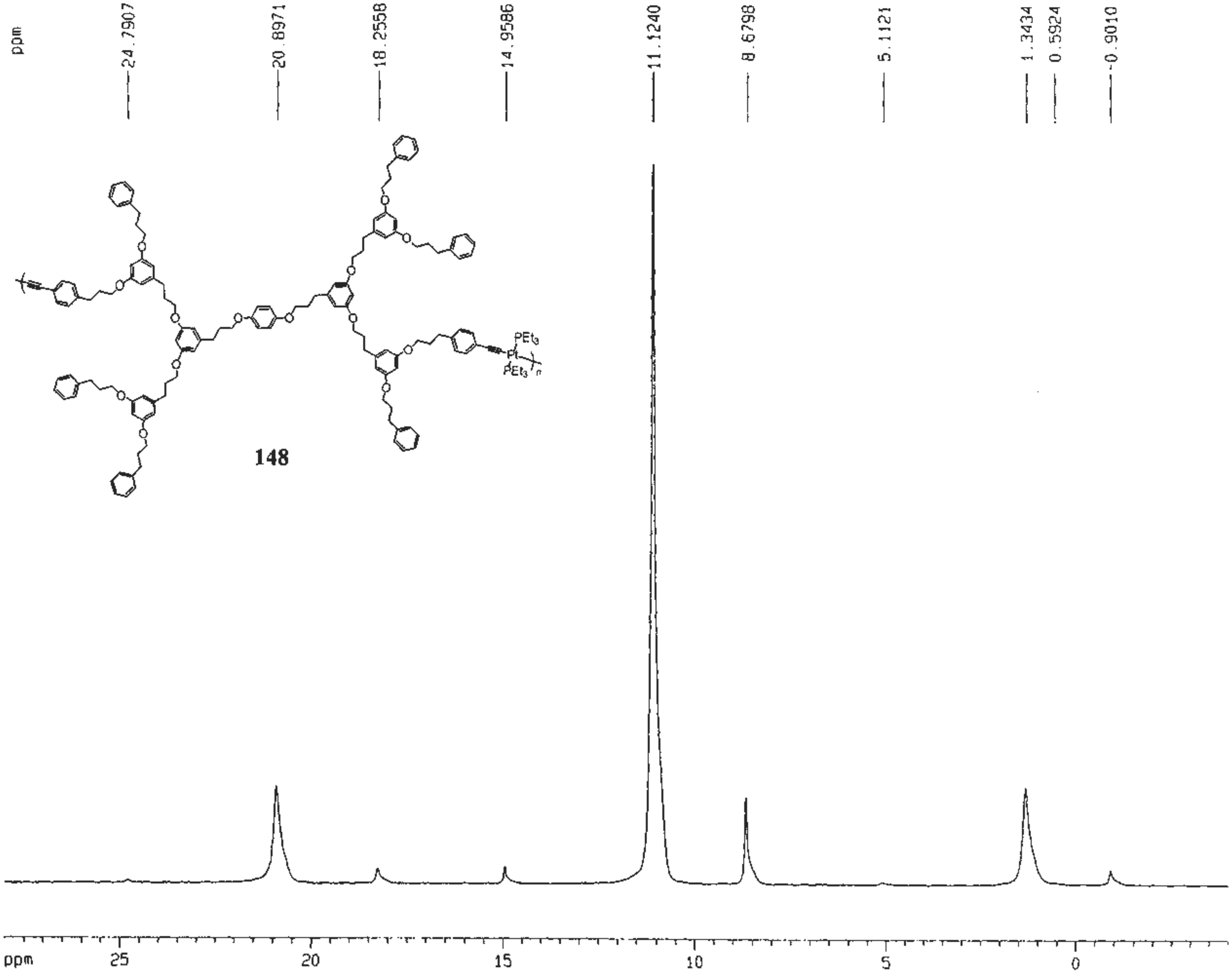
F2 - Acquisition Parameters
Date_           20080718
Time               18.58
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  2448
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 296.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677594 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.95 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37402 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(Pt-G2')P
EXPNO                  1
PROCNO                 1

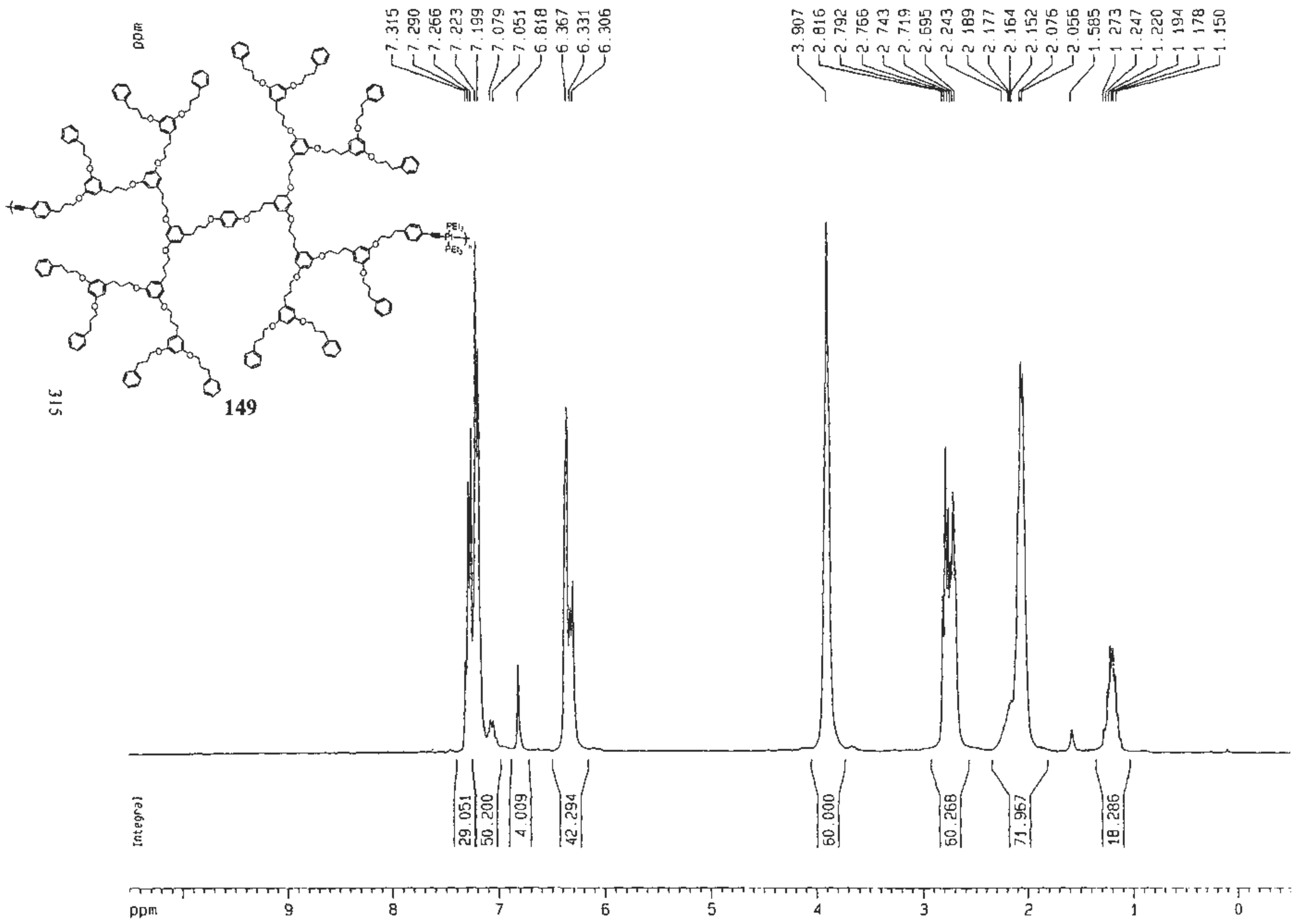
F2 - Acquisition Parameters
Date_           20080916
Time               23.09
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   700
DS                     0
SWH           37664.785 Hz
FIDRES         0.574719 Hz
AQ            0 8700404 sec
RG                4597.6
DW                13.275 usec
DE                  6.00 usec
TE                 295.2 K
D1            0 30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121 4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.97 cm
F1P               28.000 ppm
F1               3401.86 Hz
F2P               -4.000 ppm
F2               -485.98 Hz
PPMCM            1.45455 ppm/cm
HZCM           176.71977 Hz/cm
```



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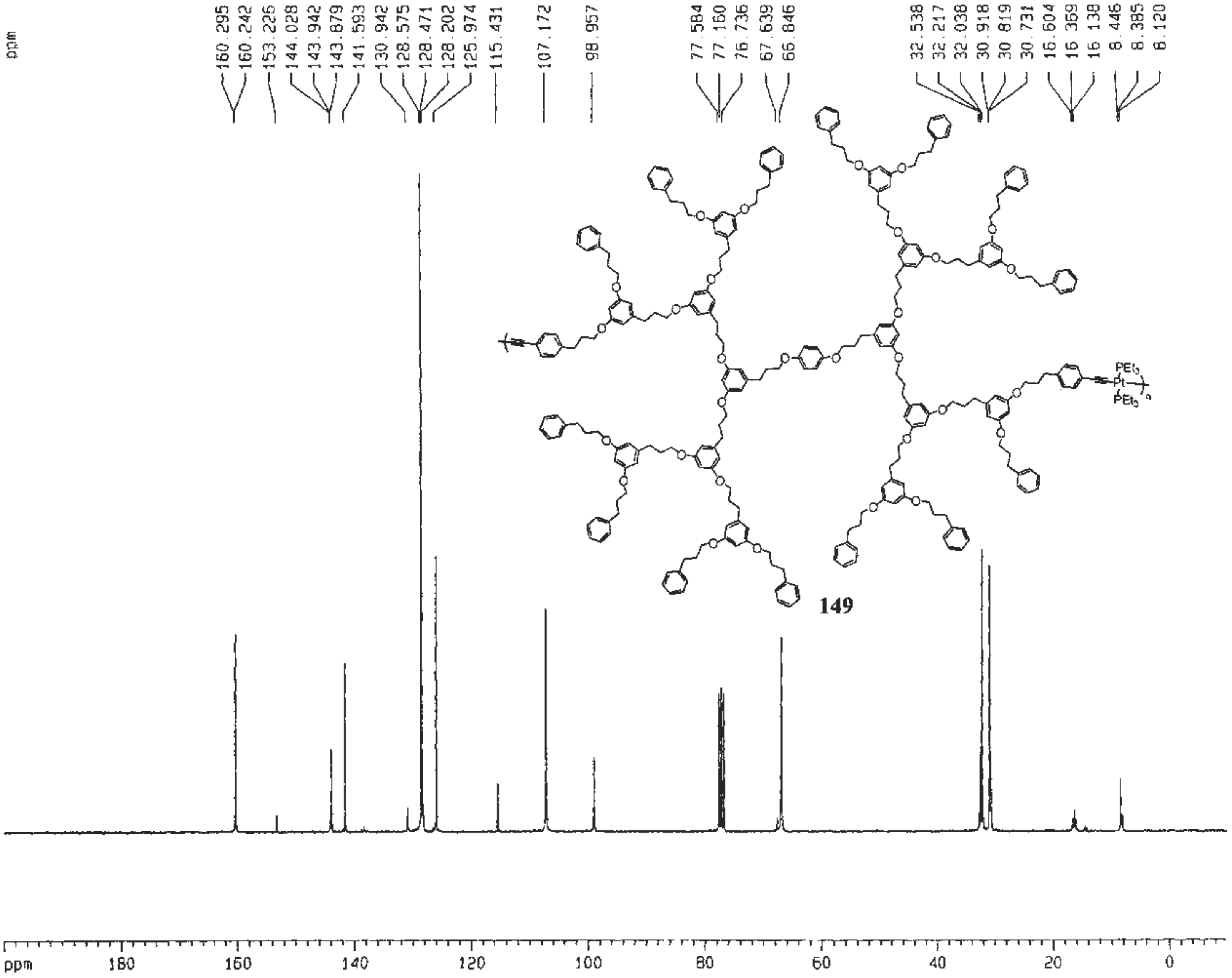
```
Current Data Parameters
NAME        Poly(Pt-G3')
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080718
Time               23.54
INSTRUM           dpx300
PROBHD   5 mm BBC BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             3601 657 Hz
FIDRES          0.219828 Hz
AQ             2.2745588 sec
RG                 101.6
DW               138.825 usec
DE                198.32 usec
TE                 296.2 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300 1300063 MHz
WDW                   EM
SSB                    0
LB                  0 30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22 00 cm
CY                  9.98 cm
F1P               10.500 ppm
F1               3151 36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(Pt-G3')C
EXPNO                  1
PROCNO                 1

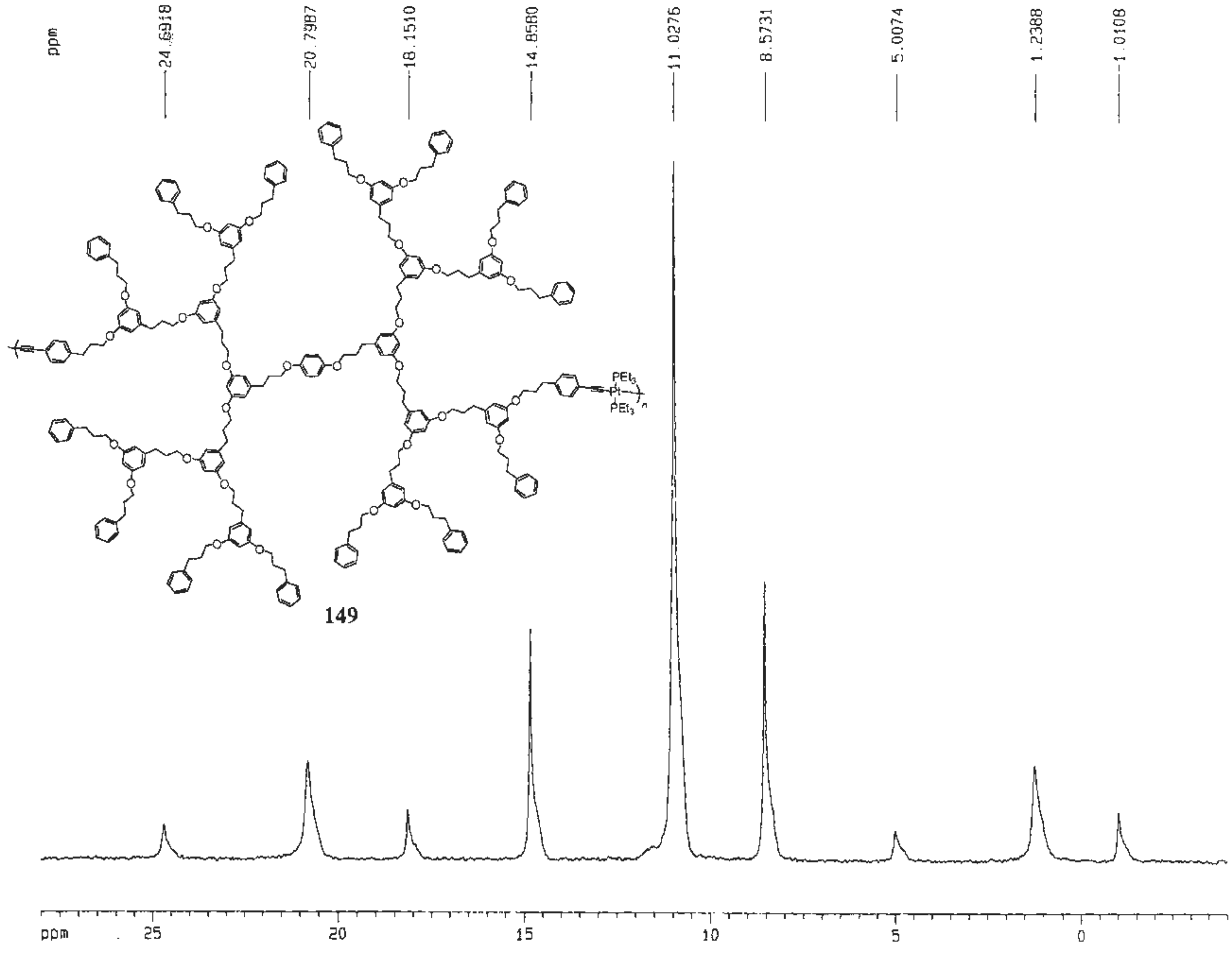
F2 - Acquisition Parameters
Date_           20080719
Time                0.13
INSTRUM           dpx300
PROBHD   5 mm QNP 8B-1H
PULPROG             zgcc
TD                 65536
SOLVENT            CDCl3
NS                  2000
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 295.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677538 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.00 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(Pt-G3')P
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080916
Time               23.28
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   900
DS                     0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                7298.2
DW                13.275 usec
DE                  6.00 usec
TE                 295.2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 13.02 cm
F1P               28.000 ppm
F1               3401.86 Hz
F2P               -4.000 ppm
F2               -485.98 Hz
PPMCM            1.45455 ppm/cm
HZCM           176.71977 Hz/cm
```

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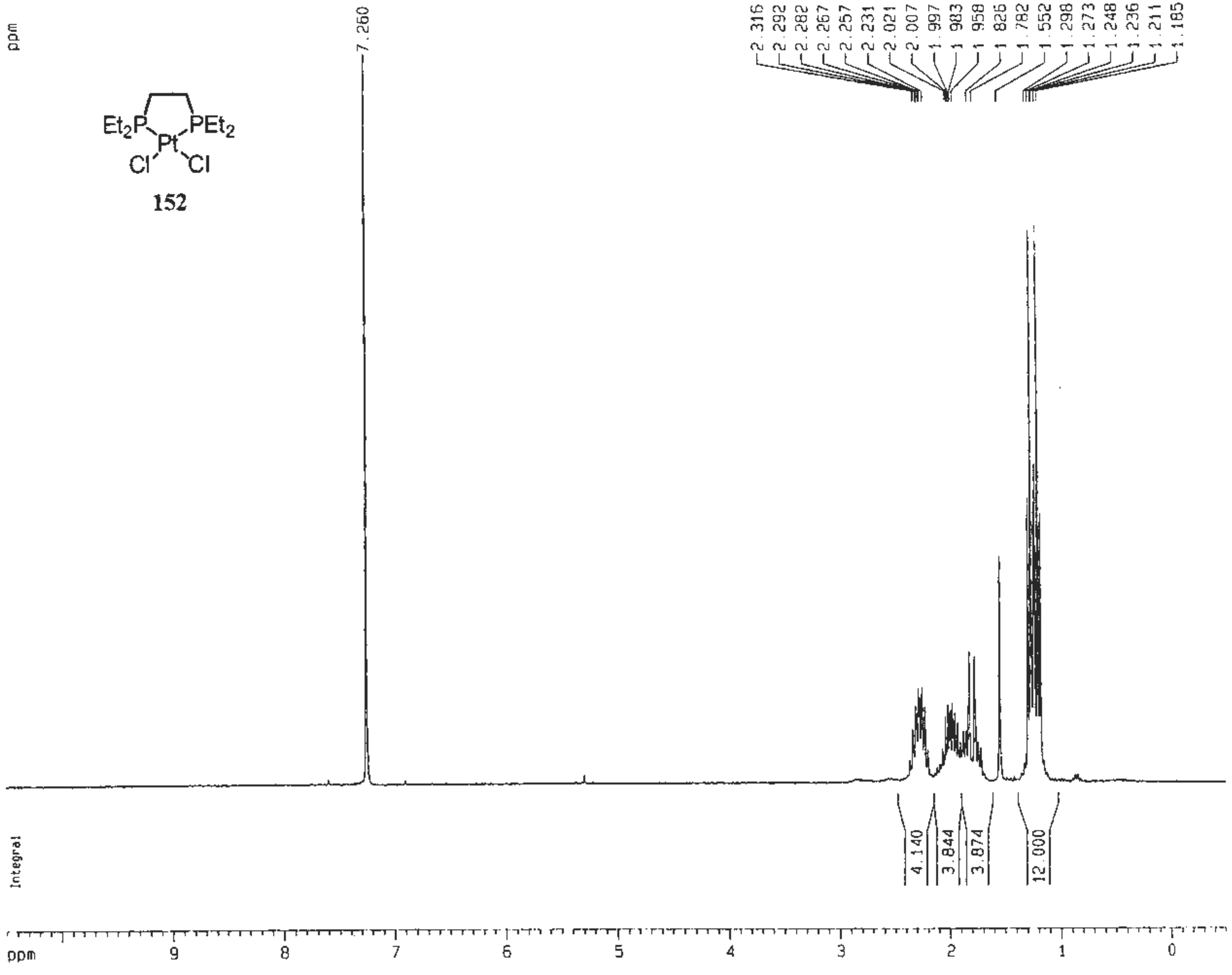


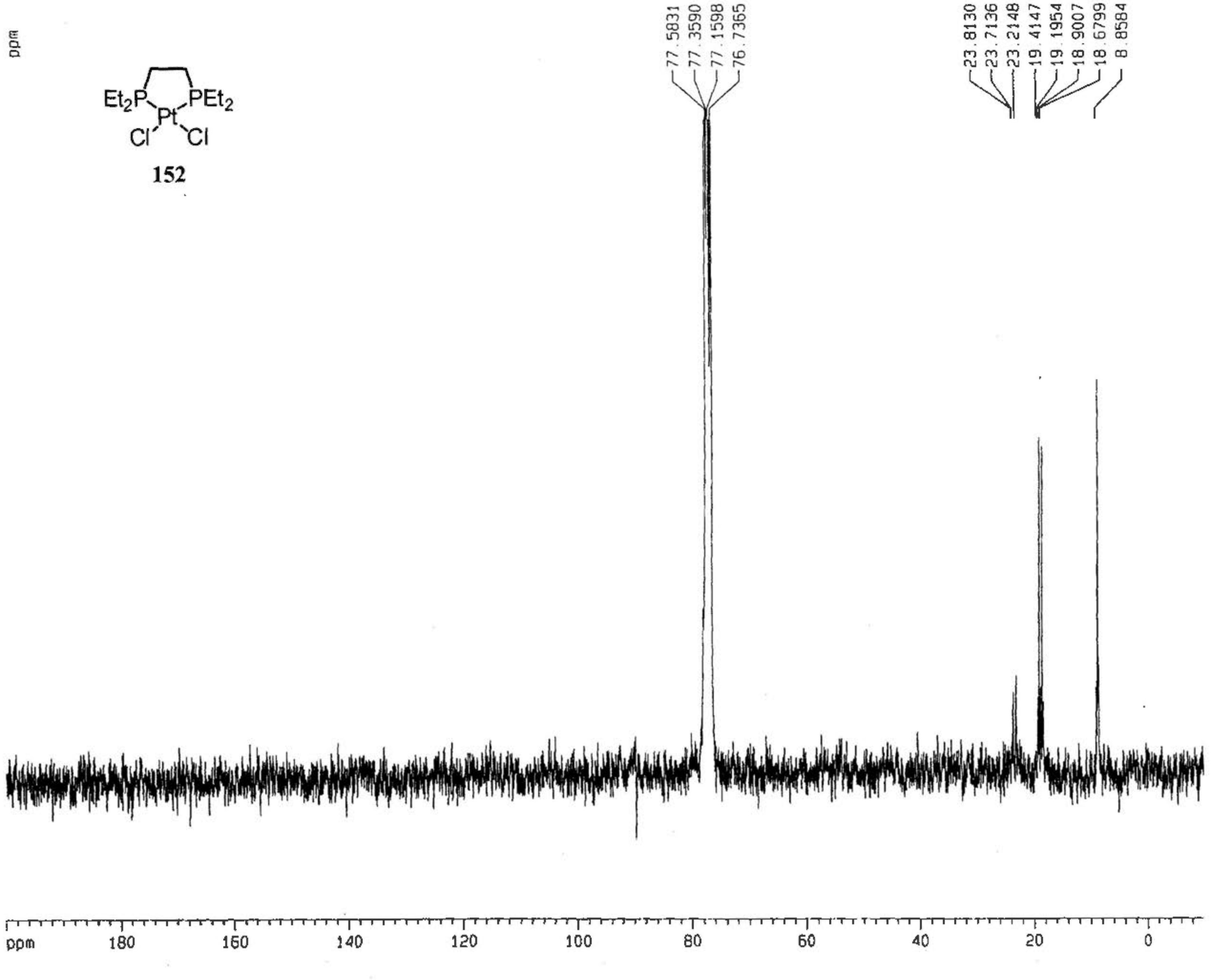
```
Current Data Parameters
NAME     cis-Pt-Linker4(Solid1)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081027
Time               16.24
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                   216
DS                     0
SWH           3601 657 Hz
FIDRES        0.219828 Hz
AQ           2 2745588 sec
RG                  2048
DW             138.825 usec
DE              198.32 usec
TE               296 2 K
D1          1 00000000 sec
MCREST      0 00000000 sec
MCWRK       0 01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  4 50 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                 16384
SF           300 1300054 MHz
WDW                   EM
SSB                    0
LB                  0 30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 15.74 cm
F1P               10 500 ppm
F1               3151.36 Hz
F2P               -0 500 ppm
F2               -150.07 Hz
PPMCM            0 50000 ppm/cm
HZCM           150.06500 Hz/cm
```



152

```
Current Data Parameters
NAME      cis-Pt-Linker4C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20081201
Time                23.22
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                  13300
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                  295.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

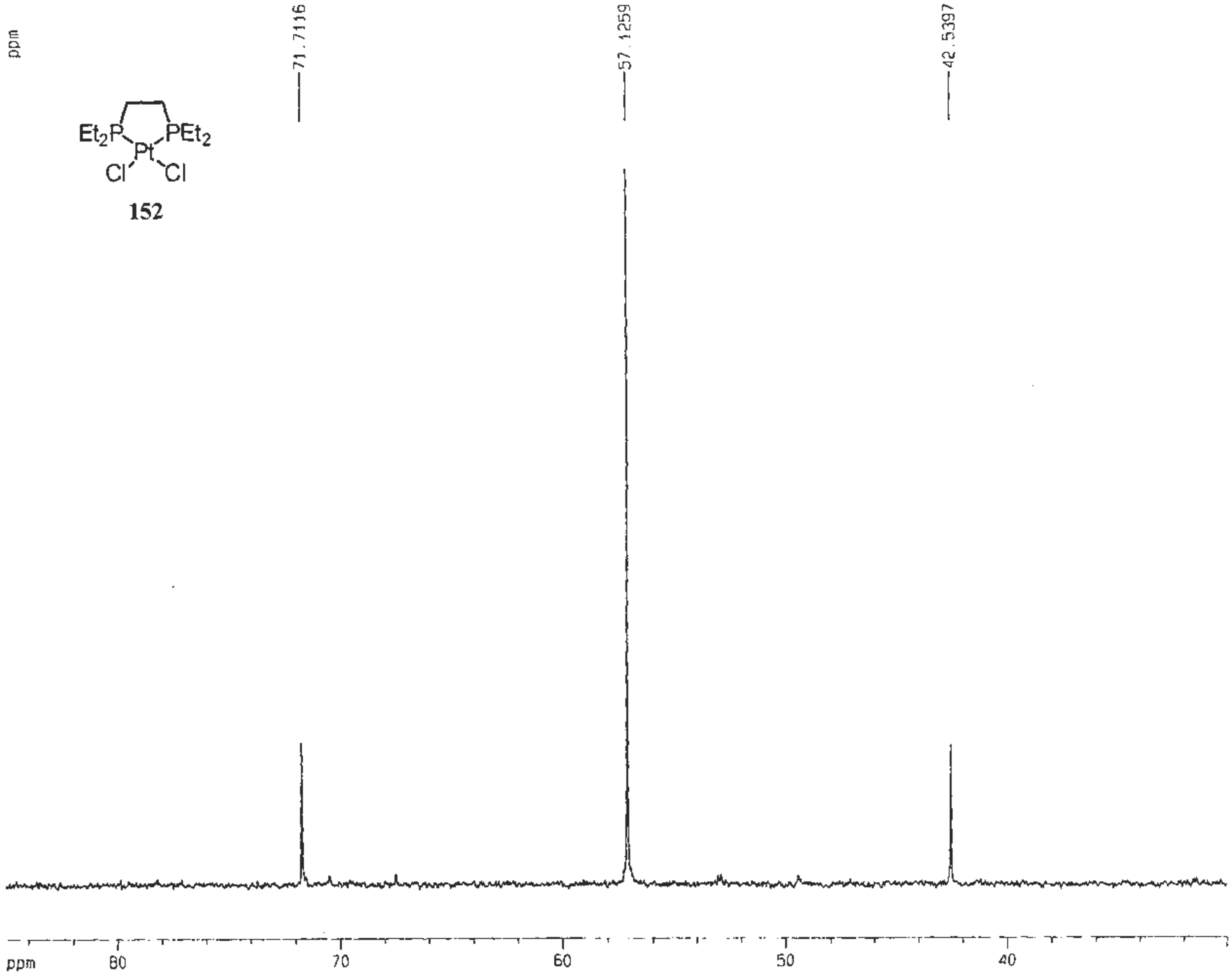
======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677388 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                 155.05 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37378 Hz/cm
```

152



```
Current Data Parameters
NAME      cis-Pt-Linker4P
EXPNO                   1
PROCNO                  1

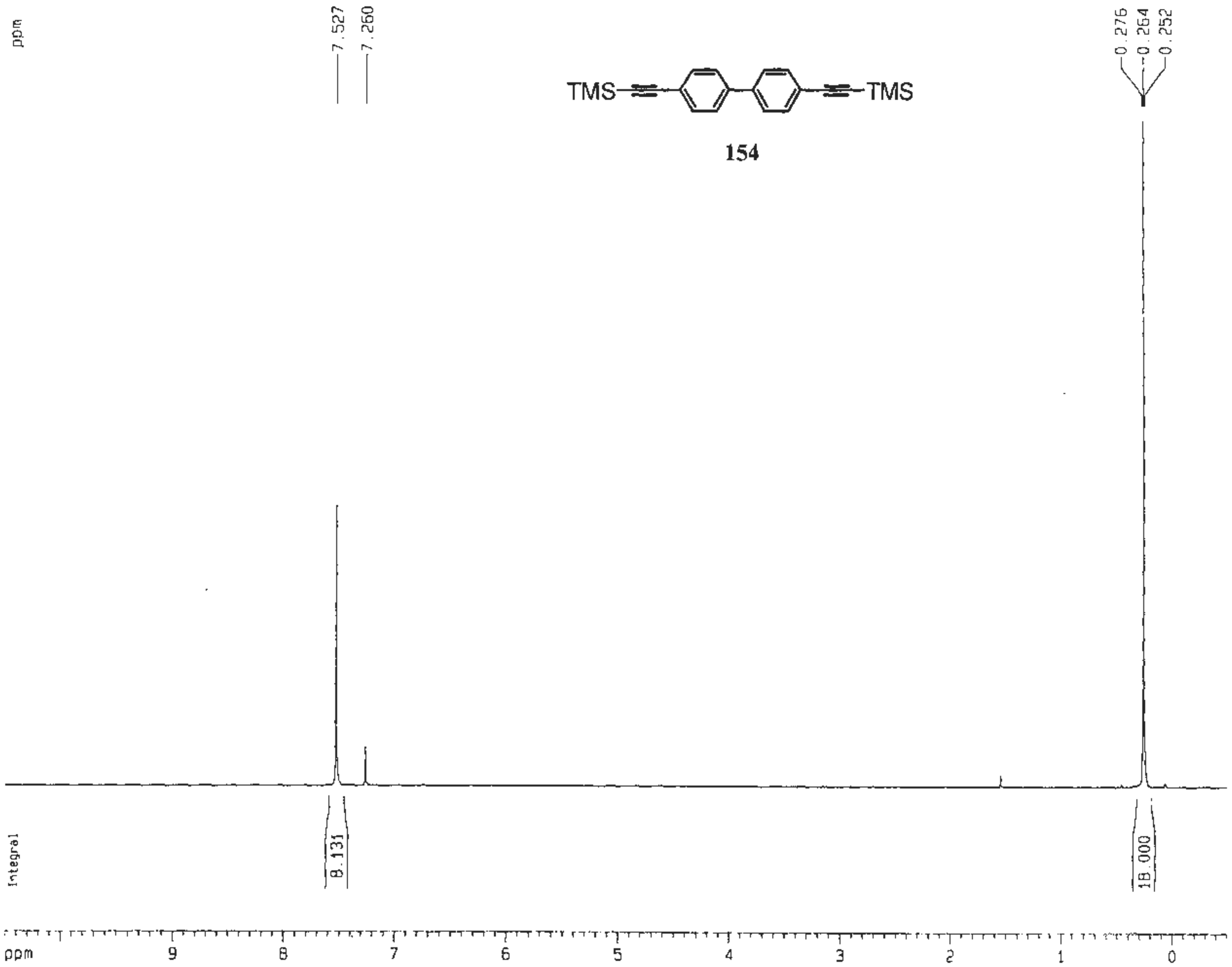
F2 - Acquisition Parameters
Date_           20081028
Time               18.57
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     0
SWH           37664 785 Hz
FIDRES         0 574719 Hz
AQ            0.8700404 sec
RG                7298 2
DW              13.275 usec
DE                6 00 usec
TE               298 2 K
D1           0 30000001 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6 00 dB
SFO1        121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100 00 usec
PL2               120 00 dB
PL12               19.00 dB
SFO2        300 1316097 MHz

F2 - Processing parameters
SI                 65536
SF          121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22 00 cm
CY                 13.00 cm
F1P               85.000 ppm
F1              10327.06 Hz
F2P               30.000 ppm
F2               3644.85 Hz
PPMCM            2 50000 ppm/cm
HZCM           303 73712 Hz/cm
```



154

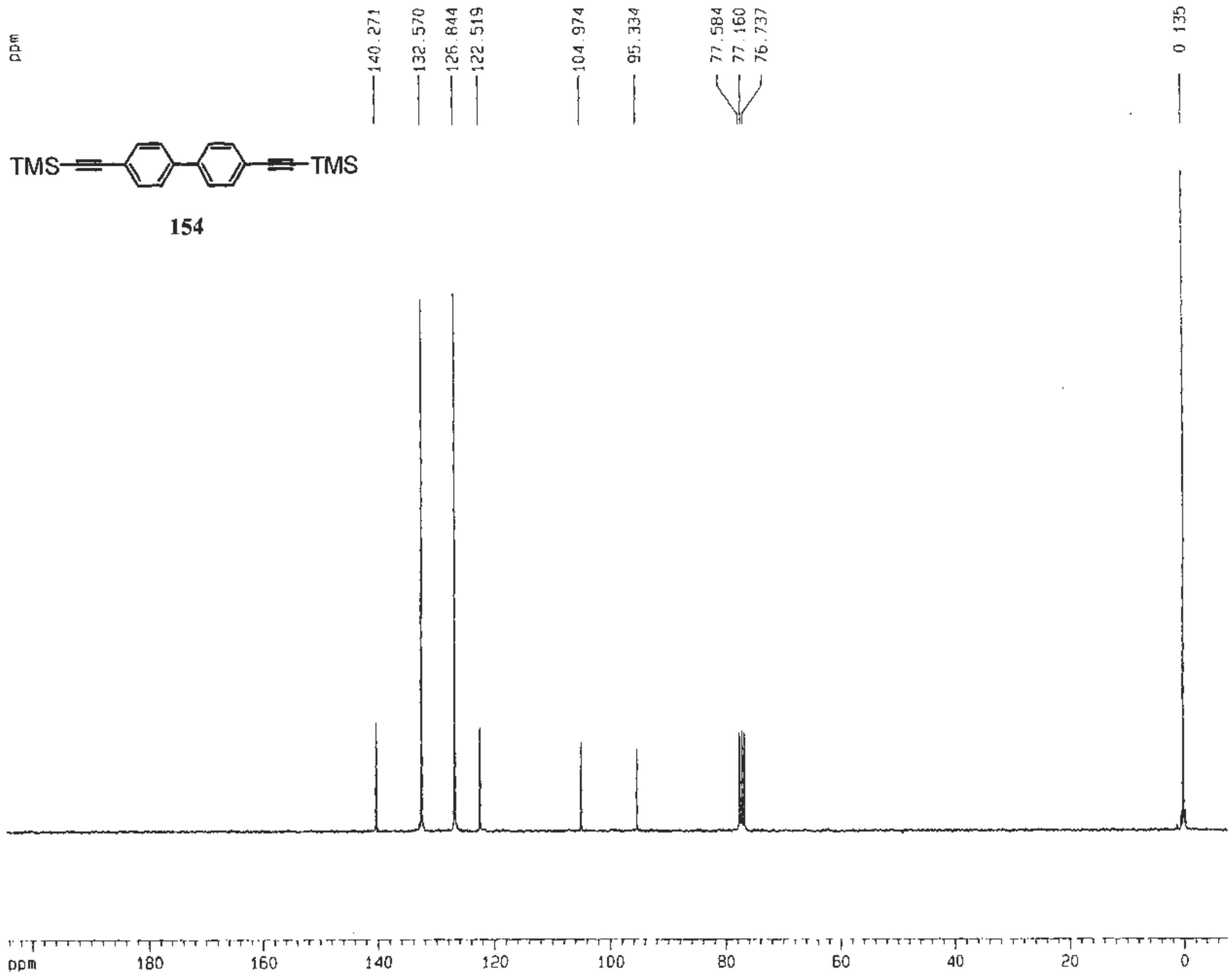
```
Current Data Parameters
NAME       TMS-BiPh-TMS
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080711
Time              19.13
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                  362
DW              138.825 usec
DE               198.32 usec
TE                295.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300064 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                11.96 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



154

```
Current Data Parameters
NAME       TMS-BiPh-TMSC
EXPNO                  1
PROCNO                 1

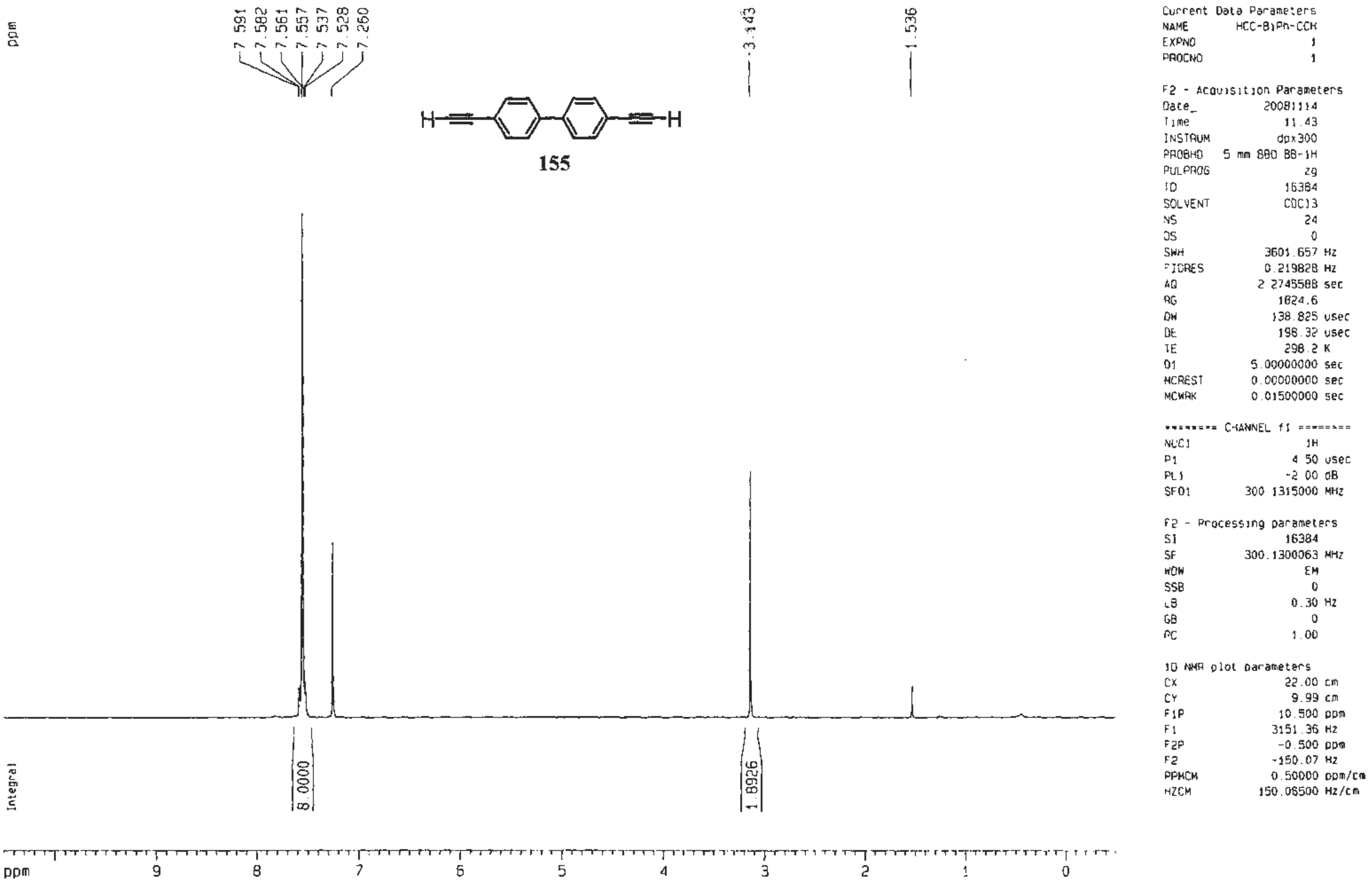
F2 - Acquisition Parameters
Date_           20081120
Time               18.34
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   452
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW               22.050 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677419 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.98 cm
F1P              204.375 ppm
F1              15423.75 Hz
F2P               -7.698 ppm
F2               -580.97 Hz
PPMCM            9.63972 ppm/cm
HZCM           727.48755 Hz/cm
```



155

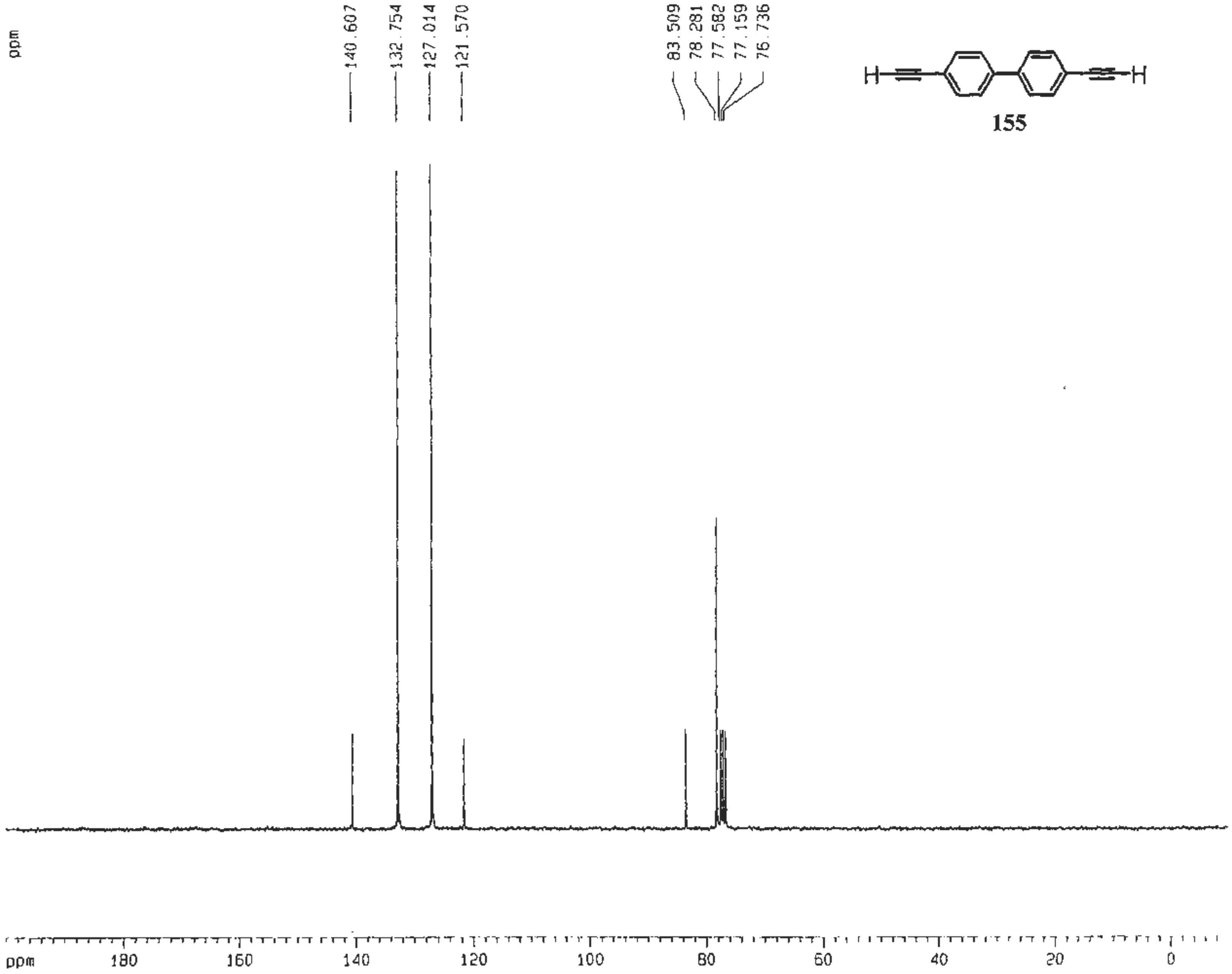
```
Current Data Parameters
NAME       HCC-BiPh-CCH
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081114
Time              11.43
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   24
DS                    0
SWH         3601.657 Hz
FIDRES      0.219828 Hz
AQ         2.2745588 sec
RG               1824.6
DW          138.825 usec
DE           198.32 usec
TE              298.2 K
D1        5.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1             4.50 usec
PL1            -2.00 dB
SFO1     300.1315000 MHz

F2 - Processing parameters
SI                16384
SF       300.1300063 MHz
WDW                  EM
SSB                   0
LB              0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX             22.00 cm
CY              9.99 cm
F1P          10.500 ppm
F1           3151.36 Hz
F2P          -0.500 ppm
F2           -150.07 Hz
PPMCM    0.50000 ppm/cm
HZCM    150.06500 Hz/cm
```



155

```
Current Data Parameters
NAME       HCC-BiPh-CCHC
EXPNO                  1
PROCNO                 1

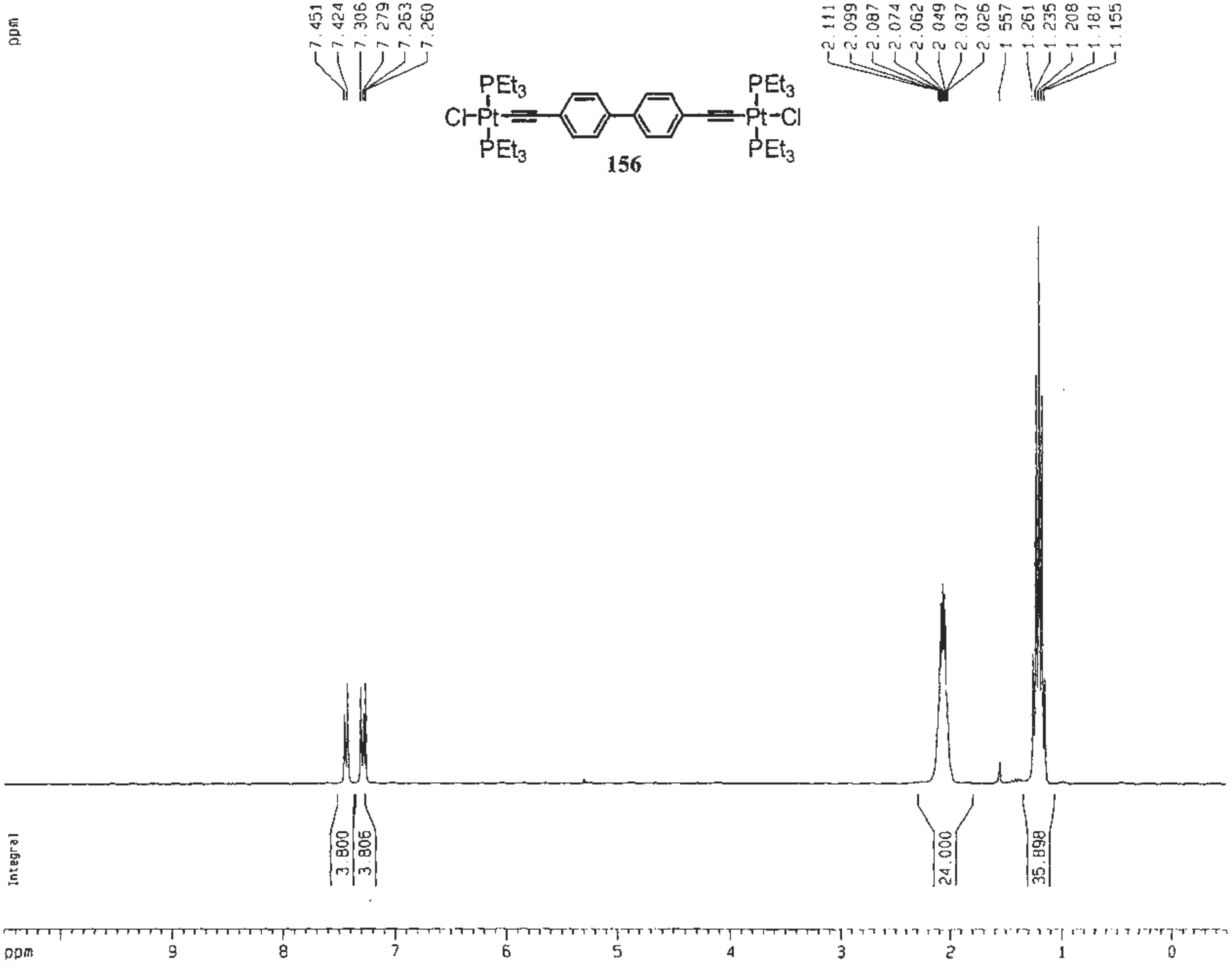
F2 - Acquisition Parameters
Date_           20081120
Time               22.17
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                  8192
DW               22.050 usec
DE                 6.00 usec
TE                297.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677431 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.97 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37384 Hz/cm
```


156

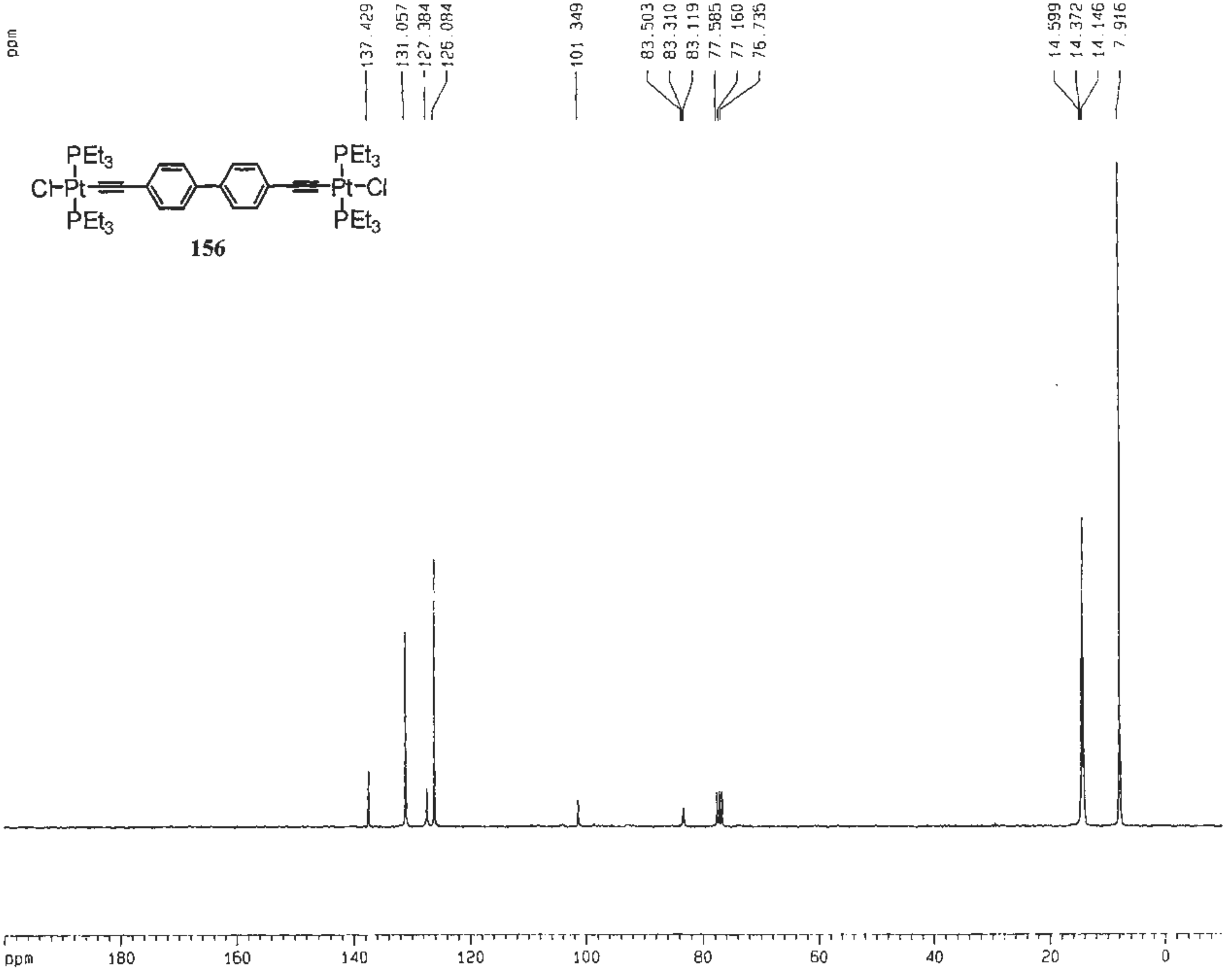
```
Current Data Parameters
NAME        L-Pt-Linker
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081120
Time              21 56
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   24
DS                    0
SWH            3601 657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                322.5
DW              138 825 usec
DE               198.32 usec
TE                295.2 K
D1           5 00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 4.50 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                16384
SF          300.1300056 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.01 cm
F1P              10.500 ppm
F1              3151 36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



156

```
Current Data Parameters
NAME          L-Pt-LinkerC
EXPNO                    1
PROCNO                   1

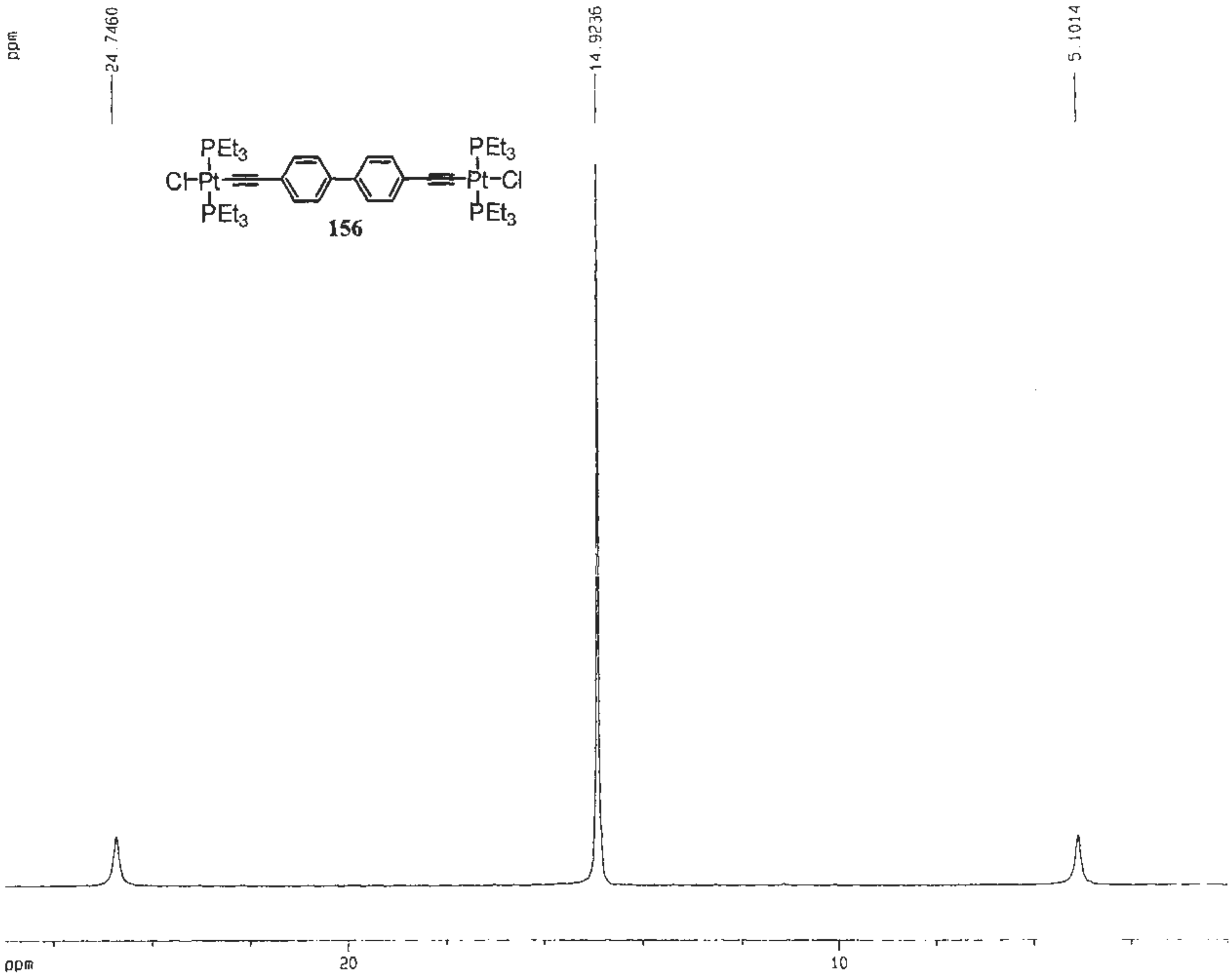
F2 - Acquisition Parameters
Date_             20081122
Time                  1 13
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     164
DS                       0
SWH              22675 736 Hz
FIDRES            0.346004 Hz
AQ               1 4451188 sec
RG                    8192
DW                  22 050 usec
DE                    6 00 usec
TE                   296.2 K
D1              1 00000000 sec
d11             0 03000000 sec
MCREST          0 00000000 sec
MCWRK           0 01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    3.00 usec
PL1                  -6 00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF              75 4677617 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   11.97 cm
F1P                200.000 ppm
F1                15093.55 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM              9.54545 ppm/cm
HZCM             720.37402 Hz/cm
```



156

```
Current Data Parameters
NAME        L-Pt-LinkerP
EXPNO                  1
PROCNO                 1

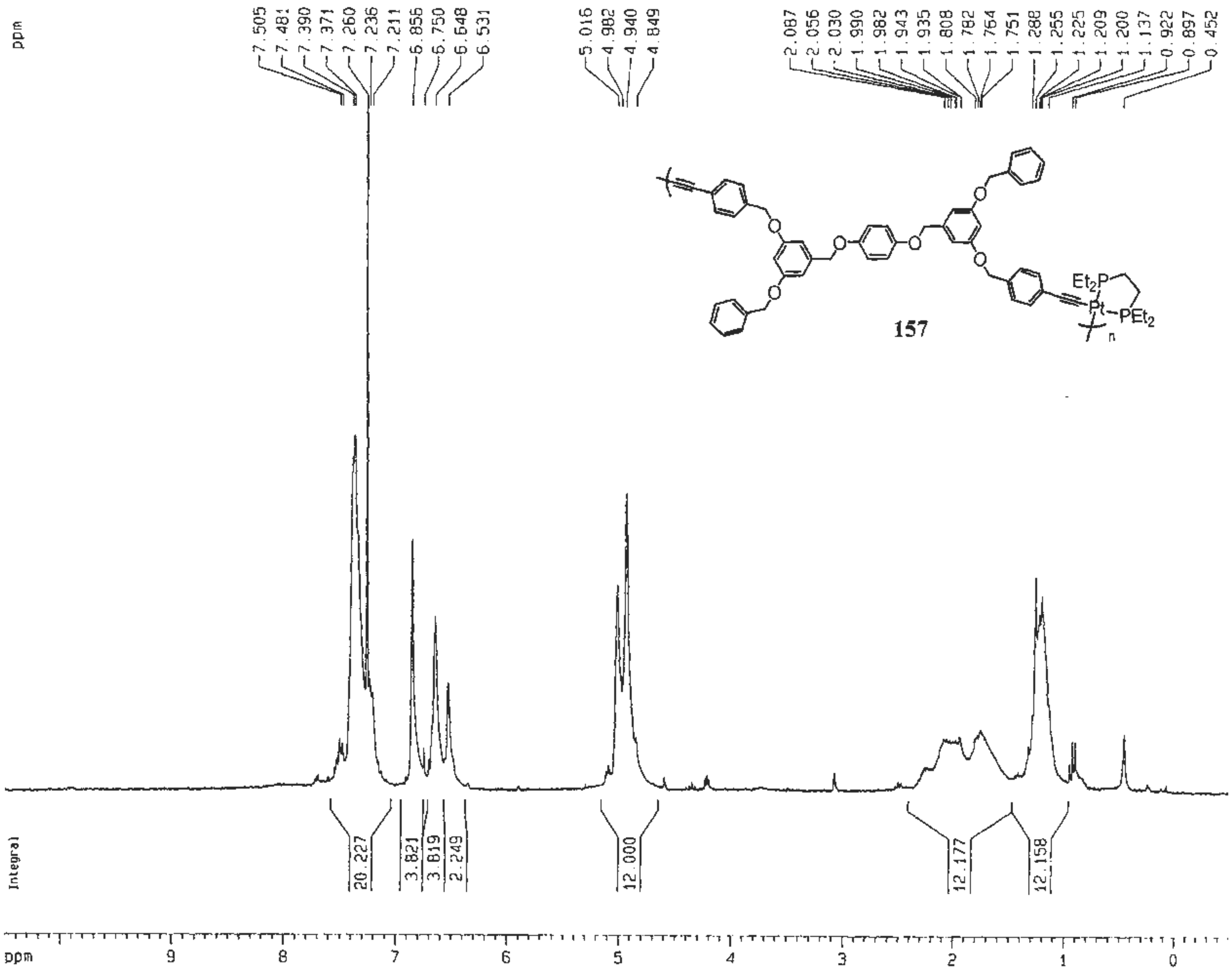
F2 - Acquisition Parameters
Date_           20081122
Time                1.31
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   112
DS                     0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                 724.1
DW                13.275 usec
DE                  6.00 usec
TE                 296.2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 13.01 cm
F1P               27.000 ppm
F1               3280.36 Hz
F2P                2.000 ppm
F2                242.99 Hz
PPMCM            1.13636 ppm/cm
HZCM           138.06232 Hz/cm
```



157

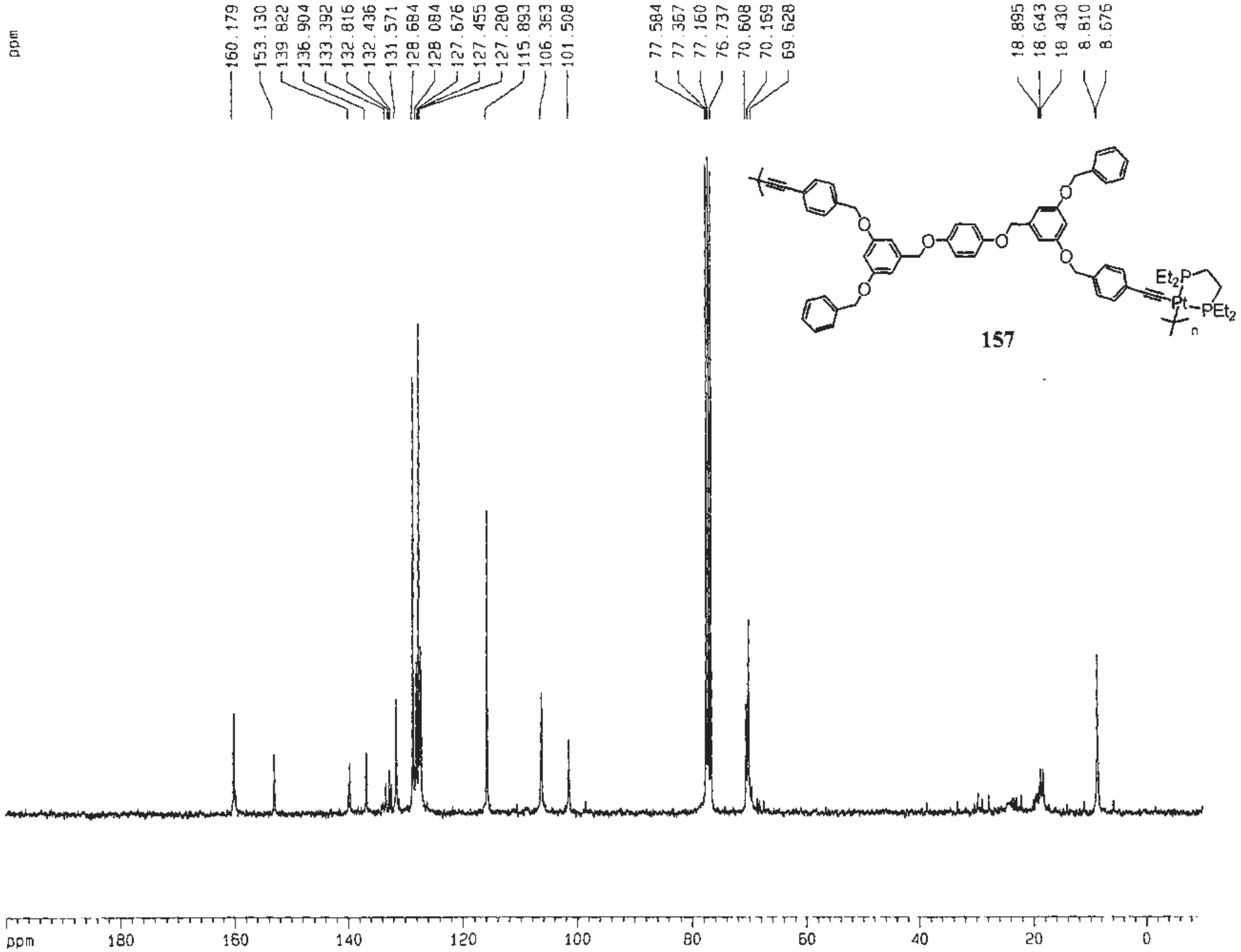
```
Current Data Parameters
NAME      Poly(cis-Pt-G1)N(ppt)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20081108
Time                  7.33
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                      64
DS                       0
SWH               3601.657 Hz
FIDRES            0.219828 Hz
AQ               2.2745588 sec
RG                   724.1
DW                 138.825 usec
DE                  198.32 usec
TE                   298.2 K
D1              5.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    4.50 usec
PL1                  -2.00 dB
SFO1           300.1315000 MHz

F2 - Processing parameters
SI                   16384
SF             300.1300060 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   19.94 cm
F1P                 10.500 ppm
F1                 3151.36 Hz
F2P                 -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.50000 ppm/cm
HZCM             150.06500 Hz/cm
```



157

```
Current Data Parameters
NAME       Poly(cis-Pt-G1)NC
EXPNO                      1
PROCNO                     1

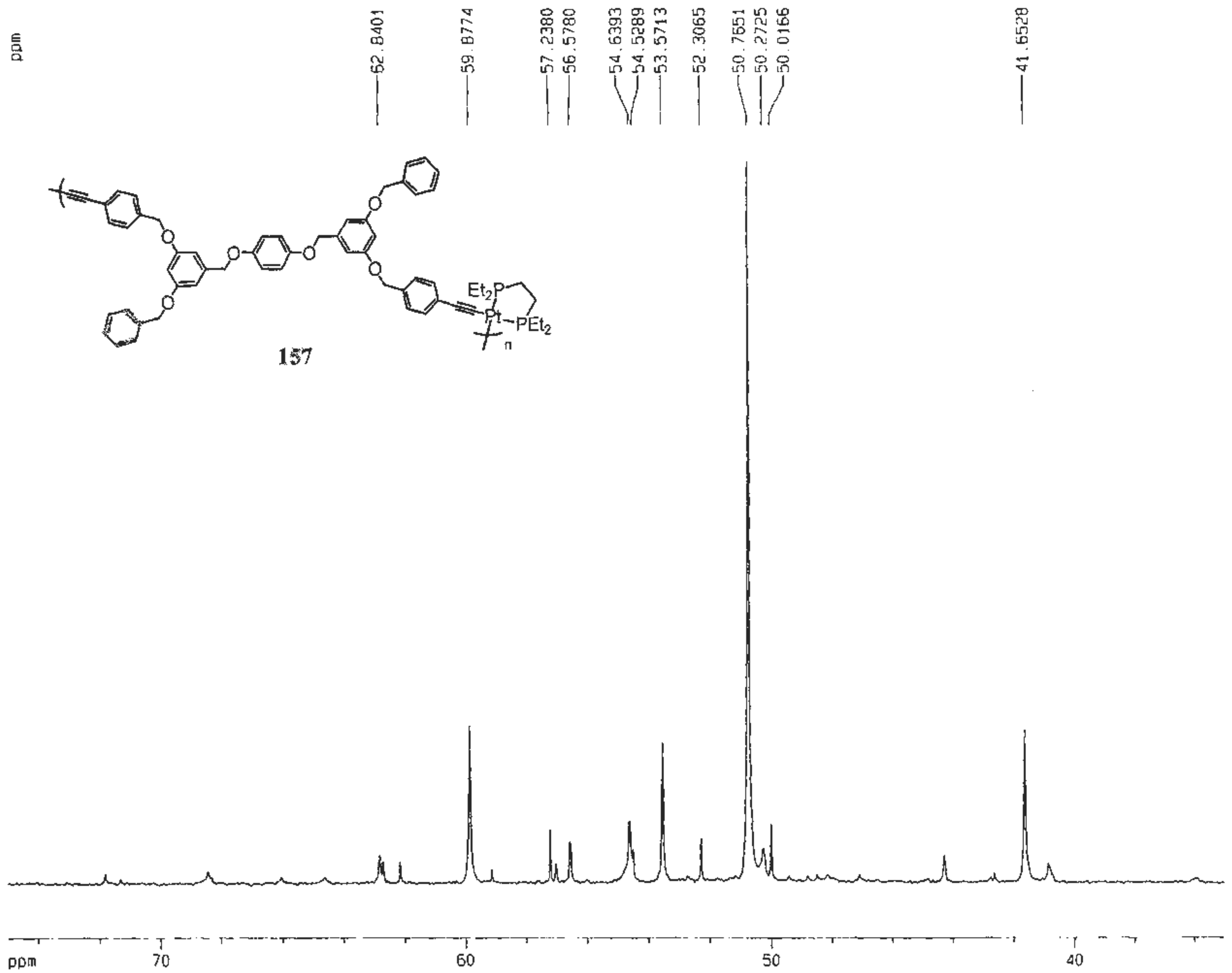
F2 - Acquisition Parameters
Date_               20081122
Time                    5.53
INSTRUM               dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zgdc
TD                     65536
SOLVENT                CDCl3
NS                      4600
DS                         0
SWH               22675 736 Hz
FIDRES             0.346004 Hz
AQ                1.4451188 sec
RG                      8192
DW                    22.050 usec
DE                      6 00 usec
TE                     296.2 K
D1                1.00000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                      3.00 usec
PL1                    -6.00 dB
SFO1              75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                   120.00 dB
PL12                   19.00 dB
SFO2            300 1315007 MHz

F2 - Processing parameters
SI                     65536
SF                75.4677423 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     22.00 cm
CY                     12.04 cm
F1P                  200.000 ppm
F1                  15093.55 Hz
F2P                  -10.000 ppm
F2                   -754 68 Hz
PPMCM                9.54545 ppm/cm
HZCM               720.37384 Hz/cm
```



157

```
Current Data Parameters
NAME      Poly(cis-Pt-G1)NP
EXPNO                  1
PROCNO                 1

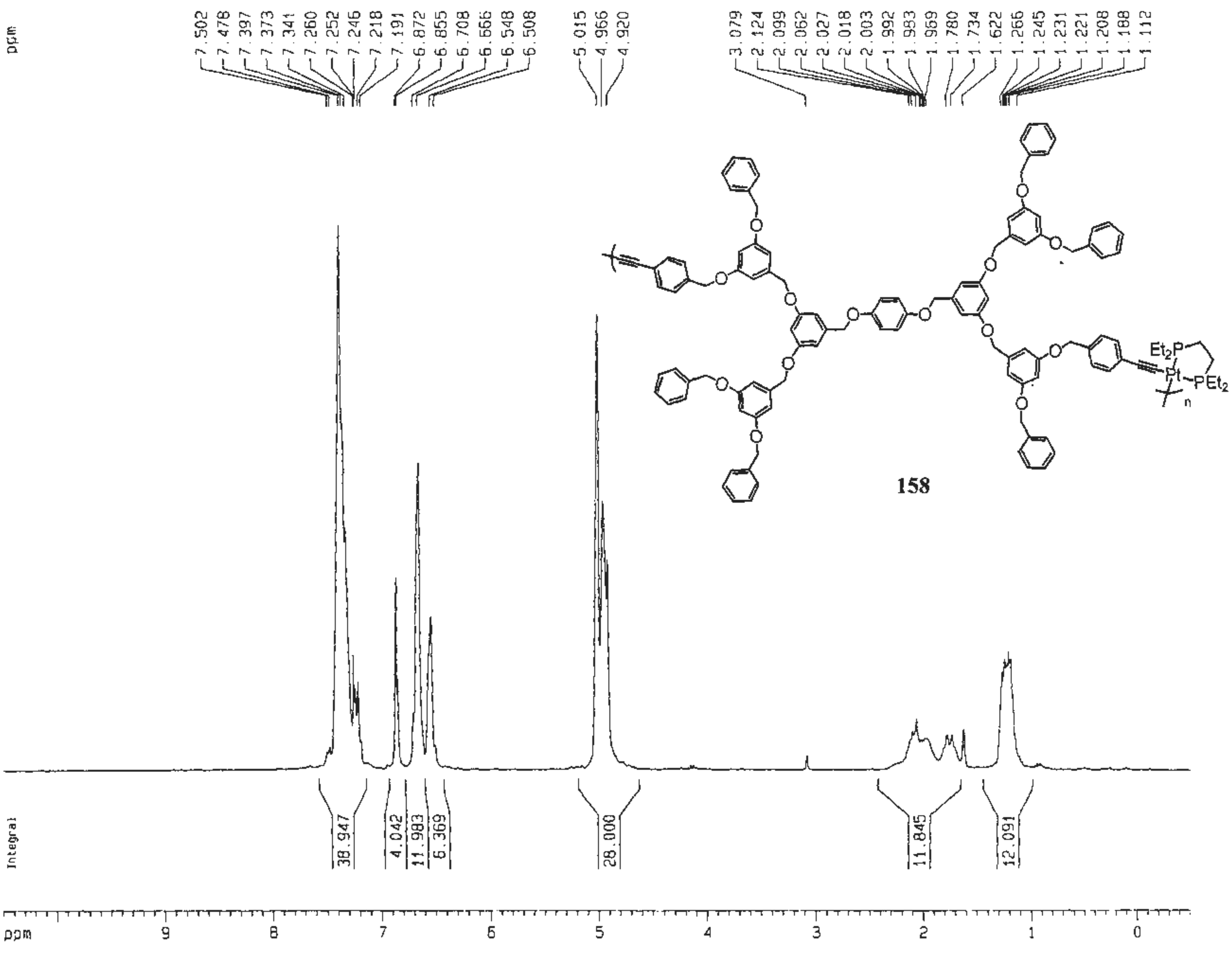
F2 - Acquisition Parameters
Date_           20081108
Time                7.55
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   601
DS                     0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                7298.2
DW                13.275 usec
DE                  6.00 usec
TE                 298.2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  3.00 usec
PL1                -6.00 dB
SFO1         121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF           121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 13.05 cm
F1P               75.000 ppm
F1               9112.11 Hz
F2P               35.000 ppm
F2               4252.32 Hz
PPMCM            1.81818 ppm/cm
HZCM           220.89972 Hz/cm
```



158

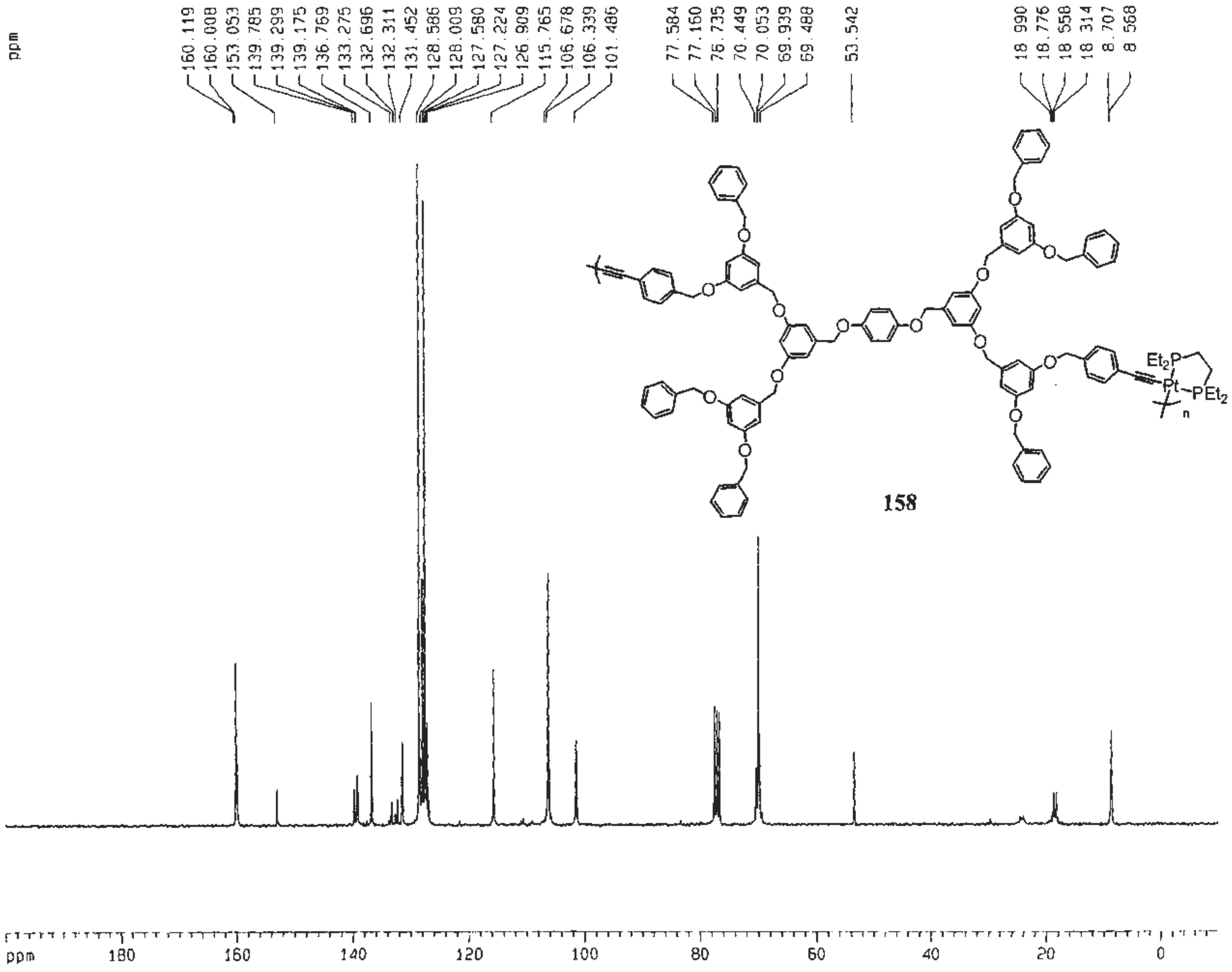
```
Current Data Parameters
NAME      Poly(cis-Pt-G2)N(ppt)
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20081122
Time                   1.06
INSTRUM              dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                  zg
TD                    16384
SOLVENT               CDCl3
NS                       32
DS                        0
SWH                3601.657 Hz
FIDRES             0.219828 Hz
AO                2.2745588 sec
RG                      128
DW                  138.825 usec
DE                   198 32 usec
TE                    296 2 K
D1               5 00000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                     4.50 usec
PL1                   -2.00 dB
SFO1            300.1315000 MHz

F2 - Processing parameters
SI                    16384
SF              300.1300063 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    22 00 cm
CY                    10 03 cm
F1P                  10 500 ppm
F1                  3151.36 Hz
F2P                  -0.500 ppm
F2                  -150.07 Hz
PPMCM               0.50000 ppm/cm
HZCM              150 06500 Hz/cm
```



158

```
Current Data Parameters
NAME       Poly(cis-Pt-G2)NC
EXPNO                    1
PROCNO                   1

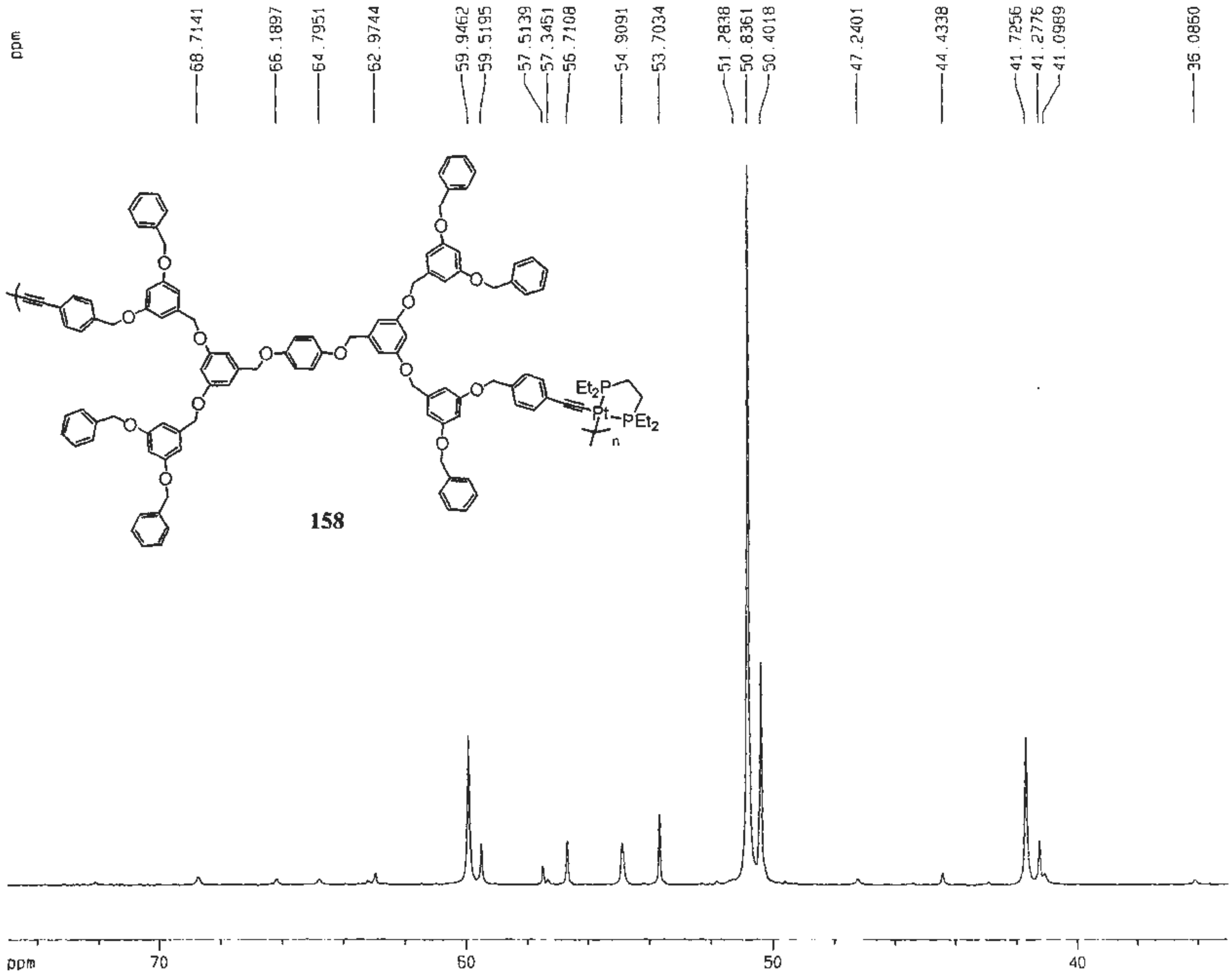
F2 - Acquisition Parameters
Date_             20081122
Time                  5.04
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     972
DS                       0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                    8192
DW                  22.050 usec
DE                    6.00 usec
TE                   296.2 K
D1              1.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    3.00 usec
PL1                  -6.00 dB
SFO1            75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2           300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF              75.4677550 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   11.99 cm
F1P                200.000 ppm
F1                15093.55 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM              9.54545 ppm/cm
HZCM             720.37402 Hz/cm
```



158

```
Current Data Parameters
NAME      Poly(cis-Pt-G2)NP
EXPNO                     1
PROCNO                    1

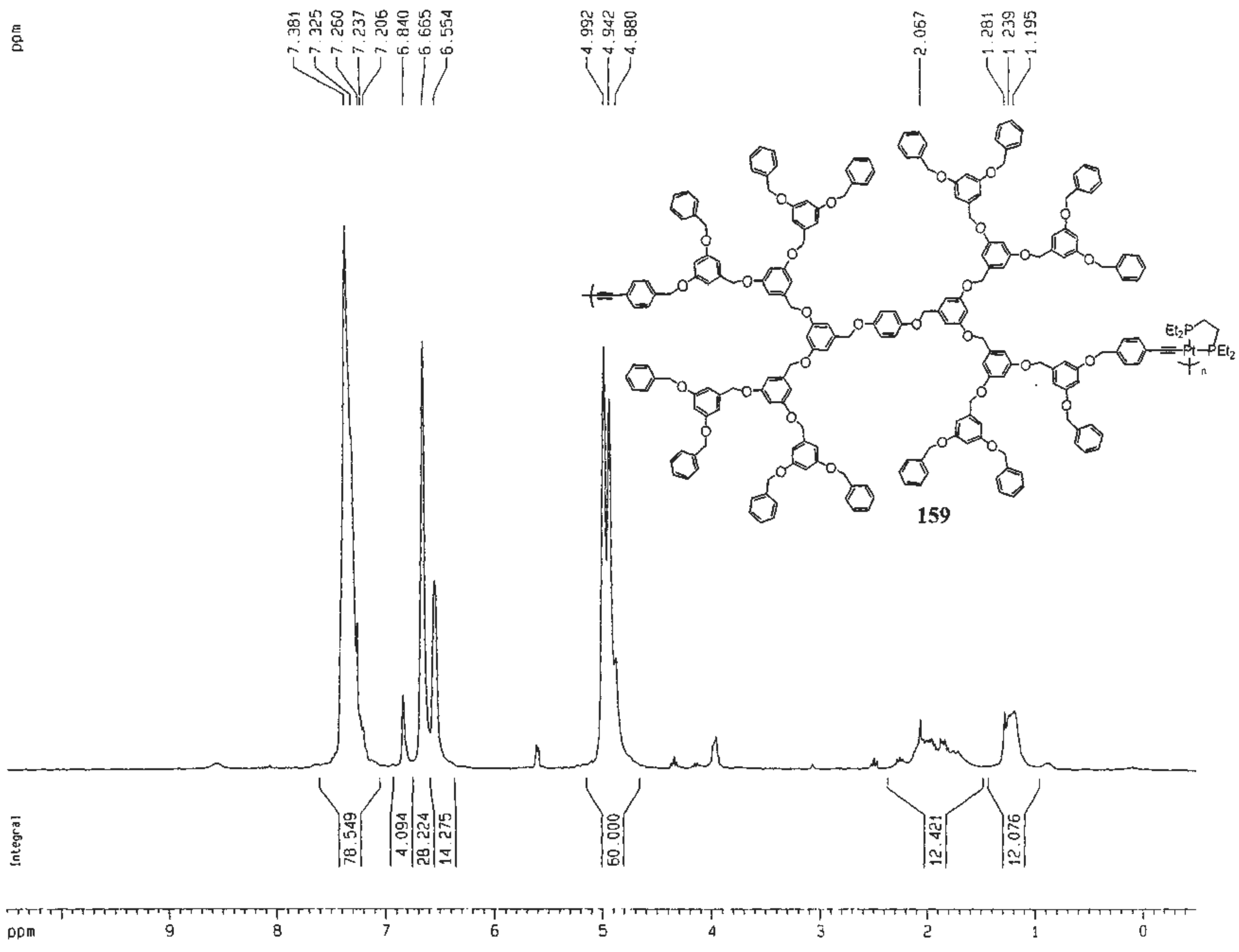
F2 - Acquisition Parameters
Date_             20081122
Time                  4.43
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     400
DS                       0
SWH              37664.785 Hz
FIDRES            0.574719 Hz
AQ               0.8700404 sec
RG                  7298.2
DW                  13.275 usec
DE                    6.00 usec
TE                   296.2 K
D1              0.30000001 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   31P
P1                    3.00 usec
PL1                  -6.00 dB
SFO1           121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2           300.1316097 MHz

F2 - Processing parameters
SI                   65536
SF             121.4948412 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   12.98 cm
F1P                 75.000 ppm
F1                 9112.11 Hz
F2P                 35.000 ppm
F2                 4252.32 Hz
PPMCM              1.81818 ppm/cm
HZCM              220.89972 Hz/cm
```



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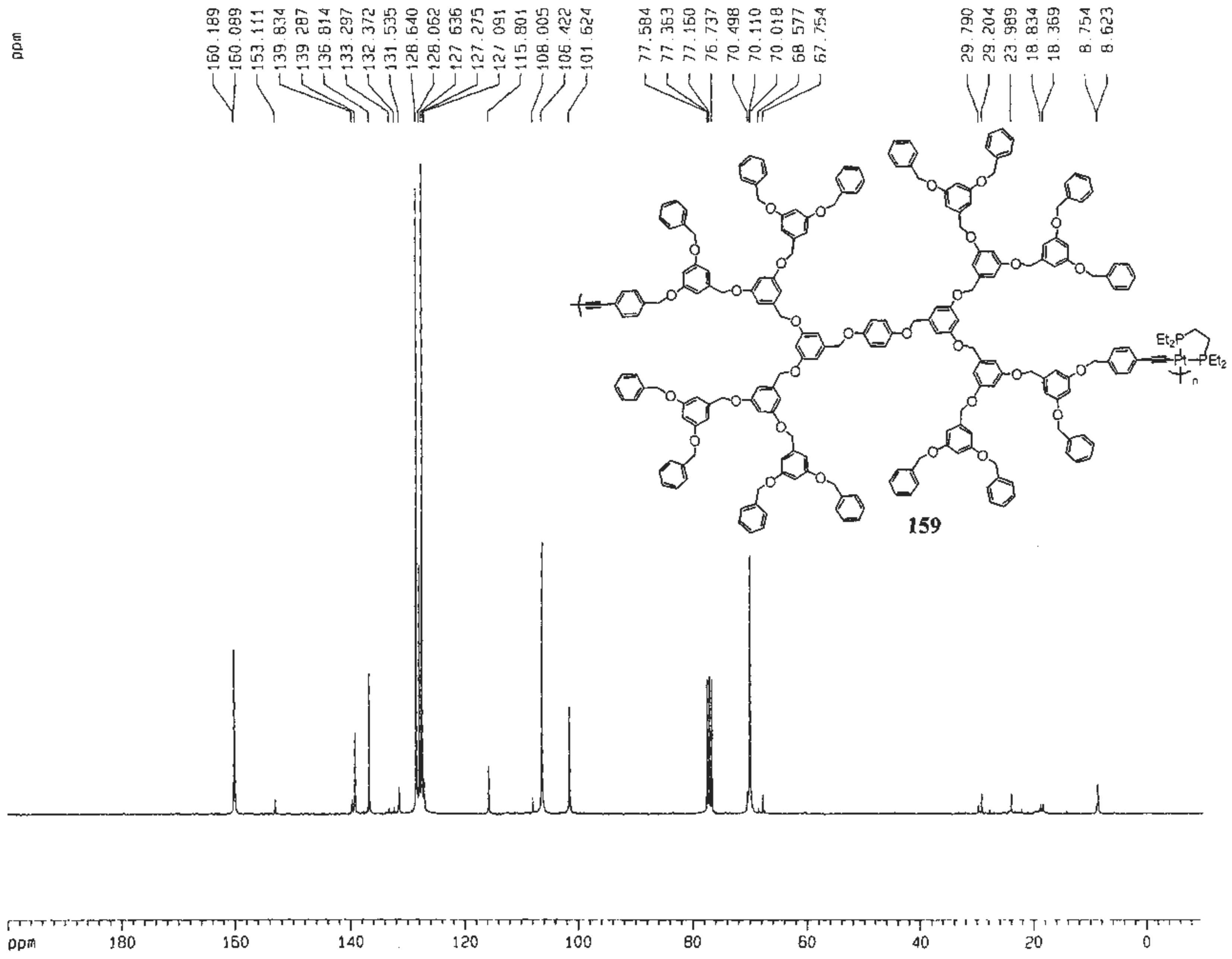
```
Current Data Parameters
NAME     Poly(cis-Pt-G3)N(ppt)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20081206
Time                  2.00
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                      32
DS                       0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                     114
DW                 138.825 usec
DE                  198.32 usec
TE                   296.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    4.50 usec
PL1                  -2.00 dB
SFO1           300.1315000 MHz

F2 - Processing parameters
SI                   16384
SF             300.1300067 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   10.04 cm
F1P                 10.500 ppm
F1                 3151.36 Hz
F2P                 -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.50000 ppm/cm
HZCM             150.06500 Hz/cm
```



159

```
Current Data Parameters
NAME      Poly(cis-Pt-G3)NC
EXPNO                     1
PROCNO                    1

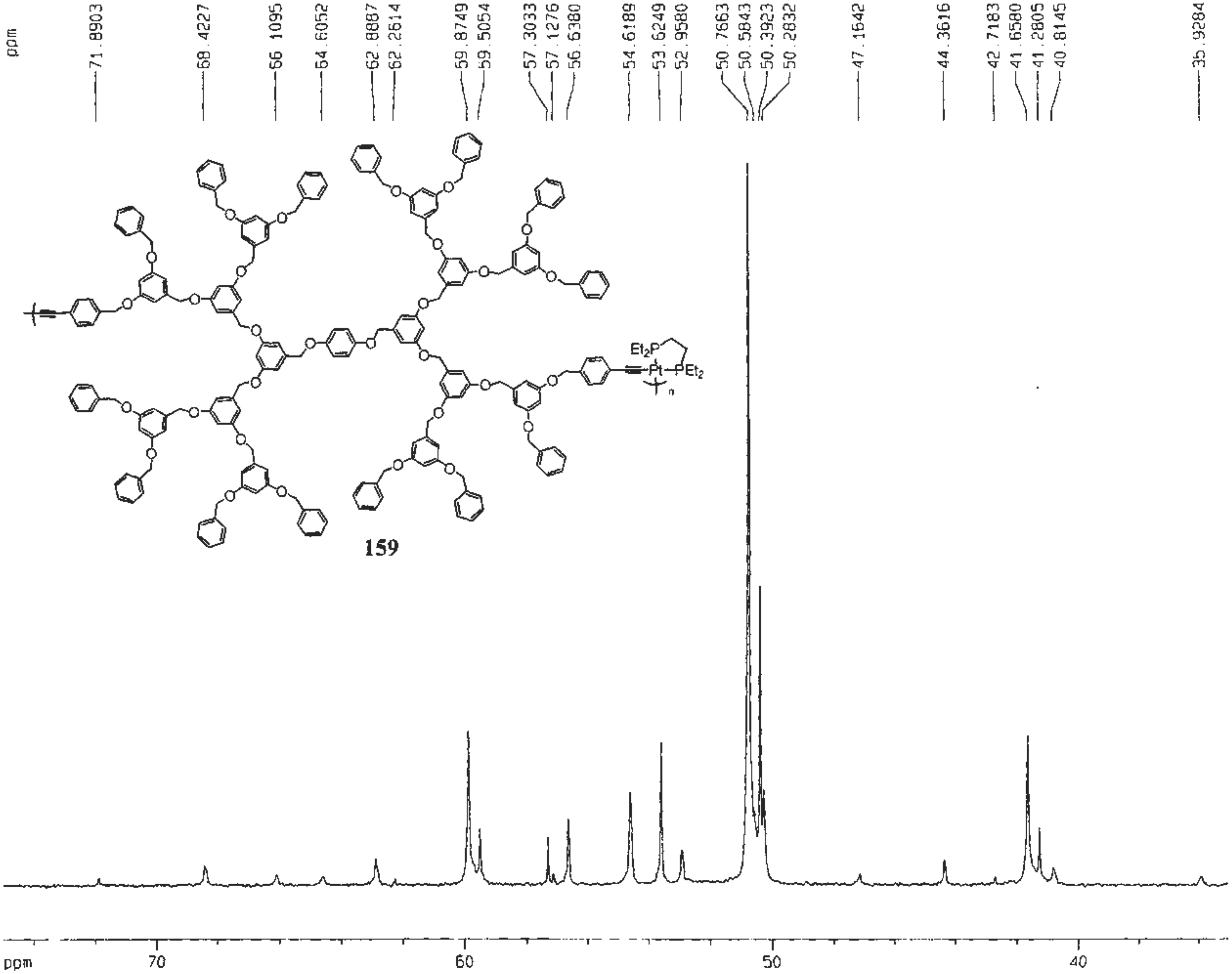
F2 - Acquisition Parameters
Date_          20081206
Time               6.23
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                 7525
DS                    0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                 8192
DW               22.050 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677483 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                12.02 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37390 Hz/cm
```



159

```
Current Data Parameters
NAME      Poly(cis-Pt-G3)NP
EXPNO                    1
PROCNO                   1

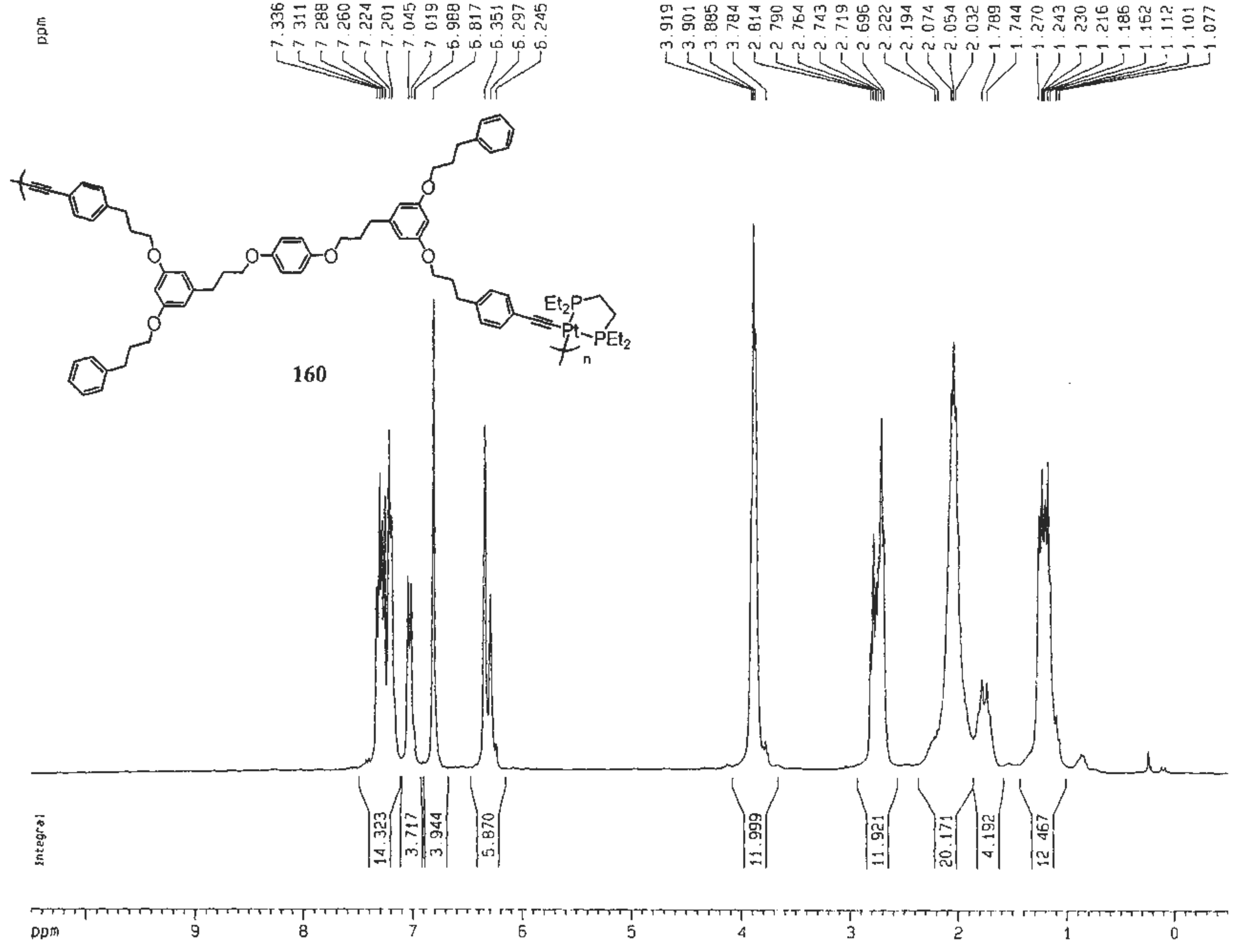
F2 - Acquisition Parameters
Date_          20081206
Time               2.37
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    0
SWH           37664.785 Hz
FIDRES         0.574719 Hz
AQ            0.8700404 sec
RG               7298.2
DW               13.275 usec
DE                 6.00 usec
TE                296.2 K
D1           0.30000001 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                31P
P1                 3.00 usec
PL1               -6.00 dB
SFO1        121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1316097 MHz

F2 - Processing parameters
SI                65536
SF          121.4948412 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                12.98 cm
F1P              75.000 ppm
F1              9112.11 Hz
F2P              35.000 ppm
F2              4252.32 Hz
PPMCM           1.81818 ppm/cm
HZCM          220.89972 Hz/cm
```



160

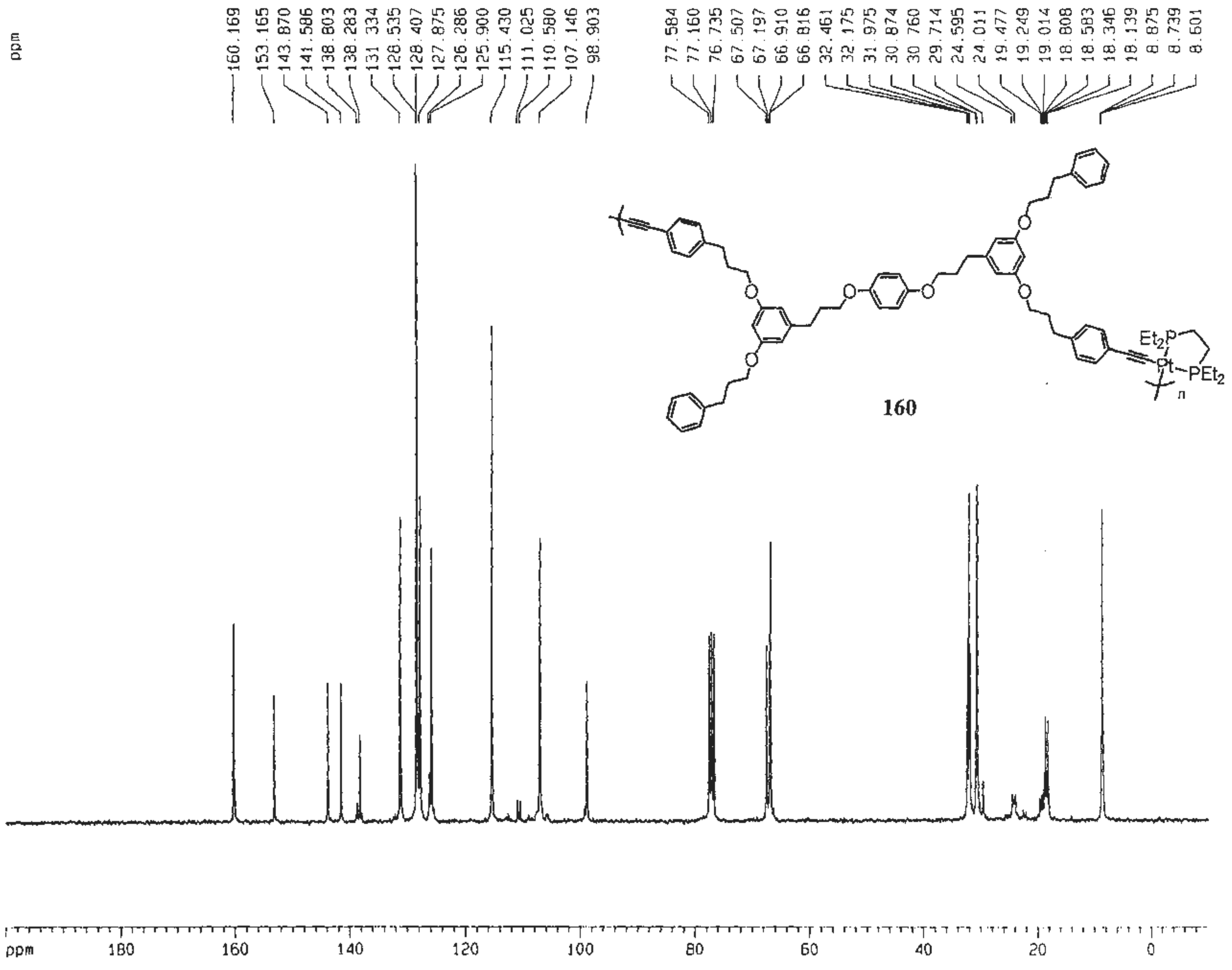
```
Current Data Parameters
NAME      Poly(cis-Pt-G1`) (ppt)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090114
Time                19.23
INSTRUM            dpx300
PROBHD   5 mm BB0 BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                     57
DW                138.825 usec
DE                 198.32 usec
TE                  297.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300074 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   9.99 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



160

```
Current Data Parameters
NAME      Poly[cis-Pt-G1']C
EXPNO                    1
PROCNO                   1

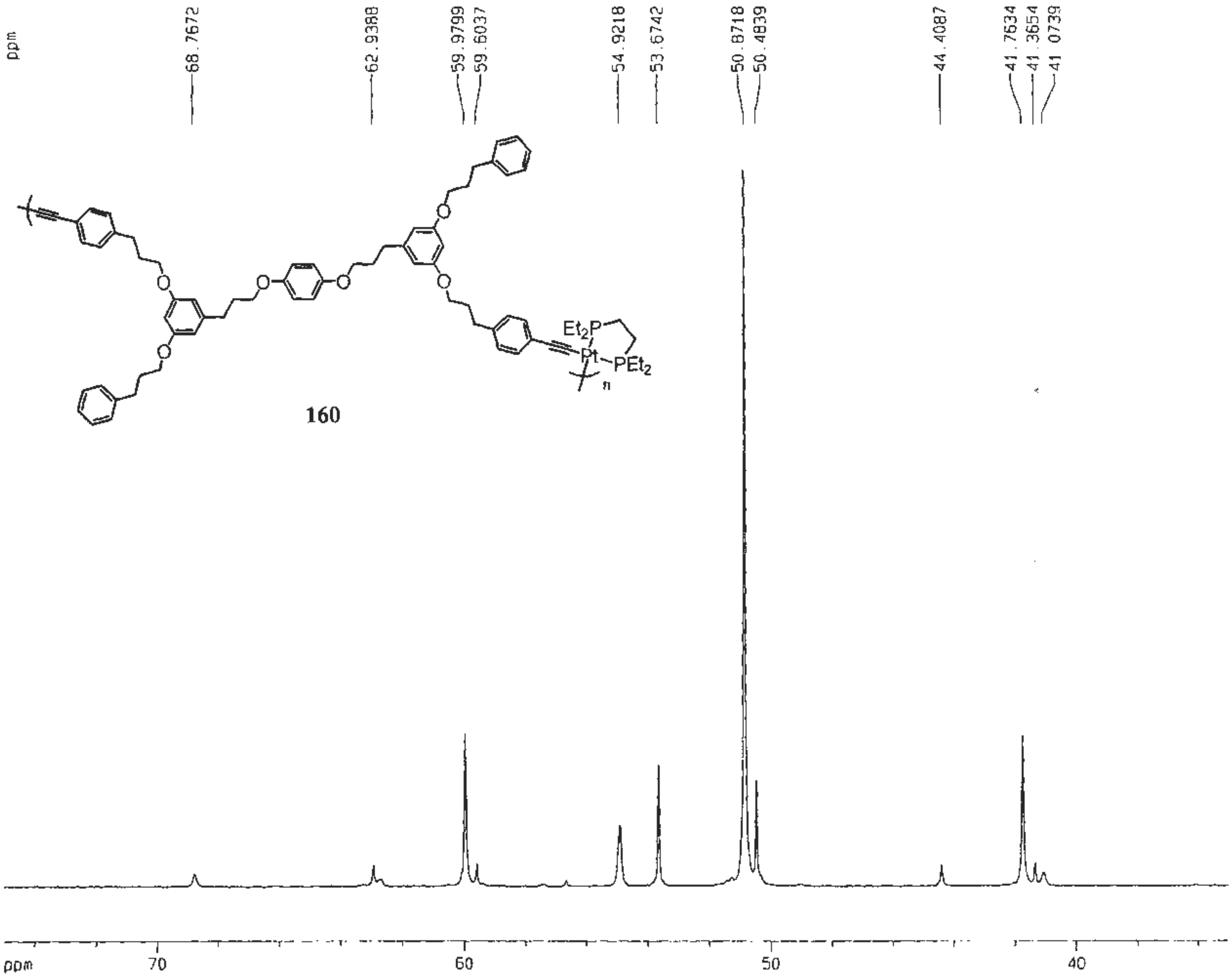
F2 - Acquisition Parameters
Date_             20090115
Time                 21.47
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                    2500
DS                       0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                    8192
DW              22.050 usec
DE                6.00 usec
TE                 297.2 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                3.00 usec
PL1               -6.00 dB
SFO1         75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2           100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                   65536
SF           75.4677516 MHz
WDW                     EM
SSB                      0
LB                 3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.99 cm
F1P            200.000 ppm
F1            15093.55 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM        9.54545 ppm/cm
HZCM        720.37390 Hz/cm
```



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```
Current Data Parameters
NAME      Poly[cis-Pt-G1']P
EXPNO                  1
PROCNO                 1

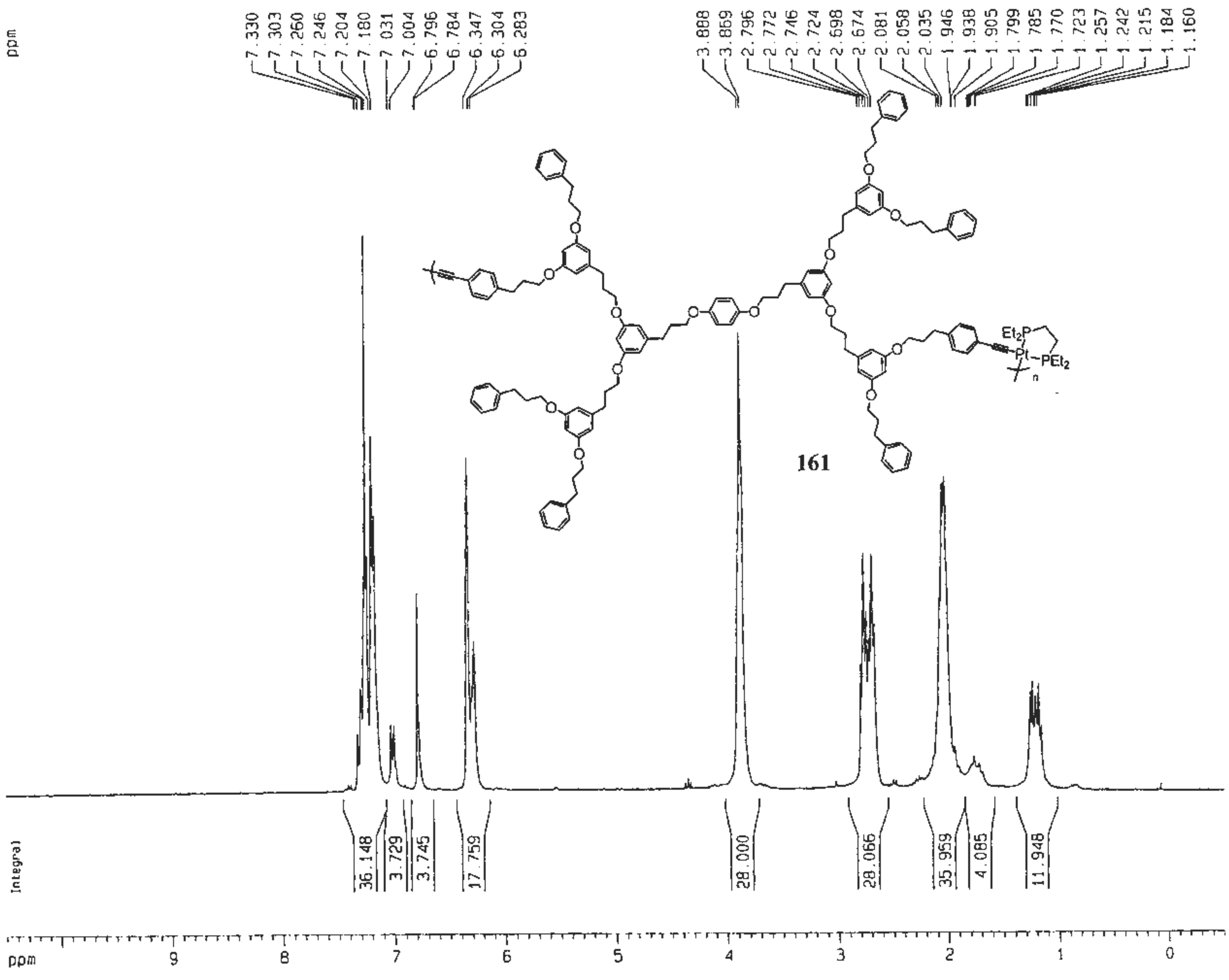
F2 - Acquisition Parameters
Date_           20090114
Time               21.49
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     0
SWH          37664.785 Hz
FIDRES        0.574719 Hz
AG           0.8700404 sec
RG                7298.2
DW              13.275 usec
DE                6.00 usec
TE               297.2 K
D1          0.30000001 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                3.00 usec
PL1              -6.00 dB
SFO1      121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2      300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF        121.4948412 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 12.97 cm
F1P               75.000 ppm
F1               9112.11 Hz
F2P               35.000 ppm
F2               4252.32 Hz
PPMCM            1.81818 ppm/cm
HZCM           220.89972 Hz/cm
```



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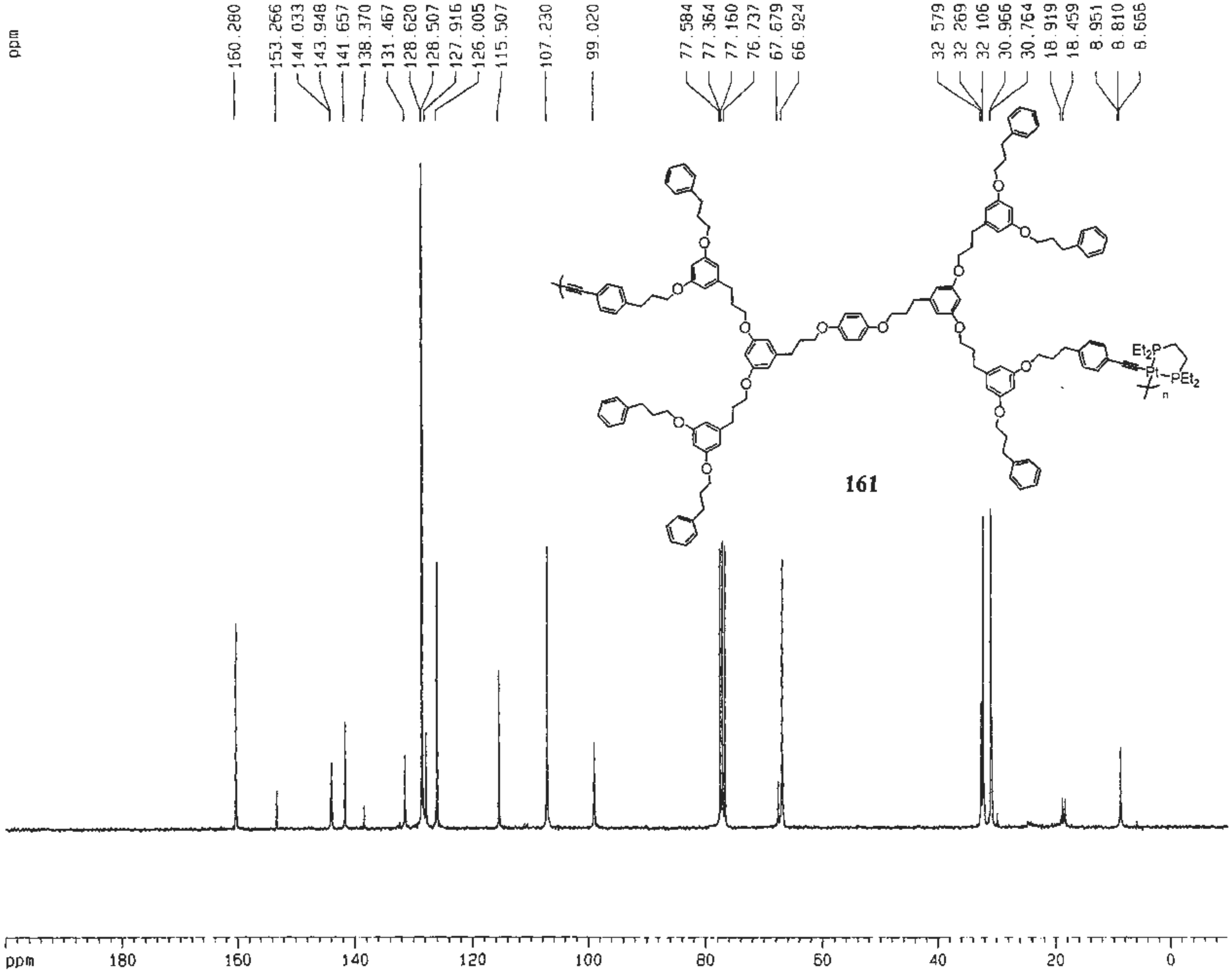
```
Current Data Parameters
NAME     Poly(cis-Pt-G2')(ppt)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090114
Time              19.03
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3601.657 Hz
FIDRES         0.219828 Hz
AQ            2.2745588 sec
RG                  362
DW              138.825 usec
DE               198.32 usec
TE                297.2 K
D1           5.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                32768
SF          300.1300063 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P              10.500 ppm
F1              3151.36 Hz
F2P              -0.500 ppm
F2              -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM          150.06500 Hz/cm
```



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```
Current Data Parameters
NAME     Poly(cis-Pt-G2')C
EXPNO                   1
PROCNO                  1

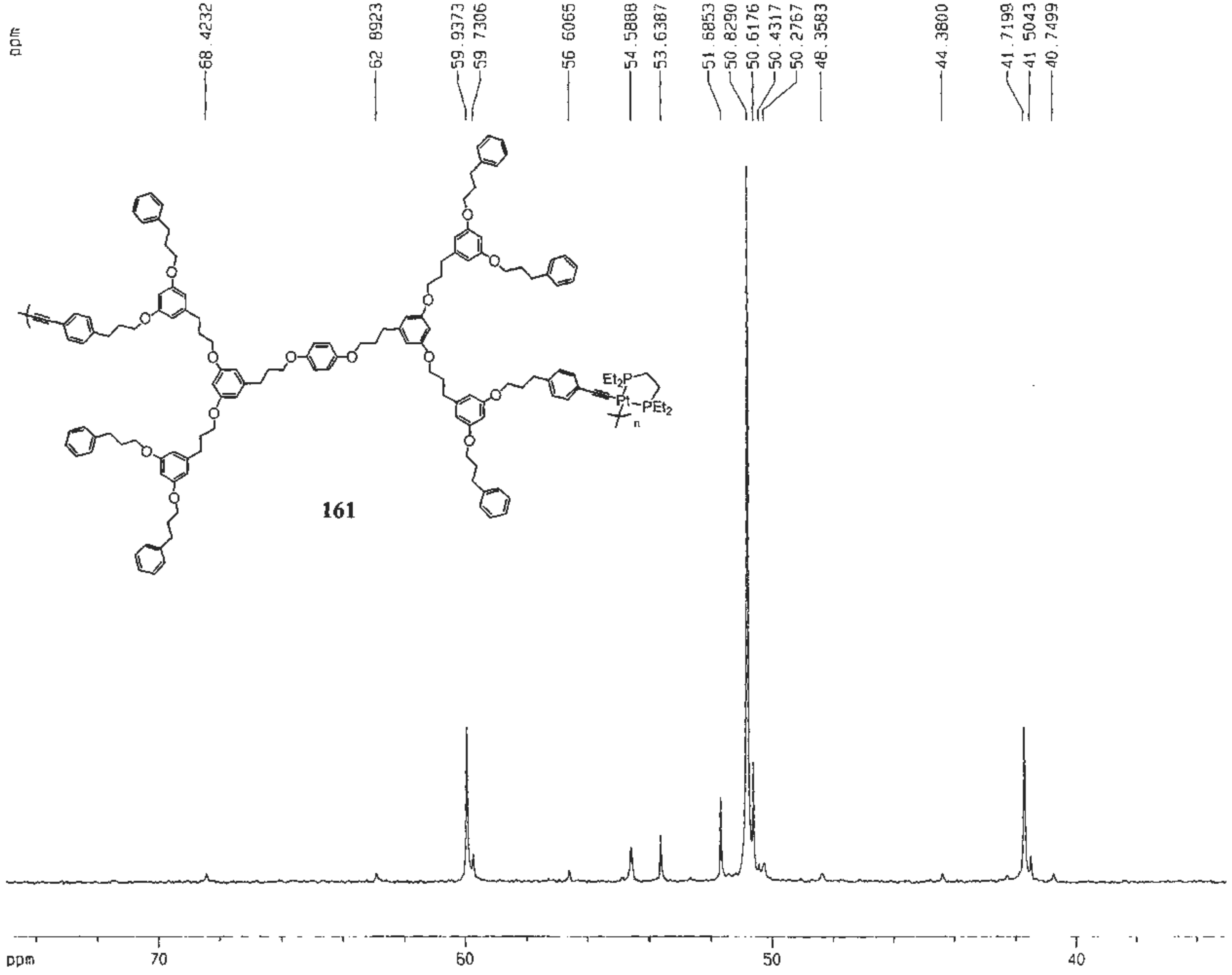
F2 - Acquisition Parameters
Date_            20090115
Time                23.38
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   4624
DS                      0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                  297.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677438 MHz
WDW                    EM
SSB                     0
LB                   2.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.06 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM             720.37384 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(cis-Pt-G2')P
EXPNO                     1
PROCNO                    1

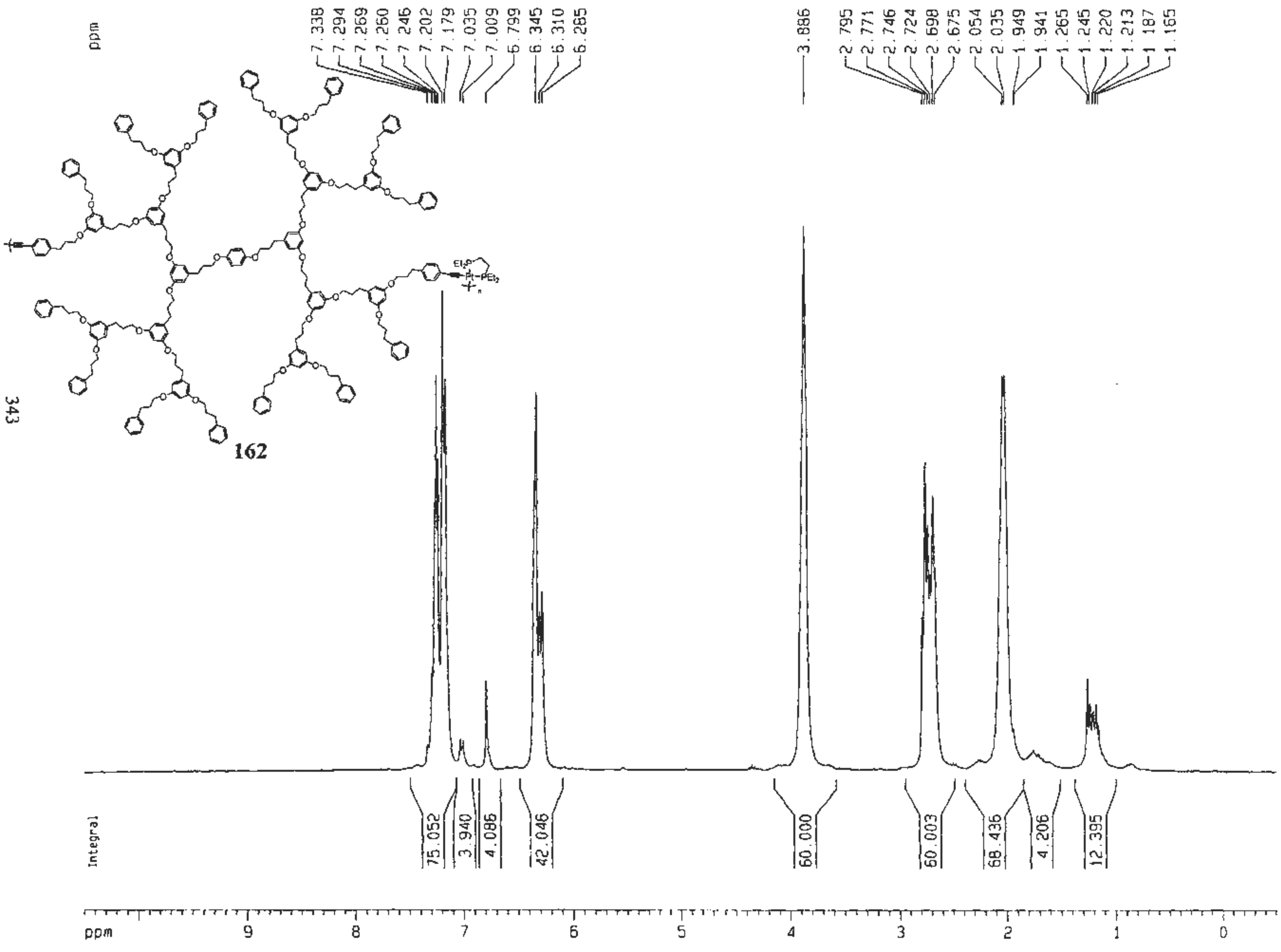
F2 - Acquisition Parameters
Date_              20090114
Time                  22.00
INSTRUM              dpx300
PROBHD    5 mm BBO BB-1H
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                      300
DS                        0
SWH              37664.785 Hz
FIDRES            0.574719 Hz
AO               0.8700404 sec
RG                   7298.2
DW                  13.275 usec
DE                    6.00 usec
TE                   297.2 K
D1              0.30000001 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    31P
P1                     3.00 usec
PL1                   -6.00 dB
SFO1          121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                  120.00 dB
PL12                  19.00 dB
SFO2          300.1316097 MHz

F2 - Processing parameters
SI                    65536
SF            121.4948412 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    22.00 cm
CY                    13.01 cm
F1P                  75.000 ppm
F1                  9112.11 Hz
F2P                  35.000 ppm
F2                  4252.32 Hz
PPMCM               1.81818 ppm/cm
HZCM              220.89972 Hz/cm
```



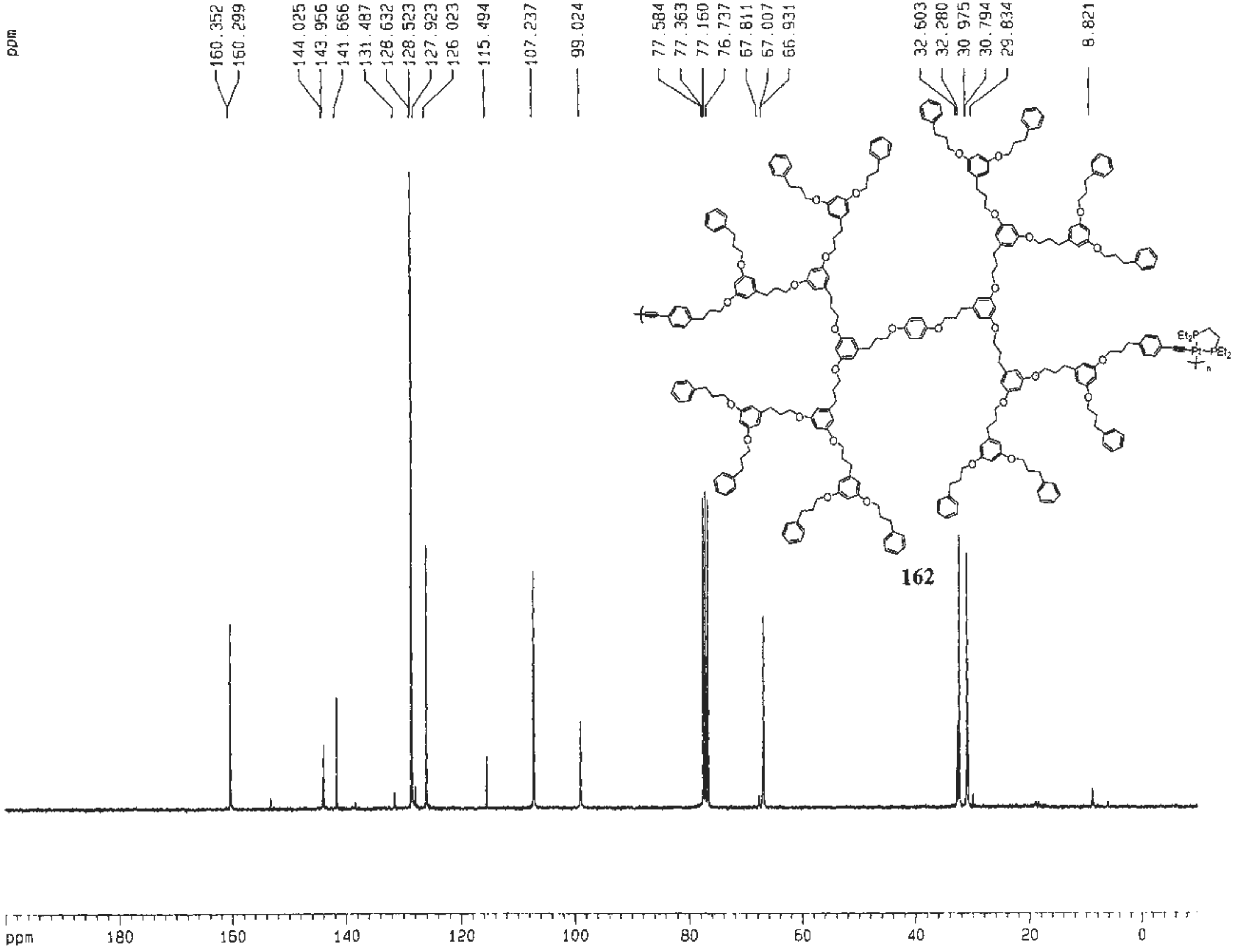
```
Current Data Parameters
NAME     Poly(cis-Pt-G3')(ppt)
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_             20090114
Time                 19.07
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                      32
DS                       0
SWH            3601.657 Hz
FIDRES         0.219826 Hz
AQ            2.2745588 sec
RG                     181
DW            138.825 usec
DE             198.32 usec
TE                297.2 K
D1         5.00000000 sec
MCREST     0.00000000 sec
MCWORK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1               9.00 usec
PL1               -2.00 dB
SFO1        300.1315000 MHz

F2 - Processing parameters
SI                   32768
SF          300.1300083 MHz
WDW                     EM
SSB                      0
LB                 0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 cm
F1P             10.500 ppm
F1             3151.36 Hz
F2P             -0.500 ppm
F2             -150.07 Hz
PPMCM       0.50000 ppm/cm
HZCM      150.06500 Hz/cm
```



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```
Current Data Parameters
NAME     Poly[cis-Pt-G3']C
EXPNO                   1
PROCNO                  1

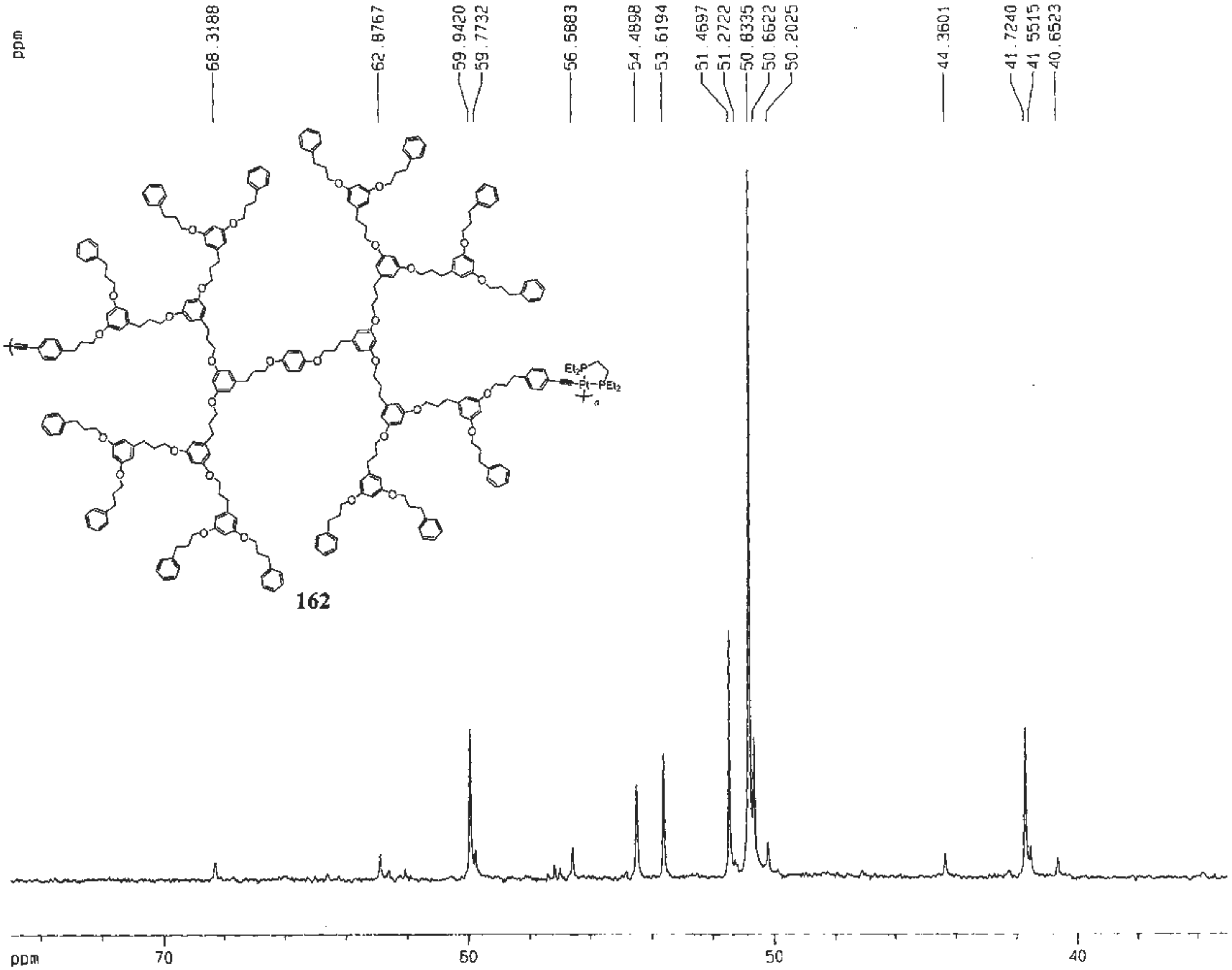
F2 - Acquisition Parameters
Date_            20090116
Time                 2.55
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   4864
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                  297.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677420 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  11.91 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37384 Hz/cm
```



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```
Current Data Parameters
NAME     Poly(cis-Pt-G3')P
EXPNO                   1
PROCNO                  1

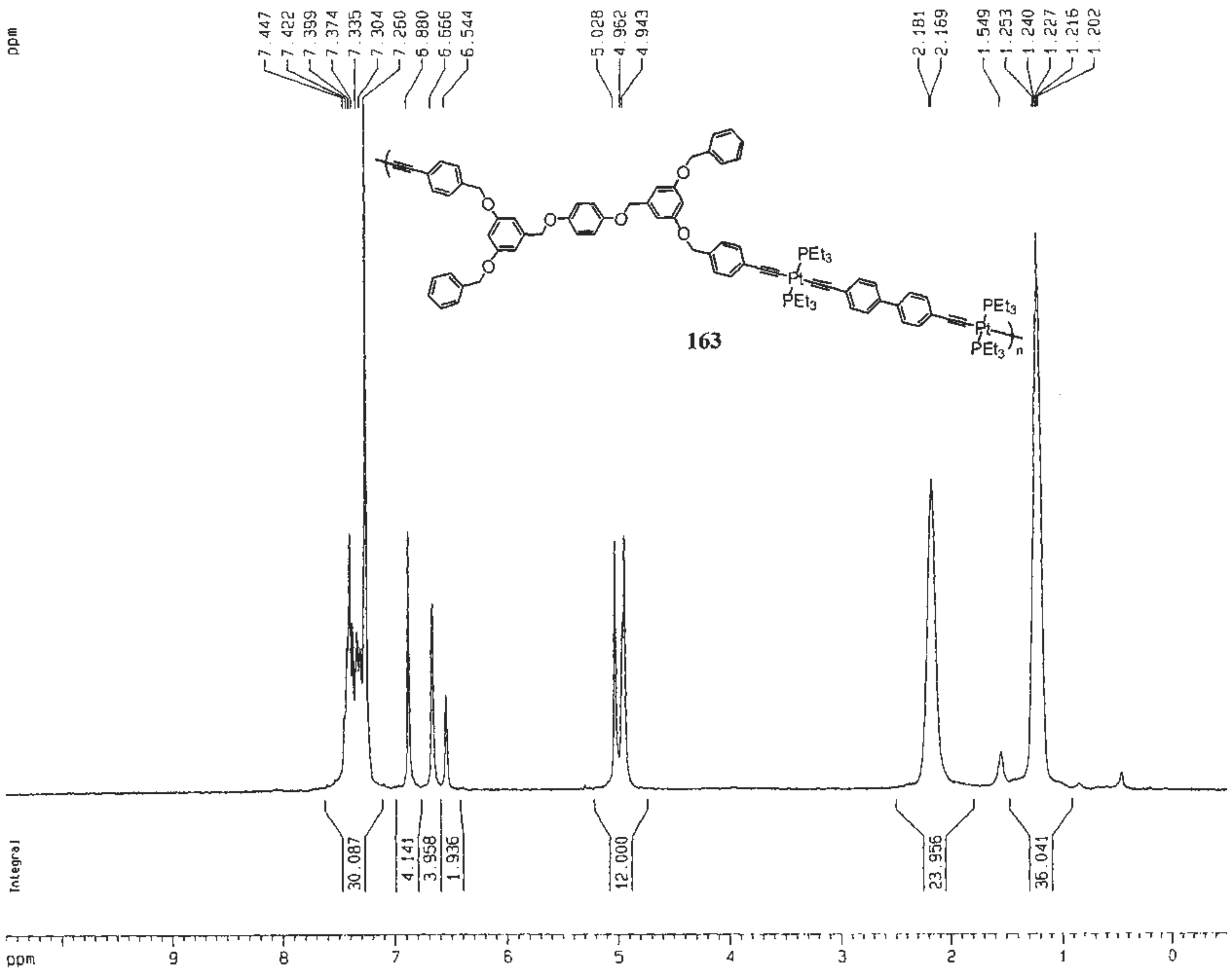
F2 - Acquisition Parameters
Date_            20090114
Time                22 35
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   1200
DS                      0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                 7298.2
DW                 13.275 usec
DE                   6.00 usec
TE                  297 2 K
D1            0.30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  31P
P1                   3.00 usec
PL1                 -6.00 dB
SFO1          121 4932237 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1316097 MHz

F2 - Processing parameters
SI                  65536
SF            121.4948412 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.98 cm
F1P                75 000 ppm
F1                9112.11 Hz
F2P                35.000 ppm
F2                4252.32 Hz
PPMCM             1.81818 ppm/cm
HZCM            220.89972 Hz/cm
```



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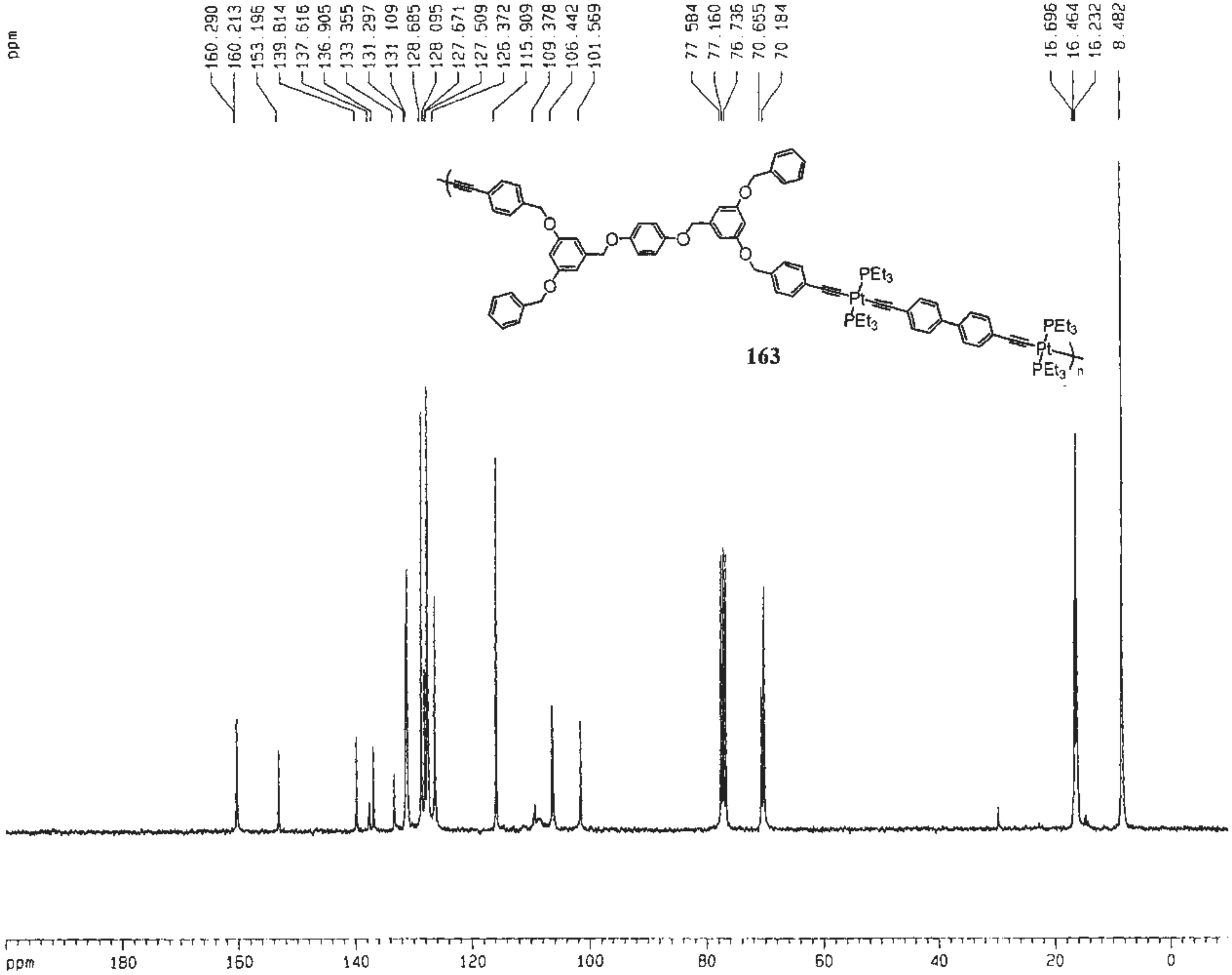
```
Current Data Parameters
NAME      Poly(L-Pt-G1)N(ppt)
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20081108
Time                  1 08
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                     128
DS                       0
SWH               3601.657 Hz
FIDRES            0.219828 Hz
AQ               2.2745588 sec
RG                   912.3
DW                 138.825 usec
DE                  198.32 usec
TE                   298.2 K
D1              5.00000000 sec
MCREST          0 00000000 sec
MCWRK           0 01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    4.50 usec
PL1                  -2.00 dB
SFO1           300.1315000 MHz

F2 - Processing parameters
SI                   16384
SF             300.1300060 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   12.91 cm
F1P                 10.500 ppm
F1                 3151 36 Hz
F2P                 -0.500 ppm
F2                 -150.07 Hz
PPMCM              0 50000 ppm/cm
HZCM             150 06500 Hz/cm
```



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```
Current Data Parameters
NAME      Poly(L-Pt-G1)NC
EXPNO                   1
PROCNO                  1

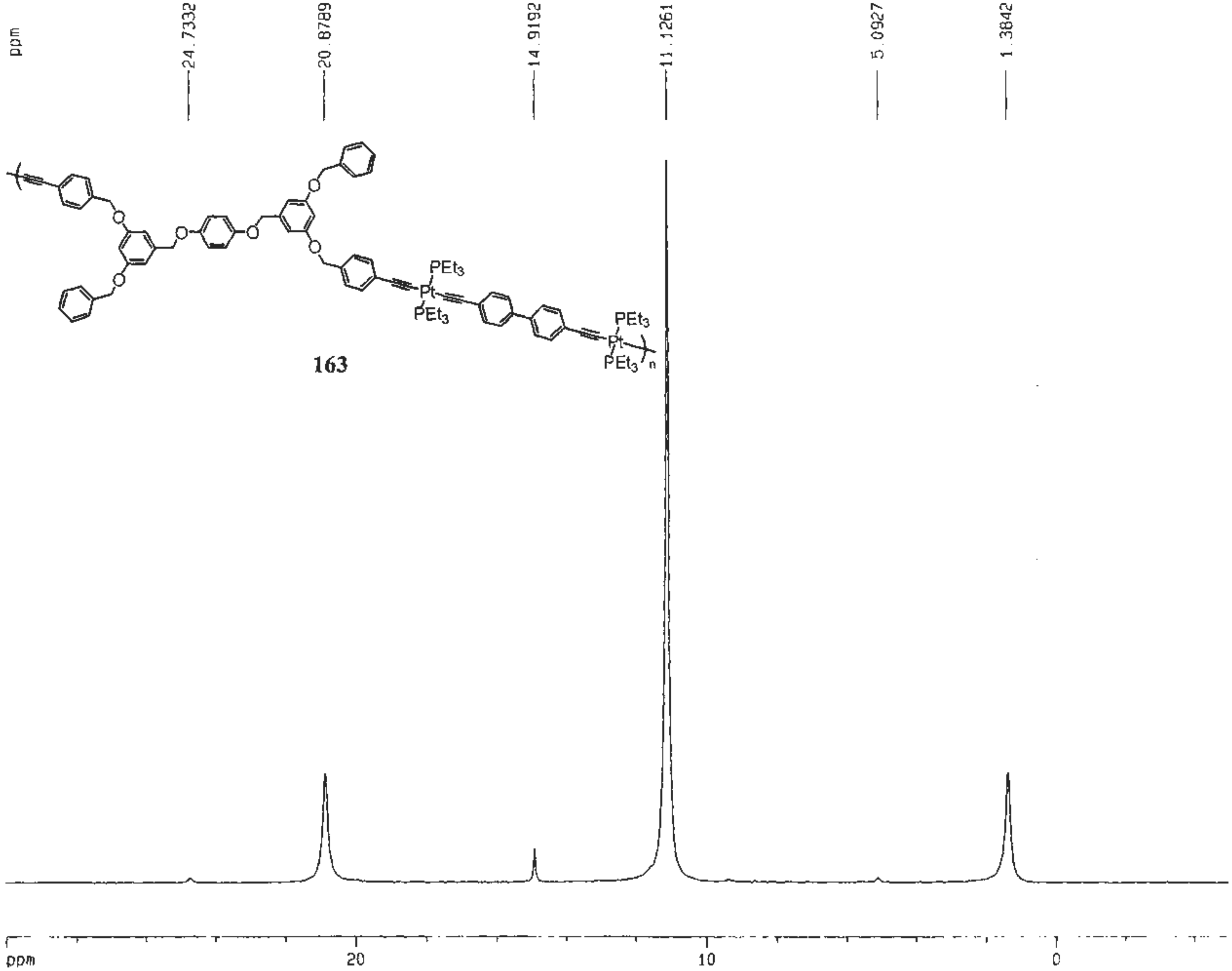
F2 - Acquisition Parameters
Date_            20081106
Time                 2.45
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   2664
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   6502
DW                 22.050 usec
DE                   6.00 usec
TE                  298.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677444 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  11.99 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37364 Hz/cm
```



163

```
Current Data Parameters
NAME       Poly(L-Pt-G1)NP
EXPNO                    1
PROCNO                   1

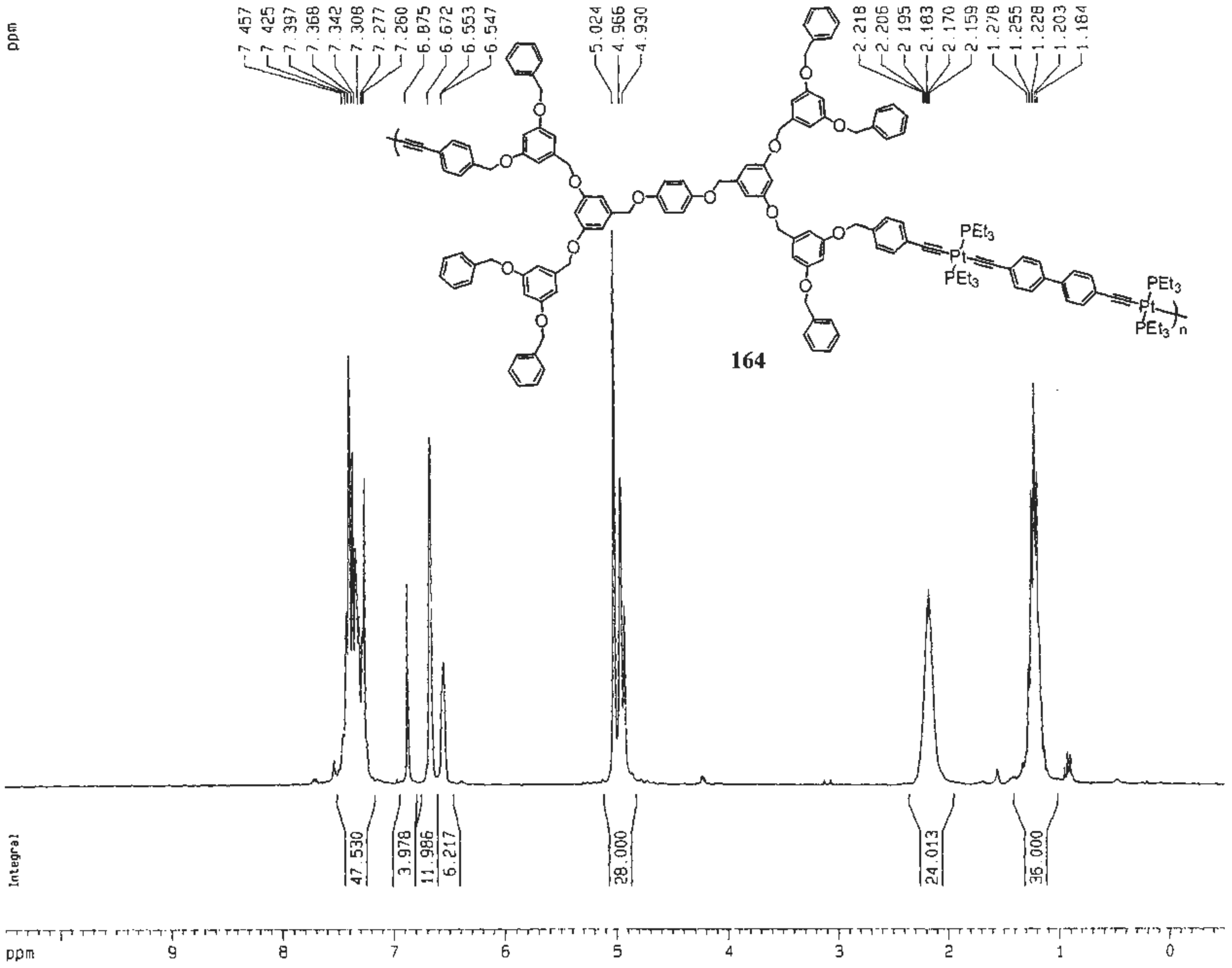
F2 - Acquisition Parameters
Date_             20081108
Time                  2.30
INSTRUM             dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     317
DS                       0
SWH           37664.785 Hz
FIDRES         0.574719 Hz
AQ            0 8700404 sec
RG                  4597 6
DW              13.275 usec
DE                6.00 usec
TE                 298.2 K
D1           0.30000001 sec
d11          0.03000000 sec
MCREST       0 00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                   31P
P1                3.00 usec
PL1               -6.00 dB
SFO1        121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2           100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2        300.1316097 MHz

F2 - Processing parameters
SI                   65536
SF          121.4948412 MHz
WDW                     EM
SSB                      0
LB                 3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                22.00 cm
CY                13.03 cm
F1P             30.000 ppm
F1              3644.85 Hz
F2P             -5.000 ppm
F2              -607.47 Hz
PPMCM         1.59091 ppm/cm
HZCM        193.28725 Hz/cm
```


164

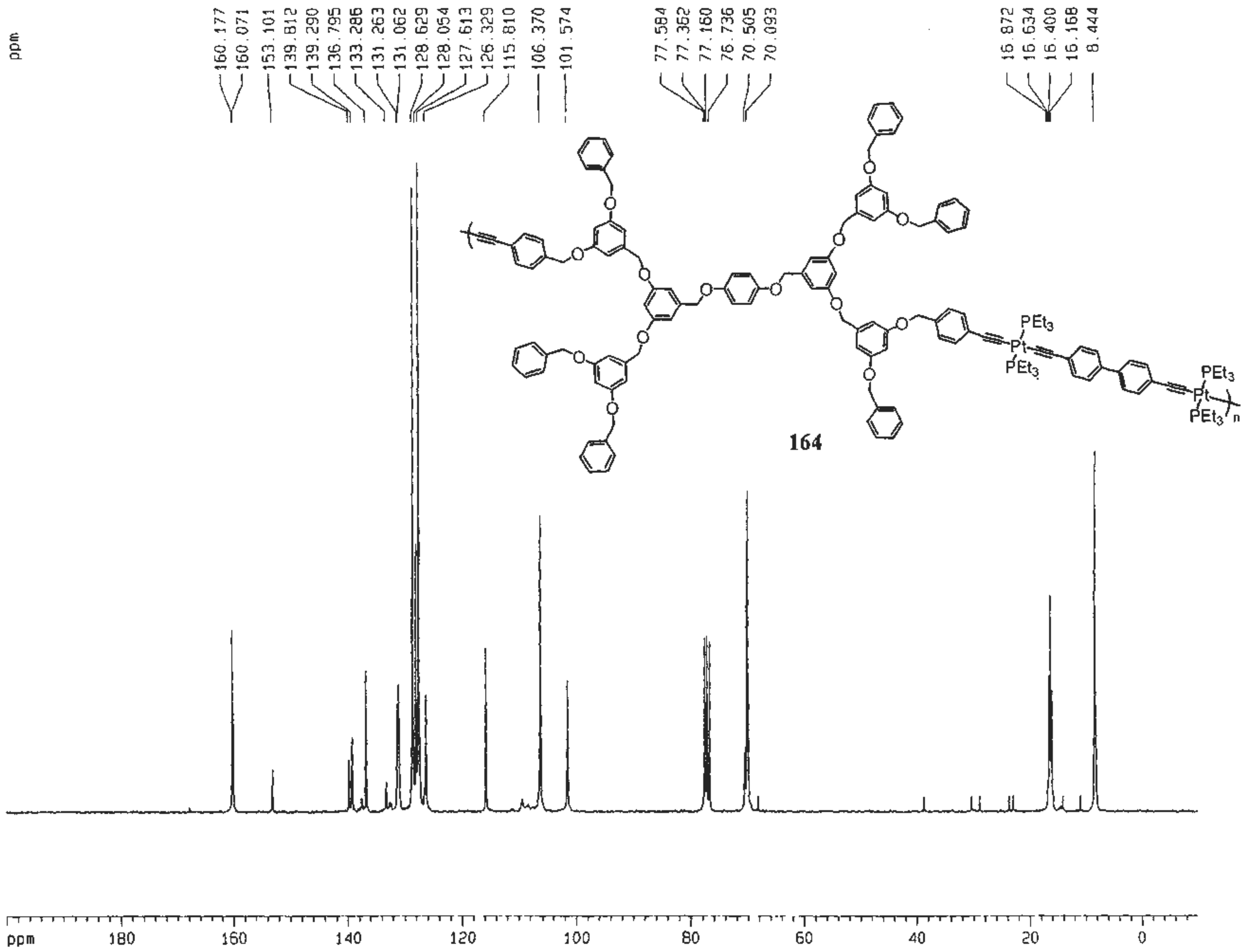
```
Current Data Parameters
NAME       Poly(L-Pt-G2)N(ppt)
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20081120
Time                     18 27
INSTRUM                 dpx300
PROBHD    5 mm BBO BB-1H
PULPROG                     zg
TD                       16384
SOLVENT                  CDCl3
NS                          32
DS                           0
SWH               3601.657 Hz
FIDRES            0.219828 Hz
AO               2.2745588 sec
RG                         362
DW                138 825 usec
DE                 198 32 usec
TE                   296.2 K
D1              5 00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                   4.50 usec
PL1                   -2.00 dB
SF01           300.1315000 MHz

F2 - Processing parameters
SI                       16384
SF             300 1300065 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                    22.00 cm
CY                     9.98 cm
F1P                 10 500 ppm
F1                 3151.36 Hz
F2P                 -0.500 ppm
F2                 -150.07 Hz
PPMCM           0.50000 ppm/cm
HZCM           150.06500 Hz/cm
```



164

```
Current Data Parameters
NAME       Poly(L-Pt-G2)NC
EXPNO                    1
PROCNO                   1

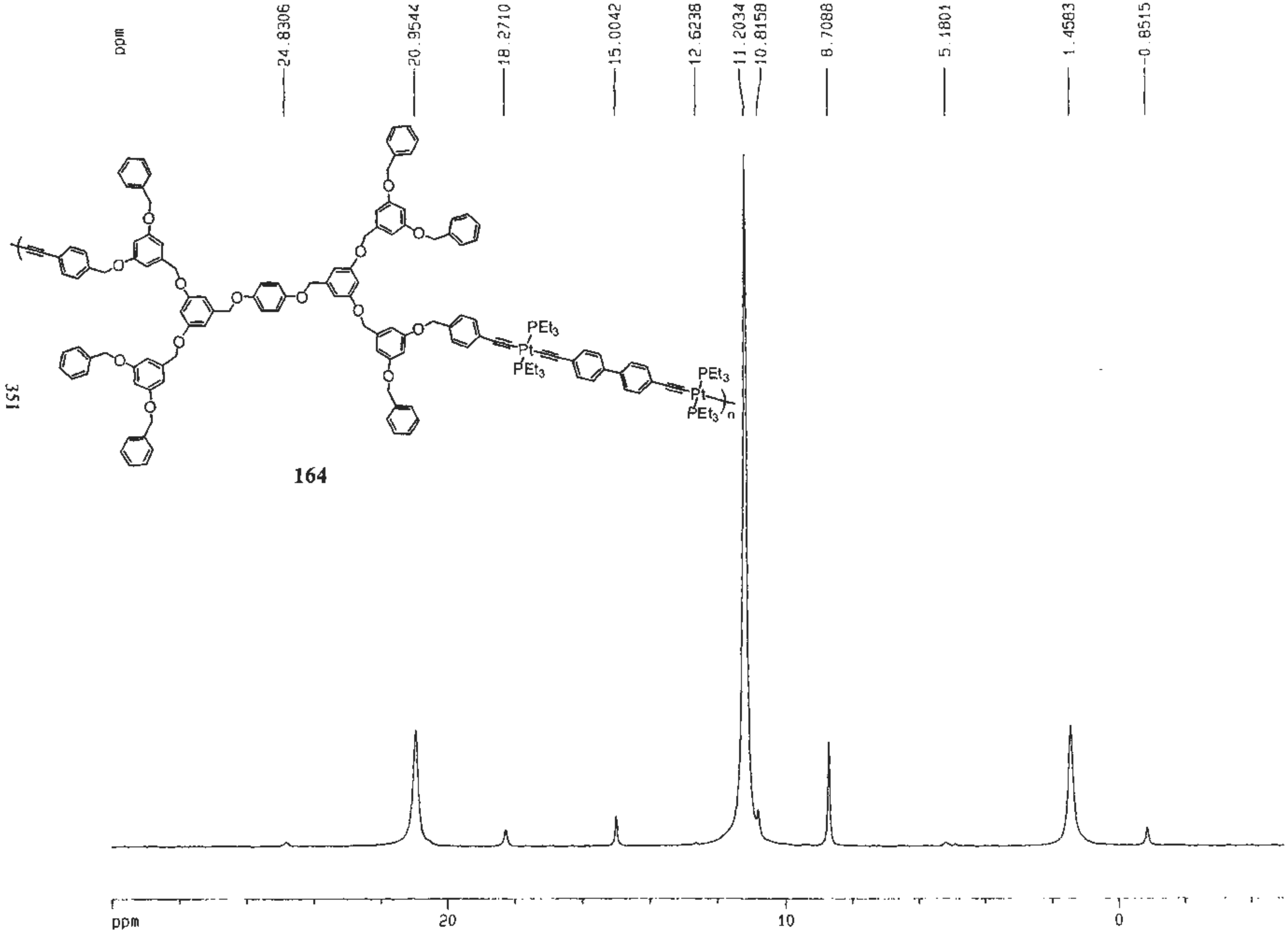
F2 - Acquisition Parameters
Date_           20081122
Time                1.46
INSTRUM           dpx300
PROBHD   5 mm BBC BB-1H
PULPROG             zgoc
TD                 65536
SOLVENT            CDCl3
NS                  4088
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 296.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677527 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.98 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37390 Hz/cm
```



164

```
Current Data Parameters
NAME     Poly(L-Pt-G2)NP
EXPNO                  1
PROCNO                 1

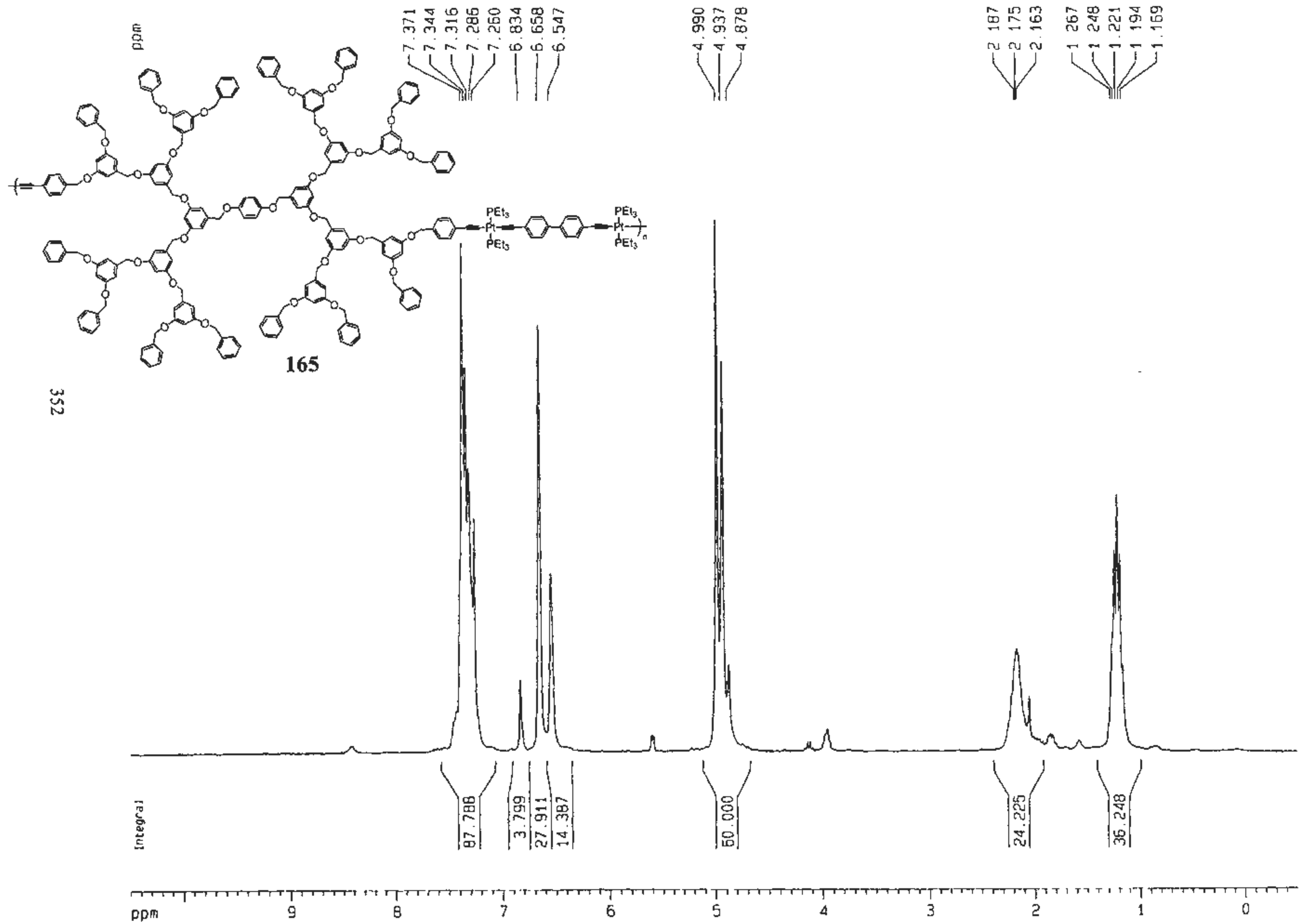
F2 - Acquisition Parameters
Date_           20081120
Time               18.59
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   320
DS                     0
SWH          37664.785 Hz
FIDRES        0.574719 Hz
AQ           0.8700404 sec
RG                  4096
DW             13.275 usec
DE               6.00 usec
TE               296.2 K
D1          0.30000001 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1               3.00 usec
PL1              -6.00 dB
SFO1       121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2          100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2       300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF         121.4948412 MHz
WDW                   EM
SSB                    0
LB                3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                22.00 cm
CY                13.02 cm
F1P             30.000 ppm
F1             3644.85 Hz
F2P             -5.000 ppm
F2             -607.47 Hz
PPMCM          1.59091 ppm/cm
HZCM         193.28725 Hz/cm
```



165

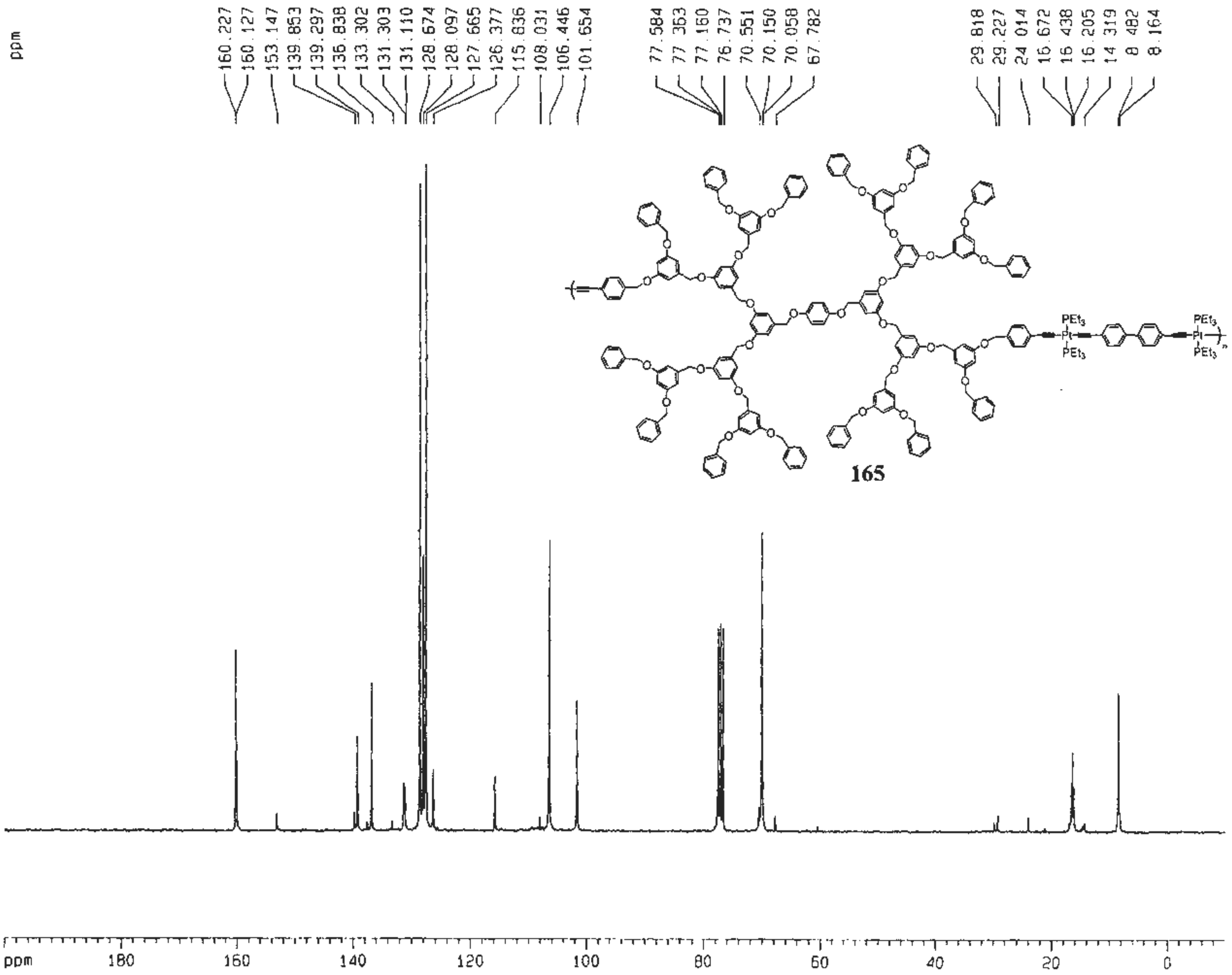
```
Current Data Parameters
NAME      Poly(L-Pt-G3)N(bpt)
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20081206
Time                   1.47
INSTRUM              dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                  zg
TD                    16384
SOLVENT               CDCl3
NS                       32
DS                        0
SWH            3601.657 Hz
FIDRES         0 219828 Hz
AQ             2 2745588 sec
RG                    287 4
DW              138 825 usec
DE               198 32 usec
TE                 295 2 K
D1           5 00000000 sec
MCREST       0 00000000 sec
MCWRK        0 01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                 4 50 usec
PL1                -2.00 dB
SFO1         300.1315000 MHz

F2 - Processing parameters
SI                    16384
SF           300.1300063 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 10.00 cm
F1P              10.500 ppm
F1               3151.36 Hz
F2P              -0.500 ppm
F2               -150.07 Hz
PPMCM        0.50000 ppm/cm
HZCM         150.06500 Hz/cm
```



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```
Current Data Parameters
NAME      Poly(L-Pt-G3)NC
EXPNO                   1
PROCNO                  1

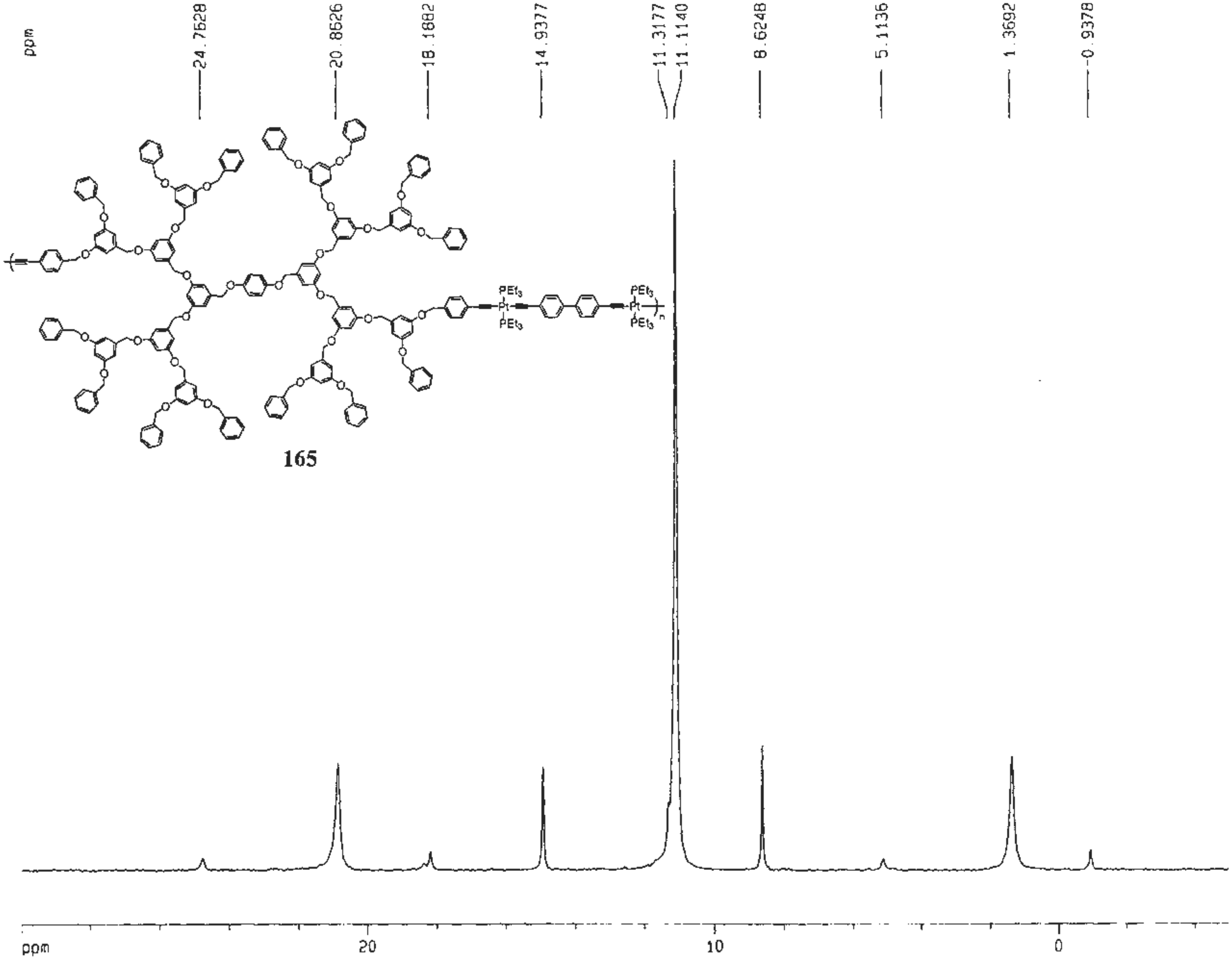
F2 - Acquisition Parameters
Date_            20081206
Time                 3.07
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   4636
DS                      0
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ              1.4451188 sec
RG                   8192
DW                 22.050 usec
DE                   6.00 usec
TE                  296.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.00 usec
PL1                 -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF             75.4677453 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.02 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37384 Hz/cm
```



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```
Current Data Parameters
NAME     Poly(L-Pt-G3)NP
EXPNO                  1
PROCNO                 1

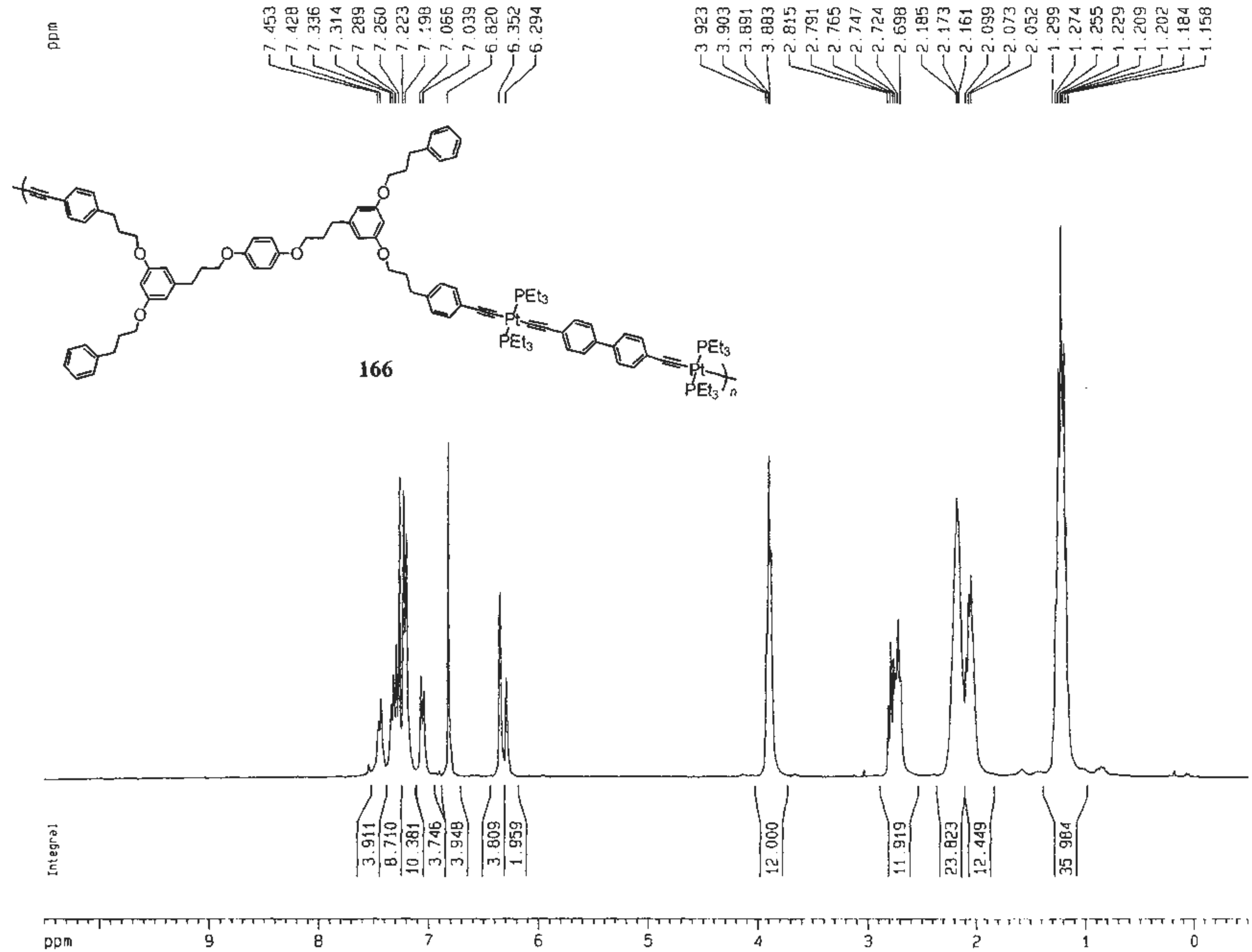
F2 - Acquisition Parameters
Date_           20081206
Time                2.51
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     0
SWH          37664.785 Hz
FIDRES        0.574719 Hz
AQ           0.8700404 sec
RG                7298.2
DW             13.275 usec
DE               6.00 usec
TE               295.2 K
D1          0.30000001 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1               3.00 usec
PL1              -6.00 dB
SFO1       121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2          100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2       300.1316097 MHz

F2 - Processing parameters
SI                 65536
SF         121.4948412 MHz
WDW                   EM
SSB                    0
LB               3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX              22.00 cm
CY              12.98 cm
F1P            30.000 ppm
F1            3644.85 Hz
F2P            -5.000 ppm
F2            -607.47 Hz
PPMCM        1.59091 ppm/cm
HZCM       193.28725 Hz/cm
```



166

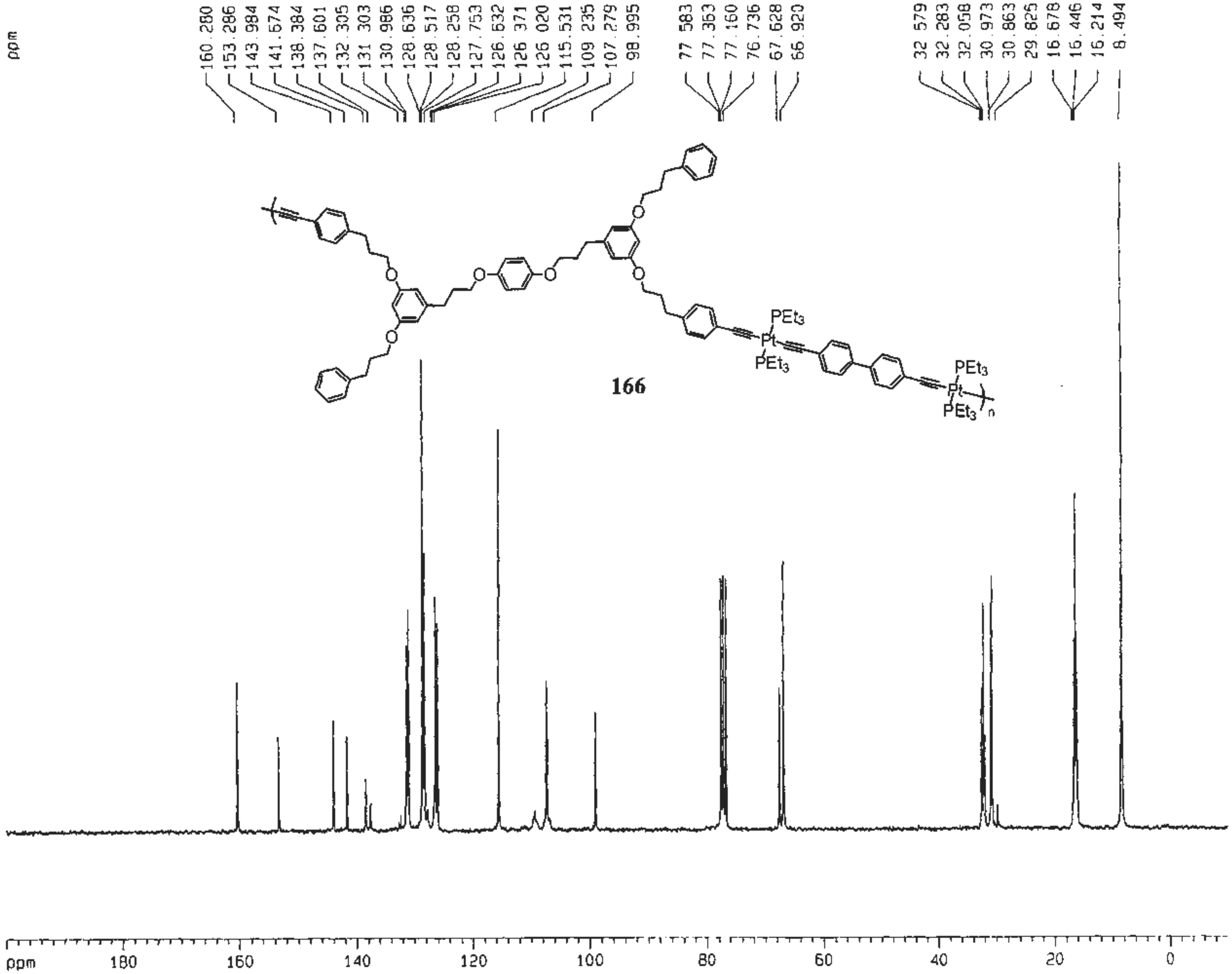
```
Current Data Parameters
NAME      Poly(L-Pt-G1') (ppt)
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090114
Time                18.28
INSTRUM            dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                zg
TD                  16384
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              3601.657 Hz
FIDRES           0.219828 Hz
AQ              2.2745588 sec
RG                  161.3
DW                138.825 usec
DE                 198.32 usec
TE                  297.2 K
D1             5.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -2.00 dB
SFO1          300.1315000 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300063 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  10.02 cm
F1P                10.500 ppm
F1                3151.36 Hz
F2P                -0.500 ppm
F2                -150.07 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.06500 Hz/cm
```



166

```
Current Data Parameters
NAME     Poly(L-Pt-G1')C
EXPNO                  1
PROCNO                 1

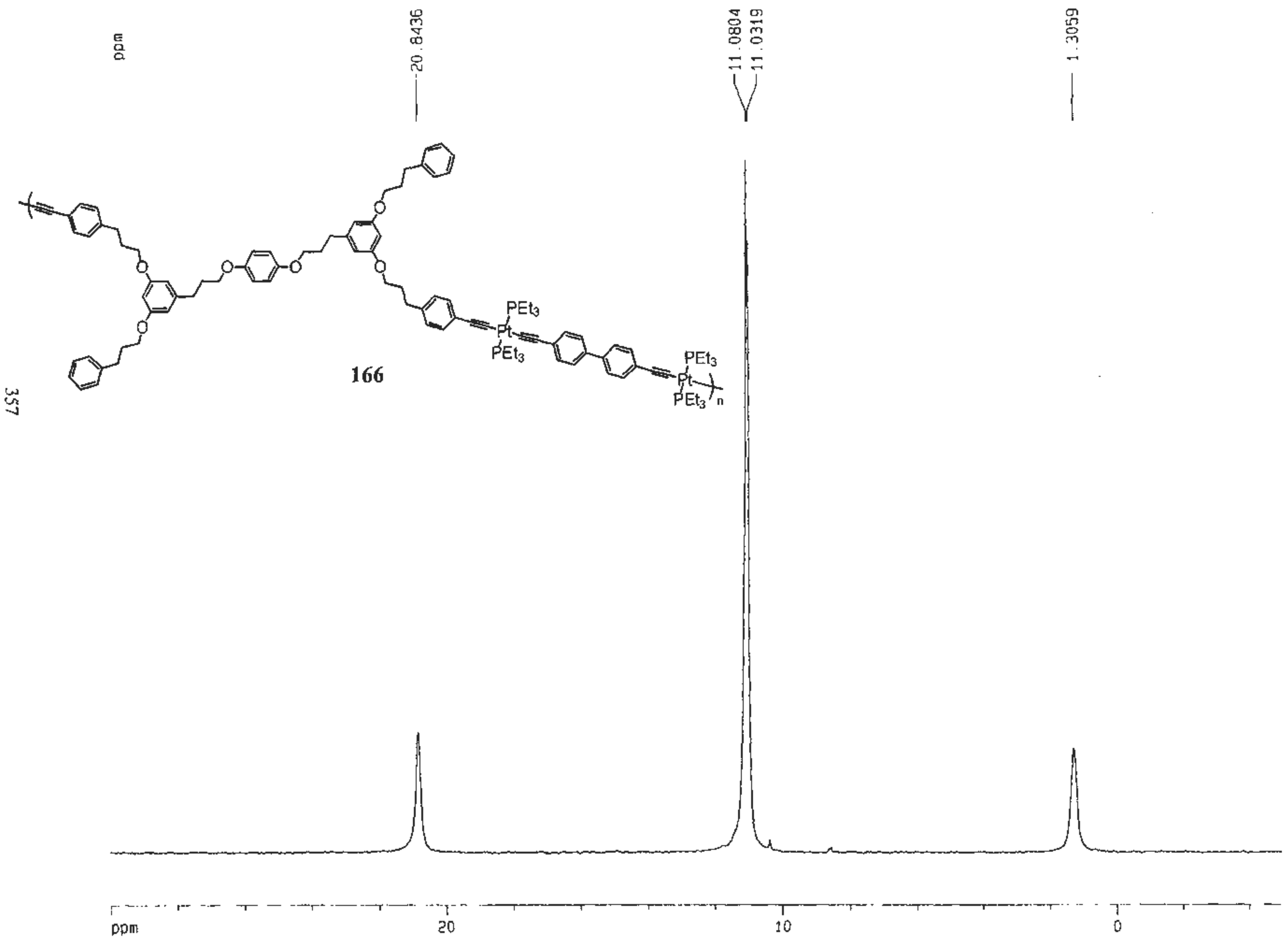
F2 - Acquisition Parameters
Date_           20090116
Time                6.22
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  4071
DS                     0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ             1.4451188 sec
RG                  8192
DW                22.050 usec
DE                  6.00 usec
TE                 297.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.00 usec
PL1                -6.00 dB
SFO1          75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               19.00 dB
SFO2         300.1315007 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677433 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 11.98 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.54545 ppm/cm
HZCM           720.37384 Hz/cm
```



166

```
Current Data Parameters
NAME      Poly(L-Pt-G1')P
EXPNO              1
PROCNO             1

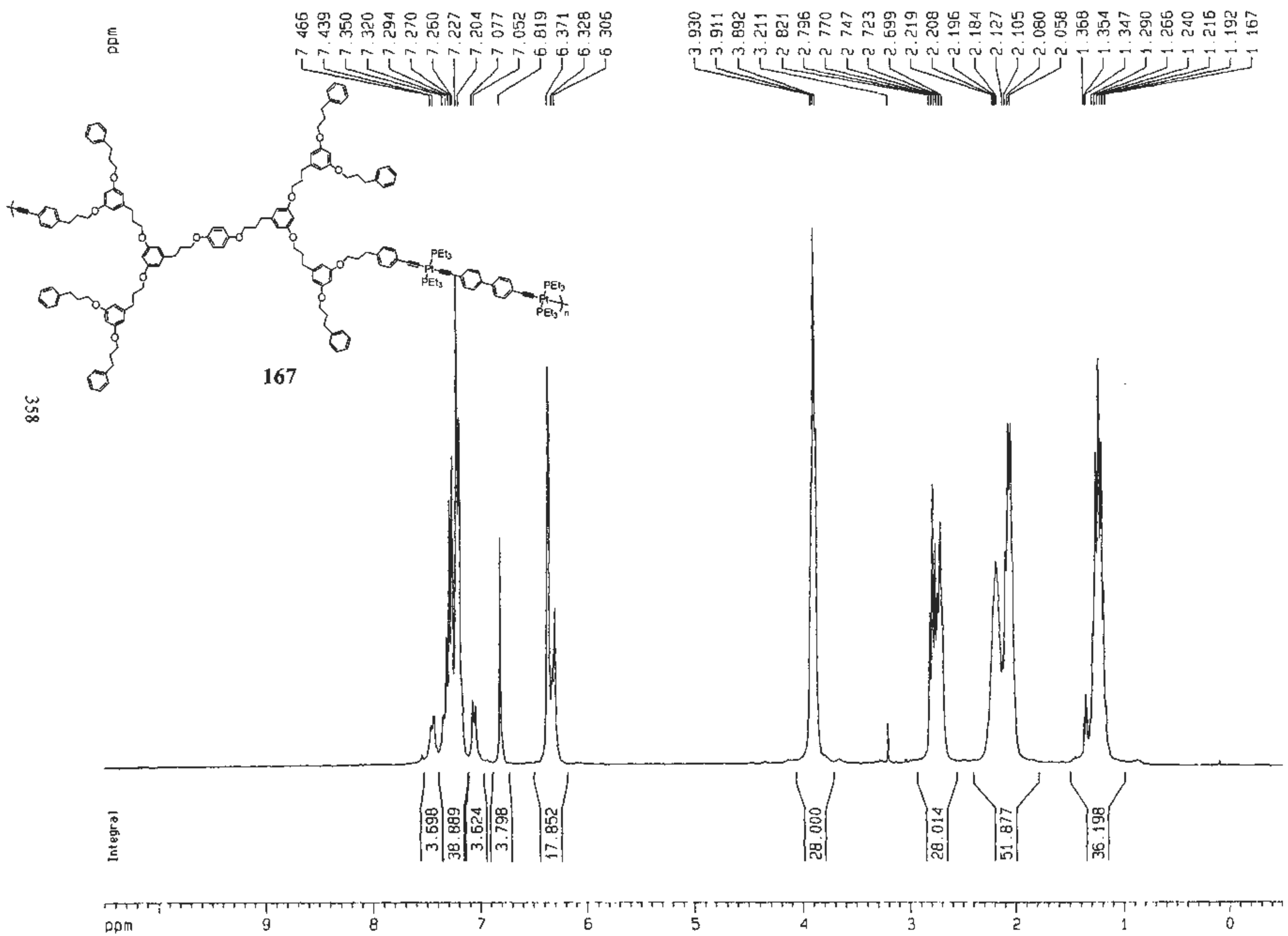
F2 - Acquisition Parameters
Date_          20090114
Time              19.34
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    0
SWH           37664.785 Hz
FIDRES         0.574719 Hz
AQ            0.8700404 sec
RG               4597.6
DW               13.275 usec
DE                 6.00 usec
TE                297.2 K
D1           0.30000001 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                31P
P1                 3.00 usec
PL1               -6.00 dB
SFO1        121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1316097 MHz

F2 - Processing parameters
SI                65536
SF          121.4948412 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                13.01 cm
F1P              30.000 ppm
F1              3644.85 Hz
F2P              -5.000 ppm
F2              -607.47 Hz
PPMCM           1.59091 ppm/cm
HZCM          193.28725 Hz/cm
```



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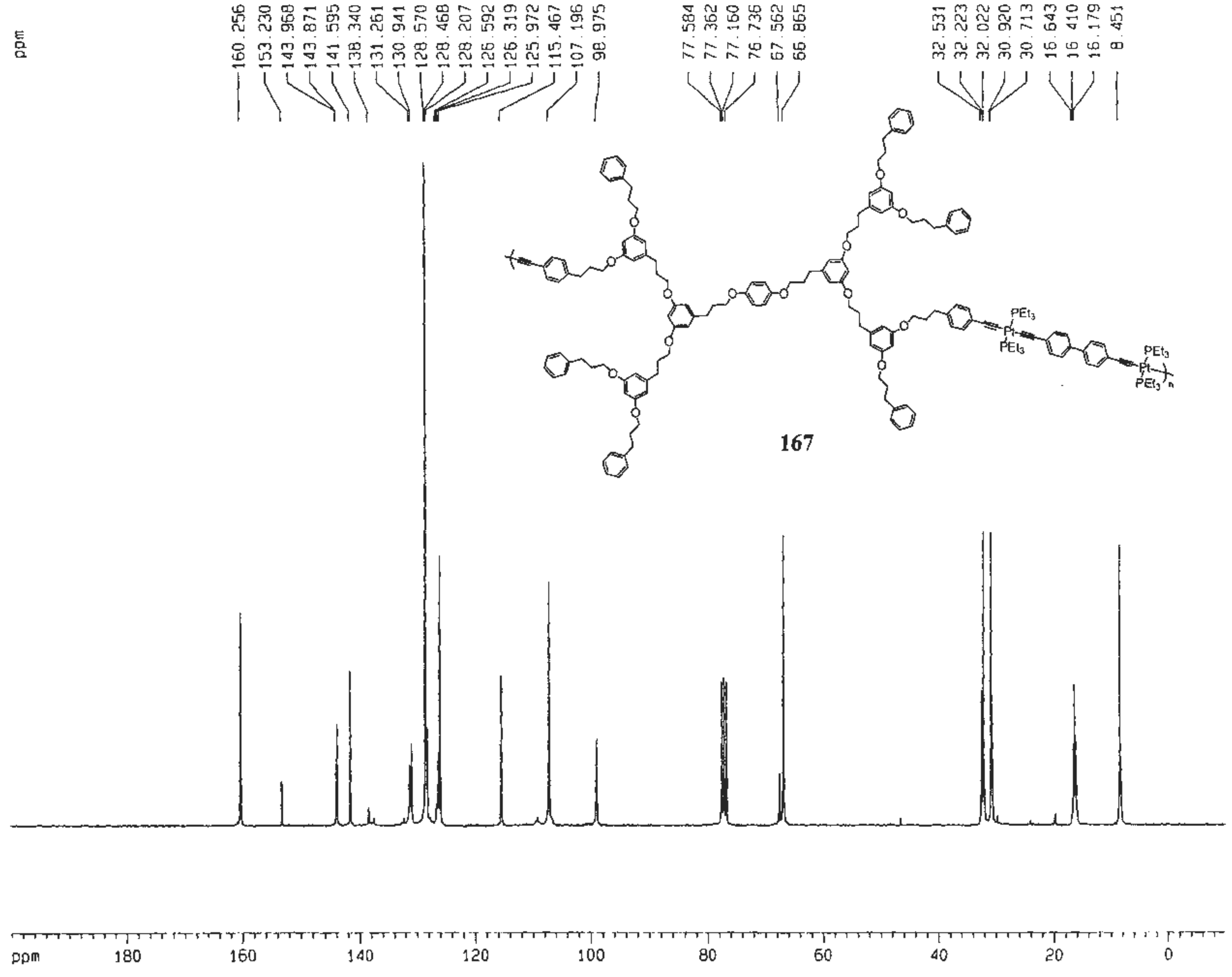
```
Current Data Parameters
NAME     Poly(L-Pt-G2')(ppt)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090114
Time               18 40
INSTRUM           dpx300
PROBHD   5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             3601.657 Hz
FIDRES          0 219828 Hz
AQ             2.2745588 sec
RG                    64
DW               138.825 usec
DE                198.32 usec
TE                 297.2 K
D1            5.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                -2.00 dB
SFO1        300 1315000 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300064 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  9.97 cm
F1P               10.500 ppm
F1               3151.36 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM         0.50000 ppm/cm
HZCM        150.06500 Hz/cm
```



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```
Current Data Parameters
NAME      Poly(L-Pt-G2')C
EXPNO              1
PROCNO             1

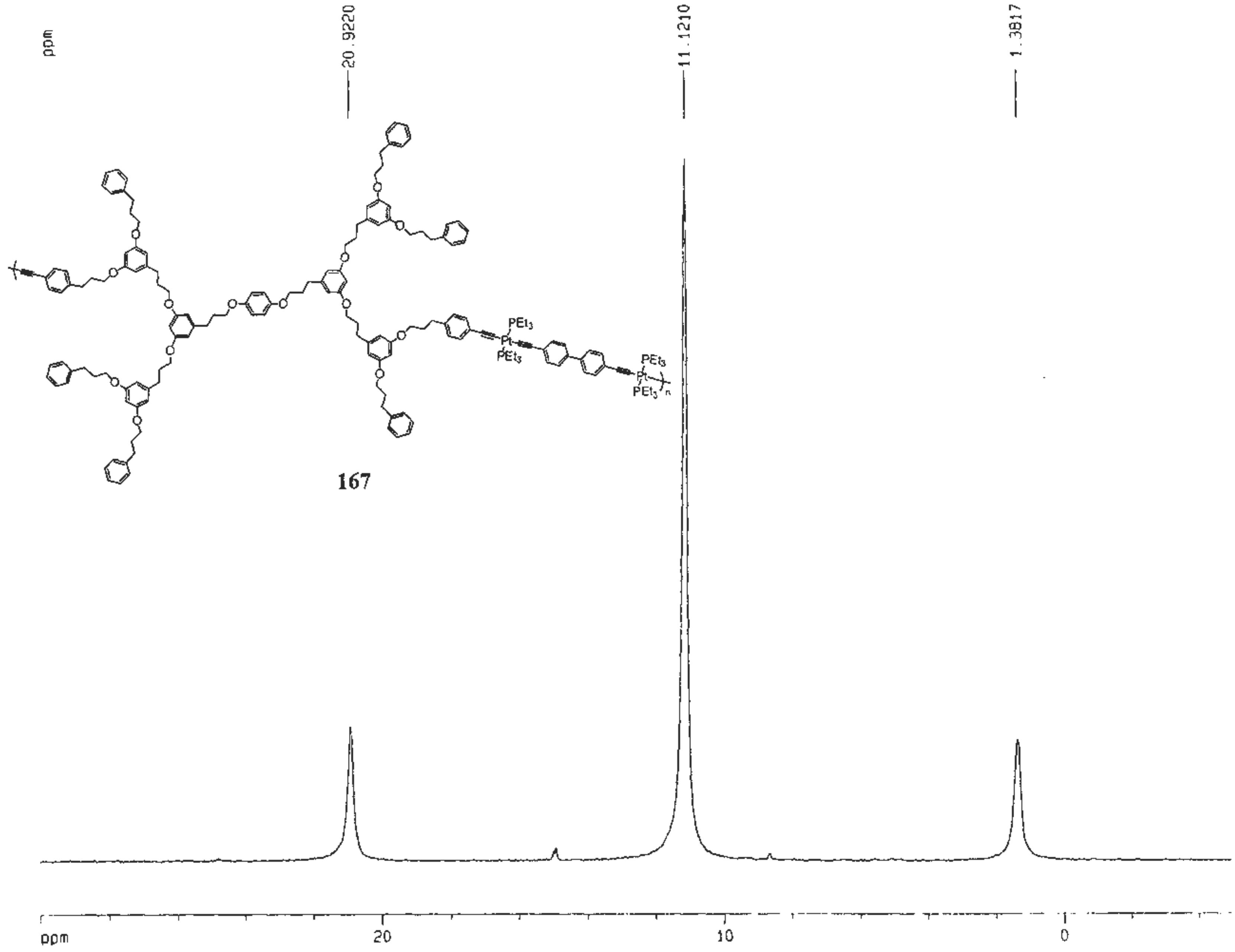
F2 - Acquisition Parameters
Date_          20090118
Time              23.39
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                 5000
DS                    0
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AO           1 4451188 sec
RG                 8192
DW               22.050 usec
DE                 6 00 usec
TE                298.2 K
D1          1.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 3.00 usec
PL1               -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100 00 usec
PL2              120 00 dB
PL12              19 00 dB
SFO2        300 1315007 MHz

F2 - Processing parameters
SI                65536
SF           75.4677520 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                11.98 cm
F1P             200.000 ppm
F1             15093.55 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.54545 ppm/cm
HZCM          720.37390 Hz/cm
```



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```
Current Data Parameters
NAME       Poly(L-Pt-G2')P
EXPNO                    1
PROCNO                   1

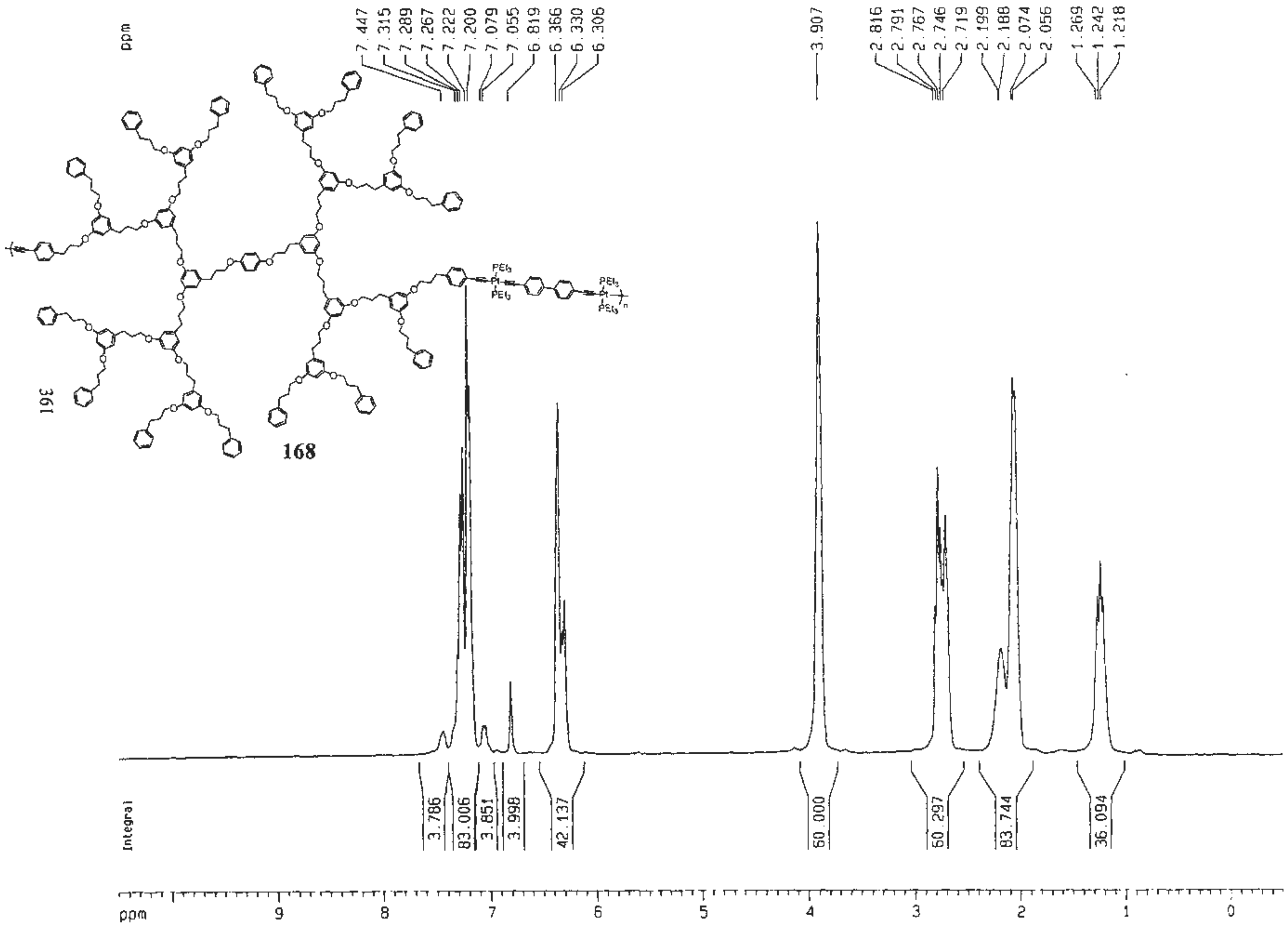
F2 - Acquisition Parameters
Date_             20090114
Time                 21.37
INSTRUM             dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     120
DS                       0
SWH            37664.785 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                  2580 3
DW                13.275 usec
DE                  6.00 usec
TE                 297.2 K
D1            0 30000001 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   31P
P1                    3.00 usec
PL1                  -6.00 dB
SFO1          121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2          300.1316097 MHz

F2 - Processing parameters
SI                   65536
SF            121.4948412 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                   12.99 cm
F1P                 30 000 ppm
F1                 3644.85 Hz
F2P                 -5.000 ppm
F2                 -607.47 Hz
PPMCM              1.59091 ppm/cm
HZCM             193.28725 Hz/cm
```



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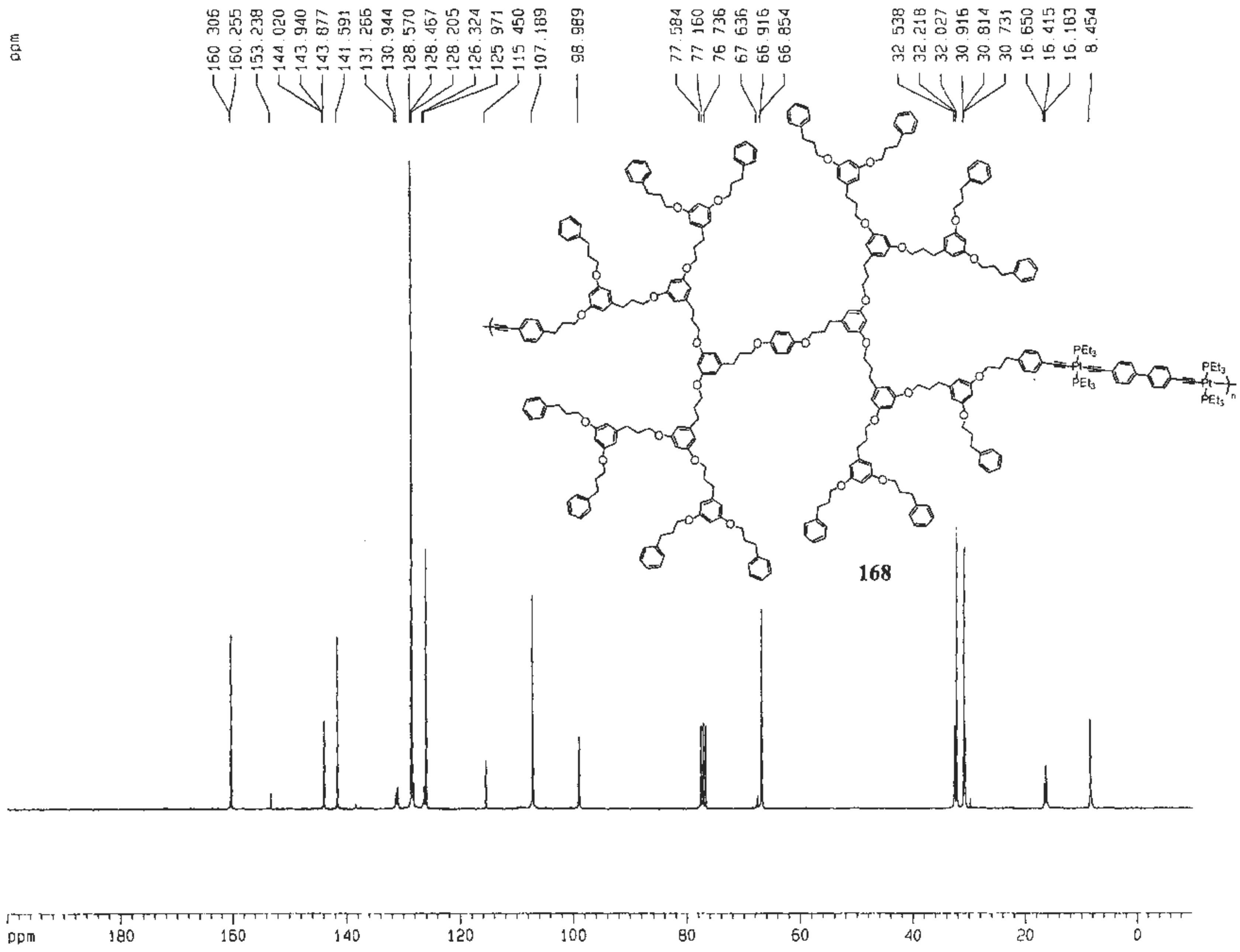
```
Current Data Parameters
NAME      Poly(L-Pt 63') (ppt)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090114
Time               18 48
INSTRUM           dpx300
PROBHD    5 mm BBO BB-1H
PULPROG               zg
TD                 16384
SOLVENT            CDCl3
NS                    32
DS                     0
SWH          3601 657 Hz
FIDRES       0.219828 Hz
AQ          2 2745588 sec
RG                    64
DW           138.825 usec
DE            198 32 usec
TE               297.2 K
D1        5.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1               9.00 usec
PL1              -2.00 dB
SFO1       300.1315000 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300061 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              22.00 cm
CY               9.96 cm
F1P            10.500 ppm
F1             3151.36 Hz
F2P            -0.500 ppm
F2             -150.07 Hz
PPMCM       0.50000 ppm/cm
HZCM      150.06500 Hz/cm
```



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```
Current Data Parameters
NAME      Poly(L-Pt-G3')C
EXPNO                   1
PROCNO                  1

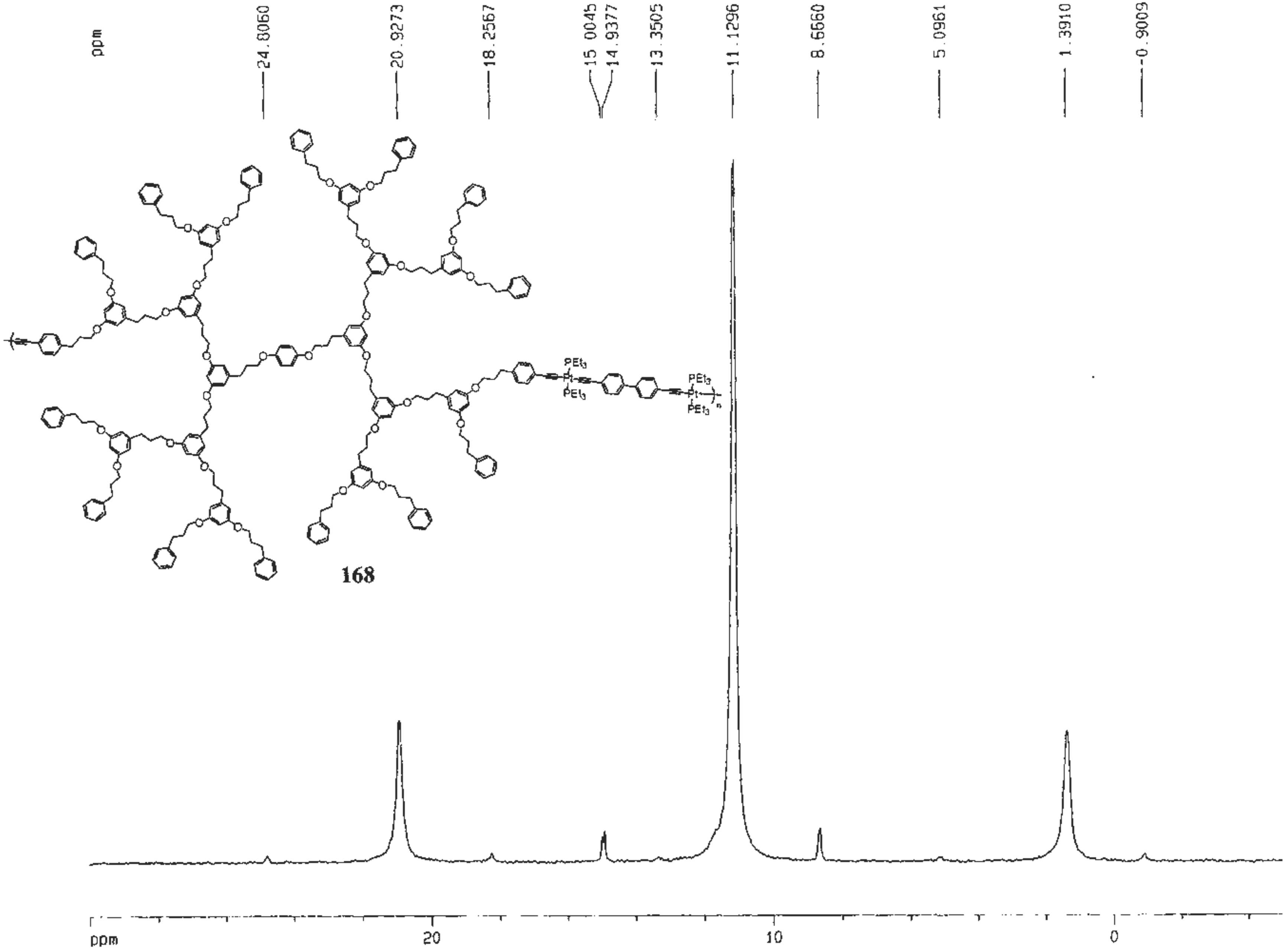
F2 - Acquisition Parameters
Date_            20090119
Time                 3.09
INSTRUM            dpx300
PROBHD    5 mm BBO BB-1H
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   7000
DS                      0
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ            1.4451188 sec
RG                   8192
DW              22.050 usec
DE                6.00 usec
TE               297.2 K
D1         1.00000000 sec
d11        0.03000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                3.00 usec
PL1              -6.00 dB
SFO1        75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SFO2       300.1315007 MHz

F2 - Processing parameters
SI                  65536
SF         75.4677538 MHz
WDW                    EM
SSB                     0
LB                  1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
CY                  12.01 cm
F1P               200.000 ppm
F1               15093.55 Hz
F2P               -10.000 ppm
F2                -754.68 Hz
PPMCM             9.54545 ppm/cm
HZCM            720.37390 Hz/cm
```



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Current Data Parameters
NAME     Poly(L-Pt-G3')P
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20090114
Time              21 42
INSTRUM          dpx300
PROBHD   5 mm BBO BB-1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  200
DS                    0
SWH          37664 785 Hz
FIDRES        0.574719 Hz
AQ           0 8700404 sec
RG               7298.2
DW               13 275 usec
DE                 6.00 usec
TE                297.2 K
D1           0.30000001 sec
d11          0 03000000 sec
MCREST       0 00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                31P
P1                 3.00 usec
PL1               -6 00 dB
SFO1        121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              19.00 dB
SFO2        300.1316097 MHz

F2 - Processing parameters
SI                65536
SF          121.4948412 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                12.96 cm
F1P              30.000 ppm
F1              3644.85 Hz
F2P              -5.000 ppm
F2              -607.47 Hz
PPMCM           1.59091 ppm/cm
HZCM          193.28725 Hz/cm
```