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Supporting Information

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Preparation of Helicenes via Olefin Metathesis. **

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Experimental Section

General.

All reactions that were performed in an anhydrous atmosphere were performed under an inert argon or nitrogen atmosphere in glassware that had previously been dried overnight at 120 °C or had been flame dried and cooled under a stream of argon or nitrogen.[1] All chemical products were obtained from Sigma-Aldrich Chemical Company or Strem Chemicals and were reagent quality. These products were used without further purification. Catalyst **4** was prepared according to a literature procedure.[2] Basic solvents were obtained from VWR. Anhydrous solvents (CH_2Cl_2, Et_2O, THF, toluene, DMF and hexanes) were dried and deoxygenated using a GlassContour system (Irvine, CA). Isolated yields reflect the mass obtained following flash column silica gel chromatography using the method reported by W. C. Still[3] and used silica gel obtained from Silicycle (40–63 nm; 230–240 mesh). Thin layer chromatography (TLC) was performed using glass backed silica gel plates (Merck GF–UV254, 0.25 mm) coated with a fluorescence indicator. Following plate development, the products were detected using a UV lamp.

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[1] D. F. Shriver, M. A. Drezdon, *The Manipulation of Air-Sensitive Compounds*; 2e Édition, éd.; Wiley: New York, 1986.
[2] R. Bujok, M. Bieniek, M. Masnyk, A. Michrowska, A. Sarosiek, H. Stepowska, D. Arlt, K. Grela, *J. Org. Chem.* **2004**, *69*, 6894-6896. See also : a) H. Wakamatsu, S. Blechert, *Angew. Chem. Int. Ed.* **2002**, *41*, 2403-2405; b) A. M. Dunne, S. Mix, S. Blechert, *Tetrahedron Lett.* **2003**, *44*, 2733-2736.
[3] W. C.Still,; M. Kahn, A. Mitra, *J. Org. Chem.* **1978**, *43*, 2923.

All nuclear magnetic resonance (NMR) experiments (^{1}H, ^{13}C) were performed using a Bruker AMX-300 and AV-300 (300 MHz, 75 MHz and 70.6 MHz) and Bruker ARX-400 and AV-400 (400 MHz and 100 MHz and 94.1 MHz) instruments. Chemical shifts are expressed in ppm and referenced via a deuterated solvent. The multiplicity of the signals are described as: s = singlet, d = doublet, t = triple t, q = quadruplet, m = multiplet and br = broad.

X-ray diffraction experiments were performed using Ebraf-Nonius CAD-3 and CAD-4 instruments at the « Laboratoire de diffraction des rayons X » at the University of Montréal.

High and low resolution mass spectrometry was performed at the « Laboratoire de spectrométrie de masse » at the University of Montréal using electrospray ionization techniques.

Additional detail of catalyst optimization :

Investigations began with converting 2,2'-binaphthol to its corresponding di-vinyl precursor **5** and subjected to various olefin metathesis catalysts (Table 1). No conversion of **5** to [5]-helicene was observed using Grubbs' 1st generation catalyst. However, traces of [5]-helicene were observed using Grubbs' 2nd generation catalyst and performing the reaction at elevated temperatures afforded the product in 60-69 % yield (Table 1, Entries 2 and 3). When conventional heating was replaced with microwave heating, a 41 % yield could be isolated after only five

minutes (Entry 4). Subsequent optimization resulted in an isolated and reproducible yield of 88 % for [5]-helicene after 25 minutes in CH_2Cl_2 at 100 °C.

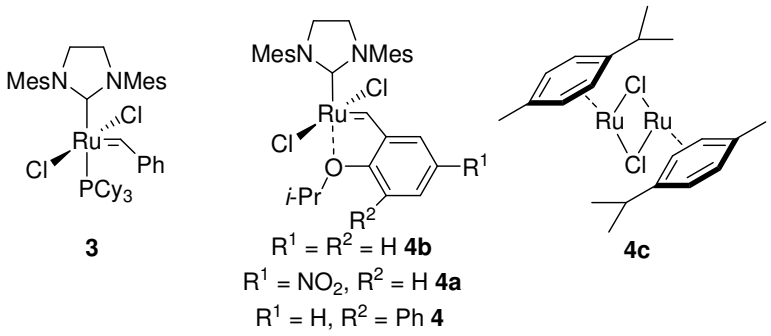
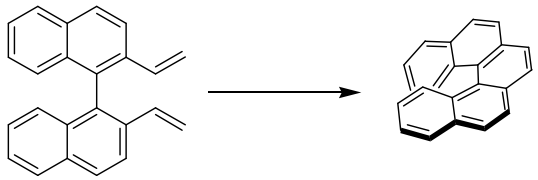


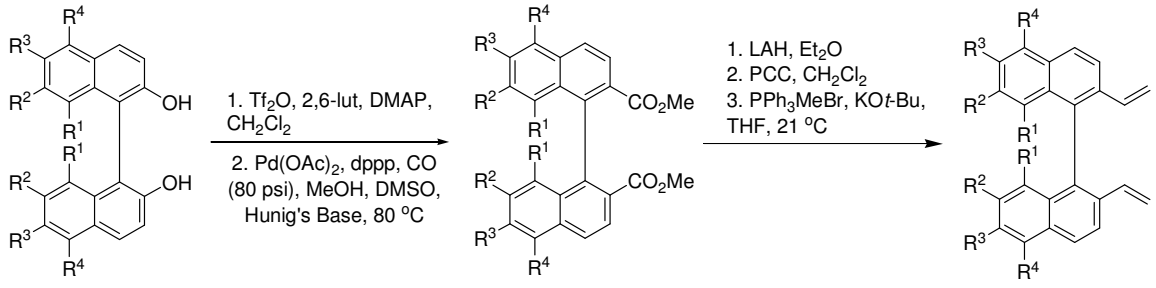
Table 1. Optimization of the formation of [5]-helicene via olefin metathesis.



Entry	Catalyst	Conditions[a]	Isolated Yield (%)
1	**Grubbs 1**	40 °C, CH_2Cl_2, 24 h	0
2	**Grubbs 2**	21 °C, PhH, 24 h	10
3	**Grubbs 2**	Δ , PhH, 24 h	60-69
4	**Grubbs 2**	150 °C (microwave), PhH, 5 min.	41
5	**Grubbs 2**	110 °C (microwave), PhH, 25 min.	75
6	**Grubbs 2**	100 °C (microwave), CH_2Cl_2, 25 min.	88
7	**4b**	21 °C, PhH, 10 d	10
8	**4b**	40 °C, PhH, 48 h	50
9	**4b**	80 °C, PhH, 24 h	90
10	**4a**	40 °C, PhH, 24 h	60
11	**4**	40 °C, PhH, 24 h	78-93
12	**4**	21 °C, PhH, 48 h	48
13	**4c**	40 °C, PhH, 24 h (1,1-diphenyl-2-propyn-1-ol, IMesHCl, AgOTf, Cs_2CO_3)	0

Although this protocol is extremely rapid, we sought to decrease the reaction temperature through the use of alternative Ru-based metathesis catalysts. Consequently, **5** was treated with Grubbs-Hoveyda catalyst **4b** at room temperature but only 10 % of the product could be isolated and the remainder of **5** was recovered (Entry 7). Gratifyingly, heating **5** at reflux in the presence of **4b** led to a 90 % yield of [5]-helicene (Entry 9). Unfortunately, a reduction in the reaction temperature to 40 °C also led to a drop in yield to 50 % (Entry 8). Maintaining the reaction temperature but exchanging **4b** for the nitro-substituted catalyst **4a** provided a slight increase in yield to 60 % (Entry 10), however the largest increase was observed using catalyst **4**. Isolated yields of 78-93 % of [5]-helicene could be obtained at 40 °C and **4** showed significant activity at room temperature, providing **5** in 48 % yield after 48 h (Entries 11 and 12 respectively). No ring closing of **5** was observed with cationic Ru-based catalyst derived from $[Ru(p\text{-cymeme})Cl_2]_2$ (Entry 13).

Preparation of di-vinyl precursors.



General procedure: All 2,2′-binaphthols were converted to the corresponding ditriflates according to literature

procedures.[4] The subsequent carboxymethylation is performed according to an established protocol.[5] The diester is then subjected to a three step protocol to convert the diester to the corresponding di-vinyl precursor. These three steps are all performed in the same day and the crude product of each step is carried forth. The di-vinyl precursor is then fully characterized and spectral data is described below. The reduction (LAH, Et_2O) and oxidation (PCC, CH_2Cl_2) on 2,2'-binaphthyl esters has been previously reported.[6] A general protocol for the Wittig reaction is as follows:

General procedure for Wittig reaction: In a flame dried round bottom flask equipped with a magnetic stirrer, was placed methylenetriphenylphosphonium bromide (1.17 g, 3.26 mmol). Anhydrous THF (10 mL) is added and the mixture is cooled to 0°C in an ice bath. Following addition of potassium *tert*-butoxide (367 mg, 3.27 mmol), the crude aldehyde (353 mg, 0.817 mmol) is added via canula as a solution of THF (5 mL). The solution is stirred for 15 minutes and quenched by the addition of water (10 mL). The organic phase is separated, dried ($MgSO_4$) and the di-vinyl

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[4] W. Zhang, Q. Xu, M. Shi, *Tetrahedron: Asym*. **2004**, *15*, 3161-3169.

[5] For the carboxymethylation of BINOL see: M. Seki, S.-I. Yamada, T. Kuroda, R. Imashiro, T. Shimizu, *Synthesis* **2000**, *12*, 1677-1680. For additional examples of carboxymethylation of 2,2'-binaphthyls see: T. Ooi, Y. Uematsu, K. Maruoka, *J. Org. Chem*. **2003**, *68*, 4576-4578.

[6] D. L. An, T. Nakano, A. Orita, J. Otera, *Angew. Chem. Int. Ed.* **2002**, *41*, 171-173.

product **1** is purified by flash column silica gel chromatography to afford the product.

2,2′-divinyl-1,1′-binaphthyl (1)[3,4] : Purification by flash column silica gel chromatography (5:1 Hex's–EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 – 7.93 (m, 6H), 7.47 (*apparent* t, *J* = 7.9 Hz, 2H), 7.53 (*apparent* t, *J* = 8.2 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.35 (dd, *J* = 17.5, 11.0 Hz, 2H), 5.84 (d, *J* = 17.5 Hz, 2H), 5.12 (d, *J* = 11.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.4, 134.7, 134.5, 133.8, 133.6, 128.6, 128.4, 127.2, 127.0, 126.4, 122.9, 115.6; HRMS (ESI^+) *m/z* 307.1481 (307.1484 calcd for $C_{24}H_{19}$ $[M+H]^+$.

6,6′-di-*p*-tolyl-2,2′-divinyl-1,1′-binaphthyl (5) : Purification by flash column silica gel chromatography (5:1 Hex's–EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (d, *J* = 1.7 Hz, 2H), 7.99 (d, *J* = 9.2 Hz, 2H), 7.95 (d, *J* = 9.2 Hz, 2H), 7.56 (m, 4H), 7.48 (dd, *J* = 7.0, 1.7 Hz, 2H), 7.32 – 7.26 (m, 4H), 7.13 (d, *J* = 8.7 Hz, 2H), 6.29 (dd, *J* = 17.5, 11.0 Hz, 2H), 5.80 (d, *J* = 17.5 Hz, 2H), 5.08 (d, *J* = 11.0 Hz, 2H), 2.39 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.4, 137.7, 137.1, 134.7, 134.0, 133.8, 133.5, 132.2, 129.5, 128.3, 127.2, 127.0, 126.0,

125.3, 122.7, 115.0, 21.0; HRMS (ESI^+) *m/z* 593.1392 (593.1386 calcd for $C_{38}H_{30}Ag$ $[M+Ag]^+$.

7,7′-dimethoxy-2,2′-divinyl-1,1′-binaphthyl (7):[7] Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded the product as an off-white solid: ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.1 Hz, 2H), 7.82(d, *J* = 3.4 Hz, 2H), 7.80 (d, *J* = 3.4 Hz, 2H), 7.13 (dd, *J* = 8.8, 2.4 Hz, 2H), 6.45 (d, *J* = 2.4 Hz, 2H), 6.35 (dd, *J* = 17.3, 12.0 Hz, 2H), 5.80 (dd, *J* = 17.3, 1.2 Hz, 2H), 5.09 (dd, *J* = 12.0, 1,2 Hz, 2H), 3.51 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.0, 135,1, 134.6, 134.3, 129.3, 128.5, 127.8, 120.1, 17.9, 114.9, 105.5, 54.9; HRMS (ESI^+) *m/z* 473.0665 (calcd 473.0668 for $C_{26}H_{22}O_2Ag$ $[M]^+$).

7-7′- bis(benzyloxy)-2,2′- divinyl -1,1′-binaphthalene (9):[8] Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded the product as an off-white solid: ^1H RMN (300 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.6 Hz, 2H), 7.85 (d, *J* = 8.9, 2H), 7.80 (d, *J* = 8.6, 2H), 7.23-7.18 (m, 10H), 7.13 (dd, *J* = 2.5, 1.5 Hz, 2H), 6.43 (d, *J* = 2.2 Hz, 2H), 6.12-6.40 (m, 2H), 5.73 (d, *J* = 16.8 Hz, 2H), 4.72 (d, *J* = 12.0

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[7] For more info on previously isolated and characterized intermediates see: W.-C. Yuan, L.-F. Cun, L.-Z. Gong, A.-Q. Mi, Y.-Z. Jiang, *Tetrahedron Lett*. **2005**, 46, 509-512.

[8] For more info on previously isolated and characterized intermediates see: a) D. Che, N. G. Andersen, S. Y. W. Lau, M. Parvez, B. A. Keay, *Tetrahedron.* **2000**, *11*, 1919-1924; b) G.P. Roth, J. A. Thomas, *Tetrahedron Lett.* **1992**, *33*, 1959-1962.

Hz, 2H), 4.70 (d, *J* = 12.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$), δ 157.4, 136.93, 135.5, 135.0, 134.6, 133.5, 129.8, 129.1, 128.8, 128.2, 128.0, 120.8, 119.2, 115.4, 107.4, 70.082; HRMS (ESI^+) *m/z* 625.1292 (625.1290 calcd for $C_{38}H_{30}O_2Ag$ $[M+Ag]^+$.

5,5′-6,6′-7,7′-8,8′-octahydro-2,2′-divinyl-1,1′-binaphthyl (11)[9] : Purification by flash column silica gel chromatography (5:1 Hex's–EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (d, *J* = 1.7 Hz, 2H), 7.47 (d, *J* = 7.6 Hz, 2H), 7.07 (d, *J* = 7.6 Hz, 2H), 6.12 (ddd, *J* = 15.2, 11.0, 0.4 Hz, 2H), 5.53 (dd, *J* = 15.2, 0.4 Hz, 4H), 4.94 (dd, *J* = 11.0, 0.4 Hz, 2H), 2.80 (t, *J* = 5.9 Hz, 4H), 2.17 – 1.95 (m,4H), 1.75 – 1.58 (m,8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.9, 137.0, 134.9, 133.1, 128.4, 128.3, 121.7, 113.2, 29.9, 27.3, 23.2, 22.7; HRMS (ESI^+) *m/z* 421.1079 (421.1081 calcd for $C_{24}H_{26}Ag$ $[M+Ag]^+$.

2,2′-divinyl-1,1′-biphenanthryl (13):[10] Purification by flash column silica gel chromatography (5:1 Hex's–EtOAc) afforded the product as an off-white solid:: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.85 (d, *J* = 8.8 Hz, 2H), 8.80 (d, *J* = 8.4 Hz,

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[9] For more info on previously isolated and characterized intermediates see: A. K. Unni, N. Takenaka, H. Yamamoto, V. H. Rawal, *J. Am. Chem. Soc.* **2005**, *127*, 1336-1337.

[10] For more info on previously isolated and characterized intermediates see T. Hattori, K. Sakurai, N. Koike, S. Miyano, H. Goto, F. Ishiya, N. Harada, *J. Am. Chem. Soc.* **1998**, *120*, 9086-9087.

2H), 8.11 (d, *J* = 11.8 Hz, 2H), 7.81 (d, *J* = 9.2 Hz, 2H), 7.69 (dt, *J* = 9.5, 1.8 Hz, 2H), 7.58 (dt, *J* = 10.5, 1.3 Hz, 2H), 7.48 (d, *J* = 12.2 Hz, 2H), 6.35 (dd, *J* = 23.4, 14.7 Hz, 2H), 5.85 (dd, *J* = 23.3, 1.1 Hz, 2H), 5.09 (dd, *J* = 14.7, 1.2 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.1, 134.9, 134.6, 131.7, 131.5, 130.1, 129.8, 128.4, 127.4, 126.7, 126.6, 124.9, 123.0, 122.7 (2 C's overlap), 115.3; HRMS (ESI$^+$) *m/z* 513.0772(513.07669 calcd for $C_{32}H_{22}Ag$ $[M+Ag]^+$.

3-vinyl-4-(2-vinylnaphthalen-1-yl)phenanthrene (15): Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (m, 3H), 7.94 (m, 2H), 7.85 (d, *J* = 8.8 Hz, 1H), 7.76 (m, 2H), 7.42 (ddd, *J* = 10.5, 7.0, 2.2 Hz, 1H), 7.28 (m, 2H), 7.18 (m, 2H), 6.87 (ddd, *J* = 7.9, 7.8, 1.5 Hz, 2H), 6.41 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.24 (dd, *J* = 17.4, 11.0 Hz, 1H), 5.77 (ddd, *J* = 17.5, 7.9, 1.1 Hz, 2H), 5.03 (ddd, *J*= 11.2, 2.8, 0.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.8, 136.8, 135.4, 134.5, 134.0, 133.5, 133.4 (2 C's overlap), 133.1, 132.6, 130.8, 129.7, 129.3, 128.6, 128.4, 128.2, 127.9, 127.6, 126.7, 126.4, 126.3, 126.2, 125.9, 125.7, 123.8, 122.9, 115.4, 115.2; HRMS (ESI$^+$) *m/z* 463.0597(463.0616 calcd for $C_{28}H_{20}Ag$ $[M+Ag]^+$.

2,2′-divinyl-1,1′-biphenanthryl (17):[11] Purification by flash column silica gel chromatography (5:1 Hex's–EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.1 Hz, 2H), 7.92 (d, *J* = 8.1 Hz, 2H), 7.85 (d, *J* = 9.4 Hz, 2H), 7.75 (app d, *J* = 8.1 Hz, 4H), 7.65 (d, *J* = 9.4 Hz, 2H), 7.28 (m, 2H), 6.82 (ddd, *J* = 9.4, 7.4, 1.5 Hz, 2H), 5.99 (dd, *J* = 18.0, 9.0 Hz, 2H), 5.48 (d, *J* = 18.0 Hz, 2H), 4.76 (d, *J* = 9.0 Hz, 2H), 2.39 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.9, 135.7, 135.0, 133.5, 133.4, 131.0, 129.5, 129.2, 128.5, 128.0, 127.4, 126.2, 125.9, 125.8, 124.7, 115.4; HRMS (ESI^+) *m/z* 513.0773(513.07669 calcd for $C_{32}H_{22}Ag$ $[M+Ag]^+$.

Preparation of helicenes.

General procedure using microwave irradiation and catalyst **3:** In a flame dried microwave vessel, equipped with a magnetic stirrer, is charged with 2,2′-divinyl-1,1′-biphenanthryl **17** (10 mg, 24.6 mmol). Anhydrous methylene chloride (1.4 mL) is added and the mixture is stirred until dissolution of all the substrate. Grubbs catalyst **3** (3.0 mg, 3.6 mmol) is added, the tube is sealed and the reaction

[11] For more info on previously isolated and characterized intermediates see: a) K. Yamamura, S. Ono, H. Ogoshi, H. Masuda, Y. Kuroda, *Synlett* **1989**, *1*, 18–19; b) M. Gingras, F. Dubois, Fabien. *Tetrahedron Lett.* **1999**, *40*, 1309–1312; c) T. Hayashi, H. Iwamura, Y. Uozumi, Y. Matsumoto, F. Ozawa, *Synthesis* **1994**, *5*, 526–32.

vessel is irradiated in a microwave at 100 °C for 60 minutes. The reaction mixture is concentrated in vacuo and purified by flash column silica gel chromatography (30:1 Hex's–EtOAc) to afford 8.1 mg of [7]–helicene **18** (81 %) as an off–white solid.

General procedure using a sealed tube reaction vessel and catalyst **4**: In a glovebox, a flame dried 100 mL sealed tube, equipped with a magnetic stirrer, is charged with 2,2'–divinyl–1,1'–biphenanthryl **17** (10 mg, 24.6 mmol). Anhydrous benzene (1.7 mL) is added and the mixture is stirred until dissolution of all the substrate. Grubbs–Hoveyda catalyst **4** (3.0 mg, 3.7 mmol) is added, the tube is sealed and the reaction vessel is immersed in a pre–heated 40°C oil bath for 24h. The reaction mixture is concentrated in vacuo and purifies by flash column silica gel chromatography (30:1 Hex's–EtOAc) to afford 8.0 mg of [7]–helicene **18** (80%) as an off–white solid.

[7]–helicene (18):[12] Purification by flash column silica gel chromatography (30:1 Hex's–EtOAc) afforded [7]–helicene as an off–white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 (s, 2H), 7.99 (d, J = 8.2 Hz, 2H), 7.91 (d, J = 8.2 Hz, 2H), 7.72

[12] a) R. El Abed, B. Ben Hassine, J.–P. Genet, M. Gorsane, A. Marinetti, *Eur. J. Org. Chem.* **2004**, *7*, 1517–1522. b) F. Teply, I. G. Stara, I. Stary, A. Kollarovic, D. Saman, L. Rulisek, P. Fiedler, *J. Am. Chem. Soc.* **2002**, *124*, 9175–9180; c) M. Gingras, F. Dubois, *Tetrahedron Lett.* **1999**, *40*, 1309–1312.

(d, *J* = 8.2 Hz, 2H), 7.48 (d, *J* = 8.2 Hz, 2H), 7.28 (dd, J = 8.2, 0.6 Hz, 2H), 7.14 (d, *J* = 8.2 Hz, 2H), 6.88 (m, 2H), 6.38 (ddd, *J* = 8.2, 5.9, 0.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.8, 132.5, 131.6, 130.3, 129.1, 128.3, 128.1, 127.6 (2 C's overlap), 127.4, 126.6, 126.0, 125.8, 125.0, 124.4; HRMS (ESI$^+$) *m/z* 485.0454 (485.0439 calcd for $C_{30}H_{18}Ag$ $[M+Ag]^+$).

[5]-helicene (2):[13] Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.6 Hz, 2H), 7.98 – 7,87 (m, 8H), 7.53 (ddd, *J* = 8.0, 6.9, 1.0 Hz, 2H), 7.28 (ddd, *J* = 9.9, 6.9, 1.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.6, 132.3, 130.8, 129.0, 127.8, 127.5, 127.3, 127.0, 126.4, 126.3, 124.4; HRMS (ESI$^+$) *m/z* 279.1168 (279.1169 calcd for $C_{22}H_{15}$ $[M+H]^+$.

2,13-di-*p*-tolyl-[5]-helicene (6): Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded **10** (90%) as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.60 (d, *J* = 8.0 Hz, 2H), 8.14(d, *J* = 2.7 Hz, 2H), 7.96 (d, *J* = 9.4 Hz, 2H), 7.88 (d, *J* = 9.4 Hz, 2H), 7.86 (s, 2H), 7.67 (d, *J* = 8.0 Hz, 4H), 7.54 (dd, *J* = 9.4, 2.7 Hz, 2H),

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[13] a) R. Pathak, K. Vandayar, W. A. L. van Otterlo, J. P. Michael, M. A. Fernandes, C. B. de Koning, *Org. Biomol. Chem.* **2004**, *2*, 3504-3509; b) D. C. Harrowven, M. I. T. Nunn, D. R. Fenwick, *Tetrahedron Lett.* **2002**, *43*, 7345-7347.

7.29 (d, J = 8.0 Hz, 1H), 2.41 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.5, 137.6, 137.2, 132.9, 132.2, 129.7, 129.5, 129.3, 127.6, 127.0, 126.9, 126.8, 126.6, 125.0, 123.4, 21.0; HRMS (ESI$^+$) *m/z* 565.1079 (565.1080 calcd for $C_{36}H_{26}Ag$ $[M]^+$). The product can be recrystallized by slow evaporation of a solution of **6** in CH_2Cl_2 (see Figure S1 below).[14]

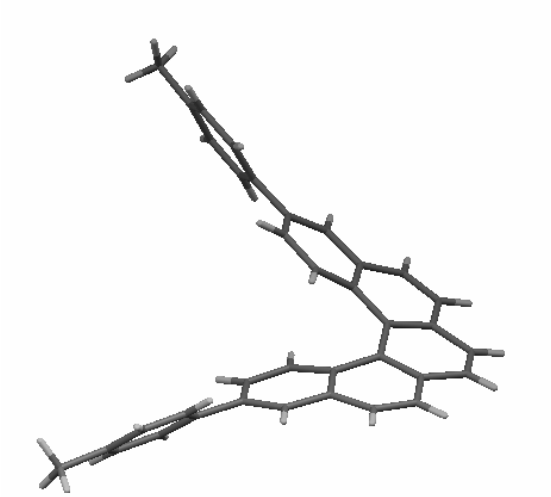


Figure S1: X-ray crystallographic analysis of 2,13-di-*p*-tolyl-[5]-helicene **(6)**.

2,13-dimethoxy-[5]-helicene (8): Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded **10** (90%) as an off-white solid (*NOTE: The product and the starting material are extremely difficult to separate by chromatography. Consequently, minor amounts of the*

[14] Crystallographic data (excluding structure factors) for the structures reported in this supporting information have been deposited with the Cambridge Cystallographic Data Centre as supplementary publication nos. CCDC-297971 (**6**) and CCDC-297972 (**12**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

starting material are observed in the 1H NMR spectrum (< 10%)): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.60 (d, *J* = 8.0 Hz, 2H), 8.14(d, *J* = 2.7 Hz, 2H), 7.96 (d, *J* = 9.4 Hz, 2H), 7.88 (d, *J* = 9.4 Hz, 2H), 7.86 (s, 2H), 7.67 (d, *J* = 8.0 Hz, 4H), 7.54 (dd, *J* = 9.4, 2.7 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 1H), 2.41 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.5, 137.6, 137.2, 132.9, 132.2, 129.7, 129.5, 129.3, 127.6, 127.0, 126.9, 126.8, 126.6, 125.0, 123.4, 21.0; HRMS (ESI^+) *m/z* 339.1379 (calcd 339.1370 for $C_{24}H_{19}O_2$ $[M+H]^+$).

2,13-dibenzyloxy-[5]-helicene : Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded the product as a pale yellow liquid: ^{1}H RMN (300 MHz, $CDCl_3$) δ 7.99 (d, *J* = 2.4 Hz, 2H), 7.93 (d, *J* = 8.8 Hz, 2H), 7.90 (d, *J* = 3.7 Hz, 2H), 7.82 (d, *J* = 8.5 Hz, 2H), 7.30 (m, 10H) 7.26 (dd, *J* = 3.2, 2.1 Hz, 2H), 4.90 (d, *J* = 11.4 Hz, 2H), 4.74 (d, *J* = 11.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$), δ 156.2, 137.1, 133.1, 131.6, 129.9, 128.9, 128.4, 128.3, 128.2, 127.8, 127.3, 126.7, 124.8, 118.7, 111.6, 77.0, 70.5, 30.2. HRMS (ESI^+) *m/z* 597.0978 (597.0976 calcd for $C_{36}H_{26}O_2Ag$ $[M+Ag]^+$.

1,2,3,4,11,12,13,14-octahydro-[5]-helicene (12) : Purification by flash column silica gel chromatography (5:1 Hex's-EtOAc) afforded the product as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.4 Hz, 2H), 7.43 (s,

2H), 7.32 (d, J = 7.6 Hz, 2H), 3.20 – 3.09 (m,1H), 3.07 – 2.96 (m,2H), 2.95 – 2.83 (m,2H), 2.69 (dt, J = 8.2, 3.5 Hz, 2H), 1.90 – 1.85 (m, 4H), 1.75 – 1.60 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.4, 133.7, 129.5, 126.4, 126.1, 124.7, 123.0, 122.0, 27.5, 26.4, 19.9, 18.9; HRMS (ESI$^+$) *m/z* 393.0778 (393.0766 calcd for $C_{22}H_{22}Ag$ $[M+Ag]^+$. The product can be recrystallized by slow evaporation of a solution of **12** in EtOAc (see Figure S2 below).[14]

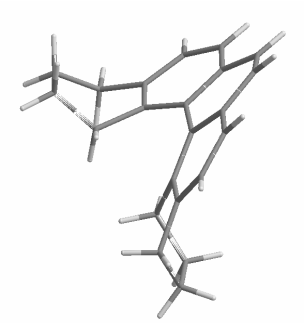


Figure S2: X-ray crystallographic analysis of 1,2,3,4,11,12,13,14-octahydro-[5]-helicene **(12)**

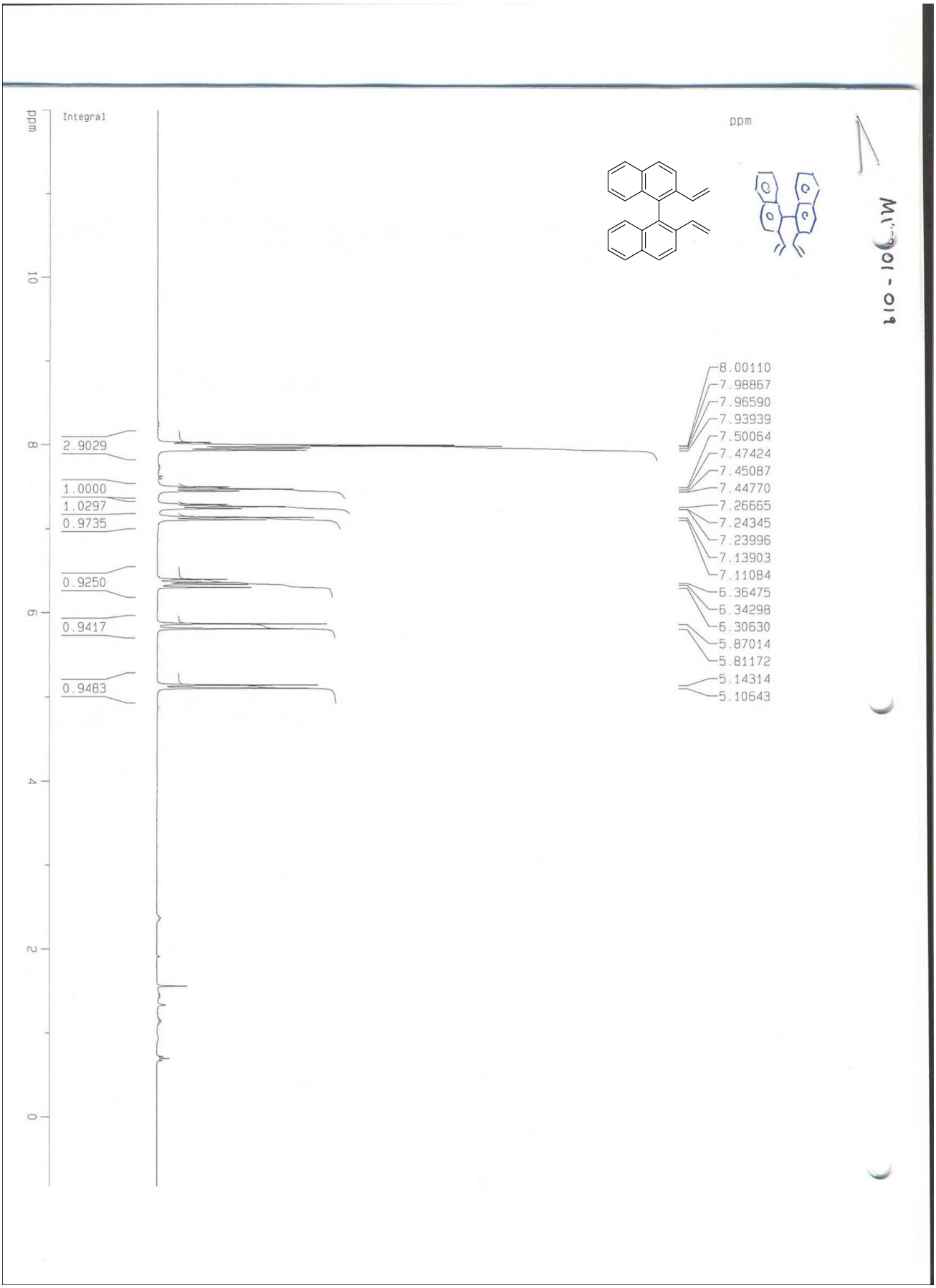
Helicene (14): Purification by flash column silica gel chromatography (30:1 Hex's–EtOAc) afforded 8.0 mg of **14** (55% brsm, 45% conversion) as an off-white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.92 (d, J = 8.7 Hz, 2H), 8.88 (d, J = 8.2 Hz, 2H), 8.17 (d, J = 8.7 Hz, 2H), 8.14 (d, J = 9.2 Hz, 2H), 7.98 (s, 2H), 7.92 (d, J = 7.8 Hz, 2H), 7.73 (dt, J = 8.2, 1.3 Hz, 2H), 7.64 (dt, J = 8.0, 1.1 Hz, 2H), 7.55 (d,

J = 9.1, Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 133.1, 132.8, 131.2, 130.2, 129.1, 129.4, 128.9, 128., 127.5 (2 C's overlap), 127.4 (2 C's overlap), 125.8, 124.3, 123.1,; HRMS (ESI$^+$) *m/z* 485.0447 (485.0439 calcd for $C_{30}H_{18}Ag$ $[M+Ag]^+$).

[6]–helicene (16):[15] Purification by flash column silica gel chromatography (30:1 Hex's–EtOAc) afforded [6]–helicene (70%) as an off–white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 2.7 Hz, 4H), 7.92 (d, *J* = 1.5 Hz, 4H), 7.82 (dd, *J* = 8.1, 0.9 Hz, 2H), 7.58 (d, *J* = 8.6 Hz, 2H), 7.20 (ddd, *J* = 14.9, 7.47 , 1.1 Hz, 2H), 6.66 (ddd, J = 10.8, 7.8, 1.4 Hz, 2H; ^{13}C NMR (75 MHz, $CDCl_3$) δ 133.9, 132.6, 132.1, 130.7, 128.8, 128.7, 128.5, 128.4, 128.1, 127.7, 127.0, 126.4, 125.5; HRMS (ESI$^+$) *m/z* 329.1335 (329.13247 calcd for $C_{26}H_{17}$ $[M+H]^+$).

[15] F. Teply, I. G. Stara, I. Stary, Ivo; A. Kollarovic, D. Saman, L. Rulisek, P. Fiedler, *J. Am. Chem. Soc.* **2002**, *124*, 9175–9180.

Spectra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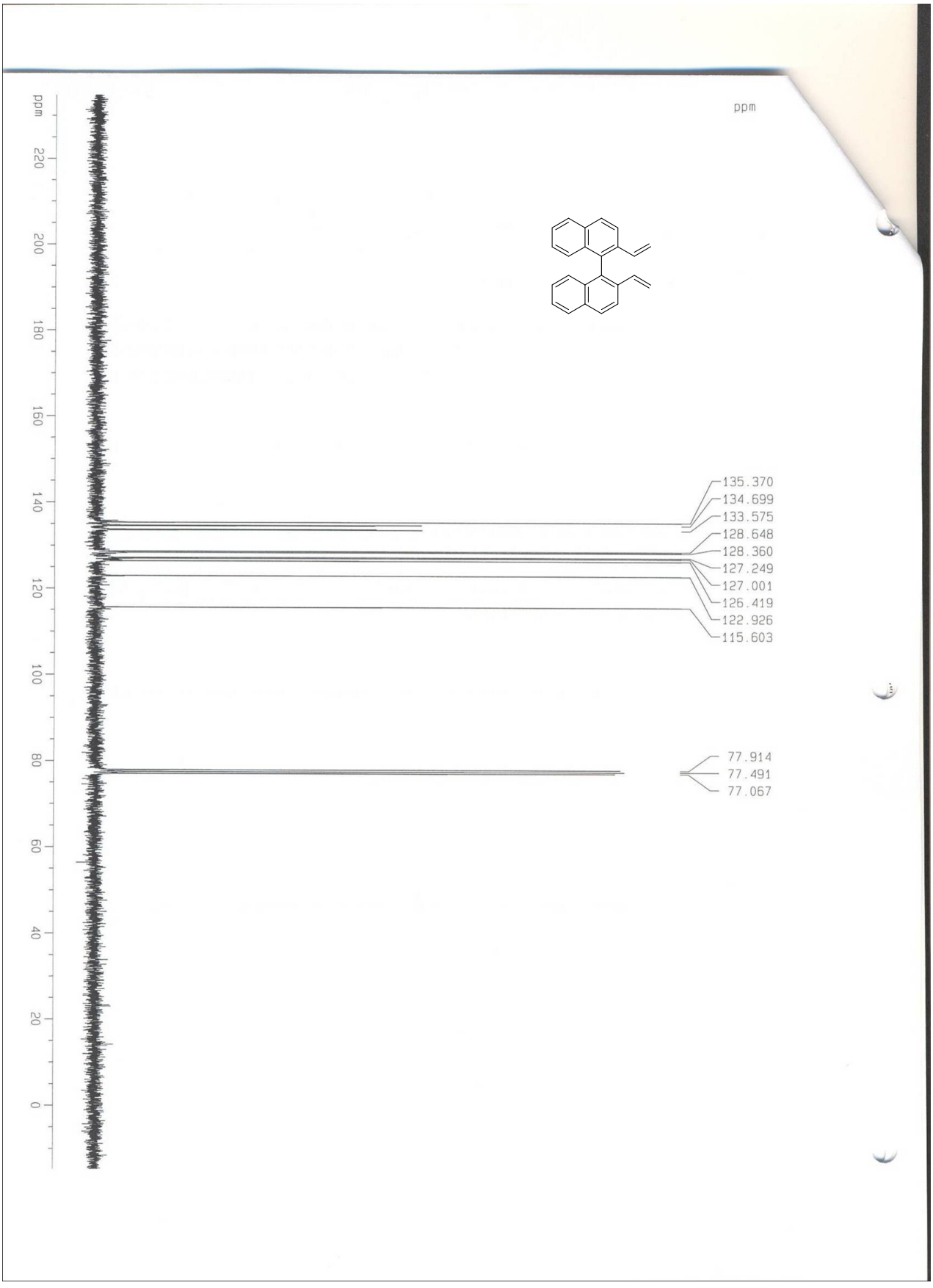
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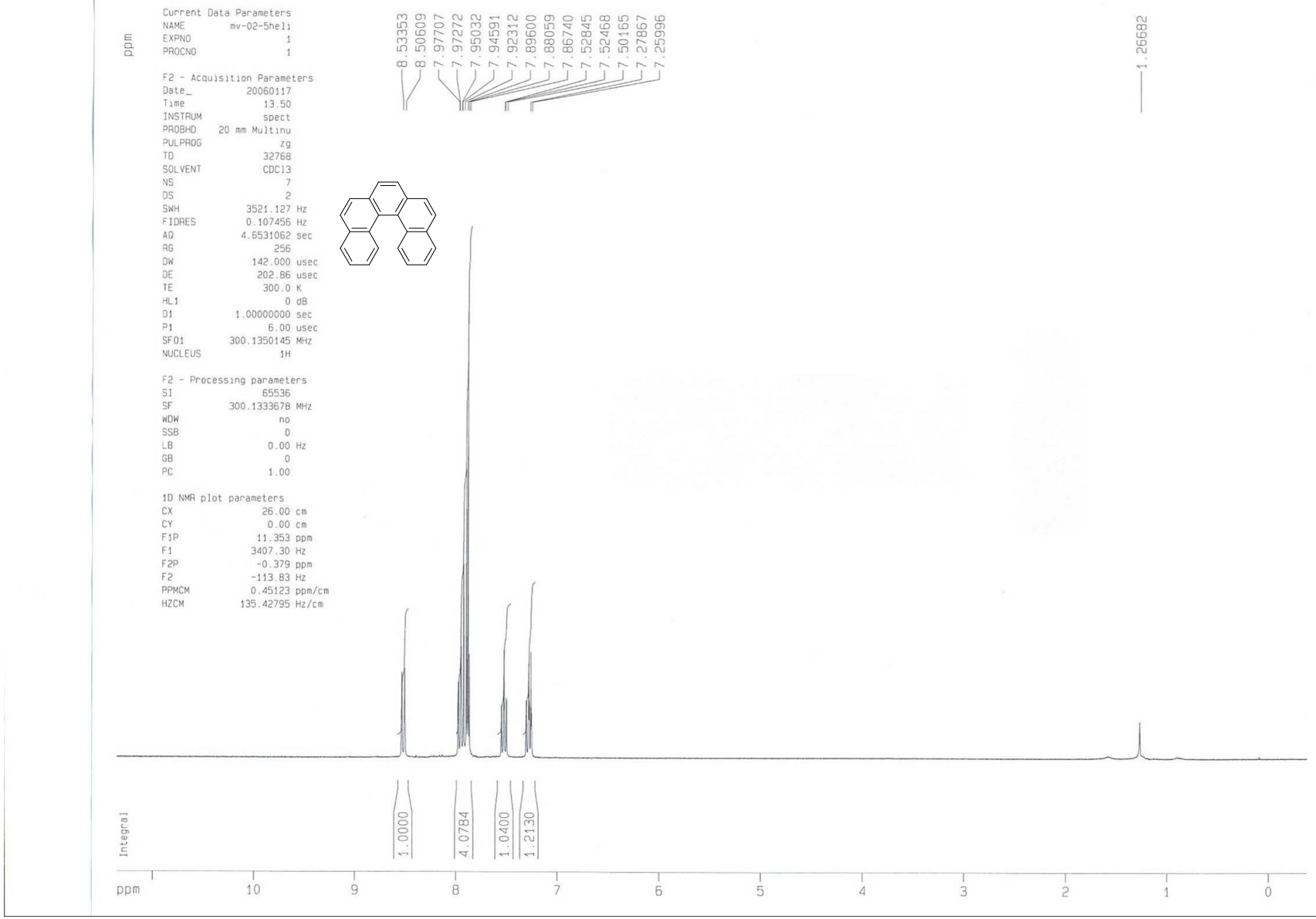
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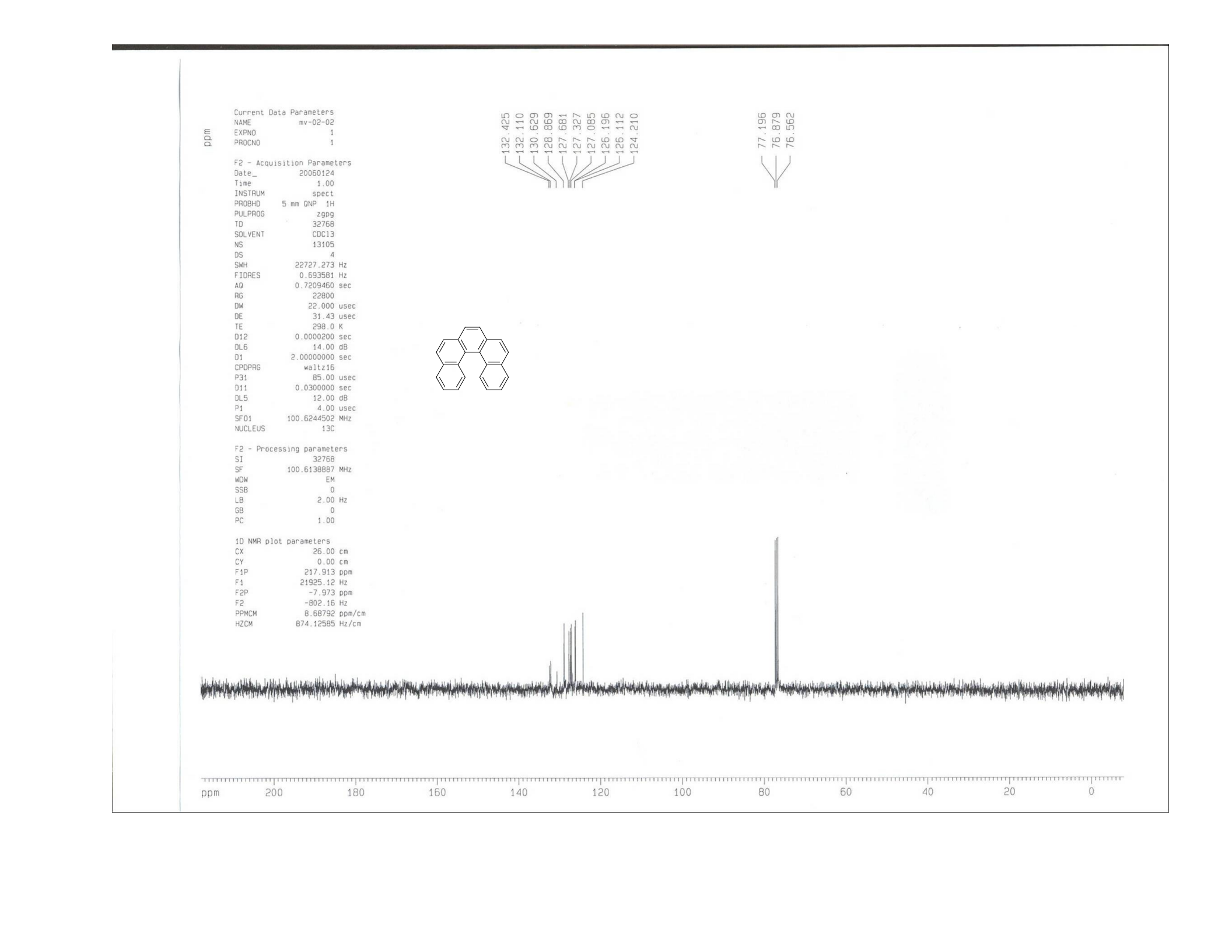
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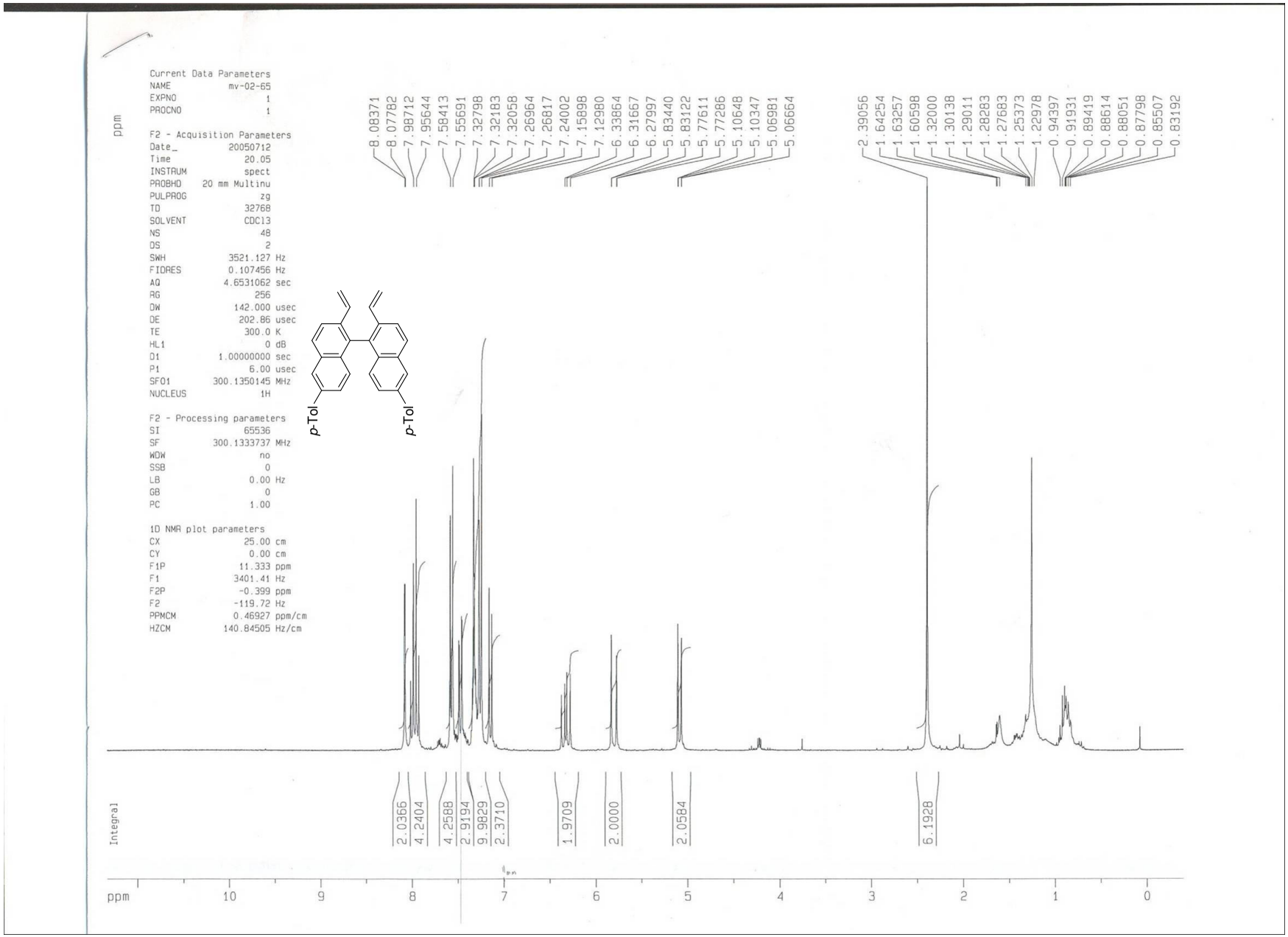
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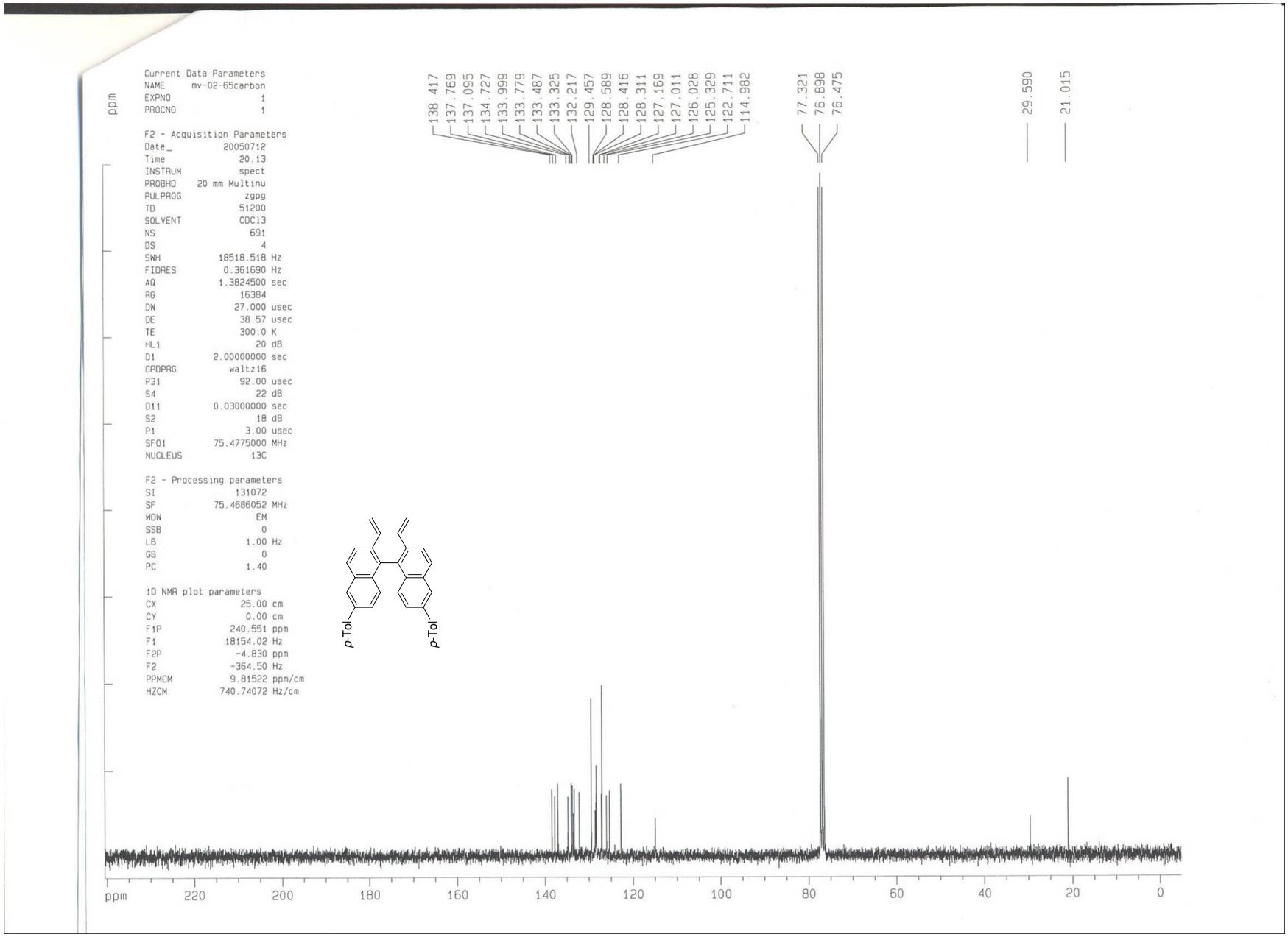
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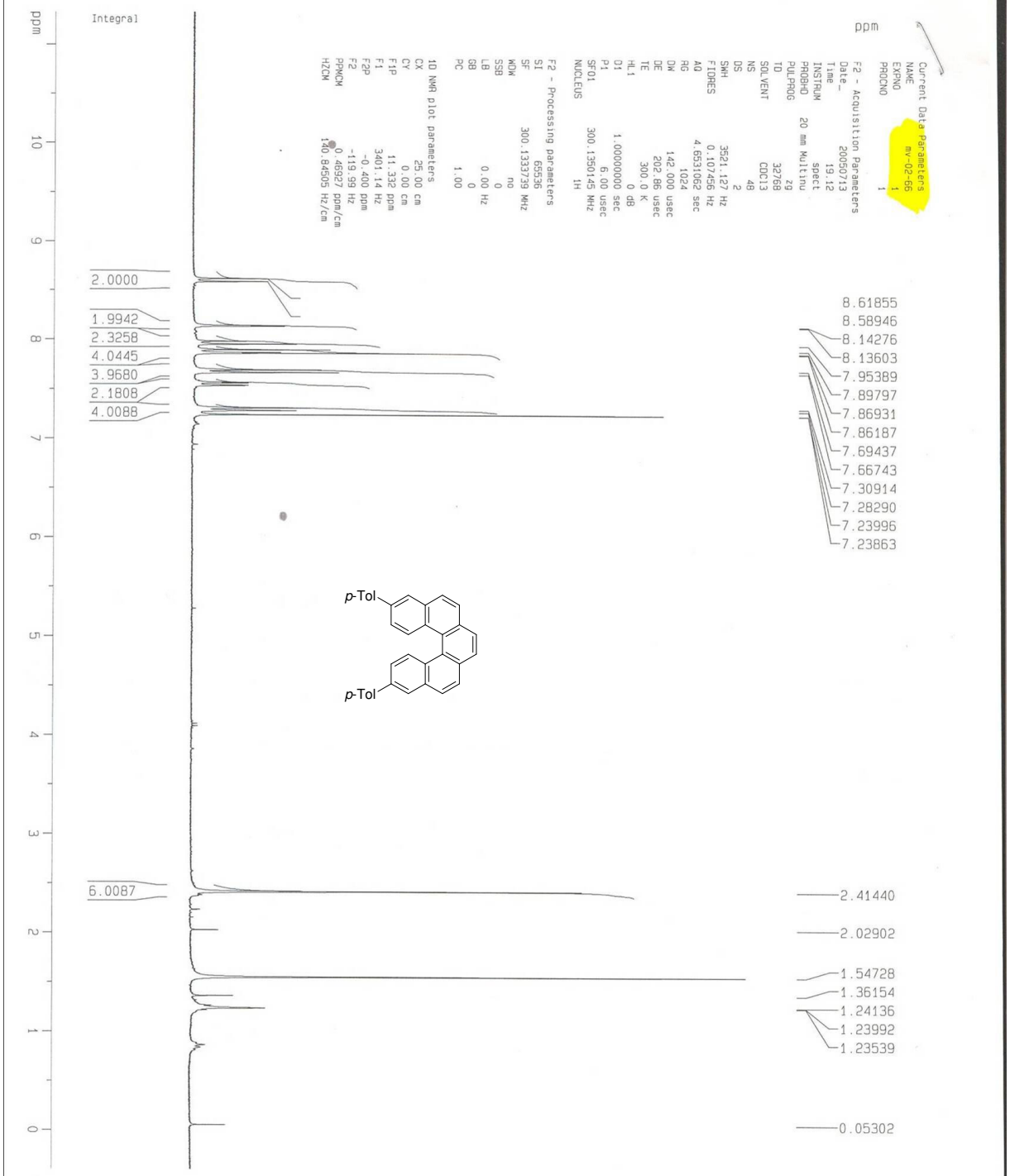
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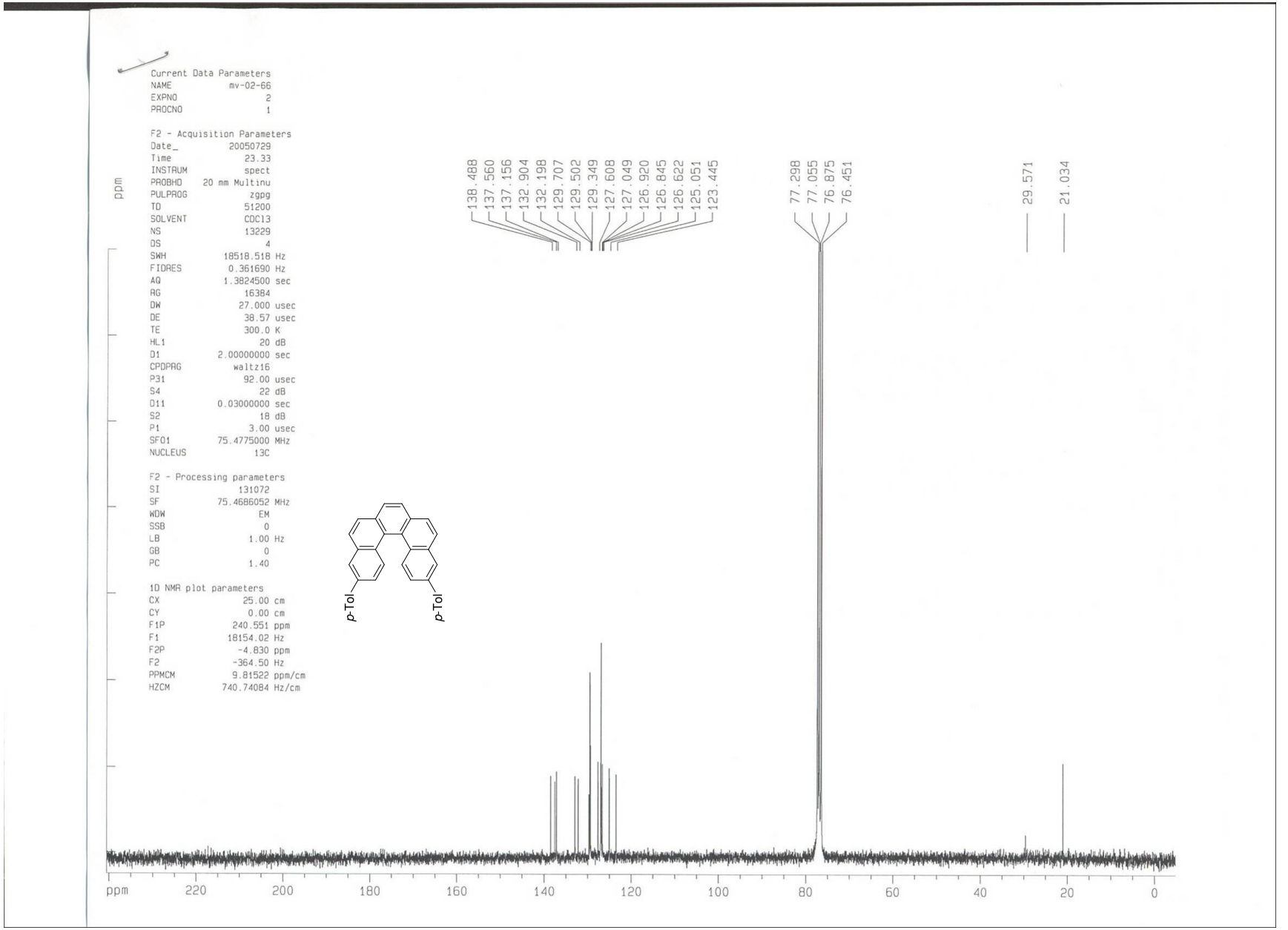
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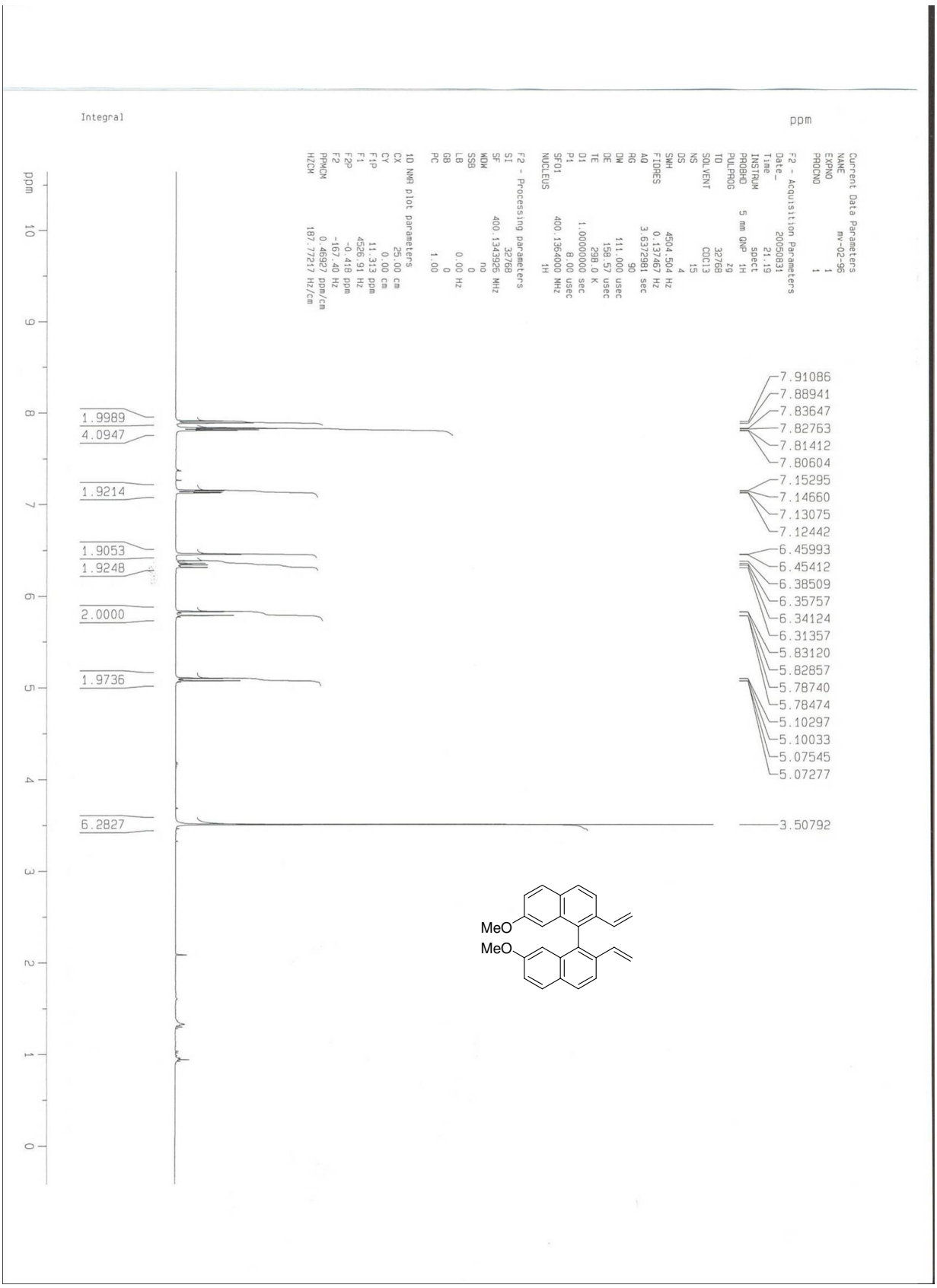
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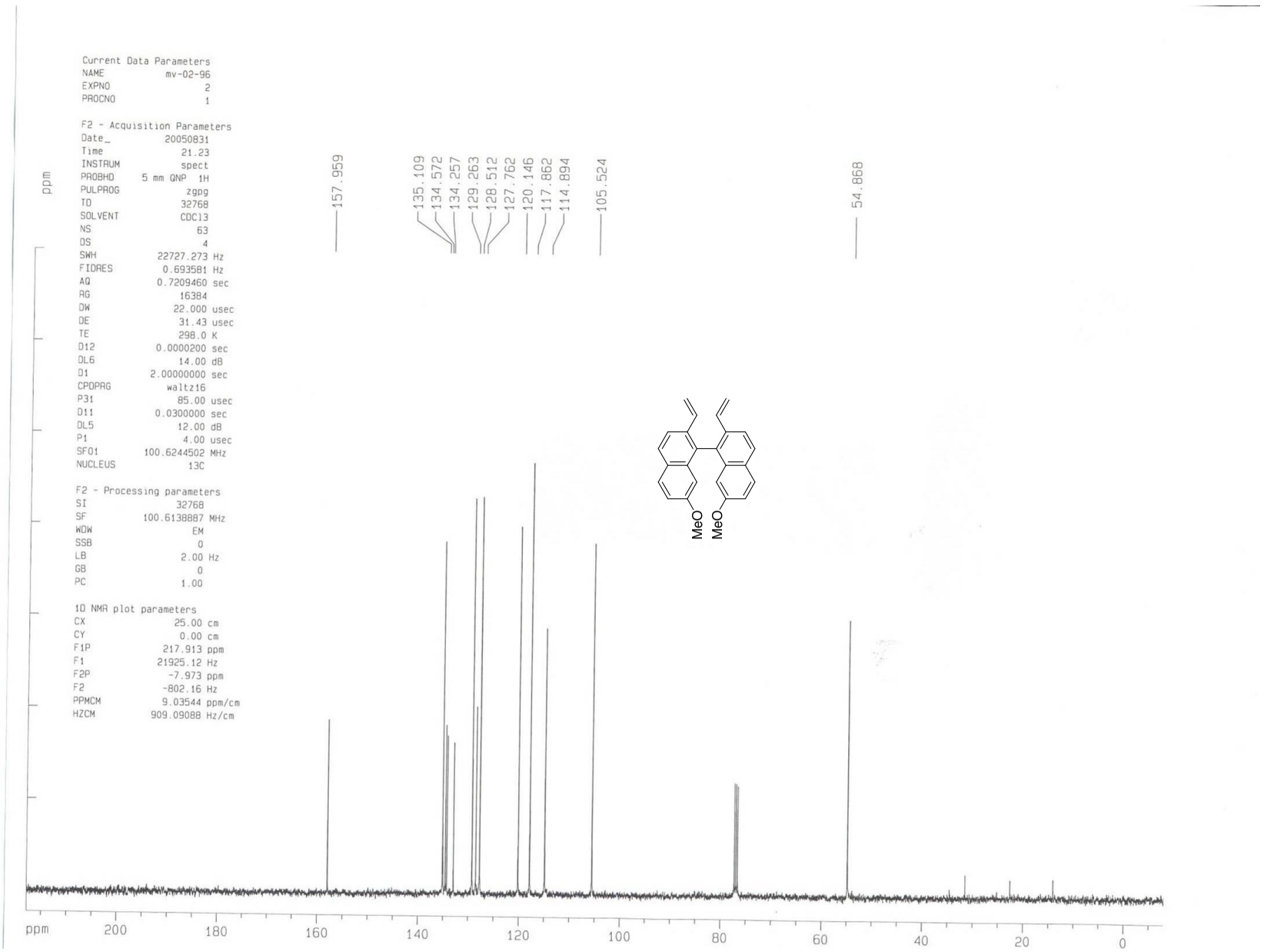
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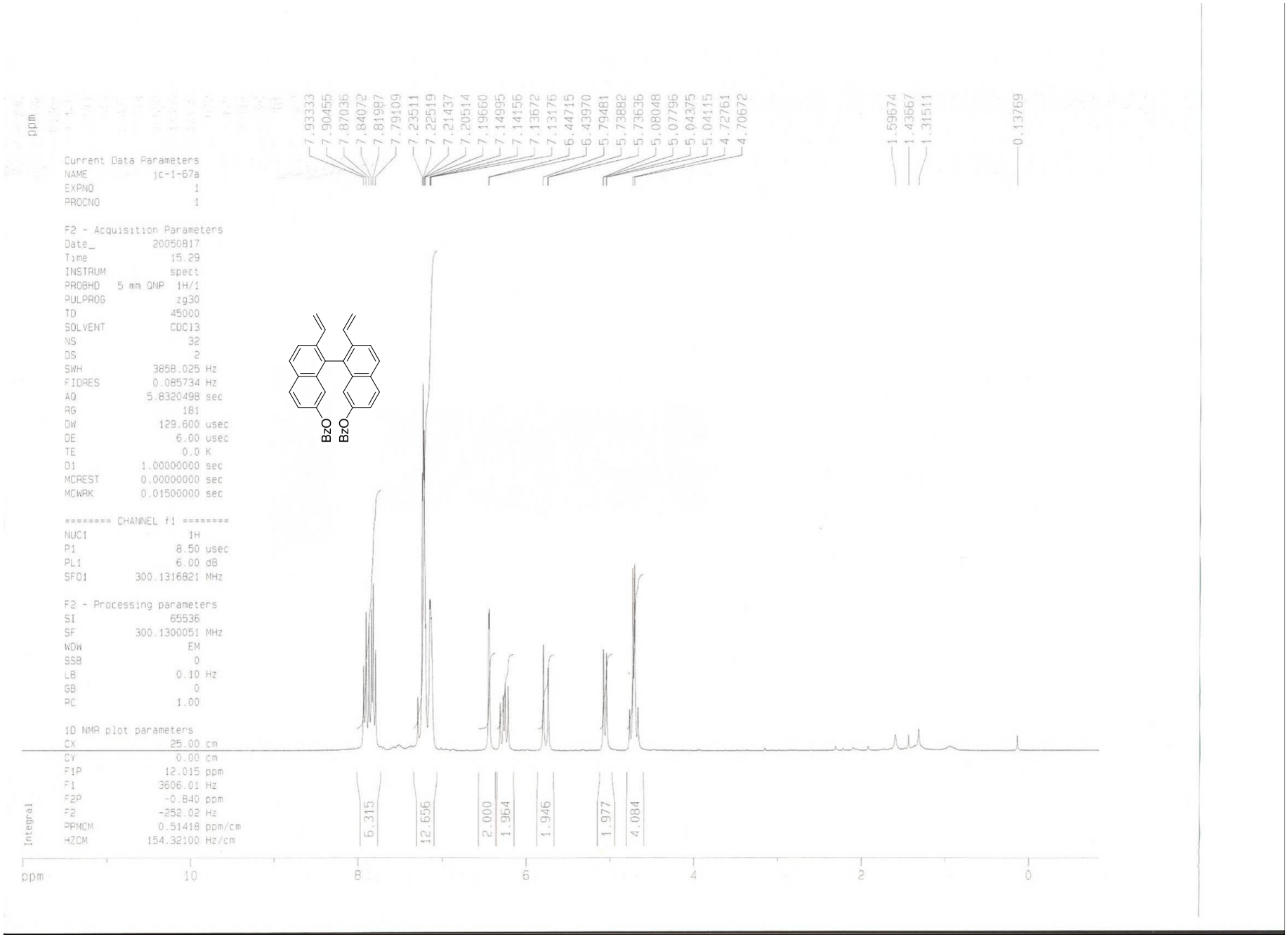
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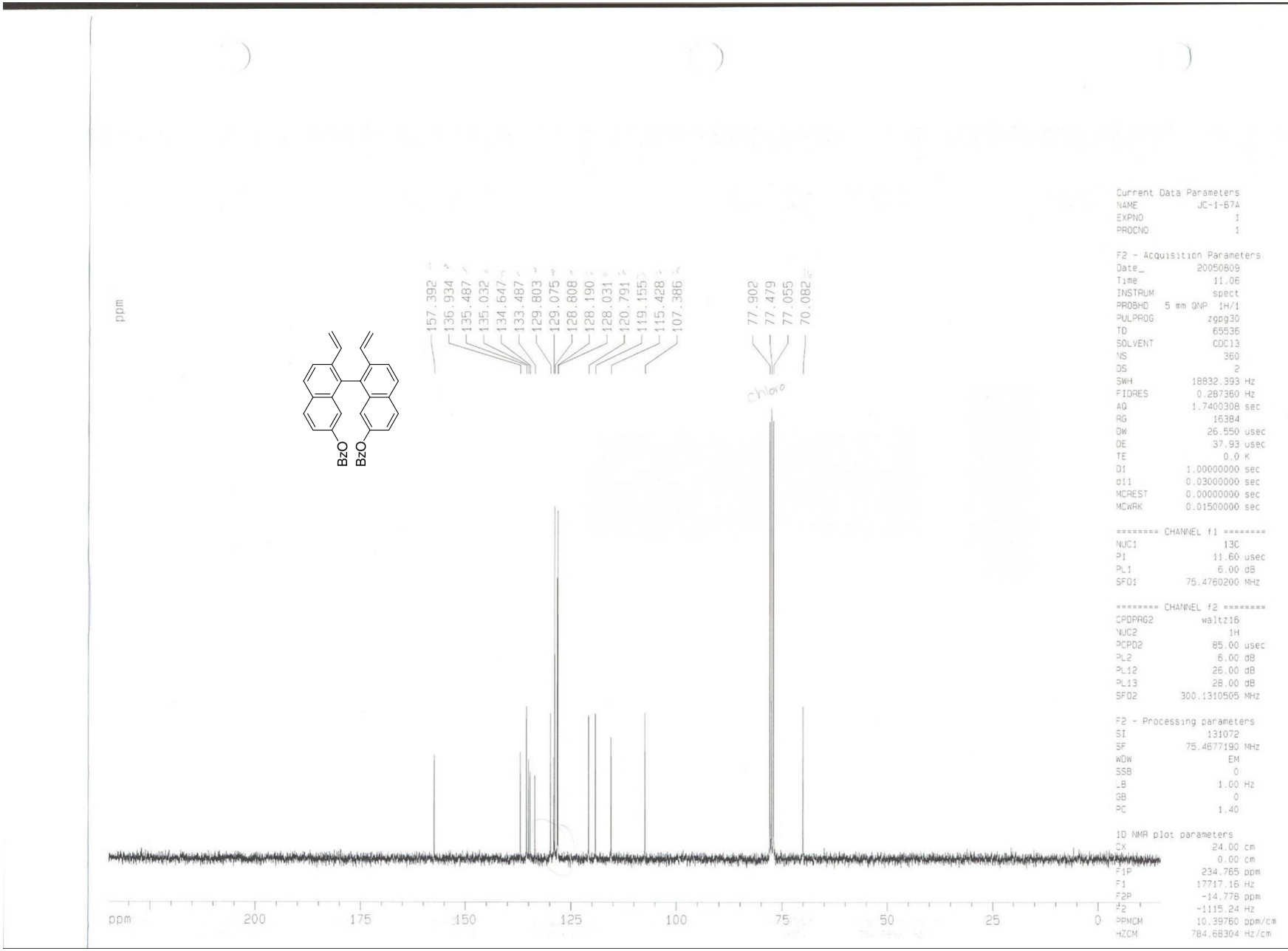
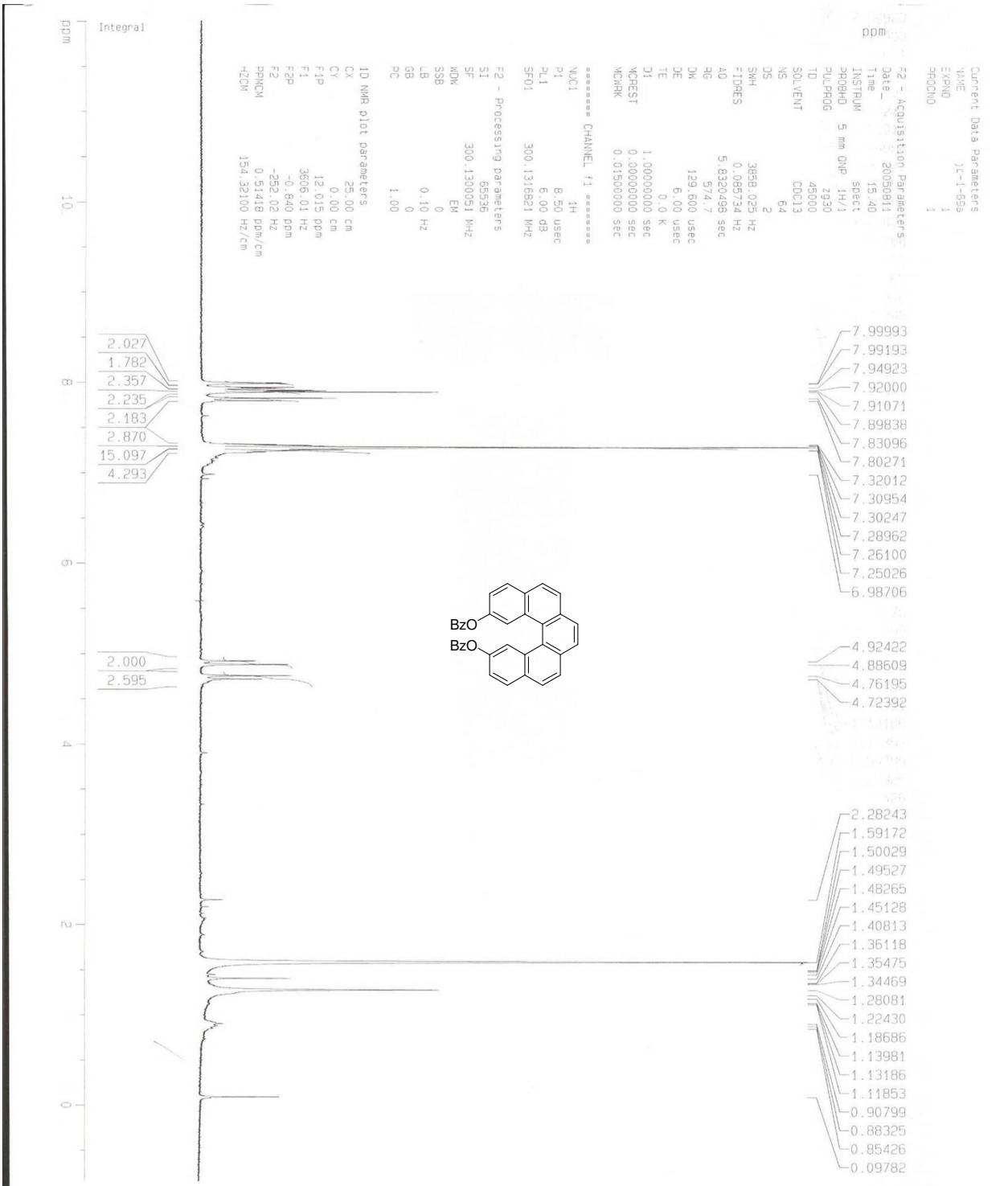
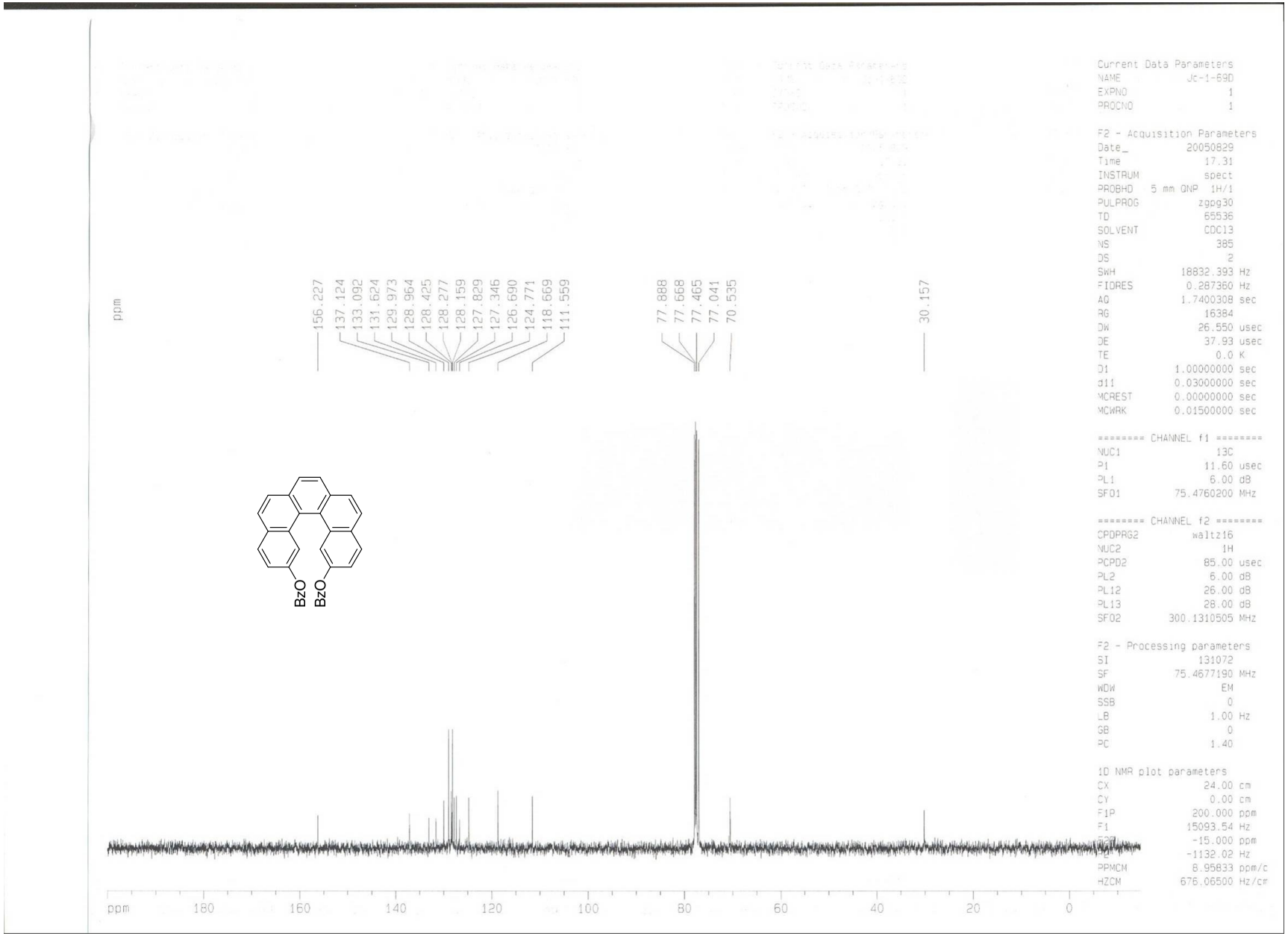
4.72761
4.70672
1.59674
1.43667
1.31511
0.13769
Current Data Parameters
NAME jc-1-67a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050817
Time 15.29
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 45000
SOLVENT CDCl3
NS 32
DS 2
SWH 3858.025 Hz
FIDRES 0.085734 Hz
AQ 5.8320498 sec
RG 161
DW 129.600 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 6.00 dB
SFO1 300.1316821 MHz
F2 - Processing parameters
SI 65536
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 12.015 ppm
F1 3606.01 Hz
F2P -0.840 ppm
F2 -252.02 Hz
PPMCM 0.51418 ppm/cm
HZCM 154.32100 Hz/cm
BzO
BzO
Integral
6.315
12.656
2.000
1.964
1.946
1.977
4.084
ppm
10
8
6
4
2
0


ppm
157.392
136.934
135.487
135.032
134.647
133.487
129.803
129.075
128.808
128.190
128.031
120.791
119.155
115.428
107.386
77.902
77.479
77.055
70.082
chloro
BzO
BzO
ppm
200
175
150
125
100
75
50
25
0
Current Data Parameters
NAME JC-1-67A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050809
Time 11.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 360
DS 2
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 16384
DW 26.550 usec
DE 37.93 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 11.60 usec
PL1 6.00 dB
SFO1 75.4760200 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 6.00 dB
PL12 26.00 dB
PL13 28.00 dB
SFO2 300.1310505 MHz
F2 - Processing parameters
SI 131072
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
CY 0.00 cm
F1P 234.765 ppm
F1 17717.16 Hz
F2P -14.778 ppm
F2 -1115.24 Hz
PPMCM 10.39760 ppm/cm
HZCM 784.68304 Hz/cm


Integral
ppm
2.027
1.782
2.357
2.235
2.183
2.870
15.097
4.293
2.000
2.595
BzO
BzO
7.99993
7.99193
7.94923
7.92000
7.91071
7.89838
7.83096
7.80271
7.32012
7.30954
7.30247
7.28962
7.26100
7.25026
6.98706
4.92422
4.88609
4.76195
4.72392
2.28243
1.59172
1.50029
1.49527
1.48265
1.45128
1.40813
1.36118
1.35475
1.34469
1.28081
1.22430
1.18686
1.13981
1.13186
1.11853
0.90799
0.88325
0.85426
0.09782



Current Data Parameters
NAME Jc-1-69D
EXPNO 1
PROCNO 1

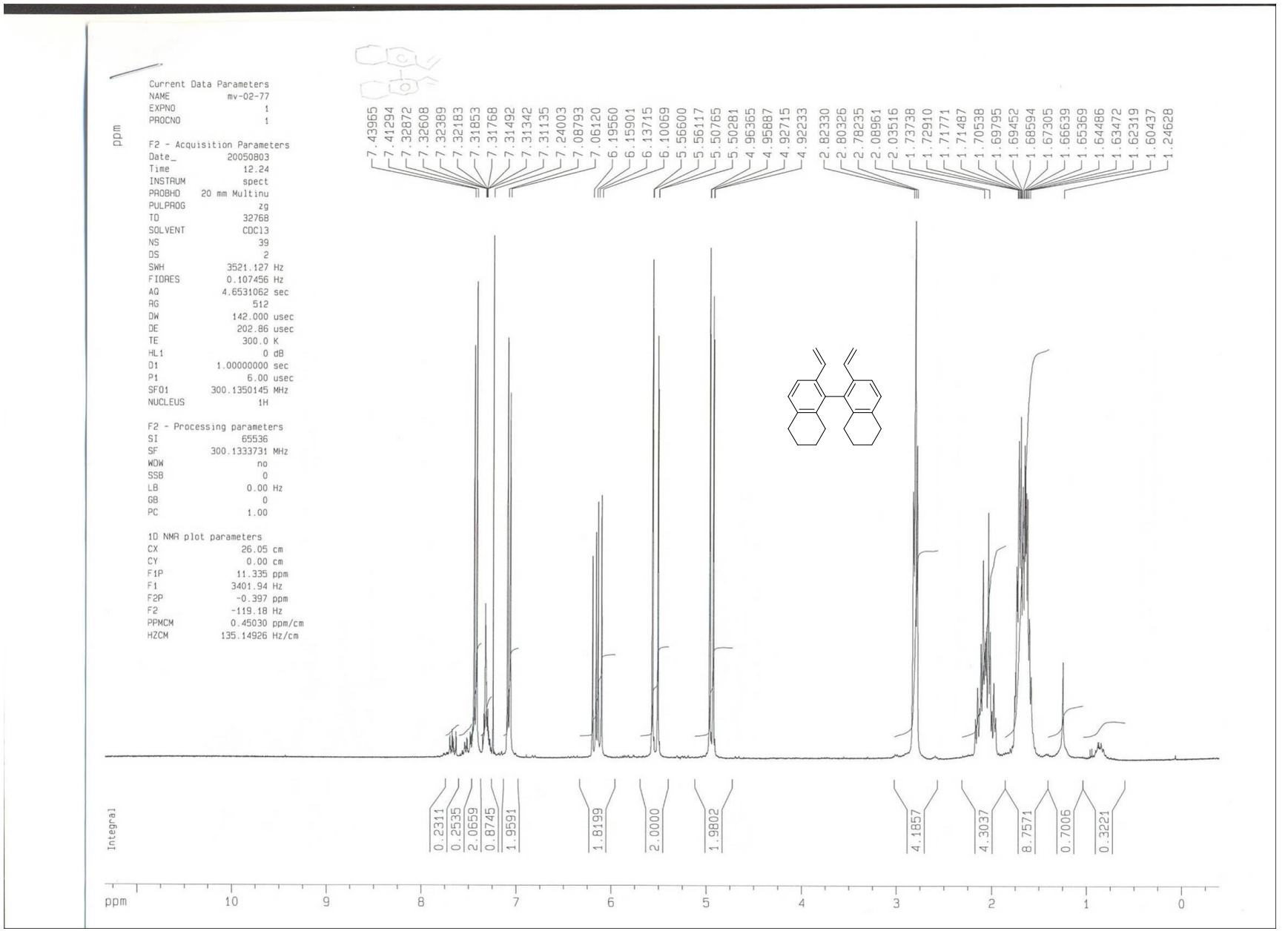
F2 - Acquisition Parameters
Date_ 20050829
Time 17.31
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 385
DS 2
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 16384
DW 26.550 usec
DE 37.93 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

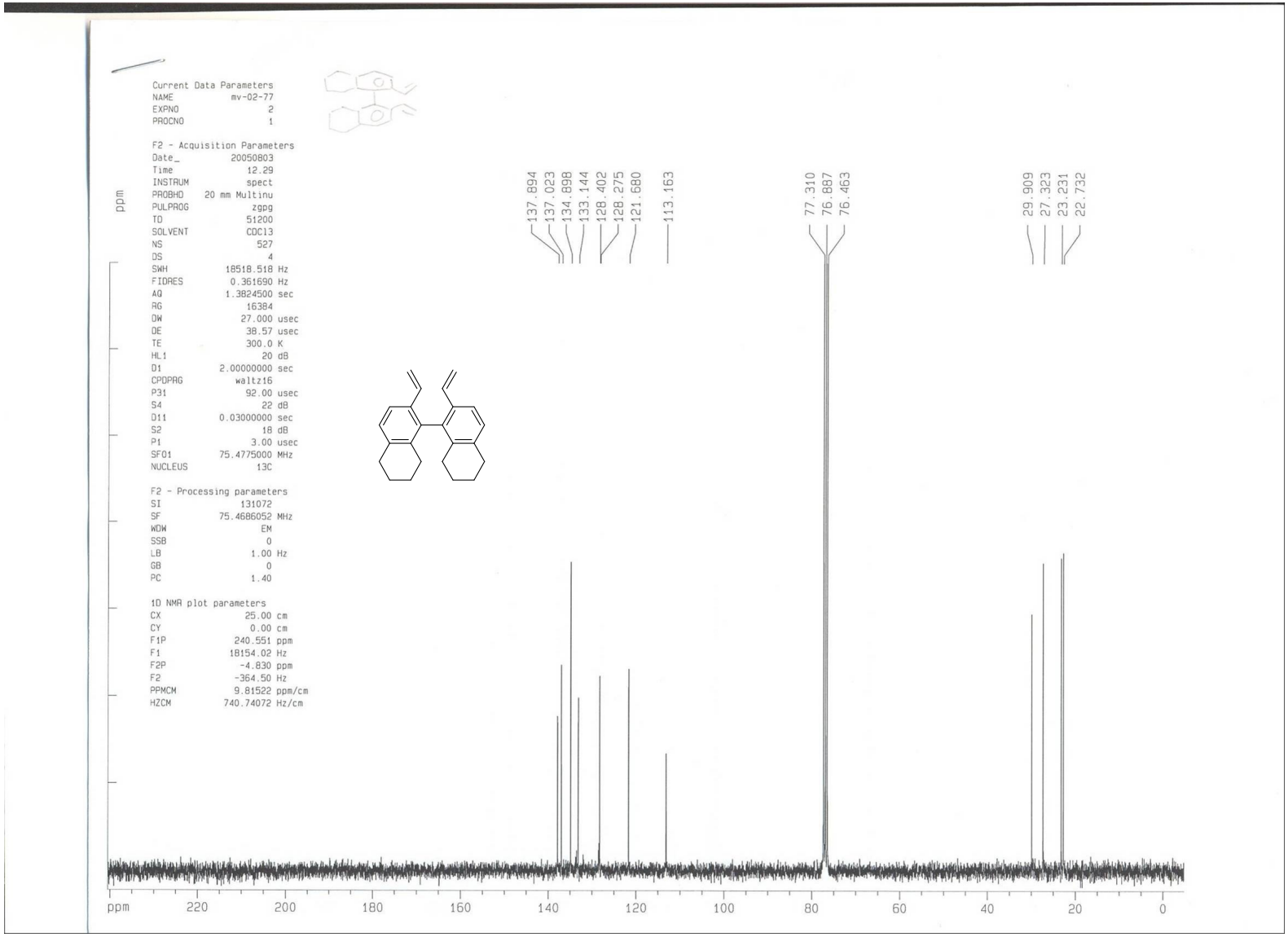
======== CHANNEL f1 ========
NUC1 13C
P1 11.60 usec
PL1 6.00 dB
SF01 75.4760200 MHz

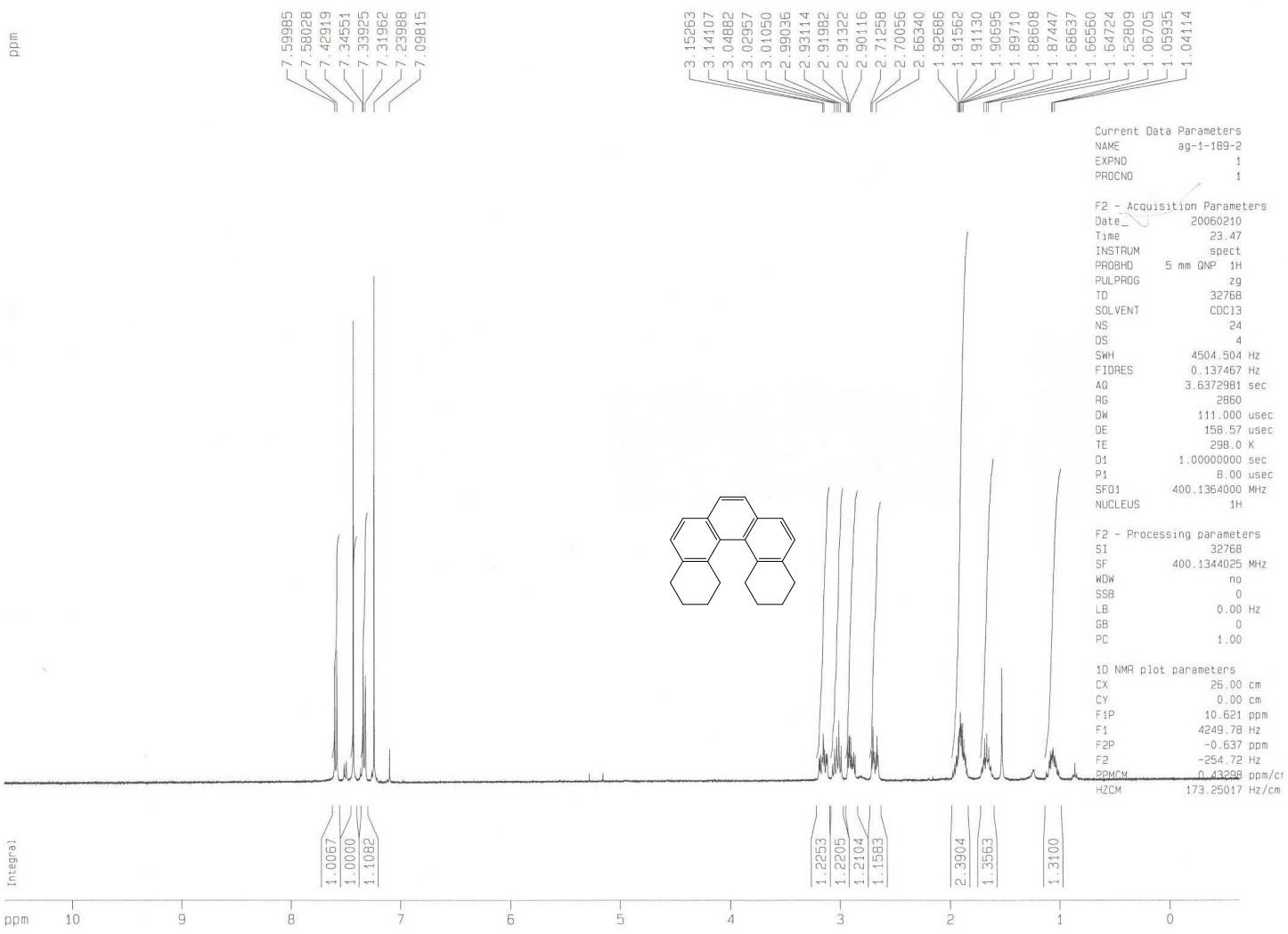
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 6.00 dB
PL12 26.00 dB
PL13 28.00 dB
SF02 300.1310505 MHz

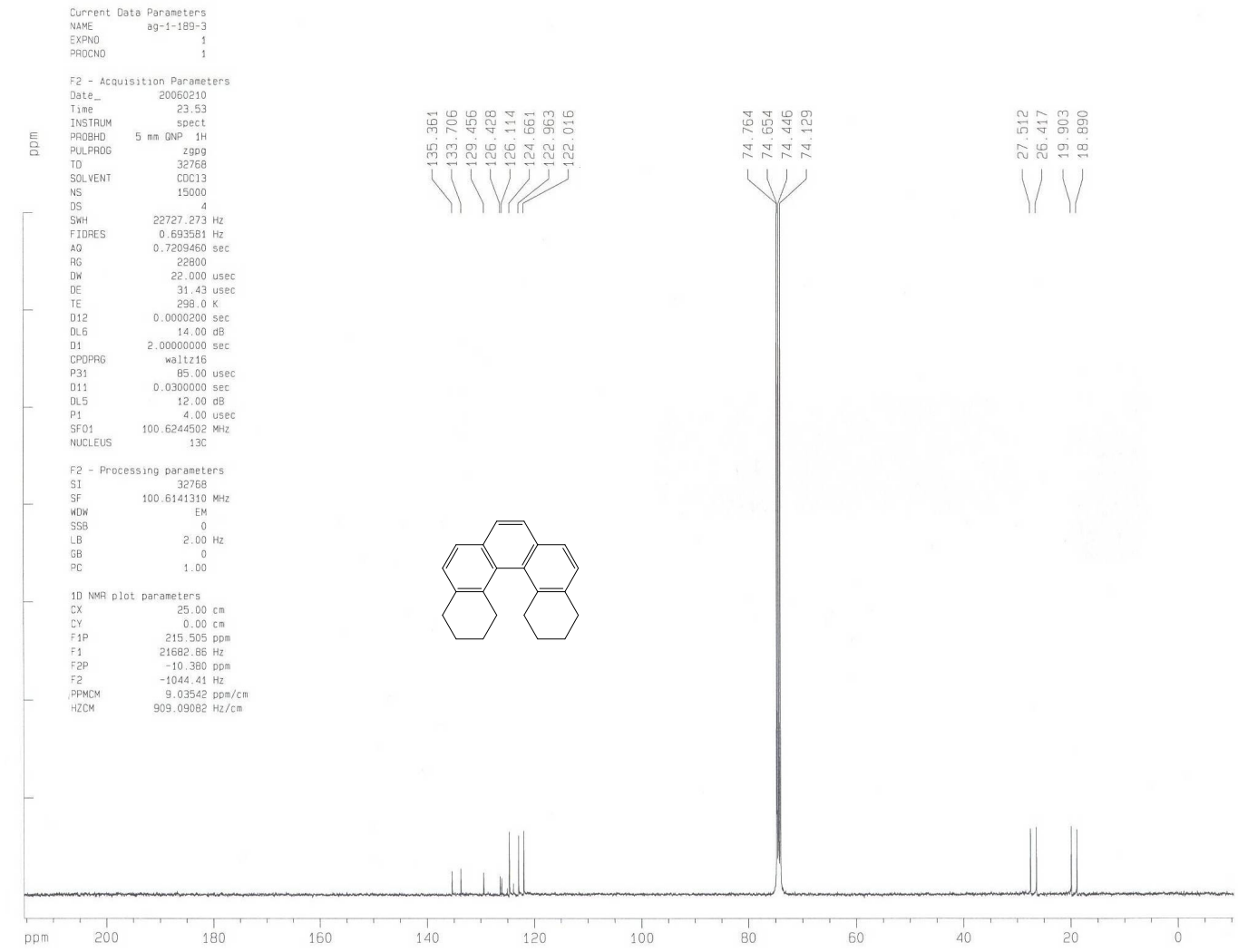
F2 - Processing parameters
SI 131072
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

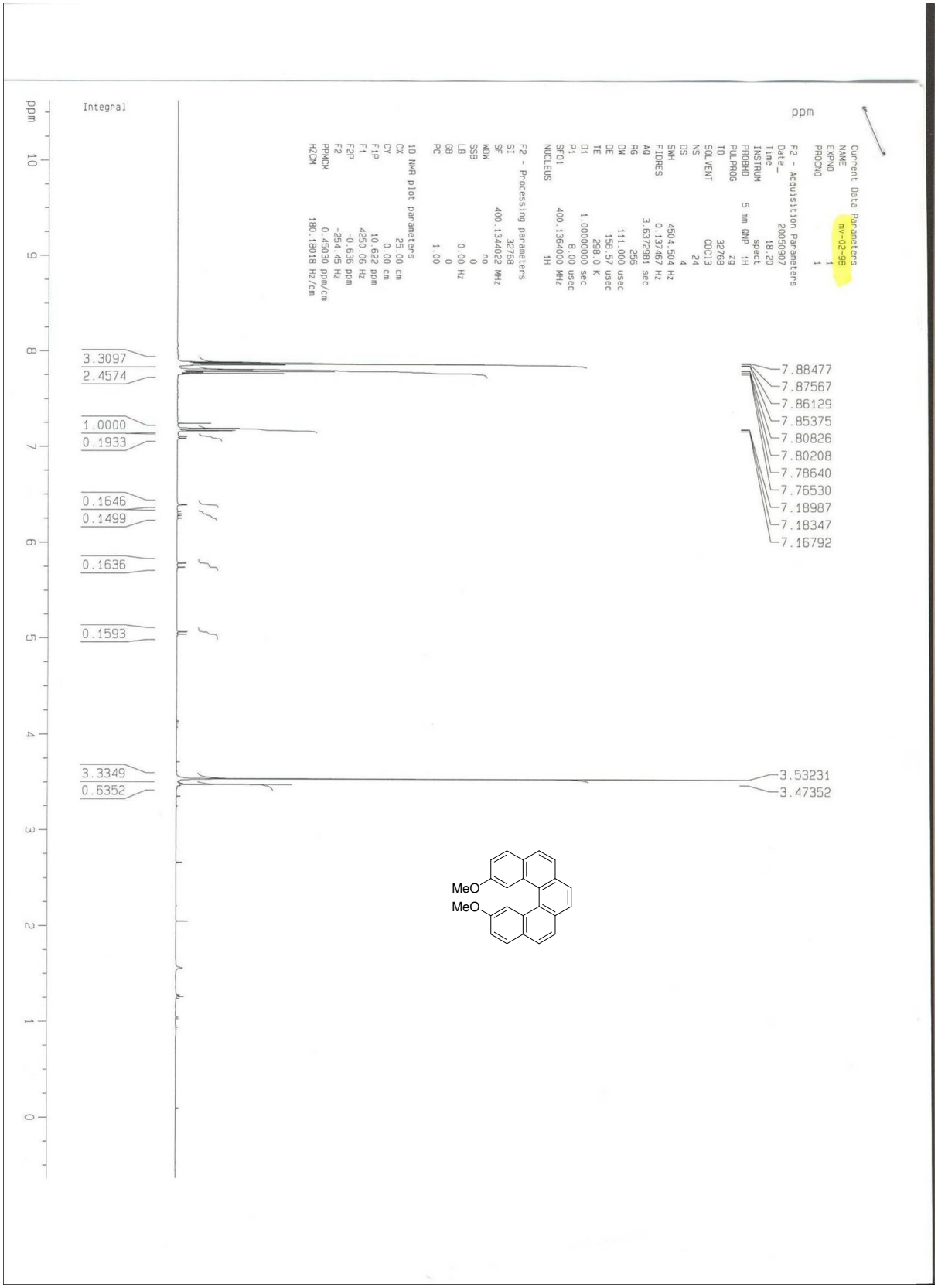
1D NMR plot parameters
CX 24.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 15093.54 Hz
F2P -15.000 ppm
F2 -1132.02 Hz
PPMCM 8.95833 ppm/c
HZCM 676.06500 Hz/cm

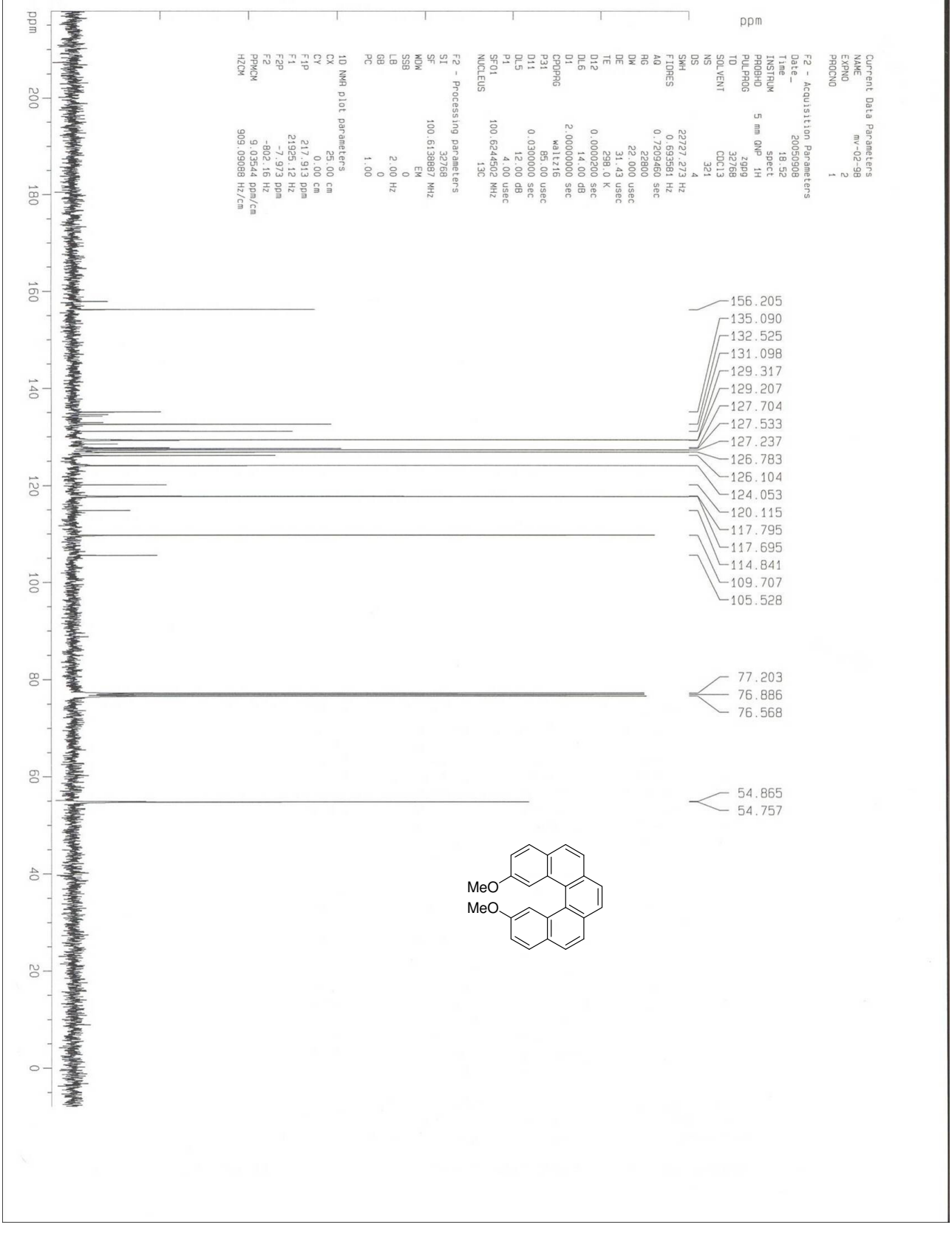

Current Data Parameters
NAME mv-02-77
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050803
Time 12.24
INSTRUM spect
PROBHD 20 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 39
DS 2
SWH 3521.127 Hz
FIDRES 0.107456 Hz
AQ 4.6531062 sec
RG 512
DW 142.000 usec
DE 202.86 usec
TE 300.0 K
HL1 0 dB
D1 1.00000000 sec
P1 6.00 usec
SF01 300.1350145 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 65536
SF 300.1333731 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 26.05 cm
CY 0.00 cm
F1P 11.335 ppm
F1 3401.94 Hz
F2P -0.397 ppm
F2 -119.18 Hz
PPMCM 0.45030 ppm/cm
HZCM 135.14926 Hz/cm
ppm
7.43965
7.41294
7.32872
7.32608
7.32389
7.32183
7.31853
7.31768
7.31492
7.31342
7.31135
7.24003
7.08793
7.06120
6.19560
6.15901
6.13715
6.10069
5.56600
5.56117
5.50765
5.50281
4.96365
4.95887
4.92715
4.92233
2.82330
2.80326
2.78235
2.08961
2.03516
1.73738
1.72910
1.71771
1.71487
1.70538
1.69795
1.69452
1.68594
1.67305
1.66639
1.65369
1.64486
1.63472
1.62319
1.60437
1.24628
Integral
0.2311
0.2535
2.0659
0.8745
1.9591
1.8199
2.0000
1.9802
4.1857
4.3037
8.7571
0.7006
0.3221
ppm
10
9
8
7
6
5
4
3
2
1
0

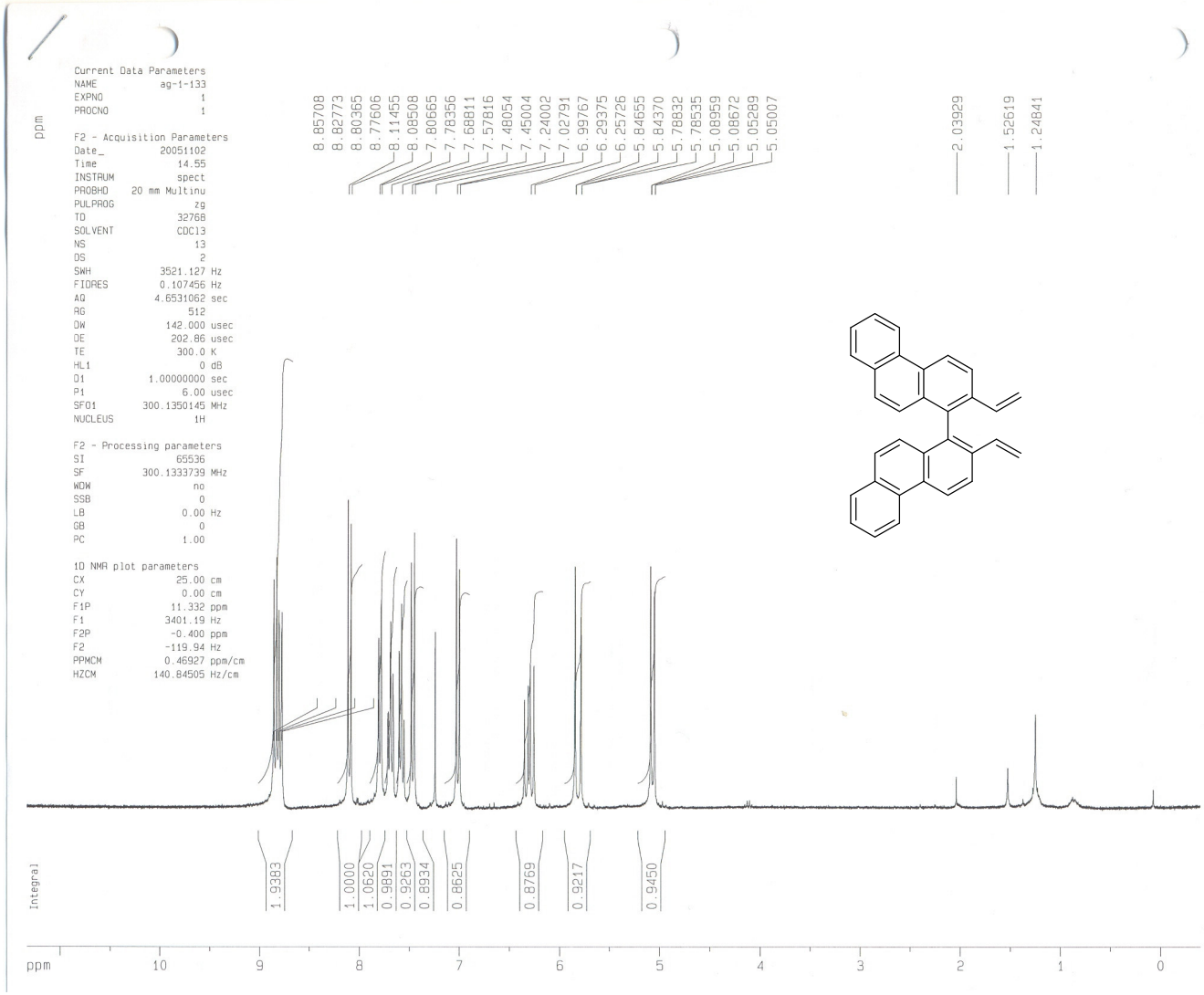

Current Data Parameters
NAME mv-02-77
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050803
Time 12.29
INSTRUM spect
PROBHD 20 mm Multinu
PULPROG zgpg
TD 51200
SOLVENT CDCl3
NS 527
DS 4
SWH 18518.518 Hz
FIDRES 0.361690 Hz
AQ 1.3824500 sec
RG 16384
DW 27.000 usec
DE 38.57 usec
TE 300.0 K
HL1 20 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 92.00 usec
S4 22 dB
D11 0.03000000 sec
S2 18 dB
P1 3.00 usec
SF01 75.4775000 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 131072
SF 75.4686052 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 240.551 ppm
F1 18154.02 Hz
F2P -4.830 ppm
F2 -364.50 Hz
PPMCM 9.81522 ppm/cm
HZCM 740.74072 Hz/cm
ppm
137.894
137.023
134.898
133.144
128.402
128.275
121.680
113.163
77.310
76.887
76.463
29.909
27.323
23.231
22.732
ppm
220
200
180
160
140
120
100
80
60
40
20
0

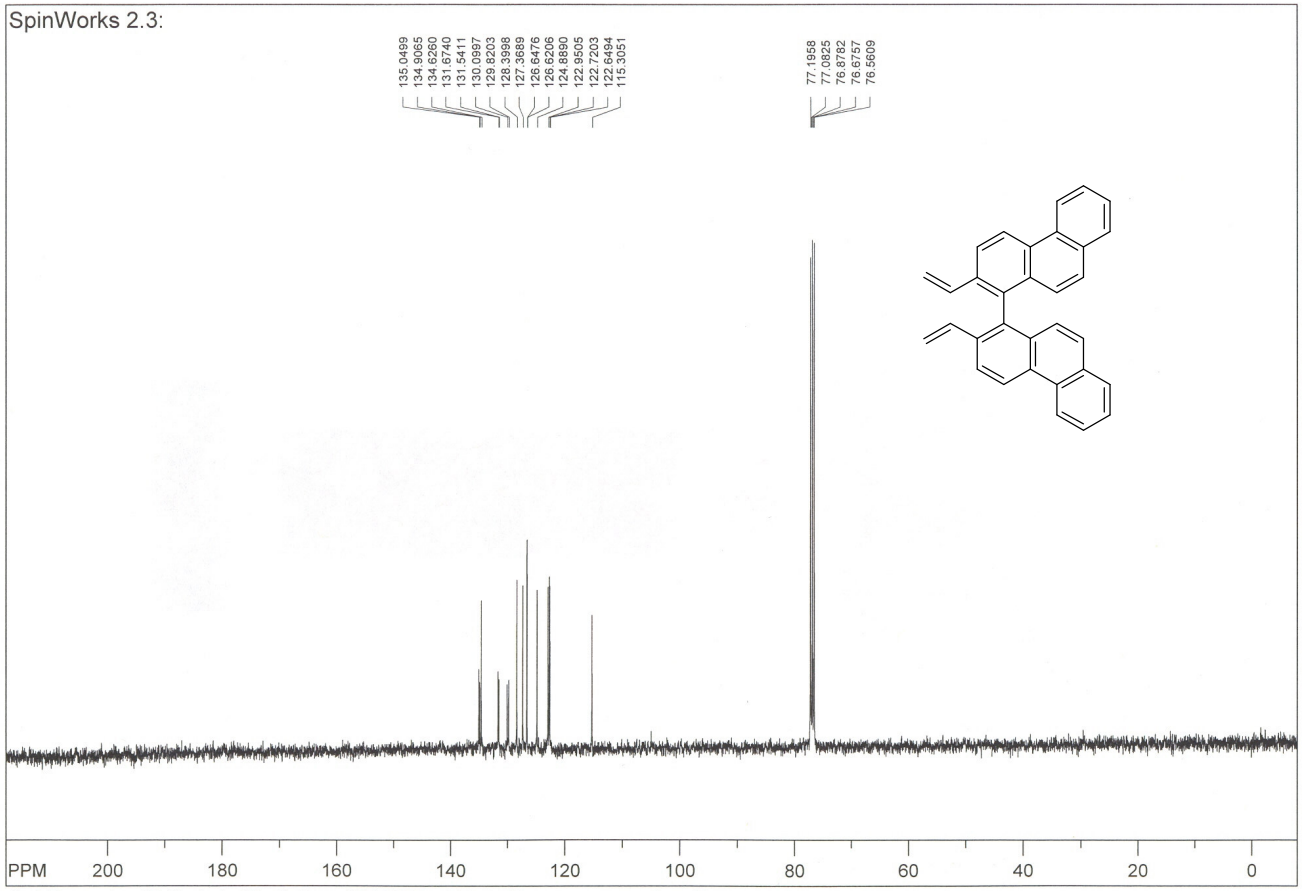

ppm
7.59985
7.58028
7.42919
7.34551
7.33925
7.31962
7.23988
7.09815
3.15263
3.14107
3.04882
3.02957
3.01050
2.99036
2.93114
2.91982
2.91322
2.90116
2.71258
2.70056
2.66340
1.92686
1.91562
1.91130
1.90695
1.89710
1.88608
1.87447
1.68637
1.66560
1.64724
1.52809
1.06705
1.05935
1.04114
Current Data Parameters
NAME ag-1-189-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060210
Time 23.47
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 24
DS 4
SWH 4504.504 Hz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 2860
DW 111.000 usec
DE 158.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 8.00 usec
SFO1 400.1364000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1344025 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 26.00 cm
CY 0.00 cm
F1P 10.621 ppm
F1 4249.78 Hz
F2P -0.637 ppm
F2 -254.72 Hz
PPMCM 0.43298 ppm/cm
HZCM 173.25017 Hz/cm
integral
1.0067
1.0000
1.1082
1.2253
1.2205
1.2104
1.1583
2.3904
1.3563
1.3100
ppm
10
9
8
7
6
5
4
3
2
1
0


Current Data Parameters
NAME ag-1-189-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060210
Time 23.53
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 15000
DS 4
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 22800
DW 22.000 usec
DE 31.43 usec
TE 298.0 K
D12 0.0000200 sec
DL6 14.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 85.00 usec
D11 0.0300000 sec
DL5 12.00 dB
P1 4.00 usec
SF01 100.6244502 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 32768
SF 100.6141310 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 215.505 ppm
F1 21682.86 Hz
F2P -10.380 ppm
F2 -1044.41 Hz
PPMCM 9.03542 ppm/cm
HZCM 909.09082 Hz/cm
ppm
135.361
133.706
129.456
126.428
126.114
124.661
122.963
122.016
74.764
74.654
74.446
74.129
27.512
26.417
19.903
18.890
ppm
200
180
160
140
120
100
80
60
40
20
0


Current Data Parameters
NAME mv-02-98
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050907
Time 18.20
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 24
DS 4
SWH 4504.504 Hz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 256
DW 111.000 usec
DE 159.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 8.00 usec
SFO1 400.1354000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1344022 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 10.622 ppm
F1 4250.06 Hz
F2P -0.635 ppm
F2 -254.45 Hz
PPMCM 0.45030 ppm/cm
HZCM 180.18018 Hz/cm
ppm
Integral
3.3097
2.4574
1.0000
0.1933
0.1646
0.1499
0.1636
0.1593
3.3349
0.6352
7.88477
7.87567
7.86129
7.85375
7.80826
7.80208
7.78640
7.76530
7.18987
7.18347
7.16792
3.53231
3.47352
MeO
MeO

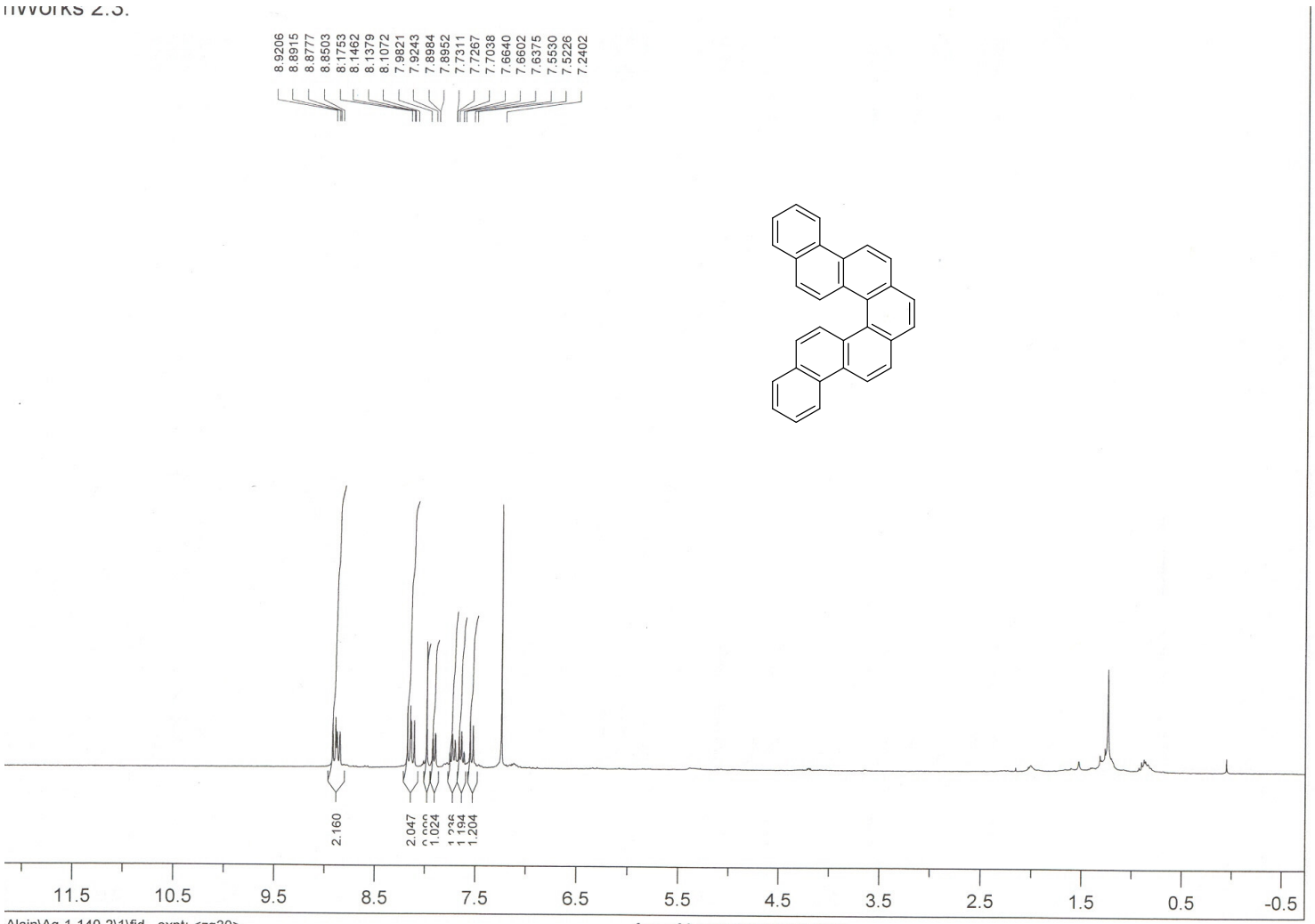

Current Data Parameters
NAME mv-02-98
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050908
Time 18.52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 321
DS 4
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 22800
DW 22.000 usec
DE 31.43 usec
TE 298.0 K
D12 0.0000200 sec
DL6 14.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 85.00 usec
D11 0.0300000 sec
DL5 12.00 dB
P1 4.00 usec
SFO1 100.6244502 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 32768
SF 100.6138887 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 217.913 ppm
F1 21925.12 Hz
F2P -7.973 ppm
F2 -802.16 Hz
PPMCM 9.03544 ppm/cm
HZCM 909.09088 Hz/cm
ppm
156.205
135.090
132.525
131.098
129.317
129.207
127.704
127.533
127.237
126.783
126.104
124.053
120.115
117.795
117.695
114.841
109.707
105.528
77.203
76.886
76.568
54.865
54.757
MeO
MeO


Current Data Parameters
NAME ag-1-133
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051102
Time 14.55
INSTRUM spect
PROBHD 20 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 13
DS 2
SWH 3521.127 Hz
FIDRES 0.107456 Hz
AQ 4.6531062 sec
RG 512
DW 142.000 usec
DE 202.86 usec
TE 300.0 K
HL1 0 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 300.1350145 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 65536
SF 300.1333739 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 11.332 ppm
F1 3401.19 Hz
F2P -0.400 ppm
F2 -119.94 Hz
PPMCM 0.46927 ppm/cm
HZCM 140.84505 Hz/cm
ppm
8.85708
8.82773
8.80365
8.77606
8.11455
8.08508
7.80665
7.78356
7.68811
7.57816
7.48054
7.45004
7.24002
7.02791
6.99767
6.29375
6.25726
5.84655
5.84370
5.78832
5.78535
5.08959
5.08672
5.05289
5.05007
2.03929
1.52619
1.24841
Integral
1.9383
1.0000
1.0620
0.9891
0.9263
0.8934
0.8625
0.8769
0.9217
0.9450
ppm
10
9
8
7
6
5
4
3
2
1
0



file: C:\Documents and Settings\p-track\Desktop\groupe\Alain\RMN\ag-1-133-2\1\fid expt: <zgpg>
transmitter freq.: 100.624450 MHz
time domain size: 32768 points
width: 22727.27 Hz = 225.862330 ppm = 0.693581 Hz/pt
number of scans: 305

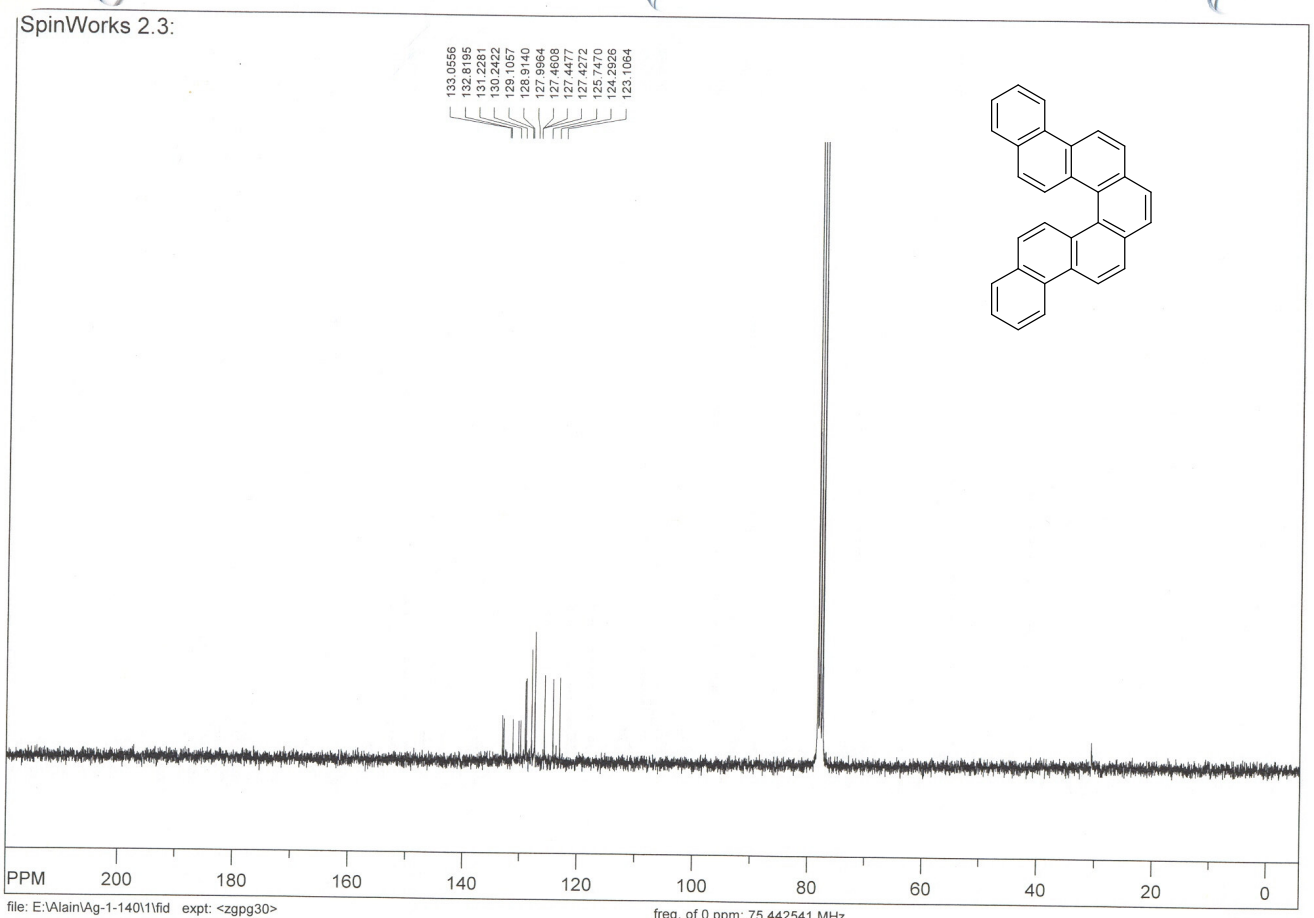
freq. of 0 ppm: 100.613889 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000
Hz/cm: 908.995 ppm/cm: 9.03354



Alain\Ag-1-140-2\1\fid expt: <zg30>
itter freq.: 300.031800 MHz
omain size: 32768 points
4006.41 Hz = 13.353285 ppm = 0.122266 Hz/pt
r of scans: 32

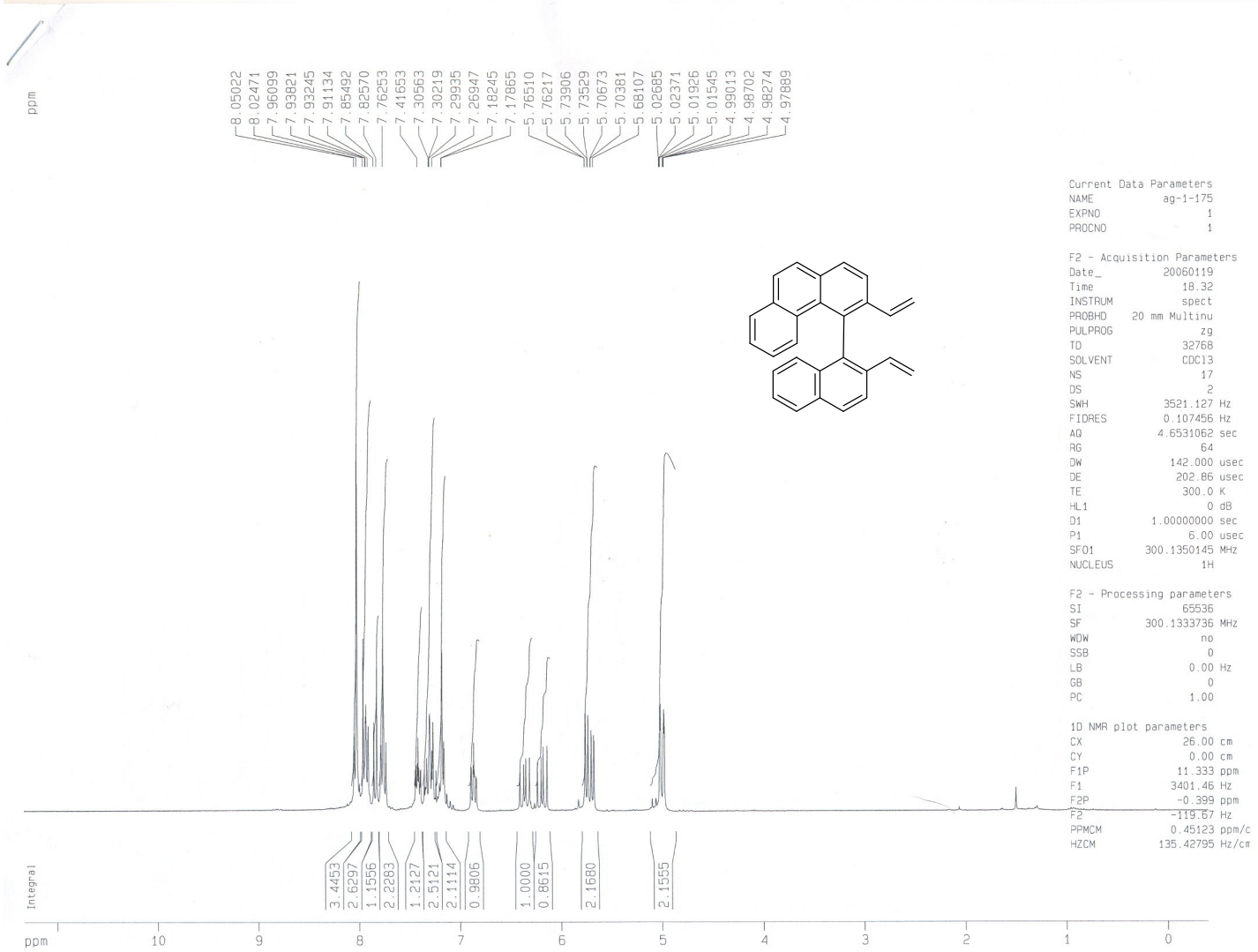
freq. of 0 ppm: 300.030017 MHz
processed size: 65536 complex points
LB: 0.000 GB: 0.0000
Hz/cm: 160.256 ppm/cm: 0.53413

SpinWorks 2.3:



file: E:\Alain\Ag-1-140\1\fid expt: <zgpg30>
transmitter freq.: 75.450604 MHz
time domain size: 49152 points
width: 17006.80 Hz = 225.403136 ppm = 0.346004 Hz/pt
number of scans: 5000

freq. of 0 ppm: 75.442541 MHz
processed size: 131072 complex points
LB: 1.000 GB: 0.0000
Hz/cm: 680.272 ppm/cm: 9.01613



Current Data Parameters

NAME	ag-1-175
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

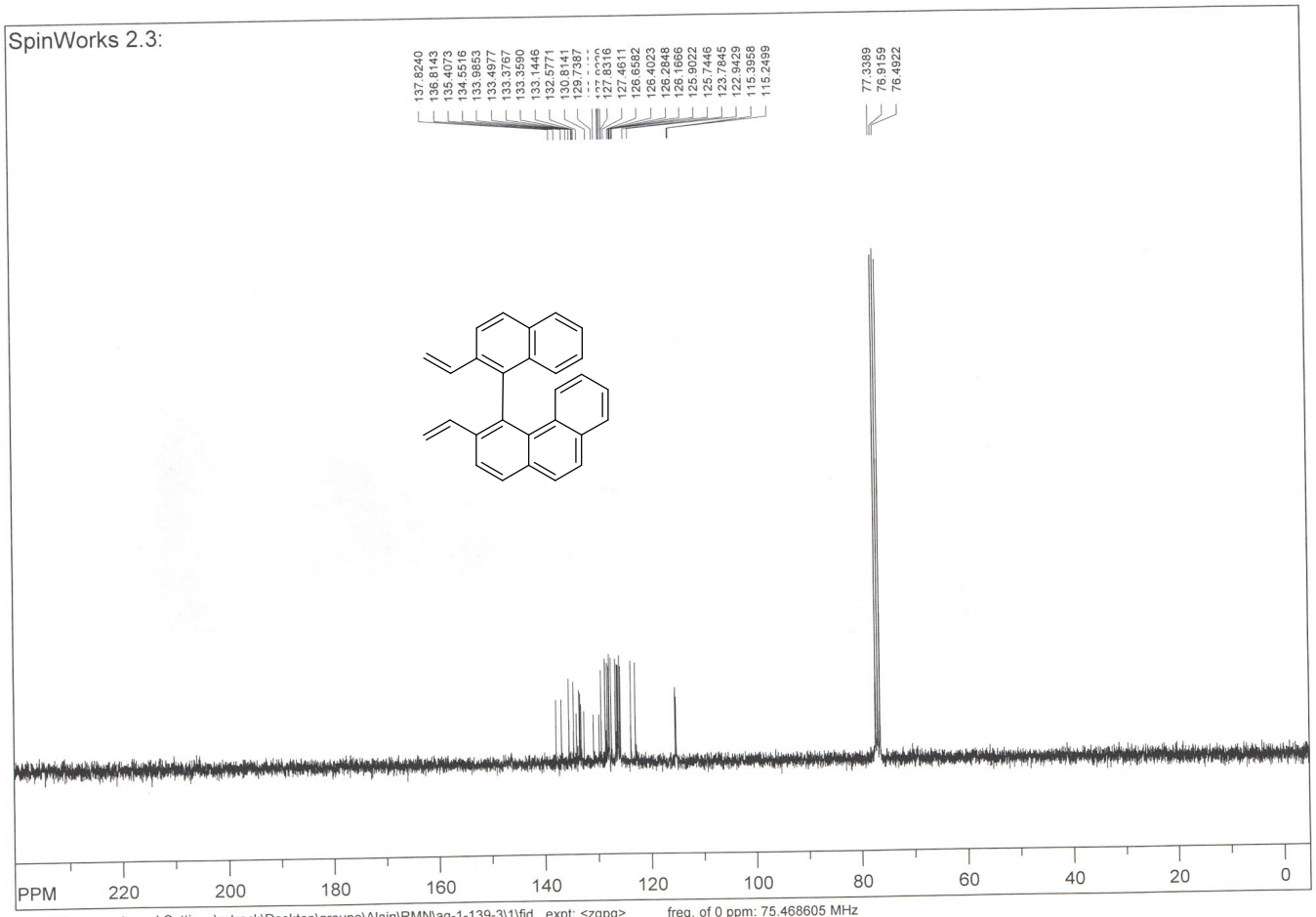
Date_	20060119
Time	18.32
INSTRUM	spect
PROBHD	20 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	17
DS	2
SWH	3521.127 Hz
FIDRES	0.107456 Hz
AQ	4.6531062 sec
RG	64
DW	142.000 usec
DE	202.86 usec
TE	300.0 K
HL1	0 dB
D1	1.00000000 sec
P1	6.00 usec
SFO1	300.1350145 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	65536
SF	300.1333736 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

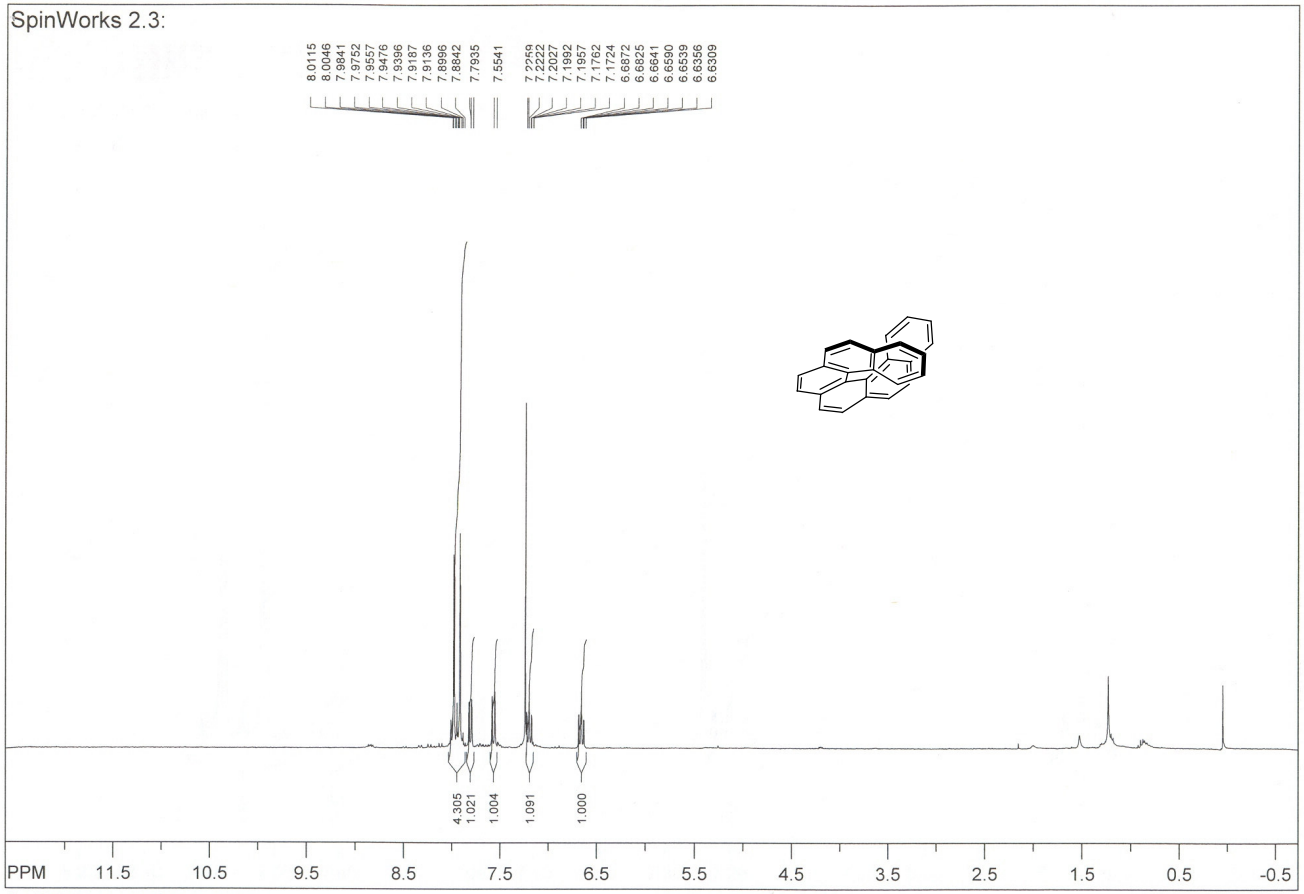
1D NMR plot parameters

CX	26.00 cm
CY	0.00 cm
F1P	11.333 ppm
F1	3401.46 Hz
F2P	-0.399 ppm
F2	-119.67 Hz
PPMCM	0.45123 ppm/c
HZCM	135.42795 Hz/cm



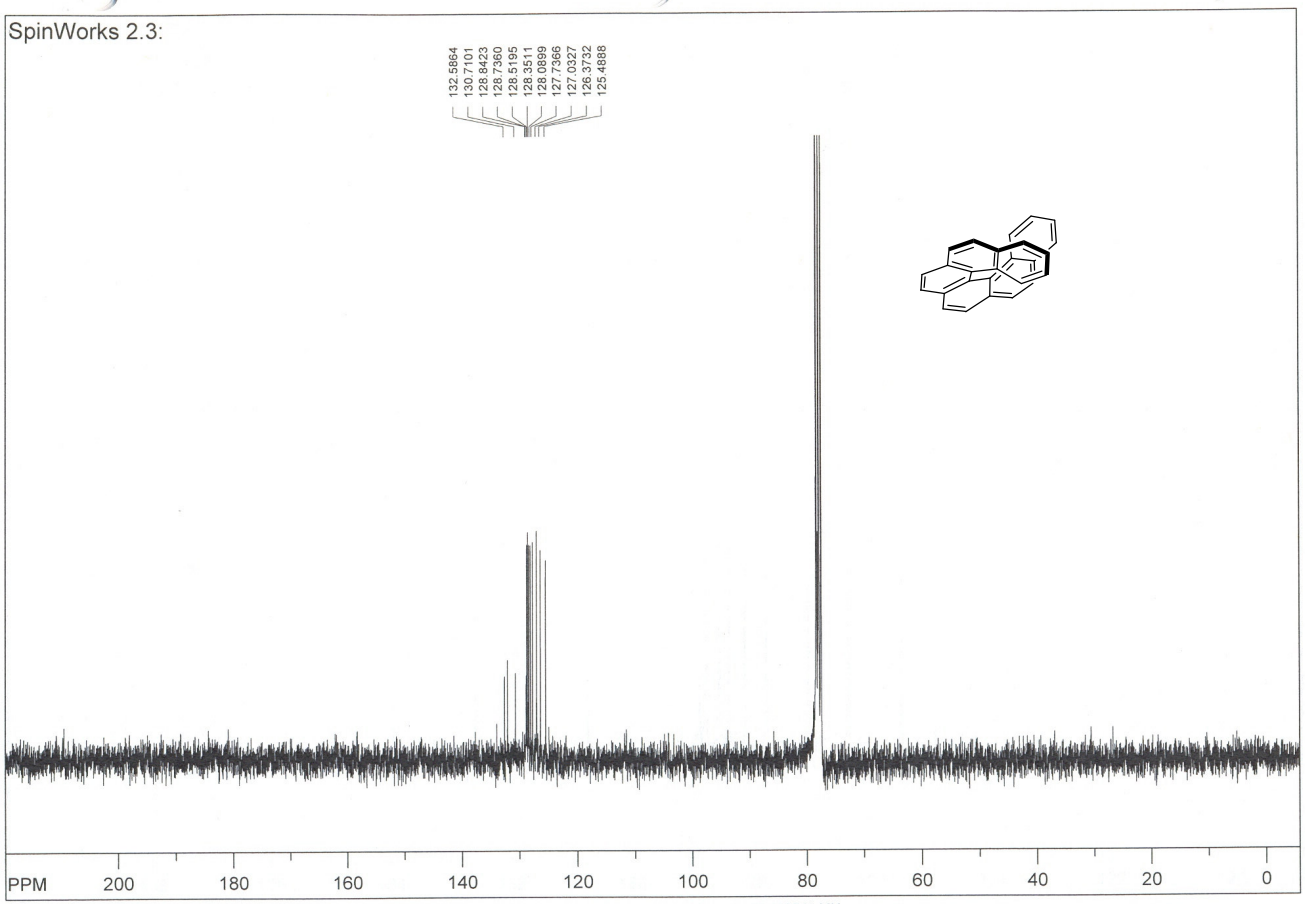
file: C:\Documents and Settings\p-track\Desktop\groupe\Alain\RMN\ag-1-139-3\1\fid expt: <zgpg>
transmitter freq.: 75.477500 MHz
time domain size: 51200 points
width: 18518.52 Hz = 245.351509 ppm = 0.361690 Hz/pt
number of scans: 300

freq. of 0 ppm: 75.468605 MHz
processed size: 131072 complex points
LB: 0.000 GB: 0.0000
Hz/cm: 740.653 ppm/cm: 9.81290



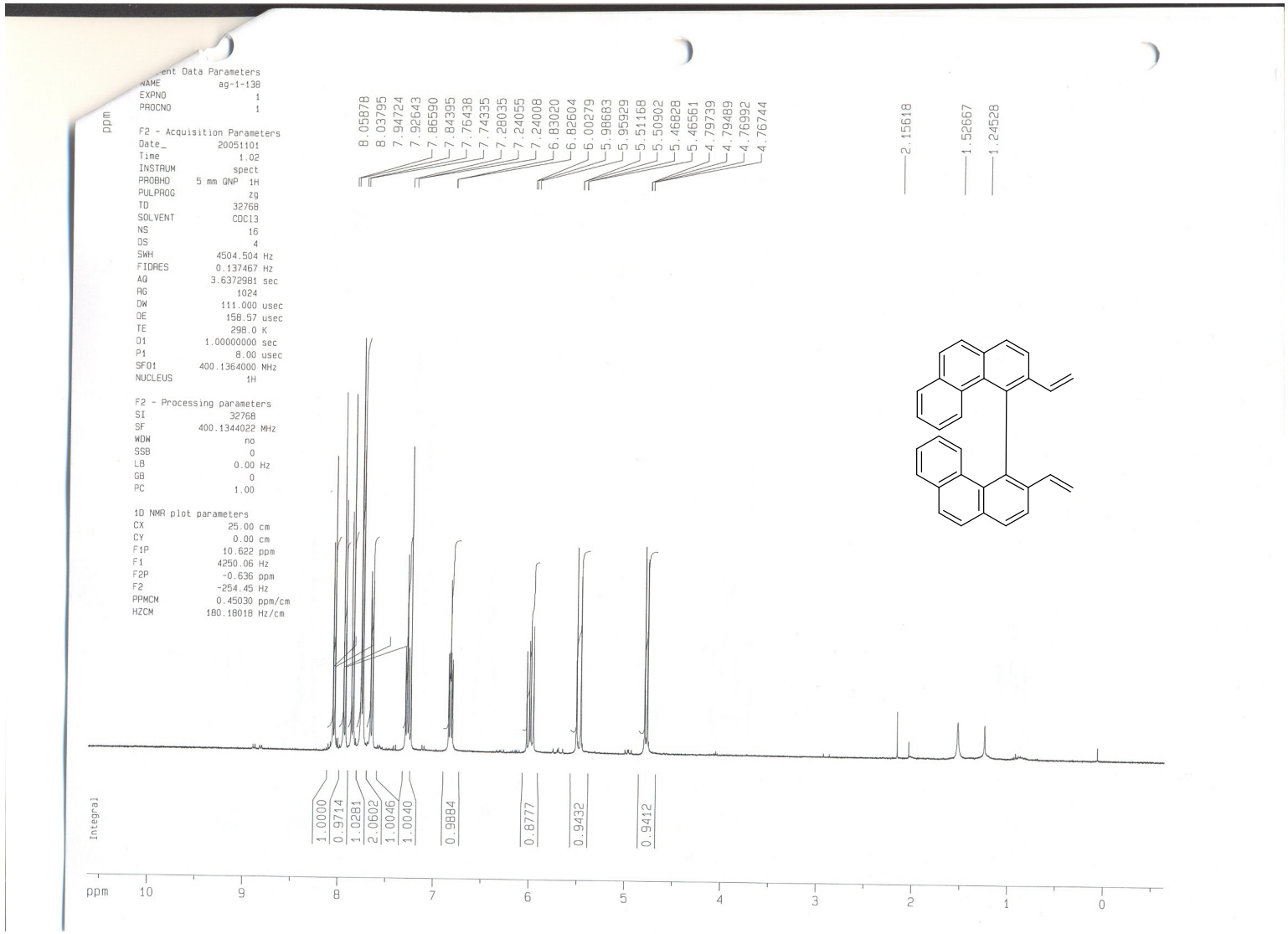
file: E:\Alain\Ag-1-146\1\fid expt: <zg30>
transmitter freq.: 300.031800 MHz
time domain size: 32768 points
width: 4006.41 Hz = 13.353285 ppm = 0.122266 Hz/pt
number of scans: 32

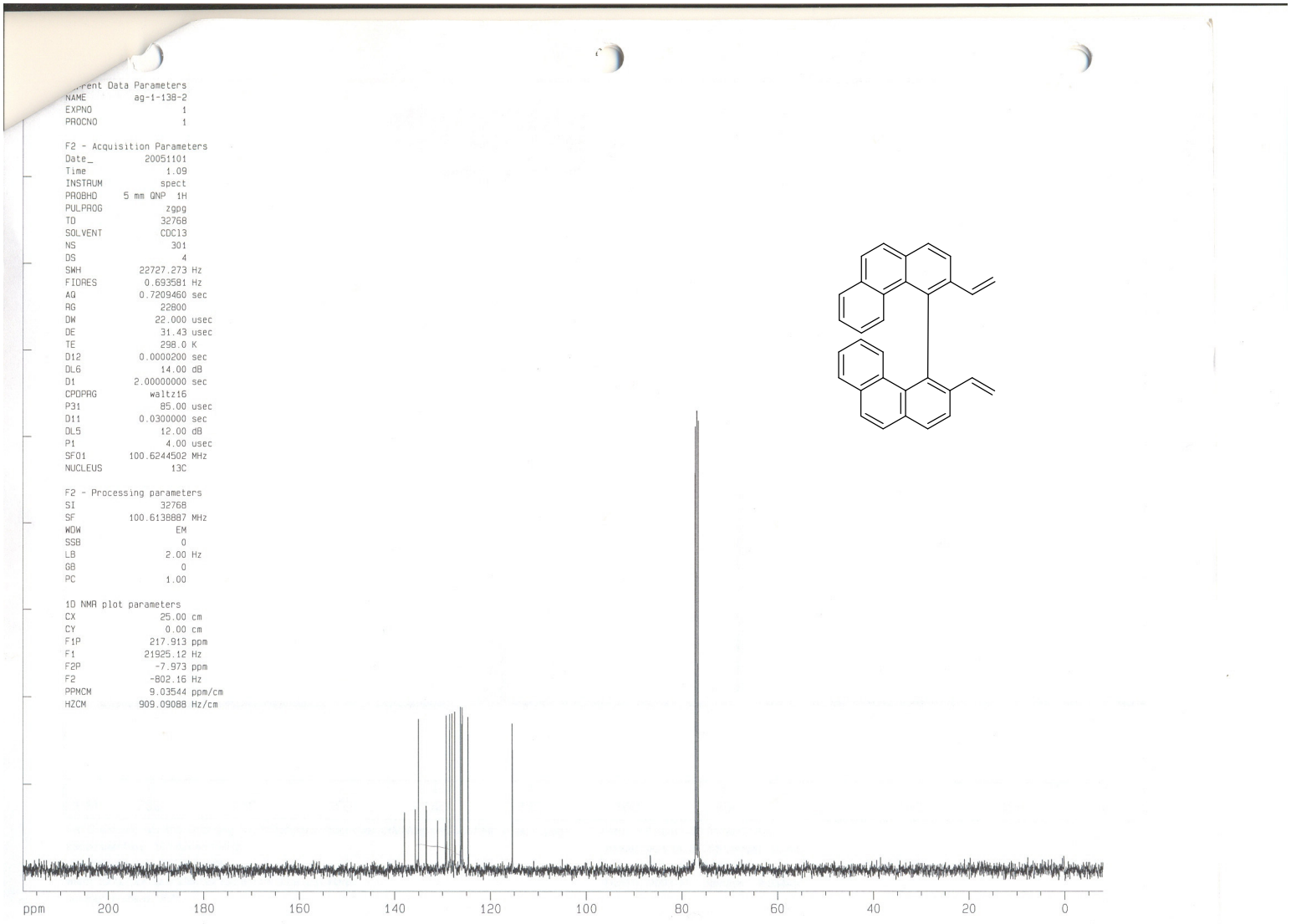
freq. of 0 ppm: 300.030017 MHz
processed size: 65536 complex points
LB: 0.300 GB: 0.0000
Hz/cm: 160.256 ppm/cm: 0.53413

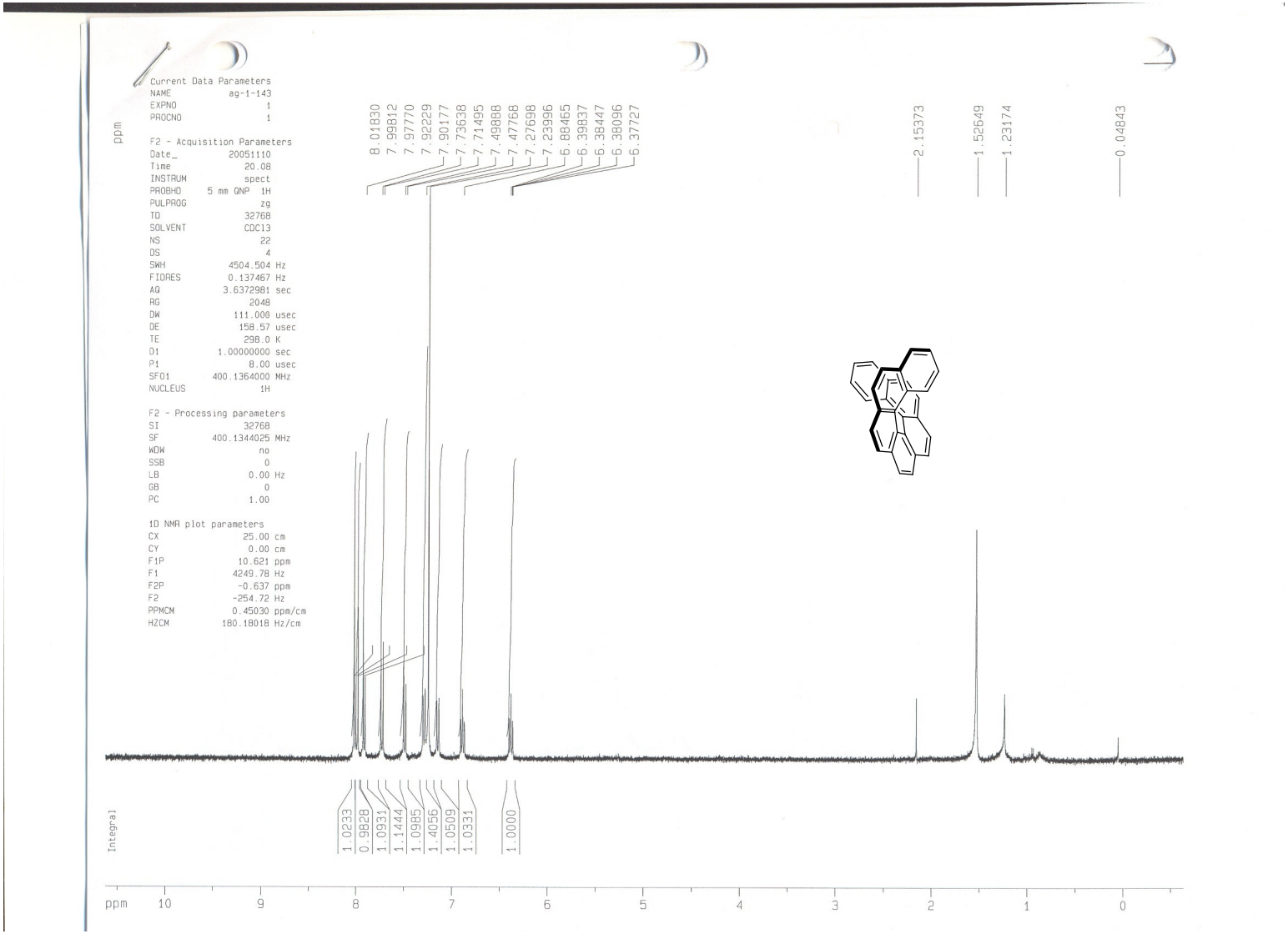


file: E:\Alain\Ag-1-146-2\1\fid expt: <zgpg30>
transmitter freq.: 75.450604 MHz
time domain size: 49152 points
width: 17006.80 Hz = 225.403136 ppm = 0.346004 Hz/pt
number of scans: 5000

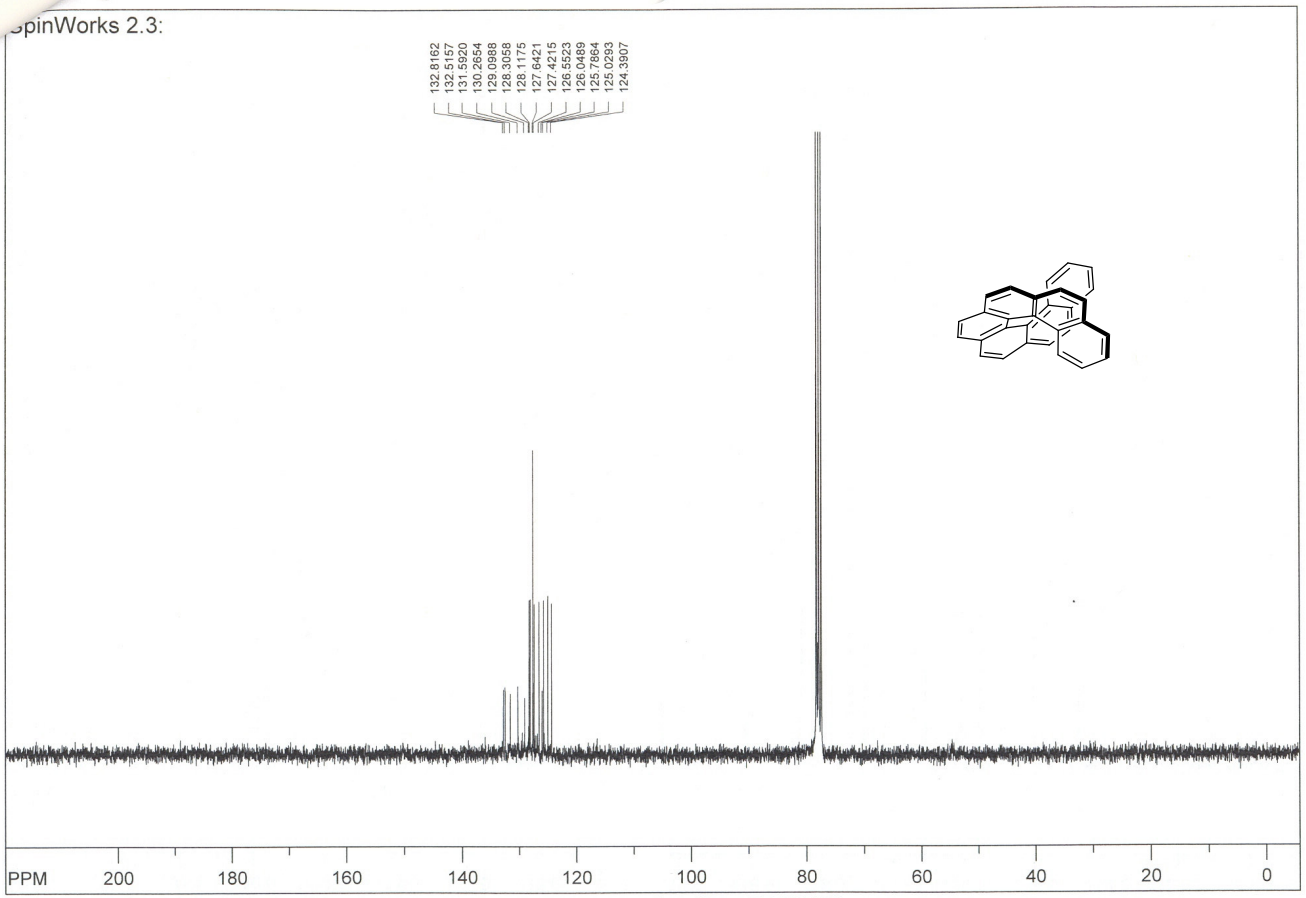
freq. of 0 ppm: 75.442541 MHz
processed size: 131072 complex points
LB: 1.000 GB: 0.0000
Hz/cm: 680.272 ppm/cm: 9.01613


ent Data Parameters
NAME ag-1-138
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051101
Time 1.02
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 4504.504 Hz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 1024
DW 111.000 usec
DE 158.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 8.00 usec
SFO1 400.1364000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1344022 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 10.622 ppm
F1 4250.06 Hz
F2P -0.636 ppm
F2 -254.45 Hz
PPMCM 0.45030 ppm/cm
HZCM 180.18018 Hz/cm
ppm
8.05878
8.03795
7.94724
7.92643
7.86590
7.84395
7.76438
7.74335
7.28035
7.24055
7.24008
6.83020
6.82604
6.00279
5.98683
5.95929
5.51168
5.50902
5.46828
5.46561
4.79739
4.79489
4.76992
4.76744
2.15618
1.52667
1.24528
Integral
1.0000
0.9714
1.0281
2.0602
1.0046
1.0040
0.9884
0.8777
0.9432
0.9412
ppm
10
9
8
7
6
5
4
3
2
1
0


Current Data Parameters
NAME ag-1-138-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051101
Time 1.09
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 301
DS 4
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 22800
DW 22.000 usec
DE 31.43 usec
TE 298.0 K
D12 0.0000200 sec
DL6 14.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 85.00 usec
D11 0.0300000 sec
DL5 12.00 dB
P1 4.00 usec
SFO1 100.6244502 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 32768
SF 100.6138887 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 217.913 ppm
F1 21925.12 Hz
F2P -7.973 ppm
F2 -802.16 Hz
PPMCM 9.03544 ppm/cm
HZCM 909.09088 Hz/cm
ppm
200
180
160
140
120
100
80
60
40
20
0


Current Data Parameters
NAME ag-1-143
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051110
Time 20.08
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 22
DS 4
SWH 4504.504 Hz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 2048
DW 111.000 usec
DE 158.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 8.00 usec
SFO1 400.1364000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1344025 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
CY 0.00 cm
F1P 10.621 ppm
F1 4249.78 Hz
F2P -0.637 ppm
F2 -254.72 Hz
PPMCM 0.45030 ppm/cm
HZCM 180.18018 Hz/cm
ppm
8.01830
7.99812
7.97770
7.92229
7.90177
7.73638
7.71495
7.49888
7.47768
7.27698
7.23996
6.88465
6.39837
6.38447
6.38096
6.37727
2.15373
1.52649
1.23174
0.04843
Integral
1.0233
0.9828
1.0931
1.1444
1.0985
1.4056
1.0509
1.0331
1.0000
ppm
10
9
8
7
6
5
4
3
2
1
0

SpinWorks 2.3:



file: E:\Alain\Ag-1-143-2\1\fid expt: <zgpg30>
transmitter freq.: 75.450604 MHz
time domain size: 49152 points
width: 17006.80 Hz = 225.403136 ppm = 0.346004 Hz/pt
number of scans: 5000

freq. of 0 ppm: 75.442541 MHz
processed size: 131072 complex points
LB: 1.000 GB: 0.0000
Hz/cm: 680.272 ppm/cm: 9.01613