Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Reductive decyanation of malononitriles and cyanoacetates using photoactivated neutral organic super-electron-donors

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Supplementary information

General experimental information

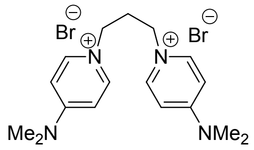
All the reactions were performed in oven-dried or flame-dried apparatus and preparation of the substrates was carried out under argon atmosphere using dry solvents. Diethyl ether, tetrahydrofuran, dichloromethane and hexane were dried with a Pure-Solv 400 solvent purification system by Innovative Technology Inc., U.S.A. A glove box (Innovative Technology Inc., U.S.A.) was used to weigh out the super electron donor (SED) into the reaction flask. All the UV reactions were carried out by using two focused UV lamps with filters (λ = 365 nm, each 100 watts) placed opposite to each other, around the reaction flask, at room temperature. All the reagents were bought from commercial suppliers and used without further purification unless stated otherwise. A Büchi rotary evaporator was used to concentrate the reaction mixtures. Thin layer chromatography (TLC) was performed using aluminium-backed sheets of silica gel and visualized under a UV lamp (254 nm). The plates were developed using vanillin or $KMnO_4$ solution. Column chromatography was performed to purify compounds by using silica gel 60 (200-400 mesh).

Proton (^1H) NMR spectra were recorded at 400 MHz on a Bruker DPX spectrometer or at 500 MHz on a Bruker DRX spectrometer. Carbon NMR (^{13}C) spectra were recorded at 100 MHz or 125 MHz respectively. The chemical shifts are quoted in parts per million (ppm) by taking tetramethylsilane as a reference ($\delta = 0$) but calibrated on the residual non-deuterated solvent signal. Signal multiplicities are abbreviated as: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; bs, broad singlet; coupling constants are given in Hertz (Hz).

Infra-Red spectra were recorded on a Perkin Elmer Spectrum One FT IR Spectrometer either pressed as discs in KBr or as films applied on NaCl crystal plates or using an ATR-IR spectrometer. Melting points were determined on a Gallenkamp Melting point apparatus. High resolution mass spectra were recorded at the EPSRC National Mass Spectrometry Service Centre, Swansea. The spectra were recorded using electron ionisation (EI), chemical ionization (CI), fast atom bombardment (FAB) or electrospray ionization (ESI) techniques as stated for each compound.

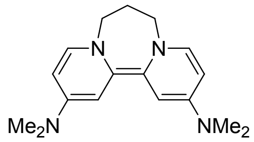
Synthesis of 4-DMAP-derived donor 8[1]

Synthesis of 1,3-*bis*(*N*',*N*'-dimethyl-4-aminopyridinium)propane dibromide[2]



A solution of 4-(dimethylamino)pyridine (9.16 g, 75 mmol, 2.5 eq.) and 1,3-dibromopropane (6.06 g, 30 mmol, 1.0 eq.) in a flask containing acetonitrile (60 mL) was stirred at reflux for 16 h, under argon. Diethyl ether (10 mL) was added to the reaction flask and the product precipitated instantaneously, and was then filtered. To precipitate more of the solid, an additional amount of diethyl ether (20 mL) was added to the filtrate. After filtration, the solid was washed with diethyl ether (3 x 50 mL) and dried under vacuum to give 1,3-bis(N',N'-dimethyl-4-aminopyridinium)propane dibromide (13 g, 97%) as a white solid m.p. 199-203 °C; [Found: (ESI$^+$) (M-Br)$^+$ 365.1338. $C_{17}H_{26}BrN_4$ (M-Br) requires 365.1335]; ν_{max}(KBr)/cm^{-1} 3027, 2725, 2468, 1649, 1571, 1403; ^{1}H-NMR (400 MHz, DMSO-d$_6$) δ 2.36 (2H, quintet, J = 7.2 Hz, $CH_2CH_2CH_2$), 3.18 (12H, s, NCH_3), 4.28 (4H, t, J = 7.2 Hz, NCH_2), 7.04 (4H, d, J = 7.6 Hz, ArH), 8.34 (4H, d, J = 7.6 Hz, ArH); ^{13}C-NMR (100 MHz, DMSO-d$_6$) δ 31.2 (CH_2), 39.8 (CH_3), 53.7 (CH_2), 107.8 (CH), 141.8 (CH), 155.8 (C); *m/z* (ESI$^+$) 367 [(M-^{81}Br)$^+$, 28%], 365 [(M-^{79}Br)$^+$, 28%], 285 (5), 143 (100).

Synthesis of *N,N,N',N'*-tetramethyl-7,8-dihydro-*6H*-dipyrido[1,2-a;2',1'-c][1,4] diazepine-2,12-diamine 8[1]



A mixture of 1,3-*bis*(*N',N'*-dimethyl-4-aminopyridinium)propane dibromide (13.38 g, 30 mmol, 1.0 eq.) and pre-washed NaH (~95%, stored in glovebox, 4.55 g, 180 mmol, 6 eq.) were taken in a Schlenk flask mounted with a dry ice condenser, and the atmosphere was made oxygen-free by flushing with argon. At this point, ammonia (75 mL) was condensed, left at reflux for 4 h and was subsequently allowed to evaporate overnight (14 h) under a steady argon flow. The flask was then transferred to an oxygen-free, moisture-free glove box. The solid was extracted with dry diethyl ether (300 mL) and the solvent was then removed by distillation and under vacuum (10-20 mbar) to give the pure *N,N,N',N'*-tetramethyl-7,8-

dihydro-6H-dipyrido-[1,2-a;2',1'-c][1,4]-diazepine-2,12-diamine **8** (7.61 g, 89%) as a purple-black, moisture-sensitive and highly oxygen-sensitive solid. ^{1}H-NMR (400 MHz, benzene-d$_6$) δ 1.00 (2H, quintet, J = 6.3 Hz, CH$_2$CH_2CH$_2$), 2.46 [12H, s, N(CH_3)$_2$], 3.03 (4H, t, J = 6.3 Hz, NCH_2), 4.91 (2H, dd, J = 7.5, 2.2 Hz, ArH), 5.14 (2H, d, J = 2.2 Hz, ArH), 5.64 (2H, d, J = 7.5 Hz, ArH); ^{13}C-NMR (100 MHz, benzene-d$_6$) δ 24.5 (CH$_2$), 40.8 (CH$_3$), 52.6 (CH$_2$), 95.8 (CH), 96.2 (CH), 116.0 (C), 138.7 (CH), 143.7 (C). The ^{1}H-NMR and ^{13}C-NMR spectral data are consistent with the previously published data of **8**.[1]

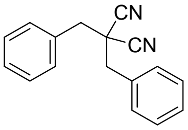
General procedure for UV-activated reductions

Under an inert atmosphere, a solution of the donor **8** in dry *N*,*N*-dimethylformamide (5 mL) was added to the flask containing the appropriate substrate in a glove-box. The sealed flask was then submitted to UV irradiation for a specified time, using two focused UV lamps (365 nm, each 100 watts). The reaction was then quenched with 1N HCl (10 mL) and extracted with diethyl ether (3 x 10 mL). The combined organic layers were then washed once again with water (10 mL), brine (10 mL) and dried over anhydrous sodium sulfate. The crude product was obtained after evaporation of solvent under reduced pressure using a rotary evaporator. Then, the crude product was adsorbed onto silica and purified by column chromatography, providing the corresponding reduced products in yields as stated.

General procedures for blank reactions

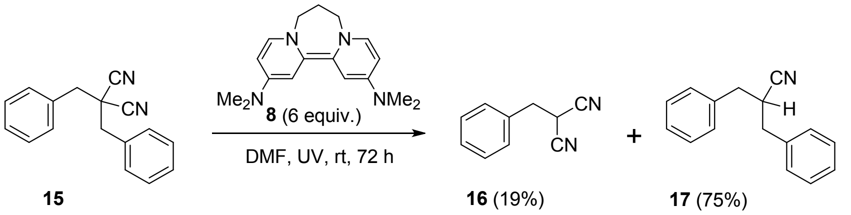
Following the preparation protocol devised for UV-activated reductions, reaction samples were prepared. Both the blank reactions and the original reaction were carried out at the same time for the same amount of time. After following the above discussed general work-up procedure, the crude product was adsorbed onto silica and purified by column chromatography, providing exclusively the starting material and the reduced product (where the reaction works) in yields as stated.

Synthesis of 2,2-dibenzylmalononitrile 15[3]



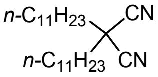
A solution of malononitrile (0.396 g, 6 mmol) in dry tetrahydrofuran (2 mL) was added slowly to the suspension of sodium hydride (~60%, 0.528 g, 13.2 mmol) in dry tetrahydrofuran (5 mL) at 0 °C under argon gas. The resulting solution was stirred for 30 min. at room temperature and benzyl bromide (2.257 g, 13.2 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The resulting reaction mixture was further refluxed for 16 h under argon gas. At this point, the reaction was quenched with water (10 mL) and extracted with diethyl ether (3 x 10 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (20% diethyl ether in petroleum ether) to afford 2,2-dibenzylmalononitrile **15** (1.316 g, 89%) as a white solid m.p. 128-130 °C; (lit.:[3] 128-130 °C); [Found: (CI$^+$ corona) (M+H)$^+$ 247.1234. $C_{17}H_{15}N_2$ (M+H) requires 247.1230]; ν_{max}(film) /cm^{-1} 3032, 2933, 2247, 1496, 1442, 1269, 1089; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.26 (4H, s, Ar*CH₂*), 7.40-7.44 (10H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 41.3 (C), 43.6 (CH_2), 115.1 (C), 129.0 (CH), 129.1 (CH), 130.4 (CH), 132.1 (C); *m/z* (CI$^+$ corona) 247 [(M+H)$^+$, 100 %], 222 (10%).

UV-activated reduction of 2,2-dibenzylmalononitrile 15



The general procedure for electron transfer reactions under UV conditions was applied to 2,2-dibenzylmalononitrile **15** (0.098 g, 0.4 mmol) using the donor **8** (0.682 g, 2.4 mmol, 6 eq.). After following the general work-up process, the crude product was purified by column chromatography (10% diethyl ether in petroleum ether) and afforded 2-benzyl-3-phenylpropanenitrile **17**[4] (0.066 g, 75%), as a white solid m.p. 88-90 °C (lit.:[4] 92-92.5 °C), and 2-benzylmalononitrile **16**[5] (0.0119 g, 19%), as a white solid m.p. 84-86 °C (lit.:[5] 88 °C).

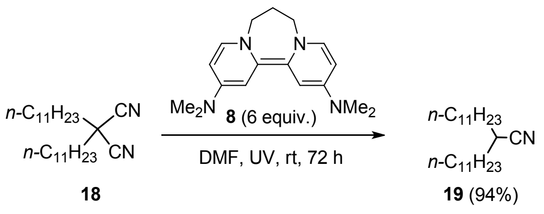
Synthesis of 2,2-diundecylmalononitrile 18



A solution of malononitrile (0.396 g, 6 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.600 g, 15 mmol) in dry tetrahydrofuran (10 mL) at 0 ^{o}C under argon gas. The resulting solution was stirred for 15 min at room temperature and 1-bromoundecane (3.104 g, 13.2 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (2% diethyl ether in petroleum ether) to yield *2,2-diundecylmalononitrile* **18** (1.598 g, 71%) as a white crystalline solid m.p. 40-42 ^{o}C. [Found: (CI corona^{+}) $(M+H)^+$ 375.3732. $C_{25}H_{47}N_2$ (M+H) requires 375.3734]; ν_{max}(film)/cm^{-1} 2958, 2914, 2846, 2247, 1469, 1379, 719; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.89 [6H, t, J = 6.8 Hz, $(CH_2)_{10}CH_3$], 1.28-1.41 (32H, m, $CCH_2CH_2(CH_2)_8CH_3$), 1.63-1.71 (4H, m, $CCH_2CH_2(CH_2)_8CH_3$), 1.89-1.94 (4H, m, $CCH_2(CH_2)_9CH_3$); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.2 (CH_3), 22.8 (CH_2), 25.7 (CH_2), 29.0 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.7 (2 x CH_2), 29.8 (C), 32.0 (CH_2), 38.0 (CH_2), 116.0 (C); *m/z* (CI corona^{+}) 375 [$(M+H)^+$, 100%], 350 [$((M-CN)+H)^+$, 26%], 327 (41).

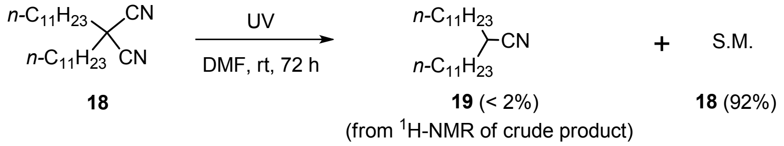
UV-activated reduction of 2,2-diundecylmalononitrile 18

(a) Reaction with photoactivated 8



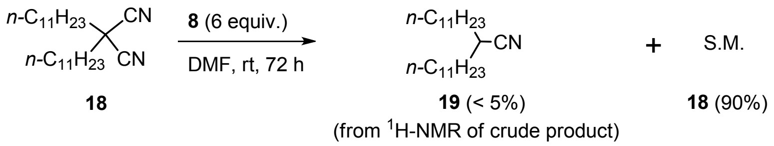
The general procedure for electron transfer reactions under UV conditions was applied to 2,2-diundecylmalononitrile **18** (0.149 g, 0.4 mmol) using the donor **8** (0.682 g, 2.4 mmol, 6 eq.) for 72 h. After following the general work-up process, the crude product was purified by column chromatography (2% diethyl ether in petroleum ether) and provided *2-undecyltridecanenitrile* **19** (0.131 g, 94%) as a white crystalline solid m.p. 28-30 ^{o}C. [Found: (CI corona^{+}) $(M+H)^+$ 350.3778. $C_{24}H_{48}N$ (M+H) requires 350.3781]; ν_{max}(film)/cm^{-1} 2986, 2922, 2852, 2241, 1465, 1377, 721; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.89 [6H, t, J = 6.8 Hz, $CH(CH_2)_{10}CH_3$], 1.27-1.37 (32H, m, $CHCH_2CH_2(CH_2)_8CH_3$), 1.39-1.66 (8H, m, CH*CH_2CH_2*(CH$_2$)$_8$CH$_3$), 2.47-2.53 (1H, m, C*H*CN); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 14.3 (CH$_3$), 22.8 (CH$_2$), 27.3 (CH$_2$), 29.3 (CH$_2$), 29.48 (CH$_2$), 29.52 (CH$_2$), 29.7 (CH$_2$), 29.8 (2 x CH$_2$), 31.8 (CH), 32.1 (CH$_2$), 32.4 (CH$_2$), 122.7 (C); *m/z* ((CI corona$^+$) 350 [(M+H)$^+$, 86%], 327 (100), 220 (24), 155 (16).

(b) Photoactivated reaction without 8



The general procedure for blank reactions was applied to 2,2-diundecylmalononitrile **18** (0.149 g, 0.4 mmol) without using donor **8**, for 72 h. After following the general work-up process, crude product was obtained. From the ^{1}H-NMR of the crude product, it was noticed that only <2% of the starting material **18** was converted to product **19**. No attempt was made to isolate this product, but column chromatography (2% diethyl ether in petroleum ether) of the crude product provided an excellent recovery of starting material **18** (92%).

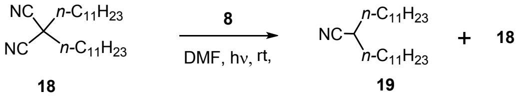
(c) With 8 under no photoactivation



The general procedure for blank reactions was applied to 2,2-diundecylmalononitrile **18** (0.149 g, 0.4 mmol) using the donor **8** (0.682 g, 2.4 mmol, 6 eq.) for 72 h. After following the general work-up process, crude product was obtained. From the ^{1}H-NMR of the crude product, it was noticed that only <5% of the starting material **18** was converted to product **19**. No attempt was made to isolate this product, but column chromatography (2% diethyl ether in petroleum ether) of the crude product provided an excellent recovery of starting material **18** (90%).

Optimisation of reaction conditions for UV-activated reduction of 18

Table 1: Optimisation of reaction conditions for decyanation of malononitriles.



	8 (equiv.)	Time (h)	Ratio[a] of **19/18**	**19**[b] (%)
i	4	48	100/0	---
ii	4	24	89/11	---
iii	4	36	100/0	92%

[a] From the ^{1}H-NMR of the crude product. [b] Isolated yields.

(i) Using 4 equivalents of 8 for 48 h

The general procedure for electron transfer reactions under UV conditions was applied to 2,2-diundecylmalononitrile **18** (0.149 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 48 h. After following the general work-up process, crude product was obtained. From the ^{1}H-NMR of the crude product, it was observed that the starting material **18** was completely consumed and provided exclusively decyanated product **19**. No attempt was made to isolate this product **19**.

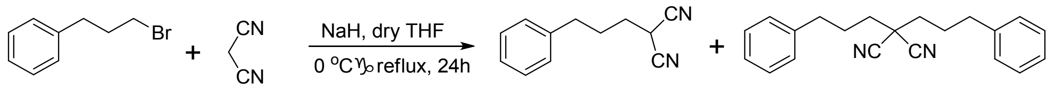
(ii) Using 4 equivalents of 8 for 24 h

The general procedure for electron transfer reactions under UV conditions was applied to 2,2-diundecylmalononitrile **18** (0.149 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 24 h. After following the general work-up process, crude product was obtained. From the ^{1}H-NMR of the crude product, it was observed that the starting material **18** was not completely consumed. The ratio of decyanated product **19** to starting material **18** in the crude product was found to be 89:11.

(iii) Using 4 equivalents of 8 for 36 h

The general procedure for electron transfer reactions under UV conditions was applied to 2,2-diundecylmalononitrile **18** (0.149 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 36 h. After following the general work-up process, crude product was obtained. From the ^{1}H-NMR of the crude product, it was observed that the starting material **18** was completely consumed. Column chromatography (2% diethyl ether in petroleum ether) of the crude product provided exclusively decyanated product **19** (92%). Spectral data of the product **19** were consistent with the previous data of the same compound.

Synthesis of 2-(3-phenylpropyl)malononitrile[6] and 2,2-bis(3-phenylpropyl)malononitrile



64%

20 (15%)

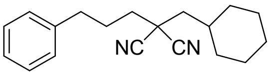
A solution of malanonitrile (1.3212 g, 20 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.600 g, 15 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and (3-bromopropyl)benzene (2.986 g, 15 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (10% diethyl ether in hexane) to yield 2-(3-phenylpropyl)malononitrile (1.753 g, 64%) as a colourless oil and *2,2-bis(3-phenylpropyl)malononitrile* **20** (0.680 g, 15%) as a white crystalline solid m.p. 78-80 °C.

For 2-(3-phenylpropyl)malononitrile: [Found: (CI corona$^+$) (M+H)$^+$ 185.1071. $C_{12}H_{13}N_2$ (M+H) requires 185.1073]; ν_{max}(film)/cm^{-1} 3026, 2922, 2866, 2240, 1602, 1496, 1454, 698; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.95-2.01 (2H, m, $CHCH_2CH_2CH_2Ar$), 2.02-2.07 (2H, m, $CHCH_2CH_2CH_2Ar$), 2.75 (2H, t, J = 7.0 Hz, $ArCH_2$), 3.67 (1H, t, J = 7.0 Hz, $CH(CN)_2$), 7.19 (2H, d, J = 7.0 Hz, ArH), 7.25 (1H, t, J = 7.0 Hz, ArH), 7.33 (2H, t, J = 7.0 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 22.6 (CH), 28.1 (CH_2), 30.2 (CH_2), 34.5 (CH_2), 112.6 (C), 126.7 (CH), 128.4 (CH), 128.8 (CH), 140.0 (C); *m/z* (CI corona$^+$) 185 [(M+H)$^+$, 100%], 141 (4).

For 2,2-bis(3-phenylpropyl)malononitrile **20**: [Found: (CI corona$^+$) (M+H)$^+$ 303.1853. $C_{21}H_{23}N_2$ (M+H) requires 303.1856]; ν_{max}(film)/cm^{-1} 3026, 2926, 2860, 2245, 1600, 1496, 1452, 702; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.88-1.91 (4H, m, $CCH_2CH_2CH_2Ar$), 1.97-2.03 (4H, m, $CCH_2CH_2CH_2Ar$), 2.72 (4H, t, J = 7.0 Hz, $ArCH_2$), 7.17 (4H, d, J = 7.0 Hz, ArH), 7.23 (2H, t, J = 7.5 Hz, ArH), 7.31 (4H, t, J = 7.5 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ

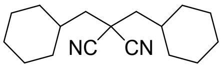
27.1 (CH_2), 34.9 (CH_2), 37.2 (CH_2), 37.7 (C), 115.7 (C), 126.6 (CH), 128.5 (CH), 128.8 (CH), 140.2 (C); *m/z* (CI corona$^+$) 303 [$(M+H)^+$, 100%], 191 (4).

Synthesis of 2-(cyclohexylmethyl)-2-(3-phenylpropyl)malononitrile 21



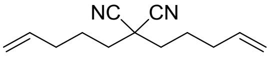
A solution of 2-(3-phenylpropyl)malononitrile (0.460 g, 2.5 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.120 g, 3 mmol) in dry tetrahydrofuran (10 mL) at 0 ^oC under argon gas. The resulting solution was stirred for 15 min at room temperature and (bromomethyl)cyclohexane (0.531 g, 3 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (10% diethyl ether in hexane) to yield *2-(cyclohexylmethyl)-2-(3-phenylpropyl)malononitrile* **21** (0.562 g, 80%) as a white solid m.p. 36-38 ^oC. [Found: (CI corona$^+$) $(M+H)^+$ 281.2013. $C_{19}H_{25}N_2$ (M+H) requires 281.2012]; ν_{max}(film)/cm^{-1} 3062, 3028, 2933, 2856, 2245, 1602, 1494, 1448, 1028, 727; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.01-1.36 (5H, m, Cy), 1.56-1.79 (6H, m, Cy), 1.89-1.96 (4H, m, CCH_2Cy, $ArCH_2CH_2CH_2C$), 1.99-2.08 (2H, m, $ArCH_2CH_2CH_2C$), 2.74 (2H, t, J = 7.2 Hz, $ArCH_2$), 7.19-7.25 (3H, m, ArH), 7.32 (2H, t, J = 7.6 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.9 (2 x CH_2), 26.0 (CH_2), 27.0 (CH_2), 33.5 (2 x CH_2), 34.9 (CH), 35.7 (CH_2), 35.9 (C), 38.7 (CH_2), 44.8 (CH_2), 116.1 (C), 126.6 (CH), 128.5 (CH), 128.8 (CH), 140.3 (C); *m/z* (CI corona$^+$) 281 [$(M+H)^+$, 100%], 256 [$((M-CN)+H)^+$, 6%], 203 (7).

Synthesis of 2,2-bis(cyclohexylmethyl)malononitrile 22[7]



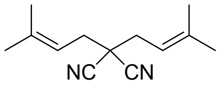
A solution of malononitrile (0.330 g, 5 mmol) in dry tetrahydrofuran (2 mL) was added slowly to a suspension of sodium hydride (~60%, 0.440 g, 11 mmol) in dry tetrahydrofuran (10 mL) at 0 ^oC under argon gas. The resulting solution was stirred for 15 min at room temperature and (bromomethyl)cyclohexane (1.77 g, 10 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (5% diethyl ether in petroleum ether) to yield 2,2-bis(cyclohexylmethyl)malononitrile **22**[7] (0.880 g, 68%) as a white solid m.p. 88-90 °C (lit.:[7] 90-92 °C). [Found: (CI corona$^+$) (M+H)$^+$ 259.2166. $C_{17}H_{27}N_2$ (M+H) requires 259.2169]; ν_{max}(film)/cm^{-1} 2918, 2848, 2242, 1446, 842; ^{1}H-NMR (400 MHz, CDCl$_3$) δ 1.04-1.23 (6H, m, Cy), 1.27-1.39 (4H, m, Cy), 1.66-1.82 (12H, m, Cy), 1.92-1.96 (4H, m, CyC*H$_2$*C); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 25.98 (CH$_2$), 26.01 (CH$_2$), 33.6 (CH$_2$), 34.2 (C), 35.6 (CH), 46.2 (CH$_2$), 116.6 (C); *m/z* (CI corona$^+$) 517 [(2M+H)$^+$, 32%], 492 (13), 467 (16), 303 (29), 259 [(M+H)$^+$, 100%], 234 (69).

Synthesis of 2,2-di(pent-4-en-1-yl)malononitrile 23[8]



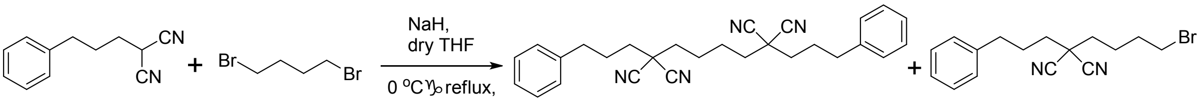
A solution of malononitrile (0.330 g, 5 mmol) in dry tetrahydrofuran (2 mL) was added slowly to a suspension of sodium hydride (~60%, 0.440 g, 11 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and 5-bromopent-1-ene (1.6392 g, 11 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (5% diethyl ether in hexane) to yield 2,2-di(pent-4-en-1-yl)malononitrile **23** (0.726 g, 72%) as a colourless oil. [Found: (CI corona$^+$) (M+H)$^+$ 203.1542. $C_{13}H_{19}N_2$ (M+H) requires 203.1543]; ν_{max}(film)/cm^{-1} 3080, 2933, 2866, 2241, 1641, 1460, 914; ^{1}H-NMR (400 MHz, CDCl$_3$) δ 1.75-1.83 (4H, m, =CHCH$_2$C*H$_2$*CH$_2$), 1.91-1.96 (4H, m, CC*H$_2$*CH$_2$CH$_2$), 2.16-2.21 (4H, m, =CHC*H$_2$*CH$_2$CH$_2$), 5.04-5.11 (4H, m, C*H$_2$*=CHCH$_2$), 5.73-5.83 (2H, m, CH$_2$=C*H*CH$_2$); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 24.7 (CH$_2$), 32.7 (CH$_2$), 37.2 (CH$_2$), 37.7 (C), 115.7 (C), 116.4 (CH$_2$), 136.7 (CH); *m/z* (CI corona$^+$) 203 [(M+H)$^+$, 100%], 178 (16).

Synthesis of 2,2-*bis*(3-methylbut-2-en-1-yl)malononitrile 25



A solution of malononitrile (0.397 g, 6 mmol) in dry tetrahydrofuran (2 mL) was added slowly to a suspension of sodium hydride (~60%, 0.528 g, 13.2 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and 1-bromo-3-methylbut-2-ene (1.967 g, 13.2 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 16 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (5% ethyl acetate in petroleum ether) to yield 2,2-*bis*(3-methylbut-2-en-1-yl)malononitrile **25** (1.047 g, 86%) as a white solid m.p. 62-64 °C. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.73 (6H, s, =CH_3), 1.83 (6H, s, CH_3), 2.66 (4H, d, J = 7.6 Hz, =CHCH_2), 5.27-5.32 (2H, m, =CHCH$_2$); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 18.4 (CH_3), 26.1 (CH_3), 35.7 (CH_2), 38.2 (C), 114.9 (CH), 115.9 (C), 140.5 (C).

Synthesis of 1,12-diphenyldodecane-4,4,9,9-tetracarbonitrile 26 and 2-(4-bromobutyl)-2-(3-phenylpropyl)malononitrile 27



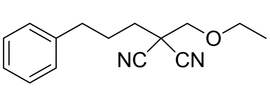
A solution of 2-(3-phenylpropyl)malononitrile (0.552 g, 3 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.132 g, 3.3 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and 1,4-dibromobutane (0.712 g, 3.3 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 16 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (20% diethyl ether in petroleum ether) to yield 1,12-diphenyldodecane-

4,4,9,9-tetracarbonitrile **26** (0.280 g, 22%) and 2-(4-bromobutyl)-2-(3-phenylpropyl) malononitrile **27** (0.462 g, 48%) as a white solid (m.p. 128-130 °C) and a thick colourless oil, respectively.

For 12-diphenyldodecane-4,4,9,9-tetracarbonitrile **26**: [Found: (CI corona$^+$) $(M+NH_4)^+$ 440.2817. $C_{28}H_{34}N_5$ (M+ NH_4) requires 440.2809]; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.73-1.77 (4H, m, $\mathrm{CCH_2C}\mathit{H_2}\mathrm{C}\mathit{H_2}\mathrm{CH_2C}$), 1.91-1.97 (8H, m, $\mathrm{C}\mathit{H_2}\mathrm{CH_2CH_2C}\mathit{H_2}\mathrm{C}$, $\mathrm{ArCH_2C}\mathit{H_2}\mathrm{CH_2C}$), 1.99-2.07 (4H, m, $\mathrm{ArCH_2CH_2C}\mathit{H_2}\mathrm{C}$), 2.75 (4H, t, J = 7.2 Hz, $\mathrm{ArC}\mathit{H_2}\mathrm{CH_2CH_2C}$), 7.19 (4H, d, J = 6.8 Hz, ArH), 7.22-7.26 (2H, m, ArH), 7.33 (4H, t, J = 7.2 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.0 (CH_2), 27.1 (CH_2), 34.8 (CH_2), 37.2 (CH_2), 37.4 (C), 37.6 (CH_2), 115.4 (C), 126.6 (CH), 128.5 (CH), 128.8 (CH), 140.1 (C); *m/z* (CI corona$^+$) 440 [(M+ $NH_4)^+$, 100%], 408 (20), 311 (22), 282 (34), 136 (16).

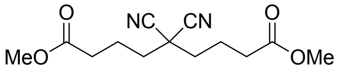
For 2-(4-bromobutyl)-2-(3-phenylpropyl) malononitrile **27**: [Found: (CI corona$^+$) $(M+NH_4)^+$ 336.1070. $C_{16}H_{23}N_3Br$ $(M+NH_4)$ requires 336.1070]; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.79-1.88 (2H, m, $\mathrm{CCH_2C}\mathit{H_2}\mathrm{CH_2CH_2Br}$), 1.91-2.08 (8H, m, $\mathrm{C}\mathit{H_2}\mathrm{CH_2C}\mathit{H_2}\mathrm{CH_2Br}$, $\mathrm{ArCH_2C}\mathit{H_2}\mathrm{C}\mathit{H_2}$), 2.76 (2H, t, J = 7.2 Hz, $\mathrm{ArC}\mathit{H_2}$), 3.43 (2H, t, J = 6.4 Hz, $\mathrm{ArC}\mathit{H_2}$), 7.19-7.21 (2H, m, ArH), 7.21-7.26 (1H, m, ArH), 7.30-7.35 (2H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 24.3 (CH_2), 27.1 (CH_2), 31.7 (CH_2), 32.2 (CH_2), 34.8 (CH_2), 36.9 (CH_2), 37.1 (CH_2), 37.6 (C), 115.5 (C), 126.6 (CH), 128.5 (CH), 128.8 (CH), 140.2 (C); *m/z* (CI corona$^+$) 338 [$(M+NH_4)^+$, ^{81}Br, 98%], 336 [$(M+NH_4)^+$, ^{79}Br, 100%], 316 (27), 256 (30), 159 (10).

Synthesis of 2-(ethoxymethyl)-2-(3-phenylpropyl)malononitrile 29



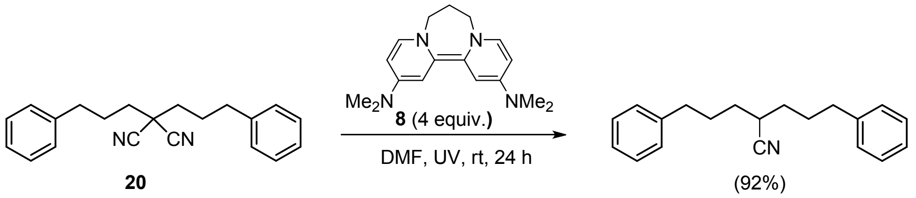
A solution of 2-(3-phenylpropyl)malononitrile (0.736 g, 4 mmol) in dry tetrahydrofuran (2 mL) was added slowly to a suspension of sodium hydride (~60%, 0.176 g, 4.4 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and (chloromethoxy)ethane (0.453 g, 4.8 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 16 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (10% ethyl acetate in petroleum ether) to yield 2-(ethoxymethyl)-2-(3-phenylpropyl)malononitrile **29** (0.621 g, 64%) as a light yellow oil. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.25 (3H, t, J = 7.0 Hz, OCH_2CH_3), 1.96-2.05 (4H, m, $ArCH_2CH_2CH_2C$), 2.76 (2H, t, J = 7.0 Hz, $ArCH_2$), 3.67 (2H, q, J = 7.0 Hz, OCH_2CH_3), 3.75 (2H, s, $CCH_2OCH_2CH_3$), 7.20 (2H, d, J = 7.0 Hz, ArH), 7.24 (1H, t, J = 7.5 Hz, ArH), 7.32 (2H, t, J = 7.0 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.9 (CH_3), 26.9 (CH_2), 33.6 (CH_2), 34.9 (CH_2), 38.6 (C), 68.2 (CH_2), 72.2 (CH_2), 114.6 (C), 126.5 (CH), 128.5 (CH), 128.8 (CH), 140.3 (C).

Synthesis of dimethyl 5,5-dicyanononanedioate 30



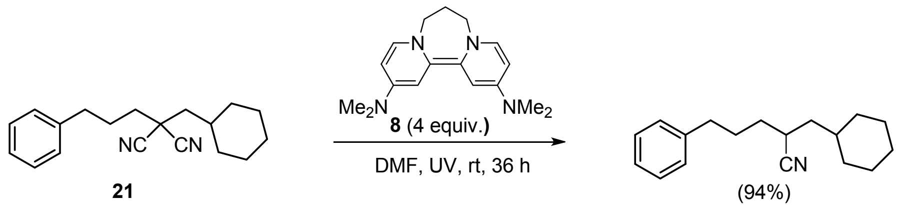
A solution of malononitrile (0.330 g, 5 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.440 g, 11 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and methyl 4-bromobutanoate (1.9914 g, 11 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (25% ethyl acetate in hexane) to yield *dimethyl 5,5-dicyanononanedioate* **30** (0.935 g, 70%) as a colourless oil. [Found: (ESI$^+$) $(M+NH_4)^+$ 284.1609. $C_{13}H_{22}N_3O_4$ $(M+NH_4)$ requires 284.1605]; ν_{max}(film)/cm^{-1} 2954, 2360, 2328, 1730, 1436, 1259, 1170; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.97-2.06 (8H, m, $CH_2CH_2CO_2CH_3$), 2.47 (4H, t, J = 6.0 Hz, CCH_2), 3.71 (6H, s, CO_2CH_3); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 20.8 (CH_2), 32.6 (CH_2), 36.6 (CH_2), 37.2 (C), 51.9 (CH_3), 115.2 (C), 172.5 (C=O); *m/z* (ESI$^+$) 284 [$(M+NH_4)^+$, 100%], 267 [$(M+H)^+$, 4%], 199 (4).

UV-activated reduction of 2,2-bis(3-phenylpropyl)malononitrile 20



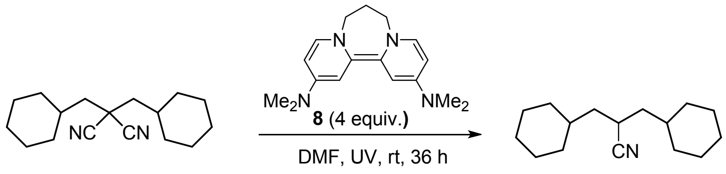
The general procedure for electron transfer reactions under UV conditions was applied to 2,2-bis(3-phenylpropyl)malononitrile **20** (0.121 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 24 h. After following the general work-up process, column chromatography (2% ethyl acetate in hexane) provided *5-phenyl-2-(3-phenylpropyl)pentanenitrile* (0.1025 g, 92%) as a colourless oil. [Found: (CI corona$^+$) (M+H)$^+$ 278.1905. $C_{20}H_{24}N$ (M+H) requires 278.1903]; ν_{max}(film)/cm^{-1} 3026, 2941, 2860, 2235, 1602, 1494, 1452, 696; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.52-1.80 (6H, m, $\mathrm{CCH_2C}H_2\mathrm{CH_2Ar}$, $\mathrm{CC}H_2\mathrm{CH_2CH_2Ar}$), 1.83-1.94 (2H, m, $\mathrm{CC}H_2\mathrm{CH_2CH_2Ar}$), 2.49-2.56 (1H, m, $\mathrm{C}H\mathrm{CN}$), 2.65 (4H, t, J = 7.6 Hz, $\mathrm{ArC}H_2$), 7.16-7.23 (6H, m, ArH), 7.30 (4H, t, J = 7.6 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 28.9 (CH_2), 31.6 (CH), 31.8 (CH_2), 35.4 (CH_2), 122.2 (C), 126.2 (CH), 128.5 (CH), 128.6 (CH), 141.4 (C); *m/z* (CI corona$^+$) 278 [(M+H)$^+$, 100%], 249 (15), 203 (13), 95 (16).

UV-activated reduction of 2-(cyclohexylmethyl)-2-(3-phenylpropyl)malononitrile 21



The general procedure for electron transfer reactions under UV conditions was applied to 2-(cyclohexylmethyl)-2-(3-phenylpropyl)malononitrile **21** (0.112 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 36 h. After following the general work-up process, column chromatography (2% diethyl ether in hexane) provided *2-(cyclohexylmethyl)-5-phenylpentanenitrile* (0.096 g, 94%) as a white solid m.p. 46-48 °C. [Found: (CI corona$^+$) (M+H)$^+$ 256.2060. $C_{18}H_{26}N$ (M+H) requires 256.2060]; ν_{max}(film)/cm^{-1} 3026, 2920, 2850, 2235, 1602, 1496, 1448, 698; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.82-0.95 (2H, m, Cy), 1.12-1.35 (4H, m, Cy), 1.44-1.90 (11H, m, Cy, $\mathrm{CHC}H_2\mathrm{Cy}$, $\mathrm{ArCH_2C}H_2\mathrm{C}H_2\mathrm{CH}$), 2.58-2.65 (1H, m, $\mathrm{C}H\mathrm{CN}$), 2.68 (2H, t, J = 7.2 Hz, $\mathrm{ArC}H_2$), 7.18-7.22 (3H, m, ArH), 7.30 (2H, t, J = 7.6 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.0 (CH_2), 26.1 (CH_2), 26.5 (CH_2), 28.9 (CH_2), 29.0 (CH_2), 32.3 (CH), 32.5 (CH_2), 33.7 (CH_2), 35.4 (CH_2), 35.5 (CH_2), 40.0 (CH), 122.5 (C), 126.1 (CH), 128.5 (CH), 128.6 (CH), 141.5 (C); *m/z* (CI corona$^+$) 256 [(M+H)$^+$, 100%], 249 (8), 95 (9).

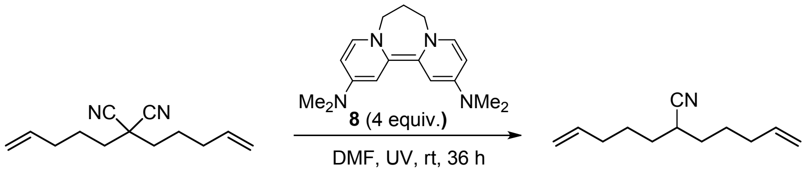
UV-activated reduction of 2,2-bis(cyclohexylmethyl)malononitrile 22



22 (90%)

The general procedure for electron transfer reactions under UV conditions was applied to 2,2-bis(cyclohexylmethyl)malononitrile **22** (0.103 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 36 h. After following the general work-up process, column chromatography (2% diethyl ether in hexane) provided *3-cyclohexyl-2-(cyclohexylmethyl)propanenitrile* (0.0836 g, 90%) as a white solid m.p. 30-32 ^{o}C. [Found: (CI corona$^+$) (M+H)$^+$ 234.2212. $C_{16}H_{28}N$ (M+H) requires 234.2216]; ν_{max}(film)/cm^{-1} 2920, 2846, 2235, 1450, 842; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.82-0.99 (4H, m, Cy), 1.11-1.35 (8H, m, Cy), 1.44-1.58 (4H, m, Cy), 1.65-1.80 (10H, m, Cy, CyCH_2CH), 2.65-2.71 (1H, m, C*H*CN); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.1 (CH_2), 26.2 (CH_2), 26.4 (CH), 26.5 (CH_2), 32.5 (CH_2), 33.7 (CH_2), 35.5 (CH), 40.5 (CH_2), 122.9 (C); *m/z* (CI corona$^+$) 251 [(M+NH$_4$)$^+$, 10%], 234 [(M+H)$^+$, 100%], 195 (7).

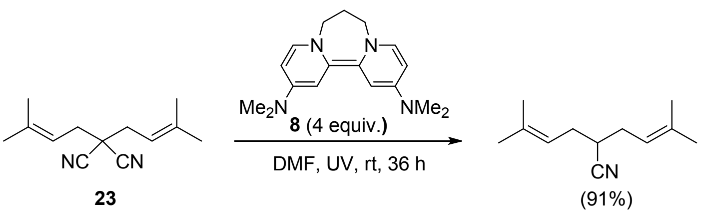
UV-activated reduction of 2,2-di(pent-4-en-1-yl)malononitrile 23



23 (91%)

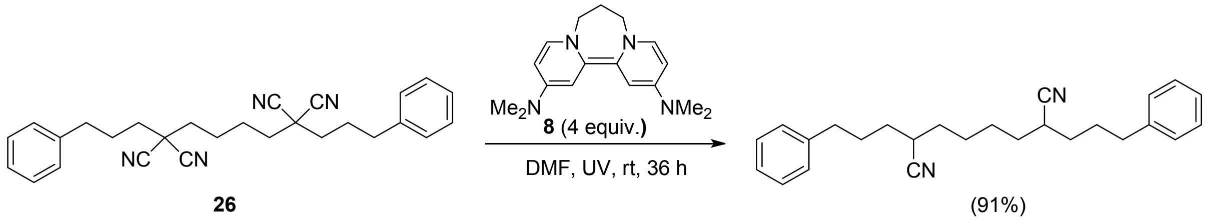
The general procedure for electron transfer reactions under UV conditions was applied to 2,2-bis(cyclohexylmethyl)malononitrile **23** (0.101 g, 0.5 mmol) using the donor **8** (0.568 g, 2.0 mmol, 4 eq.) for 36 h. After following the general work-up process, column chromatography (2% diethyl ether in hexane) provided 2-(pent-4-en-1-yl)hept-6-enenitrile (0.0806 g, 91%) as a colourless oil. [Found: (CI corona$^+$) (M+H)$^+$ 178.1587. $C_{12}H_{20}N$ (M+H) requires 178.1590]; ν_{max}(film)/cm^{-1} 3078, 2931, 2862, 2237, 1641, 1460, 910; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.53-1.69 (8H, m, =CHCH$_2$C*H$_2$*CH$_2$, =CHCH$_2$CH$_2$C*H$_2$*), 2.08-2.13 (4H, m, =CHC*H$_2$*CH$_2$CH$_2$), 2.50-2.57 (1H, m, C*H*CN), 4.98-5.07 (4H, m, C*H$_2$*=CHCH$_2$), 5.74-5.84 (2H, m, CH$_2$=C*H*CH$_2$); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 26.4 (CH_2), 31.5 (CH), 31.7 (CH_2), 33.2 (CH_2), 115.4 (CH_2), 122.3 (C), 137.8 (CH); *m/z* (CI corona$^+$) 195 [$(M+NH_4)^+$, 11%], 178 [$(M+H)^+$, 100%], 117 (14).

UV-activated reduction of 2,2-bis(3-methylbut-2-en-1-yl)malononitrile 25



The general procedure for electron transfer reactions under UV conditions was applied to 2,2-bis(3-methylbut-2-en-1-yl)malononitrile **25** (0.0809 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 36 h. After following the general work-up process, column chromatography (5% ethyl acetate in petroleum ether) provided 5-methyl-2-(3-methylbut-2-en-1-yl)hex-4-enenitrile (0.064 g, 90%) as a colourless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.65 (6H, s, =(CH_3)(CH_3)), 1.75 (6H, s, =(CH_3)(CH_3)), 2.24-2.36 (4H, m, =CHCH_2), 2.48-2.55 (1H, m, C*H*CN), 5.16-5.20 (2H, m, =C*H*CH_2); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 18.1 (CH_3), 25.9 (CH_3), 30.3 (CH_2), 32.5 (CH), 119.3 (CH), 122.4 (C), 135.9 (C).

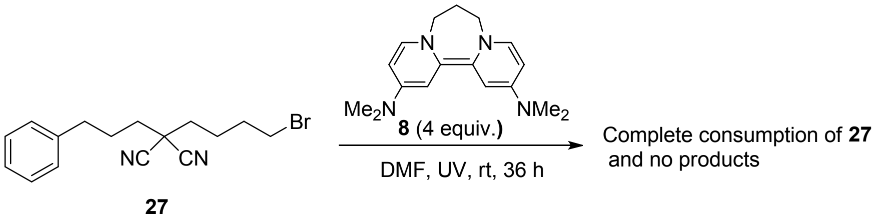
UV-activated reduction 1,12-diphenyldodecane-4,4,9,9-tetracarbonitrile 26



The general procedure for electron transfer reactions under UV conditions was applied to 1,12-diphenyldodecane-4,4,9,9-tetracarbonitrile **26** (0.1266 g, 0.3 mmol) using the donor **8** (0.341 g, 1.2 mmol, 4 eq.) for 36 h. After following the general work-up process, column chromatography (10% ethyl acetate in petroleum ether) provided 2,7-bis(3-phenylpropyl) octanedinitrile (0.102 g, 91%) as a colourless oil. [Found: (CI corona$^+$) $(M+H)^+$ 373.2640. $C_{26}H_{33}N_2$ (M+H) requires 373.2638]; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.41-1.70 (12H, m, HCCH_2CH_2CH_2CH_2CH, ArCH_2C$\mathit{H}_2$$CH_2$CH), 1.72-1.81 (2H, m, Ar$CH_2$$CH_2C\mathit{H}_2$CH), 1.83-1.96 (2H, m, Ar$CH_2$$CH_2C\mathit{H}_2$CH), 2.49-2.55 (2H, m, C*H*CN), 2.67 (4H, t, *J* = 7.2 Hz, ArCH_2), 7.18-7.23 (6H, m, ArH), 7.29-7.33 (4H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.8 (CH_2), 26.9 (CH_2), 28.9 (2 x CH_2), 31.7 (2 x CH), 31.8 (CH_2), 31.84 (CH_2), 32.07

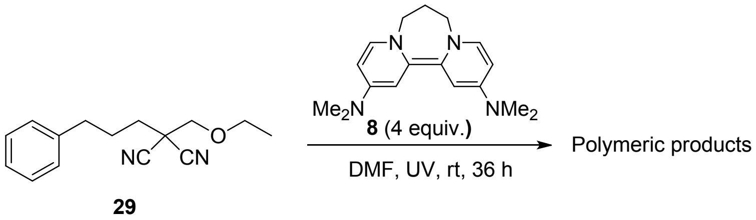
(CH_2), 32.11 (CH_2), 35.4 (2 x CH_2), 122.0 (C), 126.2 (CH), 128.5 (CH), 128.6 (CH), 141.4 (C); *m/z* (CI corona$^+$) 390 [$(M+NH_4)^+$, 44%], 373 [$(M+H)^+$, 83%], 311 (70), 260 (100), 183 (35), 134 (29).

UV-activated reduction of 2-(4-bromobutyl)-2-(3-phenylpropyl)malononitrile 27



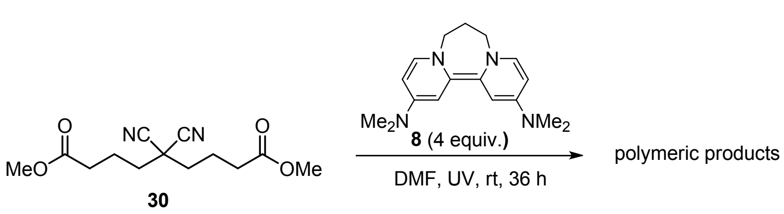
The general procedure for electron transfer reactions under UV conditions was applied to 2-(4-bromobutyl)-2-(3-phenylpropyl)malononitrile **27** (0.159 g, 0.5 mmol) using the donor **8** (0.568 g, 2.0 mmol, 4 eq.) for 36 h. After following the general work-up process, nothing was recovered from the organic phase.

UV-activated reduction of 2-(ethoxymethyl)-2-(3-phenylpropyl)malononitrile 29



The general procedure for electron transfer reactions under UV conditions was applied to 2-(ethoxymethyl)-2-(3-phenylpropyl)malononitrile **29** (0.969 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6 mmol, 4 eq.) for 36 h. After following the general work-up process, crude product was obtained. ^{1}H-NMR of the crude product and TLC showed that the reaction led to a very complex mixture of products. No attempt was made to isolate these products.

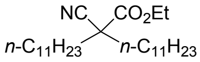
UV-activated reduction of dimethyl 5,5-dicyanononanedioate 30



The general procedure for electron transfer reactions under UV conditions was applied to dimethyl 5,5-dicyanononanedioate **30** (0.106 g, 0.4 mmol) using the donor **8** (0.454 g, 1.6

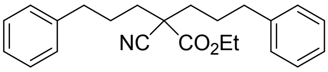
mmol, 4 eq.) for 36 h. After following the general work-up process, crude product was obtained. ^{1}H-NMR of the crude product and TLC showed that the reaction led to a very complex mixture of polymeric products. No attempt was made to isolate these products.

Synthesis of ethyl 2-cyano-2-undecyltridecanoate 31



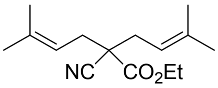
A solution of ethyl 2-cyanoacetate (0.452 g, 4 mmol) in dry tetrahydrofuran (2 mL) was added slowly to a suspension of sodium hydride (~60%, 0.352 g, 8.8 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and 1-bromoundecane (2.069 g, 8.8 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (10% diethyl ether in hexane) to yield *ethyl 2-cyano-2-undecyltridecanoate* **31** (0.896 g, 53%) as a colourless oil. [Found: (ESI$^+$) $(M+NH_4)^+$ 439.4255. $C_{27}H_{55}N_2O_2$ $(M+NH_4)$ requires 439.4258]; ν_{max}(film)/cm^{-1} 2953, 2922, 2852, 2237, 1741, 1463, 1377, 1217, 1022, 721; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.89 [6H, t, J = 7.5 Hz, $(CH_2)_{10}C\mathit{H}_3$], 1.26-1.35 (37H, m, $CH_3(C\mathit{H}_2)_9$, $(C\mathit{H}_2)_8CH_3$, $CO_2CH_2C\mathit{H}_3$), 1.54-1.58 (2H, m, $C\mathit{H}_2(CH_2)_8CH_3$), 1.74-1.79 (2H, m, $CC\mathit{H}_2(CH_2)_9CH_3$), 1.86-1.92 (2H, m, $CC\mathit{H}_2(CH_2)_9CH_3$), 4.27 (2H, q, J = 7.5 Hz, $CO_2C\mathit{H}_2CH_3$); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 14.25 (CH_3), 14.28 (CH_3), 22.8 (CH_2), 25.4 (CH_2), 29.4 (2 x CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (2 x CH_2), 32.1 (CH_2), 37.7 (CH_2), 50.2 (C), 62.6 (CH_2), 119.7 (C), 169.5 (C=O); *m/z* (ESI$^+$) 860 [$(2M+NH_4)^+$, 7%], 439 [$(M+NH_4)^+$, 100%], 351 (16), 268 (28).

Synthesis of ethyl 2-cyano-5-phenyl-2-(3-phenylpropyl)pentanoate 33



A solution of ethyl 2-cyanoacetate (0.565 g, 5 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.440 g, 11 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min. at room temperature and (3-bromopropyl)benzene (2.1898 g, 11 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 24 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (10% diethyl ether in hexane) to yield *ethyl 2-cyano-5-phenyl-2-(3-phenylpropyl)pentanoate* **33** (1.220 g, 70%) as a colourless oil. [Found: (ESI$^+$) (M+NH$_4$)$^+$ 367.2384. $C_{23}H_{31}N_2O_2$ (M+NH$_4$) requires 367.2380]; ν_{max}(film)/cm^{-1} 3026, 2935, 2864, 2240, 1737, 1452, 1219, 1182, 1095, 1018, 698; ^{1}H-NMR (500 MHz, CDCl$_3$) δ 1.29 [3H, t, J = 7.5 Hz, CO$_2$CH$_2$CH_3], 1.59-1.68 (2H, m, CCH$_2$CH_2CH$_2$Ar), 1.77-1.82 (2H, m, CCH_2CH$_2$CH$_2$Ar), 1.87-1.95 (4H, m, CCH$_2$CH_2CH$_2$Ar, CCH_2CH$_2$CH$_2$Ar), 2.65 (4H, t, J = 7.0 Hz, ArCH_2), 4.24 (2H, q, J = 7.0 Hz, CO$_2$CH_2CH$_3$), 7.16 (4H, d, J = 7.0 Hz, ArH), 7.20 (2H, t, J = 7.5 Hz, ArH), 7.29 (4H, t, J = 7.5 Hz, ArH); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 14.2 (CH$_3$), 27.0 (CH$_2$), 35.3 (CH$_2$), 36.9 (CH$_2$), 49.8 (C), 62.7 (CH$_2$), 119.3 (C), 126.2 (CH), 128.4 (CH), 128.6 (CH), 141.0 (C), 169.1 (C=O); *m/z* (ESI$^+$) 367[(M+NH$_4$)$^+$, 100%].

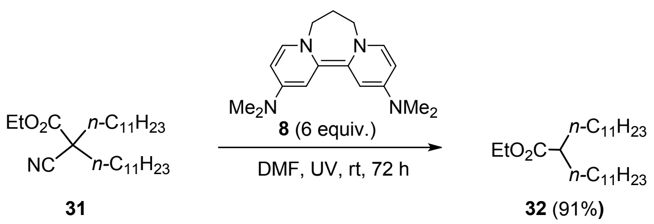
Synthesis of ethyl 2-cyano-5-methyl-2-(3-methylbut-2-en-1-yl)hex-4-enoate 34



A solution of ethyl 2-cyanoacetate (0.678 g, 6 mmol) in dry tetrahydrofuran (2 mL) was added slowly to suspension of sodium hydride (~60%, 0.528 g, 13.2 mmol) in dry tetrahydrofuran (10 mL) at 0 °C under argon gas. The resulting solution was stirred for 15 min at room temperature and 1-bromo-3-methylbut-2-ene (1.967 g, 13.2 mmol) in dry tetrahydrofuran (2 mL) was added slowly into the reaction flask. The reaction mixture was further stirred at reflux conditions for 16 h under argon gas. At this point, the reaction was quenched with water (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined organic phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (5% ethyl acetate in petroleum ether) to yield ethyl 2-cyano-5-methyl-2-(3-methylbut-2-en-1-yl)hex-4-enoate **34** (1.249 g, 83%) as a colourless oil. ^{1}H-NMR (400 MHz, CDCl$_3$) δ 1.30 (3H, t, J = 7.2 Hz, CO$_2$CH$_2$CH_3), 1.66 (6H, s, CH_3), 1.75 (6H, s, CH_3), 2.49-

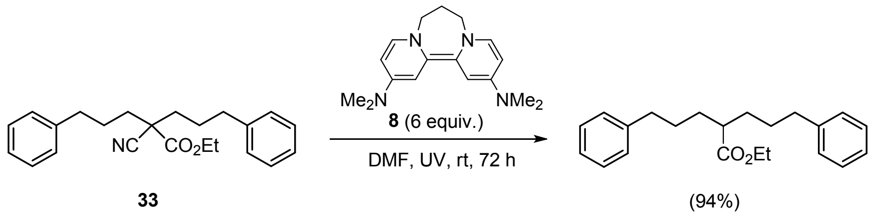
2.55 (2H, m, $=\mathrm{CHC}\mathit{H_2}$), 2.59-2.65 (2H, m, $=\mathrm{CHC}\mathit{H_2}$), 4.23 (2H, q, $J = 7.2$ Hz, $\mathrm{CO_2C}\mathit{H_2}\mathrm{CH_3}$), 5.16-5.21 (2H, m, $=\mathrm{C}\mathit{H}\mathrm{CH_2}$); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.1 (CH_3), 18.1 (CH_3), 25.9 (CH_3), 35.3 (CH_2), 50.0 (C), 62.4 (CH_2), 116.7 (CH), 119.4 (C), 137.6 (C), 168.8 (C).

UV-activated reduction of ethyl 2-cyano-2-undecyltridecanoate 31



The general procedure for electron transfer reactions under UV conditions was applied to ethyl 2-cyano-2-undecyltridecanoate **31** (0.126 g, 0.3 mmol) using the donor **8** (0.511 g, 1.8 mmol, 6 eq.) for 72 h. After following the general work-up process, column chromatography (2% diethyl ether in petroleum ether) provided *ethyl 2-undecyltridecanoate* **32** (0.108 g, 91%) as a colourless oil. [Found: (CI corona$^+$) $(M+H)^+$ 397.4041. $C_{26}H_{53}O_2$ (M+H) requires 397.4040]; $\nu_{\max}$(film)/cm^{-1} 2922, 2852, 1734, 1463, 1377, 1155, 1033, 721; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.89 [6H, t, $J = 7.0$ Hz, $\mathrm{(CH_2)_{10}C}\mathit{H_3}$], 1.24-1.34 (37H, m, $\mathrm{CH_3(C}\mathit{H_2})_9$, $\mathrm{(C}\mathit{H_2})_8\mathrm{CH_3}$, $\mathrm{CO_2CH_2C}\mathit{H_3}$), 1.41-1.46 (2H, m, $\mathrm{C}\mathit{H_2}\mathrm{(CH_2)_8CH_3}$), 1.54-1.62 (4H, m, $\mathrm{CH(C}\mathit{H_2})_2$, 1.28-2.34 (1H, m, $\mathrm{C}\mathit{H}\mathrm{CO_2Et}$), 4.14 (2H, q, $J = 7.0$ Hz, $\mathrm{CO_2C}\mathit{H_2}\mathrm{CH_3}$); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.2 (CH_3), 14.5 (CH_3), 22.8 (CH_2), 27.6 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (2 x CH_2), 29.8 (2 x CH_2), 32.1 (CH_2), 32.7 (CH_2), 45.9 (CH), 60.1 (CH_2), 176.8 (C=O); *m/z* (CI corona$^+$) 397 [$(M+H)^+$, 100%], 327 (16), 243 (23), 229 (31), 201 (32), 155 (23).

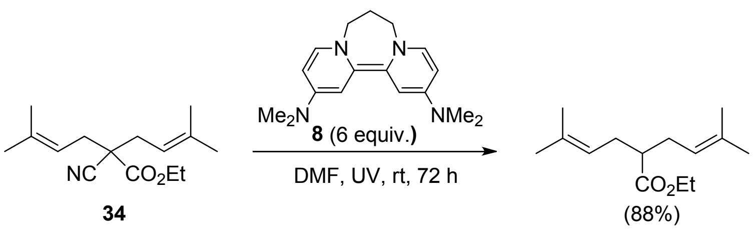
UV-activated reduction of ethyl 2-cyano-5-phenyl-2-(3-phenylpropyl)pentanoate 33



The general procedure for electron transfer reactions under UV conditions was applied to ethyl 2-cyano-5-phenyl-2-(3-phenylpropyl)pentanoate **33** (0.1396 g, 0.4 mmol) using the donor **8** (0.682 g, 2.4 mmol, 6 eq.) for 72 h. After following the general work-up process, column chromatography (5% diethyl ether in hexane) provided *ethyl 5-phenyl-2-(3-*

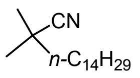
phenylpropyl)pentanoate (0.122 g, 94%) as a colourless oil. [Found: (ESI$^+$) (M+H)$^+$ 325.2169. $C_{22}H_{29}O_2$ (M+H) requires 325.2162]; ν_{max}(film)/cm^{-1} 3024, 2937, 2858, 1728, 1494, 1452, 1184, 1149, 1028, 696; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.24 [3H, t, J = 7.2 Hz, $CO_2CH_2C\mathit{H_3}$], 1.45-1.53 (2H, m, $CCH_2C\mathit{H_2}CH_2Ar$), 1.56-1.69 (6H, m, $CCH_2C\mathit{H_2}CH_2Ar$, $C\mathit{H_2}CH_2CH_2Ar$), 2.35-2.41 (1H, m, $C\mathit{H}CO_2Et$), 2.60 (4H, t, J = 7.2 Hz, $ArC\mathit{H_2}$), 4.13 (2H, q, J = 7.2 Hz, $CO_2C\mathit{H_2}CH_3$), 7.15-7.20 (6H, m, ArH), 7.26-7.30 (4H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.5 (CH_3), 29.3 (CH_2), 32.2 (CH_2), 35.9 (CH_2), 45.6 (CH), 60.2 (CH_2), 125.9 (CH), 128.4 (CH), 128.5 (CH), 142.3 (C), 176.4 (C=O); *m/z* (ESI$^+$) 342 [(M+NH_4)$^+$, 100%], 325 [(M+H)$^+$, 47%], 251 (3), 173 (5).

UV-activated reduction of ethyl 2-cyano-5-methyl-2-(3-methylbut-2-en-1-yl)hex-4-enoate 34



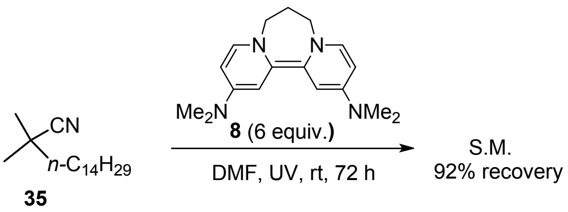
The general procedure for electron transfer reactions under UV conditions was applied to ethyl 2-cyano-5-methyl-2-(3-methylbut-2-en-1-yl)hex-4-enoate **34** (0.099 g, 0.4 mmol) using the donor **8** (0.682 g, 2.4 mmol, 6 eq.) for 72 h. After following the general work-up process, column chromatography (5% ethyl acetate in petroleum ether) provided ethyl 5-methyl-2-(3-methylbut-2-en-1-yl)hex-4-enoate (0.064 g, 90%) as a colourless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.24 (3H, t, J = 7.2 Hz, $CO_2CH_2C\mathit{H_3}$), 1.61 (6H, s, =$(CH_3)(C\mathit{H_3})$), 1.69 (6H, s, =$(C\mathit{H_3})(CH_3)$), 2.15-2.21 (2H, m, =$CHC\mathit{H_2}$), 2.26-2.39 (3H, m, $C\mathit{H}CN$, =$CHC\mathit{H_2}$), 4.12 (2H, q, J = 7.2 Hz, $CO_2C\mathit{H_2}CH_3$), 5.06-5.10 (2H, m, =$C\mathit{H}CH_2$); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 14.4 (CH_3), 17.9 (CH_3), 25.9 (CH_3), 30.5 (CH_2), 46.4 (CH), 60.2 (CH_2), 121.5 (CH), 133.6 (CH), 175.9 (C=O).

Synthesis of 2,2-dimethylhexadecanenitrile 35



Lithium diisopropylamide (1.5 M in THF, 8 mL, 12 mmol) was added dropwise into a flask containing isobutyronitrile (0.691 g, 10 mmol) in dry THF (20 mL) at -78 ^{o}C under argon gas. The reaction contents were stirred for 1 h before adding 1-bromotetradecane dropwise into the flask at -78 °C. Later, the reaction contents were allowed to warm to room temperature and stirred for 16 h under argon gas. At this point, the reaction was quenched with sat. NH_4Cl (15 mL) and extracted with diethyl ether (3 x 15 mL). The combined ether phases were washed once again with water (10 mL), brine solution (10 mL) and dried over anhydrous sodium sulfate. The concentrated solution was purified by column chromatography (5% diethyl ether in petroleum ether) to yield 2,2-dimethylhexadecanenitrile **35** (2.005 g, 76%) as a white solid m.p. 30-32 °C. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.89 (3H, t, J = 6.8 Hz, $C_{13}H_{26}CH_3$), 1.27-1.31 (22H, m, $CH_2CH_2(CH_2)_{11}CH_3$), 1.34 (6H, s, $C(CH_3)_2$); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.2 (CH_3), 22.8 (CH_2), 25.4 (CH_2), 26.8 (2 x CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (CH_2), 29.77 (5 x CH_2), 29.81 (2 x CH_2), 32.1 (CH_2), 32.5 (CH_2), 115.7 (C); *m/z* (CI corona$^+$) 203 [$(M+H)^+$, 100%], 178 (16).

UV-activated reduction of 2,2-dimethylhexadecanenitrile 35



The general procedure for electron transfer reactions under UV conditions was applied to 2,2-dimethylhexadecanenitrile **35** (0.106 g, 0.4 mmol) using the donor **8** (0.682 g, 2.4 mmol, 6 eq.) for 72 h. After following the general work-up process, the reaction provided the recovery of starting material **35** (0.098 g, 92%) only.

References

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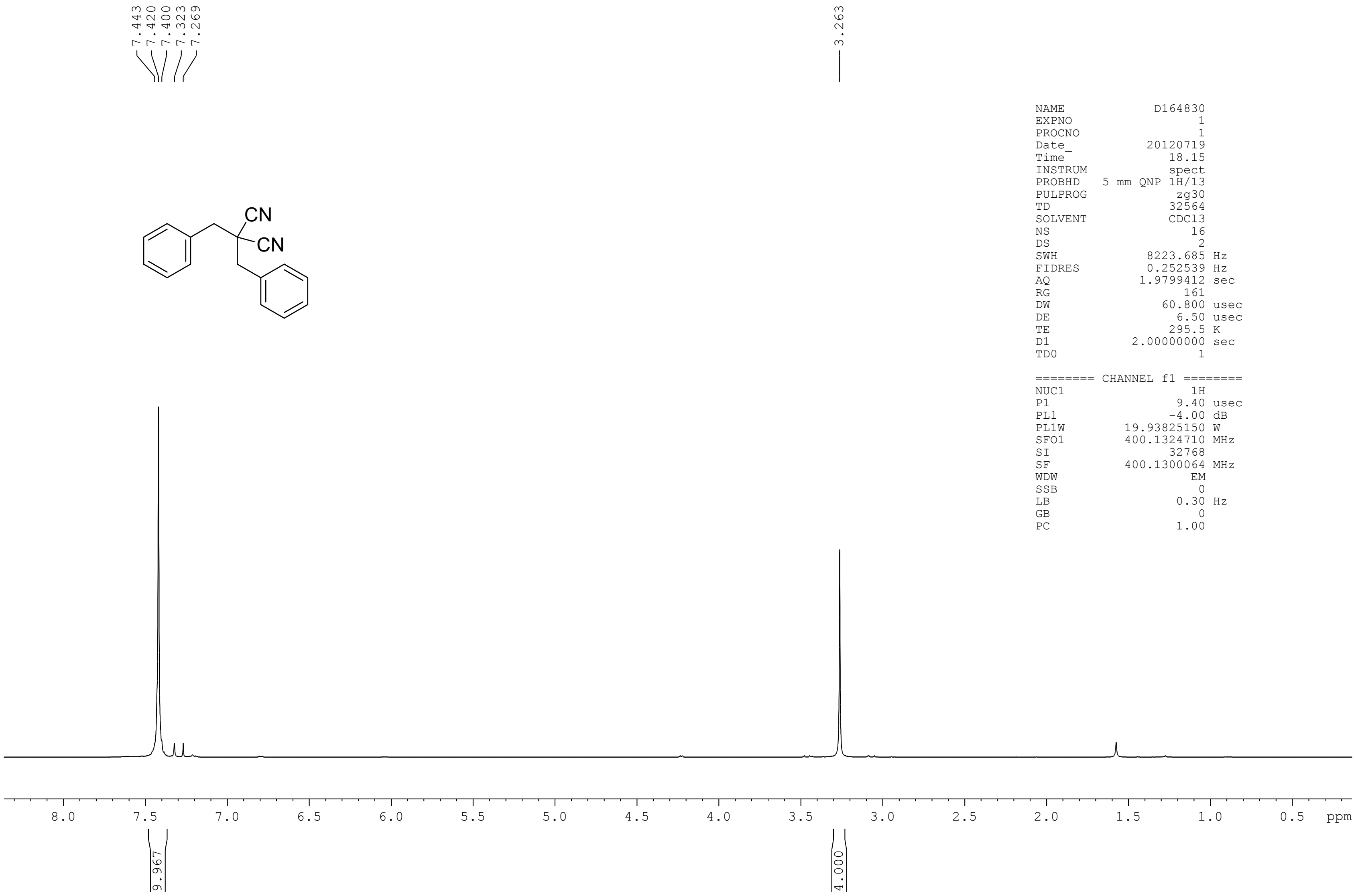

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TD0                   1

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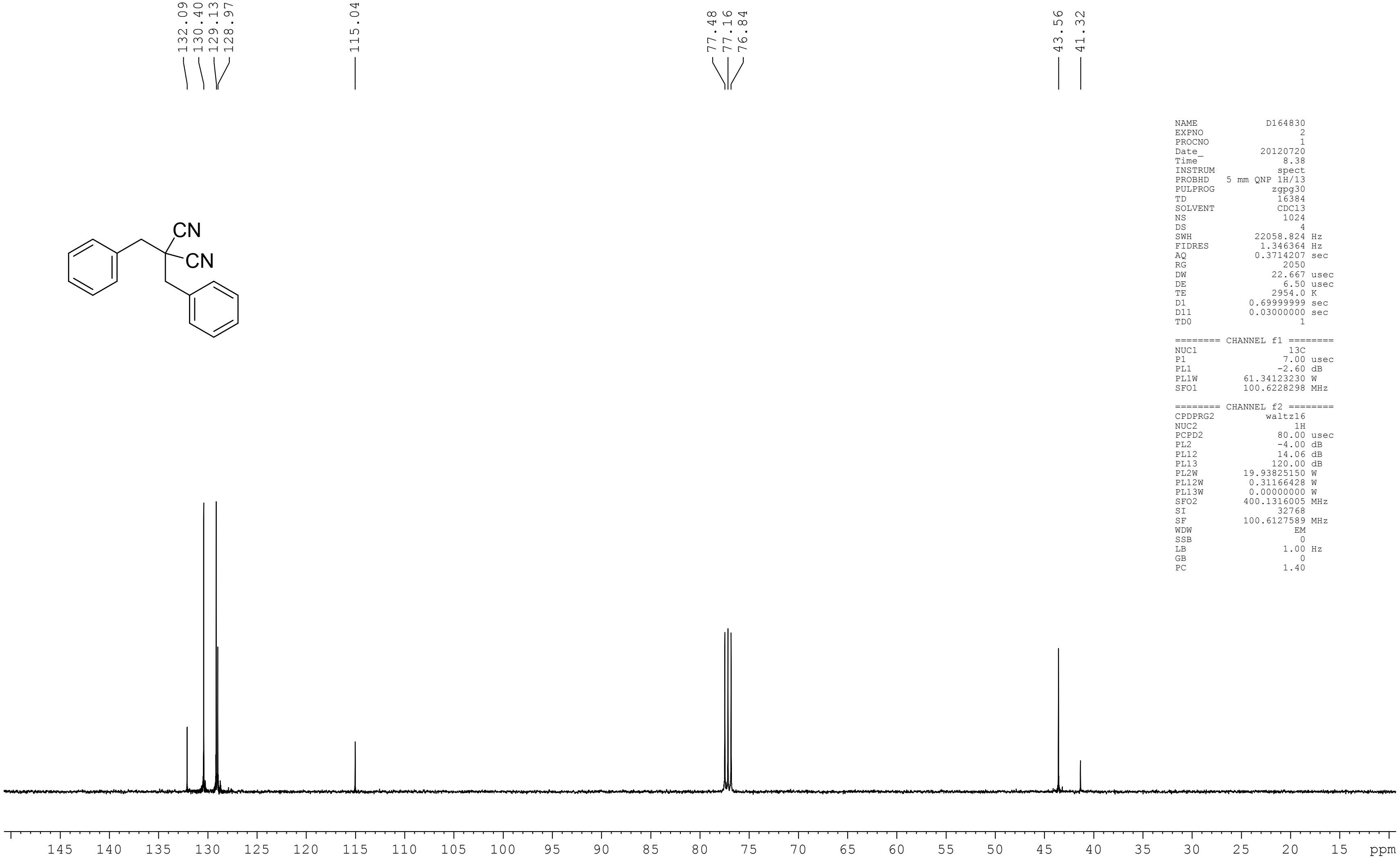

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PL13W        0.00000000 W
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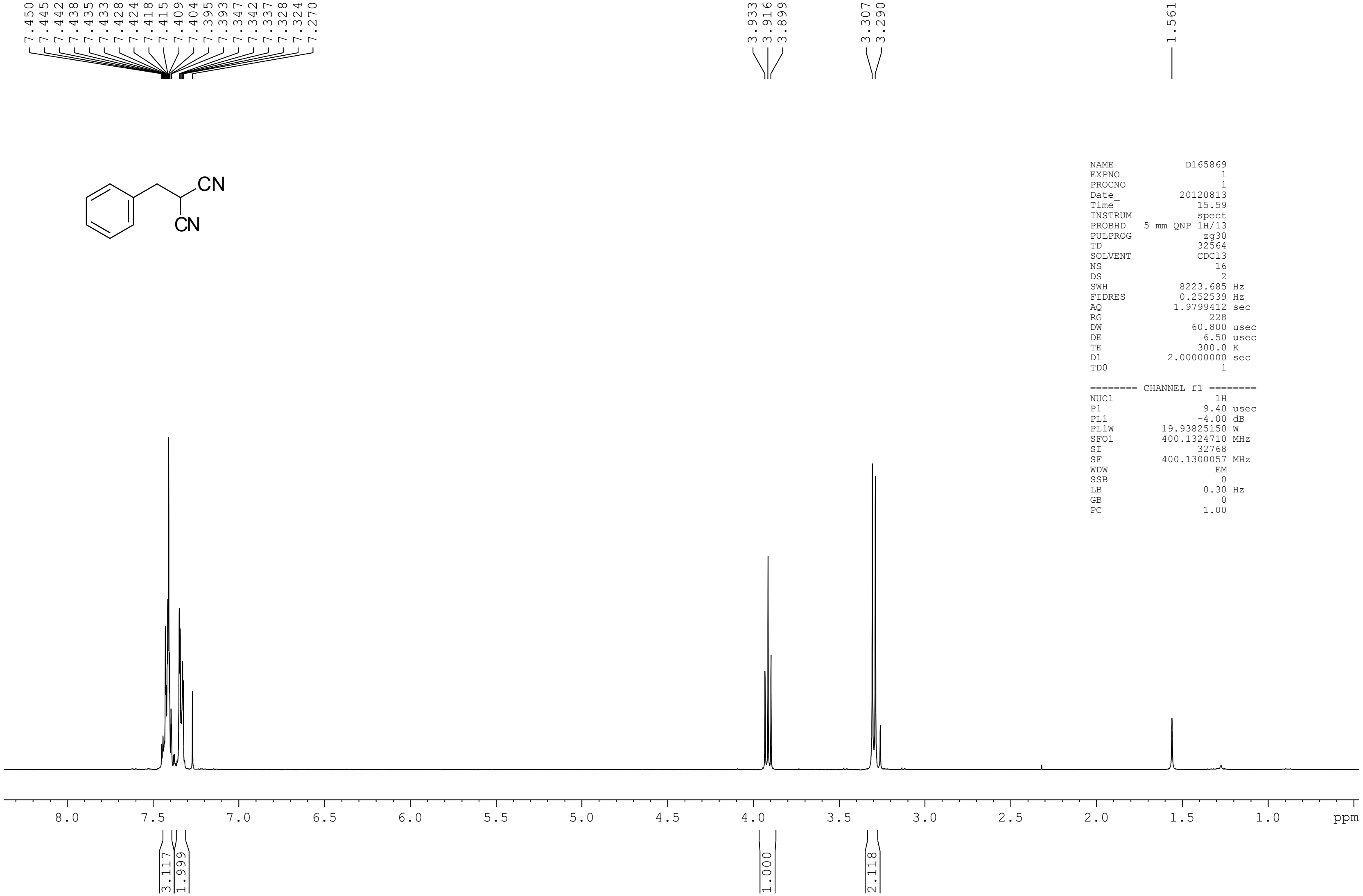

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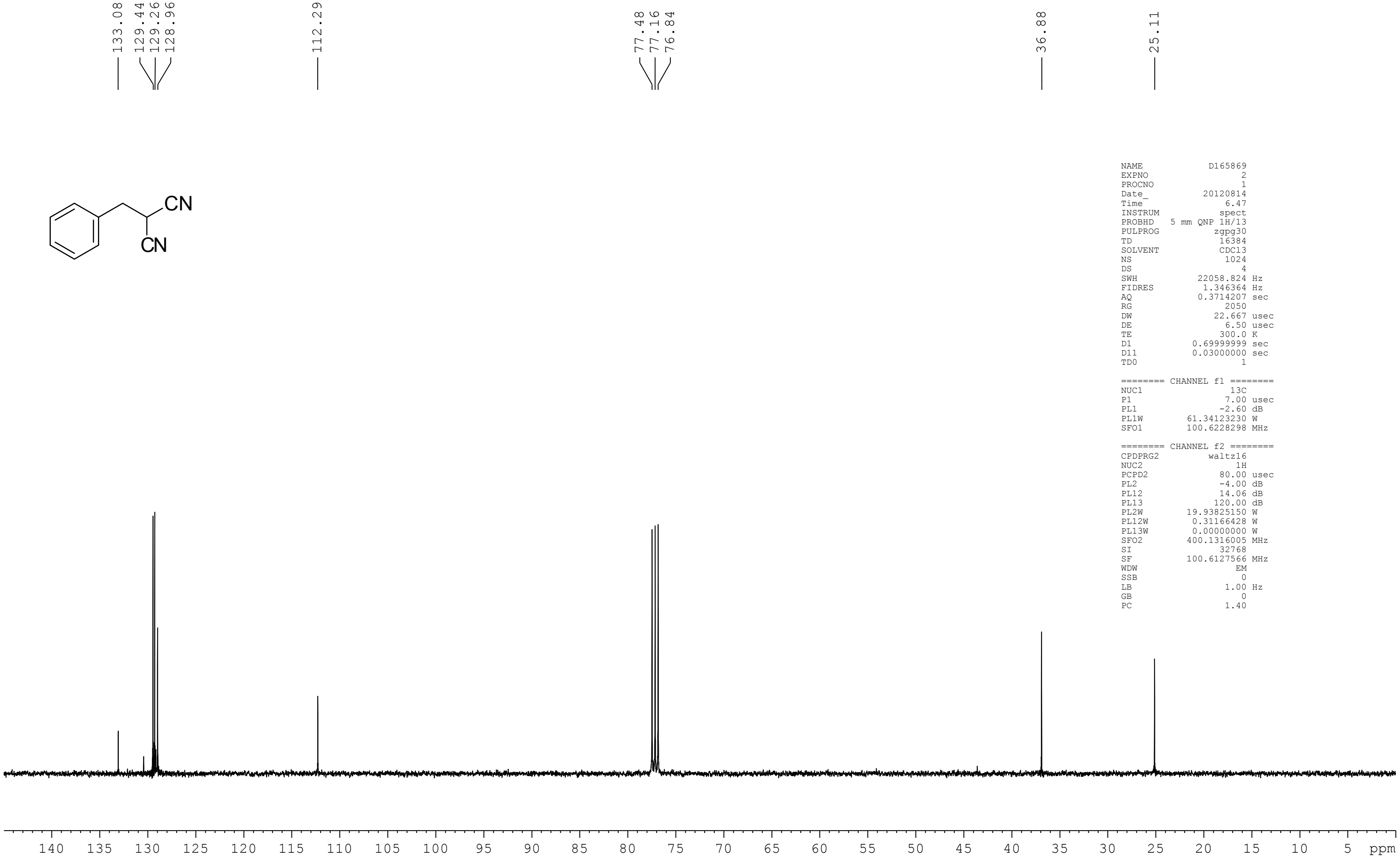
TD0                    1

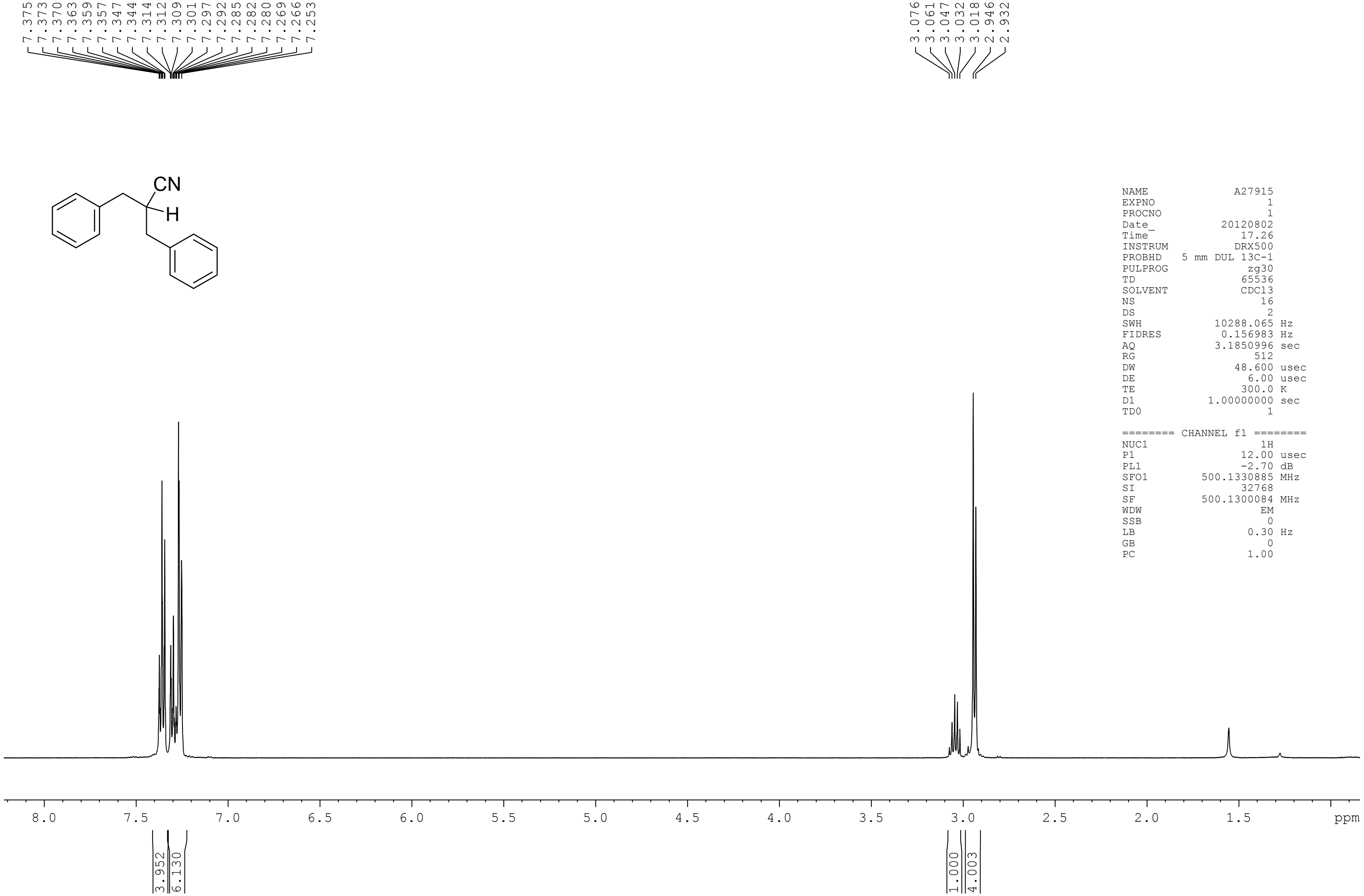
======== CHANNEL f1 ========
NUC1                  1H
P1                  9.40 usec
PL1                -4.00 dB
PL1W         19.93825150 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300057 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
NAME            D165869
EXPNO                 2
PROCNO                1
Date_          20120814
Time               6.47
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

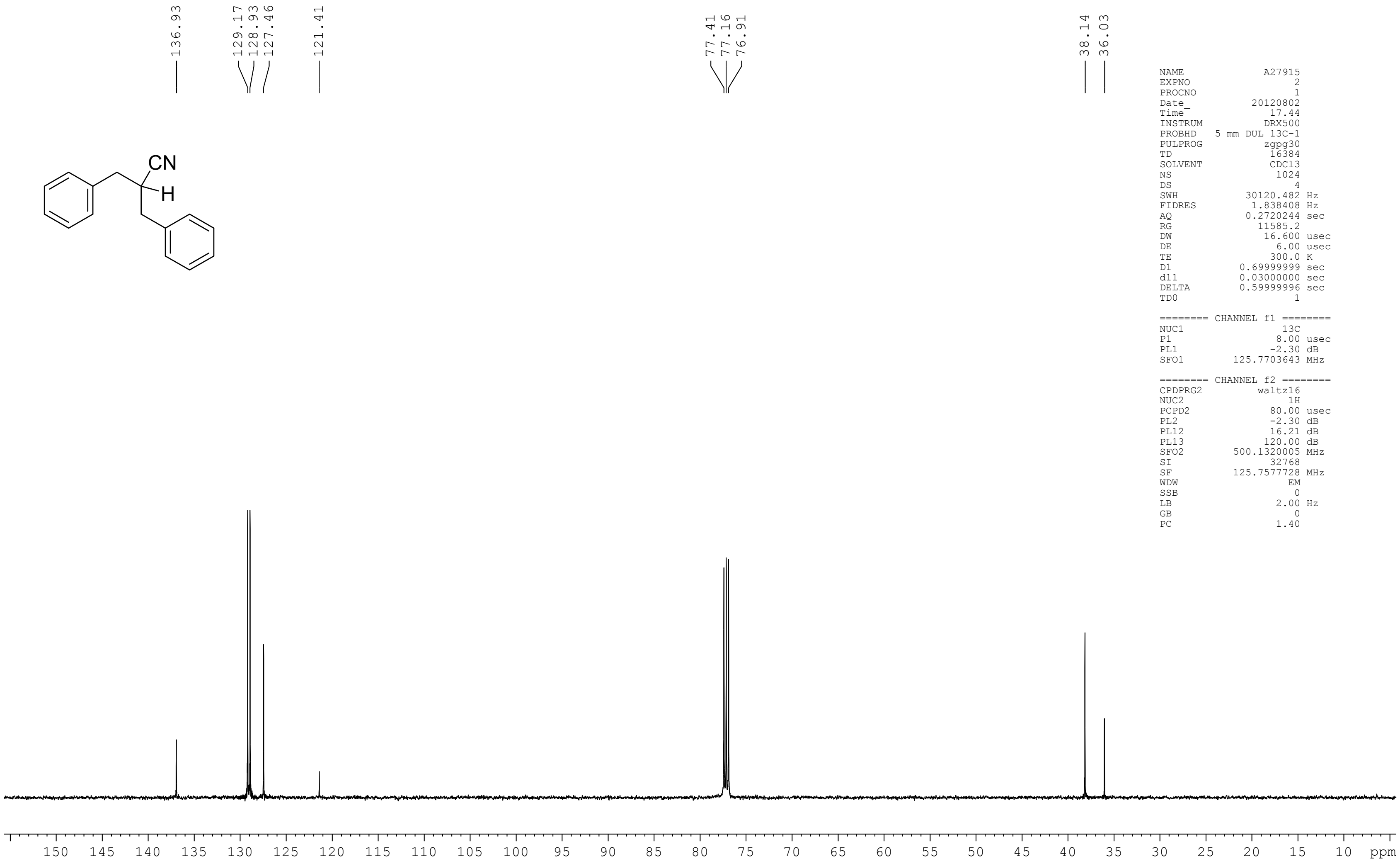
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127566 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME             A27915
EXPNO                 1
PROCNO                1
Date_          20120802
Time              17.26
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10288.065 Hz
FIDRES         0.156983 Hz
AQ            3.1850996 sec
RG                  512
DW               48.600 usec
DE                 6.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                   1

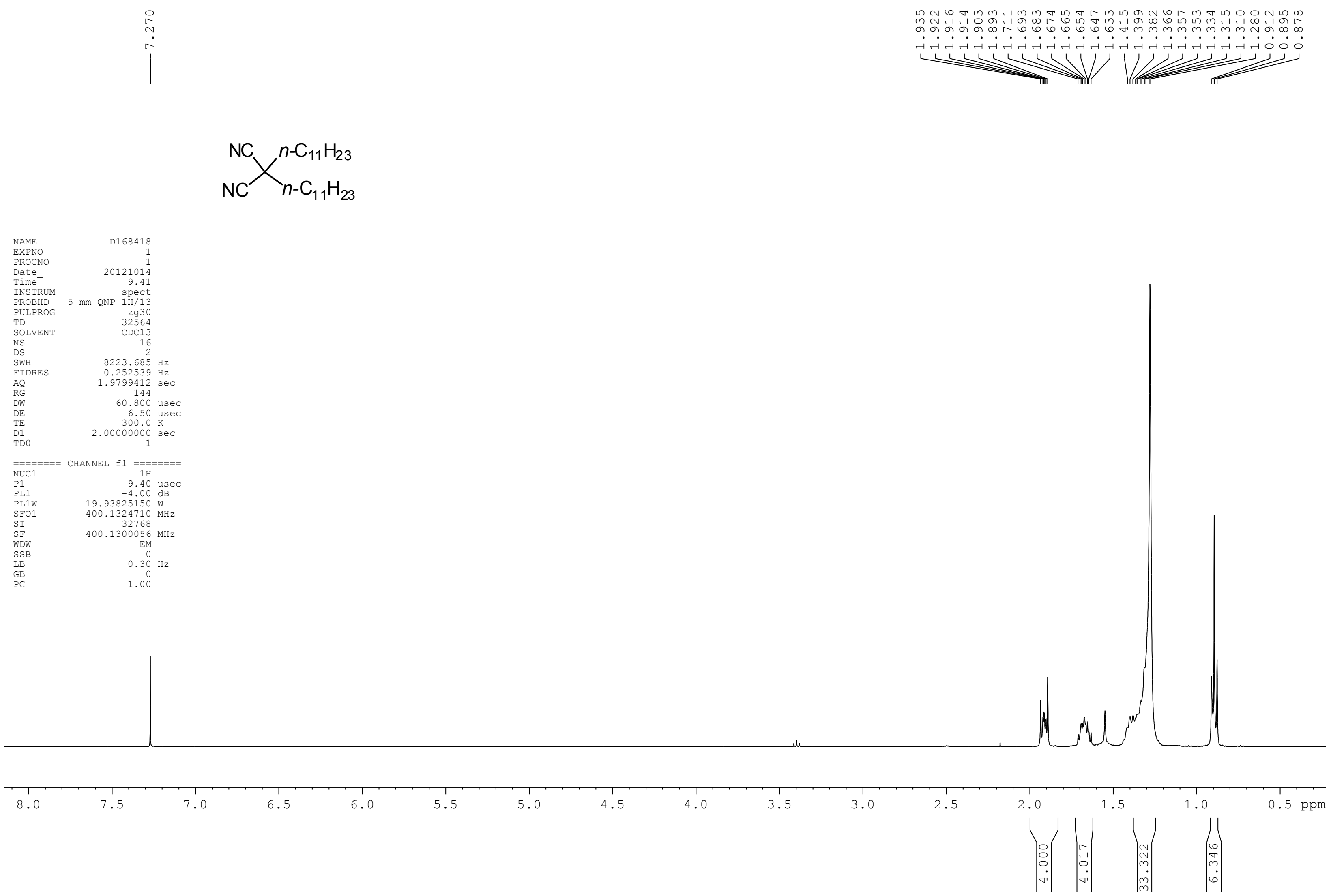
======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1               -2.70 dB
SFO1        500.1330885 MHz
SI                32768
SF          500.1300084 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME             A27915
EXPNO                 2
PROCNO                1
Date_          20120802
Time              17.44
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         1.838408 Hz
AQ            0.2720244 sec
RG              11585.2
DW               16.600 usec
DE                 6.00 usec
TE                300.0 K
D1           0.69999999 sec
d11          0.03000000 sec
DELTA        0.59999996 sec
TD0                   1

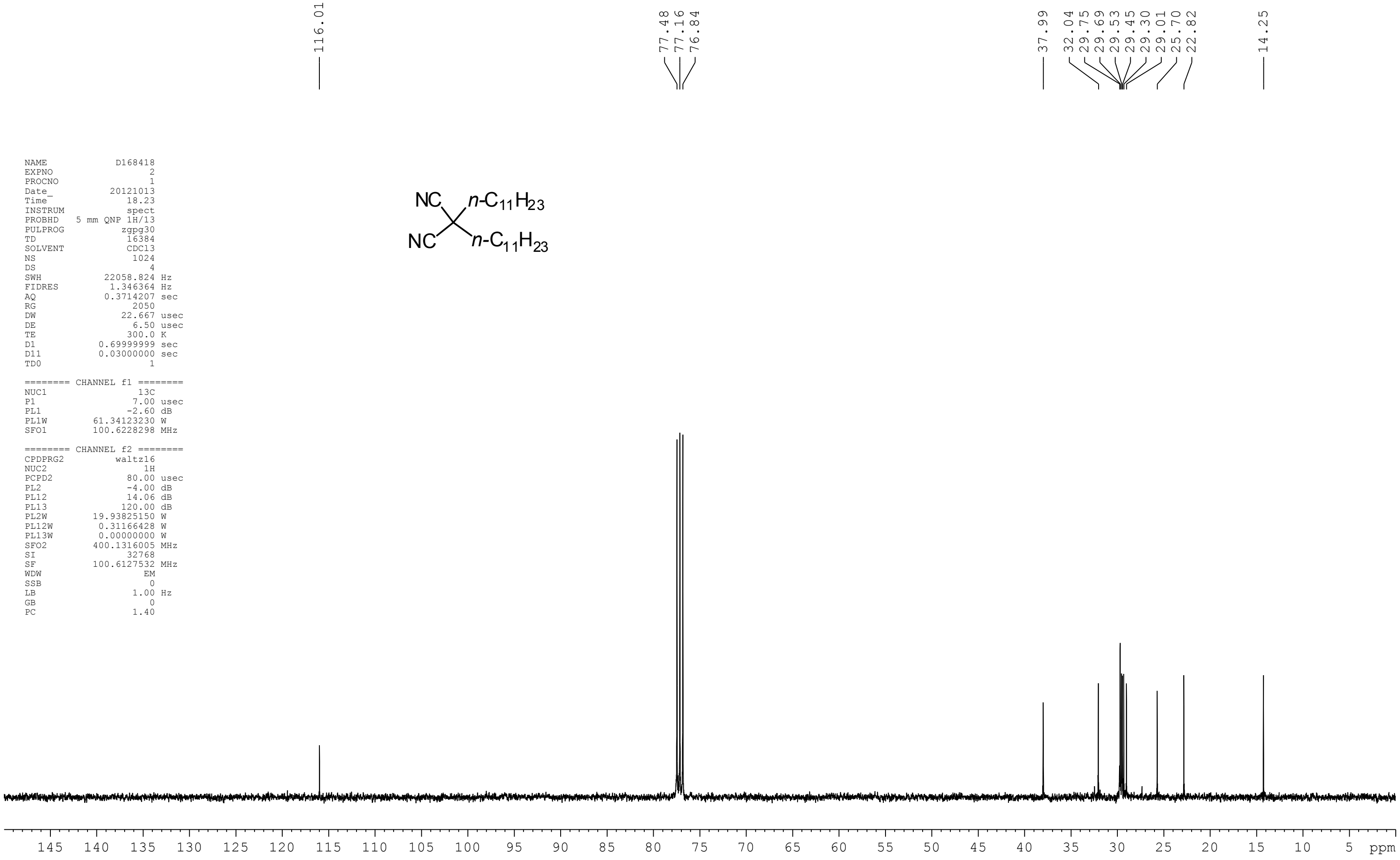
======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1               -2.30 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.30 dB
PL12              16.21 dB
PL13             120.00 dB
SFO2        500.1320005 MHz
SI                32768
SF          125.7577728 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D168418
EXPNO                 1
PROCNO                1
Date_          20121014
Time               9.41
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  144
DW               60.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

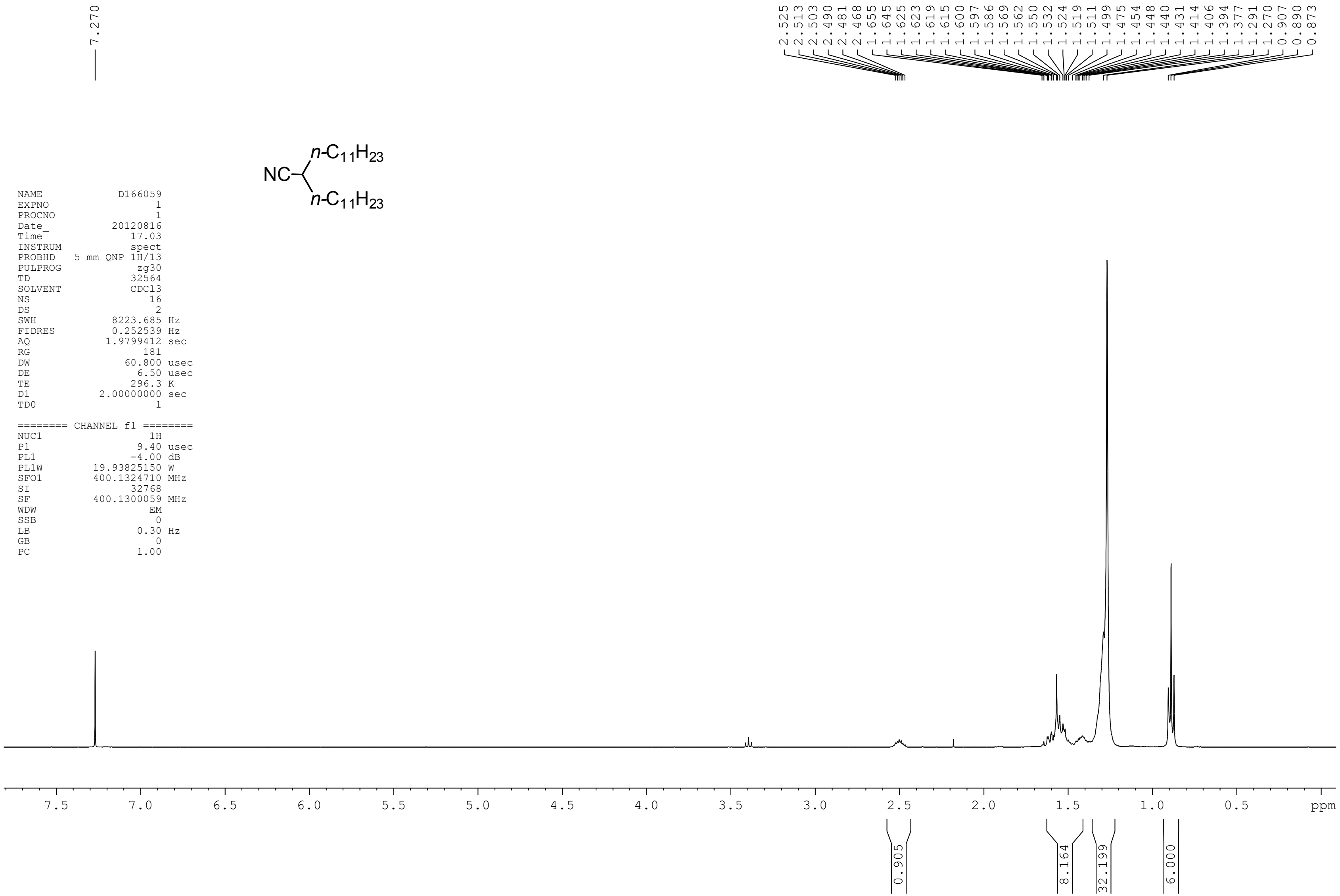
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300056 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME            D168418
EXPNO                 2
PROCNO                1
Date_          20121013
Time              18.23
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

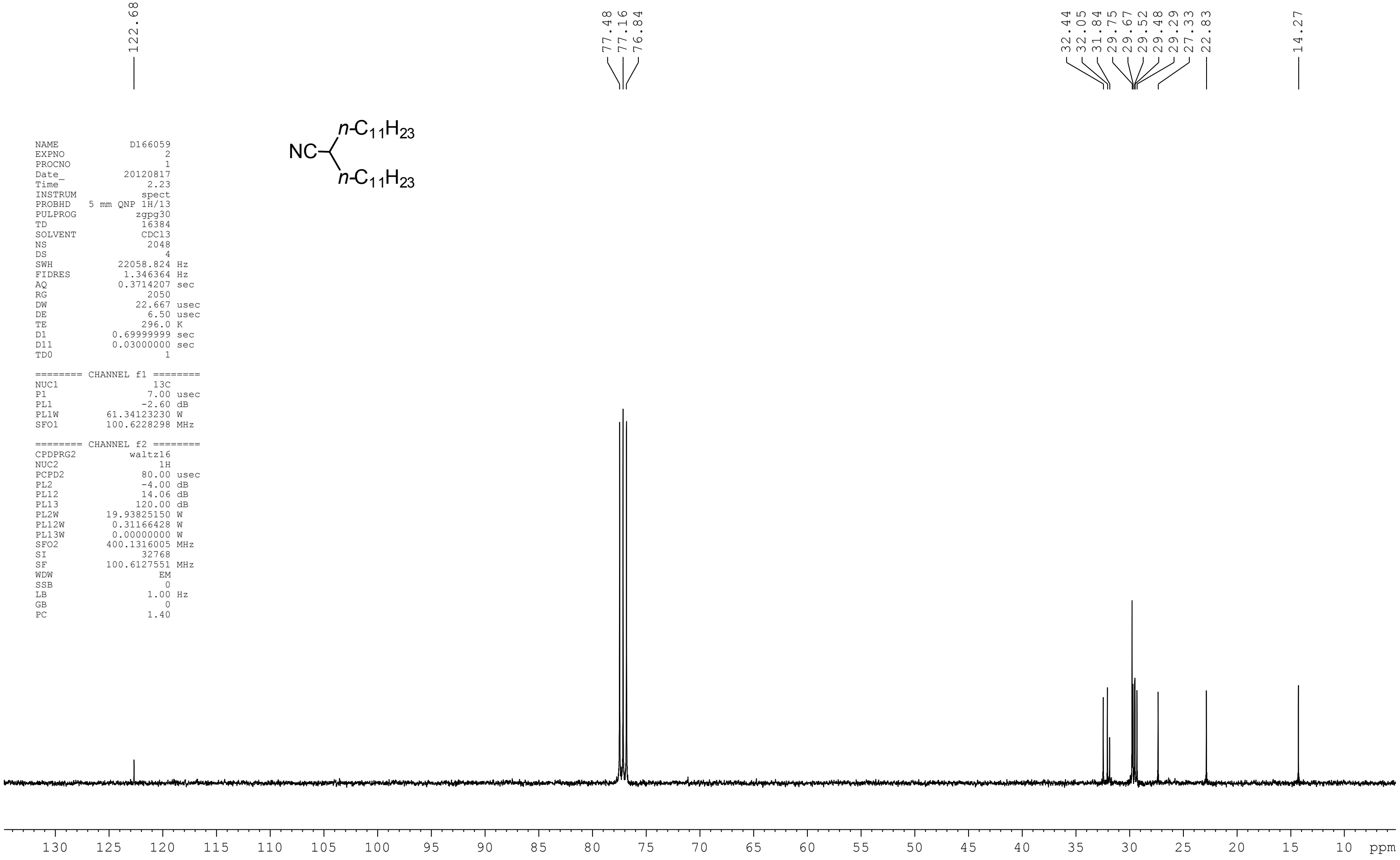
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127532 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D166059
EXPNO                 1
PROCNO                1
Date_          20120816
Time              17.03
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  181
DW               60.800 usec
DE                 6.50 usec
TE                296.3 K
D1           2.00000000 sec
TD0                   1

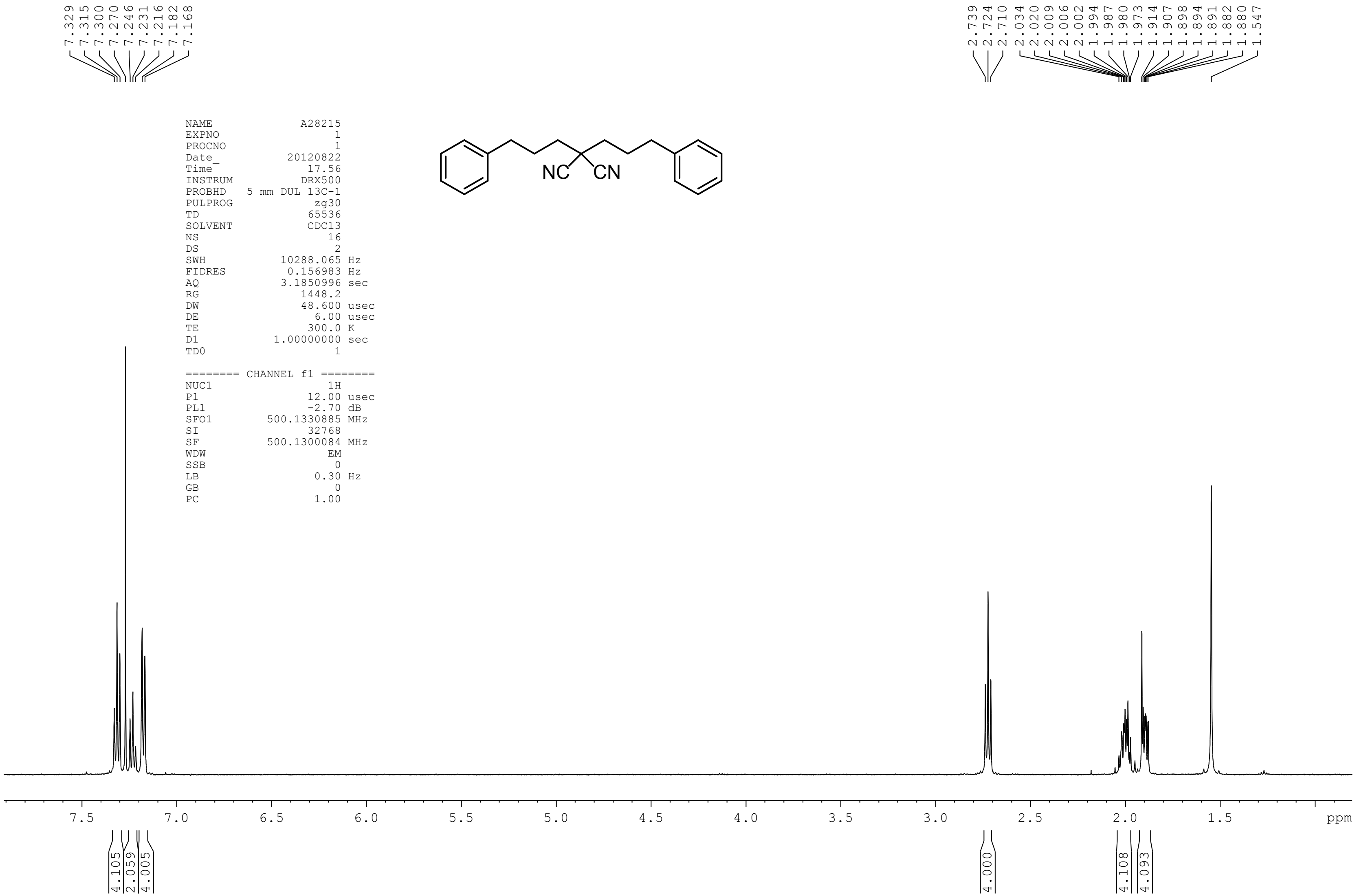
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300059 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME             D166059
EXPNO                  2
PROCNO                 1
Date_           20120817
Time                2.23
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 16384
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            22058.824 Hz
FIDRES          1.346364 Hz
AQ             0.3714207 sec
RG                  2050
DW                22.667 usec
DE                  6.50 usec
TE                 296.0 K
D1            0.69999999 sec
D11           0.03000000 sec
TD0                    1

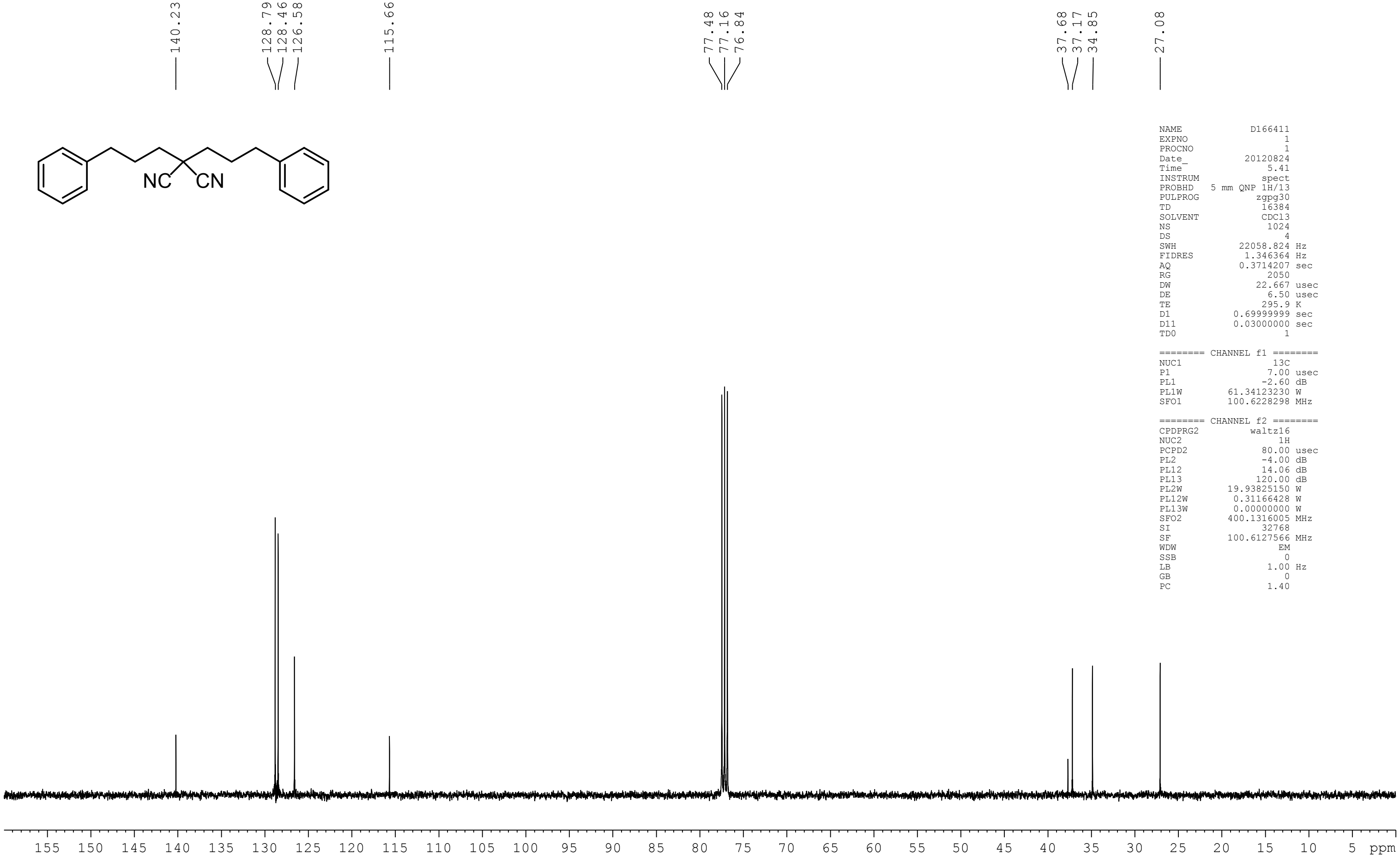
======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.60 dB
PL1W         61.34123230 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.06 dB
PL13              120.00 dB
PL2W         19.93825150 W
PL12W         0.31166428 W
PL13W         0.00000000 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127551 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



```
NAME              A28215
EXPNO                  1
PROCNO                 1
Date_           20120822
Time               17.56
INSTRUM           DRX500
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10288.065 Hz
FIDRES          0.156983 Hz
AQ             3.1850996 sec
RG                1448.2
DW                48.600 usec
DE                  6.00 usec
TE                 300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.70 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300084 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



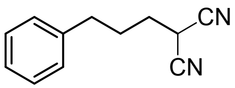
```
NAME            D166411
EXPNO                 1
PROCNO                1
Date_          20120824
Time               5.41
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                295.9 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

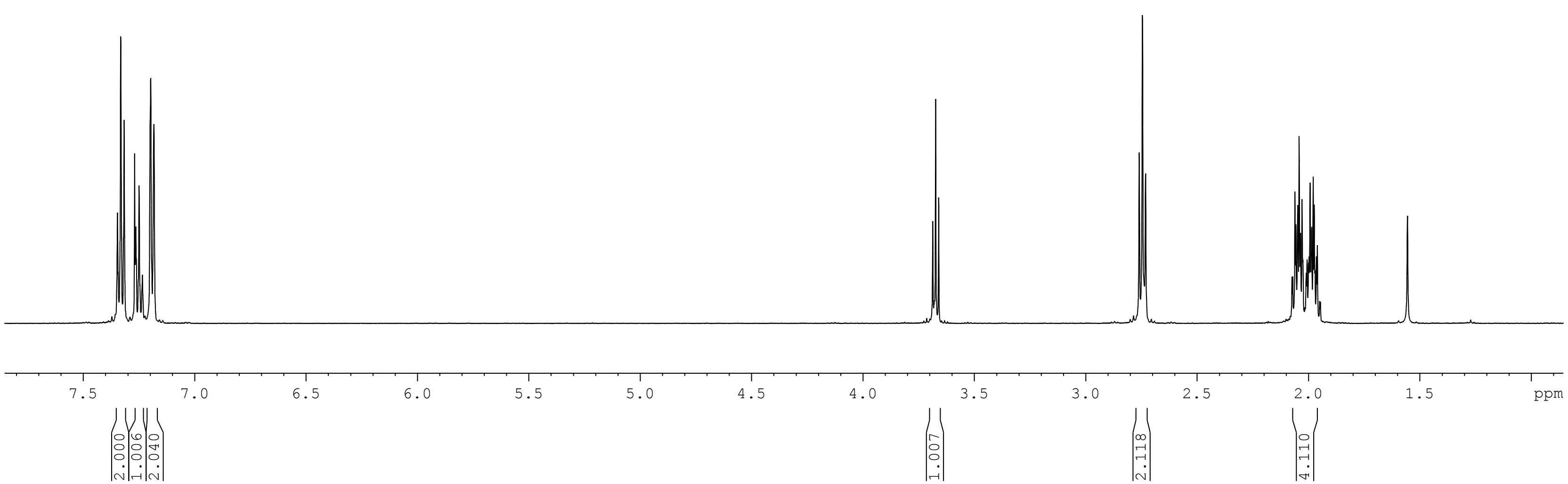
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

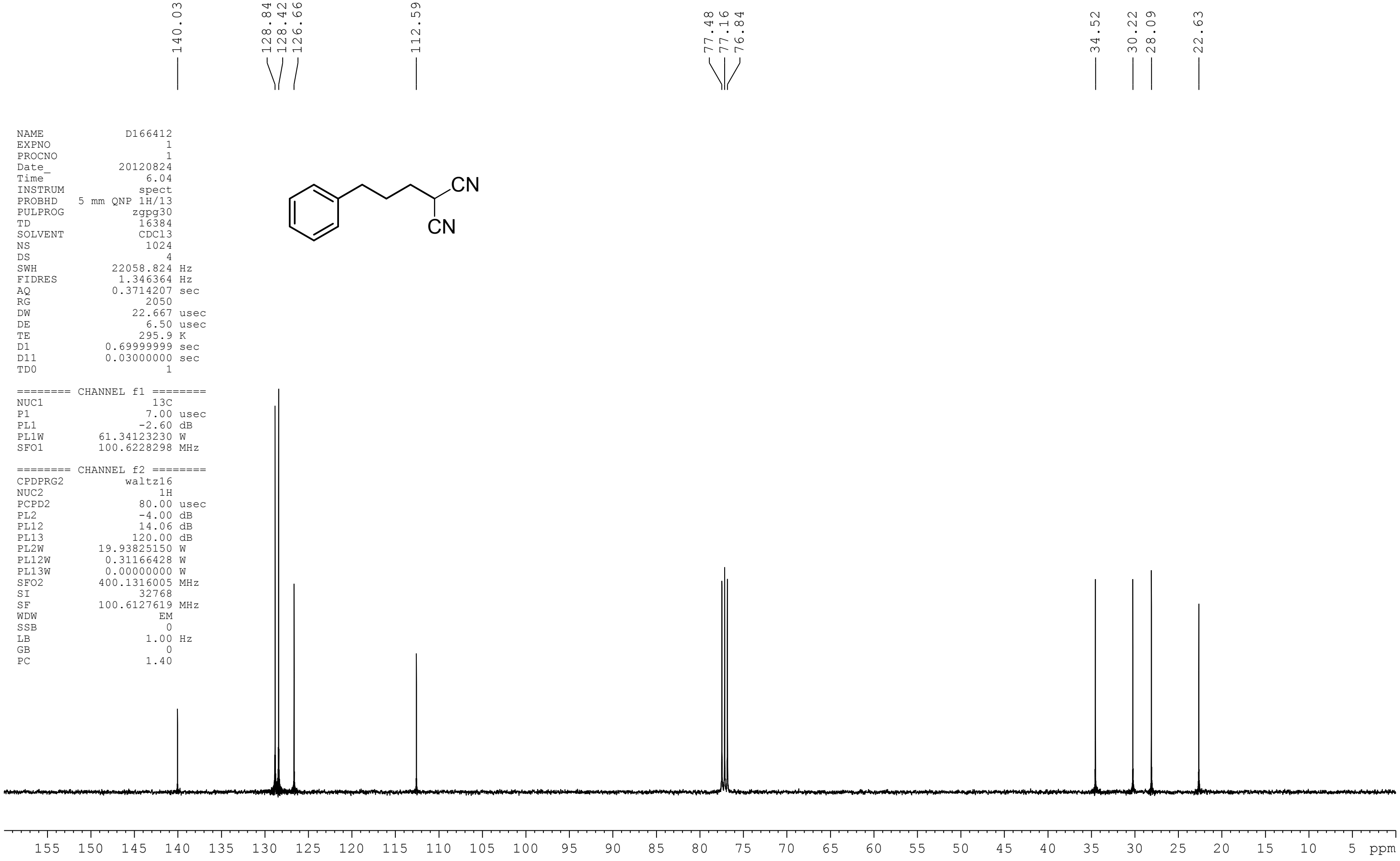
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127566 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

```
NAME             A28216
EXPNO                 1
PROCNO                1
Date_          20120822
Time              18.05
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10288.065 Hz
FIDRES         0.156983 Hz
AQ            3.1850996 sec
RG                574.7
DW               48.600 usec
DE                 6.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1               -2.70 dB
SFO1        500.1330885 MHz
SI                32768
SF          500.1300084 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



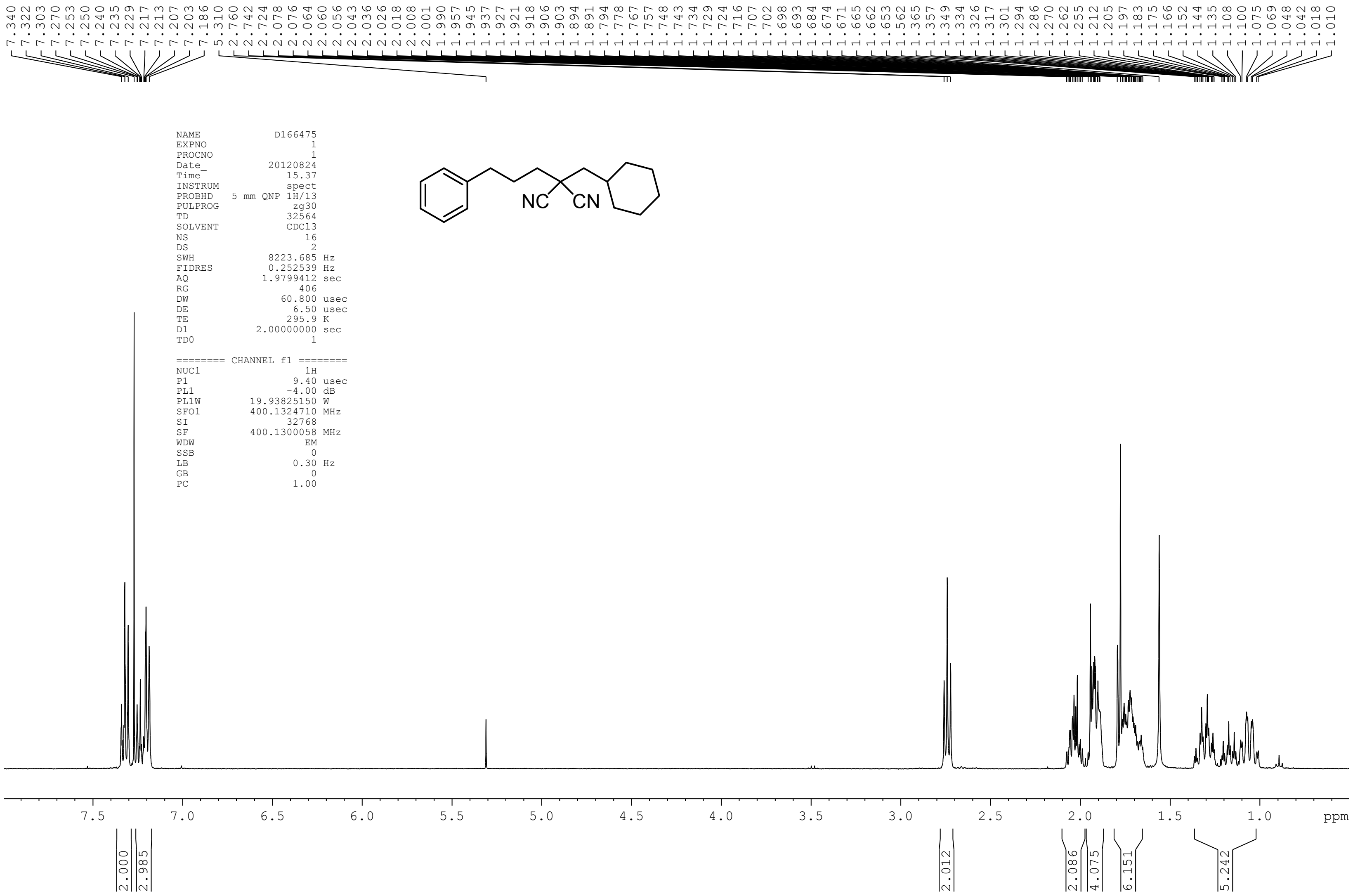




```
NAME            D166412
EXPNO                 1
PROCNO                1
Date_          20120824
Time               6.04
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                295.9 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

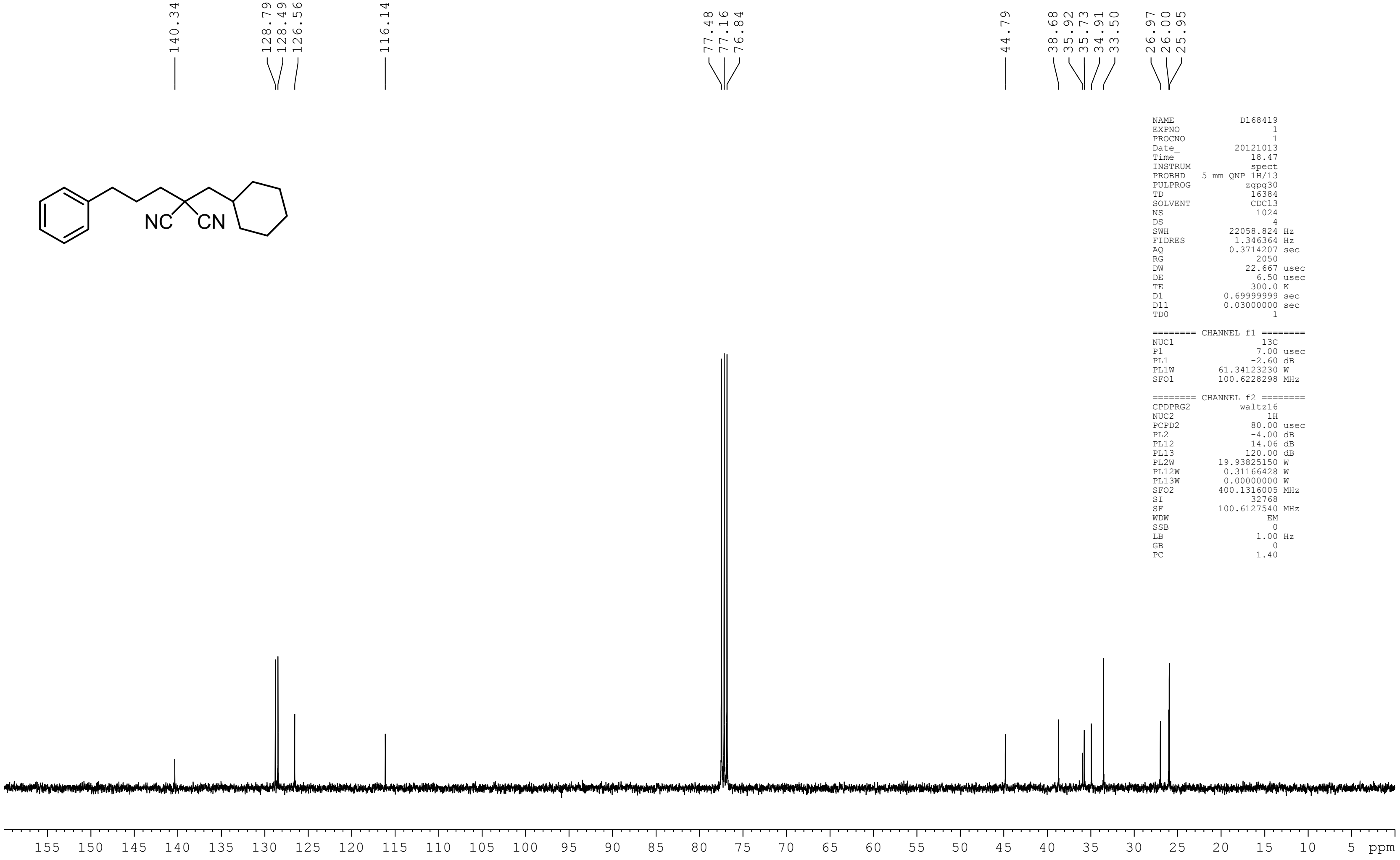
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127619 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D166475
EXPNO                 1
PROCNO                1
Date_          20120824
Time              15.37
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  406
DW               60.800 usec
DE                 6.50 usec
TE                295.9 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300058 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



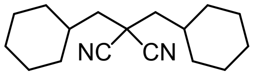
```
NAME            D168419
EXPNO                 1
PROCNO                1
Date_          20121013
Time              18.47
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

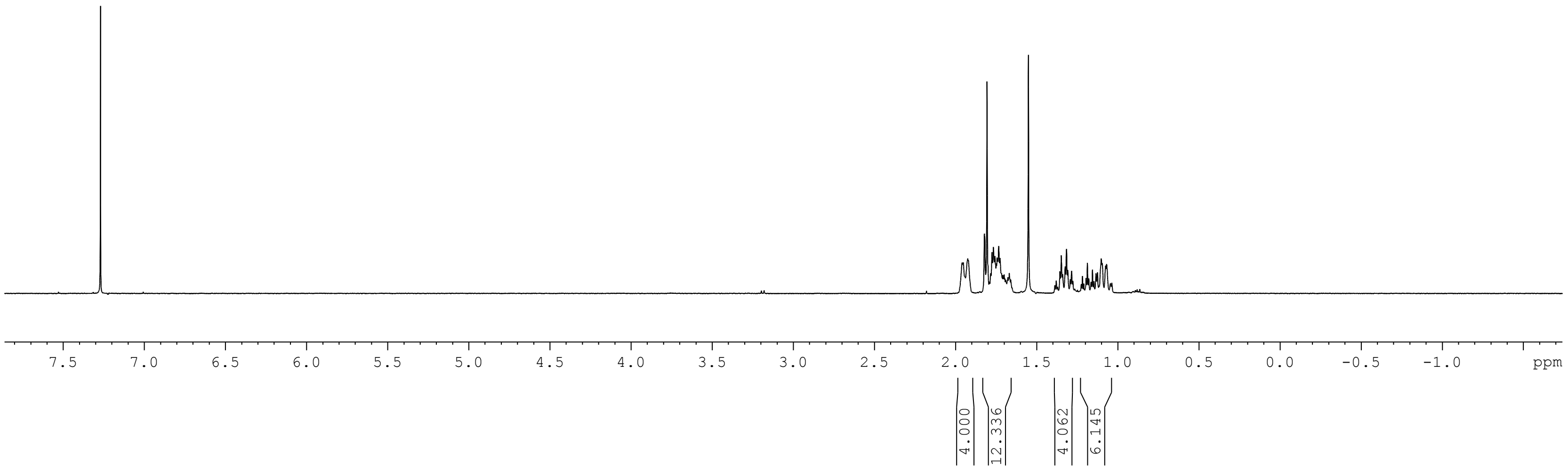
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

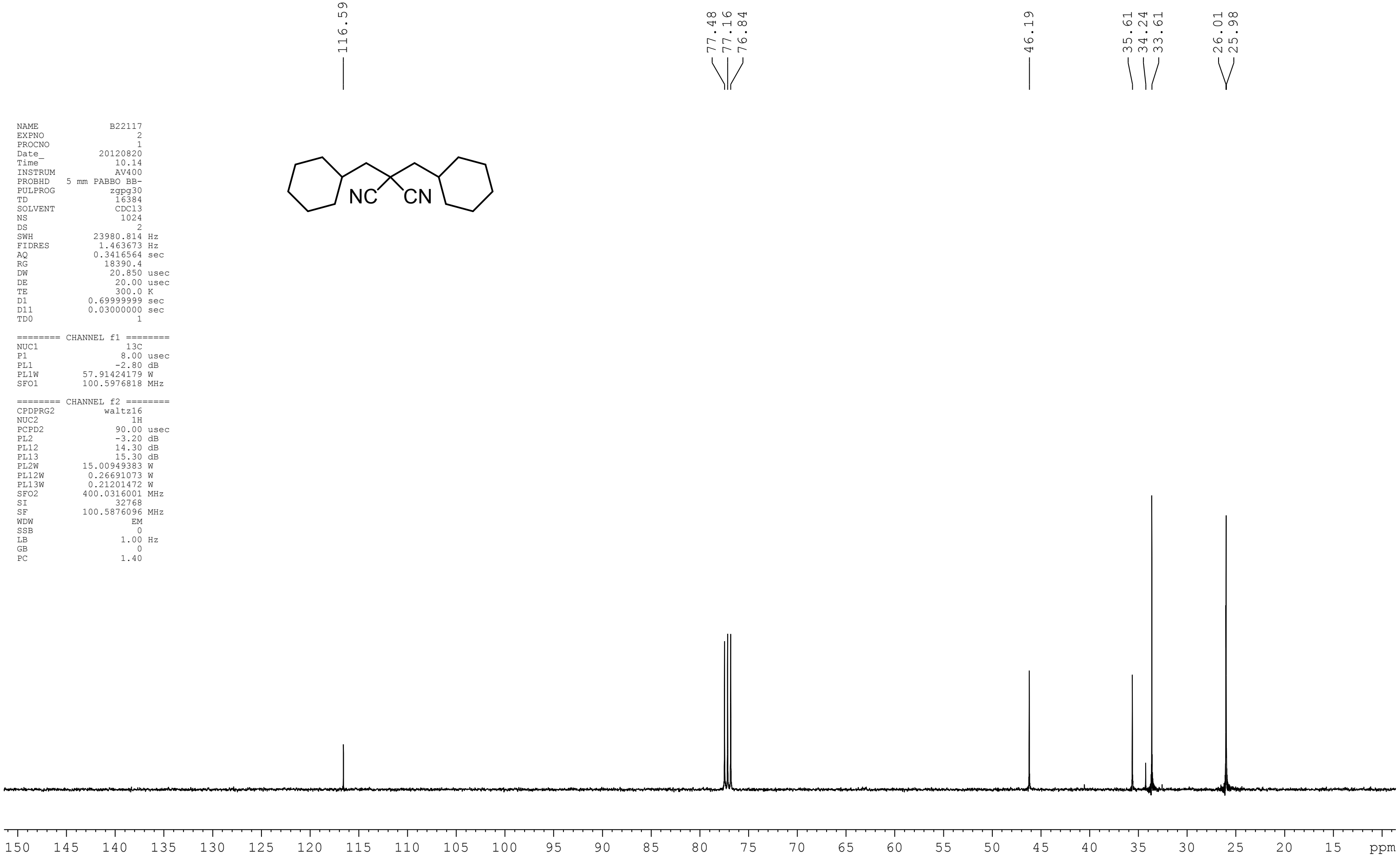
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127540 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

```
NAME              B22115
EXPNO                  1
PROCNO                 1
Date_           20120819
Time               13.02
INSTRUM            AV400
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.252629 Hz
AQ             1.9792372 sec
RG                 574.7
DW                60.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.50 dB
PL1W         12.77515030 W
SFO1         400.0324703 MHz
SI                 32768
SF           400.0300028 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



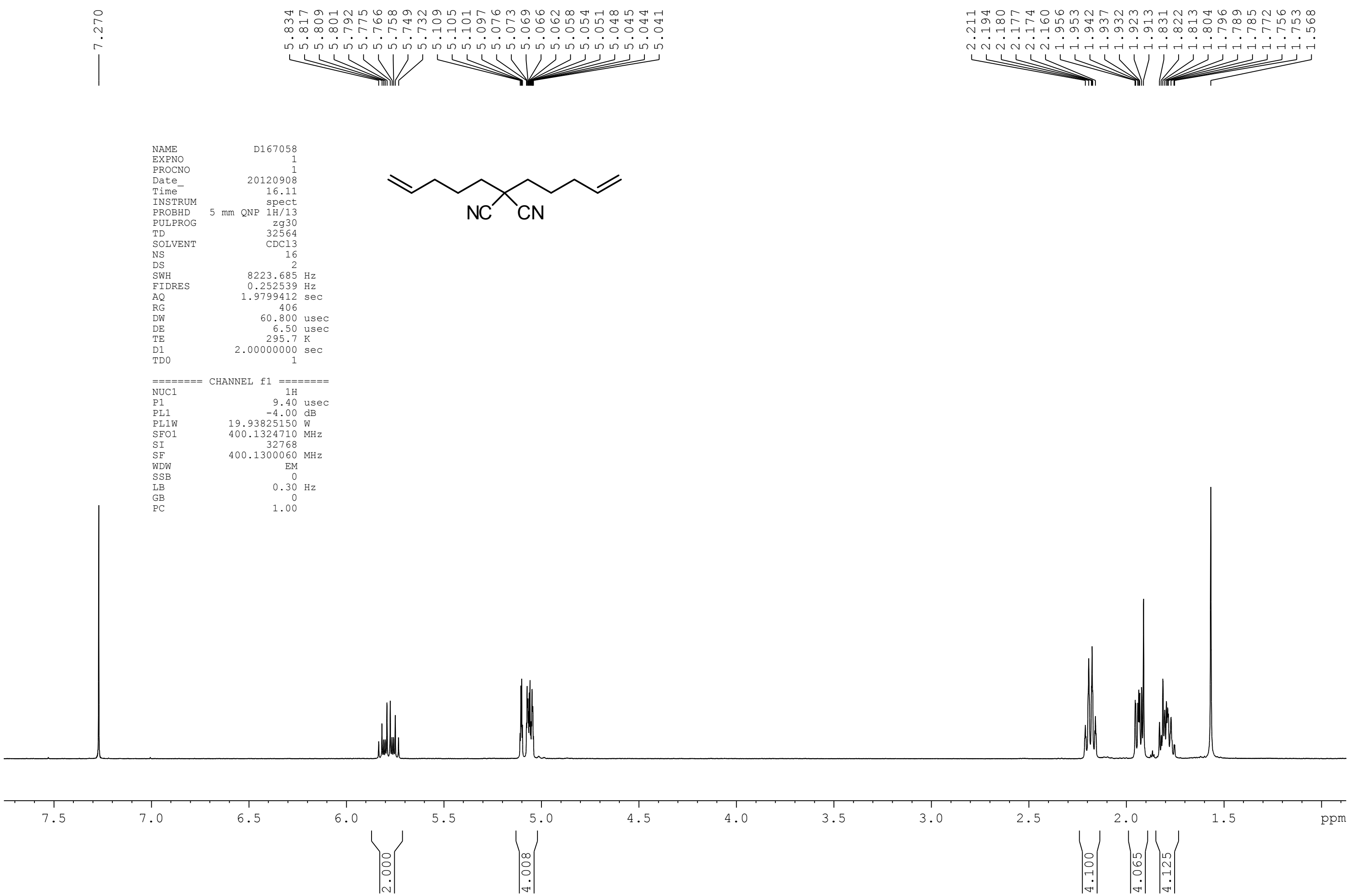




```
NAME             B22117
EXPNO                 2
PROCNO                1
Date_          20120820
Time              10.14
INSTRUM           AV400
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    2
SWH          23980.814 Hz
FIDRES        1.463673 Hz
AQ           0.3416564 sec
RG              18390.4
DW               20.850 usec
DE                20.00 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

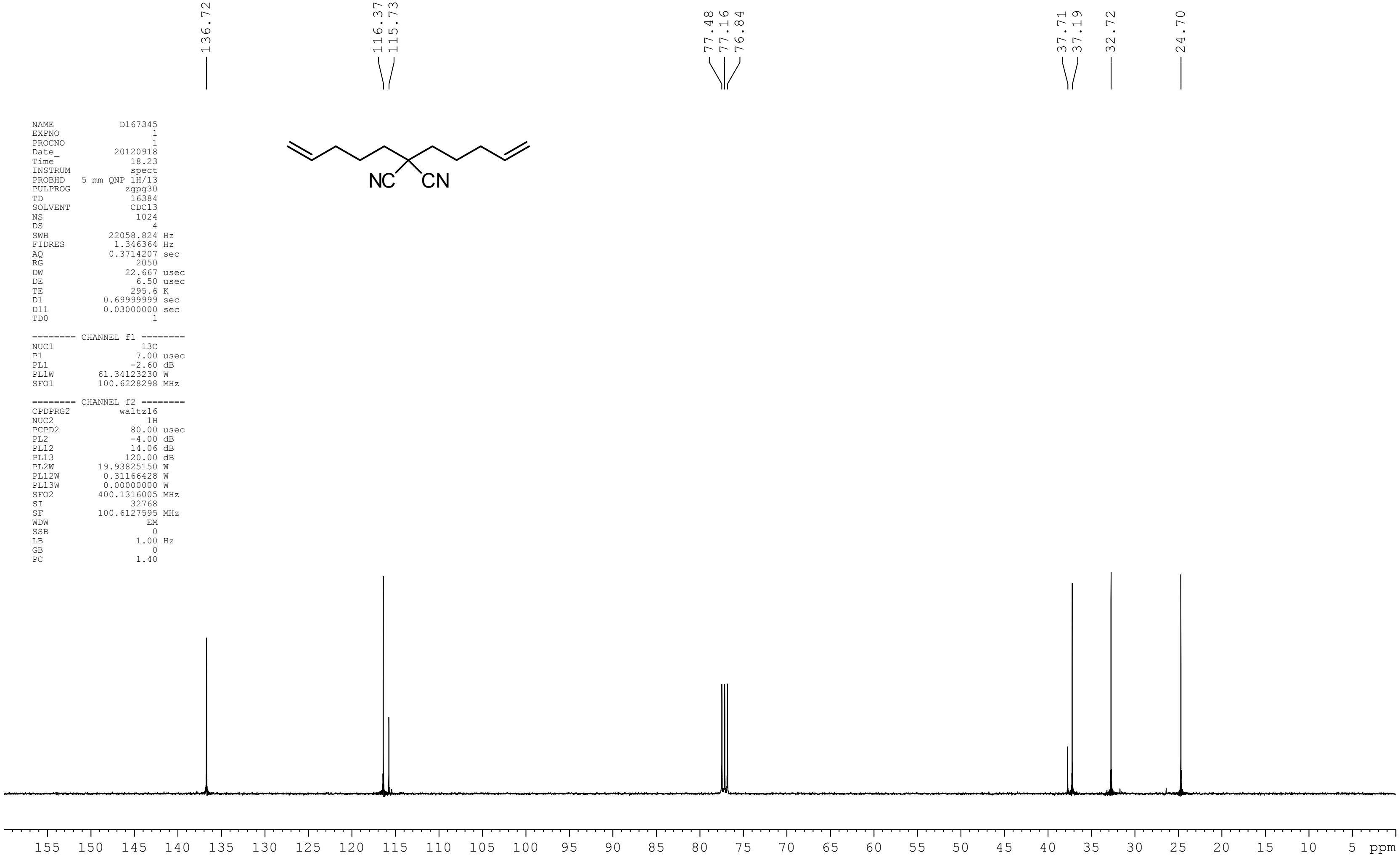
======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1               -2.80 dB
PL1W        57.91424179 W
SFO1        100.5976818 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -3.20 dB
PL12              14.30 dB
PL13              15.30 dB
PL2W        15.00949383 W
PL12W        0.26691073 W
PL13W        0.21201472 W
SFO2        400.0316001 MHz
SI                32768
SF          100.5876096 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D167058
EXPNO                 1
PROCNO                1
Date_          20120908
Time              16.11
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  406
DW               60.800 usec
DE                 6.50 usec
TE                295.7 K
D1           2.00000000 sec
TD0                   1

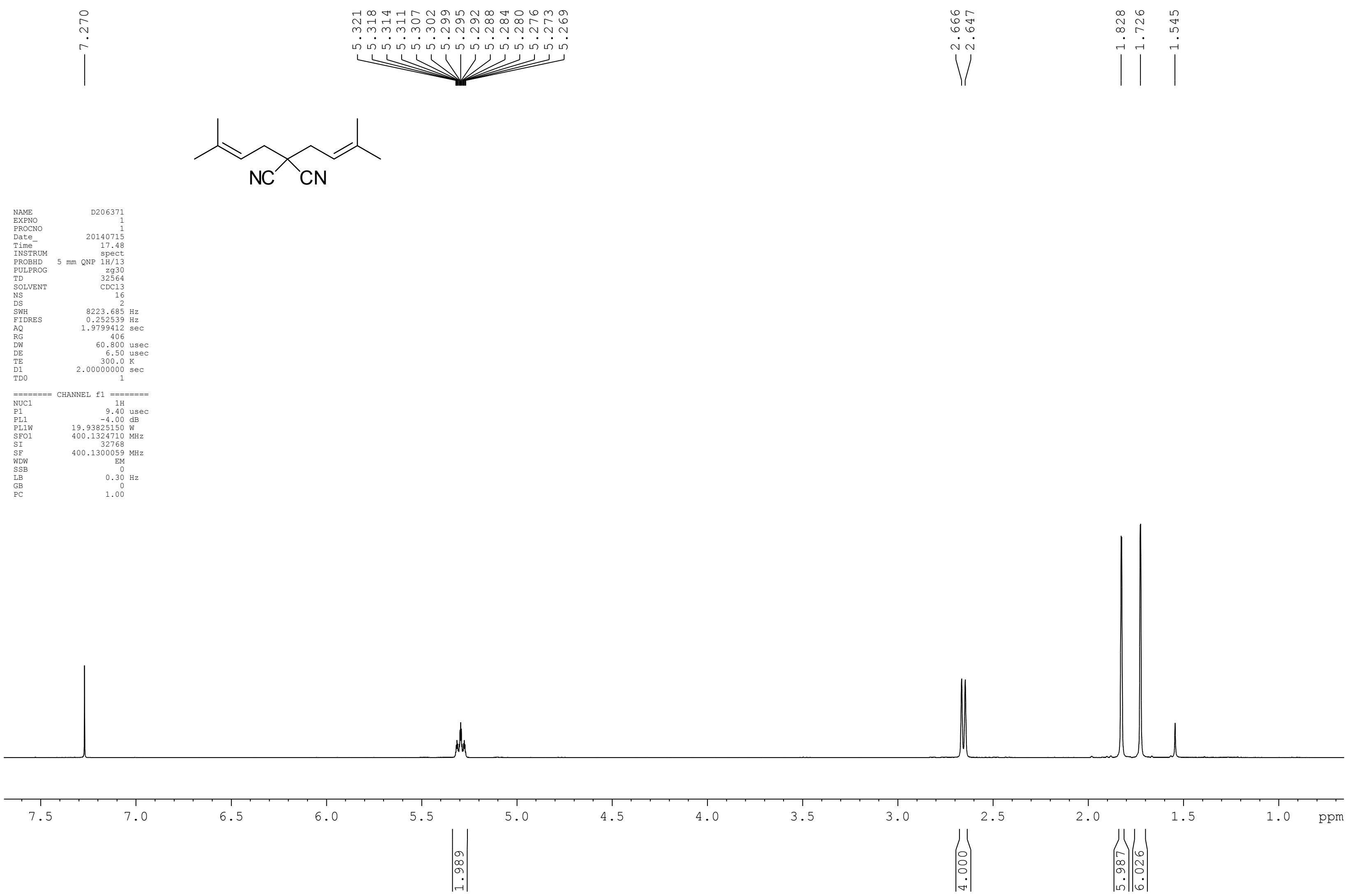
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300060 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME            D167345
EXPNO                 1
PROCNO                1
Date_          20120918
Time              18.23
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                295.6 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

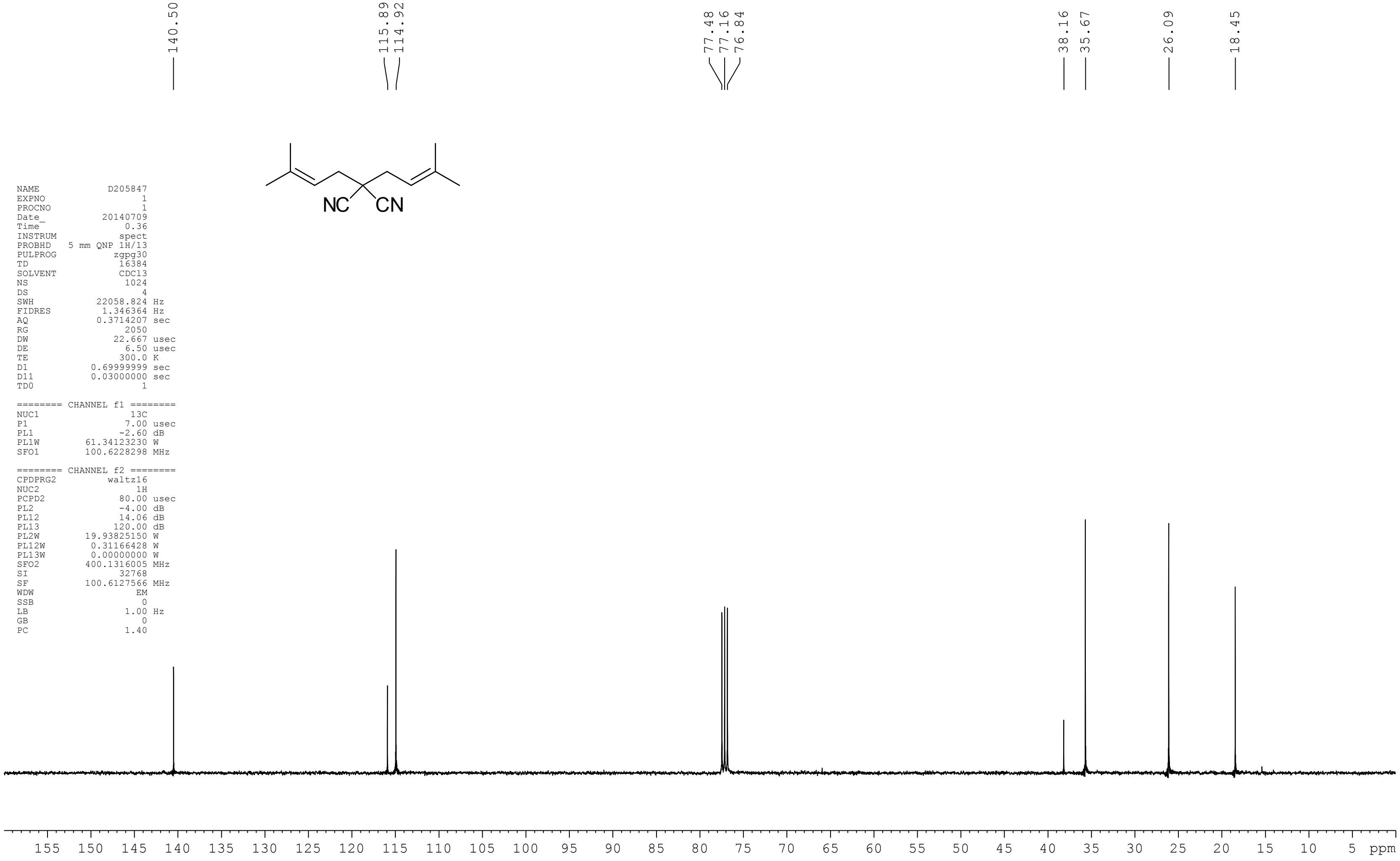
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127595 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D206371
EXPNO                 1
PROCNO                1
Date_          20140715
Time              17.48
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  406
DW               60.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

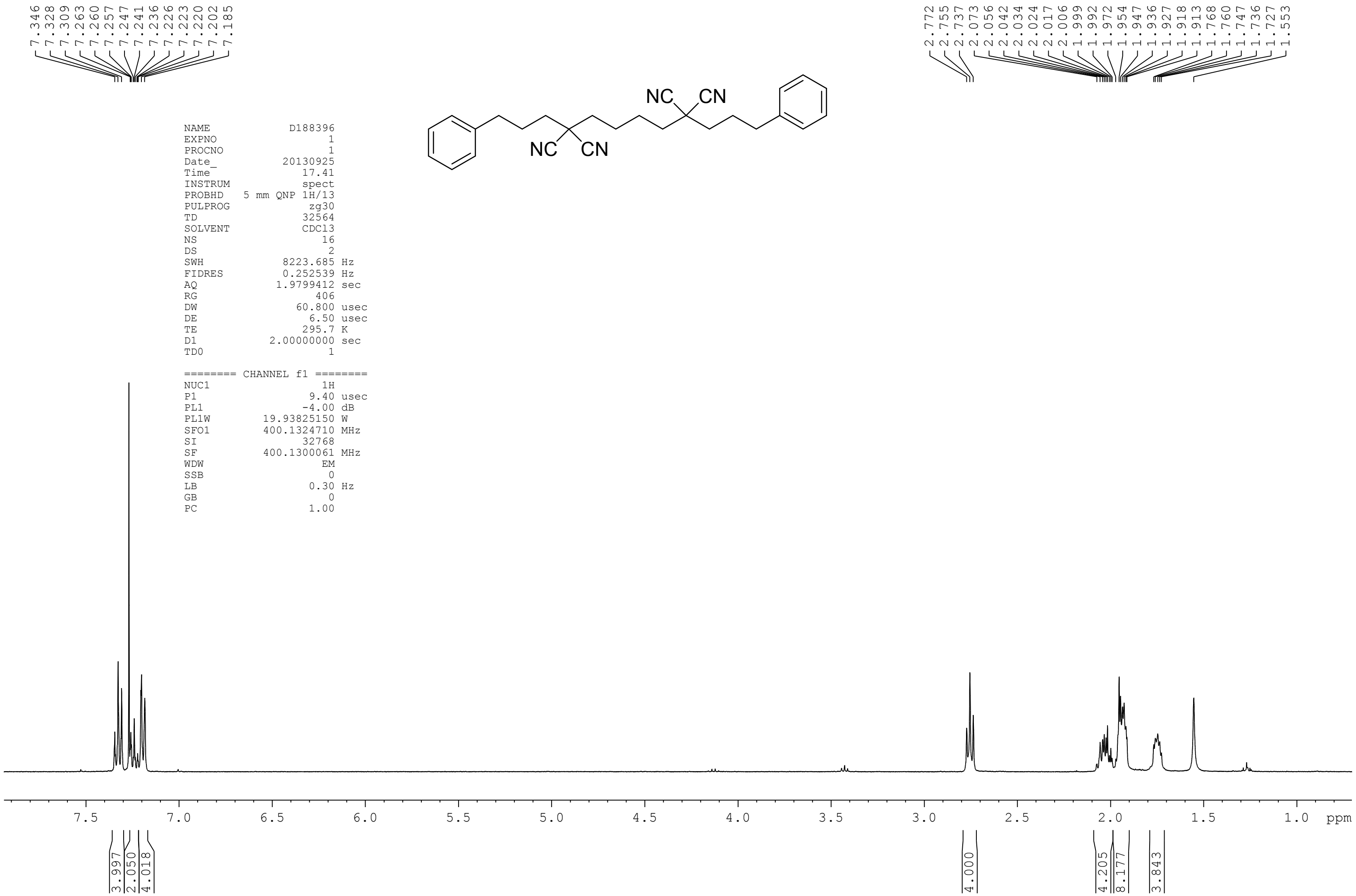
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300059 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME            D205847
EXPNO                 1
PROCNO                1
Date_          20140709
Time               0.36
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

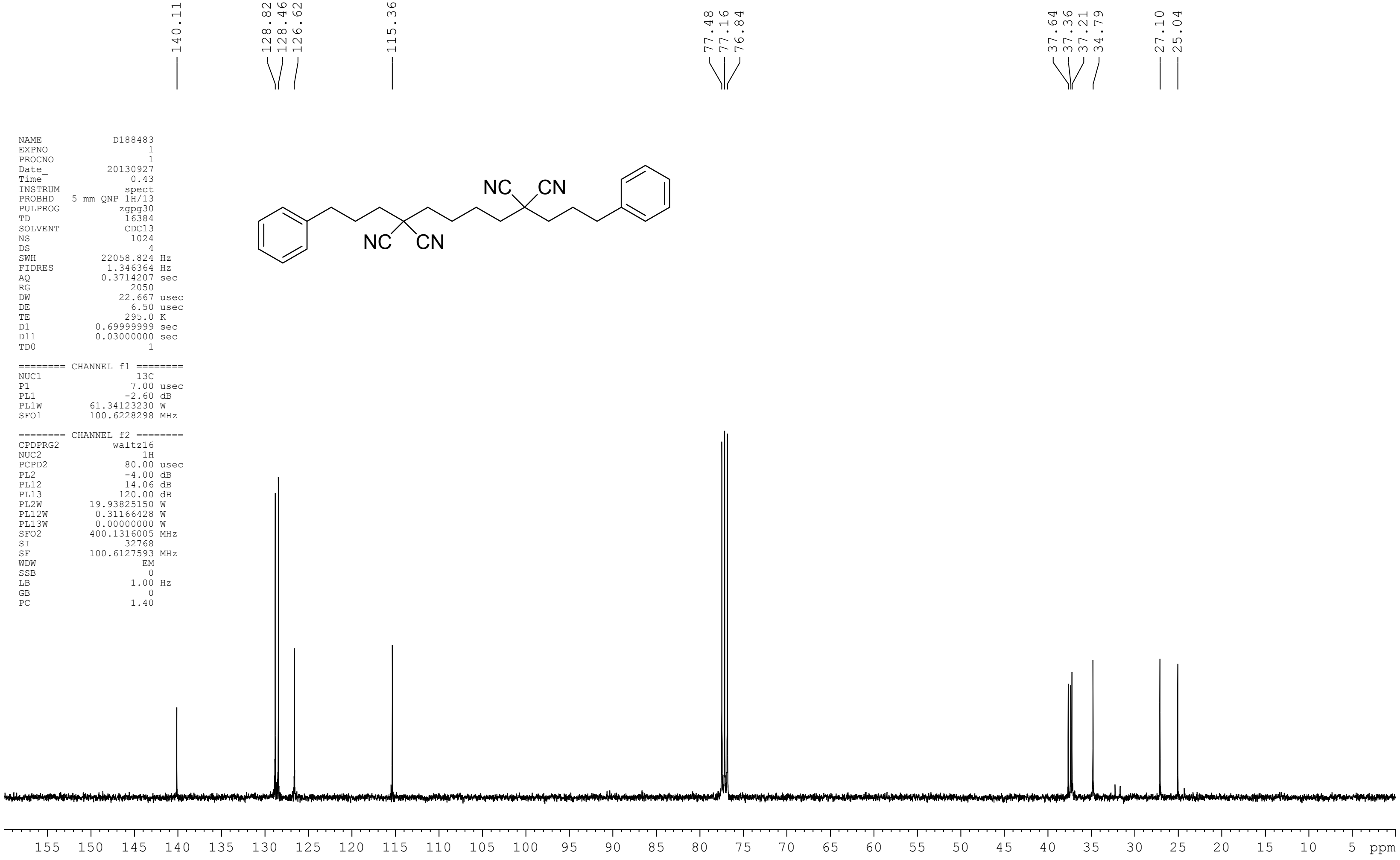
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127566 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME              D188396
EXPNO                   1
PROCNO                  1
Date_            20130925
Time                17.41
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32564
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.252539 Hz
AQ              1.9799412 sec
RG                    406
DW                 60.800 usec
DE                   6.50 usec
TE                  295.7 K
D1             2.00000000 sec
TD0                     1

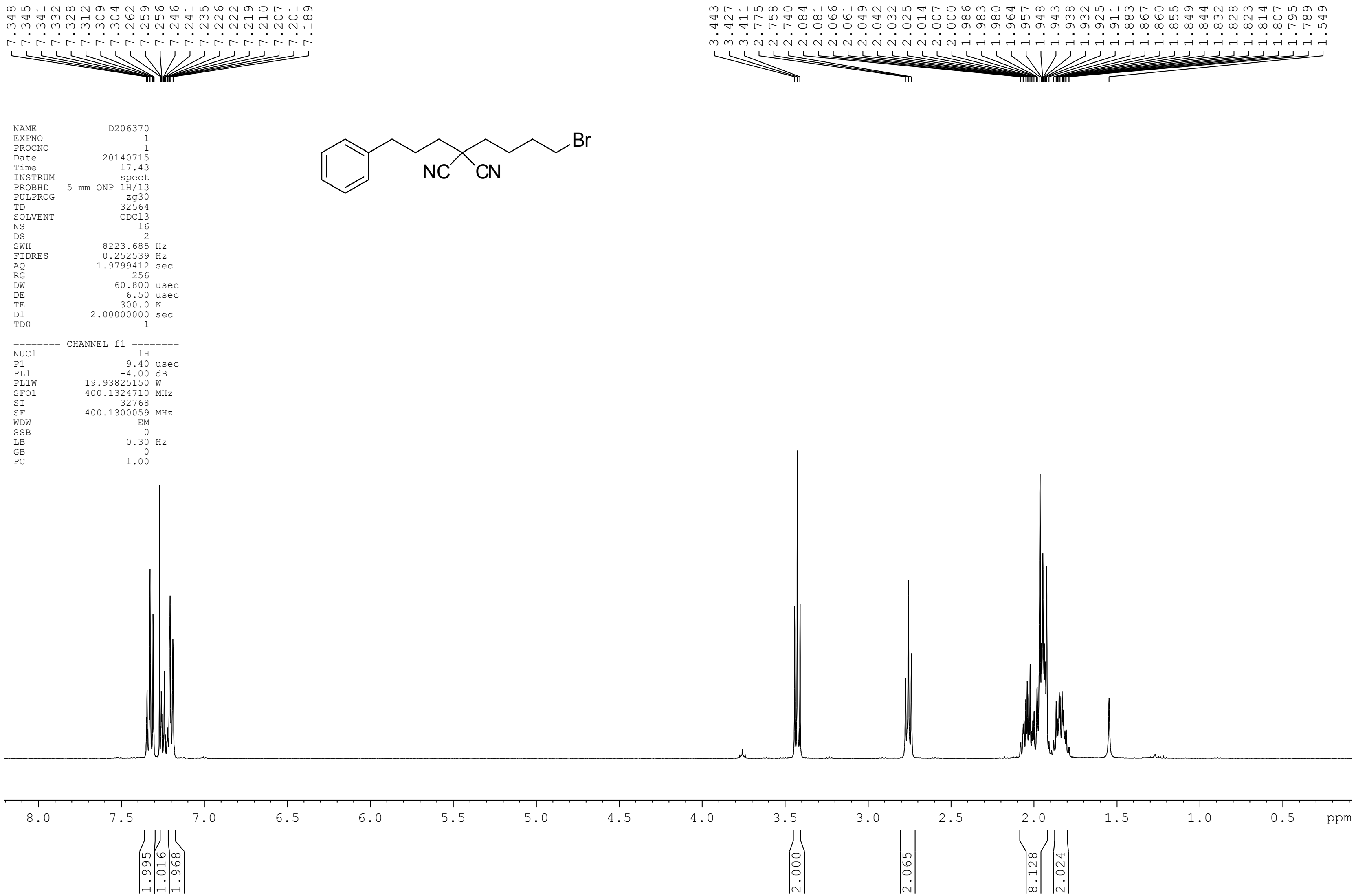
======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 usec
PL1                 -4.00 dB
PL1W          19.93825150 W
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300061 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



```
NAME             D188483
EXPNO                  1
PROCNO                 1
Date_           20130927
Time                0.43
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 16384
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                  2050
DW                22.667 usec
DE                  6.50 usec
TE                 295.0 K
D1            0.69999999 sec
D11           0.03000000 sec
TD0                    1

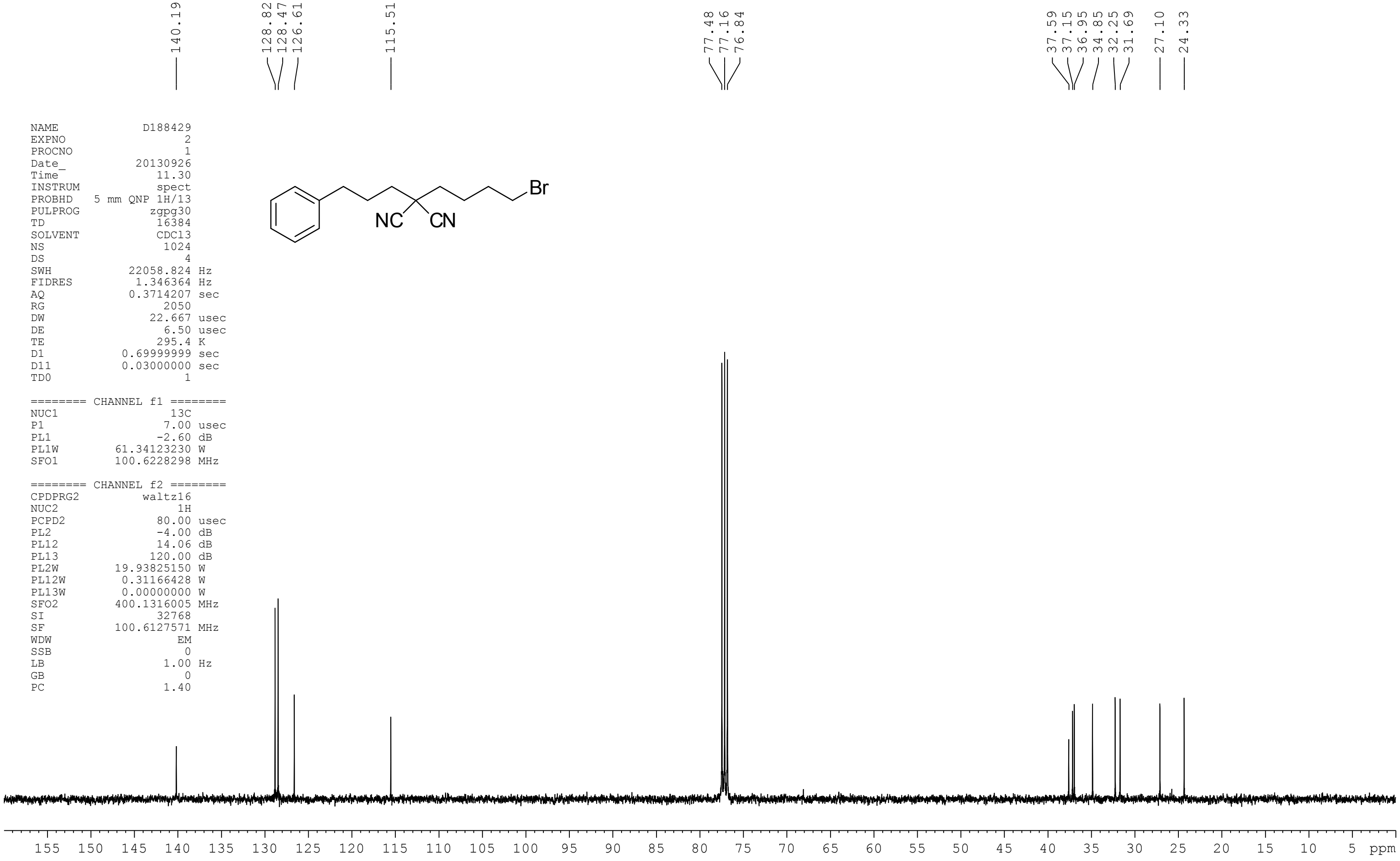
======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.60 dB
PL1W         61.34123230 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.06 dB
PL13              120.00 dB
PL2W         19.93825150 W
PL12W         0.31166428 W
PL13W         0.00000000 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127593 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



```
NAME             D206370
EXPNO                  1
PROCNO                 1
Date_           20140715
Time               17.43
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32564
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.252539 Hz
AQ             1.9799412 sec
RG                   256
DW                60.800 usec
DE                  6.50 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

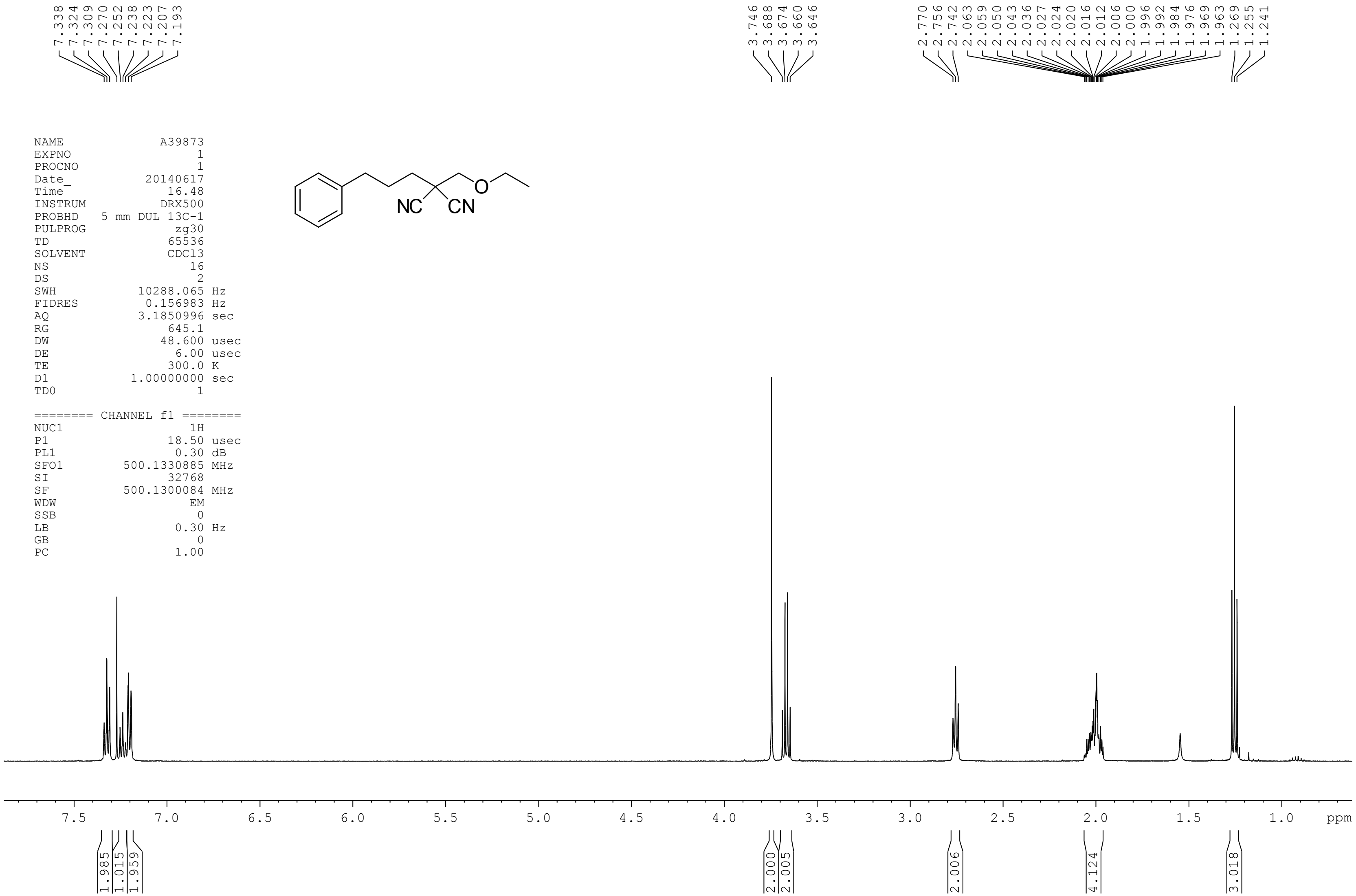
======== CHANNEL f1 ========
NUC1                  1H
P1                  9.40 usec
PL1                -4.00 dB
PL1W         19.93825150 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300059 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
NAME           D188429
EXPNO                2
PROCNO               1
Date_         20130926
Time             11.30
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               16384
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          22058.824 Hz
FIDRES        1.346364 Hz
AQ           0.3714207 sec
RG                2050
DW              22.667 usec
DE                6.50 usec
TE               295.4 K
D1          0.69999999 sec
D11         0.03000000 sec
TD0                  1

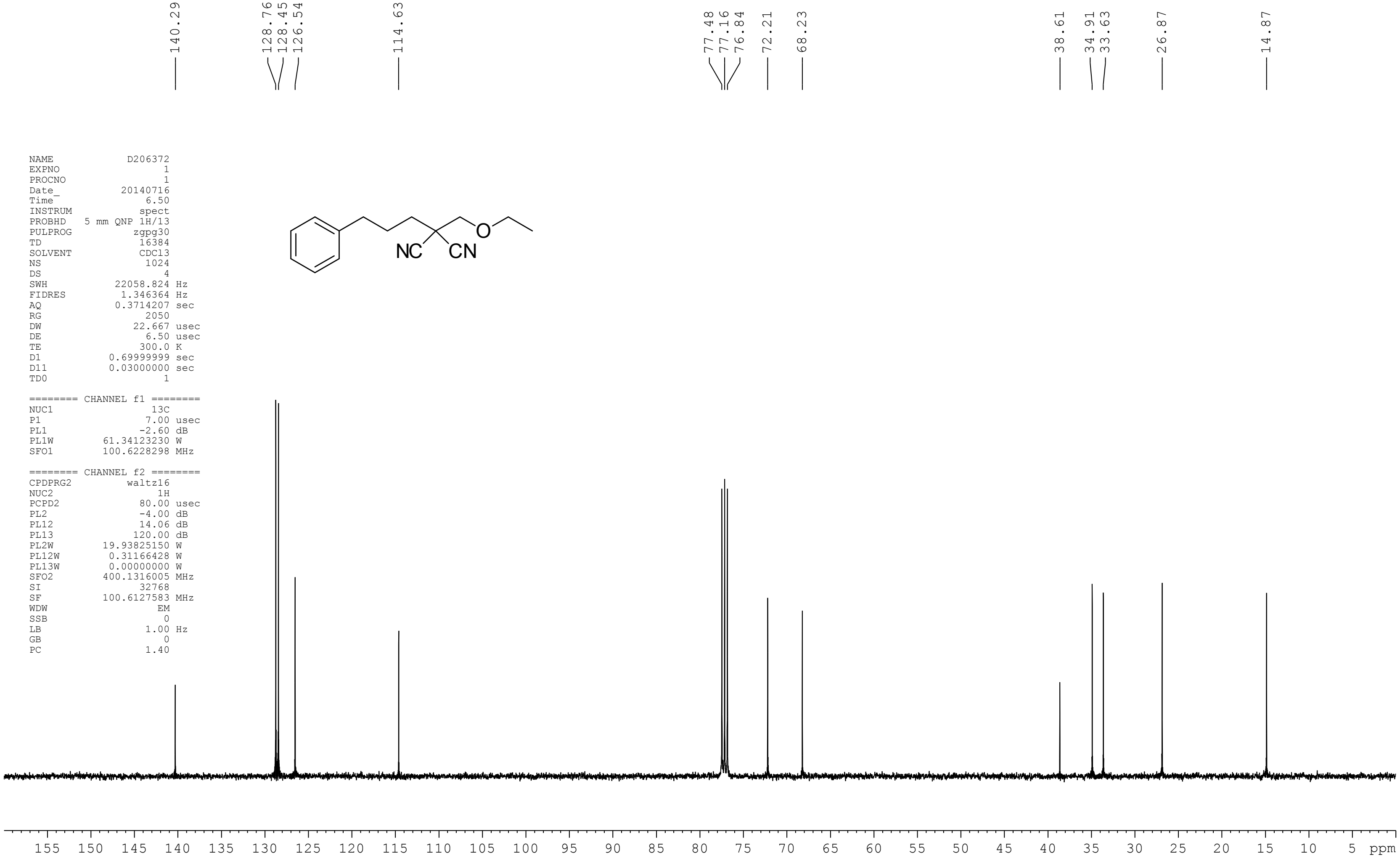
======== CHANNEL f1 ========
NUC1               13C
P1                7.00 usec
PL1              -2.60 dB
PL1W       61.34123230 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -4.00 dB
PL12             14.06 dB
PL13            120.00 dB
PL2W       19.93825150 W
PL12W       0.31166428 W
PL13W       0.00000000 W
SFO2       400.1316005 MHz
SI               32768
SF         100.6127571 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



```
NAME              A39873
EXPNO                  1
PROCNO                 1
Date_           20140617
Time               16.48
INSTRUM           DRX500
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10288.065 Hz
FIDRES          0.156983 Hz
AQ             3.1850996 sec
RG                 645.1
DW                48.600 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

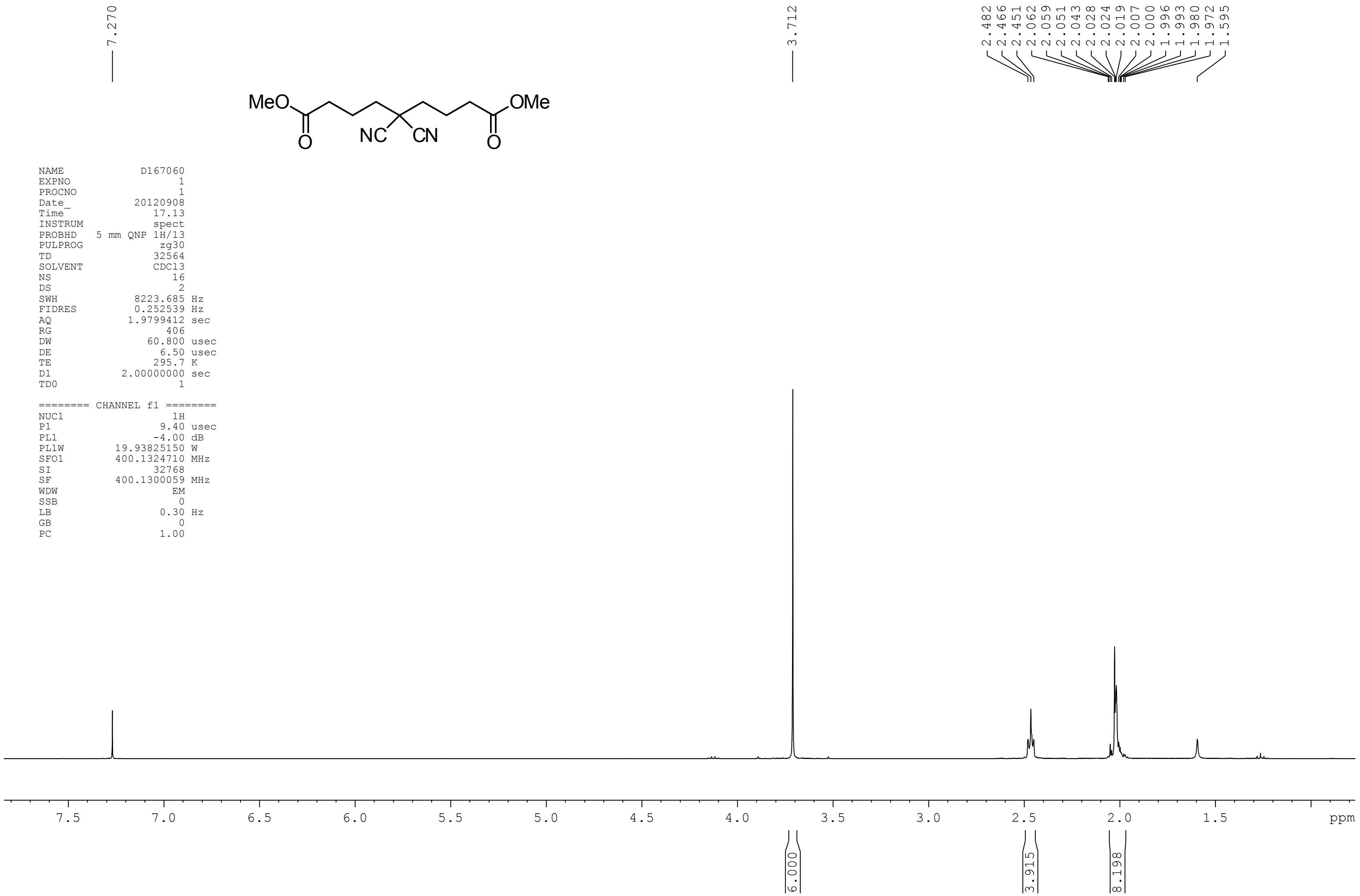
======== CHANNEL f1 ========
NUC1                  1H
P1                 18.50 usec
PL1                 0.30 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300084 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
NAME            D206372
EXPNO                 1
PROCNO                1
Date_          20140716
Time               6.50
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

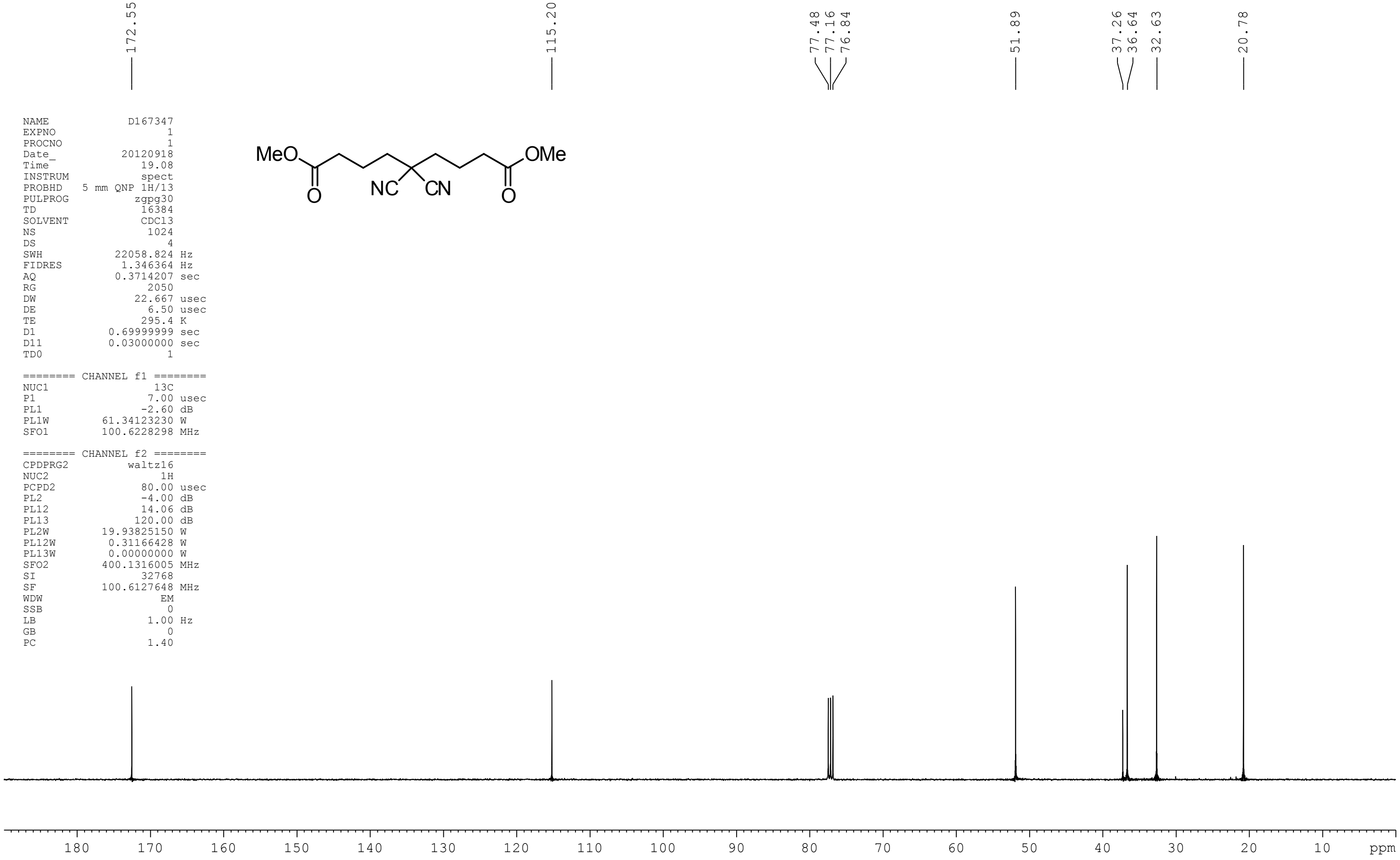
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127583 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME             D167060
EXPNO                  1
PROCNO                 1
Date_           20120908
Time               17.13
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32564
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.252539 Hz
AQ             1.9799412 sec
RG                   406
DW                60.800 usec
DE                  6.50 usec
TE                 295.7 K
D1            2.00000000 sec
TD0                    1

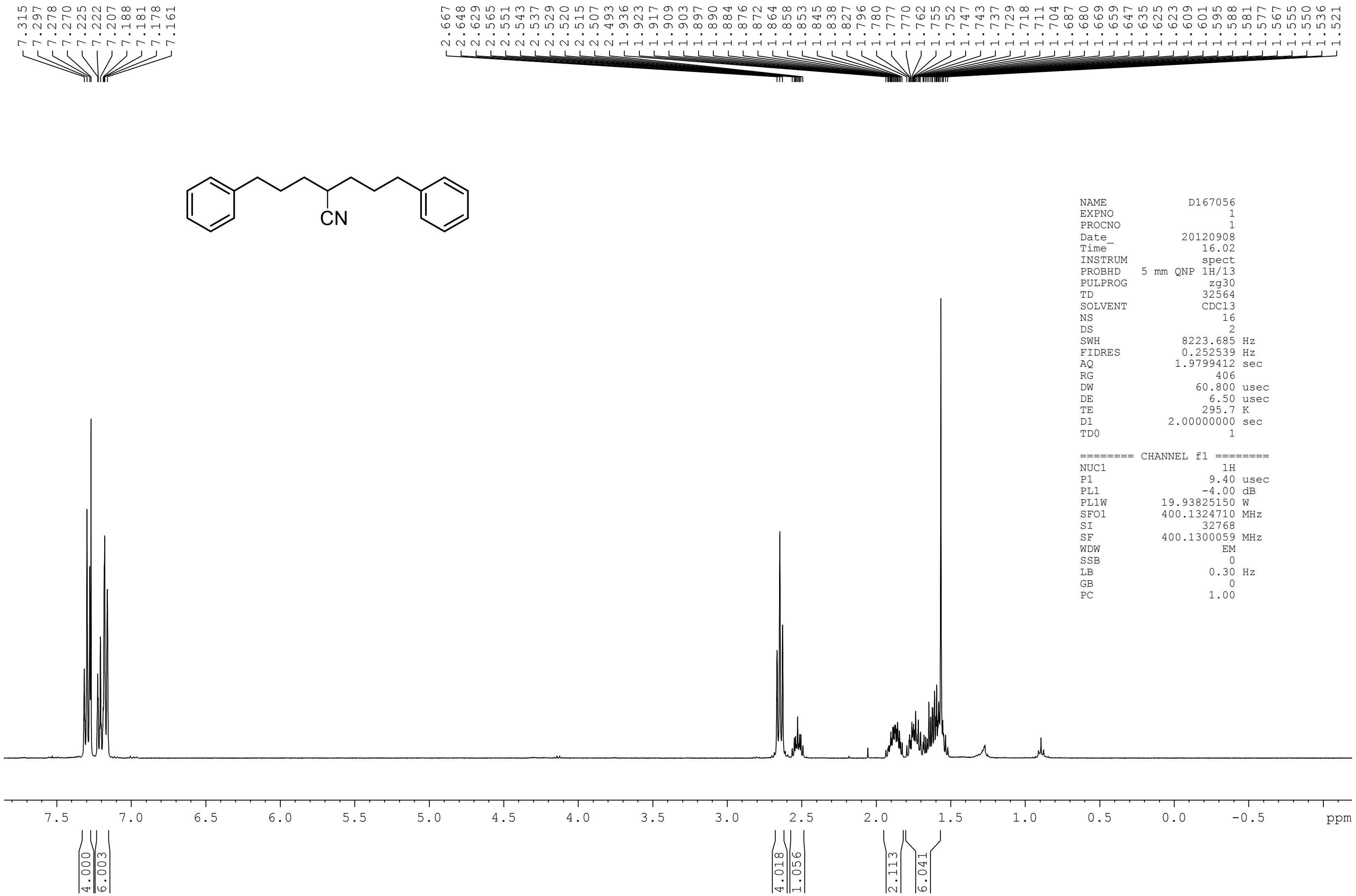
======== CHANNEL f1 ========
NUC1                  1H
P1                  9.40 usec
PL1                -4.00 dB
PL1W         19.93825150 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300059 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
NAME            D167347
EXPNO                 1
PROCNO                1
Date_          20120918
Time              19.08
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               16384
SOLVENT           CDCl3
NS                1024
DS                    4
SWH          22058.824 Hz
FIDRES        1.346364 Hz
AQ           0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                295.4 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

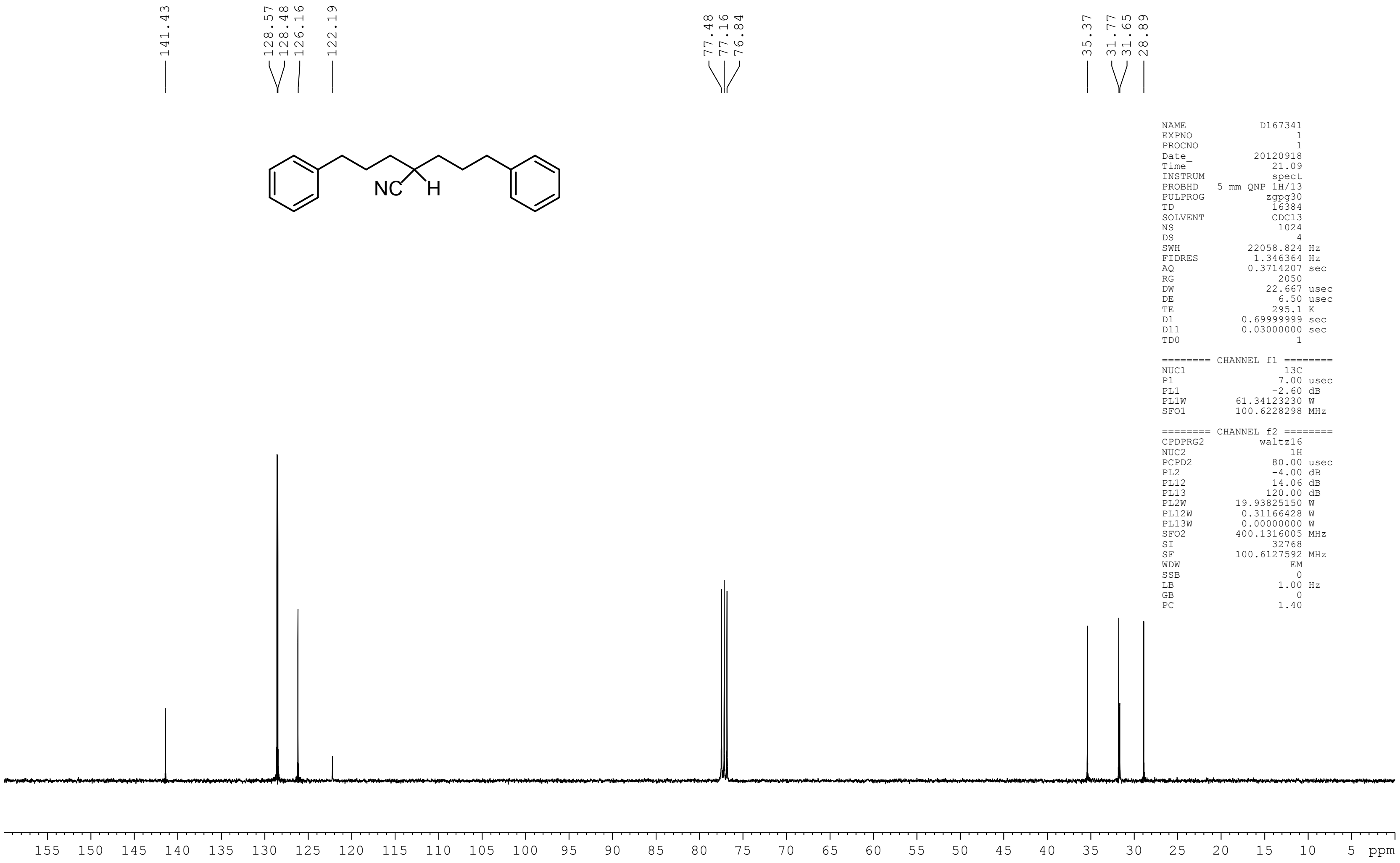
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127648 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D167056
EXPNO                 1
PROCNO                1
Date_          20120908
Time              16.02
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  406
DW               60.800 usec
DE                 6.50 usec
TE                295.7 K
D1           2.00000000 sec
TD0                   1

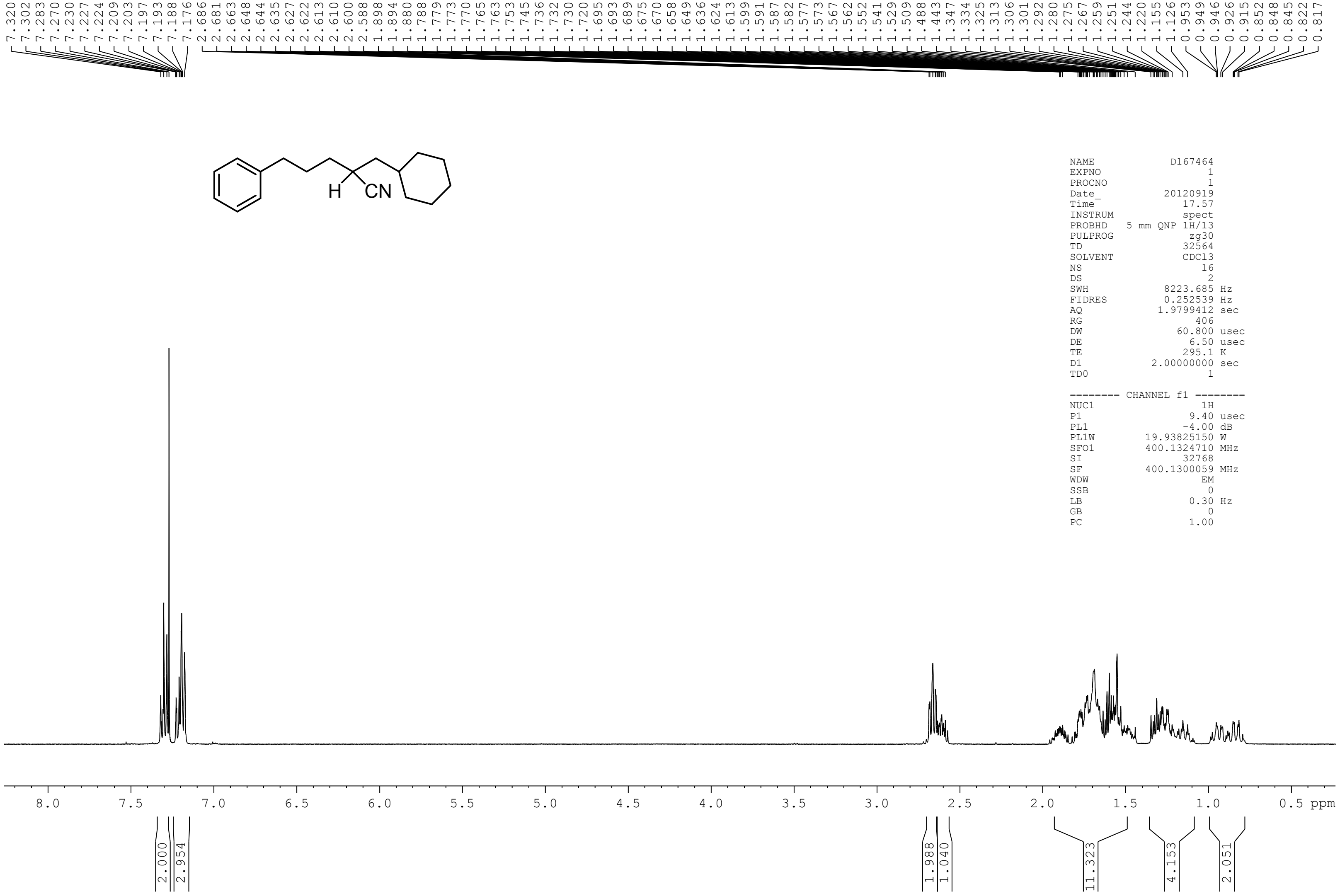
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300059 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME            D167341
EXPNO                 1
PROCNO                1
Date_          20120918
Time              21.09
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                295.1 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

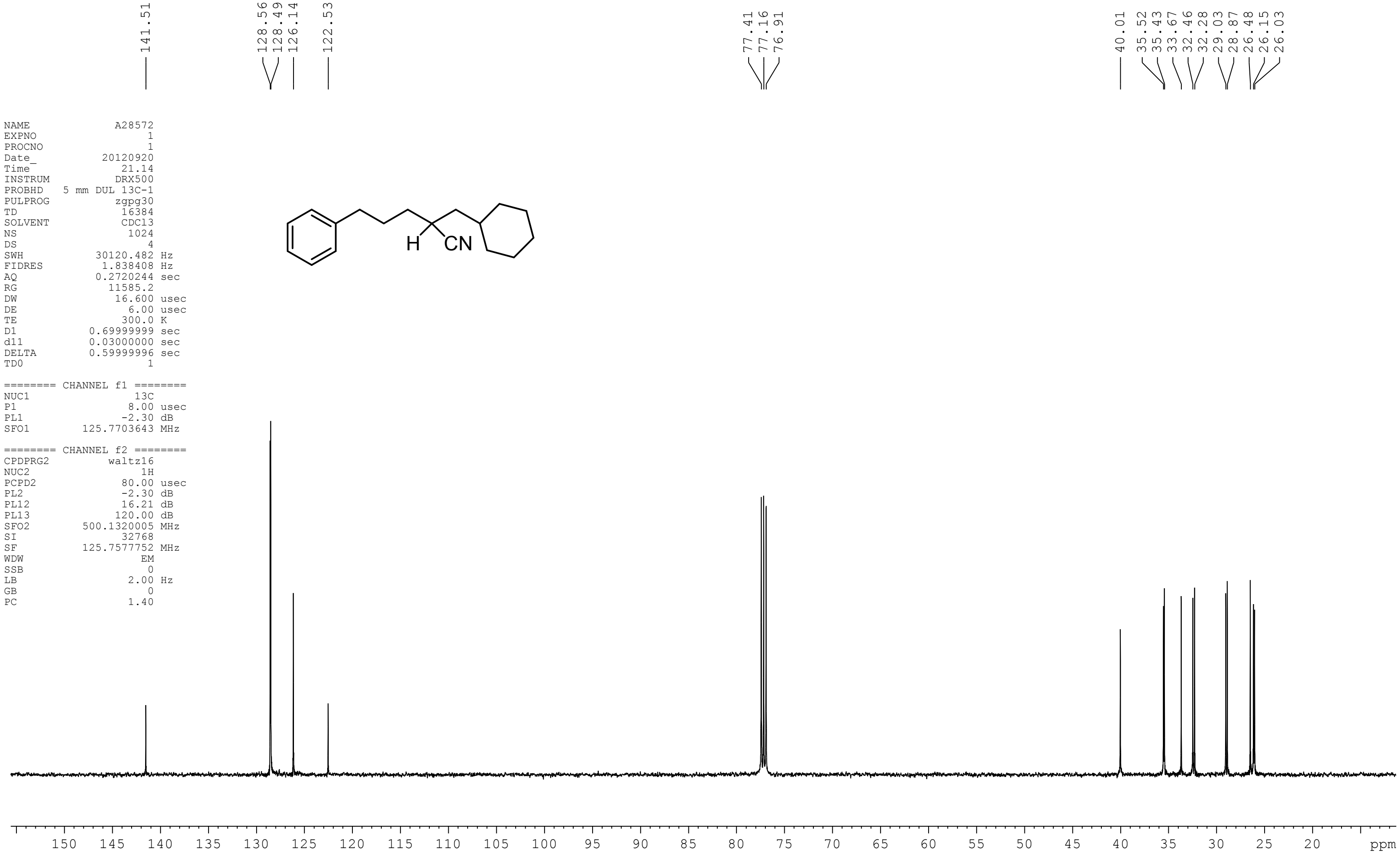
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127592 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME             D167464
EXPNO                  1
PROCNO                 1
Date_           20120919
Time               17.57
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32564
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.252539 Hz
AQ             1.9799412 sec
RG                   406
DW                60.800 usec
DE                  6.50 usec
TE                 295.1 K
D1            2.00000000 sec
TD0                    1

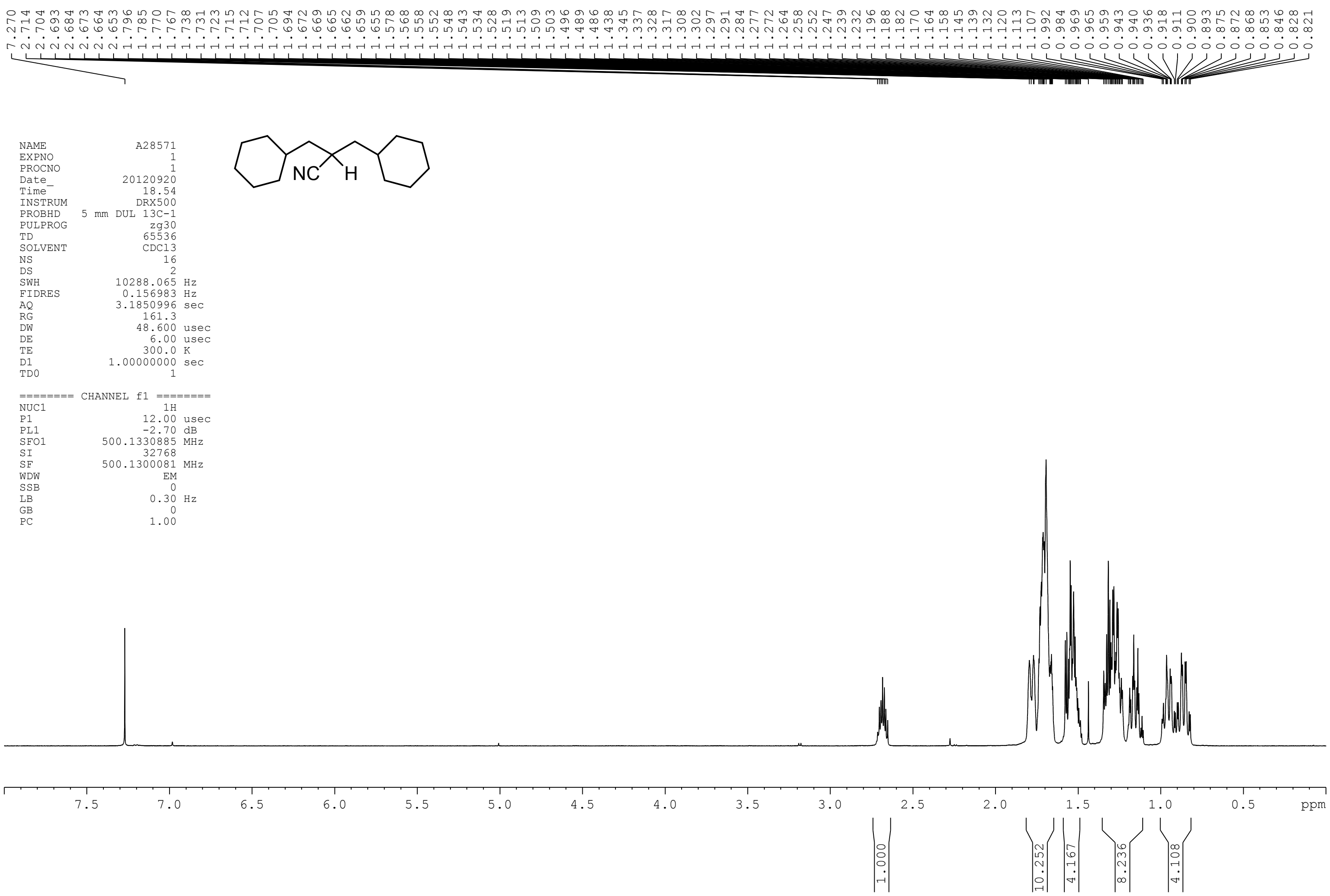
======== CHANNEL f1 ========
NUC1                  1H
P1                  9.40 usec
PL1                -4.00 dB
PL1W         19.93825150 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300059 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
NAME             A28572
EXPNO                 1
PROCNO                1
Date_          20120920
Time              21.14
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH          30120.482 Hz
FIDRES        1.838408 Hz
AQ           0.2720244 sec
RG              11585.2
DW               16.600 usec
DE                 6.00 usec
TE                300.0 K
D1           0.69999999 sec
d11          0.03000000 sec
DELTA        0.59999996 sec
TD0                   1

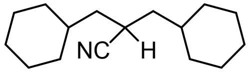
======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1               -2.30 dB
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.30 dB
PL12              16.21 dB
PL13             120.00 dB
SFO2       500.1320005 MHz
SI                32768
SF         125.7577752 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40
```



```
NAME              A28571
EXPNO                  1
PROCNO                 1
Date_           20120920
Time               18.54
INSTRUM           DRX500
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10288.065 Hz
FIDRES          0.156983 Hz
AQ             3.1850996 sec
RG                 161.3
DW                48.600 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

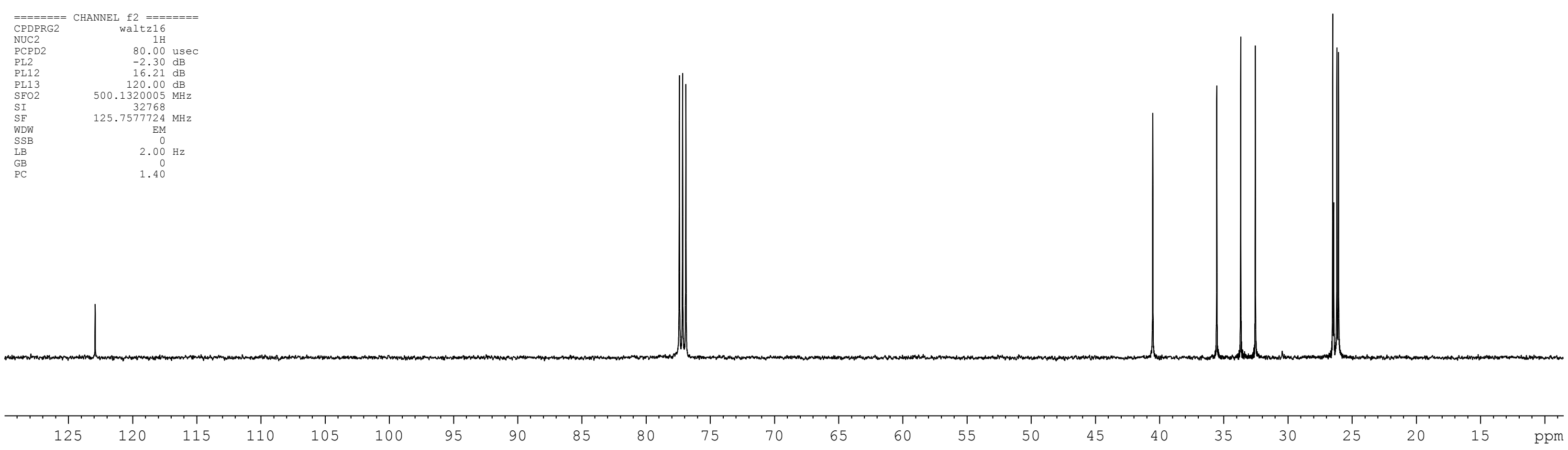
======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.70 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300081 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

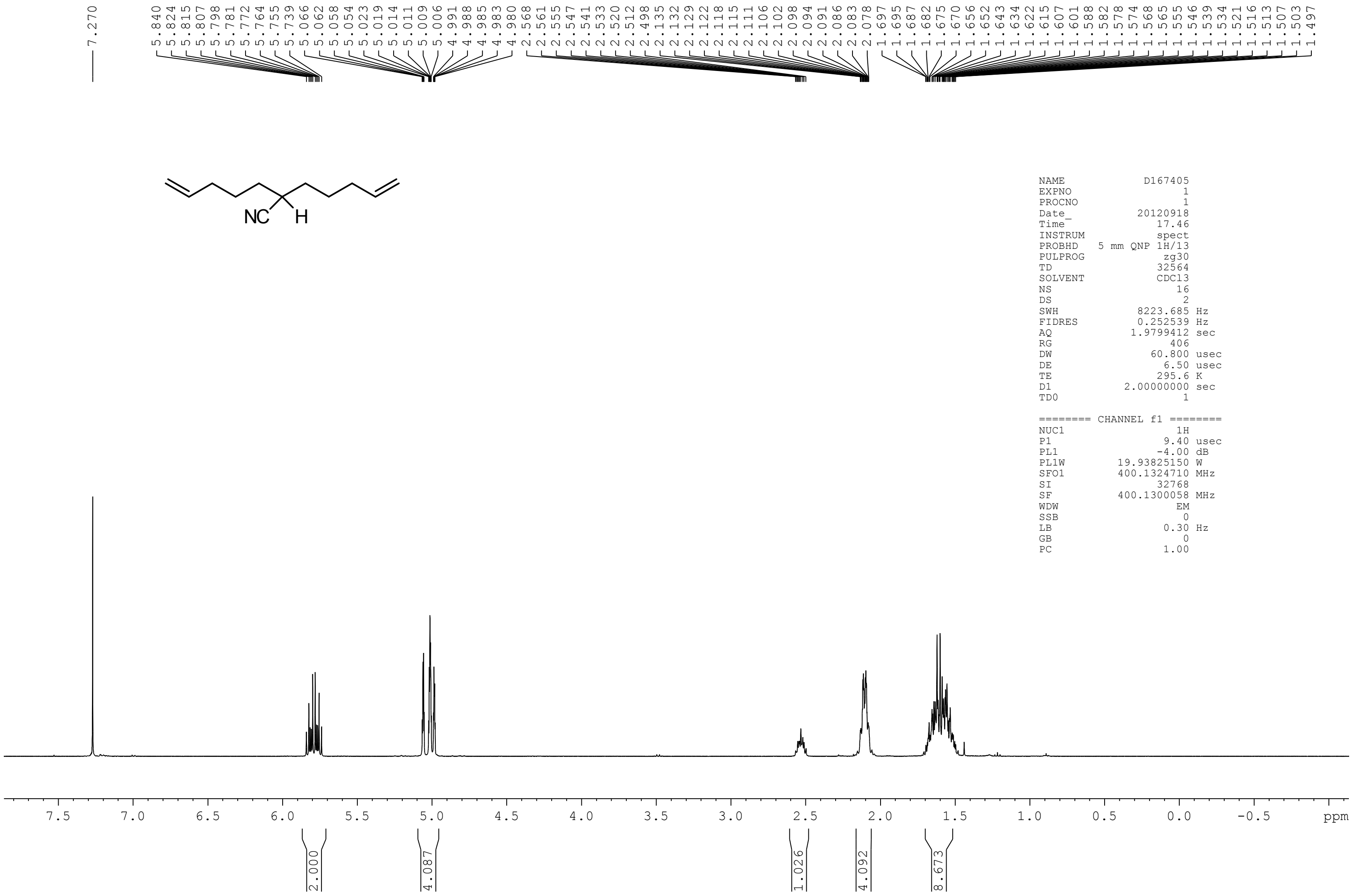


```
NAME             A28571
EXPNO                 2
PROCNO                1
Date_          20120920
Time              20.48
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         1.838408 Hz
AQ            0.2720244 sec
RG              11585.2
DW               16.600 usec
DE                 6.00 usec
TE                300.0 K
D1           0.69999999 sec
d11          0.03000000 sec
DELTA        0.59999996 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1               -2.30 dB
SFO1        125.7703643 MHz

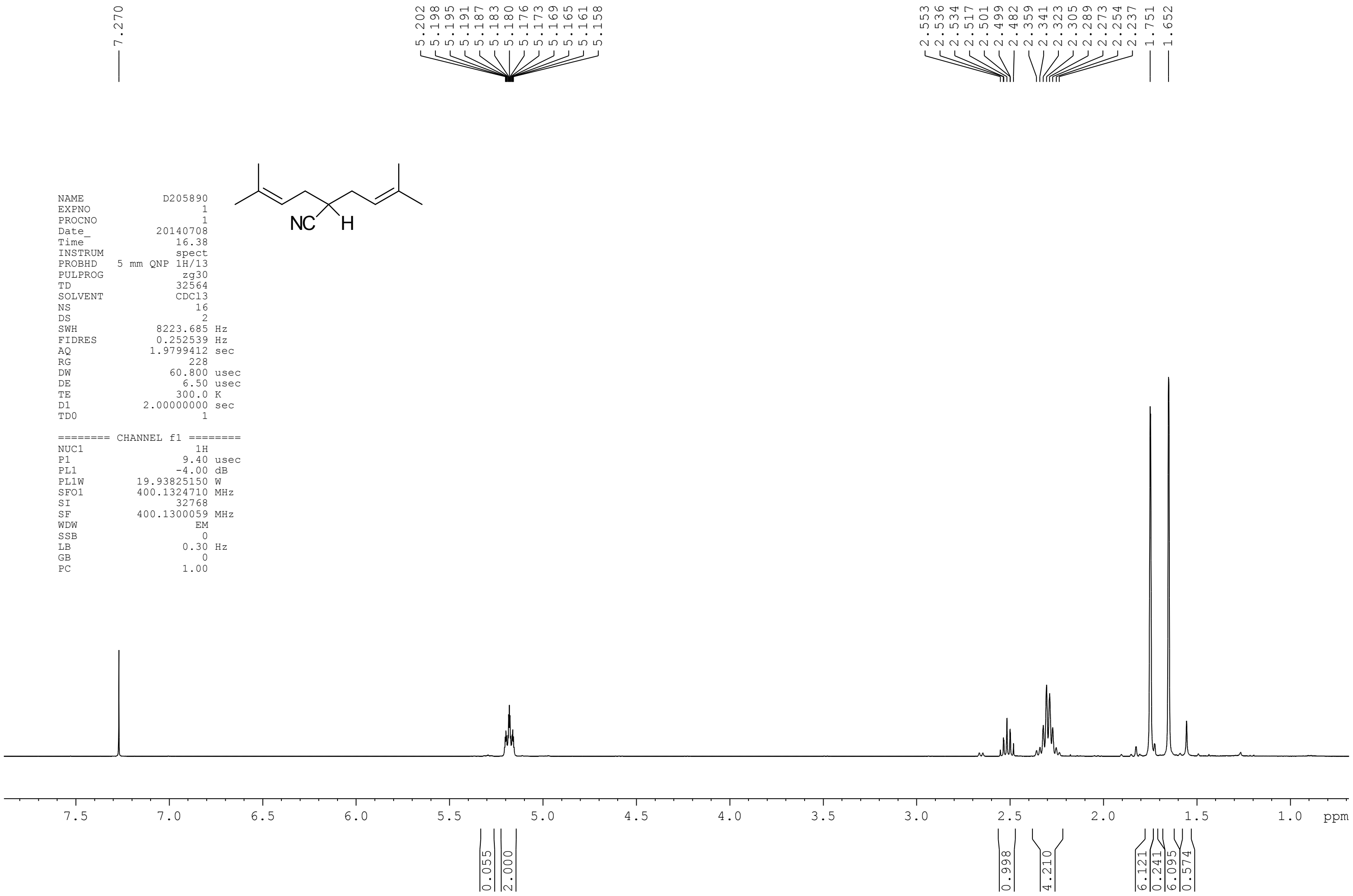
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.30 dB
PL12              16.21 dB
PL13             120.00 dB
SFO2        500.1320005 MHz
SI                32768
SF          125.7577724 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40
```





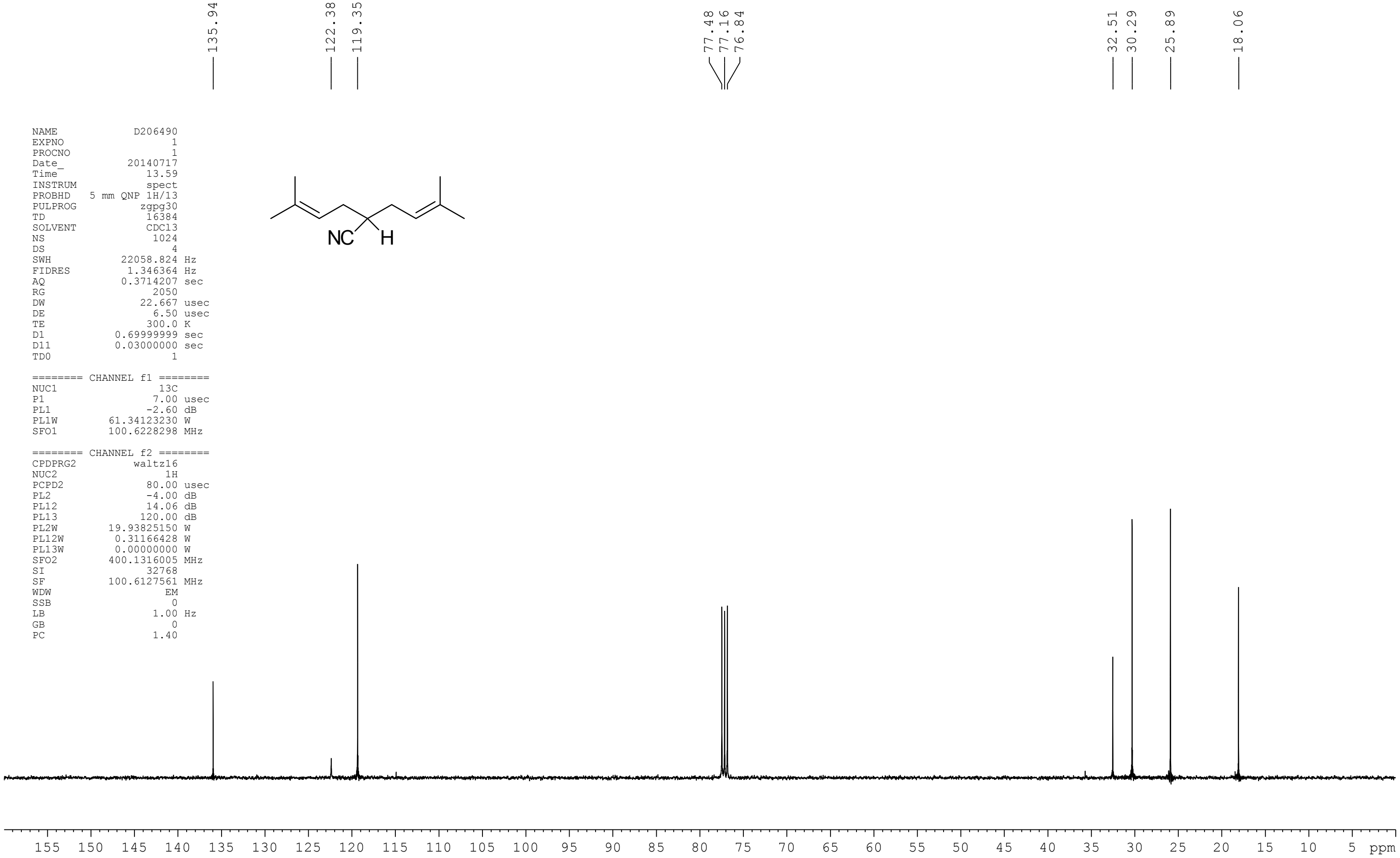
```
NAME           D167405
EXPNO                1
PROCNO               1
Date_         20120918
Time             17.46
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               32564
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8223.685 Hz
FIDRES        0.252539 Hz
AQ           1.9799412 sec
RG                 406
DW              60.800 usec
DE                6.50 usec
TE               295.6 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                9.40 usec
PL1              -4.00 dB
PL1W       19.93825150 W
SFO1       400.1324710 MHz
SI               32768
SF         400.1300058 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



```
NAME              D205890
EXPNO                   1
PROCNO                  1
Date_            20140708
Time                16.38
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32564
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.252539 Hz
AQ              1.9799412 sec
RG                    228
DW                 60.800 usec
DE                   6.50 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

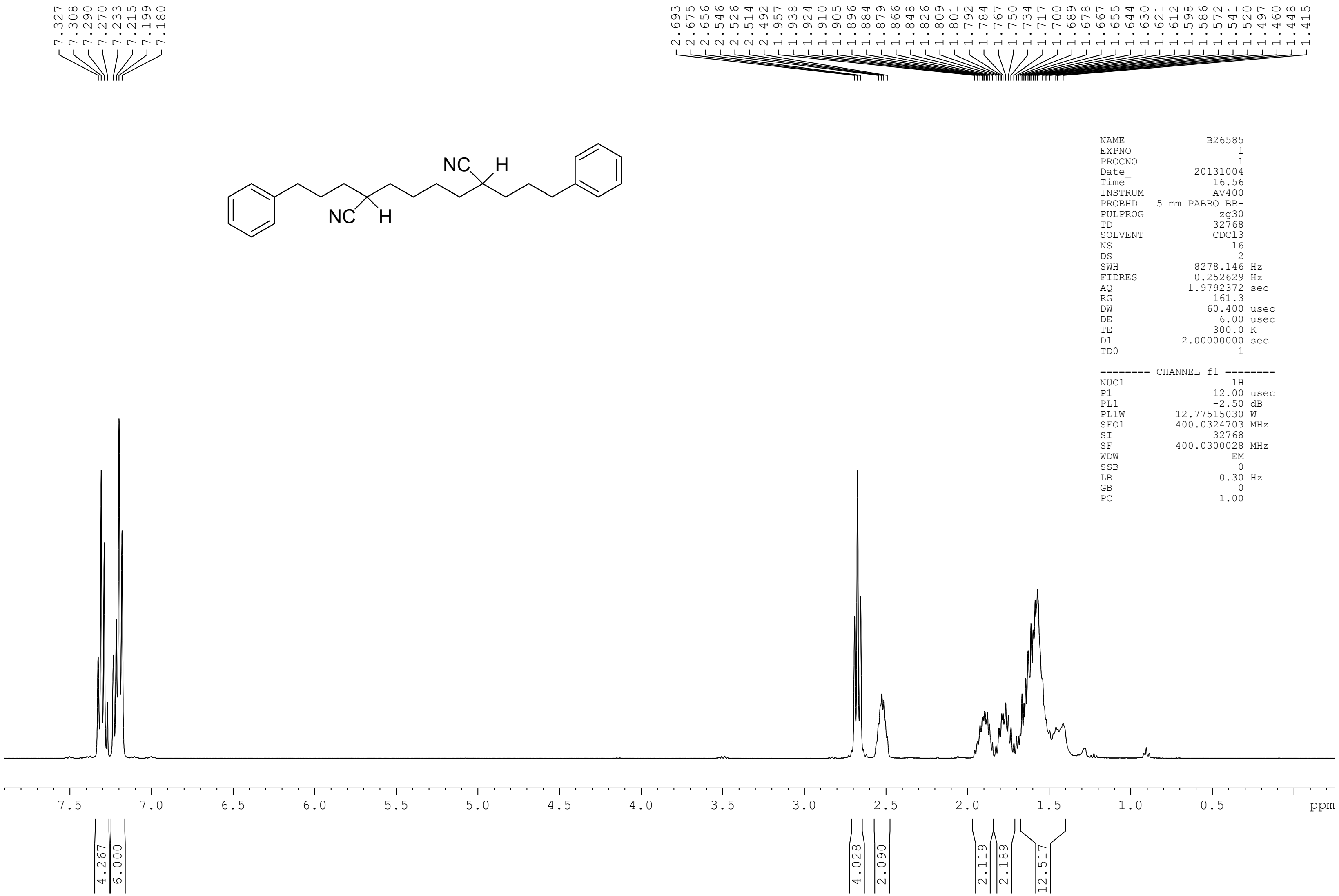
======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 usec
PL1                 -4.00 dB
PL1W          19.93825150 W
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300059 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



```
NAME            D206490
EXPNO                 1
PROCNO                1
Date_          20140717
Time              13.59
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127561 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



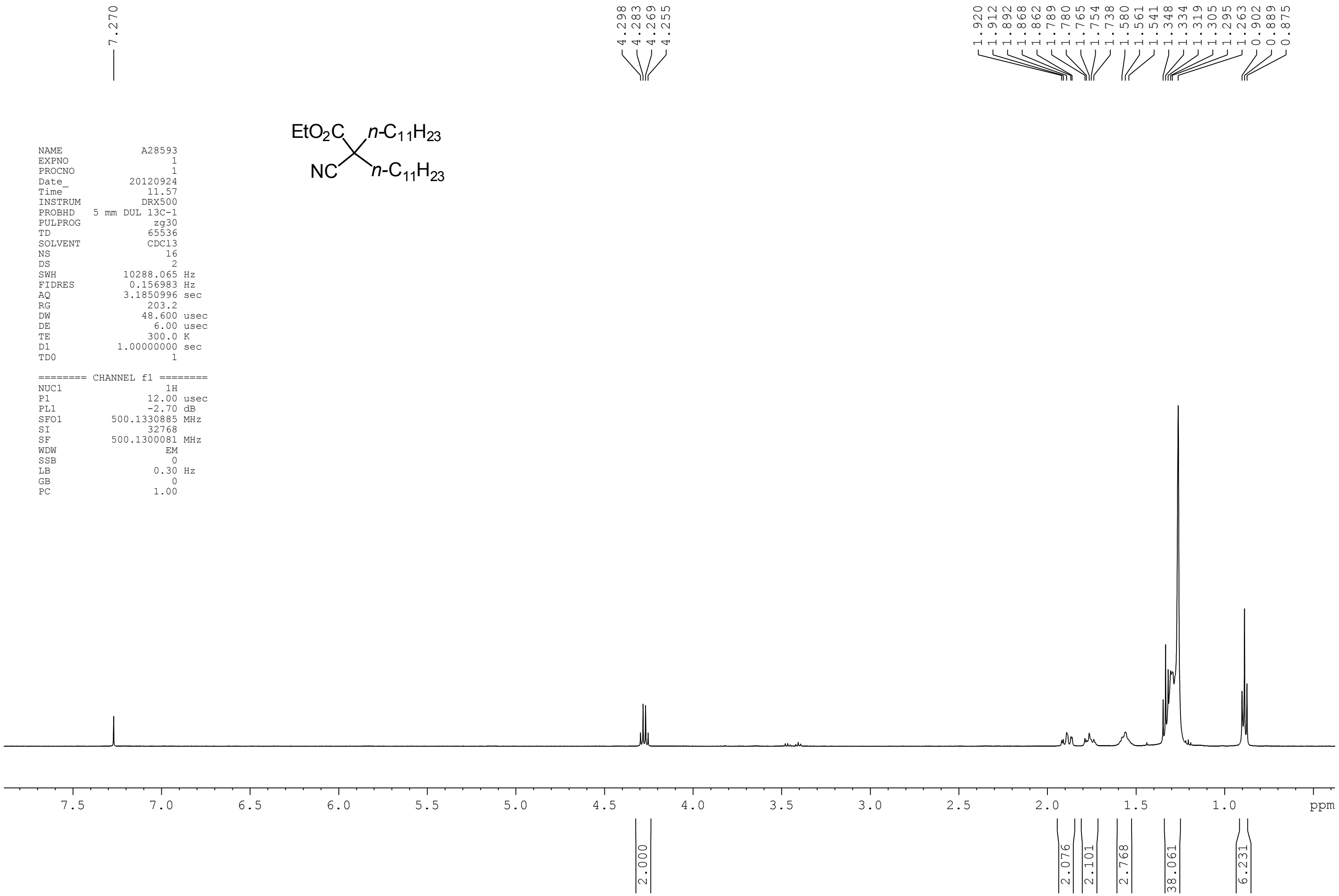
```
NAME              B26585
EXPNO                  1
PROCNO                 1
Date_           20131004
Time               16.56
INSTRUM            AV400
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.252629 Hz
AQ             1.9792372 sec
RG                 161.3
DW                60.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.50 dB
PL1W         12.77515030 W
SFO1         400.0324703 MHz
SI                 32768
SF           400.0300028 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

```
NAME             B26585
EXPNO                 2
PROCNO                1
Date_          20131004
Time              20.27
INSTRUM           AV400
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    2
SWH           23980.814 Hz
FIDRES         1.463673 Hz
AQ            0.3416564 sec
RG              20642.5
DW               20.850 usec
DE                20.00 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

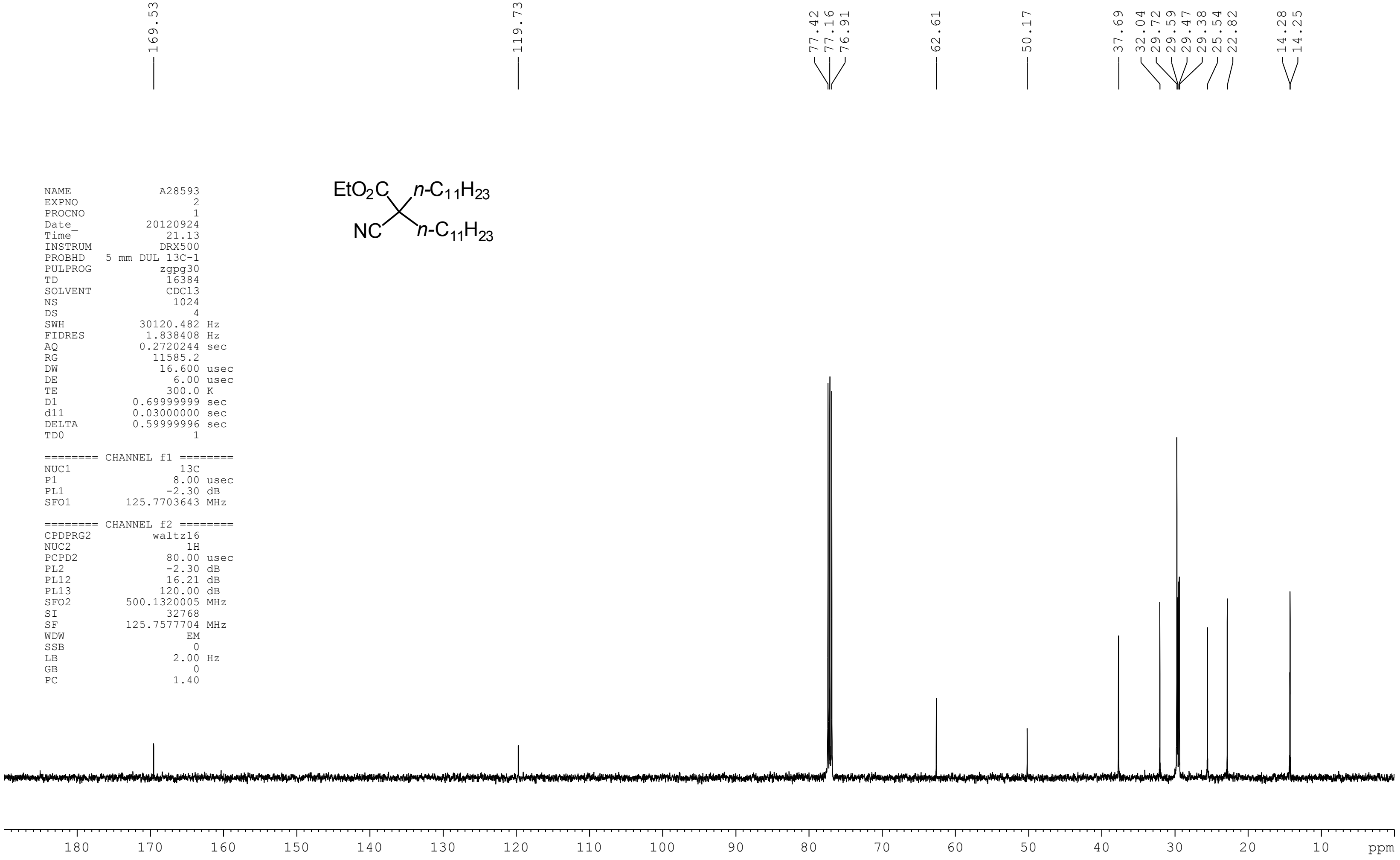
======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1               -2.80 dB
PL1W        57.91424179 W
SFO1        100.5976818 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -3.20 dB
PL12              14.30 dB
PL13              15.30 dB
PL2W        15.00949383 W
PL12W        0.26691073 W
PL13W        0.21201472 W
SFO2        400.0316001 MHz
SI                32768
SF          100.5876124 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME              A28593
EXPNO                  1
PROCNO                 1
Date_           20120924
Time               11.57
INSTRUM           DRX500
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10288.065 Hz
FIDRES         0.156983 Hz
AQ            3.1850996 sec
RG                 203.2
DW                48.600 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.70 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300081 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



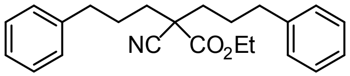
```
NAME             A28593
EXPNO                 2
PROCNO                1
Date_          20120924
Time              21.13
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         1.838408 Hz
AQ            0.2720244 sec
RG              11585.2
DW               16.600 usec
DE                 6.00 usec
TE                300.0 K
D1           0.69999999 sec
d11          0.03000000 sec
DELTA        0.59999996 sec
TD0                   1

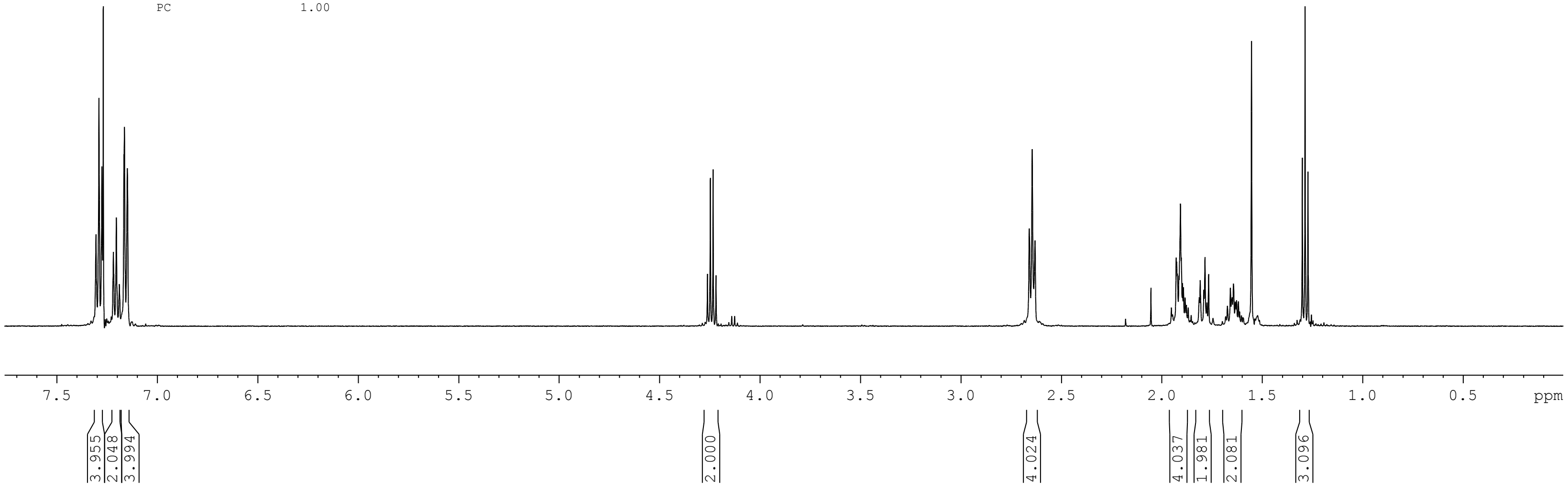
======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1               -2.30 dB
SFO1       125.7703643 MHz

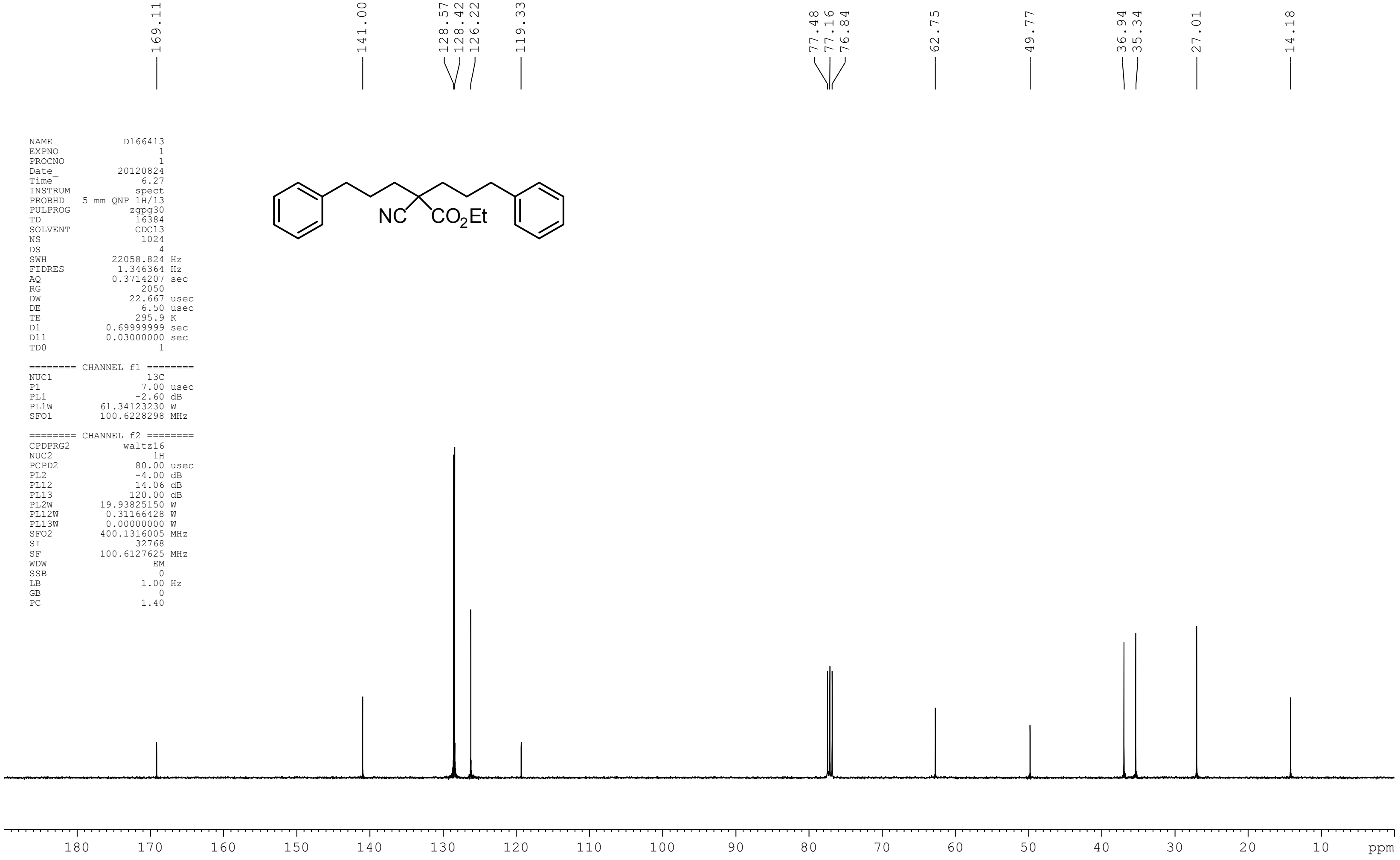
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.30 dB
PL12              16.21 dB
PL13             120.00 dB
SFO2       500.1320005 MHz
SI                32768
SF         125.7577704 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40
```

```
NAME              A28217
EXPNO                  1
PROCNO                 1
Date_           20120822
Time               18.14
INSTRUM           DRX500
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10288.065 Hz
FIDRES          0.156983 Hz
AQ             3.1850996 sec
RG                  1024
DW                48.600 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.70 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300084 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



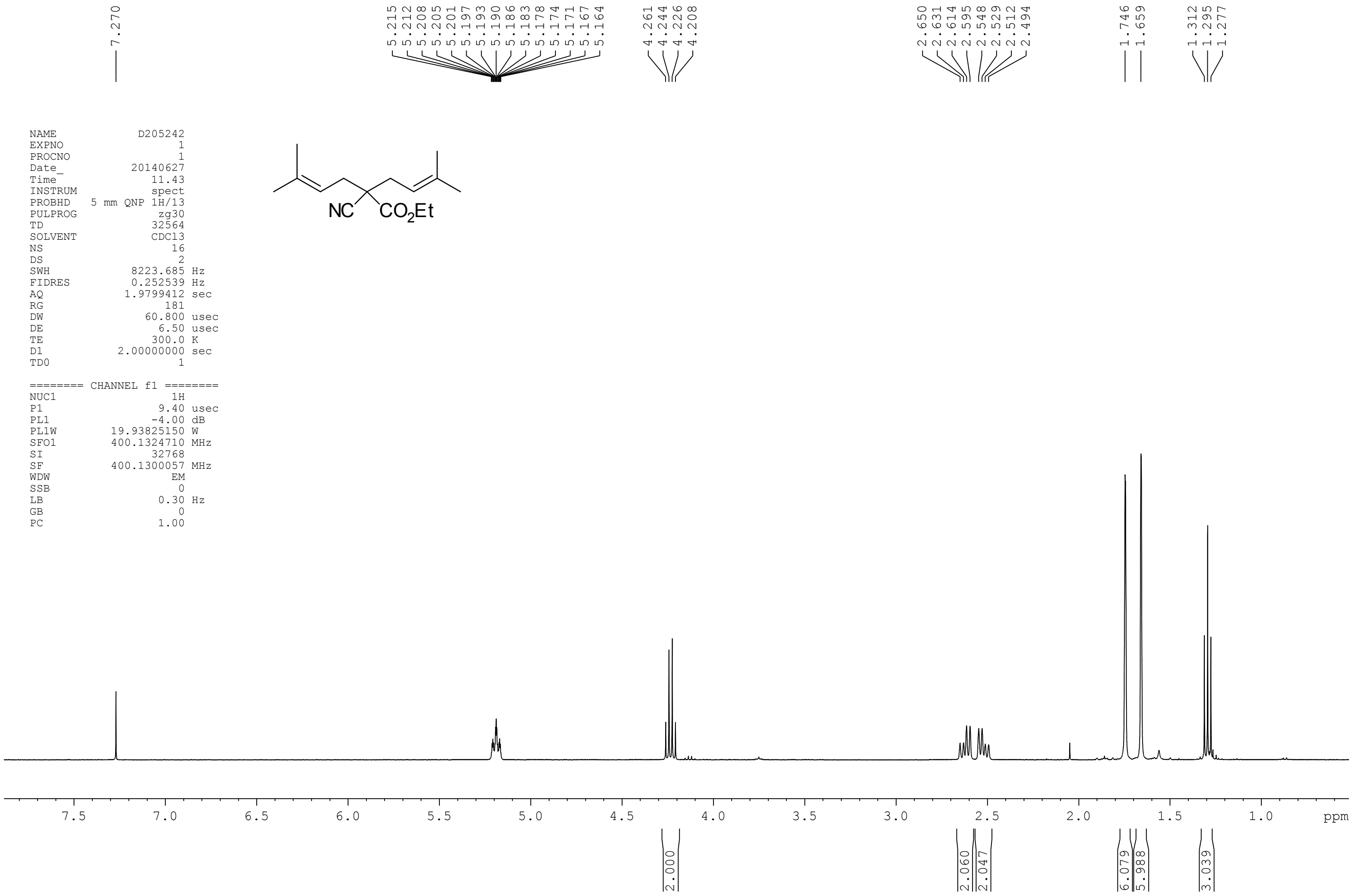




```
NAME             D166413
EXPNO                  1
PROCNO                 1
Date_           20120824
Time                6.27
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 16384
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            22058.824 Hz
FIDRES          1.346364 Hz
AQ             0.3714207 sec
RG                  2050
DW                22.667 usec
DE                  6.50 usec
TE                 295.9 K
D1            0.69999999 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.60 dB
PL1W         61.34123230 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.06 dB
PL13              120.00 dB
PL2W         19.93825150 W
PL12W         0.31166428 W
PL13W         0.00000000 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127625 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



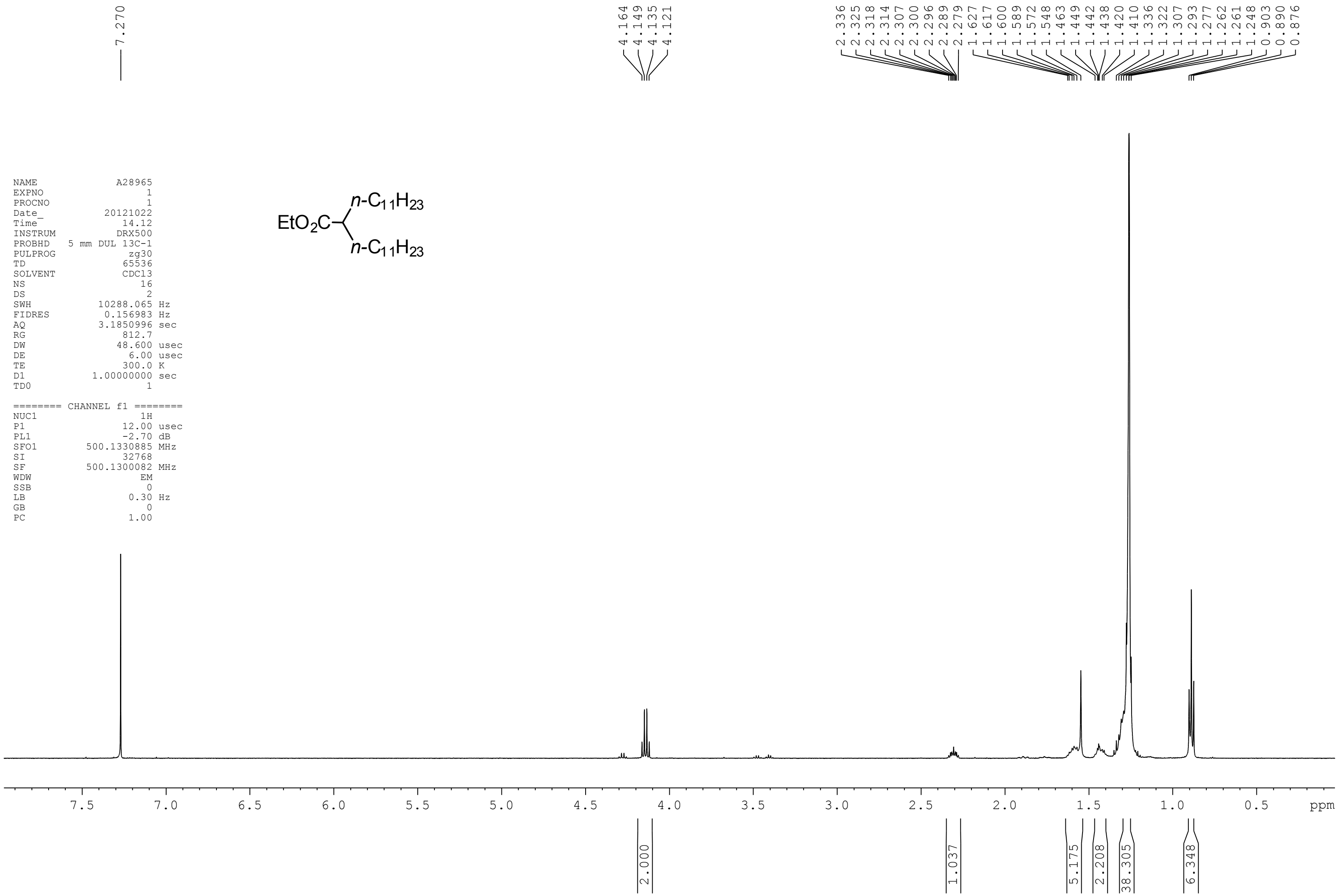
```
NAME           D205242
EXPNO                1
PROCNO               1
Date_         20140627
Time             11.43
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               32564
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8223.685 Hz
FIDRES        0.252539 Hz
AQ           1.9799412 sec
RG                 181
DW              60.800 usec
DE                6.50 usec
TE               300.0 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                9.40 usec
PL1              -4.00 dB
PL1W       19.93825150 W
SFO1       400.1324710 MHz
SI               32768
SF         400.1300057 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

```
NAME            D205846
EXPNO                 1
PROCNO                1
Date_          20140709
Time               0.13
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                300.0 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

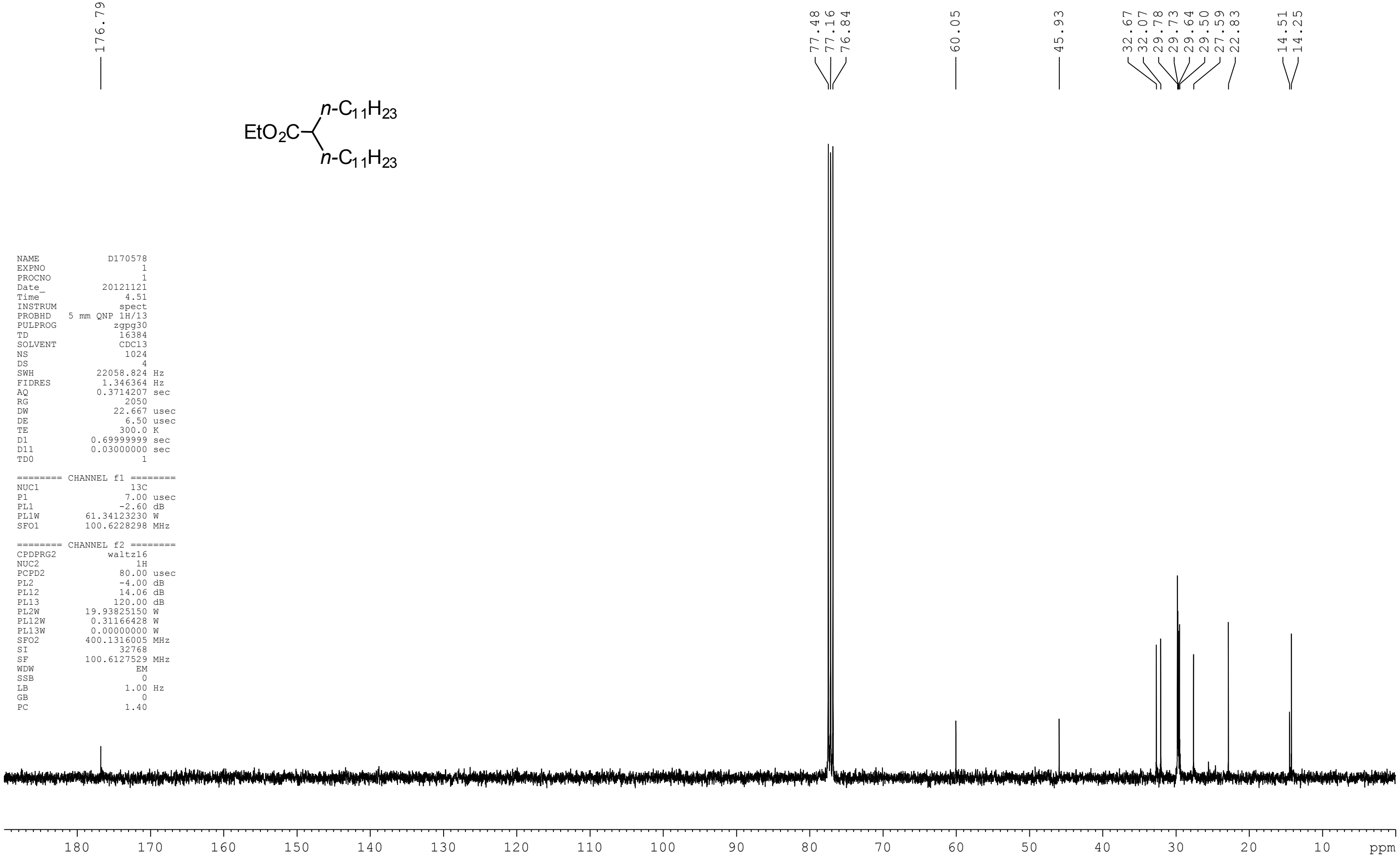
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127610 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            A28965
EXPNO                1
PROCNO               1
Date_         20121022
Time             14.12
INSTRUM         DRX500
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10288.065 Hz
FIDRES        0.156983 Hz
AQ           3.1850996 sec
RG               812.7
DW              48.600 usec
DE                6.00 usec
TE               300.0 K
D1          1.00000000 sec
TD0                  1

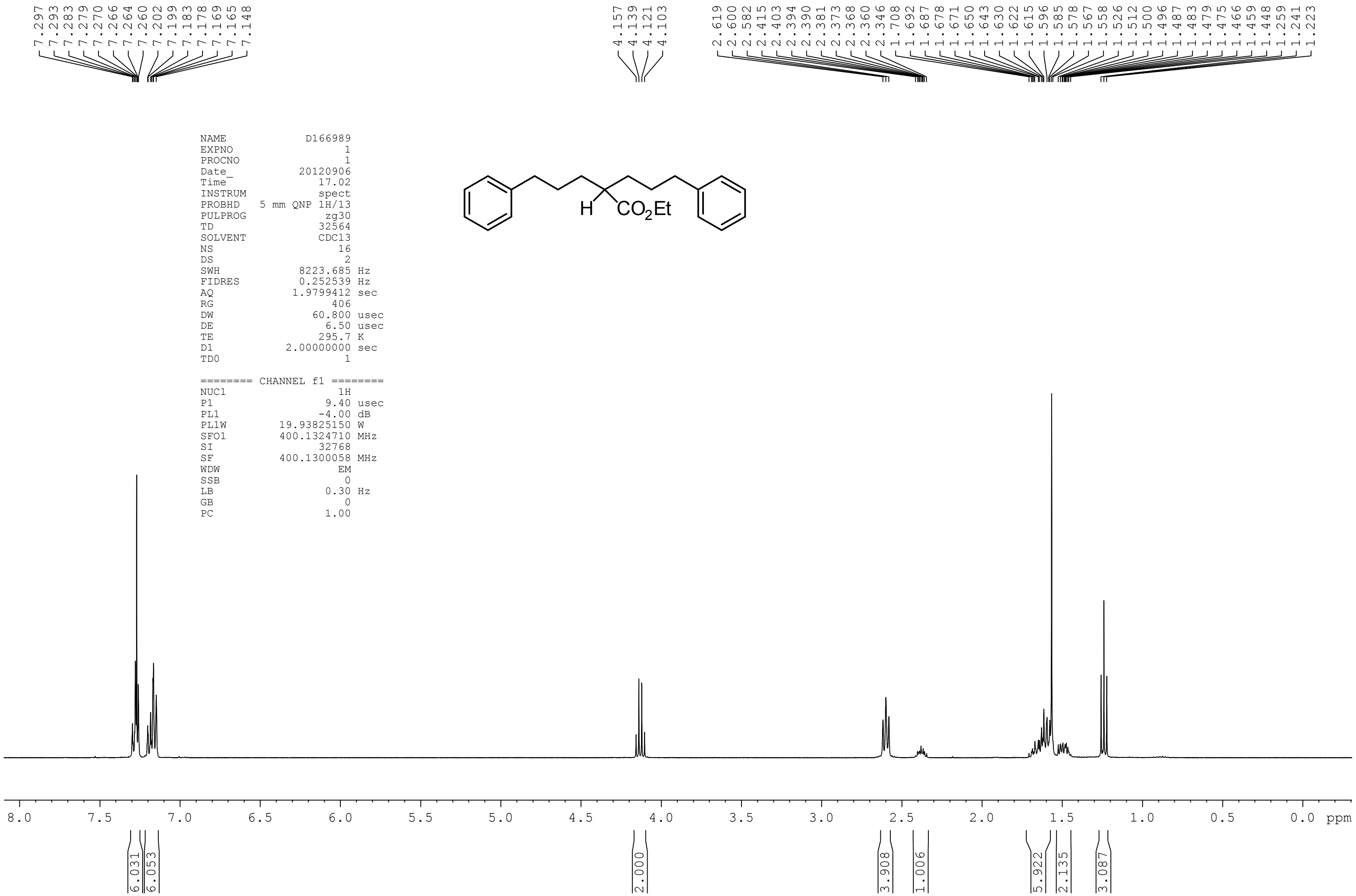
======== CHANNEL f1 ========
NUC1                1H
P1               12.00 usec
PL1              -2.70 dB
SFO1       500.1330885 MHz
SI               32768
SF         500.1300082 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



```
NAME            D170578
EXPNO                 1
PROCNO                1
Date_          20121121
Time               4.51
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               16384
SOLVENT           CDCl3
NS                1024
DS                   4
SWH          22058.824 Hz
FIDRES        1.346364 Hz
AQ           0.3714207 sec
RG                2050
DW              22.667 usec
DE                6.50 usec
TE               300.0 K
D1          0.69999999 sec
D11         0.03000000 sec
TD0                  1

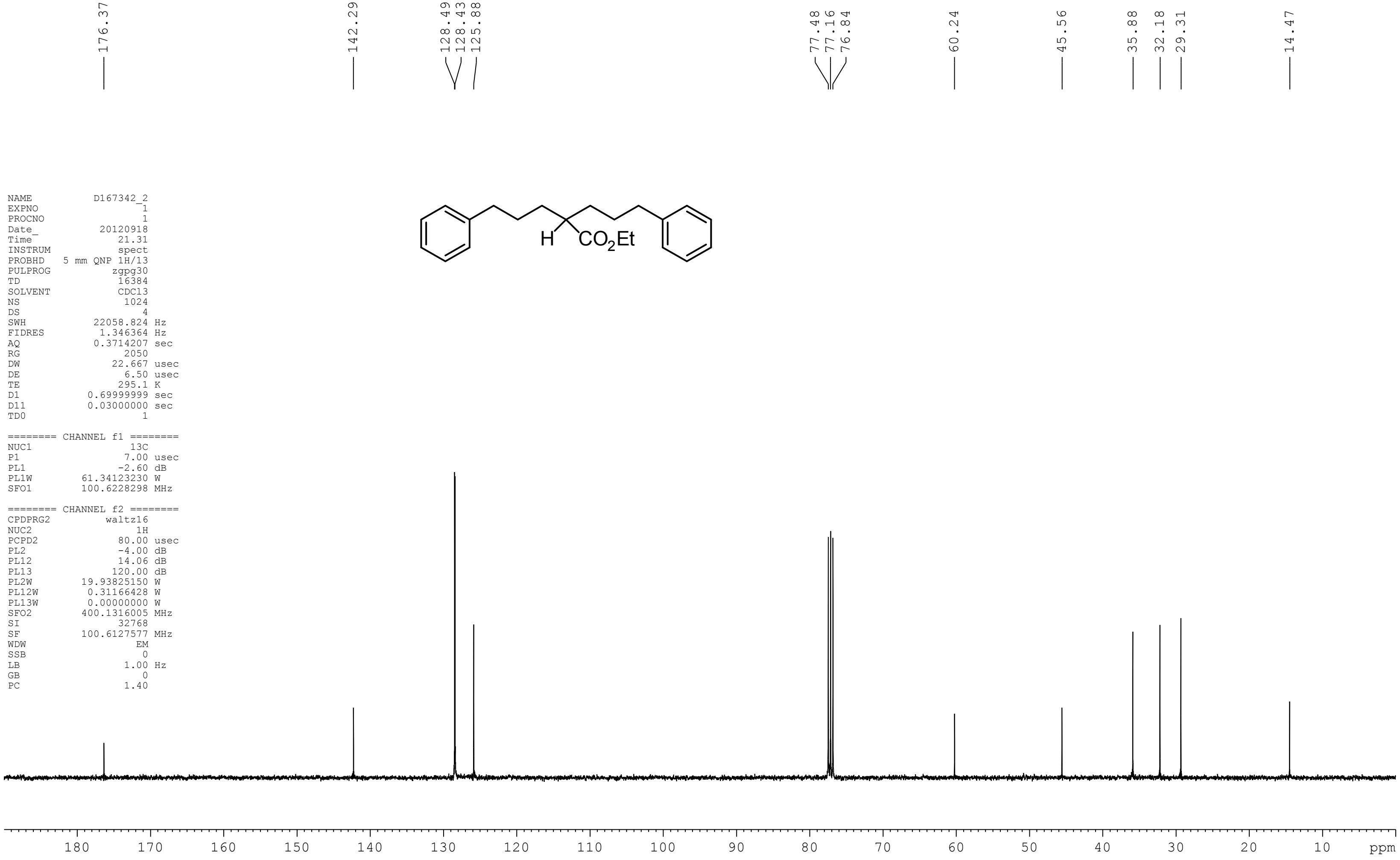
======== CHANNEL f1 ========
NUC1               13C
P1                7.00 usec
PL1              -2.60 dB
PL1W       61.34123230 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -4.00 dB
PL12             14.06 dB
PL13            120.00 dB
PL2W       19.93825150 W
PL12W       0.31166428 W
PL13W       0.00000000 W
SFO2       400.1316005 MHz
SI               32768
SF         100.6127529 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



```
NAME            D166989
EXPNO                 1
PROCNO                1
Date_          20120906
Time              17.02
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  406
DW               60.800 usec
DE                 6.50 usec
TE                295.7 K
D1           2.00000000 sec
TD0                   1

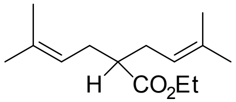
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300058 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



```
NAME          D167342_2
EXPNO                 1
PROCNO                1
Date_          20120918
Time              21.31
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                16384
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                 2050
DW               22.667 usec
DE                 6.50 usec
TE                295.1 K
D1           0.69999999 sec
D11          0.03000000 sec
TD0                   1

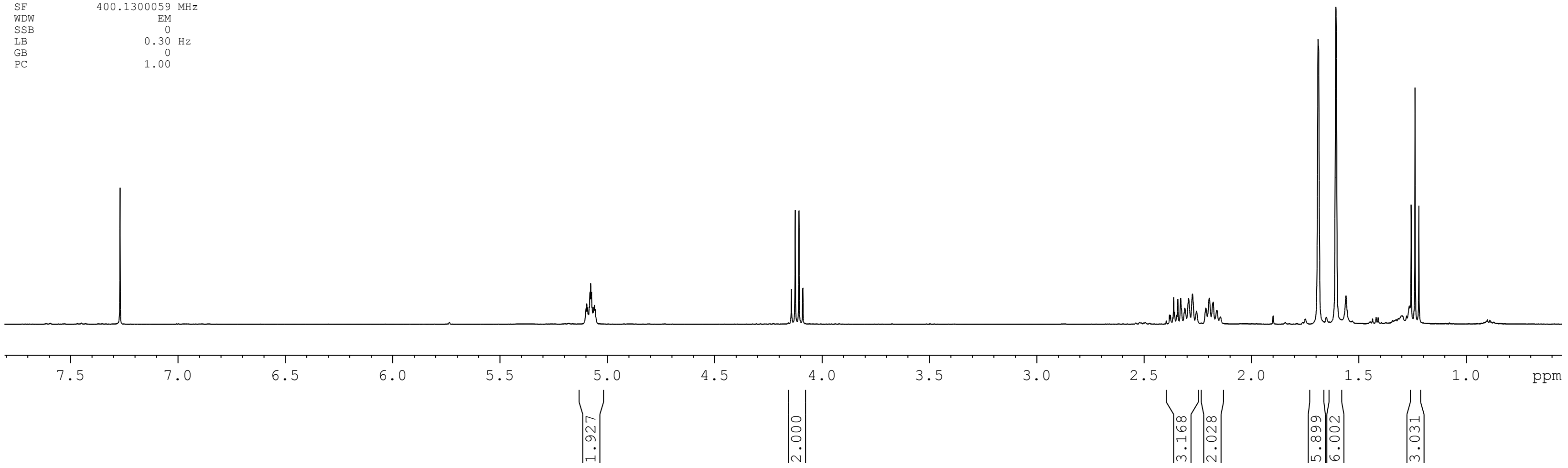
======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.60 dB
PL1W        61.34123230 W
SFO1        100.6228298 MHz

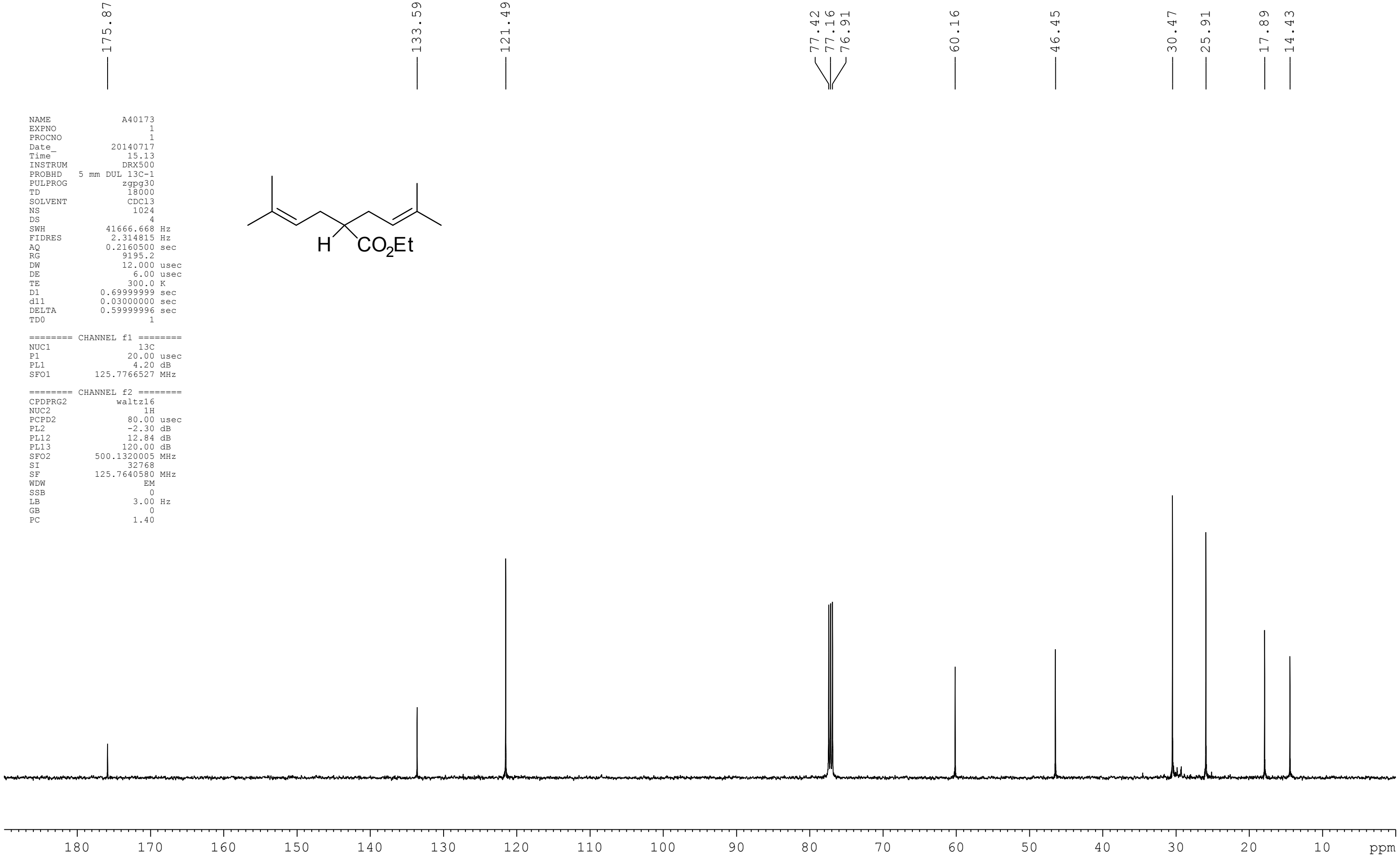
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              14.06 dB
PL13             120.00 dB
PL2W        19.93825150 W
PL12W        0.31166428 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127577 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



```
NAME            D205891
EXPNO                 1
PROCNO                1
Date_          20140708
Time              16.44
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32564
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.252539 Hz
AQ            1.9799412 sec
RG                  256
DW               60.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -4.00 dB
PL1W        19.93825150 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300059 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

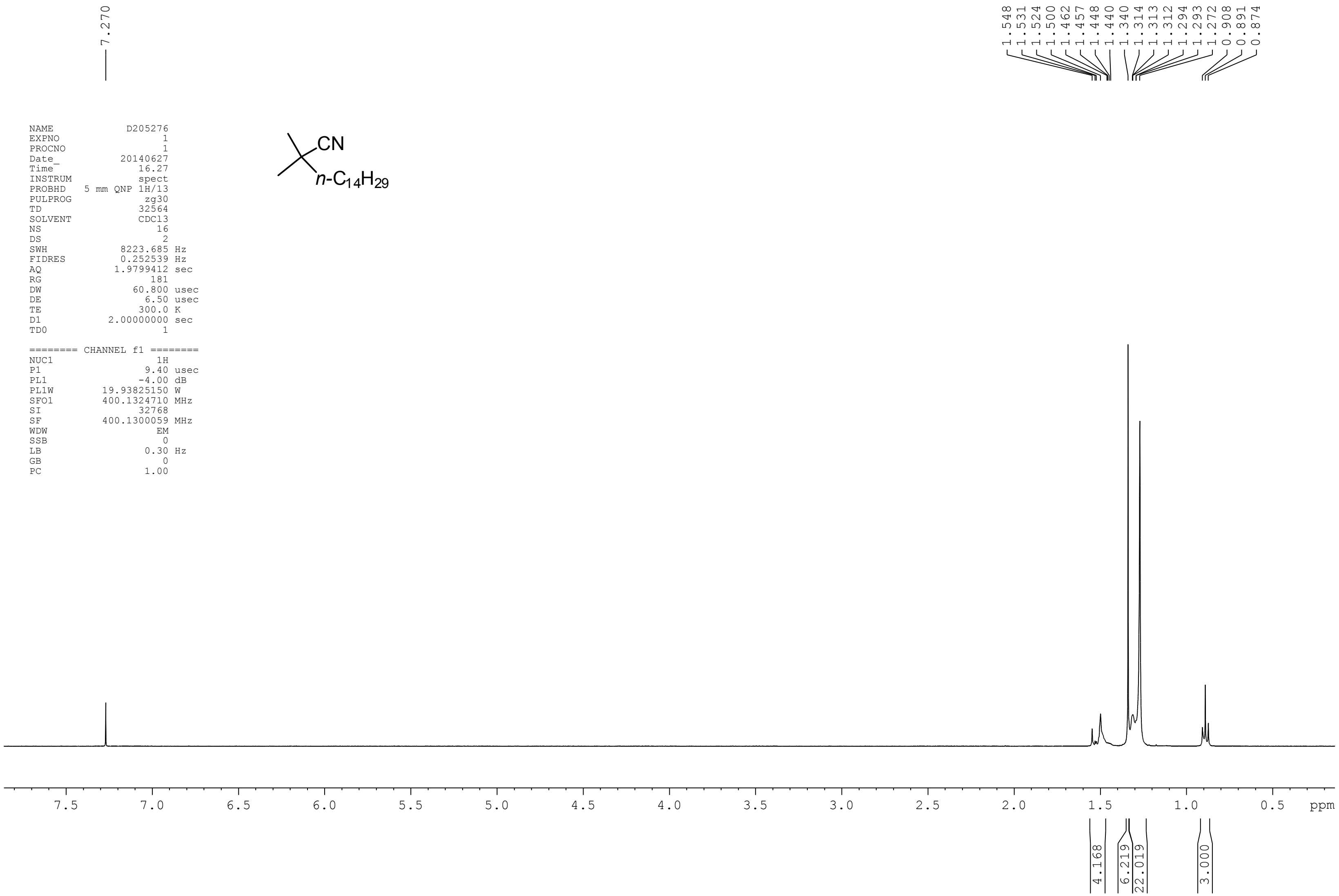




```
NAME             A40173
EXPNO                 1
PROCNO                1
Date_          20140717
Time              15.13
INSTRUM          DRX500
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                18000
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH          41666.668 Hz
FIDRES        2.314815 Hz
AQ           0.2160500 sec
RG               9195.2
DW               12.000 usec
DE                 6.00 usec
TE                300.0 K
D1          0.69999999 sec
d11         0.03000000 sec
DELTA       0.59999996 sec
TD0                   1

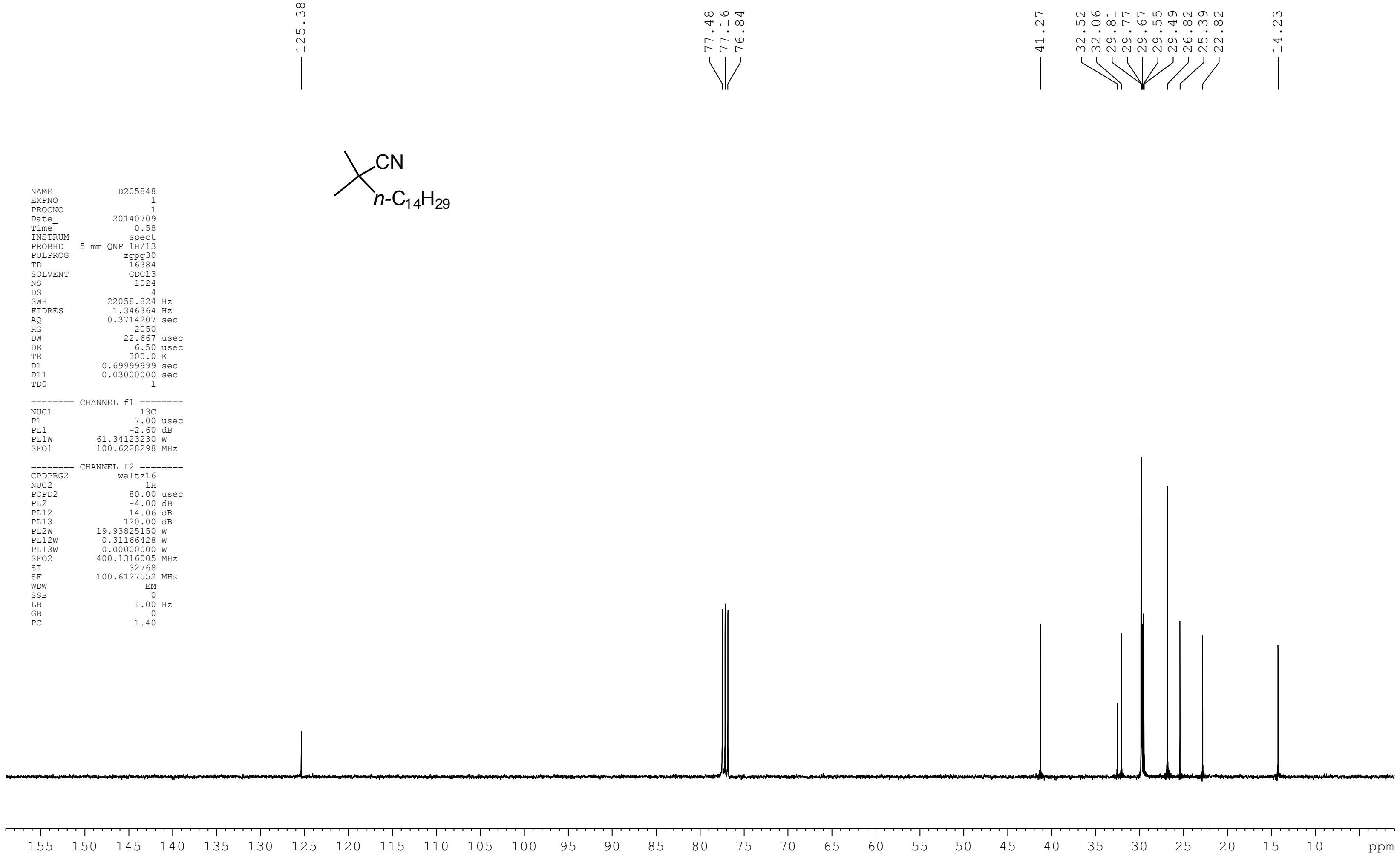
======== CHANNEL f1 ========
NUC1                13C
P1                20.00 usec
PL1                4.20 dB
SFO1        125.7766527 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.30 dB
PL12              12.84 dB
PL13             120.00 dB
SFO2        500.1320005 MHz
SI                32768
SF          125.7640580 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
```



```
NAME             D205276
EXPNO                  1
PROCNO                 1
Date_           20140627
Time               16.27
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32564
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.252539 Hz
AQ             1.9799412 sec
RG                   181
DW                60.800 usec
DE                  6.50 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.40 usec
PL1                -4.00 dB
PL1W         19.93825150 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300059 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
NAME             D205848
EXPNO                  1
PROCNO                 1
Date_           20140709
Time                0.58
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 16384
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           22058.824 Hz
FIDRES         1.346364 Hz
AQ            0.3714207 sec
RG                  2050
DW                22.667 usec
DE                  6.50 usec
TE                 300.0 K
D1            0.69999999 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.60 dB
PL1W         61.34123230 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.06 dB
PL13              120.00 dB
PL2W         19.93825150 W
PL12W         0.31166428 W
PL13W         0.00000000 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127552 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```