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Materials and Methods

Unless stated otherwise, reactions were performed under a N_2 atmosphere using flame- or oven-dried glassware and stir bars. Ambient temperature refers to 22-26 °C. Higher than ambient temperatures were maintained using pre-heated oil baths. Lower temperatures were maintained using the following dewar baths: MeCN/$CO_{2(s)}$ (−40 °C) and MeOH/ice (−20 °C). Dichloromethane and tetrahydrofuran (both of Optima® grade) were purchased from Fisher Scientific and dried by passage through an activated alumina column solvent purification system (Innovative Technology Inc. Pure Solv^{TM}). Cyclohexane (anhydrous, 99.5%) was purchased from Sigma-Aldrich and used as received. Commercially obtained reagents were used as received, unless stated otherwise. *tert*-Butyl hypochlorite (> 98.0%) and tricyclo[5.2.1.$0^{2,6}$]decan-8-one (> 95.0%, GC) were purchased from TCI America. Dess-Martin periodinane (97%) and dichloramine-T (97%) were purchased from Sigma-Aldrich. *N*-bromosuccinimide was recrystallized from H_2O and dried overnight under high vacuum. *n*-Butyllithium was purchased from Sigma-Aldrich and titrated using menthol/2,2'-bipyridine. Ethanethiol was degassed by three freeze-pump-thaw (FPT) cycles and stored in a Schlenk flask under a N_2 atmosphere; this reagent was degassed by one cycle of FPT prior to each usage. Tritylhydrazide hydrochloride was prepared according to the procedure of Baldwin *et al*.,[1] using hydrazine monohydrate (Sigma-Aldrich, reagent grade, 98%) and ca. 0.90-0.95 equiv. HCl (Fisher Scientific, Certified ACS Plus).

Reactions and compounds were visualized on EMD Millipore silica gel 60 Å F254 plates by UV fluorescence quenching (254 nm), I_2/SiO_2, PMA, or Seebach's stain. Flash column chromatography was performed on SiliCycle SiliaFlash P60 (40-63 μm particle size) using ACS or HPLC grade solvents

[1] Baldwin, J. E.; Adlington, R. M.; Bottaro, J. C.; Kolhe, J. N.; Newington, I. M.; Perry, M. W. D. *Tetrahedron* **1986**, *42*, 4235.

purchased from Fisher Scientific. ^{1}H and ^{13}C NMR spectra were measured on Bruker DRX-500 and DMX-500 spectrometers (at 500 MHz and 125 MHz, respectively) at 294-300 K. ^{1}H spectra were calibrated from internal standard TMS (δ 0.0) or solvent resonance ($CHCl_3$: 7.26, C_6D_6: 7.16). ^{13}C spectra were calibrated from solvent resonance ($CHCl_3$: 77.0). NMR data are reported as: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), and integration). ^{13}C chemical shifts arising from $^{37}Cl/^{35}Cl$ isotope effects are indicated in parentheses.[2] High-resolution mass spectral analysis was measured on an Agilent Technologies 6224 TOF LC/MS (electrospray ionization). IR spectra were measured on Nicolet 6700 FTIR spectrometer and are reported as frequency of absorption (cm^{-1}). Optical rotations were measured on a Perkin Elmer 141 polarimeter using a 100 mm path-length cell.

[2] Aliev, A. E.; Harris, K. D. M. *Magn. Reson. Chem.* **1993**, *31*, 54.

Figure S1 – Selected Variable Temperature NMR Spectra

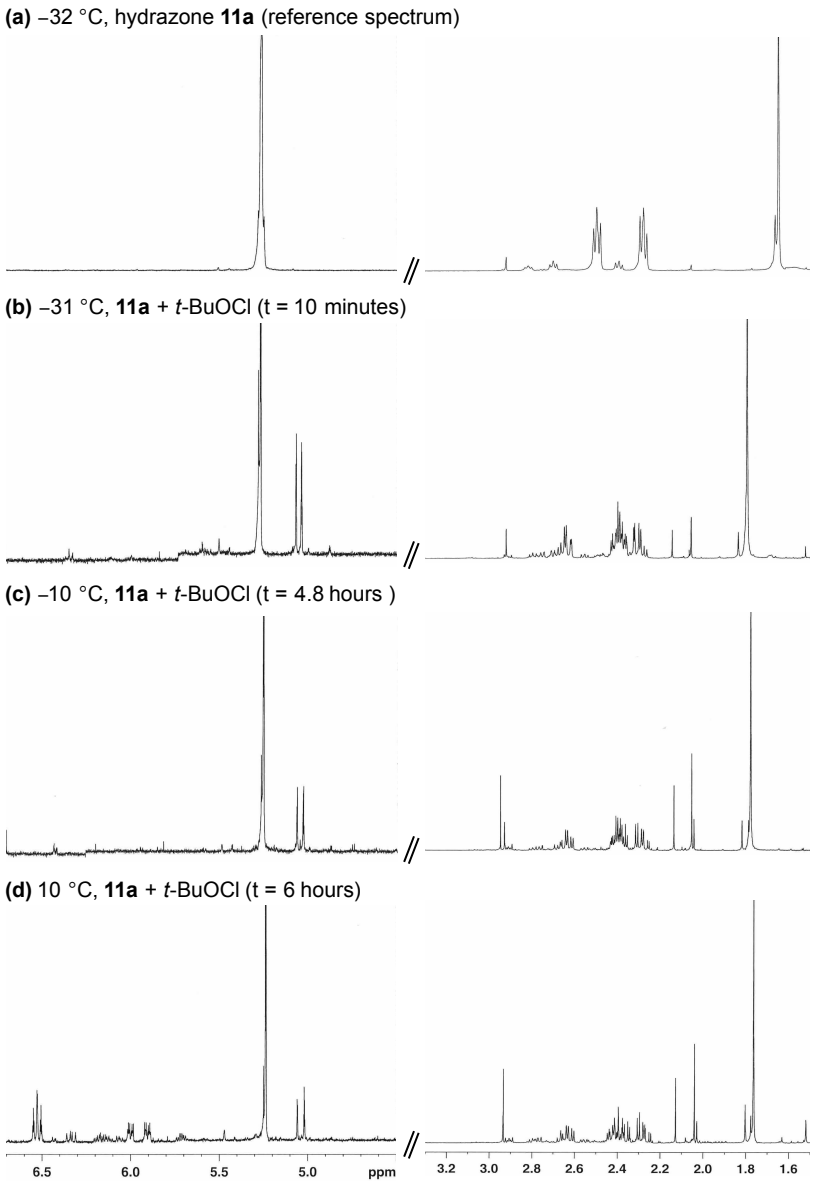
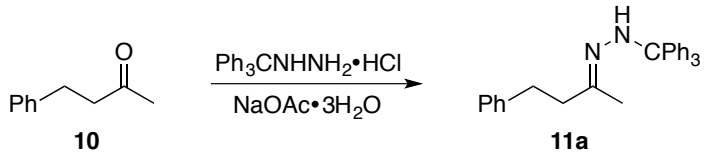


Figure S1. ^{1}H NMR evolution of **11a** + *t*-BuOCl (ca. 1.1 equiv.) in the absence of EtSH (CD_2Cl_2); Time (t) from addition of *t*-BuOCl at –78 °C. **(a)** Spectrum of hydrazone **11a** at –32 °C. **(b) 11a** + *t*-BuOCl following injection into –31 °C pre-cooled probe. **(c)** Reaction warmed to –10 °C without appreciable change. **(d)** Marked decomposition at 10 °C.

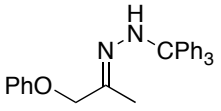
Preparation of Trityl Hydrazones

Note: The hydrazones are prone to decomposition in the presence of air at ambient temperature. Storage under an inert atmosphere at −20 °C is recommended. Trityl hydrazones are promptly subjected to halogenation conditions and are used without purification.



10 **11a**

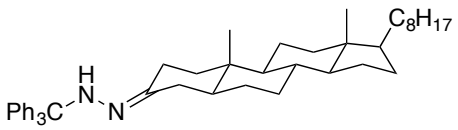
Representative Procedure: A 50 mL 14/20 recovery flask equipped with a 1-inch oval-shaped Teflon-coated magnetic stir bar was charged with Ph_3CNHNH_2•HCl (1.01g, 3.25 mmol, 1.0 equiv.) and MeOH (11 mL). The resulting light orange solution was then treated with NaOAc•$3H_2O$ (3.24M in H_2O, 1.5 mL, 4.86 mmol, 1.5 equiv.), and a color change to light yellow is observed. Benzylacetone (475 mg, 3.21 mmol, 1.0 equiv.) dissolved in 0.5 mL DCM (Fisher Scientific, Certified ACS, stabilized) was added, and the reaction immediately turned cloudy. Ketone addition was quantitated with additional DCM (2 x 0.5 mL), and the system was subsequently evacuated and backfilled with N_2 three times. Under increased N_2 flow, the reaction was stirred overnight at ambient temperature as vigorously as possible (> 600 rpm), away from light, and under a stream of N_2. The resulting white slurry was filtered using a Büchner funnel, and the hydrazone was washed sequentially with H_2O then MeOH. After drying on high vacuum (2 mmHg), **11a** was obtained as an off-white solid (953 mg, 2.36 mmol, 74% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.11 (m, 18H), 7.05-7.00 (m, 2H) 5.41 (bs, 1H), 2.62-2.55 (m, 2H), 2.43-2.36 (m, 2H), 1.70 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.6, 146.1, 142.1, 129.2, 128.3, 128.1, 127.6, 126.4, 125.6, 72.6, 40.4, 32.7, 15.0; IR (Neat film): 1492, 1448, 749, 699 cm^{-1}; HRMS (ES) calcd for $C_{29}H_{28}N_2$ $[M+H]^+$: 405.2325, found 405.2316.



11b

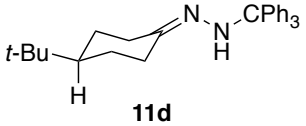
From phenoxyacetone (231 mg, 1.54 mmol), trityl hydrazone **11b** was obtained as a white solid (390 mg, 0.96 mmol, 62% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35-7.20 (m, 15H), 7.19-7.13 (m, 2H), 6.88 (tt, J = 1.0, 7.5 Hz, 1H), 6.79-6.73 (m, 2H), 5.71 (bs, 1H), 4.46 (s, 2H), 1.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.6, 145.9, 144.0, 129.3, 129.1, 127.7, 126.7, 120.6, 115.0, 72.7, 72.2, 11.7; IR

(Neat film): 3058, 1598, 1494, 1447, 1213, 1172, 1079, 1031, 901, 754, 701 cm^{-1}; HRMS (ES) calcd for $C_{28}H_{26}N_2O$ $[M+H]^+$: 407.2118, found 407.2114.



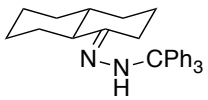
11c

From cholestanone (307 mg, 0.79 mmol), trityl hydrazone **11c** was obtained as a yellow solid as a crude mixture of hydrazone isomers (399 mg, 0.62 mmol, 78% yield). This mixture was deemed acceptably pure and was subsequently subjected to chlorinative conditions. Diagnostic NMR signals: ^{1}H NMR (500 MHz, $CDCl_3$, * denotes isomeric signal): δ 7.50-7.00 (m, 15H + 15H*), 5.53 (bs, 1H), 5.53 (bs, 1H*); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.6, 152.4*, 72.7, 72.7*.



11d

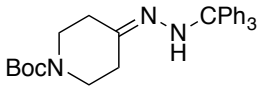
From 4-*tert*-butylcyclohexanone (482 mg, 3.12 mmol), trityl hydrazone **11d** was obtained as a white/light yellow solid (962 mg, 2.34 mmol, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39-7.34 (m, 6H), 7.29-7.24 (m, 6H), 7.23-7.17 (m, 3H), 5.53 (bs, 1H), 2.71-2.64 (m, 1H), 2.29-2.21 (m, 1H), 1.96 (app td, J = 5.0, 13.5 Hz, 1H), 1.83-1.70 (m, 2H), 1.62 (app td, J = 5.0, 13.5 Hz, 1H), 1.16 (app tt, J = 3.0, 12.0 Hz, 1H), 0.96-0.80 (m, 2H), 0.81 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.2, 145.9, 129.2, 127.6, 126.4, 72.8, 47.4, 35.3, 32.5, 27.8, 27.5, 26.6, 24.8; IR (Neat film): 3507, 2954, 1716, 1491, 1447, 1366, 1221, 1034, 905, 759, 700 cm^{-1}; HRMS (ES) calcd for $C_{29}H_{34}N_2$ $[M+H]^+$: 411.2795, found 405.2788.



11e

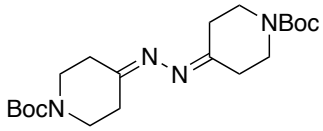
From *trans*-1-decalone (242 mg, 1.59 mmol), trityl hydrazone **11e** was obtained as a white/light yellow solid (408 mg, 1.00 mmol, 63% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.31 (m, 6H), 7.28-7.23 (m, 6H), 7.23-7.18 (m, 3H), 5.58 (bs, 1H), 2.76-2.68 (m, 1H), 1.82-1.75 (m, 1H), 1.68-1.51 (m, 7H), 1.28-0.80 (m, 7H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.6, 146.2, 129.4, 127.4, 126.3, 72.9, 49.5, 44.4,

34.4, 33.7, 27.1, 26.1, 26.1, 25.6, 24.9; IR (Neat film): 3057, 2924, 2851, 1597, 1491, 1446, 1033, 905, 731, 700 cm^{-1}; HRMS (ES) calcd for $C_{29}H_{32}N_2$ $[M+H]^+$: 409.2638, found 409.2633.



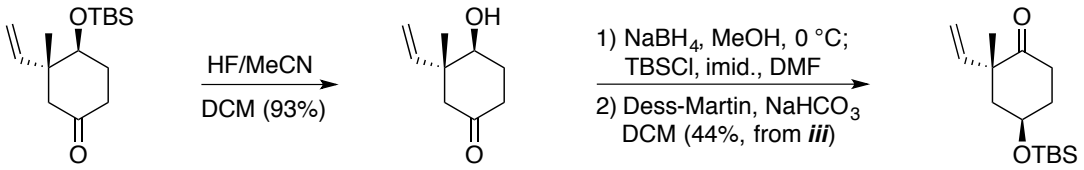
11f

From *N*-Boc-4-piperidone, trityl hydrazone **11f** was obtained as a white solid with a 7 mol % impurity, whose identity is assigned to that of corresponding azine ***i***. The hydrazone was used without further purification. ^1H NMR (500 MHz, $CDCl_3$): δ 7.37-7.31 (m, 6H), 7.31-7.24 (m, 6H), 7.24-7.19 (m, 3H), 5.54 (bs, 1H), 3.49-3.39 (m, 2H), 3.39-2.29 (m, 2H), 2.37-2.20 (m, 4H), 1.45 (s, 9H).



i

A 10 mL 14/20 recovery flask equipped a Teflon-coated magnetic stir bar was charged sequentially with *N*-Boc-4-piperidone (246 mg, 1.23 mmol, 1 equiv.), EtOH (1.25 mL), and $NH_2NH_2 \bullet H_2O$ (0.03 mL, 0.62 mmol, 0.5 equiv.), and heated to reflux for 7 hours with an attached condenser. Following filtration through a Büchner funnel, washing with EtOH, and drying under high vacuum for 2 hours, azine ***i*** was obtained as a white solid (182 mg, 0.46 mmol, 75% yield). ^1H NMR (500 MHz, $CDCl_3$): δ 3.61 (t, J = 6.0 Hz, 4H), 3.52 (t, J = 6.0 Hz, 4H), 2.57 (t, J = 6.0, 4H), 2.45 (t, J = 6.0 Hz, 4H), 1.48 (s, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 163.6, 154.6, 78.0, 44.1, 42.5, 34.5, 28.4, 28.1; IR (Neat film): 1683, 1431, 1364, 1240, 1179 cm^{-1}; HRMS (ES) calcd for $C_{20}H_{34}N_4O_4$ $[M+H]^+$: 395.2653, found 395.2659.



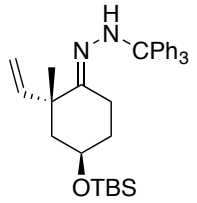
ii ***iii*** ***iv***

A 500 mL 24/40 round-bottom flask equipped with a Teflon-coated magnetic stir bar was charged with ketone ***ii*** (0.52 g, 1.94 mmol, 1 equiv.) and DCM (200 mL). The resulting solution was then treated with 80 mL of a 10% solution of HF in MeCN and stirred at ambient temperature for 21.5 hours. The reaction was carefully quenched with a total of 120 mL saturated aqueous $NaHCO_3$. In a 500 mL

separatory funnel, the reaction was partitioned between 30 mL saturated aqueous $NaHCO_3$ and 30 mL DCM. The aqueous layer was separated and extracted twice more with 40 mL portions of DCM. The combined organic layers were washed with 80 mL brine and dried over $MgSO_4$. Flash chromatography (2:3 hexanes:EtOAc) afforded alcohol ***iii*** as clear, light yellow liquid (0.28 g, 1.82 mmol, 93% yield). R_f = 0.30 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.75 (dd, *J* = 11.0, 17.5 Hz, 1H), 5.14 (dd, *J* = 1.0, 11.0 Hz, 1H), 5.10 (dd, *J* = 1.0, 17.5 Hz, 1H), 3.84 (app td, *J* = 3.5, 7.0 Hz, 1H), 2.52 (dddd, *J* = 2.0, 6.5, 8.5, 15.0 Hz, 1H), 2.44 (dd, *J* = 1.5, 14.5 Hz, 1H), 2.38 (dd, J = 1.0, 14.5 Hz, 1H), 2.30 (dddd, *J* = 1.5, 6.0, 7.5, 15.0 Hz, 1H), 2.11 (dddd, *J* = 3.5, 6.0, 8.0, 14.0, 1H) 1.98-1.87 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 210.2, 143.8, 114.7, 72.2, 48.0, 45.7, 36.8, 28.9, 20.4; IR (Neat film): 3451, 2960, 1703, 1640, 1421, 1293, 1235, 1065, 1029, 969, 921 cm^{-1}; HRMS (ES) calcd for $C_9H_{14}O_2$ $[M+H]^+$: 155.1067, found 155.1063.

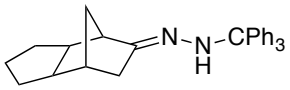
A 100 mL 14/20 recovery flask equipped with a Teflon-coated magnetic stir bar was charged with alcohol ***iii*** (0.83 g, 5.38 mmol, 1 equiv.) and MeOH (22 mL). The solution was cooled in an ice bath and subsequently treated with $NaBH_4$ (0.63 g, 16.65 mmol, 3 equiv.). After 2 hours, the reaction was quenched with 35 mL saturated aqueous NH_4Cl. In a 500 mL separatory funnel, the reaction was extracted into DCM (4 x 75 mL). The combined organic layers were dried over Na_2SO_4 and concentrated. The crude diol was subsequently dissolved in DMF (27 mL) and treated with imidazole (0.73 g, 10.76 mmol, 2 equiv.) and TBSCl (0.83 g, 5.51 mmol, 1 equiv.). After stirring for 12.5 hours at ambient temperature, the reaction was quenched with 100 mL saturated aqueous NH_4Cl and extracted with 2:1 hexanes:EtOAc (3 x 75 mL). The combined organics were washed with H_2O (2 x 50 mL), brine (50 mL), and dried over $MgSO_4$. The crude residue was subjected to careful flash chromatography (two successive columns – first using 3:1 hexanes:Et_2O, then a second using 85:15 hexanes:EtOAc) to give 0.79 g of a 10:1 diastereomeric mixture of mono-silylated diol as a clear, light yellow liquid (2.92 mmol, 54% yield over 2 steps). A 100 mL 14/20 recovery flask equipped with a Teflon-coated magnetic stir was charged with the previously purified mono-silylated diol, DCM (29 mL), $NaHCO_3$ (0.76g, 9.05 mmol, 3.1 equiv.), and Dess-Martin periodinane (1.86 g, 4.38 mmol, 1.5 equiv.). After stirring at ambient temperature for 2.5 hours, the reaction was partitioned between 50 mL saturated aqueous $NaHCO_3$ and 50 mL DCM. The aqueous layer was separated, extracted with DCM (2 x 40 mL), and dried over Na_2SO_4. The crude residue was then subjected to careful flash chromatography (95:5 hexanes:Et_2O) to yield ketone ***iv*** as a single diastereomer (0.64 g, 2.38 mmol, 82% yield). R_f =

0.70 (3:1 hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.90 (dd, *J* = 11.0, 17.5 Hz, 1H), 5.14 (d, *J* = 11.0 Hz, 1H), 5.00 (d, *J* = 17.5 Hz, 1H), 4.19 (app tt, *J* = 4.0, 10.0 Hz, 1H), 2.56 (ddd, *J* = 6.0, 13.0, 14.5 Hz, 1H), 2.39 (app dt, *J* = 4.5, 14.5 Hz, 1H), 2.12 (ddd, *J* = 3.0, 4.0, 13.5, 1H), 2.10-2.03 (m, 1H), 1.75 (app t(dd), *J* = 4.5, 10.0, 13.0 Hz, 1H), 1.69 (dd, *J* = 10.0, 13.5 Hz, 1H), 1.16 (s, 3H), 0.90 (s, 9H), 0.10 (s, 3H), 0.09 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 211.9, 142.6, 114.8, 66.5, 51.3, 47.4, 36.3, 35.5, 25.8, 24.8, 18.1, −4.7, −4.7; IR (Neat film): 2886, 2858, 1716, 1631, 1472, 1463, 1426, 1370, 1325, 1257, 1106, 1073, 991, 940, 920, 889, 866, 837, 776 cm^{-1}; HRMS (ES) calcd for $C_{15}H_{28}O_2Si$ $[M+H]^+$: 269.1931, found 269.1926.



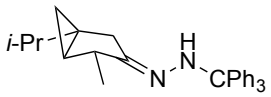
11g

From ketone ***iv*** (199 mg, 0.74 mmol), trityl hydrazone **11g** was obtained as a white solid (255 mg, 0.49 mmol, 66% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38-7.17 (m, 18H), 5.63 (bs, 1H), 5.58 (dd, *J* = 11.0, 17.5 Hz, 1H), 4.83 (dd, *J* = 1.0, 11.0 Hz, 1H), 4.52 (dd, *J* = 1.0, 17.5 Hz, 1H), 3.86 (app tt, *J* = 4.0, 10.5 Hz, 1H), 2.69-2.55 (m, 1H), 1.95-1.71 (m, 3H), 1.38-1.22 (m, 2H), 0.87 (s, 9H), 0.84 (s, 3H), 0.05 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.7, 146.1, 146.1, 129.3, 127.5, 126.4, 112.9, 73.1, 67.4, 48.0, 45.9, 34.6, 27.1, 25.9, 19.9, 18.1, −4.6, −4.6; IR (Neat film): 2929, 2856, 1714, 1491, 1448, 1254, 1105, 1073, 866, 836, 774, 701 cm^{-1}; HRMS (ES) calcd for $C_{34}H_{44}N_2OSi$ $[M+H]^+$: 525.3296, found 525.3292.



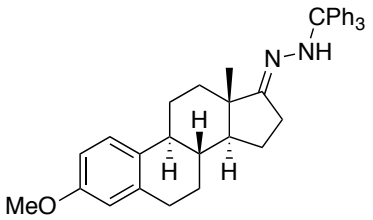
11h

From tricyclo[5.2.1.$0^{2,6}$]decan-8-one (478 mg, 3.18 mmol), trityl hydrazone **11h** was obtained as a white solid (1.047 g, 2.58 mmol, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39-7.32 (m, 6H), 7.30-7.23 (m, 6H), 7.23-7.17 (m, 3H) 5.16 (bs, 1H), 2.43 (bs, 1H), 2.19 (br d, *J* = 4.0Hz, 1H), 1.94 (dd, *J* = 4.5, 15.5 Hz, 1H), 1.89-1.81 (m, 1H), 1.81-1.68 (m, 3H), 1.67-1.58 (m, 2H), 1.49-1.40 (m, 1H), 1.25-1.12 (m, 1H), 1.10 (br d, *J* = 10.5 Hz, 1H), 0.98-0.81 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 160.4, 145.8, 129.0, 127.7, 126.4, 72.8, 47.9, 47.1, 45.2, 40.1, 34.0, 32.4, 32.3, 31.2, 27.7; IR (Neat film): 3056, 2948, 2861, 1596, 1490, 1447, 1185, 1032, 902, 742, 701 cm^{-1}; HRMS (ES) calcd for $C_{29}H_{30}N_2$ $[M+H]^+$: 407.2482, found 407.2473.



11i

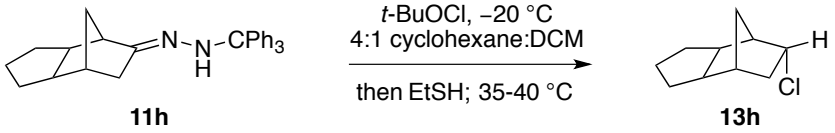
From (−)-α-thujone (484 mg, 3.18 mmol), trityl hydrazone **11i** was obtained as a light yellow solid (825 mg, 2.02 mmol, 64%, yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35-7.30 (m, 6H), 7.29-7.23 (m, 6H), 7.23-7.17 (m, 3H), 5.25 (bs, 1H), 2.44-2.36 (m, 1H), 2.19 (d, *J* = 16.5 Hz, 1H), 2.10 (d, *J* = 16.5 Hz, 1H), 1.30 (sept, *J* = 7.0 Hz, 1H), 0.94 (d, *J* = 7.0 Hz, 3H), 0.91 (d, *J* = 7.0 Hz, 3H), 0.88 (d, *J* = 7.0 Hz, 3H), 0.79 (dd, *J* = 4.0, 8.0 Hz, 1H), 0.48-0.42 (m, 1H), −0.19 (dd, J = 4.5, 4.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 160.2, 145.9, 129.1, 127.7, 126.5, 72.9, 42.4, 32.5, 31.7, 27.9, 26.6, 21.5, 20.1, 19.7, 17.1; IR (Neat film): 2957, 1491, 1448, 1033, 759, 701 cm^{-1}; HRMS (ES) calcd for $C_{29}H_{32}N_2$ $[M+H]^+$: 409.2638, found 409.2630; $[\alpha]^{21.1}_D$ +31.8° (*c* 1, $CHCl_3$).



11j

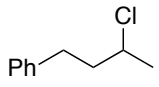
A 25 mL 14/20 recovery flask equipped with a Teflon-coated magnetic stir bar was charged with Ph_3CNHNH_2•HCl (504 mg, 1.62 mmol, 1.0 equiv.) and MeOH (4 mL), and the resulting solution was then treated with NaOAc•3H_2O (3.27M in H_2O, 0.75 mL, 2.45 mmol, 1.5 equiv.). Following addition of *O*-Me estrone (453 mg, 1.59 mmol) and DCM (12 mL), the reaction vessel was evacuated, backfilled with N_2, and stirred at ambient temperature for two weeks. During this time period, an additional total of ca. 260 mg Ph_3CNHNH_2•HCl was added. Conversion was monitored by ^{1}H NMR. Following evaporation of DCM over a stream of N_2, workup as described in the Representative Procedure provided trityl hydrzone **11j** as a white solid (856 mg, 1.58 mmol, 99% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.31 (m, 6H), 7.29-7.23 (m, 6H), 7.23-7.19 (m, 3H), 7.17 (d, *J* = 8.5 Hz, 1H), 6.69 (dd, *J* = 2.5, 8.5 Hz, 1H), 6.61 (d, *J* = 2.5 Hz, 1H), 5.24 (bs, 1H), 2.92-2.78 (m, 2H), 2.29-2.05 (m, 4H), 1.95-1.85 (m, 2H), 1.85-1.77 (m, 1H), 1.49-1.31 (m, 4H), 1.27-1.13 (m, 2H), 0.67 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.5, 157.4, 146.1, 137.8, 132.7, 129.3, 127.5, 126.4, 126.3, 113.7, 111.4, 72.9, 55.2, 52.6, 44.4, 44.2, 38.2, 34.3, 29.8, 27.2, 26.2, 24.9, 23.3, 17.0; IR (Neat film): 2928, 1609, 1498, 1448, 1255, 1033, 901, 747, 702 cm^{-1}; HRMS (ES) calcd for $C_{38}H_{40}N_2O$ $[M+H]^+$: 541.3213, found 541.3208; $[\alpha]^{21.1}_D$ +37.3° (*c* 1, $CHCl_3$).

Reductive Chlorination of Trityl Hydrazones



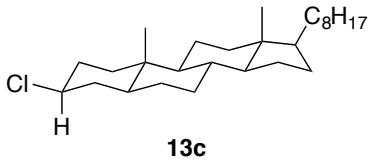
11h **13h**

Representative Procedure: An oven-dried 25 mL 14/20 recovery flask equipped with a Teflon-coated magnetic stir bar was charged with trityl hydrazone **11h** (211 mg, 0.52 mmol, 1.0 equiv), DCM (1 mL), and cyclohexane (4 mL). The resulting colorless solution was evacuated and backfilled with N_2 (3 times) and then cooled to −20 °C (external temperature). *tert*-Butyl hypochlorite (1.24M in DCM, 0.46 mL, 1.1 equiv.) at ambient temperature was added dropwise over 2 minutes to the cooled solution of hydrazone and stirred for 15 minutes. The resulting yellow solution was then frozen in a liquid N_2 bath and degassed by two FPT cycles, each time thawing in a −20 °C bath. After backfilling with N_2, the reaction was maintained at an external temperature ≤ −15 °C for at least 20 minutes. During this time EtSH was degassed once by FPT. Excess EtSH (3 mL) at ambient temperature was added to the cooled reaction without appreciable change, and the reaction flask was subsequently transferred to a pre-heated 35-40°C oil bath. After 1.5 hours, significant color dissipation was observed, and the reaction was allowed to cool to ambient temperature. In a 60 mL separatory funnel, the reaction was partitioned between 20 mL 1:1 H_2O:brine and 20 mL 1:1 Et_2O:pentane. The aqueous layer was separated and extracted once with 20 mL 1:1 Et_2O:pentane. The combined organic layers were dried over Na_2SO_4 and carefully concentrated under reduced pressure (ca. 140 mmHg). The crude residue was purified by flash chromatography (100% pentane) to afford chloride **13h** as a colorless oil with 21:1 dr, as measured by its ^{1}H NMR spectrum (73 mg, 0.43 mmol, 83% yield). R_f = 0.65 (100% pentane); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.22 (app dt, J = 4.0, 10.5 Hz, 1H), 2.63-2.53 (m, 1H), 2.25-2.16 (m, 2H), 1.97 (bd, J = 5.0 Hz, 1H), 1.96-1.85 (m, 3H), 1.70-1.62 (m, 1H), 1.49 (app d(dt), J = 2.0, 3.5, 11.0 Hz 1H), 1.29-1.17 (m, 1H), 1.15 (app dt, J = 4.0, 13.5 Hz, 1H), 1.10-1.05 (m, 1H), 1.02-0.86 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (60.6, 60.6), 47.8, 47.7, 41.1, 40.5, 39.7, 32.4, 32.2, 31.8, 27.0; IR (Neat film): 2950, 2863, 1475, 1464, 1449, 1319, 1301, 1260, 953, 922, 888, 849, 767, 749, 674 cm^{-1}; HRMS (ES) calcd for $C_{10}H_{15}Cl$ $[M+Cl]^-$: 205.0556, found 205.0550.



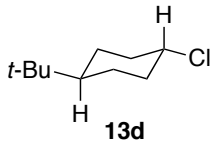
13a

From hydrazone **11a** (405 mg, 1.00 mmol), chloride **13a** was obtained as a colorless oil (138 mg, 0.82 mmol, 82% yield) following purification by flash chromatography (99:1 pentane:EtOAc). Along with **13a**, ≤ 4 mol % diethyl disulfide present accounts for < 3 wt % of the final product. The spectral data obtained are in accord with that in the literature.[3]



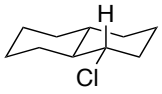
13c

From hydrazone **11c** (209.5 mg, 0.3258 mmol), chloride **13c** was obtained as a white solid (75.3 mg, 0.1850 mmol, 57% yield) following purification by flash chromatography (100% hexanes). The spectral data obtained are in accord with that in the literature.[4]



13d

From hydrazone **11d** (234.6 mg, 0.571 mmol), chloride **13d** was obtained as a colorless oil (68.5 mg, 0.392 mmol, 69% yield) following purification by flash chromatography (100% pentane). The spectral data obtained are in accord with that in the literature.[5]

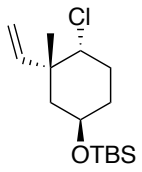


13e

From hydrazone **11e** (238 mg, 0.58 mmol), chloride **13e** was purified by flash chromatography (100% pentane) and obtained as a colorless oil with 1.3:1 dr, as measured by its ^{1}H NMR spectrum (71 mg, 0.41 mmol, 71% yield). R_f = 0.66 (100% pentane); ^{1}H NMR (500 MHz, $CDCl_3$, * denotes minor

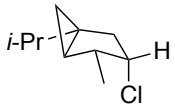
[3] Gaspar, B.; Waser, J.; Carreira, E. M.; *Org. Synth.* **2010**, *87*, 88.
[4] Mondal *et al. J. Org. Chem.* **2013**, *78*, 2118.
[5] Iwasaki *et al. J. Am. Chem. Soc.* **2014**, *136*, 1300.

diastereomer): δ 4.25-4.20 (m, 1H*), 3.58 (ddd, J = 4.25, 10.35, 11.75 Hz, 1H), 2.31-0.80 (m, 16H and 16H*); ^{13}C NMR (125 MHz, $CDCl_3$): δ (66.8, 66.6), (66.1, 66.1), 51.0, 47.4, 43.2, 38.1, 35.1, 34.9, 34.0, 33.8, 33.6, 33.2, 31.0, 30.7, 26.3, 26.3, 26.2, 26.0, 25.9, 20.0; IR (Neat film): 2928, 2854, 1447, 1257, 1231, 1030, 915, 737 cm^{-1}; HRMS (ES) calcd for $C_{10}H_{17}Cl$ $[M]^-$: 172.1019, found 172.1023.



13g

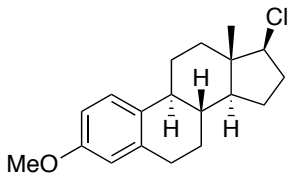
From hydrazone **11g** (220 mg, 0.42 mmol), chloride **13g** was purified by two successive columns (first using 4:1 hexanes:$CHCl_3$, then a second using 9:1 hexanes:acetone) and obtained as a single diastereomer as a colorless oil (60 mg, 0.21 mmol, 50% yield). The crude ^{1}H NMR indicated a dr of 2.8:1. R_f = 0.40 (2:1 hexanes:$CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.13 (dd, *J* = 11.0, 18.0 Hz, 1H), 5.20 (ddd, *J* = 0.5, 1.0, 11.0 Hz, 1H), 5.15 (dd, *J* = 1.0, 17.5 Hz, 1H), 3.82 (tt, *J* = 4.5, 10.5 Hz, 1H), 3.76 (dd, *J* = 4.0, 12.0 Hz, 1H), 2.11-1.98 (m, 2H), 1.95-1.86 (m, 1H), 1.85-1.73 (m, 1H), 1.44-1.32 (m, 2H), 1.17 (s, 3H), 0.87 (9H), 0.04 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.3, 114.5, (69.0, 69.0), 66.6, 46.9, 43.0, 35.6, 31.7, 28.7, 25.8, 18.1, −4.7, −4.7; IR (Neat film): 2953, 2931, 2858, 1462, 1371, 1252, 1106, 1005, 917, 865, 836, 794, 775 cm^{-1}; HRMS (ES) calcd for $C_{15}H_{29}ClOSi$ $[M]^+$: 288.1676, found 288.1662.



13i

From hydrazone **11i** (240.1 mg, 0.588 mmol), chloride **13i** was purified by flash chromatography (100% pentane) and obtained as a colorless oil with 2.6:1 dr, as measured by its ^{1}H NMR spectrum (71.3 mg, 0.413 mmol, 70% yield). R_f = 0.7 (100% pentane); ^{1}H NMR (500 MHz, $CDCl_3$, * denotes minor diastereomer): δ 3.92 (app dt, *J* = 7.0, 10.5, 1H), 3.90-3.84 (m, 1H*), 2.38 (ddd, *J* = 1.5, 8.0, 14.5 Hz, 1H*), 2.26 (dq, *J* = 7.0, 7.0 Hz, 1H), 2.17 (qd, *J* = 3.0, 7.0 Hz, 1H*), 2.08 (dd, *J* = 7.5, 12.5 Hz, 1H), 1.89 (dd, J = 1.0, 10.5 Hz, 1H), 1.79 (dd, *J* = 3.5, 14.5 Hz, 1H*), 1.30 (sept, *J* = 7.0 Hz, 1H), 1.28 (sept, *J* = 7.0 Hz, 1H*), 1.06 (d, *J* = 7.0 Hz, 3H*), 1.02 (d, *J* = 7.0 Hz, 3H), 0.97 (d, *J* = 7.0 Hz, 3H), 0.93 (d, *J* = 7.0 Hz, 3H*), 0.91 (d, *J* = 7.0 Hz, 3H), 0.90 (d, *J* = 7.0 Hz, 3H*), 0.89-0.83 (m, 1H*), 0.57 (ddd, *J* = 1.5, 5.0, 8.5 Hz, 1H*), 0.41 (dd, *J* = 4.0, 5.5 Hz, 1H), 0.29 (ddd, *J* = 1.5, 5.5, 8.5 Hz, 1H); ^{13}C NMR

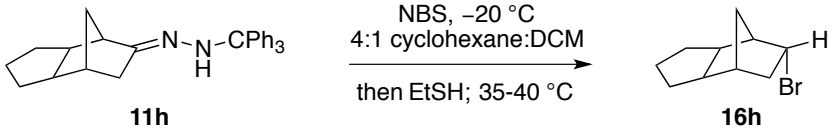
(125 MHz, $CDCl_3$): δ 65.6, 66.6, 60.0, 60.0, 47.0, 38.2, 37.9, 35.1, 33.0, 32.3, 29.7, 28.4, 20.6, 20.1, 20.1, 19.7, 19.6, 18.9, 17.0, 13.49; IR (Neat film): 2959, 2872, 1456, 1375, 888, 846, 789 cm^{-1}; HRMS (ES) calcd for $C_{10}H_{17}Cl$ $[M+Na]^+$: 195.0911, found 195.0911.



13j

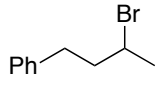
An oven-dried 25 mL 14/20 recovery flask equipped with a Teflon-coated magnetic stir bar was charged with trityl hydrzaone **11j** (111 mg, 0.21 mmol, 1.0 equiv) and THF (2.5 mL) and subsequently evacuated and backfilled with N_2 (3 times). After cooling to −40 °C (external temperature), *n*-butyllithium (1.6M in hexanes, 0.14 mL, 1.1 equiv.) was added dropwise over 1 minute, and the resulting deep red-orange solution was stirred for an additional 30 minutes. An ambient temperature solution of dichloramine-T (60.6 mg, 0.25 mmol, 1.2 equiv.) in 0.6 mL THF was added dropwise over 1 minute to the cooled lithiated hydrazone, and immediate color dissipation to a light yellow solution was observed. After stirring for an addition 10 minutes, the reaction was frozen in a liquid N_2 bath and degassed by two FPT cycles, each time thawing in a −15 °C bath. After backfilling with N_2, the reaction was maintained at external temperature ≤ −15 °C for 35 minutes. Excess EtSH (1.5 mL) at ambient temperature was added to the cooled reaction, and the reaction flask was subsequently transferred to a pre-heated 35-40°C oil bath. After 50 minutes, the reaction was allowed to cool to ambient temperature and subjected to workup as in the representative procedure for chlorination. The crude residue was purified by flash chromatography (3 successive columns – 9:1 hexanes:Et_2O, 4:1 hexanes:$CHCl_3$, 1:1 hexanes:$CHCl_3$) to afford chloride **13j** as a white solid with a slight tint of yellow (42 mg, 0.14 mmol, 67% yield). R_f = 0.45 (1:1 hexanes:$CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6): δ 7.12 (d, *J* = 8.5 Hz, 1H), 6.79 (dd, *J* = 3.0, 8.5 Hz, 1H), 6.70 (d, *J* = 3.0 Hz, 1H), 3.51 (app t, *J* = 9.0 Hz, 1H), 3.42 (s, 3H), 2.76-2.61 (m, 2H), 2.13-2.05 (m, 1H), 2.05-1.90 (m, 3H), 1.80-1.71 (m, 1H), 1.62-1.55 (m, 1H), 1.38-1.25 (m, 2H), 1.25-0.93 (m, 4H), 0.76 (s, 3H), 0.67 (ddd, *J* = 7.5, 11.0, 12.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.5, 137.8, 132.3, 126.3, 113.8, 111.5, 69.1, 55.2, 50.7, 44.5, 43.8, 39.4, 36.3, 32.0, 29.8, 27.4, 26.2, 23.7, 12.6; IR (Neat film): 2913, 1653, 1608, 1559, 1540, 1501, 1457, 1251, 1038, 859, 824 cm^{-1}; HRMS (ES) calcd for $C_{19}H_{25}ClO$ $[M]^+$: 304.1594, found 304.1605; $[\alpha]^{21.1}_D$ +82.0° (*c* 1, $CHCl_3$).

Reductive Bromination of Trityl Hydrazones



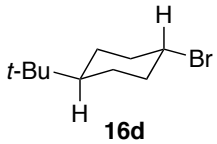
11h **16h**

Representative Procedure: An oven-dried 25 mL 14/20 recovery flask equipped with a Teflon-coated magnetic stir bar was charged with trityl hydrazone **11h** (233 mg, 0.57 mmol, 1.0 equiv), DCM (1 mL), and cyclohexane (4 mL). The resulting colorless solution was evacuated and backfilled with N_2 (3 times) and then cooled to −20 °C (external temperature). *N*-bromosuccinimide (0.4M in THF, 1.1 equiv.) at ambient temperature was added dropwise over 2 minutes to the cooled solution of hydrazone and stirred for 15 minutes. The resulting yellow reaction was then frozen in a liquid N_2 bath and degassed by two FPT cycles, each time thawing in a −20 °C bath. After backfilling with N_2, the reaction was maintained at an external temperature ≤ −15 °C for at least 20 minutes. During this time EtSH was degassed once by FPT. Excess EtSH (4 mL) at ambient temperature was added to the cooled reaction without appreciable change, and the reaction flask was subsequently transferred to a pre-heated 35-40 °C oil bath. After 1 hour, significant color dissipation was observed, and the reaction was allowed to cool to ambient temperature. In a 60 mL separatory funnel, the reaction was partitioned between 20 mL 1:1 H_2O:brine and 20 mL 1:1 Et_2O:pentane. The aqueous layer was separated and extracted once with 20 mL 1:1 Et_2O:pentane. The combined organic layers were dried over Na_2SO_4 and carefully concentrated under reduced pressure (ca. 140 mmHg). The crude residue was purified by flash chromatography (100% pentane) to afford bromide **16h** as a colorless oil (74 mg, 0.34 mmol, 60% yield). R_f = 0.62 (100% pentane); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.28 (app dt, *J* = 4.0, 10.5 Hz, 1H), 2.61-2.51 (m, 1H), 2.27 (ddd, *J* = 4.5, 10.5, 13.5, 1H), 2.22-2.18 (m, 1H), 1.97-1.84 (m, 4H), 1.70-1.62 (m, 1H), 1.46 (app d(dt), *J* = 2.0, 3.5, 10.5, 1H), 1.32 (ddd, *J* = 3.5, 4.0, 13.5, 1H), 1.29-1.17 (m, 1H), 1.12-1.06 (m, 1H), 1.01-0.87 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 53.5, 47.9, 47.8, 42.0, 41.1, 41.1, 32.3, 32.0, 32.0, 27.0; IR (Neat film): 2949, 2863, 1474, 1449, 1317, 1240, 1209, 952, 879, 759, 745, 634 cm^{-1}; HRMS (ES) calcd for $C_{10}H_{15}Br$ $[M+^{81}Br]^-$: 294.9526, found 294.9513.



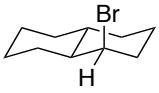
16a

From hydrazone **11a** (147.1 mg, 0.3636 mmol), bromide **16a** was obtained as a colorless oil (50.1 mg, 0.2351 mmol, 65% yield) following purification by flash chromatography (100% pentane). The spectral data obtained are in accord with that in the literature.[6]



16d

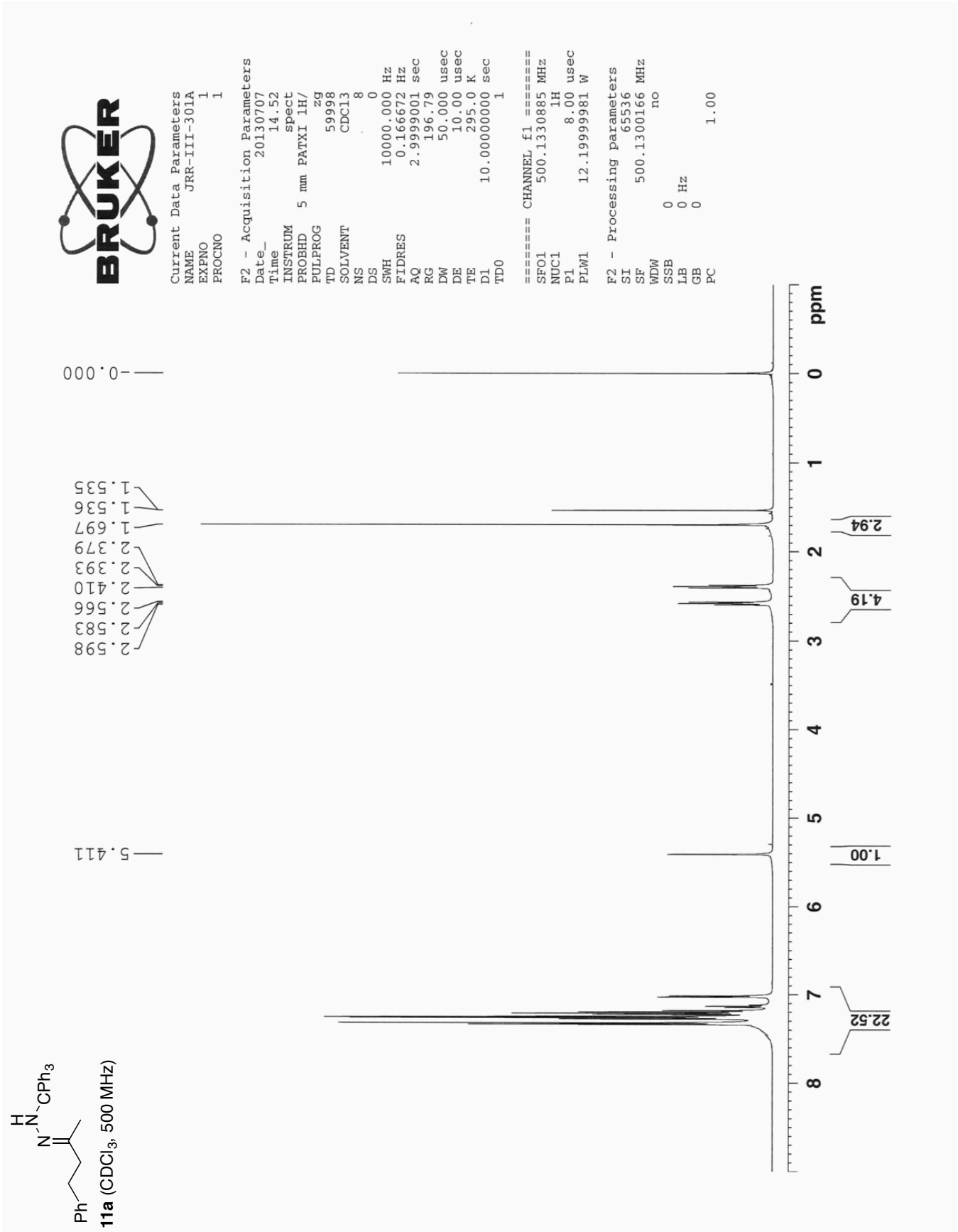
From hydrazone **11d** (246.8 mg, 0.6011 mmol), bromide **16d** was obtained as a colorless oil (91.5 mg, 0.4175 mmol, 69% yield) following purification by flash chromatography (100% pentane). The spectral data obtained are in accord with that in the literature.[5]



16e

From hydrazone **11e** (204.6 mg, 0.5008 mmol), bromide **16e** was obtained as a colorless oil (52.8 mg, 0.2432 mmol, 49% yield) following purification by flash chromatography (100% pentane). ^{1}H NMR (500 MHz, $CDCl_3$, * denotes minor diastereomer): δ 4.45-4.41 (m, 1H), 3.79 (ddd, J = 4.5, 10.5, 12.0 Hz, 1H*), 2.40-0.80 (m, 16H + 16H*); ^{13}C NMR (125 MHz, $CDCl_3$): δ 63.0, 61.5, 51.4, 47.6, 44.1, 39.4, 35.7, 35.6, 34.0, 33.9, 33.6, 33.4, 32.7, 32.5, 27.3, 26.5, 26.4, 26.2, 26.0, 20.8; IR (Neat film): 2925, 2853, 1447 cm^{-1}; HRMS (ES) calcd for $C_{10}H_{17}Br$ $[M+H]^+$: 217.0586, found 217.0586.

[6] Dai *et al. Nat. Chem.* **2011**, *3*, 140.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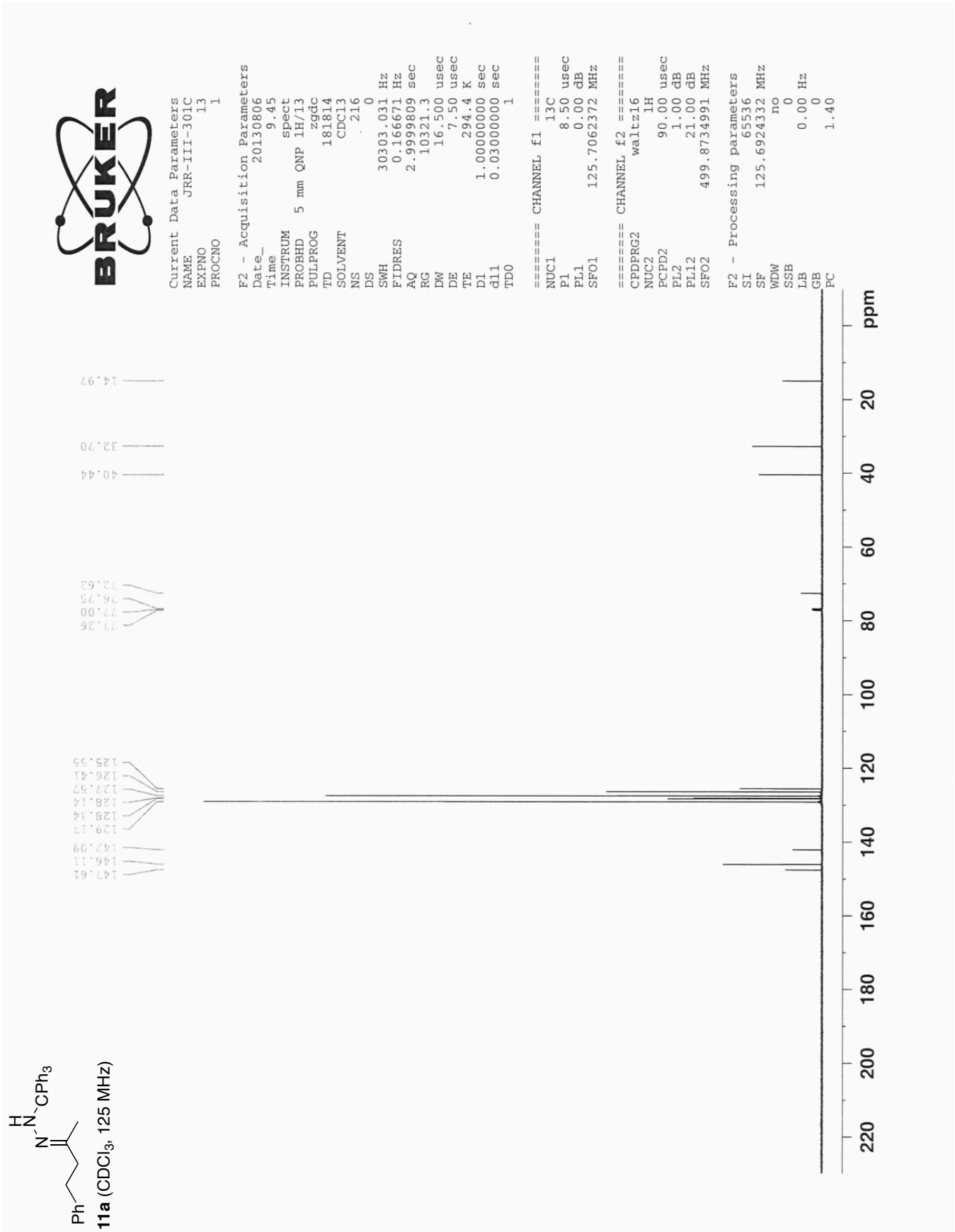
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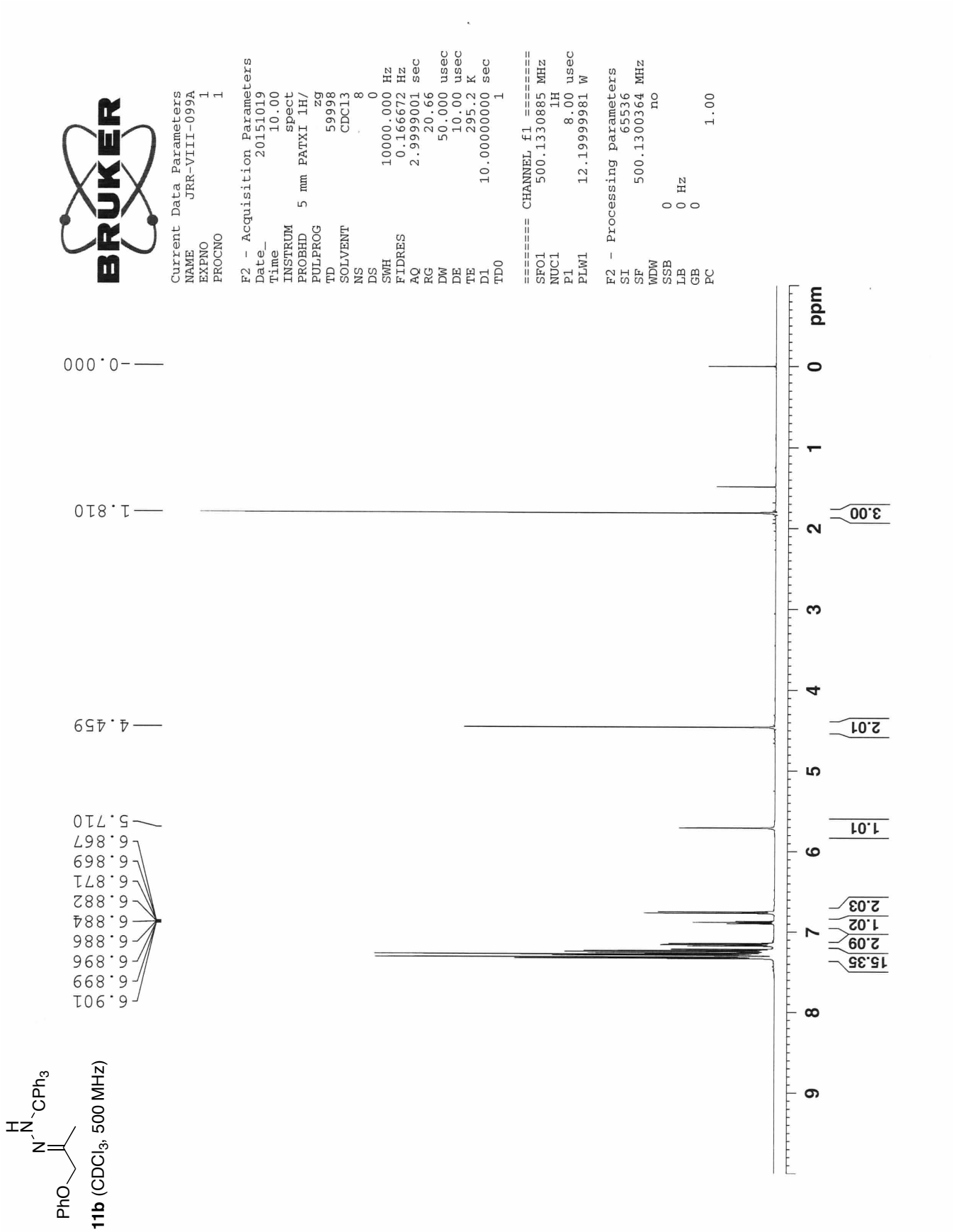
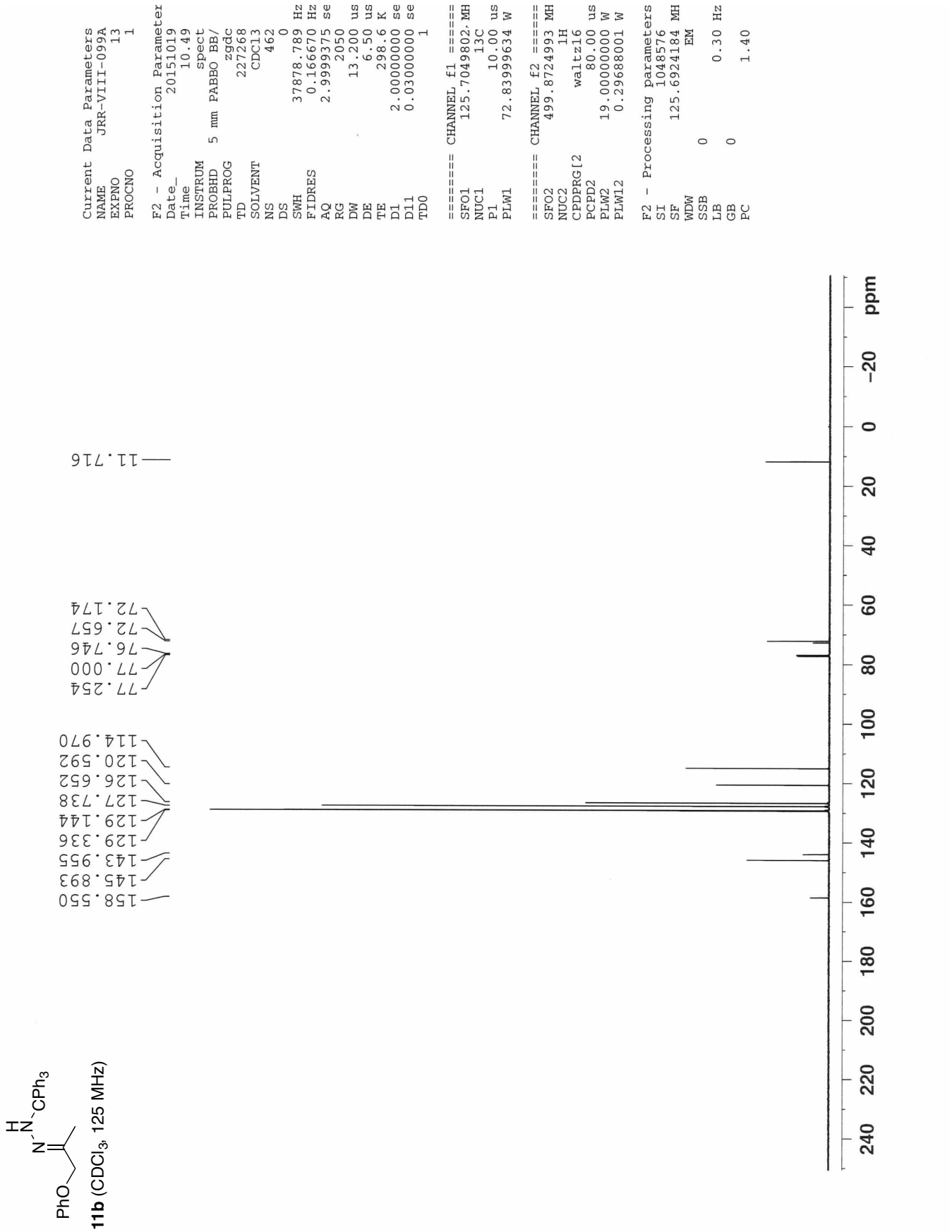
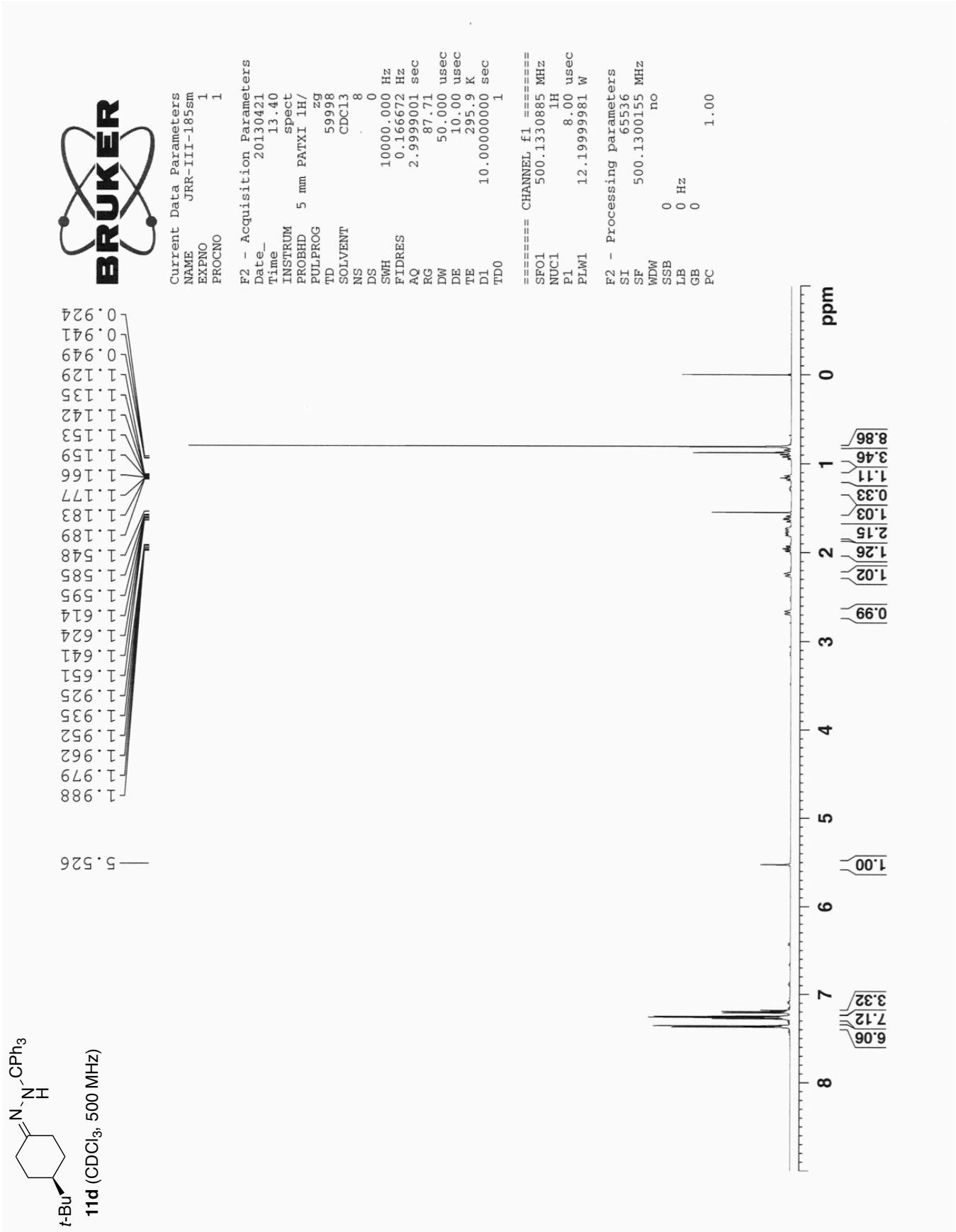
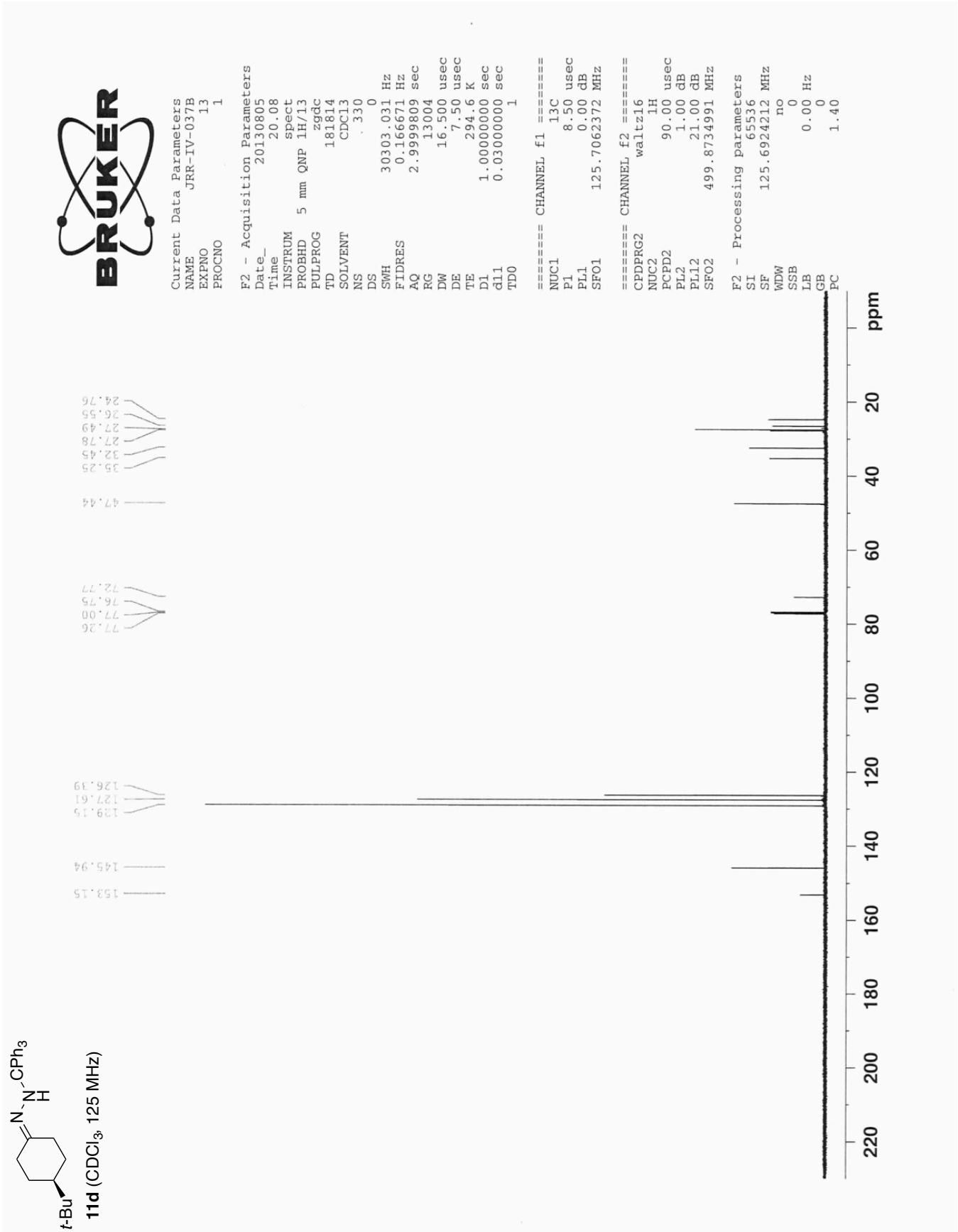
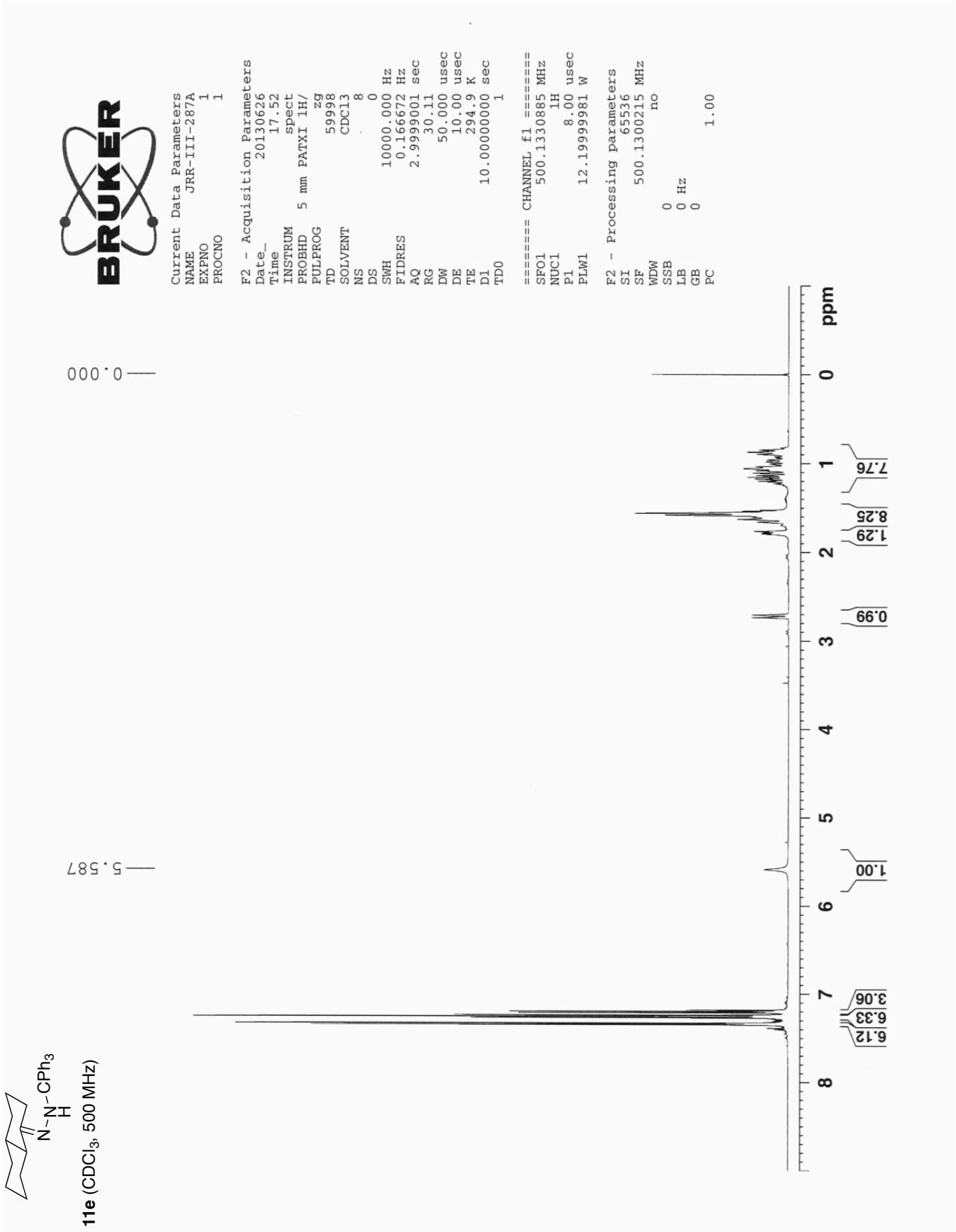
77.26
125.55
126.41
127.57
128.14
128.34
129.17
142.09
146.11
147.61
220
200
180
160
140
120
100
80
60
40
20
ppm

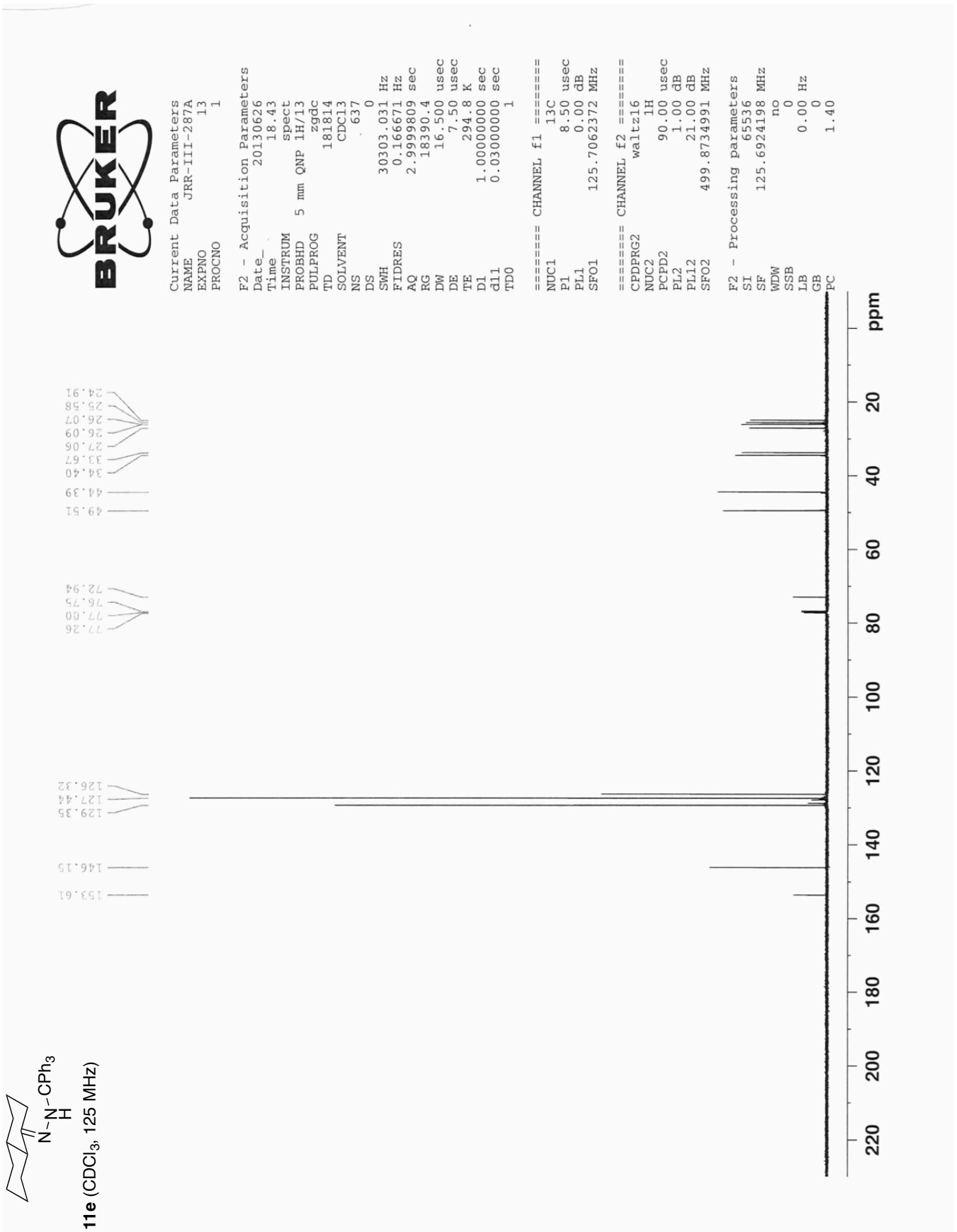

11b (CDCl3, 500 MHz)
PhO
N
H
N
CPh3
6.901
6.899
6.896
6.886
6.884
6.882
6.871
6.869
6.867
5.710
4.459
1.810
0.000
ppm
3.00
2.01
1.01
2.03
1.02
2.09
15.35

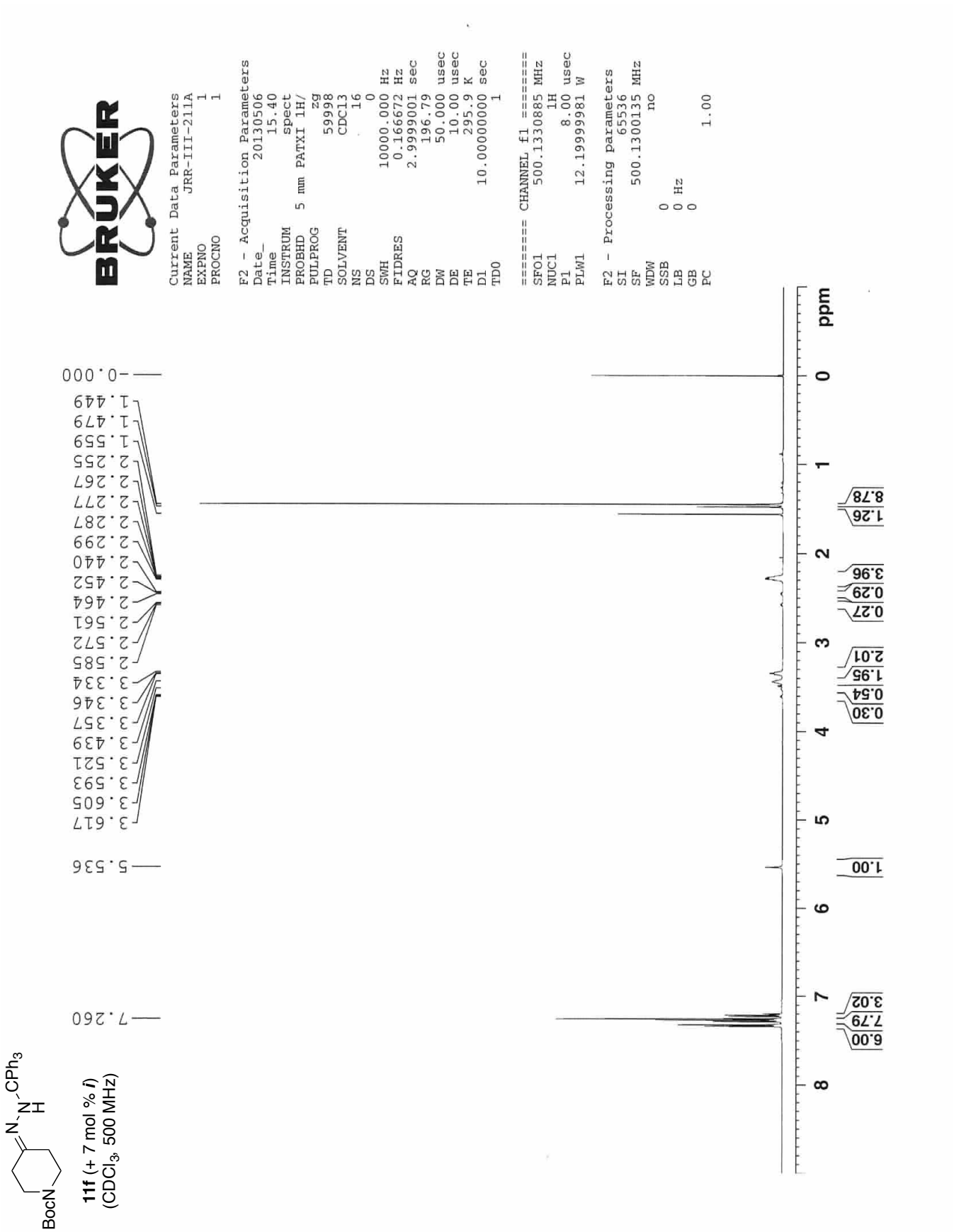

PhO
H
N
N
CPh3
11b (CDCl3, 125 MHz)
158.550
145.893
143.955
129.336
129.144
127.738
126.652
120.592
114.970
77.254
77.000
76.746
72.657
72.174
11.716
240 220 200 180 160 140 120 100 80 60 40 20 0 −20 ppm
Current Data Parameters
NAME JRR-VIII-099A
EXPNO 13
PROCNO 1
F2 - Acquisition Parameter
Date_ 20151019
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgdc
TD 227268
SOLVENT CDCl3
NS 462
DS 0
SWH 37878.789 Hz
FIDRES 0.166670 Hz
AQ 2.9999375 se
RG 2050
DW 13.200 us
DE 6.50 us
TE 298.6 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 125.7049802 MH
NUC1 13C
P1 10.00 us
PLW1 72.83999634 W
======== CHANNEL f2 ======
SFO2 499.8724993 MH
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 us
PLW2 19.00000000 W
PLW12 0.29688001 W
F2 - Processing parameters
SI 1048576
SF 125.6924184 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

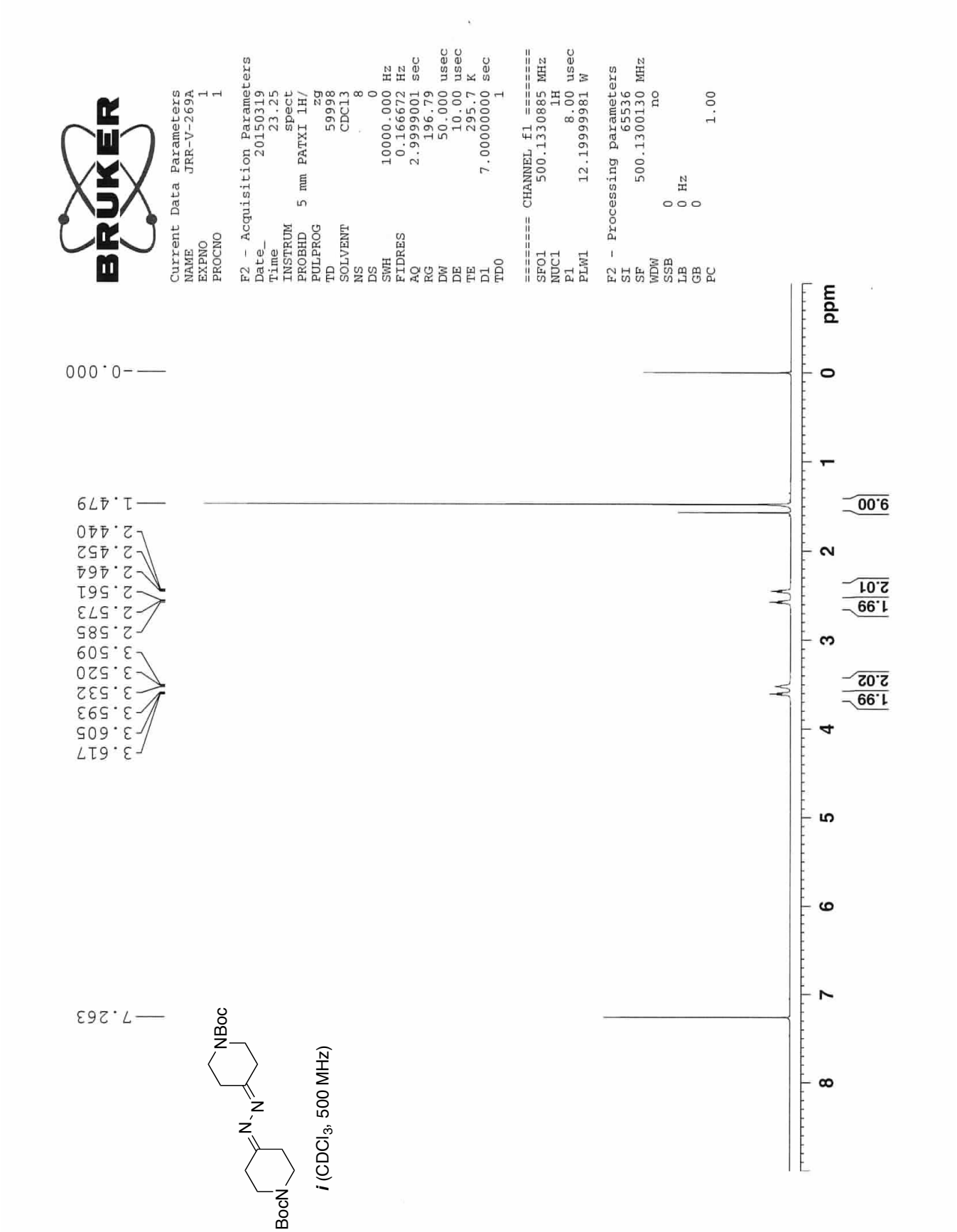

11d (CDCl3, 500 MHz)
t-Bu
CPh3
Current Data Parameters
NAME JRR-III-185sm
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130421
Time 13.40
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.99999001 sec
RG 87.71
DW 50.000 usec
DE 10.00 usec
TE 295.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300155 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
8.86
3.46
1.11
0.33
1.03
2.15
1.26
1.02
0.99
1.00
6.06
7.12
3.32
5.526
1.988
1.979
1.962
1.952
1.935
1.925
1.651
1.641
1.624
1.614
1.595
1.585
1.548
1.189
1.183
1.177
1.166
1.159
1.153
1.142
1.135
1.129
0.949
0.941
0.924

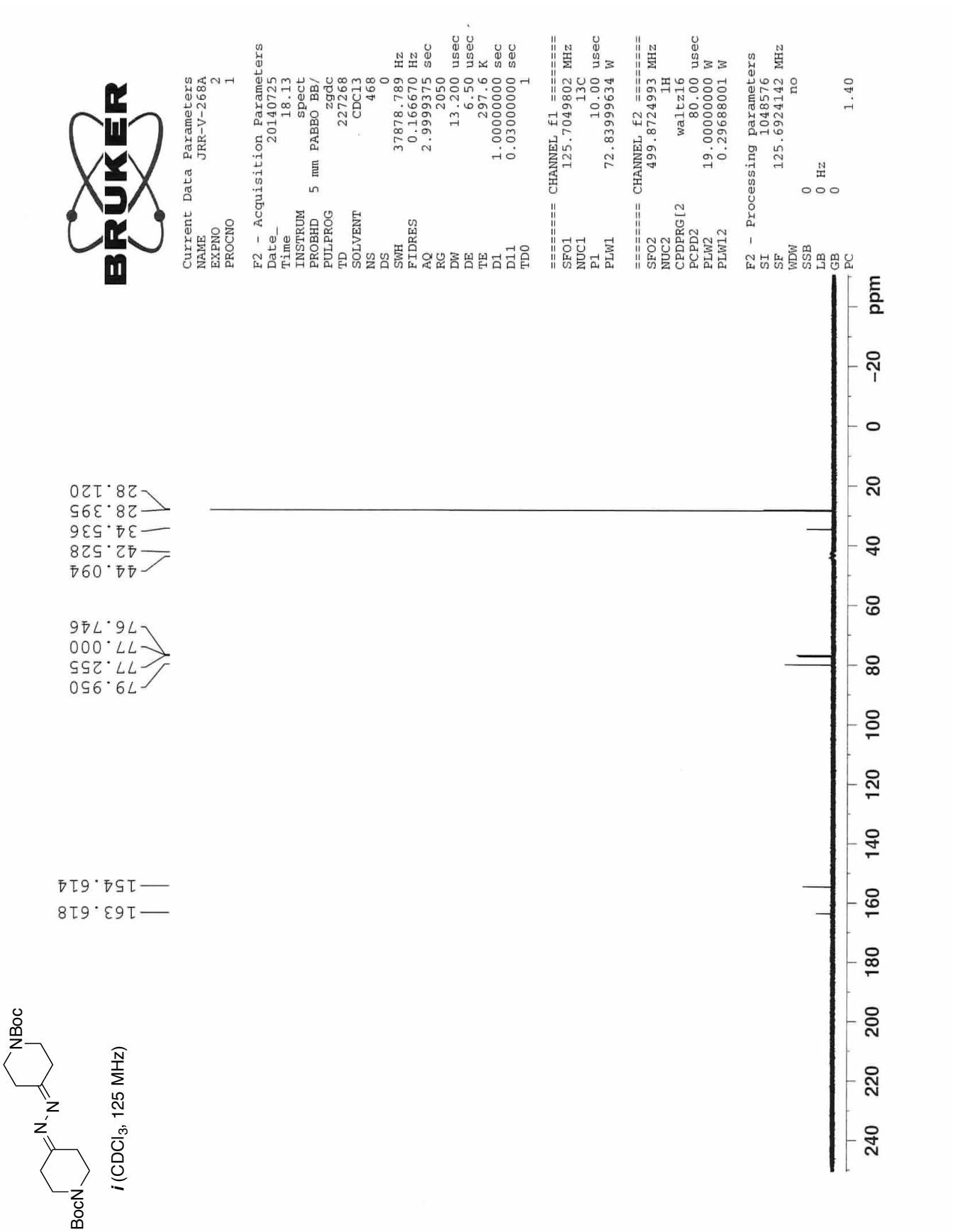

t-Bu
N
N
H
CPh3
11d (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-037B
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130805
Time 20.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 330
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 13004
DW 16.500 usec
DE 7.50 usec
TE 294.6 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924212 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
153.15
145.94
129.15
127.61
126.19
77.26
77.00
76.75
72.77
47.44
35.25
32.45
27.78
27.49
26.55
24.76
220
200
180
160
140
120
100
80
60
40
20
ppm

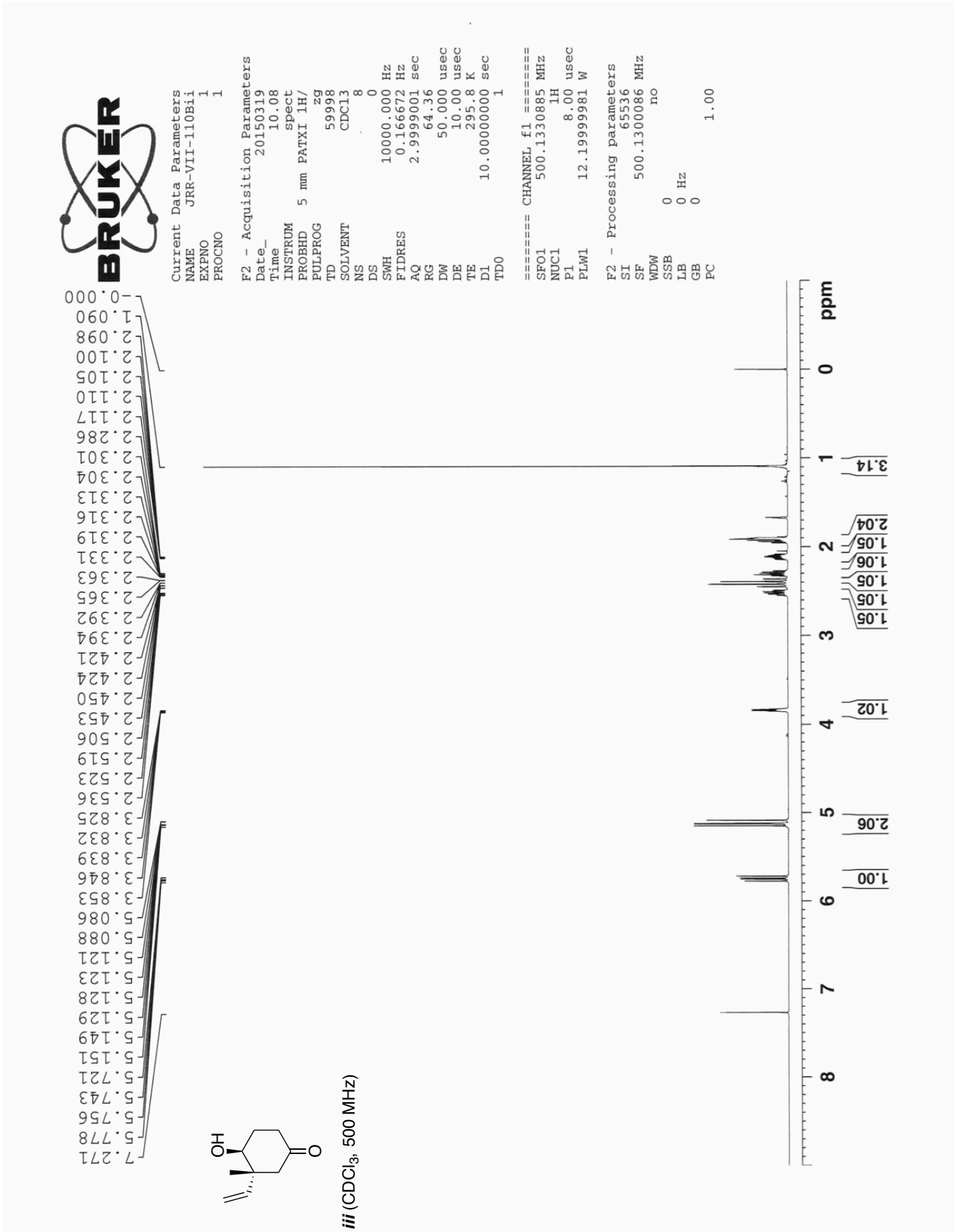

11e (CDCl3, 500 MHz)
N–N–CPh3
H
BRUKER
Current Data Parameters
NAME JRR-III-287A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130626
Time 17.52
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 30.11
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300215 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
0.000
5.587
ppm
0
1
2
3
4
5
6
7
8
7.76
1.29
8.25
0.99
1.00
6.12
6.33
3.06

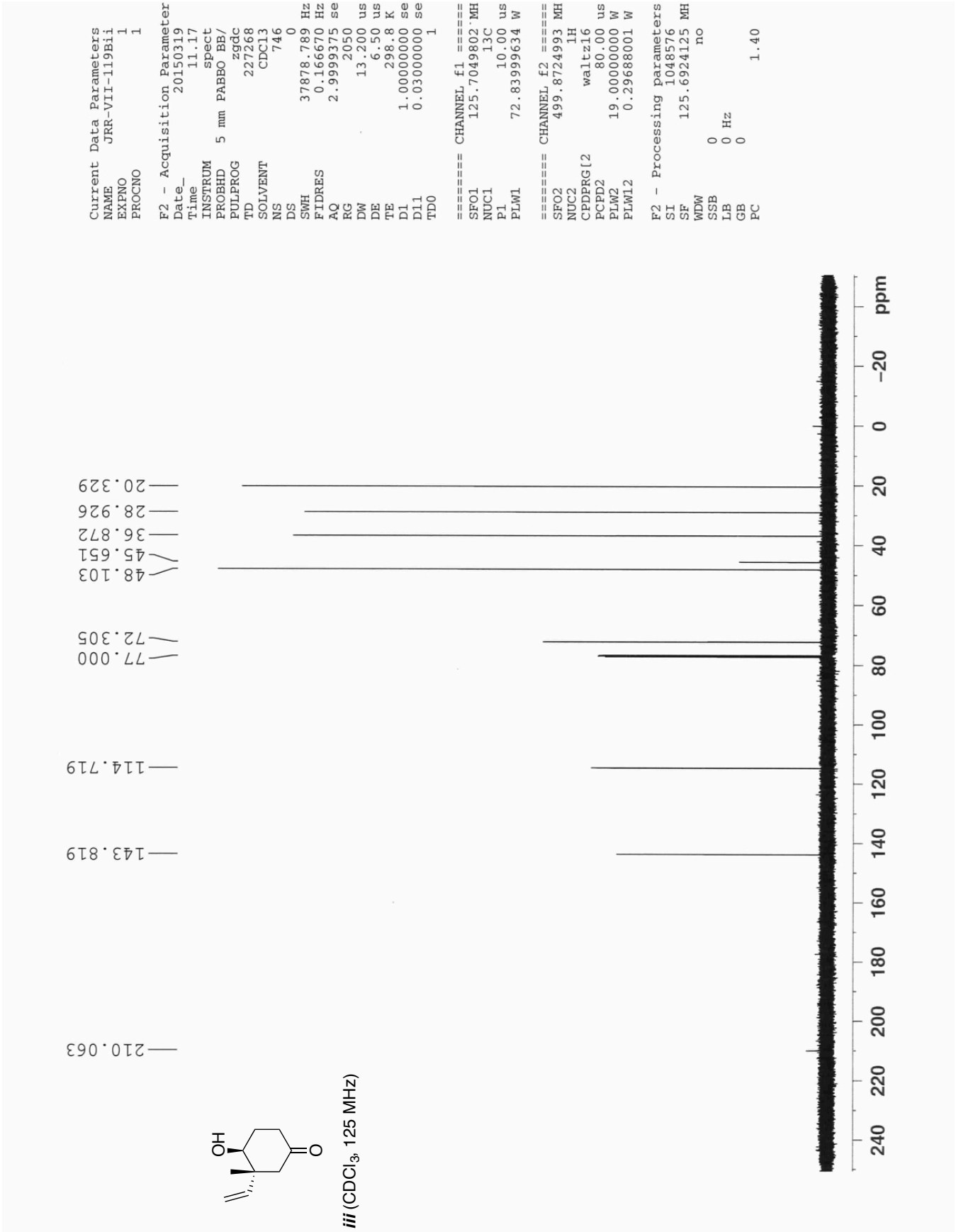

N–N–CPh3
H
11e (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-III-287A
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130626
Time 18.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 637
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 18390.4
DW 16.500 usec
DE 7.50 usec
TE 294.8 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924198 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
153.61
146.15
129.35
127.44
126.32
77.26
77.00
76.75
72.94
49.51
44.39
34.40
33.67
27.06
26.09
26.07
25.58
24.91
220
200
180
160
140
120
100
80
60
40
20
ppm


BocN
N
N
H
CPh3
11f (+ 7 mol % i)
(CDCl3, 500 MHz)
7.260
5.536
3.617
3.605
3.593
3.521
3.439
3.357
3.346
3.334
2.585
2.572
2.561
2.464
2.452
2.440
2.299
2.287
2.277
2.267
2.255
1.559
1.479
1.449
0.000
BRUKER
Current Data Parameters
NAME JRR-III-211A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130506
Time 15.40
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 196.79
DW 50.000 usec
DE 10.00 usec
TE 295.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300135 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
8.78
1.26
3.96
0.29
0.27
2.01
1.95
0.54
0.30
1.00
3.02
7.79
6.00
ppm

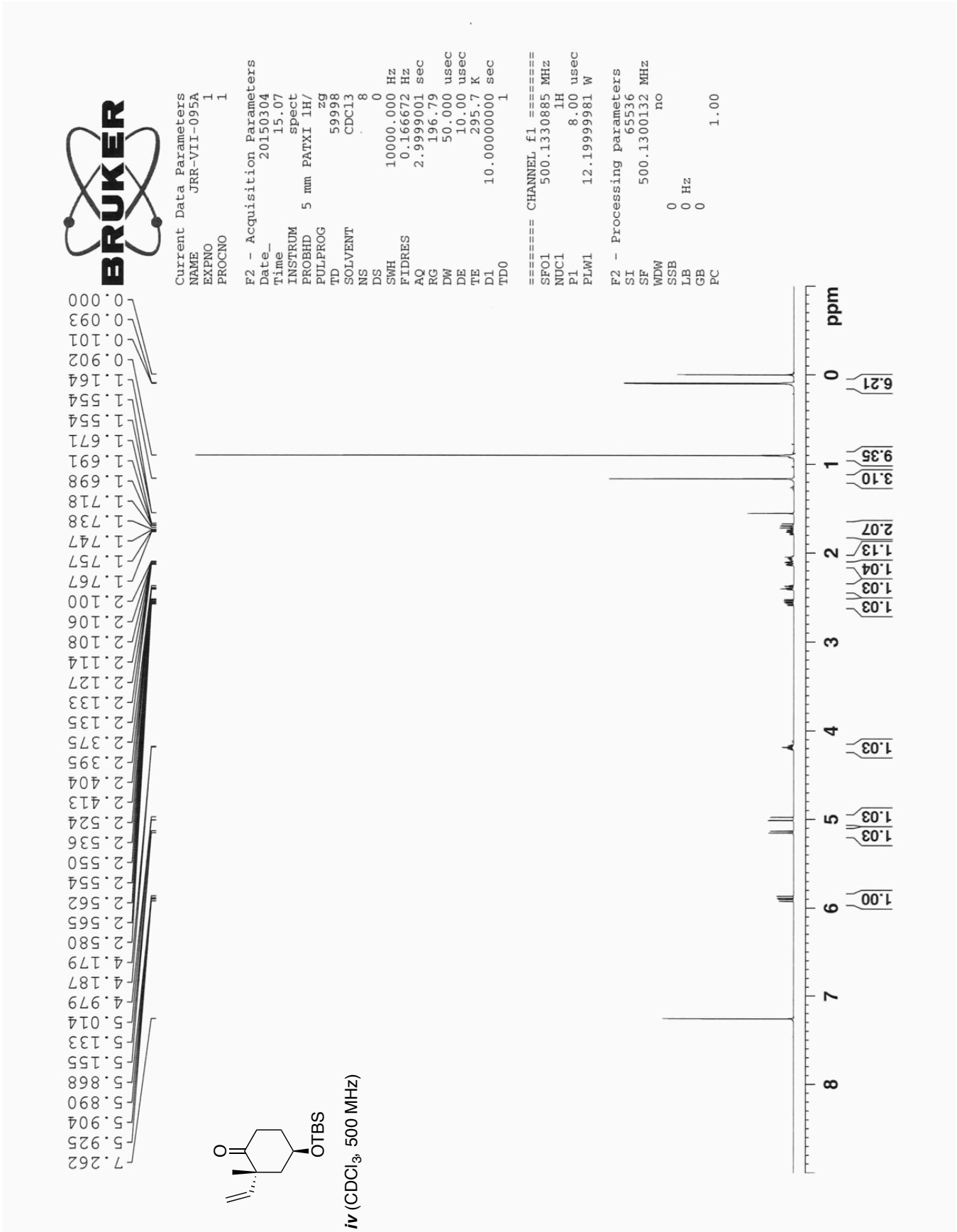

BRUKER
Current Data Parameters
NAME JRR-V-269A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150319
Time 23.25
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.99990001 sec
RG 196.79
DW 50.000 usec
DE 10.00 usec
TE 295.7 K
D1 7.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300130 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.263
3.617
3.605
3.593
3.532
3.520
3.509
2.585
2.573
2.561
2.464
2.452
2.440
1.479
0.000
9.00
2.01
1.99
2.02
1.99
ppm
NBoc
BocN
i ($CDCl_3$, 500 MHz)

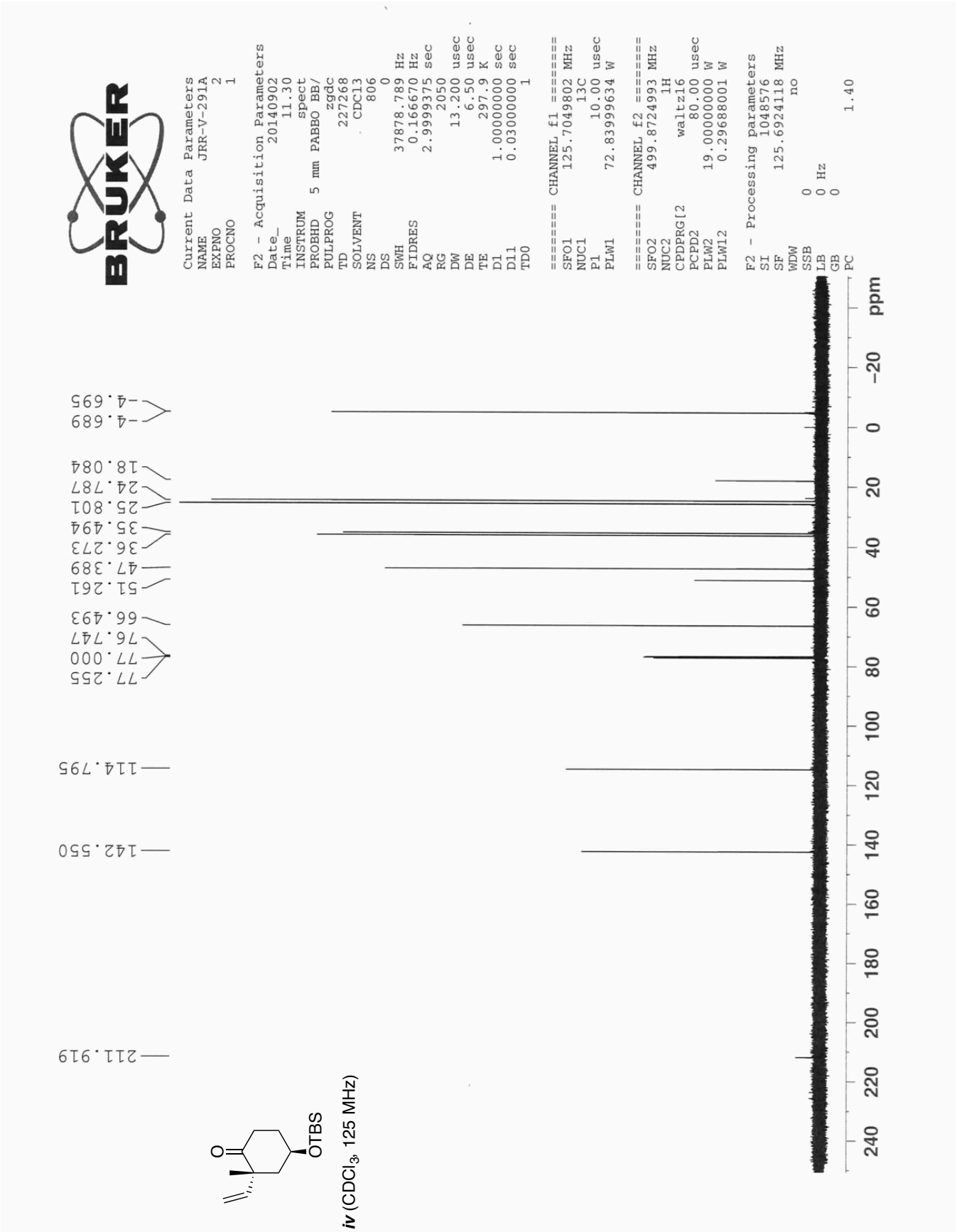

NBoc
N
N
BocN
i ($CDCl_3$, 125 MHz)
163.618
154.614
79.950
77.255
77.000
76.746
44.094
42.528
34.536
28.395
28.120
ppm


BRUKER
Current Data Parameters
NAME JRR-VII-110Bii
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150319
Time_ 10.08
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 64.36
DW 50.000 usec
DE 10.00 usec
TE 295.8 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300086 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
3.14
2.04
1.05
1.06
1.05
1.05
1.05
1.02
2.06
1.00
OH
O
iii (CDCl3, 500 MHz)

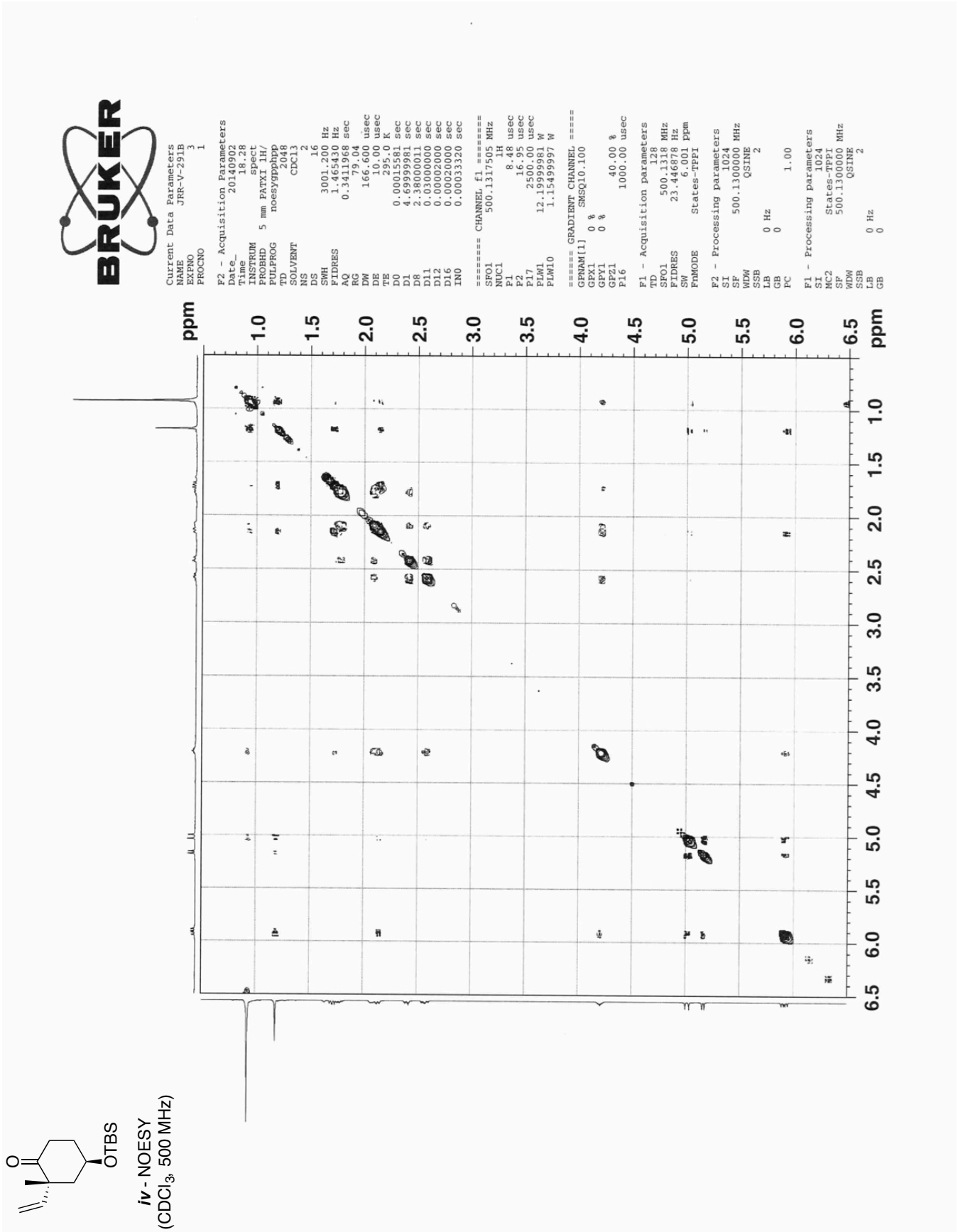


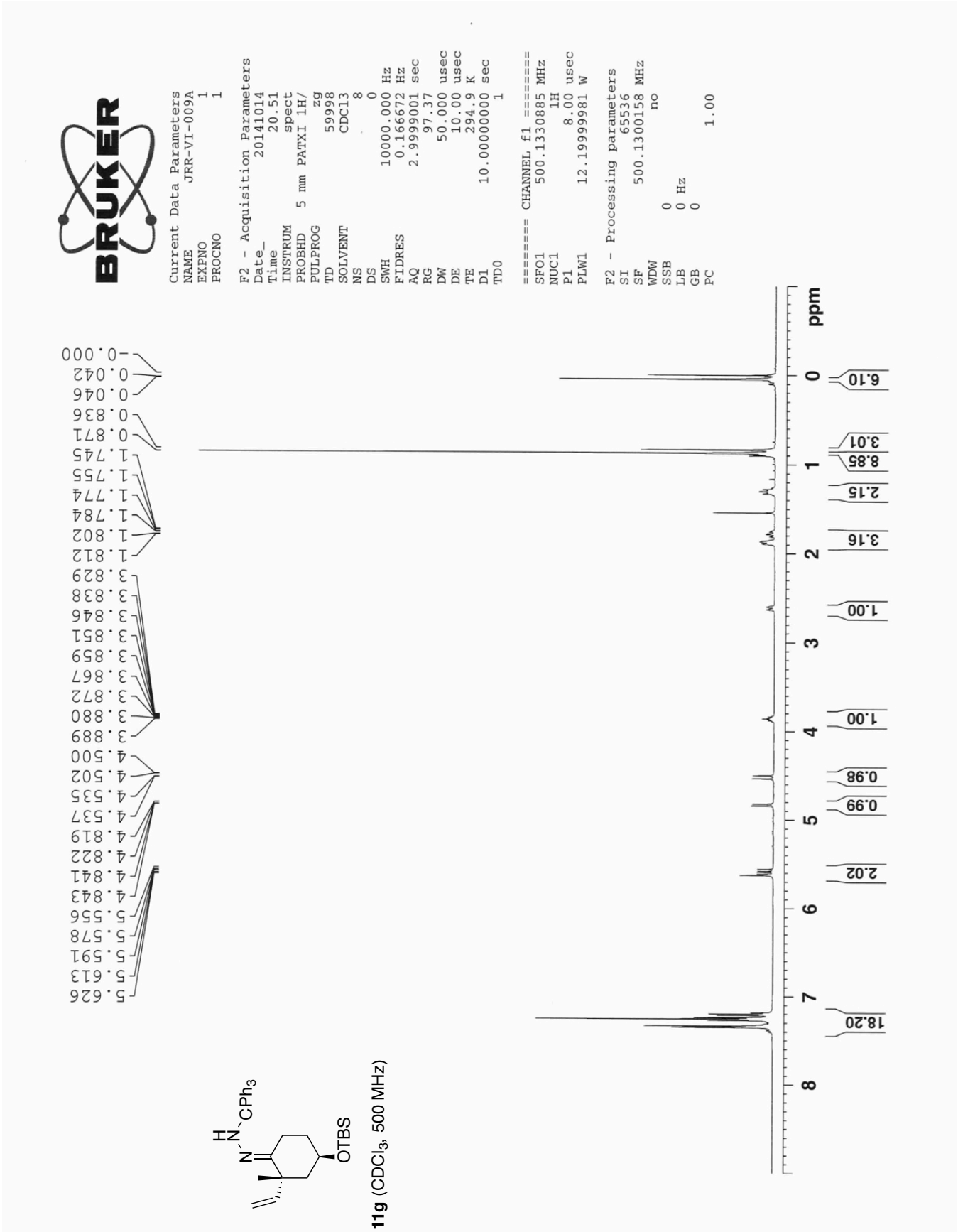
iii ($CDCl_3$, 125 MHz)


BRUKER
Current Data Parameters
NAME JRR-VII-095A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150304
Time 15.07
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 196.79
DW 50.000 usec
DE 10.00 usec
TE 295.7 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300132 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
6.21
9.35
3.10
2.07
1.13
1.04
1.03
1.03
1.03
1.03
1.03
1.00
OTBS
O
iv (CDCl$_3$, 500 MHz)

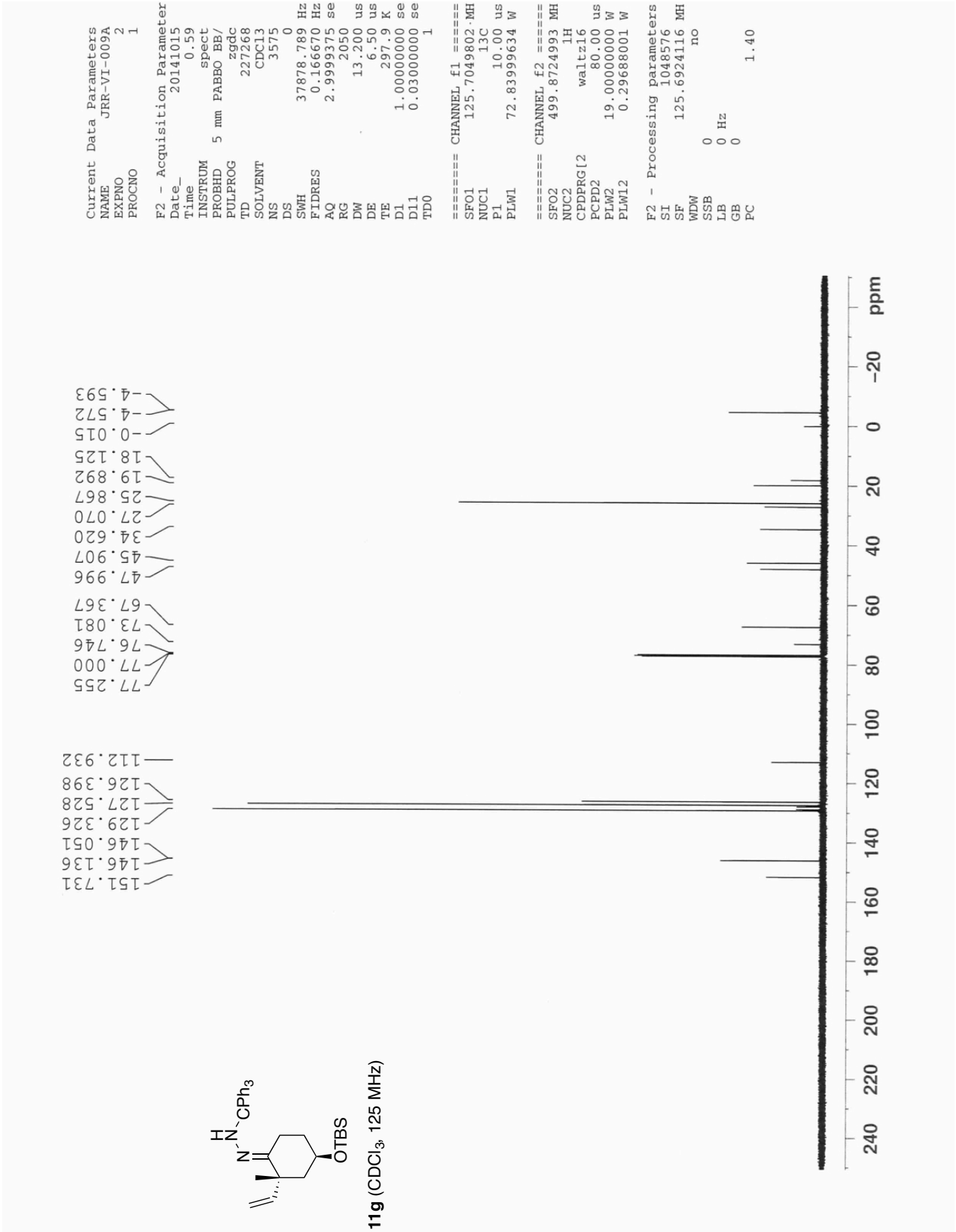


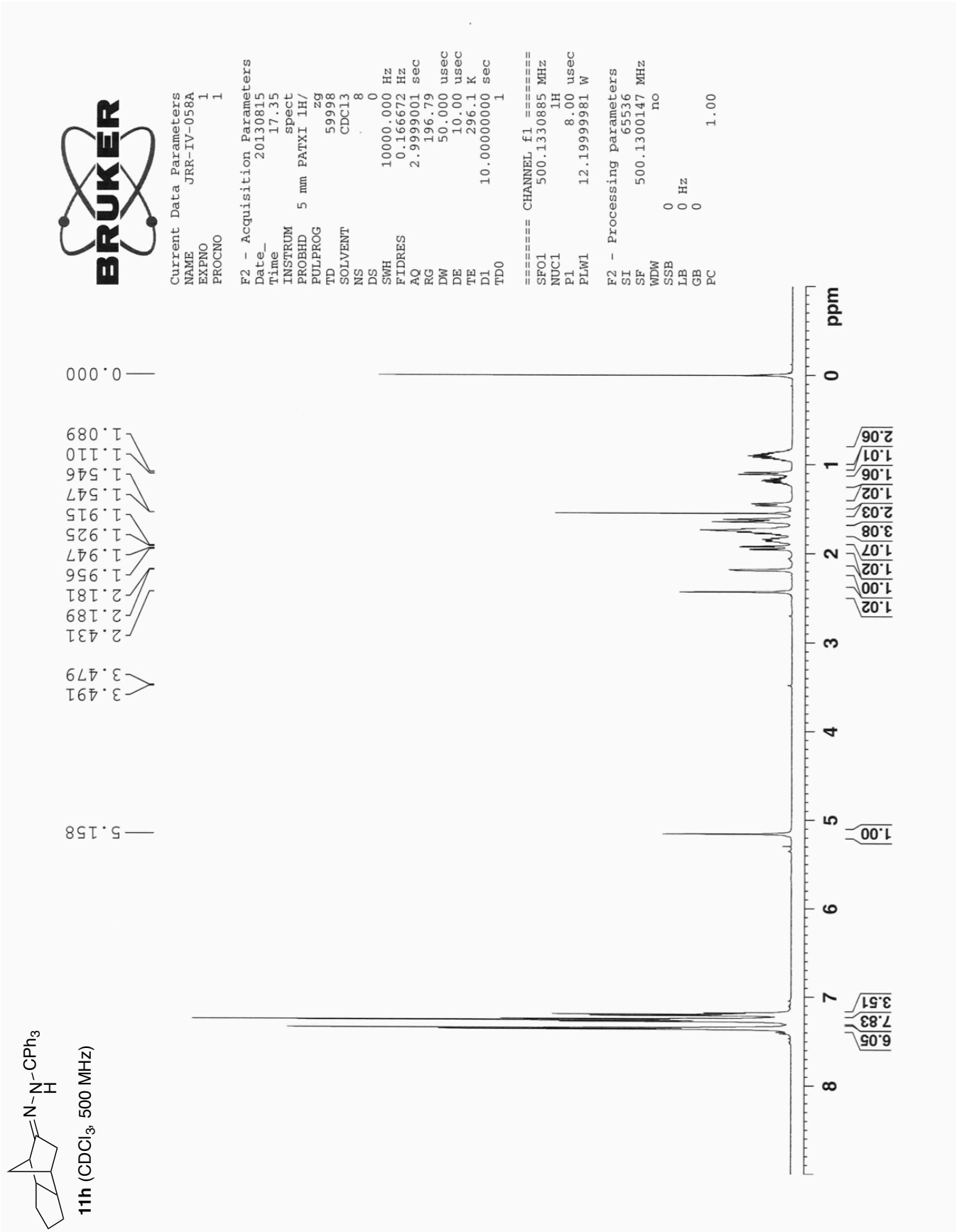
iv ($CDCl_3$, 125 MHz)

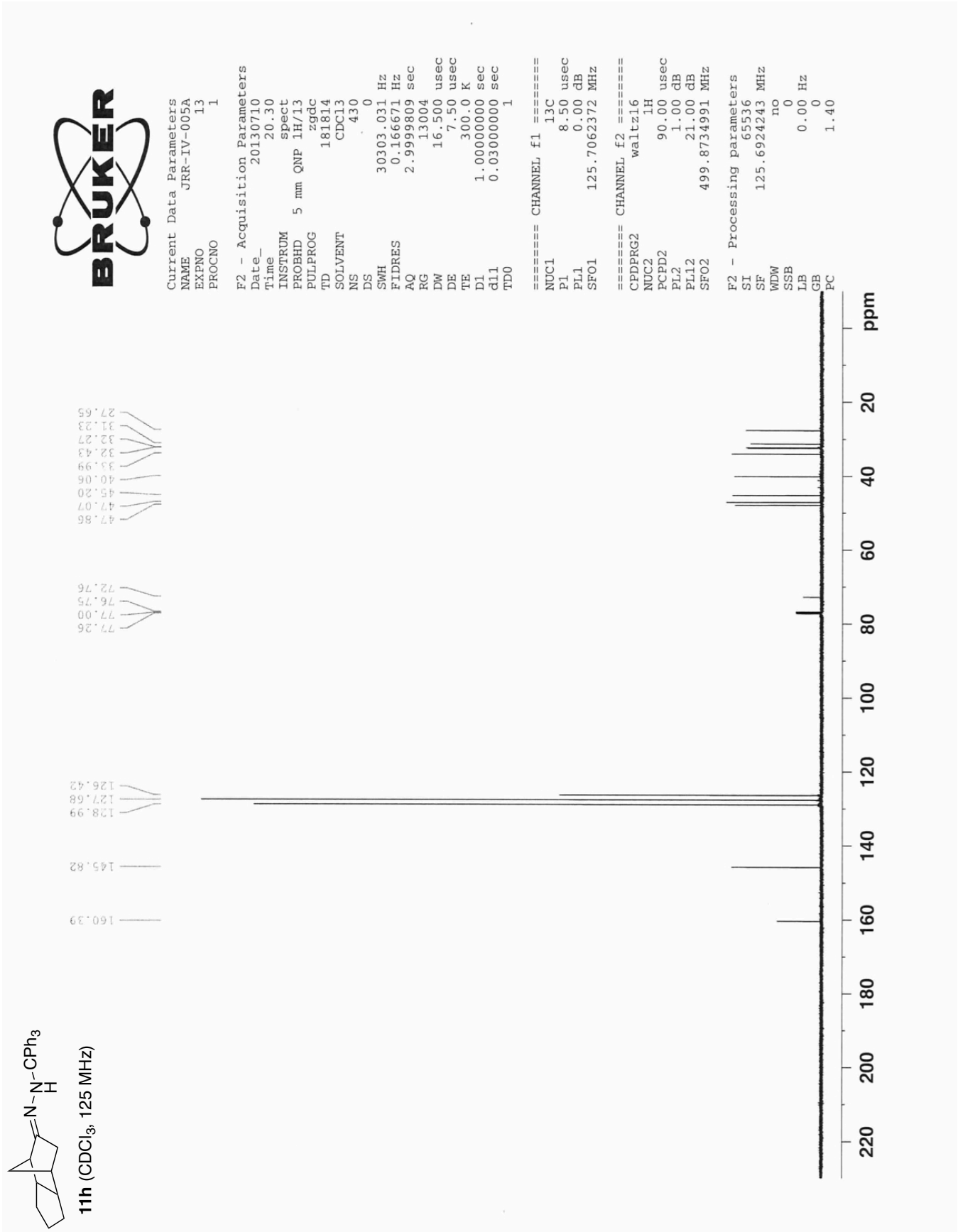

OTBS
O
iv - NOESY
($CDCl_3$, 500 MHz)
BRUKER
ppm
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

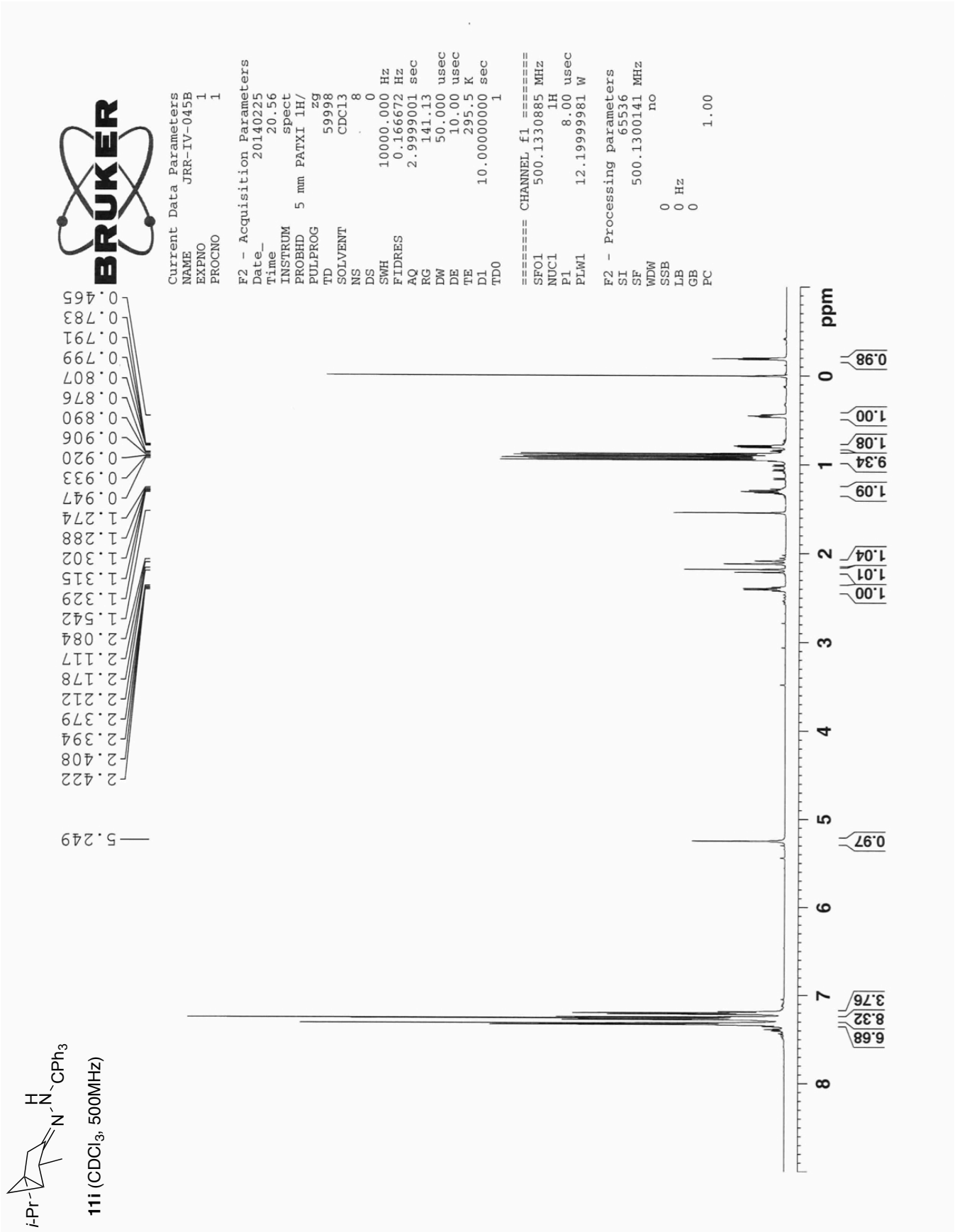


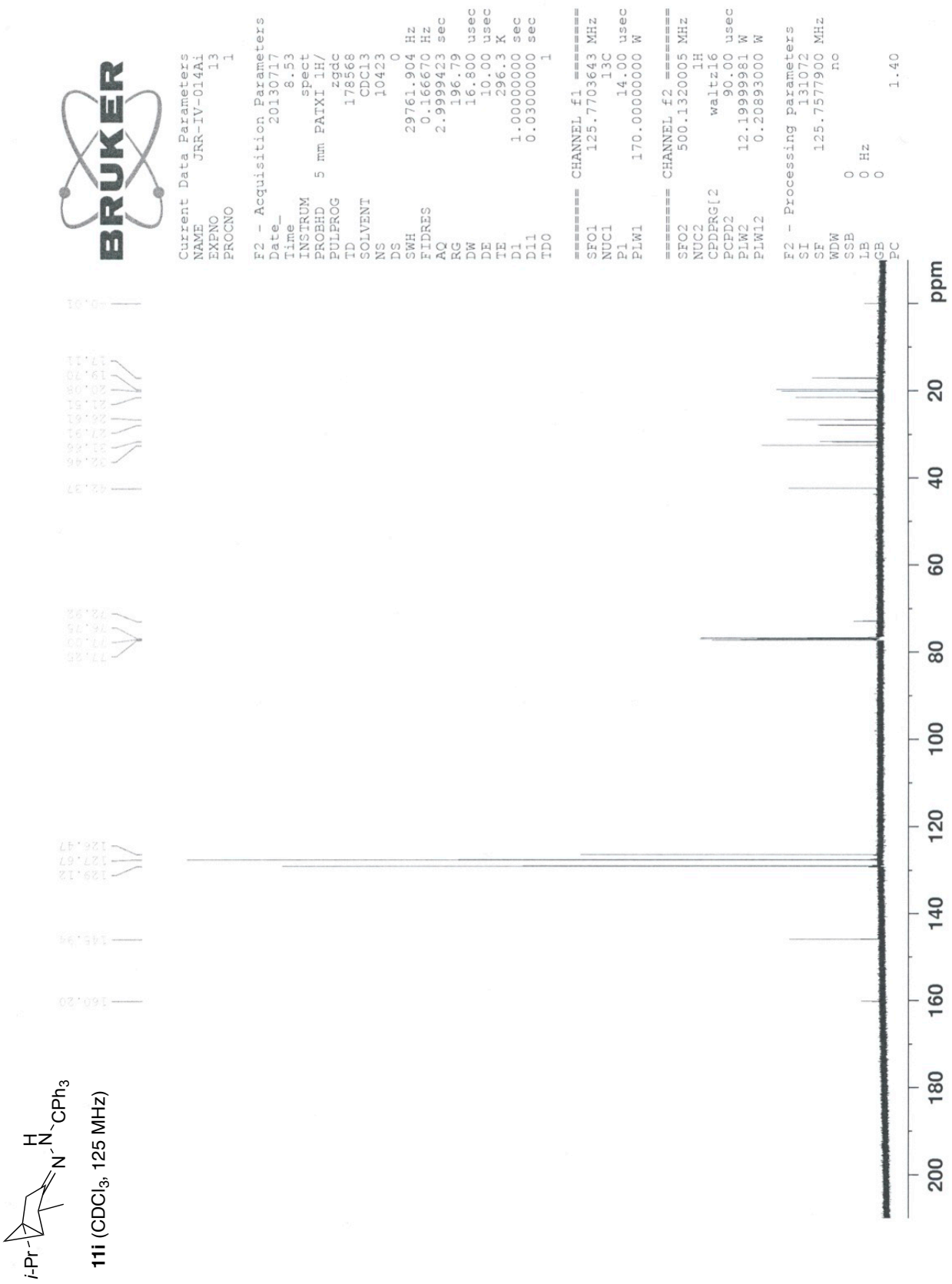
11g ($CDCl_3$, 500 MHz)

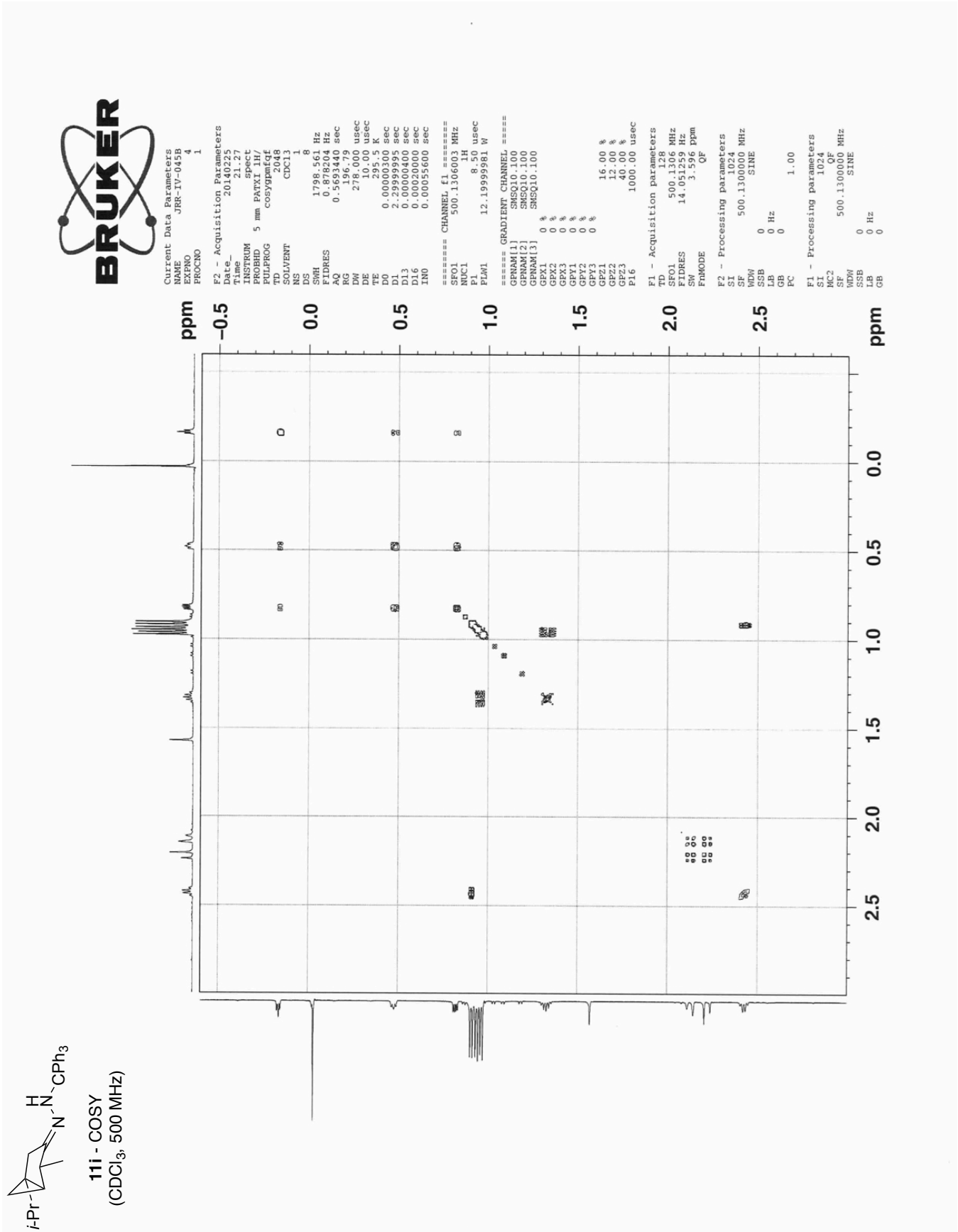

Current Data Parameters
NAME JRR-VI-009A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameter
Date_ 20141015
Time 0.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgdc
TD 227268
SOLVENT CDCl3
NS 3575
DS 0
SWH 37878.789 Hz
FIDRES 0.166670 Hz
AQ 2.99999375 se
RG 2050
DW 13.200 us
DE 6.50 us
TE 297.9 K
D1 1.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 125.7049802 MH
NUC1 13C
P1 10.00 us
PLW1 72.83999634 W
======== CHANNEL f2 ======
SFO2 499.8724993 MH
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 us
PLW2 19.00000000 W
PLW12 0.29688001 W
F2 - Processing parameters
SI 1048576
SF 125.6924116 MH
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
151.731
146.136
146.051
129.326
127.528
126.398
112.932
77.255
77.000
76.746
73.081
67.367
47.996
45.907
34.620
27.070
25.867
19.892
18.125
-0.015
-4.572
-4.593
240 220 200 180 160 140 120 100 80 60 40 20 0 −20 ppm
OTBS
CPh3
11g (CDCl3, 125 MHz)

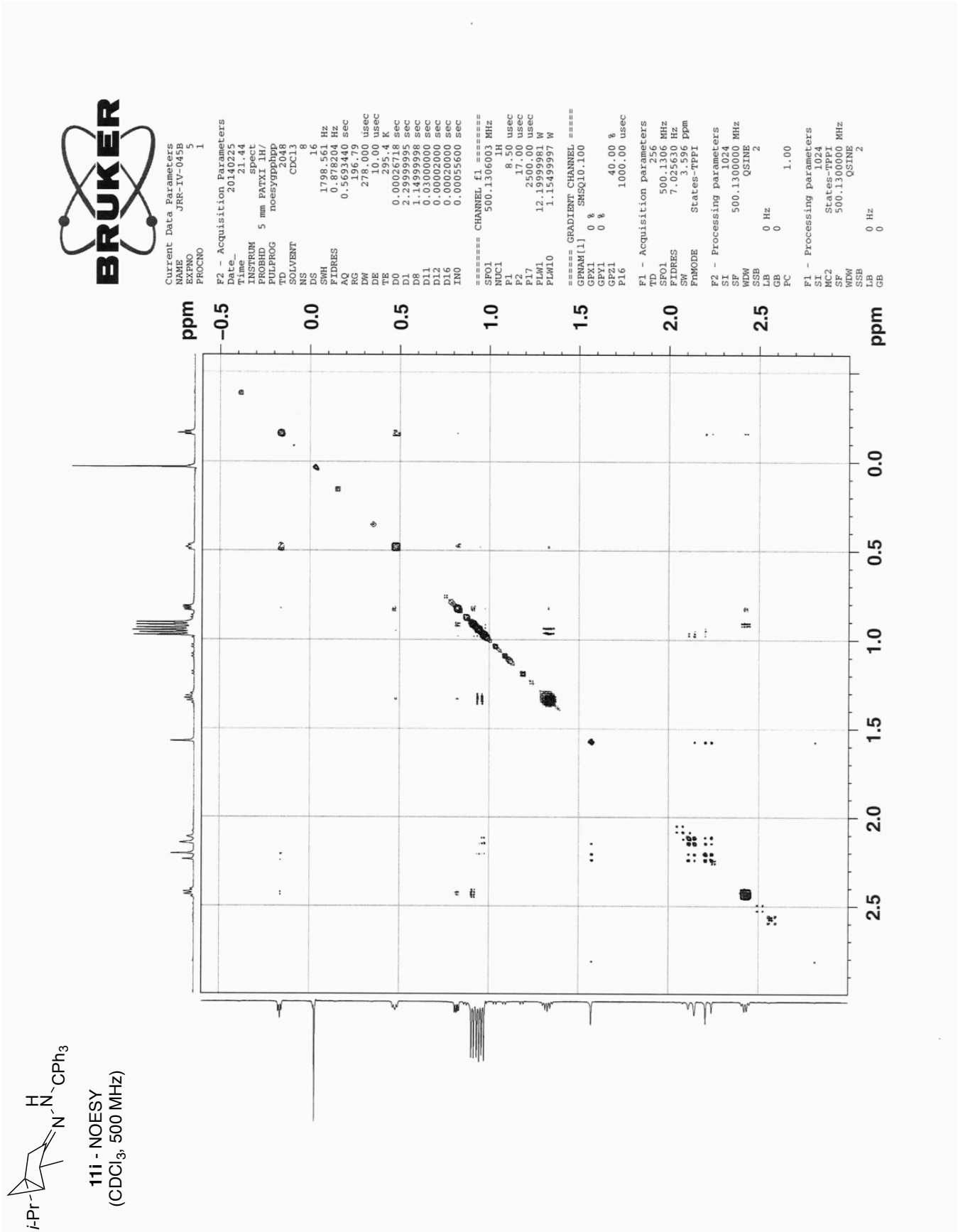

11h (CDCl3, 500 MHz)
N–N–CPh3
H
BRUKER
Current Data Parameters
NAME JRR-IV-058A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130815
Time 17.35
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 196.79
DW 50.000 usec
DE 10.00 usec
TE 296.1 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300147 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
0.000
1.089
1.110
1.546
1.547
1.915
1.925
1.947
1.956
2.181
2.189
2.431
3.479
3.491
5.158
ppm
1.02
1.00
1.02
1.07
3.08
2.03
1.02
1.06
1.01
2.06
1.00
6.05
7.83
3.51

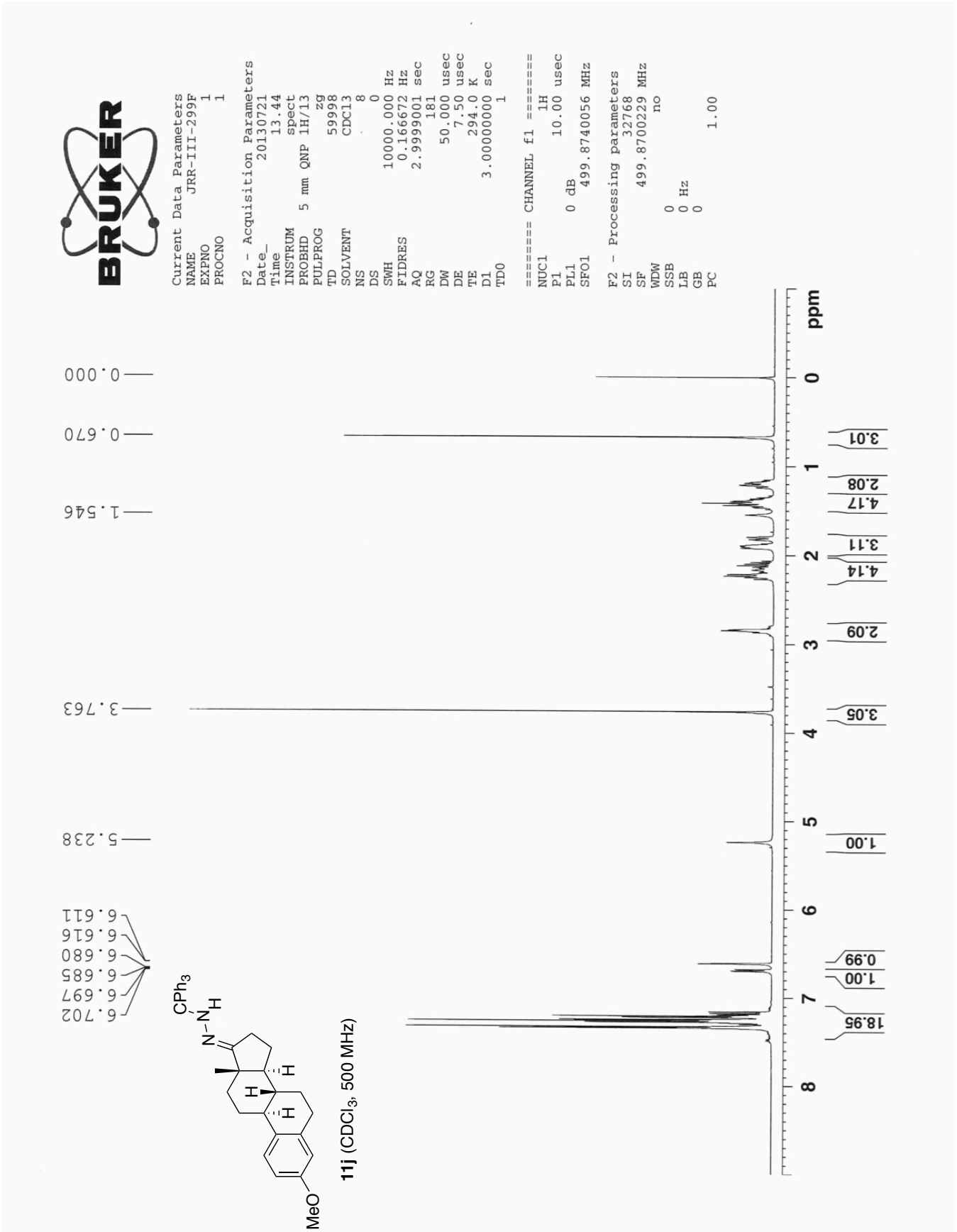

N–N–CPh3
H
11h (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-005A
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130710
Time 20.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 430
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 13004
DW 16.500 usec
DE 7.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924243 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
160.39
145.82
128.99
127.68
126.42
77.26
76.75
72.76
47.86
47.07
45.20
40.06
35.99
32.43
32.27
31.33
27.65
220 200 180 160 140 120 100 80 60 40 20 ppm

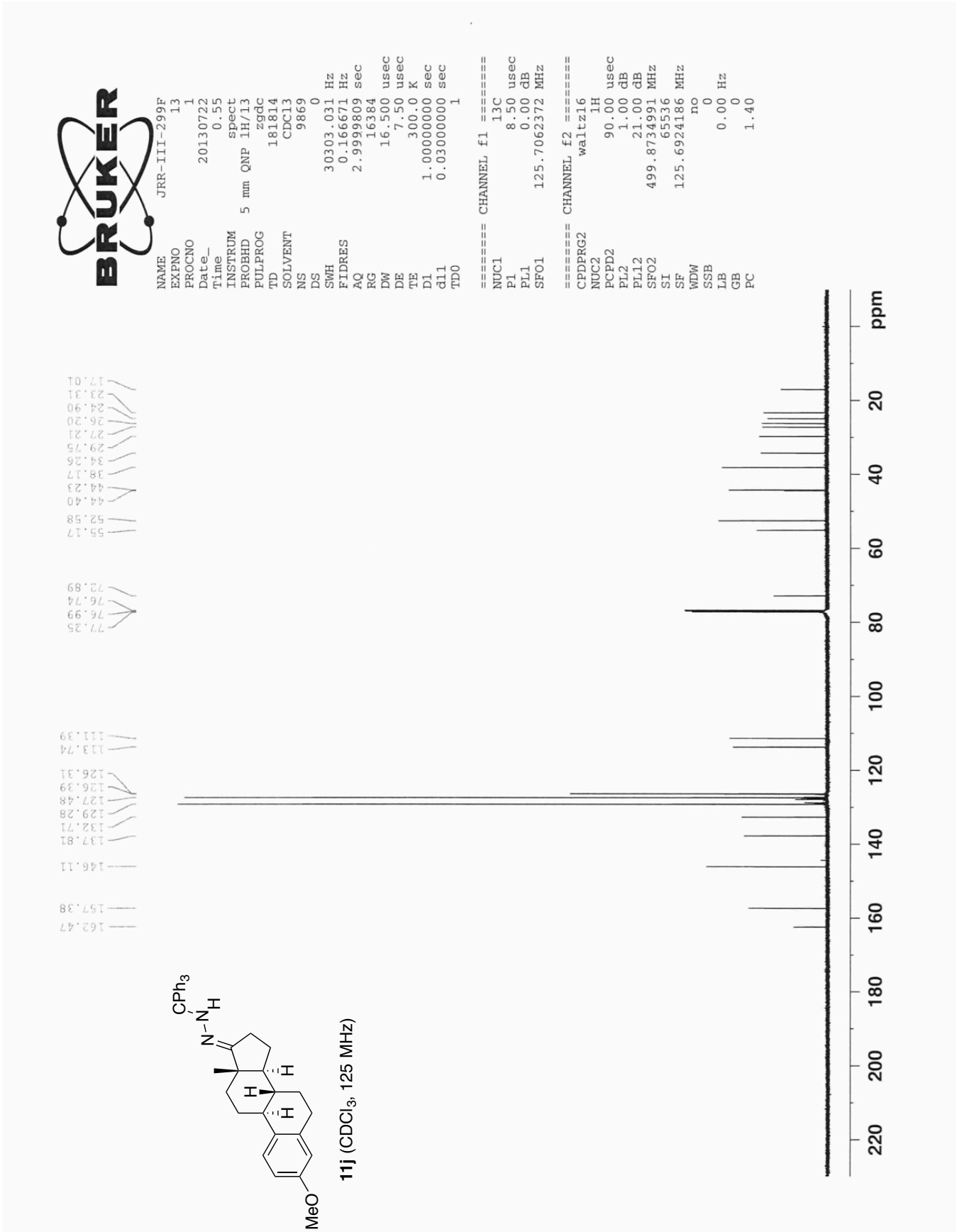

i-Pr
H
N
N
CPh3
11i (CDCl3, 500MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-045B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140225
Time 20.56
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.99999001 sec
RG 141.13
DW 50.000 usec
DE 10.00 usec
TE 295.5 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300141 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
0.98
1.00
1.08
9.34
1.09
1.00
1.01
1.04
0.97
6.68
8.32
3.76
5.249
2.422
2.408
2.394
2.379
2.212
2.178
2.117
2.084
1.542
1.329
1.315
1.302
1.288
1.274
0.947
0.933
0.920
0.906
0.890
0.876
0.807
0.799
0.791
0.783
0.465

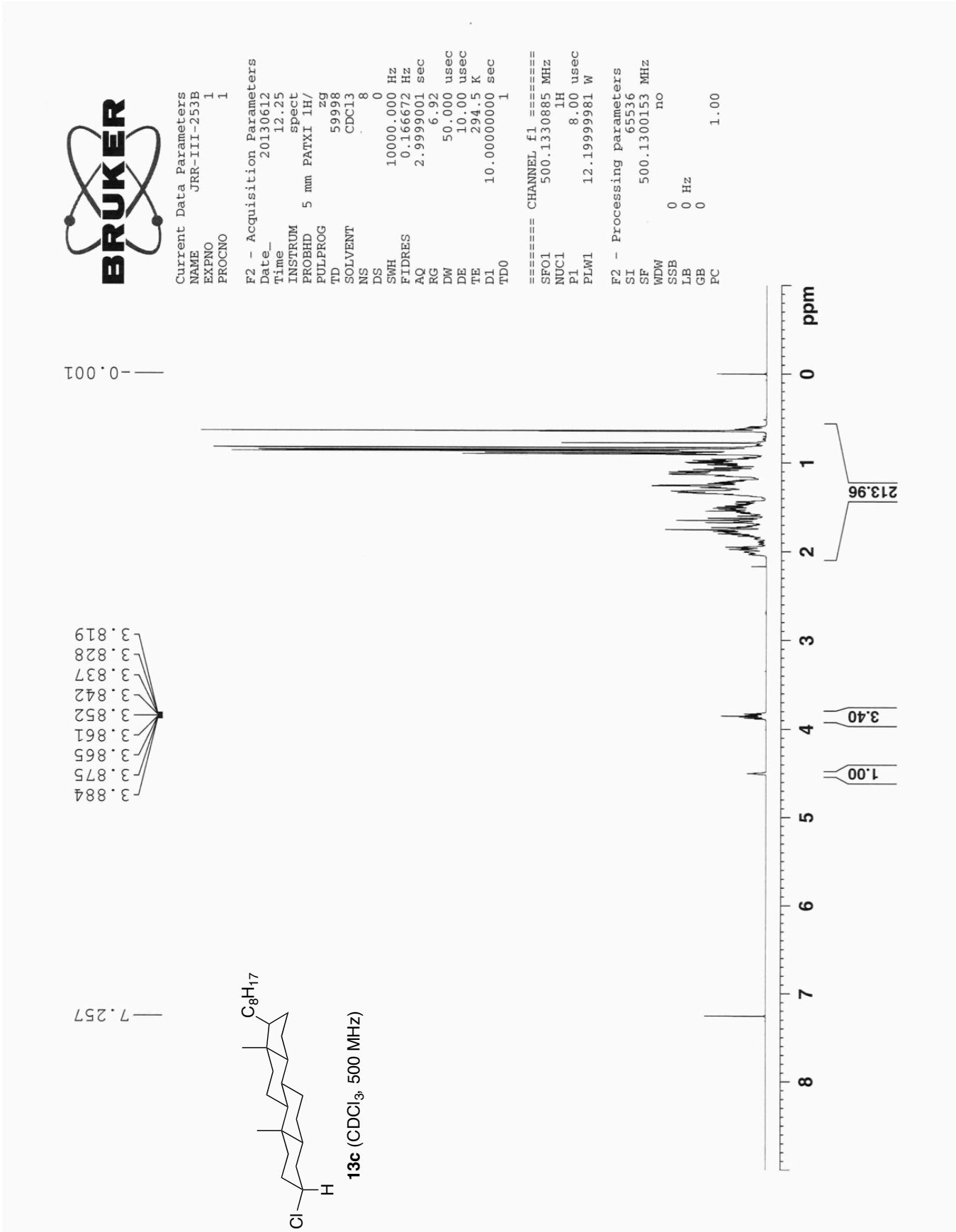

i-Pr
H
N
N
CPh3
11i (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-014Ai
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130717
Time 8.53
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgdc
TD 178568
SOLVENT CDCl3
NS 1043
DS 0
SWH 29761.904 Hz
FIDRES 0.166670 Hz
AQ 2.9999423 sec
RG 196.79
DW 16.800 usec
DE 10.00 usec
TE 296.3 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703643 MHz
NUC1 13C
P1 14.00 usec
PLW1 170.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.19999981 W
PLW12 0.20893000 W
F2 - Processing parameters
SI 131072
SF 125.7577900 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
ppm
200
180
160
140
120
100
80
60
40
20

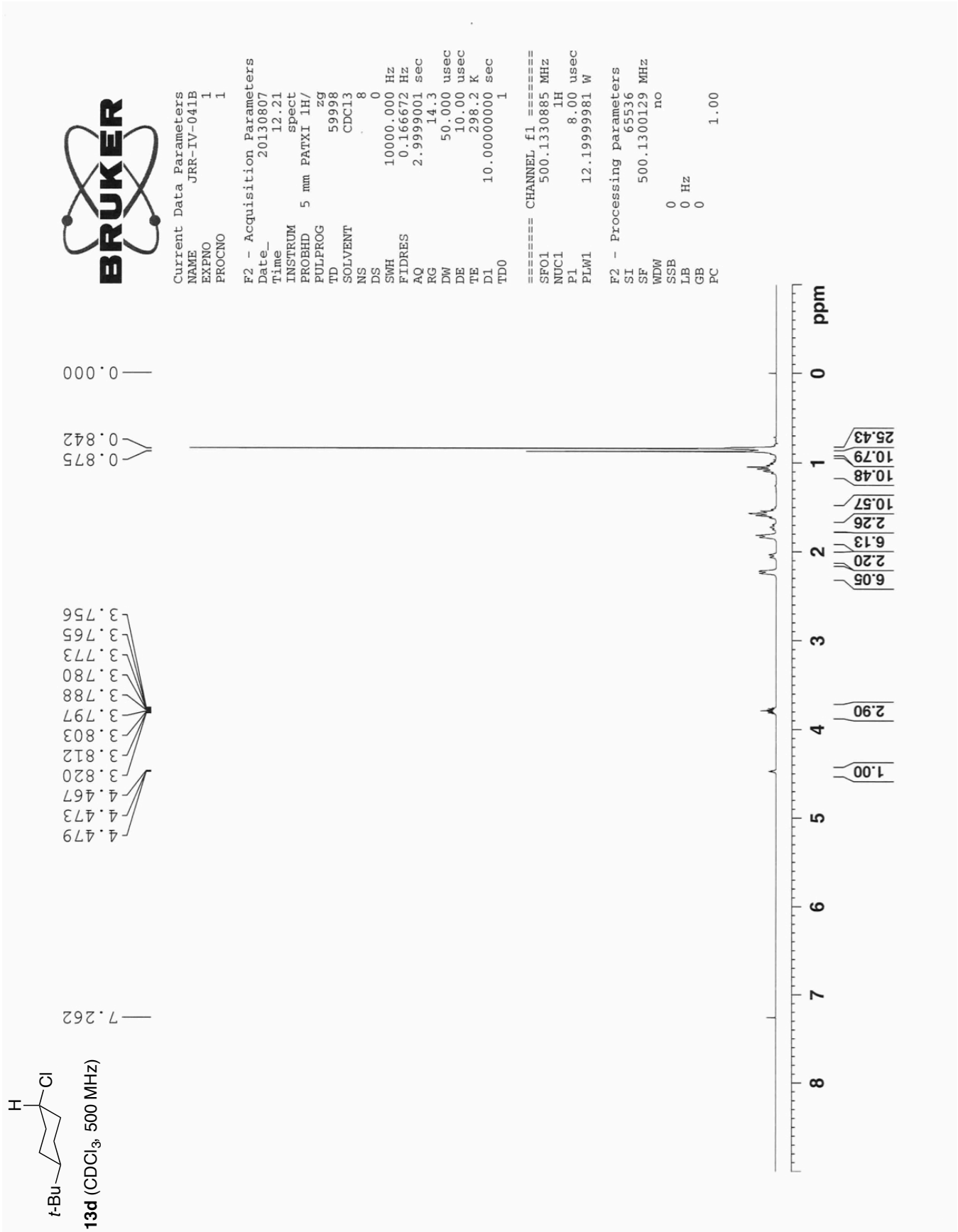

i-Pr
H
N
N
CPh3
11i - COSY
(CDCl3, 500 MHz)
BRUKER
ppm
−0.5
0.0
0.5
1.0
1.5
2.0
2.5

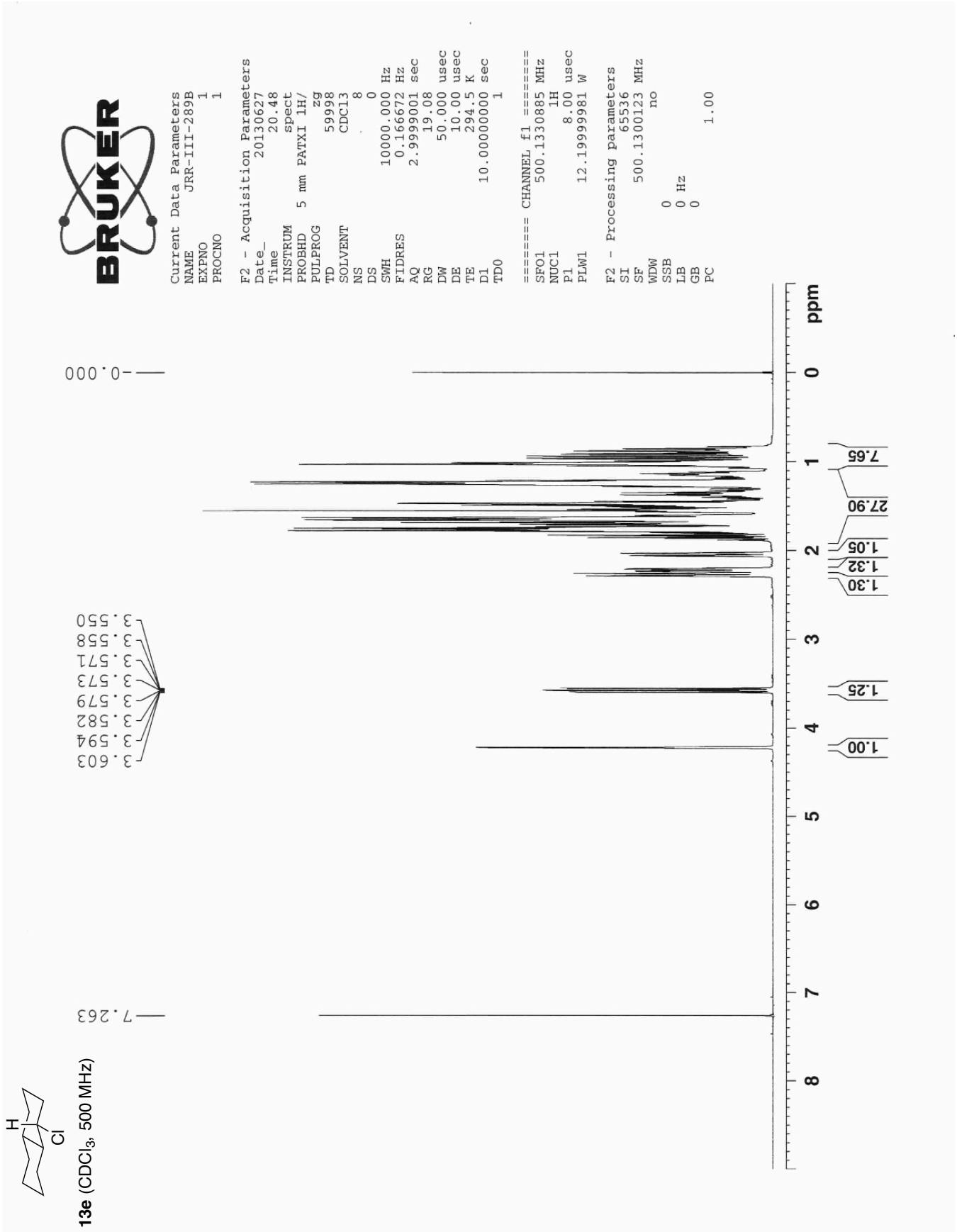

i-Pr
H
N
N
CPh3
11i - NOESY
(CDCl3, 500 MHz)
BRUKER
ppm
−0.5
0.0
0.5
1.0
1.5
2.0
2.5

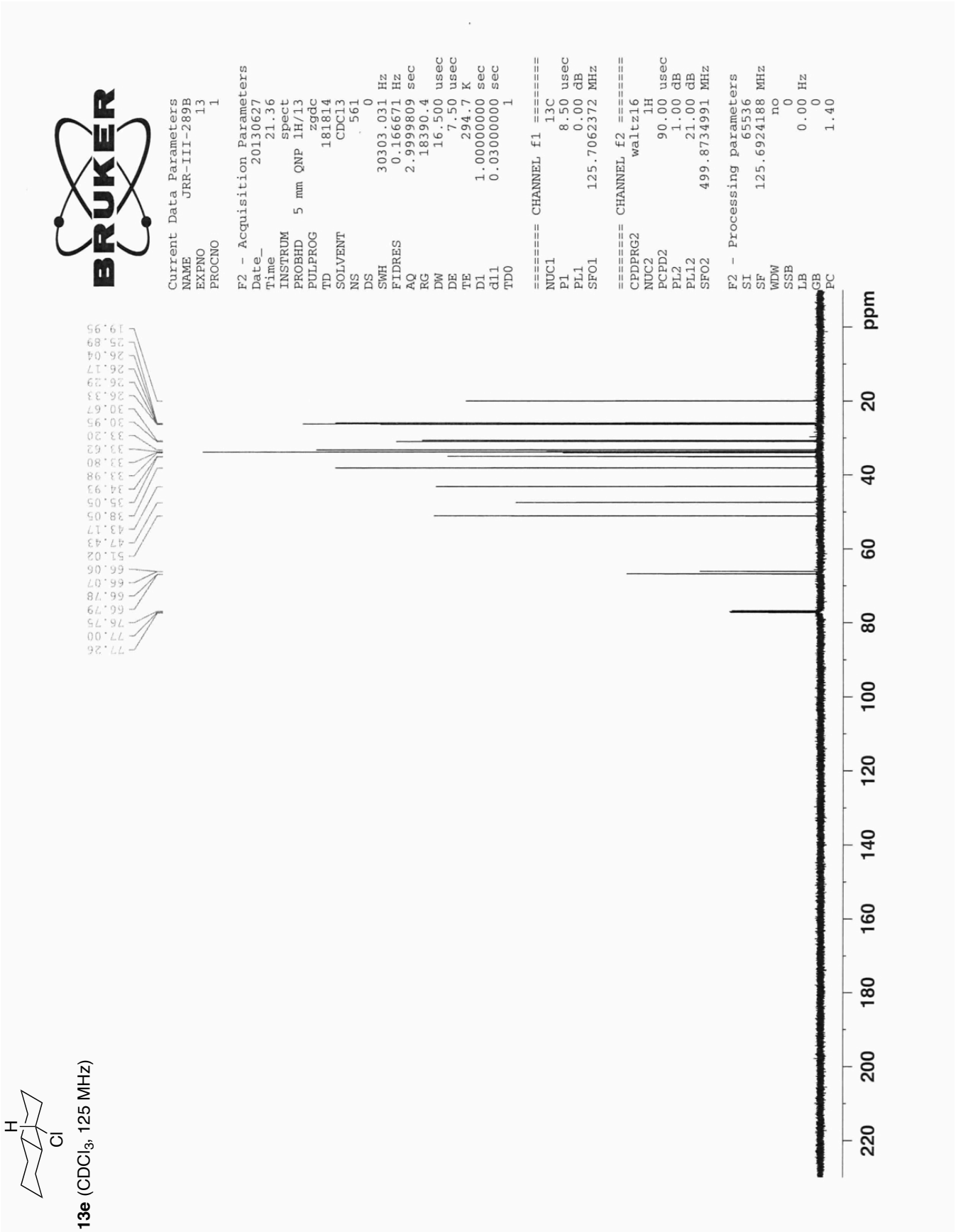

BRUKER
Current Data Parameters
NAME JRR-III-299F
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130721
Time 13.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 181
DW 50.000 usec
DE 7.50 usec
TE 294.0 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0 dB
SFO1 499.8740056 MHz
F2 - Processing parameters
SI 32768
SF 499.8700229 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
0.000
0.670
1.546
3.763
5.238
6.611
6.616
6.680
6.685
6.697
6.702
3.01
2.08
4.17
3.11
4.14
2.09
3.05
1.00
0.99
1.00
18.95
ppm
CPh3
N
N
H
H
H
MeO
11j (CDCl3, 500 MHz)

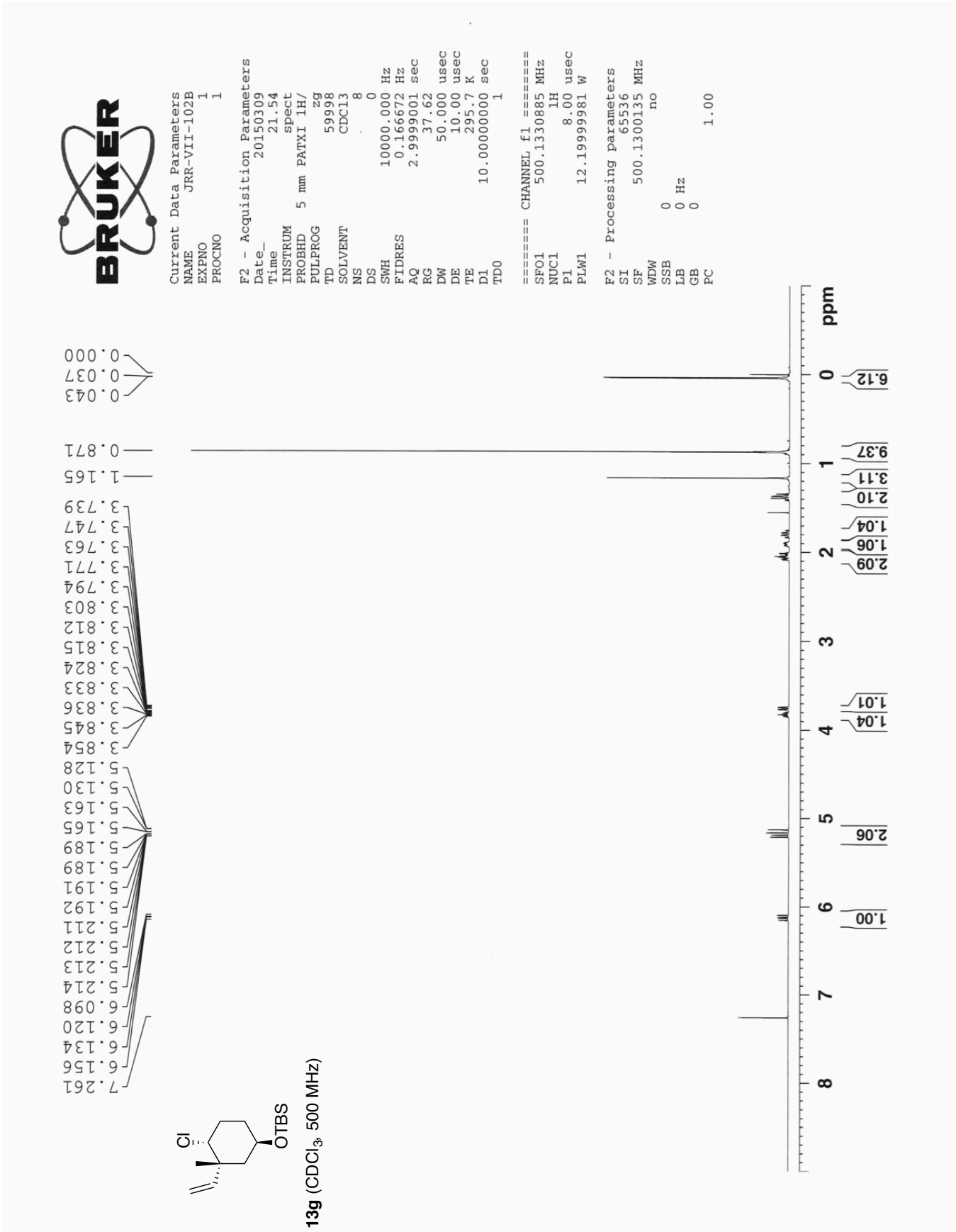

BRUKER
NAME JRR-III-299F
EXPNO 13
PROCNO 1
Date_ 20130722
Time 0.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 9869
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.99999809 sec
RG 16384
DW 16.500 usec
DE 7.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
SI 65536
SF 125.6924186 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
CPh3
N
N
H
H
H
MeO
11j (CDCl3, 125 MHz)
ppm

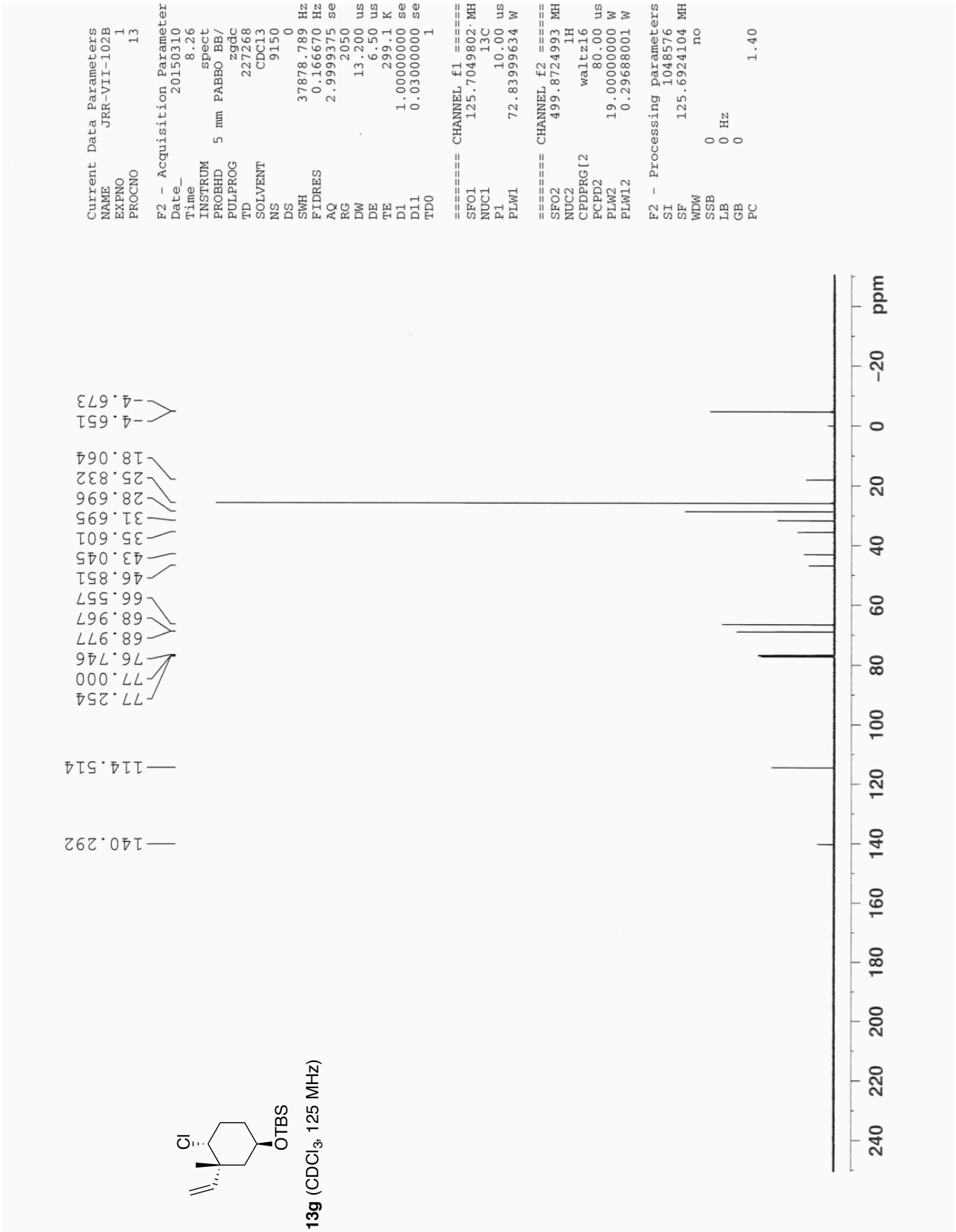

Current Data Parameters
NAME JRR-III-253B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130612
Time 12.25
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 6.92
DW 50.000 usec
DE 10.00 usec
TE 294.5 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300153 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.257
3.884
3.875
3.865
3.861
3.852
3.842
3.837
3.828
3.819
0.001
213.96
3.40
1.00
ppm
13c (CDCl3, 500 MHz)
C8H17
Cl
H

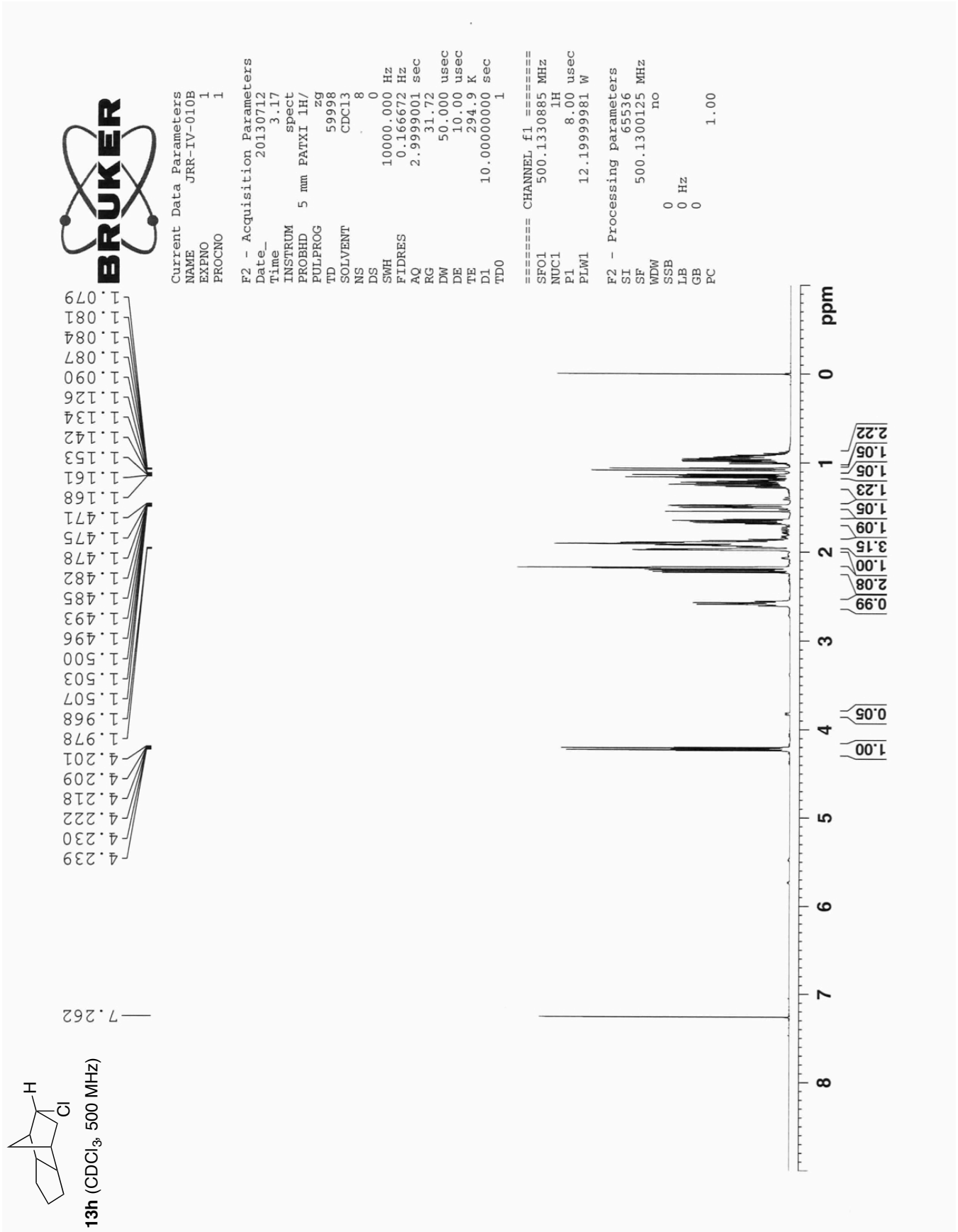

H
Cl
t-Bu
13d (CDCl3, 500 MHz)
7.262
4.479
4.473
4.467
3.820
3.812
3.803
3.797
3.788
3.780
3.773
3.765
3.756
0.875
0.842
0.000
1.00
2.90
6.05
2.20
6.13
2.26
10.57
10.48
10.79
25.43
ppm
0
1
2
3
4
5
6
7
8
BRUKER
Current Data Parameters
NAME JRR-IV-041B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130807
Time 12.21
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 14.3
DW 50.000 usec
DE 10.00 usec
TE 298.2 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300129 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

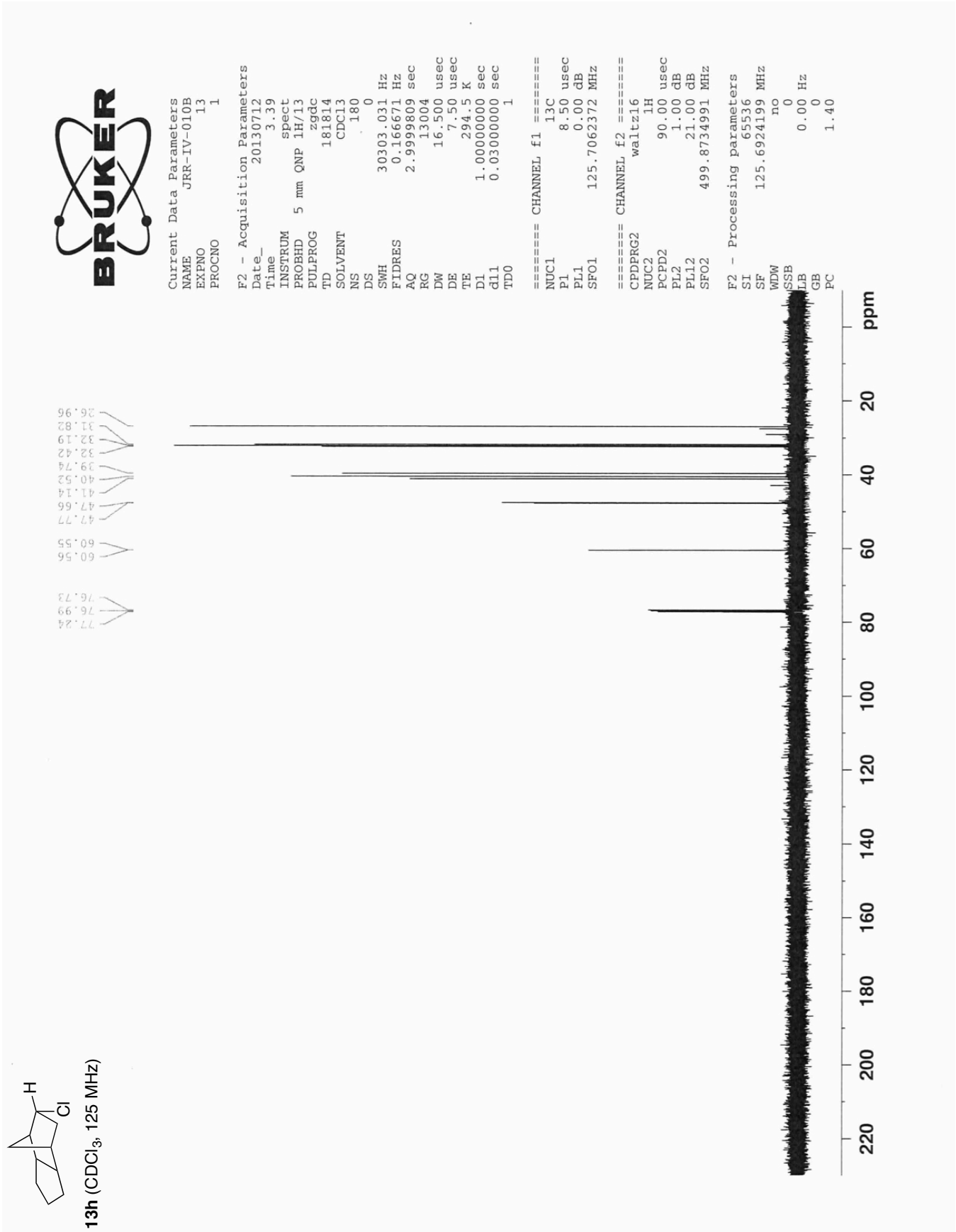

Current Data Parameters
NAME JRR-III-289B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130627
Time 20.48
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.99990001 sec
RG 19.08
DW 50.000 usec
DE 10.00 usec
TE 294.5 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
0.000
7.263
3.603
3.594
3.582
3.579
3.573
3.571
3.558
3.550
7.65
27.90
1.05
1.32
1.30
1.25
1.00
ppm
H
Cl
13e (CDCl3, 500 MHz)

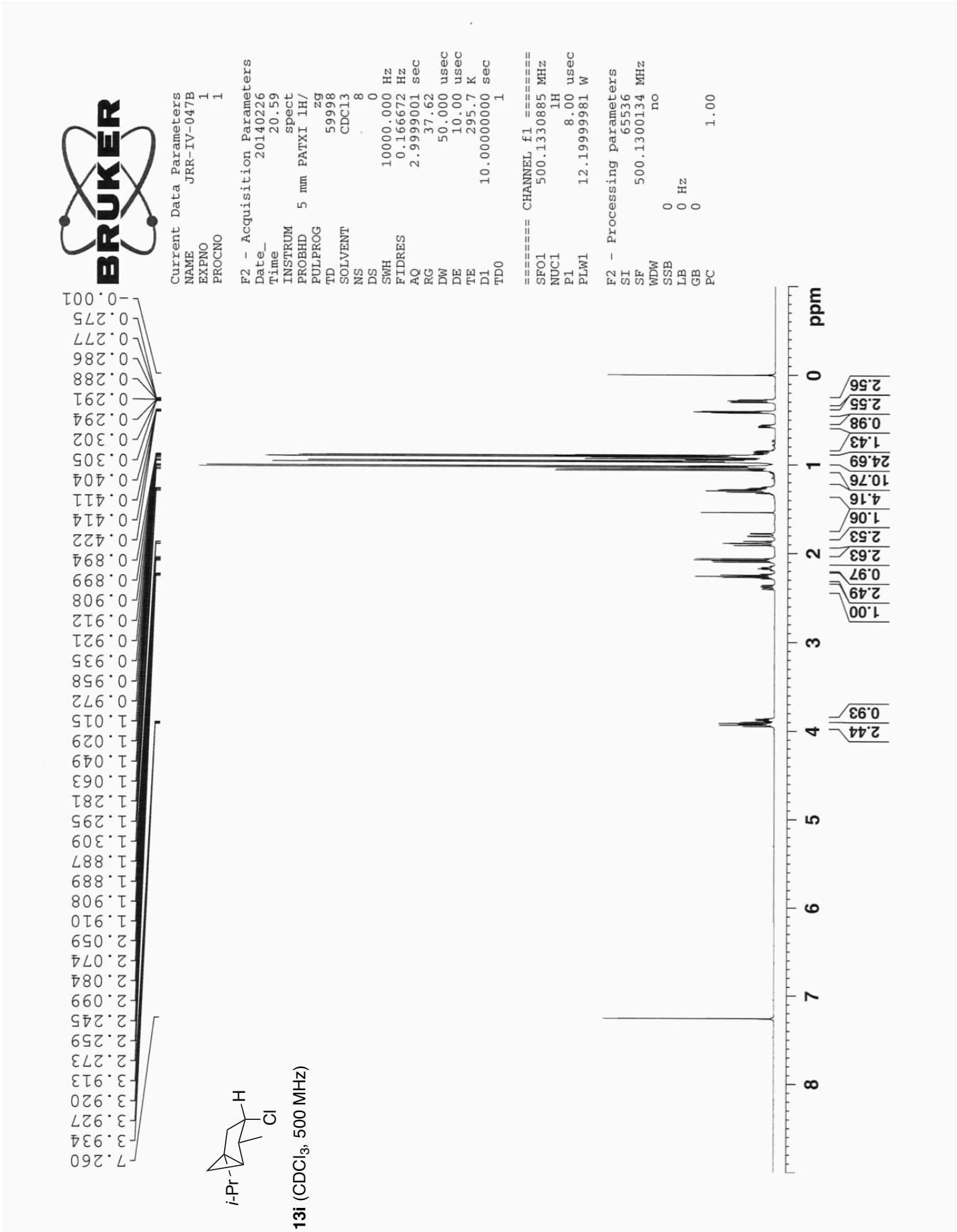

H
Cl
13e (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-III-289B
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130627
Time 21.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 561
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 18390.4
DW 16.500 usec
DE 7.50 usec
TE 294.7 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924188 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
19.95
25.89
26.04
26.17
26.29
26.33
30.67
30.95
33.20
33.62
33.80
33.98
34.93
35.05
38.05
43.17
47.43
51.02
66.06
66.07
66.78
66.79
76.75
77.00
77.26
220
200
180
160
140
120
100
80
60
40
20
ppm

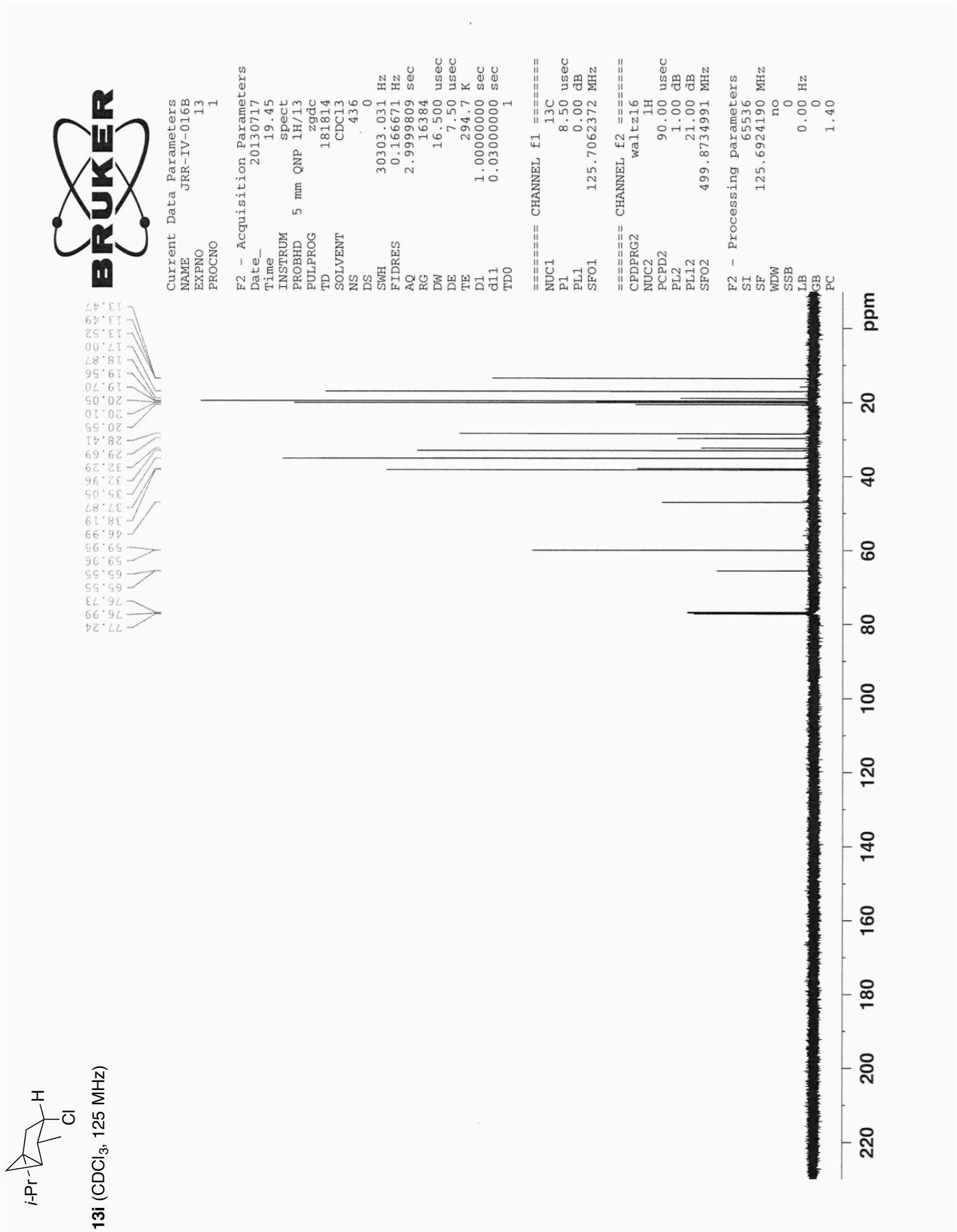

BRUKER
Current Data Parameters
NAME JRR-VII-102B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150309
Time 21.54
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 37.62
DW 50.000 usec
DE 10.00 usec
TE 295.7 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300135 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
0.043
0.037
0.000
0.871
1.165
3.739
3.747
3.763
3.771
3.794
3.803
3.812
3.815
3.824
3.833
3.836
3.845
3.854
5.128
5.130
5.163
5.165
5.189
5.189
5.191
5.192
5.211
5.212
5.213
5.214
6.098
6.120
6.134
6.156
7.261
6.12
9.37
3.11
2.10
1.04
1.06
2.09
1.04
1.01
2.06
1.00
ppm
Cl
OTBS
13g (CDCl3, 500 MHz)

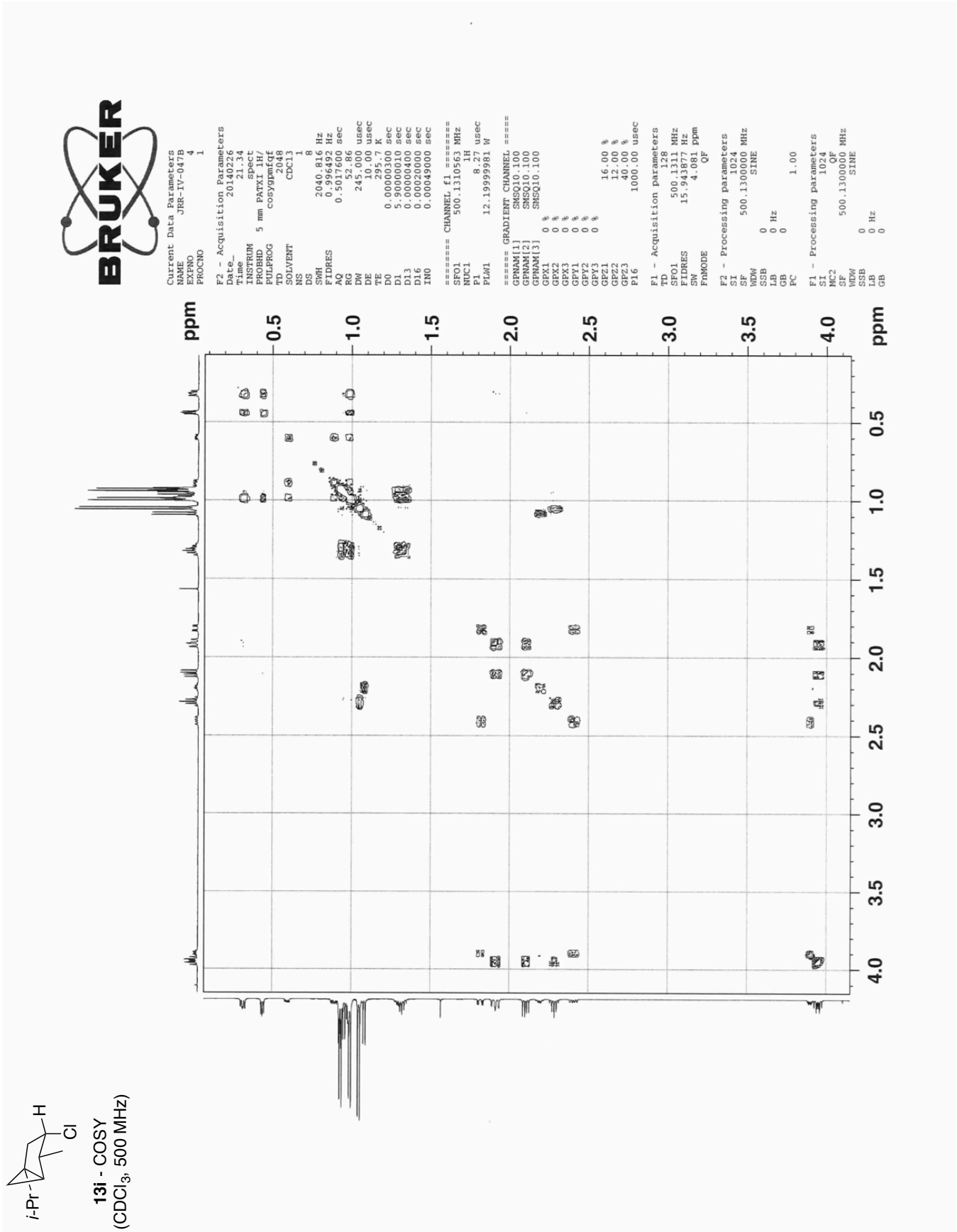

Current Data Parameters
NAME JRR-VII-102B
EXPNO 1
PROCNO 13
F2 - Acquisition Parameter
Date_ 20150310
Time 8.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgdc
TD 227268
SOLVENT CDCl3
NS 9150
DS 0
SWH 37878.789 Hz
FIDRES 0.166670 Hz
AQ 2.9999375 se
RG 2050
DW 13.200 us
DE 6.50 us
TE 299.1 K
D1 1.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 125.7049802 MH
NUC1 13C
P1 10.00 us
PLW1 72.83999634 W
======== CHANNEL f2 ======
SFO2 499.8724993 MH
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 us
PLW2 19.00000000 W
PLW12 0.29688001 W
F2 - Processing parameters
SI 1048576
SF 125.6924104 MH
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
140.292
114.514
77.254
77.000
76.746
68.977
68.967
66.557
46.851
43.045
35.601
31.695
28.696
25.832
18.064
-4.651
-4.673
240 220 200 180 160 140 120 100 80 60 40 20 0 -20 ppm
Cl
OTBS
13g ($CDCl_3$, 125 MHz)

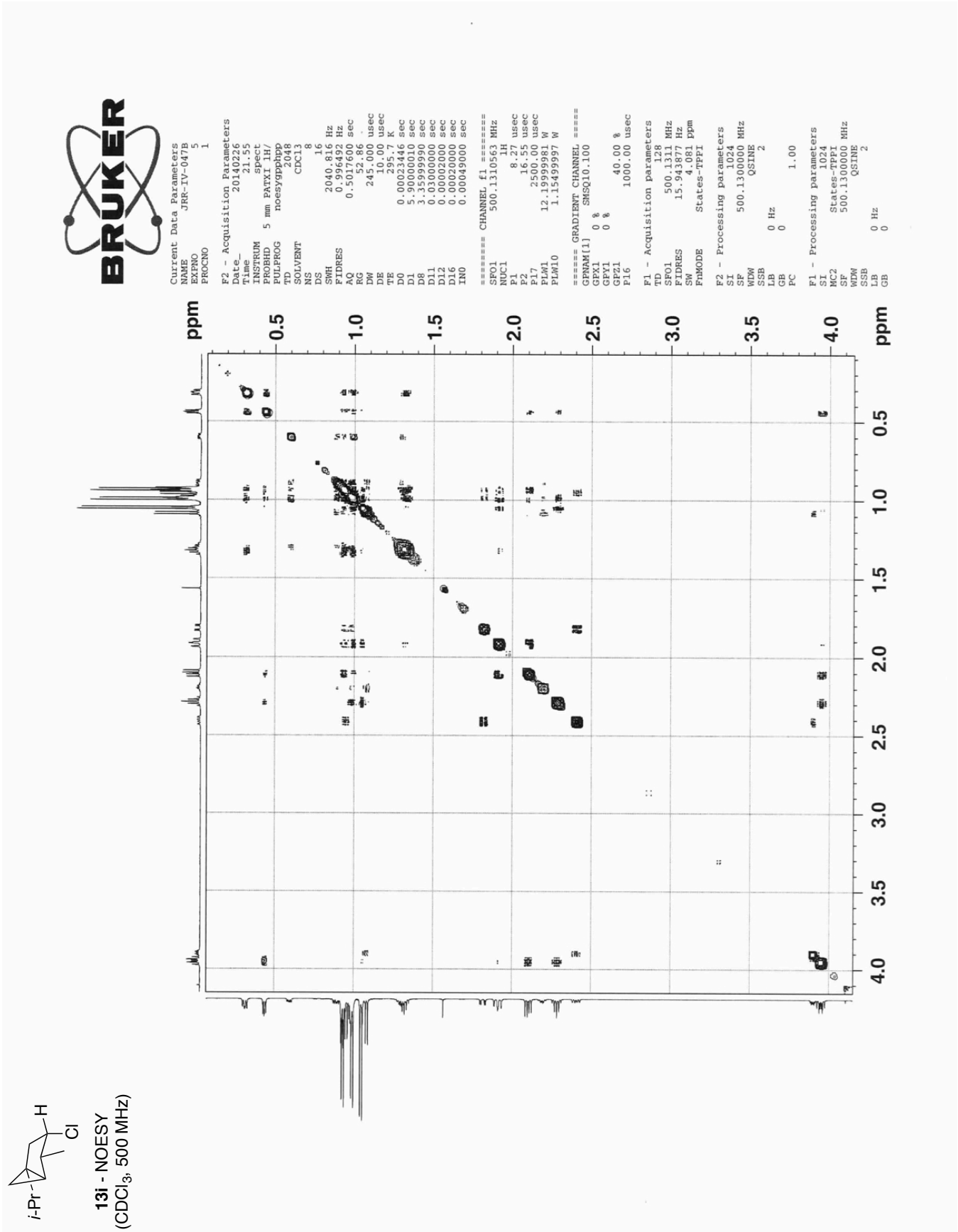

13h (CDCl3, 500 MHz)
Cl
H
7.262
1.079
1.081
1.084
1.087
1.090
1.126
1.134
1.142
1.153
1.161
1.168
1.471
1.475
1.478
1.482
1.485
1.493
1.496
1.500
1.503
1.507
1.968
1.978
4.201
4.209
4.218
4.222
4.230
4.239
BRUKER
Current Data Parameters
NAME JRR-IV-010B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130712
Time 3.17
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 31.72
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
1.00
0.05
0.99
2.08
1.00
3.15
1.09
1.05
1.23
1.05
1.05
2.22

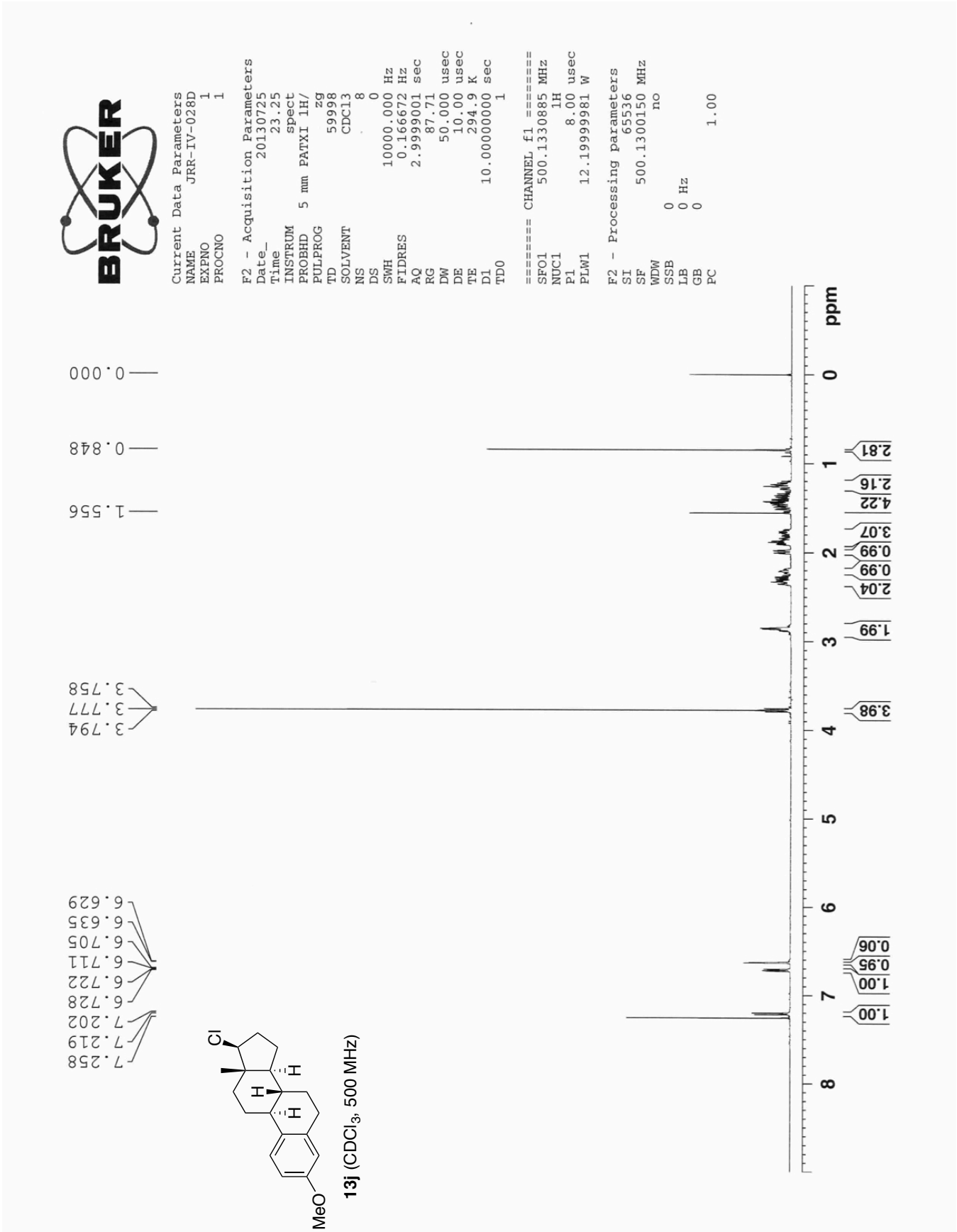

H
Cl
13h (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-010B
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130712
Time 3.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 180
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 13004
DW 16.500 usec
DE 7.50 usec
TE 294.5 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924199 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
77.24
76.99
76.73
60.56
60.55
47.77
47.66
41.14
40.52
39.74
32.42
32.19
31.82
26.96
220
200
180
160
140
120
100
80
60
40
20
ppm

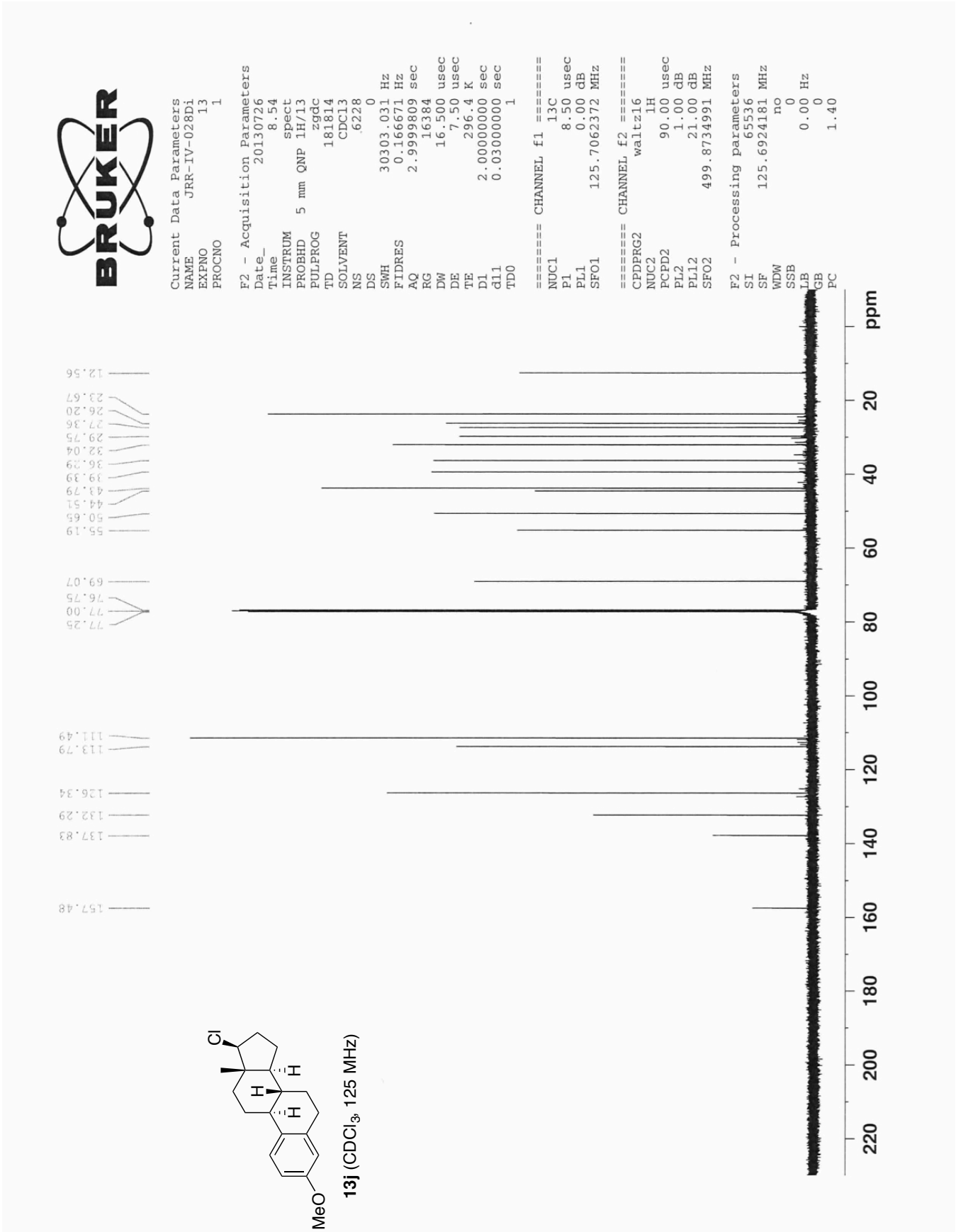

BRUKER
Current Data Parameters
NAME JRR-IV-047B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140226
Time 20.59
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 37.62
DW 50.000 usec
DE 10.00 usec
TE 295.7 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300134 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
i-Pr
H
Cl
13i (CDCl3, 500 MHz)


i-Pr
H
Cl
13i (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-016B
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130717
Time 19.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 436
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 16384
DW 16.500 usec
DE 7.50 usec
TE 294.7 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
13.47
13.49
13.52
17.00
18.87
19.56
19.70
20.05
20.10
20.55
28.41
29.69
32.29
32.96
35.05
37.87
38.19
46.99
59.95
59.96
65.55
65.55
76.73
76.99
77.24
220
200
180
160
140
120
100
80
60
40
20
ppm

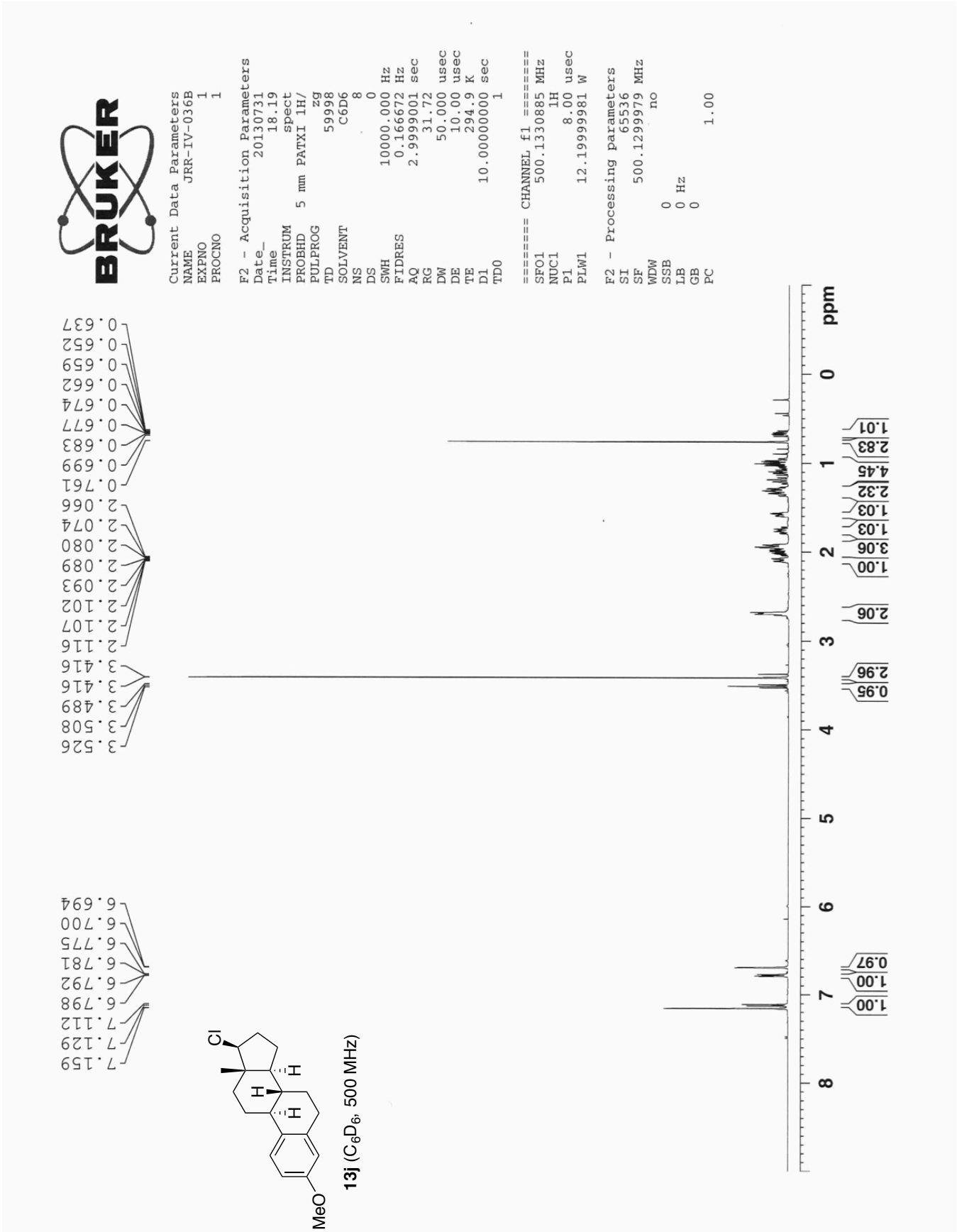

BRUKER
Current Data Parameters
NAME JRR-IV-047B
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140226
Time 21.34
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG cosygpmfqf
TD 2048
SOLVENT CDCl3
NS 1
DS 8
SWH 2040.816 Hz
FIDRES 0.996492 Hz
AQ 0.5017600 sec
RG 52.86
DW 245.000 usec
DE 10.00 usec
TE 295.7 K
D0 0.00000300 sec
D1 5.90000010 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00049000 sec
======== CHANNEL f1 ========
SFO1 500.1310563 MHz
NUC1 1H
P1 8.27 usec
PLW1 12.19999981 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 16.00 %
GPZ2 12.00 %
GPZ3 40.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 128
SFO1 500.1311 MHz
FIDRES 15.943877 Hz
SW 4.081 ppm
FnMODE QF
F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
i-Pr
H
Cl
13i - COSY
(CDCl3, 500 MHz)

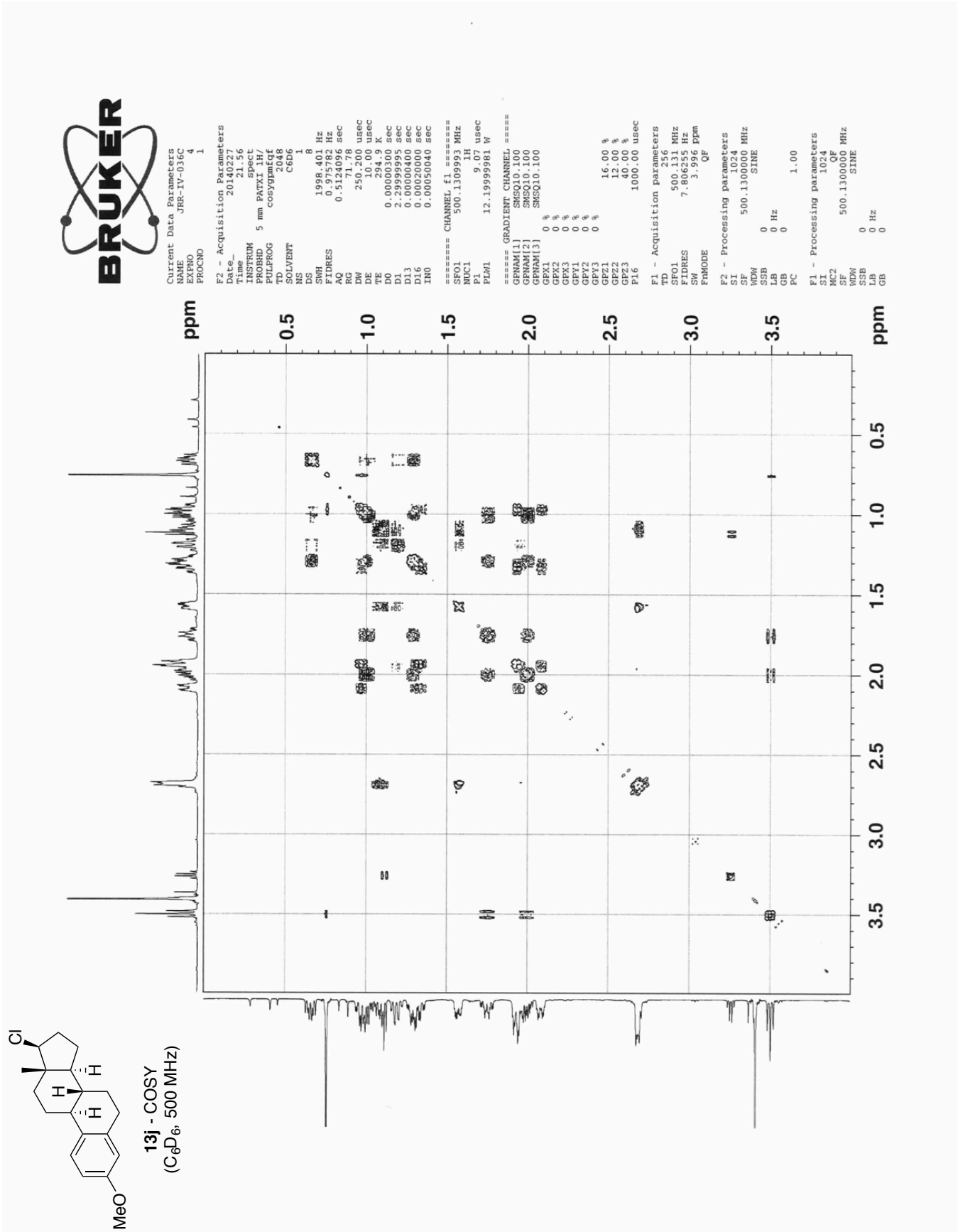

i-Pr
H
Cl
13i - NOESY
(CDCl3, 500 MHz)
BRUKER
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0

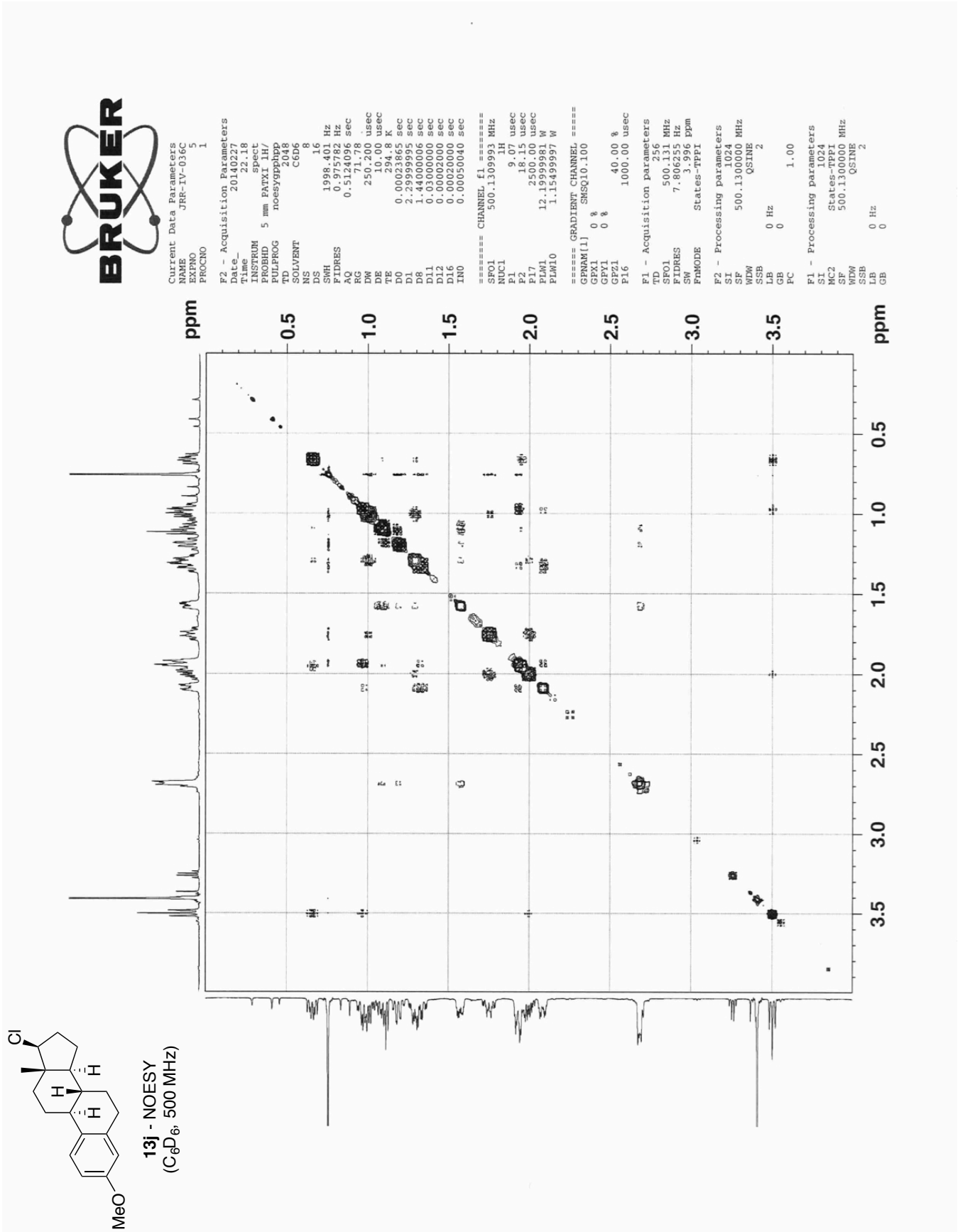

Current Data Parameters
NAME JRR-IV-028D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130725
Time 23.25
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 87.71
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300150 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.258
7.219
7.202
6.728
6.722
6.711
6.705
6.635
6.629
3.794
3.777
3.758
1.556
0.848
0.000
2.81
2.16
4.22
3.07
0.99
0.99
2.04
1.99
3.98
1.00
0.95
1.00
0.06
ppm
Cl
H
H
H
MeO
13j (CDCl3, 500 MHz)

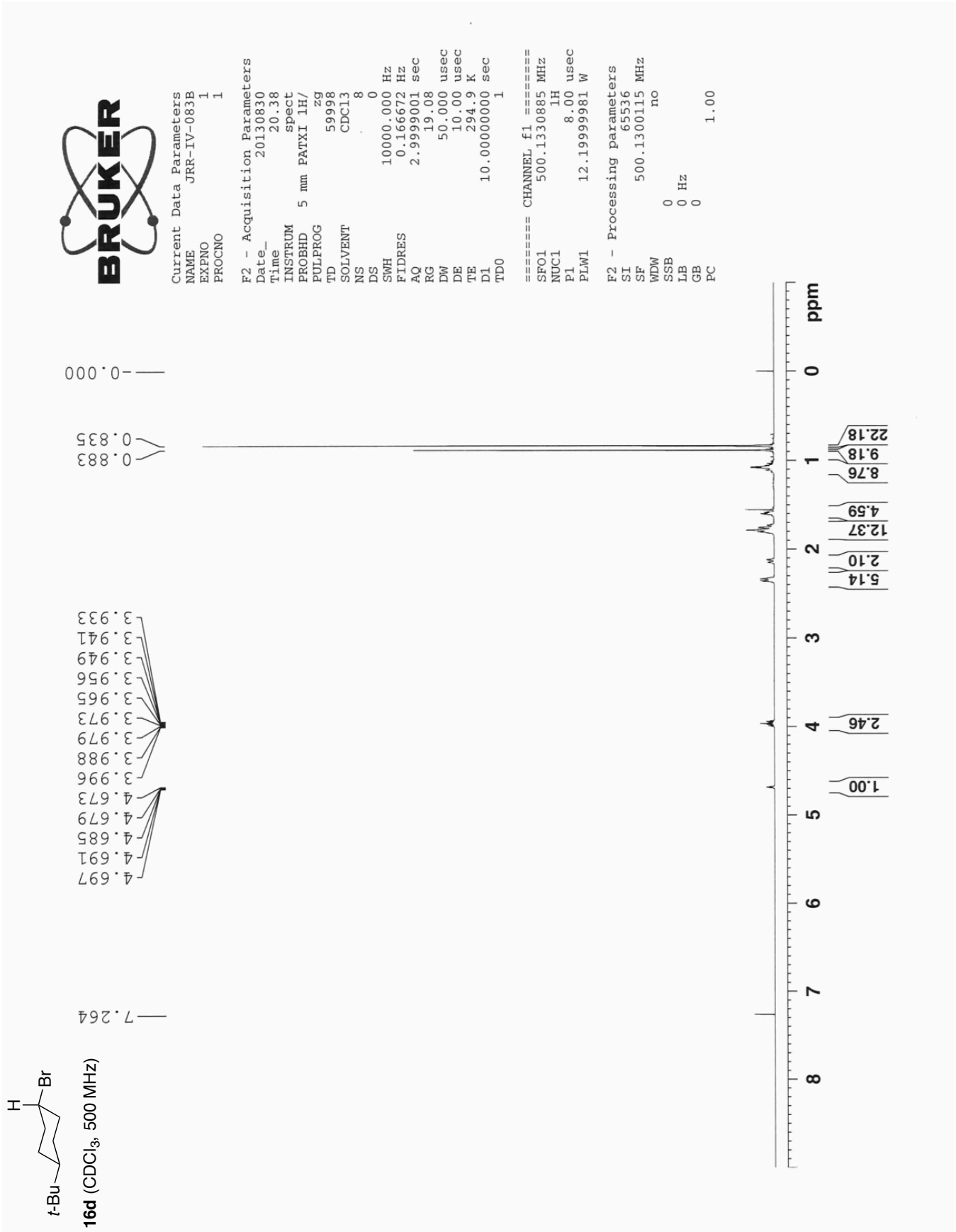


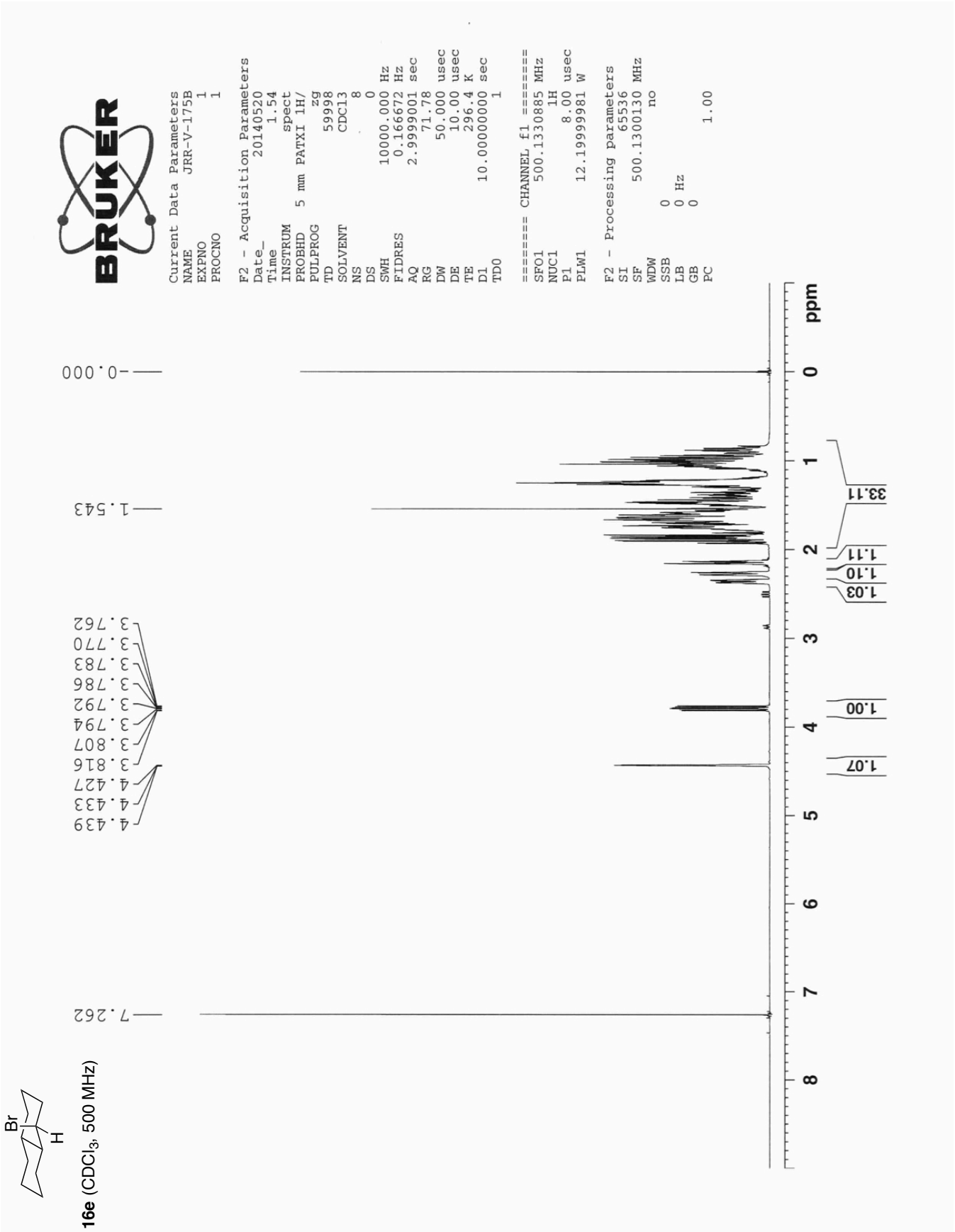
13j ($CDCl_3$, 125 MHz)

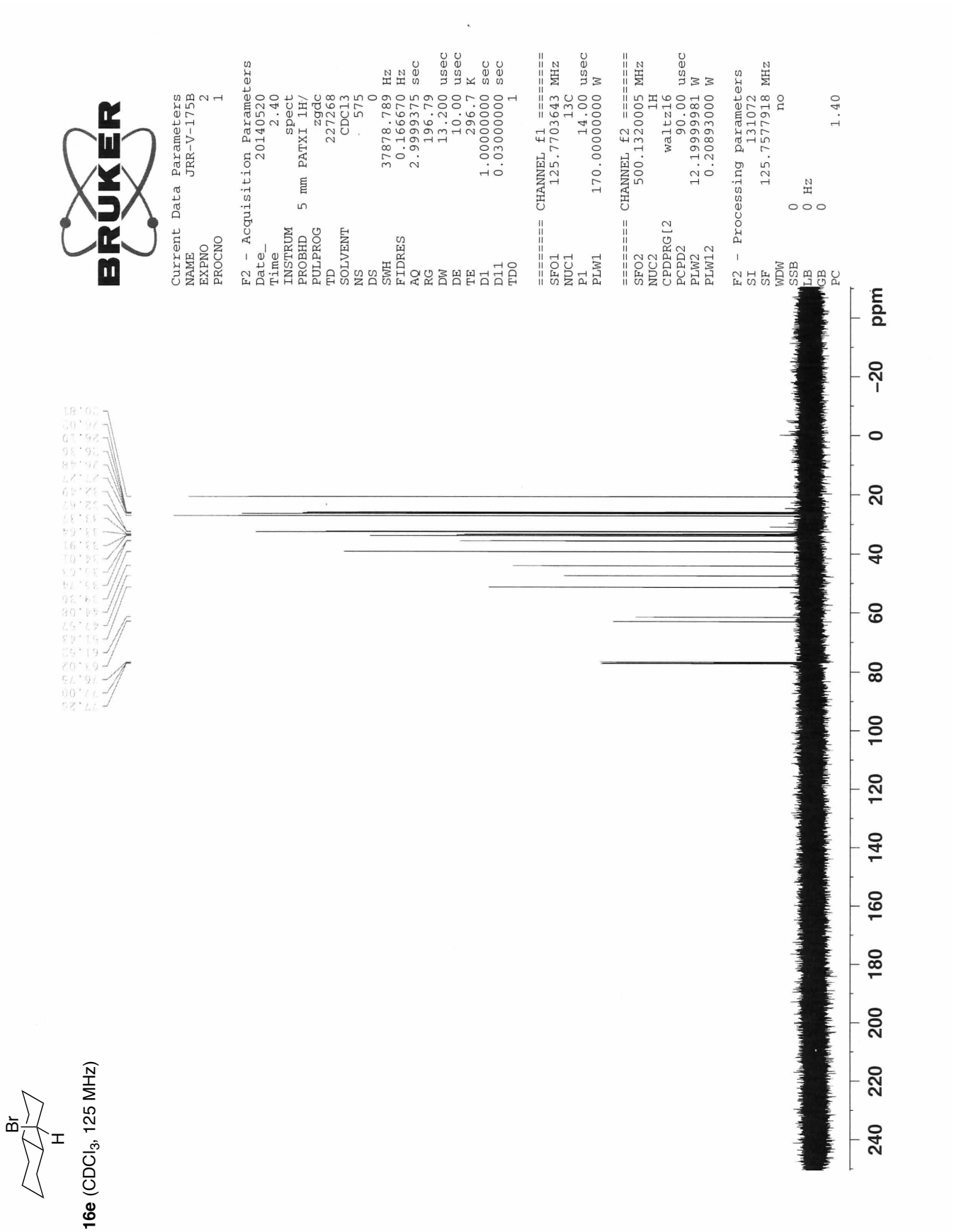

Current Data Parameters
NAME JRR-IV-036B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130731
Time_ 18.19
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT C6D6
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.99999001 sec
RG 31.72
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1299979 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
BRUKER
7.159
7.129
7.112
6.798
6.792
6.781
6.775
6.700
6.694
3.526
3.508
3.489
3.416
3.416
2.116
2.107
2.102
2.093
2.089
2.080
2.074
2.066
0.761
0.699
0.683
0.677
0.674
0.662
0.659
0.652
0.637
0.97
1.00
1.00
2.96
0.95
2.06
1.00
3.06
1.03
1.03
2.32
4.45
2.83
1.01
ppm
Cl
H
H
H
MeO
13j (C6D6, 500 MHz)

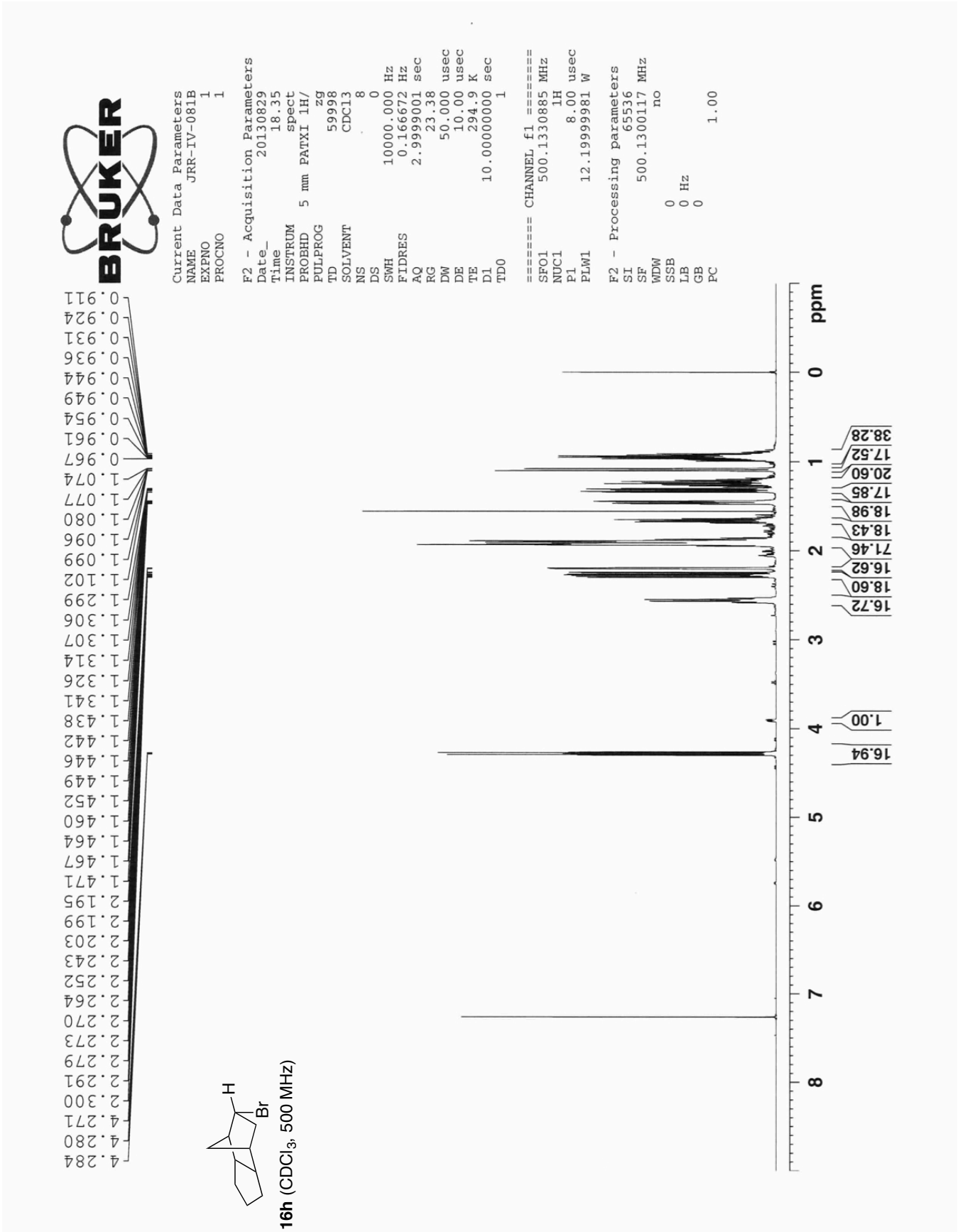

BRUKER
Current Data Parameters
NAME JRR-IV-036C
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140227
Time 21.56
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG cosygpmfqf
TD 2048
SOLVENT C6D6
NS 1
DS 8
SWH 1998.401 Hz
FIDRES 0.975782 Hz
AQ 0.5124096 sec
RG 71.78
DW 250.200 usec
DE 10.00 usec
TE 294.9 K
D0 0.00000300 sec
D1 2.29999995 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00050040 sec
======== CHANNEL f1 ========
SFO1 500.1309993 MHz
NUC1 1H
P1 9.07 usec
PLW1 12.19999981 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 16.00 %
GPZ2 12.00 %
GPZ3 40.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.131 MHz
FIDRES 7.806255 Hz
SW 3.996 ppm
FnMODE QF
F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
Cl
H
H
H
MeO
13j - COSY
(C_6D_6, 500 MHz)

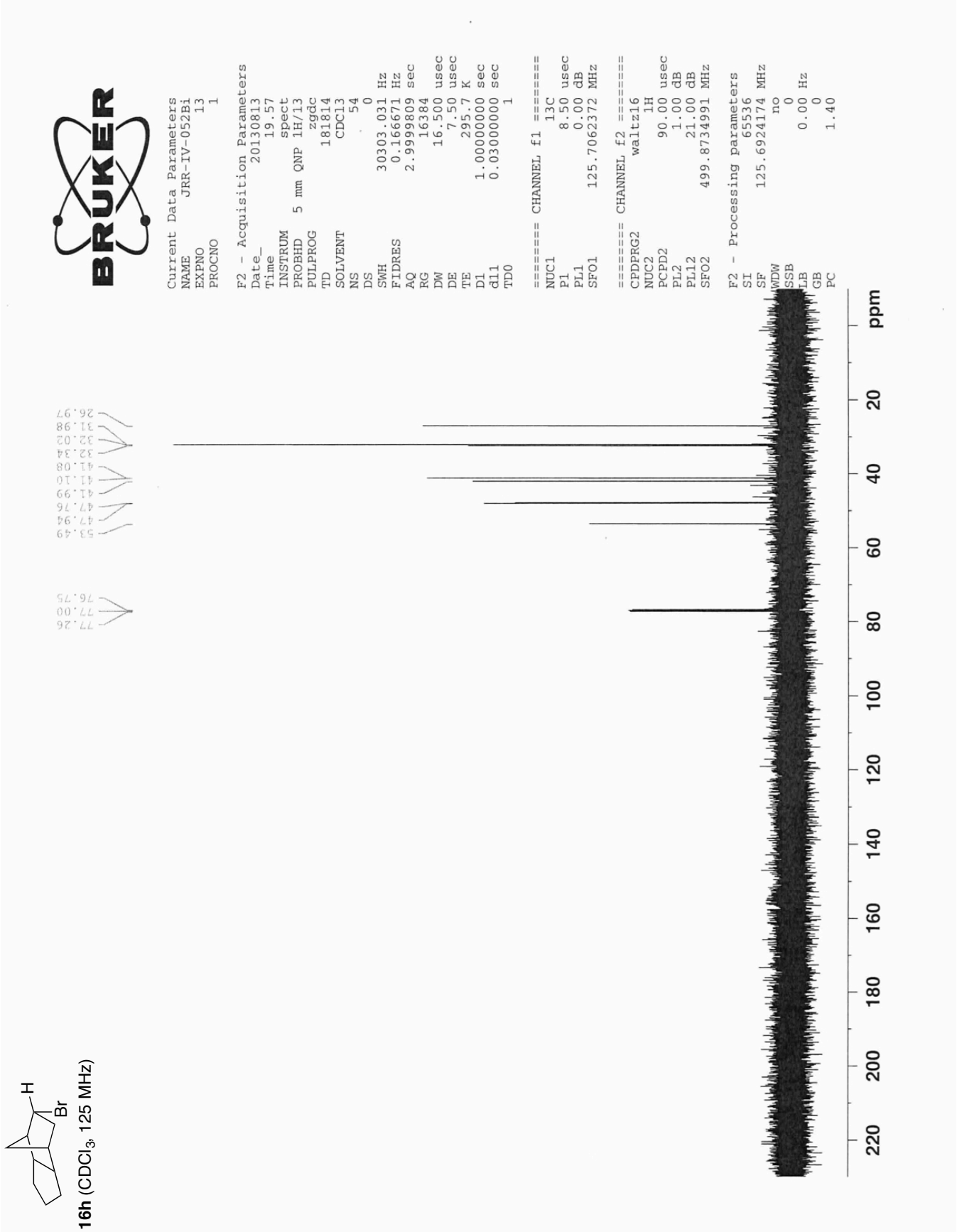

BRUKER
Current Data Parameters
NAME JRR-IV-036C
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140227
Time 22.18
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG noesygpphpp
TD 2048
SOLVENT C6D6
NS 8
DS 16
SWH 1998.401 Hz
FIDRES 0.975782 Hz
AQ 0.5124096 sec
RG 71.78
DW 250.200 usec
DE 10.00 usec
TE 294.8 K
D0 0.00023865 sec
D1 2.29999995 sec
D8 1.44000006 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00050040 sec
======== CHANNEL f1 ========
SFO1 500.1309993 MHz
NUC1 1H
P1 9.07 usec
P2 18.15 usec
P17 2500.00 usec
PLW1 12.19999981 W
PLW10 1.15499997 W
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPX1 0 %
GPY1 0 %
GPZ1 40.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 256
SFO1 500.131 MHz
FIDRES 7.806255 Hz
SW 3.996 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
Cl
H
H
H
MeO
13j - NOESY
(C_6D_6, 500 MHz)


H
Br
t-Bu
16d (CDCl3, 500 MHz)
7.264
4.697
4.691
4.685
4.679
4.673
3.996
3.988
3.979
3.973
3.965
3.956
3.949
3.941
3.933
0.883
0.835
0.000
Current Data Parameters
NAME JRR-IV-083B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130830
Time 20.38
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 19.08
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300115 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
1.00
2.46
5.14
2.10
12.37
4.59
8.76
9.18
22.18


Br
H
16e (CDCl3, 500 MHz)
7.262
1.543
0.000
4.439
4.433
4.427
3.816
3.807
3.794
3.792
3.786
3.783
3.770
3.762
33.11
1.11
1.10
1.03
1.00
1.07
ppm
Current Data Parameters
NAME JRR-V-175B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140520
Time 1.54
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 71.78
DW 50.000 usec
DE 10.00 usec
TE 296.4 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300130 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00


Br
H
16e (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-V-175B
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140520
Time 2.40
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgdc
TD 227268
SOLVENT CDCl3
NS 575
DS 0
SWH 37878.789 Hz
FIDRES 0.166670 Hz
AQ 2.99999375 sec
RG 196.79
DW 13.200 usec
DE 10.00 usec
TE 296.7 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703643 MHz
NUC1 13C
P1 14.00 usec
PLW1 170.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.19999981 W
PLW12 0.20893000 W
F2 - Processing parameters
SI 131072
SF 125.7577918 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 0 −20 ppm


BRUKER
Current Data Parameters
NAME JRR-IV-081B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130829
Time 18.35
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDC13
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999001 sec
RG 23.38
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.19999981 W
F2 - Processing parameters
SI 65536
SF 500.1300117 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
4.284 4.280 4.271 2.300 2.291 2.279 2.273 2.270 2.264 2.252 2.243 2.203 2.199 2.195 1.471 1.467 1.464 1.460 1.452 1.449 1.446 1.442 1.438 1.341 1.326 1.314 1.307 1.306 1.299 1.102 1.099 1.096 1.080 1.077 1.074 0.967 0.961 0.954 0.949 0.944 0.936 0.931 0.924 0.911
H
Br
16h (CDCl3, 500 MHz)
8 7 6 5 4 3 2 1 0 ppm
16.94 1.00 16.72 18.60 16.62 71.46 18.43 18.98 17.85 20.60 17.52 38.28


H
Br
16h (CDCl3, 125 MHz)
BRUKER
Current Data Parameters
NAME JRR-IV-052Bi
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130813
Time 19.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 181814
SOLVENT CDCl3
NS 54
DS 0
SWH 30303.031 Hz
FIDRES 0.166671 Hz
AQ 2.9999809 sec
RG 16384
DW 16.500 usec
DE 7.50 usec
TE 295.7 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7062372 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 65536
SF 125.6924174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
77.26
77.00
76.75
53.49
47.94
47.76
41.99
41.10
41.08
32.34
32.02
31.98
26.97
220
200
180
160
140
120
100
80
60
40
20
ppm