Organocatalytic Dimerization of Ketoketenes

Supporting information

Ahmad A. Ibrahim, Gero D. Harzmann, and Nessan J. Kerrigan*

Department of Chemistry, Oakland University, 2200 N. Squirrel Rd., Rochester, Michigan 48309, USA.

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General Information.

Unless otherwise stated all reactions were carried out in flame dried glassware under a nitrogen atmosphere using standard inert atmosphere techniques. Diethyl ether and THF were dried using sodium benzophenone stills, CH_2Cl_2 and toluene were dried using calcium hydride stills, methanol was dried using a magnesium methoxide still, and *N*,*N*-dimethylethylamine was distilled from potassium hydroxide under nitrogen.[1] Tri-*n*-butylphosphine, trimethylphosphine (1.0 M solution in toluene), lithium iodide, *n*-butyl lithium (2.5 M in hexane), $LiAlH_4$ (1.0 M in Et_2O) and 2-hydroxypyridine were purchased from Aldrich Chemical Co. and used as received. Iatrobeads (Bioscan, 6RS-8060, 60μM particle size), and TLC plates (Sorbent Technologies, UV254, 250μM) were used as received. Methylphenylketene, methyl-*p*-tolylketene, ethylphenylketene, diphenylketene, 6-methoxy-naphthalenylmethylketene, isobutylphenylketene, ethyl-2-thiophenylketene, and cyclopentylphenylketene were prepared according to literature procedures.[2-4]

NMR spectra were recorded on a Bruker DPX Avance 200 spectrometer (200 MHz for ^1H and 50 MHz for ^{13}C). NMR chemical shifts were reported relative to TMS (0 ppm) for ^1H and to $CDCl_3$ (77.23 ppm) for ^{13}C spectra.

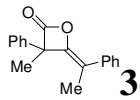
High resolution mass spectra were obtained from the College of Sciences Major Instrumentation Cluster at Old Dominion University. Low resolution mass spectra were recorded on a GC/MS Hewlett Packard HP 6890 GC instrument with a 5973 mass selective detector. IR spectra were recorded on a Bio Rad FTS-175C spectrometer.

Analytical high performance liquid chromatography (HPLC) was performed using a diode array detector (deuterium lamp, 190-600 nm) using a Daicel Chiralpak AD column (25x 0.46 cm) (Daicel chemical Ind., Ltd.) on a Perkin Elmer 235C instrument, with HPLC-grade isopropanol and hexanes as the eluting solvents.

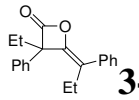
[1] Armarego, W. L. F.; Perrin, D. D. Purification of Laboratory Chemicals, 4th ed., Butterworth Heinemann, 2002.
[2] Hodous, B. L.; Fu, G. C. *J. Am. Chem. Soc.* **2002**, *124*, 10006-10007.
[3] Wiskur, S. L.; Fu, G. C. *J. Am. Chem. Soc.* **2005**, *127*, 6176-6177.
[4] Allen, A. D.; Baigrie, L. M.; Gong, L.; Tidwell, T. T. *Can. J. Chem.* **1991**, 69, 138-145.

Method A for dimerization of ketoketenes: Ketoketene (1.39 mmol, 1.0 equiv.) was dissolved in CH_2Cl_2 (1.4 mL). LiI (0.42 mmol, 0.3 equiv.) was dissolved in Et_2O (1.4 mL) and was then transferred to the flask containing the ketoketene solution and the resulting solution (0.5 M of ketoketene in solvent) was cooled to 0℃. Tri-*n*-butylphosphine (0.14 mmol, 0.1 equiv.) was added in one portion, and stirred for the indicated time at the indicated temperature. The reaction was then diluted with CH_2Cl_2 (5 mL) and quenched by adding deionized water (10 mL). The layers were separated, the aqueous layer was extracted with CH_2Cl_2 (2 × 5 mL), and the combined organics were dried over anhydrous sodium sulphate. The solvent was removed under reduced pressure to provide the crude product for ^{1}H NMR/GCMS analysis. 10% EtOAc/hexane (20 mL) and dichloromethane (5 mL) were added to the crude residue, which was passed through a plug column of neutral silica (iatrobeads, 2 × 2 cm, 10 g) and was eluted with 10% EtOAc/Hexane (100 mL). Finally solvent was removed under reduced pressure to yield the desired ketoketene dimer in high purity (≥95%), in most cases, as determined by GCMS and HPLC analysis, and confirmed by ^{1}H and ^{13}C NMR spectroscopy.

Method B for dimerization of ketoketenes: To a solution of ketoketene in CH_2Cl_2 (0.5 M), the indicated phosphine (with indicated equivalents) was added at 0℃ or -78℃. The reaction was stirred at the indicated temperature for the indicated time. The reaction was typically opened to air and stirred for 10 minutes before the solvent was removed under reduced pressure, and the crude product was purified as detailed below.

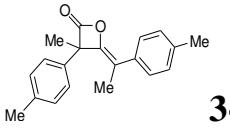


3-Methyl-3-phenyl-4-(1-phenyl-ethylidene)-oxetan-2-one (4a): (Method A) Methylphenylketene (197 mg, 1.48 mmol), stirred for 1 hour at 0℃, and was isolated as a colorless oil (152 mg, 78%). The *Z*:*E* ratio was determined to be >16:1 by ^{1}H NMR analysis; IR (thin film) 1881, 1844, 1699, 1140 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.52-7.16 (m, 10H), 1.92 (s, 3H), 1.86 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 171.4, 146.9, 136.2, 135.2, 129.4, 128.6, 128.6, 127.6, 127.4, 126.3, 108.6, 64.4, 19.6, 15.6; MS (EI 70 eV): m/z 264, 132, 104, 78; (M^++Na) HRMS m/z calcd for $C_{18}H_{16}O_2Na$: 287.1043; found: 287.1039.

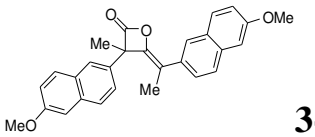


3-Ethyl-3-phenyl-4-(1-phenyl-propylidene)-oxetan-2-one (4b): (Method A) Ethylphenylketene (709 mg, 4.85 mmol)**,** stirred for 4 hours at 0℃, was isolated as a colorless oil (546 mg, 77%). The *Z*:*E* ratio was determined to be 37:1 by HPLC analysis; IR (thin film): 1857, 1699, 1140cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.42-7.20 (m, 10H), 2.38-2.02 (m, 4H), 1.15 (t, *J* = 7.4 Hz, 3H), 0.83 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 170.9, 143.9, 135.4, 134.7, 129.3, 128.6, 128.2, 127.6, 126.5, 116.5, 70.1, 26.3, 22.9, 12.8, 10.0; (EI 70 eV): m/z 292, 146, 117, 103, 91, 77; MS (M^++Na) HRMS m/z calcd for $C_{20}H_{20}O_2Na$: 315.1355; found: 315.1351.

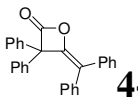
(-)-3-Ethyl-3-phenyl-4-(1-phenyl-propylidene)-oxetan-2-one ((-)-4b): (Method B): Ethylphenylketene (57 mg, 0.39 mmol) was dissolved in CH_2Cl_2 (0.35 mL) and was cooled to -78 ℃. (*R*,*R*)-Binaphane (27 mg, 0.04 mmol) was dissolved in CH_2Cl_2 (0.35 mL) and cooled to -78 ℃. The phosphepine solution was then transferred via syringe to the flask containing the ketoketene solution. The reaction was stirred at -78℃ for 48 hours, after which the solvent was removed under reduced pressure. The crude product was treated with dry isopropanol (3 mL) to dissolve the crude ketoketene dimer and precipitate the phosphepine. The mixture was filtered under nitrogen using a Schlenk filter stick. The precipitate was washed with dry isopropanol (2 × 3 mL), the phosphine was recovered, and the solvent was removed from the filtrate under reduced pressure. The crude product was then dissolved in 5% EtOAc/hexane (6 mL) and CH_2Cl_2 (1.5 mL), before being passed through a plug of neutral silica (3.3 g). Elution with 5% EtOAc/hexane (60 mL), followed by solvent removal under reduced pressure yielded a colorless oil (36 mg, 65%). The *Z*:*E* ratio was determined to be >37:1 by HPLC analysis. HPLC analysis of the purified compound on a Daicel Chiralpak AD column (25 x 0.46 cm), eluted with hexane:isopropanol (98:2), showed that (-)-**4b** was afforded in 80% e.e. (R_{t1} = 4.1 min, R_{t2} = 4.9 min with a ratio 9.5: 83.2); $[\alpha]_D$=-25.2 °; IR (thin film): 1857, 1699, 1140cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.42- 7.20 (m, 10H), 2.38-2.02 (m, 4H), 1.15 (t, *J* = 7.4 Hz, 3H), 0.83 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 170.9, 143.9, 135.4, 134.7, 129.3, 128.6, 128.2, 127.6, 126.5, 116.5, 70.1, 26.3, 22.9, 12.8, 10.0; (EI 70 eV): m/z 292, 146, 117, 103, 91, 77; MS (M^++Na) HRMS m/z calcd for $C_{20}H_{20}O_2Na$: 315.1355; found: 315.1351.



3-Methyl-3-p-tolyl-4-(1-p-tolyl-ethylidene)-oxetan-2-one (4c): (Method A) Methyl-*p*-tolylketene (552 mg, 3.78 mmol)**,** stirred for 3 hours at 0 ℃, and was isolated as a colorless oil (498 mg, 90%). The *Z*:*E* ratio was determined to be 39:1 by HPLC analysis; IR (thin film): 1880, 1845, 1698, 1140 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.24-7.09 (m, 8H), 2.27 (s, 6H), 1.87 (s, 3H), 1.81 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 171.7, 146.6, 138.4, 137.3, 133.3, 132.3, 130.0, 129.2, 127.2, 126.1, 108.3, 64.1, 21.3, 21.2, 19.5, 15.5; MS (EI 70 eV) m/z 292, 146, 118, 91; (M^++Na) HRMS m/z calcd for $C_{20}H_{20}O_2Na$: 315.1355; found: 315.1354.

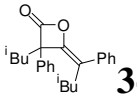


3-(6-Methoxy-naphthalen-2-yl)-4-[1-(6-methoxy-naphthalen-2-yl)-ethylidene]-3-methyl-oxetan-2-one (4d): (Method A) 6-Methoxy-naphthylmethyl ketene (266 mg, 1.25 mmol), was stirred for 2 hours at 0 ℃, and isolated as an off-white solid (164 mg, 62%). The Z:E ratio was determined to be >16:1 by HPLC analysis; mp 179-184 °C; IR (thin film): 1880, 1699cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.91-7.57 (m, 9H), 7.19-7.12 (m, 3H), 3.91 (s, 3H), 3.90 (s, 3H), 2.10 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 171.7, 158.4, 158.2, 147.1, 134.4, 133.9, 131.6, 130.3, 129.9, 129.9, 128.9, 128.1, 126.9, 126.1, 125.9, 125.1, 124.5, 119.7, 119.3, 108.6, 105.7, 64.6, 55.5, 29.9, 19.4, 15.5, 14.4; MS (EI 70 eV) m/z 424, 378, 212, 184; (M^++Na) HRMS m/z calcd for $C_{28}H_{24}O_4Na$: 447.1567; found: 447.1564.

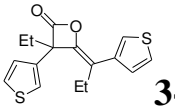


4-Benzhydrylidene-3,3-diphenyl-oxetan-2-one (4e): (Method B) Diphenylketene (269 mg, 1.38 mmol) and Me_3P (1.0 M in toluene, 277 μL, 0.28 mmol), stirred for 3 days at room temperature. The crude product was triturated with isopropanol to afford an off-white solid (210 mg, 78%); mp 140-144 °C; IR (thin film): 1859, 1671, 1167 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.25-6.68 (m, 20H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 169.8, 148.0, 136.8, 135.9, 130.8, 129.5, 128.9, 128.6, 128.4, 128.2, 128.0, 127.9, 126.5, 118.7, 74.5; MS (EI 70 eV): m/z

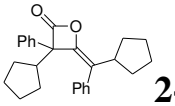
388, 194, 165, 105; (M^++Na) HRMS m/z calcd for $C_{28}H_{20}O_2Na$: 411.1355; found: 411.1346.



3-Isobutyl-4-(3-methyl-1-phenyl-butylidene)-3-phenyl-oxetan-2-one (4f): (Method B) Isobutylphenylketene (185 mg, 1.06 mmol) and tri-*n*-butylphosphine (27 μL, 0.11 mmol), stirred for 4 hours at room temperature, purified through a plug of iatrobeads eluted with 10% EtOAc/hexane, solvent was removed under reduced pressure, was isolated as a colorless oil (155 mg, 84%) which was composed of an inseparable mixture of **4f**:2,4-isobutyl-2,4-phenylcyclobutanedione isomer in the ratio 7:1. The *Z*:*E* ratio of **4f** was determined to be 28:1 by GCMS analysis; IR (thin film): 1870, 1699, 1190 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, TMS): δ 7.52-7.22 (m, 10H), 2.33-1.96 (m, 4H), 1.62-1.41 (m, 2H), 1.07 (d, *J* = 3.8 Hz, 3H), 1.04 (d, *J* = 3.8 Hz, 3H), 0.77 (d, *J* = 6.6 Hz, 3H), 0.45 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 171.5, 136.2, 135.2, 129.1, 128.4, 128.3, 127.5, 126.5, 126.3, 114.6, 68.2, 41.5, 38.1, 26.2, 25.8, 24.4, 23.6, 22.9, 21.6; MS (EI 70 eV): m/z 174, 131, 103; (M^++Na) HRMS calcd for $C_{24}H_{28}O_2Na$: 371.1981; found: 371.1980.

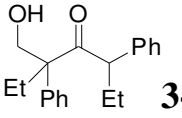


3-Ethyl-3-thiophen-3-yl-4-(1-thiophen-3-yl-propylidene)-oxetan-2-one (4g): (Method A) Ethyl(2-thiophenyl)ketene (162 mg, 1.06 mmol), was stirred for 7 hours at 0 ℃, isolated as a colorless oil (127 mg, 78%). The *Z*:*E* ratio was determined to be 20:1 by HPLC analysis; IR (thin film): 1859, 1695, 1411 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, TMS) δ 7.30-7.16 (m, 4H), 7.05-7.02 (m, 2H), 2.39-2.01 (m, 4H), 1.09 (t, *J* = 7.3 Hz, 3H), 0.92 (m, *J* = 7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 170.1, 144.5, 136, 135.4, 127.4, 127.1, 126.1, 125.5, 122.6, 122.4, 111.3, 67.5, 26.1, 22.6, 13.5, 9.7; MS (EI 70 eV): m/z 152, 123, 97; (M^++Na) HRMS m/z calcd for $C_{16}H_{16}O_2S_2Na$: 327.0484; found: 327.0485.



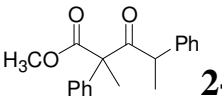
2-Cyclopentyl-3-(cyclopentyl-phenyl-methylene)-2-phenyl-cyclobutanone (4h): (Method B) Cyclopentylphenylketene (223 mg, 1.20 mmol),

Me_3P (1.0 M in toluene, 240 μL, 0.24 mmol), stirred for 2 days at room temperature, purified by column chromatography over neutral silica (iatrobeads) with gradient elution 0.5-0.75% EtOAc/hexane, and the solvent was removed under reduced pressure to yield **4h** as a colorless oil (135 mg, 60%), isolated as an inseparable mixture of diastereomers. The *Z*:*E* ratio was determined to be 3:1 by GCMS analysis; IR (thin film): 1850, 1702, 1118, 1034 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, TMS) for the mixture of diastereomers: δ 7.48-7.02 (m, 10H), 2.95-2.13 (m, 2H), 2.07-0.80 (m, 10H); ^{13}C NMR (50 MHz, $CDCl_3$) for major diastereomer: δ 170.7, 145.7, 135.9, 134.2, 130.3, 129.1, 128.8, 128.6, 128.2, 127.7, 127.6, 126.9, 118.5, 71.3, 43.0, 41.2, 30.8, 30.5, 29.7, 28.6, 25.7, 25.6, 25.4, 25.1; MS (EI 70 eV): m/z 186, 129, 115, 91, 77; (M^++Na) HRMS m/z calcd for $C_{26}H_{28}O_2Na$: 395.1982; found: 395.1978.

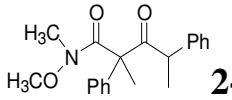


3-Hydroxymethyl-3,5-diphenyl-heptan-4-one (7) : 4b (133.6 mg, 0.46 mmol) was dissolved in THF (0.92 mL) and the solution (0.5M) was cooled to -78℃. After five minutes, $LiAlH_4$ (1M in Et_2O, 0.46 mL, 0.46 mmol) was added at -78℃. The reaction was stirred for 50 min until TLC showed complete consumption of reactant and then the reaction was quenched with MeOH (0.3 mL) at -78℃. The reaction was worked up with CH_2Cl_2 (10 mL) and HCl (1M, 10 mL). Aqueous saturated NaCl solution was added to aid separation of the phases. The aqueous phase was extracted with CH_2Cl_2 (3 × 4 mL), and the combined organic layers were dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure. The resulting crude product was purified on a plug of neutral silica (iatrobeads) using a gradient of solvents from 10% EtOAc/hexane to 20% EtOAc/hexane to afford **7** as a white solid (98.3 mg, 74%), isolated as an inseparable mixture of diastereomers, with a d.r. = 1:1 as determined by GCMS analysis; IR (thin film): 3460, 1697, 1030 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, TMS) for the mixture of diastereomers: δ 6.83-7.21 (m, 10H), 4.22 (d, *J* = 11.8 Hz, 1H), 3.83 (d, *J* = 11.8 Hz, 1H), 3.51 (dd, *J* = 6.6, 8.4 Hz, 1H), 2.24-2.15 (m, 2H), 1.8 - 1.71 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H), 0.59 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) for the mixture of diastereomers: δ 213.4, 139.4, 137.9, 128.8, 128.5, 128.0, 127.9, 127.5, 126.8, 63.7, 61.8, 56.1, 28.5, 23.5, 12.1, 8.6; MS (EI 70 eV):

m/z 266, 147, 119, 91, 77; (M^++Na) HRMS m/z calcd for $C_{20}H_{24}O_2Na$: 319.1669; found: 319.1662.

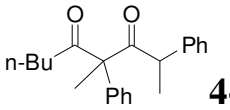


2-Methyl-3-oxo-2,4-diphenyl-pentanoic acid methyl ester (8): *n*-BuLi (2.5 M in hexane, 0.2ml, 0.5mmol) was added to a stirred solution of MeOH (2.06 mmol, 83 μL) in THF (1.85 mL) at 0 ℃ and stirred for 10 min. Compound **4a** (136.0 mg, 0.51 mmol) was dissolved in THF (5 mL) and the solution was now added dropwise to the solution of MeOLi at 0 ℃. The reaction was stirred for 85 min until TLC showed complete consumption of reactant. Then the reaction was quenched with HCl (1M, 10 mL) and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 × 10 mL), and the combined organics were dried over Na_2SO_4. The solvent was removed under reduced pressure. The resulting product **8** was obtained quantitatively as a colorless oil, isolated as an inseparable mixture of diastereomers, with a d.r. = 1.6:1.0 as determined by GCMS analysis; IR (thin film): 1716, 1251, 1208 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, TMS) for the mixture of diastereomers: δ 7.26-6.99 (m, 8H), 6.83-6.78 (m, 2H), 3.95 (q, *J* = 7 Hz, 1H), 3.85 (q, *J* = 6.8 Hz, 1H), 3.71 (s, 3H), 3.42 (s, 3H), 1.66 (s, 3H), 1.61 (s, 3H), 1.31 (d, *J* = 7.0 Hz, 3H), 1.24 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) for the mixture of diastereomers: δ 208.2, 207.5, 172.9, 172.3, 141.4, 138.3, 137.5, 128.6, 128.5, 128.1, 128.0, 128.0, 127.5, 127.0, 126.7, 65.5, 65.4, 52.7, 52.5, 49.7, 49.5, 29.9, 22.4, 22.2, 21.9, 21.5; MS (EI 70 eV): m/z 296, 164, 132, 105, 77; (M^++Na) HRMS m/z calcd for $C_{19}H_{20}O_3Na$: 319.1305; found: 319.1298.

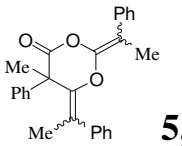


2-Methyl-3-oxo-2,4-diphenyl-pentanoic acid methoxy-methyl-amide (9): 4a (117.7 mg, 0.45 mmol) was dissolved in CH_2Cl_2 (2.23 ml, 0.2 M). After a few minutes the Weinreb amine (0.89 mmol, 65 μl) and 2-Hydroxypyridine (0.05 mmol, 4.2 mg) were added. The reaction was stirred for 4 days until TLC showed complete consumption of reactant and then the reaction was quenched with H_2O (10 mL). The layers were separated and the aqueous phase was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure. The resulting crude product was purified by flash column chromatography on neutral

silica (iatrobeads, 12.5 g) using a gradient of solvents from 5% EtOAc/hexane to 15% EtOAc/hexane to afford **9** as a solidifying white oil (106.1 mg, 90%), isolated as an inseparable mixture of diastereomers, with a d.r. = 1.4:1.0 as determined by GCMS analysis; IR (thin film): 1714, 1652, 1180 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, TMS) for the mixture of diastereomers: δ 7.31-6.86 (m, 10H), 4.02-3.89 (m, 1H), 3.16 (s, 3H), 3.06 (s, 1H), 3.04 (s, 1H), 2.97 (s, 1H), 1.71 (s, 3H), 1.53 (s, 3H), 1.32 (d, *J* = 6.8 Hz, 3H), 1.30 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) for the mixture of diastereomers: δ 207.4, 206.9, 141.7, 141.2, 139.6, 138.6, 128.7, 128.4, 128.3, 128.1, 128.1, 127.8, 127.6, 127.2, 127.0, 126.6, 64.5, 64.2, 59.9, 59.6, 50.1, 49.3, 33.6, 23.1, 21.9, 21.7, 21.5; MS (EI 70 eV): m/z 325, 193, 162, 132, 105, 77; (M^++Na) HRMS m/z calcd for $C_{20}H_{23}NO_3Na$: 348.1570; found: 348.1564.

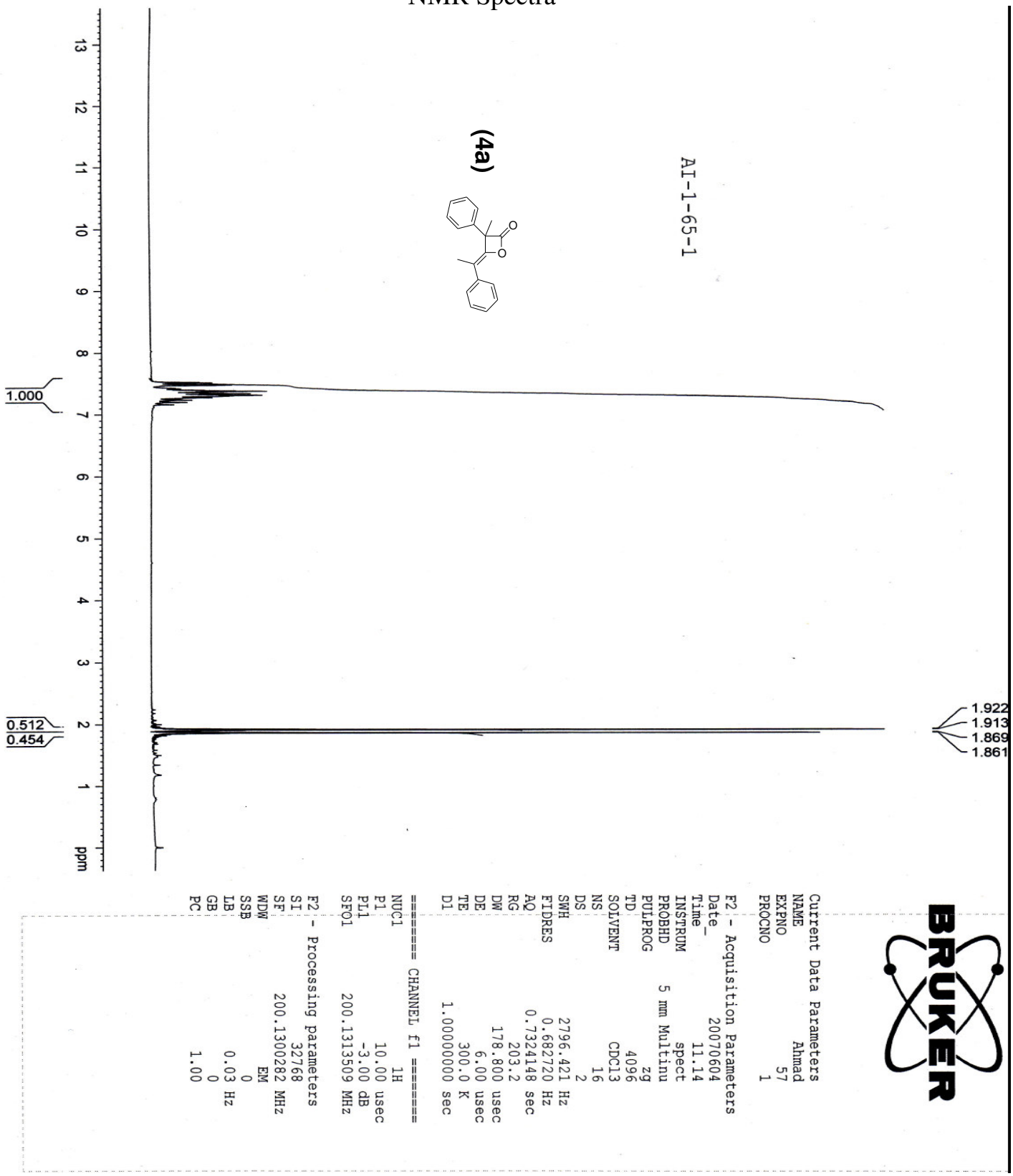


4-Methyl-2,4-diphenyl-nonane-3,5-dione (10): 4a (160 mg, 0.61 mmol) was dissolved in THF (4.8 mL), and *n*-BuLi (2.5 M in hexane, 0.48 mL, 1.2 mmol) was added dropwise over five minutes at -78 ℃, stirred for 15 min and was quenched by adding water (2 mL) at -78 ℃. The reaction was then warmed up to room temperature, brine (8 mL) and CH_2Cl_2 (5 mL) were added, and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 5 mL), the combined organics were dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure to yield a colorless oil (148 mg, 93%), isolated as an inseparable mixture of diastereomers, with a d.r. = 8:1 by GCMS analysis; IR (thin film): 1712 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$,TMS) for the major diastereomer: δ 7.29-6.74 (m, 10H), 3.88 (q, *J* = 6.8 Hz, 1H), 2.47-2.15 (m, 2H), 1.56 (s, 3H), 1.51-1.39 (m, 2H), 1.31 (d, *J* = 6.8 Hz, 3H), 1.24-1.10 (m, 2H), 0.77 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) for the major diastereomer: δ 210.8, 210.7, 141.0, 137.7, 128.9, 129.2, 128.7, 128.6, 128.1, 128.0, 127.0, 126.4, 70.6, 49.8, 39.3, 26.3, 22.4, 21.3, 19.9, 14.7; MS (EI 70 eV): m/z 322, 238, 190, 132, 105, 77; (M^++Na) HRMS m/z calcd for $C_{22}H_{26}O_2Na$: 345.1825; found: 345.1819.



5,5-Dimethyl-2,6-bis-(1-phenyl-ethylidene)-[1,3]dioxan-4-one(5a): ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 7.44-6.92 (m, 15H), 2.02 (s, 3H), 1.83 (s, 3H), 1.36 (s, 3H); selected signals from ^{13}C NMR (50 MHz, $CDCl_3$): δ 168.3, 144.4, 143.7, 141.6, 139.6, 137.3, 136.2, 135.2, 128.4, 128.1, 127.6, 127.2, 126.6, 120.6, 98.1, 54.2, 25.0, 19.3, 15.0; MS (EI 70 eV): m/z 396, 221, 132, 104.

NMR Spectra


AI-1-65-1
(4a)
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0.454
1.922
1.913
1.869
1.861
ppm
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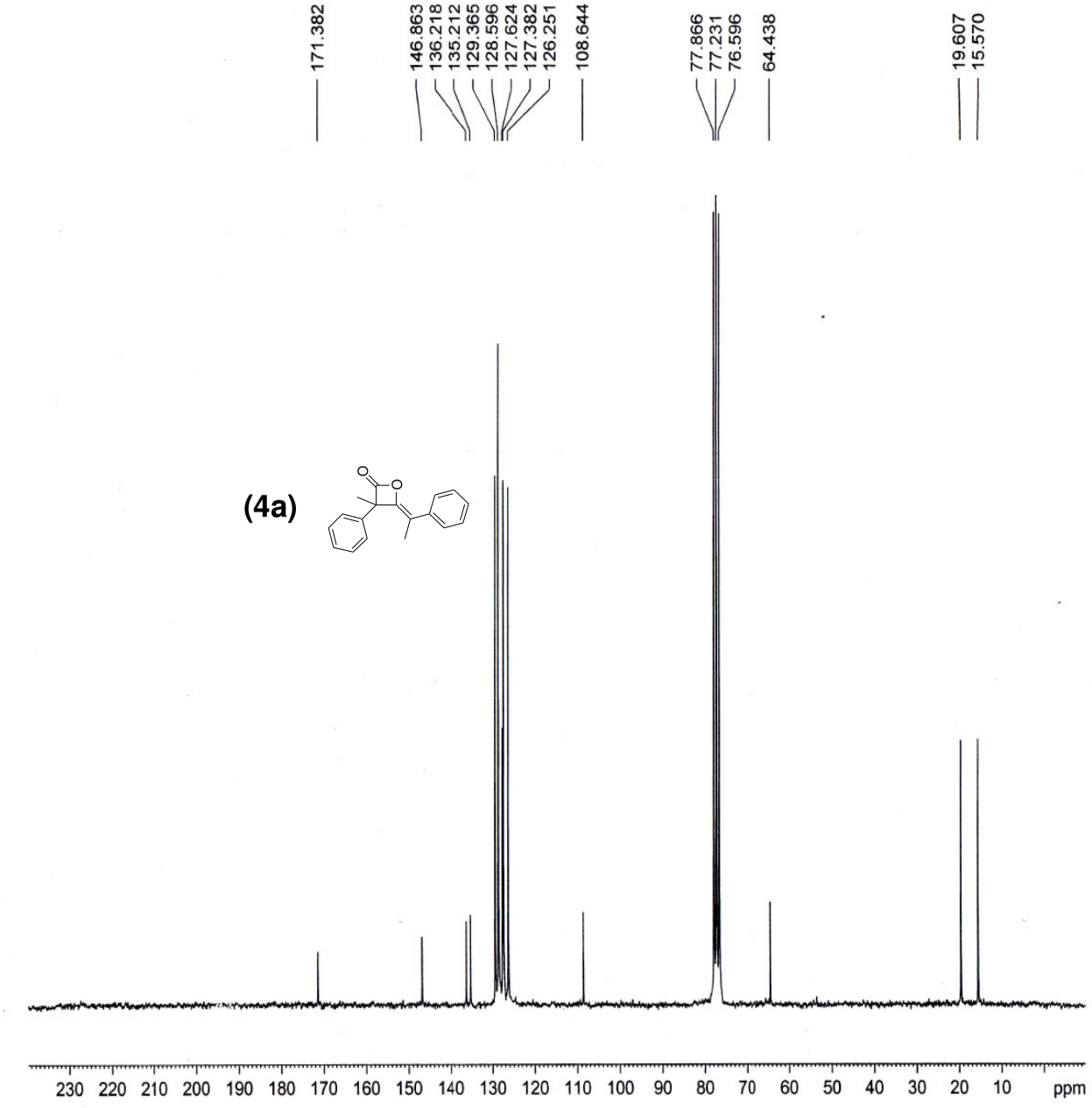
EXPNO 57
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070604
Time_ 11.14
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 4096
SOLVENT CDCl3
NS 16
DS 2
SWH 2796.421 Hz
FIDRES 0.682720 Hz
AQ 0.7324148 sec
RG 203.2
DW 178.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -3.00 dB
SFO1 200.1313509 MHz
F2 - Processing parameters
SI 32768
SF 200.1300282 MHz
WDW EM
SSB 0
LB 0.03 Hz
GB 0
PC 1.00

AI-1-65-1



BRUKER

Current Data Parameters

NAME	Ahmad
EXPNO	72
PROCNO	1

F2 - Acquisition Parameters

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Time	8.00
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	37686
SOLVENT	CDC13
NS	9500
DS	4
SWH	12562.814 Hz
FIDRES	0.333355 Hz
AQ	1.4999528 sec
RG	90.5
DW	39.800 usec
DE	6.00 usec
TE	300.0 K
D1	4.00000000 sec
d11	0.03000000 sec

======== CHANNEL f1 ========

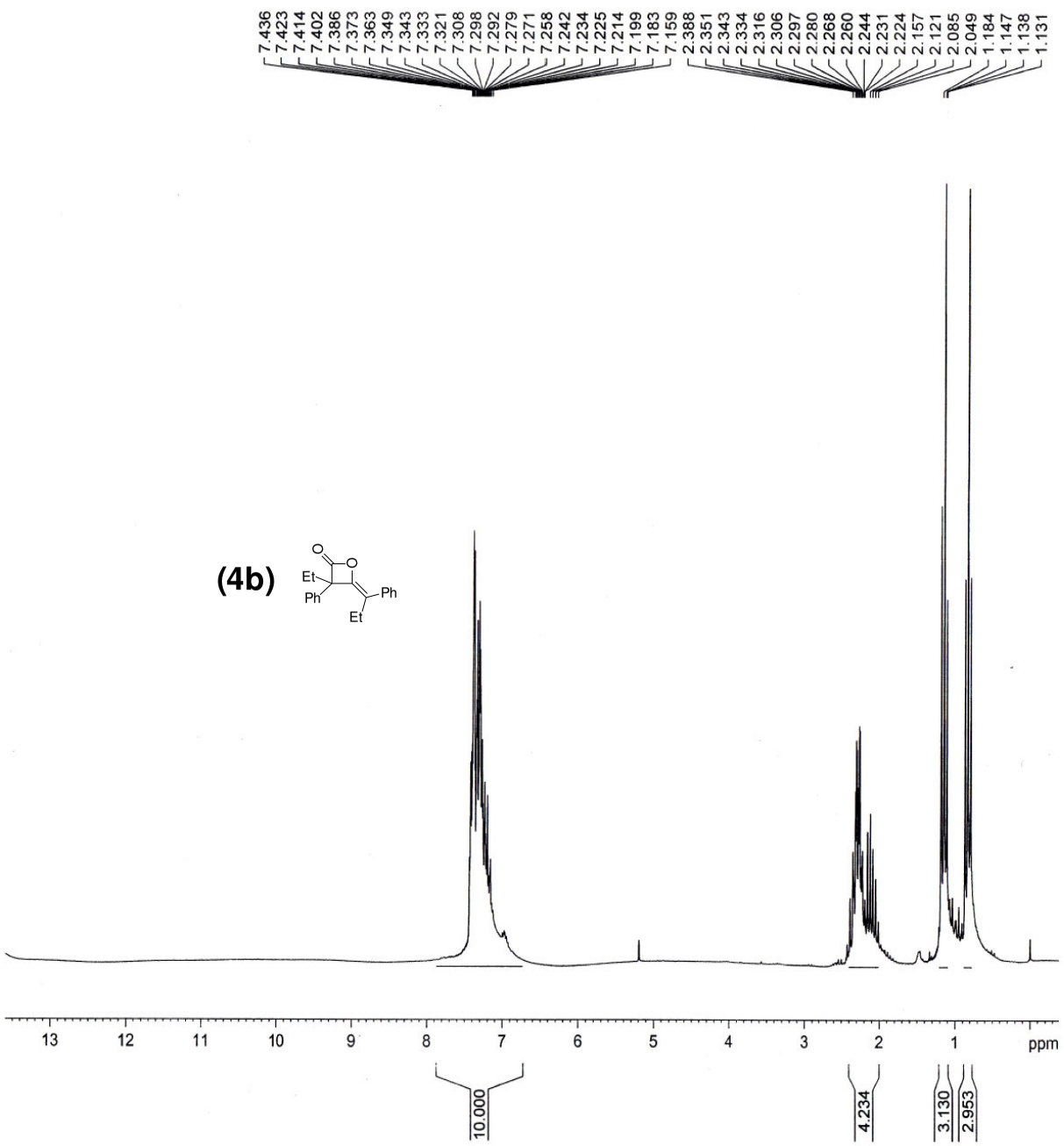
NUC1	13C
P1	6.00 usec
PL1	1.00 dB
SFO1	50.3285046 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-4.00 dB
PL12	16.72 dB
SFO2	200.1300000 MHz

F2 - Processing parameters

SI	32768
SF	50.3227316 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



BRUKER

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Current Data Parameters
NAME          G Harzmann
EXPNO                 68
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080319
Time               20.17
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG              zg
TD                 4096
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            2796.421 Hz
FIDRES         0.682720 Hz
AQ             0.7324148 sec
RG                812.7
DW                178.800 usec
DE                  6.00 usec
TE                300.0 K
D1             3.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1                -3.00 dB
SFO1        200.1313509 MHz

F2 - Processing parameters
SI                 32768
SF          200.1300287 MHz
WDW                   EM
SSB                    0
LB                  0.03 Hz
GB                     0
PC                  1.00
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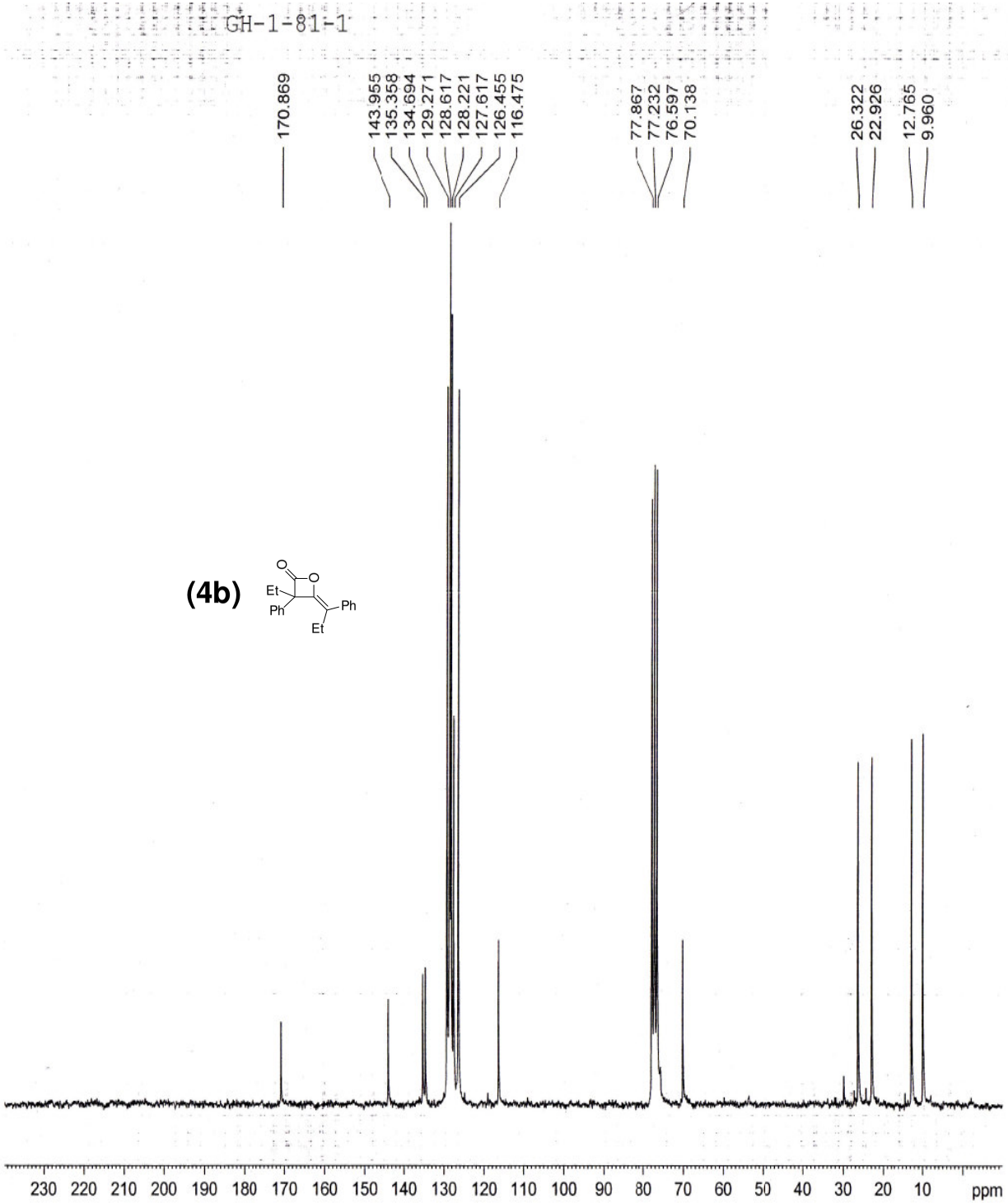

BRUKER

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Current Data Parameters
NAME          G Harzmann
EXPNO                 69
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080320
Time               10.53
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG            zgdc
TD                37686
SOLVENT           CDCl3
NS                 9500
DS                    4
SWH           12562.814 Hz
FIDRES         0.333355 Hz
AQ             1.4999528 sec
RG                 90.5
DW                39.800 usec
DE                 6.00 usec
TE                300.0 K
D1             4.00000000 sec
d11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 usec
PL1                1.00 dB
SFO1         50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2               -4.00 dB
PL12              16.72 dB
SFO2        200.1300000 MHz

F2 - Processing parameters
SI                32768
SF           50.3227212 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
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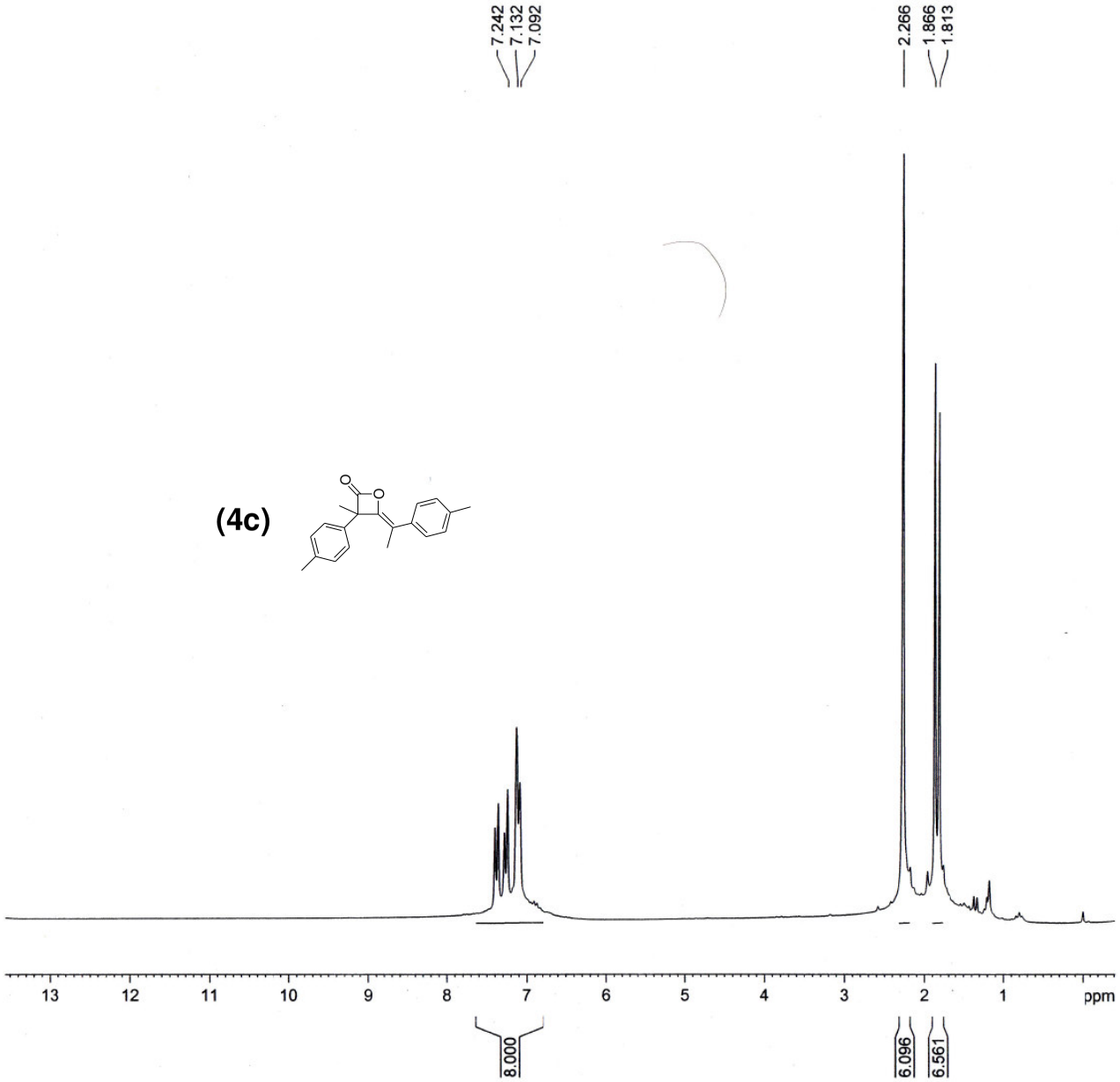
GH1-58-1

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Current Data Parameters
NAME          G Harzmann
EXPNO                 45
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080212
Time               12.58
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG              zg
TD                 4096
SOLVENT           CDCl3
NS                   16
DS                    2
SWH             2796.421 Hz
FIDRES          0.682720 Hz
AQ              0.7324148 sec
RG                  114
DW                178.800 usec
DE                  6.00 usec
TE                 300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                 10.00 usec
PL1                -3.00 dB
SFO1         200.1313509 MHz

F2 - Processing parameters
SI                 32768
SF           200.1300329 MHz
WDW                  EM
SSB                   0
LB                 0.03 Hz
GB                    0
PC                 1.00
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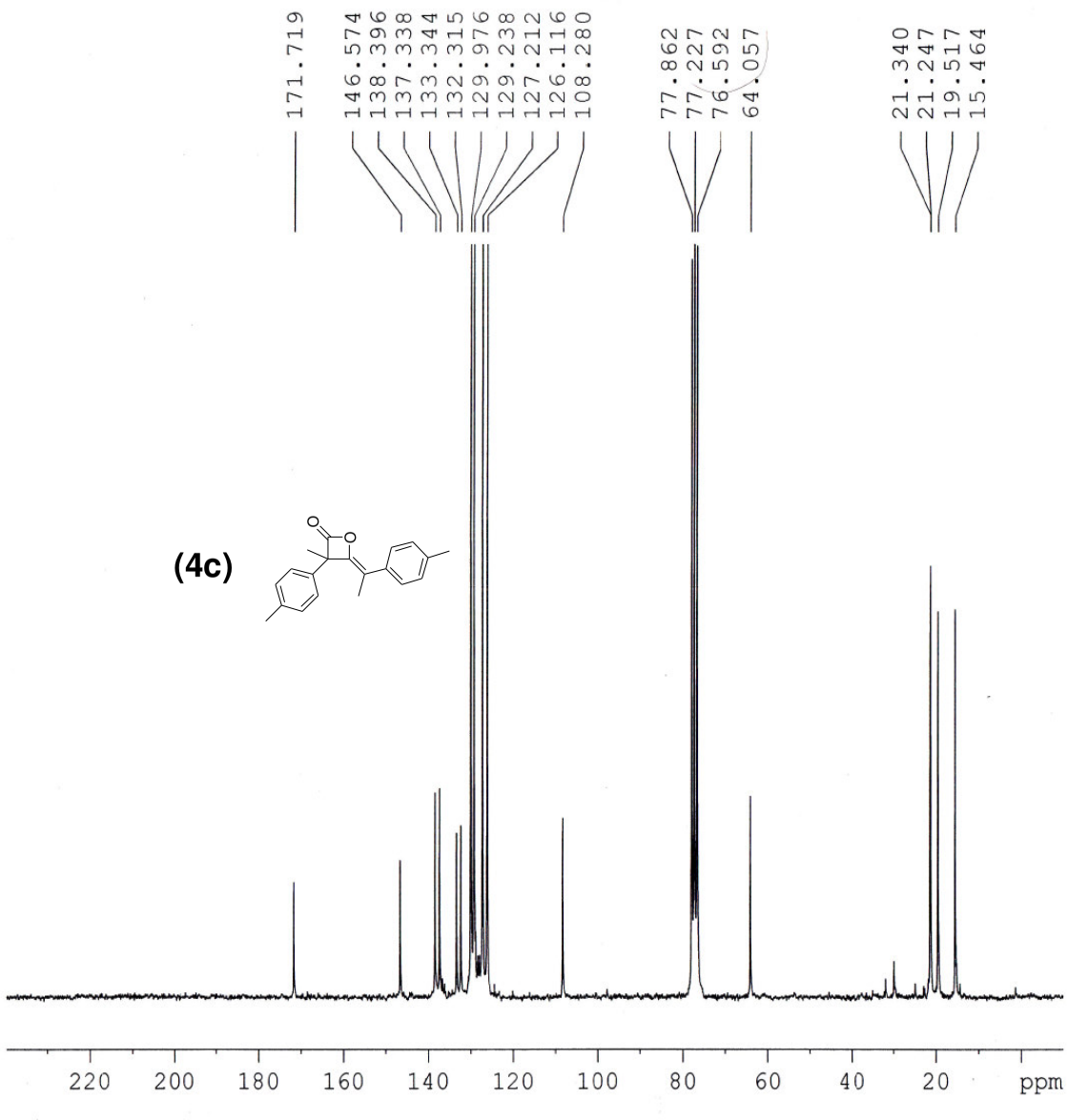
GH1-58-1 (Carbon)

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Current Data Parameters
NAME          G Harzmann
EXPNO                 46
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080212
Time               10.03
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG            zgdc
TD                37686
SOLVENT            DMSO
NS                 9500
DS                    4
SWH           12562.814 Hz
FIDRES         0.333355 Hz
AQ            1.4999528 sec
RG                 90.5
DW                39.800 use
DE                 6.00 use
TE                300.0 K
D1            4.00000000 sec
d11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 use
PL1                1.00 dB
SFO1         50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             100.00 use
PL2                -4.00 dB
PL12               16.72 dB
SFO2        200.1300000 MHz

F2 - Processing parameters
SI                 32768
SF           50.3227235 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40
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nk1-28-1

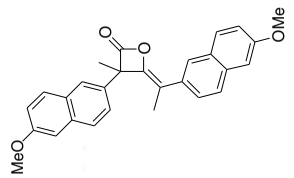
7.910 7.850 7.842 7.795 7.780 7.761 7.719 7.575 7.566 7.190 7.165 7.146 7.125

3.913 3.903 3.895 3.881

2.100 2.091 2.085 2.042 2.035 2.013 2.004

0.007 0.000

(4d)

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Current Data Parameters
NAME          N_Kerrigan
EXPNO                 32
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080302
Time              20.32
INSTRUM           spect
PROBHD     5 mm Multinu
PULPROG             zg
TD                4096
SOLVENT           CDCl3
NS                  16
DS                   2
SWH            2796.421 Hz
FIDRES         0.682720 Hz
AQ             0.7324148 sec
RG                90.5
DW               178.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -3.00 dB
SFO1        200.1313509 MHz

F2 - Processing parameters
SI                32768
SF          200.1300148 MHz
WDW                  EM
SSB                   0
LB                 0.03 Hz
GB                    0
PC                 1.00
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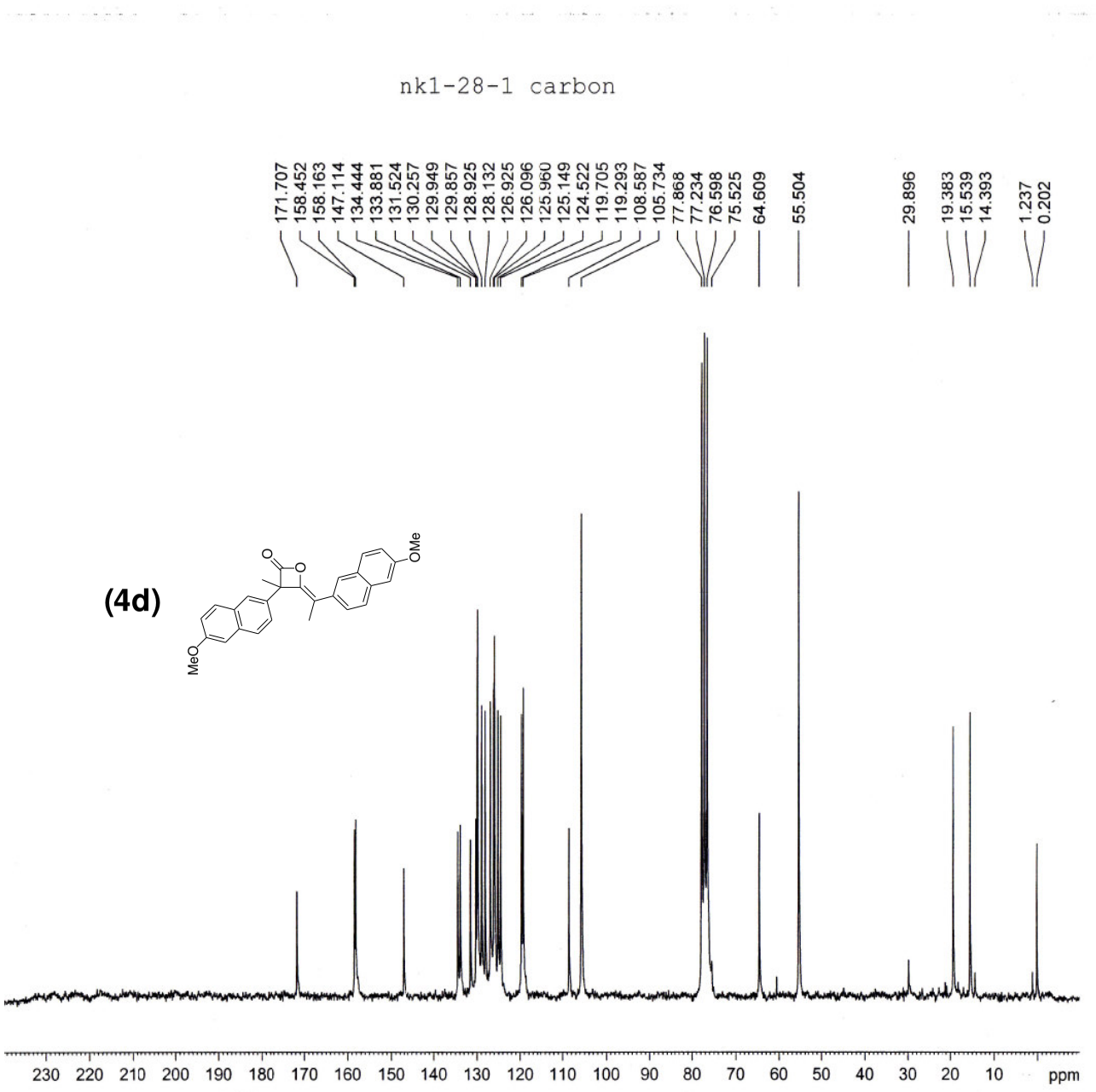


BRUKER

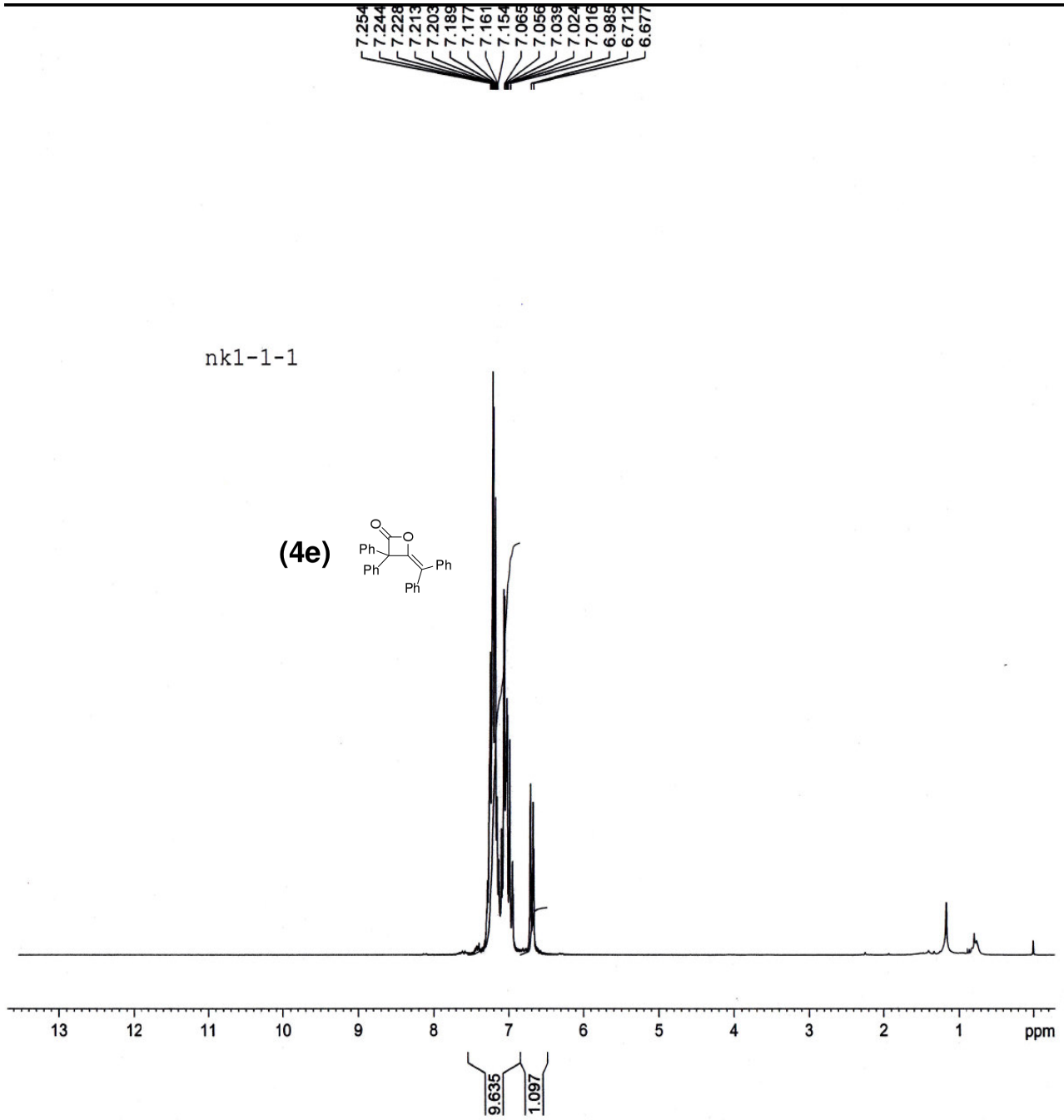
Current Data Parameters
NAME N_Kerrigan
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080303
Time 11.14
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgdc
TD 37686
SOLVENT DMSO
NS 9500
DS 4
SWH 12562.814 Hz
FIDRES 0.333355 Hz
AQ 1.4999528 sec
RG 57
DW 39.800 usec
DE 6.00 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 1.00 dB
SFO1 50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 16.72 dB
SFO2 200.1300000 MHz

F2 - Processing parameters
SI 32768
SF 50.3227220 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



BRUKER

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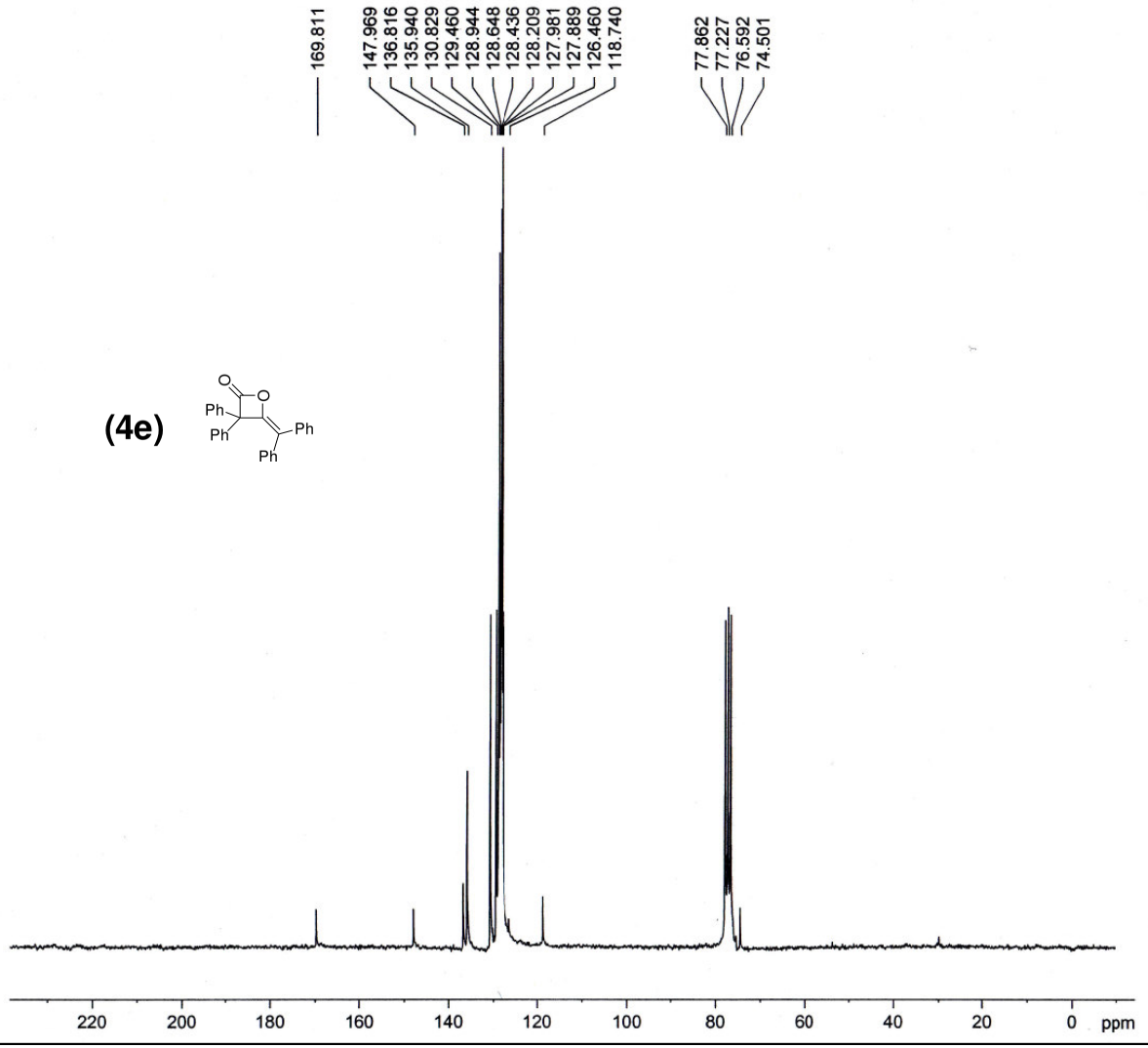
Current Data Parameters
NAME          N_Kerrigan
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061210
Time               19.45
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG              zg
TD                 4096
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            2796.421 Hz
FIDRES         0.682720 Hz
AQ             0.7324148 sec
RG                  90.5
DW                178.800 usec
DE                  6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -3.00 dB
SFO1        200.1313509 MHz

F2 - Processing parameters
SI                 32768
SF          200.1300402 MHz
WDW                   EM
SSB                    0
LB                  0.03 Hz
GB                     0
PC                  1.00
```

diphenylketene dimer



(4e)

BRUKER

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Current Data Parameters
NAME          N-Kerrigan
EXPNO                 19
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080125
Time                9.50
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG            zgdc
TD                37686
SOLVENT            DMSO
NS                 9500
DS                    4
SWH           12562.814 Hz
FIDRES         0.333355 Hz
AQ             1.4999528 sec
RG                 71.8
DW                39.800 usec
DE                 6.00 usec
TE                300.0 K
D1            4.00000000 sec
d11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 usec
PL1                1.00 dB
SFO1          50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               -4.00 dB
PL12              16.72 dB
SFO2         200.1300000 MHz

F2 - Processing parameters
SI                32768
SF           50.3227220 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
```

nk1-20-1

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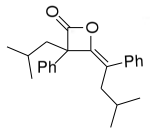
(4f)



BRUKER

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Current Data Parameters
NAME                 N_Kerrigan
EXPNO                        24
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080222
Time                      18.45
INSTRUM                   spect
PROBHD          5 mm Multinu
PULPROG                     zg
TD                        4096
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   2796.421 Hz
FIDRES                0.682720 Hz
AQ                    0.7324148 sec
RG                          181
DW                    178.800 usec
DE                      6.00 usec
TE                      300.0 K
D1                    1.00000000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                      10.00 usec
PL1                      -3.00 dB
SFO1                200.1313509 MHz

F2 - Processing parameters
SI                        32768
SF                  200.1300110 MHz
WDW                          EM
SSB                           0
LB                       0.03 Hz
GB                            0
PC                         1.00
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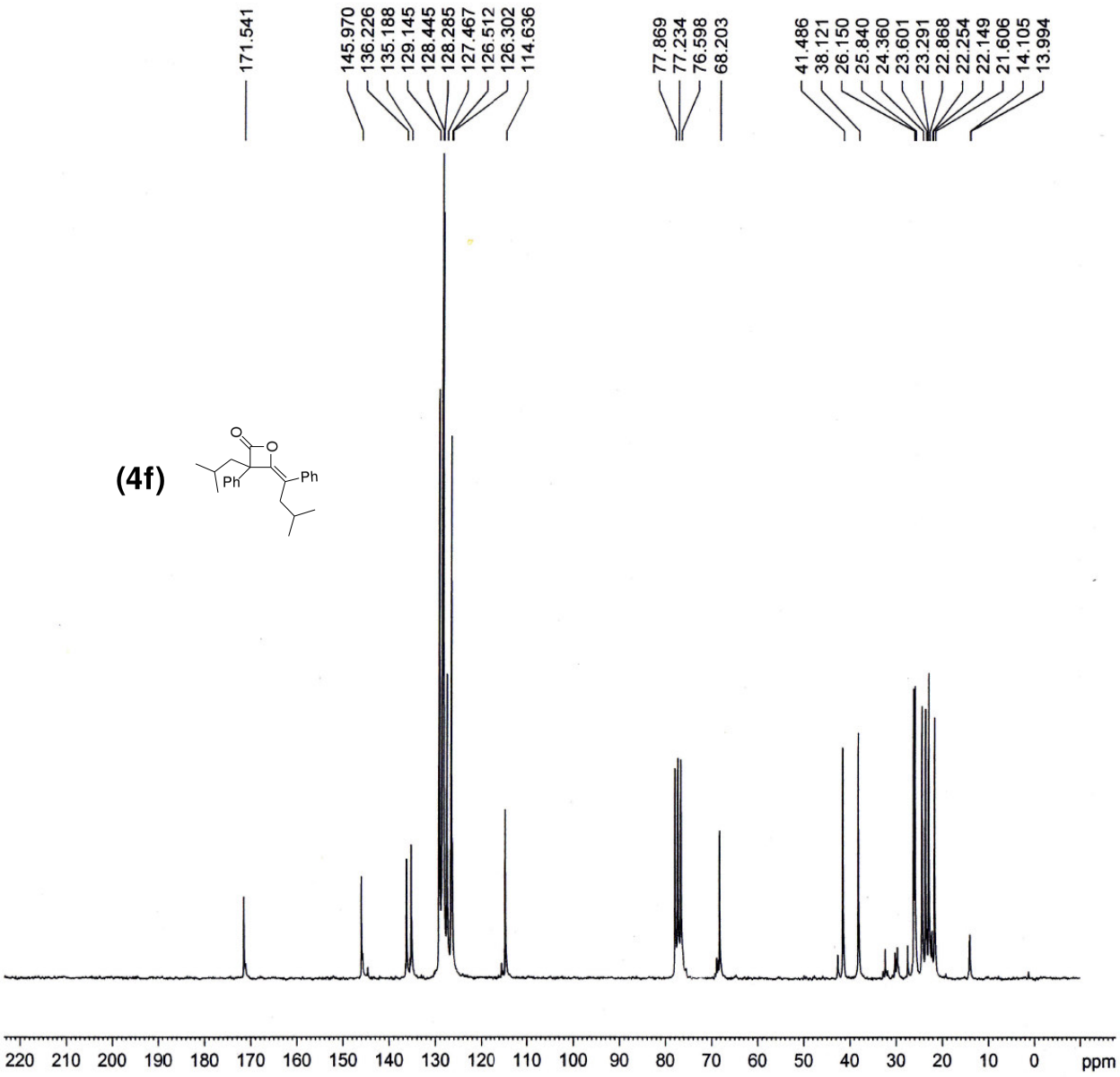
nk1-20-1



(4f)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Current Data Parameters
NAME          N_Kerrigan
EXPNO                 26
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080224
Time              12.00
INSTRUM           spect
PROBHD     5 mm Multinu
PULPROG           zgdc
TD               37686
SOLVENT          CDCl3
NS                9500
DS                   4
SWH           12562.814 Hz
FIDRES         0.333355 Hz
AQ             1.4999528 sec
RG                  57
DW                39.800 usec
DE                 6.00 usec
TE                300.0 K
D1           4.00000000 sec
d11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 usec
PL1                1.00 dB
SFO1         50.3285046 MHz

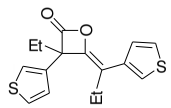
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               -4.00 dB
PL12              16.72 dB
SFO2        200.1300000 MHz

F2 - Processing parameters
SI                32768
SF           50.3227247 MHz
WDW                EM
SSB                 0
LB                3.00 Hz
GB                  0
PC                1.40
```

nk1-41-1pr

7.312 7.297 7.283 7.273 7.265 7.257 7.243 7.234 7.226 7.219 7.211 7.182 7.069 7.061 7.044 7.036 2.370 2.334 2.299 2.291 2.262 2.254 2.247 2.240 2.224 2.218 2.208 2.196 2.182 2.169 2.154 2.146 2.132 2.097 2.061 1.497 1.185 1.142 1.128 1.121 1.114 1.105 1.097 1.090 1.083 1.069 1.054 1.047 1.040 1.034

(4g)

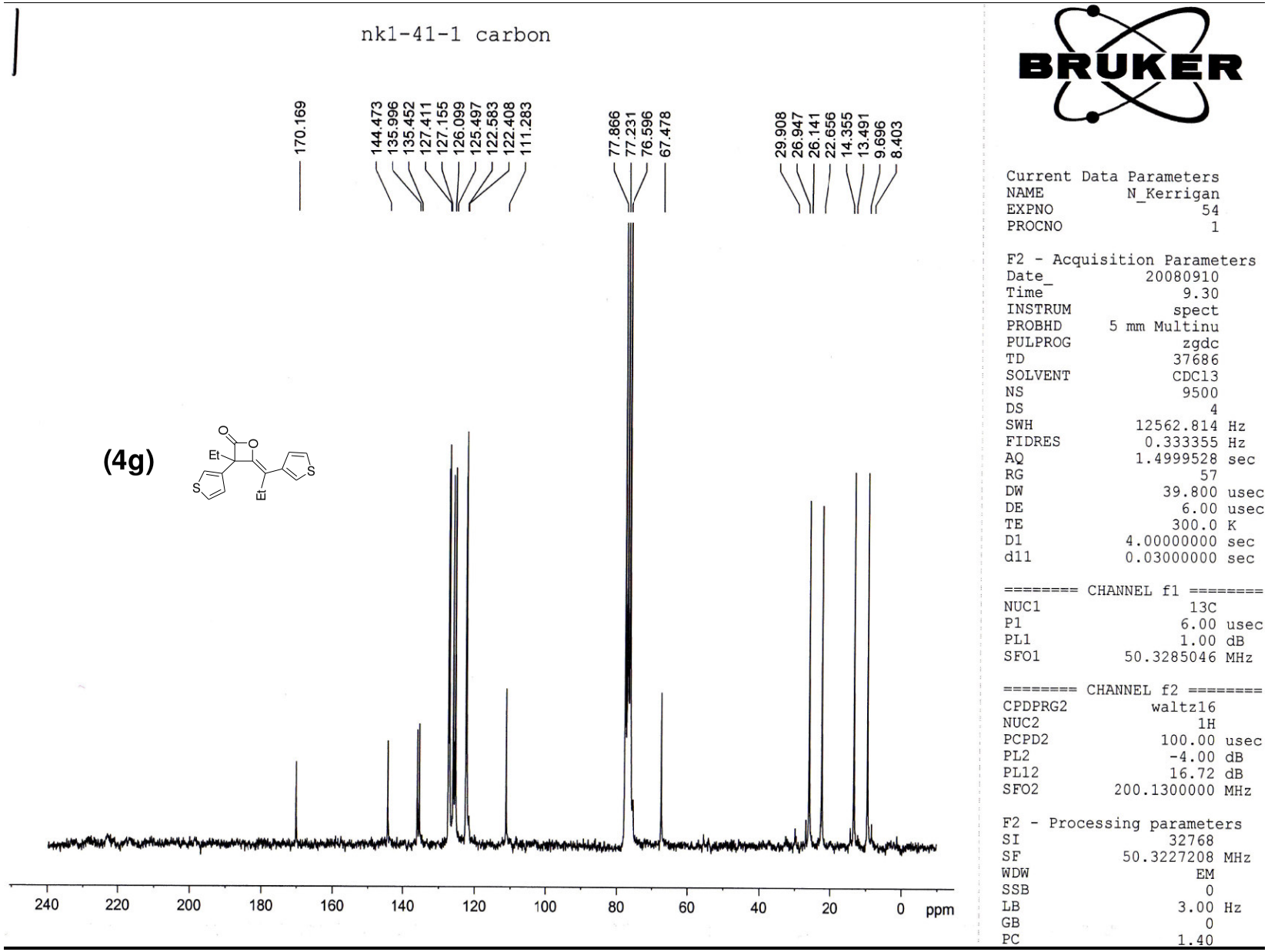



```
Current Data Parameters
NAME          N_Kerrigan
EXPNO                 53
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080909
Time               18.45
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG              zg
TD                 4096
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           2796.421 Hz
FIDRES        0.682720 Hz
AQ            0.7324148 sec
RG                 362
DW              178.800 usec
DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -3.00 dB
SFO1        200.1313509 MHz

F2 - Processing parameters
SI                32768
SF          200.1300241 MHz
WDW                  EM
SSB                   0
LB                 0.03 Hz
GB                    0
PC                 1.00
```



BRUKER

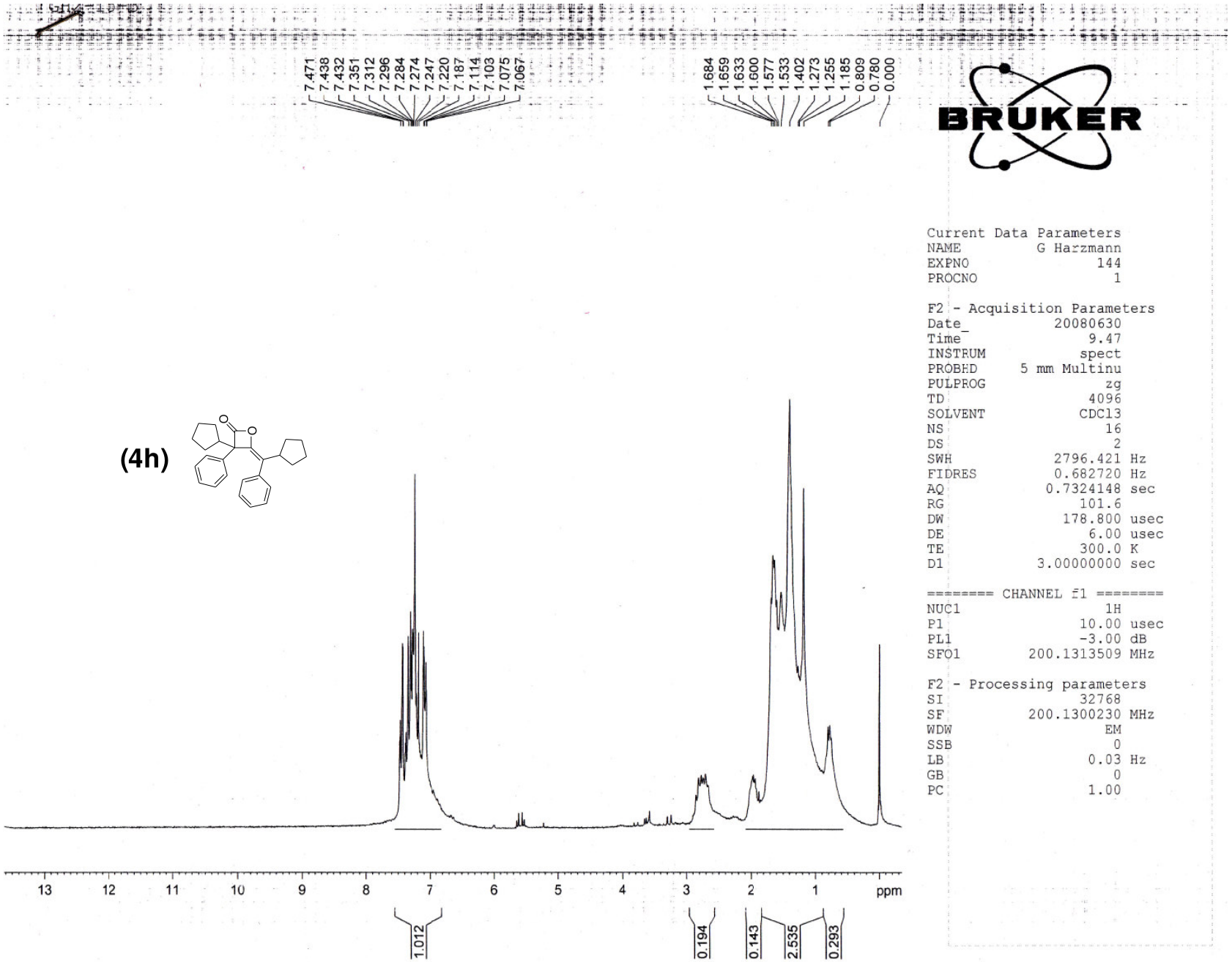
Current Data Parameters
NAME N_Kerrigan
EXPNO 54
PROCNO 1

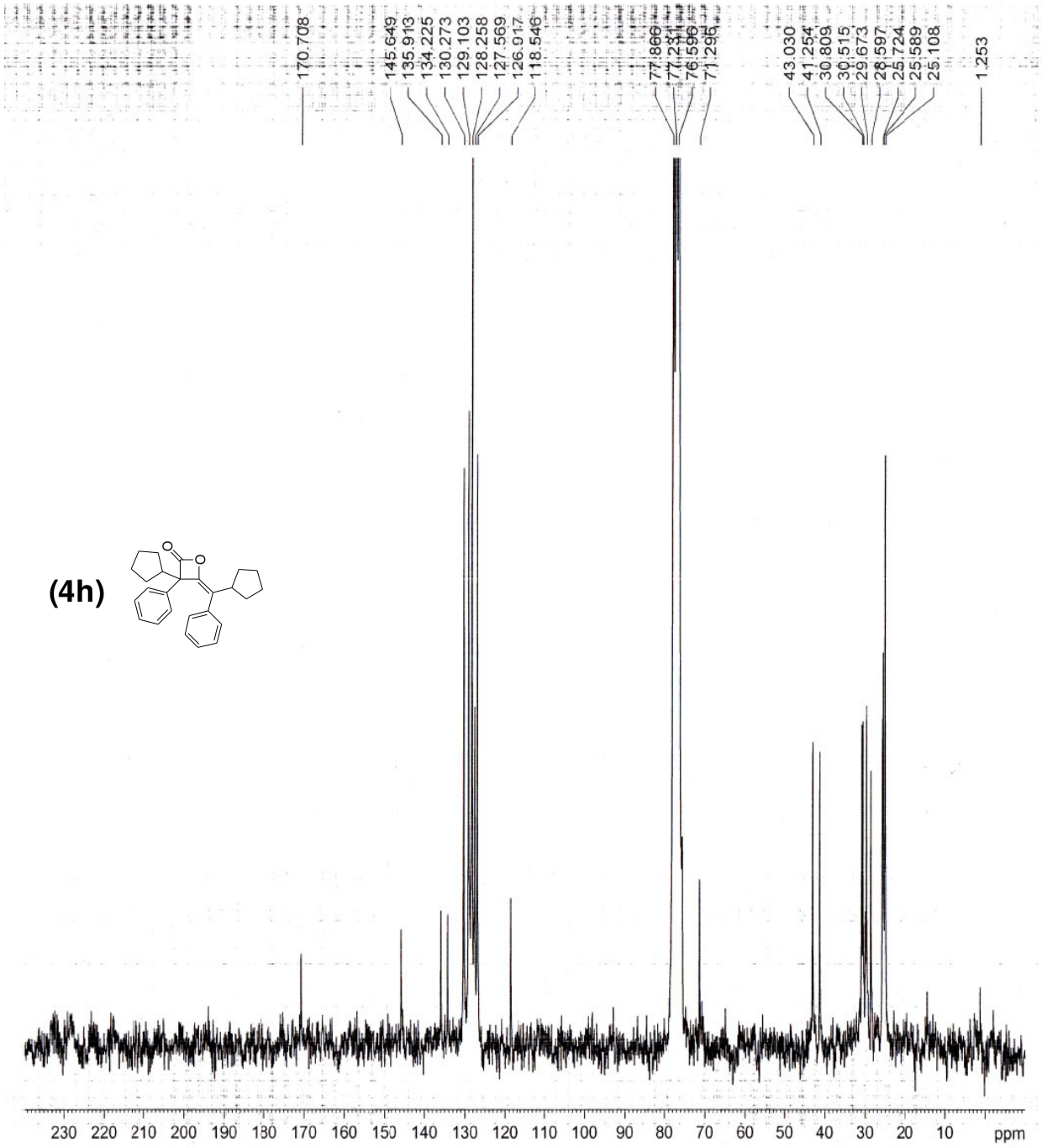
F2 - Acquisition Parameters
Date_ 20080910
Time 9.30
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgdc
TD 37686
SOLVENT CDCl3
NS 9500
DS 4
SWH 12562.814 Hz
FIDRES 0.333355 Hz
AQ 1.4999528 sec
RG 57
DW 39.800 usec
DE 6.00 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 1.00 dB
SFO1 50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 16.72 dB
SFO2 200.1300000 MHz

F2 - Processing parameters
SI 32768
SF 50.3227208 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40


7.471
7.438
7.432
7.351
7.312
7.296
7.284
7.274
7.247
7.220
7.187
7.114
7.103
7.075
7.067
1.684
1.659
1.633
1.600
1.577
1.533
1.402
1.273
1.255
1.185
0.809
0.780
0.000
BRUKER
(4h)
Current Data Parameters
NAME G Harzmann
EXPNO 144
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080630
Time 9.47
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 4096
SOLVENT CDCl3
NS 16
DS 2
SWH 2796.421 Hz
FIDRES 0.682720 Hz
AQ 0.7324148 sec
RG 101.6
DW 178.800 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -3.00 dB
SFO1 200.1313509 MHz
F2 - Processing parameters
SI 32768
SF 200.1300230 MHz
WDW EM
SSB 0
LB 0.03 Hz
GB 0
PC 1.00
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.012
0.194
0.143
2.535
0.293



(4h)

BRUKER

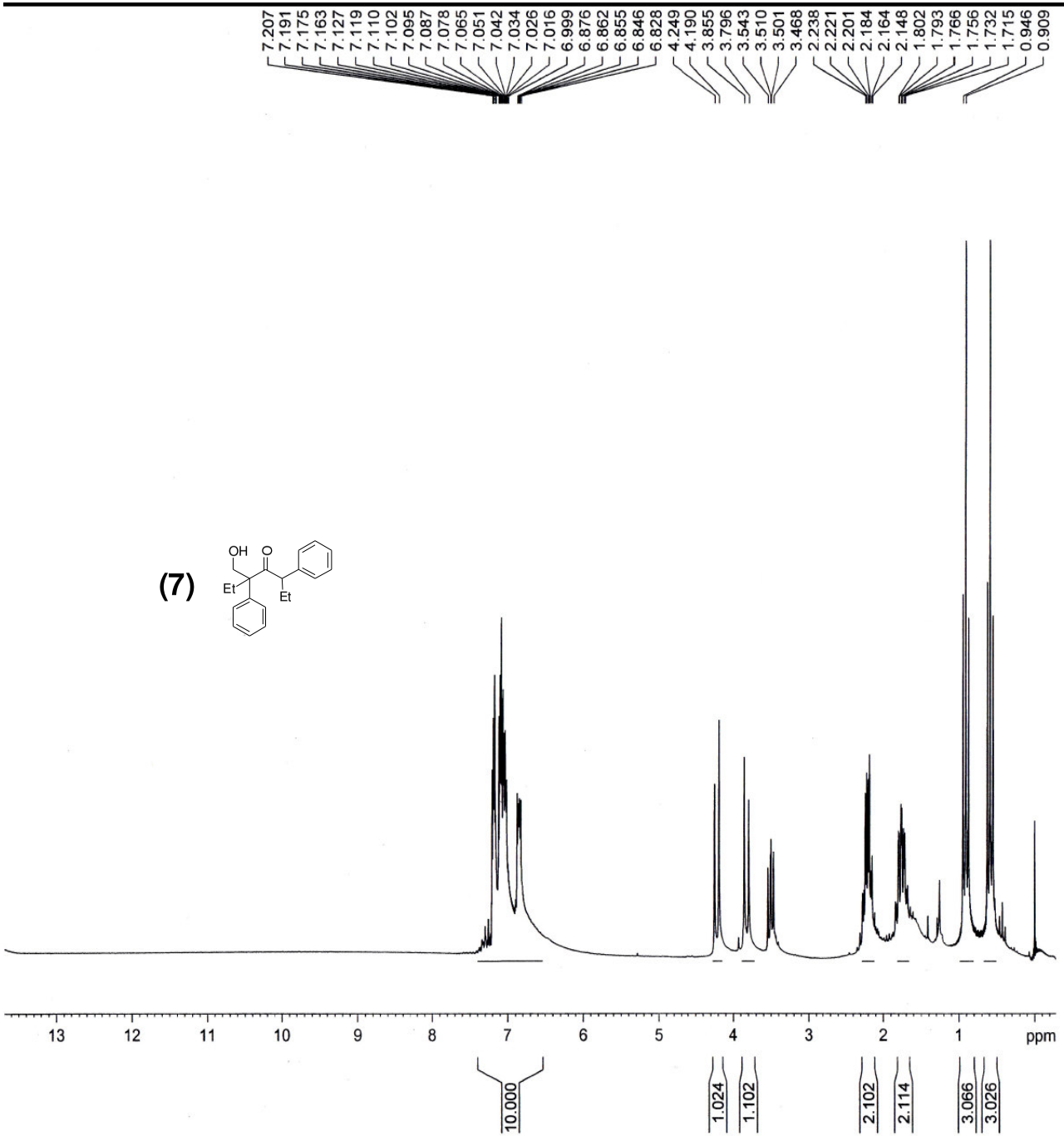
```
Current Data Parameters
NAME          G Harzmann
EXPNO                153
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080719
Time               18.26
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG            zgdc
TD                37686
SOLVENT           CDCl3
NS                15042
DS                    4
SWH           12562.814 Hz
FIDRES         0.333355 Hz
AQ             1.4999528 sec
RG                 80.6
DW                39.800 usec
DE                 6.00 usec
TE                300.0 K
D1            4.00000000 sec
d11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 usec
PL1                1.00 dB
SFO1         50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                -4.00 dB
PL12               16.72 dB
SFO2        200.1300000 MHz

F2 - Processing parameters
SI                 32768
SF           50.3227193 MHz
WDW                EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
```



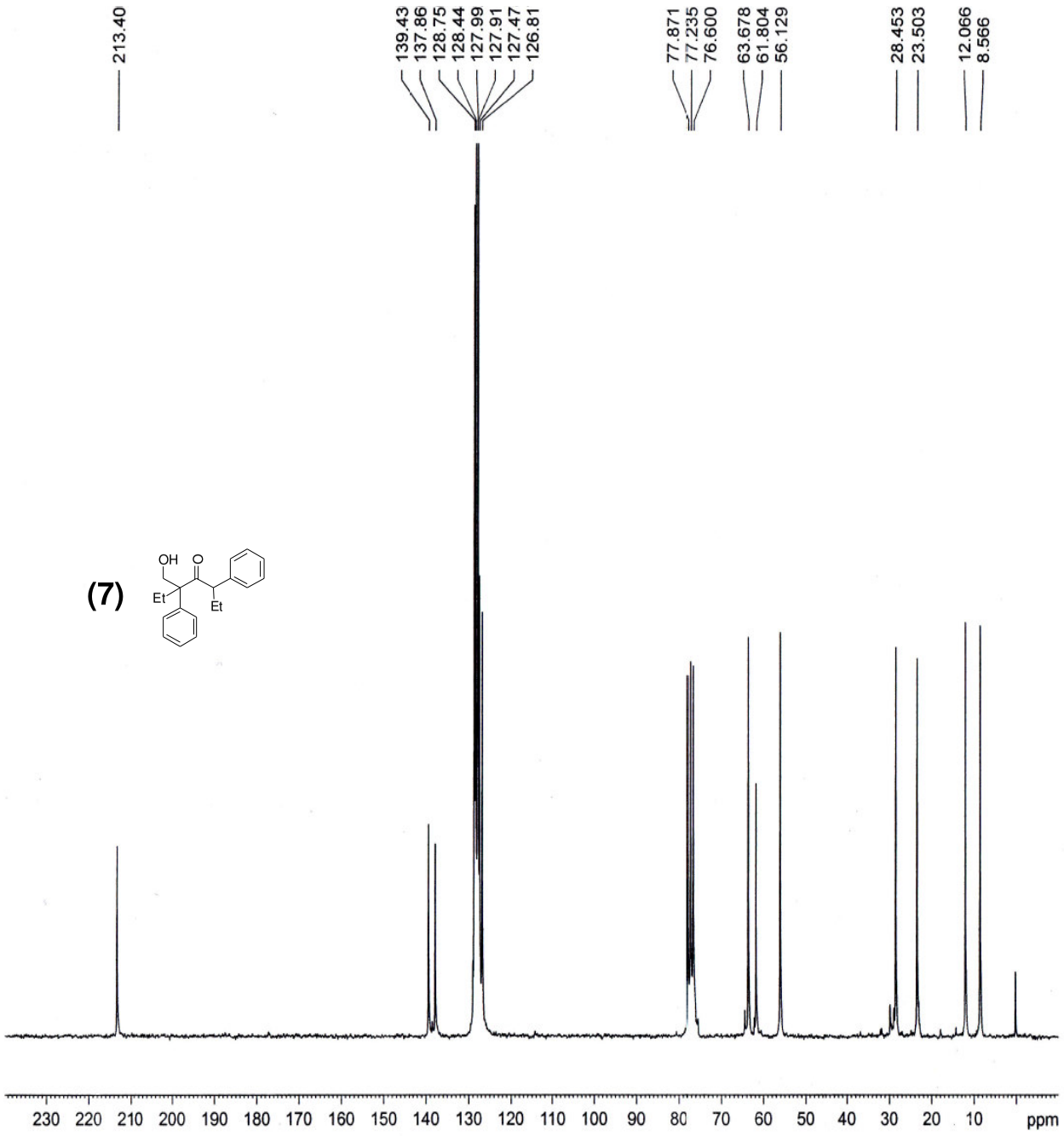
BRUKER

Current Data Parameters
NAME G Harzmann
EXPNO 88
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080501
Time 11.36
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 4096
SOLVENT CDCl3
NS 16
DS 2
SWH 2796.421 Hz
FIDRES 0.682720 Hz
AQ 0.7324148 sec
RG 90.5
DW 178.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -3.00 dB
SFO1 200.1313509 MHz

F2 - Processing parameters
SI 32768
SF 200.1300093 MHz
WDW EM
SSB 0
LB 0.03 Hz
GB 0
PC 1.00



BRUKER

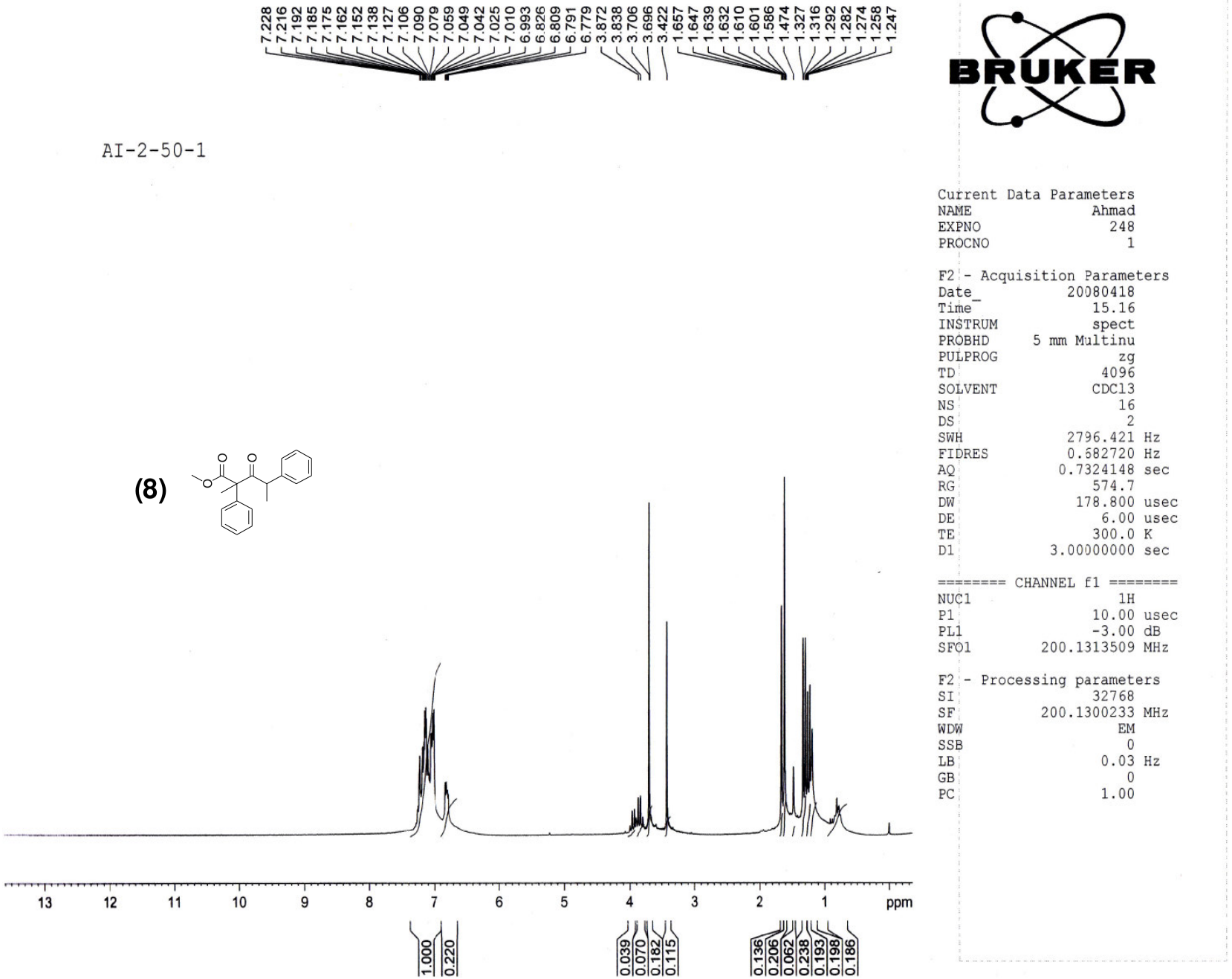
```
Current Data Parameters
NAME          N_Kerrigan
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080406
Time              11.58
INSTRUM           spect
PROBHD     5 mm Multinu
PULPROG           zgdc
TD               37686
SOLVENT          CDCl3
NS                9500
DS                   4
SWH          12562.814 Hz
FIDRES        0.333355 Hz
AQ           1.4999528 sec
RG                90.5
DW              39.800 usec
DE               6.00 usec
TE              300.0 K
D1           4.00000000 sec
d11          0.03000000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                6.00 usec
PL1               1.00 dB
SFO1         50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2              -4.00 dB
PL12             16.72 dB
SFO2        200.1300000 MHz

F2 - Processing parameters
SI                32768
SF          50.3227224 MHz
WDW                EM
SSB                 0
LB               3.00 Hz
GB                  0
PC               1.40
```




```
Current Data Parameters
NAME                 Ahmad
EXPNO                  248
PROCNO                   1

F2 - Acquisition Parameters
Date_             20080418
Time                 15.16
INSTRUM              spect
PROBHD     5 mm Multinu
PULPROG                zg
TD                   4096
SOLVENT              CDCl3
NS                     16
DS                      2
SWH               2796.421 Hz
FIDRES            0.682720 Hz
AQ                0.7324148 sec
RG                   574.7
DW                 178.800 usec
DE                   6.00 usec
TE                  300.0 K
D1             3.00000000 sec

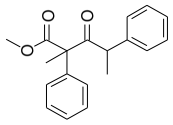
======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -3.00 dB
SFO1          200.1313509 MHz

F2 - Processing parameters
SI                   32768
SF            200.1300233 MHz
WDW                    EM
SSB                     0
LB                   0.03 Hz
GB                      0
PC                   1.00
```

AI-2-50-1

208.220
207.525
172.917
172.254
141.349
138.274
137.528
128.603
128.477
128.145
127.957
127.489
127.031
126.708
77.872
77.237
76.602
65.495
65.393
52.727
52.481
49.696
49.478
29.910
22.351
22.157
21.912
21.458

(8)



BRUKER

Current Data Parameters
NAME Ahmad
EXPNO 249
PROCNO 1

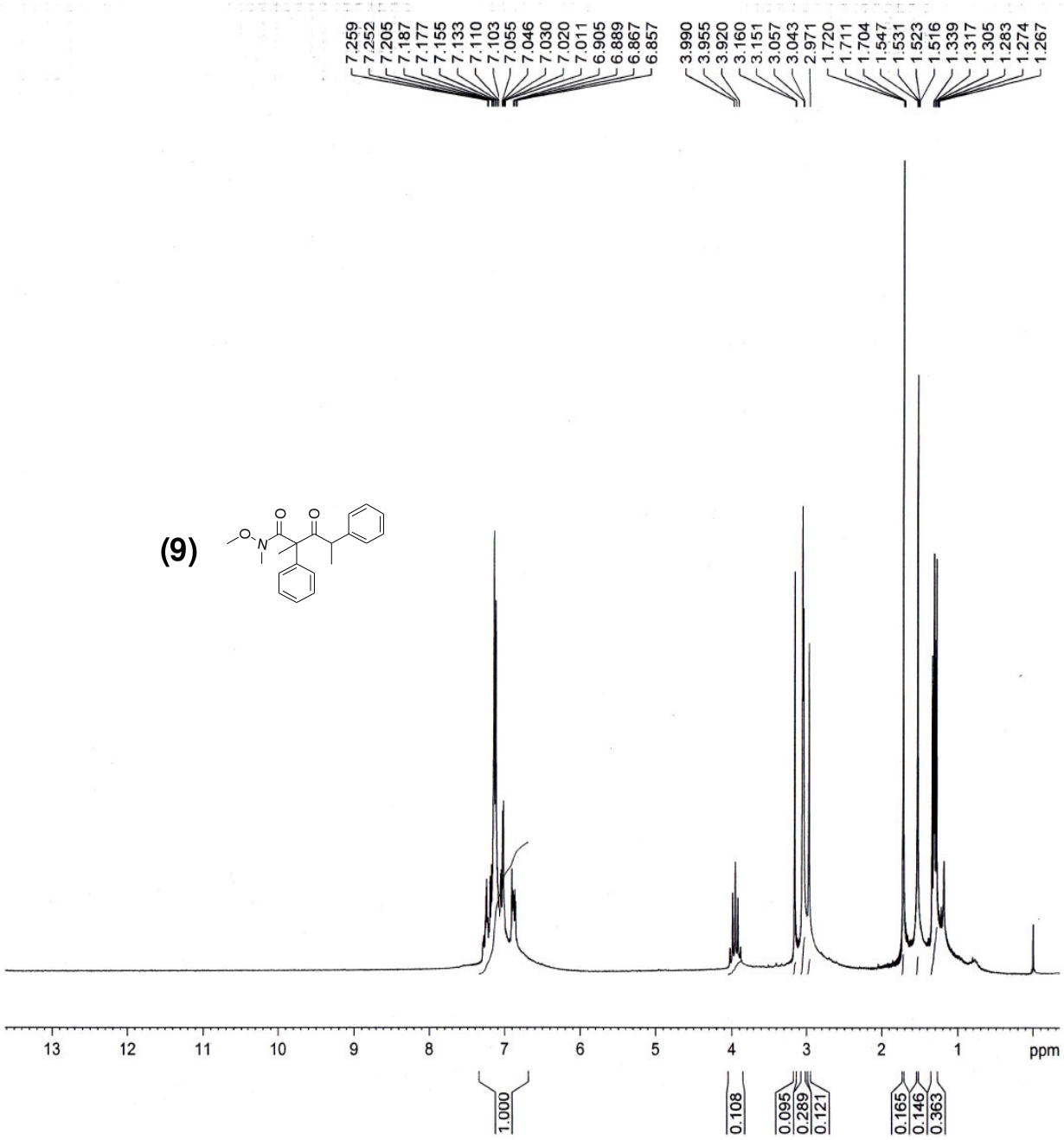
F2 - Acquisition Parameters
Date_ 20080422
Time 10.38
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgdc
TD 37686
SOLVENT DMSO
NS 9500
DS 4
SWH 12562.814 Hz
FIDRES 0.333355 Hz
AQ 1.4999528 sec
RG 90.5
DW 39.800 usec
DE 6.00 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 1.00 dB
SFO1 50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 16.72 dB
SFO2 200.1300000 MHz

F2 - Processing parameters
SI 32768
SF 50.3227193 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

(9)



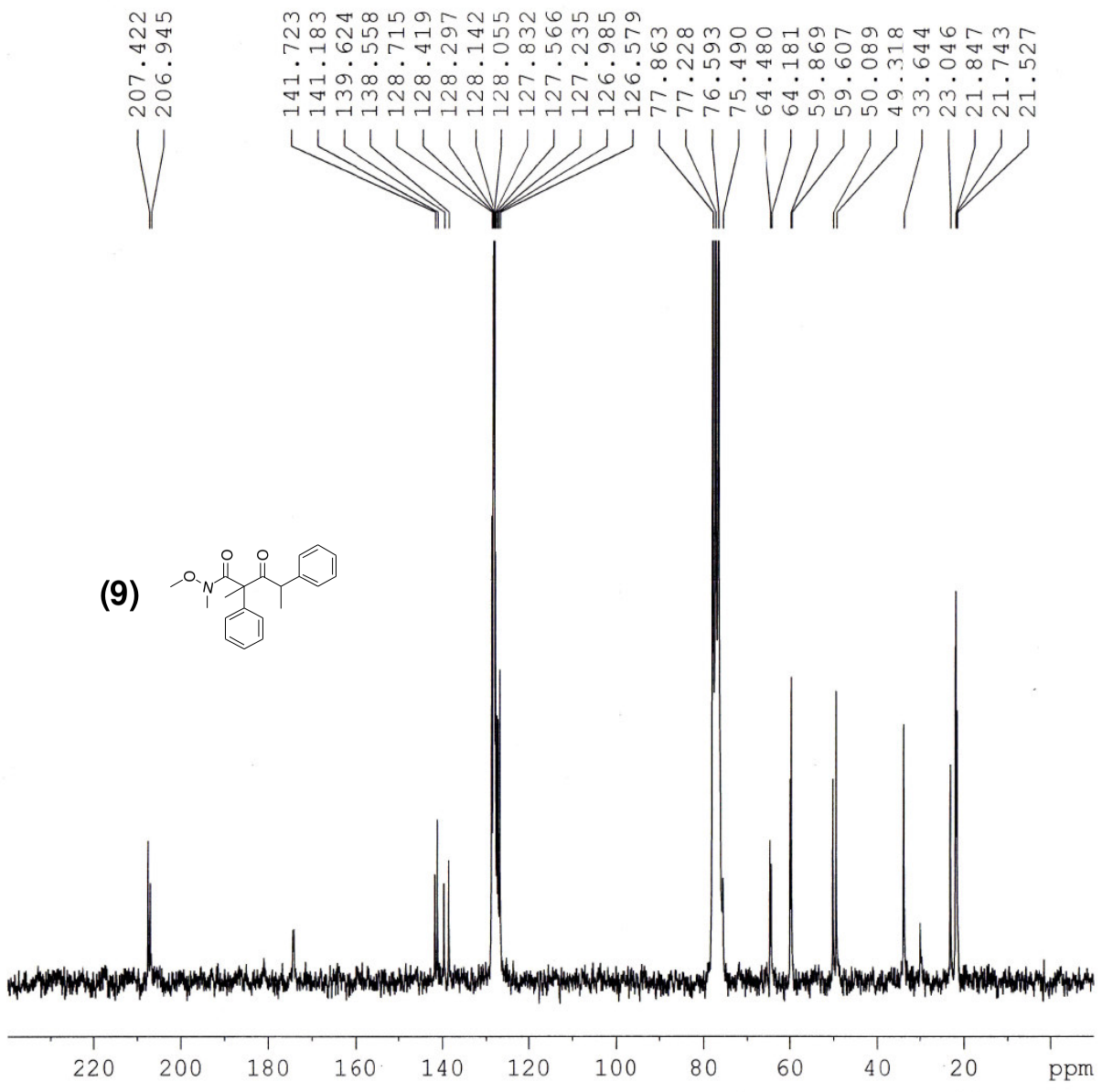

```
Current Data Parameters
NAME          G Harzmann
EXPNO                 92
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080506
Time              17.06
INSTRUM           spect
PROBHD     5 mm Multinu
PULPROG             zg
TD                4096
SOLVENT           CDCl3
NS                  16
DS                   2
SWH            2796.421 Hz
FIDRES          0.682720 Hz
AQ              0.7324148 sec
RG                90.5
DW               178.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                10.00 usec
PL1               -3.00 dB
SFO1        200.1313509 MHz

F2 - Processing parameters
SI                32768
SF          200.1300232 MHz
WDW                 EM
SSB                  0
LB                0.03 Hz
GB                   0
PC                1.00
```

GH1-99-1 (Carbon)



(9)

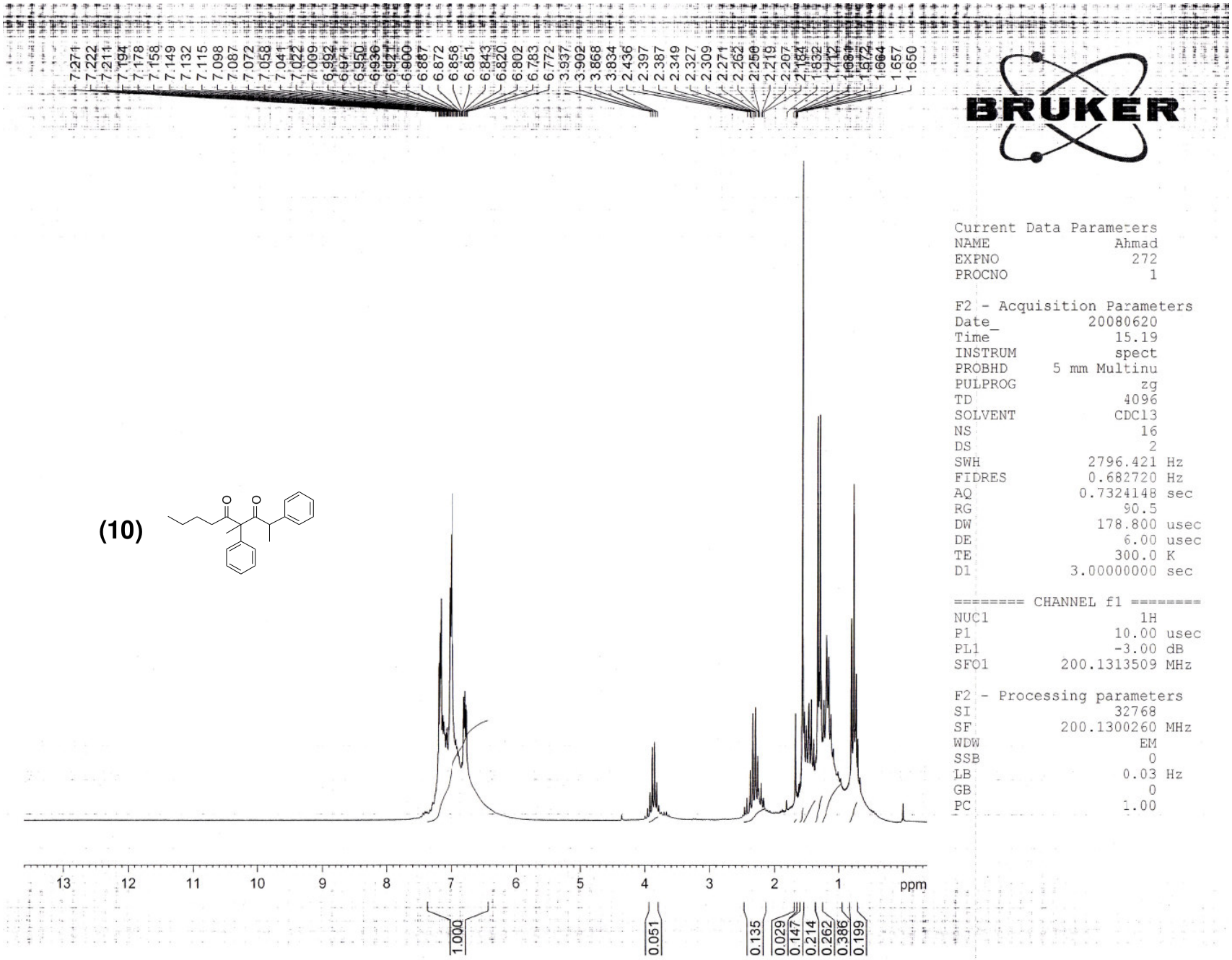

```
Current Data Parameters
NAME          G Harzmann
EXPNO                 93
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080507
Time                9.52
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG             zgdc
TD                 37686
SOLVENT            CDCl3
NS                  9482
DS                     4
SWH            12562.814 Hz
FIDRES          0.333355 Hz
AQ              1.4999528 sec
RG                  71.8
DW                39.800 use
DE                 6.00 use
TE                300.0 K
D1           4.00000000 sec
d11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 use
PL1                1.00 dB
SFO1         50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             100.00 use
PL2                -4.00 dB
PL12               16.72 dB
SFO2         200.1300000 MHz

F2 - Processing parameters
SI                 32768
SF           50.3227197 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40
```

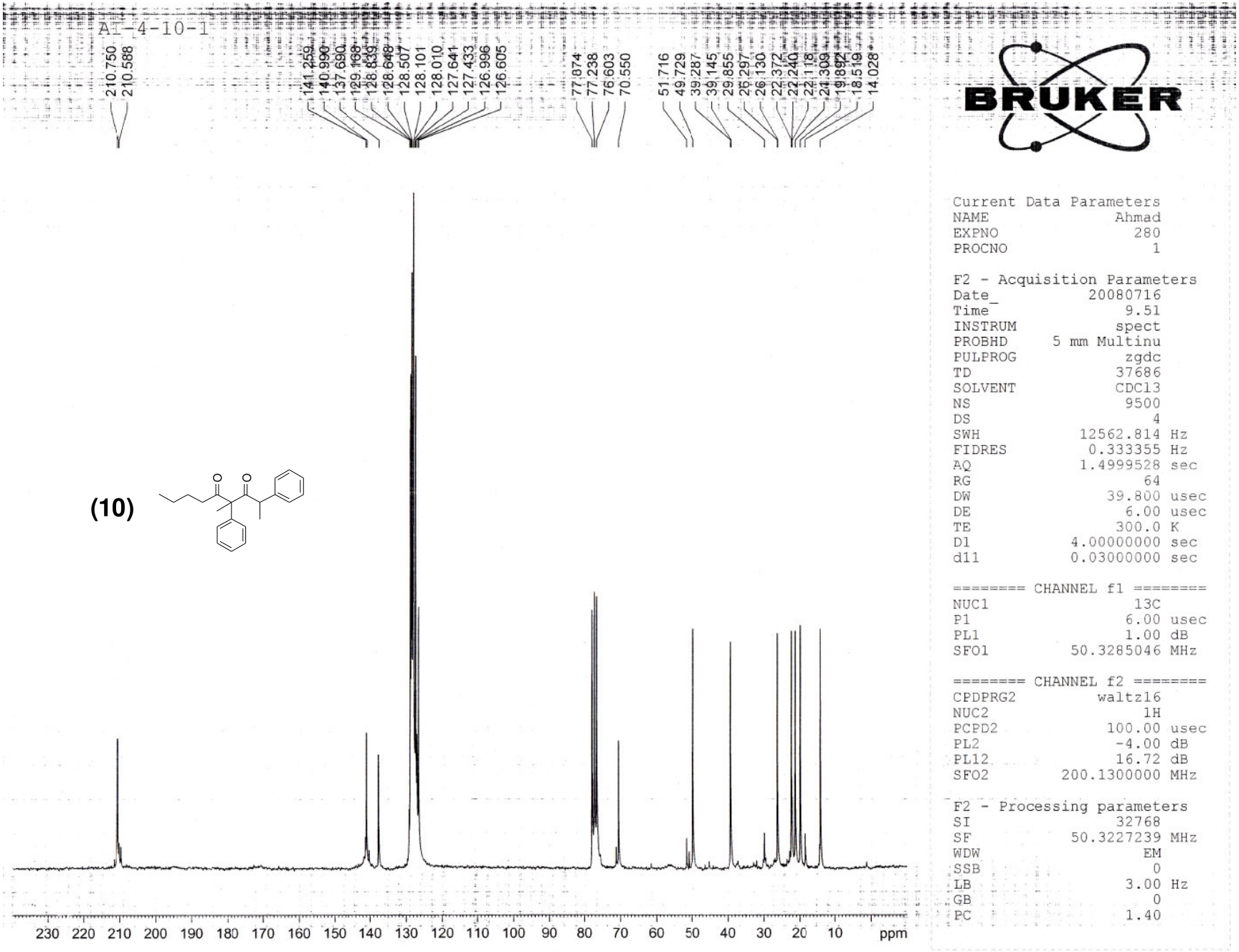



```
Current Data Parameters
NAME                  Ahmad
EXPNO                   272
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080620
Time                  15.19
INSTRUM               spect
PROBHD      5 mm Multinu
PULPROG                 zg
TD                    4096
SOLVENT               CDCl3
NS                      16
DS                       2
SWH                2796.421 Hz
FIDRES             0.682720 Hz
AQ                 0.7324148 sec
RG                      90.5
DW                   178.800 usec
DE                     6.00 usec
TE                    300.0 K
D1              3.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   10.00 usec
PL1                  -3.00 dB
SFO1          200.1313509 MHz

F2 - Processing parameters
SI                    32768
SF            200.1300260 MHz
WDW                     EM
SSB                      0
LB                    0.03 Hz
GB                       0
PC                    1.00
```



BRUKER

```
Current Data Parameters
NAME                 Ahmad
EXPNO                  280
PROCNO                   1

F2 - Acquisition Parameters
Date_             20080716
Time                 9.51
INSTRUM             spect
PROBHD     5 mm Multinu
PULPROG              zgdc
TD                  37686
SOLVENT             CDC13
NS                   9500
DS                      4
SWH              12562.814 Hz
FIDRES           0.333355 Hz
AQ               1.4999528 sec
RG                     64
DW                 39.800 usec
DE                  6.00 usec
TE                 300.0 K
D1              4.00000000 sec
d11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  6.00 usec
PL1                 1.00 dB
SFO1          50.3285046 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -4.00 dB
PL12               16.72 dB
SFO2         200.1300000 MHz

F2 - Processing parameters
SI                   32768
SF            50.3227239 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40
```