# Pd-Catalyzed Carbonylative Conjugate Addition of Dialkylzinc Reagents to Unsaturated Carbonyls

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## Supporting Information

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## General Information

$^1$H NMR spectra were recorded on Varian Unity Inova 500 MHz and Varian Gemini 400 MHz spectrometers. Chemical shifts are reported in ppm with the solvent resonance as the internal standard ($CDCl_3$: 7.24 ppm, $C_6D_6$: 7.16 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, sept = septet, b = broad, m = multiplet), coupling constants (Hz) and integration. $^{13}C\{^1H\}$NMR spectra were recorded on Varian Unity Inova 500 MHz (125 MHz) and Varian Gemini 400 MHz (100 MHz) spectrometers. Chemical shifts are reported in ppm with the solvent resonance as the internal standard ($CDCl_3$: 77.00 ppm, $C_6D_6$: 128.0 ppm). Infrared (IR) spectra were recorded on a Bruker α-P Spectrometer. Frequencies are reported in wavenumbers ($cm^{-1}$). High-resolution mass spectrometry (HRMS) and low-resolution mass spectrometry (ESI) was performed at Boston College, Chestnut Hill, MA.

Liquid chromatography was performed using forced flow (flash chromatography) on silica gel ($SiO_2$, 230 x 450 Mesh) purchased from Silicycle. Thin layer chromatography was performed on 25 μm silica gel glass backed plates from Silicycle. Visualization was performed using ultraviolet light (254 nm), ceric ammonium molybdate (CAM), and potassium permanganate ($KMnO_4$).

All reactions were conducted in oven- or flame-dried glassware under an inert atmosphere of nitrogen or argon. Tetrahydrofuran and dichloromethane were purified using Pure Solv MD-4 solvent purification system, from Innovative Technology, Inc., by passing the solvent through two activated alumina columns after being purged with argon. Trimethylsilyl chloride, *trans*-2-nonenal, cinnamaldehyde, 3-methyl-2-butenal, 2-methyl-2-pentenal, *trans*-1-phenyl-2-buten-1-one, isovaleraldehyde, and benzaldehyde were distilled from calcium hydride. Triphenylphosphine was recrystallized from ethanol. Methyl 4-(3-oxo-3-phenyl-1-propenyl)benzoate, 4-chlorochalcone, 4-methoxychalcone were purchased from Acros Organics. Triethylsilyl chloride was purchased from Gelest, Inc. and used without further purification. Acetic acid was purchased from Fisher Scientific and used without further purification. Tris(dibenzylideneacetone)dipalladium (0) and tricyclohexylphosphine were purchased from Strem Chemicals, Inc. and used without further purification. Triphenylsilyl chloride, diphenylmethylsilyl chloride, dimethylphenylsilyl chloride, tertrabutylammonium flouride, *trans*-4-phenyl-3-buten-2-one, cyclohexenone, 4,4-dimethyl-2-cyclohexenone, 4-hexen-3-one were purchased from Aldrich and used without further purification.

## Experimental Procedures

### *I. Representative Procedure for Conjugate Addition with Solid α,β-Unsaturated Carbonyls.*

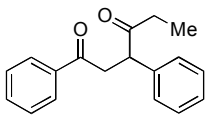
In the glove box, $Pd_2(dba)_3$ (6.9 mg, 7.5 μmol) and triphenylphosphine (4.7 mg, 18 μmol) were added to an oven-dried roundbottom flask charged with a magnetic stir bar. The flask was sealed with a rubber septum, removed from the dry box, and placed under atmosphere of argon. To the flask was added tetrahydrofuran (1.0 mL) and was allowed to stir for 30 minutes. Next, CO (balloon) was added to the mixture and the mixture was vented for one minute; the vent was closed and freshly distilled trimethylsilyl chloride (88 μL, 690 μmol) was added followed by a solution of chalcone (62.5 mg, 300 μmol) in tetrahydrofuran (1.0 mL). The mixture was stirred for 10 minutes prior to dropwise addition of diethyl zinc (44 μL, 390 μmol). The mixture was then allowed to stir for 3 hours under a CO (g) atmosphere. It was then passed through a plug of

$SiO_2$ (60% diethyl ether/hexanes). The material was concentrated *in vacuo* by rotary evaporation and then diluted with tetrahydrofuran (4.2 mL). The mixture was cooled to 0 °C and treated with acetic acid (26 μL, 450 μmol) and TBAF (450 μL, 450 μmol, 1.0 M in THF). The reaction mixture stirred for 10 minutes and was quenched with saturated aqueous sodium bicarbonate and the organic layer was separated. The aqueous layer was washed with diethyl ether (5 X 5 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified on silica gel (15% diethyl ether/hexanes) to afford a clear, colorless oil (67.9 mg, 85.0% yield).

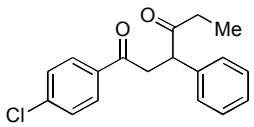
***11. Representative Procedure for Conjugate Addition with Liquid α,β-Unsaturated Carbonyls.***

In the glove box, $Pd_2(dba)_3$ (6.9 mg, 7.5 μmol) and triphenylphosphine (4.7 mg, 18 μmol) were added to an oven-dried roundbottom flask charged with a magnetic stir bar. The flask was sealed with a rubber septum, removed from the dry box, and placed under atmosphere of argon. To the flask was added tetrahydrofuran (2.0 mL) and was allowed to stir for 30 minutes. Next, CO (balloon) was added to the mixture and the mixture was vented for one minute; the vent was closed and freshly distilled trimethylsilyl chloride (88 μL, 690 μmol) was added and followed by a solution of *trans*-2-nonenal (50.0 μL, 300 μmol). The mixture was stirred for 10 minutes prior to dropwise addition of diethyl zinc (44 μL, 390 μmol). The mixture was then allowed to stir for 4 hours under a CO(g) atmosphere. It was then passed through a plug of $SiO_2$ (60% diethyl ether/hexanes). The material was concentrated *in vacuo* by rotary evaporation and then diluted with tetrahydrofuran (4.2 mL). The mixture was cooled to 0 °C and treated with acetic acid (26 μL, 450 μmol) and TBAF (450 μL, 450 μmol, 1.0 M in THF). The reaction mixture stirred for 10 minutes and was quenched with saturated aqueous sodium bicarbonate and the organic layer was separated. The aqueous layer was washed with diethyl ether (5 X 5 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified on silica gel (15% diethyl ether/hexanes) to afford a clear, colorless oil (46.4 mg, 78.0% yield).

## *III. Full Characterization*

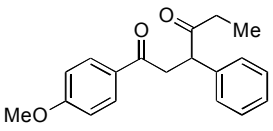



**1,3-diphenylhexane-1,4-dione (Compound S-1):** Purified on $SiO_2$ (15% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.94 (bd, J = 8.3 Hz, 2H); 7.52 (tt, J = 6.7, 1.2 Hz, 1H); 7.44-7.39 (m, 2H); 7.35-7.31 (m, 2H); 7.28-7.24 (m, 2H); 4.42 (dd, J = 10.2, 3.5 Hz, 1H); 4.03 (dd, J = 18.0, 10.2 Hz, 1H); 3.11 (dd, J = 18.0, 3.5 Hz, 1H); 2.64 (dq, J = 18.0, 7.4 Hz, 1H); 2.50 (dq, J = 18.0, 7.2 Hz, 1H); 1.00 (t, J = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.5, 198.7, 138.8, 137.0, 133.2, 129.2, 129.1, 129.0, 128.6, 127.6, 53.5, 42.4, 35.2, 8.0; IR (neat) 3028, 1715, 1683, 1398, 753, 700, 690; HRMS (ESI+) for $C_{18}H_{18}O_2$ [M+H]: calculated: 267.1385, found: 267.1385.

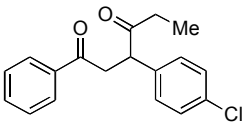



**1-(4-chlorophenyl)-3-phenylhexane-1,4-dione (Compound S-2):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to a afford a clear oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.90 (bd, J = 6.9 Hz, 2H); 7.42 (bd,

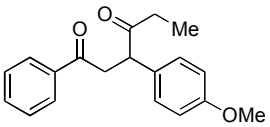
J = 6.8 Hz, 2H); 7.37-7.34 (m, 2H); 7.30-7.27 (m, 3H); 4.42 (dd, J = 10.0, 4.0 Hz, 1H); 4.01 (dd, J = 17.9, 10.1 Hz, 1H); 3.08 (dd, J = 18.1, 3.7 Hz, 1H); 2.63 (dq, J = 17.9, 7.4 Hz, 1H); 2.51 (dq, J = 18.1, 7.3 Hz, 1H); 1.02 (t, J = 7.3 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 209.8, 197.0, 139.6, 138.1, 134.8, 129.5, 129.1, 128.8, 128.2, 127.6, 53.0, 42.3, 34.9, 7.8; IR (neat)1714, 1683, 1589, 1400, 1090, 992, 835, 755, 700 ; HRMS (ESI+) for $C_{18}H_{17}ClO_2$ [M]: calculated: 301.0995, found 301.0999.



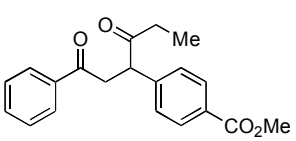

**1-(4-methoxyphenyl)-3-phenylhexane-1,4-dione (Compound S-3):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to a afford a pale yellow oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.94 (bd, J = 9.1 Hz, 2H); 7.36-7.34 (m, 2H); 7.33-7.26 (m, 3H); 6.91 (bd, J = 9.0 Hz, 2H); 4.42 (dd, J = 10.2, 3.6 Hz, 1H); 4.00 (dd, J = 17.9, 10.3 Hz, 1H); 3.85 (s, 3H); 3.11 (dd, J = 17.8, 3.6 Hz, 1H); 2.66 (dq, J = 17.9, 7.4 Hz, 1H); 2.52 (dq, J = 17.9, 7.4 Hz, 1H); 1.02 (t, J = 7.1 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 210.0, 196.7, 163.5, 138.4, 130.3, 129.6, 129.0, 128.3, 127.4, 113.7, 55.4, 53.0, 42.2, 35.0, 7.8; IR (neat) 1714, 1683, 1580, 1533, 1510, 1249, 1180, 1030, 1002, 655, 631; HRMS (ESI+) for $C_{19}H_{20}O_3$ [M+1]: calculated: 297.1490, found 297.1498.




**3-(4-chlorophenyl)-1-phenylhexane-1,4-dione (Compound S-4):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a white solid. mp= 100-102 °C; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.92 (bd, J = 8.2 Hz, 2H); 7.52 (tt, J = 6.7, 1.4 Hz, 1H); 7.41 (bt, J = 8.0 Hz, 2H); 7.30-7.27 (m, 2H); 7.22-7.19 (m, 2H); 4.38 (dd, J = 10.0, 3.7 Hz, 1H); 3.98 (dd, J = 18.2, 10.2 Hz, 1H); 3.10 (dd, J = 18.0, 3.9 Hz, 1H); 2.63 (dq, J = 18.0, 7.2 Hz, 1H); 2.48 (dq, J = 18.0, 7.3 Hz, 1H); 0.99 (t, J = 7.3 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 209.5, 197.8, 136.7, 136.3, 133.4, 133.2, 129.6, 129.2, 128.5, 128.0, 52.2, 42.3, 35.1, 7.8; IR (neat) 2916, 1714, 1681, 1480, 1237, 1110, 1019, 748, 689; HRMS (ESI+) for $C_{18}H_{17}ClO_2$ [M]: calculated: 301.0995, found: 301.0994.

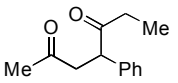



**3-(4-methoxyphenyl)-1-phenylhexane-1,4-dione (Compound S-5):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a white solid. mp= 89-90 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.87 (bd, J = 8.3 Hz, 2H); 7.52 (tt, J = 6.7, 1.4 Hz, 1H); 7.36 (bt, J = 7 Hz, 2H); 7.12 (bd, J = 7.8 Hz, 2H); 6.80 (bd, J = 7.8 Hz, 2H); 4.35 (dd, J = 10.2, 3.8 Hz, 1H); 3.98 (dd, J = 18.0, 10.2 Hz, 1H); 3.77 (s, 3H); 3.08 (dd, J = 18.0, 3.9 Hz, 1H); 2.63 (dq, J = 18.0, 7.4 Hz, 1H); 2.49 (dq, J = 18.0, 7.5 Hz, 1H); 0.99 (t, J = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.2, 198.3, 158.9, 136.5, 133.1, 130.2, 129.3, 128.5, 128.0, 114.4, 55.2, 52.0, 42.4, 34.8, 7.8; IR (neat) 2973, 2936, 1714, 1683, 1609, 1510, 1249, 1116, 993, 690, 681; LRMS (ESI+) for $C_{19}H_{20}O_3$ [M+H]: calculated: 297.2, found: 297.2.

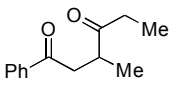



**Methyl 4-(1,4-dioxo-1-phenylhexan-3-yl)benzoate (Compound S-6):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a pale yellow solid. mp= 85-87 °C $^1$H NMR (400 MHz, $CDCl_3$) δ 7.97 (bd, J = 8.2 Hz, 2H); 7.90 (bd, J = 8.4 Hz, 2H); 7.50 (tt, J = 4.8, 1.2 Hz, 1H); 7.39 (bt, J = 7.8 Hz, 2H); 7.33 (bd, J = 8.3 Hz, 2H); 4.46 (dd, J = 10.0, 3.7 Hz, 1H); 4.01 (dd, J = 18.0, 10.2 Hz, 1H); 3.86 (s, 3H); 3.12 (dd, J = 18.0, 4.0 Hz, 1H); 2.63 (dq, J = 18.0, 7.2

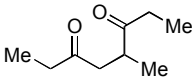
Hz, 1H); 2.45 (dq, J = 18.0, 7.3 Hz, 1H); 0.97 (t, J = 7.3 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 209.2, 197.7, 166.6, 143.4, 136.2, 133.3, 130.3, 129.4, 128.5, 128.3, 128.0, 52.9, 52.1, 42.2, 35.2, 7.7; IR (neat) 2918, 1714, 1681, 1608, 1414, 1276, 1110, 762, 748, 706, 689; HRMS (ESI+) for $C_{20}H_{20}O_4$ [M+H]: calculated: 325.1440, found: 325.1448.



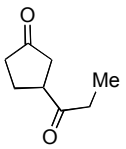

**4-phenylheptane-2,5-dione (Compound S-7):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.54-7.42 (m, 5H); 4.46 (dd, J = 10.4, 4.0 Hz, 1H); 3.71 (dd, J = 18.0, 10.4 Hz, 1H); 2.81 (dd, H = 18.0, 3.7 Hz, 1H); 2.71 (dq, J = 17.8, 3.4 Hz, 2H); 2.39 (s, 3H); 1.20 (t, J = 7.4 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 209.8, 206.7, 138.1, 129.0, 128.1, 127.4, 52.9, 46.6, 34.7, 29.8, 7.7; IR (neat) 2963, 1711, 1515, 1491, 1356, 850, 749, 701, 690; LRMS (ESI+) for $C_{13}H_{16}O_2$ [M+H]: calculated: 205.1, found: 205.1.



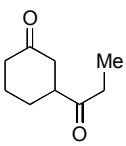

**3-methyl-1-phenylhexane-1,4-dione (Compound S-8):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.92 (bd, J = 8.2 Hz, 2H); 7.52 (tt, J = 6.7, 1.4 Hz, 1H); 7.41 (bt, J = 7.8 Hz, 2H); 3.52 (dd, J = 18.0, 9.0 Hz, 1H); 3.25-3.17 (m, 1H); 2.91 (dd, J = 18.0, 4.5 Hz, 1H); 2.64 (dq, J = 7.2, 1.8 Hz, 2H); 1.15 (d, J = 7.2 Hz, 3H); 1.06 (t, J = 7.3 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 214.4, 199.0, 136.9, 133.8, 129.1, 128.4, 42.5, 41.8, 35.1, 17.7, 8.0; IR (neat) 3061, 1712, 1683, 1458, 1353, 1216, 749, 690; HRMS (ESI+) for $C_{13}H_{16}O_2$ [M+H]: calculated: 205.1229, found: 205.1220.




**4-methyloctane-3,6-dione (Compound S-9):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.95-2.86 (m, 1H); 2.81 (dd, J = 17.6, 9.4 Hz, 1H); 2.43 (dq, J = 7.4, 1.0 Hz, 2H); 2.36-2.18 (m, 3H); 0.93 (d, J = 7.1 Hz, 3H); 0.90 (t, J = 7.3 Hz, 3H); 0.88 (t, J = 7.5 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 214.5, 210.0, 45.7, 40.9, 36.0, 34.6, 17.1, 7.9, 7.8; IR (neat) 2974, 1710, 1459, 1376, 1356, 1116; HRMS (ESI+) for $C_9H_{16}O_2$ [M+H]: calculated: 157.1229, found: 157.1234.

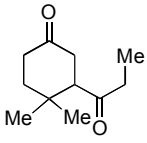



**3-propionylcyclopentanone (Compound S-10):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 3.22 (pentet, J = 8.5 Hz, 1H); 2.59 (dq, J = 18.0, 7.2 Hz, 1H); 2.58-2.42 (m, 2H); 2.36-2.10 (m, 4H); 1.96 (ddt, J = 16.8, 9.6, 8.2 Hz, 1H); 1.05 (dd, J = 7.2, 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 217.1, 211.9, 47.9, 40.8, 37.9, 34.9, 26.7, 7.9; IR (neat) 2974, 1739, 1705, 1459, 1406, 1374, 1138, 1113; HRMS (ESI+) for $C_9H_{14}O_2$ [M+H]: calculated: 141.0915, found: 141.0912.

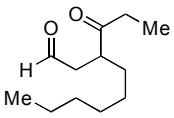



**3-propionylcyclohexanone (Compound S-11):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.85 (tt, J = 11.0, 4.3 Hz, 1H); 2.55 (dq, J = 18.8, 7.2 Hz, 1H); 2.53-2.24 (m, 5H); 2.09-1.99 (m, 2H); 1.76-1.62 (m, 2H); 1.03 (t, J = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 211.0, 209.9, 49.8, 42.5, 40.8, 34.1, 27.3, 24.8, 7.5; IR (neat) 2939, 1708,

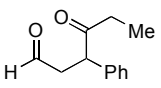
1450, 1417, 1224, 1123, 964; HRMS (ESI+) for $C_9H_{14}O_2$ [M+H]: calculated: 155.1072, found: 155.1066.



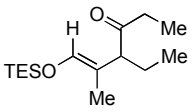
**4,4-dimethyl-3-propionylcyclohexanone (Compound S-12):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.81 (dd, J = 8.8, 4.9 Hz, 1H); 2.52 (dq, J = 18.4, 7.3 Hz, 1H); 2.50-2.33 (m, 3H); 2.30-2.19 (m, 2H); 1.81 (ddd, J = 12.7, 6.1, 6.1 Hz, 1H); 1.57 (ddd, J = 14.6 ,9.2, 5.6 Hz, 1H); 1.10 (s, 3H); 1.05 (s, 3H); 0.98 (dd, J = 7.2, 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 212.5, 209.8, 57.1, 39.9, 38.4, 37.5, 33.0, 28.8, 23.3, 7.8; IR (neat) 2958, 1712, 1458, 1415, 1369, 1144; HRMS (ESI+) for $C_{11}H_{18}O_2$ [M+H]: calculated: 183.1385, found: 183.1390.



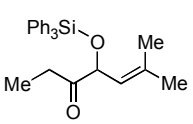
**3-propionylnonanal (Compound S-13):** Purified on $SiO_2$ (15% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 9.74 (s, 1H); 3.06-2.94 (m, 2H); 2.66-2.52 (m, 2H); 2.49 (dd, J = 17.9, 3.5 Hz, 1H); 1.63-1.56 (m, 1H); 1.44-1.36 (m, 1H); 1.30-1.27 (m, 8H); 1.07 (t, J = 7.1 Hz, 3H); 0.88 (t, J = 6.9 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 213.3, 200.7, 45.1, 44.9, 35.3, 31.6, 31.5, 29.2, 27.0, 22.5, 13.9, 7.6; IR (neat) 2935, 2927, 1709, 1459, 1390, 1109, 741, 735; HRMS (ESI+) for $C_{12}H_{22}O_2$ [M+H]: calculated: 199.1698, found: 199.1702.



**4-oxo-3-phenylhexanal (Compound S-14):** Purified on $SiO_2$ (15% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 9.80 (s, 1H); 7.40-7.20 (m, 5H); 4.13 (dd, J = 9.7, 5.2 Hz, 1H); 3.27 (dd, J = 17.6, 10.0 Hz, 1H); 2.56 (dd, J = 17.4, 5.2 Hz, 1H); 2.41 (dq, J = 18.0, 7.7 Hz, 2H); 0.98 (t, J = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 209.2, 200.0, 137.8, 129.1, 128.1, 127.6, 51.8, 46.8, 34.6, 7.8; IR (neat) 2976, 1712, 1493, 1454, 1120, 755, 701; LRMS (ESI+) for $C_{12}H_{14}O_2$ [M+H]: calculated: 191.2, found: 191.2.

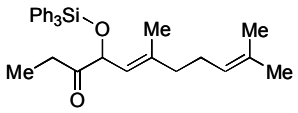


***trans*-4-ethyl-5-methyl-6-(triethylsilyloxy)hex-5-en-3-one (Compound S-15):** Purified on $SiO_2$ (5% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 6.22 (s, 1H); 2.86 (dd, J = 7.0, 7.0 Hz, 1H); 2.64 (dq, J = 17.5, 7.3 Hz, 1H); 2.40 (dq, J = 17.5, 7.5 Hz, 1H); 1.82 (dtd, J = 14.6, 7.2, 7.2 Hz, 1H); 1.62-1.51 (m, 1H); 1.54 (s, 3H); 1.12-1.08 (m, 3H); 1.08 (t, J = 7.6 Hz, 9H); 0.87 (t, J = 7.2 Hz, 3H); 0.76 (q, J = 8.2 Hz, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 212.0, 137.9, 115.0, 57.4, 34.0, 21.1, 11.9, 9.8, 7.9, 6.6, 4.6; IR (neat) 2958, 2877, 1712, 1660, 1235, 1169, 1125, 1005; HRMS (ESI+) for $C_{15}H_{30}O_2Si$ [M+H]: calculated: 271.2093, found: 271.2095.

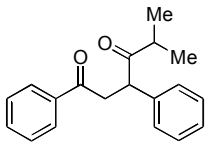


**6-methyl-4-(triphenylsilyloxy)hept-5-en-3-one (Compound S-16):** Purified on $SiO_2$ (10% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.64 (bd, J = 6.6 Hz, 6H); 7.46-7.37 (m, 9H); 5.21-5.18 (m, 1H); 4.93 (d, J = 9.0 Hz, 1H); 2.60 (dq, J = 18.4, 7.4 Hz, 1H); 2.44 (dq, J = 18.3, 7.3 Hz, 1H); 1.64 (d, J = 1.2, 3H); 1.41 (d, J = 1.2 Hz, 3H); 0.96 (t, J = 7.4 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 210.5, 138.3, 135.4, 134.0, 130.0, 127.8, 122.5, 77.2, 30.8, 25.7,

18.4, 7.3; IR (neat) 2974, 2917, 1731, 1618, 1428, 1156, 1115, 710, 698, 506; HRMS (ESI+) for $C_{26}H_{28}O_2Si$ [M+H]: calculated: 401.1905, found: 401.1920.




**(*E*)-6,10-dimethyl-4-(triphenylsilyloxy)undeca-5,9-dien-3-one (Compound S-17):** Purified on $SiO_2$ (gradient 0-5% diethyl ether/hexanes, stain in CAM) to afford an inseperable 2:1 mixture of the *E*:*Z* isomers (see spectra) as a colorless oil. $^1$H NMR (500 MHz, $C_6D_6$) δ 7.81-7.73 (m, 6H); 7.20-7.14 (m, 9H); 5.36 (bd, J = 8.6 Hz, 1H); 5.16 (d, J = 8.6 Hz, 1H); 5.07-5.02 (m, 1H); 2.55 (dq, J = 18.1, 7.3 Hz, 1H); 2.30 (dq, J = 18.1, 7.3 Hz, 1H); 1.99-1.91 (m, 2H); 1.89-1.81 (m, 2H); 1.61 (s, 3H); 1.48 (s, 3H); 1.39 (s, 3H); 0.96 (t, J = 7.4 Hz, 3H); $^{13}$C NMR (125 MHz, $C_6D_6$) δ 207.9, 141.0, 135.5, 134.3, 131.2, 130.0, 127.9, 124.0, 123.0, 77.6, 39.4, 30.4, 26.1, 25.4, 17.4, 16.6, 7.4; IR (neat) 2918, 1731, 1428, 1156, 834, 710, 506; LRMS (ESI+) for $C_{31}H_{36}O_2Si$ [M+H]: calculated: 469.2, found: 469.2.




**5-methyl-1,3-diphenylhexane-1,4-dione (Compound S-18):** Purified on $SiO_2$ (15% diethyl ether/hexanes, stain in CAM) to afford a colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.89-7.83 (m, 2H); 7.46 (tt, J = 6.8, 1.5 Hz, 1H); 7.37-7.34 (m, 2H); 7.28-7.25 (m, 2H); 7.21-7.18 (m, 3H); 4.52 (dd, J = 10.2, 3.7 Hz, 1H); 3.95 (dd, J = 18.1, 10.3 Hz, 1H); 3.00 (dd, J = 18.0, 3.6 Hz, 1H); 2.72 (sept, J = 7.1 Hz, 1H); 1.16 (d, J = 7.0 Hz, 3H); 0.84 (dd, J = 6.8 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 213.0, 198.1, 138.3, 136.5, 133.1, 129.0, 128.5, 128.3, 128.0, 127.4, 51.8, 42.5, 39.8, 19.2, 18.3; IR (neat) 2966, 2901, 1711, 1685, 1598, 1493, 1449, 1239, 1017, 753, 699; LRMS (ESI+) for $C_{19}H_{20}O_2$ [M+H]: calculated: 281.3, found: 281.2.

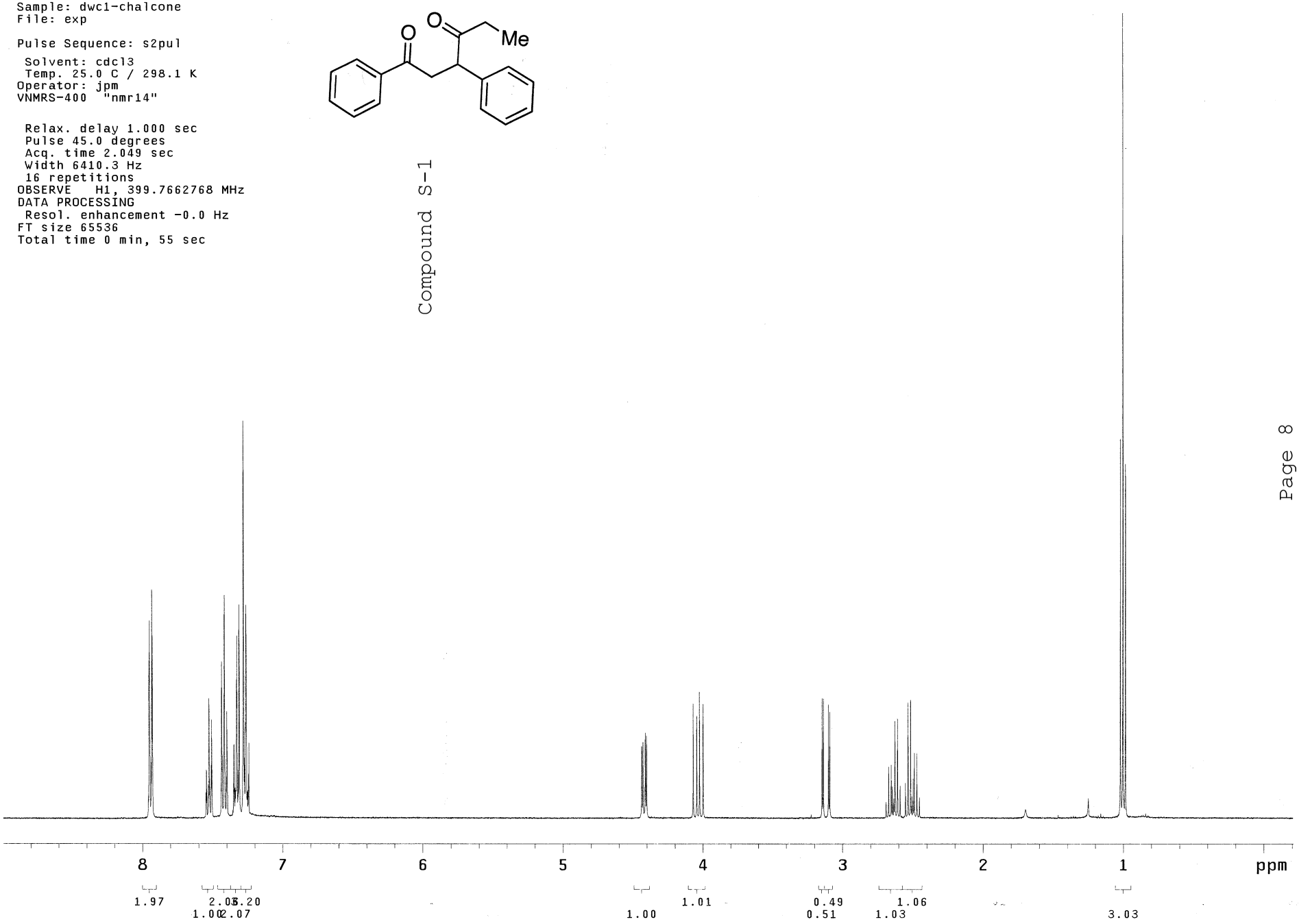
Sample: dwc1-chalcone
File: exp

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7662768 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 55 sec



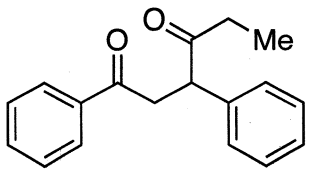

Compound S-1



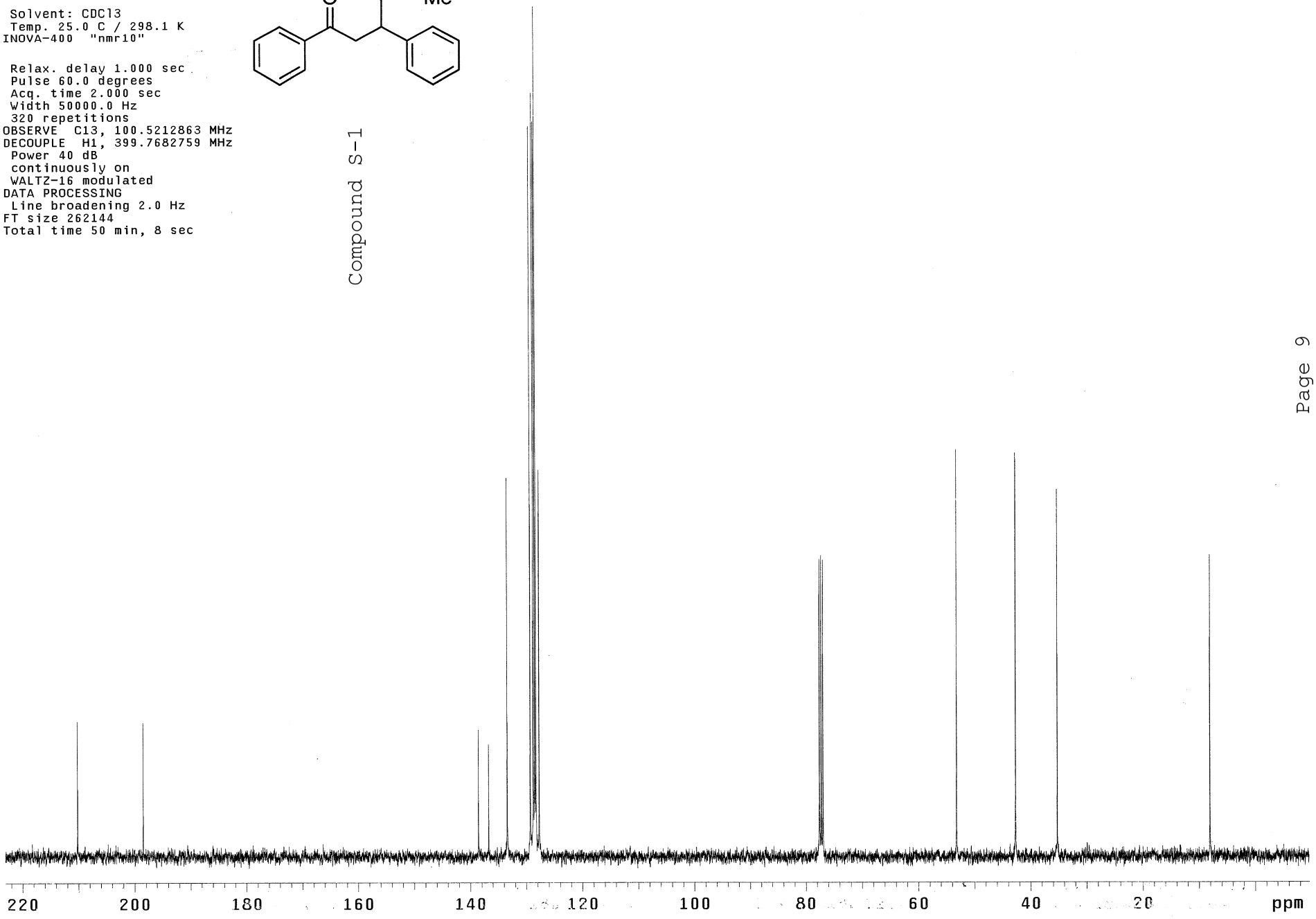
dwc1-296-1C

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "nmr10"

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.000 sec
Width 50000.0 Hz
320 repetitions
OBSERVE C13, 100.5212863 MHz
DECOUPLE H1, 399.7682759 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 262144
Total time 50 min, 8 sec




Compound S-1



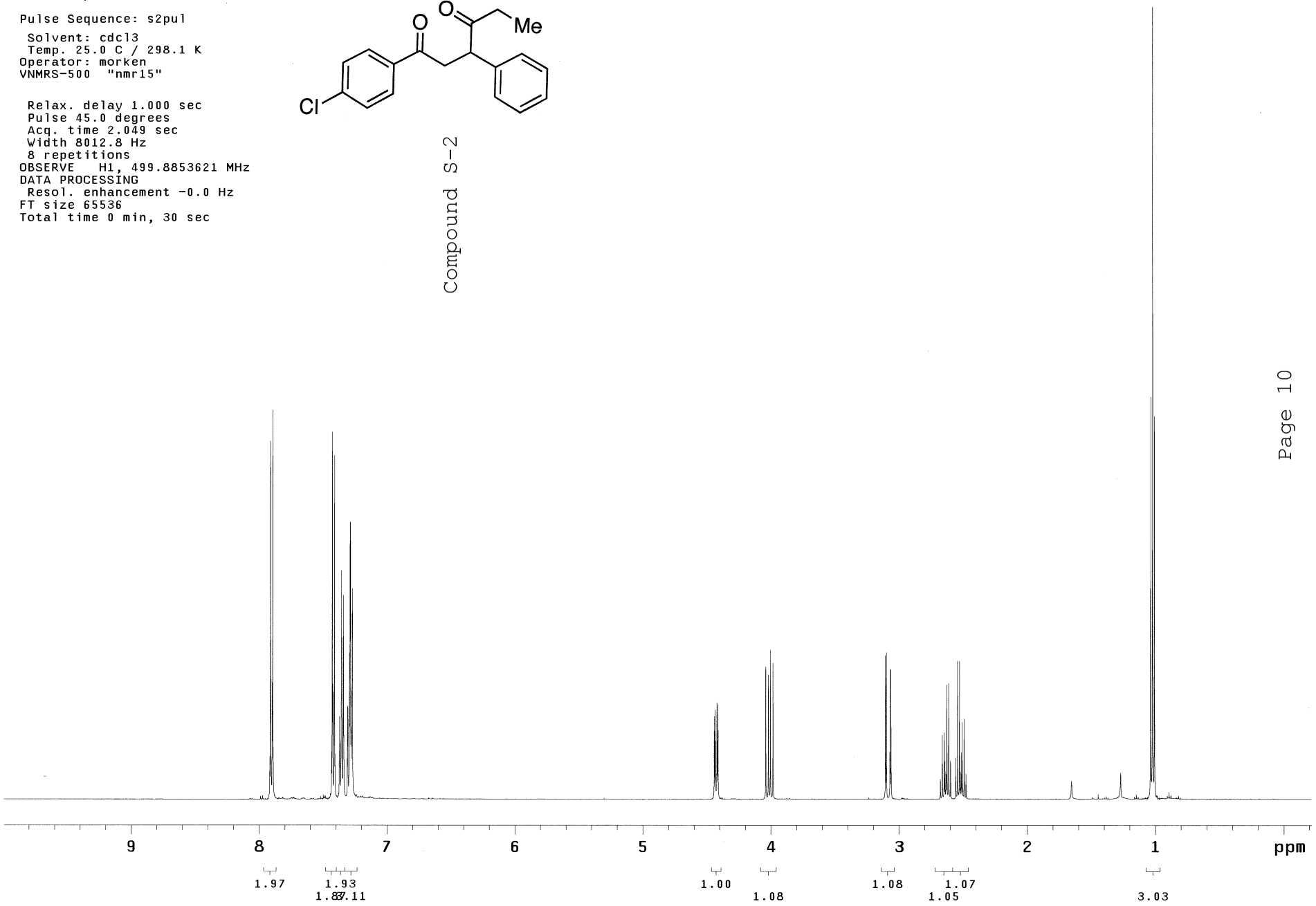
Sample: dwc1-258-pureH
File: exp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: morken
VNMRS-500 "nmr15"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 8012.8 Hz
 8 repetitions
OBSERVE H1, 499.8853621 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec



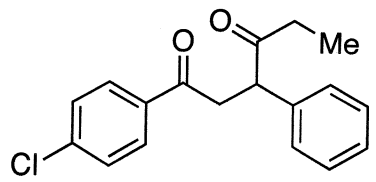

Compound S-2



Sample: dwc1-258-pureC
File: exp

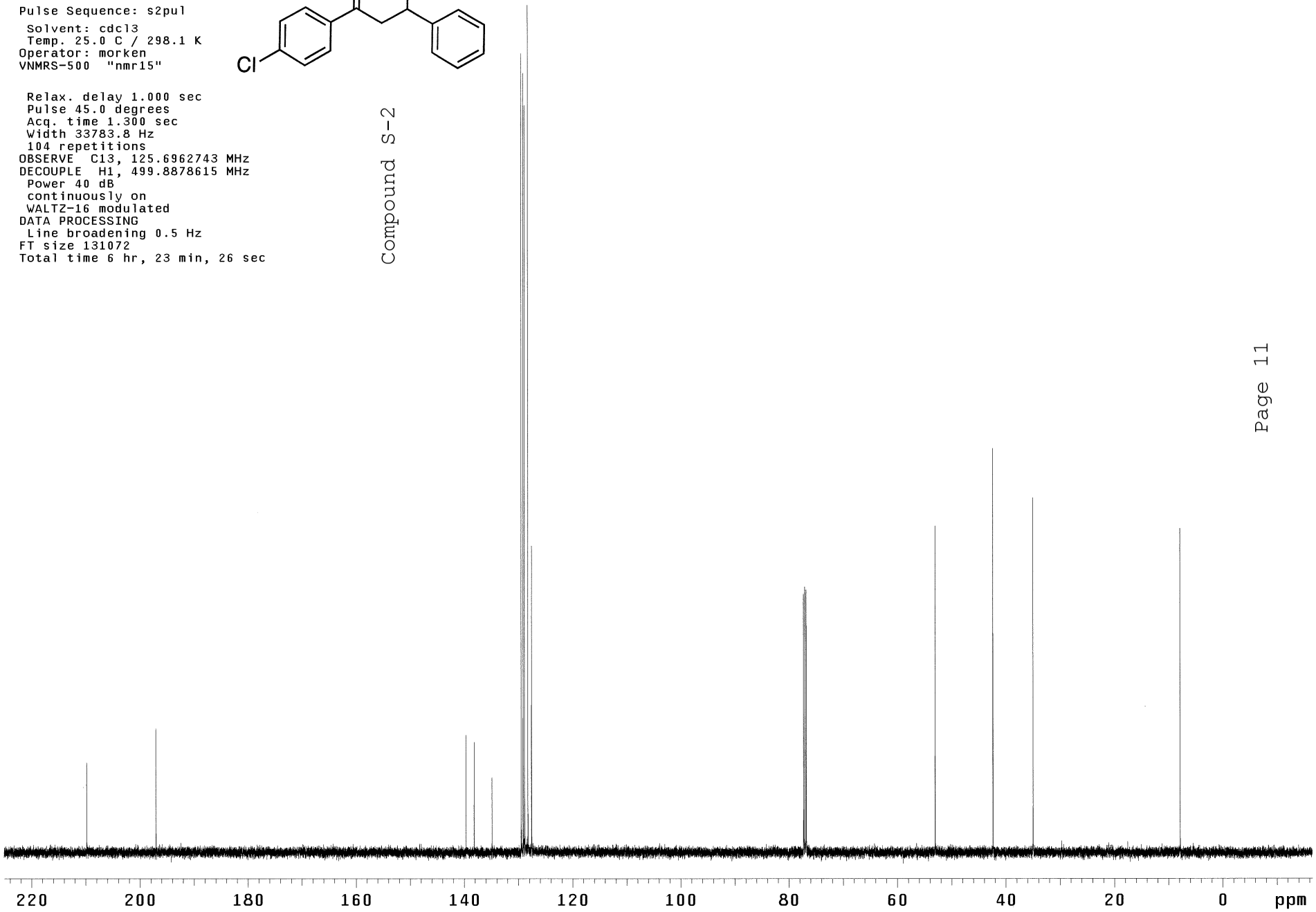
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: morken
VNMRS-500 "nmr15"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 33783.8 Hz
 104 repetitions
OBSERVE C13, 125.6962743 MHz
DECOUPLE H1, 499.8878615 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 23 min, 26 sec




Compound S-2

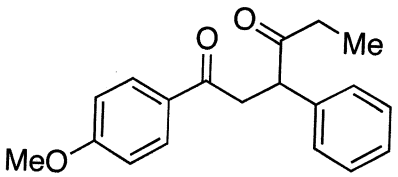




Sample: dwc1-260-pureH
File: exp

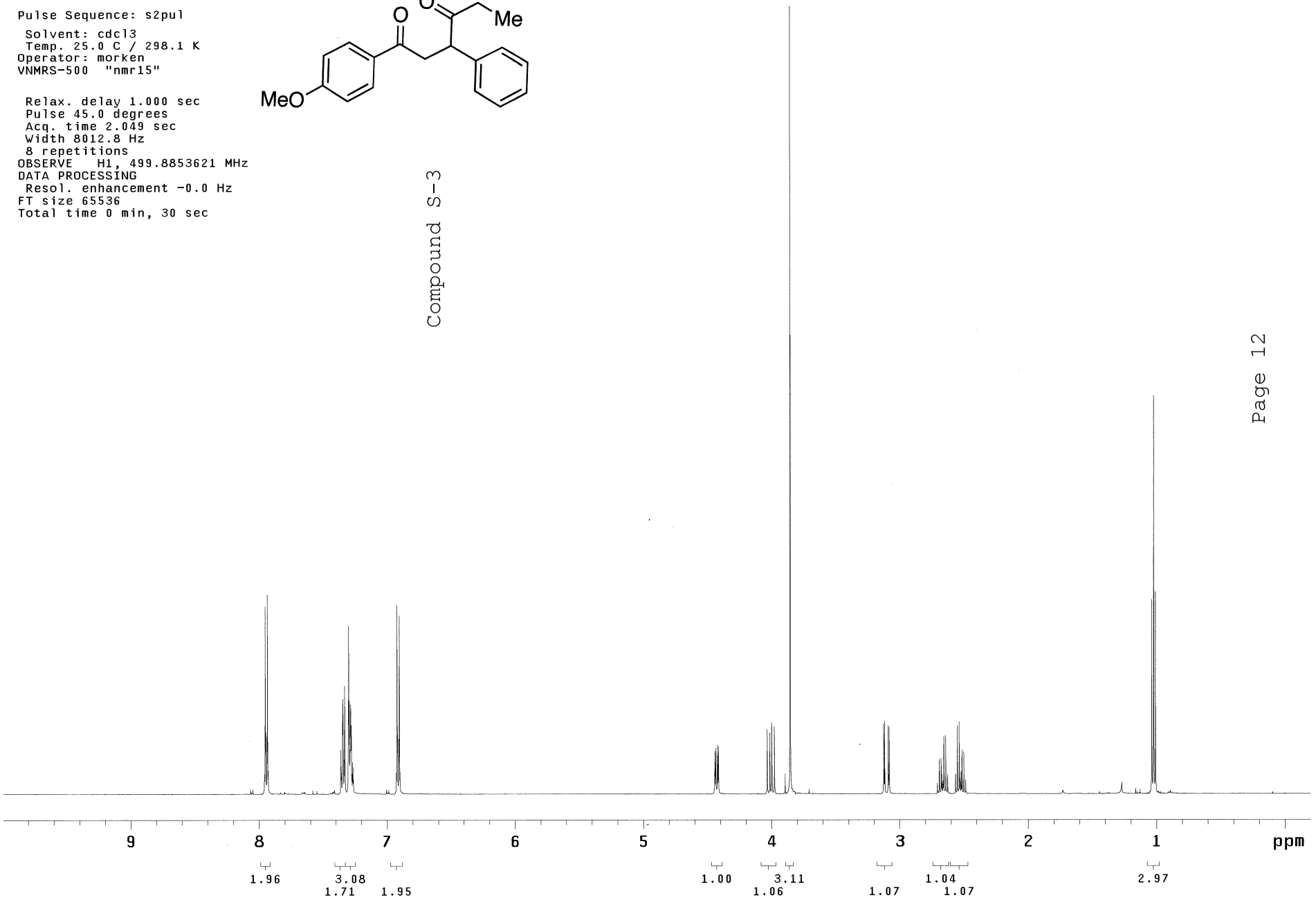
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: morken
VNMRS-500 "nmr15"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 8012.8 Hz
 8 repetitions
OBSERVE H1, 499.8853621 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec




Compound S-3

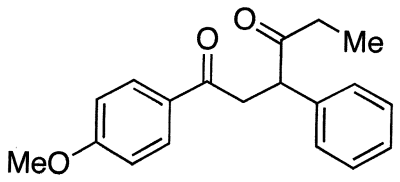




Sample: dwc1-260-pureC
File: exp

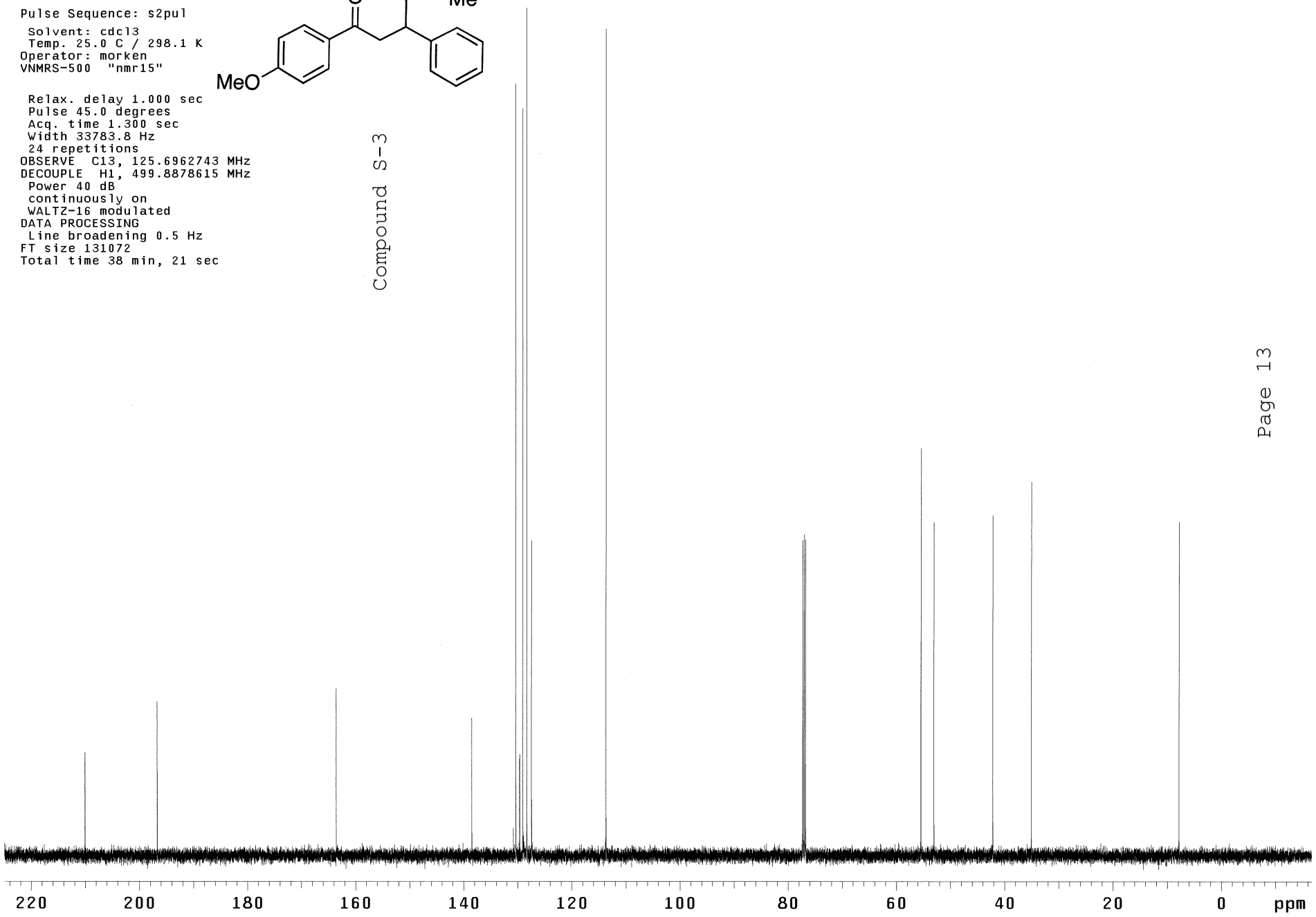
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: morken
VNMRS-500  "nmr15"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 33783.8 Hz
 24 repetitions
OBSERVE  C13, 125.6962743 MHz
DECOUPLE  H1, 499.8878615 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 21 sec




Compound S-3

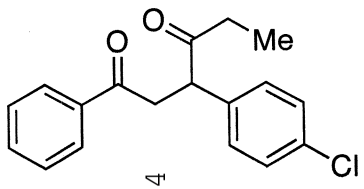




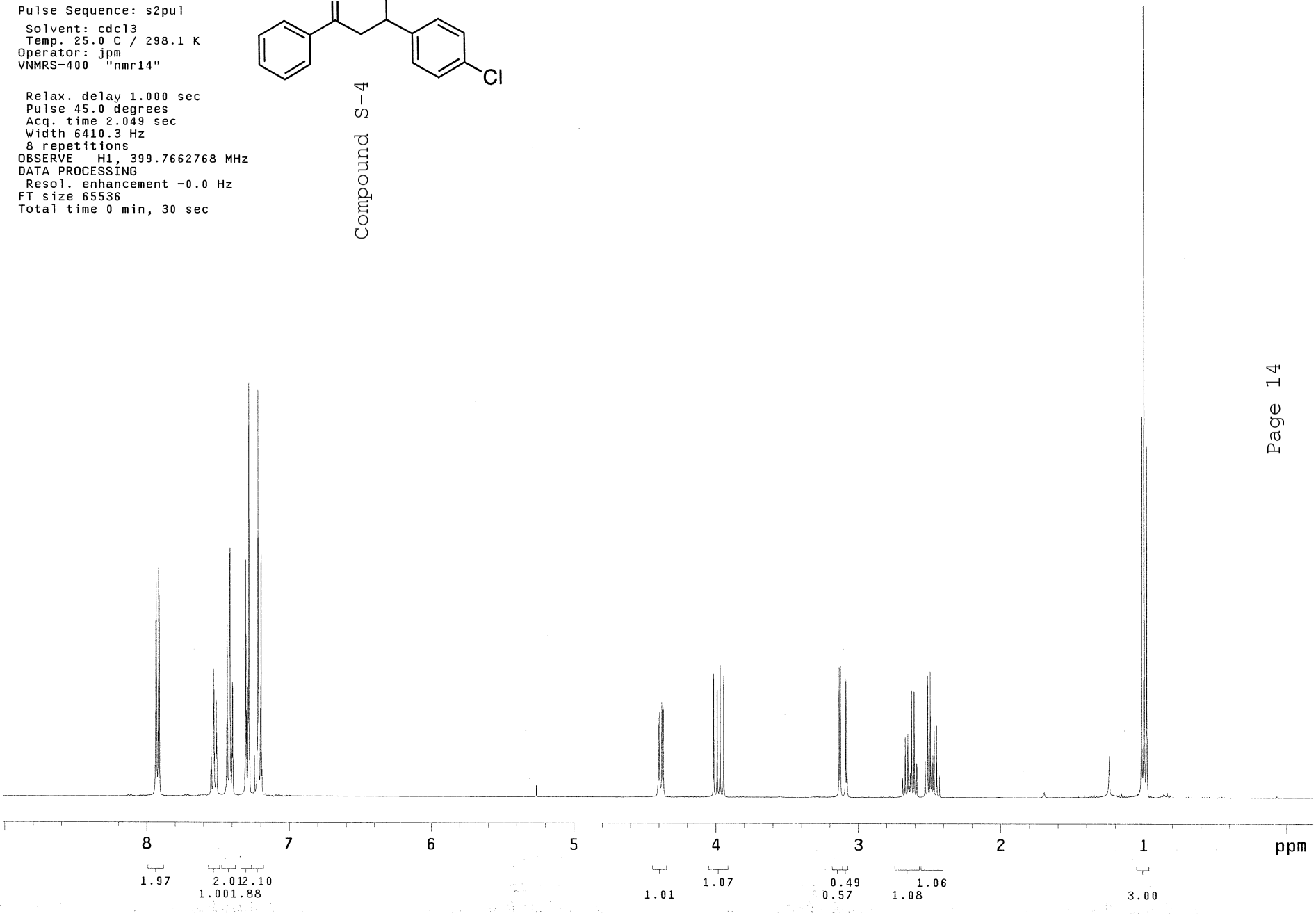
Sample: dwc1-4Cl-chalcone
File: exp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE H1, 399.7662768 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec



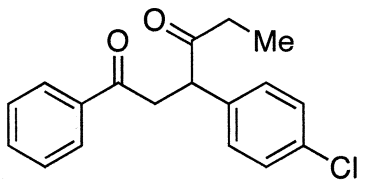

Compound S-4



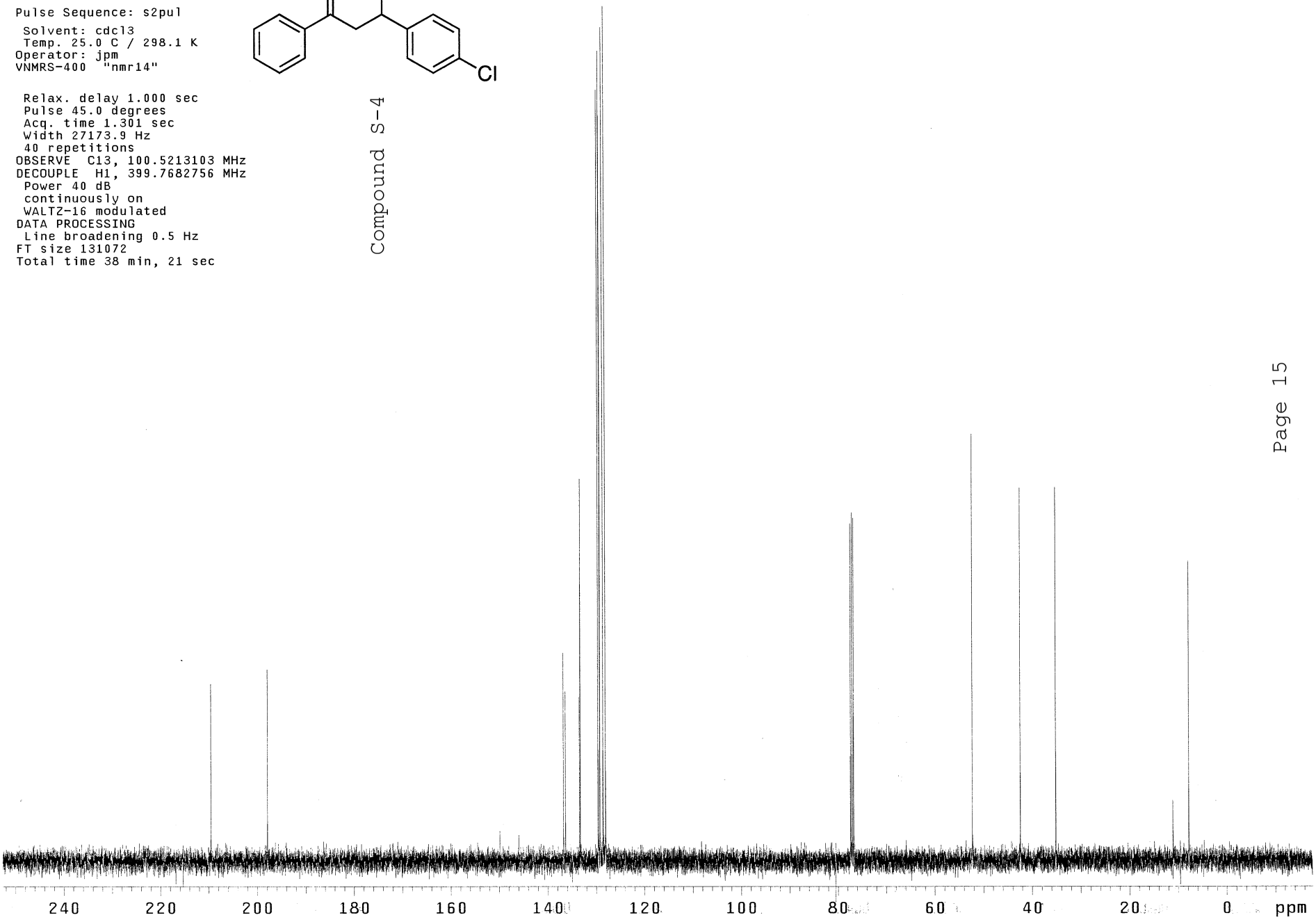
Sample: dwc1-4Cl-chalconeC
File: exp

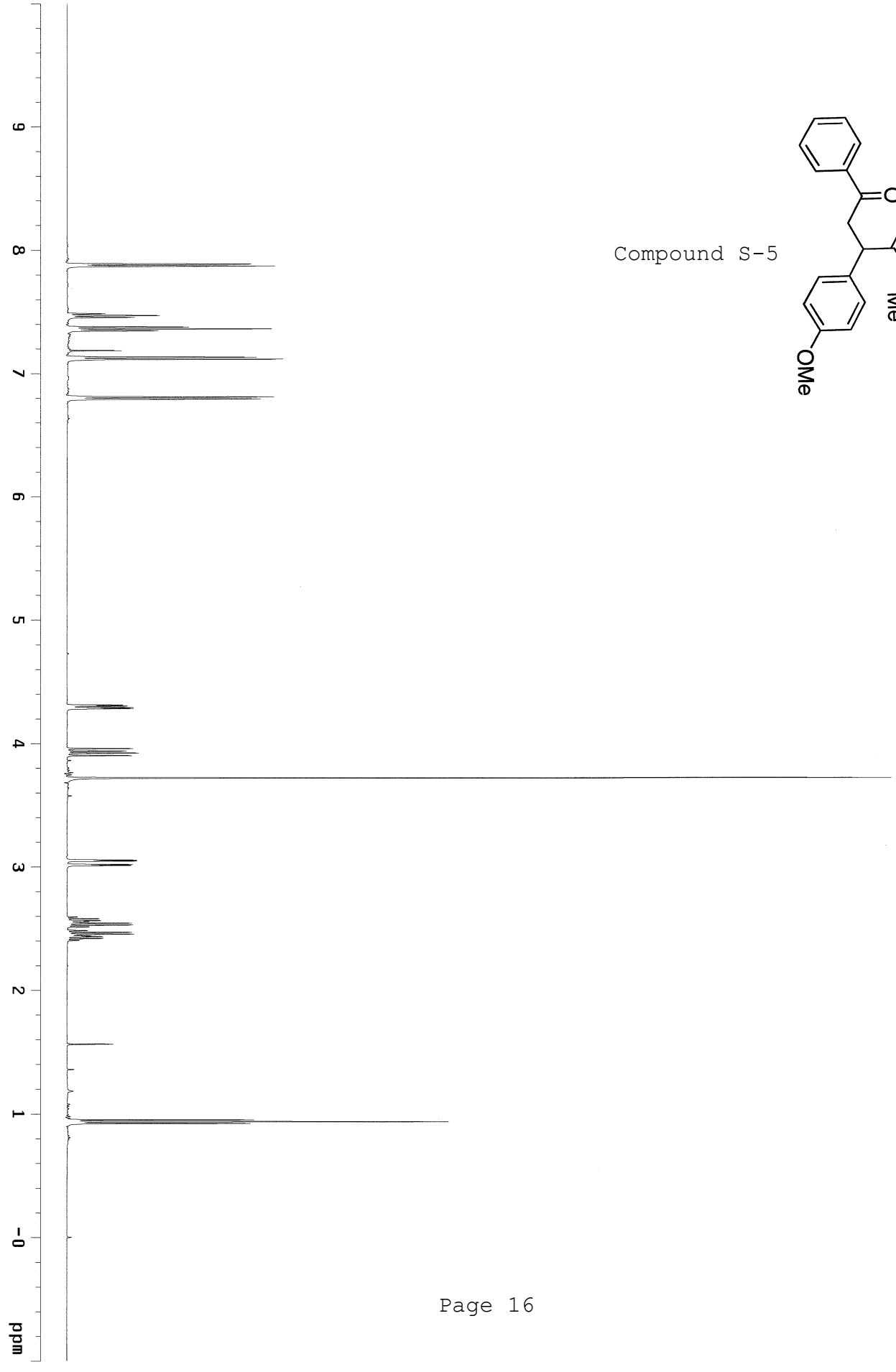
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.301 sec
 Width 27173.9 Hz
 40 repetitions
OBSERVE C13, 100.5213103 MHz
DECOUPLE H1, 399.7682756 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 21 sec

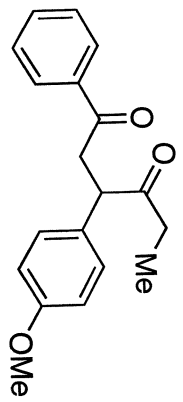


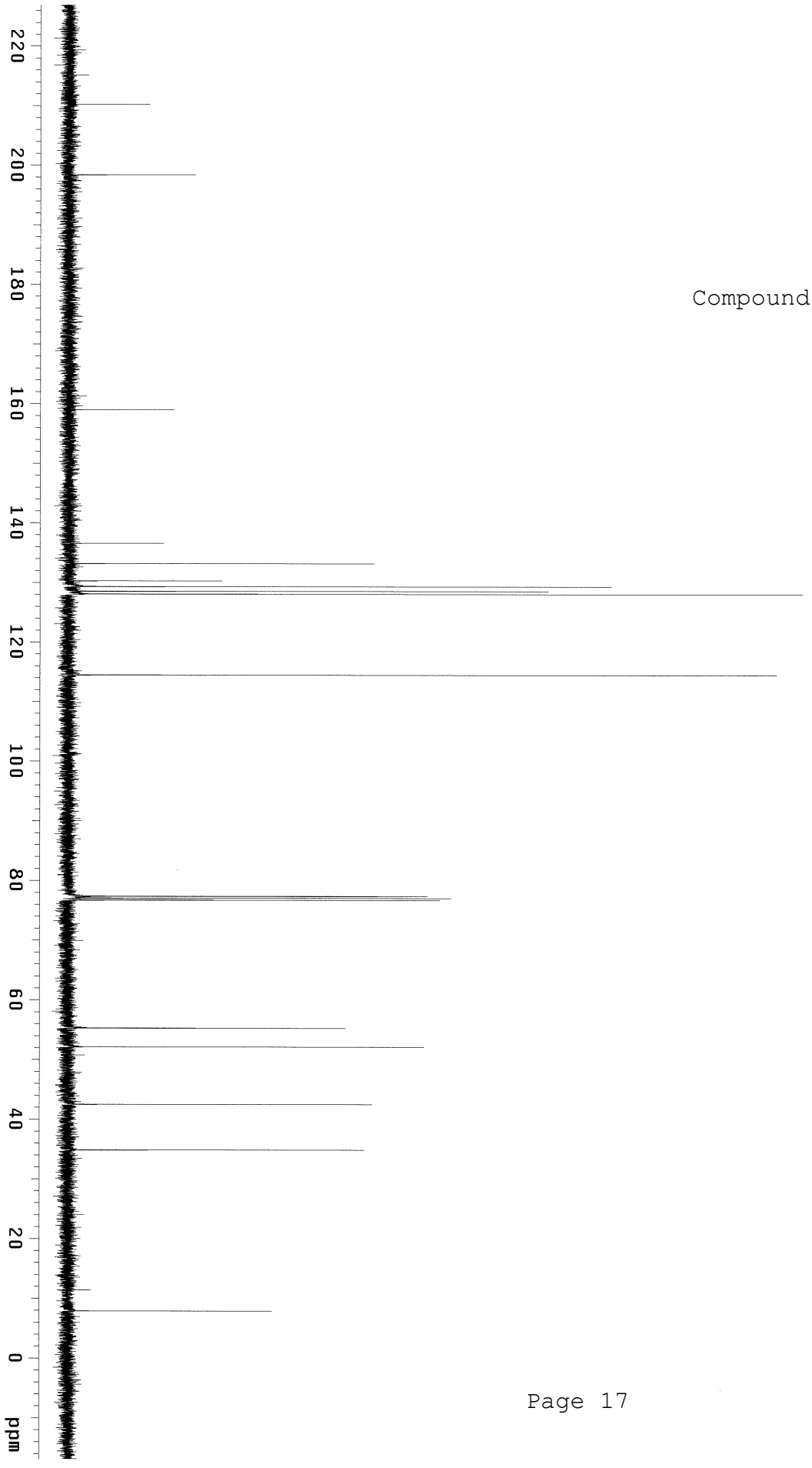

Compound S-4



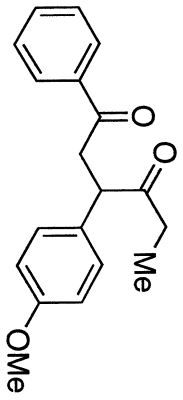



Compound S-5





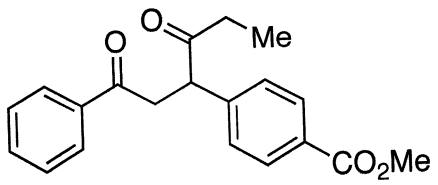

Compound S-5



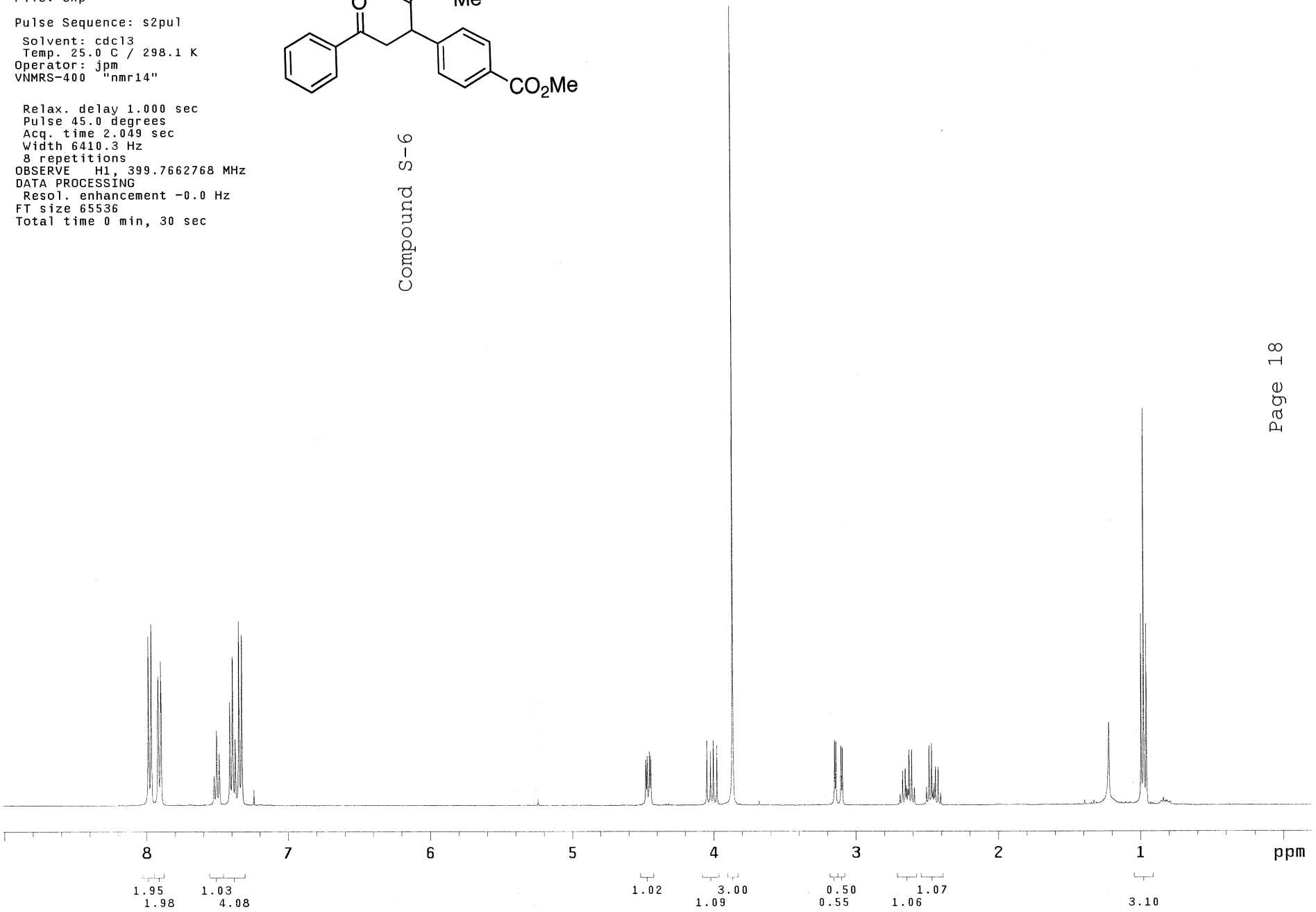
Sample: dwc1-ester-chalcone
File: exp

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7662768 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec



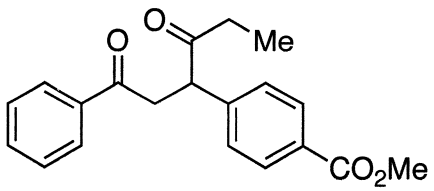

Compound S-6



Sample: dwc1-ester-cha
File: exp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1
Operator: jpm
VNMRS-400 "nmr14"

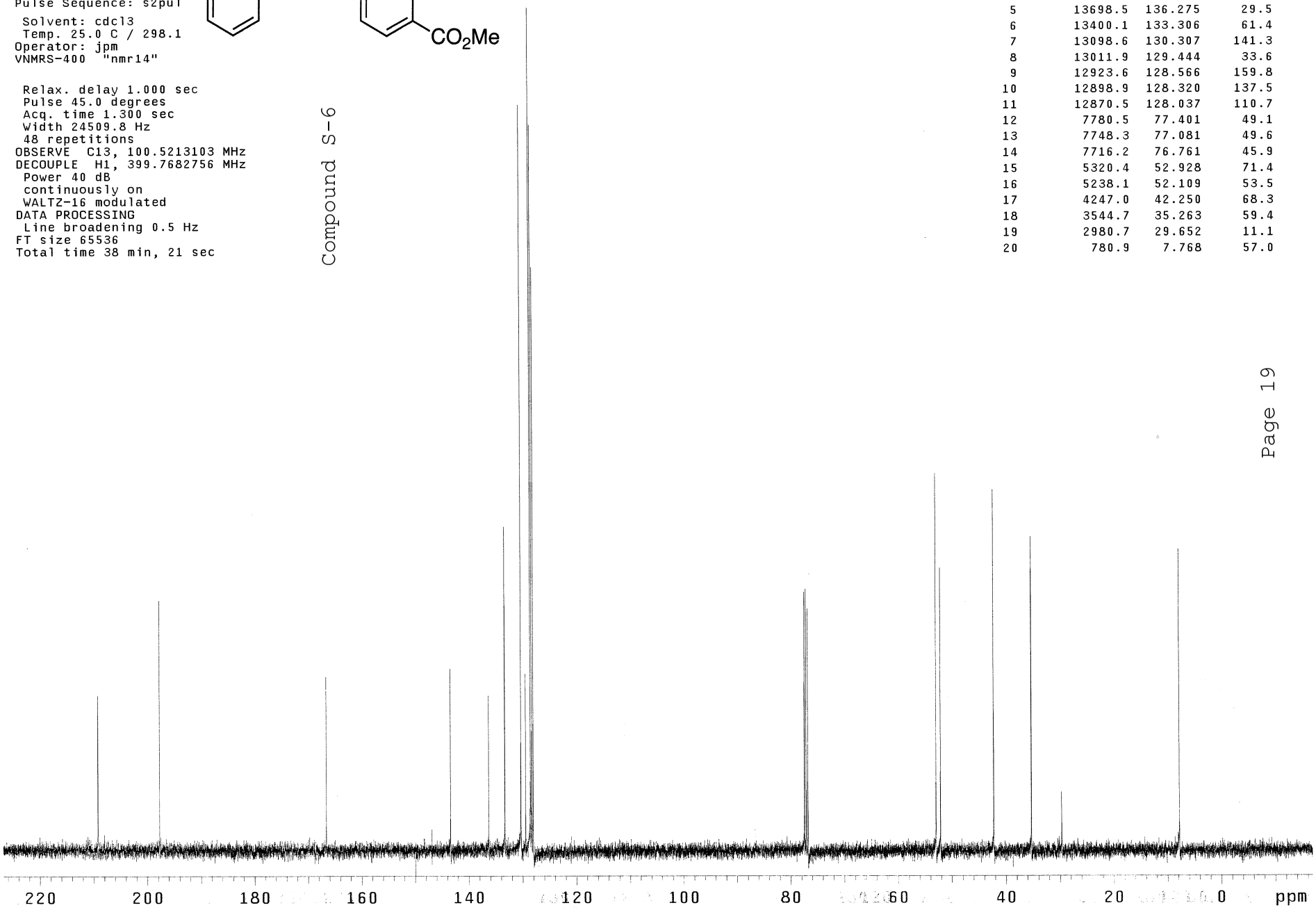
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 48 repetitions
OBSERVE C13, 100.5213103 MHz
DECOUPLE H1, 399.7682756 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec




Compound S-6

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 21028.0 | 209.189 | 29.6 |
| 2 | 19874.6 | 197.715 | 47.5 |
| 3 | 16745.8 | 166.589 | 33.1 |
| 4 | 14414.3 | 143.396 | 34.6 |
| 5 | 13698.5 | 136.275 | 29.5 |
| 6 | 13400.1 | 133.306 | 61.4 |
| 7 | 13098.6 | 130.307 | 141.3 |
| 8 | 13011.9 | 129.444 | 33.6 |
| 9 | 12923.6 | 128.566 | 159.8 |
| 10 | 12898.9 | 128.320 | 137.5 |
| 11 | 12870.5 | 128.037 | 110.7 |
| 12 | 7780.5 | 77.401 | 49.1 |
| 13 | 7748.3 | 77.081 | 49.6 |
| 14 | 7716.2 | 76.761 | 45.9 |
| 15 | 5320.4 | 52.928 | 71.4 |
| 16 | 5238.1 | 52.109 | 53.5 |
| 17 | 4247.0 | 42.250 | 68.3 |
| 18 | 3544.7 | 35.263 | 59.4 |
| 19 | 2980.7 | 29.652 | 11.1 |
| 20 | 780.9 | 7.768 | 57.0 |





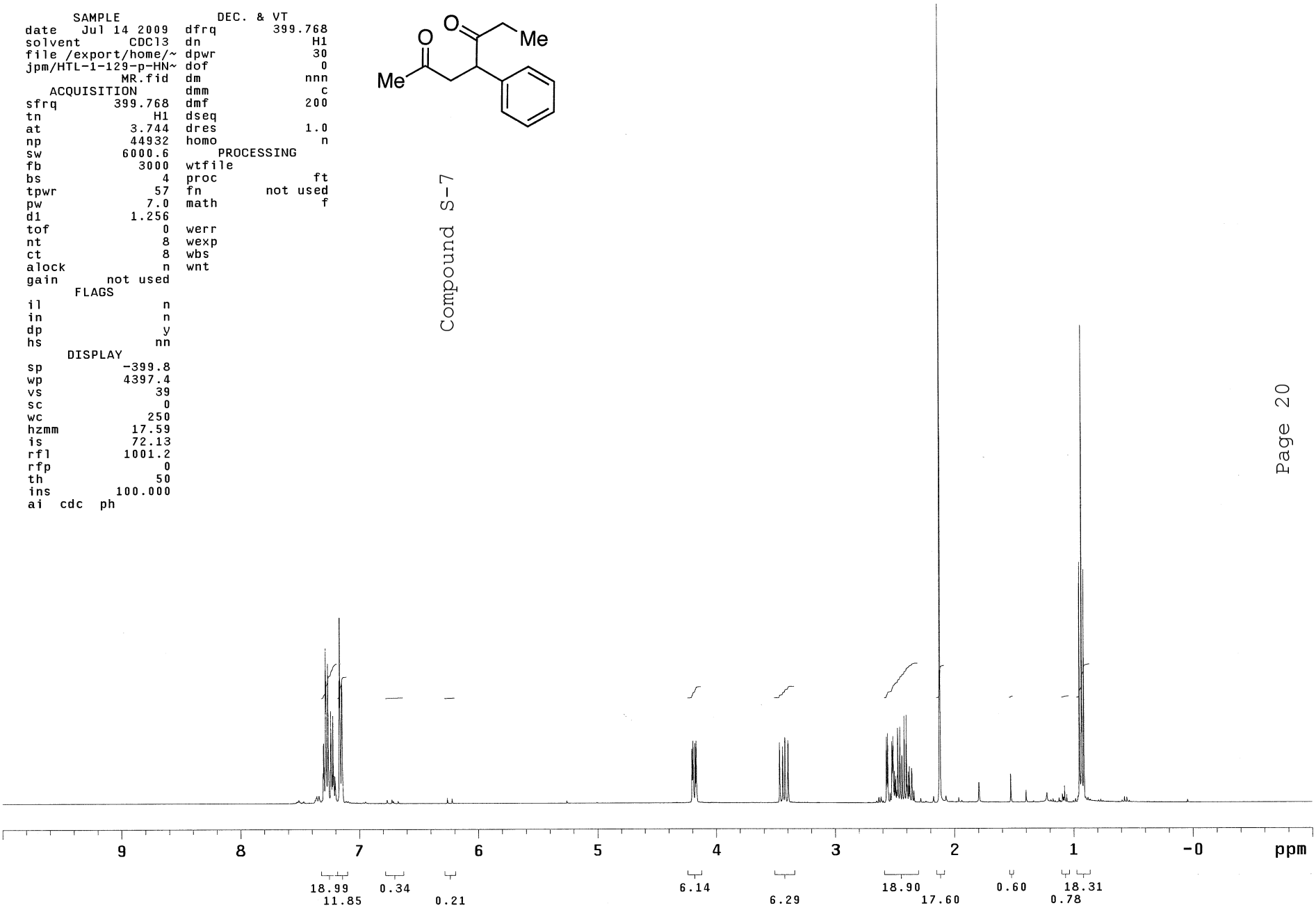
HTL-1-129-p-HNMR

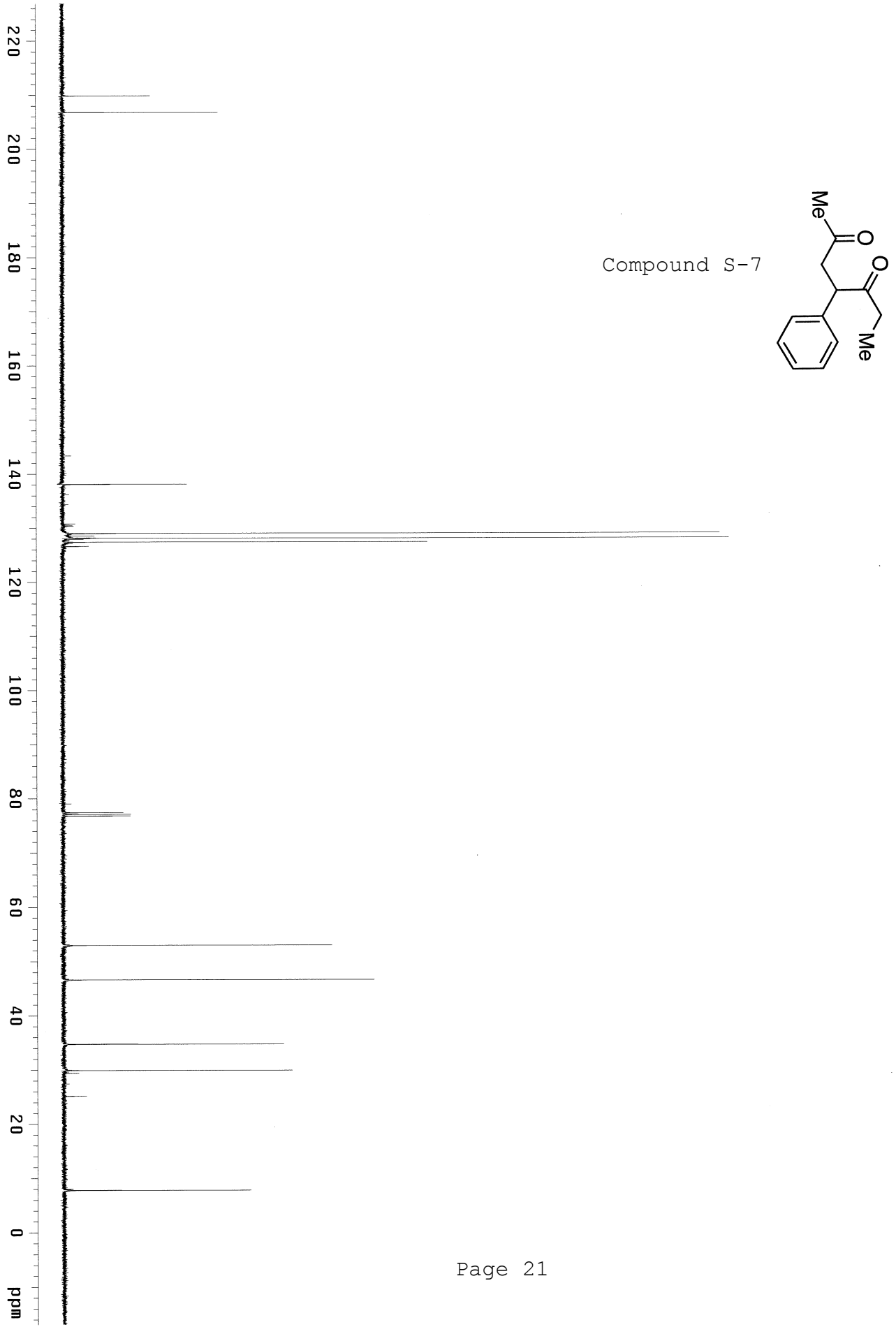
exp3 std1h

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Jul 14 2009 | dfrq | 399.768 |
| solvent | CDCl3 | dn | H1 |
| file | /export/home/~ | dpwr | 30 |
| | jpm/HTL-1-129-p-HN~ | dof | 0 |
| | MR.fid | dm | nnn |
| ACQUISITION | | dmm | c |
| sfrq | 399.768 | dmf | 200 |
| tn | H1 | dseq | |
| at | 3.744 | dres | 1.0 |
| np | 44932 | homo | n |
| sw | 6000.6 | PROCESSING | |
| fb | 3000 | wtfile | |
| bs | 4 | proc | ft |
| tpwr | 57 | fn | not used |
| pw | 7.0 | math | f |
| d1 | 1.256 | | |
| tof | 0 | werr | |
| nt | 8 | wexp | |
| ct | 8 | wbs | |
| alock | n | wnt | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | -399.8 | | |
| wp | 4397.4 | | |
| vs | 39 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 17.59 | | |
| is | 72.13 | | |
| rfl | 1001.2 | | |
| rfp | 0 | | |
| th | 50 | | |
| ins | 100.000 | | |
| ai cdc ph | | | |




Compound S-7

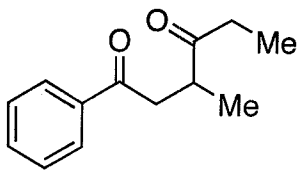





Compound S-7

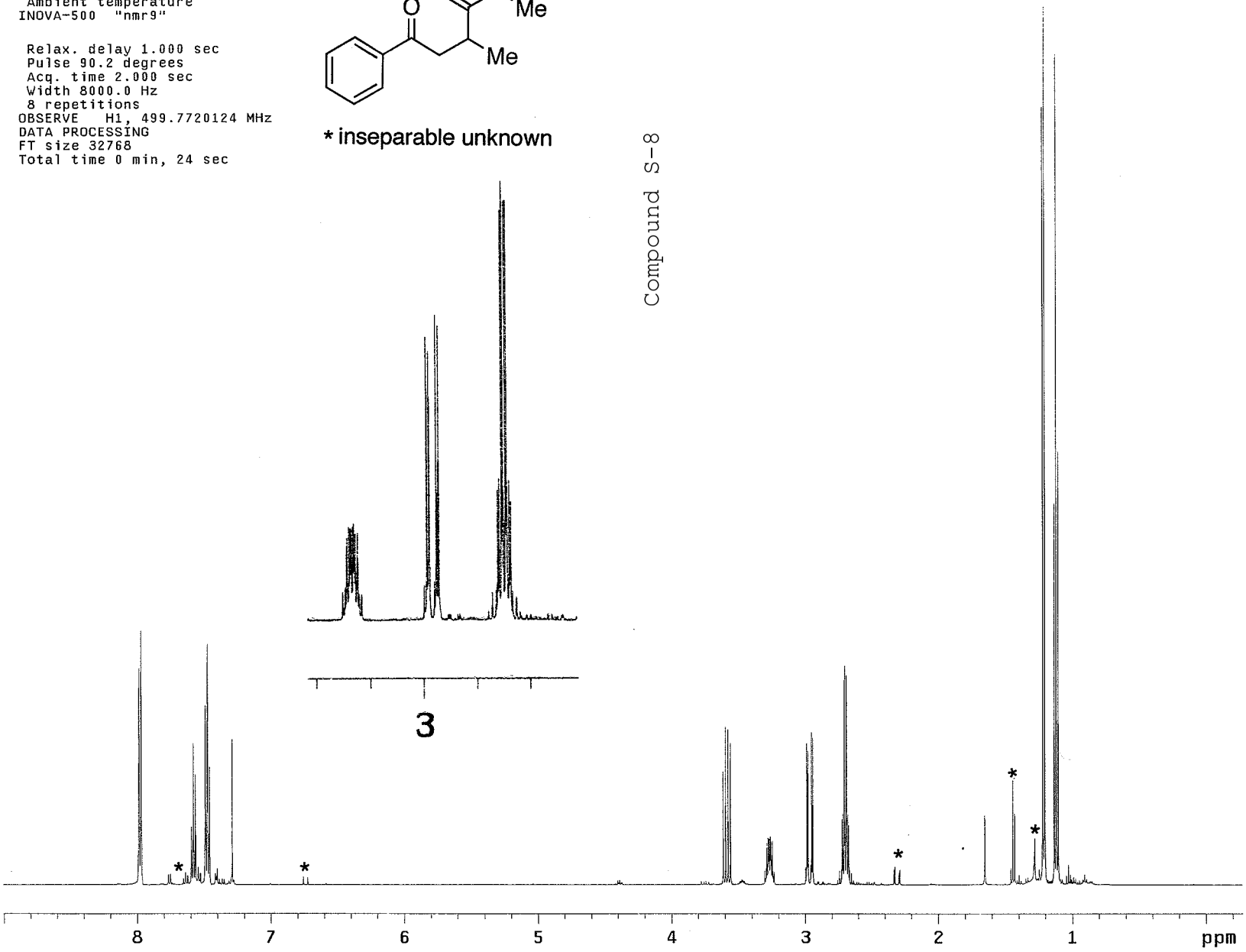
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr9"

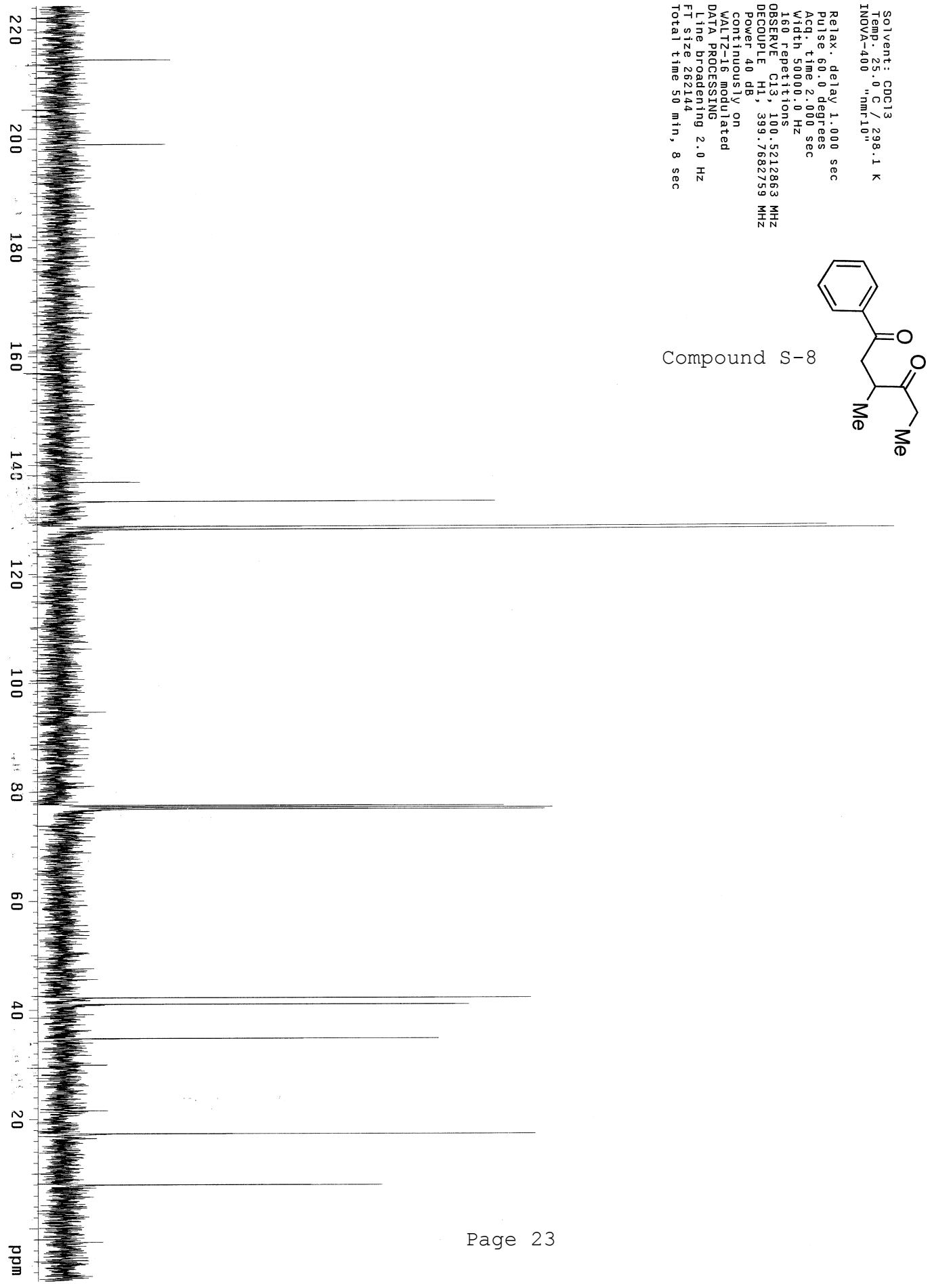
Relax. delay 1.000 sec
Pulse 90.2 degrees
Acq. time 2.000 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7720124 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec




* inseparable unknown

Compound S-8




Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "nmr10"
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.000 sec
Width 50000.0 Hz
160 repetitions
OBSERVE C13, 100.5212863 MHz
DECOUPLE H1, 399.7682759 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 262144
Total time 50 min, 8 sec
O
O
Me
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

Compound S-8

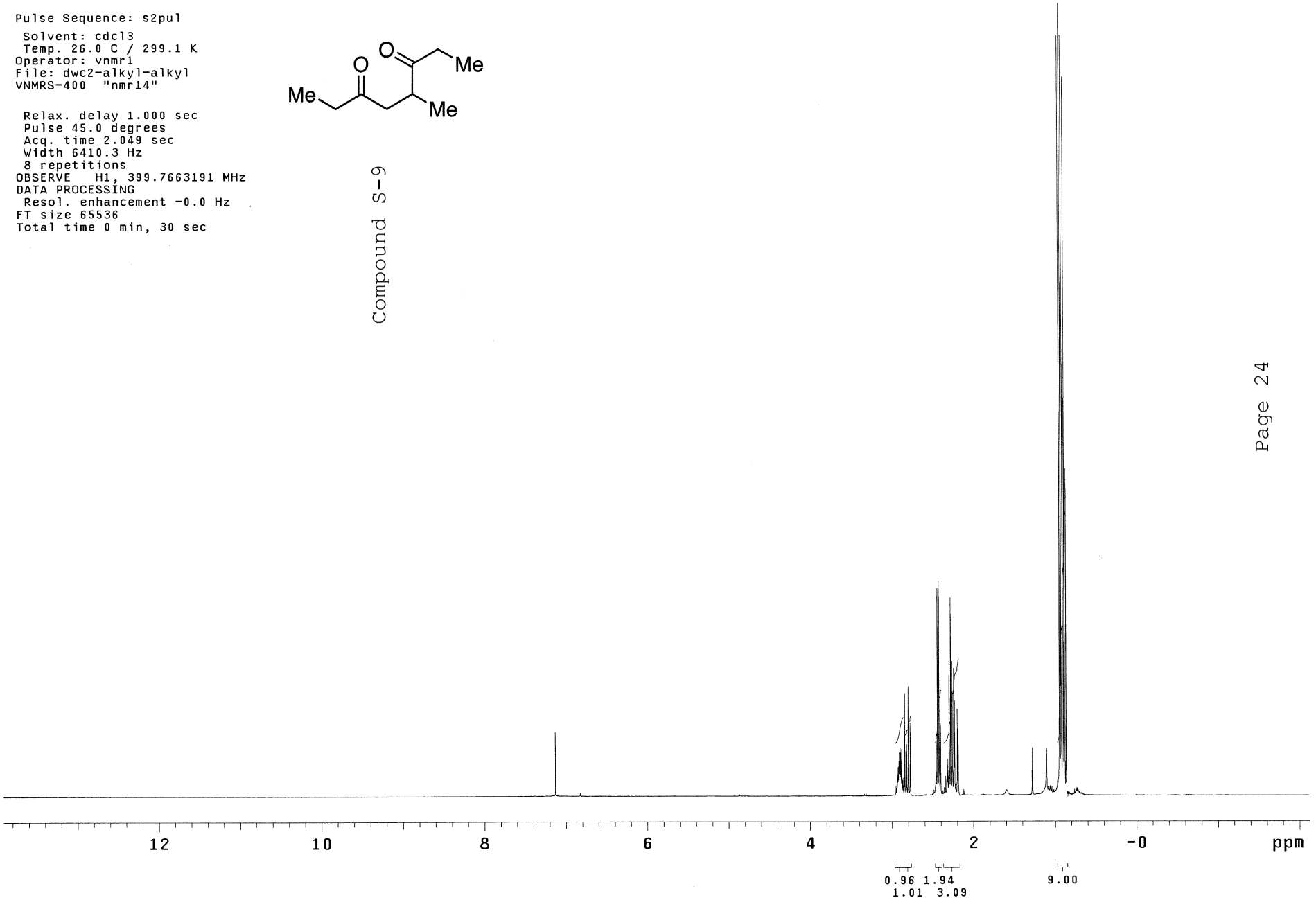
Sample: dwc2-alkyl-alkyl
File: /home/vnmr1/vnmrsys/data/Custar/dwc2-alkyl-alkyl.fid

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 26.0 C / 299.1 K
Operator: vnmr1
File: dwc2-alkyl-alkyl
VNMRS-400  "nmr14"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE   H1, 399.7663191 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec



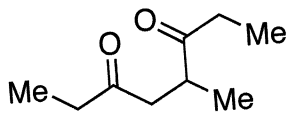

Compound S-9



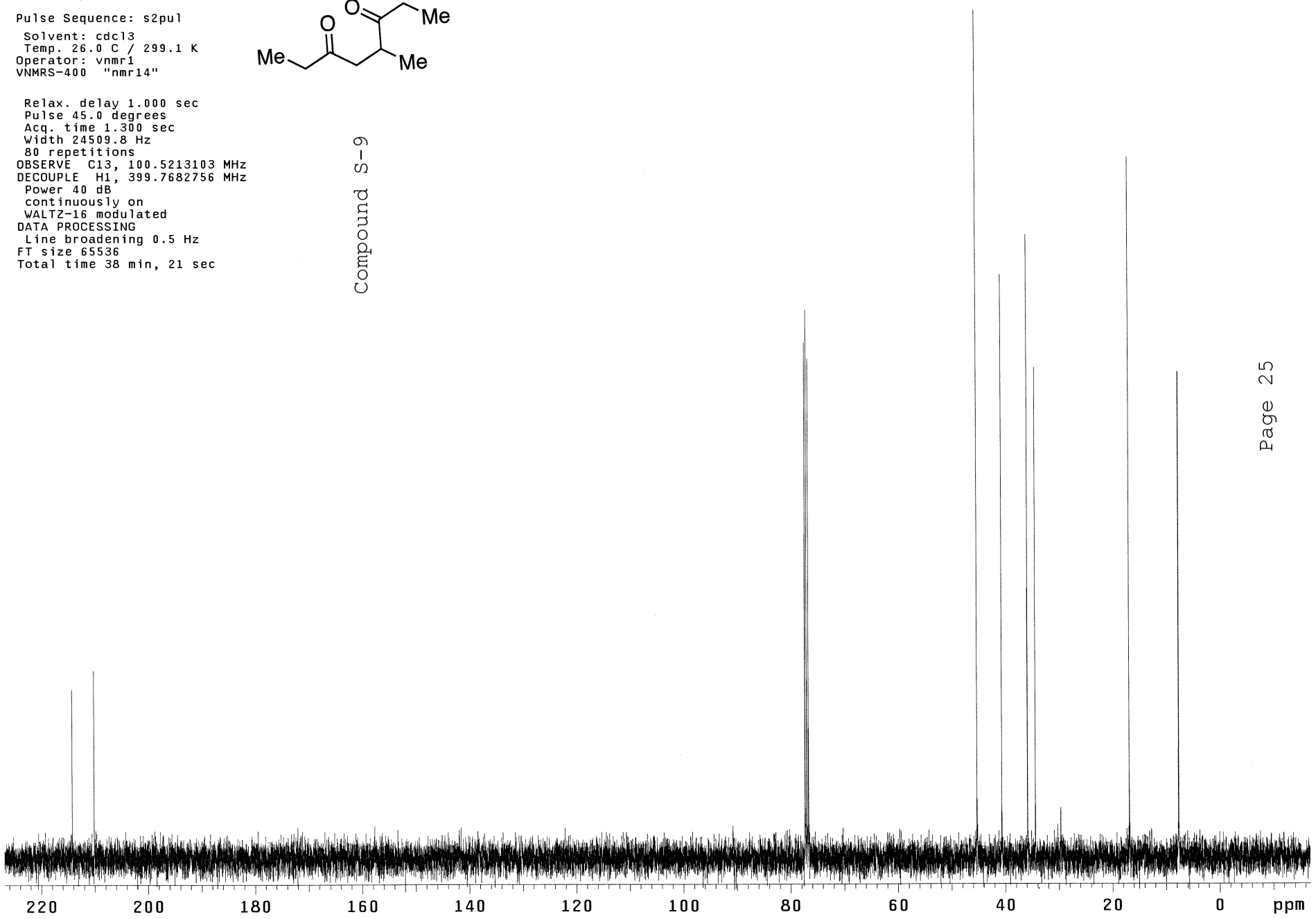
Sample: dwc2-alkyl-alkyl_C
File: exp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 26.0 C / 299.1 K
Operator: vnmr1
VNMRS-400  "nmr14"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 80 repetitions
OBSERVE  C13, 100.5213103 MHz
DECOUPLE  H1, 399.7682756 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec



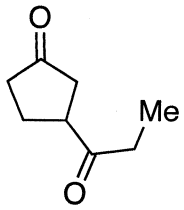

Compound S-9



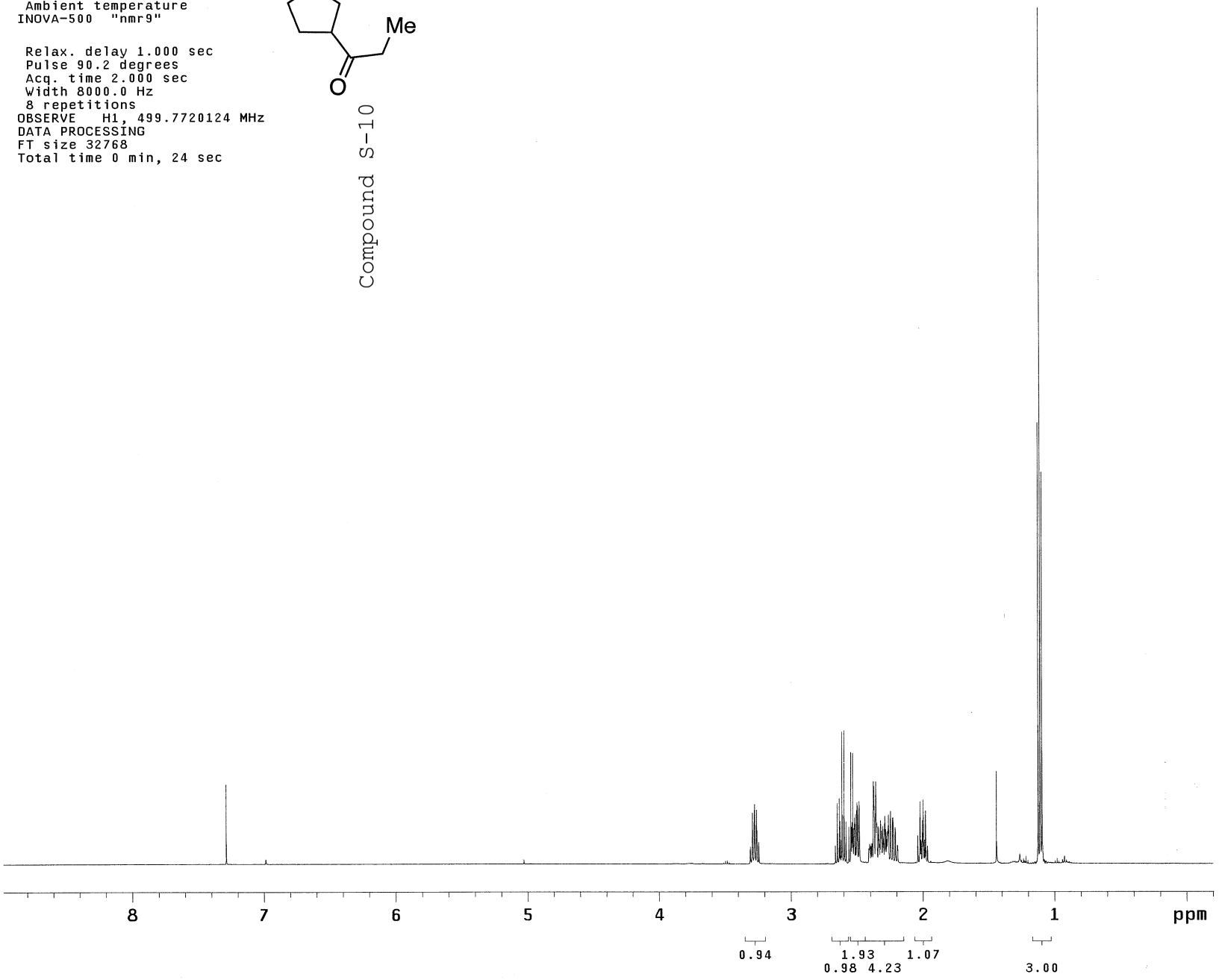
dwc1-291-1

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr9"

Relax. delay 1.000 sec
Pulse 90.2 degrees
Acq. time 2.000 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7720124 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec



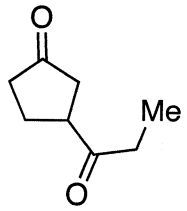

Compound S-10



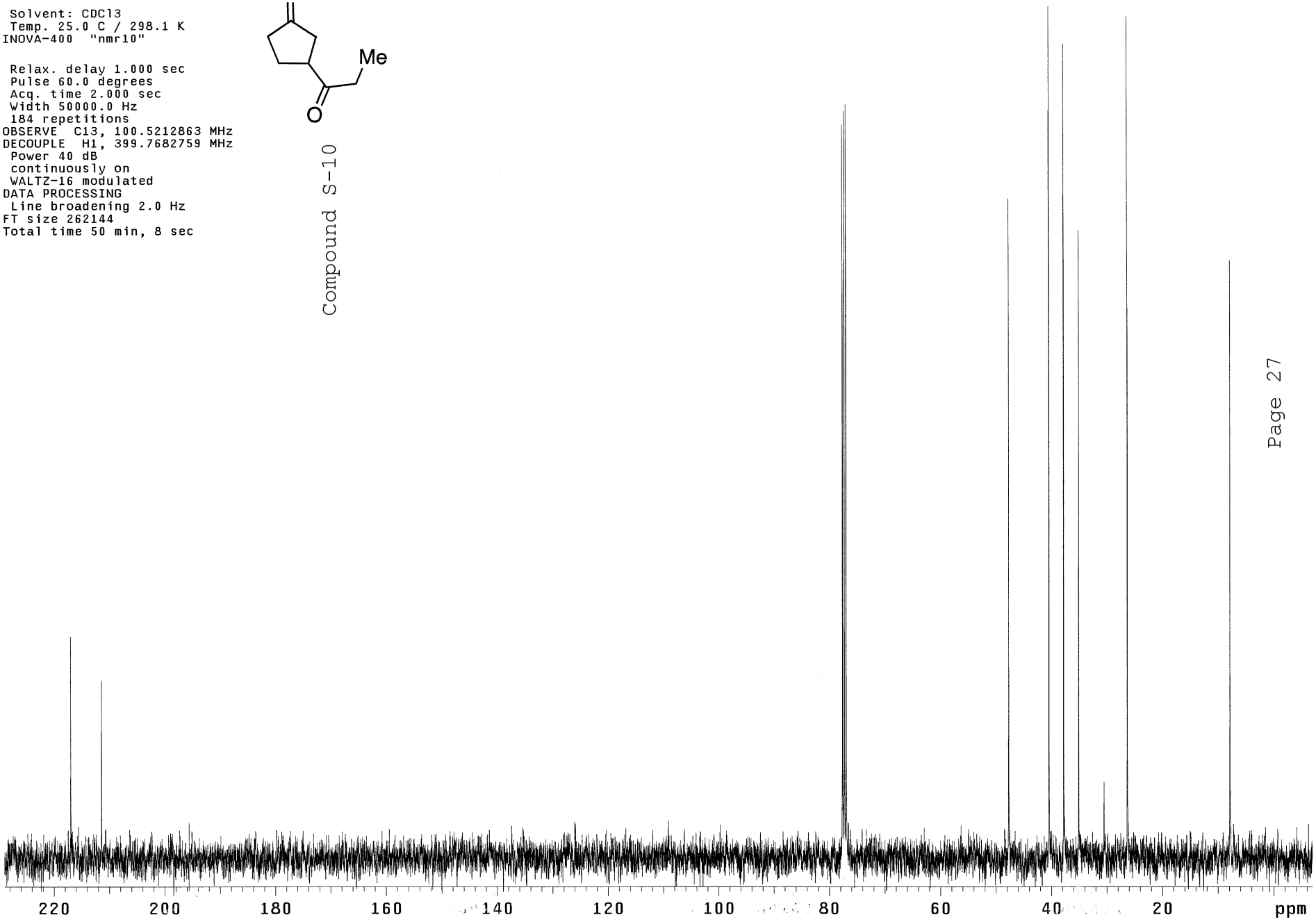
dwc1-291-1-C

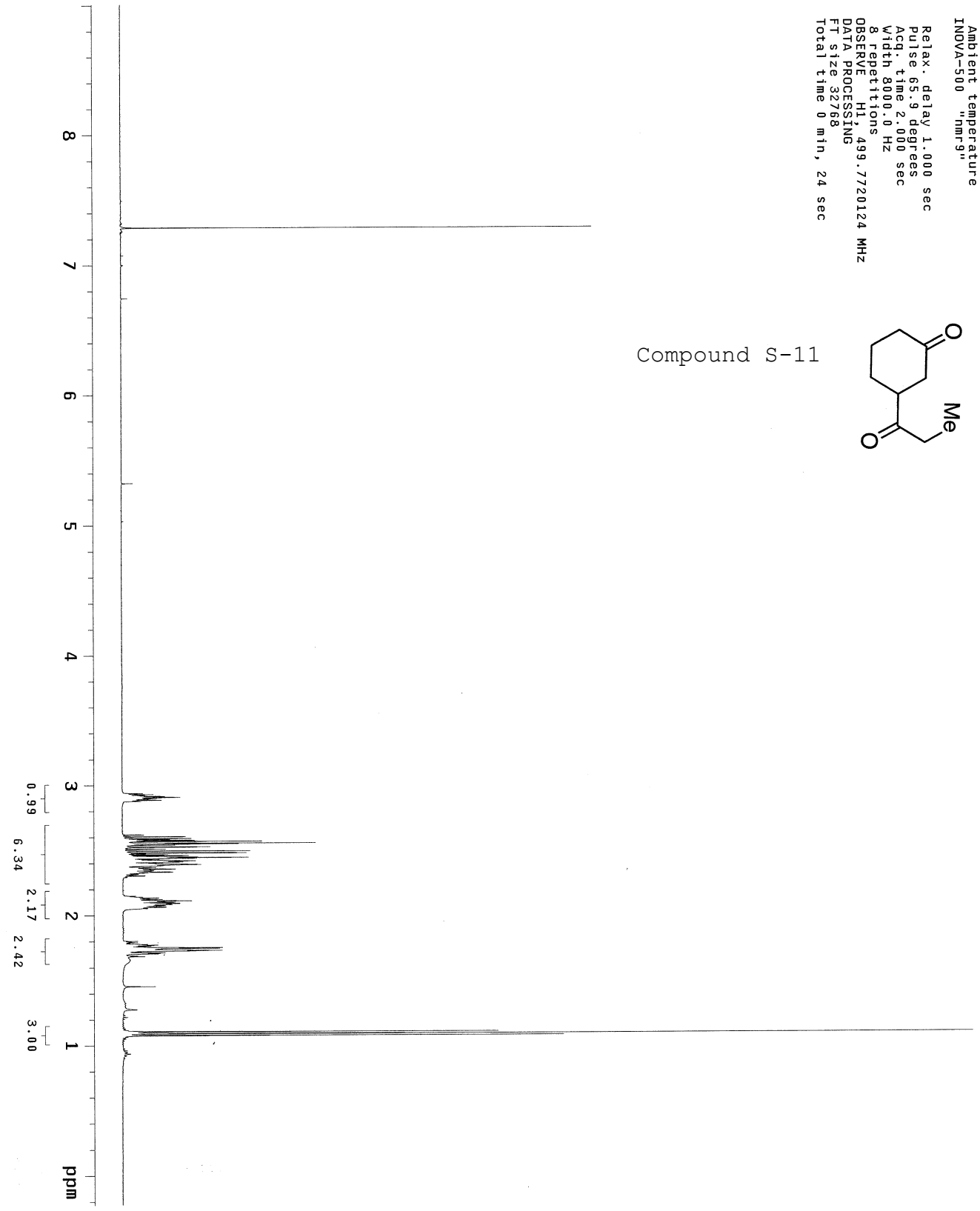
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "nmr10"

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.000 sec
Width 50000.0 Hz
184 repetitions
OBSERVE C13, 100.5212863 MHz
DECOUPLE H1, 399.7682759 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 262144
Total time 50 min, 8 sec




Compound S-10

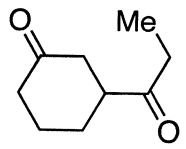



dwc1-cyclohexenone_1H
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr9"
Relax. delay 1.000 sec
Pulse 65.9 degrees
Acq. time 2.000 sec
width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7720124 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec
Compound S-11
O
Me
O
8
7
6
5
4
3
2
1
ppm
0.99
6.34
2.17
2.42
3.00

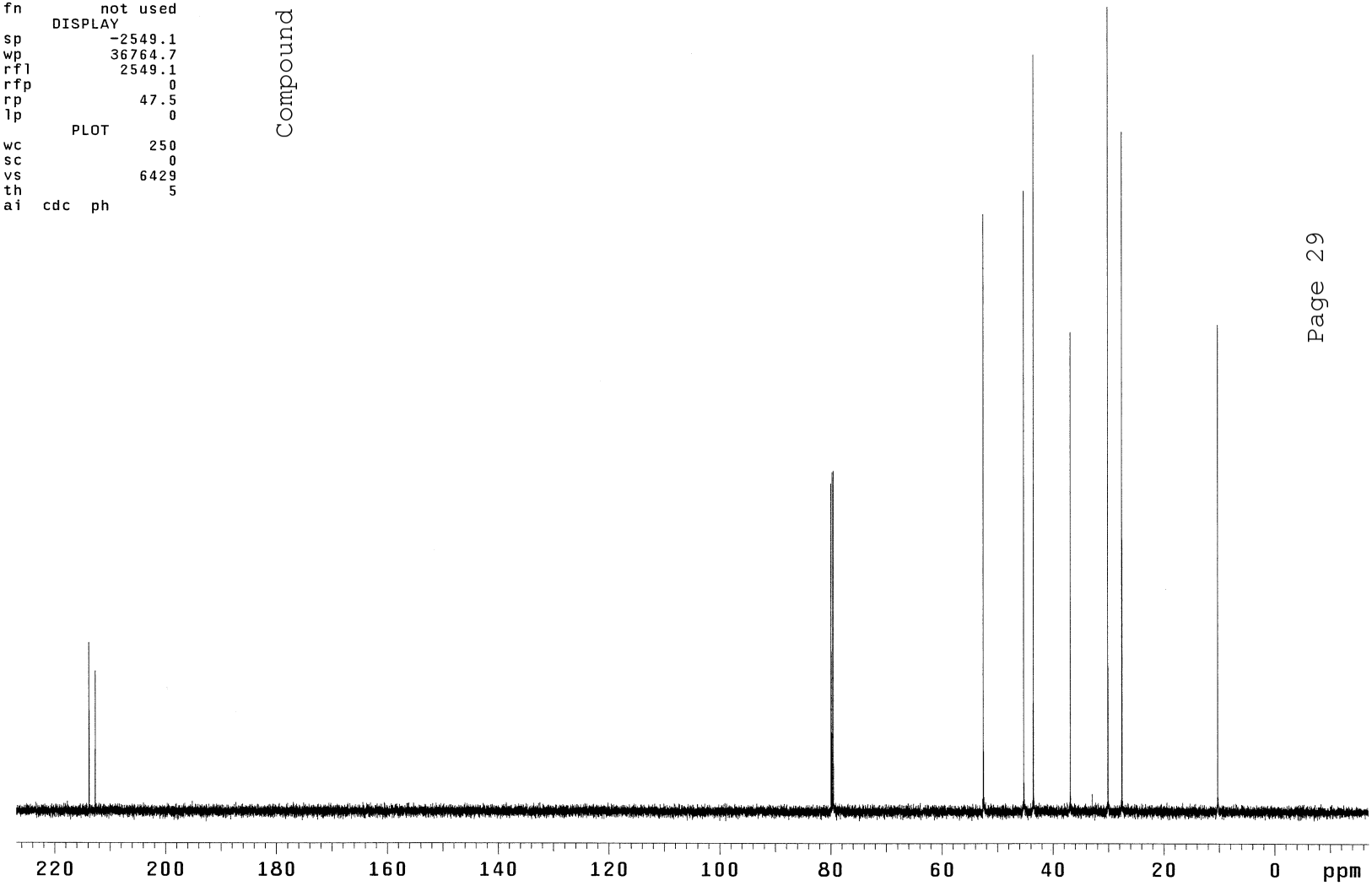
Std carbon

exp32 Carbon

| SAMPLE | | SPECIAL | |
|---|---|---|---|
| date | Jul 17 2009 | temp | 26.0 |
| solvent | cdcl3 | gain | 30 |
| file | exp | spin | 20 |
| ACQUISITION | | hst | 0.008 |
| sw | 36764.7 | pw90 | 7.600 |
| at | 1.300 | alfa | 10.000 |
| np | 95624 | FLAGS | |
| fb | 17000 | il | n |
| bs | 16 | in | n |
| d1 | 1.000 | dp | y |
| nt | 256 | hs | nn |
| ct | 32 | PROCESSING | |
| TRANSMITTER | | lb | 0.50 |
| tn | C13 | fn | not used |
| sfrq | 150.809 | DISPLAY | |
| tof | 1140.3 | sp | -2549.1 |
| tpwr | 57 | wp | 36764.7 |
| pw | 3.800 | rfl | 2549.1 |
| DECOUPLER | | rfp | 0 |
| dn | H1 | rp | 47.5 |
| dof | 0 | lp | 0 |
| dm | yyy | PLOT | |
| dmm | w | wc | 250 |
| dpwr | 46 | sc | 0 |
| dmf | 14706 | vs | 6429 |
| | | th | 5 |
| | | ai cdc ph | |




Compound S-11



Sample: dwc2-52-1
File: exp

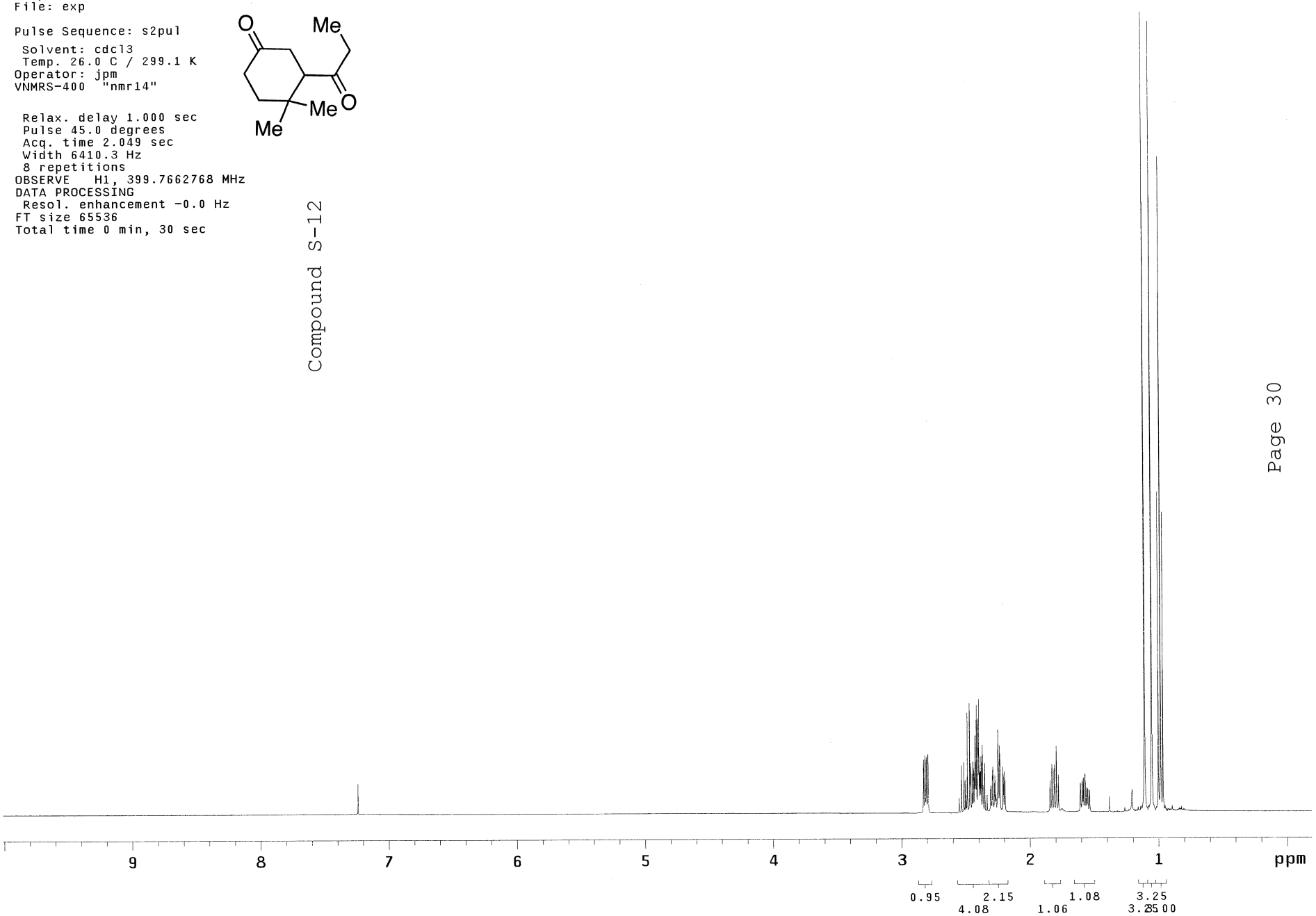
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: jpm
VNMRS-400 "nmr14"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7662768 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec




Compound S-12



Sample: dwc2-52-2-C
File: exp

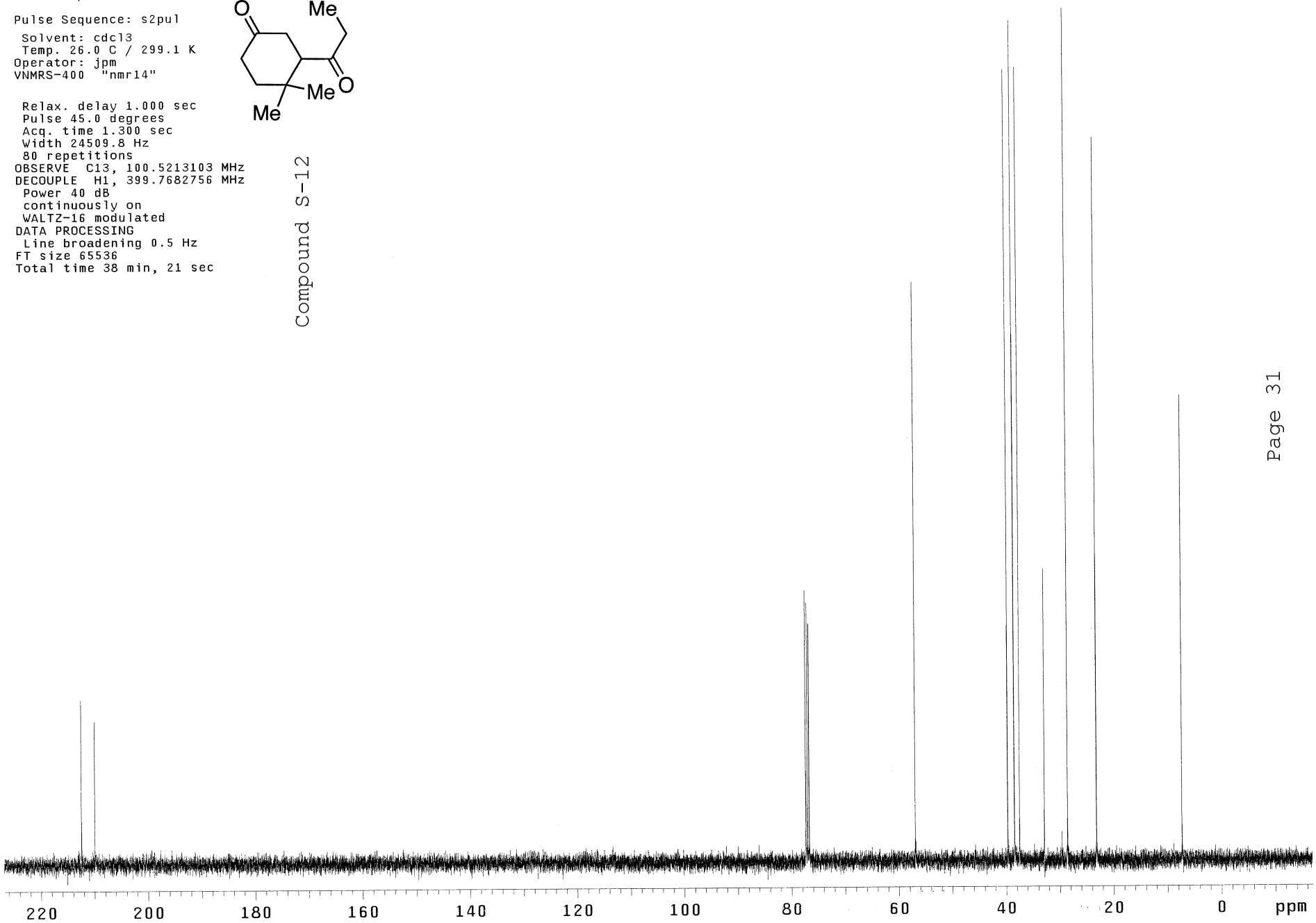
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 26.0 C / 299.1 K
Operator: jpm
VNMRS-400 "nmr14"




 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 80 repetitions
OBSERVE C13, 100.5213103 MHz
DECOUPLE H1, 399.7682756 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec

Compound S-12

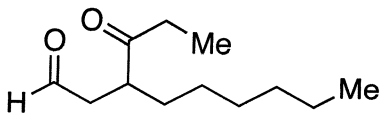




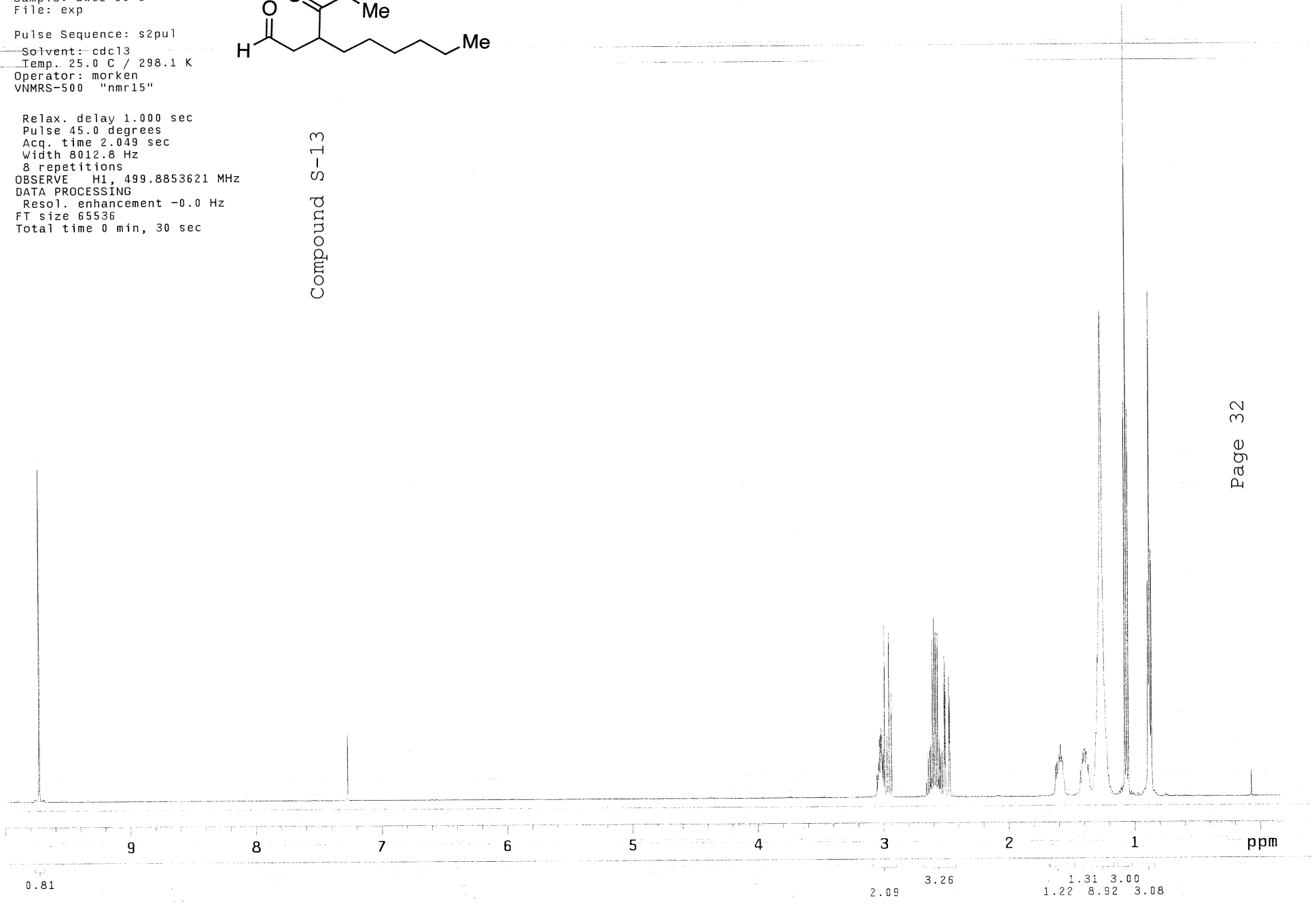
Sample: dwc2-69-3
File: exp

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: morken
VNMRS-500 "nmr15"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
8 repetitions
OBSERVE H1, 499.8853621 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec

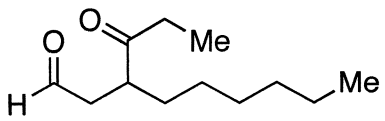



Compound S-13



STANDARD 1H OBSERVE - profile

Sample: dwc2-69-C
File: exp



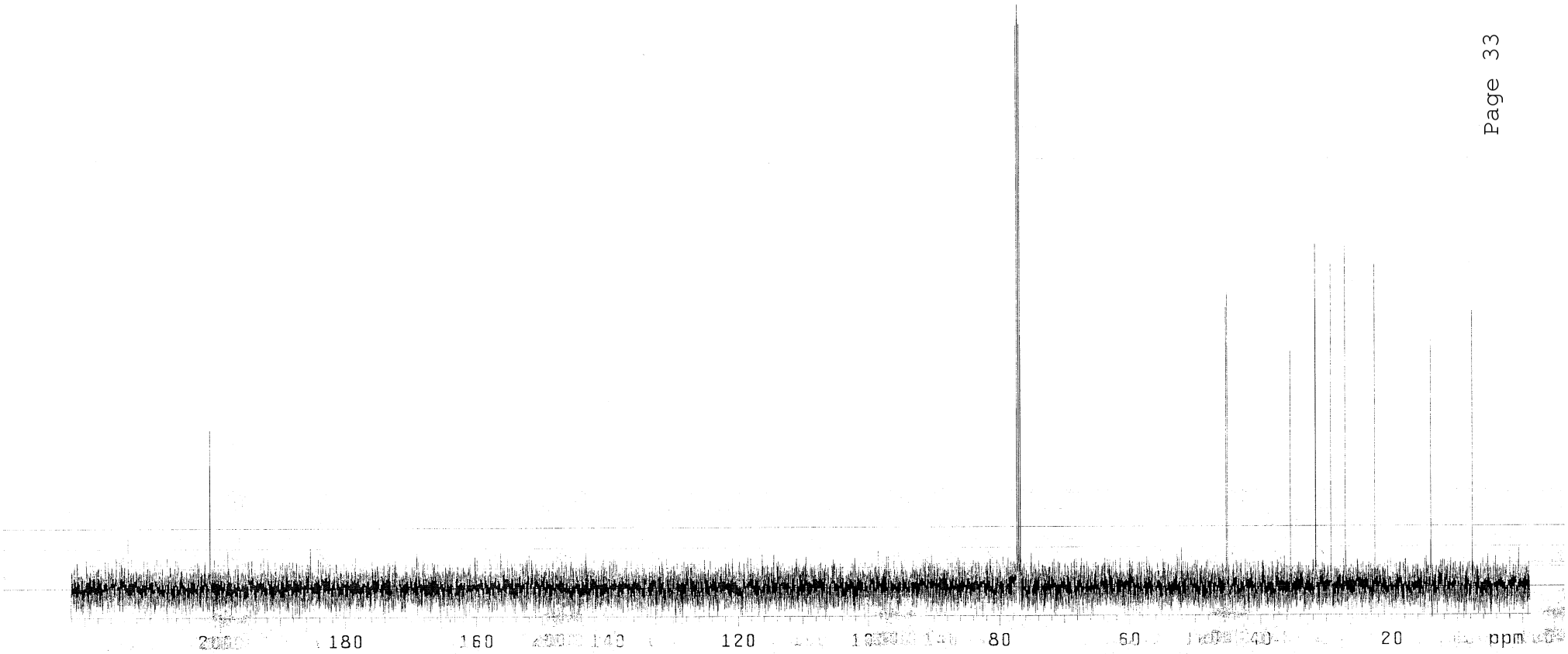

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: morken
VNMRS-500 "nmr15"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 33783.8 Hz
416 repetitions
OBSERVE C13, 125.6962743 MHz
DECOUPLE H1, 499.8878615 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 21 sec

Compound S-13

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 26813.5 | 213.319 | 8.6 |
| 2 | 25232.4 | 200.741 | 27.0 |
| 3 | 9712.3 | 77.268 | 95.3 |
| 4 | 9679.8 | 77.009 | 98.8 |
| 5 | 9647.8 | 76.755 | 95.5 |
| 6 | 5678.0 | 45.172 | 47.3 |
| 7 | 5655.8 | 44.996 | 49.7 |
| 8 | 4446.4 | 35.375 | 40.2 |
| 9 | 3966.5 | 31.556 | 58.0 |
| 10 | 3965.0 | 31.544 | 48.7 |
| 11 | 3673.2 | 29.223 | 54.5 |
| 12 | 3395.3 | 27.012 | 57.6 |
| 13 | 2830.4 | 22.517 | 54.5 |
| 14 | 1757.1 | 13.979 | 41.8 |
| 15 | 962.2 | 7.655 | 46.7 |

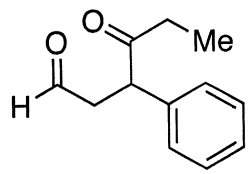




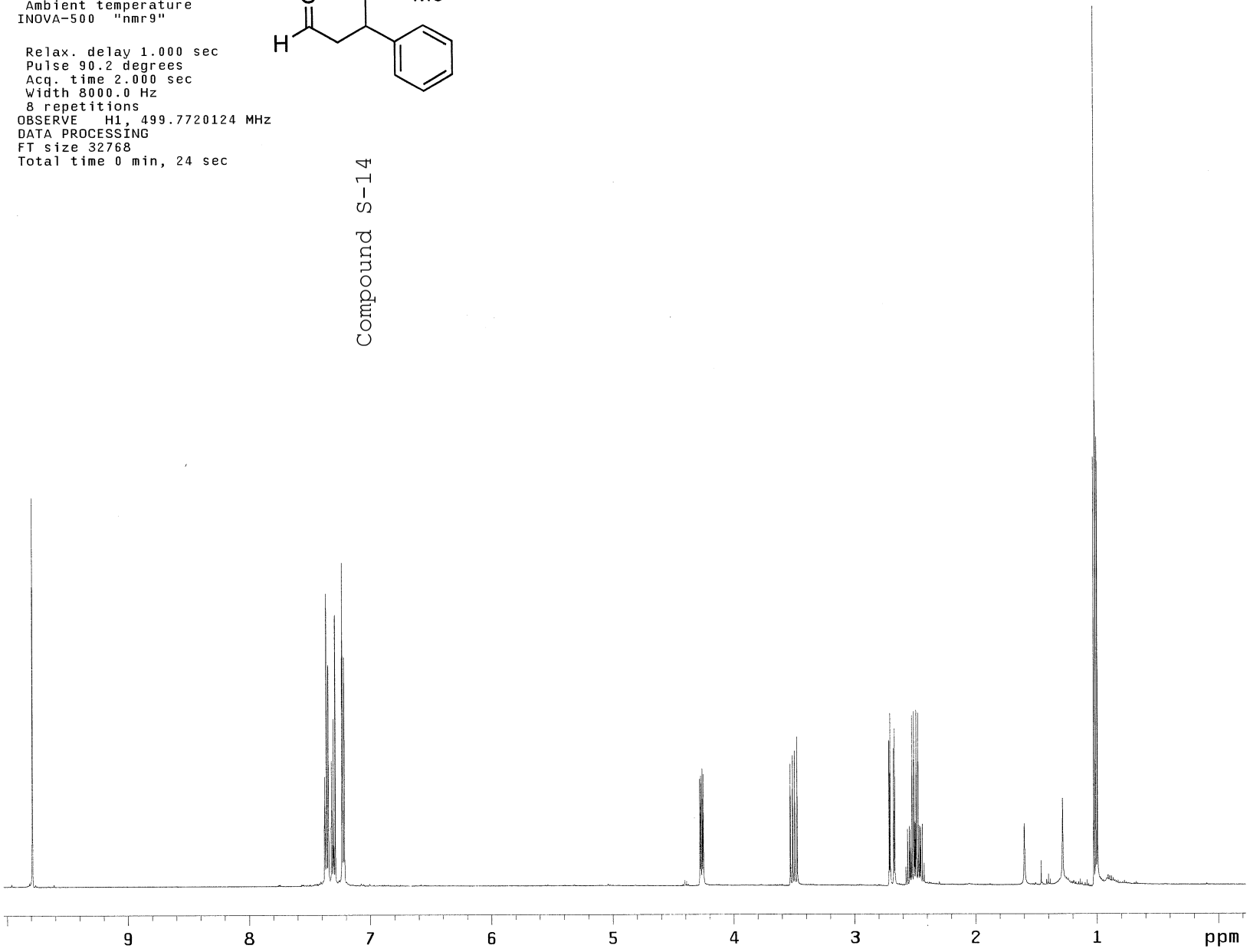
cinna2

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr9"

Relax. delay 1.000 sec
Pulse 90.2 degrees
Acq. time 2.000 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7720124 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec



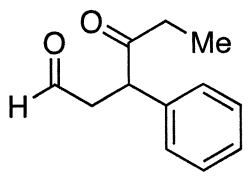

Compound S-14



Sample: dwc2-cinnamaldehyde_C
File: exp

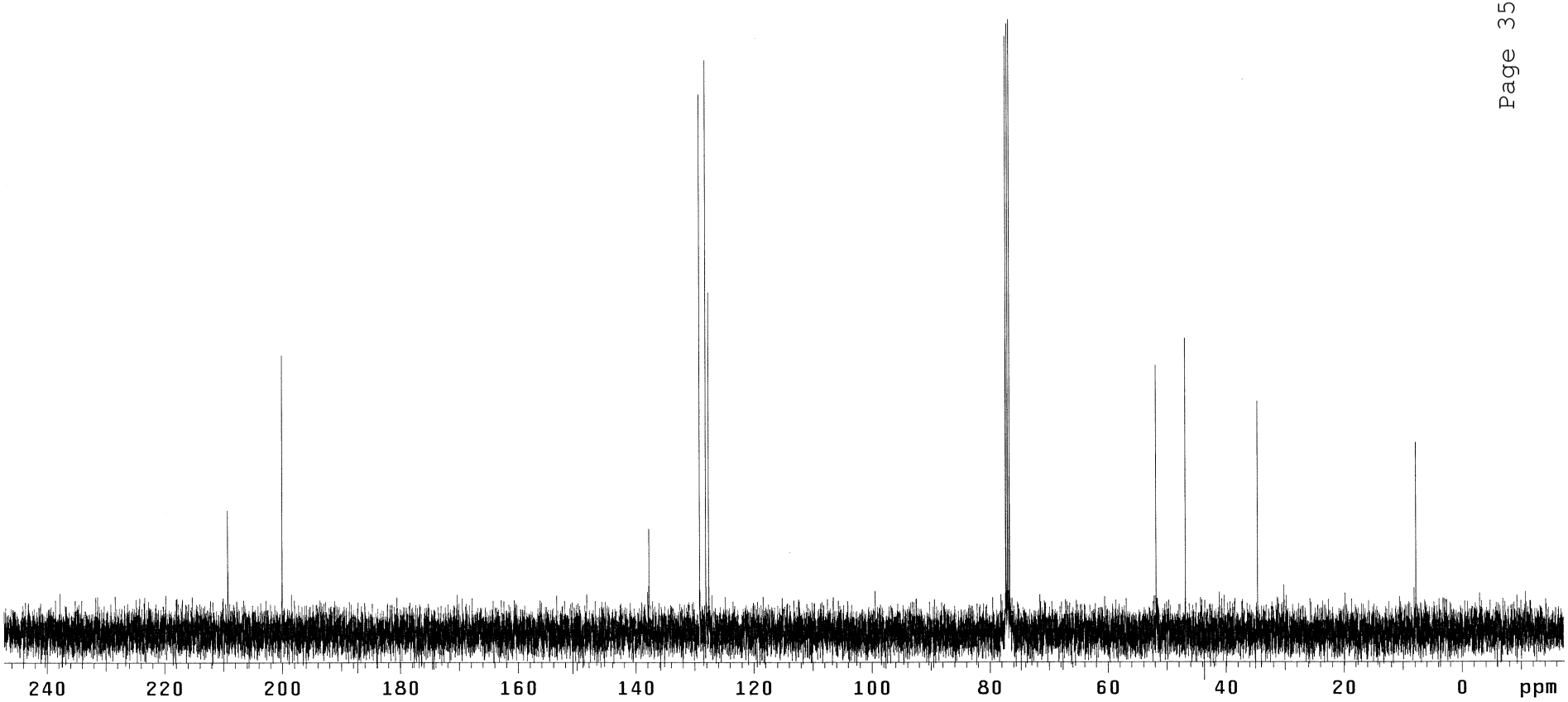
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 26.0 C / 299.1 K
Operator: vnmr1
VNMRS-400  "nmr14"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.301 sec
 Width 26595.7 Hz
 96 repetitions
OBSERVE  C13, 100.5213103 MHz
DECOUPLE  H1, 399.7682756 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 21 sec




Compound S-14

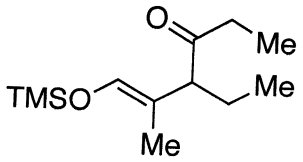




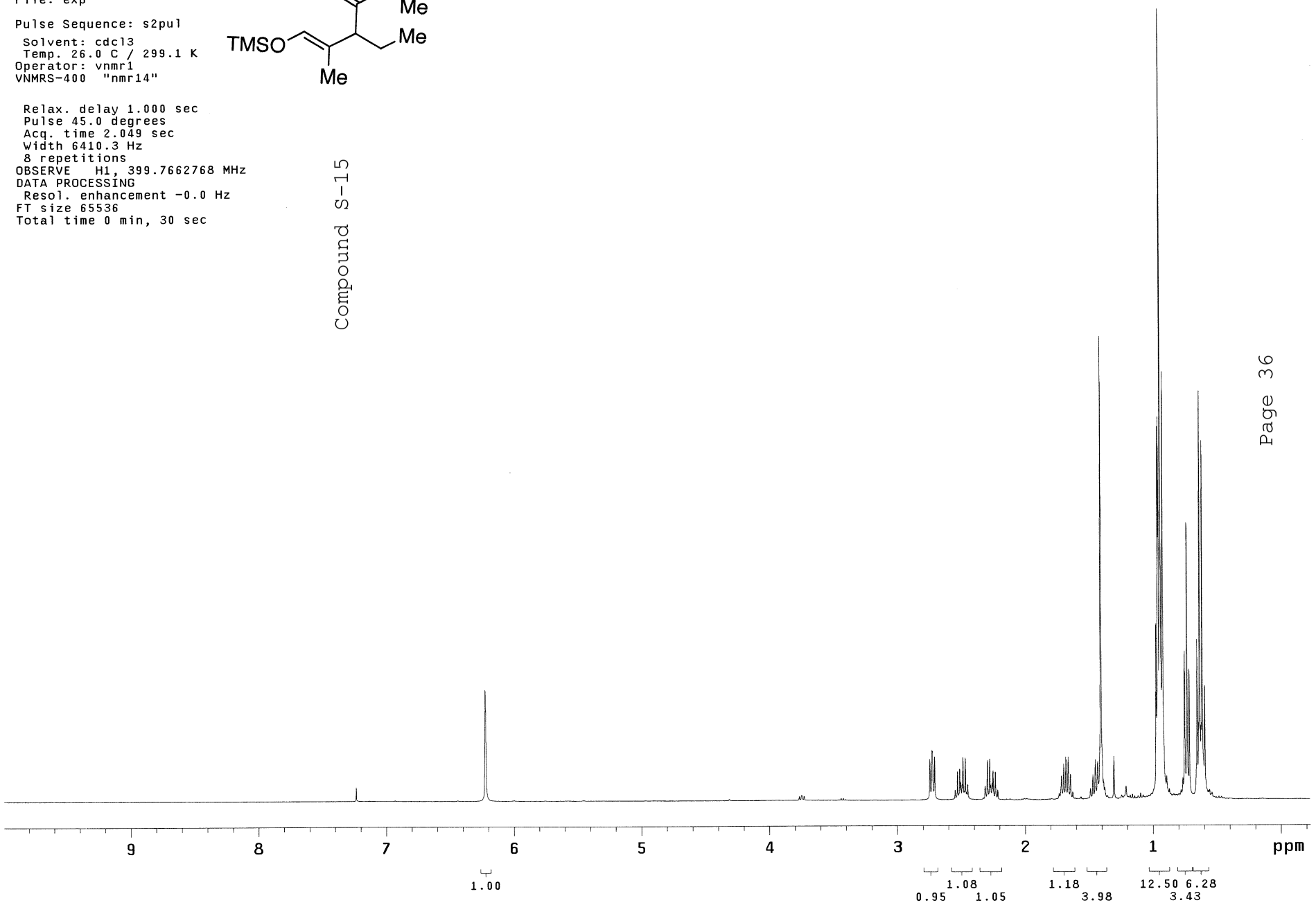
Sample: a-substitution
File: exp

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: vnmr1
VNMRS-400 "nmr14"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7662768 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec

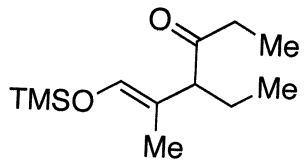



Compound S-15



Sample: a-substitution
File: exp

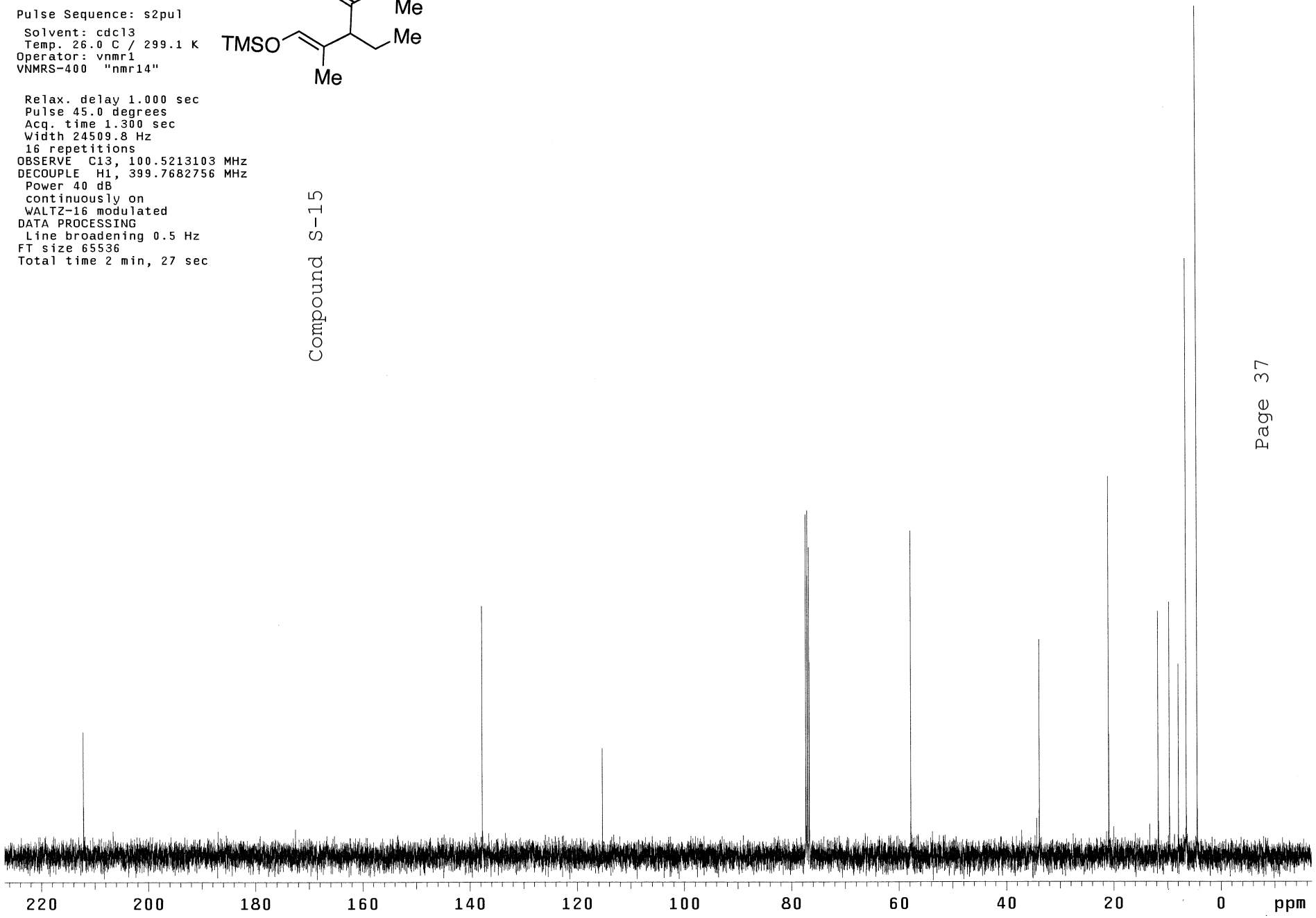
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 26.0 C / 299.1 K
Operator: vnmr1
VNMRS-400  "nmr14"

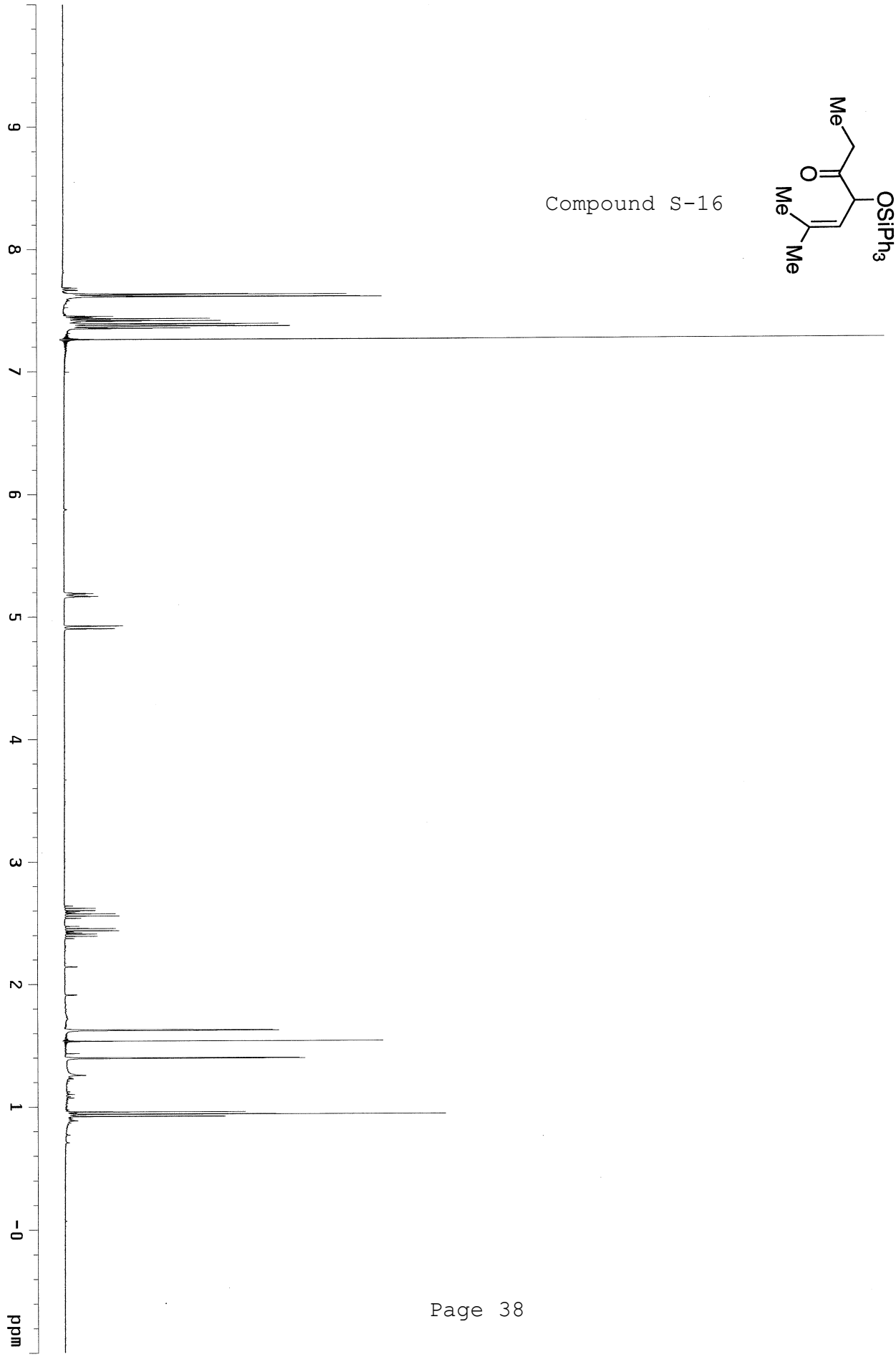



 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 16 repetitions
OBSERVE  C13, 100.5213103 MHz
DECOUPLE  H1, 399.7682756 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 27 sec

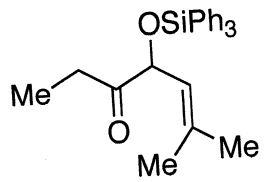
Compound S-15







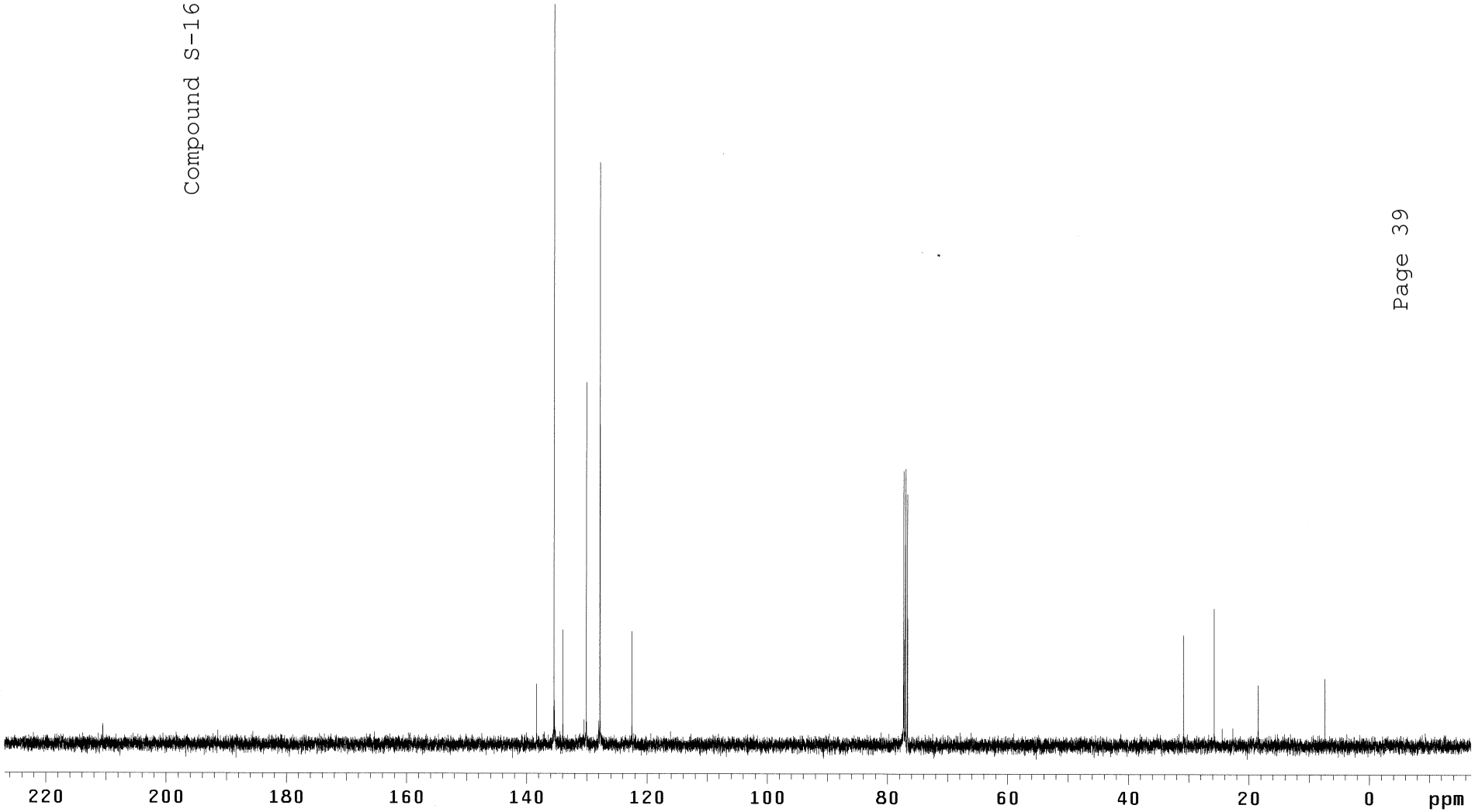

Compound S-16

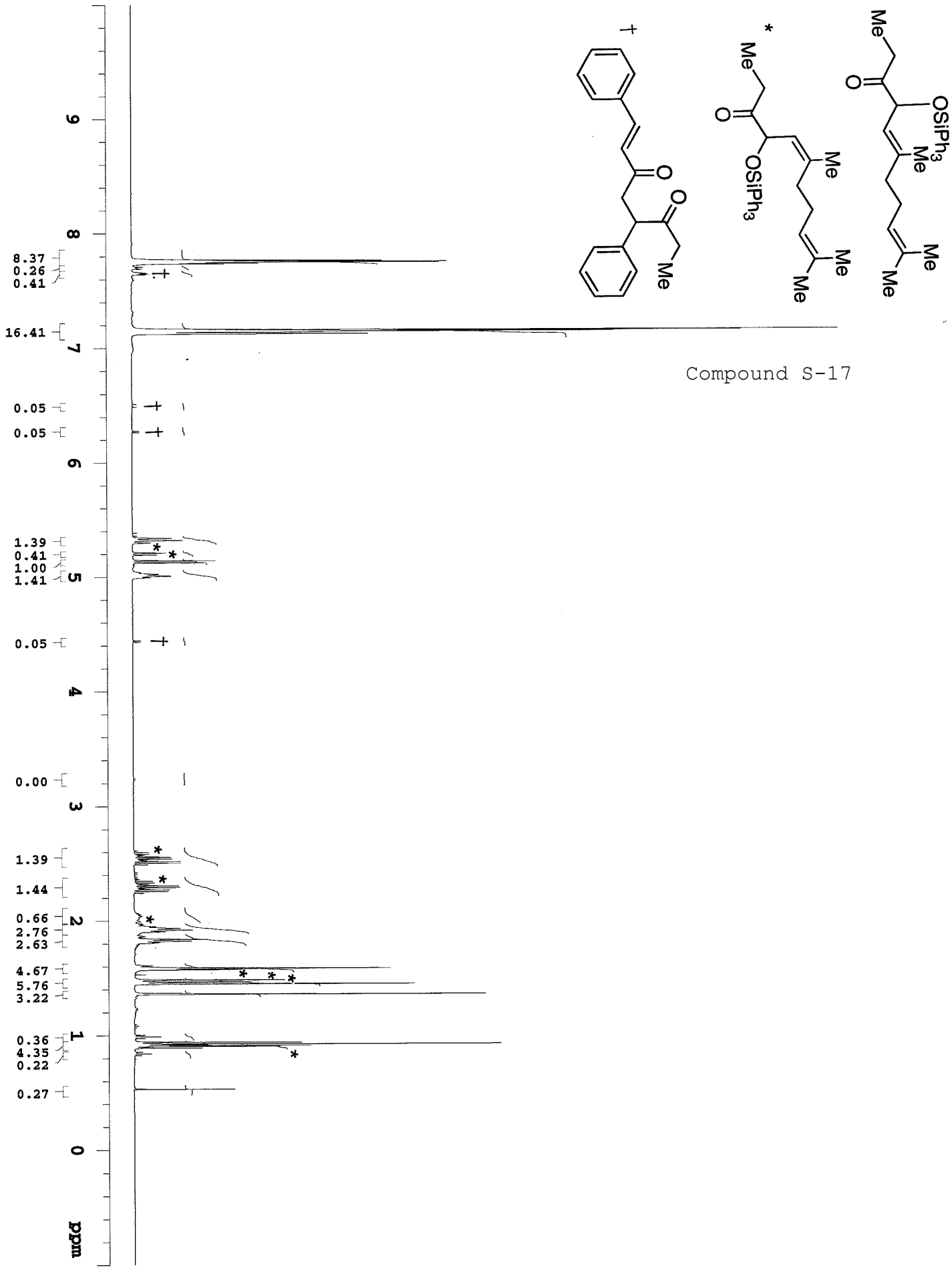



| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 13902.0 | 138.299 | 10.2 |
| 2 | 13613.2 | 135.426 | 126.0 |
| 3 | 13466.6 | 133.968 | 19.5 |
| 4 | 13073.2 | 130.054 | 61.6 |
| 5 | 12847.3 | 127.807 | 99.1 |
| 6 | 12314.7 | 122.509 | 19.2 |
| 7 | 7770.8 | 77.305 | 46.6 |
| 8 | 7764.0 | 77.238 | 17.9 |
| 9 | 7738.6 | 76.985 | 47.0 |
| 10 | 7706.4 | 76.665 | 42.5 |
| 11 | 3098.9 | 30.828 | 18.6 |
| 12 | 2588.0 | 25.746 | 23.2 |
| 13 | 1853.5 | 18.439 | 10.2 |
| 14 | 739.8 | 7.359 | 11.3 |

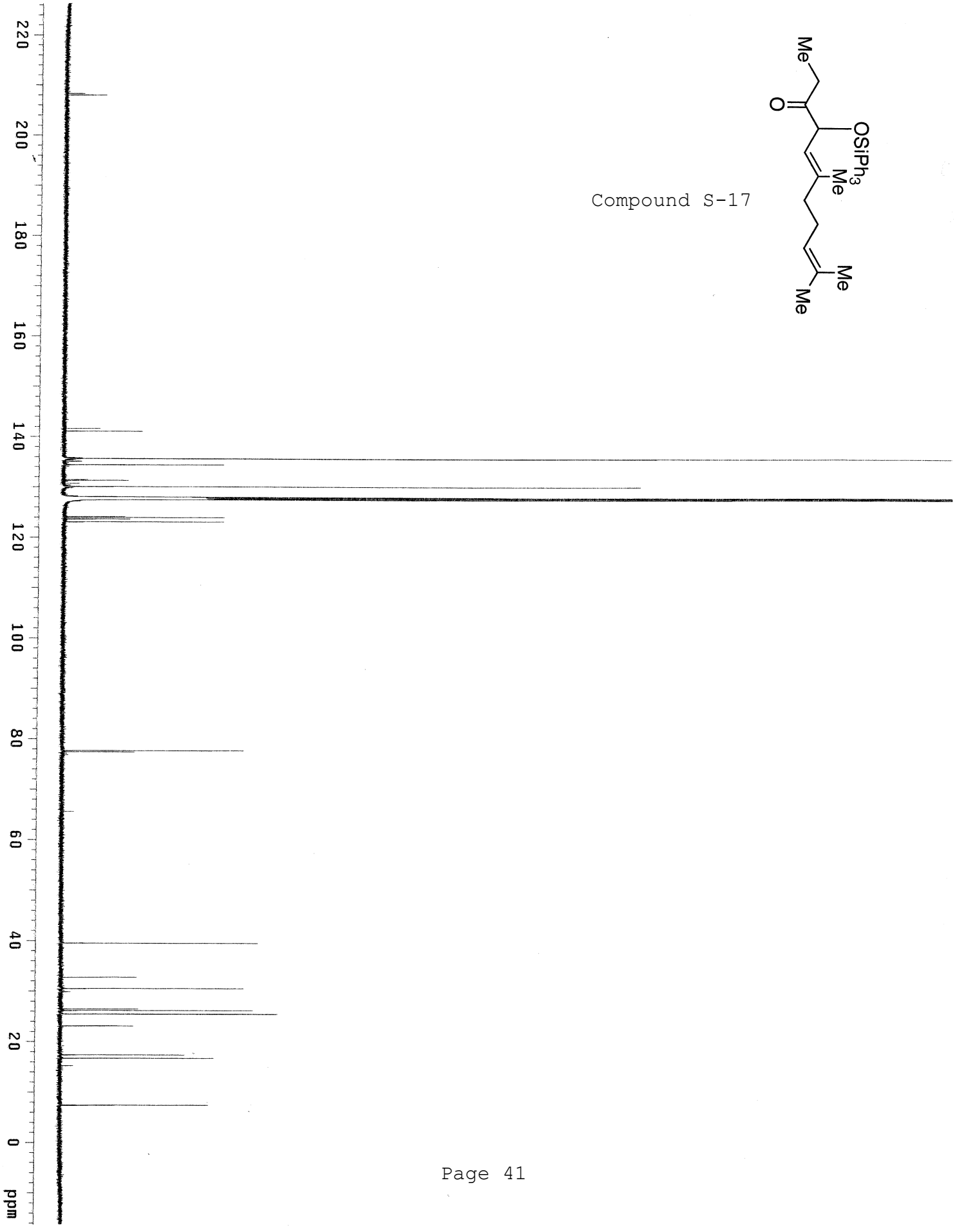
Compound S-16





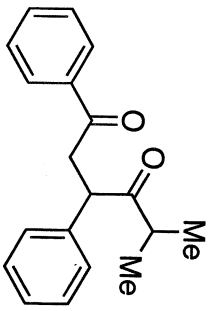


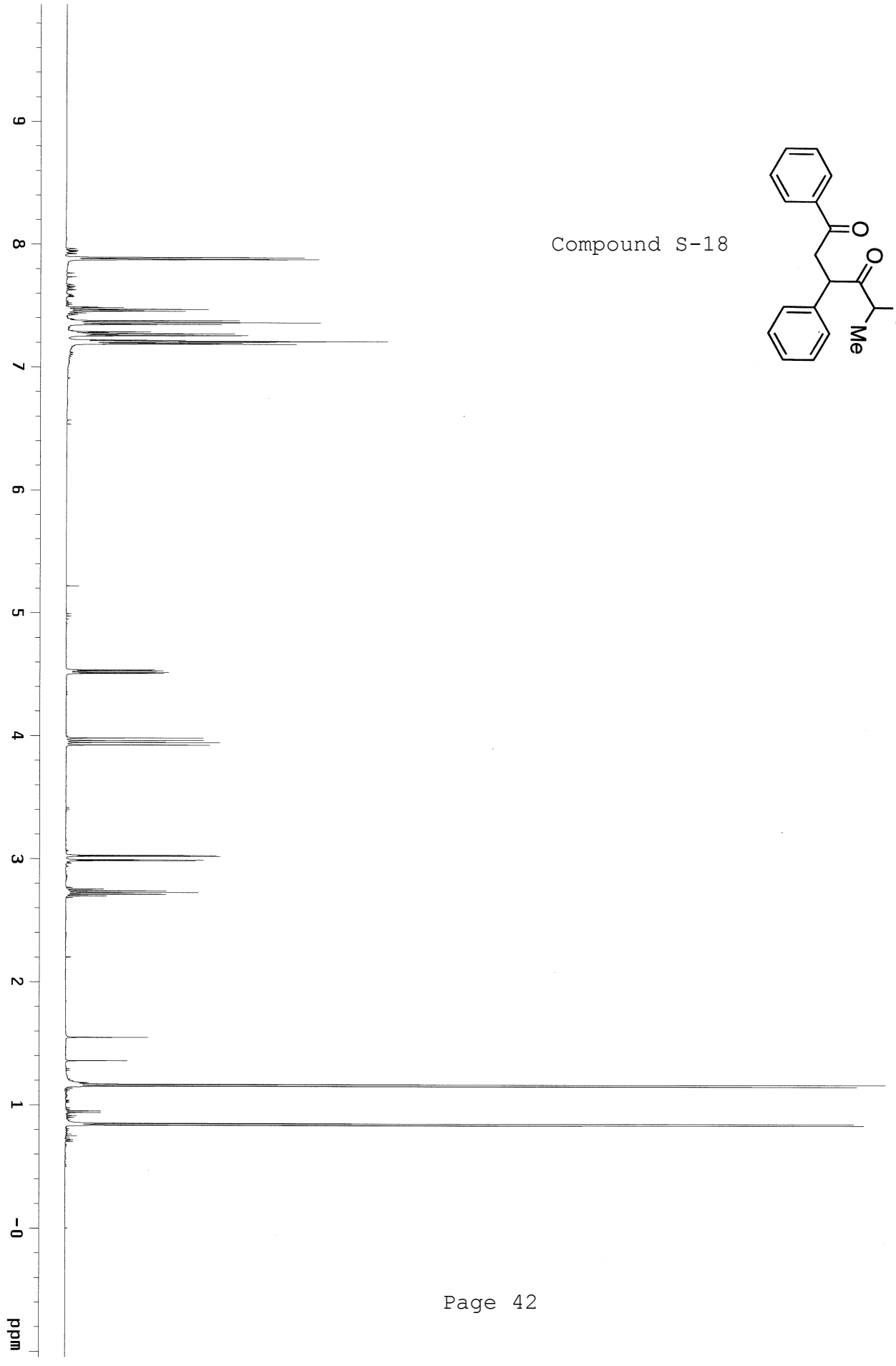

Compound S-17




Compound S-17

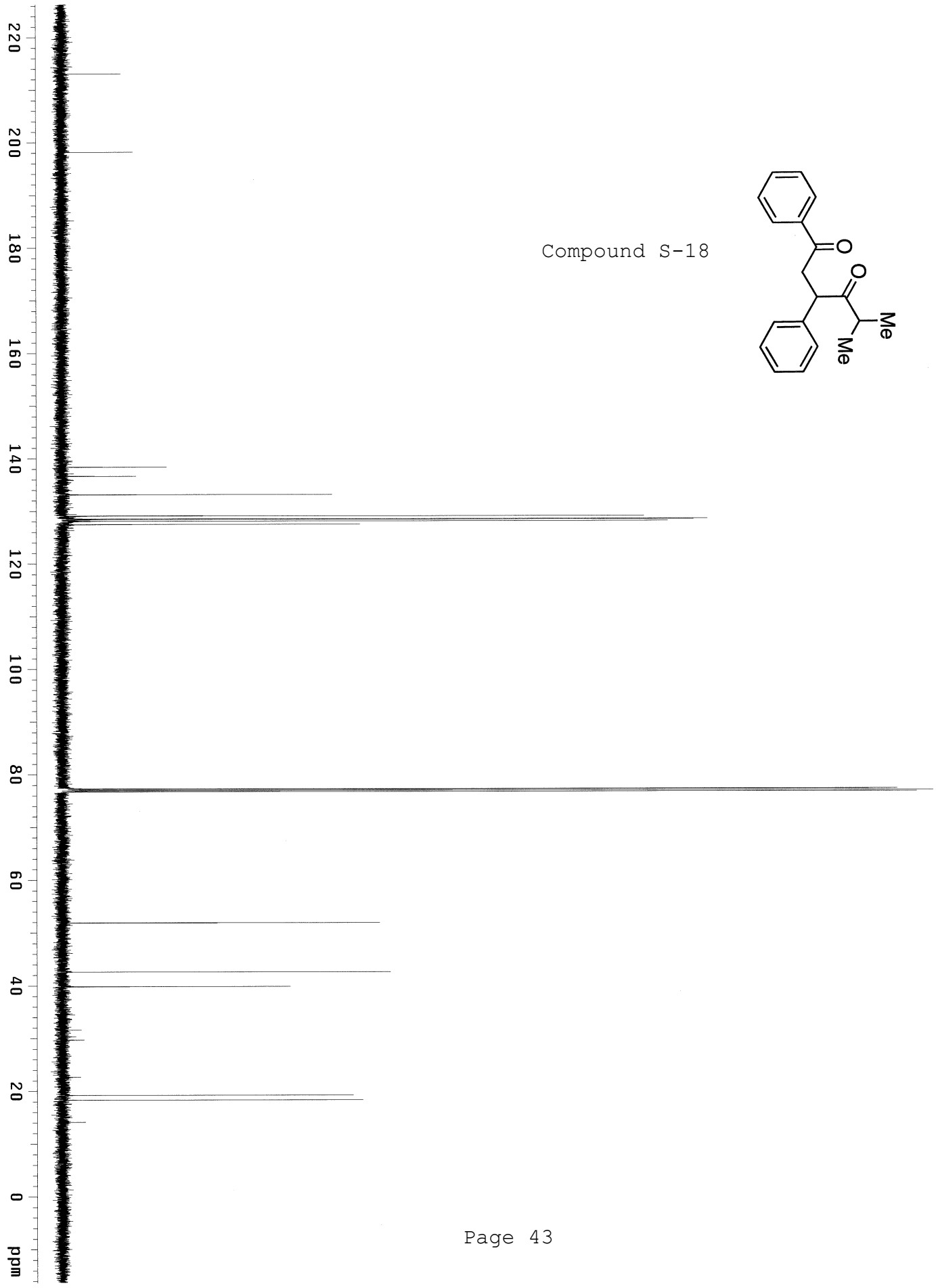
Compound S-18



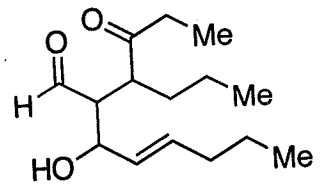
Compound S-18



Solvent: CDCl3
Ambient temperature
GEMINI-400BB "nmr8"

Relax. delay 2.000 sec
Pulse 40.4 degrees
Acq. time 3.000 sec
Width 5998.8 Hz
16 repetitions
OBSERVE H1, 400.0268509 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 29 sec




Tentatively Aldol
Adduct 5