Supporting Information

Total Synthesis of (±)-α- and β-Lycoranes by Sequential Chemoselective Conjugate Addition–Stereoselective Nitro-Michael Cyclization of ω-Nitro-α,β,ψ,ω-unsaturated Ester

Tomohisa Yasuhara,[†] Katsumi Nishimura,[‡] Mitsuaki Yamashita,[‡] Naoshi Fukuyama,[‡] Ken-ichi Yamada,[‡] Osamu Muraoka,[†] and Kiyoshi Tomioka*[‡]

[†]School of Pharmaceutical Sciences, Kinki University, 3-4-1 Kowakae, Higashi-osaka, Osaka 577-8502, Japan, and [‡]Graduate School of Pharmaceutical Sciences, Kyoto University, Yoshida, Sakyo-ku, Kyoto 606-8501, Japan

General. The extracts were dried over Na_2SO_4 unless otherwise noted. Purification was carried out using silica gel column chromatography. NMR (500 or 270 MHz for proton, 125 or 67.8 MHz for carbon) was measured in $CDCl_3$ and chemical shift and *J* value were presented in ppm relative to internal tetramethylsilane and Hz, respectively. The wavenumbers of maximum absorption peaks of IR spectroscopy were presented in cm^{-1}. All melting points were uncorrected.

***tert*-Butyl 7-hydroxyhept-2-enoate (2*E*/2*Z* = 9/1) (10):** A mixture **9** (0.5 g, 4.9 mol) and $Ph_3P{=}CHCO_2{}^tBu$ (1.8g, 9.8 mmol) in toluene (10 mL) was stirred at 60 °C for 10 min, and treated with hexane (50 mL) at 0 °C, and then filtrated. After concentration, the mixture was again treated with hexane (50 mL) at 0 °C, and then filtrated. Concentration and chromatography (hexane/AcOEt = 3/2) gave **10** as a colorless oil (0.891 g, 91%). ^{1}H NMR: 1.48 (9H, s), 1.53–1.65 (4H, m), 2.21 (1.8H, ddt, *J* = 1.4, 6.7, 7.4, *E*), 2.64 (0.2H, ddt, *J* = 1.8, 7.5, 7.6, *Z*), 3.67 (2H, t, *J* = 6.4), 5.69 (0.1H, dt, *J* = 11.6, 1.8, *Z*), 5.75 (0.9H, dt, *J* = 15.9, 1.4, *E*), 6.13 (0.1H, dt, *J* = 11.6, 7.5, *Z*), 6.85 (0.9H, dt, *J* = 15.9, 6.7, *E*). ^{13}C NMR: 24.2 (*E*), 25.1 (*Z*), 28.1 (*E*), 28.2 (*Z*), 31.7, 32.0, 62.4 (*Z*), 62.5 (*E*), 80.1, 121.7 (*Z*), 123.2 (*E*), 147.5 (*E*), 148.5 (*Z*), 166.1. IR (neat): 3440, 2985, 1735, 1705. EIMS (*m/z*): 201 (M^++H), 144 (M^+–^{t}Bu+H), 126 (M^+–O^{t}Bu+H).

***tert*-Butyl (2*E*)-7-oxohept-2-enoate (11)**: To a solution of **10** (400 mg, 2.0 mmol) above in toluene (7 mL), DMSO (7 mL), pyridine (0.16 mL, 2.0 mmol), freshly distilled trifluoroacetic acid (0.08 mL, 1.0 mmol) and DCC (1.2 g, 6.0 mmol) were successively added at rt. The mixture was stirred for 18 h at rt, and then diluted with toluene (30 mL), and then filtrated. The whole was washed with water (30 mL x 3) and brine (20 mL). Concentration and chromatography (hexane/AcOEt = 10/1) gave **11** as a colorless oil (358 mg, 90%). ^{1}H NMR: 1.48 (9H, s), 1.80 (2H, tt, *J* = 7.2, 7.3), 2.23 (2H, ddt, *J* = 1.6, 7.0, 7.3), 2.49 (2H, dt, *J* = 1.4, 7.2), 5.76 (1H, dt, *J* = 15.6, 1.6), 6.81 (1H, dt, *J* = 15.6, 7.0), 9.78 (1H, t, *J* = 1.4). ^{13}C NMR (125 MHz): 20.4, 28.1, 31.1, 43.0, 80.2, 124.0, 146.2, 165.8, 201.7. IR (neat): 2900, 1710, 1650. EIMS (*m/z*): 199 (M^++H), 142 (M^+–^{t}Bu+H), 125 (M^+–O^{t}Bu).

***tert*-Butyl (2*E*)-7-hydroxy-8-nitrooct-2-enoate (12)**: A mixture of **11** (198 mg, 1.0 mmol), nitromethane (0.17 mL, 1.0 mmol), and triethylamine (2 drops) in THF (2 mL) was stirred at reflux for 12 h. Concentration and chromatography (hexane/AcOEt = 8/1) gave **12** as a colorless oil (219 mg, 84%). ^{1}H NMR: 1.40–1.70 (4H, m), 1.44 (9H, s), 2.19 (2H, dt, *J* = 6.7, 6.4), 3.02 (1H, brs), 4.29–4.41 (3H, m), 5.72 (1H, d, *J* = 15.6), 6.78 (1H, dt, *J* = 6.7, 15.6). ^{13}C NMR: 23.7, 28.1, 31.4, 33.0, 68.3, 80.3, 80.6, 123.6, 146.8, 166.1. IR (neat): 3420, 2900, 1700. EIMS (*m/z*): 260 (M^++H), 203 (M^+–^{t}Bu+H), 186 (M^+–O^{t}Bu). Anal. Calcd. for $C_{12}H_{21}NO_5$: C, 55.58; H, 8.16; N, 5.40. Found. C, 55.60; H, 8.21; N, 5.62.

***tert*-Butyl 8-nitrooct-2,7-dienoate (8)**: A solution of **12** (900 mg, 3.5 mmol), trifluoroacetic anhydride (0.5 mL, 3.5 mmol), and triethylamine (0.9 mL, 7.0 mmol) in THF (10 mL) was stirred at 0 °C for 1 h, and diluted with $CHCl_3$ (20 mL), and then washed with water (20 mL), satd NH_4Cl (20 mL), and brine (20 mL). Combined water layers were extracted with $CHCl_3$ (20 mL x 2). Concentration and chromatography (hexane/AcOEt = 8/1) gave **8** as a colorless oil (804 mg, 96%). ^{1}H NMR: 1.49 (9H, s), 1.70 (2H, tt, *J* = 7.4, 7.7), 2.25 (2H, dt, *J* = 6.9, 7.4), 2.31 (2H, dt, *J* = 7.3, 7.7), 5.77 (1H, d, *J* = 15.6), 6.81 (1H, dt, *J* = 15.6, 6.9), 6.99 (1H, d, *J* = 13.4), 7.26 (1H, dt, *J* = 13.4, 7.3). ^{13}C NMR: 26.1, 27.7, 28.1, 31.1, 80.4, 124.2, 141.6, 145.7, 145.7, 165.7. IR (neat): 2900, 1710, 1650.

FAB-MS (*m/z*): 242 (M^++H), 186, 168, 57. HRMS–FAB: $[M+H]^+$ Calcd. for $C_{12}H_{19}NO_4$, 242.1392; Found, 242.1389.

***tert*-Butyl 7-benzo[1,3]dioxol-5-yl-8-nitrooct-2-enoate (6)**: A hexane solution of BuLi (1.0 mL, 1.5 mmol) was added at –78 °C over 1 min to a solution of 5-bromobenzo[1,3]dioxole (301 mg, 1.5 mmol) in THF (10 mL), and then the mixture was stirred for 0.5 h at –78 °C. The whole was added to a solution of **8** (241 mg, 1.0 mmol) in THF (5 mL) at –78 °C over 5 min. The mixture was stirred for 15 min at –78 °C, and then successively treated with MeOH (1 mL), satd NH_4Cl (20 mL), and brine (20 mL). The mixture was extracted with AcOEt (20 mL x 3). Concentration and chromatography (hexane/AcOEt = 50/1 to 1/1) gave **6** as a pale yellow oil (340 mg, 94%). ^{1}H NMR: 1.33 (2H, dddd, 7.3, 7.4, 7.7, 8.0), 1.43 (9H, s), 1.63 (2H, m), 2.12 (2H, m), 3.34 (1H, m), 4.45 (1H, dd, *J* = 8.0, 11.9), 4.48 (1H, dd, *J* = 7.0, 11.9), 5.66 (1H, d, *J* = 15.6), 5.94 (2H, s), 6.60–6.65 (2H, m), 6.70–6.79 (2H, m). ^{13}C NMR: 25.4, 28.1, 31.6, 32.5, 44.0, 80.2, 81.0, 101.2, 107.4, 108.7, 121.0, 123.5, 132.6, 146.7, 147.5, 148.2, 165.9. FTIR (neat): 2978, 1708, 1651. EIMS (*m/z*): 363 (M^+), 306 (M^+–^{t}Bu) 162 ($C_{10}H_{10}O_2^+$), 148 ($C_9H_8O_2^+$), 135 ($C_8H_7O_2^+$). HRMS–FAB: $[M+H]^+$ Calcd. for $C_{19}H_{25}NO_6$, 363.1682. Found, 363.1677.

***tert*-Butyl (3-benzo[1,3]dioxol-5-yl-2-nitrocyclohexyl)acetate (5a and 5b)**: A mixture of **6** (500 mg, 1.3 mmol), CsF (400 mg, 2.6 mmol), and myristyltrimethylammonium bromide (60 mg, 0.1 mmol) in THF (14 mL) was stirred at rt for 24 h, and was then treated with brine (20 mL). The mixture was extracted with AcOEt (20 mL x 3). Concentration and chromatography (hexane/AcOEt = 10/1) gave a 1.5:1 mixture of **5a** and **5b** as a pale yellow oil (522 mg, 94%). Crystallization from $CHCl_3$/hexane gave diastereomerically pure **5a** as colorless cubes of mp 123–125 °C (239 mg) in 43% yield and a mother liquid in 51% yield as pale yellow oil (**5a**/**5b** = 1/3). **5a:** ^{1}H NMR: 1.24–1.33 (1H, m), 1.42 (9H, s), 1.50–1.55 (2H, m), 1.83 (1H, m), 1.95 (1H, m), 2.02 (1H, m), 2.12 (1H, dd, *J* = 8.9, 15.9), 2.27 (1H, dd, *J* = 3.1, 15.9), 2.44 (1H, m), 3.05 (1H, ddd, *J* = 3.4, 11.0, 11.3), 4.47 (1H, dd, *J* = 11.0, 11.0), 5.90 (2H, s), 6.59 (1H, d, *J* = 7.9), 6.65 (1H, s), 6.69 (1H, d, *J* = 7.9). ^{13}C NMR: 24.9, 28.0, 30.1, 32.9, 38.3, 38.9, 48.4, 81.1, 95.4, 101.0, 107.3, 108.5, 120.6, 134.0, 146.7, 147.8, 170.2. FTIR ($CHCl_3$):

2936, 1720, 1551. EIMS (*m/z*): 363 (M^+), 290 (M^+–O^{t}Bu), 260 (M^+–$C_5H_{11}O_2$). Anal. Calcd. for $C_{19}H_{25}NO_6$: C, 62.80; H, 6.93; N, 3.85. Found: C, 62.75; H, 7.02; N, 3.62.

A 1:3 mixture of **5a** and **5b**: ^{1}H NMR: 1.24–1.33 (0.25H, m, **5a**), 1.43 (6.50H, s, **5b**), 1.44 (2.25H, s, **5a**), 1.43–2.44 (6.75H, m), 2.48 (0.75H, dd, *J* = 2.0, 13.8, **5b**), 2.49 (0.75H, dd, *J* = 6.3, 13.8, **5b**), 3.07 (1H, brs), 3.18 (0.75H, ddd, *J* = 4.3, 11.9, 12.0, **5b**), 4.49 (0.25H, dd, *J* = 10.9, 11.2, **5a**), 4.82 (0.75H, dd, *J* = 4.6, 11.9, **5b**), 5.91 (1.5H, s, **5b**), 5.92 (0.5H, s, **5a**), 6.61–6.74 (3H, m). ^{13}C NMR (125 MHz): 19.9 (**5b**), 24.9 (**5a**), 27.99 (**5b**), 28.02 (**5a**), 29.3 (**5b**), 30.1 (**5a**), 32.9 (**5a**), 33.5 (**5b**), 33.8 (**5b**), 34.6 (**5b**), 38.3 (**5a**), 38.9 (**5a**), 41.7 (**5b**), 48.4 (**5a**), 81.05 (**5b**), 81.13 (**5a**), 92.1 (**5b**), 95.4 (**5a**), 101.0 (**5a**, **5b**), 107.3 (**5a**), 107.5 (**5b**), 108.40 (**5a**), 108.44 (**5b**), 120.3 (**5b**), 120.6 (**5a**), 134.0 (**5a**), 134.8 (**5b**), 146.6 (**5b**), 146.8 (**5a**), 147.77 (**5b**), 147.81 (**5a**), 170.2 (**5a**), 170.6 (**5b**).

***tert*-Butyl 1,2-*trans*-2,3-*trans*-2-amino-3-benzo[1,3]dioxol-5-ylcyclohexylacetate (4a)**: To a solution of **5a** (1.6 g, 4.4 mmol) in EtOH (60.0 mL), 10% HCl (10 mL) and Zn powder (1.0 g, 15.4 mmol) were added at rt. After stirring for 1 d, the mixture was filtrated and concentrated. The residue was diluted with AcOEt (40 mL), and then washed with satd $NaHCO_3$ (20 mL) and brine (20 mL). Concentration and chromatography gave **4a** as a colorless oil (1.46 g, 99%). ^{1}H NMR: 1.26 (1H, brs), 1.43–1.48 (3H, m), 1.45 (9H, s), 1.75–1.85 (3H, m), 2.14 (1H, brs), 2.27 (1H, brs), 2.48 (1H, dd, *J* = 9.5, 9.8), 2.64 (1H, brd, *J* = 9.8), 5.93 (2H, s), 6.65 (1H, d, *J* = 8.0), 6.70 (1H, s), 6.74 (1H, d, *J* = 8.0). ^{13}C NMR: 25.8, 28.0, 31.8, 34.5, 39.9, 42.0, 53.6, 59.0, 79.9, 100.7, 107.5, 108.2, 120.8, 138.1, 145.9, 147.7, 172.7. FTIR (neat): 3375, 2927, 1717. EIMS (*m/z*): 333 (M^+), 277 (M^+–^{t}Bu+H). HRMS–FAB: Calcd. for $C_{19}H_{27}NO_4$ 333.1940. Found 333.1922.

3a,7a-*trans*-7,7a-*trans*-7-Benzo[1,3]dioxol-5-yl-octahydroindol-2-one (14a): A solution of **4a** (1.46 g, 4.4 mmol) and sodium (500 mg, 22 mmol) in MeOH (60 mL) was stirred at rt for 24 h, and was then treated with brine (10 mL). The mixture was extracted with AcOEt (40 mL x 3). Concentration and recrystallization from $CHCl_3$/hexane gave **14a** as colorless needles of mp 215-217 °C (1.12 g, 98%). ^{1}H NMR: 1.38 (1H, m), 1.49–1.59 (2H, m), 1.89–2.04 (4H, m), 2.11 (1H, dd, *J* = 12.9, 15.8), 2.38 (1H, dd, *J* = 6.5, 15.8), 2.55 (1H, ddd, *J* = 2.8, 10.1, 11.0), 3.15 (1H, dd, *J* = 10.1, 10.1), 5.28 (1H, brs), 5.94

(2H, s), 6.63 (1H, d, J = 8.0), 6.68 (1H, s), 6.35 (1H, d, J = 8.0). ^{13}C NMR: 26.1, 28.1, 33.0, 38.0, 44.9, 48.4, 65.3, 101.0, 107.0, 108.5, 120.0, 136.3, 146.5, 148.1, 177.6. FTIR ($CHCl_3$): 3422, 1686. EIMS (*m/z*): 259 (M^+). Anal. Calcd. for $C_{15}H_{17}NO_3$: C, 69.48; H, 6.61; N, 5.40. Found: C, 62.75; H, 6.90; N, 5.52.

3a,7a-*trans*-7,7a-*trans*-7-Benzo[1,3]dioxol-5-yloctahydroindole (15a): A mixture of **14a** (259 mg, 1.0 mmol) and $LiAlH_4$ (76 mg, 2.0 mmol) in THF (5 mL) was stirred at reflux for 3 h and then successively treated with water (1 mL), 8% NaOH (3 mL), and water (3 mL). The mixture was extracted with $CHCl_3$ (20 mLx3). Concentration and chromatography ($CHCl_3$/MeOH = 10/1) afforded **15a** as a pale yellow oil (241 mg, 98%). ^{1}H NMR: 1.17–1.25 (1H, m), 1.39–1.45 (4H, m), 1.68 (1H, brs), 1.81–1.85 (2H, m), 1.95–1.99 (2H, m), 2.39–2.44 (2H, m), 2.90–2.99 (2H, m), 5.91 (2H, s), 6.68–6.74 (3H, m). ^{13}C NMR: 26.6, 29.9, 30.6, 33.8, 43.4, 45.3, 50.0, 69.0, 100.7, 107.3, 108.2, 120.2, 128.5, 135.9, 145.7. FTIR (neat): 3422, 2922, 1480. EIMS (*m/z*): 245 (M^+).

Methyl 3a,7a-*trans*-7,7a-*trans*-7-benzo[1,3]dioxol-5-yloctahydroindole-1-carboxylate (16): Methyl chloroformate (30 mg, 0.31 mmol) was added at 0 °C over 1 min to a solution of **15a** (64 mg, 0.26 mmol) and triethylamine (0.052 ml. 0.39 mmol) in $CHCl_3$ (3 mL). The mixture was stirred at rt for 1 h, and then diluted with $CHCl_3$ (20 mL), and was washed with 10% HCl (20 mL), satd $NaHCO_3$ (20 mL) and brine (20 mL). Concentration and chromatography gave **16** as a colorless oil (77 mg, 98%). ^{1}H NMR: 1.25–1.50 (3H, m), 1.64–1.98 (6H, m), 2.61 (1H, m), 3.05 (3H, s), 3.07 (1H, m), 3.30 (1H, m), 3.75 (1H, m), 5.89 (1H, d. J = 10.7), 5.90 (1H, d, J = 10.7), 6.60–6.62 (1H, m), 6.69–6.62 (2H, m). ^{13}C NMR: 26.5, 29.6, 30.3, 33.8, 49.0, 49.5, 51.3, 51.7, 68.3, 100.5, 107.5, 107.9, 120.5, 139.7, 145.3, 147.1, 153.7. FTIR (neat): 2932, 1686. EIMS (*m/z*): 303 (M^+).

7-Oxo-β-lycorane (17): A mixture of **16 (**50 mg, 0.17 mmol) and freshly distilled $POCl_3$ (2 mL) was heated at 90 °C for 20 h in a sealed glass tube. The mixture was slowly poured into cold water (5 mL) with stirring. The water layer was made alkaline (pH 11) with 8% NaOH, and then extracted with $CHCl_3$ (20 mL x 3). Concentration and chromatography gave **17** as white amorphous of mp

157–158 °C (33 mg, 90%). ^{1}H NMR: 1.25–1.58 (5H, m), 2.03 (3H, m), 2.14 (1H, m), 2.34 (1H, brs), 2.69 (1H, dd, *J* = 11.0, 12.6), 2.80 (1H, dd, *J* = 11.0, 11.7), 3.58 (1H, m), 3.79 (1H, dd, *J* = 9.2, 11.9), 5.99 (2H, s), 6.69 (1H, s), 7.58 (1H, s). ^{13}C NMR: 26.1, 27.2, 28.8, 29.7, 41.0, 44.7, 45.3, 66.4, 101.5, 103.7, 108.4, 125.6, 136.8, 146.4, 150.4, 159.8. FTIR ($CHCl_3$): 3020, 1639. EIMS (*m/z*): 271 (M^+), 153, 110.

β-Lycorane (2): A mixture of **17** (54 mg, 0.20 mmol) and $LiAlH_4$ (22 mg, 0.57 mmol) in THF (2 mL) was stirred at rt for 3 h and was then successively treated with water (0.5 mL), 8% NaOH (1.0 mL), and water (1.0 mL). The water layer was extracted with $CHCl_3$ (10 mL x 3). Concentration and recrystallization from hexane gave **2** as colorless plates of mp 86–88 °C (47 mg, 90%). ^{1}H NMR: 1.15–1.21 (2H, m), 1.45–1.54 (3H, m), 1.67 (1H, brs), 1.93–2.06 (3H, m), 2.26–2.34 (2H, m), 2.50 (1H, dd, *J* = 10.4, 11.3), 3.35 (1H, d, *J* = 14.1), 3.38 (1H, m), 4.07 (1H, d, *J* = 14.1), 5.89 (1H, brs), 5.90 (1H, brs), 6.51 (1H, s), 6.72 (1H, s). ^{13}C NMR: 26.5, 28.3, 28.9, 30.1, 41.8, 43.0, 53.9, 57.3, 71.9, 100.7, 105.4, 106.9, 128.5, 131.3, 145.6, 146.2. FTIR ($CHCl_3$): 2928, 1593, 1485. EIMS (*m/z*): 257 (M^+).

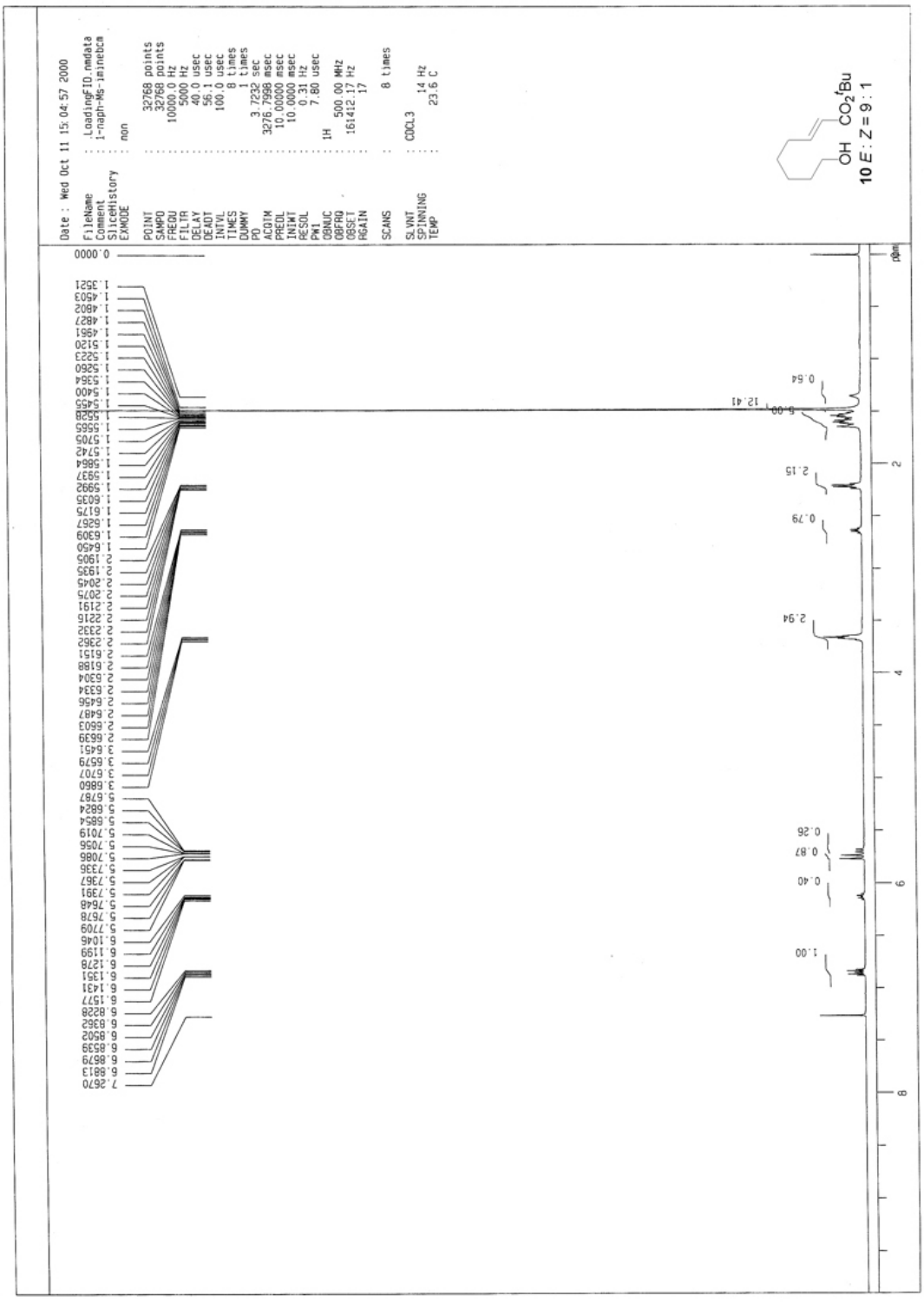
***tert*-Butyl 1,2-*cis*-2,3-*trans*-2-amino-3-benzo[1,3]dioxol-5-ylcyclohexylacetate (4b)**: To a solution of a 1:3 mixture of **5a** and **5b** (1.7 g, 4.7 mmol) in EtOH (50 mL), 10% HCl (2 mL) and Zn powder (3.5 mg, 350 mmol) were added at 0 °C. The mixture was stirred at rt for 2 d, filtrated, and then concentrated. The residue was diluted with AcOEt (40 mL), and washed with satd $NaHCO_3$ (20 mL) and brine (20 mL). Concentration and chromatography gave **4a** as a colorless oil (303 mg, 19%) and **4b** as a colorless oil (1.21 g, 77%). **4b**: ^{1}H NMR: 1.21–1.29 (1H, m), 1.43 (1H, m), 1.45 (9H, s), 1.51–1.57 (2H, m), 1.74–1.82 (2H, m), 2.13 (2H, brs), 2.26 (1H, dd, *J* = 9.5, 15.1), 2.37 (1H, ddd, *J* = 3.5, 10.8, 12.5), 2.40–2.50 (1H, m), 2.69 (1H, dd, *J* = 4.3, 15.1), 3.08 (1H, dd, *J* = 4.6, 10.8), 5.92 (2H, s), 6.65–6.78 (3H, m). ^{13}C NMR: 20.5, 28.1, 29.3, 32.7, 34.0, 36.2, 45.9, 56.7, 80.2, 100.8, 107.6, 108.3, 121.0, 137.7, 146.1, 147.9, 173.5. FTIR (neat): 3449, 3020, 1717. FABMS (*m/z*): 333 (M^+), 277 (M^+–^{t}Bu+H). HRMS–FAB: Calcd. for $C_{19}H_{27}NO_4$ 333.1940. Found 333.1921.

3a,7a-*cis*-7,7a-*trans*-7-Benzo[1,3]dioxol-5-yl-octahydroindol-2-one (14b): A solution of **4b** (1.0 g, 3

mmol) and sodium (340 g, 15 mmol) in MeOH (30 mL) was stirred at rt for 2 d, and treated with brine (10 mL). The mixture was extracted with AcOEt (30 mL x 3). Concentration and recrystallization from $CHCl_3$/hexane afforded **14b** as white amorphous of mp 192–193 °C (683 mg, 88%). ^{1}H NMR: 1.37–154 (2H, m), 1.63–1.90 (4H, m), 2.21 (1H, dd, *J* = 8.6, 16.5), 2.36 (1H, m), 2.37 (1H, dd, *J* = 12.4, 16.5), 2.74 (1H, m), 3.88 (1H, dd, *J* = 7.2, 10.3), 5.54 (1H, brs), 5.94 (2H, s), 6.62 (1H, d, *J* = 7.8), 6.67 (1H, s), 6.74 (1H, d, *J* = 7.8). ^{13}C NMR: 21.1, 26.0, 30.6, 33.4, 34.9, 48.3, 60.0, 100.9, 107.5, 108.3, 120.7, 137.0, 146.3, 147.9, 177.7. FTIR ($CHCl_3$): 3425, 2936, 1686. EIMS (*m/z*): 260 (M^++H).

3a,7a-*trans*-7,7a-*trans*-7-Benzo[1,3]dioxol-5-yloctahydroindole (15b): A mixture of **14b** (100 mg, 0.39 mmol) and $LiAlH_4$ (30 mg, 0.8 mmol) in THF (2 mL) was stirred at reflux for 2 h, and then treated with water (1 mL), 8% NaOH (3 mL) and water (3 mL). The mixture was extracted with $CHCl_3$ (20 mLx3). Concentration and chromatography ($CHCl_3$/MeOH = 10/1) gave **15b** as a pale yellow oil (80 mg, 94%). ^{1}H NMR: 1.26 (1H, brs), 1.40–1.86 (7H, m), 2.27–2.50 (4H, m), 3.01 (1H, ddd, *J* = 8.1, 8.4, 10.5), 3.12 (1H, dd, *J* = 6.5, 10.5), 5.93 (2H, s), 6.64 (1H, dd, *J* = 1.4, 7.8), 6.70 (1H, d, *J* = 1.4), 6.74 (1H, d, *J* = 7.8). ^{13}C NMR: 21.2, 26.2, 27.2, 32.4, 38.1, 43.3, 44.9, 63.4, 100.8, 107.8, 108.2, 120.8, 138.7, 146.0, 147.8. FTIR (neat): 3691, 2927, 1485. EIMS (*m/z*): 246 (M^++H).

α-Lycorane (1): To a solution of **15b** (120 mg, 0.49 mmol) in THF (3 mL), Eschenmoser's salt ($Me_2N{=}CH_2I$, 138 mg, 0.75 mmol) was added at rt. The mixture was stirred at 40 °C for 19 h, and then treated with 10% NaOH (2 mL) and brine (20 mL). Water layer was extracted with $CHCl_3$ (20 mL x 3). Concentration and recrystallization from hexane gave **1** as colorless plates of mp 93–94 °C (101 mg, 80%). ^{1}H NMR: 1.12–1.26 (2H, m), 1.50–1.94 (6H, m), 2.19–2.30 (1H, m), 2.34–2.47 (1H, m), 2.52 (1H, dd, *J* = 6.8, 10.0), 2.84 (1H, ddd, *J* = 3.5, 9.1, 9.7), 3.19 (1H, ddd, *J* = 8.1, 8.1, 9.7), 3.75 (1H, d, *J* = 15.1), 4.14 (1H, d, *J* = 15.1), 5.90 (2H, s), 6.60 (1H, s), 6.70 (1H, s). ^{13}C NMR: 20.7, 24.9, 26.0, 27.7, 33.8, 36.8, 54.2, 54.4, 64.5, 100.6, 104.4, 106.9, 128.3, 134.8, 145.4, 146.2. FTIR (neat): 2928, 1481, 1381. EIMS (*m/z*): 257 (M^+).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DEADT 56.1 usec
INTVL 100.0 usec
TIMES 8 times
DUMMY 1 times
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ACQTM 3276.7998 msec
PREDL 10.00000 msec
INIWT 10.0000 msec
RESOL 0.31 Hz
PW1 7.80 usec
OBNUC 1H
OBFRQ 500.00 MHz
OBSET 161412.17 Hz
AGAIN 17
SCANS 8 times
SLVNT CDCl3
SPINNING 14 Hz
TEMP 23.6 C
OH CO2tBu
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12.41
6.00
2.15
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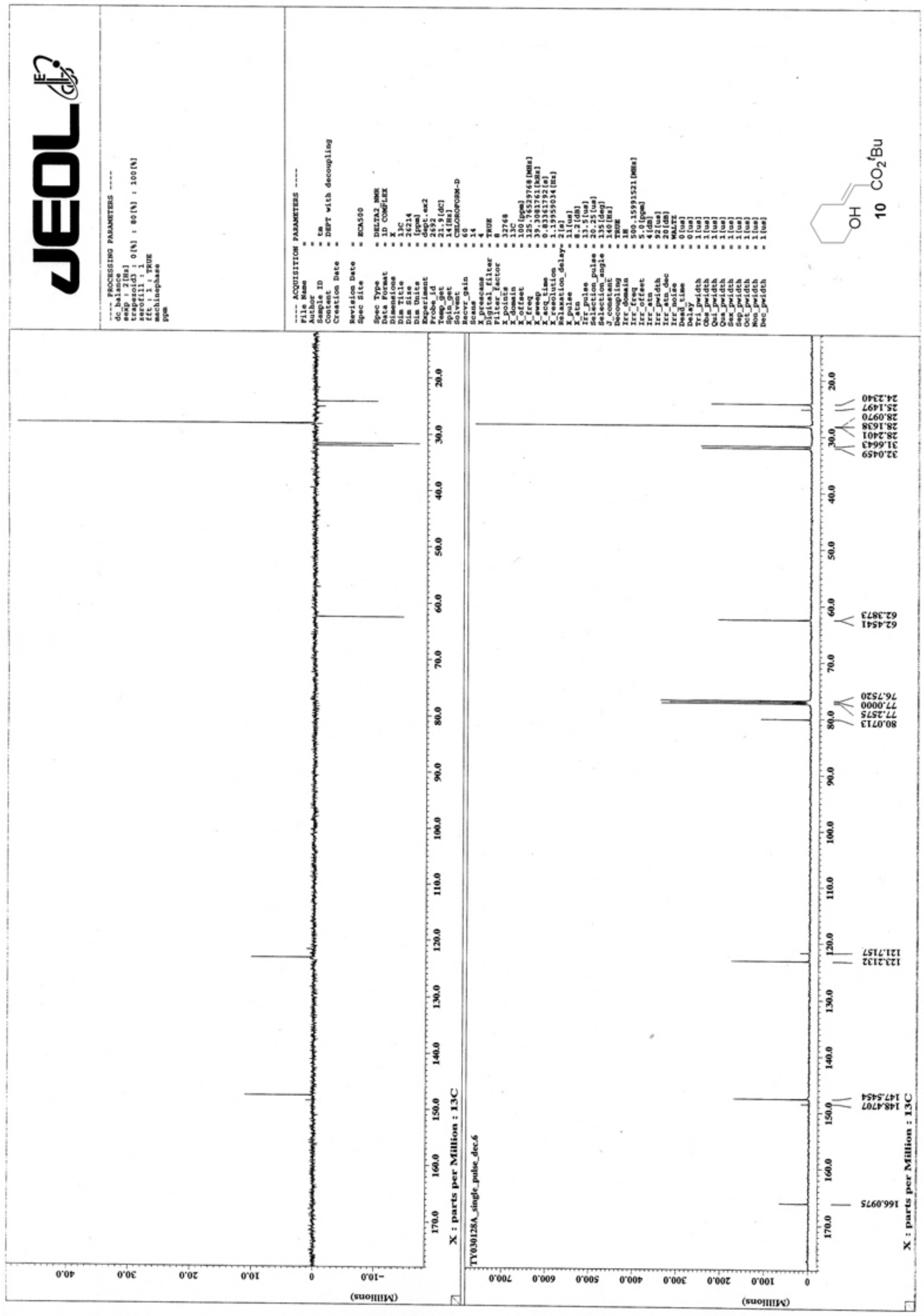
2.94
0.26
0.87
0.40
1.00
ppm


JEOL
10
X : parts per Million : 13C
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147.5454
123.2132
121.7157
80.0713
77.2575
77.0000
76.7520
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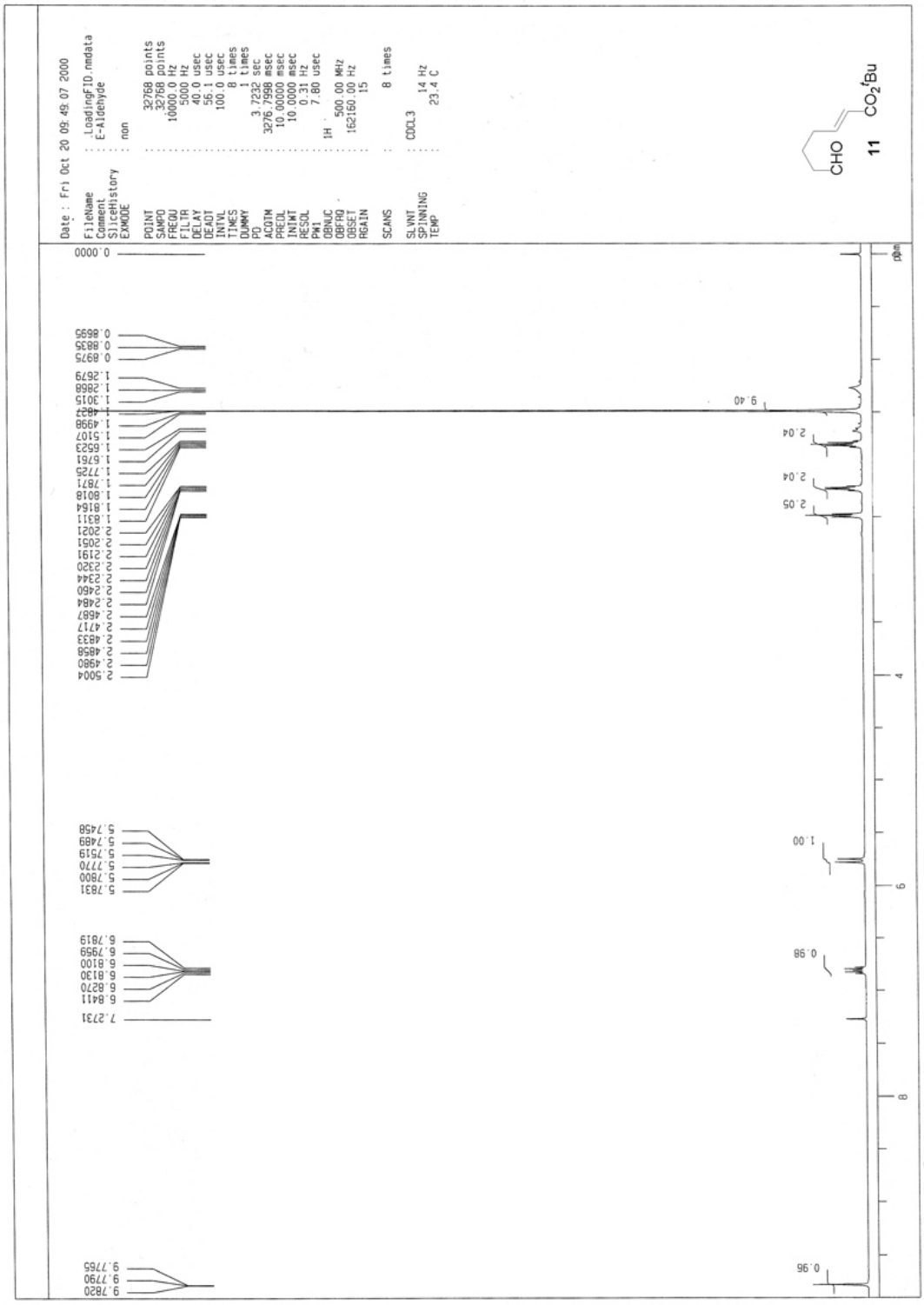
62.3873
32.0459
31.6643
28.2401
28.1638
28.0970
25.1497
24.2340
(Millions)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TIMES 8 times
DUMMY 1 times
PD 3.7232 sec
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PREDL 10.00000 msec
INIWT 10.0000 msec
RESOL 0.31 Hz
PW1 7.80 usec
OBNUC 1H
OBFRQ 500.00 MHz
OBSET 162160.00 Hz
AGAIN 15
SCANS 8 times
SLVNT CDCL3
SPINNING 14 Hz
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CO2tBu
11
0.0000
0.8695
0.8835
0.8975
1.2679
1.2868
1.3015
1.4827
1.4998
1.5107
1.6523
1.6761
1.7725
1.7871
1.8018
1.8164
1.8311
2.2021
2.2051
2.2191
2.2320
2.2344
2.2460
2.2684
2.4687
2.4717
2.4833
2.4858
2.4980
2.5004
5.7458
5.7489
5.7519
5.7770
5.7800
5.7831
5.7819
5.7959
5.8100
5.8130
5.8270
5.8411
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9.7820
9.40
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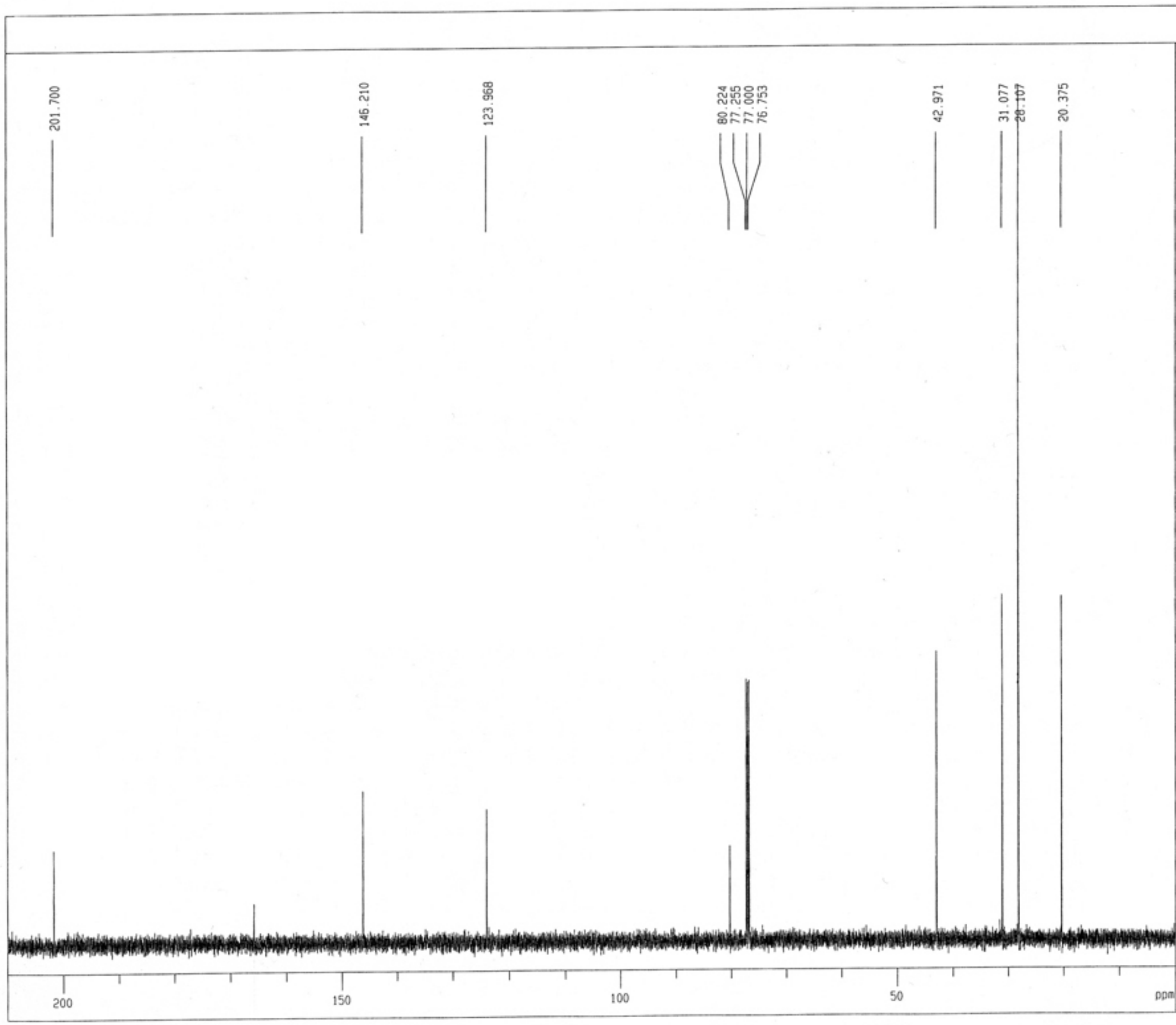
2.04
2.05
1.00
0.98
0.96
ppm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77.000
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20.375
200
150
100
50
ppm
Date :
FileName : .LoadingFID.nmdata
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INTVL : 29.5 usec
TIMES : 512 times
DUMMY : 1 times
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PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 1.03 Hz
PW1 : 6.40 usec
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OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 15
IRNUC : 1H
IRFRQ : 500.00 MHz
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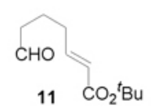
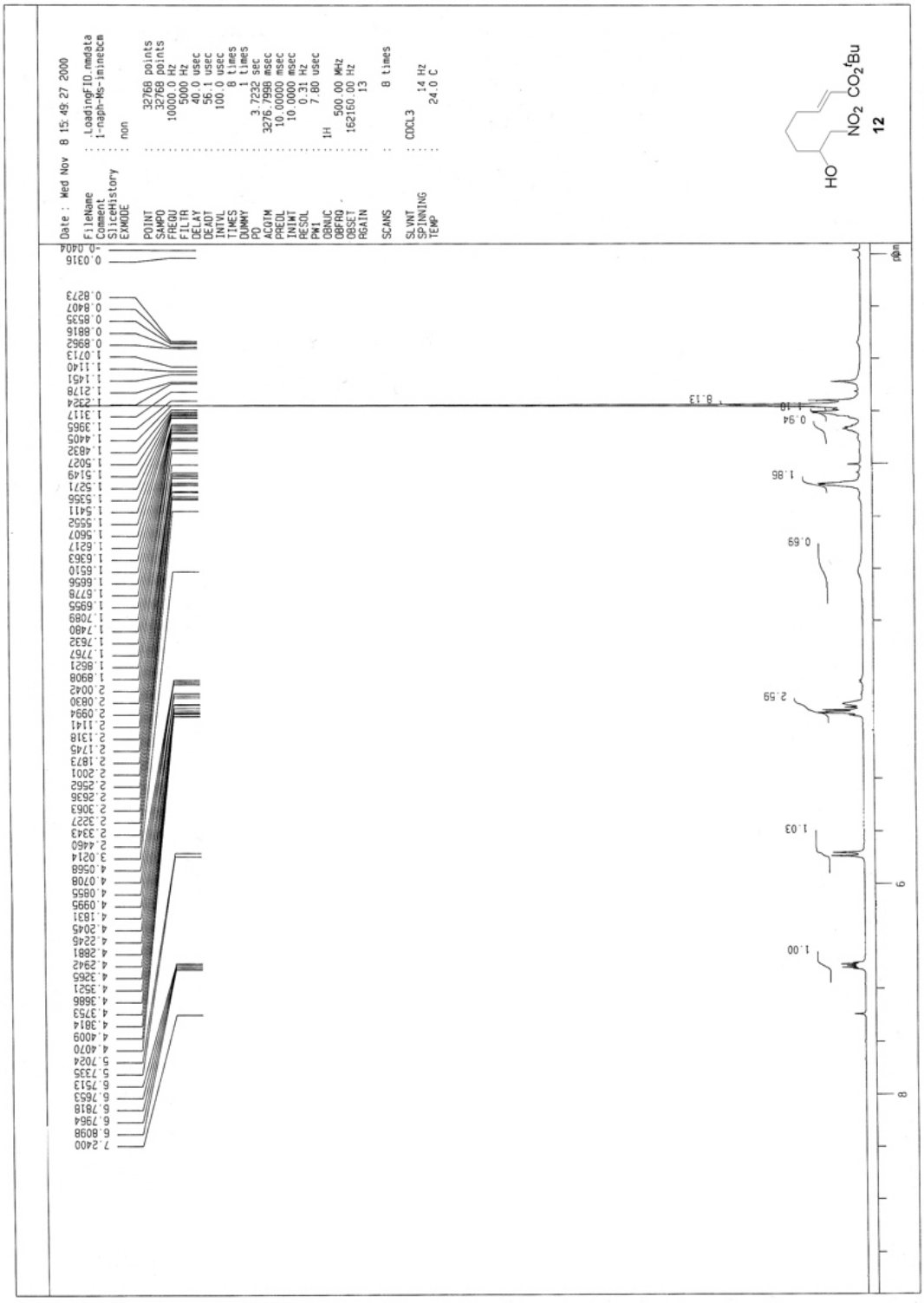
SPINNING : 14 Hz
TEMP : 24.3 C


CHO
$CO_2{}^tBu$

11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INTVL 100.0 usec
TIMES 8 times
DUMMY 1 times
PD 3.7232 sec
ACQTM 3276.7998 msec
PREDL 10.00000 msec
INIWT 10.0000 msec
RESOL 0.31 Hz
PW1 7.80 usec
1H
OBNUC
OBFRQ 500.00 MHz
OBSET 162160.00 Hz
AGAIN 13
SCANS 8 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 24.0 C
HO
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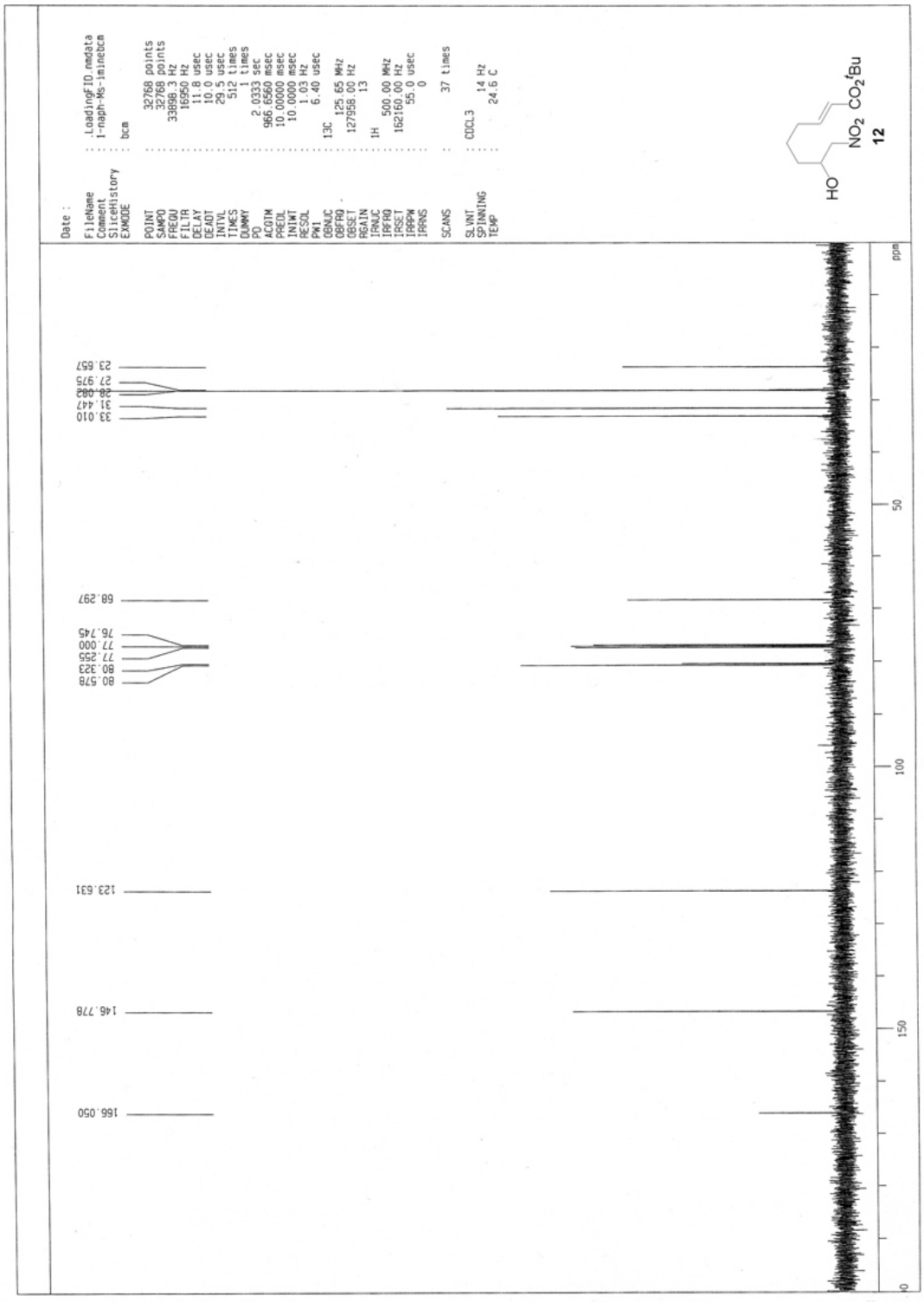
12
8.13
1.10
0.94
1.86
0.69
2.59
1.03
1.00
ppm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FILTR 16950 Hz
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DEADT 10.0 usec
INTVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 10.00000 msec
INIWT 10.0000 msec
RESOL 1.03 Hz
PW1 6.40 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
AGAIN 13
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRPPW 55.0 usec
IRPNS 0
SCANS 37 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 24.6 C
HO
NO2 CO2tBu
12
23.657
27.975
28.082
31.447
33.010
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ppm
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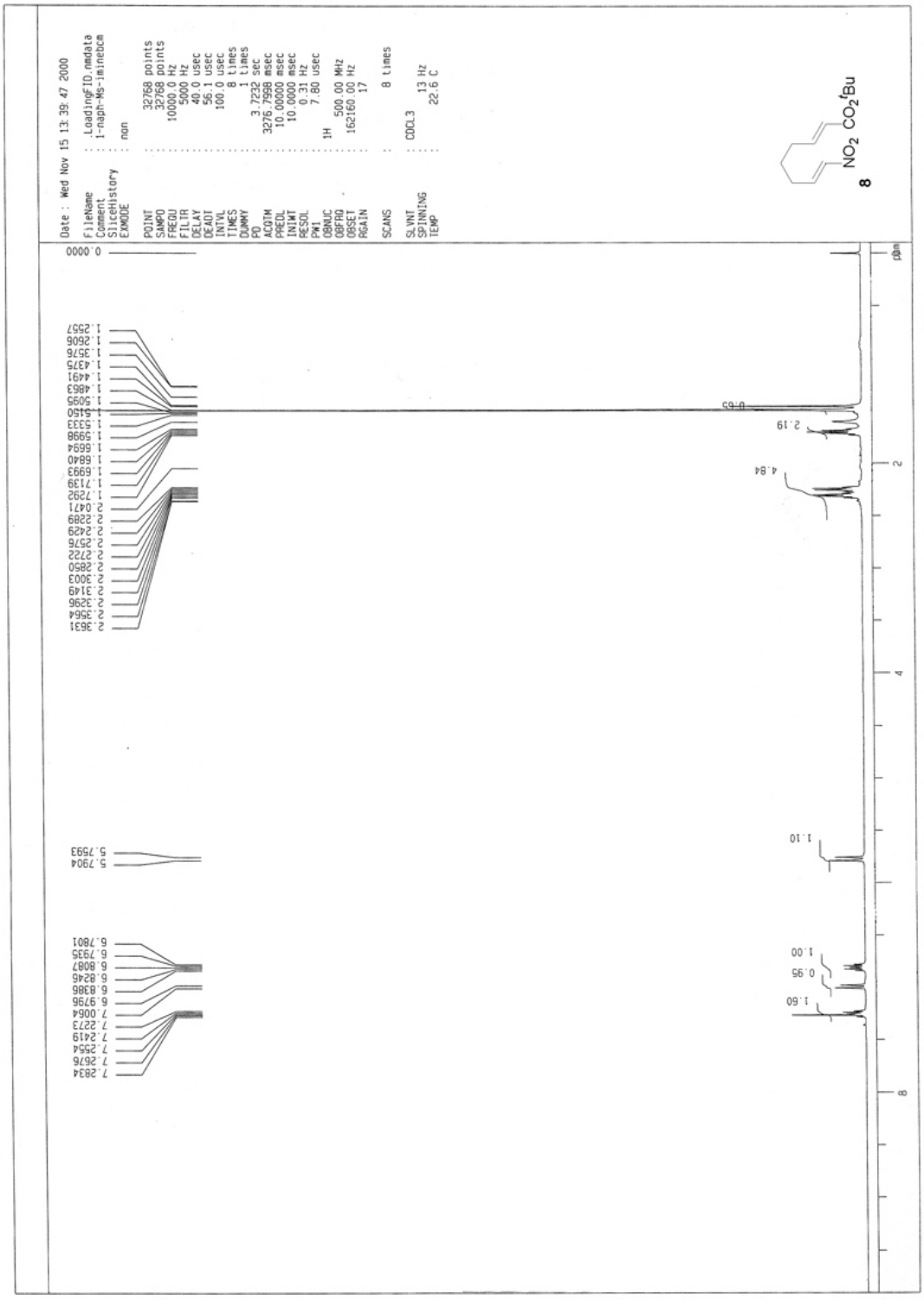
50
100
150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DEADT 56.1 usec
INTVL 100.0 usec
TIMES 8 times
DUMMY 1 times
PD 3.7232 sec
ACQTM 3276.7998 msec
PREDL 10.00000 msec
INIWT 10.0000 msec
RESOL 0.31 Hz
PW1 7.80 usec
OBNUC 1H
OBFRQ 500.00 MHz
OBSET 162160.00 Hz
AGAIN 17
SCANS 8 times
SLVNT CDCL3
SPINNING 13 Hz
TEMP 22.6 C
8
NO2 CO2tBu
0.0000
1.2557
1.2606
1.3576
1.4375
1.4491
1.4863
1.5095
1.5150
1.5333
1.5998
1.6694
1.6840
1.6993
1.7139
1.7292
2.0471
2.2289
2.2429
2.2576
2.2722
2.2850
2.3003
2.3149
2.3296
2.3564
2.3631
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5.7904
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6.8087
6.8246
6.8386
6.9796
7.0064
7.2273
7.2419
7.2554
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7.2834
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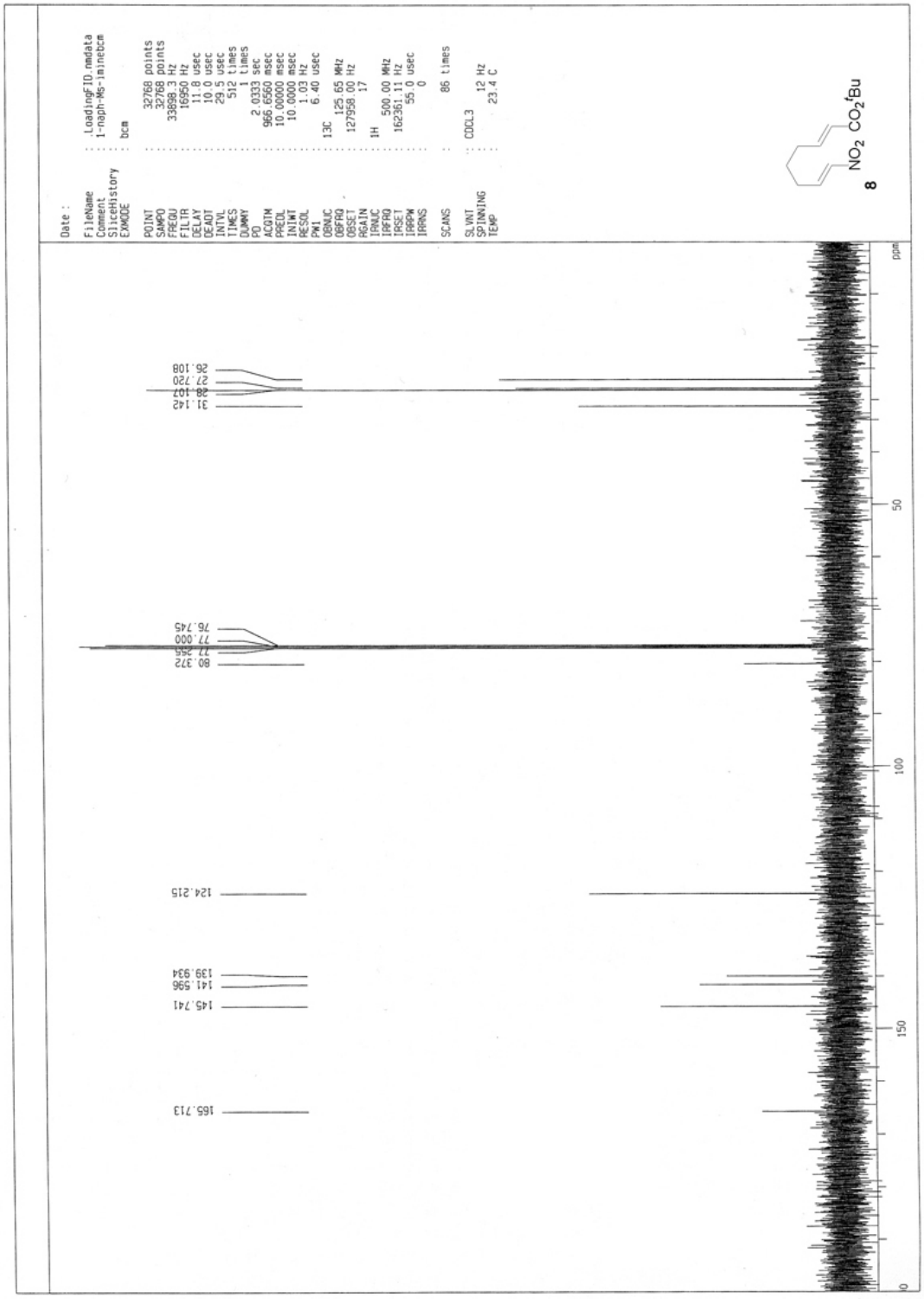
2.19
4.84
1.10
1.00
0.95
1.60
ppm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FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 512 times
DUMMY : 1 times
PD : 2.0333 sec
ACQTM : 966.6560 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 1.03 Hz
PW1 : 6.40 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
AGAIN : 17
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162361.11 Hz
IRPPW : 55.0 usec
IRPNS : 0
SCANS : 86 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 23.4 C
8
NO2 CO2tBu
26.108
27.720
28.107
31.142
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80.372
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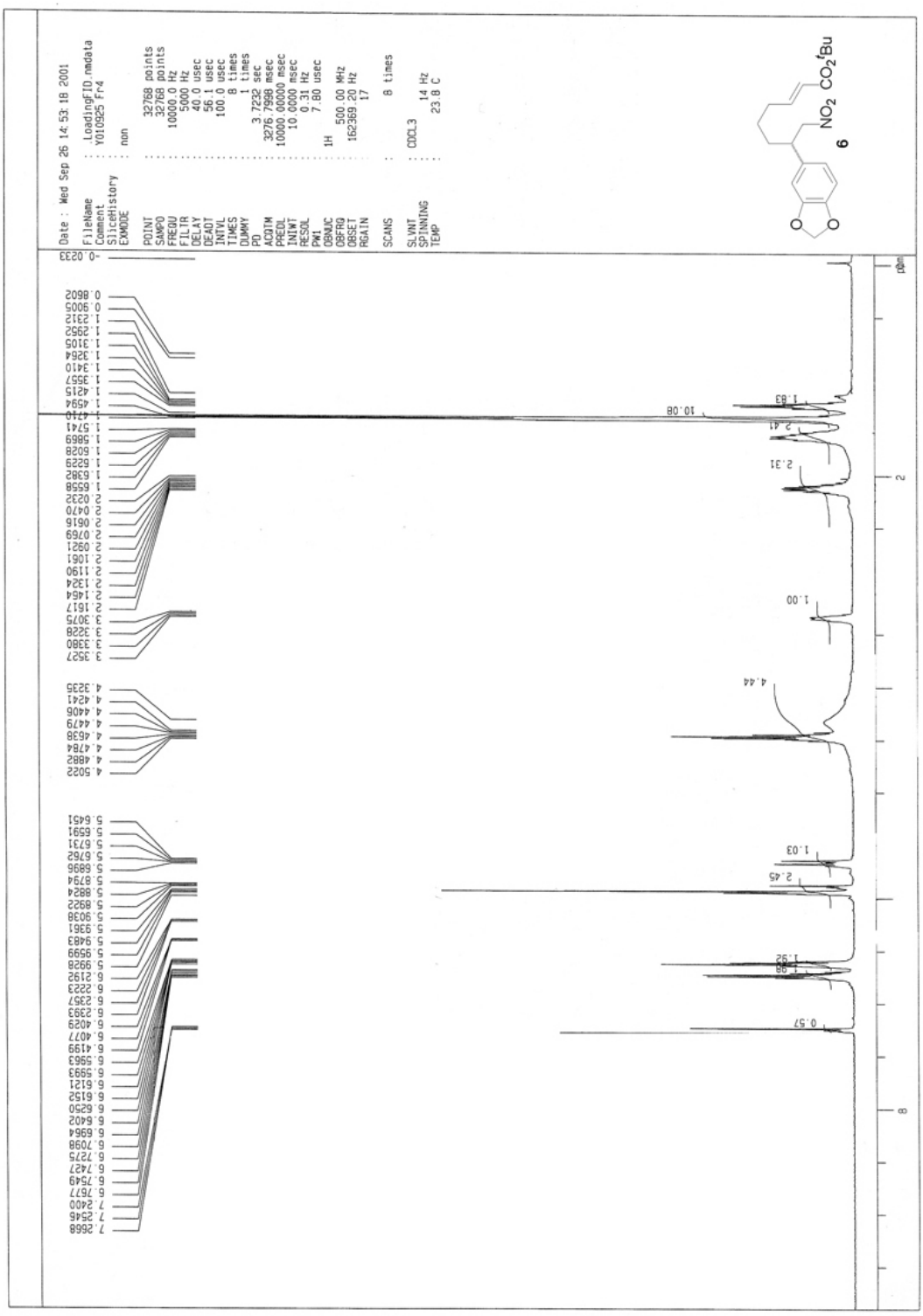
ppm
0
50
100
150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DEADT 56.1 usec
INTVL 100.0 usec
TIMES 8 times
DUMMY 1 times
PD 3.7232 sec
ACQTM 3276.7998 msec
PREDL 10000.00000 msec
INLVT 10.00000 msec
RESOL 0.31 Hz
PWX 7.80 usec
OBNUC 1H 500.00 MHz
OBFRQ 162369.20 Hz
OBSET 17
RGAIN
SCANS 8 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 23.8 C
6
NO2 CO2tBu
10.08
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1.00
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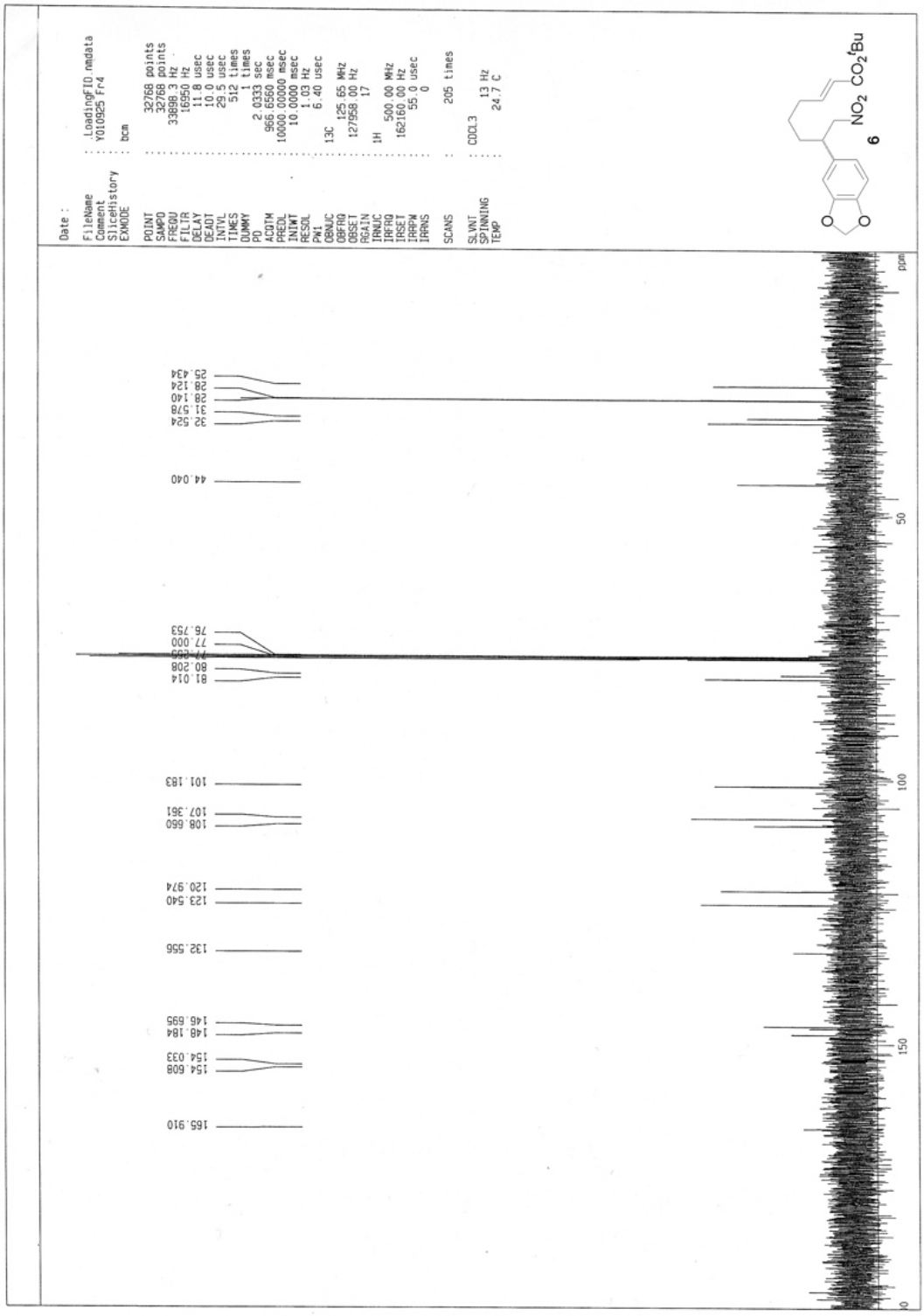
1.03
2.45
1.98
1.92
0.57
ppm
2
8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DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 512 times
DUMMY : 1 times
PD : 2.0333 sec
ACQTM : 966.6560 msec
PREDL : 10000.00000 msec
INIWT : 10.0000 msec
RESOL : 1.03 Hz
PW1 : 6.40 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
AGAIN : 17
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRRPW : 55.0 usec
IRRNS : 0
SCANS : 205 times
SLVNT : CDCl3
SPINNING : 13 Hz
TEMP : 24.7 C
6
29.434
29.124
28.140
31.578
32.524
44.040
76.753
77.000
77.255
80.208
81.014
101.183
107.361
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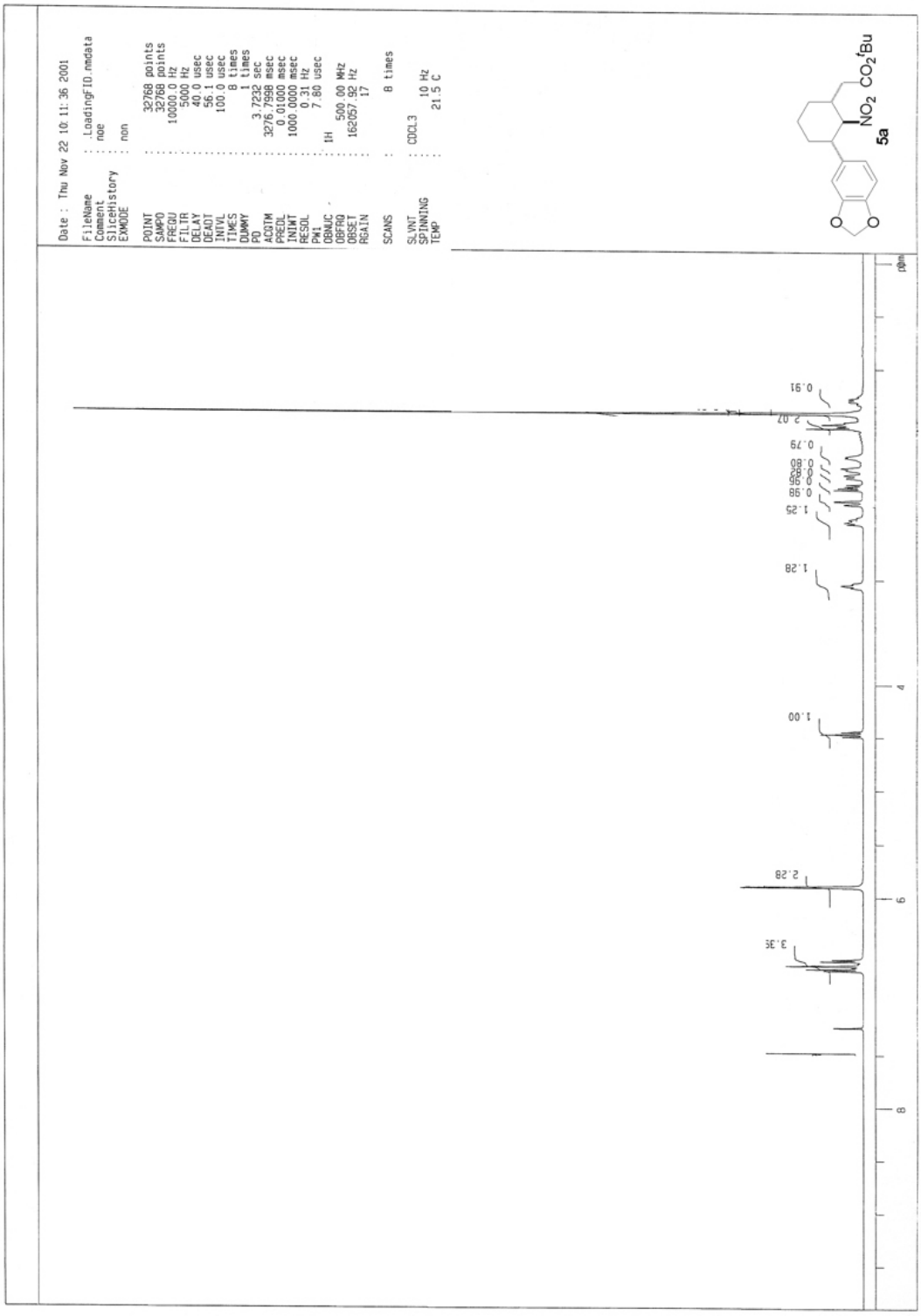
100
150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DEADT : 56.1 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 7.80 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162057.92 Hz
RGAIN : 17
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 21.5 C
NO2 CO2tBu
5a
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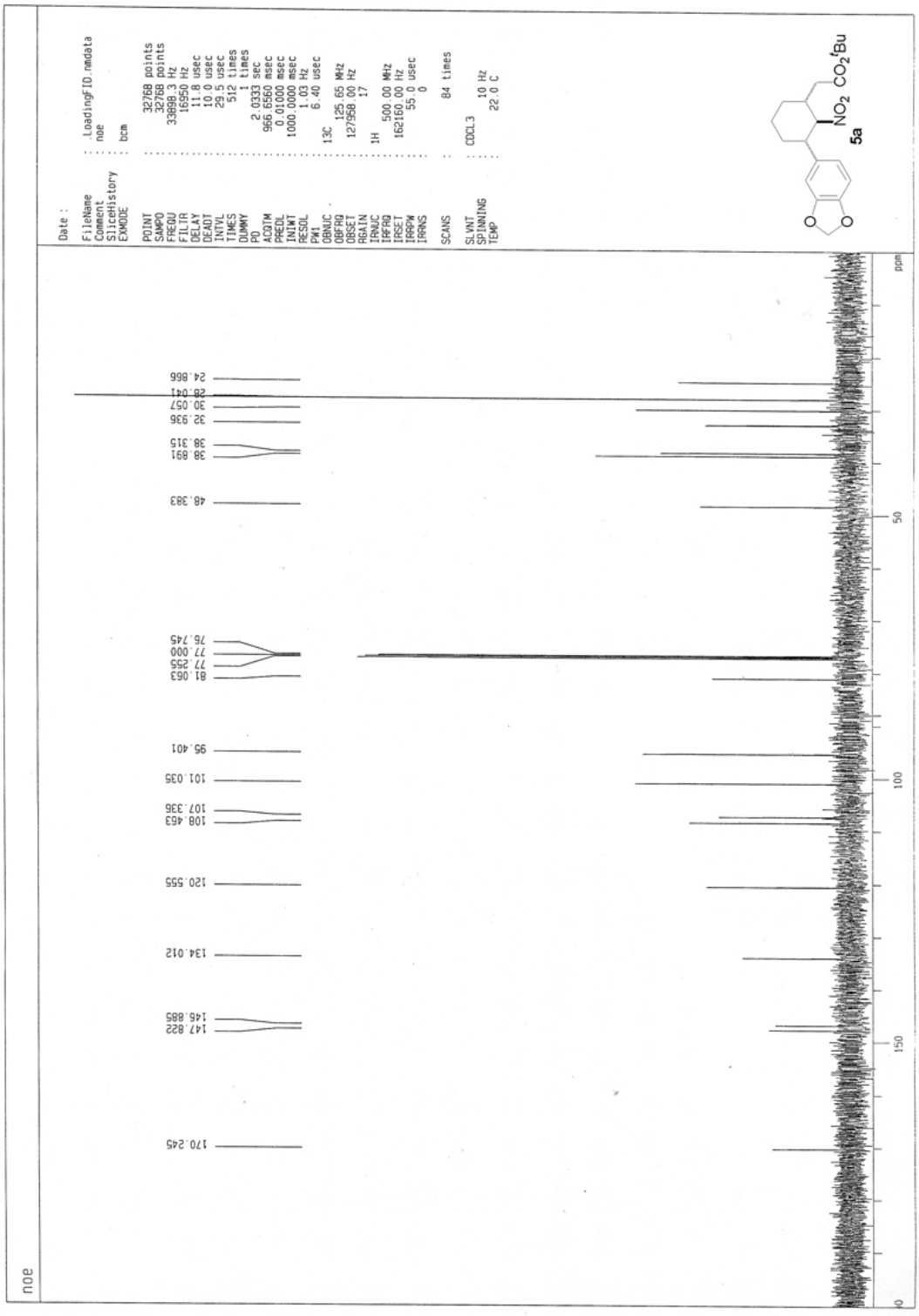
2.07
0.79
0.98
1.25
1.28
1.00
2.28
3.35
4
6
8
(ppm)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Date :
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FILTR 16950 Hz
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DEADT 10.0 usec
INTVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
AQSEL 0.0000 msec
INIWT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.40 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
AGAIN 17
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRRPW 55.0 usec
IRRNS 0
SCANS 84 times
SLVNT CDCL3
SPINNING 10 Hz
TEMP 22.0 C
24.866
28.041
30.057
32.936
38.315
38.891
48.383
76.745
77.000
77.255
81.063
95.401
101.035
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120.555
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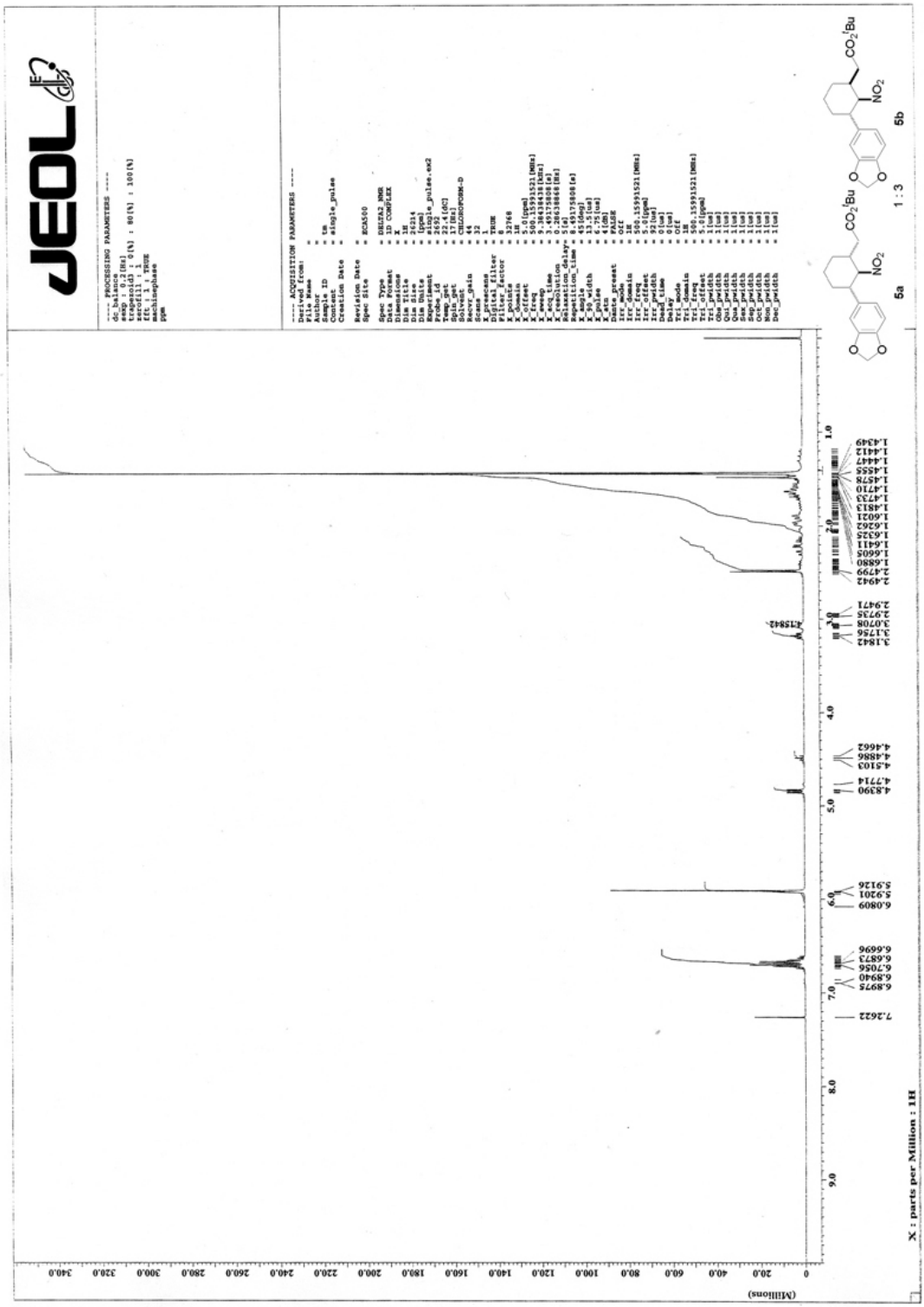
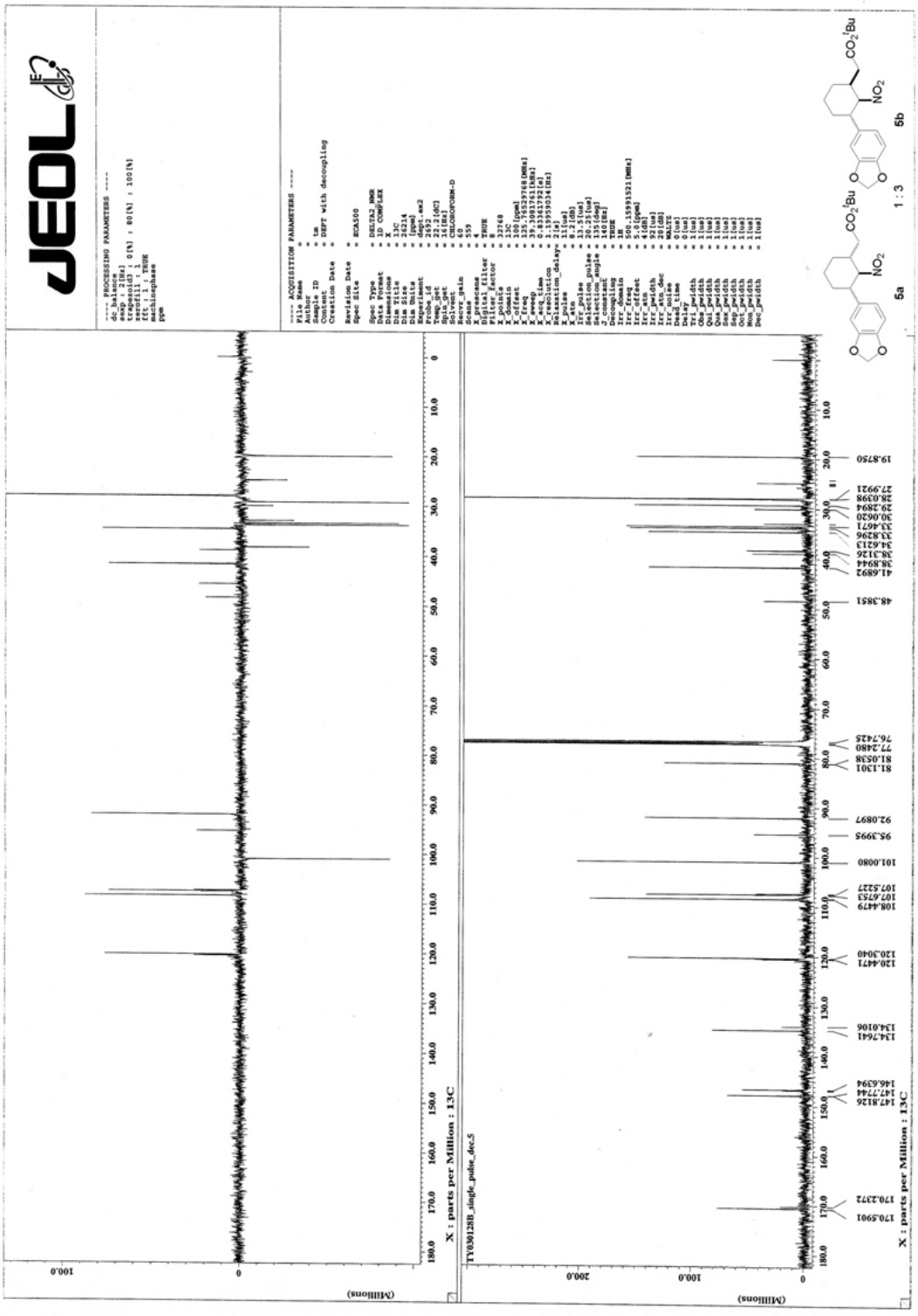
145.885
147.822
170.245
ppm
0
50
100
150
5a


JEOL
5a
1 : 3
5b
X : parts per Million : 1H


JEOL
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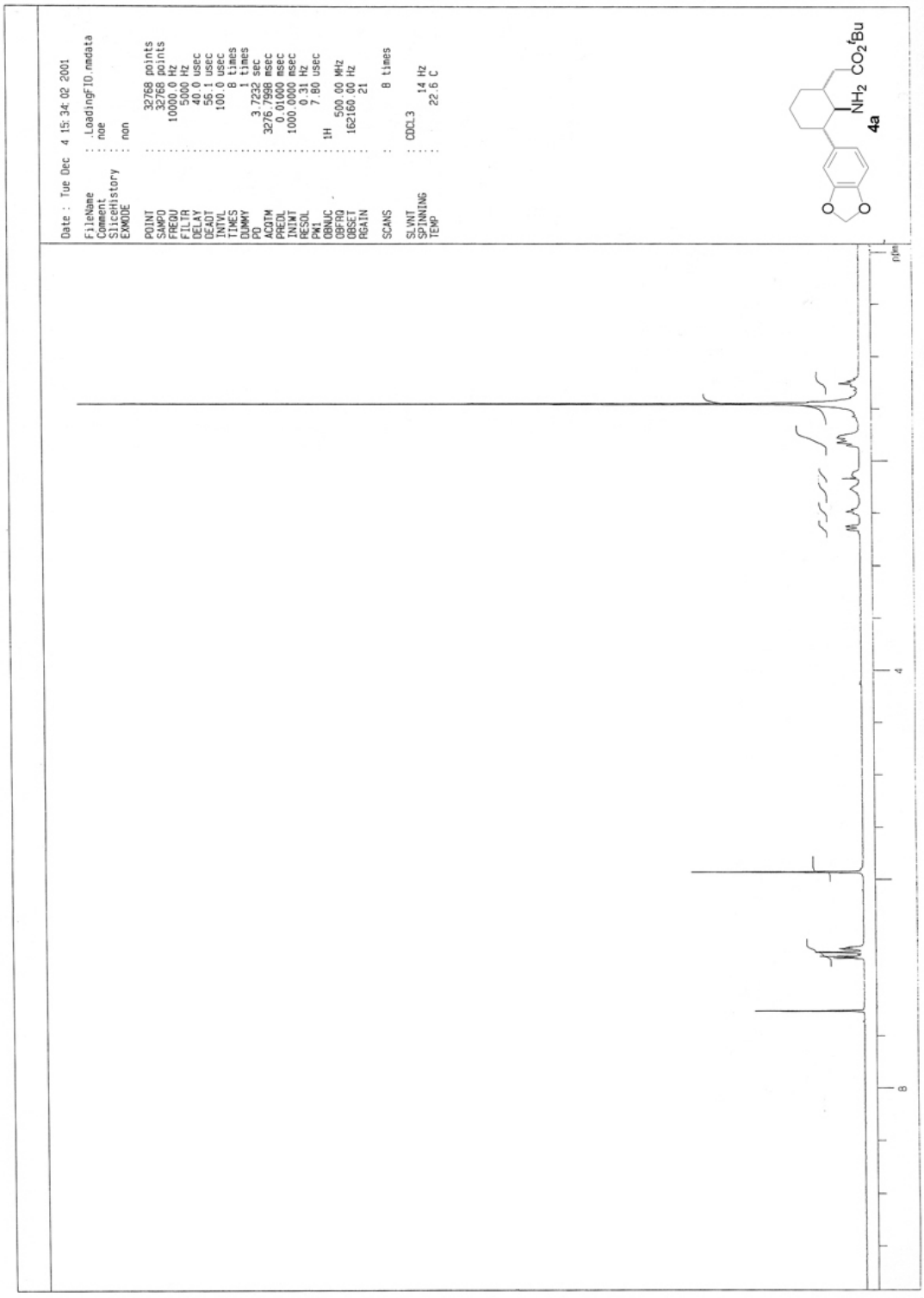
5b
X : parts per Million : 13C
TY050128B_single_pulse_dec.5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FILTR : 5000 Hz
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DEADT : 56.1 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 7.80 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
AGAIN : 21
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 14 Hz
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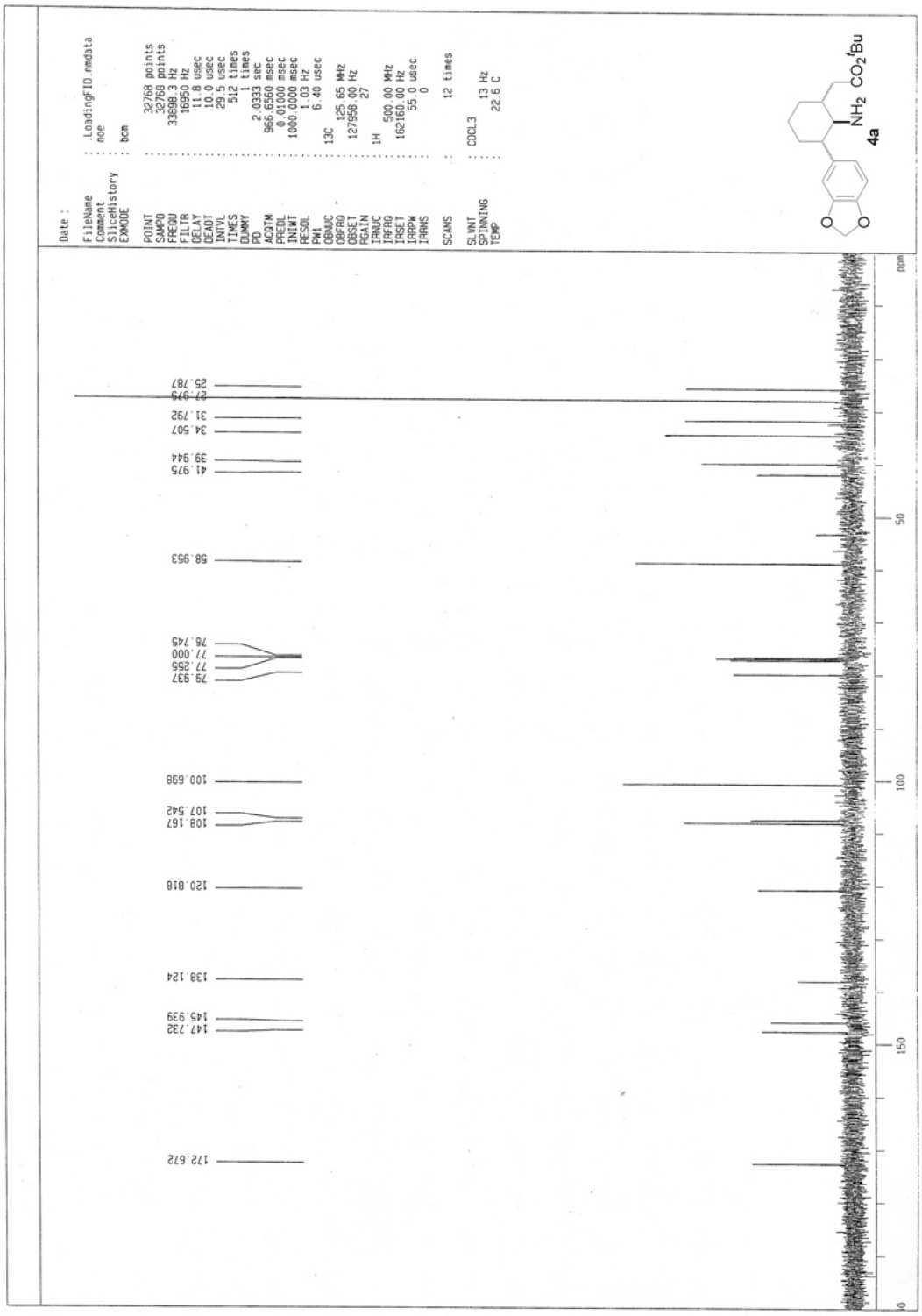
NH2
CO2tBu
4a
8
4
ppm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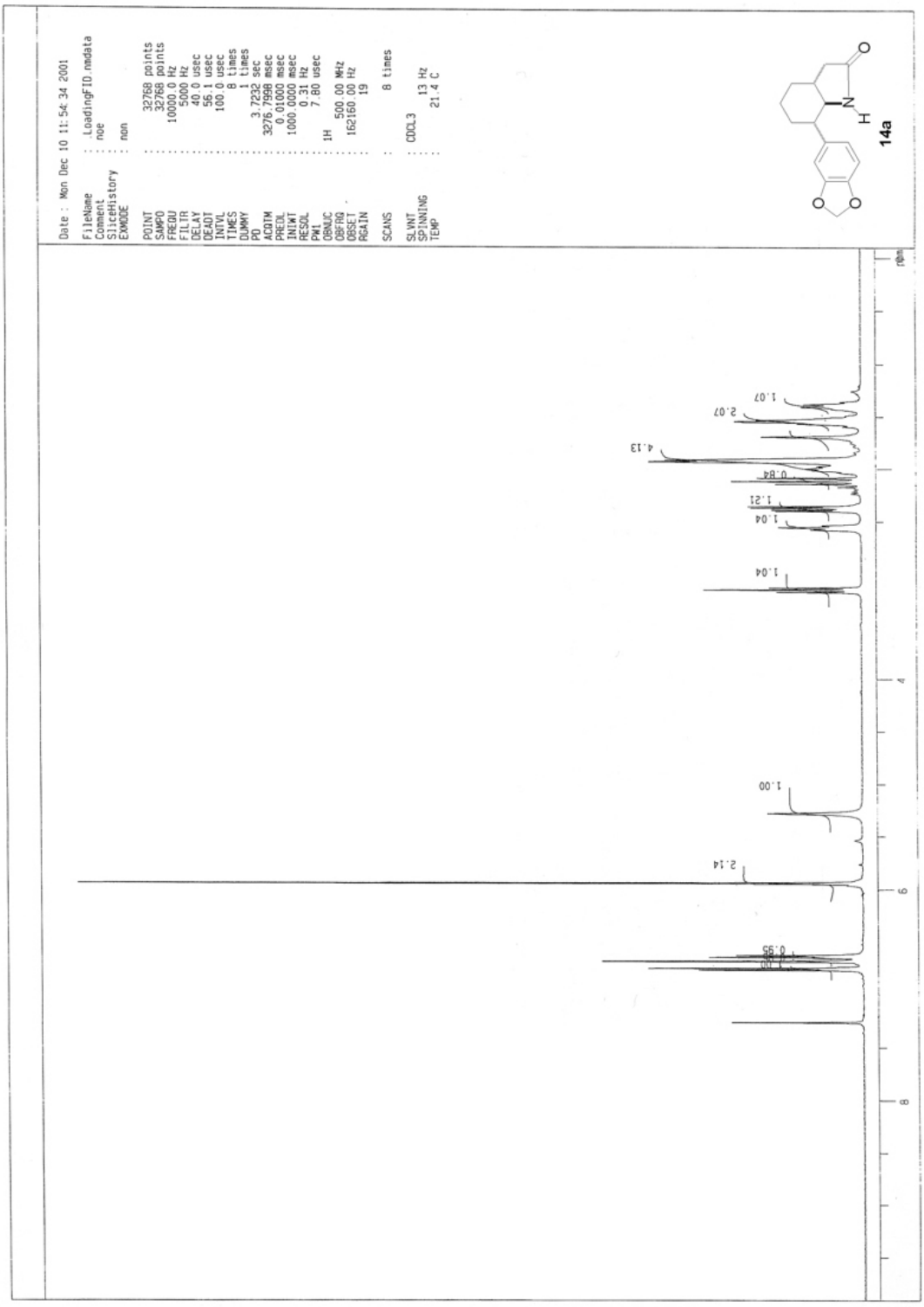
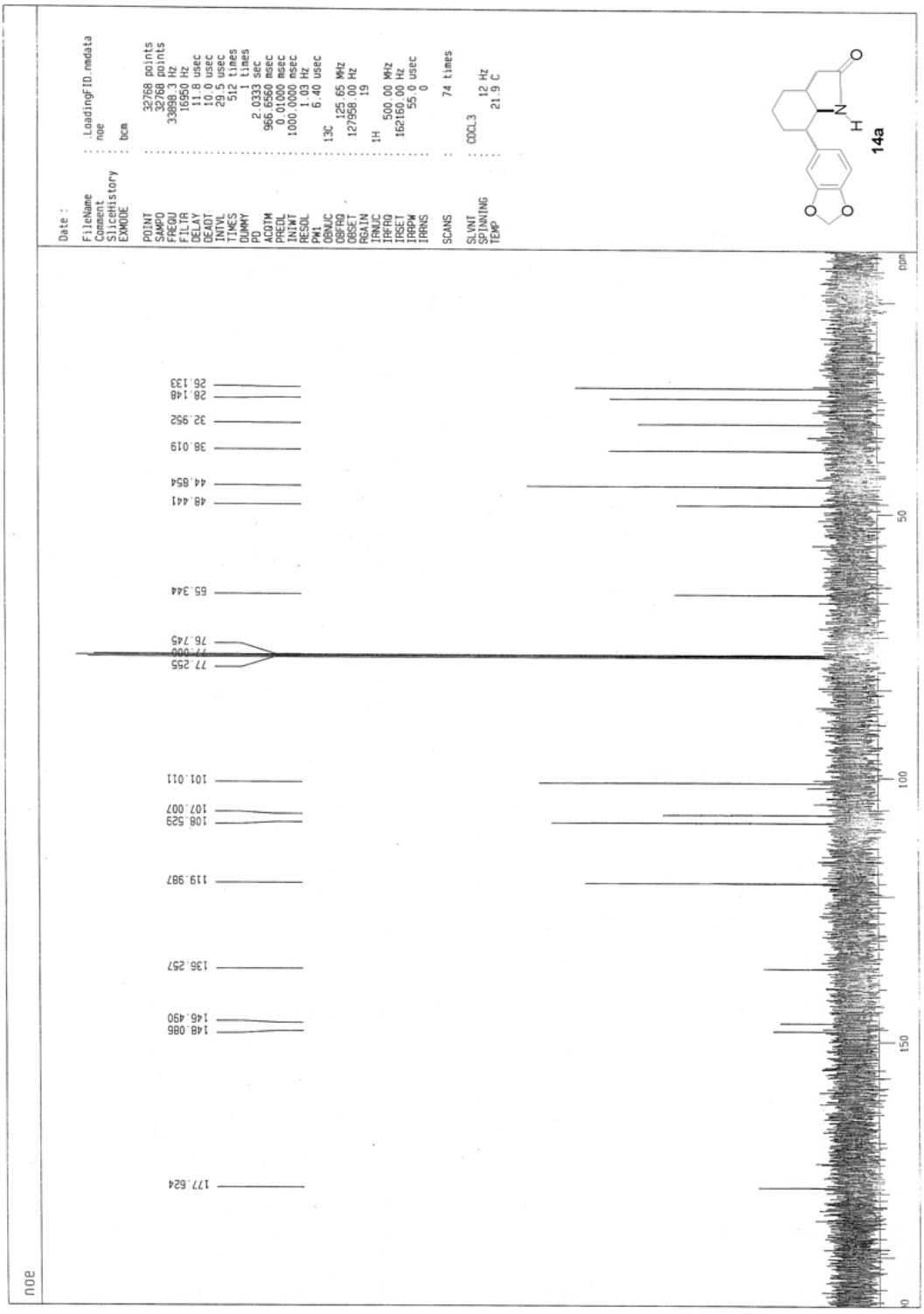
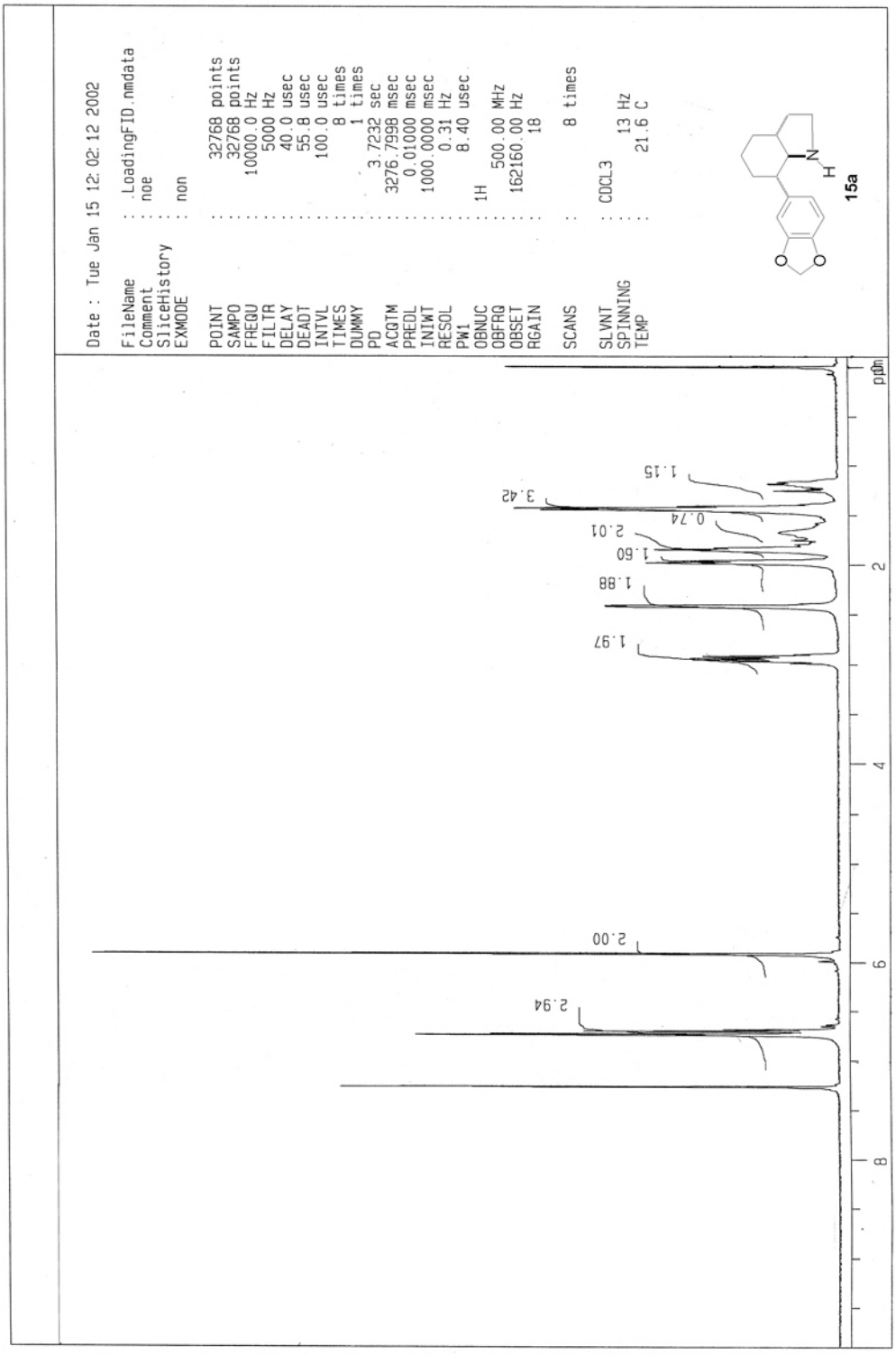
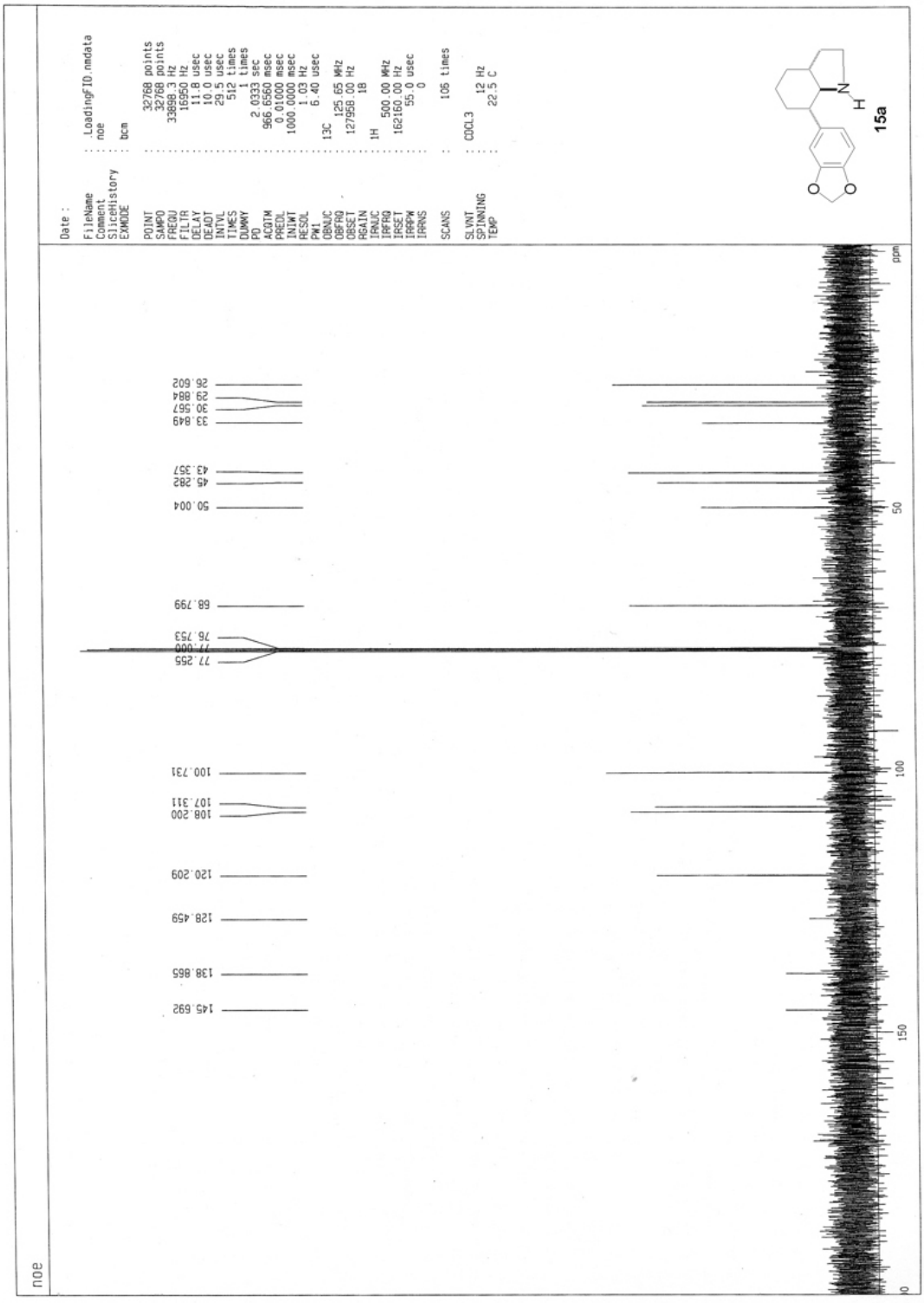
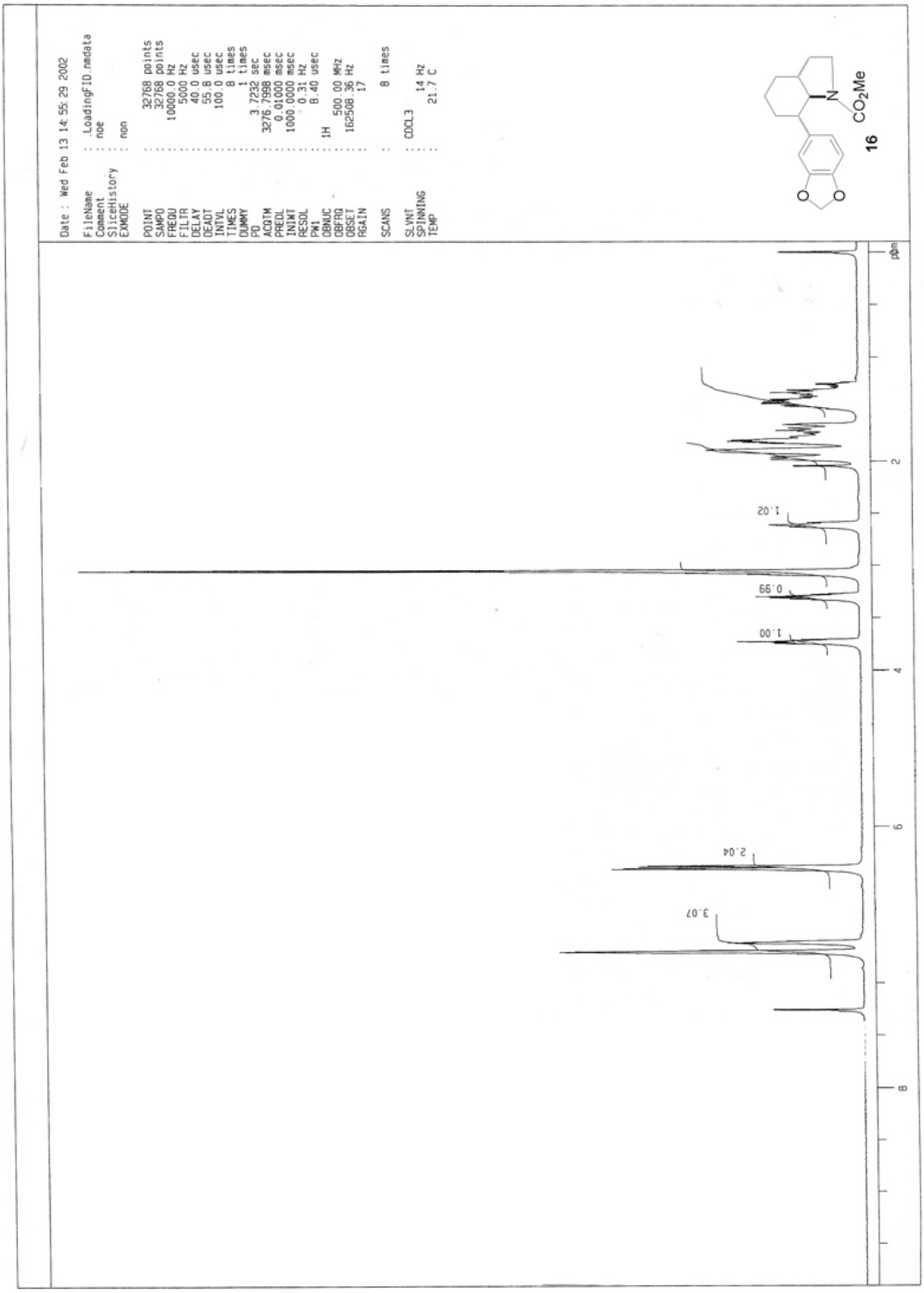
FileName : LoadingFID.nmdata
Comment : noe
SliceHistory : : bcm
EXMODE
POINT 32768 points
SAMPD 32768 points
FREQU 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INIVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 1.03 Hz
PWLX 6.40 usec
OBNUC 13C 125.65 MHz
OBFRQ 127958.00 Hz
OBSET 27
RGAIN
IRNUC 1H 500.00 MHz
IRFRQ 162160.00 Hz
IRSET 55.0 usec
IRRPW 0
IRNS
SCANS 12 times
SLVNT CDCL3 13 Hz
SPINNING 22.6 C
TEMP
NH2 CO2tBu
4a
25.787
27.975
31.792
34.507
39.944
41.975
58.953
76.745
77.000
77.255
79.937
100.698
107.542
108.167
120.818
138.124
145.939
147.732
172.672
ppm
0
50
100
150

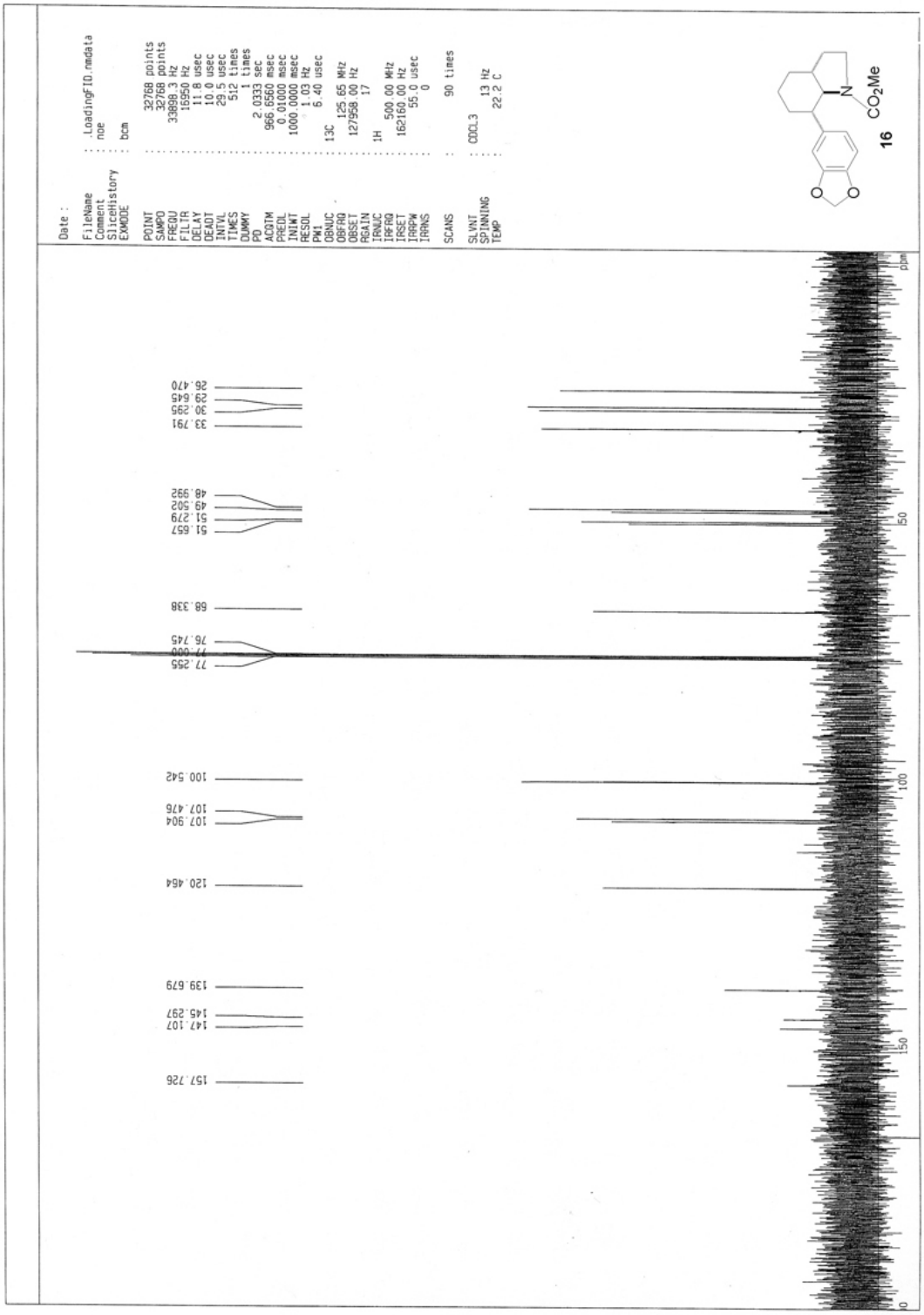

Date : Mon Dec 10 11:54:34 2001
FileName : LoadingFID.nmrdata
Comment : noe
SiteHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 50000 Hz
DELAY : 40.0 usec
DEADT : 56.1 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 7.80 usec
OBNUC : 1H 500.00 MHz
OBFRQ : 162160.00 Hz
OBSET : 19
AGAIN :
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.4 C
14a
1.07
2.07
4.13
0.84
1.21
1.04
1.04
1.00
2.14
0.95
ppm

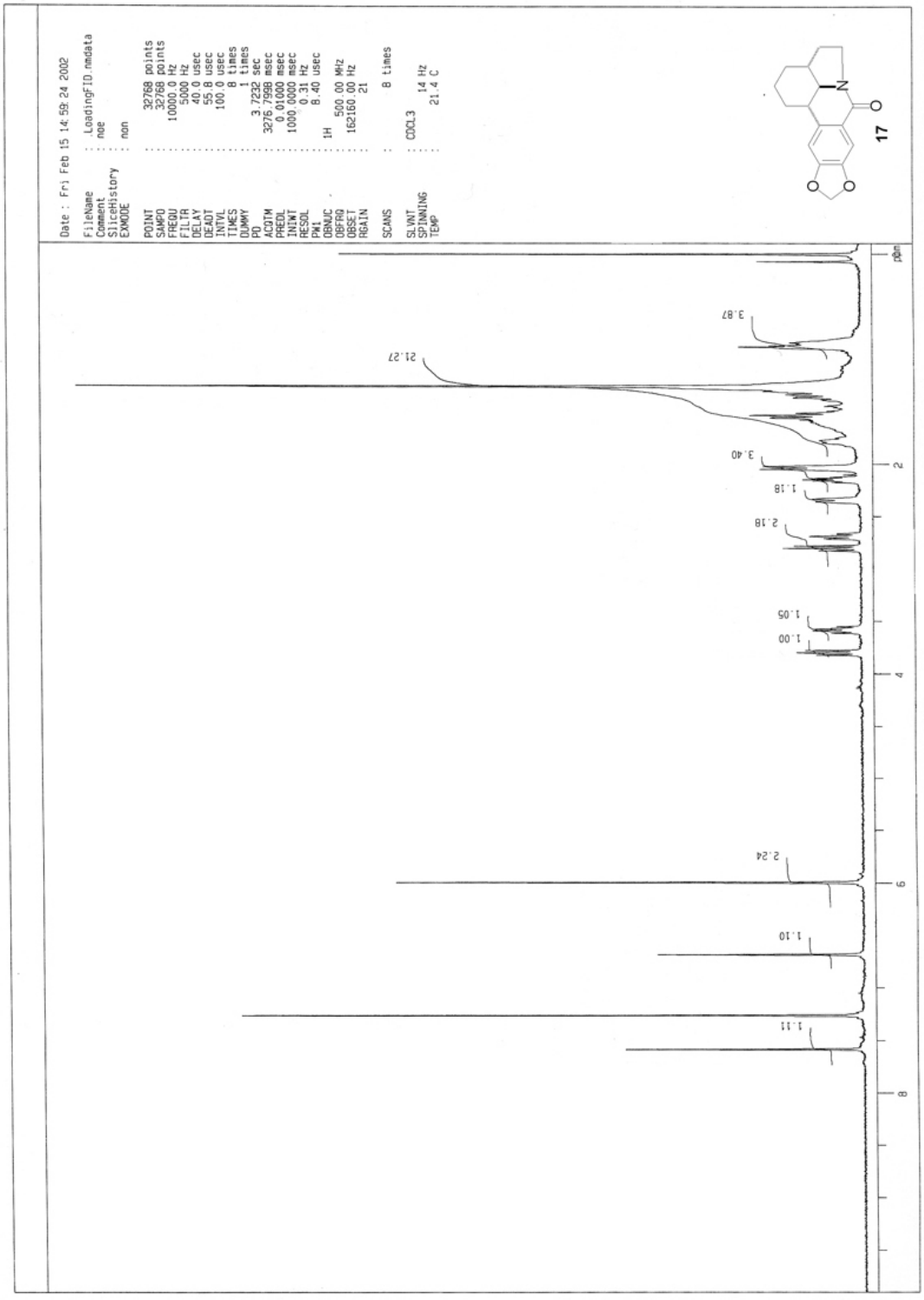

Date :
FileName .LoadingFID.nmrdata
Comment noe
SliceHistory
EXMODE bcm
POINT 32768 points
SAMPO 32768 points
SWEEP 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.40 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
AGAIN 19
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRRPW 55.0 usec
IRRNS 0
SCANS 74 times
SLVNT CDCL3
SPINNING 12 Hz
TEMP 21.9 C
14a
26.133
26.148
32.952
38.019
44.854
48.441
65.344
76.745
77.000
77.255
101.011
107.007
108.529
119.987
136.257
146.490
148.086
177.624
ppm
50
100
150
noe

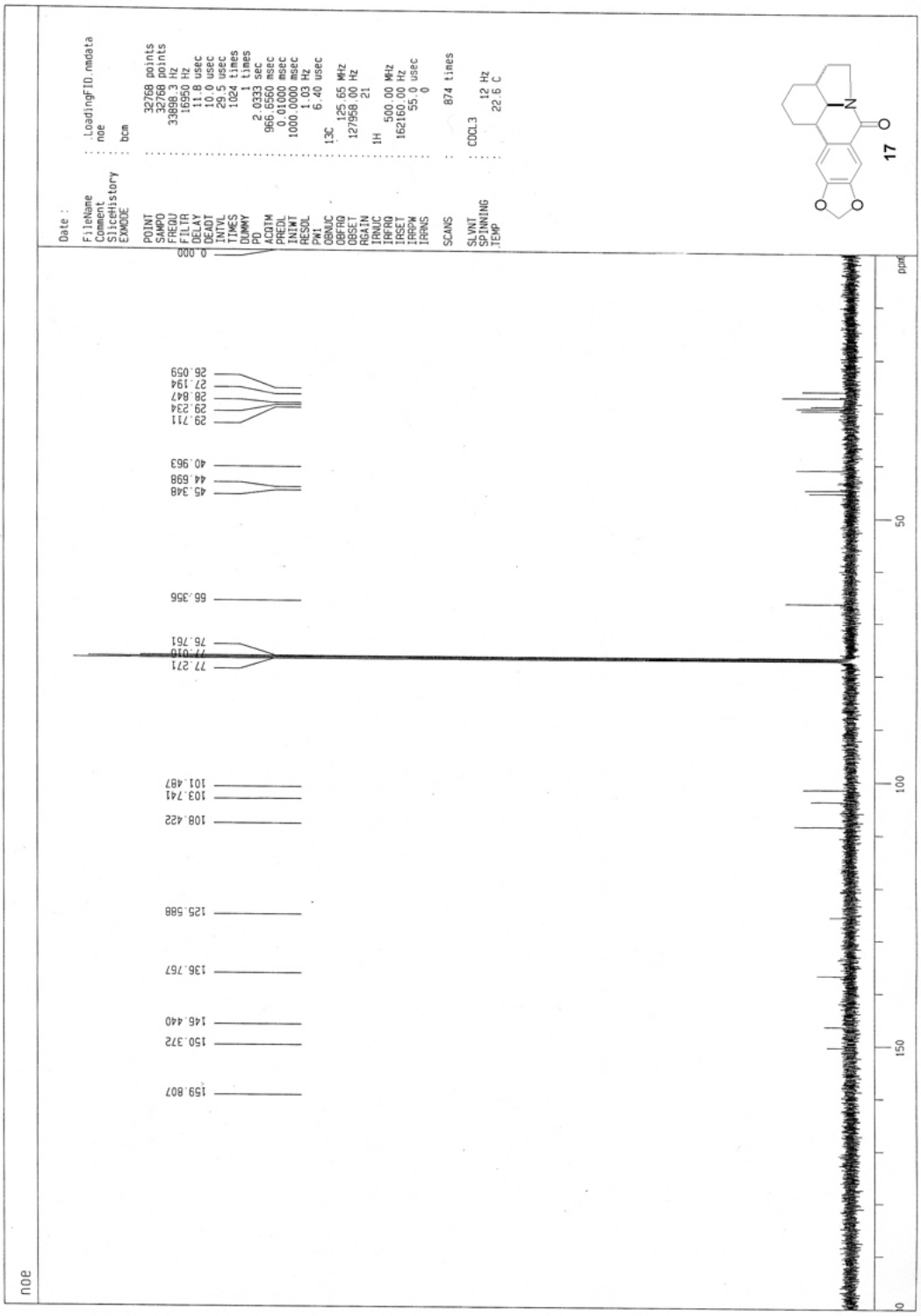

Date : Tue Jan 15 12:02:12 2002
FileName : LoadingFID.nmdata
Comment : noe
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 55.8 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 8.40 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
AGAIN : 18
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.6 C
15a
2.00
2.94
1.97
1.88
1.60
2.01
0.74
3.42
1.15
8
6
4
2
ppm

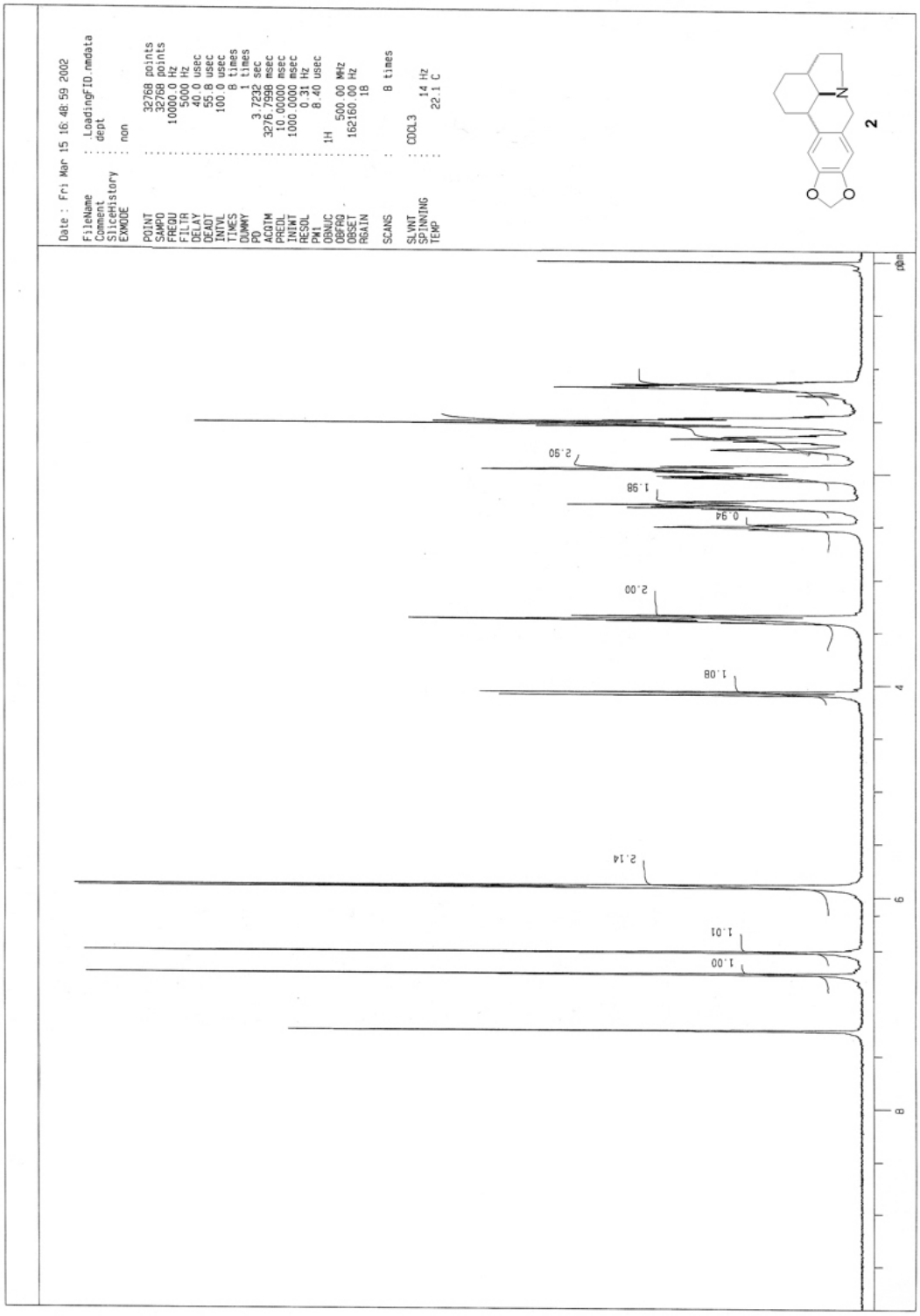

noe
Date :
FileName : LoadingFID.nmdata
Comment : noe
SliceHistory : bcm
EXMODE :
POINT 32768 points
SAMPD 32768 points
FREQU 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.40 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
AGAIN 18
IFNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRAPW 55.0 usec
IRNS 0
SCANS 106 times
SLVNT CDCl3
SPINNING 12 Hz
TEMP 22.5 C
15a
26.602
29.884
30.567
33.849
43.357
45.282
50.004
68.799
76.753
77.000
77.255
100.731
107.311
108.200
120.209
128.459
138.865
145.692
ppm
0
50
100
150

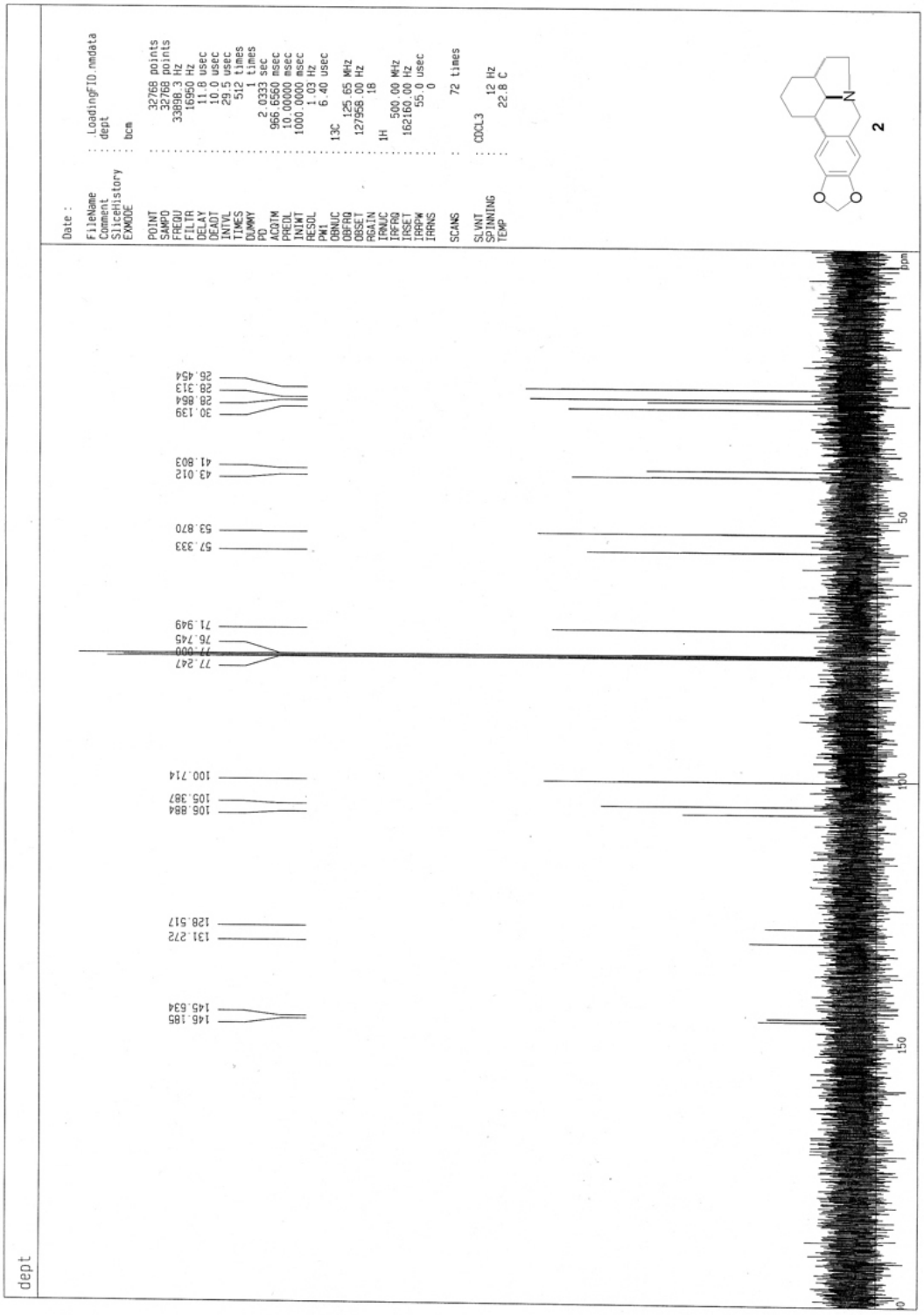

Date : Wed Feb 13 14:55:29 2002
FileName : LoadingFID.nmrdata
Comment : noe
SliceHistory : non
EXMODE :
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 55.8 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 8.40 usec
OBNUC : 1H 500.00 MHz
OBFRQ : 162508.36 Hz
OBSET : 17
AGAIN :
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 21.7 C
16
CO2Me
1.02
0.99
1.00
2.04
3.07
ppm

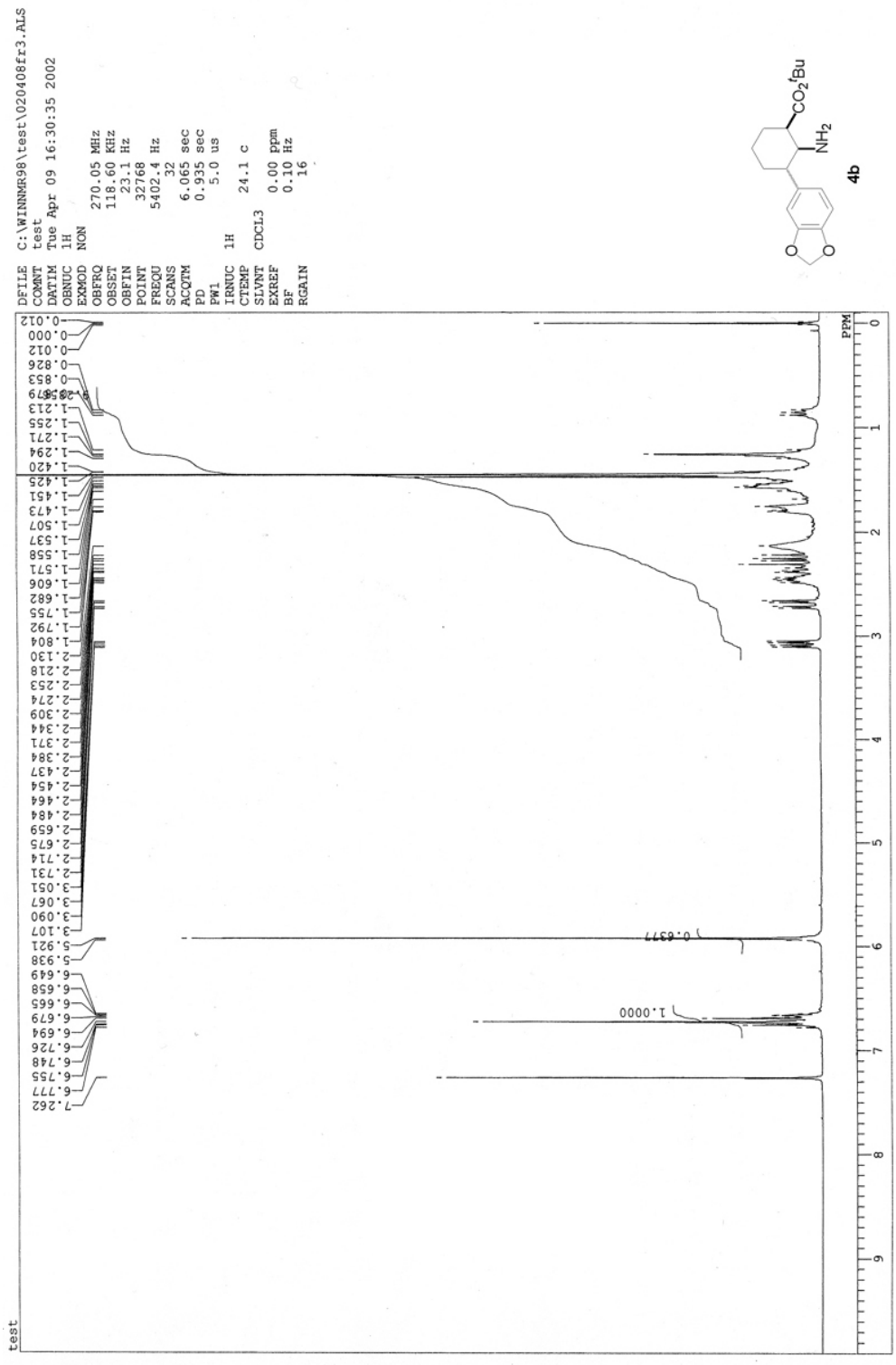

Date :
FileName : LoadingFID.rmdata
Comment : noe
SliceHistory : ...
EXMODE : bcm
POINT : 32768 points
SWPO : 32768 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 512 times
DUMMY : 1 times
PD : 2.0333 sec
ACQTM : 966.6560 msec
PREPUL : 0.0000 msec
TNIWT : 1000.0000 msec
RESOL : 1.03 Hz
PW1 : 6.40 usec
OBNUC : 13C 125.65 MHz
OBFRQ : 127958.00 Hz
OBSET : 17
RGAIN : 1H
TNNUC : 500.00 MHz
IRFRQ : 162160.00 Hz
TRSET : 55.0 usec
TRPW : 0
TRNS : ...
SCANS : 90 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 22.2 C
16
CO2Me
28.470
29.645
30.295
33.791
48.992
49.502
51.279
51.657
68.338
76.745
77.000
77.255
100.542
107.476
107.904
120.464
139.679
145.297
147.107
157.726
ppm

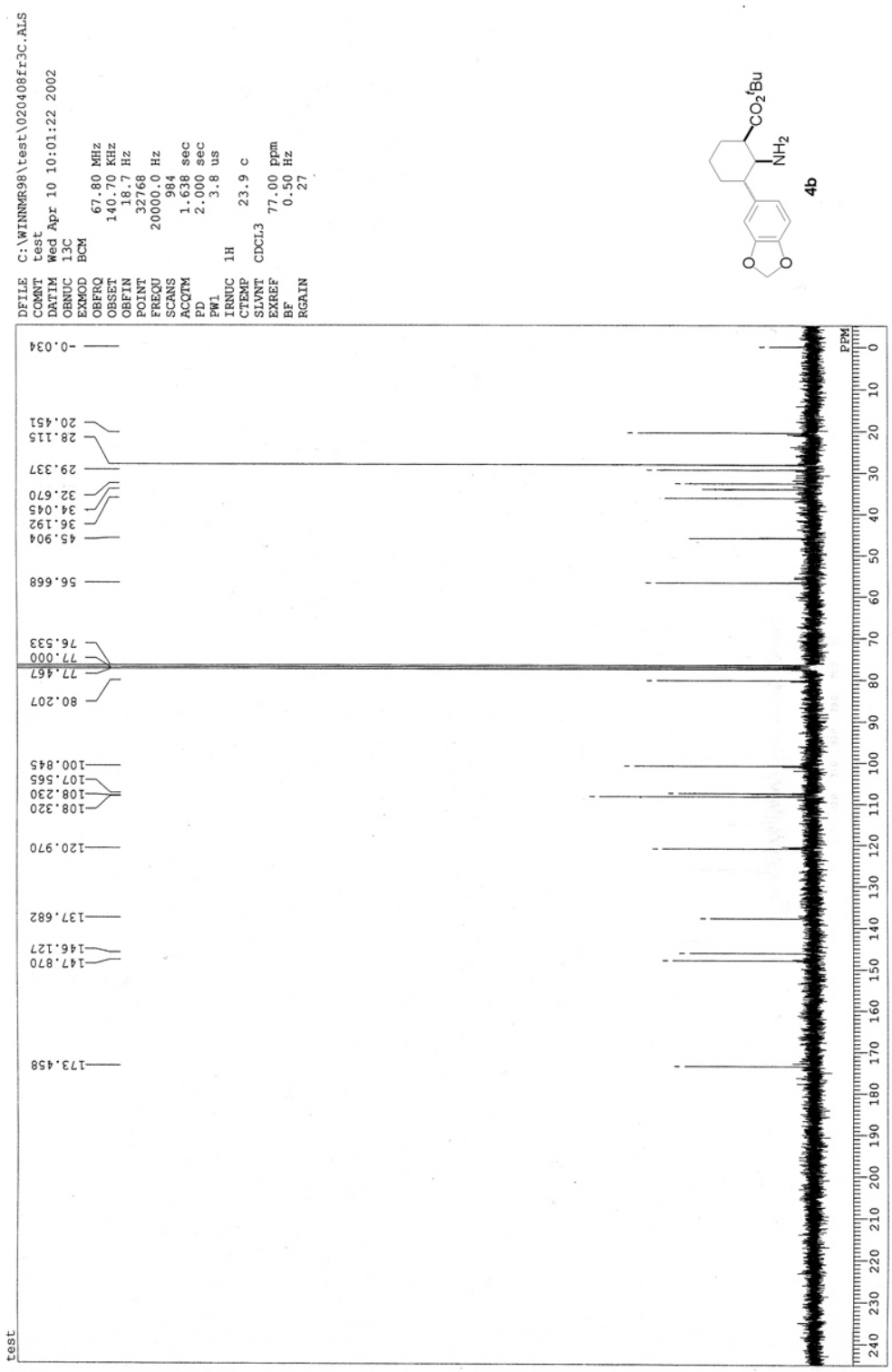

Date : Fri Feb 15 14:59:24 2002
FileName : LoadingFID.nmrdata
Comment : noe
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPD : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 55.8 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 8.40 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 21
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 21.4 C
17
3.87
21.27
3.40
1.18
2.18
1.05
1.00
2.24
1.10
1.11
ppm

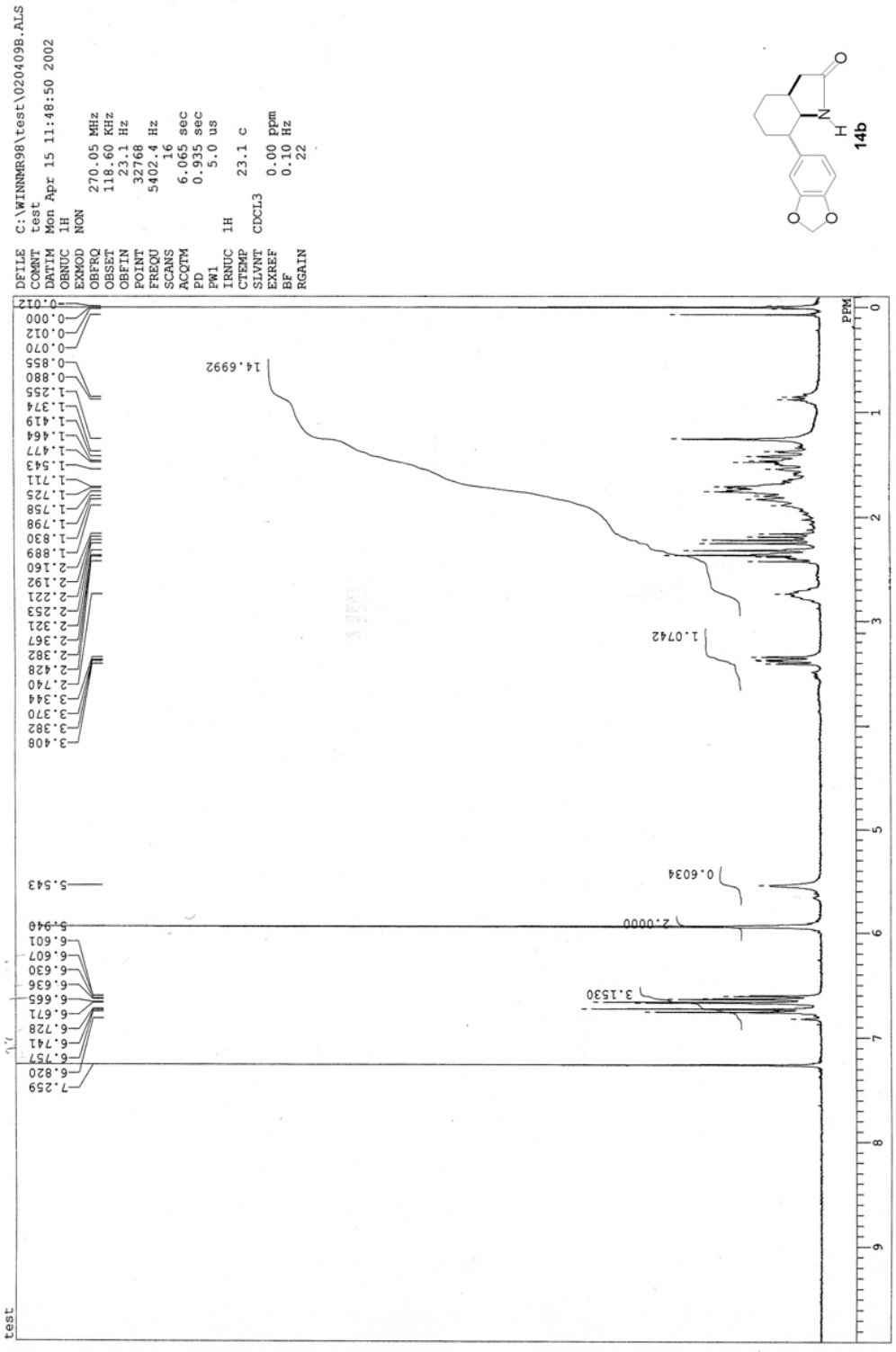

noe
Date :
FileName : LoadingFID nmdata
Comment : noe
SliceHistory :
EXMODE : bcm
POINT 32768 points
SAMPD 32768 points
FREQU 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 1024 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.40 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
AGAIN 21
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRPPW 55.0 usec
IRPNS 0
SCANS 874 times
SLVNT CDCL3
SPINNING 12 Hz
TEMP 22.6 C
17
26.059
27.194
28.847
29.234
29.711
40.963
44.698
45.348
66.356
76.761
77.010
77.271
101.487
103.741
108.422
125.588
136.767
146.440
150.372
159.807
ppm
0
50
100
150

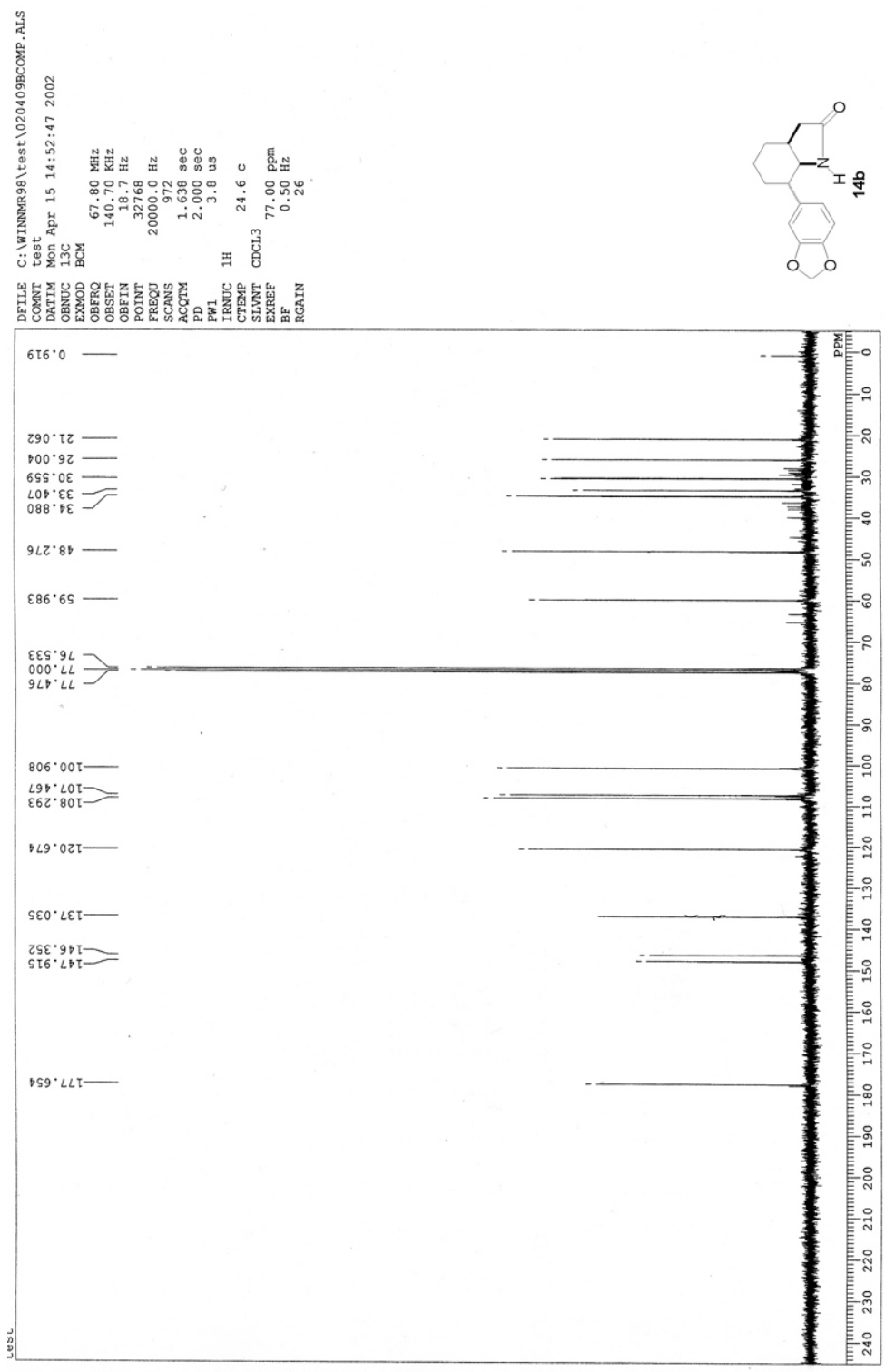

Date : Fri Mar 15 16:48:59 2002
FileName : LoadingFID.nmrdata
Comment : dept
SliceHistory :
EXMODE : non
POINT 32768 points
SAMPO 32768 points
FREQU 10000.0 Hz
FILTR 5000 Hz
DELAY 40.0 usec
DEADT 55.8 usec
INTVL 100.0 usec
TIMES 8 times
DUMMY 1 times
PD 3.7232 sec
ACQTM 3276.7998 msec
PREDL 10.00000 msec
AVRAT 1000.0000 msec
RESOL 0.31 Hz
PWT 8.40 usec
OBNUC 1H 500.00 MHz
OBFRQ 162160.00 Hz
OBSET 18
RGAIN
SCANS 8 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 22.1 C
2
2.14
1.01
1.00
2.00
1.08
2.50
1.98
0.94
ppm

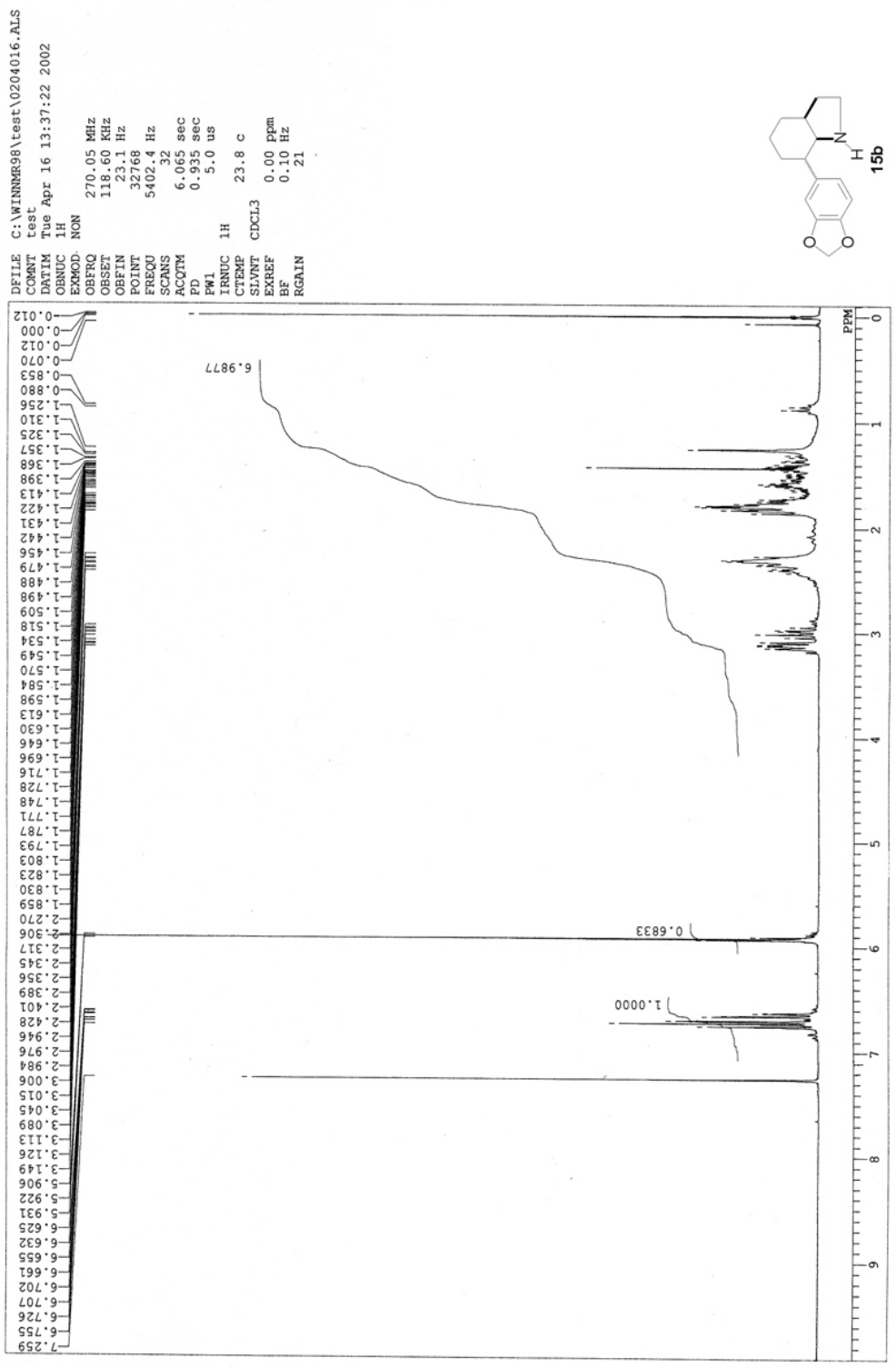

dept
Date :
FileName : LoadingFID.nmdata
Comment : dept
SliceHistory : : bcm
EXMODE :
POINT 32768 points
SAMPO 32768 points
FREQU 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 10.00000 msec
INIWT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.40 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
AGAIN 18
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRPW 55.0 usec
IRPWR 0
SCANS 72 times
SLVNT CDCL3
SPINNING 12 Hz
TEMP 22.8 C
2
O
O
N
26.454
28.313
28.864
30.139
41.803
43.012
53.870
57.333
71.949
76.745
77.000
77.247
100.714
105.387
105.884
128.517
131.272
145.634
146.185
ppm
0
50
100
150

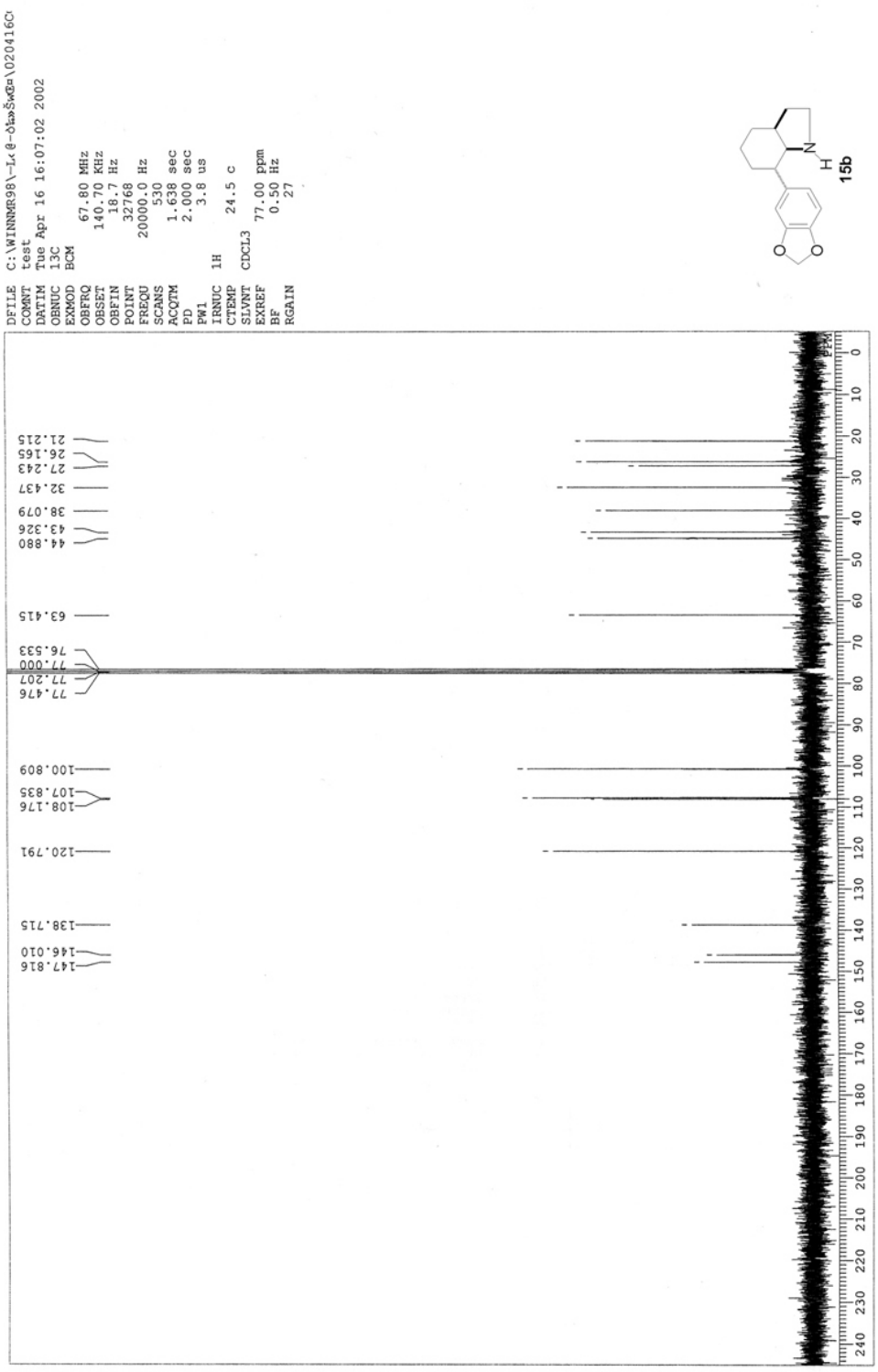

DFILE C:\WINNMR98\test\020408fr3.ALS
COMMT test
DATIM Tue Apr 09 16:30:35 2002
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 118.60 KHz
OBFIN 23.1 Hz
POINT 32768
FREQU 5402.4 Hz
SCANS 32
ACQTM 6.065 sec
PD 0.935 sec
PW1 5.0 us
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 16
test
PPM
1.0000
0.8377
4b

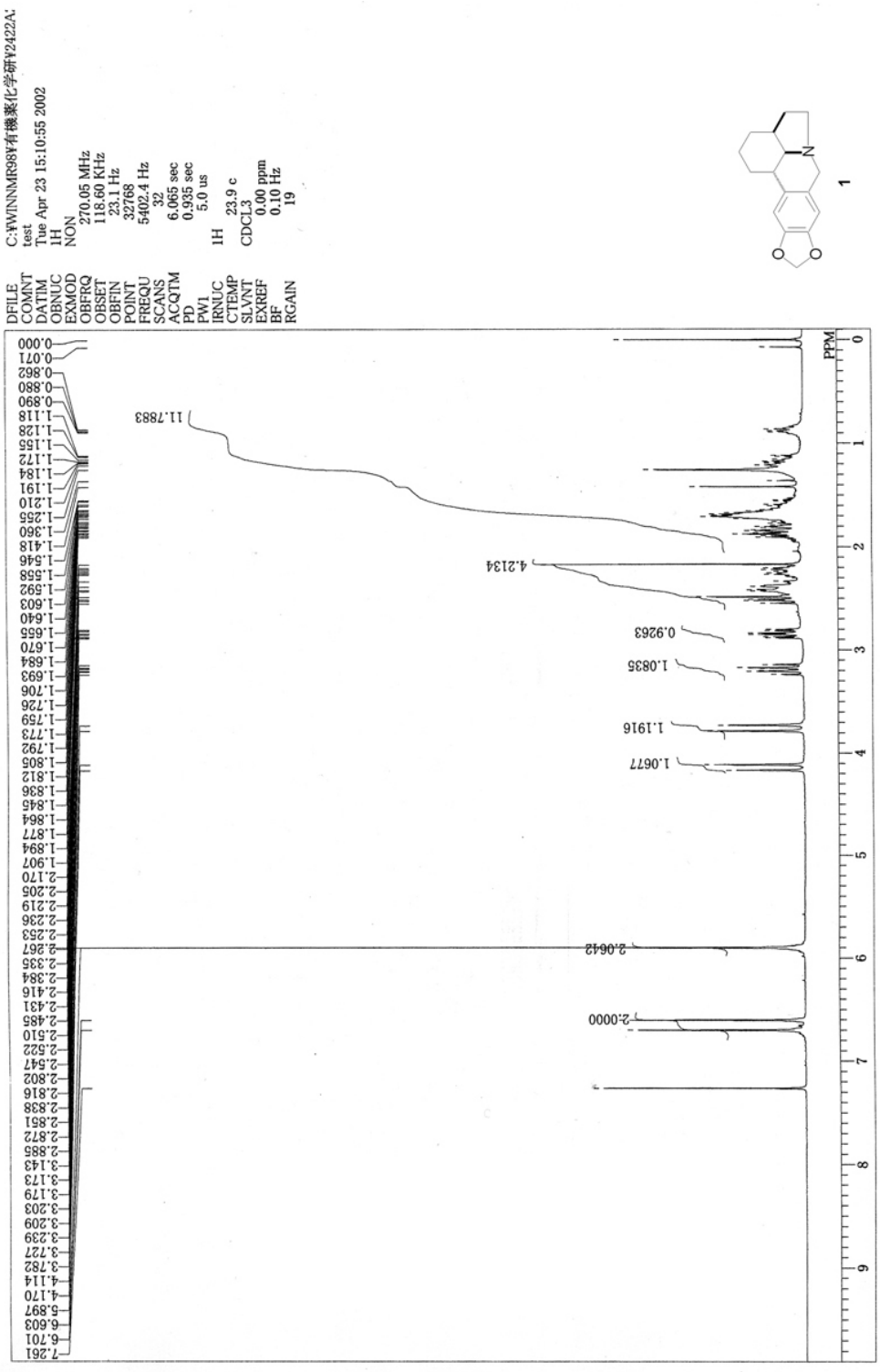

DFILE C:\WINNMR98\test\020408fr3C.ALS
COMNT test
DATIM Wed Apr 10 10:01:22 2002
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 140.70 KHz
OBFIN 18.7 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 984
ACQTM 1.638 sec
PD 2.000 sec
PW1 3.8 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 27
-0.034
20.451
28.115
29.337
32.670
34.045
36.192
45.904
56.668
76.533
77.000
77.467
80.207
100.845
107.565
108.230
108.320
120.970
137.682
146.127
147.870
173.458
PPM
test
4b

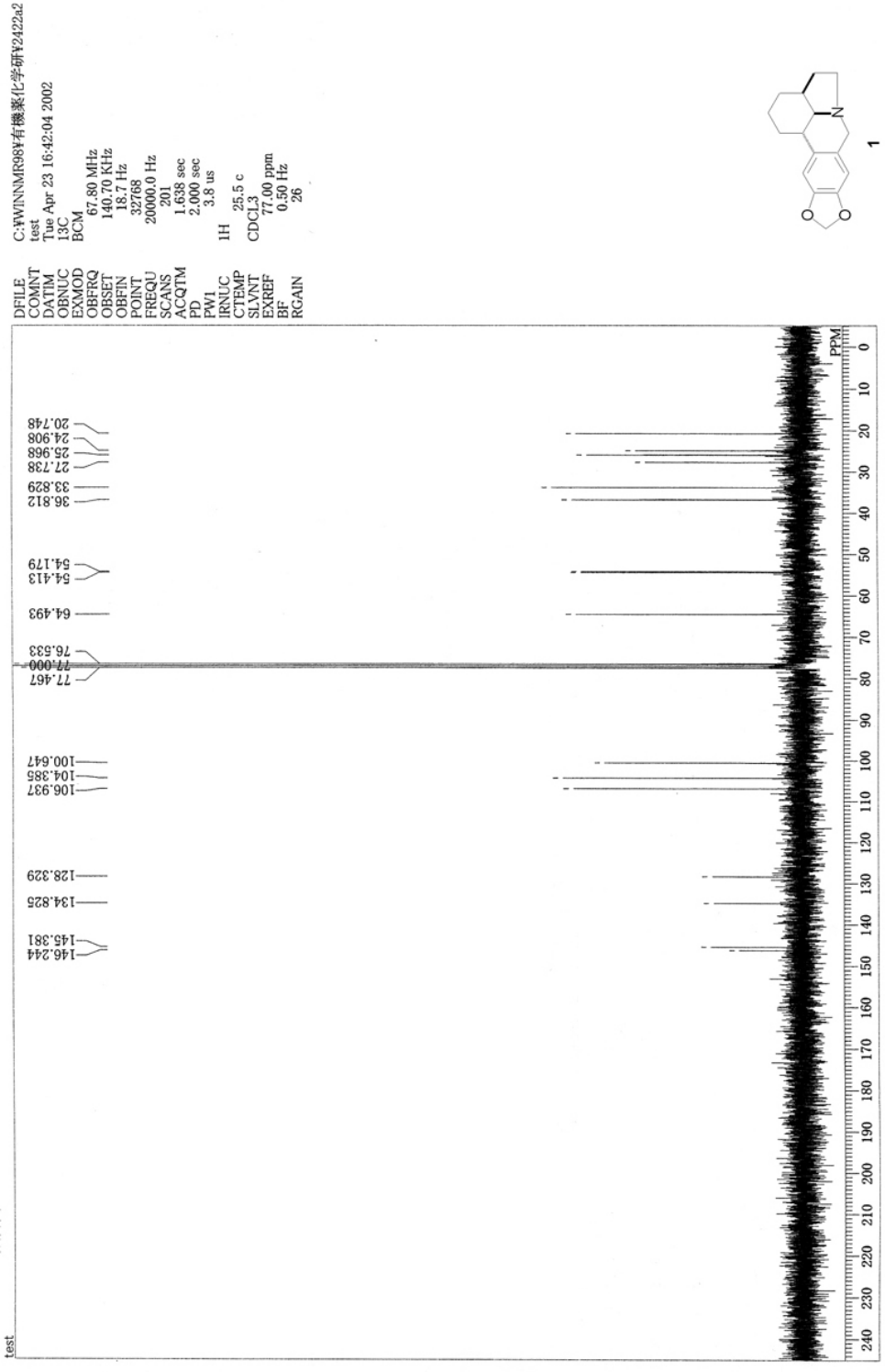

C:\WINNMR98\test\020409B.ALS
test
Mon Apr 15 11:48:50 2002
1H
NON
270.05 MHz
118.60 KHz
23.1 Hz
32768
5402.4 Hz
16
6.065 sec
0.935 sec
5.0 us
1H
23.1 c
CDCL3
0.00 ppm
0.10 Hz
22
14.6992
1.0742
0.6034
2.0000
3.1530
PPM
14b


DFILE C:\WINNMR98\test\020409BCOMP.ALS
COMNT test
DATIM Mon Apr 15 14:52:47 2002
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSFT 140.70 KHz
OBFIN 18.7 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 972
ACQTM 1.638 sec
PD 2.000 sec
PW1 3.8 us
IRNUC 1H
CTEMP 24.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 26
0.919
21.062
26.004
30.559
33.407
34.880
48.276
59.983
76.533
77.000
77.476
100.908
107.467
108.293
120.674
137.035
146.352
147.915
177.654
PPM
14b


DFILE C:\WINNMR98\test\0204016.ALS
COMNT test
DATIM Tue Apr 16 13:37:22 2002
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 118.60 KHz
OBFIN 23.1 Hz
POINT 32768
FREQU 5402.4 Hz
SCANS 32
ACQTM 6.065 sec
PD 0.935 sec
PW1 5.0 us
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 21
6.9877
0.6833
1.0000
PPM
15b


DFILE C:\WINNMR98\...\020416C
COMNT test
DATIM Tue Apr 16 16:07:02 2002
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 140.70 KHz
OBFIN 18.7 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 530
ACQTM 1.638 sec
PD 2.000 sec
PW1 3.8 us
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 27
21.215
26.165
27.243
32.437
38.079
43.326
44.880
63.415
76.533
77.000
77.207
77.476
100.809
107.835
108.176
120.791
138.715
146.010
147.816
15b


C:\FWINNMR98\有機薬化学研\2422A
test
Tue Apr 23 15:10:55 2002
1H
NON
270.05 MHz
118.60 KHz
23.1 Hz
32768
5402.4 Hz
32
6.065 sec
0.935 sec
5.0 us
1H
23.9 c
CDCL3
0.00 ppm
0.10 Hz
19
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
11.7883
4.2134
0.9263
1.0835
1.1916
1.0677
2.0642
2.0000
PPM
1


DFILE C:¥WINNMR98¥有機薬化学研¥2422a2
COMNT test
DATIM Tue Apr 23 16:42:04 2002
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 140.70 KHz
OBFIN 18.7 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 201
ACQTM 1.638 sec
PD 2.000 sec
PW1 3.8 us
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 26
test
20.748
24.908
25.968
27.738
33.829
36.812
54.179
54.413
64.493
76.533
77.000
77.467
100.647
104.385
106.937
128.329
134.825
145.381
146.244
PPM
1