Supporting Information

Asymmetric Organocatalysis of 4 + 3 Cycloaddition Reactions

Michael Harmata,* Sunil K. Ghosh, Xuechuan Hong, Sumrit Wacharasindhu, and Patrick Kirchhoefer

Department of Chemistry
University of Missouri-Columbia
Columbia, Missouri 65211

General Information:

All reactions were carried out under an atmosphere of nitrogen in oven-dried glassware. Dichloromethane was freshly distilled from CaH_2. Furans were distilled immediately prior to use. Trifluoroacetic acid (TFA), chloroform, n-butylamine, and (2*S*, 5*S*)-2-(1′, 1′-Dimethylethyl)-3-methyl-5-phenylmethyl-4-imidazolidinone (**4**) were purchased from Aldrich and used without further purification. Chromatographic separations were carried out using Silicycle ultra pure silica gel (230-400 mesh). Thin layer chromatography was performed on EM Reagent 0.25 nm silica gel 60-F plates. Visualization of the developed chromatogram was performed by UV light and vanillin stain solution followed by heating.

Melting points were measured with a Fisher-Johns melting point apparatus. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. Optical rotations were measured on a Jasco DIP-370 digital polarimeter. ^{1}H NMR were recorded on a Bruker ARX-250 (250 MHz), DRX-300 (300 MHz), DRX-500 (500 MHz) spectrometer and are reported in ppm (δ) from tetramethylsilane (TMS: δ 0.0 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet, ddd = doublet of doublet of doublet), coupling constants (Hz), and integration. ^{13}C NMR spectra were recorded on a Bruker ARX-250 (62.5 MHz), DRX-300 (75 MHz), and DRX-500 (125 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with solvent resonance as the internal standard ($CDCl_3$: δ 77.0 ppm). Analytical high performance liquid chromatography (HPLC) was performed on a Varian Pro Star model 500 using Chiralpak AD or Chiralcel OD-H column. Silyl enol ethers[1] and dialkyl furans[2] were prepared according to published methods.

General Experimental Procedure:

To a solution of (2*S*, 5*S*)-2-(1′, 1′-Dimethylethyl)-3-methyl-5-phenylmethyl-4-imidazolidinone (**4**) in CH_2Cl_2 (1 mL) was charged with the appropriate acid and then placed in a bath of desired temperature. The solution was stirred for 10 min before the addition of silyl enol ether (**1, 5-7**) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, the furan (**12-15**) (2-5 equiv) was added to it. The resulting solution was stirred at

constant temperature as mentioned in the table. The reaction mixture was then quenched with cold water and extracted with diethyl ether. The separated organic layer was dried over $MgSO_4$ and concentrated. The residue was purified by flash chromatography to afford the 4+3 cycloadducts. For the measurement of enantiomeric excess, the product was treated with 2-3 equiv of n-butylamine in $CHCl_3$ to give the corresponding pyrrole derivative.

Compound 8 (Table 1): To a solution of **4** (24.6 mg, 0.10 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.7 μL, 0.10 mmol). This solution was then cooled to 0 ^oC and stirred for 10 min before the addition of **1** (85 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, furan (363 μL, 5 mmol) was added. The resulting solution was stirred at this temperature for 96 h. The reaction mixture was quenched with cold water, extracted with ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (30 % EtOAc/hexanes) to afford **8** (*endo*) in 8 % yield as a colorless oil. IR (neat) 2973, 1725, 1708, 1335 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 9.82 (t, *J* = 1.0 Hz, 1H), 6.35 (dd, *J* = 1.7, 6.1 Hz, 1H), 6.22 (dd, *J* = 1.7, 6.1 Hz, 1H), 5.06 (ddd, *J* = 1.3, 2.6, 4.8 Hz, 1H), 4.95 (dd, *J* = 1.6, 4.6 Hz, 1H), 3.41 (ddd, *J* = 4.8, 6.0, 7.4 Hz, 1H), 2.86 (ddd, *J* = 1.3, 7.5, 18.8 Hz, 1H), 2.83 (dd, *J* = 5.1, 15.6 Hz, 1H), 2.36 (dd, *J* = 1.2, 15.6 Hz, 1H), 2.14 (ddd, *J* = 0.8, 6.0, 18.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.1, 199.2, 135.3, 131.2, 80.2, 78.1, 51.4, 45.6, 39.4.

Compound 9 (Table 1): To a solution of **8** (8 mg, 0.05 mmol) in $CHCl_3$ (2 mL) was added n-butylamine (15 μL, 0.14 mmol) and stirred for 6 h at room temperature. The solvent was removed and purified by a short silica gel column chromatography (10 % EtOAc/hexanes) to afford **9** (6 mg, 63%) as a colorless oil. Enantiomeric excess was determined by HPLC using a Chiralcel OD-H column [hexanes/isopropanol 99:1; flow rate 0.7 ml/min; t_r = 33.87 min and 42.70 min; 50% ee]; $[\alpha]^{25}_D$ 16.0 (c 0.30, $CHCl_3$); IR (neat) 2954, 2931, 1480 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 6.58 (dd, *J* = 1.6, 5.8 Hz, 1H), 6.41 (d, *J* = 2.6 Hz, 1H), 5.96 (d, *J* = 2.7 Hz, 1H), 5.87 (dd, *J* = 1.8, 5.8 Hz, 1H), 5.36 (d, *J* = 1.3 Hz, 1H), 5.14 (dd, *J* = 1.7, 6.0 Hz, 1H), 3.67 (t, *J* = 7.3 Hz, 1H), 3.12 (dd, *J* = 6.1, 15.7 Hz, 1H), 2.29 (d, *J* = 15.7 Hz, 1H), 1.72-1.60 (m, 2H), 1.39-1.24(m, 2H),

0.92 (t, J = 7.3 Hz, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 139.9, 125.1, 122.0, 120.1, 117.3, 102.5, 77.3, 77.0, 45.9, 33.1, 26.1, 19.9, 13.6.

Compound 16: (Table 2, entry 1): To a solution of **4** (24.6 mg, 0.10 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.7 μL, 0.10 mmol). This solution was then cooled to –78 ^oC and stirred for 10 min before the addition of **1** (85 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, **12** (265 μL, 2.5 mmol) was added. The resulting solution was stirred for 96 h. The reaction mixture was quenched by cold water, extracted by ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (20 % EtOAc/hexanes) to afford **16** (*endo)* in 64 % yield as a colorless oil; IR (neat) 2980, 1722, 1707 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$): δ 9.83 (t, J = 0.9 Hz, 1H), 6.02 (d, J = 5.8 Hz, 1H), 5.92 (d, J = 5.8 Hz, 1H), 3.17 (dd, J = 4.4, 8.6 Hz, 1H), 2.73 (ddd, J = 1.8, 8.6, 17.0 Hz, 1H), 2.60 (d, J = 15.4 Hz, 1H), 2.41 (d, J = 15.4 Hz, 1H), 2.22 (dd, J = 4.4, 16.9 Hz, 1H), 1.49 (d, J = 3.6 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.3, 199.8, 138.0, 134.3, 86.6, 84.3, 55.7, 50.7, 39.4, 23.2, 21.8. Anal. calcd for $C_{11}H_{14}O_3$: C, 68.02; H, 7.27. Found: C, 68.03; H, 6.98.

Pyrrole derivative of compound 16: To a solution of **16** (10 mg, 0.05 mmol) in $CHCl_3$ (2 mL) was added n-butylamine (11 μL, 0.10 mmol) and stirred for 6 h at room temperature. The solvent was removed and purified by a short silica gel column chromatography (8 % EtOAc/hexanes) to afford the product (8 mg, 67%) as a colorless oil. Enantiomeric excess was determined by HPLC using a Chiralpak AD column [hexanes/isopropanol 98:2; flow rate 0.5 ml/min; t_r = 10.24 min and 11.86 min; 89% ee]; $[\alpha]^{25}_D$ 49.1 (c 0.66, $CHCl_3$); IR (neat) 2962, 1480, 1442 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$): δ 6.40 (d, J = 2.7 Hz, 1H), 6.25 (d, J = 5.6 Hz, 1H), 5.98 (d, J = 2.7 Hz, 1H), 5.58 (d, J = 5.6 Hz, 1H), 3.66 (t, J = 7.3 Hz, 3H), 2.77 (d, J = 15.8 Hz, 1H), 2.37 (d, J = 15.8 Hz, 1H), 1.72-1.55 (m, 2H), 1.68 (s, 3H), 1.55 (s, 3H), 1.39-1.26 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 142.5, 129.1, 124.2, 123.8, 117.1, 101.4, 83.7, 83.2, 46.1, 33.0, 32.8, 24.8, 20.0 19.9, 13.6.

Compound 17 (Table 2, entry 6): To a solution of **4** (24.6 mg, 0.10 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.7 μL, 0.10 mmol). This solution was then cooled to –60 ^oC, stirred for 10 min, followed by the addition of **1** (85 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, **13** (310 mg, 2.5 mmol) was added. The resulting

solution was stirred at this temperature for 22 h. The reaction mixture was quenched by cold water, extracted by ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (15 % EtOAc/hexanes) to afford **17** (*endo)* in 55 % yield as a colorless oil; IR (neat) 2970, 1711, 1708 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 9.82-9.80 (m, 1H), 6.00 (d, *J* = 5.9 Hz, 1H), 5.86 (d, *J* = 5.9 Hz, 1H), 3.18 (dd, *J* = 4.2, 8.8 Hz, 1H), 2.67 (ddd, *J* = 2.0, 8.8, 16.8 Hz, 1H), 2.56 (d, *J* = 15.3 Hz, 1H), 2.37 (d, *J* = 15.3 Hz, 1H), 2.20 (ddd, *J* = 0.5, 4.1, 16.8 Hz, 1H), 1.83-1.68 (m, 4H), 0.96 (t, *J* = 7.4 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 206.9, 199.9, 136.9, 133.6, 89.3, 87.3, 54.8, 49.5, 39.1, 29.2, 27.0, 8.0, 7.7. Anal. calcd for $C_{13}H_{18}O_3$: C, 70.24; H, 8.16. Found: C, 69.90; H, 7.95.

Pyrrole derivative of compound 17: To a solution of **17** (Table 3, entry 3) (30 mg, 0.14 mmol) in $CHCl_3$ (2 mL) was added butylamine (11 μL, 0.10 mmol) and stirred for 6 h at room temperature. The solvent was removed and the residue purified by a short silica gel column chromatography (8 % EtOAc/hexanes) to afford the product (25 mg, 71%) as a colorless oil. Enantiomeric excess was determined by HPLC using a Chiralpak AD column [hexanes/isopropanol 98:2; flow rate 0.5 ml/min; t_r = 9.04 min and 11.85 min; 81.3 % ee]; $[\alpha]^{25}_D$ 35.8 (c 1.48, $CHCl_3$). IR (neat) 2960, 1482, 1454 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 6.41 (d, *J* = 2.5 Hz, 1H), 6.24 (d, *J* = 5.6 Hz, 1H), 5.97 (d, *J* = 2.6 Hz, 1H), 5.61 (d, *J* = 5.7 Hz, 1H), 3.67 (t, *J* = 7.4 Hz, 2H), 2.75 (d, *J* = 15.7 Hz, 1H), 2.35 (d, *J* = 15.7 Hz, 1H), 2.15-2.02 (m, 2H), 1.87 (q, *J* = 7.5 Hz, 2H), 1.70-1.64 (m, 2H), 1.37-1.26 (m, 2H), 1.09-1.01 (m, 6H), 0.92 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 141.5, 127.5, 124.7, 123.5, 117.0, 101.4, 86.8, 86.3, 46.0, 33.0, 31.5, 30.9, 26.0, 19.9, 13.6, 8.2, 8.1.

Compound 18 (Table 2, entry 9): To a solution of **4** (22.1 mg, 0.09 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.0 μL, 0.09 mmol). This solution was then cooled to –78 ^oC and stirred for 10 min before the addition of **5** (96 mg, 0.45 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, **14** (342mg, 2.25 mmol) was added. The resulting solution was stirred at this temperature for 95 h. The reaction mixture was quenched with cold water, extracted with ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (20 % EtOAc/hexanes) to afford **18** (*endo)* in 74 % yield as a colorless oil. IR (neat) 2962, 2864, 2733, 1719 cm^{-1}; ^1H NMR (300

MHz, $CDCl_3$): δ 9.82-9.80 (m, 1H), 5.99 (d, *J* = 6.2 Hz, 1H), 5.86 (d, *J* = 5.7 Hz, 1H), 3.17 (dd, *J* = 4.2, 8.9 Hz, 1H), 2.67 (ddd, *J* = 2.5, 10.0,17.5 Hz, 1H), 2.56 (d, *J* = 12.4 Hz, 1H), 2.37 (d, *J* = 15.3 Hz, 1H), 2.21 (dd, *J* = 4.1, 17.5 Hz, 1H), 1.82-1.61 (m, 4H), 1.61-1.41 (m, 2H), 1.41-1.21 (m, 2H), 1.03-0.88, (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.8, 199.9, 136.9, 133.6, 88.9, 86.9, 55.1, 49.8, 39.1, 38.6, 36.5, 17.1, 16.9, 14.3, 14.2; HRMS calcd for $C_{15}H_{22}O_3Na$ $[M + Na]^+$ 273.14611, found 273.14780.

Pyrrole derivative of compound 18: To a solution of **18** (30 mg, 0.119 mmol) in $CHCl_3$ (6 mL) was added n-butylamine (23 μL, 0.239 mmol) and stirred for 10 h at room temperature. The solvent was removed and purified by a short silica gel column chromatography (10 % EtOAc/hexanes) to afford the product as a colorless oil (34 mg, 99%). Enantiomeric excess was determined by HPLC using a Chiralcel AD column [hexane/isopropanol 98:2; flow rate 0.5 ml/min; t_r = 9.60 and 11.18 min; 85%ee]; IR: 2990, 2962, 1486, 1466, 1271 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$): δ 6.41 (d, *J*= 2.5 Hz, 1H), 6.23 (d, *J* = 5.7 Hz, 1H), 5.97 (d, *J* = 2.7 Hz, 1H), 5.60 (d, *J* = 5.5 Hz, 1H), 3.67 (t, *J* = 7.3 Hz, 2H), 2.75 (d, *J* = 15.7 Hz, 1H), 2.34 (d, *J* = 15.7), 2.07-1.28 (m, 12H), 1.07-0.90 (m, 9H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 141.6, 127.5, 124.5, 123.7, 117.0, 101.3, 86.3, 85.9, 46.0, 40.6, 35.5, 33.0, 31.8, 19.9, 17.3, 14.6, 13.6.

Compound 19 (Table 2, entry 11): To a solution **4** (24.6 mg, 0.10 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.7 μL, 0.10 mmol). This solution was cooled to -35 ^{o}C and stirred for 10 min before the addition of **1** (85 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, **15** (270 mg, 1 mmol) was added. The resulting solution was stirred at this temperature for 12 h. The reaction mixture was quenched with cold water, extracted with ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (15 % EtOAc/hexanes) to afford **19** as *endo:exo* isomers (3.7:1) in 56 % yield as a white crystalline solid. *Endo* isomer: R_f = 0.43 (25% EtOAc/hexanes); mp 222-224 ^{o}C; IR (film) 2835, 1719, 1709 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$): δ 9.81 (s, 1H), 7.64-7.18 (m, 13H), 7.00-6.97 (m, 1H), 4.26 (dd, *J* = 2.2, 9.9 Hz, 1H), 3.28 (d, *J* = 14.8 Hz, 1H), 3.14 (d, *J* = 14.8 Hz, 1H), 2.63 (dd, *J* = 9.9, 17.5 Hz, 1H), 2.37 (dd, *J* = 2.2, 17.5 Hz, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 205.6, 198.9, 146.7, 141.3, 139.9, 137.2, 129.1, 128.9, 128.5, 128.3, 128.0, 127.9, 125.8, 122.8, 121.7, 88.6, 85.9, 52.5, 50.7, 40.0; Anal. calcd for $C_{25}H_{20}O_3$: C, 81.50; H 5.47. Found: C, 81.71; H,

5.51; *Exo* isomer: R_f = 0.21 (25% EtOAc/hexanes); mp 172-173 ^oC; IR (film) 2835, 1719, 1709 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 9.57 (t, *J* = 1.9 Hz, 1H), 7.64-7.18 (m, 14H), 3.70 (t, *J* = 6.5 Hz, 1H), 3.28 (d, *J* = 15.4 Hz, 1H), 3.11 (d, *J* = 15.4 Hz, 1H), 2.68 (d, *J* = 1.3 Hz, 1H), 2.65 (d, *J* = 1.9 Hz, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 206.8, 198.8, 145.0, 143.6, 140.2, 138.6, 129.0, 128.8, 128.6, 128.5, 128.2, 125.9, 125.0, 121.3, 121.2, 87.3, 85.8, 53.8, 49.2, 42.5. Anal. calcd for $C_{25}H_{20}O_3$: C, 81.50; H 5.47. Found: C, 81.56; H, 5.68.

Pyrrole derivative of compound 19: To a solution of **19** (30 mg, 0.02 mmol) in $CHCl_3$ (2 mL) was added n-butylamine (10 μL, 0.09 mmol) and stirred for 6 h at room temperature. The solvent was removed and purified by a short silica gel column chromatography (8 % EtOAc/hexanes) to afford the product as a colorless solid (25 mg, 76%). *Endo*: Enantiomeric excess was determined by HPLC using a Chiralcel OD-H column [hexanes/isopropanol 98:2; flow rate 1 ml/min; t_r = 8.25 min and 12.42 min; 12 % ee]; *Exo*: Enantiomeric excess was determined by HPLC using a Chiralcel OD-H column [hexanes/isopropanol 98:2; flow rate 1 ml/min; t_r = 9.46 min and 13.70 min; 68 % ee]; IR (film) 2953, 2925, 1486 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 8.00-7.95 (m, 2H), 7.75-7.70 (m, 2H), 7.53-7.33 (m, 6H), 7.11-6.94 (m, 4H), 6.45 (d, *J* = 2.8 Hz, 1H), 6.08 (d, *J* = 2.8 Hz, 1H), 3.72-3.64 (m, 2H), 3.48 (d, *J* = 15.1 Hz, 1H), 3.15 (d, *J* = 15.0 Hz, 1H), 1.70-1.58 (m, 2H), 1.33-1.21 (m, 2H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 150.8, 144.9, 143.3, 139.0, 128.2, 128.0, 127.5, 127.1, 126.1, 125.4, 124.0, 123.1, 121.5, 118.8, 118.5, 103.0, 86.4, 84.6, 46.1, 33.7, 33.0, 19.9, 13.6.

Compound 21 (Eqn. 2): To a solution of **4** (24.6 mg, 0.10 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.7 μL, 0.10 mmol). This solution was cooled to –30 ^oC and stirred for 10 min before the addition of **20** (92 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, **12** (265 μL, 2.5 mmol) was added. The resulting solution was stirred at this temperature for 96 h. The reaction mixture was quenched with cold water, extracted with ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (20 % EtOAc/hexanes) to afford **21** (*endo*) (67 mg, 64%) as a colorless oil; IR(neat) 2974, 2921, 1723, 1705 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 9.84 (dd, *J* = 1.2, 3.1 Hz, 1H), 6.04 (d, *J* = 5.9 Hz, 1H), 5.95 (d, *J* = 5.9 Hz, 1H), 3.23 (dd, *J* = 4.2, 9.1 Hz, 1H), 2.70 (ddd, *J* = 1.9, 9.1, 16.8 Hz, 1H), 2.61 (q, *J* = 7.0

Hz, 1H), 2.21 (ddd, *J* = 0.8, 4.1, 16.7 Hz, 1H), 1.51 (s, 3H), 1.47 (s, 3H), 1.00 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.7, 200.0, 136.7, 135.6, 87.8, 86.7, 55.8, 54.6, 39.7, 21.9, 21.8, 10.1; HRMS calcd for $C_{12}H_{16}O_3Na$ $[M + Na]^+$ 231.0992, found 231.0992.

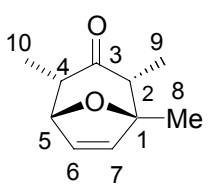
Pyrrole derivative of compound 21: To a solution of **21** (21 mg, 0.1 mmol) in $CHCl_3$ (3 mL) was added n-butylamine (14.6 μL, 0.2 mmol) and refluxed for 24 hrs. The solvent was removed and the residue purified by a short silica gel column chromatography (8 % EtOAc/hexanes) to afford the product (9 mg, 37%) as a colorless oil. Enantiomeric excess was determined by HPLC using a Chiralpak AD column [hexanes/isopropanol 98:2; flow rate 0.5 ml/min; t_r = 9.96 min and 10.59 min; 9 % ee]; $[\alpha]^{25}_D$ 8.0 (c 0.85, $CHCl_3$); IR(neat) 2966, 2929, 2868, 1589, 1454 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.38-6.29 (m, 2H), 5.95 (d, *J* = 2.8 Hz, 1H), 5.65 (d, *J* = 5.7 Hz, 1H), 3.81-3.69 (m, 2H), 2.97 (q, *J* = 7.1 Hz, 1H), 1.78-1.66 (m, 2H), 1.63 (s, 3H), 1.57 (s, 3H), 1.42-1.25 (m, 2H), 1.16 (d, *J* = 7.1 Hz, 3H) 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.7, 129.5, 128.7, 124.1, 117.7, 101.4, 87.1, 83.4, 47.1, 38.8, 33.1, 22.6, 20.2, 20.1, 13.7, 13.1.

Compound 22 (Eqn. 2): To a solution of **4** (24.6 mg, 0.10 mmol) in CH_2Cl_2 (1 mL) was added TFA (7.7 μL, 0.10 mmol). This solution was cooled to –30 ^{o}C and stirred for 10 min before the addition of **20** (92 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After stirring for an additional 10 min, furan (181 μL, 2.5 mmol) was added. The resulting solution was stirred at this temperature for 96 h. The reaction mixture was quenched with cold water, extracted with ether (3 X 5 mL), dried over $MgSO_4$, and concentrated. The residue was purified by flash chromatography (20 % EtOAc/hexanes) to afford **22** (*endo*) (63 mg, 64%) as a colorless oil. IR (neat) 2970, 1728, 1703 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.84 (s, 1H), 6.36 (dd, *J* = 1.5, 4.7 Hz, 1H), 6.26 (dd, *J* = 1.5, 6.1 Hz, 1H), 4.93 (dd, *J* = 1.5, 4.5 Hz, 1H), 4.88 (dd, *J* = 1.5, 4.5 Hz, 1H), 3.42 (m, 1H), 2.89 (m, 1H), 2.82 (dd, *J* = 7.9, 17.5 Hz, 1H), 2.12 (dd, *J* = 5.71, 7.5 Hz, 1H), 0.97 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.9, 199.5, 134.2, 132.8, 82.7, 81.2, 50.6, 50.4, 39.5, 10.0; HRMS calcd for $C_{10}H_{12}O_3Na$ $[M + Na]^+$ 203.0679, found 203.0680.

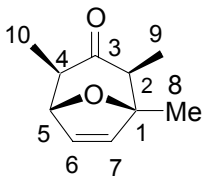
Pyrrole derivative of compound 22: To a solution of **22** (28 mg, 0.15 mmol) in $CHCl_3$ (3 mL) was added n-butylamine (56 μL, 0.77 mmol) and refluxed for 24 hrs. The solvent was removed and purified by a short silica gel column chromatography (8 %

EtOAc/hexanes) to afford the product as a colorless oil (14 mg, 43%). Enantiomeric excess was determined by HPLC using a Chiralpak AD column [hexanes/isopropanol 98:2; flow rate 0.5 ml/min; t_r = 17.73 min and 24.33 min; 7% ee]; IR (neat) 2970, 2917, 1589, 1470 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 6.72 (dd, *J* = 1.5, 5.8 Hz, 1H), 6.35 (d, *J* = 2.3 Hz, 1H), 5.94 (d, *J* = 2.7 Hz, 1H), 5.88 (dd, *J* = 1.7, 5.9 Hz, 1H), 5.25 (d, *J* = 1.7 Hz, 1H), 4.96 (dd, *J* = 1.5, 5.7 Hz, 1H), 1.70 (m, 2H), 1.32 (m, 2H), 1.14 (d, *J* = 7.1 Hz, 3H) 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 142.0, 125.3, 120.3, 117.9, 112.7, 102.6, 82.0, 79.0, 47.2, 33.1, 32.6, 20.1, 13.7, 13.2.

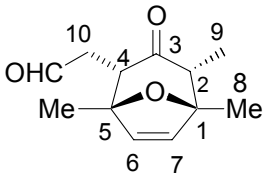
Relative Stereochemistry of 21:



endo **24**



exo **24**



21

The determination of the relative stereochemistry of **21** was made by NOESY experiments.[3] From these experiments, we observed the following cross peaks: CH_2-10 and H-6 (intense), CH_3-9 and H-7 (intense). We can therefore assign to **21**, the structure shown. Further support for this stereochemical assignment comes from a comparison of the chemical shifts of H-2, H-8, H-9 in **21** with the endo and exo isomers of **24** (Table 1).[4] Further support was from a comparison of ^{13}C chemical shift of C-4, C-8 and C-9 in **21** with the endo and exo isomers of **24** (Table 2).[4]

Table 1. ^1H Chemical shifts, δ (ppm), in $CDCl_3$ of **21** and diastereoisomeric pairs of **24**

Compound	H-2	H-4	H-6	H-7	H-8	H-9
endo **24**	2.57	2.77	6.25	6.12	1.51	0.96
exo **24**	2.26	2.26	6.19	6.04	1.39	1.26
21	2.61	3.23	6.04	5.95	1.51	1.0

Table 2. ^{13}C Chemical shifts, δ (ppm), in $CDCl_3$ of **21** and diastereoisomeric pairs of **24**

Compound	C-2	C-4	C-6	C-7	C-8	C-9
endo **24**	49.27	55.36	136.31	132.89	21.33	9.75
exo **24**	48.78	53.35	137.75	133.29	19.68	14.54
21	54.58	55.8	136.68	135.59	21.9	10.1

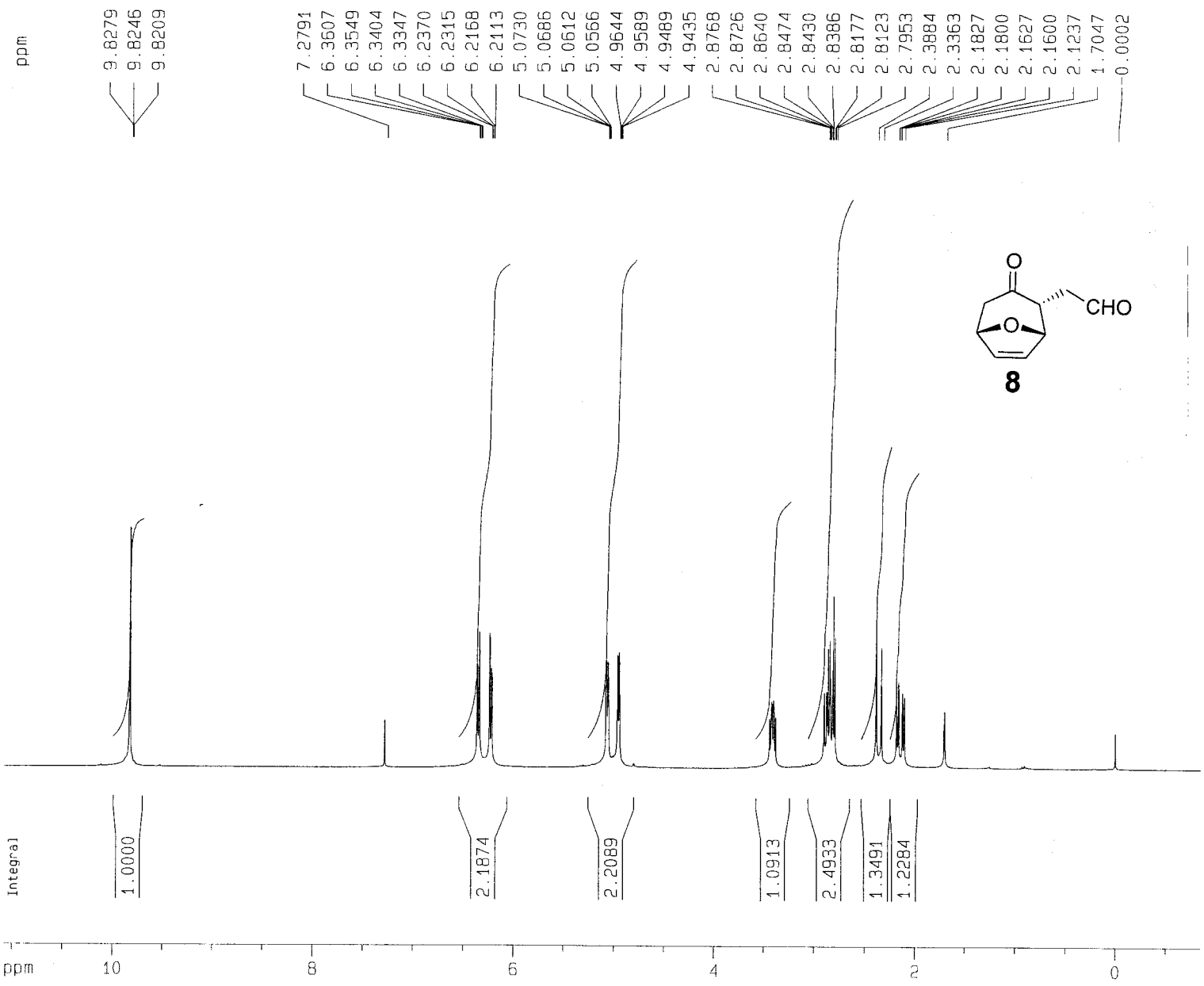
[1] Ohno, M.; Mori, K.; Hattori, T.; Eguchi, S. *J. Org. Chem*., **1990**, 55, 6086.

[2] McKeown, N. B.; Chambrier, I.; Cook, M. J. *J. Chem. Soc., Perkin Trans. 1* **1990**, 1169.

[3] Montana, A. M.; Grima G. M.; Garcia, F. *Magn. Reson. Chem.* **1999**, 37, 507-511.

[4] Montana, A. M.; Ribes S.; Grima G. M.; Garcia, F. *Magn. Reson. Chem.* **1998**, 36, 174-180.

sg-4-120-furan adduct-1H NMR



Current Data Parameters

NAME	sg-4-120
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

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Time	20.41
INSTRUM	drx300
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	228.1
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	7.50 usec
PL1	0.00 dB
SF01	300.1318534 MHz

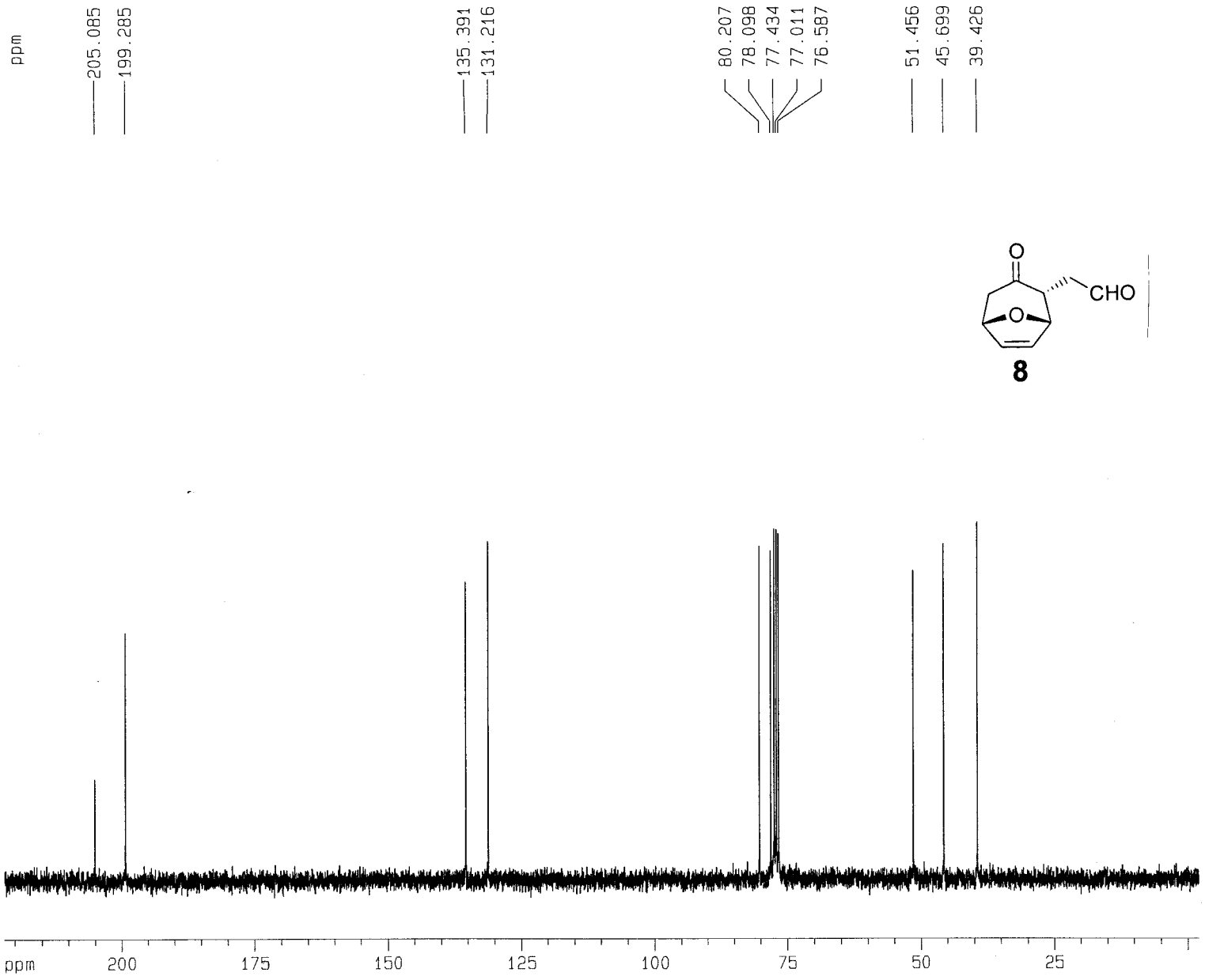
F2 - Processing parameters

SI	32768
SF	300.1300001 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.30

1D NMR plot parameters

CX	20.00 cm
CY	4.00 cm
F1P	11.080 ppm
F1	3325.35 Hz
F2P	-0.845 ppm
F2	-253.58 Hz
PPMCM	0.59623 ppm/cm
HZCM	178.94669 Hz/cm

sg-4-120-furan adduct-13C NMR



Current Data Parameters

NAME	sg-4-120
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020702
Time	20.52
INSTRUM	drx300
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	306
DS	4
SWH	18832.578 Hz
FIDRES	0.287362 Hz
AQ	1.7400136 sec
RG	32768
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec

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

NUC1	13C
P1	7.75 usec
PL1	6.00 dB
SF01	75.4760107 MHz

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

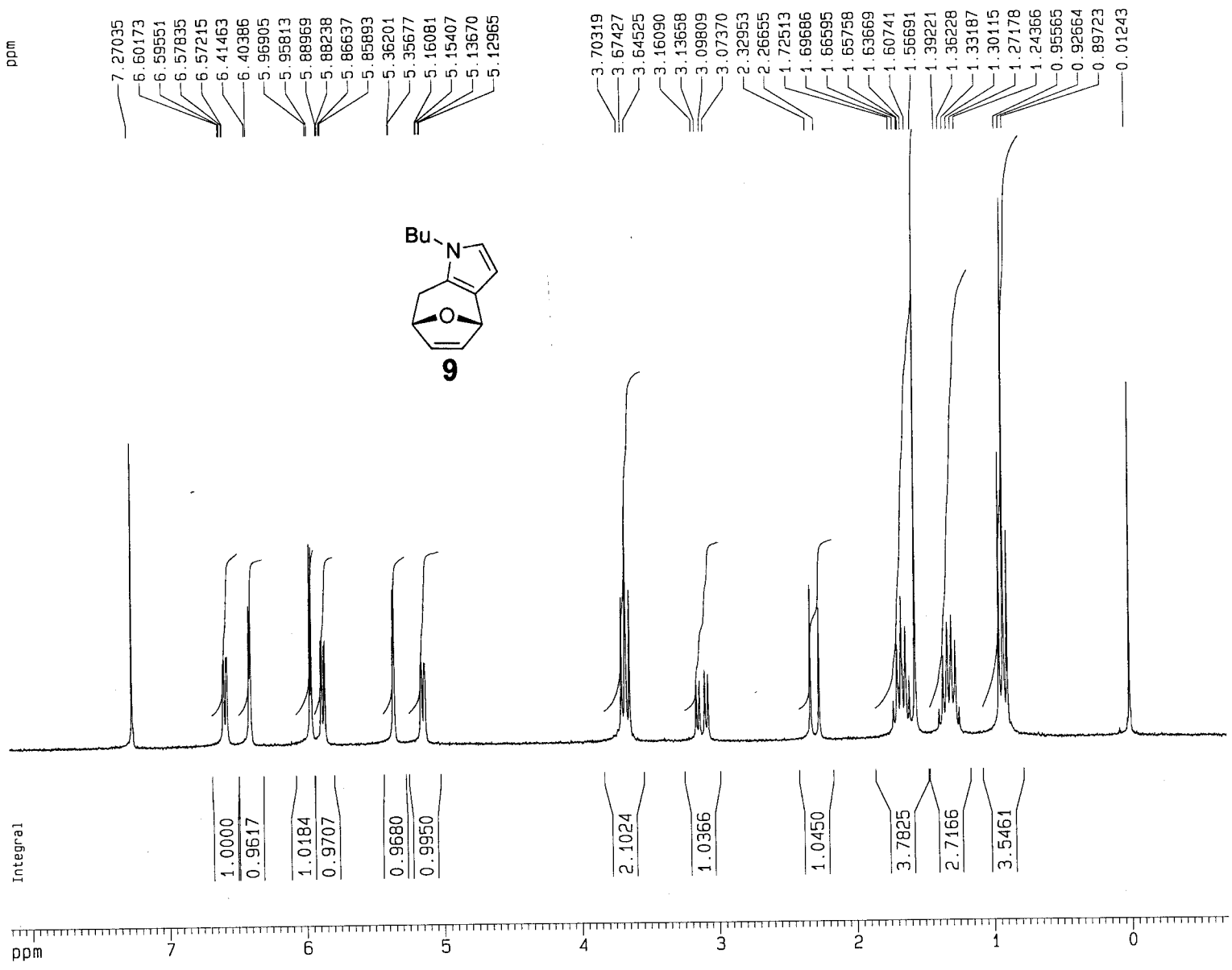
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	24.50 dB
SF02	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677519 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	222.039 ppm
F1	16756.80 Hz
F2P	-2.064 ppm
F2	-155.79 Hz
PPMCM	11.20518 ppm/cm
HZCM	845.62946 Hz/cm



Current Data Parameters

NAME	sg-4-furan
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

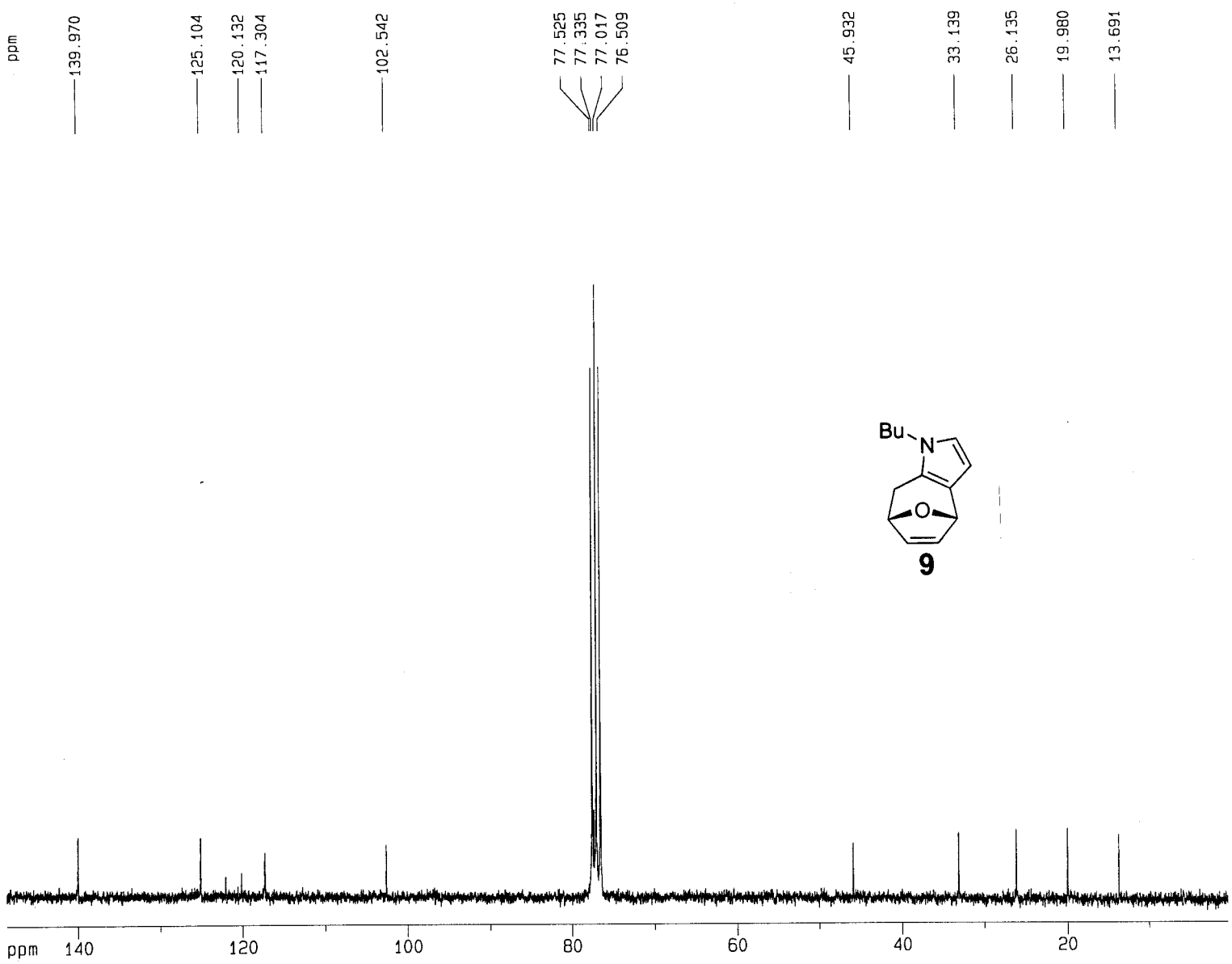
Date_	20020713
Time	13.50
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	4096
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300049 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	9.00 cm
F1P	8.163 ppm
F1	2041.93 Hz
F2P	-0.694 ppm
F2	-173.63 Hz
PPMCM	0.44288 ppm
HZCM	110.77803 Hz/



Current Data Parameters

NAME	sg-4-furan
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

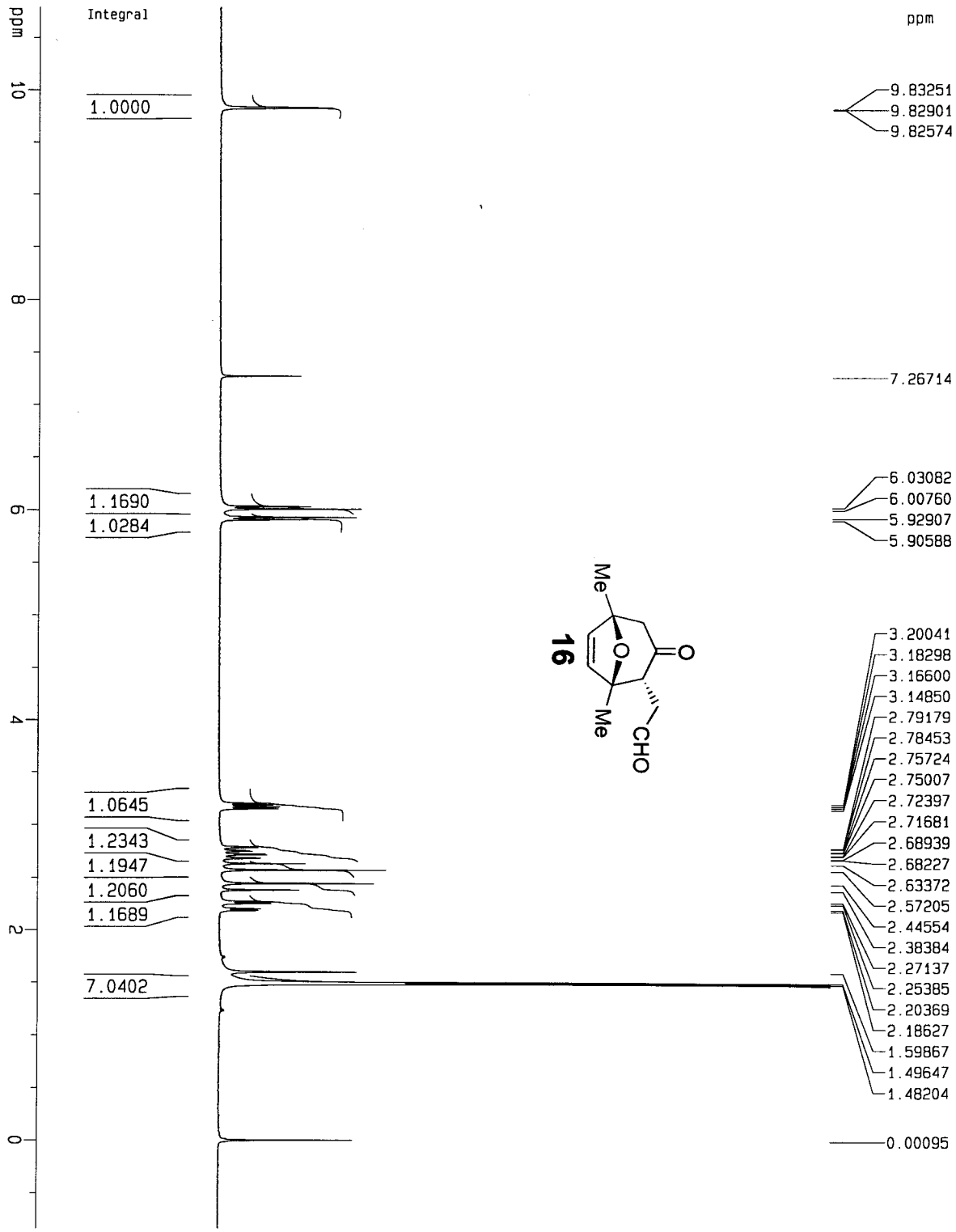
Date_	20020713
Time	13.55
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	3716
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952392 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	148.694 ppm
F1	9352.17 Hz
F2P	0.474 ppm
F2	29.80 Hz
PPMCM	7.41103 ppm
HZCM	466.11865 Hz/

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Current Data Parameters
NAME                  sg-4-43-1
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20020603
Time                   15.22
INSTRUM                arx250
PROBHD     5 mm QNP  1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               5208.333 Hz
FIDRES             0.158946 Hz
AQ                 3.1457779 sec
RG                      2860
DW                 96.000 use
DE                 137.14 use
TE                     300.0 K
D1                 1.00000000 sec
P1                     8.70 use
SFO1          250.1315321 MHz
NUCLEUS                   1H

F2 - Processing parameters
SI                        16384
SF            250.1300056 MHz
WDW                       EM
SSB                        0
LB                  0.20 Hz
GB                         0
PC                      1.50

1D NMR plot parameters
CX                  20.00 cm
CY                  12.50 cm
F1P                10.792 ppm
F1                2699.42 Hz
F2P                -0.838 ppm
F2                 -209.60 Hz
PPMCM              0.58150 ppm
HZCM              145.45097 Hz/
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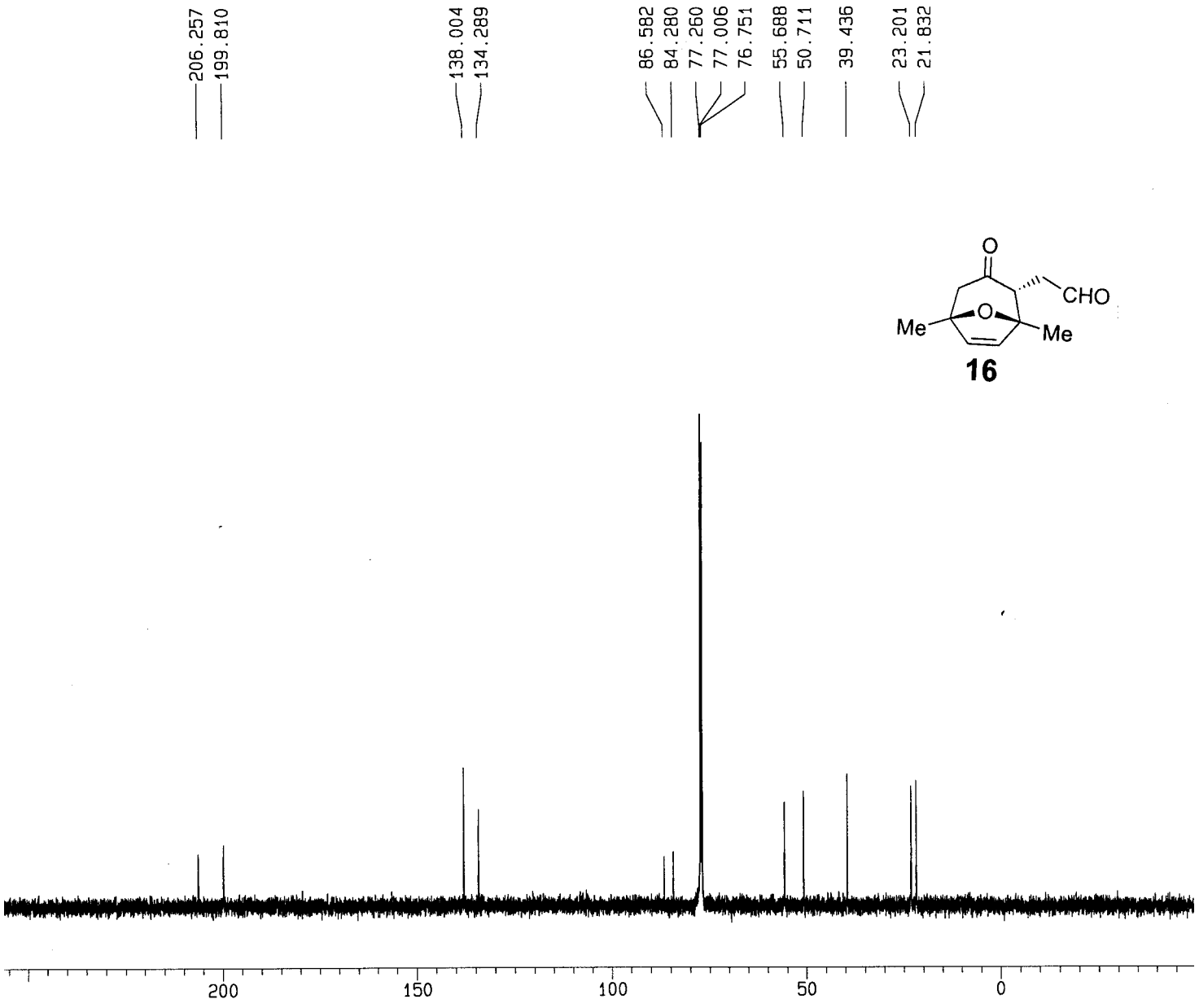


16

Current Data Parameters

NAME	sg-4-43
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020603
Time	17.45
INSTRUM	DRX500
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	450
DS	4
SWH	39681.812 Hz
FIDRES	0.605496 Hz
AQ	0.8258188 sec
RG	16384
DW	12.600 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
d11	0.03000000 sec

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

NUC1	13C
P1	8.24 usec
PL1	3.00 dB
SFO1	125.7713108 MHz

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

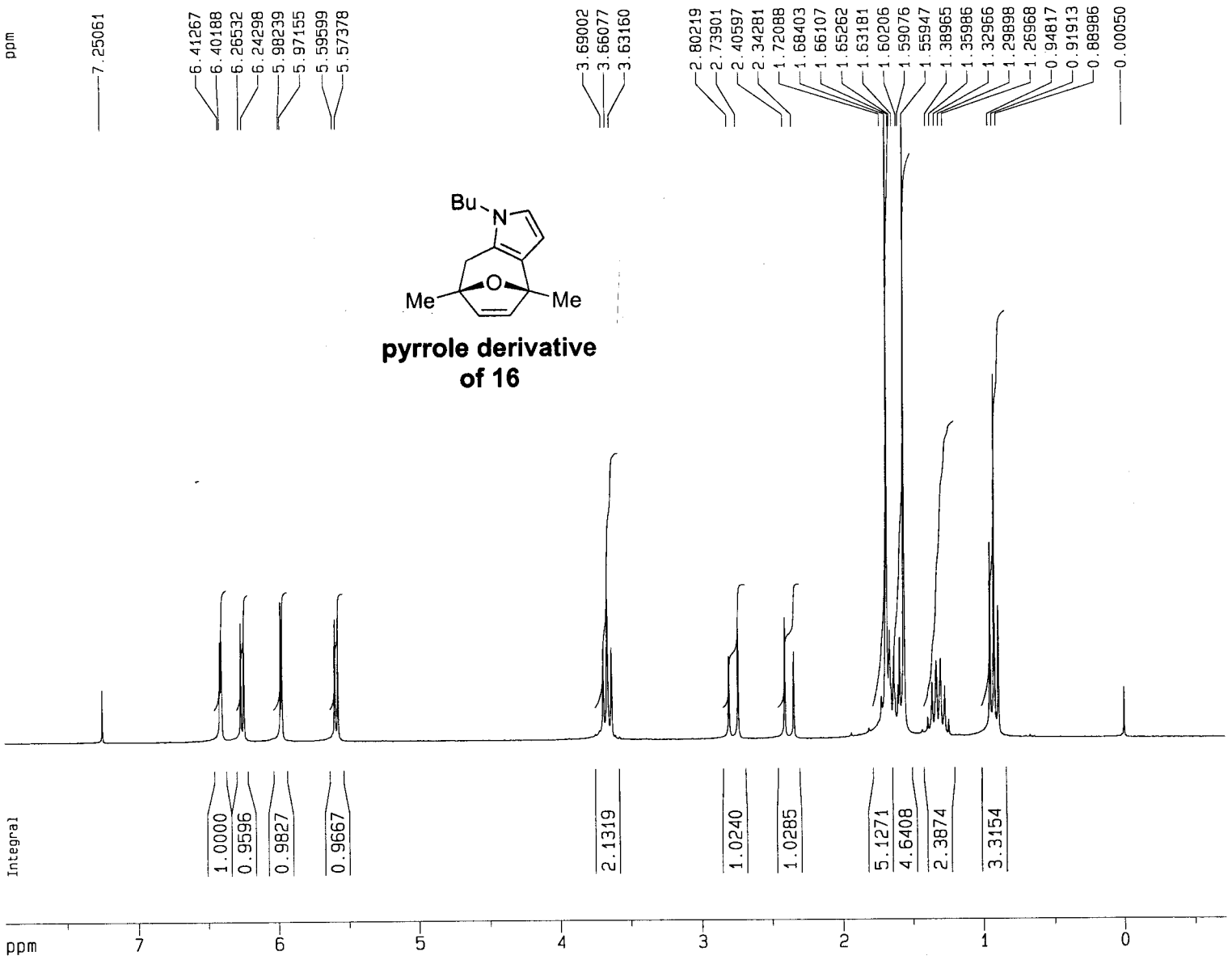
CPDPRG2	waltz16
NUC2	1H
PCPD2	107.00 usec
PL2	0.00 dB
PL12	21.00 dB
SFO2	500.1320005 MHz

F2 - Processing parameters

SI	32768
SF	125.7577923 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	8.00 cm
F1P	266.058 ppm
F1	33458.86 Hz
F2P	-49.484 ppm
F2	-6222.98 Hz
PPMCM	15.77709 ppm/cm
HZCM	1984.09204 Hz/cm



Current Data Parameters

NAME	sg-4-51
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

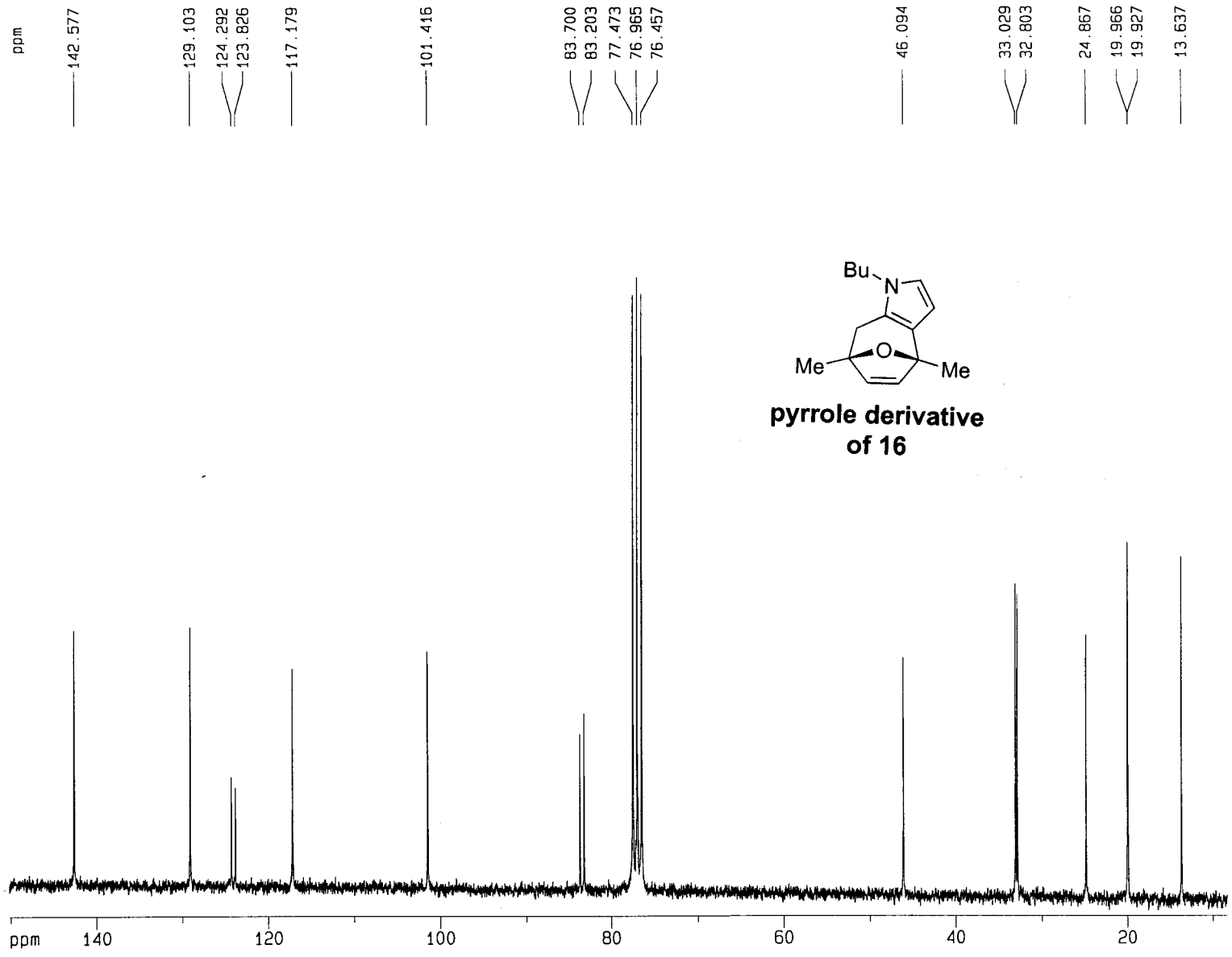
Date_	20020629
Time	12.19
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	1024
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300097 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	7.943 ppm
F1	1986.69 Hz
F2P	-0.713 ppm
F2	-178.40 Hz
PPMCM	0.43279 ppm
HZCM	108.25461 Hz/



Current Data Parameters

NAME	sg-4-51
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

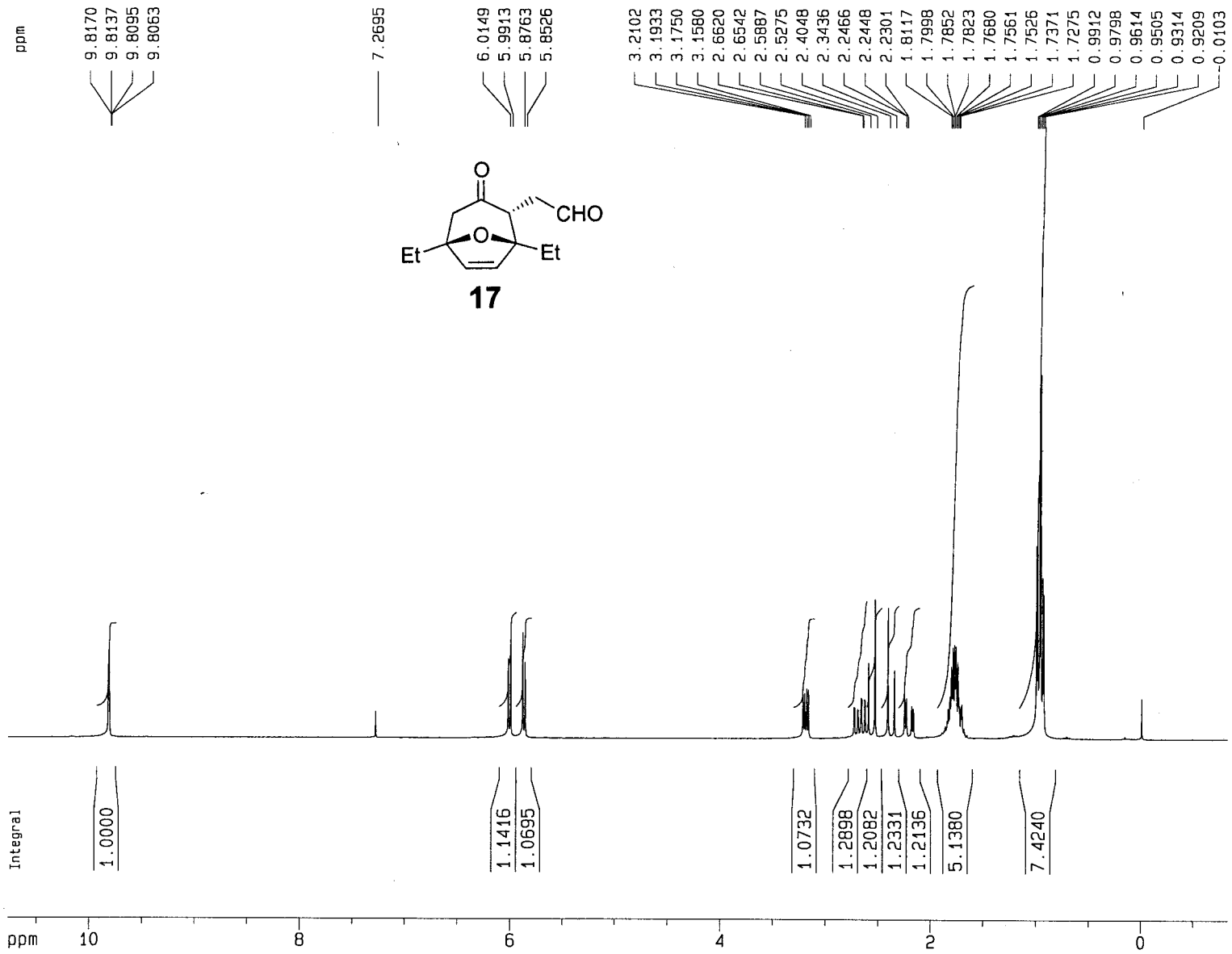
Date_	20020629
Time	14.43
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	4123
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952440 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	150.212 ppm
F1	9447.60 Hz
F2P	8.366 ppm
F2	526.19 Hz
PPMCM	7.09228 ppm
HZCM	446.07059 Hz/



Current Data Parameters

NAME	sg-4-103-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

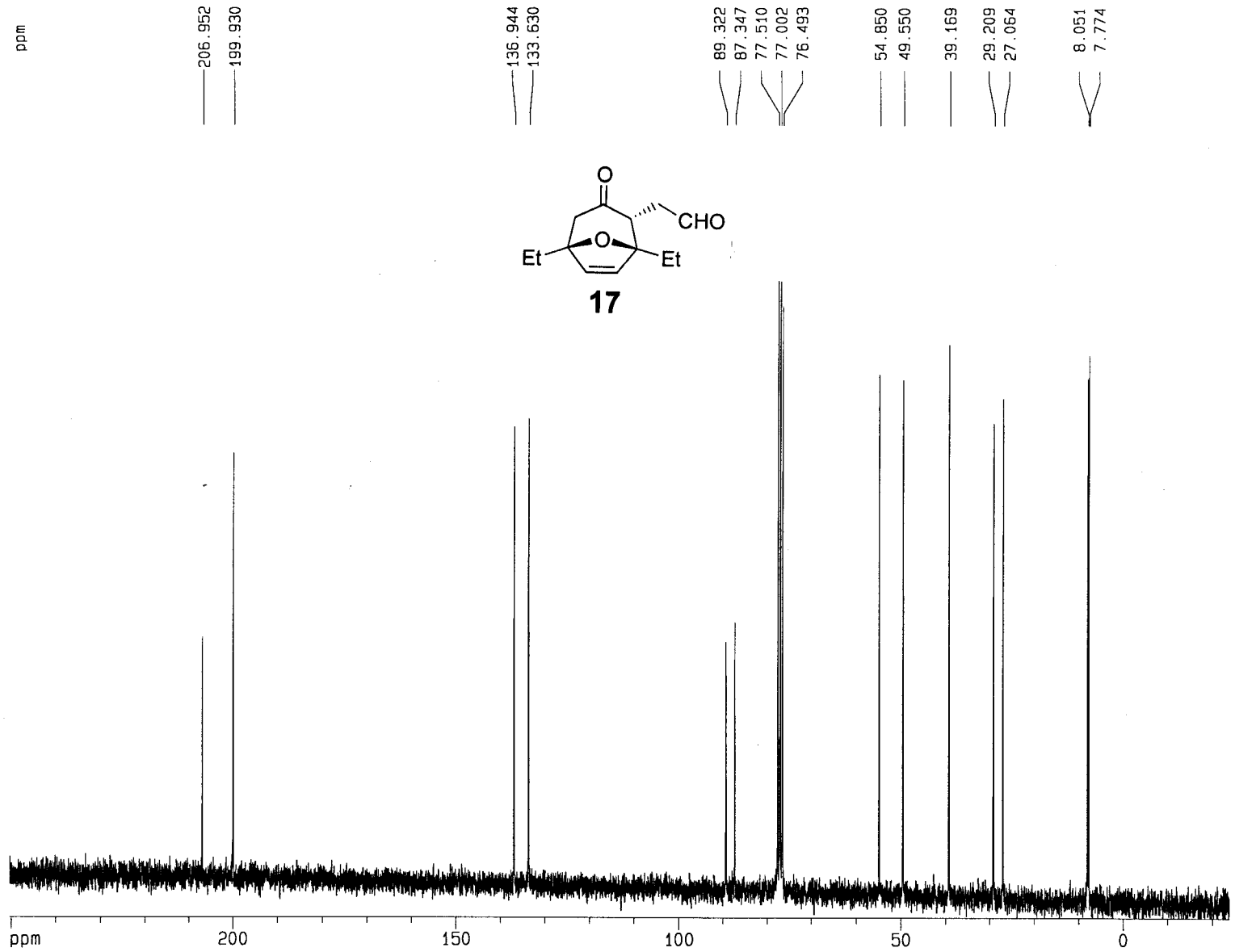
Date_	20020623
Time	17.49
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	715
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300049 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	10.775 ppm
F1	2695.05 Hz
F2P	-0.829 ppm
F2	-207.30 Hz
PPMCM	0.58017 ppm
HZCM	145.11768 Hz/



Current Data Parameters

NAME	sg-4-103-1
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

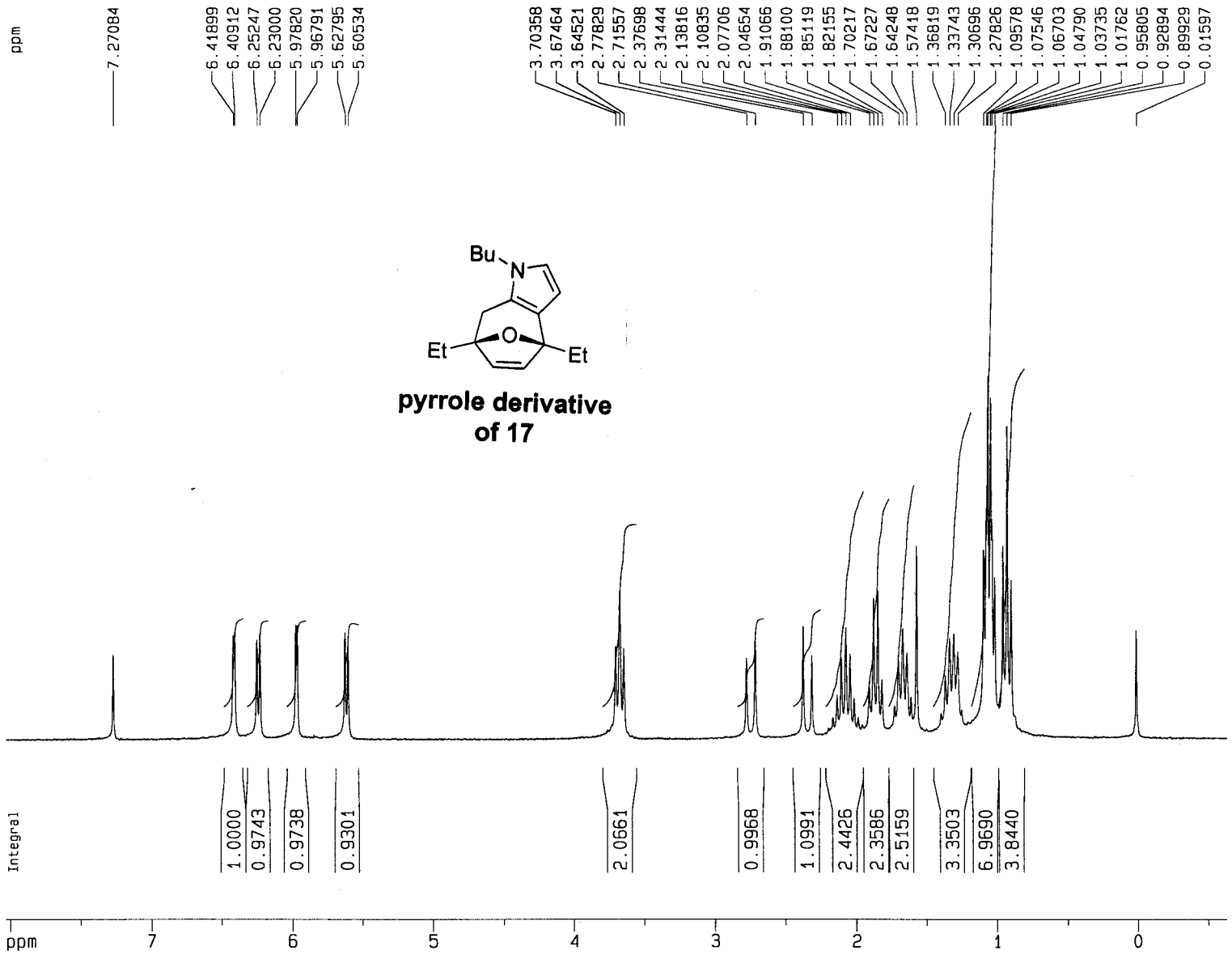
Date_	20020623
Time	18.03
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	1238
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952419 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	250.354 ppm
F1	15746.05 Hz
F2P	-23.775 ppm
F2	-1495.33 Hz
PPMCM	13.70643 ppm
HZCM	862.06891 Hz/



Current Data Parameters

NAME	sg-4-105
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

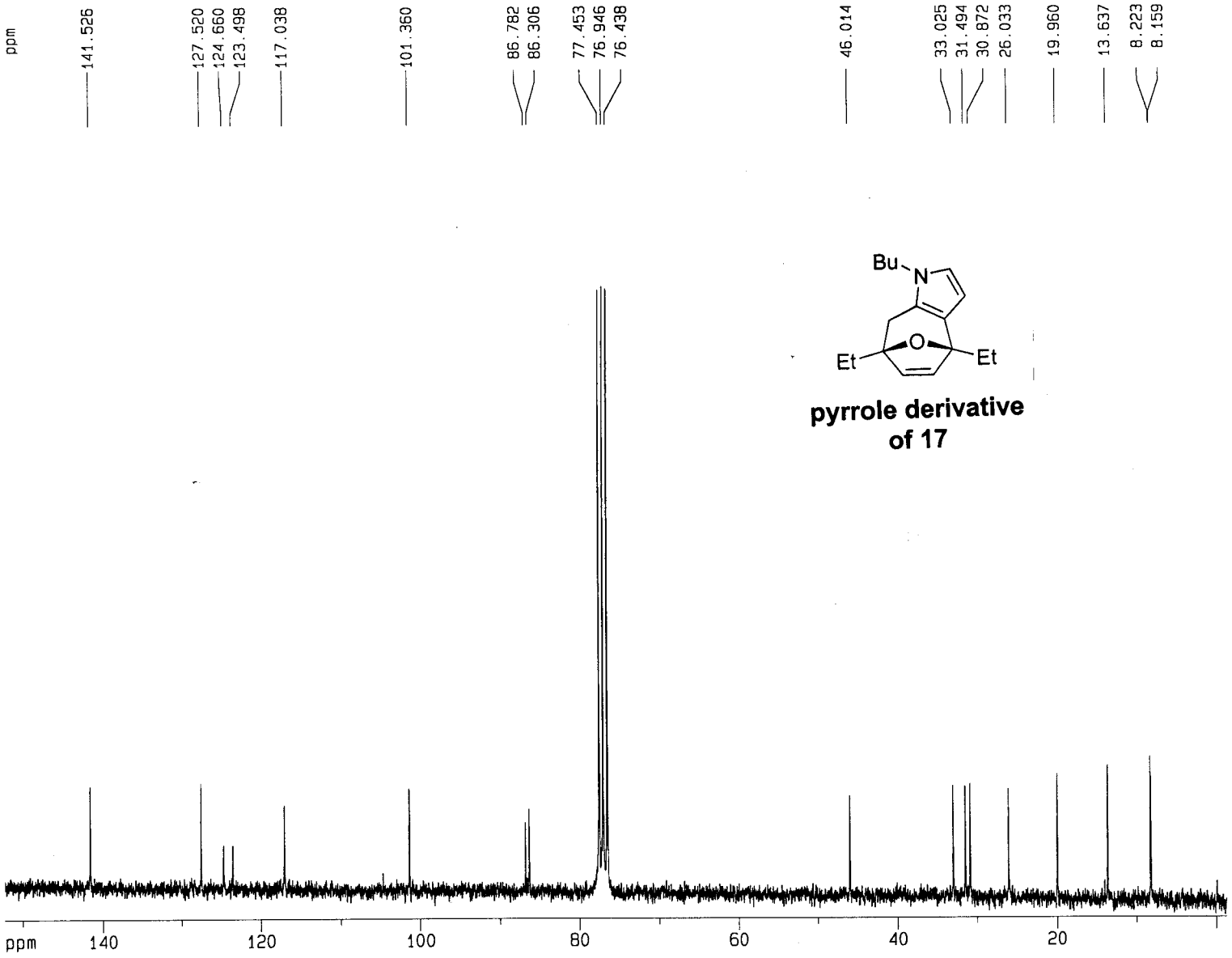
Date_	20020624
Time	16.46
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	1430
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300049 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	8.033 ppm
F1	2009.22 Hz
F2P	-0.630 ppm
F2	-157.61 Hz
PPMCM	0.43314 ppm
HZCM	108.34129 Hz/



Current Data Parameters

NAME	sg-4-105
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020624
Time	16.51
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	2959
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

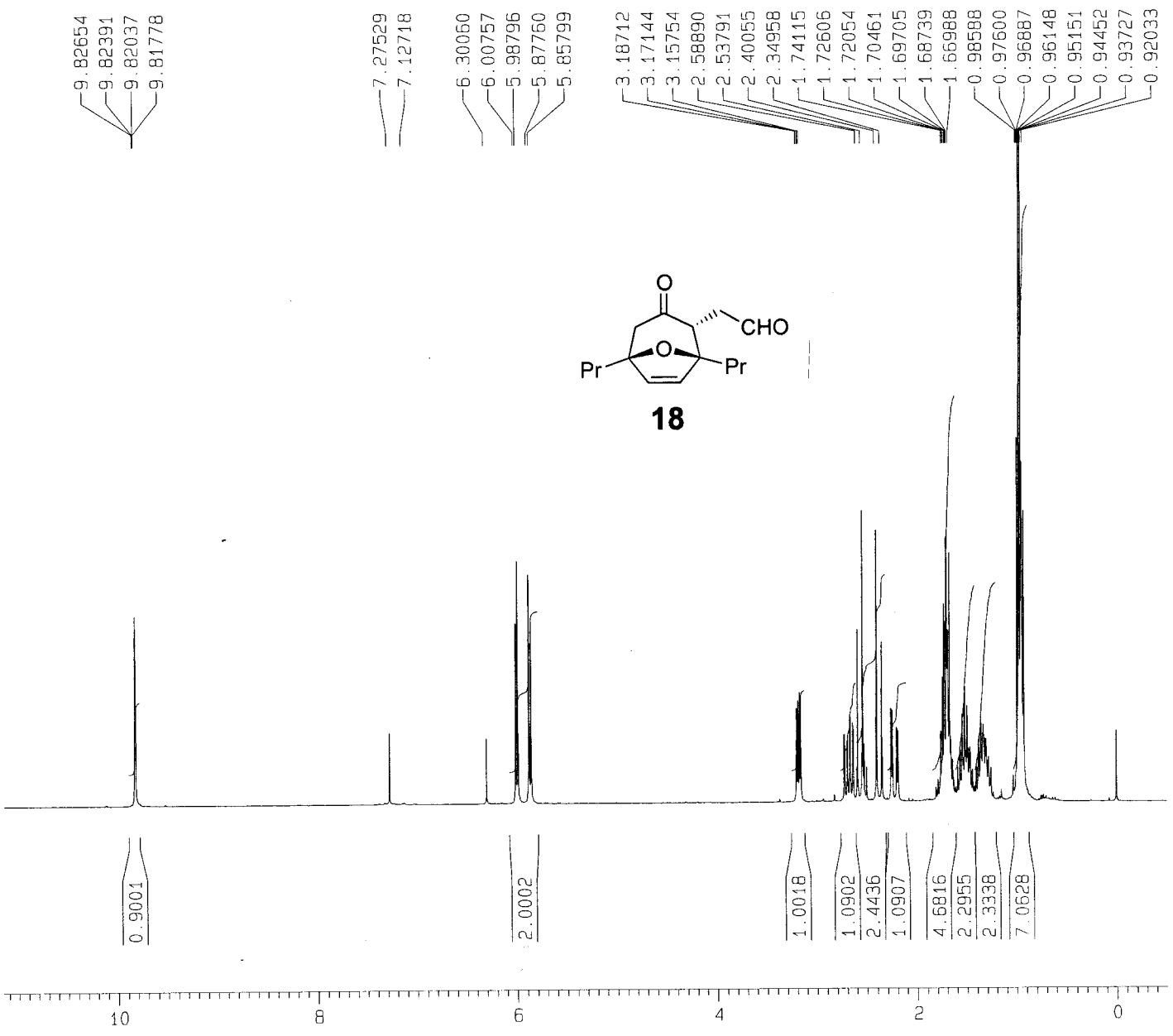
F2 - Processing parameters

SI	32768
SF	62.8952440 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	152.264 ppm
F1	9576.67 Hz
F2P	-1.280 ppm
F2	-80.48 Hz
PPMCM	7.67717 ppm
HZCM	482.85727 Hz/

sw-IV-110-A11H NMR



18

Current Data Parameters

NAME	sw-IV-110-A1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20021210
Time	21.12
INSTRUM	drx300
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	143.7
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
D31	0.00000000 sec

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

NUC1	1H
P1	7.50 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

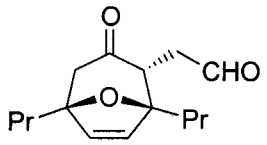
SI	32768
SF	300.1300016 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.30

1D NMR plot parameters

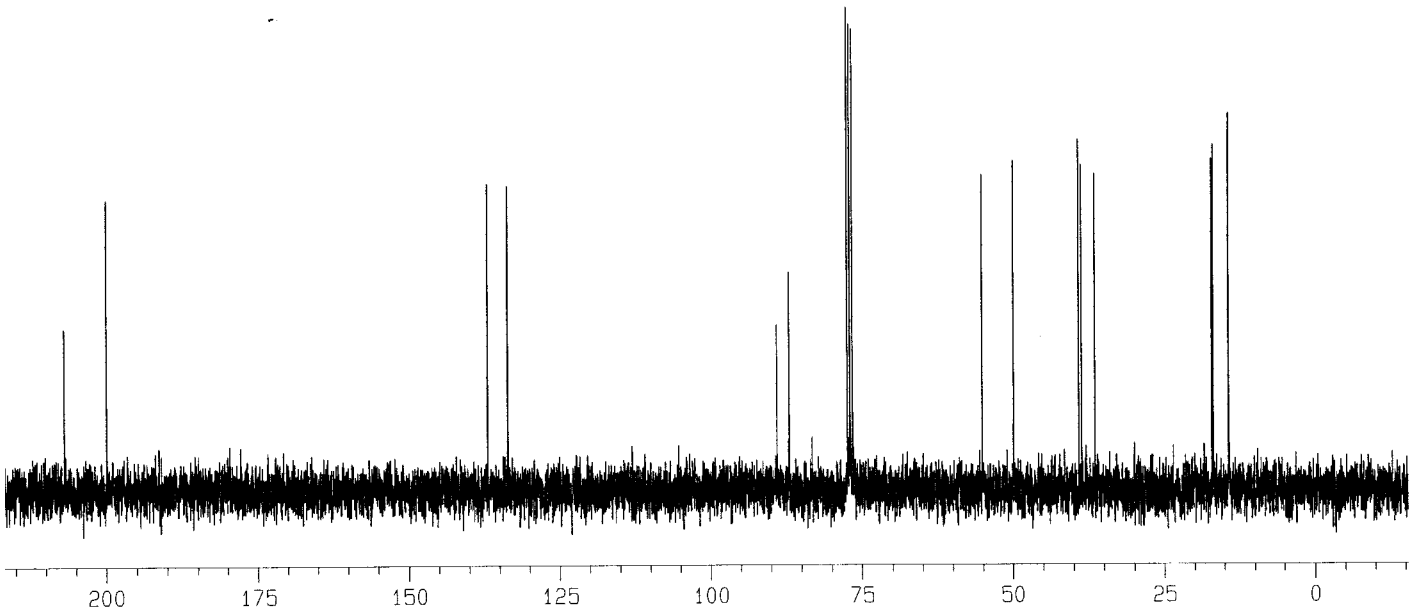
CX	20.00 cm
CY	12.50 cm
F1P	12.000 ppm
F1	3601.56 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.62500 ppm/cm
HZCM	187.58125 Hz/cm

sw-IV-110-A113C NMR

206.888
199.943
136.896
133.607
88.987
86.978
77.421
76.998
76.574
55.179
49.895
39.191
38.688
36.509
17.199
16.951
14.343
14.296



18




```
Current Data Parameters
NAME        sw-IV-110-A1
EXPNO                  2
PROCNO                 1

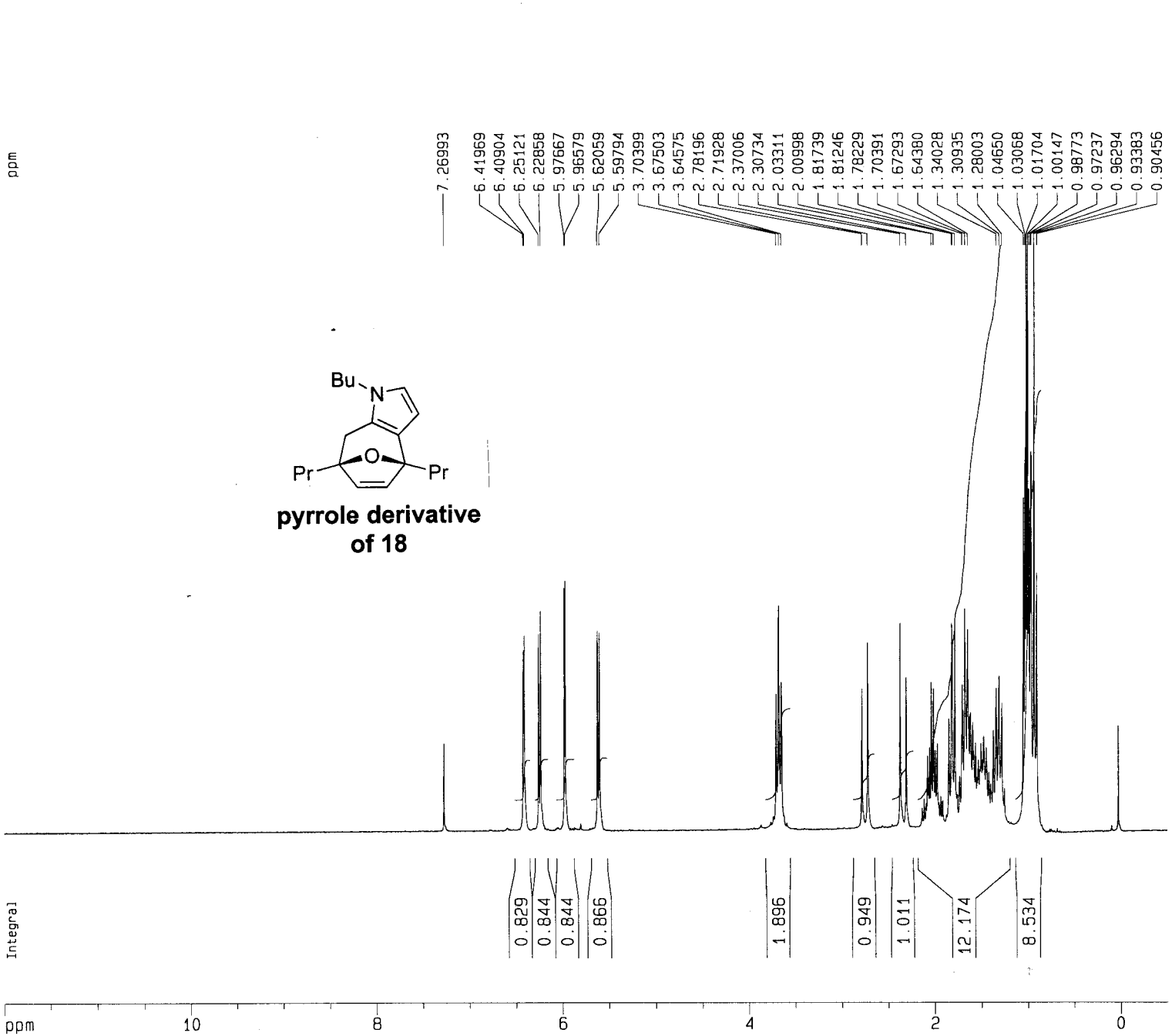
F2 - Acquisition Parameters
Date_           20021210
Time               21.14
INSTRUM          drx300
PROBHD   5 mm Multinucl
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  173
DS                    4
SWH          18850.141 Hz
FIDRES        0.287630 Hz
AQ            1.7383924 sec
RG                32768
DW               26.525 usec
DE               37.89 usec
TE               300.0 K
D1           1.00000000 sec
d11          0.03000000 sec
D31          0.00000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 7.75 usec
PL1                6.00 dB
SFO1         75.4760107 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              24.50 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF         75.4677514 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX               20.00 cm
CY                5.00 cm
F1P             234.330 ppm
F1            17684.39 Hz
F2P            -15.447 ppm
F2            -1165.75 Hz
PPMCM         12.48887 ppm/cm
HZCM         942.50702 Hz/cm
```



Current Data Parameters

NAME	sw-IV-116-A1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

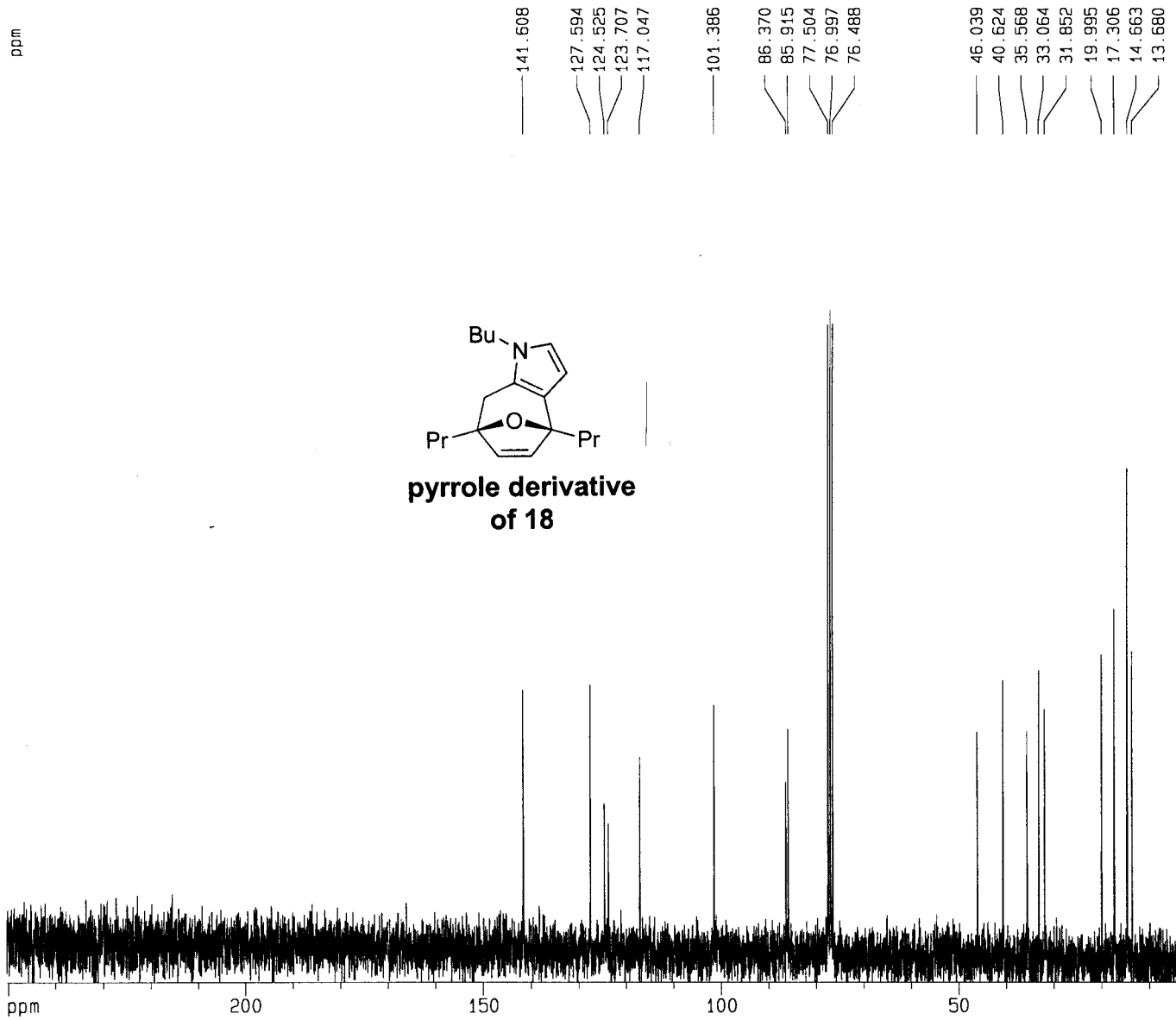
Date_	20021211
Time	11.44
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	715
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300049 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	12.000 ppm
F1	3001.56 Hz
F2P	-0.500 ppm
F2	-125.07 Hz
PPMCM	0.62500 ppm
HZCM	156.33125 Hz/



pyrrole derivative of 18

Current Data Parameters

NAME	sw-IV-116-A1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

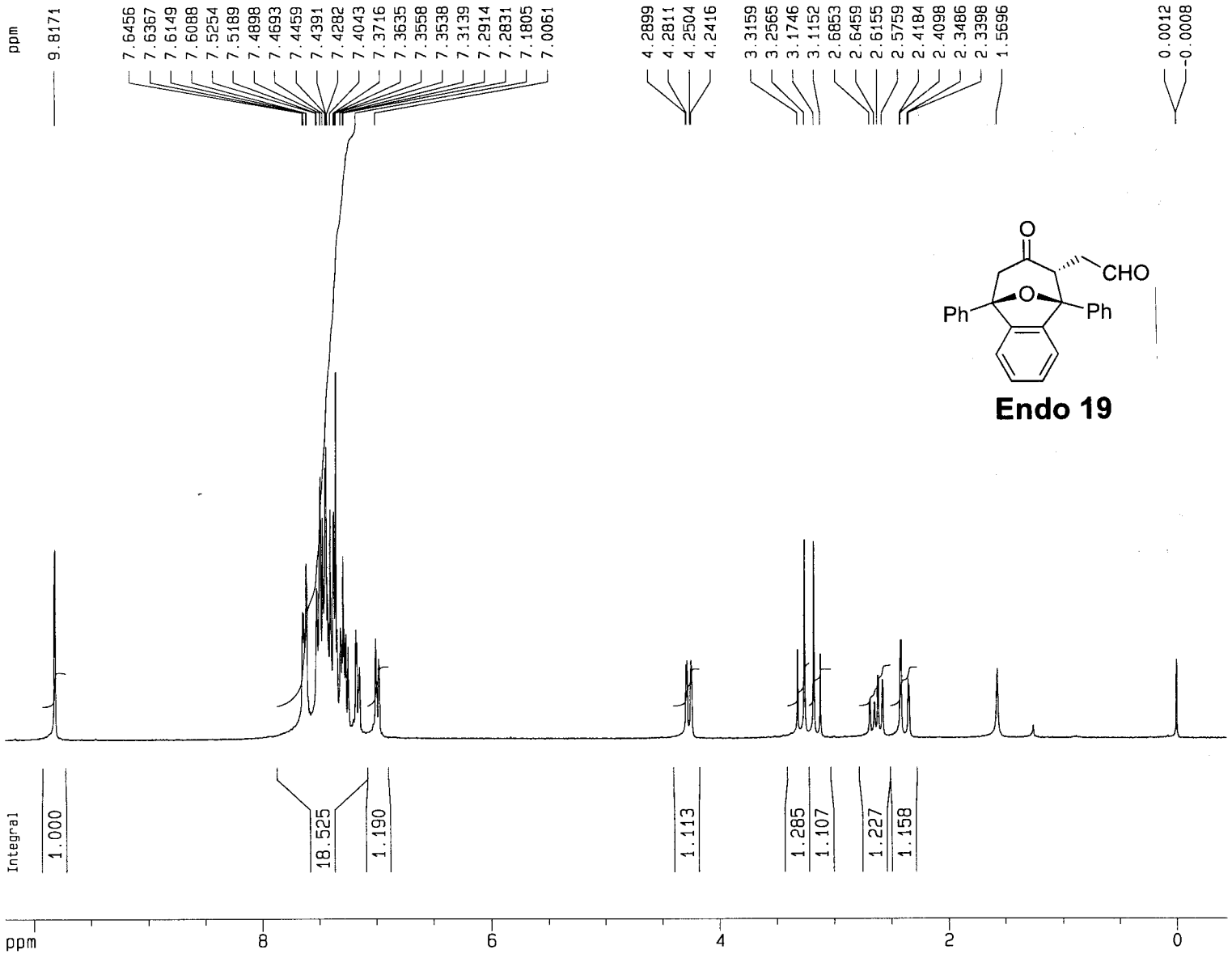
Date_	20021211
Time	11.47
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	172
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952419 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	250.388 ppm
F1	15748.23 Hz
F2P	-23.740 ppm
F2	-1493.14 Hz
PPMCM	13.70643 ppm
HZCM	862.06891 Hz/



Endo 19

Current Data Parameters

NAME	sg-4-77-1-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

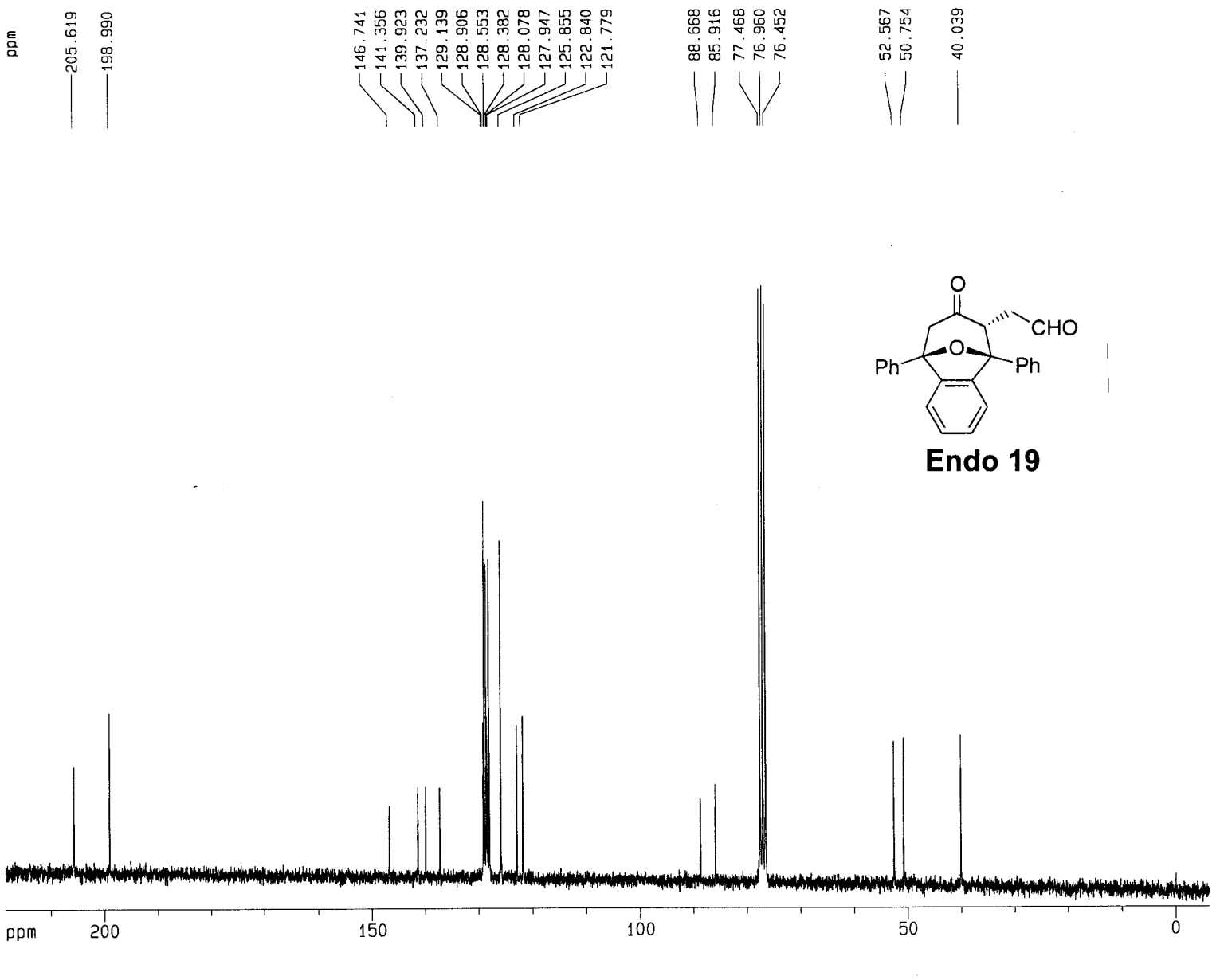
Date_	20020629
Time	15.46
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	1024
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300100 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	10.258 ppm
F1	2565.80 Hz
F2P	-0.436 ppm
F2	-109.02 Hz
PPMCM	0.53469 ppm
HZCM	133.74112 Hz/



Current Data Parameters

NAME	sg-4-77-1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

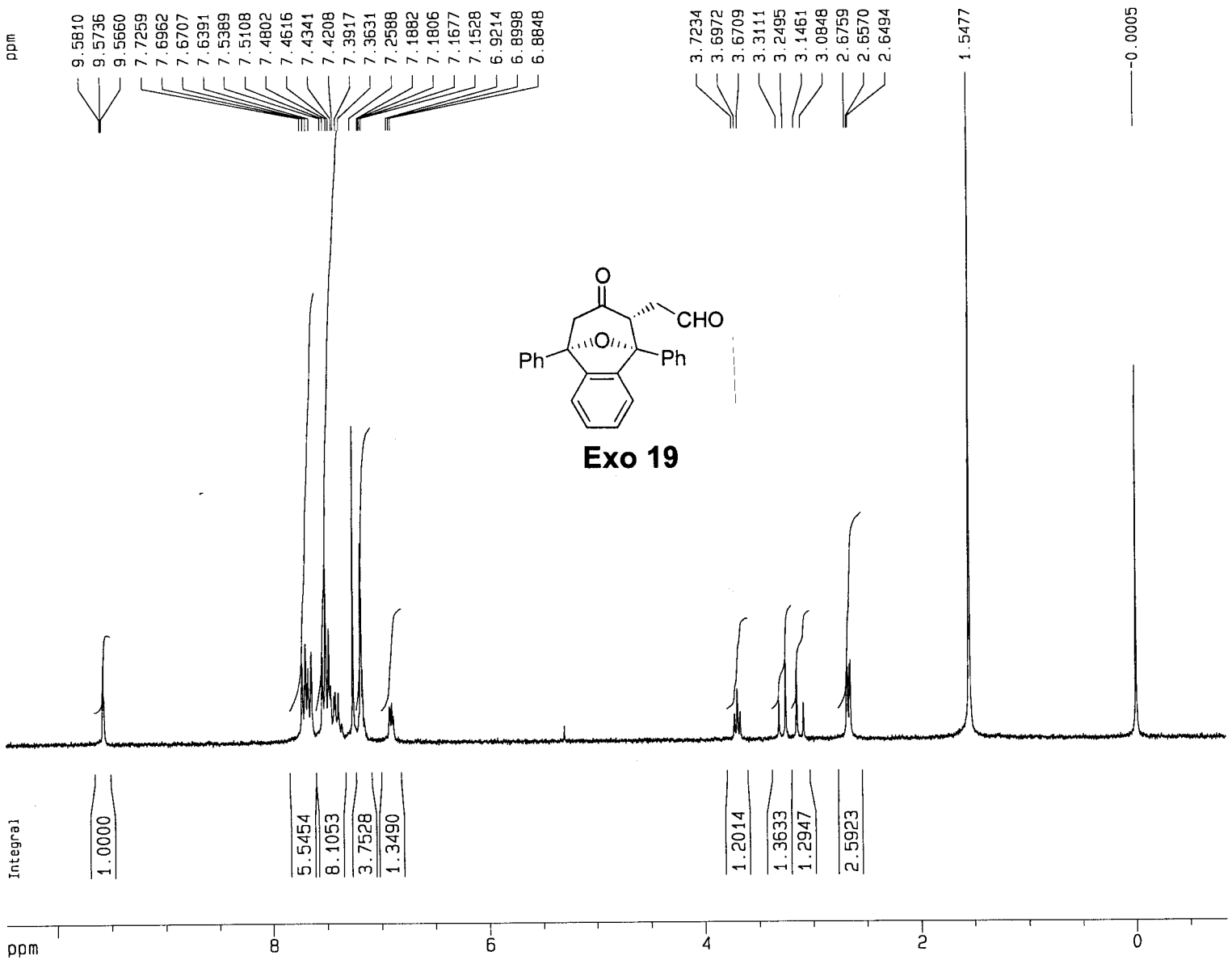
Date_	20020628
Time	18.58
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	3250
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SF01	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952440 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	218.478 ppm
F1	13741.24 Hz
F2P	-6.243 ppm
F2	-392.68 Hz
PPMCM	11.23608 ppm
HZCM	706.69604 Hz/



Current Data Parameters

NAME	sg-4-77-4-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

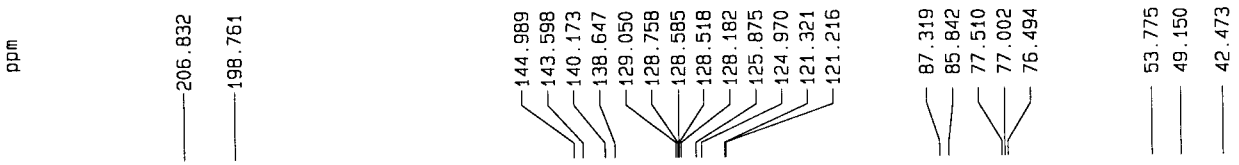
Date_	20020608
Time	21.29
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	11400
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

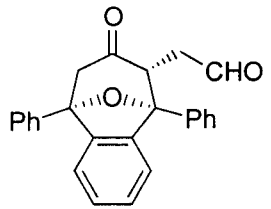
F2 - Processing parameters

SI	16384
SF	250.1300078 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	10.479 ppm
F1	2621.04 Hz
F2P	-0.833 ppm
F2	-208.48 Hz
PPMCM	0.56561 ppm
HZCM	141.47635 Hz/





Exo 19

Current Data Parameters

NAME	sg-4-77-4-2
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

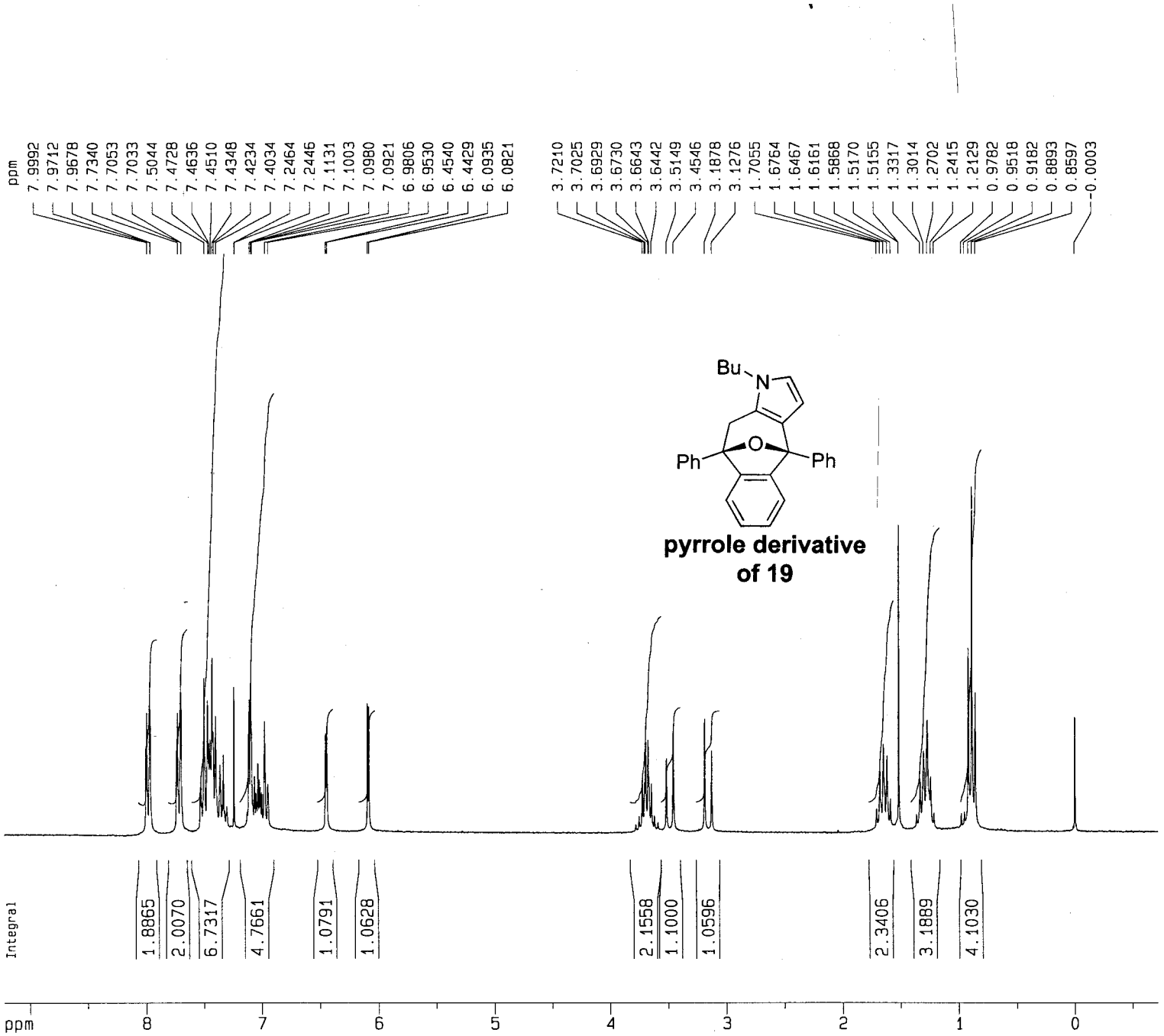
Date_	20020608
Time	22.03
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	2371
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952408 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	235.908 ppm
F1	14837.48 Hz
F2P	-2.900 ppm
F2	-182.42 Hz
PPMCM	11.94041 ppm
HZCM	750.99475 Hz/



Current Data Parameters

NAME	sg-4-78
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

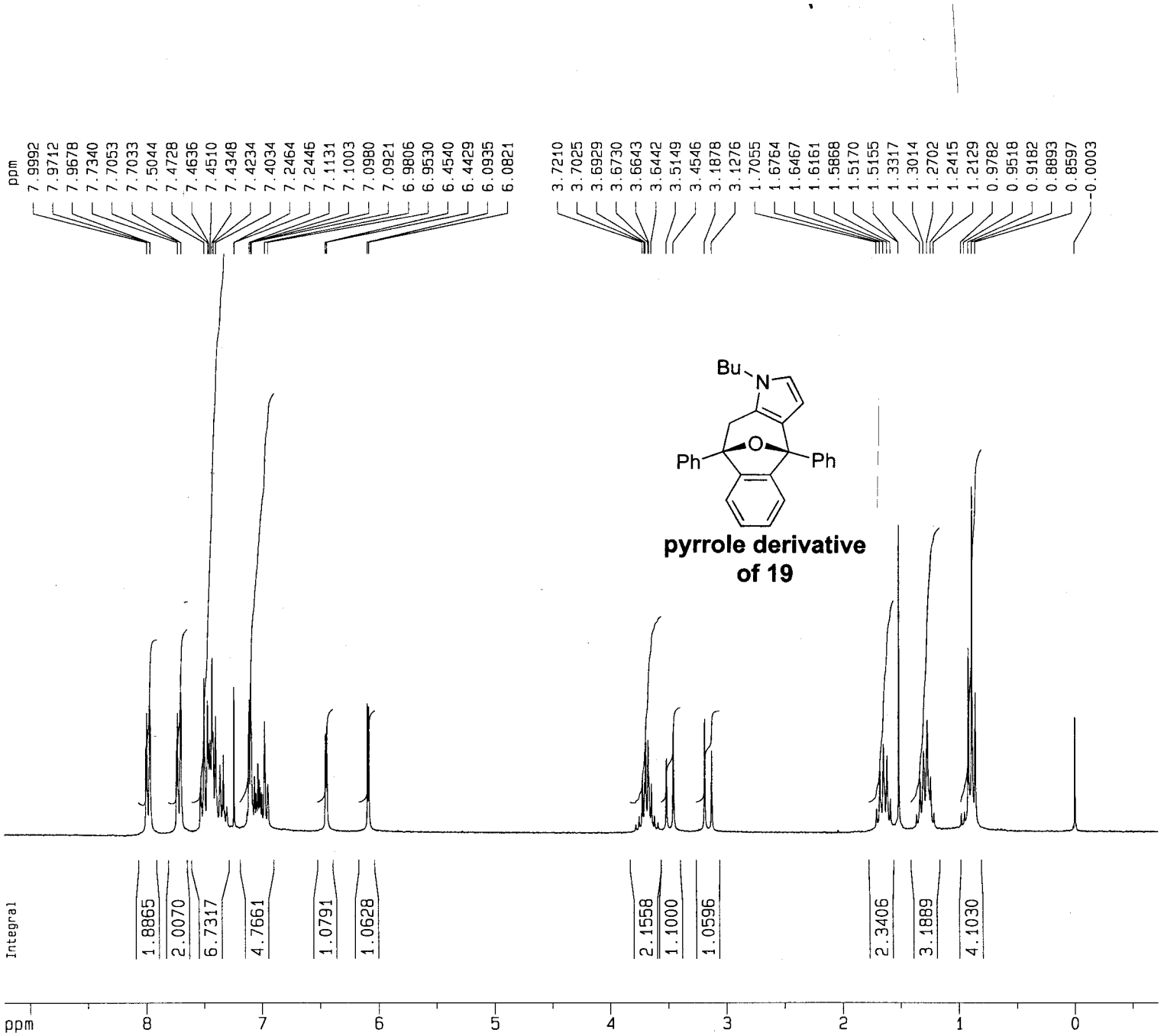
Date_	20020629
Time	16.03
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	2048
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300110 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	9.229 ppm
F1	2308.42 Hz
F2P	-0.718 ppm
F2	-179.67 Hz
PPMCM	0.49736 ppm
HZCM	124.40448 Hz/



Current Data Parameters

NAME	sg-4-78
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

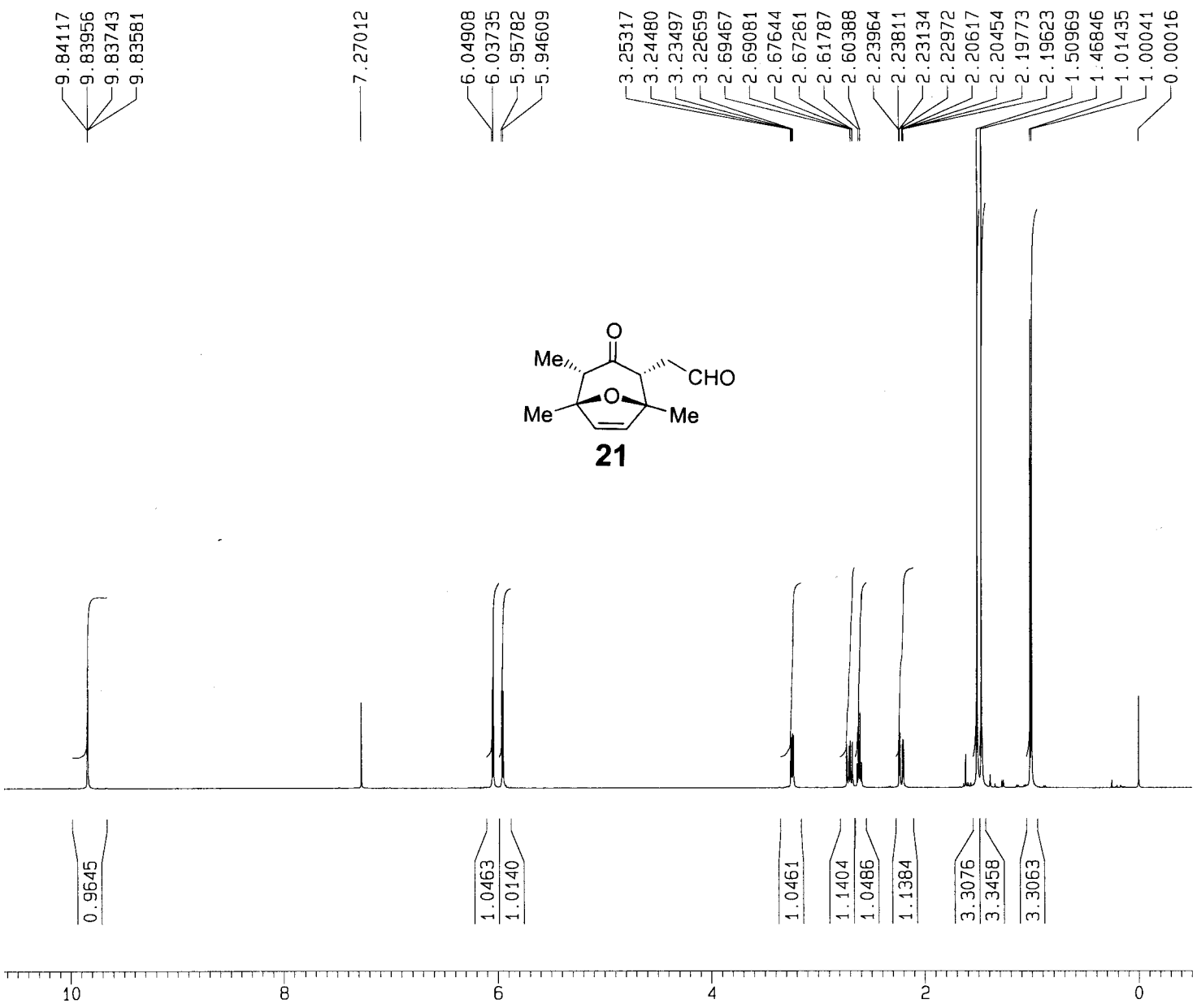
Date_	20020629
Time	16.03
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	2048
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300110 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	9.229 ppm
F1	2308.42 Hz
F2P	-0.718 ppm
F2	-179.67 Hz
PPMCM	0.49736 ppm
HZCM	124.40448 Hz/



21

Current Data Parameters

NAME	XH-IV-4-A2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20021125
Time	23.06
INSTRUM	DRX500
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	45056
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.229283 Hz
AQ	2.1807604 sec
RG	64
DW	48.400 usec
DE	6.00 usec
TE	296.7 K
D1	1.00000000 sec

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

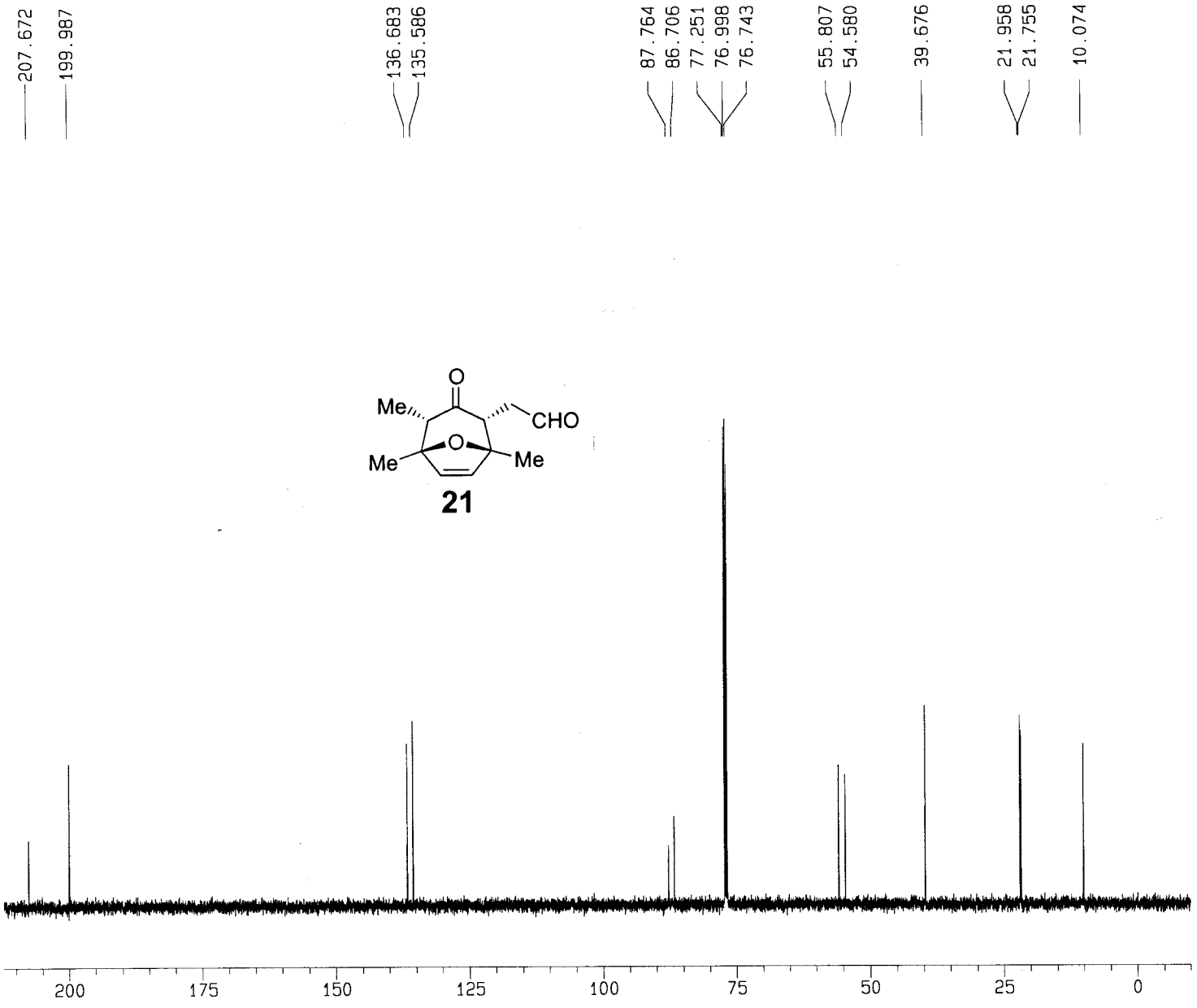
NUC1	1H
P1	13.25 usec
PL1	-3.00 dB
SFO1	500.1330885 MHz

F2 - Processing parameters

SI	32768
SF	500.1300084 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	11.000 ppm
F1	5501.43 Hz
F2P	-0.500 ppm
F2	-250.07 Hz
PPMCM	0.57500 ppm/cr
HZCM	287.57477 Hz/cm



21

```
Current Data Parameters
NAME                  XH-IV-4-A2
EXPNO                          2
PROCNO                         1

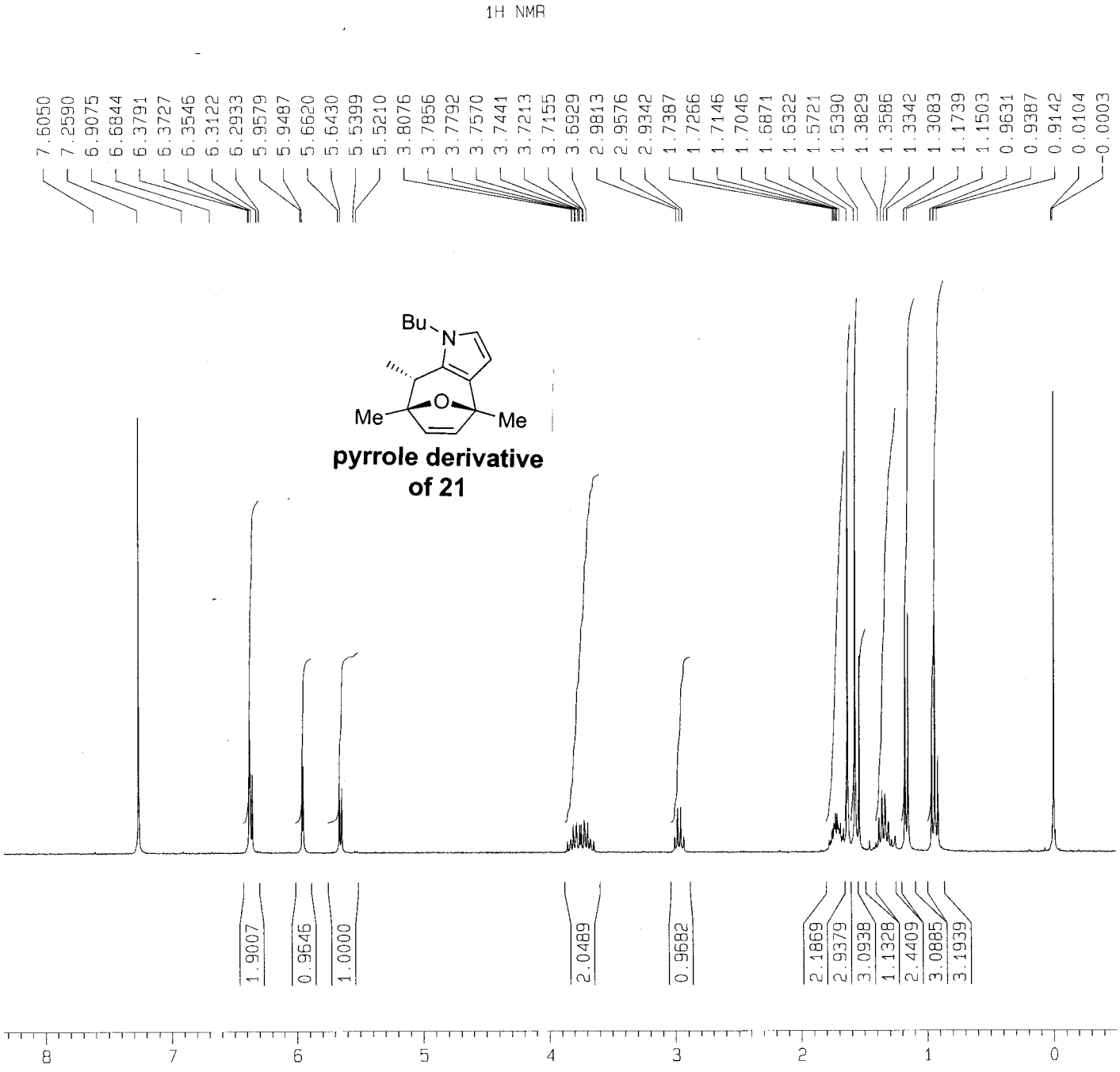
F2 - Acquisition Parameters
Date_                   20021125
Time                       23.08
INSTRUM                   DRX500
PROBHD   5 mm Multinucl
PULPROG                   zgdc30
TD                         65536
SOLVENT                    CDCl3
NS                           150
DS                             4
SWH                    39681.812 Hz
FIDRES                 0.605496 Hz
AQ                     0.8258188 sec
RG                         16384
DW                        12.600 usec
DE                         6.00 usec
TE                        298.0 K
D1                     1.00000000 sec
d11                    0.03000000 sec

======== CHANNEL f1 ========
NUC1                         13C
P1                        7.90 usec
PL1                       3.00 dB
SFO1                125.7713108 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    88.00 usec
PL2                       0.00 dB
PL12                     21.00 dB
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577935 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                        20.00 cm
CY                         8.00 cm
F1P                     220.000 ppm
F1                    27666.71 Hz
F2P                     -10.000 ppm
F2                    -1257.58 Hz
PPMCM                  11.50000 ppm/cm
HZCM                1446.21472 Hz/cm
```




```
Current Data Parameters
NAME                XH-IV-9-1
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20021127
Time                  14.59
INSTRUM              drx300
PROBHD    5 mm Multinucl
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       2
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                      64
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                 1.00000000 sec
D31                0.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    7.50 usec
PL1                   0.00 dB
SF01           300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300065 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.30

1D NMR plot parameters
CX                   20.00 cm
CY                    8.00 cm
F1P                  9.000 ppm
F1                2701.17 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM              0.47500 ppm/cm
HZCM             142.56175 Hz/cm
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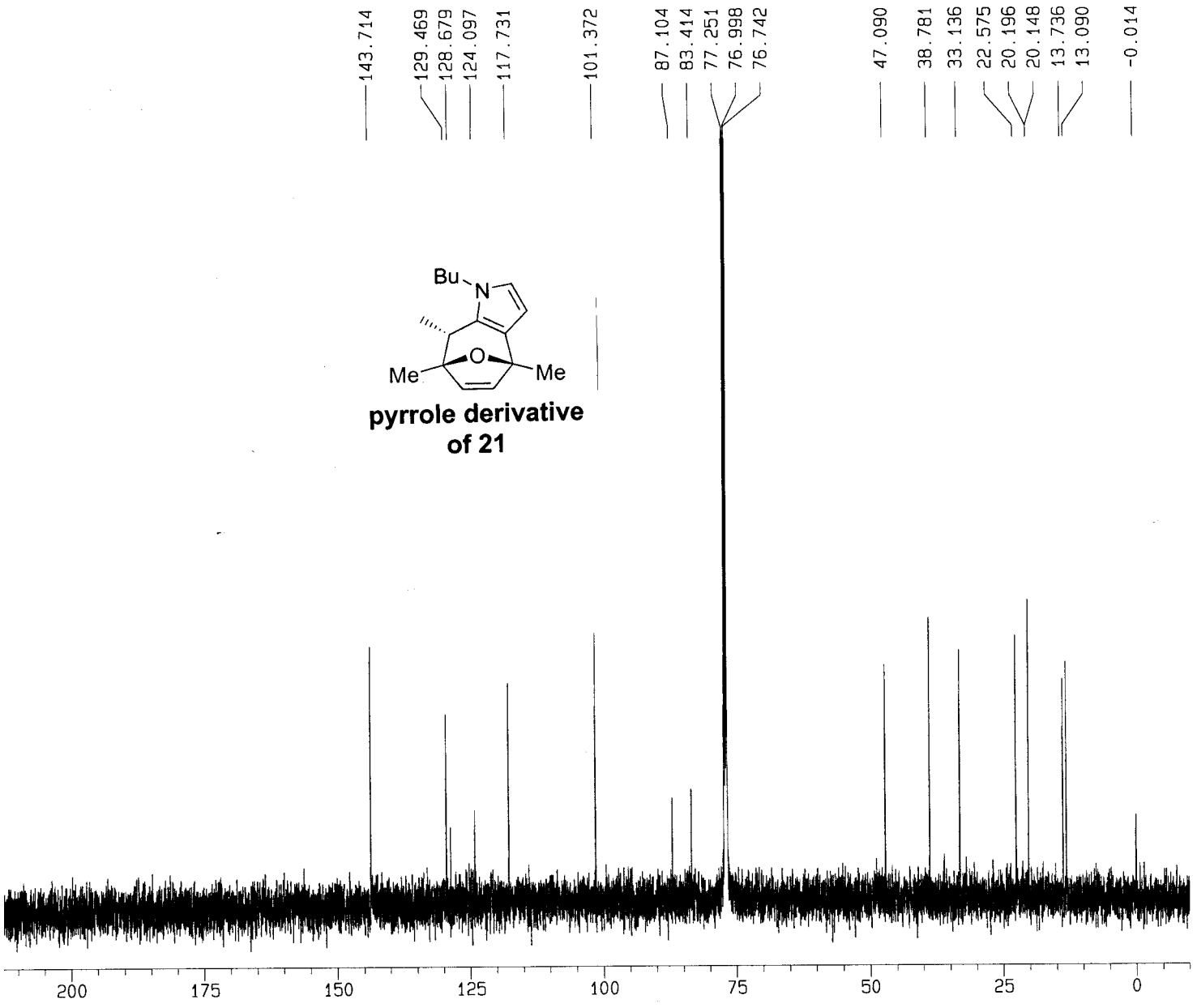


pyrrole derivative of 21

```
Current Data Parameters
NAME                  XH-IV-9-1
EXPNO                         2
PROCNO                        1

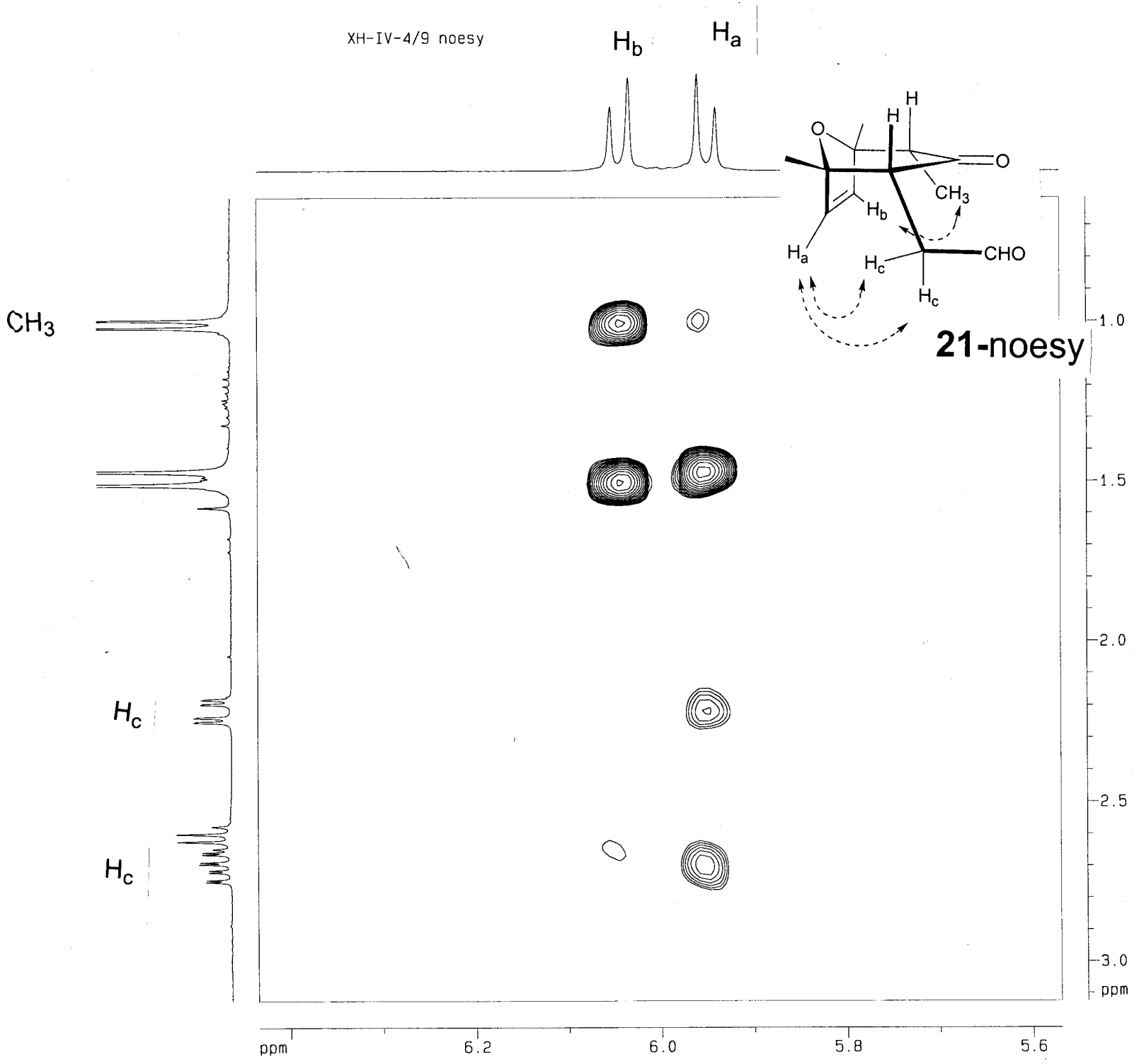
F2 - Acquisition Parameters
Date_              20021127
Time                    15.20
INSTRUM                DRX500
PROBHD   5 mm Multinucl
PULPROG                zgdc30
TD                      65536
SOLVENT                 CDCl3
NS                       2013
DS                          4
SWH                39681.812 Hz
FIDRES             0.605496 Hz
AQ                 0.8258188 sec
RG                      16384
DW                     12.600 usec
DE                      6.00 usec
TE                     298.0 K
D1                1.00000000 sec
d11               0.03000000 sec

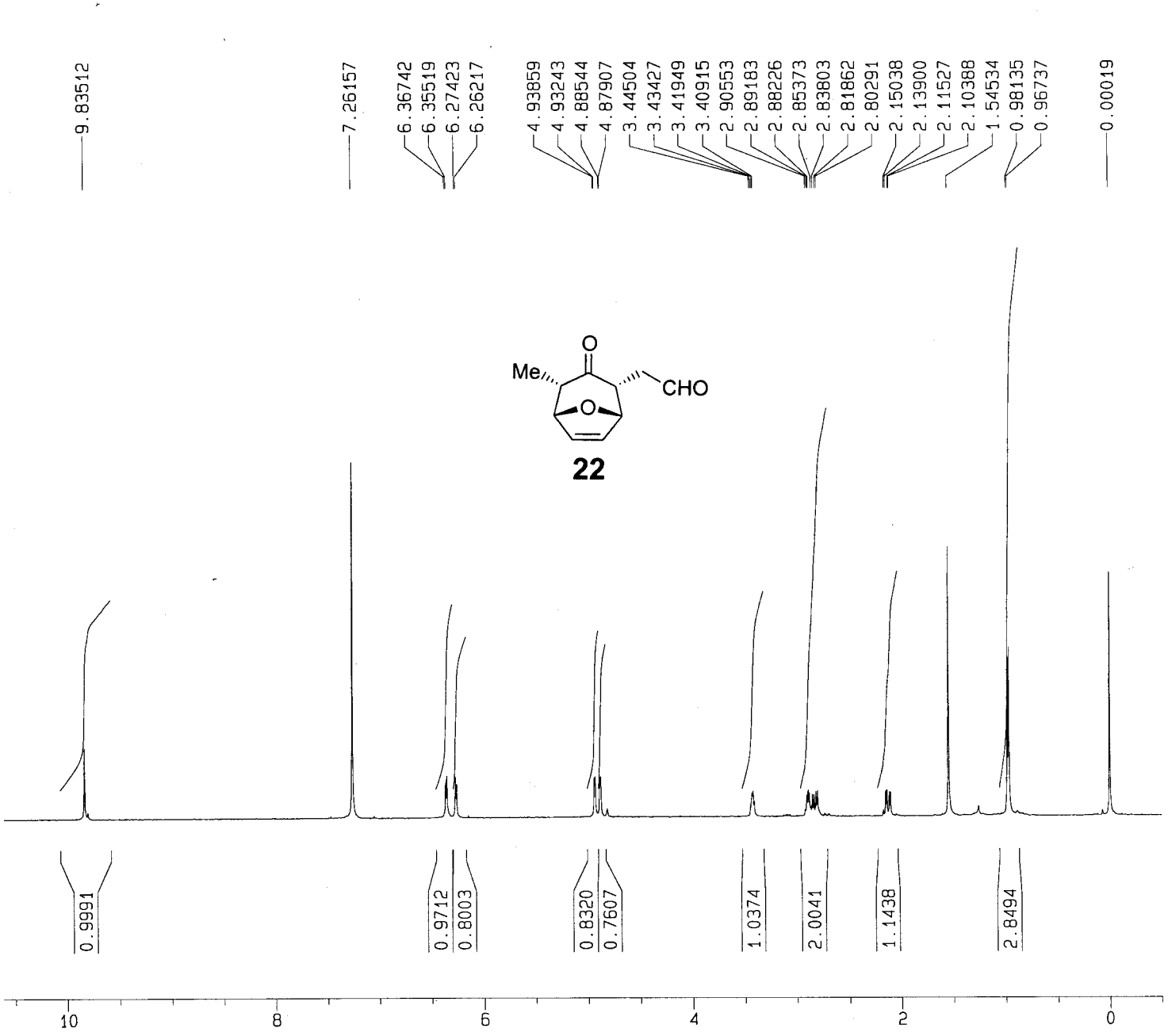
======== CHANNEL f1 ========
NUC1                      13C
P1                       7.90 usec
PL1                      3.00 dB
SFO1              125.7713108 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  88.00 usec
PL2                     0.00 dB
PL12                   21.00 dB
SFO2              500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                125.7577923 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
CY                       100.00 cm
F1P                     220.000 ppm
F1                    27666.71 Hz
F2P                     -10.000 ppm
F2                    -1257.58 Hz
PPMCM                  11.50000 ppm/cm
HZCM                1446.21460 Hz/cm
```


XH-IV-4/9 noesy
Hb
Ha
H
H
O
O
CH3
Hb
Ha
Hc
CHO
Hc
21-noesy
CH3
Hc
Hc
1.0
1.5
2.0
2.5
3.0
ppm
ppm
6.2
6.0
5.8
5.6



Current Data Parameters

NAME	XH-IV-8-1-A1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20021212
Time	17.09
INSTRUM	DRX500
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	57344
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.180151 Hz
AQ	2.7754996 sec
RG	64
DW	48.400 usec
DE	6.00 usec
TE	296.7 K
D1	1.00000000 sec

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

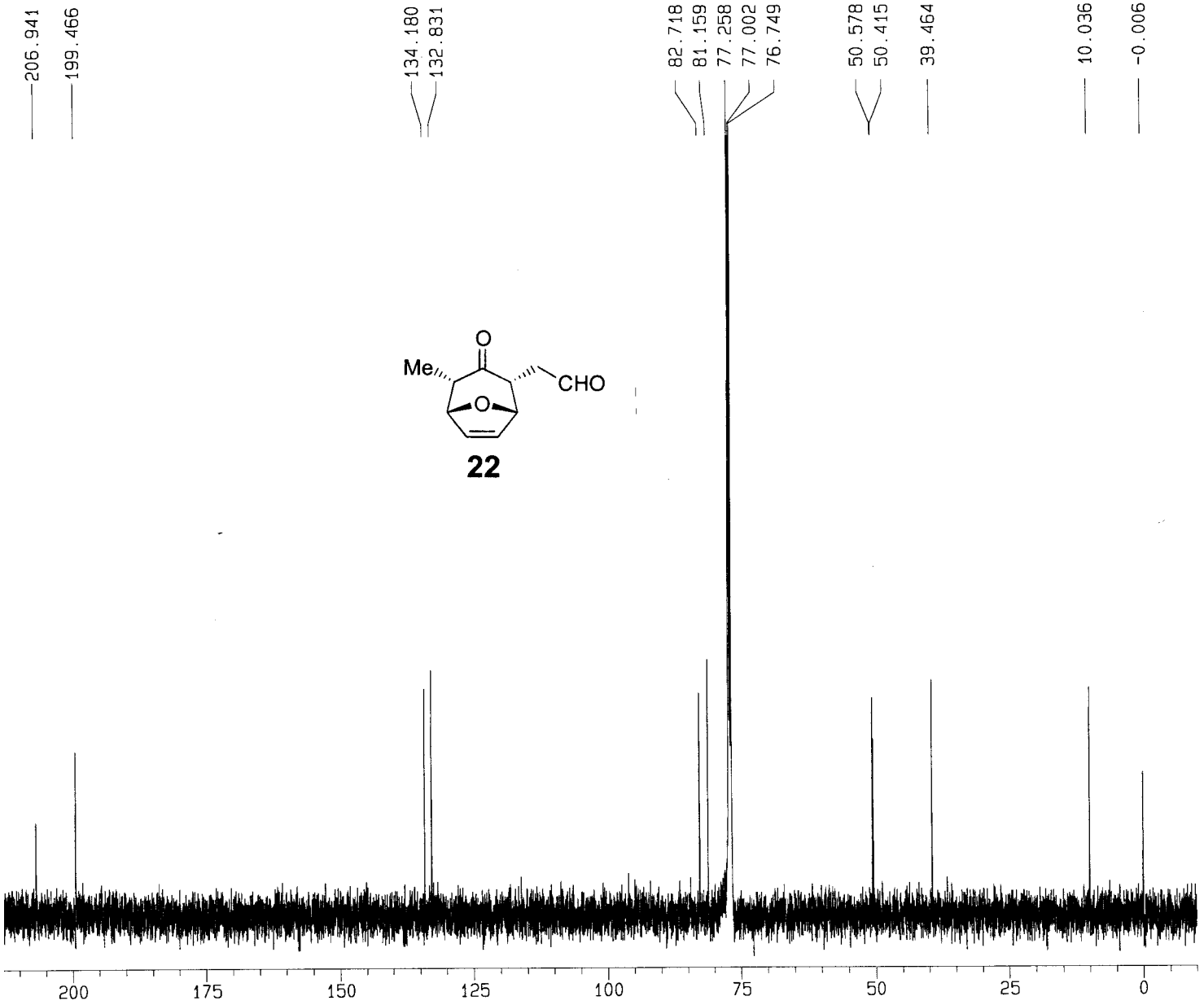
NUC1	1H
P1	13.25 usec
PL1	-3.00 dB
SFO1	500.1330885 MHz

F2 - Processing parameters

SI	32768
SF	500.1300128 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	6.00 cm
F1P	11.000 ppm
F1	5501.43 Hz
F2P	-0.500 ppm
F2	-250.07 Hz
PPMCM	0.57500 ppm/cr
HZCM	287.57477 Hz/cm



22

Current Data Parameters

NAME	XH-IV-8-1-A1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20021212
Time	17.13
INSTRUM	DRX500
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	6807
DS	4
SWH	39681.812 Hz
FIDRES	0.605496 Hz
AQ	0.8258188 sec
RG	16384
DW	12.600 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
d11	0.03000000 sec

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

NUC1	13C
P1	7.90 usec
PL1	3.00 dB
SFO1	125.7713108 MHz

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

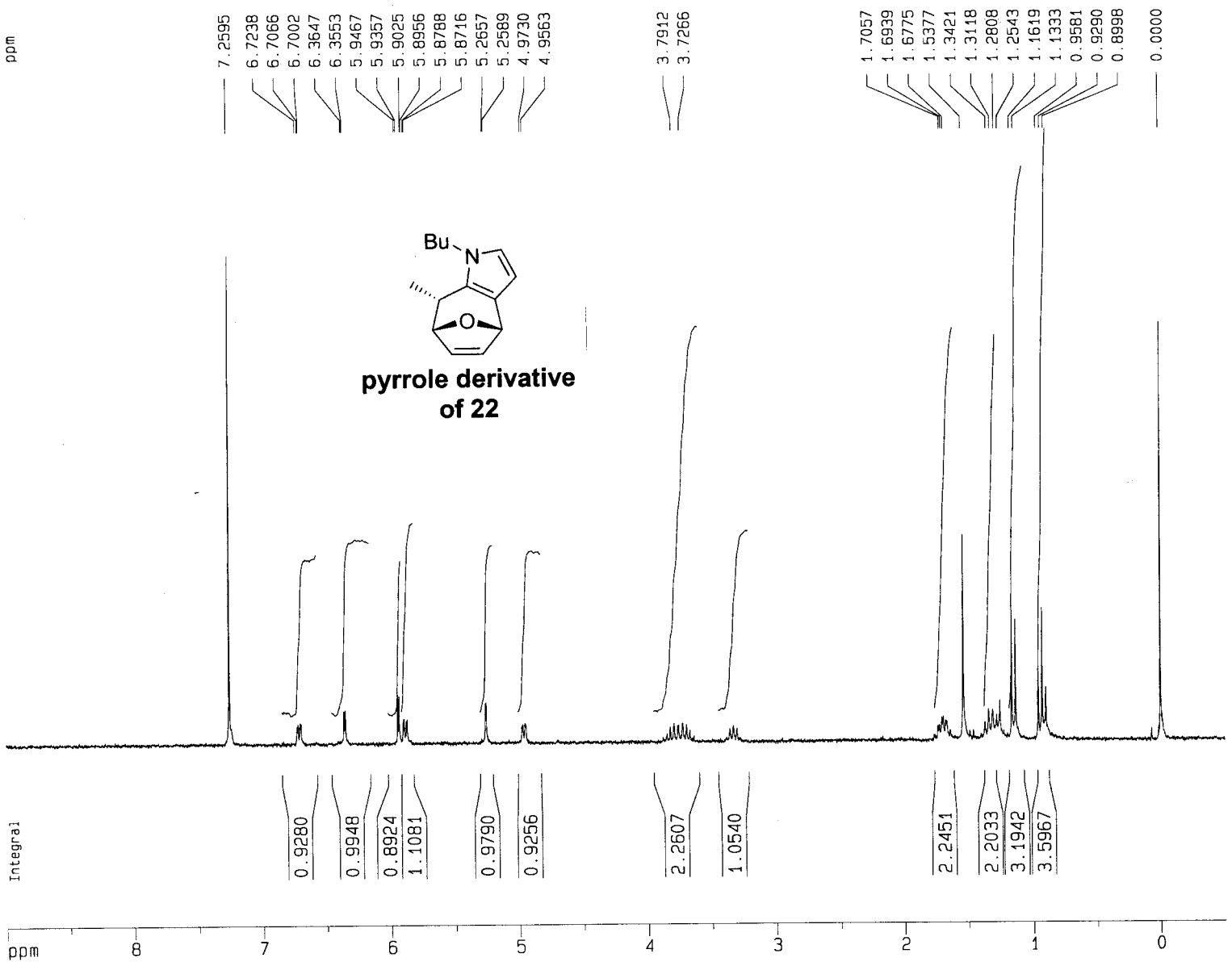
CPDPRG2	waltz16
NUC2	1H
PCPD2	88.00 usec
PL2	0.00 dB
PL12	21.00 dB
SFO2	500.1320005 MHz

F2 - Processing parameters

SI	32768
SF	125.7577910 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	150.00 cm
F1P	220.000 ppm
F1	27666.71 Hz
F2P	-10.000 ppm
F2	-1257.58 Hz
PPMCM	11.50000 ppm/cm
HZCM	1446.21460 Hz/cm



Current Data Parameters

NAME	XH-IV-24-a1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

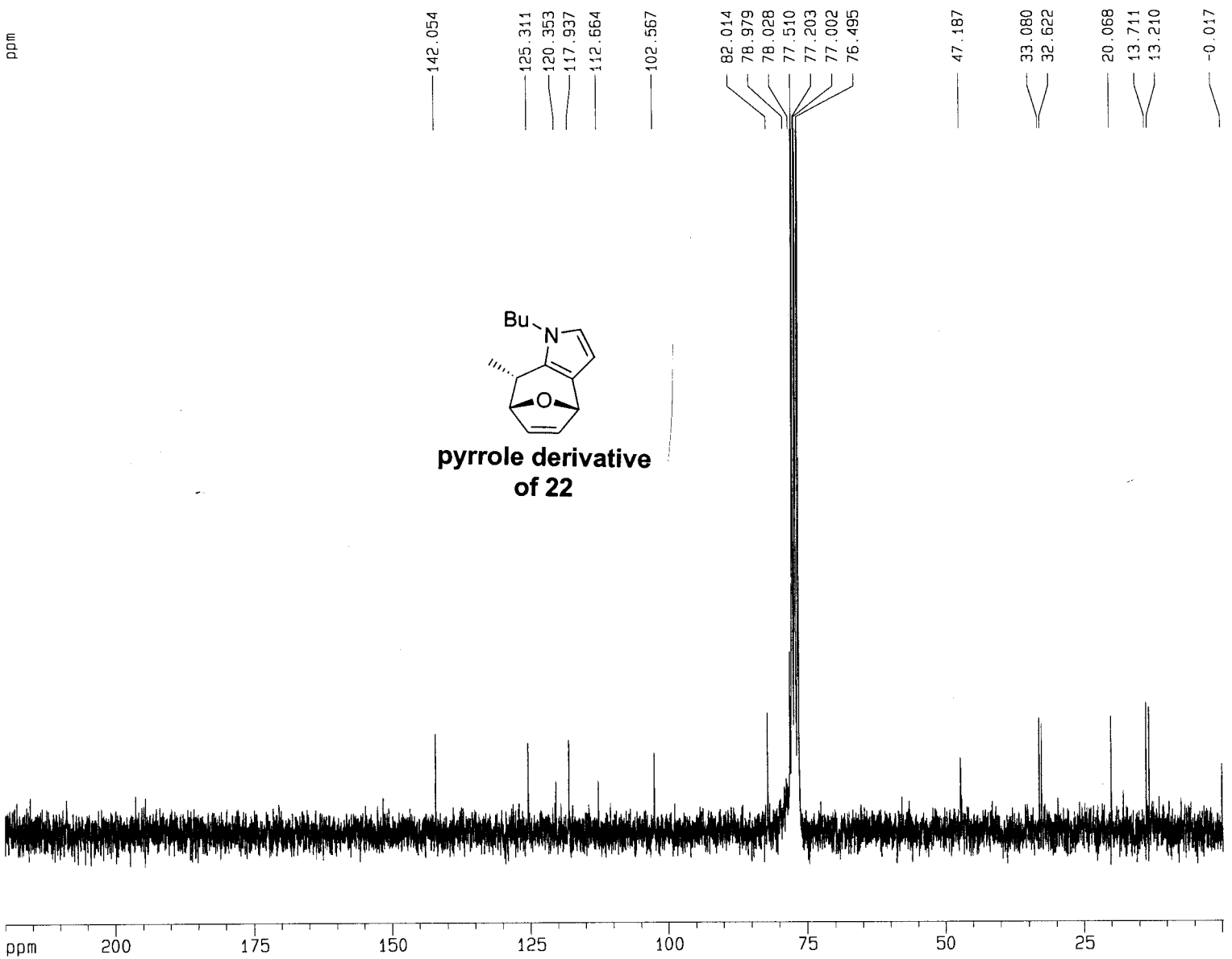
Date_	20021214
Time	20.32
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	256
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300078 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	8.00 cm
F1P	9.000 ppm
F1	2251.17 Hz
F2P	-0.500 ppm
F2	-125.07 Hz
PPMCM	0.47500 ppm
HZCM	118.81175 Hz/



Current Data Parameters

NAME	XH-IV-24-a1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

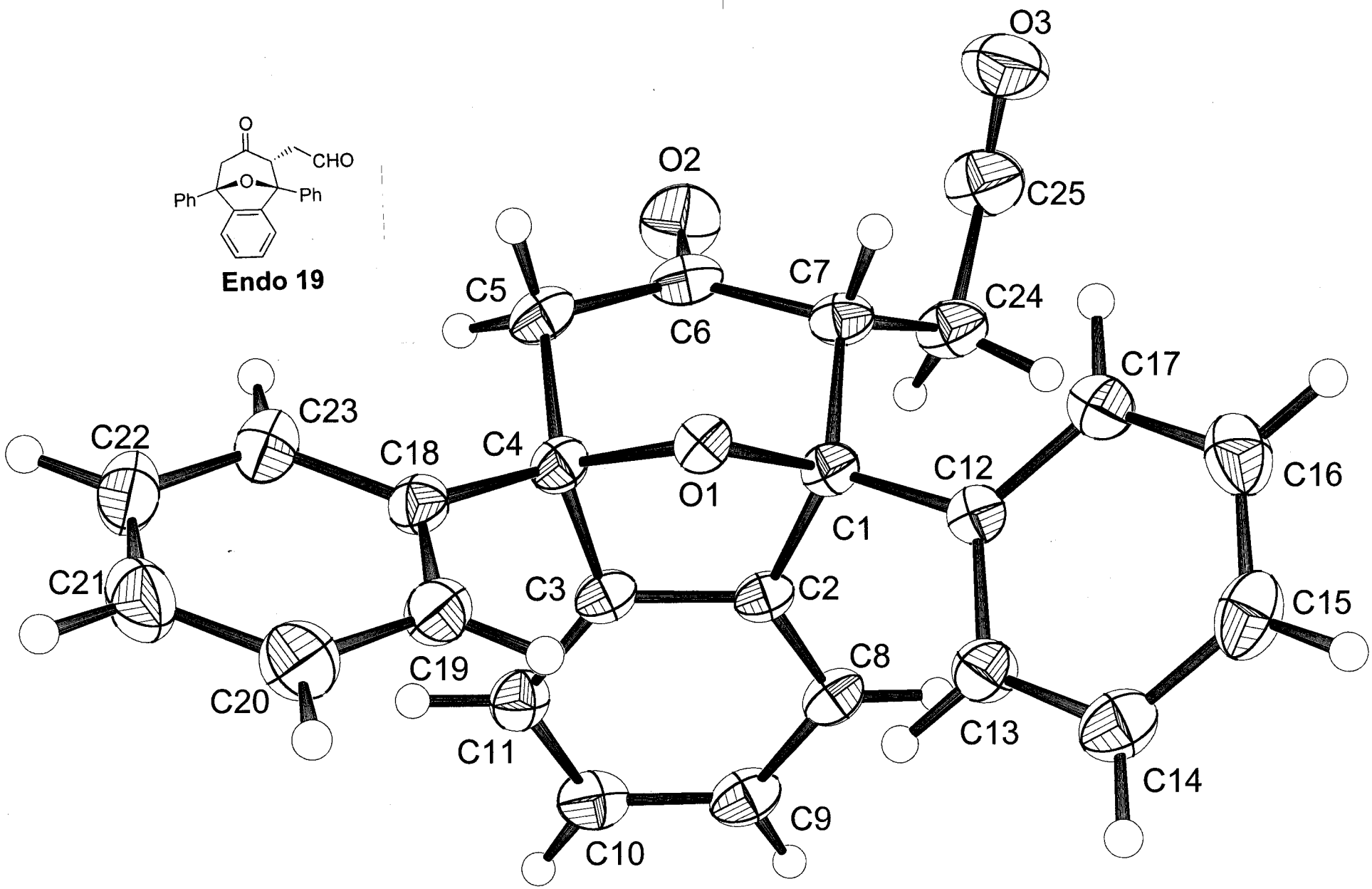
Date_	20021215
Time	8.00
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	28185
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952398 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	100.00 cm
F1P	220.000 ppm
F1	13836.95 Hz
F2P	-0.200 ppm
F2	-12.58 Hz
PPMCM	11.01000 ppm
HZCM	692.47656 Hz/


O
CHO
Ph
O
Ph
Endo 19
O3
O2
C25
C5
C7
C24
C6
C17
C23
C22
C18
C4
O1
C12
C16
C1
C21
C3
C2
C15
C8
C19
C20
C13
C11
C14
C9
C10

Identification code	SG-77-1
Empirical formula	C25 H20 O3
Formula weight	368.41
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2~1~/c
Unit cell dimensions	a = 12.0876(11) A alpha = 90 deg. b = 10.3640(10) A beta = 92.407(2) deg. c = 14.9187(14) A gamma = 90 deg.
Volume	1867.3(3) A^3
Z, Calculated density	4, 1.310 Mg/m^3
Absorption coefficient	0.085 mm^-1
F(000)	776
Crystal size	0.45 x 0.45 x 0.20 mm
Theta range for data collection	1.69 to 27.11 deg.
Limiting indices	-15<=h<=14, -12<=k<=13, -17<=l<=19
Reflections collected / unique	11122 / 4105 [R(int) = 0.0274]
Completeness to theta = 27.11	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9832 and 0.9627
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4105 / 0 / 253
Goodness-of-fit on F^2	1.024
Final R indices [I>2sigma(I)]	R1 = 0.0375, wR2 = 0.0843
R indices (all data)	R1 = 0.0636, wR2 = 0.0943
Largest diff. peak and hole	0.215 and -0.160 e.A^-3

displacement parameters (A 2 x 10 3) for sg771.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	6391(1)	6338(1)	1174(1)	24(1)
O(2)	7439(1)	2820(1)	1872(1)	48(1)
O(3)	9842(1)	3415(1)	995(1)	57(1)
C(1)	7394(1)	6280(1)	1747(1)	24(1)
C(2)	6931(1)	6239(1)	2681(1)	24(1)
C(3)	5842(1)	5812(1)	2591(1)	25(1)
C(4)	5588(1)	5530(1)	1599(1)	25(1)
C(5)	5911(1)	4126(1)	1386(1)	31(1)
C(6)	7120(1)	3877(1)	1623(1)	32(1)
C(7)	7954(1)	4977(1)	1537(1)	27(1)
C(8)	7405(1)	6540(1)	3520(1)	28(1)
C(9)	6748(1)	6448(1)	4259(1)	34(1)
C(10)	5650(1)	6058(1)	4163(1)	35(1)
C(11)	5184(1)	5722(1)	3326(1)	30(1)
C(12)	8049(1)	7486(1)	1540(1)	24(1)
C(13)	7681(1)	8654(1)	1876(1)	31(1)
C(14)	8189(1)	9810(1)	1673(1)	37(1)
C(15)	9084(1)	9805(2)	1125(1)	39(1)
C(16)	9458(1)	8657(2)	781(1)	40(1)
C(17)	8942(1)	7497(1)	984(1)	32(1)
C(18)	4433(1)	5916(1)	1273(1)	27(1)
C(19)	4180(1)	7220(1)	1157(1)	34(1)
C(20)	3118(1)	7600(2)	895(1)	44(1)
C(21)	2290(1)	6690(2)	761(1)	45(1)
C(22)	2528(1)	5398(2)	885(1)	42(1)
C(23)	3598(1)	5010(2)	1129(1)	34(1)
C(24)	8992(1)	4682(1)	2120(1)	35(1)
C(25)	9661(1)	3596(2)	1766(1)	46(1)

Table 3. Bond lengths [A] and angles [deg] for sg771.

O(1)-C(4)	1.4485(15)
O(1)-C(1)	1.4554(15)
O(2)-C(6)	1.2141(17)
O(3)-C(25)	1.194(2)
C(1)-C(12)	1.5179(18)
C(1)-C(2)	1.5251(18)
C(1)-C(7)	1.5479(17)
C(2)-C(3)	1.3891(18)
C(2)-C(8)	1.3902(18)
C(3)-C(11)	1.3844(19)
C(3)-C(4)	1.5264(18)
C(4)-C(18)	1.5130(18)
C(4)-C(5)	1.5432(18)
C(5)-C(6)	1.511(2)
C(6)-C(7)	1.530(2)
C(7)-C(24)	1.5274(18)
C(8)-C(9)	1.388(2)
C(9)-C(10)	1.390(2)
C(10)-C(11)	1.392(2)
C(12)-C(17)	1.3885(19)
C(12)-C(13)	1.3913(19)
C(13)-C(14)	1.386(2)
C(14)-C(15)	1.383(2)
C(15)-C(16)	1.380(2)
C(16)-C(17)	1.394(2)
C(18)-C(23)	1.3894(19)
C(18)-C(19)	1.395(2)
C(19)-C(20)	1.383(2)
C(20)-C(21)	1.383(2)
C(21)-C(22)	1.380(2)
C(22)-C(23)	1.388(2)
C(24)-C(25)	1.496(2)

C(4)-O(1)-C(1)	105.99(9)
O(1)-C(1)-C(12)	105.99(10)
O(1)-C(1)-C(2)	102.06(9)
C(12)-C(1)-C(2)	115.06(10)
O(1)-C(1)-C(7)	106.14(10)
C(12)-C(1)-C(7)	116.18(10)
C(2)-C(1)-C(7)	109.91(10)
C(3)-C(2)-C(8)	120.80(12)
C(3)-C(2)-C(1)	107.63(11)
C(8)-C(2)-C(1)	131.57(12)
C(11)-C(3)-C(2)	121.17(12)
C(11)-C(3)-C(4)	130.98(12)
C(2)-C(3)-C(4)	107.84(11)
O(1)-C(4)-C(18)	109.40(10)
O(1)-C(4)-C(3)	101.69(10)
C(18)-C(4)-C(3)	113.96(11)
O(1)-C(4)-C(5)	105.97(10)
C(18)-C(4)-C(5)	114.90(11)
C(3)-C(4)-C(5)	109.79(11)
C(6)-C(5)-C(4)	111.29(11)
O(2)-C(6)-C(5)	121.13(13)
O(2)-C(6)-C(7)	119.74(13)
C(5)-C(6)-C(7)	119.14(11)
C(24)-C(7)-C(6)	109.35(11)
C(24)-C(7)-C(1)	114.54(11)
C(6)-C(7)-C(1)	109.77(11)
C(9)-C(8)-C(2)	118.14(13)
C(8)-C(9)-C(10)	120.89(13)
C(9)-C(10)-C(11)	120.93(13)
C(3)-C(11)-C(10)	118.01(13)
C(17)-C(12)-C(13)	118.46(13)
C(17)-C(12)-C(1)	123.61(12)
C(13)-C(12)-C(1)	117.77(11)
C(14)-C(13)-C(12)	121.47(13)
C(15)-C(14)-C(13)	119.40(14)

C(16)-C(15)-C(14)	120.04(13)
C(15)-C(16)-C(17)	120.37(14)
C(12)-C(17)-C(16)	120.26(14)
C(23)-C(18)-C(19)	118.79(13)
C(23)-C(18)-C(4)	121.74(12)
C(19)-C(18)-C(4)	119.39(12)
C(20)-C(19)-C(18)	120.44(14)
C(21)-C(20)-C(19)	120.29(15)
C(22)-C(21)-C(20)	119.72(15)
C(21)-C(22)-C(23)	120.24(14)
C(22)-C(23)-C(18)	120.49(14)
C(25)-C(24)-C(7)	113.13(12)
O(3)-C(25)-C(24)	125.61(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for sg771.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	26(1)	22(1)	23(1)	1(1)	-2(1)	0(1)
O(2)	56(1)	23(1)	64(1)	6(1)	-1(1)	9(1)
O(3)	53(1)	66(1)	54(1)	-16(1)	7(1)	15(1)
C(1)	25(1)	22(1)	24(1)	-1(1)	-3(1)	3(1)
C(2)	30(1)	15(1)	25(1)	1(1)	0(1)	1(1)
C(3)	33(1)	17(1)	26(1)	1(1)	-1(1)	0(1)
C(4)	29(1)	22(1)	25(1)	0(1)	0(1)	-2(1)
C(5)	40(1)	21(1)	31(1)	-3(1)	-2(1)	-3(1)
C(6)	44(1)	23(1)	27(1)	-4(1)	0(1)	4(1)
C(7)	32(1)	24(1)	26(1)	-2(1)	-1(1)	6(1)
C(8)	35(1)	22(1)	27(1)	0(1)	-4(1)	-1(1)
C(9)	48(1)	29(1)	23(1)	0(1)	-3(1)	-1(1)
C(10)	49(1)	29(1)	28(1)	3(1)	9(1)	-2(1)
C(11)	35(1)	24(1)	32(1)	3(1)	4(1)	-4(1)
C(12)	25(1)	24(1)	22(1)	2(1)	-2(1)	2(1)
C(13)	33(1)	25(1)	33(1)	1(1)	4(1)	0(1)
C(14)	47(1)	24(1)	41(1)	2(1)	1(1)	-1(1)
C(15)	44(1)	34(1)	38(1)	11(1)	-3(1)	-13(1)
C(16)	36(1)	48(1)	35(1)	7(1)	6(1)	-6(1)
C(17)	32(1)	34(1)	31(1)	-1(1)	3(1)	2(1)
C(18)	28(1)	30(1)	23(1)	-2(1)	0(1)	-2(1)
C(19)	32(1)	31(1)	39(1)	-4(1)	-3(1)	1(1)
C(20)	37(1)	43(1)	52(1)	-4(1)	-5(1)	9(1)
C(21)	28(1)	66(1)	42(1)	-4(1)	-3(1)	5(1)
C(22)	33(1)	57(1)	35(1)	-1(1)	-1(1)	-14(1)
C(23)	35(1)	37(1)	31(1)	3(1)	-2(1)	-7(1)
C(24)	38(1)	30(1)	35(1)	-4(1)	-7(1)	9(1)
C(25)	42(1)	45(1)	49(1)	-7(1)	-11(1)	18(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for sg771.

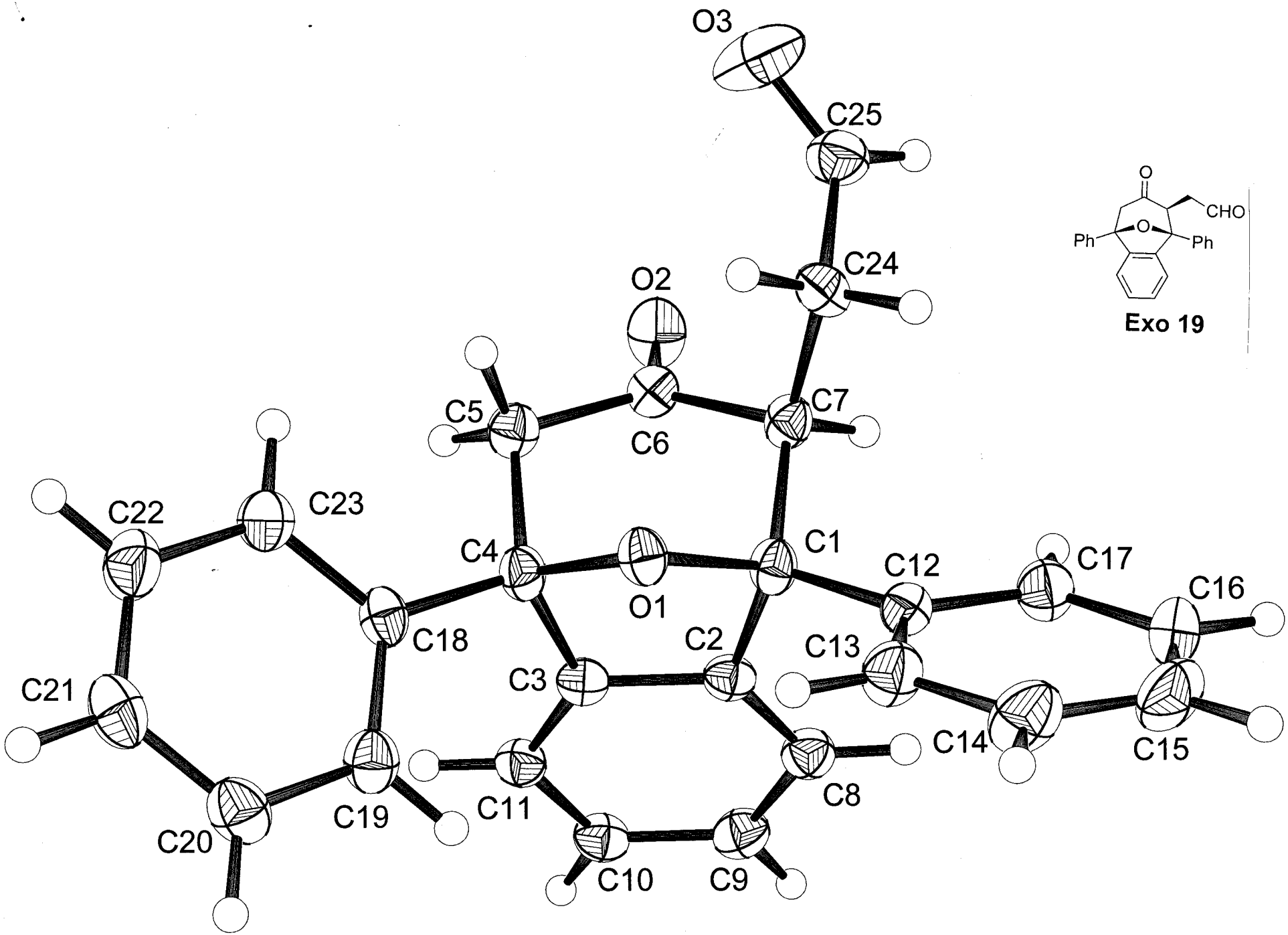
	x	y	z	U(eq)
H(5A)	5450	3527	1728	37
H(5B)	5763	3956	739	37
H(7)	8174	5005	899	33
H(8)	8157	6802	3586	34
H(9)	7054	6654	4838	40
H(10)	5210	6021	4675	42
H(11)	4438	5438	3261	36
H(13)	7067	8660	2254	37
H(14)	7925	10599	1908	45
H(15)	9441	10592	986	47
H(16)	10072	8657	403	47
H(17)	9202	6712	741	38
H(19)	4741	7852	1259	40
H(20)	2957	8489	806	53
H(21)	1560	6953	584	54
H(22)	1958	4773	802	50
H(23)	3759	4117	1198	41
H(24A)	9459	5466	2163	41
H(24B)	8771	4463	2733	41
H(25)	9968	2998	2190	55

Table 6. Torsion angles [deg] for sg771.

C(4)-O(1)-C(1)-C(12)	-157.54(10)
C(4)-O(1)-C(1)-C(2)	-36.76(11)
C(4)-O(1)-C(1)-C(7)	78.36(11)
O(1)-C(1)-C(2)-C(3)	21.06(12)
C(12)-C(1)-C(2)-C(3)	135.32(11)
C(7)-C(1)-C(2)-C(3)	-91.27(12)
O(1)-C(1)-C(2)-C(8)	-158.92(13)
C(12)-C(1)-C(2)-C(8)	-44.66(18)
C(7)-C(1)-C(2)-C(8)	88.76(16)
C(8)-C(2)-C(3)-C(11)	2.49(19)
C(1)-C(2)-C(3)-C(11)	-177.49(12)
C(8)-C(2)-C(3)-C(4)	-178.36(11)
C(1)-C(2)-C(3)-C(4)	1.66(13)
C(1)-O(1)-C(4)-C(18)	158.44(10)
C(1)-O(1)-C(4)-C(3)	37.61(11)
C(1)-O(1)-C(4)-C(5)	-77.15(11)
C(11)-C(3)-C(4)-O(1)	155.19(13)
C(2)-C(3)-C(4)-O(1)	-23.85(12)
C(11)-C(3)-C(4)-C(18)	37.59(19)
C(2)-C(3)-C(4)-C(18)	-141.44(11)
C(11)-C(3)-C(4)-C(5)	-92.91(16)
C(2)-C(3)-C(4)-C(5)	88.05(12)
O(1)-C(4)-C(5)-C(6)	51.50(14)
C(18)-C(4)-C(5)-C(6)	172.42(11)
C(3)-C(4)-C(5)-C(6)	-57.58(14)
C(4)-C(5)-C(6)-O(2)	148.45(14)
C(4)-C(5)-C(6)-C(7)	-32.16(17)
O(2)-C(6)-C(7)-C(24)	-21.61(18)
C(5)-C(6)-C(7)-C(24)	159.00(12)
O(2)-C(6)-C(7)-C(1)	-148.04(13)
C(5)-C(6)-C(7)-C(1)	32.56(16)
O(1)-C(1)-C(7)-C(24)	-176.01(11)
C(12)-C(1)-C(7)-C(24)	66.49(15)
C(2)-C(1)-C(7)-C(24)	-66.36(14)
O(1)-C(1)-C(7)-C(6)	-52.57(13)
C(12)-C(1)-C(7)-C(6)	-170.07(11)
C(2)-C(1)-C(7)-C(6)	57.08(13)
C(3)-C(2)-C(8)-C(9)	-2.28(19)
C(1)-C(2)-C(8)-C(9)	177.69(13)
C(2)-C(8)-C(9)-C(10)	0.3(2)
C(8)-C(9)-C(10)-C(11)	1.5(2)
C(2)-C(3)-C(11)-C(10)	-0.66(19)
C(4)-C(3)-C(11)-C(10)	-179.59(13)
C(9)-C(10)-C(11)-C(3)	-1.3(2)
O(1)-C(1)-C(12)-C(17)	-100.48(14)
C(2)-C(1)-C(12)-C(17)	147.56(12)
C(7)-C(1)-C(12)-C(17)	17.11(18)
O(1)-C(1)-C(12)-C(13)	74.71(14)
C(2)-C(1)-C(12)-C(13)	-37.25(16)
C(7)-C(1)-C(12)-C(13)	-167.70(12)
C(17)-C(12)-C(13)-C(14)	-0.5(2)
C(1)-C(12)-C(13)-C(14)	-175.92(13)
C(12)-C(13)-C(14)-C(15)	-0.1(2)
C(13)-C(14)-C(15)-C(16)	0.5(2)
C(14)-C(15)-C(16)-C(17)	-0.3(2)
C(13)-C(12)-C(17)-C(16)	0.7(2)
C(1)-C(12)-C(17)-C(16)	175.90(12)
C(15)-C(16)-C(17)-C(12)	-0.4(2)
O(1)-C(4)-C(18)-C(23)	143.46(12)
C(3)-C(4)-C(18)-C(23)	-103.48(15)
C(5)-C(4)-C(18)-C(23)	24.45(18)
O(1)-C(4)-C(18)-C(19)	-39.92(16)
C(3)-C(4)-C(18)-C(19)	73.14(16)
C(5)-C(4)-C(18)-C(19)	-158.94(13)
C(23)-C(18)-C(19)-C(20)	-0.7(2)
C(4)-C(18)-C(19)-C(20)	-177.38(13)
C(18)-C(19)-C(20)-C(21)	1.2(2)
C(19)-C(20)-C(21)-C(22)	-0.4(2)

C(20)-C(21)-C(22)-C(23)	-1.0(2)
C(21)-C(22)-C(23)-C(18)	1.6(2)
C(19)-C(18)-C(23)-C(22)	-0.7(2)
C(4)-C(18)-C(23)-C(22)	175.90(13)
C(6)-C(7)-C(24)-C(25)	70.21(16)
C(1)-C(7)-C(24)-C(25)	-166.13(13)
C(7)-C(24)-C(25)-O(3)	40.8(2)

Symmetry transformations used to generate equivalent atoms:


O3
C25
C24
O2
C5
C6
C7
C22
C23
C4
O1
C1
C12
C17
C16
C18
C2
C13
C21
C3
C14
C15
C8
C19
C11
C20
C10
C9

O
CHO
Ph
O
Ph

Exo 19

Table 1. Crystal data and structure refinement for sg77.

Identification code	SG-77-2
Empirical formula	C25 H20 O3
Formula weight	368.41
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2~1~/c
Unit cell dimensions	a = 13.772(2) A alpha = 90 deg. b = 16.105(3) A beta = 95.199(4) deg. c = 8.5772(14) A gamma = 90 deg.
Volume	1894.6(5) A^3
Z, Calculated density	4, 1.292 Mg/m^3
Absorption coefficient	0.084 mm^-1
F(000)	776
Crystal size	0.50 x 0.15 x 0.05 mm
Theta range for data collection	1.48 to 27.19 deg.
Limiting indices	-17<=h<=15, -20<=k<=20, -10<=l<=10
Reflections collected / unique	11734 / 4162 [R(int) = 0.1185]
Completeness to theta = 27.19	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9958 and 0.9592
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4162 / 0 / 254
Goodness-of-fit on F^2	0.921
Final R indices [I>2sigma(I)]	R1 = 0.0546, wR2 = 0.0966
R indices (all data)	R1 = 0.1973, wR2 = 0.1377
Extinction coefficient	0.0084(11)
Largest diff. peak and hole	0.249 and -0.227 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for sg77.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	7909(1)	9688(1)	3119(2)	24(1)
O(2)	5088(1)	9138(1)	3510(2)	38(1)
O(3)	5999(2)	7953(1)	6515(3)	58(1)
C(1)	7353(2)	10273(2)	3981(3)	24(1)
C(2)	6863(2)	10812(2)	2674(3)	23(1)
C(3)	6833(2)	10357(2)	1282(4)	22(1)
C(4)	7296(2)	9512(2)	1661(3)	24(1)
C(5)	6490(2)	8918(2)	2114(3)	27(1)
C(6)	5950(2)	9275(2)	3430(3)	26(1)
C(7)	6545(2)	9764(2)	4716(3)	25(1)
C(8)	6432(2)	11587(2)	2721(4)	28(1)
C(9)	5983(2)	11915(2)	1337(4)	30(1)
C(10)	5955(2)	11463(2)	-51(4)	27(1)
C(11)	6374(2)	10682(2)	-87(4)	27(1)
C(12)	8033(2)	10744(2)	5166(3)	24(1)
C(13)	9036(2)	10754(2)	5035(4)	33(1)
C(14)	9655(2)	11190(2)	6126(4)	41(1)
C(15)	9291(2)	11621(2)	7340(4)	42(1)
C(16)	8292(2)	11624(2)	7456(4)	37(1)
C(17)	7672(2)	11184(2)	6388(4)	31(1)
C(18)	7933(2)	9159(2)	470(3)	24(1)
C(19)	8589(2)	9686(2)	-196(4)	30(1)
C(20)	9202(2)	9386(2)	-1274(4)	34(1)
C(21)	9166(2)	8559(2)	-1697(4)	37(1)
C(22)	8517(2)	8030(2)	-1059(4)	37(1)
C(23)	7898(2)	8332(2)	21(4)	32(1)
C(24)	6962(2)	9142(2)	5977(3)	28(1)
C(25)	6154(2)	8680(2)	6673(4)	34(1)

Table 3. Bond lengths [A] and angles [deg] for sg77.

O(1)-C(1)	1.458(3)
O(1)-C(4)	1.472(3)
O(2)-C(6)	1.215(3)
O(3)-C(25)	1.197(3)
C(1)-C(12)	1.521(4)
C(1)-C(2)	1.526(4)
C(1)-C(7)	1.559(4)
C(2)-C(8)	1.385(4)
C(2)-C(3)	1.398(4)
C(3)-C(11)	1.385(4)
C(3)-C(4)	1.525(4)
C(4)-C(18)	1.517(4)
C(4)-C(5)	1.541(4)
C(5)-C(6)	1.520(4)
C(6)-C(7)	1.531(4)
C(7)-C(24)	1.546(4)
C(8)-C(9)	1.392(4)
C(9)-C(10)	1.392(4)
C(10)-C(11)	1.387(4)
C(12)-C(17)	1.394(4)
C(12)-C(13)	1.396(4)
C(13)-C(14)	1.397(4)
C(14)-C(15)	1.382(4)
C(15)-C(16)	1.388(4)
C(16)-C(17)	1.389(4)
C(18)-C(23)	1.386(4)
C(18)-C(19)	1.399(4)
C(19)-C(20)	1.394(4)
C(20)-C(21)	1.380(4)
C(21)-C(22)	1.384(4)
C(22)-C(23)	1.401(4)
C(24)-C(25)	1.506(4)

C(1)-O(1)-C(4)	105.53(19)
O(1)-C(1)-C(12)	109.9(2)
O(1)-C(1)-C(2)	102.2(2)
C(12)-C(1)-C(2)	114.3(2)
O(1)-C(1)-C(7)	107.0(2)
C(12)-C(1)-C(7)	114.3(2)
C(2)-C(1)-C(7)	108.2(2)
C(8)-C(2)-C(3)	121.2(3)
C(8)-C(2)-C(1)	130.8(3)
C(3)-C(2)-C(1)	107.9(2)
C(11)-C(3)-C(2)	120.1(3)
C(11)-C(3)-C(4)	131.9(3)
C(2)-C(3)-C(4)	107.8(3)
O(1)-C(4)-C(18)	108.8(2)
O(1)-C(4)-C(3)	101.9(2)
C(18)-C(4)-C(3)	116.5(2)
O(1)-C(4)-C(5)	106.0(2)
C(18)-C(4)-C(5)	114.3(2)
C(3)-C(4)-C(5)	108.1(2)
C(6)-C(5)-C(4)	111.4(2)
O(2)-C(6)-C(5)	121.3(3)
O(2)-C(6)-C(7)	121.0(3)
C(5)-C(6)-C(7)	117.6(3)
C(6)-C(7)-C(24)	108.1(2)
C(6)-C(7)-C(1)	109.5(2)
C(24)-C(7)-C(1)	113.0(2)
C(2)-C(8)-C(9)	118.4(3)
C(8)-C(9)-C(10)	120.4(3)
C(11)-C(10)-C(9)	120.9(3)
C(3)-C(11)-C(10)	118.9(3)
C(17)-C(12)-C(13)	118.6(3)
C(17)-C(12)-C(1)	121.0(3)
C(13)-C(12)-C(1)	120.4(3)
C(12)-C(13)-C(14)	120.0(3)
C(15)-C(14)-C(13)	121.1(3)

C(14)-C(15)-C(16)	119.0(3)
C(15)-C(16)-C(17)	120.4(3)
C(16)-C(17)-C(12)	121.0(3)
C(23)-C(18)-C(19)	118.5(3)
C(23)-C(18)-C(4)	122.6(3)
C(19)-C(18)-C(4)	118.8(3)
C(20)-C(19)-C(18)	120.8(3)
C(21)-C(20)-C(19)	120.0(3)
C(20)-C(21)-C(22)	120.0(3)
C(21)-C(22)-C(23)	120.1(3)
C(18)-C(23)-C(22)	120.6(3)
C(25)-C(24)-C(7)	110.9(2)
O(3)-C(25)-C(24)	124.7(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for sg77.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	21(1)	29(1)	23(1)	-3(1)	2(1)	1(1)
O(2)	21(1)	50(2)	43(2)	-11(1)	6(1)	-6(1)
O(3)	52(2)	29(2)	96(2)	6(1)	22(2)	-3(1)
C(1)	20(2)	26(2)	26(2)	-5(2)	5(1)	3(1)
C(2)	20(2)	24(2)	24(2)	2(2)	2(1)	-2(1)
C(3)	16(2)	25(2)	25(2)	1(2)	2(1)	-2(1)
C(4)	24(2)	28(2)	20(2)	-4(1)	0(1)	-2(1)
C(5)	26(2)	27(2)	28(2)	-2(2)	3(2)	0(1)
C(6)	24(2)	25(2)	28(2)	-1(2)	2(2)	0(1)
C(7)	24(2)	24(2)	27(2)	-4(1)	6(2)	3(1)
C(8)	27(2)	28(2)	30(2)	0(2)	4(2)	-1(2)
C(9)	31(2)	25(2)	36(2)	2(2)	4(2)	1(1)
C(10)	25(2)	30(2)	26(2)	5(2)	-1(2)	0(1)
C(11)	27(2)	28(2)	26(2)	0(2)	4(2)	-4(1)
C(12)	25(2)	23(2)	25(2)	-1(2)	3(2)	0(1)
C(13)	29(2)	34(2)	36(2)	-5(2)	6(2)	-4(2)
C(14)	31(2)	40(2)	53(2)	-6(2)	3(2)	-9(2)
C(15)	41(2)	38(2)	46(2)	-7(2)	-8(2)	-14(2)
C(16)	47(2)	35(2)	29(2)	-10(2)	2(2)	-5(2)
C(17)	29(2)	35(2)	31(2)	-3(2)	4(2)	-3(2)
C(18)	22(2)	28(2)	22(2)	-2(2)	2(1)	7(1)
C(19)	26(2)	33(2)	30(2)	-4(2)	3(2)	2(2)
C(20)	26(2)	43(2)	34(2)	-1(2)	8(2)	-1(2)
C(21)	34(2)	46(2)	32(2)	-4(2)	11(2)	8(2)
C(22)	40(2)	33(2)	37(2)	-7(2)	7(2)	6(2)
C(23)	35(2)	29(2)	34(2)	-2(2)	10(2)	4(2)
C(24)	29(2)	33(2)	22(2)	3(2)	3(2)	-2(2)
C(25)	36(2)	33(2)	36(2)	4(2)	10(2)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for sg77.

	x	y	z	U(eq)
H(5A)	6022	8817	1188	32
H(5B)	6784	8379	2450	32
H(7)	6102	10159	5205	30
H(8)	6442	11888	3675	34
H(9)	5693	12450	1339	36
H(10)	5645	11694	-986	33
H(11)	6346	10374	-1035	32
H(13)	9298	10465	4203	39
H(14)	10337	11191	6034	49
H(15)	9717	11911	8083	51
H(16)	8031	11928	8270	44
H(17)	6991	11182	6492	38
H(19)	8616	10256	89	36
H(20)	9645	9750	-1716	40
H(21)	9587	8354	-2426	44
H(22)	8491	7461	-1353	44
H(23)	7451	7966	450	39
H(24A)	7393	8740	5501	33
H(24B)	7358	9445	6814	33
H(25)	5738	8989	7282	41

Table 6. Torsion angles [deg] for sg77.

C(4)-O(1)-C(1)-C(12)	-158.6(2)
C(4)-O(1)-C(1)-C(2)	-36.8(2)
C(4)-O(1)-C(1)-C(7)	76.7(2)
O(1)-C(1)-C(2)-C(8)	-162.5(3)
C(12)-C(1)-C(2)-C(8)	-43.8(4)
C(7)-C(1)-C(2)-C(8)	84.8(3)
O(1)-C(1)-C(2)-C(3)	22.3(3)
C(12)-C(1)-C(2)-C(3)	141.0(2)
C(7)-C(1)-C(2)-C(3)	-90.4(3)
C(8)-C(2)-C(3)-C(11)	0.6(4)
C(1)-C(2)-C(3)-C(11)	176.3(2)
C(8)-C(2)-C(3)-C(4)	-175.4(2)
C(1)-C(2)-C(3)-C(4)	0.4(3)
C(1)-O(1)-C(4)-C(18)	160.7(2)
C(1)-O(1)-C(4)-C(3)	37.0(2)
C(1)-O(1)-C(4)-C(5)	-76.0(2)
C(11)-C(3)-C(4)-O(1)	162.1(3)
C(2)-C(3)-C(4)-O(1)	-22.7(3)
C(11)-C(3)-C(4)-C(18)	43.9(4)
C(2)-C(3)-C(4)-C(18)	-140.9(3)
C(11)-C(3)-C(4)-C(5)	-86.5(4)
C(2)-C(3)-C(4)-C(5)	88.8(3)
O(1)-C(4)-C(5)-C(6)	54.3(3)
C(18)-C(4)-C(5)-C(6)	174.1(2)
C(3)-C(4)-C(5)-C(6)	-54.4(3)
C(4)-C(5)-C(6)-O(2)	146.9(3)
C(4)-C(5)-C(6)-C(7)	-37.2(3)
O(2)-C(6)-C(7)-C(24)	88.9(3)
C(5)-C(6)-C(7)-C(24)	-86.9(3)
O(2)-C(6)-C(7)-C(1)	-147.6(3)
C(5)-C(6)-C(7)-C(1)	36.6(3)
O(1)-C(1)-C(7)-C(6)	-54.5(3)
C(12)-C(1)-C(7)-C(6)	-176.5(2)
C(2)-C(1)-C(7)-C(6)	55.0(3)
O(1)-C(1)-C(7)-C(24)	66.1(3)
C(12)-C(1)-C(7)-C(24)	-55.8(3)
C(2)-C(1)-C(7)-C(24)	175.6(2)
C(3)-C(2)-C(8)-C(9)	-1.4(4)
C(1)-C(2)-C(8)-C(9)	-176.1(3)
C(2)-C(8)-C(9)-C(10)	1.2(4)
C(8)-C(9)-C(10)-C(11)	-0.1(4)
C(2)-C(3)-C(11)-C(10)	0.5(4)
C(4)-C(3)-C(11)-C(10)	175.3(3)
C(9)-C(10)-C(11)-C(3)	-0.7(4)
O(1)-C(1)-C(12)-C(17)	-163.9(2)
C(2)-C(1)-C(12)-C(17)	81.8(3)
C(7)-C(1)-C(12)-C(17)	-43.6(4)
O(1)-C(1)-C(12)-C(13)	17.0(4)
C(2)-C(1)-C(12)-C(13)	-97.3(3)
C(7)-C(1)-C(12)-C(13)	137.3(3)
C(17)-C(12)-C(13)-C(14)	0.8(4)
C(1)-C(12)-C(13)-C(14)	179.9(3)
C(12)-C(13)-C(14)-C(15)	-0.5(5)
C(13)-C(14)-C(15)-C(16)	-0.6(5)
C(14)-C(15)-C(16)-C(17)	1.4(5)
C(15)-C(16)-C(17)-C(12)	-1.1(5)
C(13)-C(12)-C(17)-C(16)	0.0(4)
C(1)-C(12)-C(17)-C(16)	-179.1(3)
O(1)-C(4)-C(18)-C(23)	108.3(3)
C(3)-C(4)-C(18)-C(23)	-137.3(3)
C(5)-C(4)-C(18)-C(23)	-10.0(4)
O(1)-C(4)-C(18)-C(19)	-71.2(3)
C(3)-C(4)-C(18)-C(19)	43.2(4)
C(5)-C(4)-C(18)-C(19)	170.5(3)
C(23)-C(18)-C(19)-C(20)	-0.7(4)
C(4)-C(18)-C(19)-C(20)	178.8(3)
C(18)-C(19)-C(20)-C(21)	0.1(5)
C(19)-C(20)-C(21)-C(22)	0.4(5)

C(20)-C(21)-C(22)-C(23)	-0.2(5)
C(19)-C(18)-C(23)-C(22)	0.9(4)
C(4)-C(18)-C(23)-C(22)	-178.6(3)
C(21)-C(22)-C(23)-C(18)	-0.4(5)
C(6)-C(7)-C(24)-C(25)	-59.8(3)
C(1)-C(7)-C(24)-C(25)	178.7(2)
C(7)-C(24)-C(25)-O(3)	113.1(4)

Symmetry transformations used to generate equivalent atoms: