

**SUPPORTING INFORMATION**  
**FOR**

**Total Syntheses of Yingzhaosu A and of its C(14)-Epimer**  
**Including the First Evaluation of their Antimalarial and**  
**Cytotoxic Activities**

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## General

Compounds **8** and **15** (as an inseparable mixture) were synthesized and purified as described earlier.<sup>1</sup>

<sup>1</sup>H, <sup>13</sup>C and <sup>31</sup>P NMR as well as DEPT were recorded at 400MHz or 250 MHz NMR. COSY, NOE-difference and HMQC NMR spectra were recorded at 100MHz. Residual CHCl<sub>3</sub> (7.27 ppm for <sup>1</sup>H NMR) and CDCl<sub>3</sub> (77.00 ppm for <sup>13</sup>C NMR) peaks were used as internal standards. Infrared spectra were measured on a FT-IR instrument as thin films or in KBr pellets. Specific rotations, HRMS and X-ray analysis were performed using commercial instruments and software packages. HRMS were performed using the Desorption Chemical Ionization (DCI) Technique. Melting points are uncorrected. TLC analysis was performed on precoated silica gel plates (UV detection) followed by development with either alkaline KMnO<sub>4</sub> or a solution of N,N-dimethyl-*p*-phenylenediamine dihydrochloride in water/methanol/acetic acid (stain specific for peroxides), stabilized by 2–3 mL of conc. HCl per 150 mL of the stain solution.\

Flash chromatography was performed on Silica gel 60 (230-400 mesh). Medium pressure liquid chromatography (MPLC) was performed on glass columns (*l* = 460 mm, *d* = 26 or 43 mm, filled with silica gel (15–25 μm particle size). For analytical and semi-preparative HPLC a commercial HPLC instrument with an UV detector and direct-phase columns was used. Retention times (*R*<sub>t</sub>) are given for analytical HPLC. Semi-preparative direct-phase HPLC was performed on a 250 x 10mm column (Si-60 (10 μm); flow rate: 2.5 - 3 mL/min). Analytical direct-phase HPLC was performed on a, 250 x 4 mm column (Si-60 (5 μm); flow rate: 1 mL/min).

All reagents were used as received from the supplier with the following exceptions: Titanium tetrachloride, trimethylsilyl chloride and trimethyl boroxine were distilled immediately prior to use. Pyridine and 2,6-lutidine was distilled over KOH pellets. The purity of MCPBA was determined by iodometric titration (purity noted). Commercial CH<sub>2</sub>Cl<sub>2</sub> was distilled over P<sub>2</sub>O<sub>5</sub>. THF was distilled from

sodium/benzophenone. All reactions specified as dry were carried out in oven dried glassware under an atmosphere of dry argon.

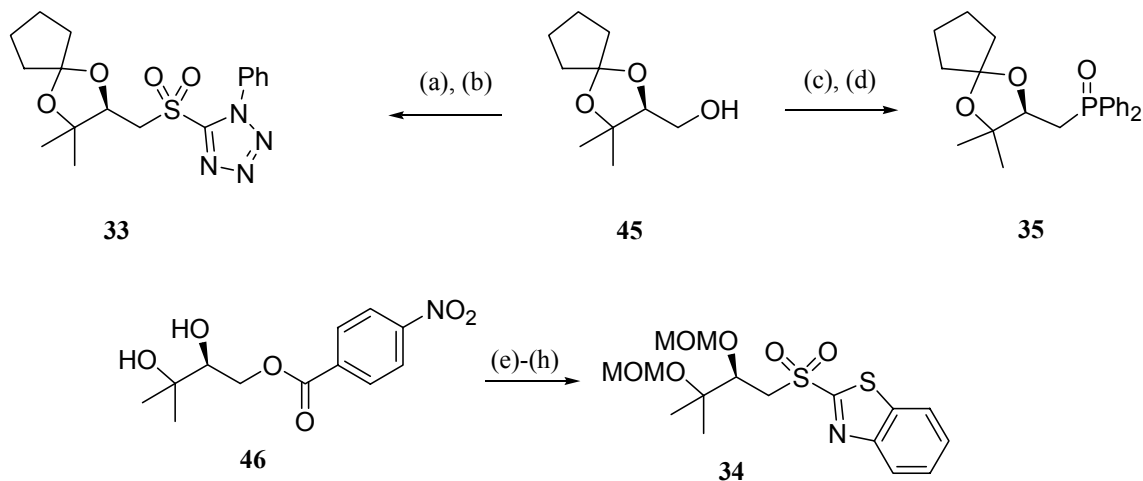
Unless otherwise stated the purity of all the title compounds was estimated to be  $\geq 95\%$  by  $^1\text{H}$  NMR and analytical HPLC determination. The ratios of diastereomers were determined by integration of the relevant separated signals in  $^1\text{H}$  NMR spectra.

The purity of all the *in vitro* tested compounds was judged to be  $\geq 95\%$  by 400 MHz  $^1\text{H}$  NMR. The purity of the *in vivo* tested compounds was  $\geq 97\%$  according to the same criteria.

## Synthesis of Julia-Kociensky sulfones **33-34** and Horner-Wittig reagent **35**

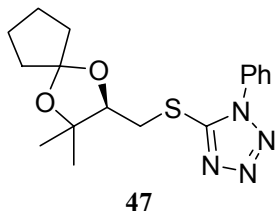
Compounds **33** and **35** were synthesized from the readily available<sup>3</sup> alcohol **45** (see Scheme I). Compound **34** was synthesized in four steps from the readily available<sup>3</sup> alcohol **46** (Scheme I).

Scheme S1



(a) 1.1 equiv.  $\text{Ph}_3\text{P}$ , 1.1 equiv. DEAD, 1.3 equiv. 1-phenyl-1*H*-tetrazole-5-thiol, THF, 98% yield. (b) cat.  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$ , 4 equiv.  $\text{H}_2\text{O}_2$ ,  $\text{H}_2\text{O}/\text{EtOH}$ , 95% yield. (c) 2 equiv.  $\text{Ph}_3\text{P}$ , 4.5 equiv. imidazole, 1.9 equiv.  $\text{I}_2$ , 1,4-dioxane, 65-70°C, 94% yield. (d) 1.1 equiv.  $\text{Ph}_2\text{PH}$ , 1.1 equiv. *n*-BuLi, THF 0 °C. then exposed to atmospheric air, 49% yield. (e) 4 equiv. MOMBr, 3 equiv. 2,6-di-*tert*-butyl-4-methyl-pyridine, 94% yield. (f) 2 equiv.  $\text{K}_2\text{CO}_3$ ,  $\text{H}_2\text{O}/\text{EtOH}$ , 87%. (g) 1.1 equiv.  $\text{Ph}_3\text{P}$ , 1.1 equiv. DEAD, 1.3 equiv. benzothiazole-2-thiol, THF, 90% yield. (h) cat.  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$ , 1 equiv.  $\text{NaHCO}_3$ , 4 equiv.  $\text{H}_2\text{O}_2$ ,  $\text{H}_2\text{O}/\text{EtOH}$ , 86% yield.

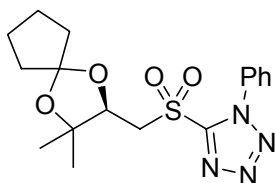
### 5-[(2*S*)-3,3-Dimethyl-1,4-dioxaspiro[4.4]non-2-ylsulfanyl]-1-phenyl-1*H*-tetraazole (**47**):



To a solution of alcohol **45** (233 mg; 1.25 mmol) in dry THF (11 mL) were added triphenyl phosphine (1.3 equiv.; 426 mg; 1.62 mmol), 1-phenyl-1*H*-tetrazole-5-thiol (1.3 equiv.; 289 mg; 1.62 mmol) and DEAD (1.3 equiv.; 0.26 mL; 1.62 mmol). The reaction mixture was stirred for 45 min., poured into 35 mL saturated sodium bicarbonate and extracted with dichloromethane (4×30 mL). The combined organic extracts were

washed with brine, dried ( $\text{Na}_2\text{SO}_4$ ) and the solvent removed under reduced pressure. Flash chromatography (15% ethyl acetate/hexane) afforded 426 mg (1.22 mmol, 98% yield) of the desired sulfide **47** as a colourless oil;  $R_f$  = 0.21 (15% ethyl acetate/hexane).

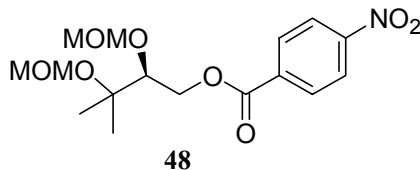
**5-[(2*S*)-3,3-Dimethyl-1,4-dioxaspiro[4.4]non-2-ylsulfonyl]-1-phenyl-1*H*-tetrazole (**33**):**



**33**

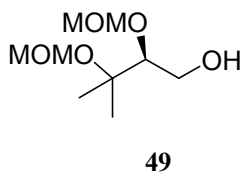
Sulfide **47** (106 mg; 0.31 mmol) was dissolved in ethanol (2.2 mL) and a catalytic amount (0.10 equiv.; 37 mg; 0.03 mmol) ammonium molybdate tetrahydrate was added. The solution was cooled to 0°C and a 30% hydrogenperoxide solution (1.6 equiv.; 0.55 mL) was added and the resulting reaction mixture was stirred for 22 h. at room temperature. The reaction mixture was poured into brine (30 mL) and extracted with 4×20 mL dichloromethane. The combined extracts were dried ( $\text{Na}_2\text{SO}_4$ ) and the solvent removed under reduced pressure. Flash chromatography (17% ethyl acetate/hexane) afforded **33** (111 mg; 95% yield) as a white solid.  $R_f$  = 0.45 (30% ethyl acetate/hexane);  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  1.09 (s, 3H), 1.32 (s, 3H), 1.46-1.80 (comp. m, 8H), 3.63 (dd,  $J$  = 2.6, 14.8 Hz, 1H), 3.69 (dd,  $J$  = 9.8, 14.8 Hz, 1H), 4.13 (dd,  $J$  = 2.6, 9.8 Hz, 1H), 7.63 (m, 5H, Ar);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  22.3 ( $\text{CH}_3$ ), 23.0 ( $\text{CH}_2$ ), 23.1 ( $\text{CH}_2$ ), 25.0 ( $\text{CH}_3$ ), 37.6 ( $\text{CH}_2$ ), 37.8 ( $\text{CH}_2$ ), 56.4 ( $\text{CH}_2$ ), 79.7 ( $\text{CH-O-}$ ), 118.4 (C), 125.5 (CH, Ar), 127.3 (CH, Ar), 129.2 (CH, Ar), 130.7 (CH, Ar), 132.8 (CH, Ar), 135.4 (C, Ar), 153.5 (C=N, tetrazole).

**(2S)-2,3-Di(methoxymethoxy)-3-methylbutyl 4-nitrobenzoate (48):**



Diol **46** (578 mg; 2.14 mmol) and 4-methyl-2,6-di-*t*-butyl-pyridine (3 equiv.; 1.37 g; 6.44 mmol) was dissolved in freshly dried chloroform (22 mL). Methoxymethylbromide (4 equiv.; 0.7 mL; 8.6 mmol) was added to the solution drop wise over 5 min. The resulting reaction mixture was stirred for 4 h. Poured into water (50 mL) at 4°C and extracted with 4×20 mL dichloromethane. The combined extracts were washed with saturated sodium bicarbonate (45 mL) and dried (Na<sub>2</sub>SO<sub>4</sub>). The solvent was removed under reduced pressure. Flash chromatography (gradient: 15% to 35% ethyl acetate/hexane) afforded the title compound **48** (728 mg; 94 % yield) as a brownish oil: *R*<sub>f</sub> = 0.66 (50% ethyl acetate/hexane); <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>): δ 1.33 (s, 3H), 1.38 (s, 3H), 3.35 (s, 3H), 3.38 (s, 3H), 3.80 (dd, *J* = 2.5, 7.3 Hz, 1H), 4.44 (dd, *J* = 7.3, 11.8 Hz, 1H), 4.73 (d, *J* = 6.7 Hz, 1H), 4.75 (d, *J* = 7.3 Hz, 1H), 4.78 (dd, *J* = 2.5, 11.8 Hz, 1H), 4.82 (d, *J* = 7.3 Hz, 1H), 4.84 (d, *J* = 6.7 Hz, 1H), 8.28 (m, 4H); <sup>13</sup>C NMR (62.5 MHz, CDCl<sub>3</sub>): δ 22.1 (CH<sub>3</sub>), 24.0 (CH<sub>3</sub>), 55.4 (CH<sub>3</sub>), 56.1 (CH<sub>3</sub>), 66.5 (CH<sub>2</sub>), 82.0 (CH), 91.2 (CH<sub>2</sub>), 97.7 (CH<sub>2</sub>), 123.6 (2×CH, Ar), 130.7 (2×CH, Ar), 164.7 (C=O, ester).

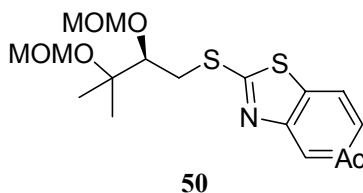
**(2S)-2,3-Di(methoxymethoxy)-3-methylbutan-1-ol (49):**



Nitrobenzoate **48** (716 mg; 2 mmol) was dissolved in ethanol (16.5 mL) and potassium carbonate (2 equiv.; 550 mg; 4 mmol) was added. The solution was stirred for 6 h. Potassiumhydrogensulfate (2 equiv.; 542 mg; 4mmol) was added followed by 100 mL dichloromethane and drying material (Na<sub>2</sub>SO<sub>4</sub>). The resulting heterogeneous mixture was stirred for 12 h. and filtered. The solvent was removed under reduced pressure.

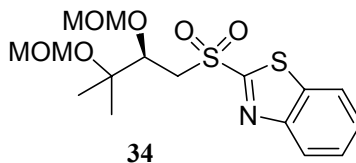
Flash chromatography (gradient: 15% to 40% ethyl acetate/hexane) afforded hygroscopic alcohol **49** as an yellowish oil (317 mg; 87 % yield):  $R_f$  = 0.35 (30% ethyl acetate/hexane), which was used without further purification but after careful drying in a desiccator over phosphorouspentoxide under vacuum.

**2-[(2*R*)-2,3-Di(methoxymethoxy)-3-methylbutylsulfanyl]-1,3-benzothiazole (**50**):**



To a solution of alcohol **49** (361 mg; 1.74 mmol) in dry THF (18 mL) was added triphenyl phosphine (1.3 equiv.; 593 mg; 2.2 mmol), benzothiazolemercaptane (1.5 equiv.; 435 mg; 2.6 mmol) and DEAD (1.3 equiv.; 0.4 mL; 2.2 mmol). The reaction mixture was stirred for 2 h., poured into saturated sodium bicarbonate (100 mL) and extracted with dichloromethane (5×30 mL). The combined extracts were dried ( $\text{Na}_2\text{SO}_4$ ) and the solvent removed under reduced pressure. Two flash chromatographies (gradient: 6% to 20% ethyl acetate/hexane) afforded 561 mg (1.56 mmol, 90% yield) of benzothiazole sulfide **50** as an oil:  $R_f$  = 0.53 (30% ethyl acetate/hexane);  $^1\text{H}$  NMR (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.36 (s, 3H), 1.38 (s, 3H), 3.38 (dd,  $J$  = 7.9, 13.3 Hz, 1H), 3.41 (s, 3H), 3.44 (s, 3H), 3.80 (dd,  $J$  = 3.3, 7.9 Hz, 1H), 3.96 (dd,  $J$  = 3.3, 13.3 Hz, 1H), 4.76 (d,  $J$  = 6.7 Hz, 1H), 4.79 (d,  $J$  = 7.2 Hz, 1H), 4.85 (d,  $J$  = 7.3 Hz, 1H), 4.87 (d,  $J$  = 6.7 Hz, 1H), 7.29 (m, 1H), 7.41 (m, 1H), 7.75 (m, 1H), 7.83 (m, 1H).

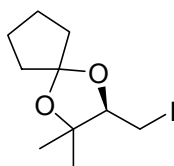
**2-[(2*R*)-2,3-Di(methoxymethoxy)-3-methylbutylsulfonyl]-1,3-benzothiazole (**34**):**





Sulfide **50** (551 mg; 1.54 mmol) was dissolved in ethanol (14 mL) and a catalytic amount (0.10 equiv.; 190 mg; 0.15 mmol) ammonium molybdate tetrahydrate was added followed by sodium bicarbonate (80 mg) and a 30% hydrogen peroxide solution (1.6 equiv.; 3.4 mL). The solution became yellow then orange. The reaction mixture was stirred for 24 h. To the resulting yellowish solution was added a 25% solution of ethyl acetate in hexane (100 mL). The solution was washed with saturated sodium bicarbonate. The water phase was extracted with dichloromethane (4×25 mL). The combined organic solutions were dried (MgSO<sub>4</sub>) and the solvent removed under reduced pressure. Flash chromatography (30% ethyl acetate/hexane) afforded the desired sulfone **34** (518 mg; 86% yield) as a clear oil. *R*<sub>f</sub> = 0.28 (30% ethyl acetate/hexane); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 1.23 (s, 3H), 1.33 (s, 3H), 3.24 (s, 3H), 3.62 (s, 3H), 3.38 (dd, *J* = 8.0, 15.5 Hz, 1H), 4.13 (dd, *J* = 2.3, 15.5 Hz, 1H), 4.17 (dd, *J* = 2.3, 8.0 Hz, 1H), 4.60 (d, *J* = 7.4 Hz, 1H), 4.64 (d, *J* = 7.4 Hz, 1H), 4.72 (d, *J* = 6.6 Hz, 1H), 4.95 (d, *J* = 6.6 Hz, 1H), 7.59 (m, 1H), 7.64 (m, 1H), 8.02 (m, 1H), 8.22 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 21.2 (CH<sub>3</sub>), 23.9 (CH<sub>3</sub>), 55.3 (CH<sub>3</sub>), 56.6 (CH<sub>3</sub>), 57.5 (CH<sub>2</sub>), 77.7 (C), 78.3 (CH), 91.1 (CH<sub>2</sub>), 98.1 (CH<sub>2</sub>), 122.3 (CH), 125.4 (CH), 127.6 (CH), 128.0 (CH), 136.8 (C), 152.7 (C), 166.4 (C=N, thiazole).

**(2*R*)-3,3-(Dimethyl-1,4-dioxaspiro[4.4]non-2-yl)-methyl iodide (51):**

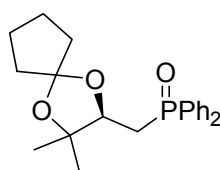


**51**

Alcohol **45** (242 mg; 1.3 mmol), triphenylphosphine (2.1 equiv.; 716 mg; 2.7 mmol) and imidazole (4.6 equiv.; 411 mg; 5.98 mmol) were dissolved in dry 1,4-dioxane (5 mL). Upon addition of iodine (2.0 equiv.; 660 mg; 2.6 mmol) a weakly exothermic reaction was observed. The mixture was stirred at room temperature overnight and then heated to 70°C for 1.5 h. The reaction mixture was poured into cold water (50 mL) and extracted with ether (2×50 mL). The combined extracts were washed with brine (50 mL) and dried (MgSO<sub>4</sub>).

The solvent was removed under reduced pressure. Two flash chromatographies (10% ether/hexane) afforded iodide **51** (362 mg; 94%) as an oil.  $R_f$  = 0.41 (10% ether/hexane);  $^1\text{H}$  NMR (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.12 (s, 3H), 1.32 (s, 3H), 1.56-1.87 (comp m, 8H) 3.07 (dd,  $J$  = 5.8, 10.3 Hz, 1H), 3.19 (dd,  $J$  = 7.9, 10.3 Hz, 1H), 3.90 (dd,  $J$  = 5.8, 7.9 Hz, 1H);  $^{13}\text{C}$  NMR (62.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.1 ( $\text{CH}_2$ ), 21.7 ( $\text{CH}_3$ ), 23.3 ( $\text{CH}_2$ ), 23.7 ( $\text{CH}_2$ ), 26.7 ( $\text{CH}_3$ ), 38.1 ( $\text{CH}_2$ ), 38.2 ( $\text{CH}_2$ ), 79.8 (C), 83.7 (CH), 116.7 (C).

**(3S)-3-(Diphenyl-phosphinoylmethyl)-2,2-dimethyl-1,4-dioxaspiro[4.4]nonane (35):**

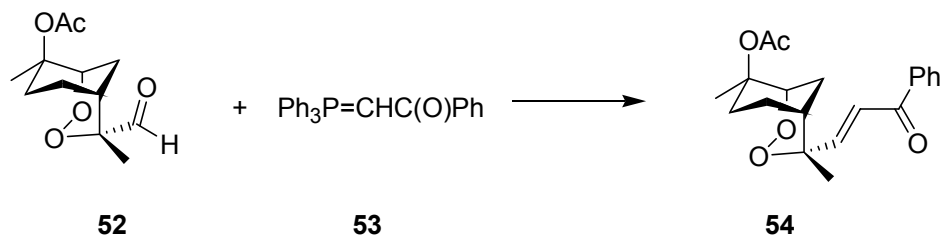


**35**

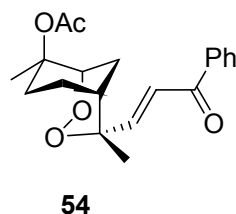
Diphenylphosphine (1.1 equiv.; 251 mg; 1.348 mmol) was dissolved in dry THF (3 mL) and the solution cooled to 0°C. A hexane solution of *n*-butyllithium (1.1 equiv.; *ca.* 1.4 M; 0.96 mL; 1.348 mmol) was added drop wise affording a deep orange solution. The reaction mixture was stirred for 30 min. at 0°C and then a solution of iodide **51** in dry THF (1.5 mL + 2×0.5 mL) was added via canula. The colour changed to pale yellow. The reaction mixture was stirred for 30 min at 0°C and then for 2.5 h. at room temperature. The reaction mixture was poured into water (50 mL) and extracted with chloroform (100 mL). The organic phase was washed with water and the solvent removed under reduced pressure. Under the entire workup no special precautions were taken to exclude atmospheric oxygen. Four flash chromatographies (25% ethyl acetate/hexane) afforded Phosphinate **35** (170 mg; 49% yield) as a colourless solid.  $R_f$  = 0.25 (25% ethyl acetate/hexane);  $^1\text{H}$  NMR (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.14 (s, 3H), 1.24 (s, 3H), 1.29-1.80 (comp m, 8H) 2.30 (ddd,  $J$  = 3.2, 15.0, 16.8 Hz, 1H), 2.58 (ddd,  $J$  = 7.4, 9.4, 16.8 Hz, 1H), 3.94 (dd,  $J$  = 3.2, 9.4 Hz, 1H).  $^{31}\text{P}$  NMR (101.25 MHz,  $\text{CDCl}_3$ ):  $\delta$  30.5.

## Model studies; Wittig olefination of aldehyde **52**

Scheme S2



(1*R*,4*R*,5*R*,8*R*)-8-Acetoxy-4,8-dimethyl-4-(*E*)-(3-oxo-3-phenyl-1-propenyl)-2,3-dioxabicyclo[3.3.1]nonane (**54**).



A mixture of (1*R*,4*R*,5*R*,8*R*)-8-acetoxy-4,8-dimethyl-4-formyl-2,3-dioxabicyclo[3.3.1]nonane (**52**)<sup>4,5</sup> (16.0 mg, 0.066 mmol) and benzoylmethylene)triphenylphosphorane (**53**) (150 mg, 0.396 mmol) in dry  $\text{CH}_2\text{Cl}_2$  (1.5 ml) was stirred at rt under argon for 30 days. At that time TLC analysis revealed approximately 90% consumption of aldehyde **52**. The mixture was directly subjected to flash chromatography (elution with hexane-benzene-ethyl acetate 4:2:1) to give the title compound **54** (10.8 mg, 48%) as a yellowish oil,  $R_f$  = 0.43 (in ethyl acetate/benzene/hexane 3:6:11);  $^1\text{H}$  NMR (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.32 (s, Me(11), 3H), 1.72 (s, Me(10), 3H), 1.78-1.90 (m, 4H), 2.01-2.15 (m, 1H), 2.04 (s, MeC(O), 3H), 2.18-2.42 (m, 2H), 4.44 (br s, H(1), 1H), 7.06 (d,  $J$  = 15.6 Hz, =C(14)HC(O), 1H), 7.37 (d,  $J$  = 15.6 Hz, =C(13)H, 1H), 7.47-7.53 (m, 2H), 7.54-7.62 (m, 1H), 8.03 (ddd,  $J$  = 7.0, 1.1, 1.1 Hz, 2H);  $^{13}\text{C}$  NMR (100MHz,  $\text{C}_6\text{D}_6$ ):  $\delta$  21.8 (C(11)H<sub>3</sub>), 21.8 (C(6)H<sub>2</sub>), 22.9(CH<sub>3</sub>C(O)), 23.4 (C(10)H<sub>3</sub>), 25.2 (C(6)H<sub>2</sub>), 31.2 (C(5)H), 33.6 (C(7)H<sub>2</sub>), 78.0 (C(1)H), 82.4 (C(4)), 84.4 (C(8)), 125.3 (=C(13)H), 128.8 (2CH), 128.9 (2CH), 132.7 (CH), 150.4 (=C(14)HC(O)), 189.1 (C=O). A small amount of the

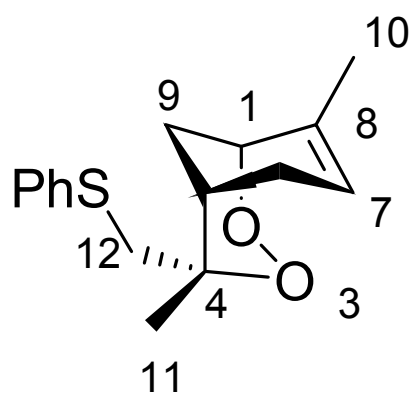
starting aldehyde **52** [0.5 mg, 5%,  $R_f$  = 0.32 (10% ethyl acetate in hexane)] was recovered by an additional flash chromatography of the less polar fractions.

### References for Supporting Information

- (1) Korshin, E. E.; Hoos, R.; Szpilman, A. M.; Posner, G. H.; Bachi, M. D. *Tetrahedron* **2002**, *58*, 2449-2469.
- (2) (a) Mair, R. D.; Hall, R. T. In *Organic Peroxides*; Swern, D., Ed.; Wiley Interscience: London, 1971; Vol. 2, p 553. (b) Knappe, E.; Peteri, D. *Z. Anal. Chem.* **1962**, *190*, 386-389.
- (3) A. B. Smith III, J. Kingerywood, T. L. Leenay, E. G. Nolen and T. Sunazuka, *J. Am. Chem. Soc.* **1992**, *114*, 1438-1449
- (4) The aldehyde **52** was prepared in 92% yield from (1*R*,4*R*,5*R*,8*R*)-8-acetoxy-4,8-dimethyl-4-phenylsulfinylmethyl-2,3-dioxabicyclo[3.3.1]nonane<sup>2</sup> through a Pummerer reaction with TFAC and 2,6-lutidine in conditions which are very similar to the described for the synthesis of unsaturated aldehydes **21-24** (see Scheme 4 and Experimental Section of this paper).
- (5) Bachi, M. D.; Korshin, E. E.; Hoos, R.; Szpilman, A. M.; Cumming, J. N.; Plyopradith, P.; Xie, S. J.; Shapiro, T. A.; Posner, G. H. *J. Med. Chem.* **2003**, *46*, 2516-2533.

**Copies of  $^1\text{H}$ ,  $^{13}\text{C}$ , DEPT, COSY and HMQC NMR spectra**

Unsaturated sulfide **16a**:

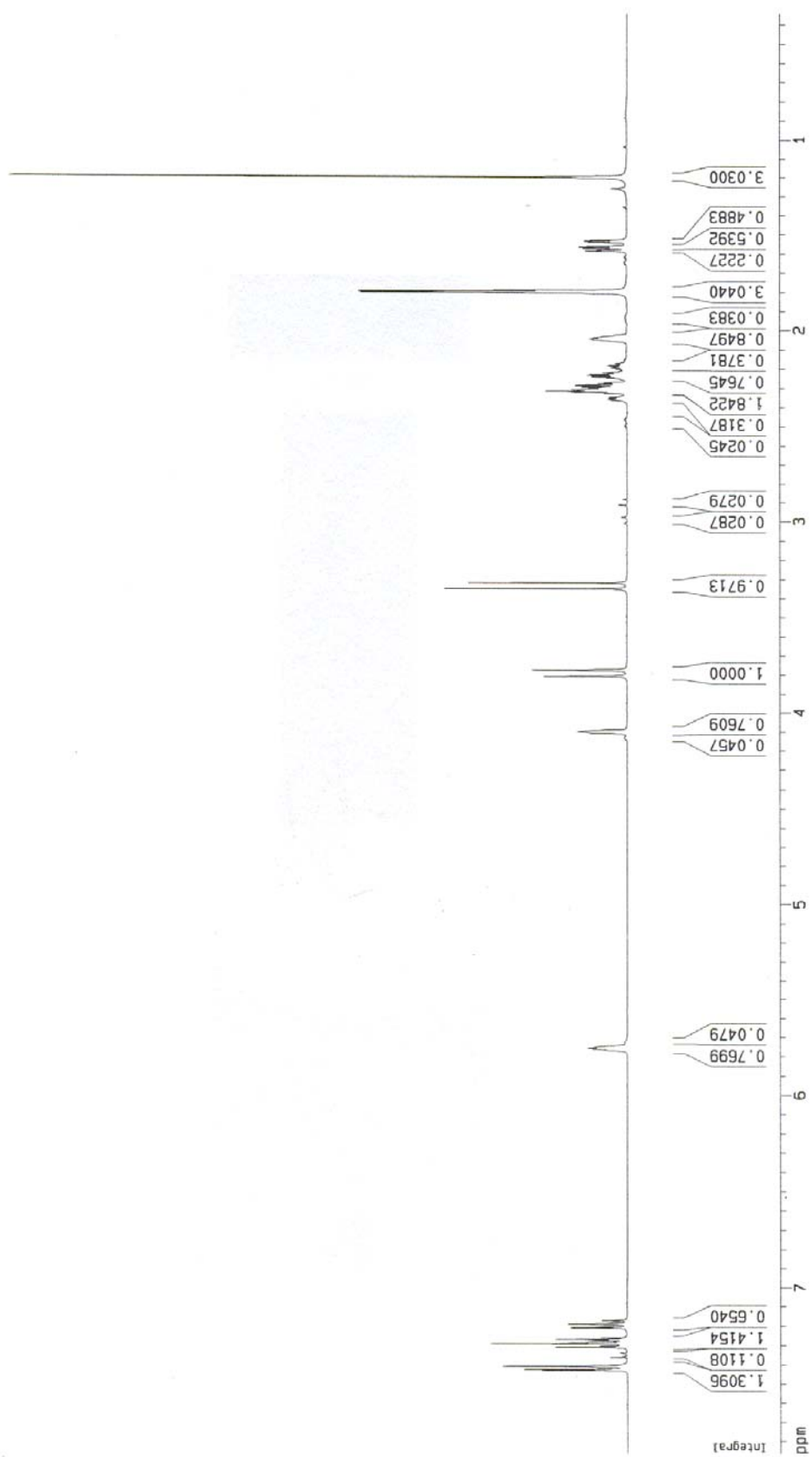


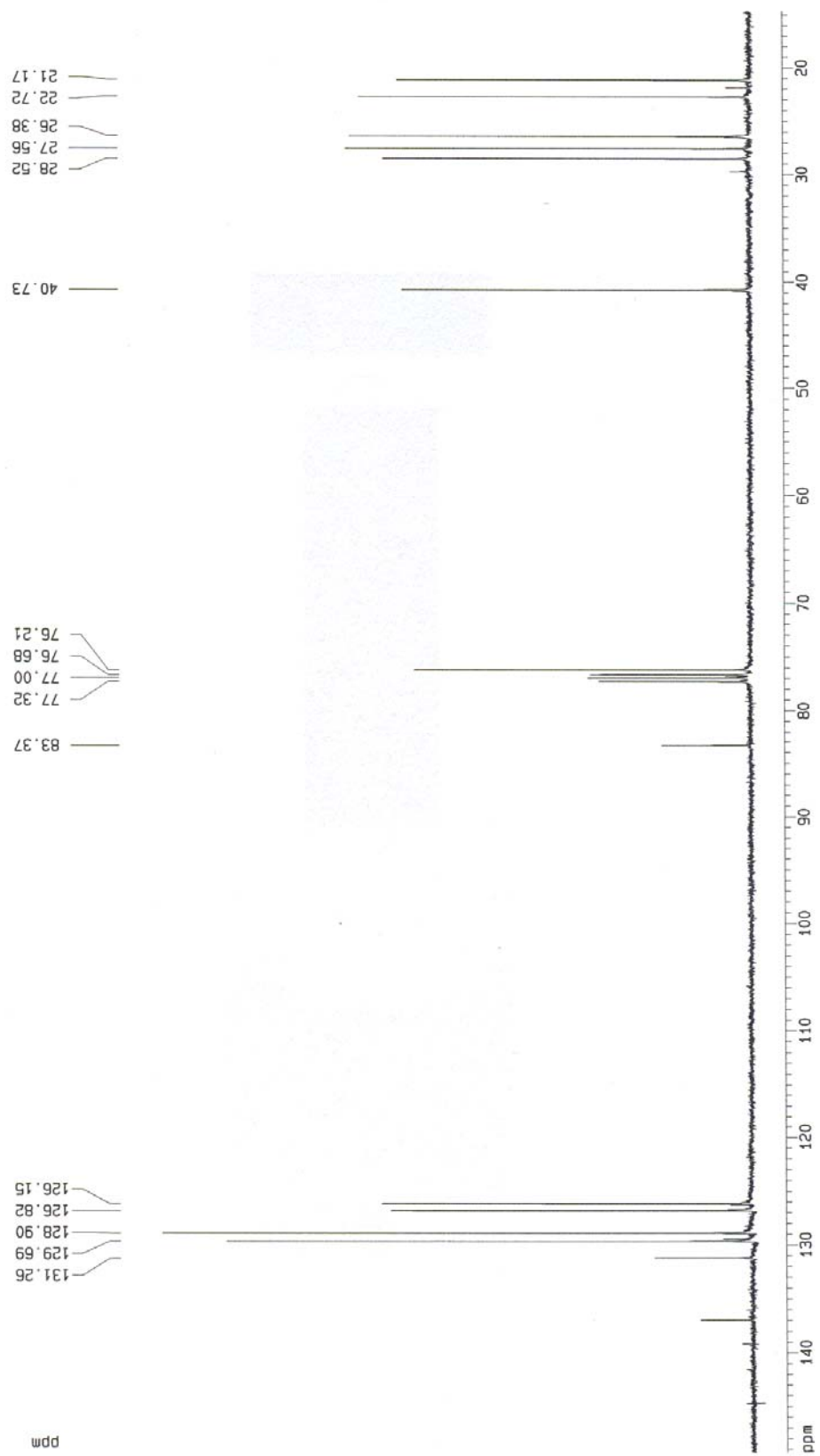
**16a**

Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

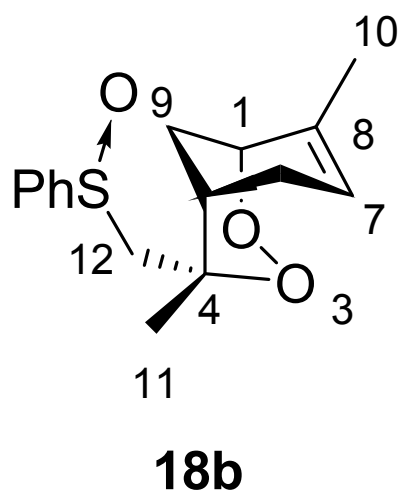
$^{13}\text{C}$  NMR spectra







Unsaturated sulfoxide **18b**:



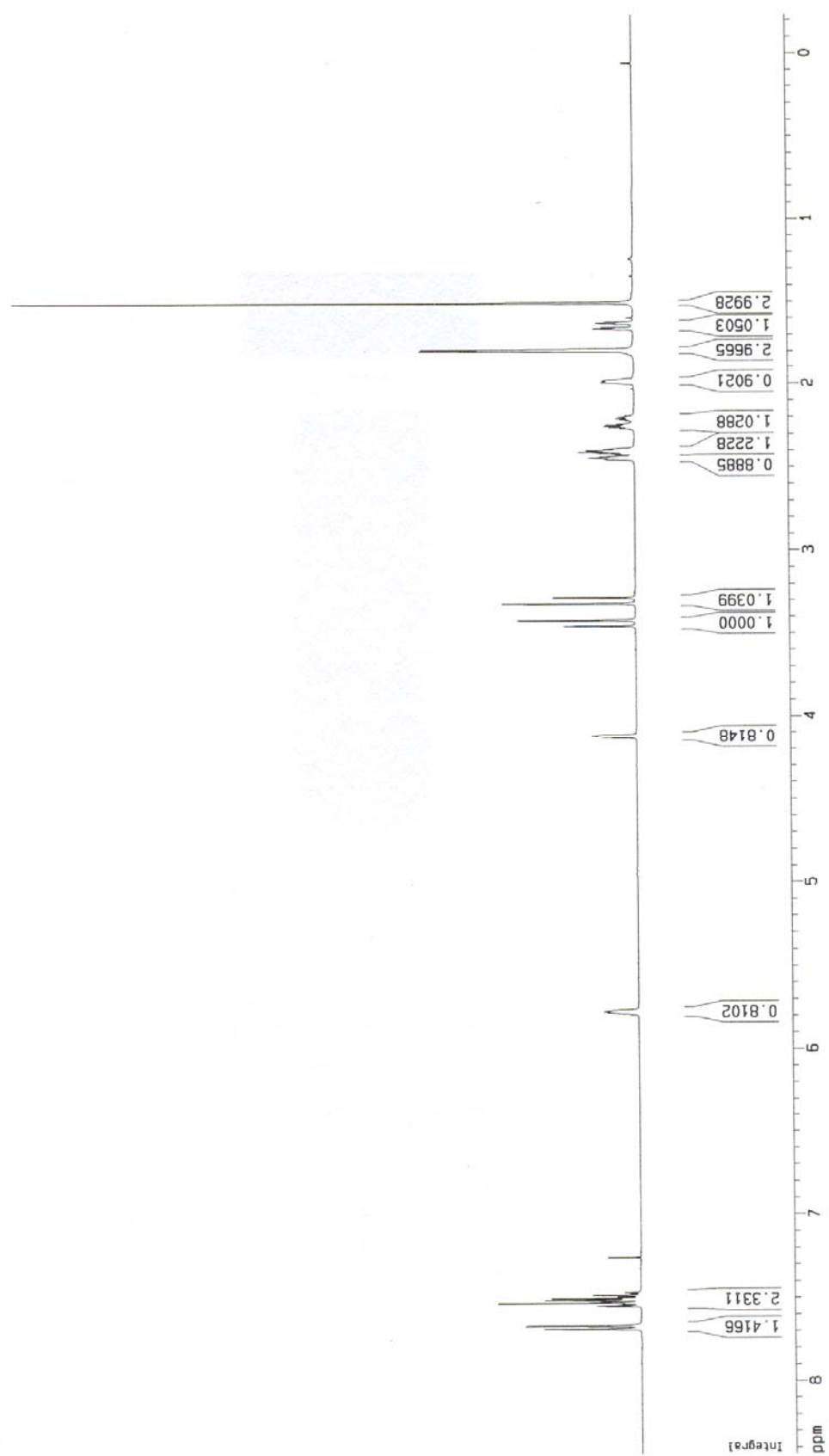
Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

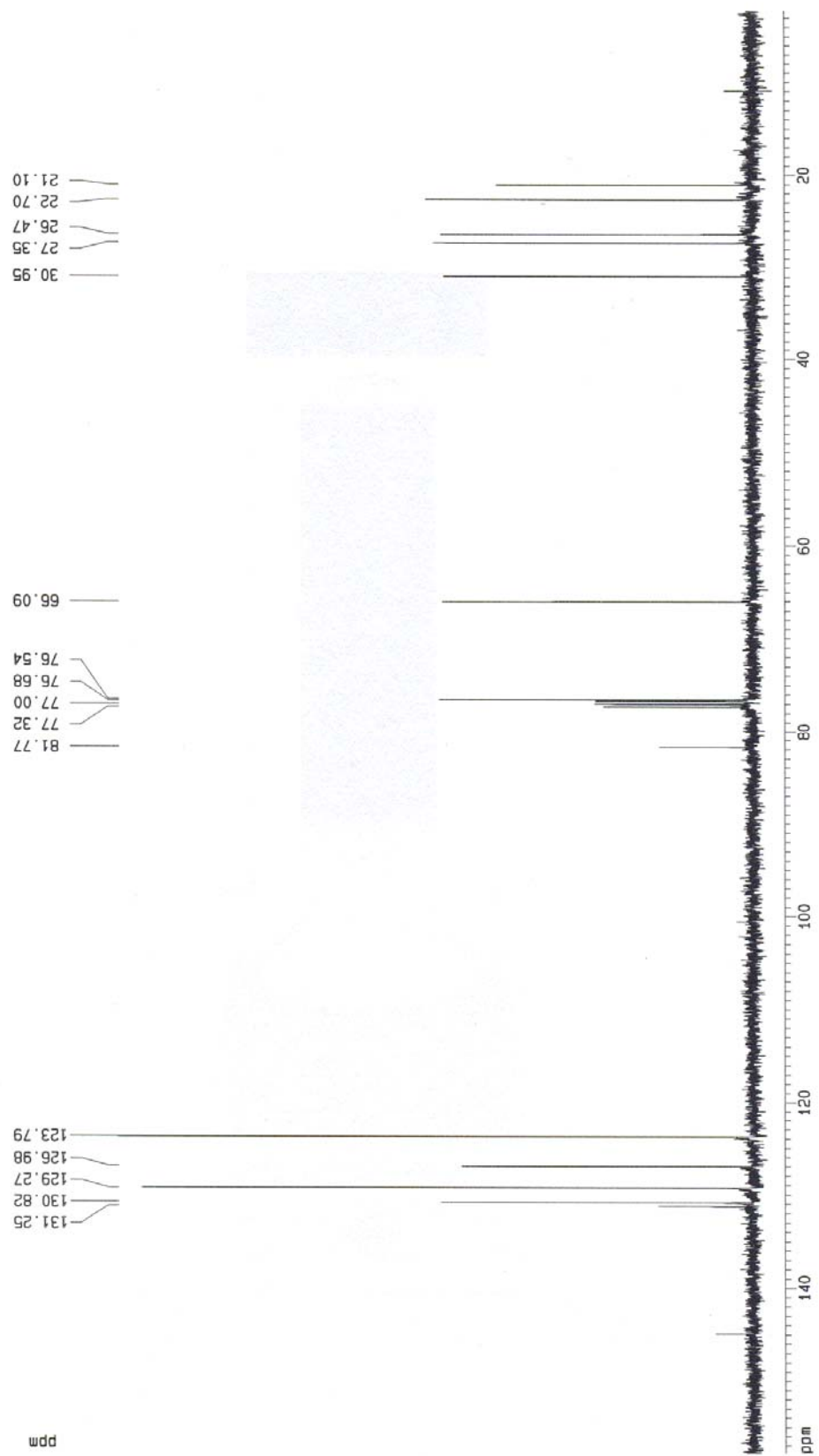
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

COSY spectra

HMQC spectra





Current Data Parameters  
NAME edicosy952.3  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters

Date 200530  
Time 15.55  
PULPROG cosy45  
SOLVENT ccd13  
AQ 0.1957640 sec  
FIDRES 3.130008 Hz  
DN 156.0 usec  
RG 128  
NUCLEUS 1H  
HL1 1.0B  
D1 0.900000 sec  
P1 12.6 usec  
D0 0.000030 sec  
DE 222.9 usec  
SF01 400.1362351 MHz  
SNH 3205.13 Hz  
TD 1024  
NS 4  
DS 4  
IN0 0.0003120 sec

F1 - Acquisition Parameters

NUC0 1  
TD 256  
SF01 400.1362 MHz  
FIDRES 12.519917 Hz  
SN 8.010 ppm

F1 - Processing parameters

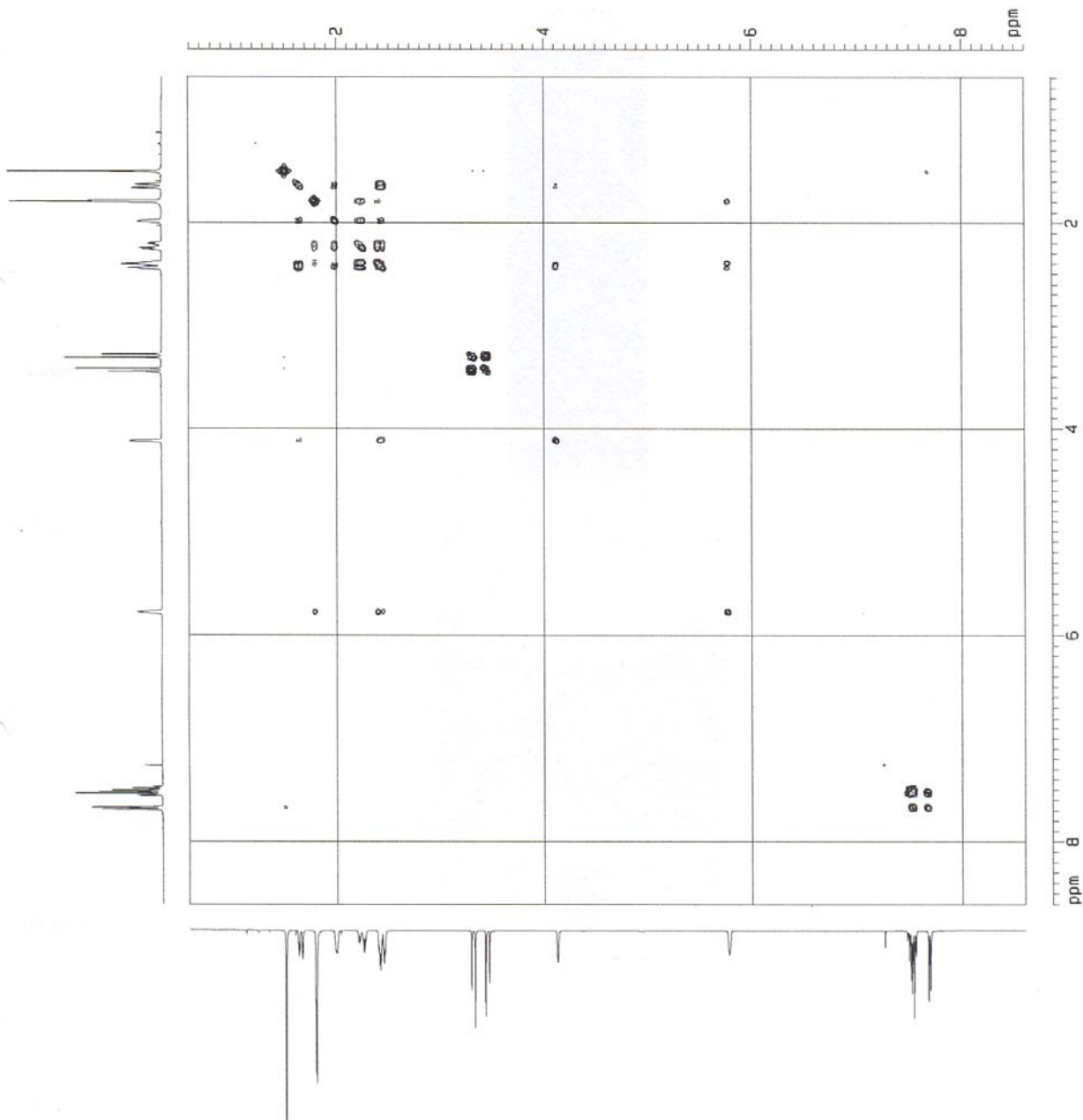
SI 512  
MC2 512  
SF 400.1343965 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

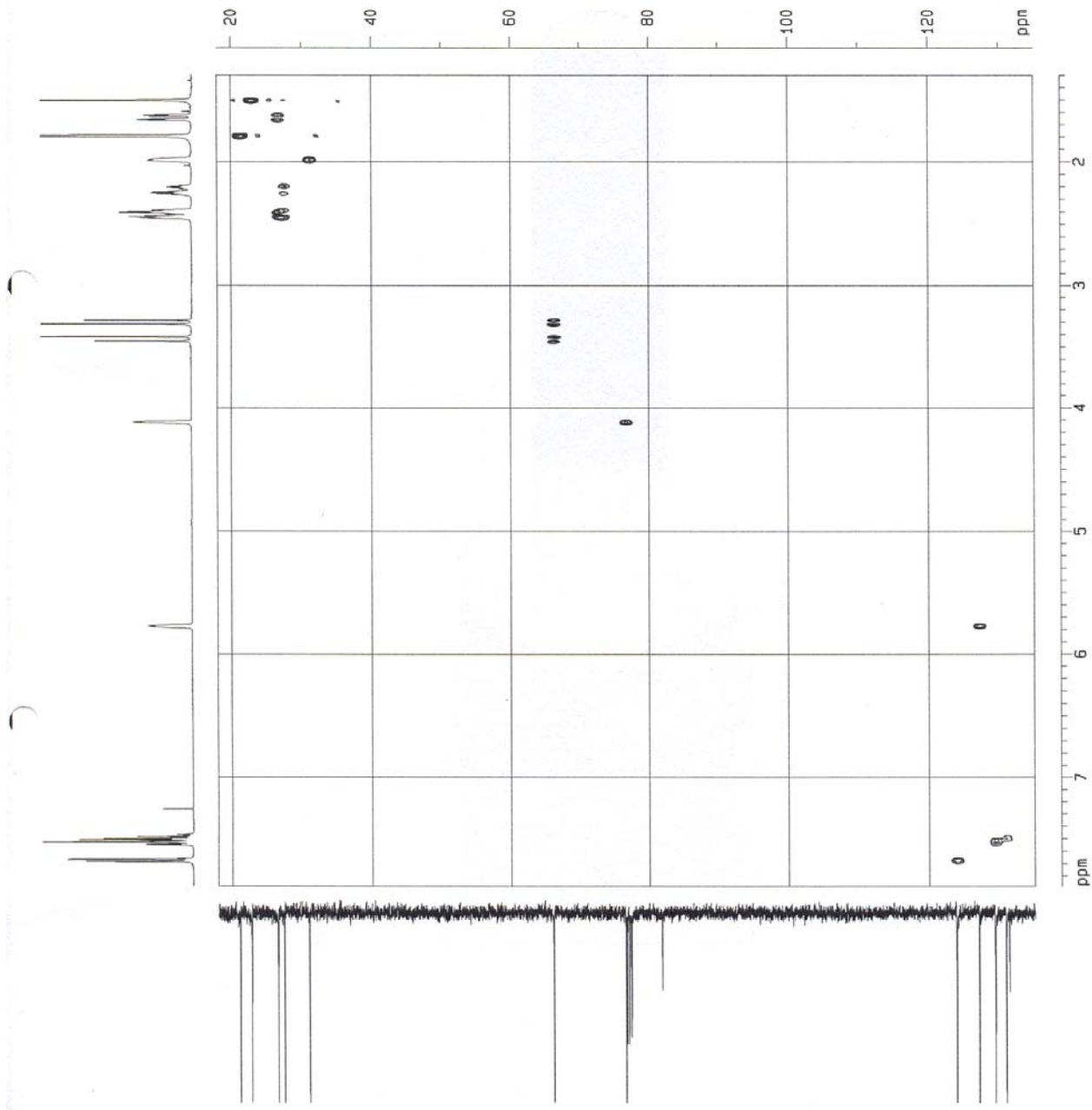
F1 - Processing parameters

SI 512  
MC2 512  
SF 400.1343965 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

2D NMR plot parameters

CX2 15.00 cm  
CX1 15.00 cm  
F2PL0 8.600 ppm  
F2L0 3441.16 Hz  
F2PHI 0.550 ppm  
F2H1 236.03 Hz  
F1PL0 8.600 ppm  
F1L0 3441.16 Hz  
F1PHI 0.550 ppm  
F1H1 236.06 Hz  
F2PMON 0.53401 ppm/cm  
F2FOM 213.67522 Hz/cm  
F1PMON 0.53400 ppm/cm  
F1FOM 213.67325 Hz/cm





Current Data Parameters  
 NAME 611010552-10  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 960530  
 Time 16:23  
 PULPROG invetd  
 SOLVENT CDCl3  
 AQ 0.1507640 sec  
 FIDRES 3.150000 Hz  
 BW 126.0 ussc  
 SFO1 400.1323651 MHz  
 NUC1 13C  
 NUC2 1H  
 D1 2.0000000 sec  
 P1 13.0 ussc  
 D2 0.0040000 sec  
 P2 25.0 ussc  
 P4 14.0 ussc  
 SFO2 100.6214220 MHz  
 D7 0.6000000 sec  
 P3 7.0 ussc  
 D13 0.0000000 sec  
 D10 0.0000000 sec  
 IL0 20.00 dB  
 DE 222.9 ussc  
 SFO1 400.1323651 MHz  
 SWH 3265.13 Hz  
 TD 1024  
 P31 80.0 ussc  
 NS 16  
 DS 4  
 IN0 0.000150 sec

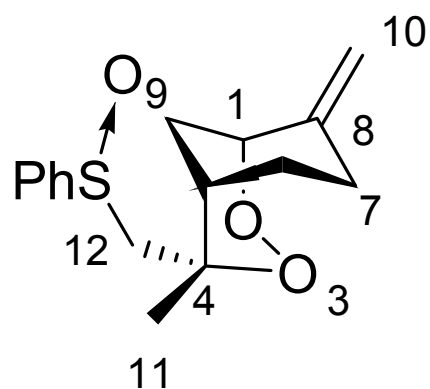
F1 - Acquisition parameters  
 NQ0 128  
 TD 128  
 SFO1 100.6214 MHz  
 FIDRES 102.795782 Hz  
 SW 130.766 ppm

F1 - Processing parameters  
 SI 1024  
 N2 1024  
 SF 400.1323651 MHz  
 WDW SINE  
 SSF 2  
 LB 0.00 Hz  
 GB 0

F1 - Processing parameters  
 SI 256  
 N2 256  
 SF 100.6138556 MHz  
 WDW EM  
 SSF 1  
 LB -10.00 Hz  
 GB 0.33

2D NMR plot parameters  
 D12 15.00 cm  
 D11 15.00 cm  
 F2P10 7.985 ppm  
 F2L0 3155.20 Hz  
 F2P1C 1.259 ppm  
 F2H1 519.72 Hz  
 F1P10 1355.001 ppm  
 F1L0 1358.921 Hz  
 F1P1C 18.098 ppm  
 F1H1 1814.86 Hz  
 F2P1CM 0.43910 ppm/cm  
 F2H1CM 175.69776 Hz/cm  
 F1P1CM 7.73987 ppm/cm  
 F1H1CM 764.67401 Hz/cm

Unsaturated sulfoxide **19b**:



**19b**

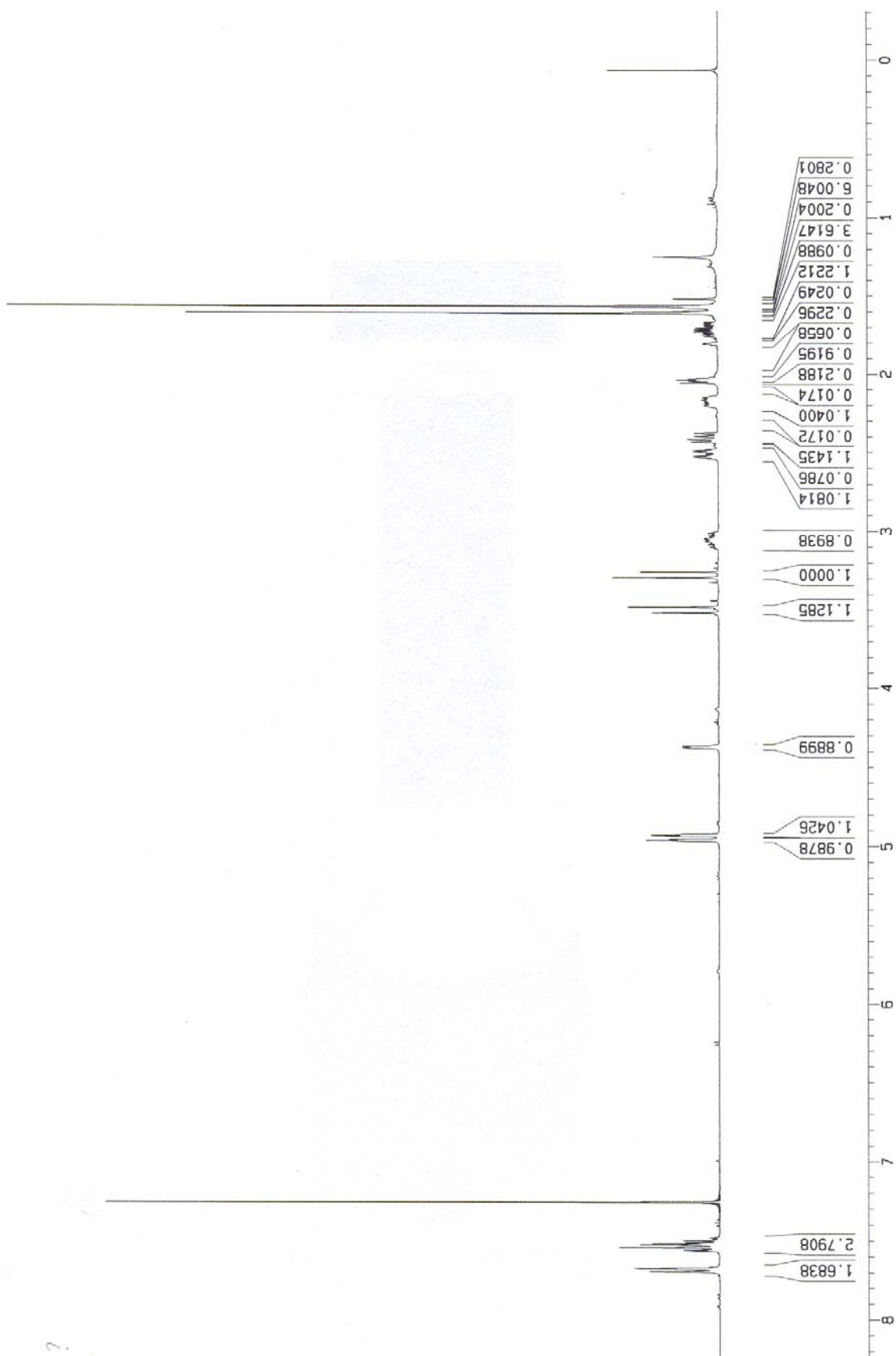
Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

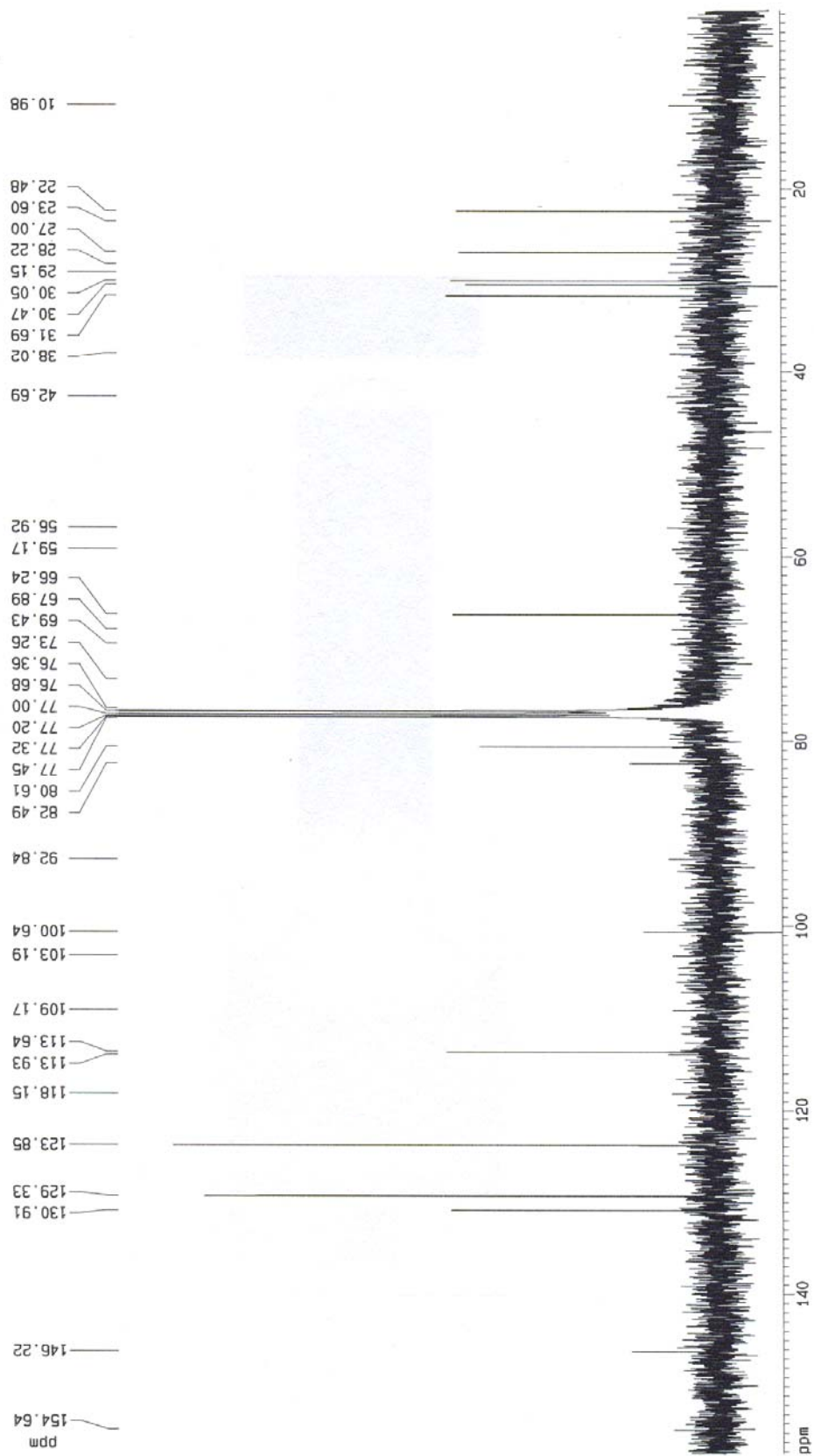
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra





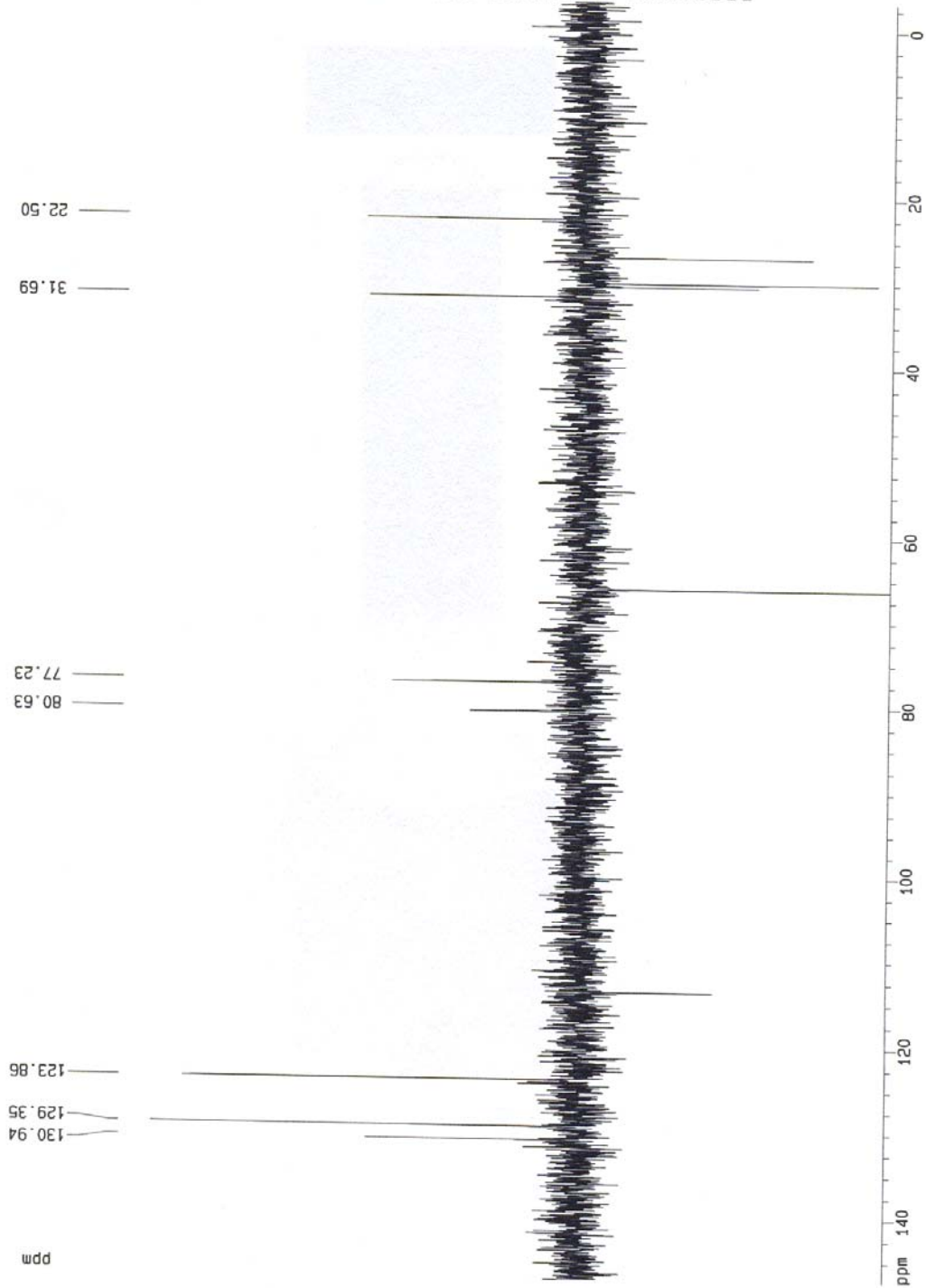


Current Data Parameters  
 NAME edik30.5-89510  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 960631  
 Time 4.42  
 PULPROG zgpg30  
 SOLVENT DMSO  
 AQ 1.048986 sec  
 FIDRES 0.476837 Hz  
 DW 16.0 usec  
 RG 32768  
 NUCLEUS 13C  
 H1 1 dB  
 D1 3.0000000 sec  
 S1 1 dB  
 P3 10.3 usec  
 SF02 400.1360000 MHz  
 D2 0.0035714 sec  
 P4 20.6 usec  
 P2 6.2 usec  
 S2 12.4 usec  
 DE 22.9 usec  
 SF01 100.6223697 MHz  
 SMH 31250.00 Hz  
 TD 65536  
 P31 100.0 usec  
 NS 5069  
 DS 2

F1 - Processing parameters  
 S1 131072  
 W2 65536  
 SF 100.6136722 MHz  
 WDW EN  
 SSB 0  
 LB 2.00 Hz  
 GB 0

ID NMR plot parameters  
 CX 23.10 cm  
 F1P 147.260 ppm  
 F1 14816.44 Hz  
 F2P -3.310 ppm  
 F2 -333.07 Hz  
 FPMCH 6.51808 ppm/cm  
 HZCM 655.50979 Hz/cm



Current Data Parameters  
edikcosy8951.2 in  $\text{CO}_2$

## F2 - Acquisition Parameters

Date		960820C				
Time	18.03					
PALPROG	COSYAS					
SOLVENT	CDCl <sub>3</sub>					
AQ	0.215600 sec					
FIDRES	2.325149 Hz					
DW	219.0 usec					
RG	4095					
HOLDUS	1H					
HLI	1 dB					
D1	1.0000000 sec					
P1	12.8 usrc					
D0	0.000030 sec					
DE	300.0 USEC					
FSFO1	400.1378750 MHz					
NK1	2380.95 Hz					
TD	1024					
NS	16					
TNO	4					
ZND	0.0004260 SEC					

## F1 - Acquisition parameters

INDO	1
TD	256
SF01	400.1358 MHz
FIDRES	9.300656 Hz
SN	5.950 dB

### F1 - Processing parameters

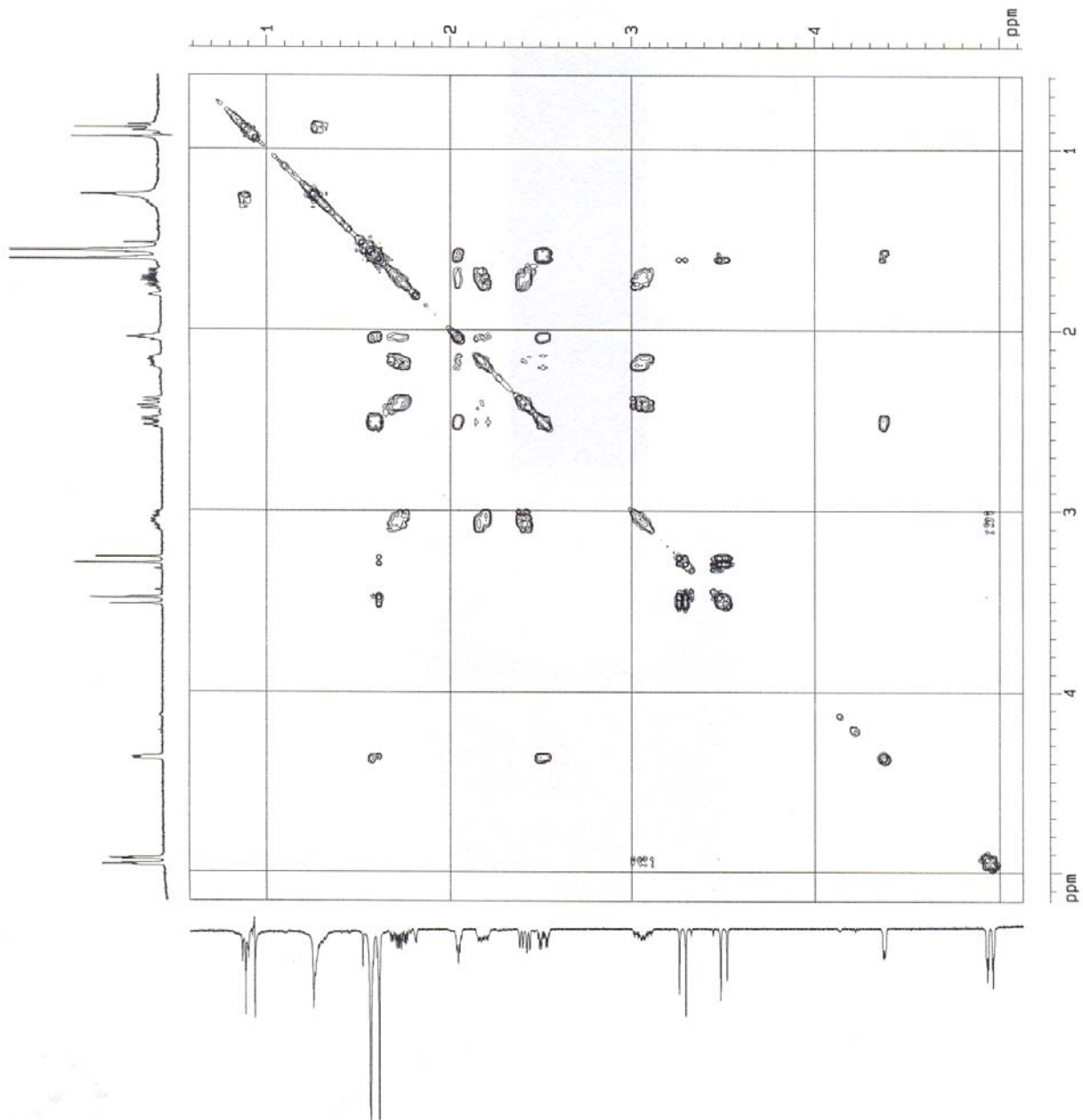
SI	512
MC2	OF
SF	400.1343945 MHz
WOM	SINE
SSB	0
LB	0.00 Hz
SB	0

## F1 - Processing parameters

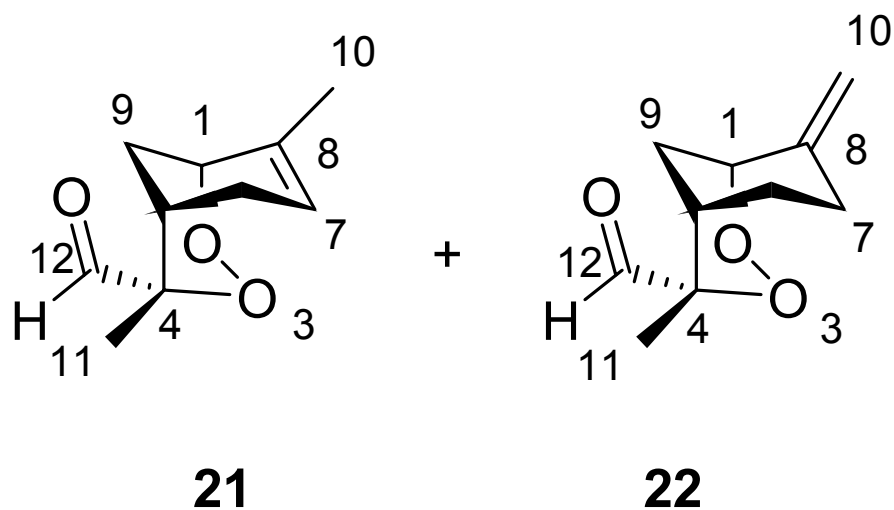
SI	512
MC2	OF
SF	400.1343945 MHz
NDW	SINE
SSB	0
LB	0.00 HZ
FR	0

## 20 NMR plot parameters

2X2	45.00 cm
2X4	45.00 cm
2P5.0	5.160 ppm
2L0	205.4 69 Hz
2PH1	0.593 ppm
2H1	237.12 Hz
1P0	5.125 ppm
1L0	2050.73 Hz
1PH1	0.581 ppm
1H1	232.45 Hz
3P3MCM	0.30449 ppm/cm
2PHMCM	121.83760 Hz/cm
1PPMCM	0.30254 ppm/cm
1H2MCM	121.21855 Hz/cm



Mixture of unsaturated aldehyde **21** and **22**:



Recorded at 400 MHz ( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

COSY spectra

Current Data Parameters  
 NAME as2.24a150300H  
 EXPRNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20000315  
 Time 19.00  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 58  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.244532 Hz  
 AQ 2.0447731 sec  
 RG 256  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SF01 400.1320007 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300049 MHz  
 WDM EM  
 SSB 0  
 LB 0.01 Hz  
 GB 0  
 PC 0.40

1D NMR plot parameters  
 CX 20.00 cm  
 FLP 11.000 ppm  
 F1 4401.43 Hz  
 F2P -0.200 ppm  
 F2 -80.03 Hz  
 PPMCM 0.56000 ppm/cm  
 HZCM 224.07280 Hz/cm

1.07715  
 1.17374  
 1.82763  
 1.83205  
 1.83361  
 1.83602  
 1.83812  
 1.84243

9.91170

ppm

3.0016  
 0.6389  
 1.4361  
 0.4090  
 2.8605  
 1.2606  
 0.2955  
 3.0400  
 0.1990  
 0.2616  
 0.2021

0.7530  
 0.1585

0.3659

0.7471

0.7154

Integral

0 2 4 6 8 10 ppm

Current Data Parameters  
 NAME as2.24a150300C  
 EXPNO 1  
 PROCNO 1

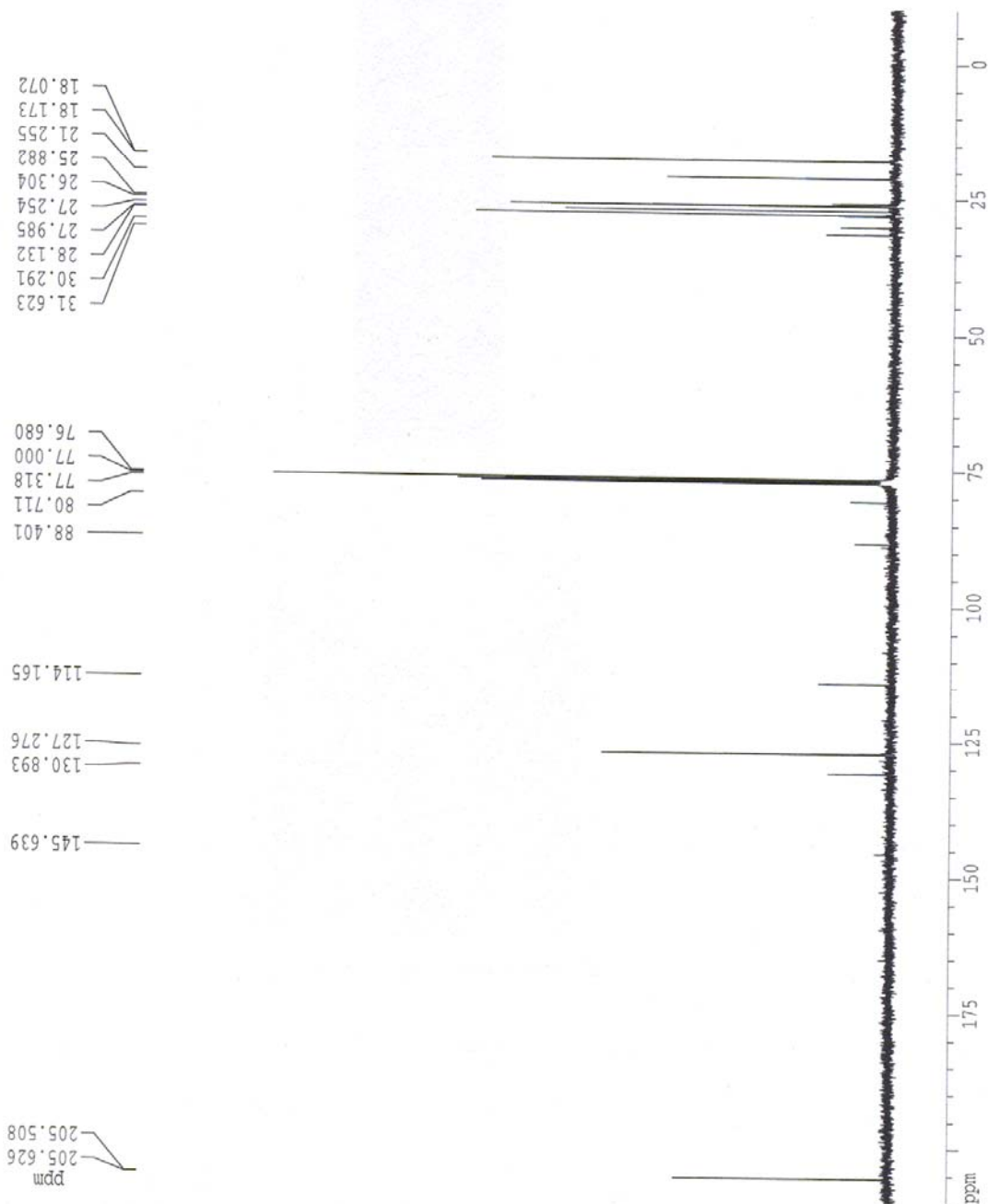
F2 - Acquisition Parameters  
 Date\_ 20000315  
 Time 19.21  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zgpgc  
 TD 65536  
 SOLVENT CDC13  
 NS 1556  
 DS 4  
 SWH 30120.462 Hz  
 FIDRES 0.459602 Hz  
 AQ 1.0879476 sec  
 RG 2896.3  
 DW 16.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.20 usec  
 PL1 -6.00 dB  
 SFO1 100.6227903 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waitz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 120.00 dB  
 PL12 14.00 dB  
 SFO2 400.1316005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127717 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 FIP 210.000 ppm  
 F1 21128.68 Hz  
 F2P -10.000 ppm  
 F2 -1006.13 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 1106.74048 Hz/cm



# as2-24a COSY

Current Data Parameters  
NAME as2.24a1500Co  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20000315  
Time 21:56  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 1024  
SOLVENT CDCl3  
NS 8  
DS 1  
SWH 3834.356 Hz  
FIDRES 3.74488 Hz  
AQ 0.133576 sec  
RG 260  
RG2 260  
DW 130.400 usec  
DE 6.00 usec  
TE 300.0 K  
D2 0.0000000 sec  
D1 1.0000000 sec  
DI 1.0000000 sec  
IMD 0.0000000 sec

## ===== CHANNEL f1 =====

NUC1 1H  
P1 13.00 usec  
PL1 -4.00 dB  
SFO1 400.132023 MHz

## F1 - Acquisition Parameters

NUC2 13C  
TD 256  
SFO2 400.1316 MHz  
FIDRES 14.97752 Hz  
SW 9.583 ppm

## F2 - Processing parameters

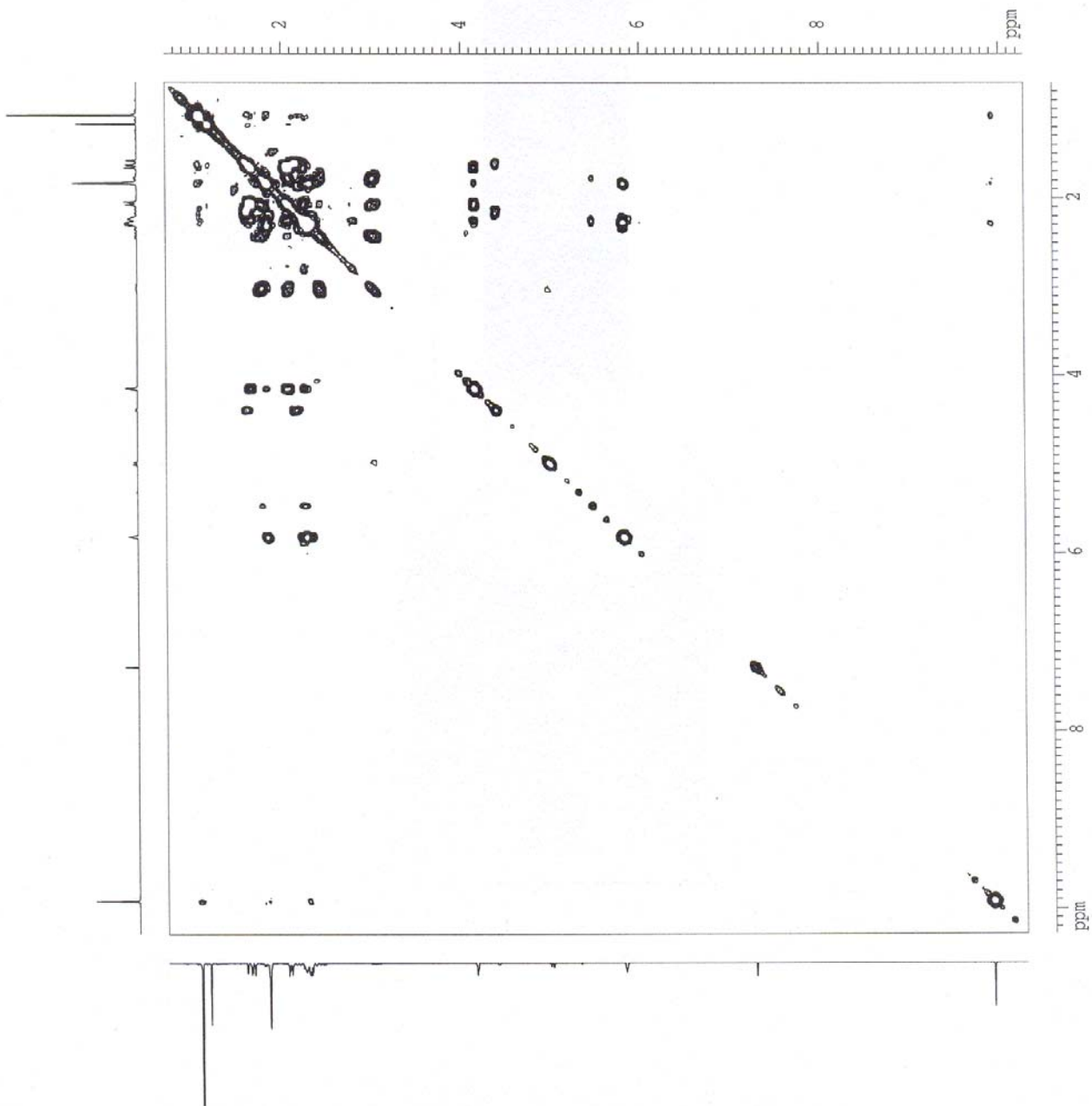
SI 512  
SF 400.1300640 MHz  
WDW Q9INE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## F1 - Processing parameters

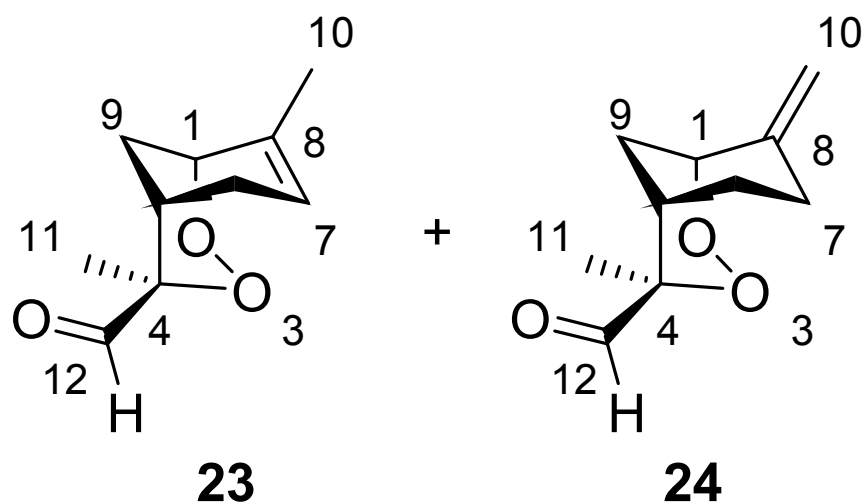
SI 512  
SF 400.1294623 MHz  
WDW Q9INE  
SSB 0  
LB 0.00 Hz  
GB 0

## 2D NMR plot parameters

CH2 15.00 cm  
CH1 15.00 cm  
F2F1 13.282 ppm  
F2F0 41.156 Hz  
F2F1 0.703 ppm  
F2F0 281.10 Hz  
F2F1 10.585 ppm  
F2F0 41.154 Hz  
F2F1 0.703 ppm  
F2F0 281.08 Hz  
F2F1 0.63885 ppm/cm  
F2F0 255.62372 Hz/cm  
F2F1 0.63885 ppm/cm  
F2F0 255.62372 Hz/cm



Mixture of unsaturated aldehydes **23** and **24**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra

Current Data Parameters  
 NAME as2.24b150300H  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20000315  
 Time 22.53  
 INSTRUM spect  
 PROHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 100  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.244532 Hz  
 AQ 2.0447731 sec  
 RG 256  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

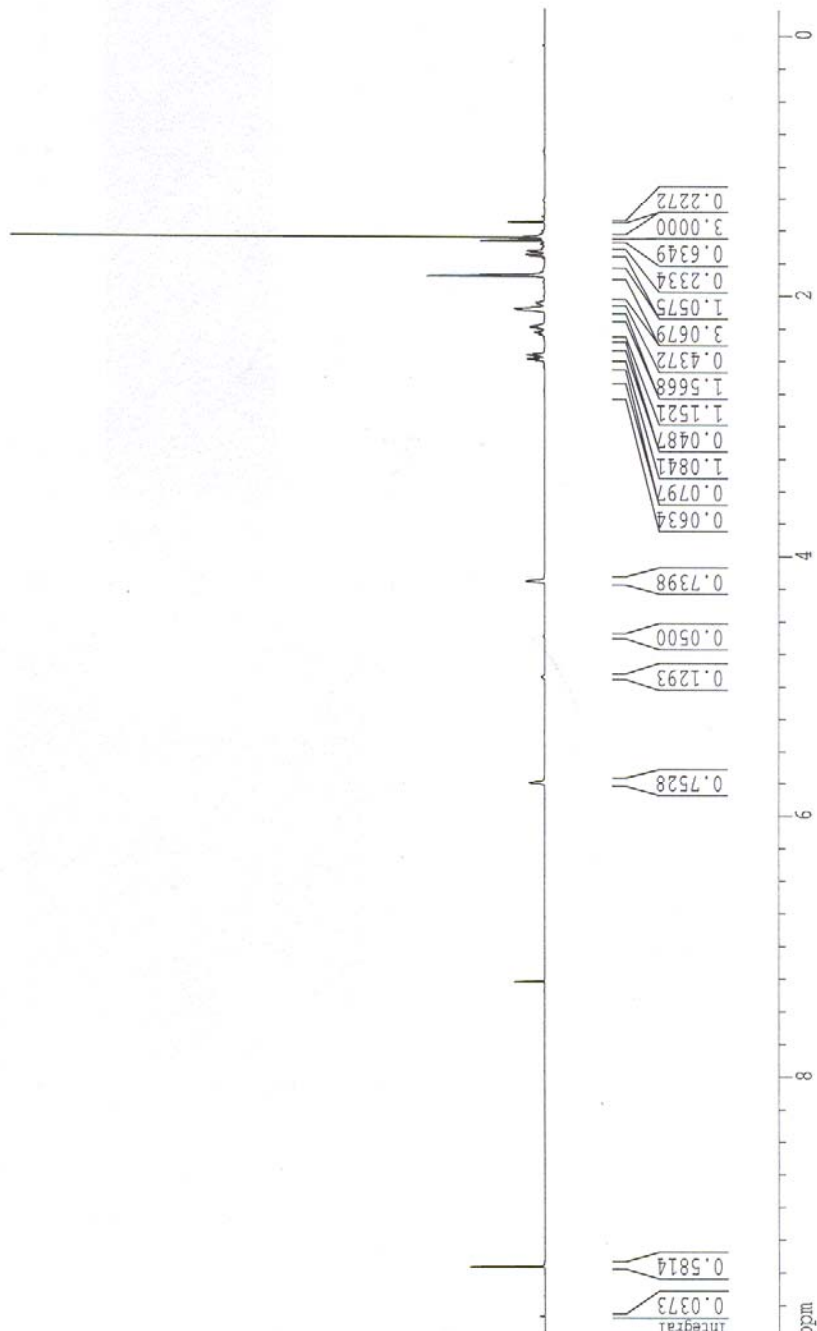
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SF01 400.1320007 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300040 MHz  
 WDW EM  
 SSB 0  
 LB 0.01 Hz  
 GB 0  
 PC 0.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10,000 ppm  
 F1 4001.30 Hz  
 F2P -0.200 ppm  
 F2 -80.03 Hz  
 PPMCM 0.51000 ppm/cm  
 HZCM 204.06630 Hz/cm

1.43756  
 1.55679  
 1.58136  
 1.83845  
 1.84268  
 1.84492  
 1.84914  
 1.85340

9.45751  
 ppm





Current Data Parameters  
 NAME as2.24b150300C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20000315  
 Time 23.51  
 INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 952  
 DS 4  
 SWH 30120.462 Hz  
 FIDRES 0.459602 Hz  
 AQ 1.0879476 sec  
 RG 1149.4  
 DW 16.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.20 usec  
 PL1 -6.00 dB  
 SFO1 100.6227903 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCDP2 80.00 usec  
 PL2 120.00 dB  
 PL12 14.00 dB  
 SFO2 400.1316005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127708 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

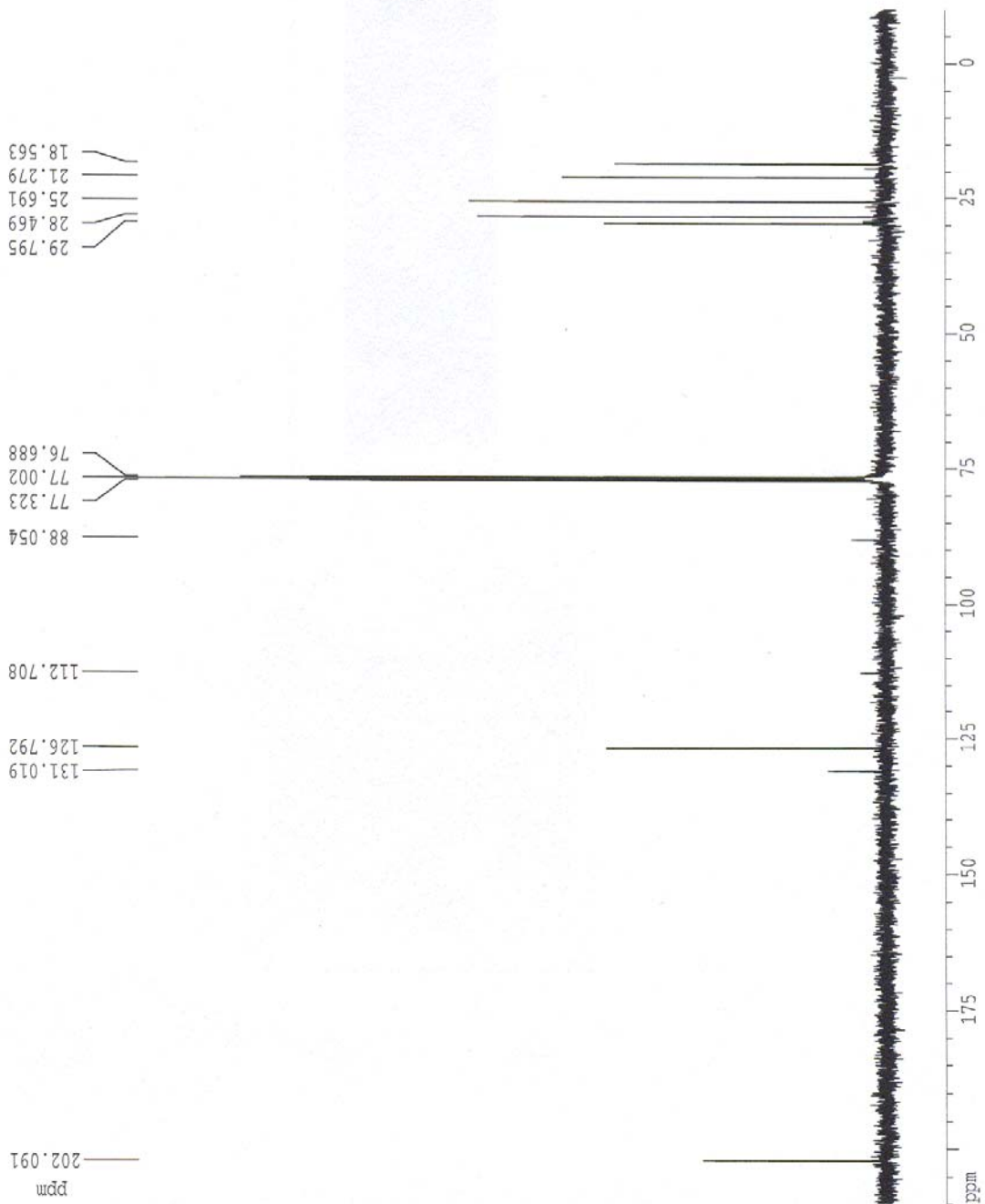
ID NMR plot parameters  
 CX 20.00 cm  
 F1P 210.000 ppm  
 F1 21128.68 Hz  
 F2P -10.000 ppm  
 F2 -1006.13 Hz  
 PPNM 11.00000 ppm/cm  
 HZCM 1106.74048 Hz/cm

29.795  
 28.469  
 25.691  
 21.279  
 18.563

88.054  
 77.323  
 77.002  
 76.688

112.708  
 131.019  
 126.792

202.091



Current Data Parameters  
 NAME as2.24b150300D  
 EXFNO 1  
 PROCNO 1

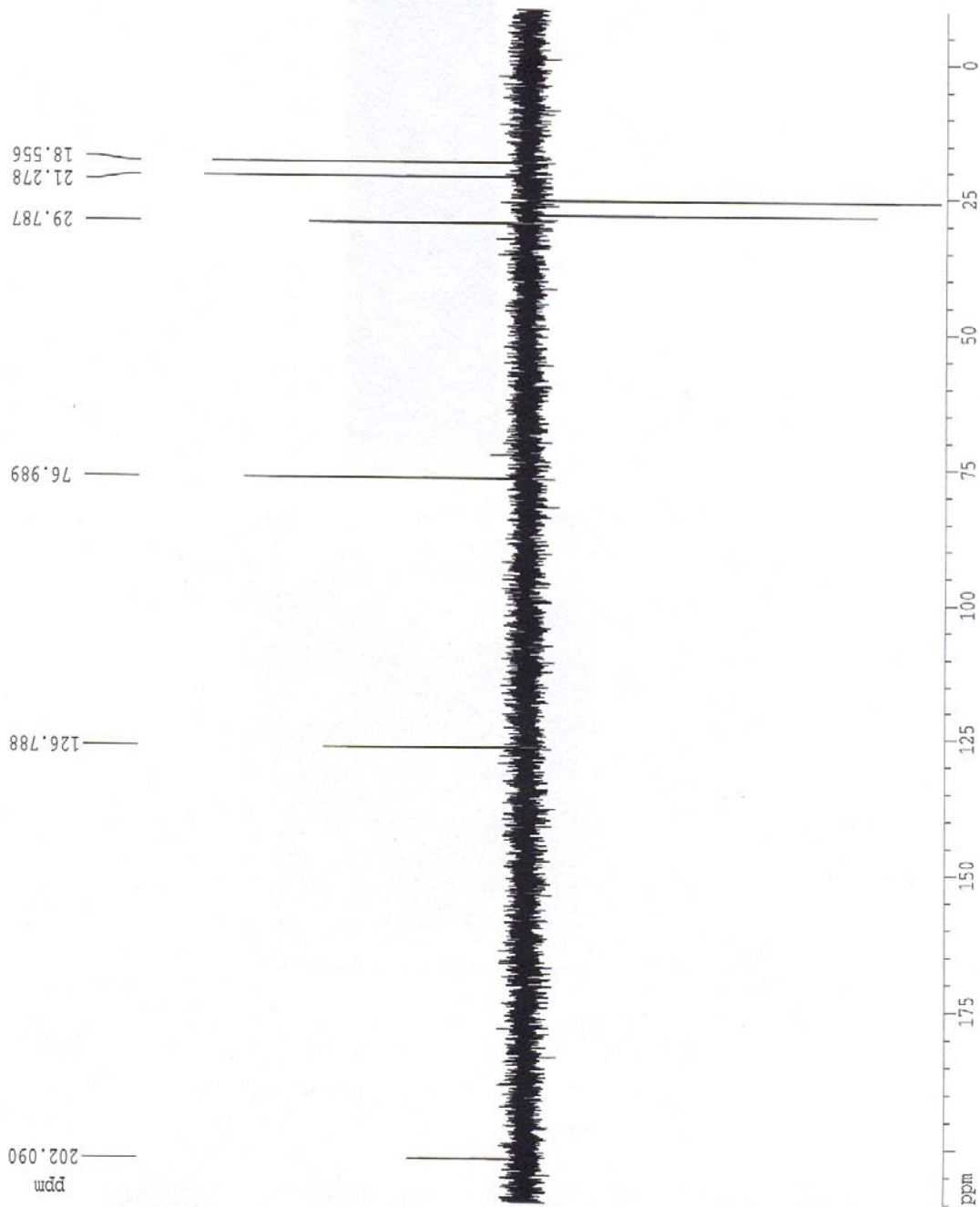
F2 - Acquisition Parameters  
 Date\_ 20000316  
 Time 0.09  
 INSTRUM spect  
 PROBHD 5 mm QNP1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 300  
 DS 8  
 SMH 25125.629 Hz  
 FIDRES 0.383397 Hz  
 AQ 1.3042164 sec  
 RG 11585.2  
 DW 19.900 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 D2 0.0045000 sec  
 D12 0.00002000 sec  
 DELTA 6366.18261719 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.20 usec  
 P2 12.40 usec  
 PL1 -6.00 dB  
 SFO1 100.6227903 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P3 13.05 usec  
 P4 26.10 usec  
 PCPD2 90.00 usec  
 PL2 -6.00 dB  
 PL12 14.00 dB  
 SFO2 400.1308003 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127715 MHz  
 NQM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

ID NMR plot parameters  
 CX 20.00 cm  
 FIP 210.000 ppm  
 F1 21128.68 Hz  
 F2 -10.000 ppm  
 F2 -1006.13 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 1106.74948 Hz/cm



# as2-24B COSY

Current Data Parameters  
NAME  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20000316  
Time 0.22  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 1024  
SOLVENT CDCl3  
NS 8  
DS 16  
SWH 3531.071 Hz  
FIDRES 3.448314 Hz  
AQ 0.1450484 sec  
RG 362  
DM 141.600 usec  
DE 6.00 usec  
TE 300.0 K  
DO 0.0000000 sec  
D1 1.0000000 sec  
INQ 0.0000000 sec

## ===== CHANNEL f1 =====

NUC1 1H  
P1 13.00 usec  
PL1 -4.00 dB  
SFO1 400.132794 MHz

## F1 - Acquisition Parameters

ND0 1  
TD 203  
SFO1 400.1322 MHz  
FIDRES 17.394651 Hz  
SW 8.925 ppm

## F2 - Processing Parameters

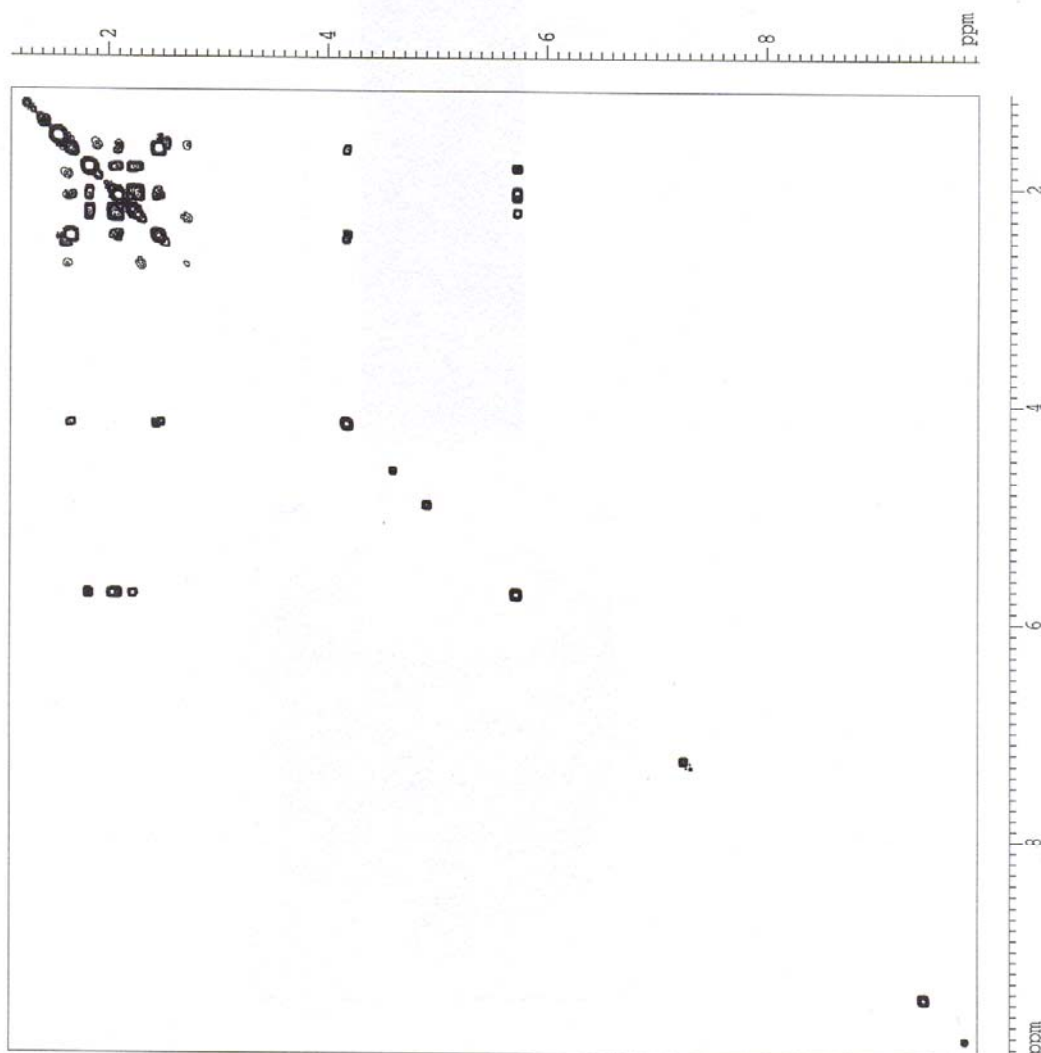
SI 512  
SF 400.1300040 MHz  
WDW QSI  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## F1 - Processing Parameters

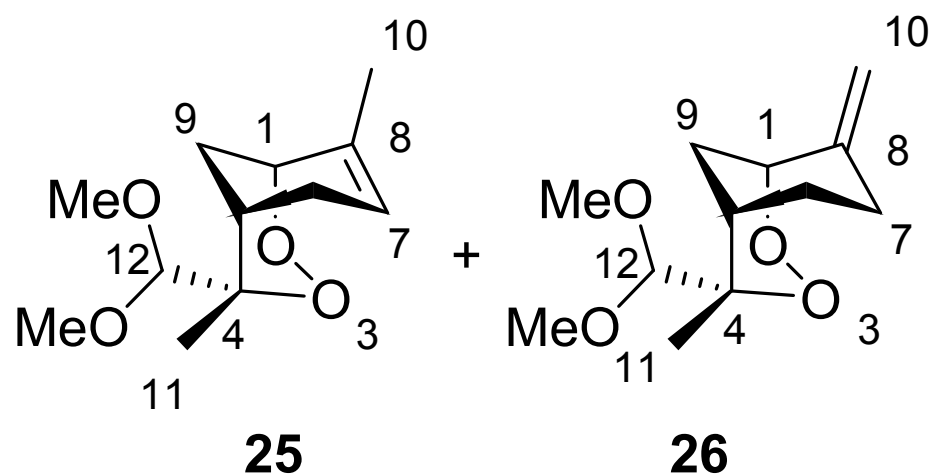
SI 512  
SF 400.1299915 MHz  
WDW QSI  
SSB 0  
LB 0.00 Hz  
GB 0

## 2D NMR plot parameters

CH2 15.00 cm  
CH1 15.00 cm  
F2FLO 9.949 ppm  
F2LO 3980.97 Hz  
F2FHI 1.124 ppm  
F2HI 449.90 Hz  
F2FLO 9.938 ppm  
F2LO 3976.33 Hz  
F2FHI 1.113 ppm  
F2HI 445.26 Hz  
F2FMIN 0.5852 ppm/cm  
F2FMAX 235.4891 ppm/cm  
F2FMIN 0.5852 ppm/cm  
F2FMAX 235.4891 ppm/cm



Mixture of unsaturated acetals **25** and **26**:

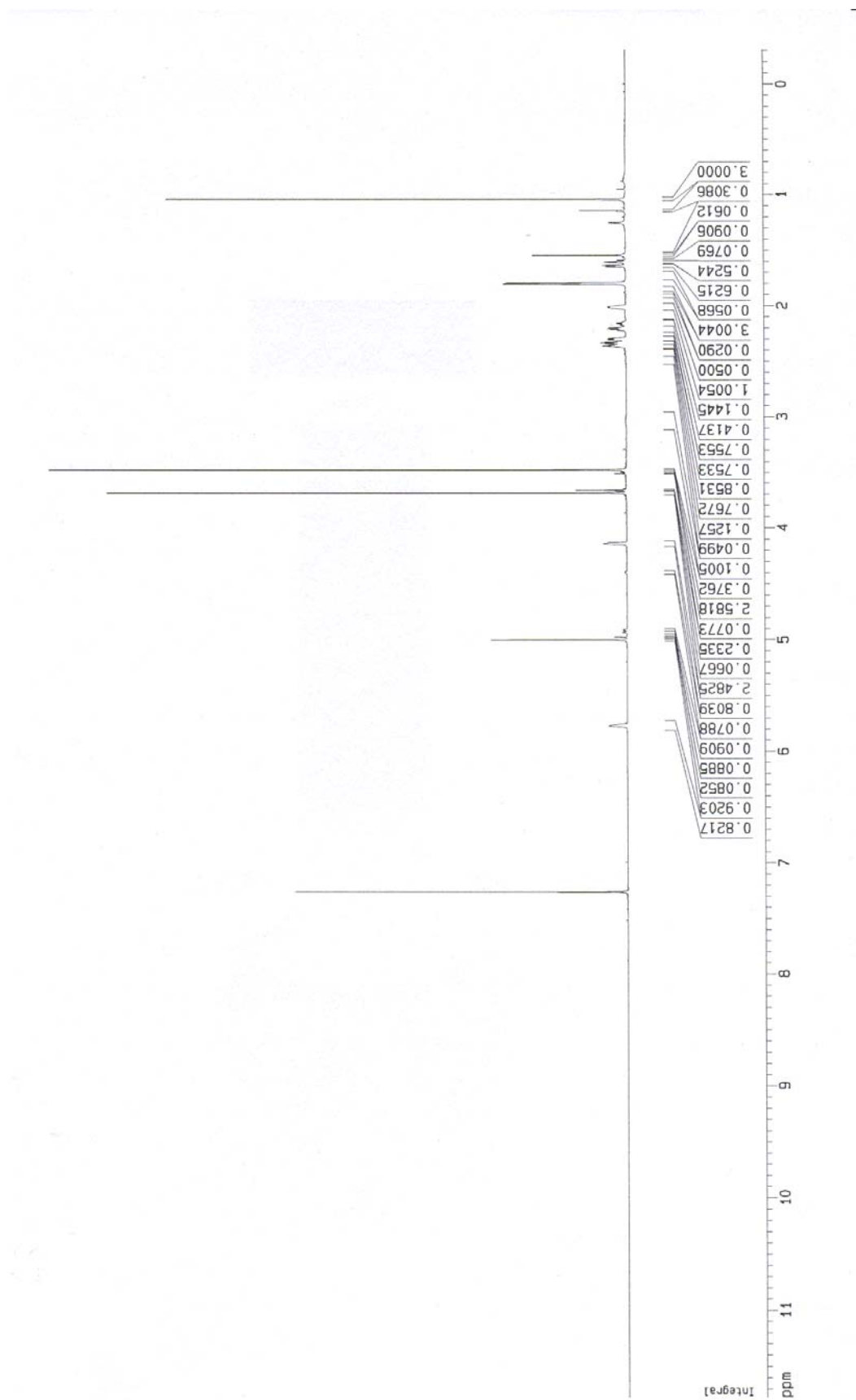


Recorded at 400 MHz ( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra



Current Data Parameters  
 NAME ed1.1.229A46 21710  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

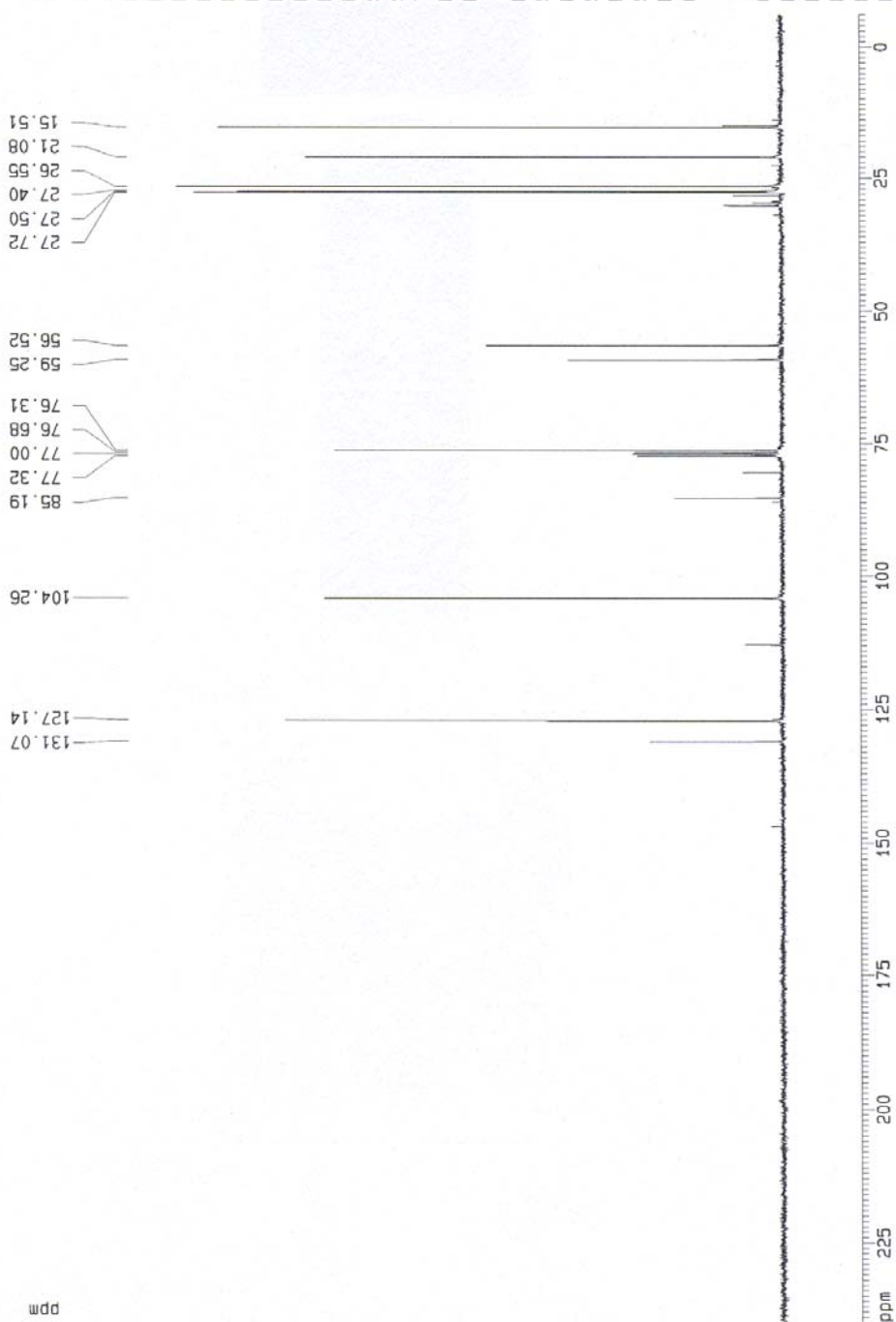
Date 980105  
 Time 1.00  
 PULPROG zg.cpd  
 SOLVENT CDCl3  
 AQ 1.0485960 sec  
 FIDRES 0.476837 Hz  
 DW 16.0 usec  
 RG 32768  
 NUCLEUS 13C  
 HL1 20 dB  
 D1 3.0000000 sec  
 P31 100.0 usec  
 D2 0.0031000 sec  
 P1 7.0 usec  
 DE 22.9 usec  
 SF01 100.6230000 MHz  
 SWH 31250.00 Hz  
 TO 65536  
 NS 340  
 DS 2

F1 - Processing parameters

SI 131072  
 MC2 GF  
 SF 100.6138782 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters

CX 21.95 cm  
 F1P 239.868 ppm  
 F1 24134.01 Hz  
 F2P -6.019 ppm  
 F2 -605.58 Hz  
 PPMCM 11.20100 ppm/c  
 HZCM 1126.97644 Hz/cm



Current Data Parameters  
NAME ~~ec211114~~ 217-  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

Date 980105  
Time 1.29  
PULPROG dept135  
SOLVENT CDCl3  
AQ 1.0485960 sec  
FIDRES 0.476837 Hz  
AQ 15.0 usec  
RG 32768  
NUCLEUS 13C

F1 - Processing parameters

SI 131072  
SF 100.6138008 MHz  
AQ 22.9 usec  
SF01 100.6233997 MHz  
SH 31250.00 Hz  
TD 65536  
P31 100.0 usec  
NS 545  
DS 4

1D NMR plot parameters

CX 23.10 cm  
FIP 197.874 ppm  
F1 18908.92 Hz  
F2P -33.080 ppm  
F2 -3328.27 Hz  
PPHOM 9.99801 ppm/cm  
HZCM 1005.83850 Hz/cm

15.48  
21.07  
27.46

56.52  
59.25

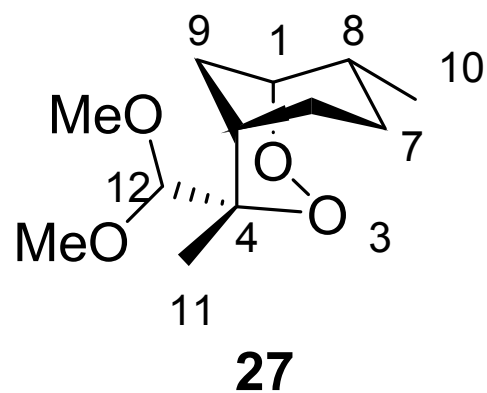
76.29

104.22

127.13



Saturated acetal **27**:



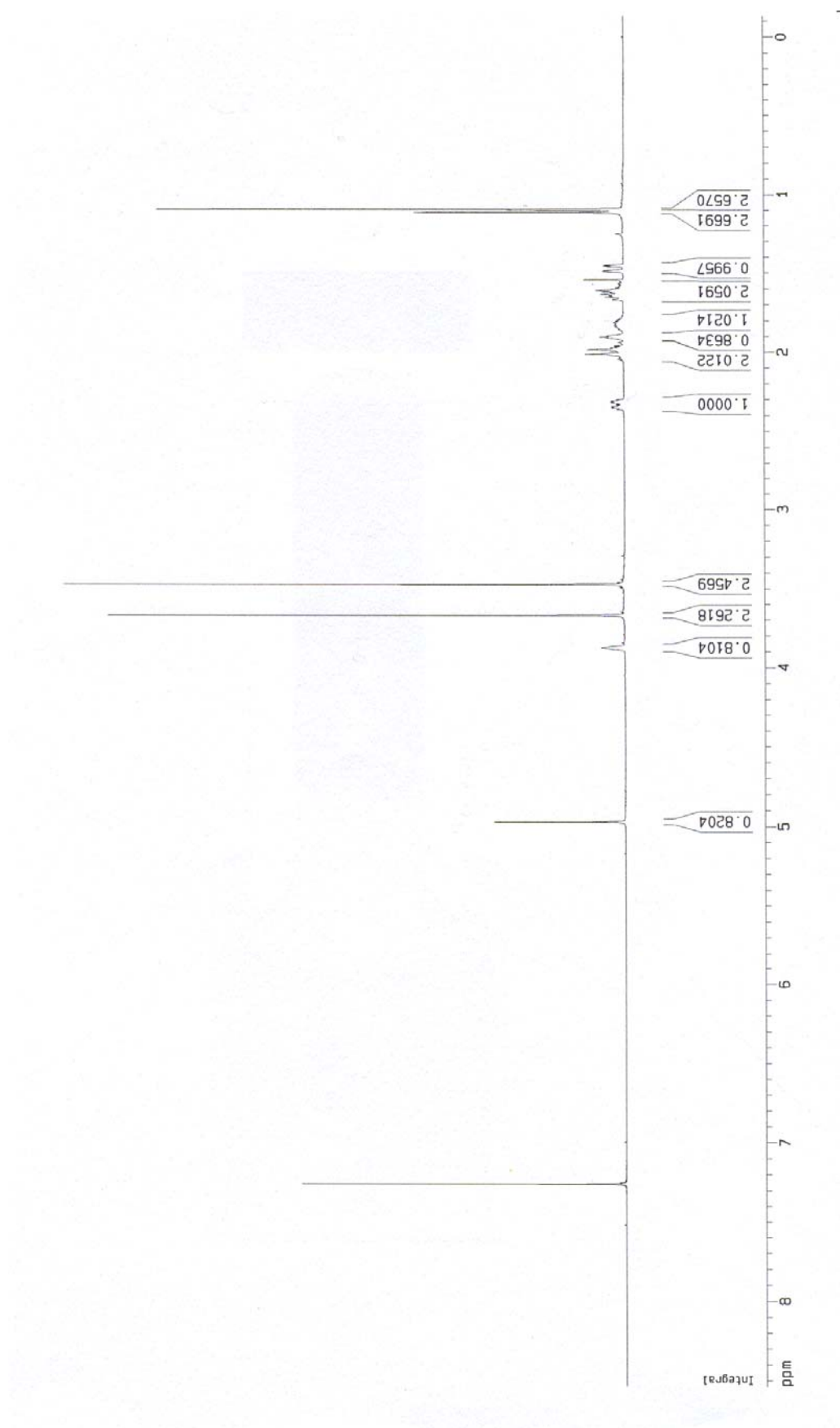
Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra



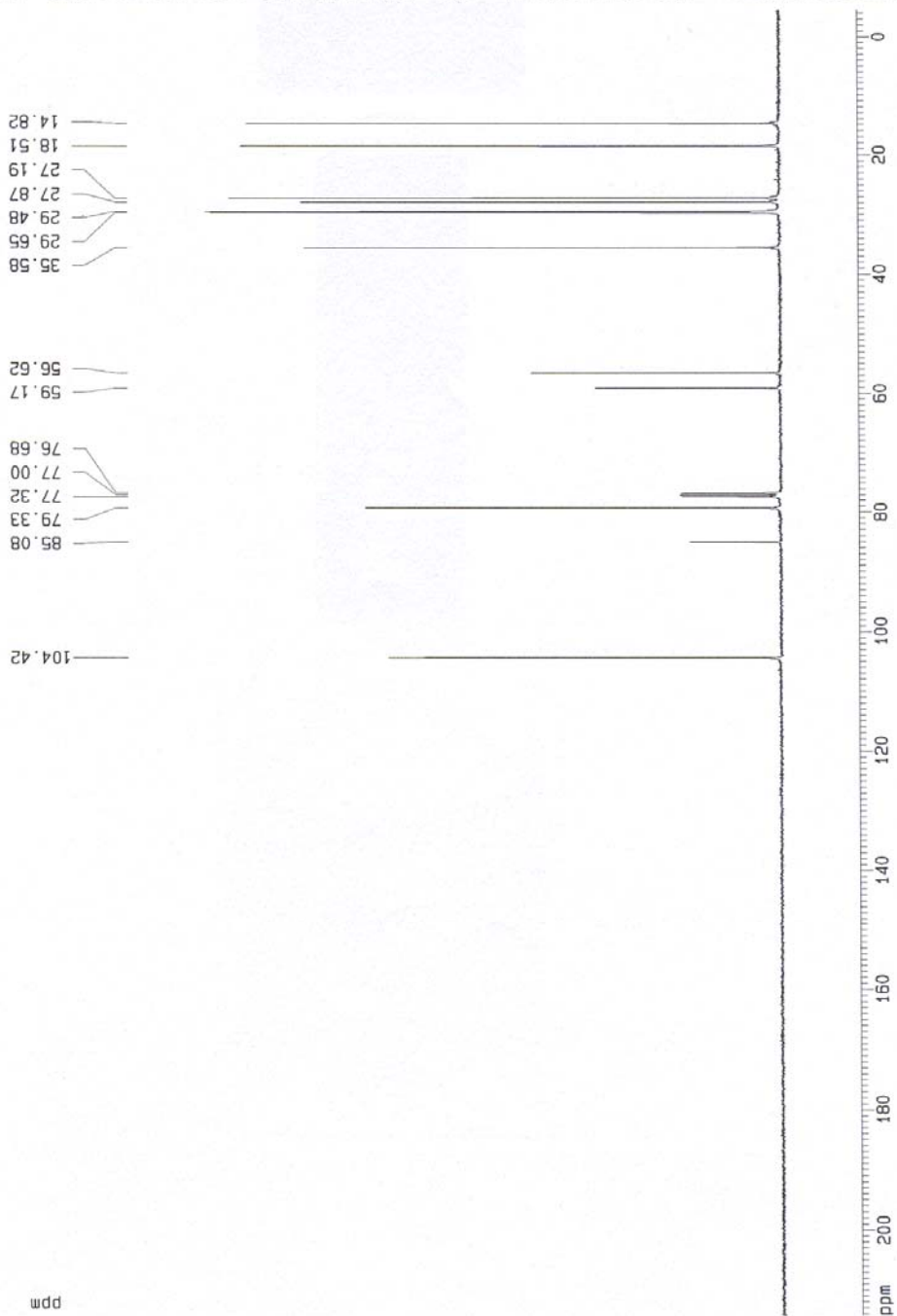


Current Data Parameters  
 NAME ed11.1.2351C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 980111  
 Time 22.55  
 PULPROG zg.cpd  
 SOLVENT CDCl3  
 AQ 1.0485960 sec  
 FIDRES 0.476837 Hz  
 DW 15.0 usec  
 RG 16384  
 NUCLEUS 13C  
 HL1 20 dB  
 D1 3.000000 sec  
 P31 100.0 usec  
 D2 0.0031000 sec  
 P1 7.0 usec  
 DE 22.9 usec  
 SF01 100.6230000 MHz  
 SWH 31250.00 Hz  
 TD 65536  
 NS 578  
 DS 2

F1 - Processing parameters  
 SI 131072  
 MC2 GF  
 SF 100.6138780 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters  
 CX 21.95 cm  
 F1P 214.291 ppm  
 F1 21560.60 Hz  
 F2P -4.498 ppm  
 F2 -452.60 Hz  
 PPMCM 9.96662 ppm/c  
 HZCM 1002.78021 Hz/cm

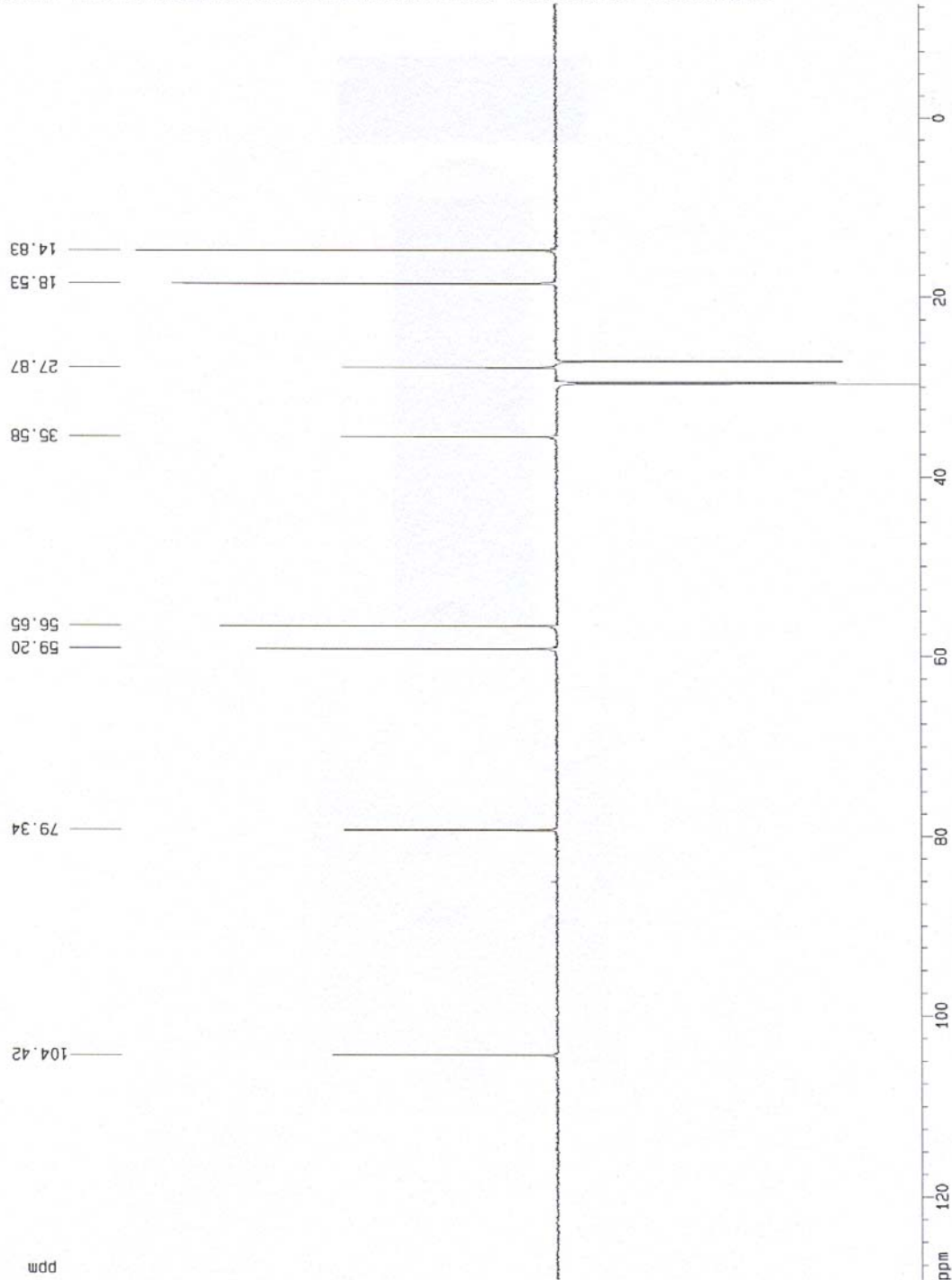


Current Data Parameters  
 NAME ed11.1.2510  
 EXPNO 1  
 PROCNO 1

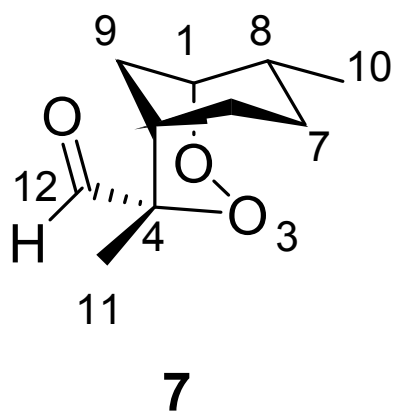
F2 - Acquisition Parameters  
 Date 980111  
 Time 23.51  
 PULPROG dept135  
 SOLVENT DMS  
 AQ 1.048560 sec  
 FIDRES 0.476837 Hz  
 DM 16.0 usec  
 RG 32768  
 NUCLEUS 13C  
 HL1 1 dB  
 D1 2.000000 sec  
 S1 1 dB  
 P3 10.3 usec  
 SF02 400.1350000 MHz  
 D2 0.0035714 sec  
 P4 20.6 usec  
 P1 6.2 usec  
 P2 12.4 usec  
 S2 23 dB  
 DE 22.9 usec  
 SF01 100.6223697 MHz  
 SWH 31250.00 Hz  
 TD 65536  
 P31 100.0 usec  
 NS 329  
 DS 4

F1 - Processing parameters  
 SI 131072  
 MC2 GF  
 SF 100.6138765 MHz  
 XCN EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters  
 CX 23.10 cm  
 F1P 129.134 ppm  
 F1 12992.65 Hz  
 F2P -12.765 ppm  
 F2 -1284.70 Hz  
 PPM0H 6.14298 ppm/cm  
 HZ0H 618.05575 Hz/cm



Saturated aldehyde **7**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

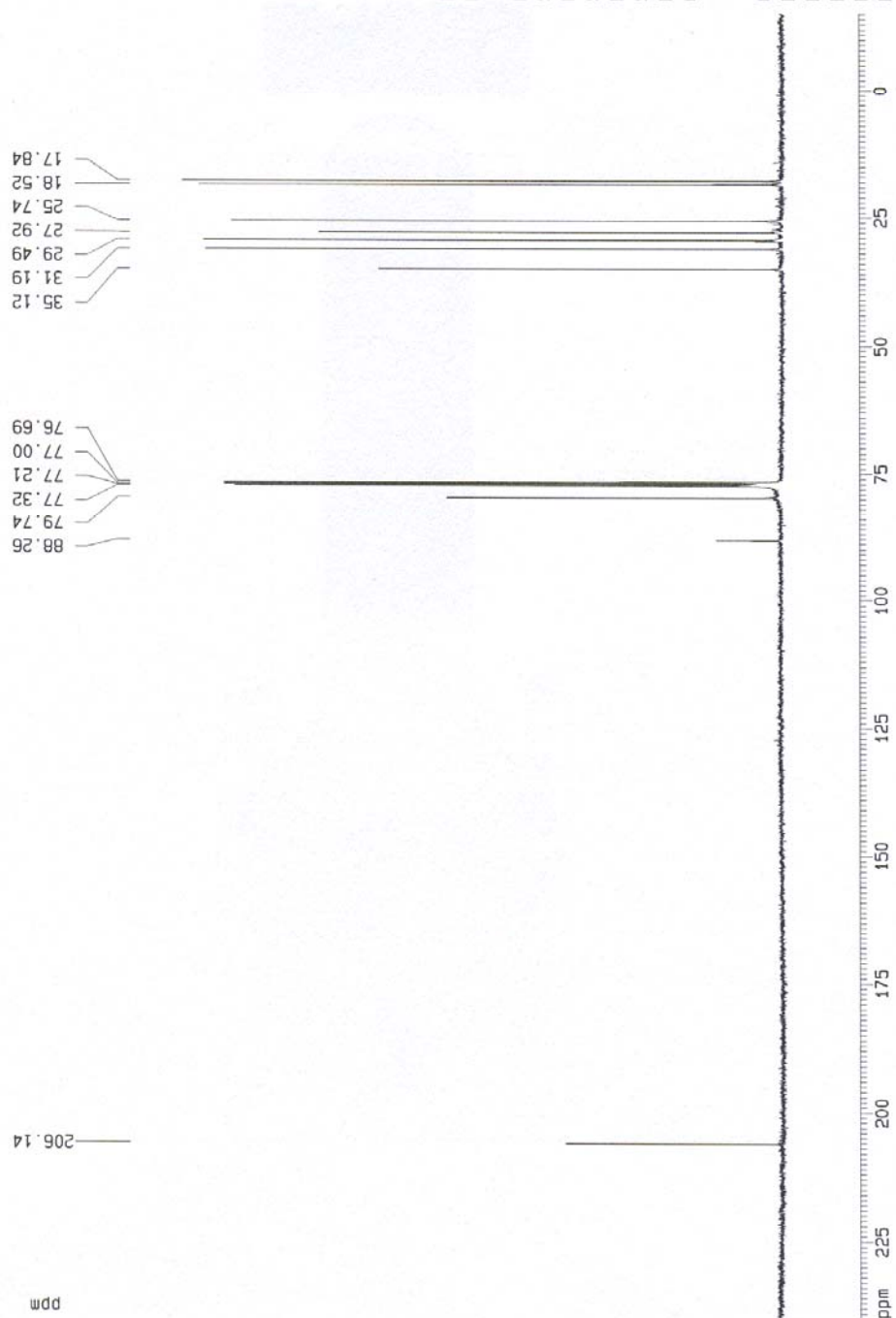


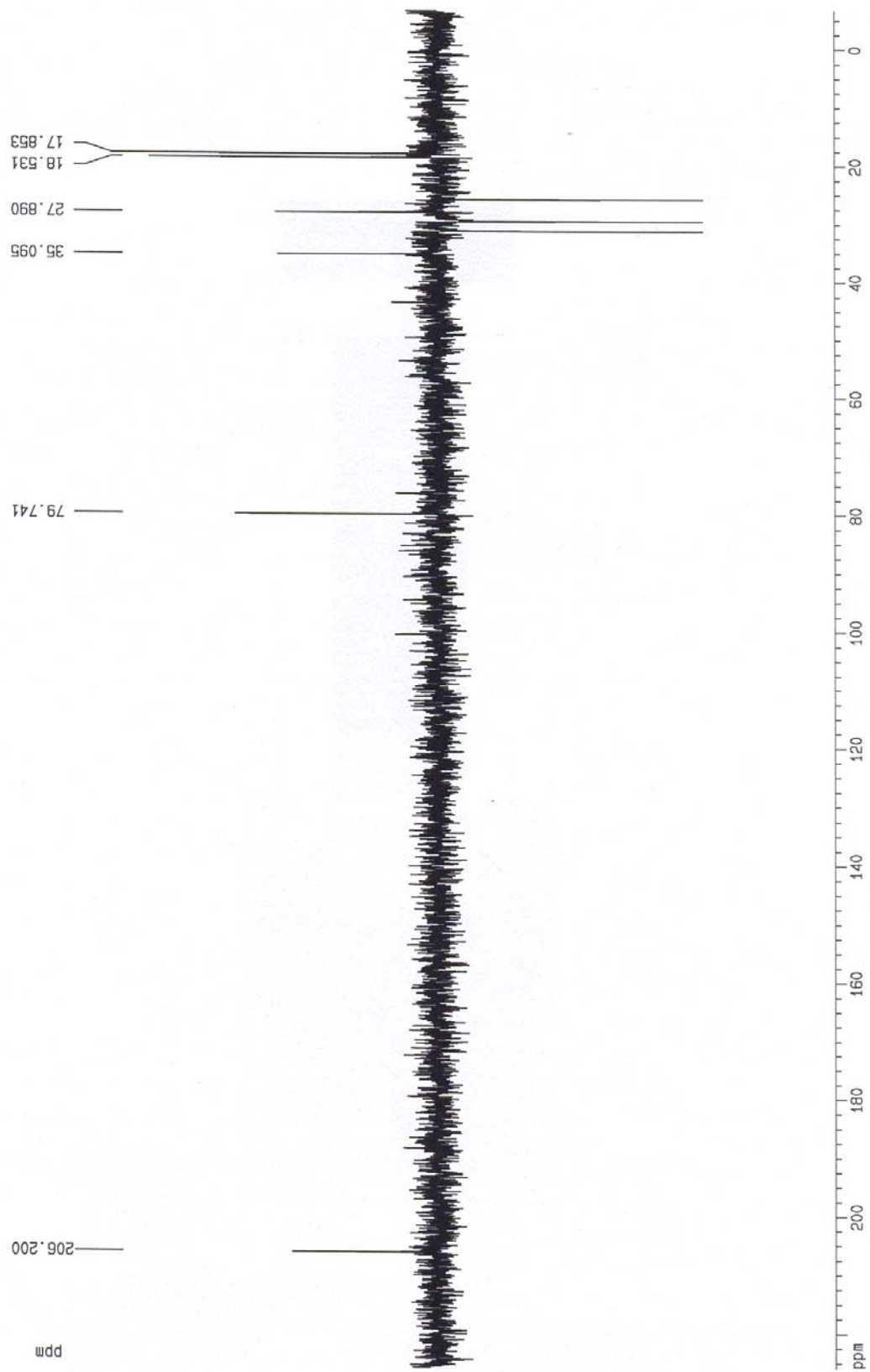
Current Data Parameters  
NAME ed11.1.2361C  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date 980112  
Time 3.18  
PULPROG zg.cpd  
SOLVENT COC13  
AQ 1.0485960 sec  
FIDRES 0.476837 Hz  
DQ 15.0 usec  
RG 16384  
NUCLEUS 13C  
HL1 20 dB  
D1 3.0000000 sec  
P31 100.0 usec  
D2 0.0031000 sec  
P1 7.0 usec  
DE 22.9 usec  
SF01 100.6230000 MHz  
SWH 31250.00 Hz  
TD 65536  
NS 4756  
DS 2

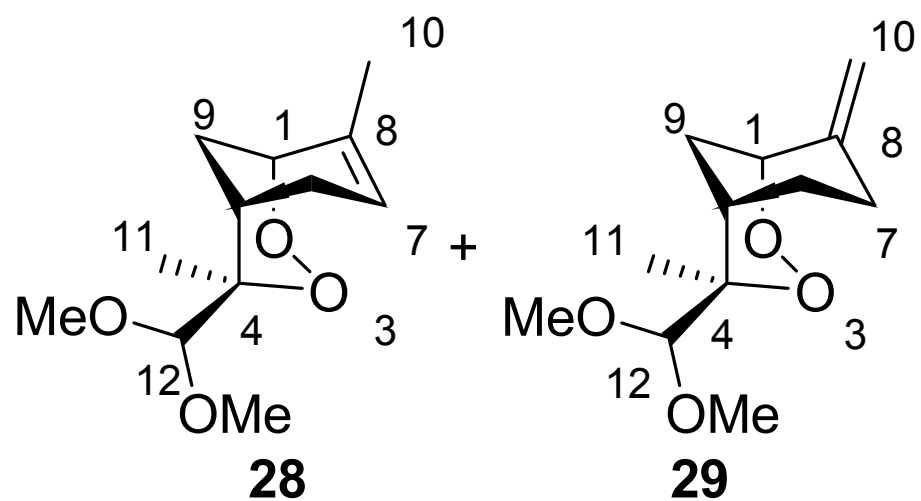
F1 - Processing parameters  
SI 131072  
MC2 GF  
SF 100.6138732 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0

1D NMR plot parameters  
CX 21.95 cm  
F1P 239.917 ppm  
F1 24139.01 Hz  
F2P -15.104 ppm  
F2 -1519.69 Hz  
PPMCM 11.61714 ppm/c  
HZCM 1168.84558 Hz/cm





Mixture of unsaturated acetals **28** and **29**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

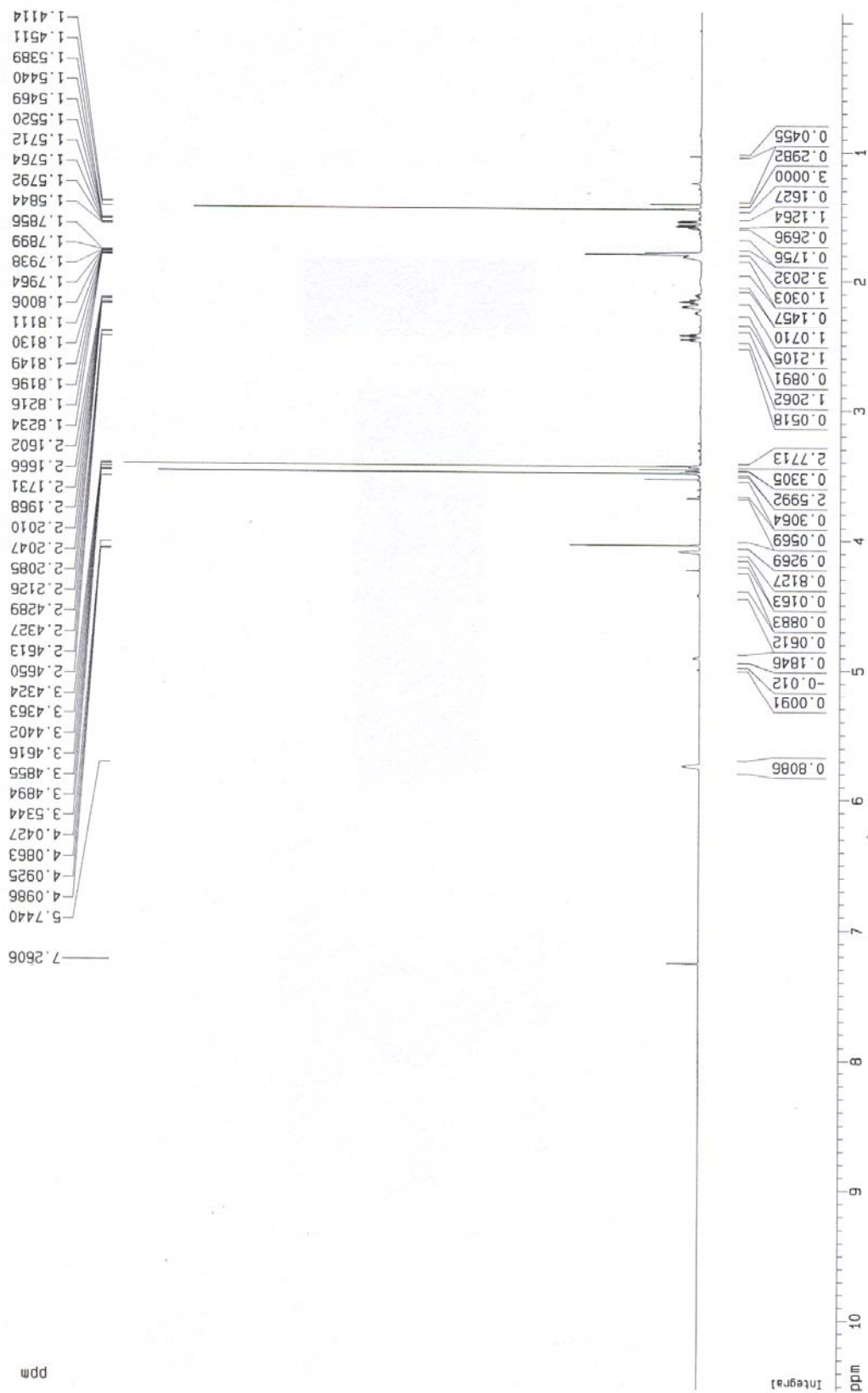
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra





Current Data Parameters  
 NAME ed11.1.2321C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

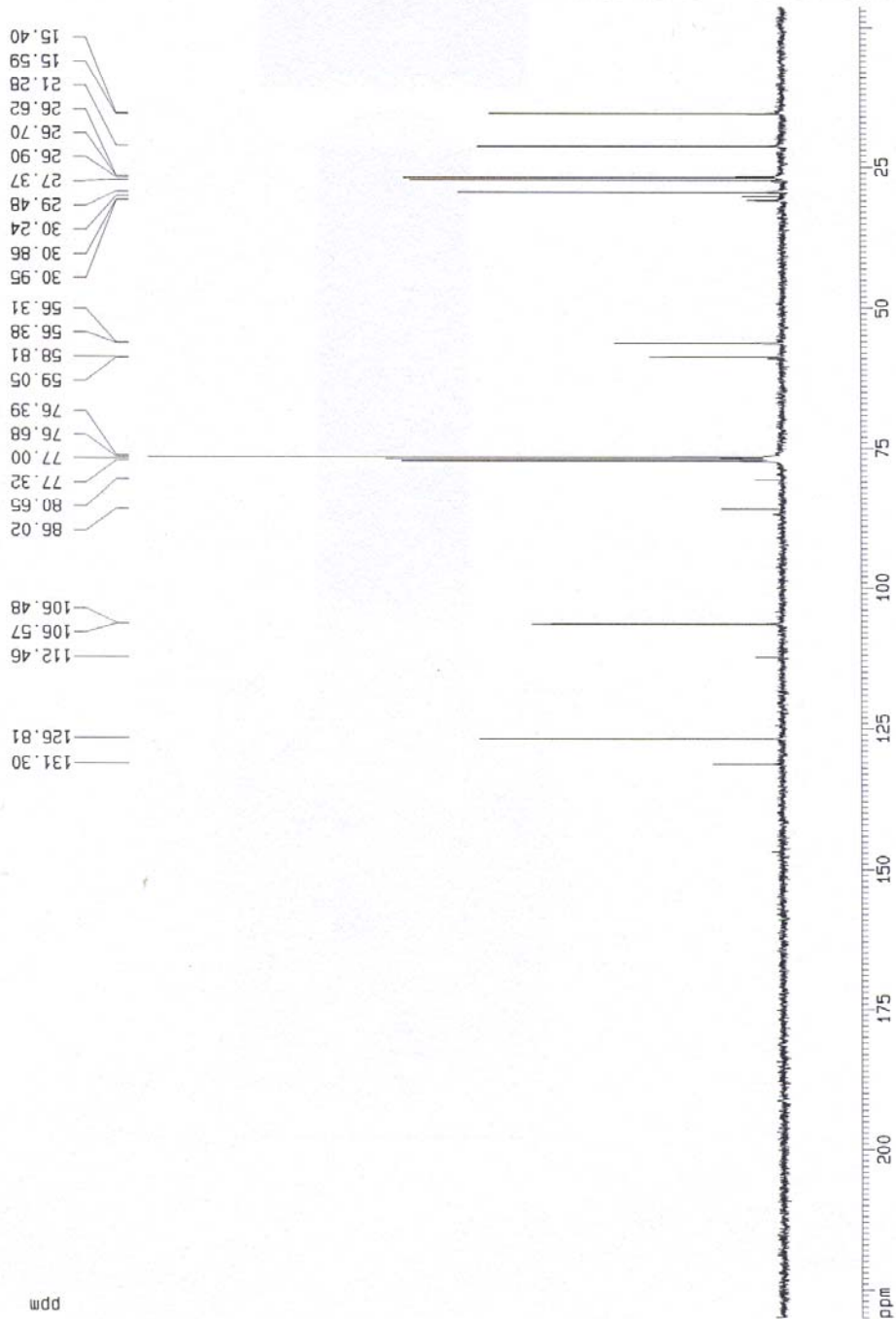
Date 980111  
 Time 17.01  
 PULPROG zg.cpd  
 SOLVENT CDC13  
 AQ 1.0485960 sec  
 FIDRES 0.476837 Hz  
 DW 16.0 usec  
 RG 16384  
 NUCLEUS 13C  
 HL1 20 dB  
 D1 3.0000000 sec  
 P31 100.0 usec  
 D2 0.0031000 sec  
 P1 7.0 usec  
 DE 22.9 usec  
 SF01 100.6230000 MHz  
 SWH 31250.00 Hz  
 TD 65536  
 NS 1093  
 DS 2

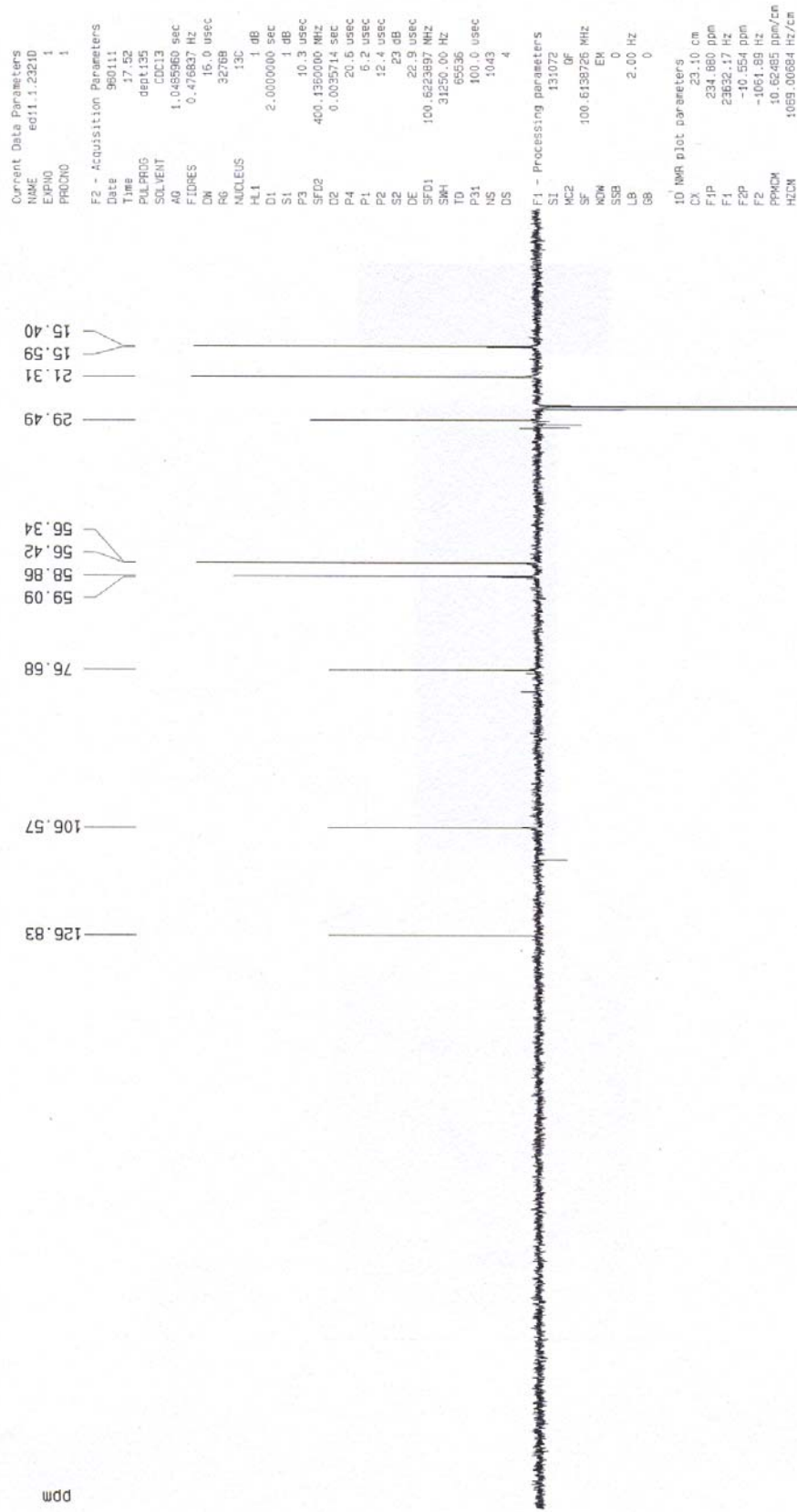
F1 - Processing parameters

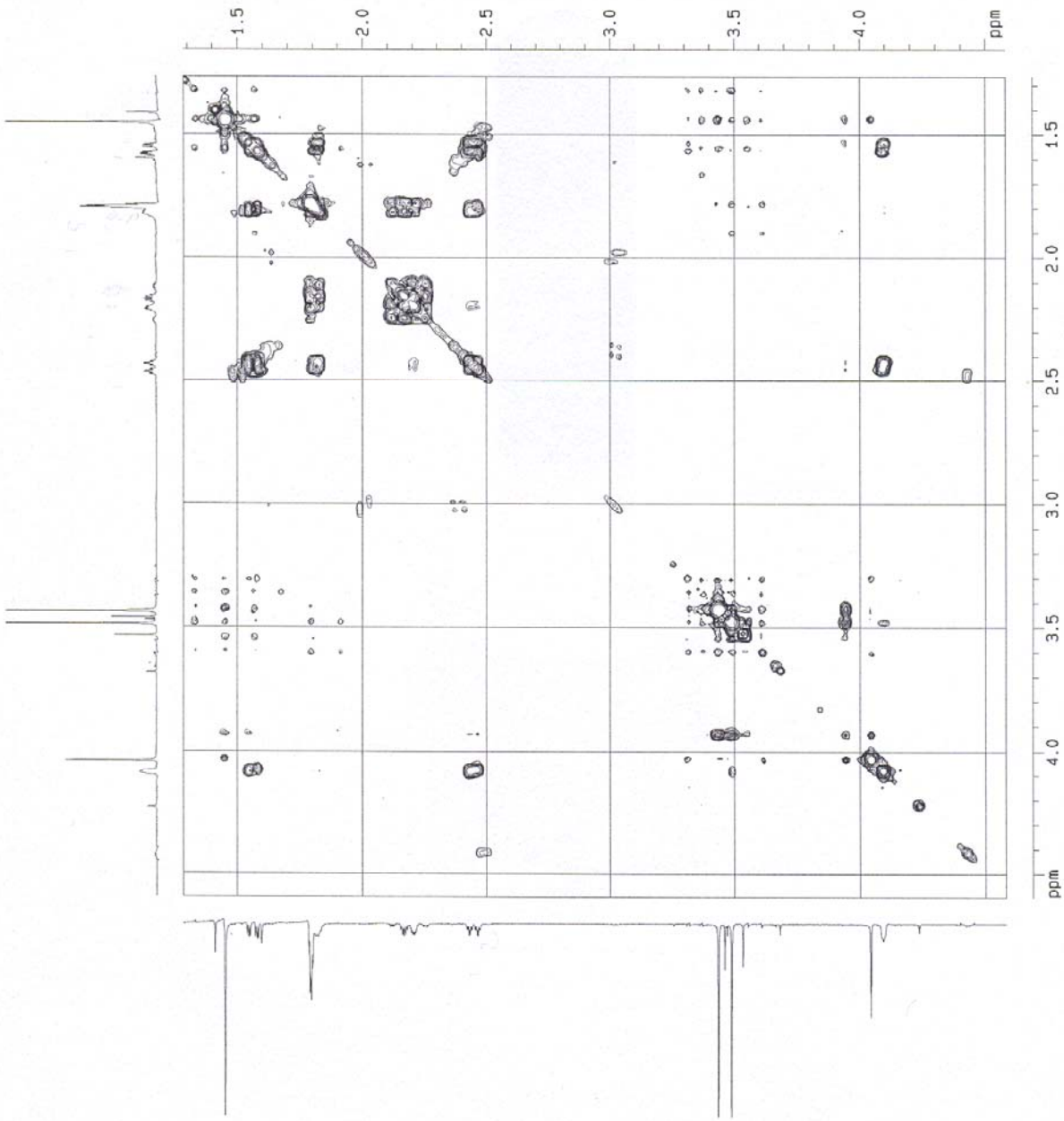
SI 131072  
 MC2 GF  
 SF 100.6138740 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters

CX 21.95 cm  
 F1P 230.014 ppm  
 F1 23142.59 Hz  
 F2P -3.692 ppm  
 F2 -371.51 Hz  
 PPMCM 10.54616 ppm/c  
 HZCM 1071.15100 Hz/cm







Current Data Parameters  
 NAME ed11.1.231Co  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 980111  
 Time 18:47  
 PULPROG zgpg30  
 SOLVENT ccd13  
 AQ 0.2078520 sec  
 FIDRES 2.405326 Hz  
 DM 203.0 usec  
 RG 256  
 NUC1 1H  
 NUC2 13C  
 H1 1.000000 sec  
 D1 12.6 usec  
 P1 0.000030 sec  
 DE 299.0 usec  
 SF01 400.1363721 MHz  
 SWH 2463.05 Hz  
 TD 1024  
 NS 4  
 DS 4  
 INO 0.0004050 sec

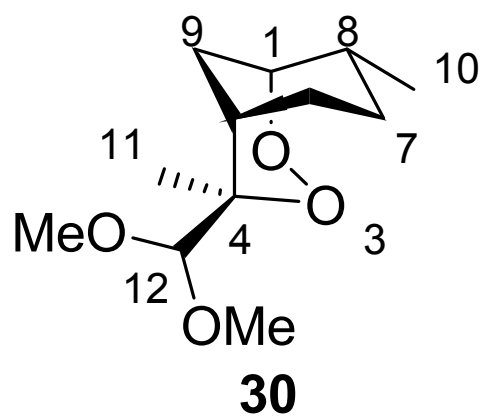
F1 - Acquisition Parameters  
 NS 1  
 TD 256  
 SF01 400.136 MHz  
 FIDRES 9.621238 Hz  
 SN 5.155 sps

F1 - Processing parameters  
 SI 512  
 MC2 0F  
 SF 400.1343951 MHz  
 KW STINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

F1 - Processing parameters  
 SI 512  
 MC2 0F  
 SF 400.1343943 MHz  
 KW STINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CY1 15.00 cm  
 F2PL0 4.592 ppm  
 F2PL1 1837.54 Hz  
 F2PH1 1.262 ppm  
 F2H1 504.98 Hz  
 F1PL0 4.590 ppm  
 F1PL1 1832.74 Hz  
 F1PH1 1.266 ppm  
 F1H1 514.63 Hz  
 F2PPMCM 0.22202 ppm/cm  
 F2HZCM 88.83881 Hz/cm  
 F1PPMCM 0.21961 ppm/cm  
 F1HZCM 87.87407 Hz/cm

Saturated acetal **30**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ):

$^1\text{H}$  NMR spectra (in  $\text{C}_6\text{D}_6$ )

$^{13}\text{C}$  NMR spectra (in  $\text{C}_6\text{D}_6$ )

DEPT spectra (in  $\text{CDCl}_3$ )

COSY spectra (in  $\text{C}_6\text{D}_6$ )

HMQC spectra (in  $\text{C}_6\text{D}_6$ )

Current Data Parameters  
 NAME e3.12.435A1.h  
 EXPNO 2  
 PROCNO 1

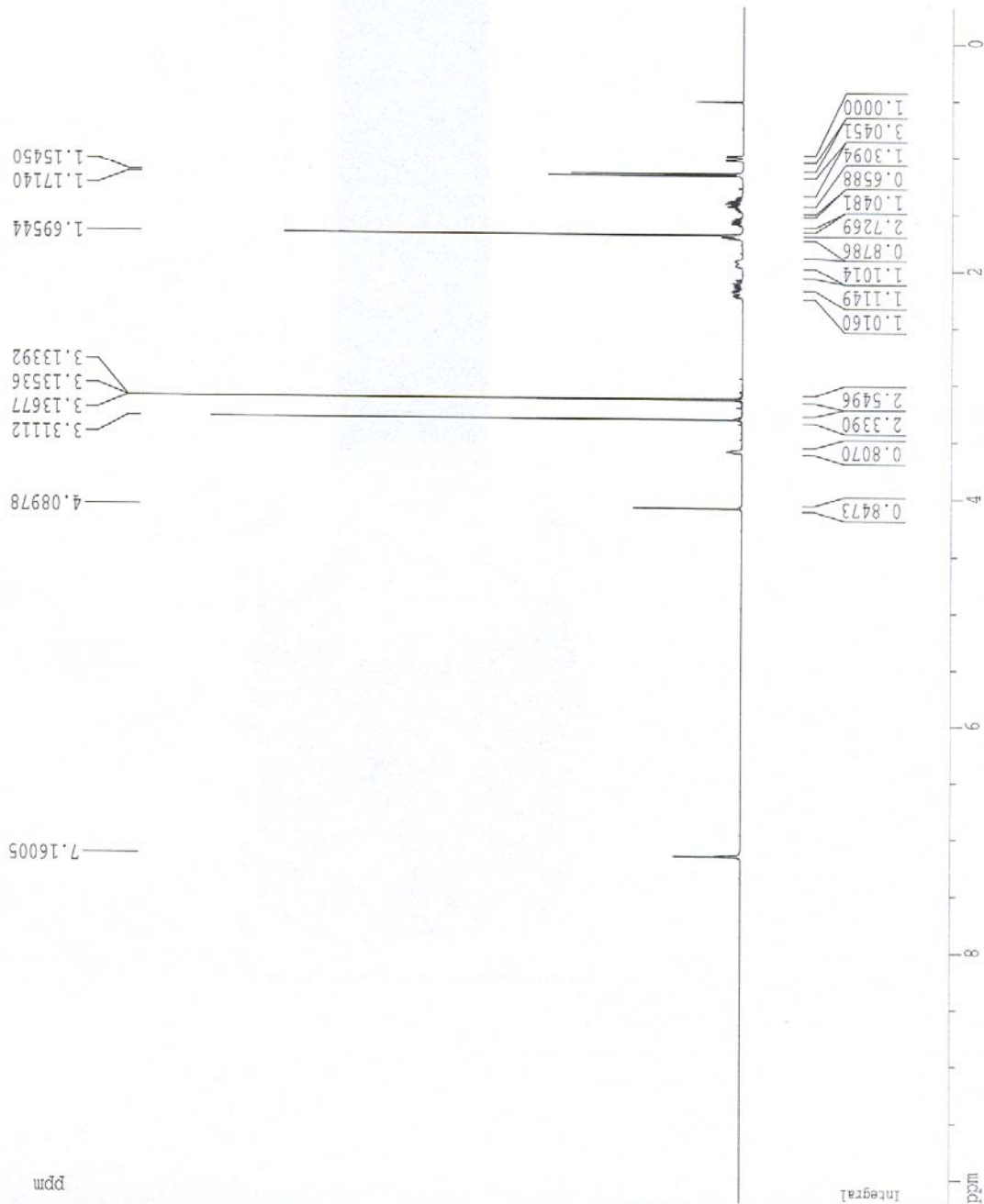
F2 - Acquisition Parameters  
 Date\_ 20020312  
 Time 17.19  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT C6D6  
 NS 250  
 DS 0  
 SMH 8012.820 Hz  
 FIDRES 0.244532 Hz  
 AQ 2.0447731 sec  
 RG 101.6  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.50000000 sec

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

NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SF01 400.1320007 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300443 MHz  
 WDM EM  
 SSB 0  
 LB 0.01 Hz  
 GB 0  
 PC 0.40

1D NMR plot parameters  
 CX 20.00 cm  
 FIP 10.210 ppm  
 F1 4085.34 Hz  
 F2P -0.319 ppm  
 F2 -127.68 Hz  
 PPMCM 0.52646 ppm/cm  
 HZCM 210.65096 Hz/cm



Current Data Parameters  
NAME e3.12.435A1PC  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20020312  
Time 19:52  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 65536  
SOLVENT *C<sub>6</sub>D<sub>6</sub>*  
NS 2876  
DS 4  
SWH 30120.482 Hz  
FIDRES 0.459602 Hz  
AQ 1.0879476 sec  
RG 512  
DW 16.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 6.20 usec  
PL1 -6.00 dB  
SFO1 100.6227903 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 120.00 dB  
PL12 14.00 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127494 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 175.982 ppm  
F1 17706.04 Hz  
F2P -10.621 ppm  
F2 -1068.61 Hz  
PPMCM 9.33015 ppm/cm  
HZCM 938.73248 Hz/cm

16.084  
19.024  
26.933  
30.079  
30.811  
30.844  
35.874

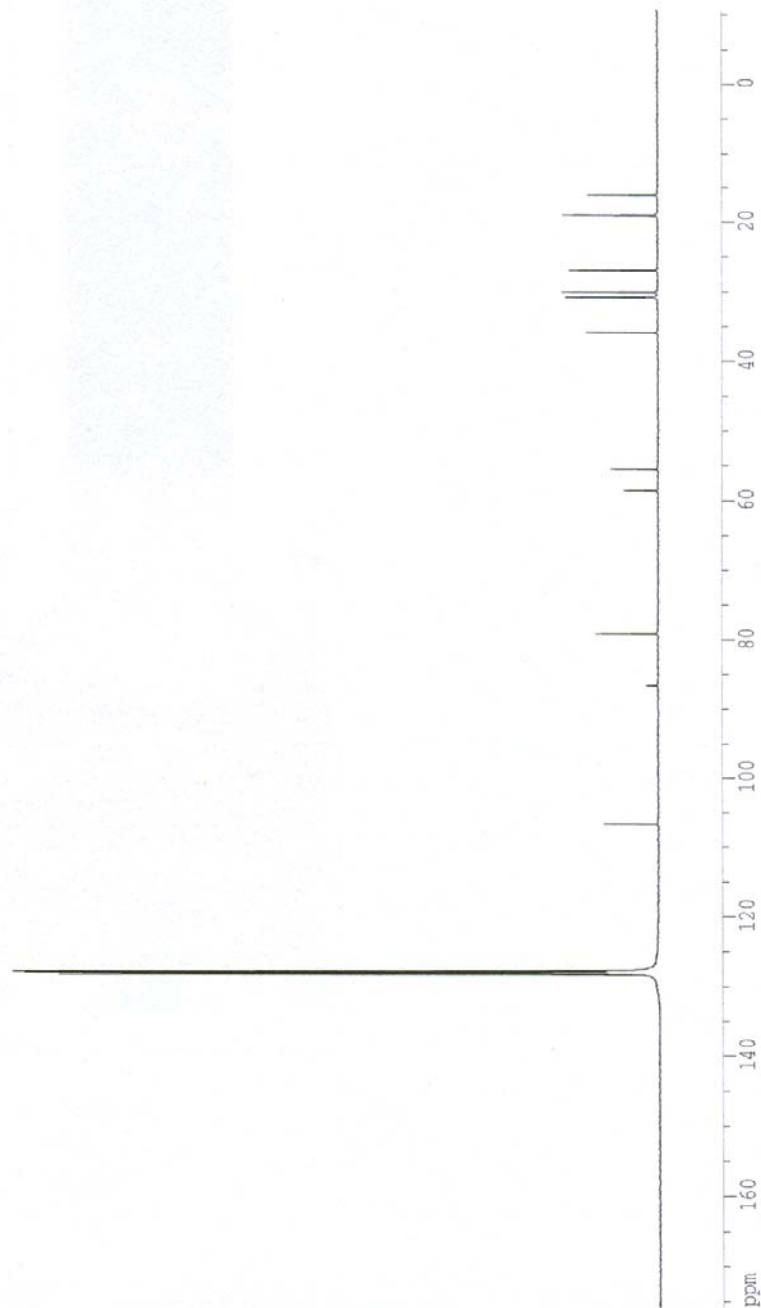
55.478  
58.629

79.145  
86.595

106.707

127.757  
127.884  
127.998  
128.239

ppm



Current Data Parameters  
 NAME e3.12.435A1.D  
 EXPNO 1  
 PROCNO 1

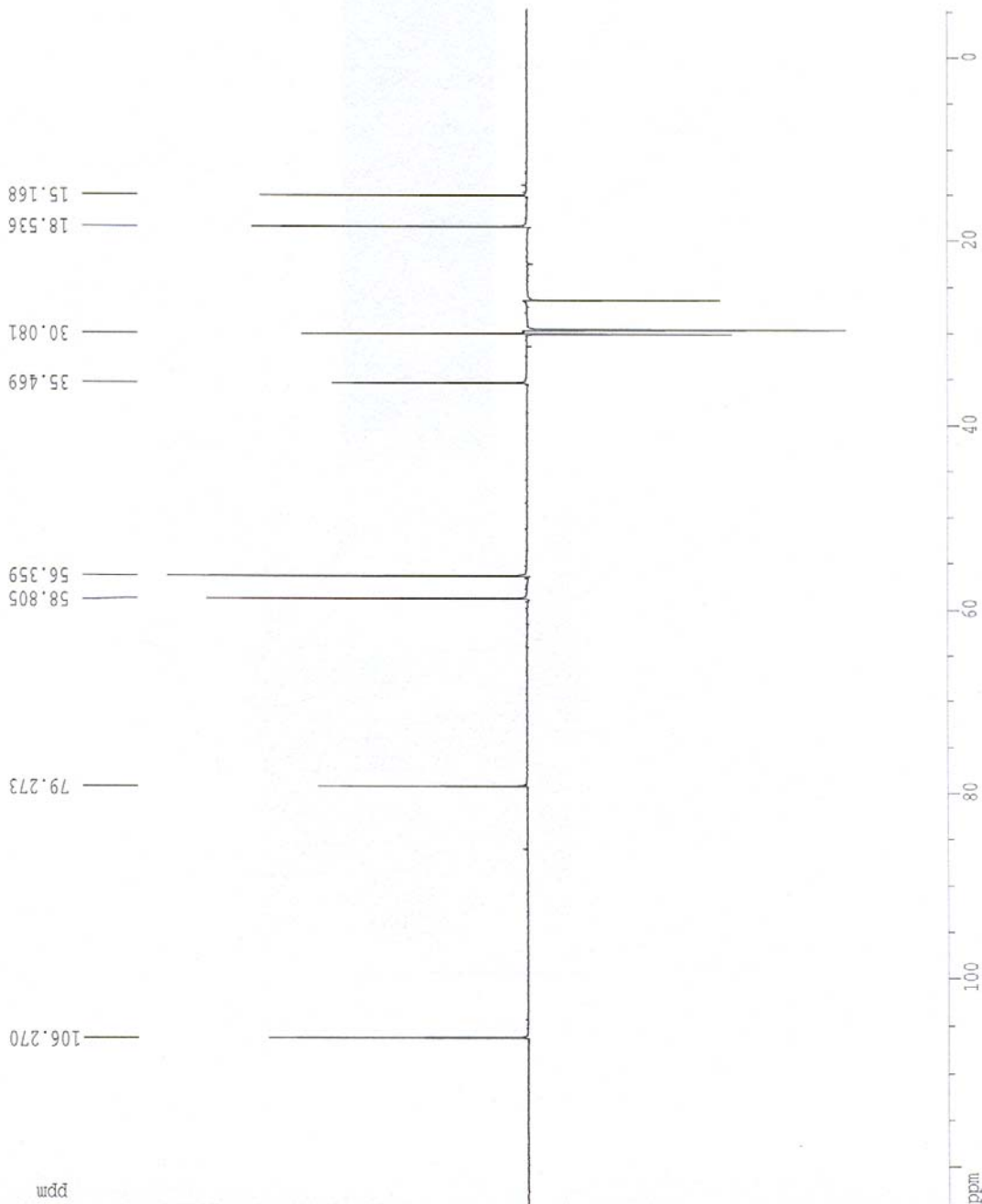
F2 - Acquisition Parameters  
 Date\_ 20020312  
 Time 16.54  
 INSTRUM spect  
 PROSD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 700  
 DS 8  
 SWH 25125.629 Hz  
 FIDRES 0.183387 Hz  
 AQ 1.3042164 sec  
 RG 14596.5  
 DW 19.900 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 D2 0.00345000 sec  
 D12 0.00002000 sec  
 DELTA 6366.18261719 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.20 usec  
 P2 12.40 usec  
 PL1 -6.00 dB  
 SF01 100.6227903 MHz

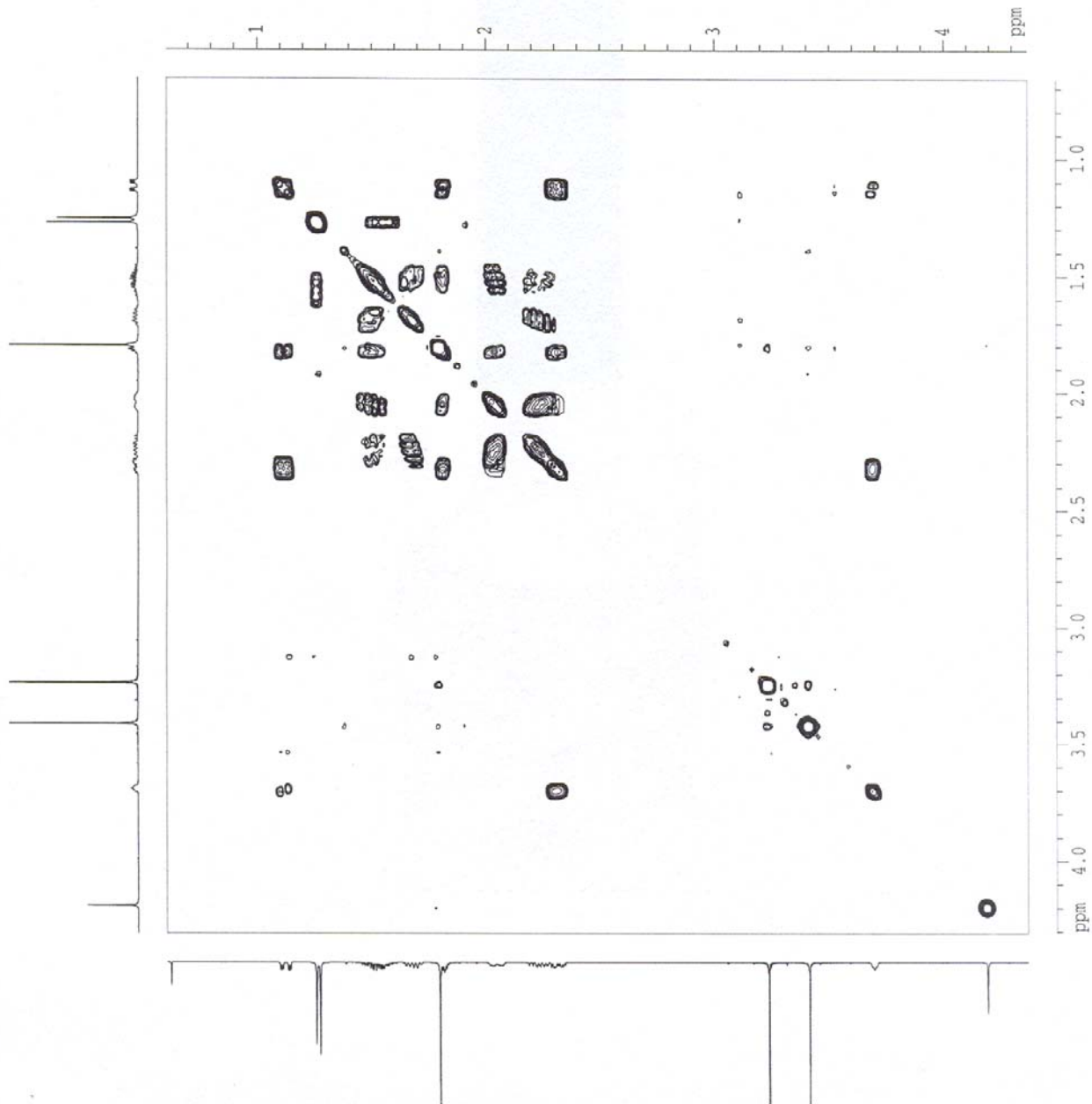
===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P3 13.05 usec  
 P4 26.10 usec  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 14.00 dB  
 SF02 400.1306003 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127784 MHz  
 MDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 124.146 ppm  
 F1 12490.67 Hz  
 F2P -5.196 ppm  
 F2 -522.83 Hz  
 PPMCM 6.46712 ppm/cm  
 HZCM 650.67493 Hz/cm







Current Data Parameters  
 NAME e3.12.4381.co  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20020312  
 Time 17.47  
 INSTRUM spect  
 PULPROG 5 mm QNP 1H  
 TD 1024  
 SOLVENT CDCl3  
 NS 12  
 DS 16  
 SWE 2185.315 Hz  
 FIDRES 2.134096 Hz  
 AQ 0.2343412 sec  
 ZG 228.1  
 DQ 228.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D0 0.00000000 sec  
 O1 1.00000000 sec  
 D1 0.00000000 sec  
 D2 0.00000000 sec  
 D3 0.00000000 sec  
 D4 0.00000000 sec  
 D5 0.00000000 sec  
 D6 0.00000000 sec  
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 D71 0.00000000 sec  
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 D94 0.00000000 sec  
 D95 0.00000000 sec  
 D96 0.00000000 sec  
 D97 0.00000000 sec  
 D98 0.00000000 sec  
 D99 0.00000000 sec  
 D100 0.00000000 sec

F1 - Acquisition Parameters

NAME e3.12.4381.co  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20020312  
 Time 17.47  
 INSTRUM spect  
 PULPROG 5 mm QNP 1H  
 TD 1024  
 SOLVENT CDCl3  
 NS 12  
 DS 16  
 SWE 2185.315 Hz  
 FIDRES 2.134096 Hz  
 AQ 0.2343412 sec  
 ZG 228.1  
 DQ 228.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D0 0.00000000 sec  
 O1 1.00000000 sec  
 D1 0.00000000 sec  
 D2 0.00000000 sec  
 D3 0.00000000 sec  
 D4 0.00000000 sec  
 D5 0.00000000 sec  
 D6 0.00000000 sec  
 D7 0.00000000 sec  
 D8 0.00000000 sec  
 D9 0.00000000 sec  
 D10 0.00000000 sec  
 D11 0.00000000 sec  
 D12 0.00000000 sec  
 D13 0.00000000 sec  
 D14 0.00000000 sec  
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 D16 0.00000000 sec  
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 D91 0.00000000 sec  
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 D95 0.00000000 sec  
 D96 0.00000000 sec  
 D97 0.00000000 sec  
 D98 0.00000000 sec  
 D99 0.00000000 sec  
 D100 0.00000000 sec

F2 - Processing Parameters

SI 512  
 SF 400.1300004 MHz  
 WDW QF  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing Parameters

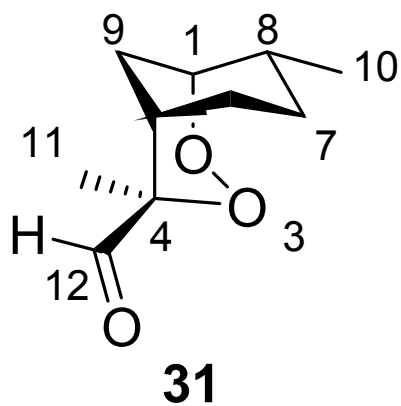
SI 512  
 SF 400.1300004 MHz  
 WDW QF  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

2D NMR plot parameters

CT2 15.00 cm  
 CT1 15.00 cm  
 F2LO 1731.24 Hz  
 F2HI 261.54 Hz  
 F2F0 4.349 ppm  
 F2F1 1744.13 Hz  
 F2F2 0.603 ppm  
 F2F3 241.46 Hz  
 F2F4 0.24321 ppm/cm  
 F2F5 97.31480 Hz/cm  
 F2F6 0.25103 ppm/cm  
 F2F7 100.44479 Hz/cm



Saturated aldehyde **31**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

Current Data Parameters  
 NAME as2.25a150300H  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20000315  
 Time 17.27  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 72  
 DS 0  
 SMH 8012.820 Hz  
 FIDRES 0.244532 Hz  
 AQ 2.0447731 sec  
 RG 228.1  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.0000000 sec

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

NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SFO1 400.1320007 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300040 MHz  
 WDW EM  
 SSB 0  
 LB 0.01 Hz  
 GB 0  
 PC 0.40

1D NMR plot parameters  
 CX 20.00 cm  
 FIP 10.000 ppm  
 F1 4001.30 Hz  
 F2P -0.100 ppm  
 F2 -40.01 Hz  
 PPMCM 0.50500 ppm/cm  
 HZCM 202.06566 Hz/cm

1.59486  
 1.46111  
 1.10613  
 1.08993

9.67632  
 ppm

1.0456  
 0.8550  
 3.0559  
 2.7872  
 1.1542  
 2.9000  
 3.1113

0.7340

Integral  
 0.5501



Current Data Parameters  
 NAME as2.25a150300C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20000315  
 Time 18.05  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 900  
 DS 4  
 SNH 30120.482 Hz  
 FIDRES 0.459602 Hz  
 AQ 1.0879476 sec  
 RG 2896.3  
 DW 16.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec

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

NUC1 13C  
 P1 6.20 usec  
 PL1 -6.00 dB  
 SFO1 100.6227903 MHz

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

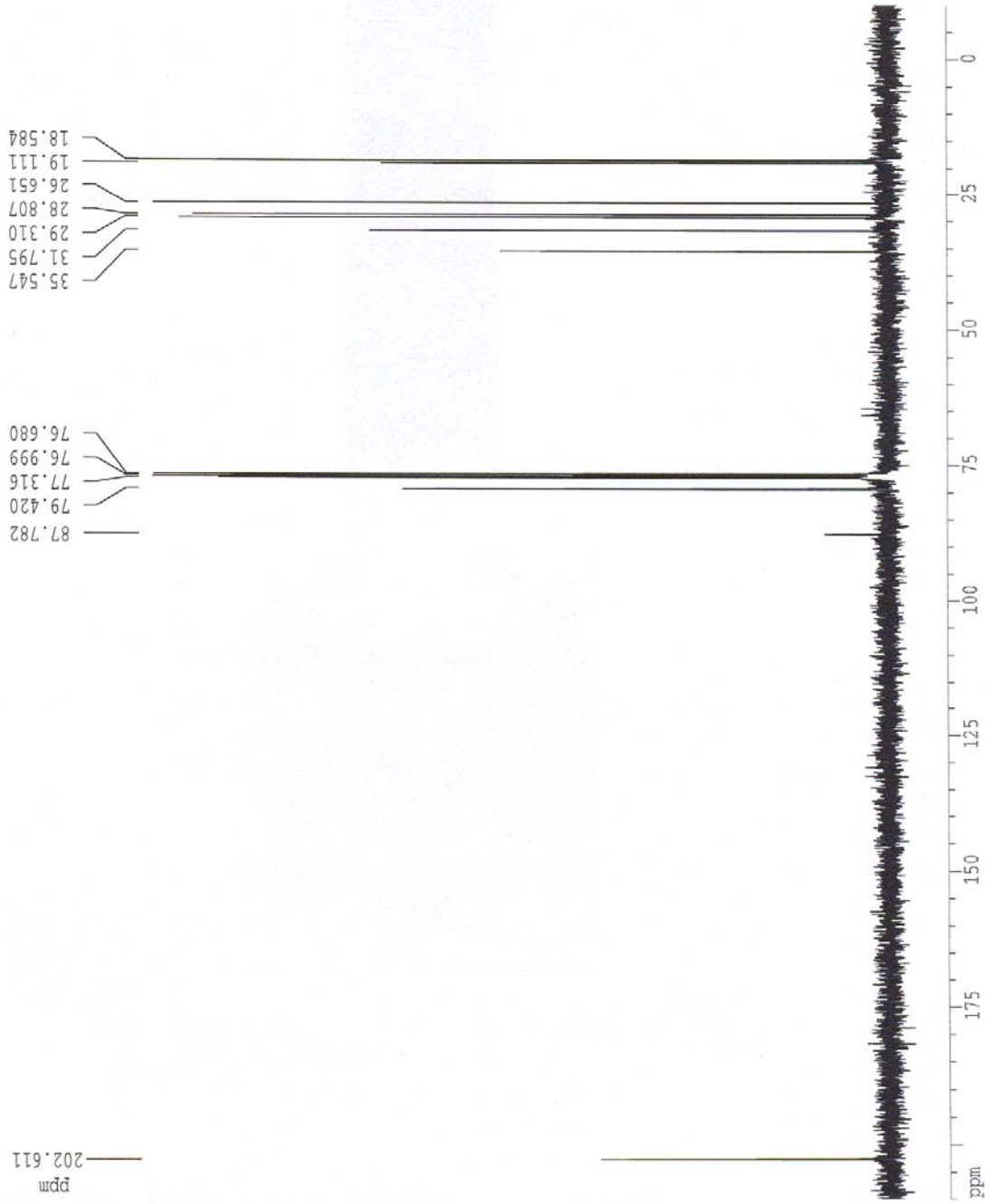
CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 120.00 dB  
 PL12 14.00 dB  
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768  
 SF 100.6127717 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters

CX 20.00 cm  
 F1P 210.000 ppm  
 F1 21128.68 Hz  
 F2P -10.000 ppm  
 F2 -1006.13 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 1106.74048 Hz/cm



Current Data Parameters  
NAME as2.25a150300D  
EXPNO 1  
PROCNO 1

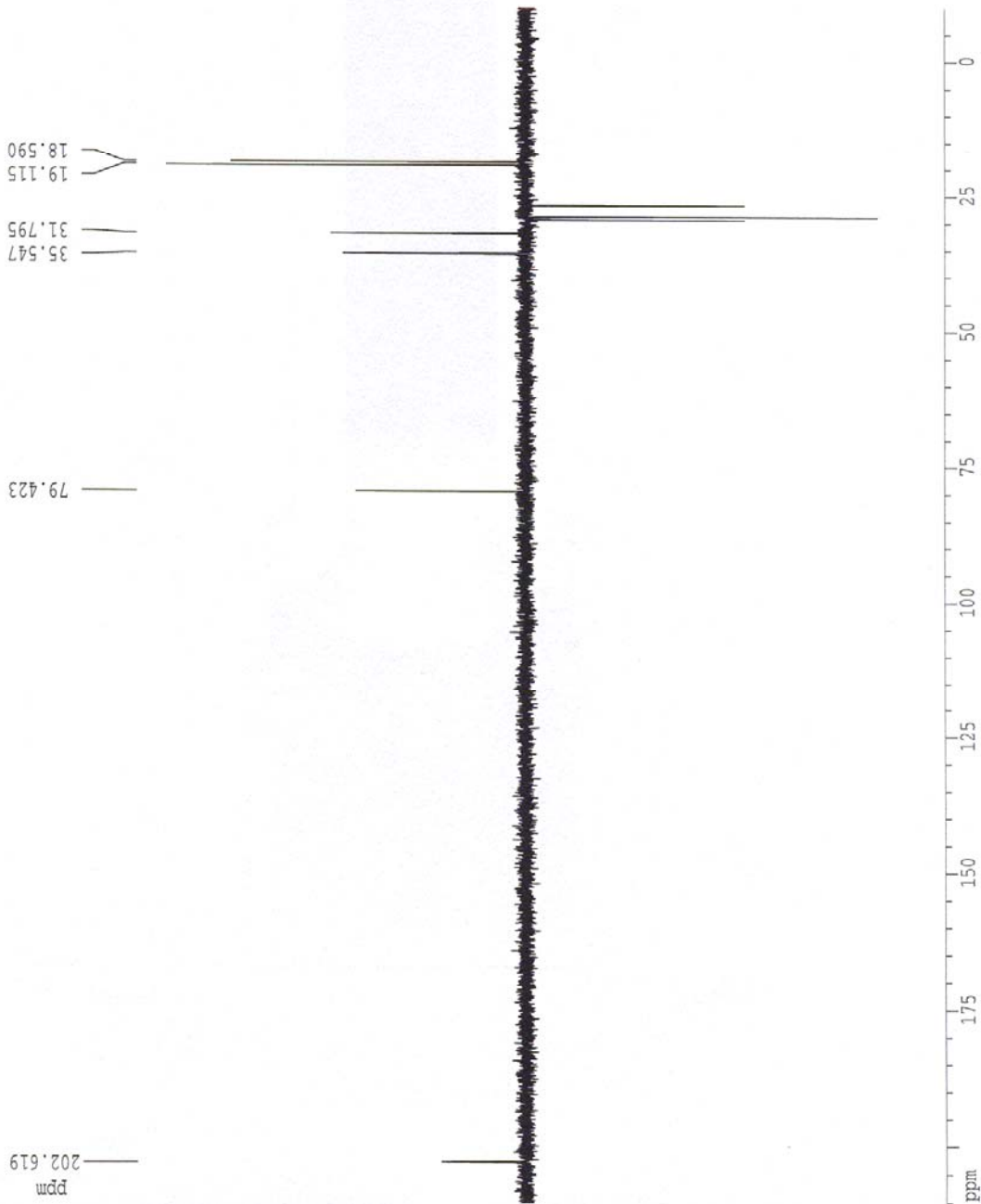
F2 - Acquisition Parameters  
Date\_ 20000315  
Time 18.29  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 450  
DS 8  
SWH 25125.629 Hz  
FIDRES 0.383387 Hz  
AQ 1.3042164 sec  
RG 11585.2  
DW 19.900 usec  
DE 6.00 usec  
TE 300.0 K  
D1 3.00000000 sec  
D2 0.00345000 sec  
D12 0.0002000 sec  
DELTA 6366.18261719 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 6.20 usec  
PL1 12.40 usec  
SFO1 100.627903 MHz

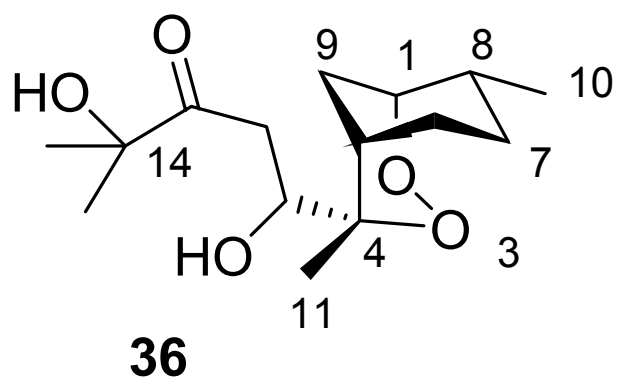
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
P2 13.05 usec  
PL2 26.10 usec  
SFO2 400.1306003 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127715 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
FLP 210.000 ppm  
F1 21128.68 Hz  
F2 -100.00 ppm  
F2 -1006.13 Hz  
PPM/M 11.00000 ppm/cm  
HZCM 1106.74043 Hz/cm



Aldol adduct **36**:

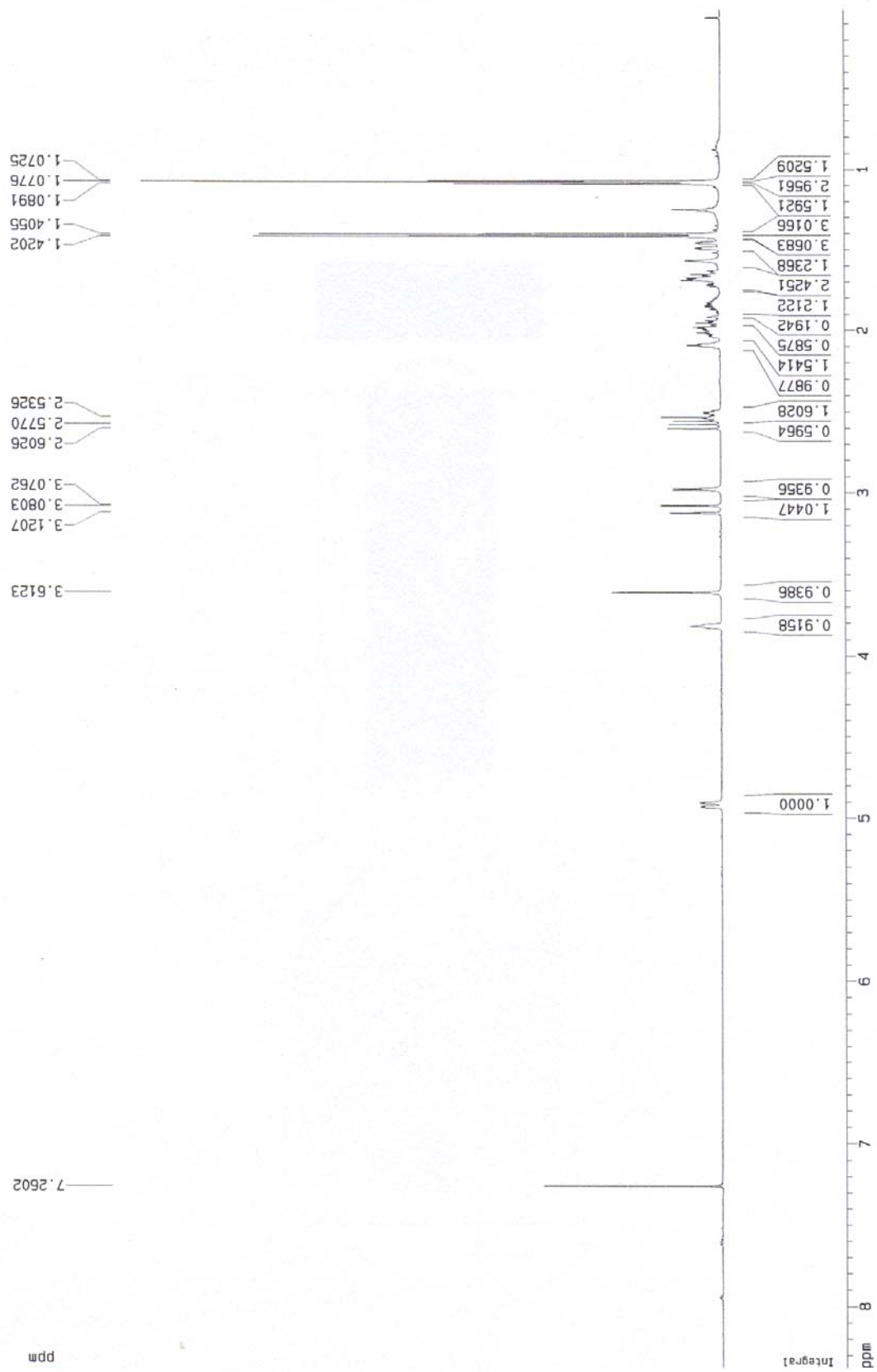


Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

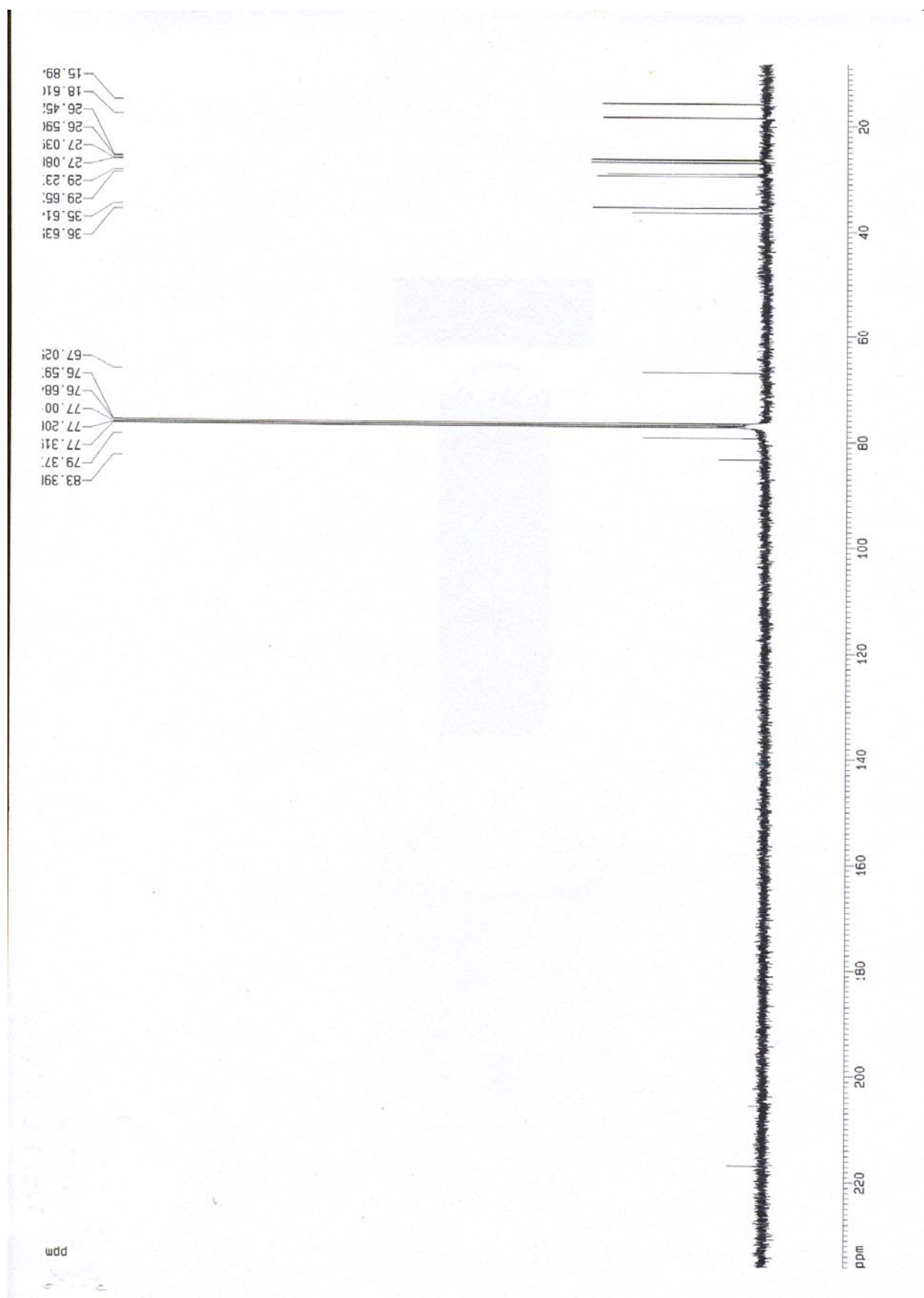
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra







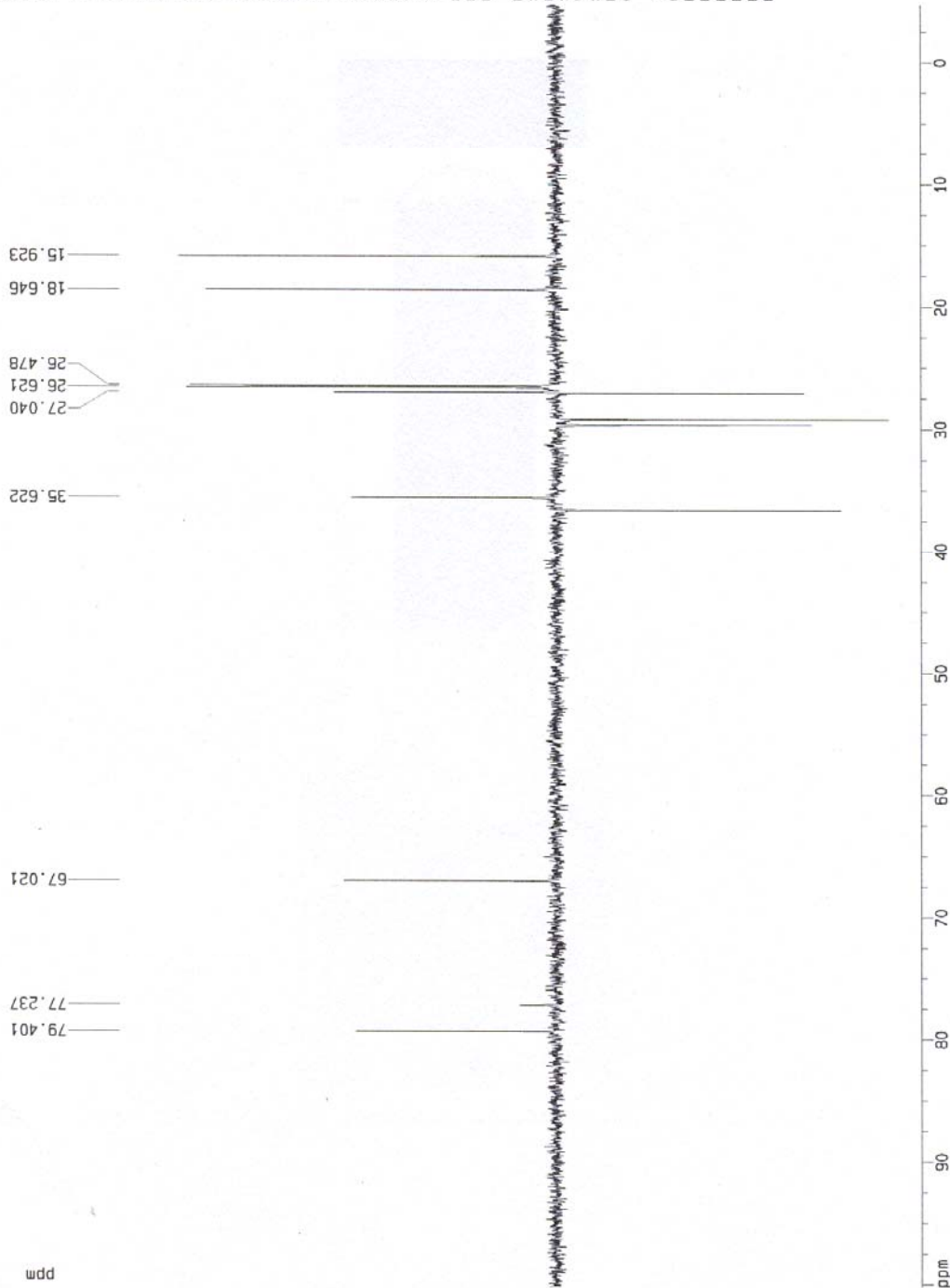
Current Data Parameters  
 NAME ed15.3.25510  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 990316  
 Time 7.16  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 AQ 1.048560 sec  
 FIDRES 0.476837 Hz  
 DN 16.0 usec  
 RG 32768  
 NUC1 13C

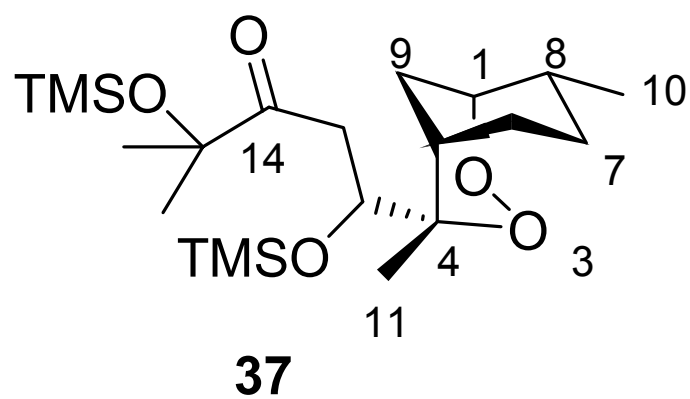
HL1 1 dB  
 D1 2.000000 sec  
 S1 1 dB  
 P3 19.3 usec  
 SF02 400.136000 MHz  
 C2 0.0035714 sec  
 P4 20.6 usec  
 P1 6.2 usec  
 P2 12.4 usec  
 S2 23 dB  
 DE 22.9 usec  
 SF01 100.622387 MHz  
 SWH 31250.00 Hz  
 TO 65536  
 P31 100.0 usec  
 NS 5669  
 DS 4

F1 - Processing parameters  
 SI 131072  
 SF 100.6138710 MHz  
 DE  
 EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters  
 CX 23.10 cm  
 FIP 100.233 ppm  
 F1 10084.79 Hz  
 F2 -4.747 ppm  
 F2 -477.57 Hz  
 PPMON 4.54455 ppm/cm  
 NDCM 457.24478 Hz/cm



Aldol adduct **37**:

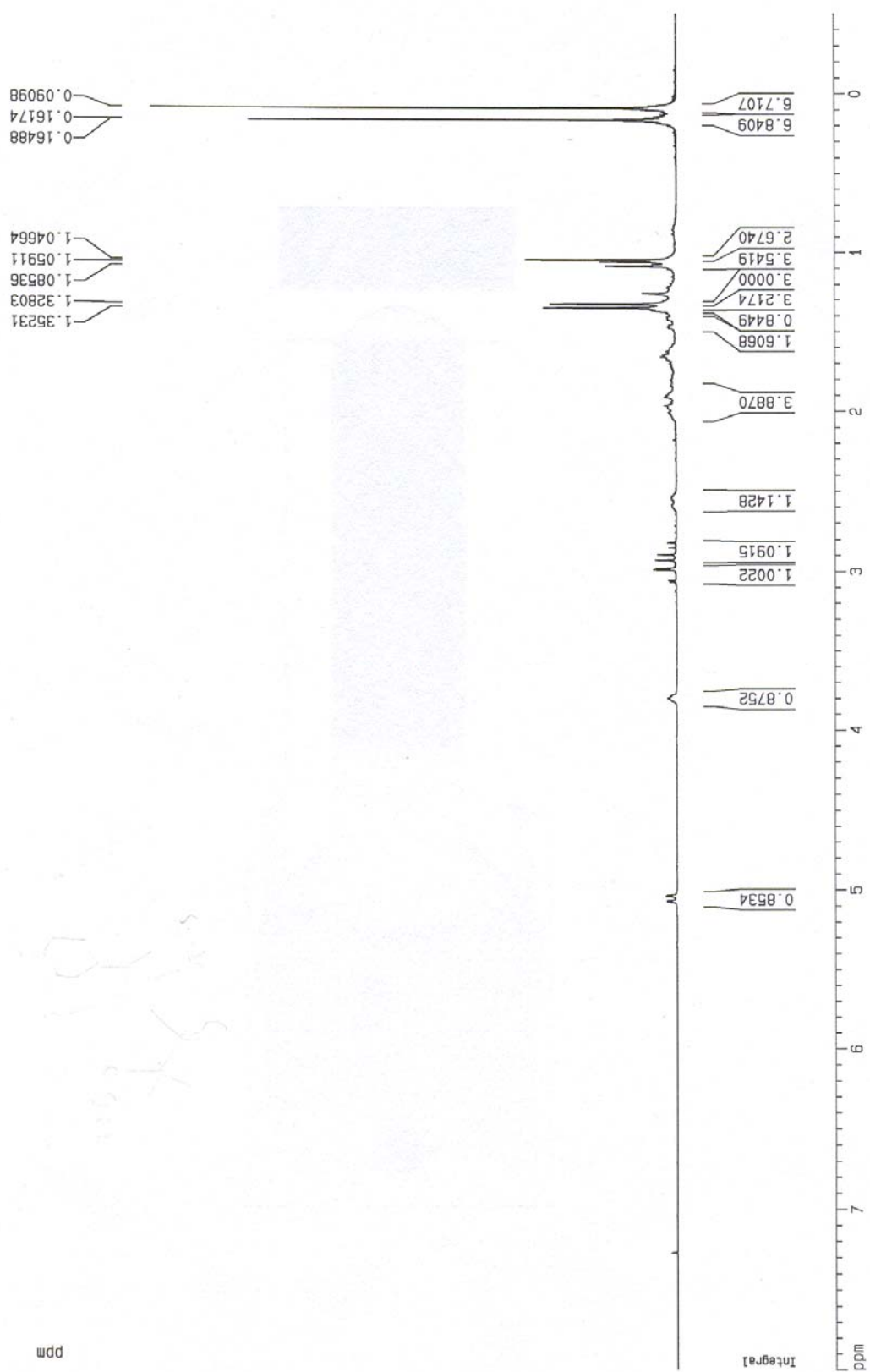


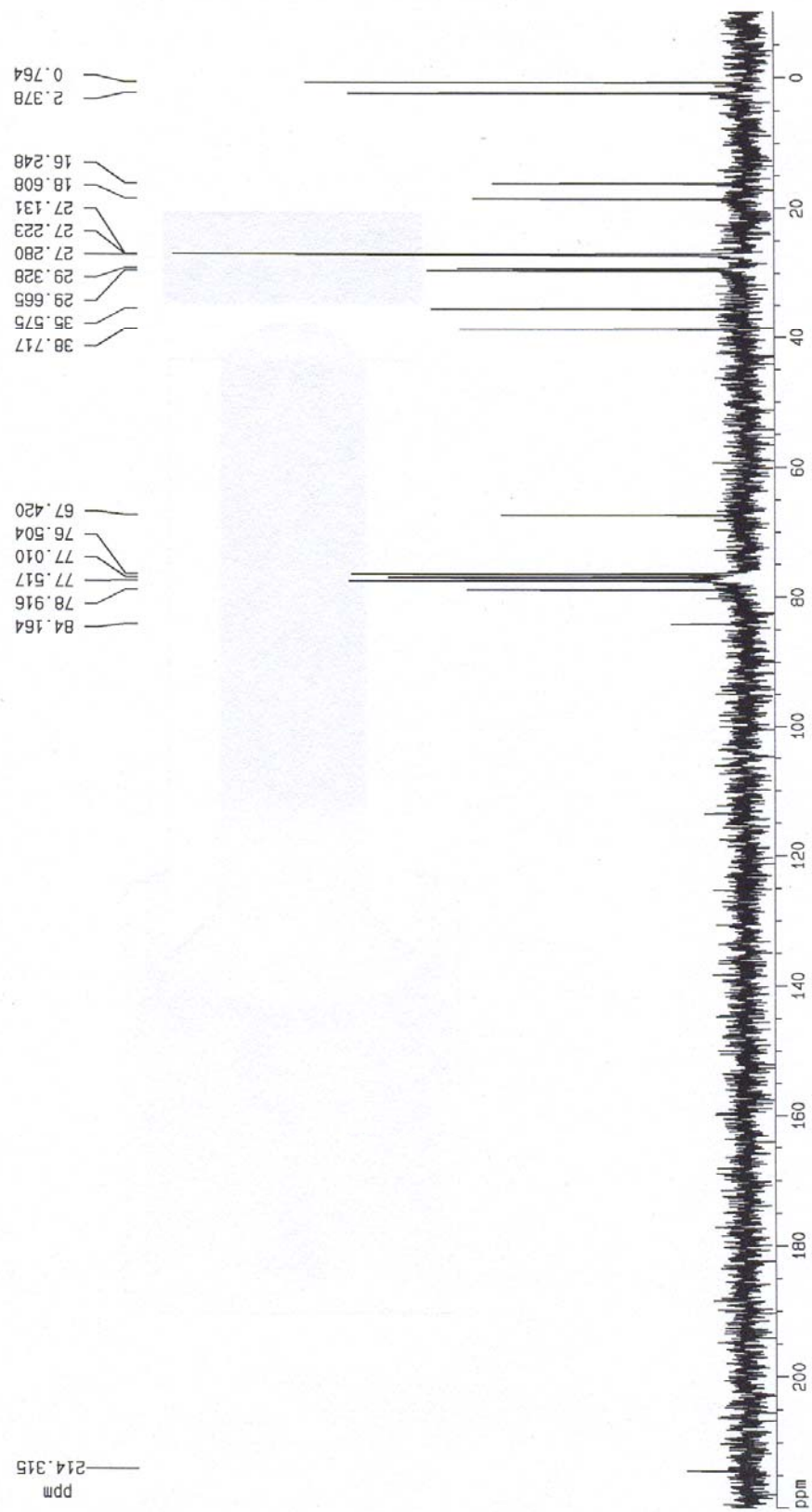
Recorded at 250 MHz( $^1\text{H}$ ) and 66.7 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

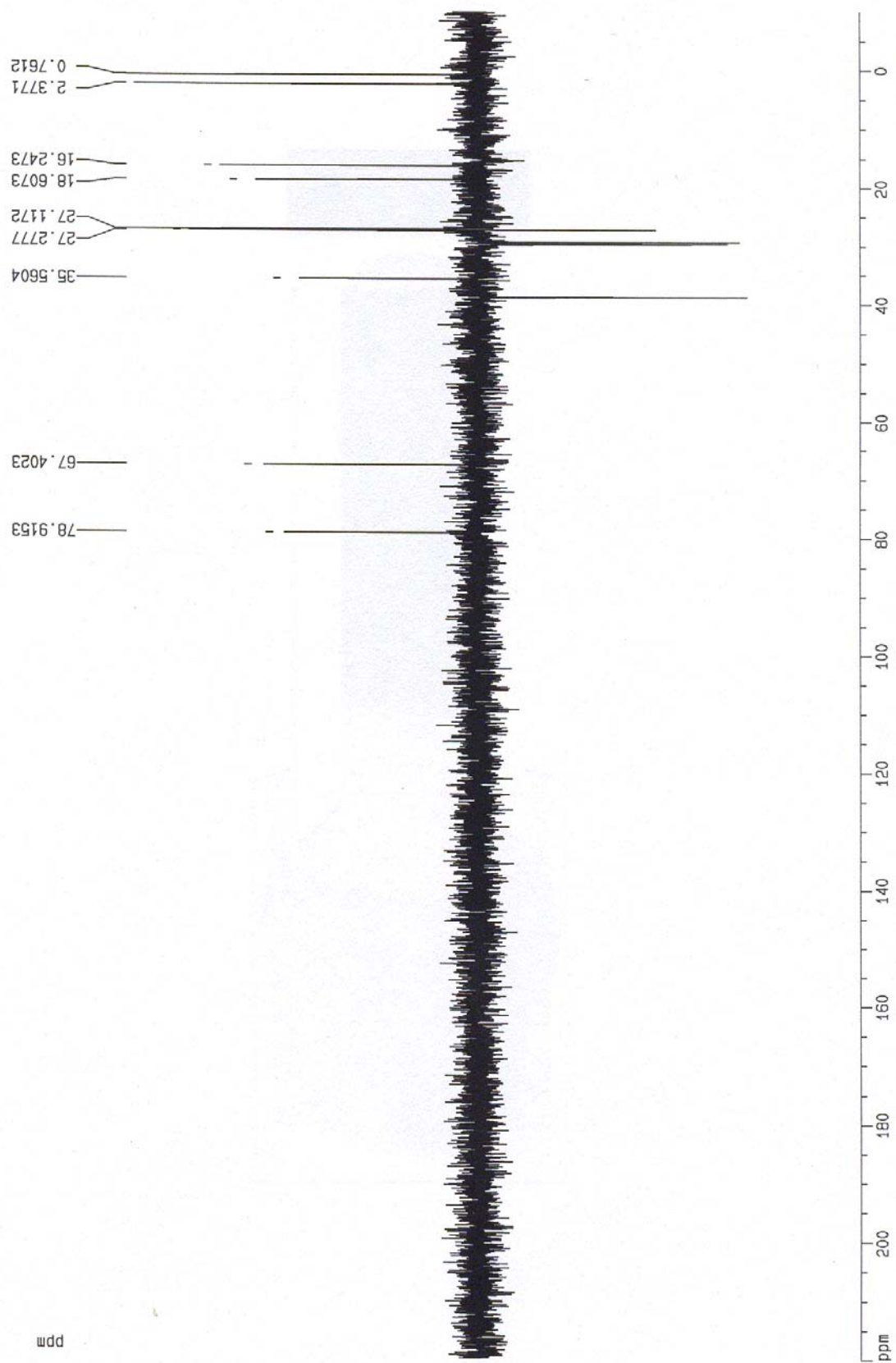
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

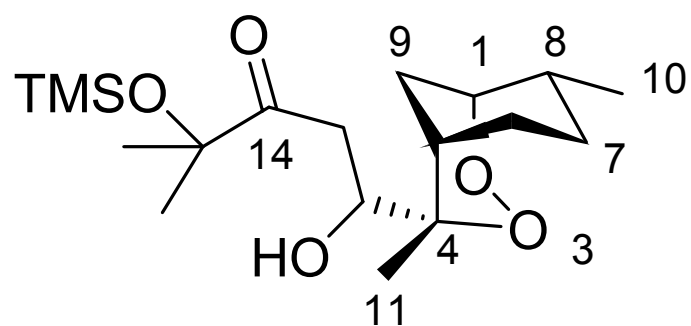
DEPT spectra







Aldol adduct **38**:



**38**

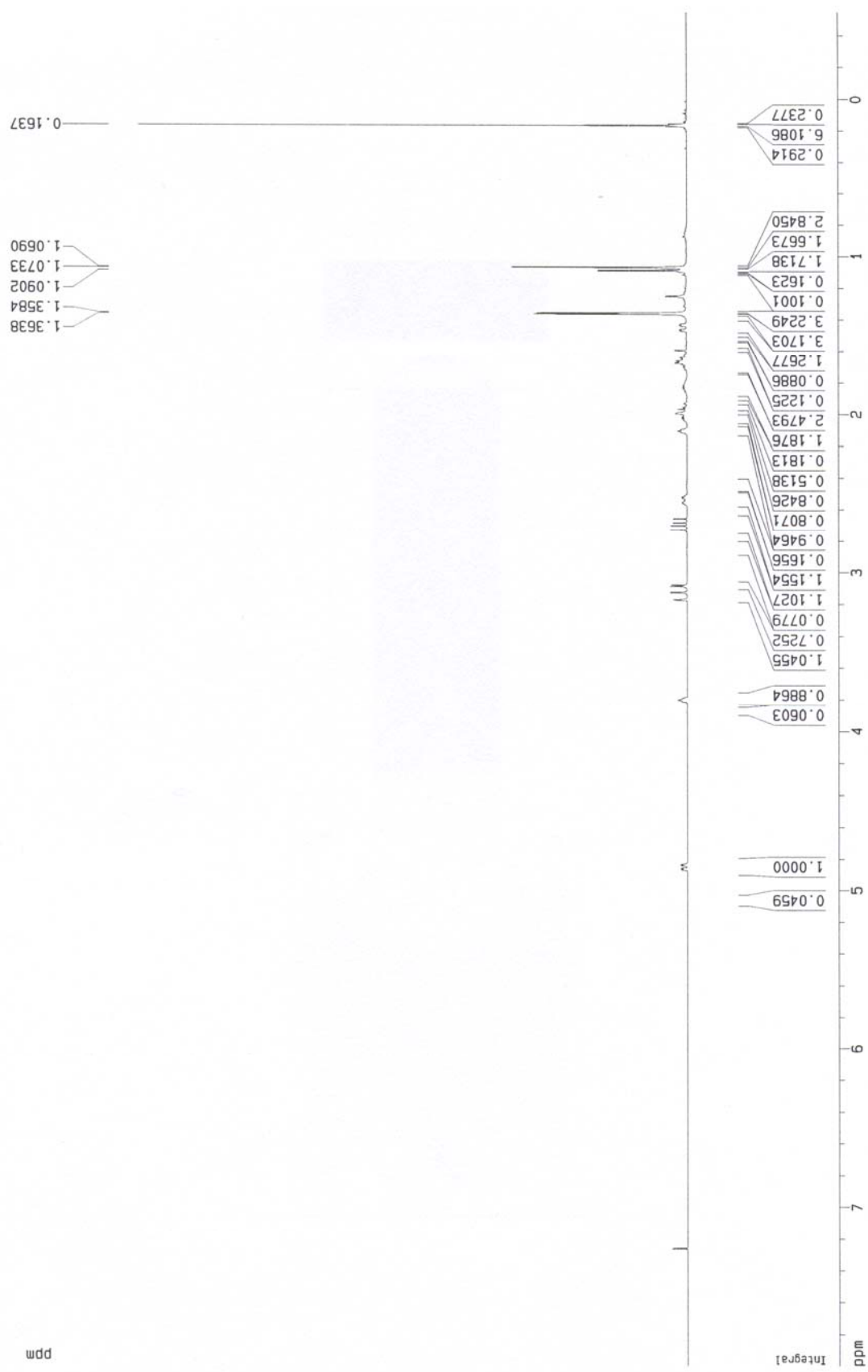
Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

COSY spectra

HMQC spectra





Current Data Parameters  
 NAME ed22.2.25081C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date 980223  
 Time 2.25  
 PULPROG zg.cpd  
 SOLVENT CDC13  
 AQ 1.0485960 sec  
 FIDRES 0.476837 Hz  
 DW 15.0 usec  
 RG 32768  
 NUCLEUS 13C  
 HL1 20 dB  
 D1 3.0000000 sec  
 P31 100.0 usec  
 D2 0.0031000 sec  
 P1 7.0 usec  
 DE 22.9 usec  
 SF01 100.6230000 MHz  
 SWH 31250.00 Hz  
 TD 65536  
 NS 544  
 DS 2

F1 - Processing parameters

SI 131072  
 MC2 GF  
 SF 100.6138701 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters

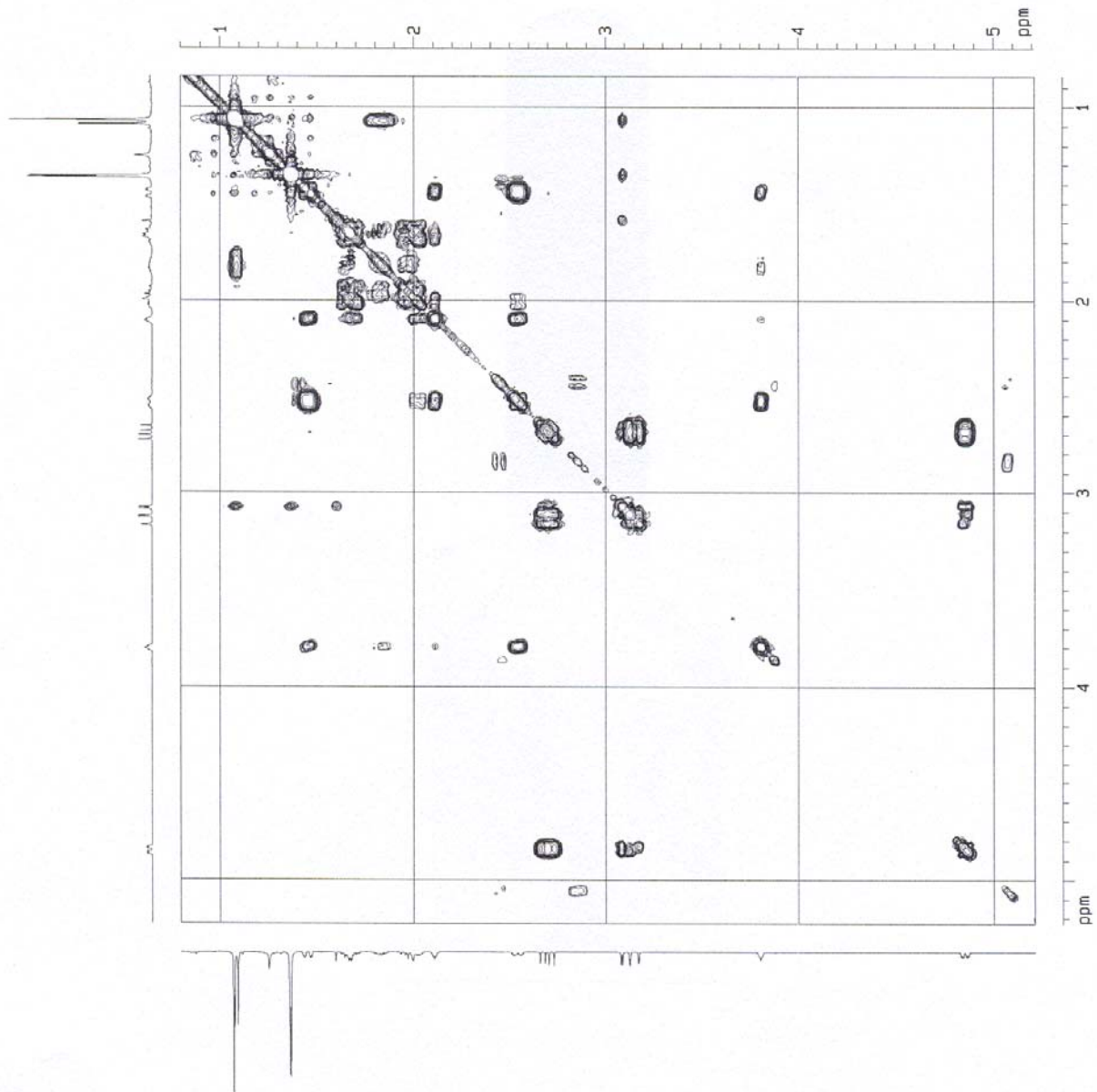
CX 21.95 cm  
 F1P 245.986 ppm  
 F1 24749.62 Hz  
 F2P -64.607 ppm  
 F2 -6500.38 Hz  
 PPMCM 14.14864 ppm/c  
 HZCM 1423.54907 Hz/cm

83.49  
 80.26  
 79.26  
 77.32  
 77.00  
 76.68  
 66.92  
 37.17  
 35.76  
 29.70  
 29.27  
 27.25  
 27.20  
 27.07  
 26.98  
 18.62  
 15.86  
 2.28

217.67

ppm

ppm



Current Data Parameters  
 NAME ed22.2 25CB10  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date 980223  
 Time 2:50  
 PULPROG csg45  
 SOLVENT cdc13  
 AQ 0.1812680 sec  
 FIDRES 2.756651 Hz  
 CN 377.0 usec  
 RG 128  
 NUC1 1H  
 NUC2 13C  
 H1 1 dB  
 D1 1.5000000 sec  
 P1 12.8 usec  
 D0 0.0000000 sec  
 TE 293.2 usec  
 SF01 400.1363893 MHz  
 SNH 2824.66 Hz  
 TD 1024  
 DS 4  
 INO 0.0003540 sec

F1 - Acquisition Parameters

NO 1  
 TD 256  
 SF01 400.135 MHz  
 FIDRES 11.034687 Hz  
 SN 7.055.000

F1 - Processing Parameters

SI 512  
 SF 400.1343895 MHz  
 SFO 400.1343895 MHz  
 SINE 0  
 LB 0.00 Hz  
 GB 0

F1 - Processing Parameters

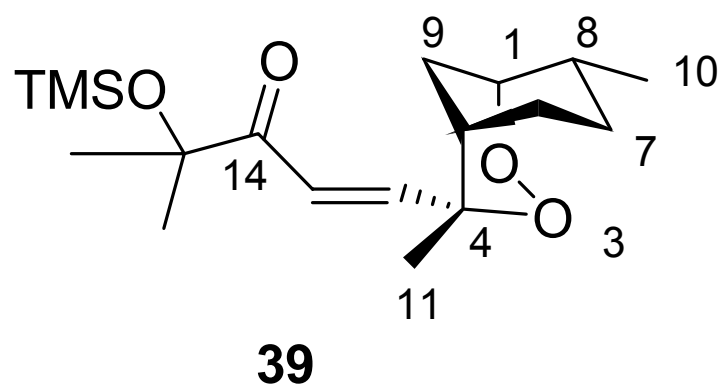
SI 512  
 SF 400.1347363 MHz  
 SFO 400.1347363 MHz  
 SINE 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters

CK2 15.00 cm  
 CK1 15.00 cm  
 F2P.D 5.226 ppm  
 F2L.D 2090.99 Hz  
 F2PHI 0.841 ppm  
 F2H1 335.49 Hz  
 F1P.D 5.212 ppm  
 F1L.D 2085.49 Hz  
 F1PHI 0.600 ppm  
 F1H1 319.84 Hz  
 F2P.MW 0.29232 ppm/cm  
 F2L.MW 116.96581 Hz/cm  
 F1P.MW 0.29416 ppm/cm  
 F1L.MW 117.70334 Hz/cm



Unsaturated ketone **39**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

COSY spectra

Current Data Parameters  
 NAME as2.20b170200H  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20000217  
 Time 17.20  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDC13  
 NS 29  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.244532 Hz  
 AQ 2.0447731 sec  
 RG 32  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.0000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SFO1 400.1320007 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300040 MHz  
 WDW EM  
 SSB 0  
 LB 0.01 Hz  
 GB 0  
 PC 0.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 9.000 ppm  
 F1 3601.17 Hz  
 F2P -1.000 ppm  
 F2 -400.13 Hz  
 PPMCM 0.50000 ppm/cm  
 HZCM 200.06500 Hz/cm

1.38484  
 1.36679  
 1.22691  
 1.11904  
 1.10214  
 0.21528  
 0.12378  
 0.12060

7.03971  
 7.03316

ppm

9.8542  
 0.9166  
 3.2374  
 3.3367  
 3.3793  
 3.0000  
 1.0408  
 2.3408  
 2.0060  
 2.0468  
 1.0179

0.8499

1.4411

Integral



```

Current Data Parameters
NAME      as2.20b170200C
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20000217
Time      17.29
INSTRUM   spect
PROBHD    5 mm QNP 1H
PULPROG   zgpgc
TD         65536
SOLVENT   CDCl3
NS         315
DS         4
SWH        30120.482 Hz
FIDRES     0.459602 Hz
AQ         1.0879476 sec
RG         3649.1
LW         16.600 usec
DE         6.00 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec

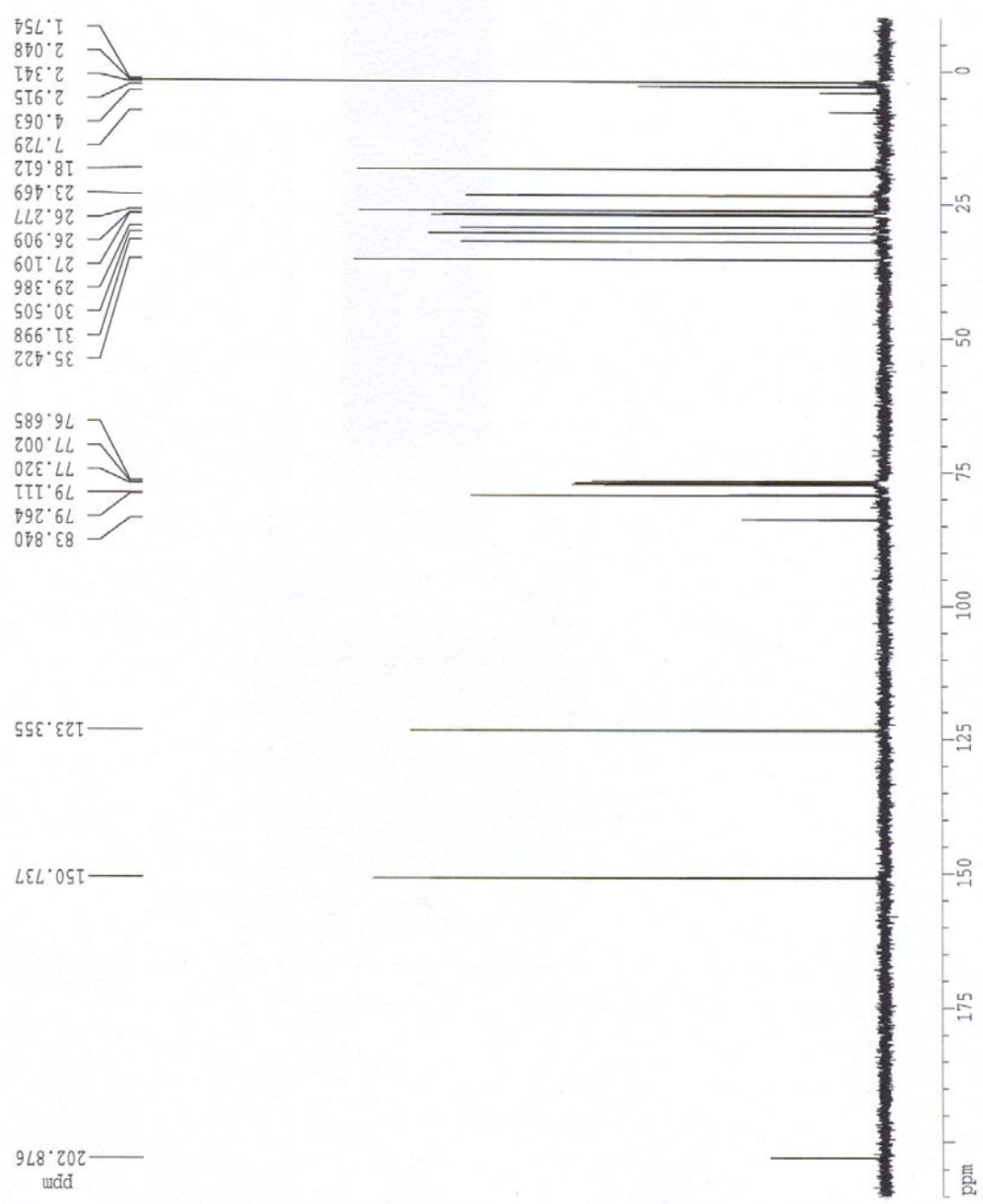
===== CHANNEL f1 =====
NUC1       13C
P1         6.20 usec
PL1        -6.00 dB
SFO1       100.6227903 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         120.00 dB
PL12        14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI          32768
SF          100.6127727 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

1D NMR plot parameters
CX          20.00 cm
F1P         210.000 ppm
F1          21128.68 Hz
F2P         -10.000 ppm
F2          -1006.13 Hz
PPMCM       11.00000 ppm/cm
HZCM        1106.74048 Hz/cm

```





# as2-20B COSY

Current Data Parameters  
 NAME as2-20B122060  
 EXPNO 4  
 PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20000217  
 Time 17.56  
 INSTRUM spect  
 PROBRD 5 mm QNP 1H  
 PULPROG cosy45  
 TD 1024  
 SOLVENT CDCl3  
 NS 8  
 DS 16  
 SWH 3255.208 Hz  
 FIDRES 1.178914 Hz  
 AQ 0.1571364 sec  
 RG 101.5  
 DM 153.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 TO 0.0000000 sec  
 TR 1.0000000 sec  
 TD 1  
 INO 0.0000000 sec

## ===== CHANNEL f1 =====

NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SFO1 400.1313819 MHz

## F1 - Acquisition Parameters

ND0 1  
 TD 163  
 SFO1 400.1316 MHz  
 FIDRES 19.970604 Hz  
 SW 8.135 ppm

## F2 - Processing parameters

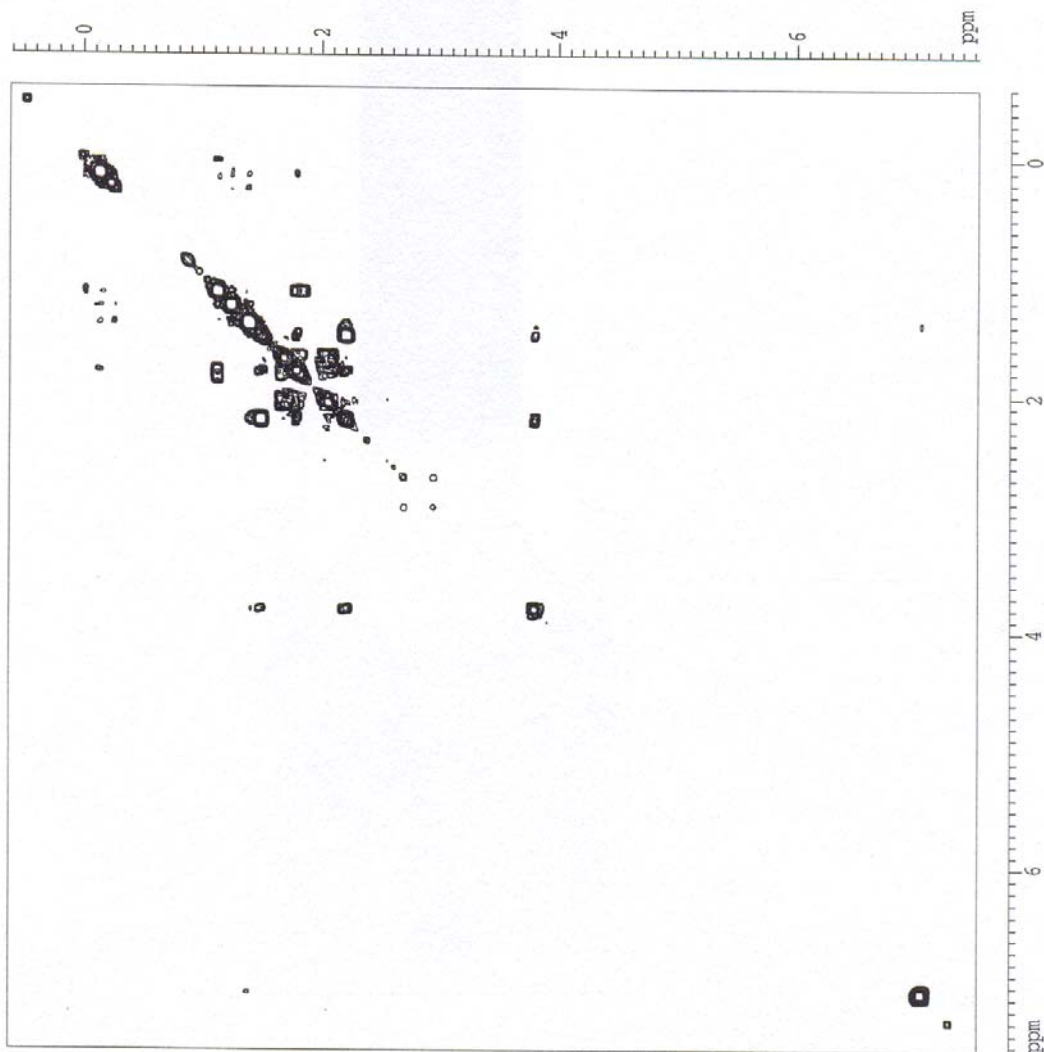
SI 512  
 SF 400.1299984 MHz  
 NS 0  
 DS 0  
 SW 0.00 Hz  
 TE 300.0 K  
 PC 1.00

## F1 - Processing parameters

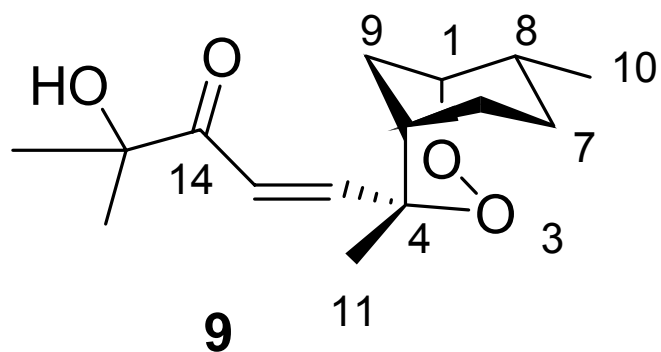
SI 512  
 SF 400.1302150 MHz  
 NS 0  
 DS 0  
 SW 0.00 Hz  
 PC 1.00

## 2D NMR plot parameters

SI 512  
 SF 400.1302150 MHz  
 NS 0  
 DS 0  
 SW 0.00 Hz  
 PC 1.00



Unsaturated ketone **9**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

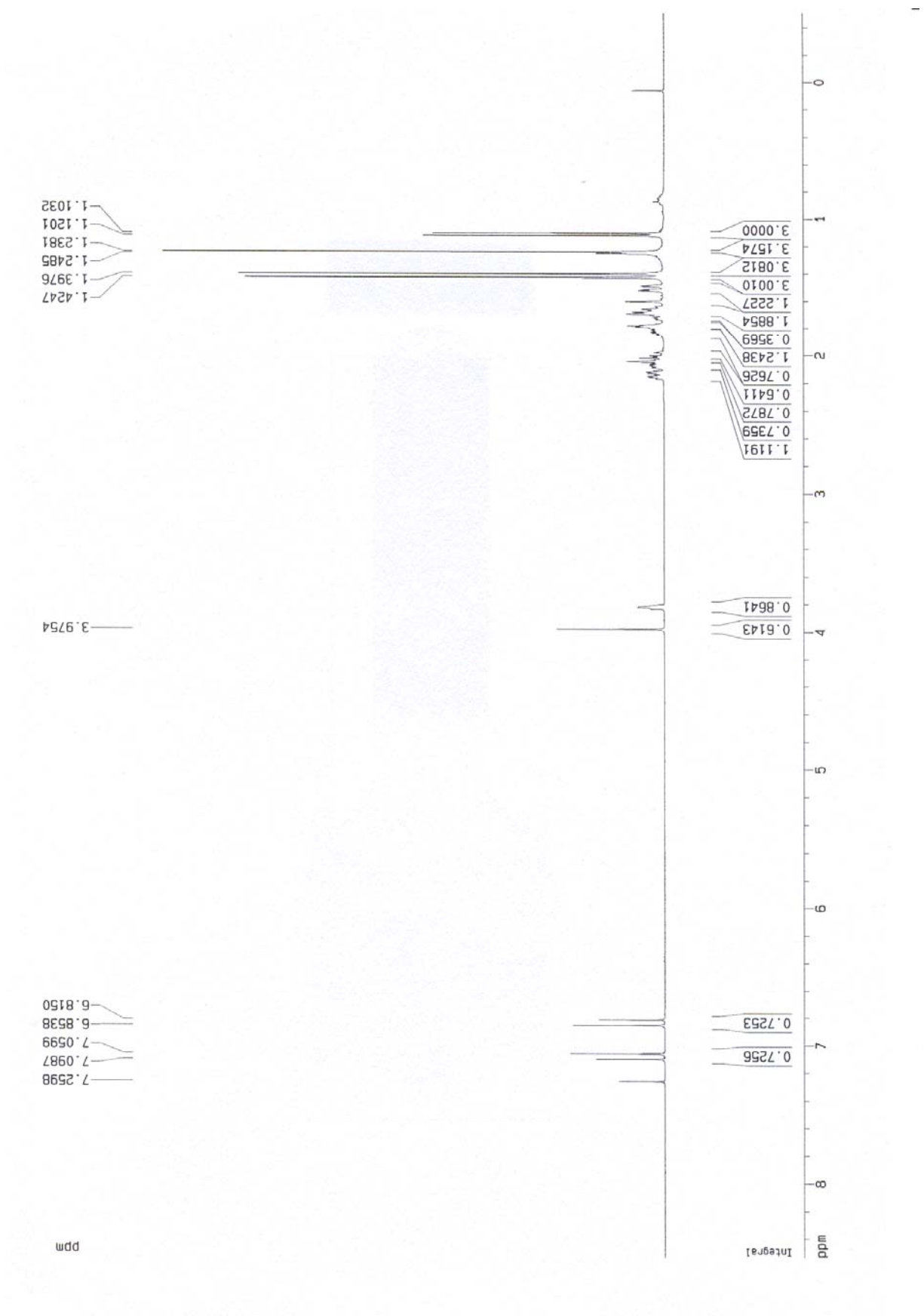
$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra

HMQC spectra





Current Data Parameters  
 NAME ed22.2.254.1C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date 980222  
 Time 16.44  
 PULPROG zg.cpd  
 SOLVENT CDCl3  
 AQ 1.0485960 sec  
 FIDRES 0.476837 Hz  
 DW 15.0 usec  
 RG 32768  
 NUCLEUS 13C  
 HL1 20 dB  
 D1 3.000000 sec  
 P31 100.0 usec  
 D2 0.0031000 sec  
 P1 7.0 usec  
 DE 22.9 usec  
 SF01 100.6230000 MHz  
 SWH 31250.00 Hz  
 TD 65536  
 NS 826  
 DS 2

F1 - Processing parameters

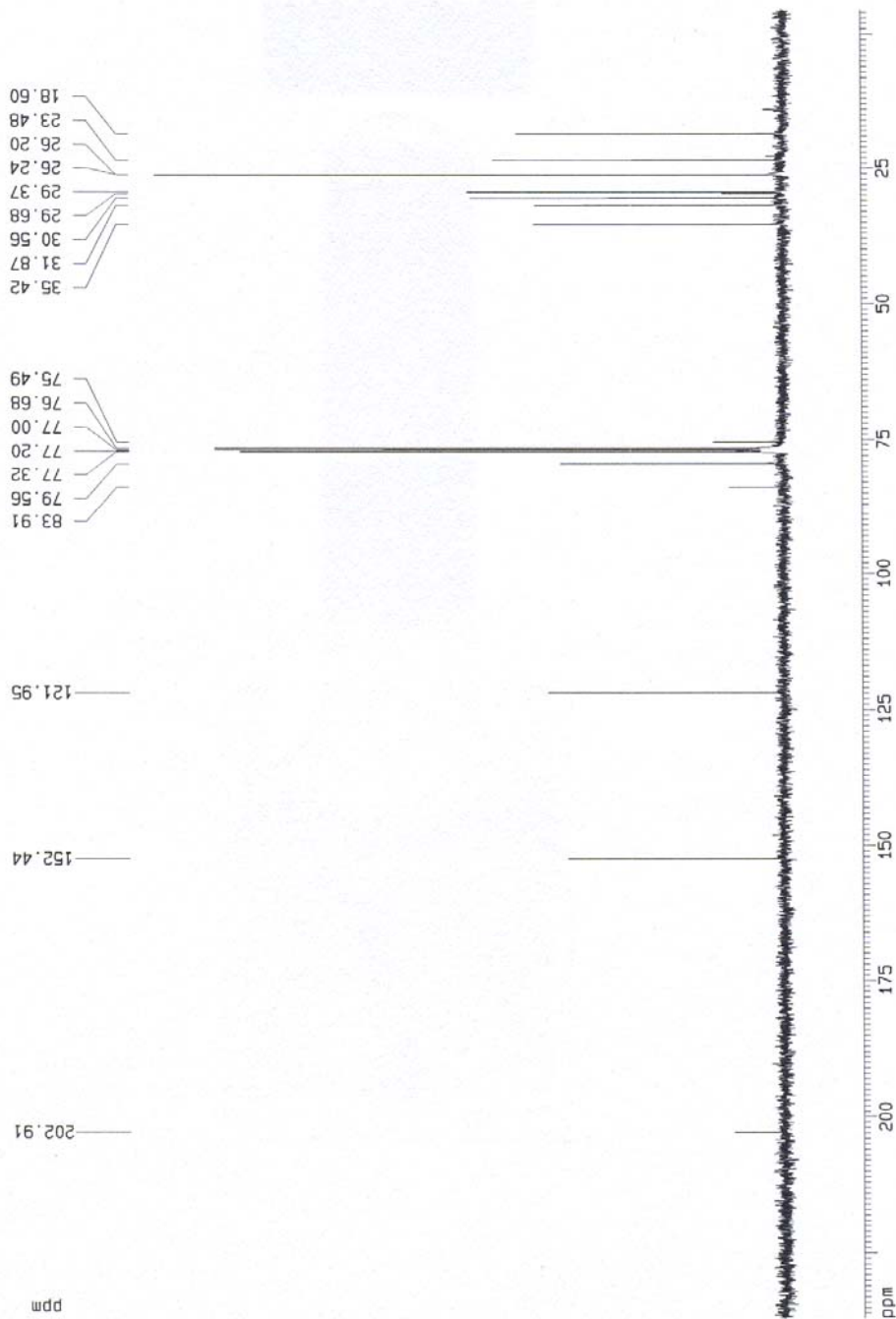
SI 131072  
 MC2 GF  
 SF 100.6138706 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0

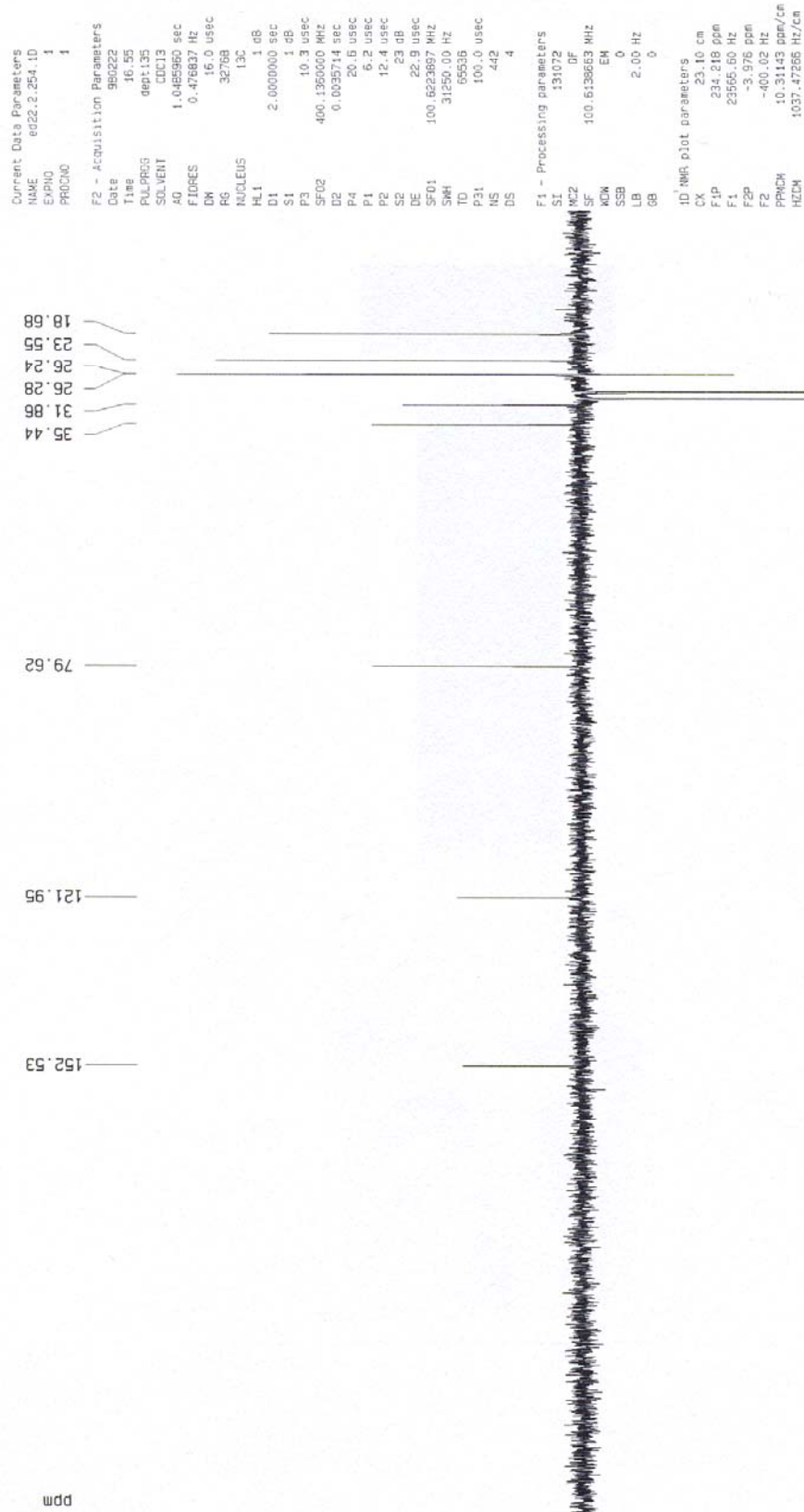
1D NMR plot parameters

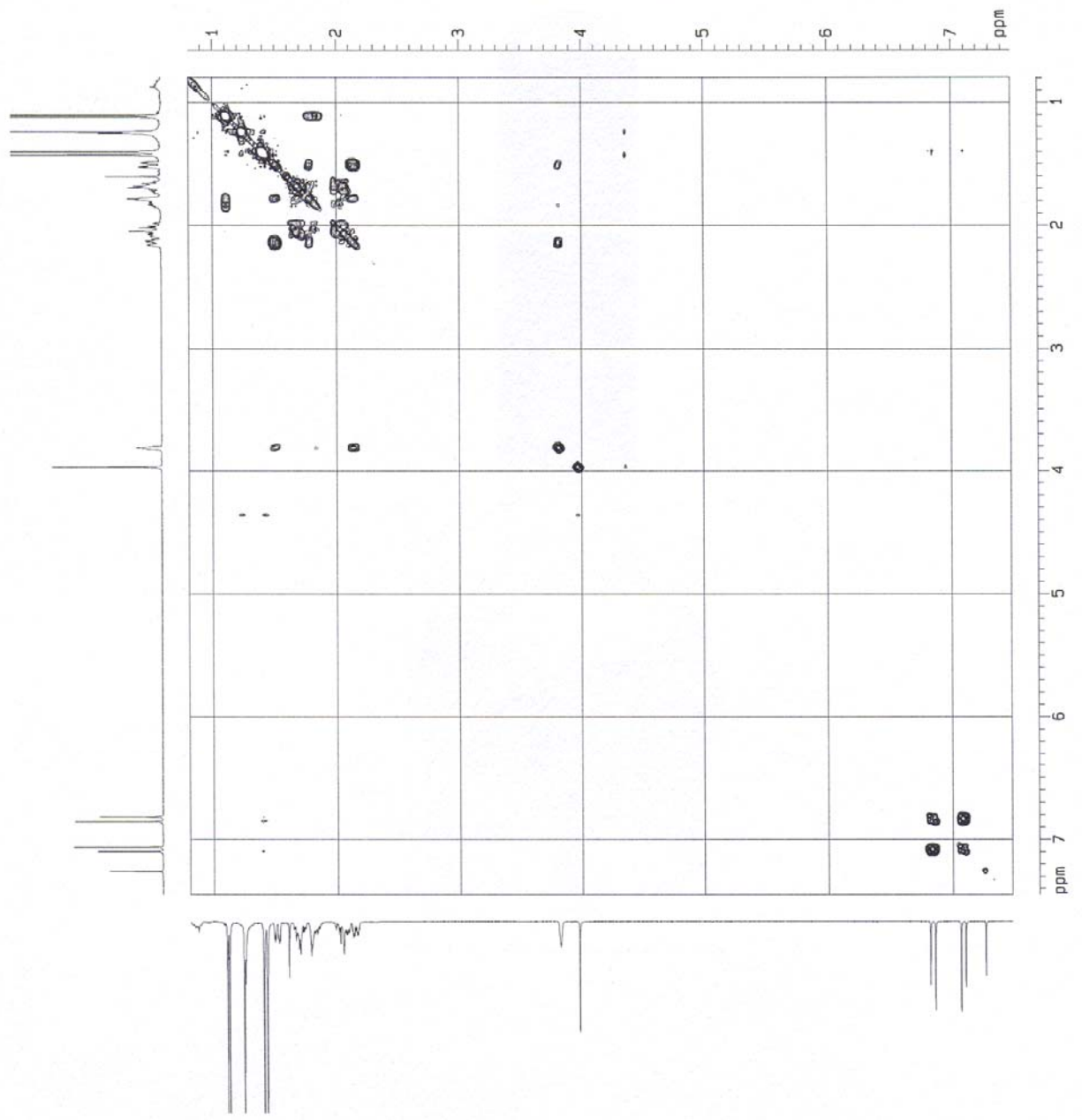
CX 21.95 cm  
 F1P 236.898 ppm  
 F1 23835.26 Hz  
 F2P -4.420 ppm  
 F2 -444.76 Hz  
 PPMCH 10.99294 ppm/c  
 HZCM 1106.04187 Hz/cm

18.60  
 23.48  
 26.20  
 26.24  
 29.37  
 29.68  
 30.56  
 31.87  
 35.42  
 75.49  
 76.68  
 77.00  
 77.20  
 77.32  
 79.56  
 83.91

121.95  
 152.44  
 202.91







Current Data Parameters  
 NAME BD22.2 2541.Co  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date 990222  
 Time 20.01  
 PULPROG zgpg30  
 SOLVENT cdcl3  
 A0 0.1618120 sec  
 FIDRES 3.090388 Hz  
 CN 158.0 usec  
 RG 256  
 NUCLEUS 1H  
 H1 1.48  
 D1 1.5000000 sec  
 P1 12.6 usec  
 D0 0.0000330 sec  
 DE 225.7 usec  
 SF01 400.1361405 MHz  
 SNH 3164.56 Hz  
 TD 1024  
 AS 4  
 CS 4  
 INO 0.0003160 sec

F1 - Acquisition Parameters

M00 1  
 TD 256  
 SF01 400.136 MHz  
 FIDRES 12.361505 Hz  
 SN 7.509 ppm

F1 - Processing Parameters

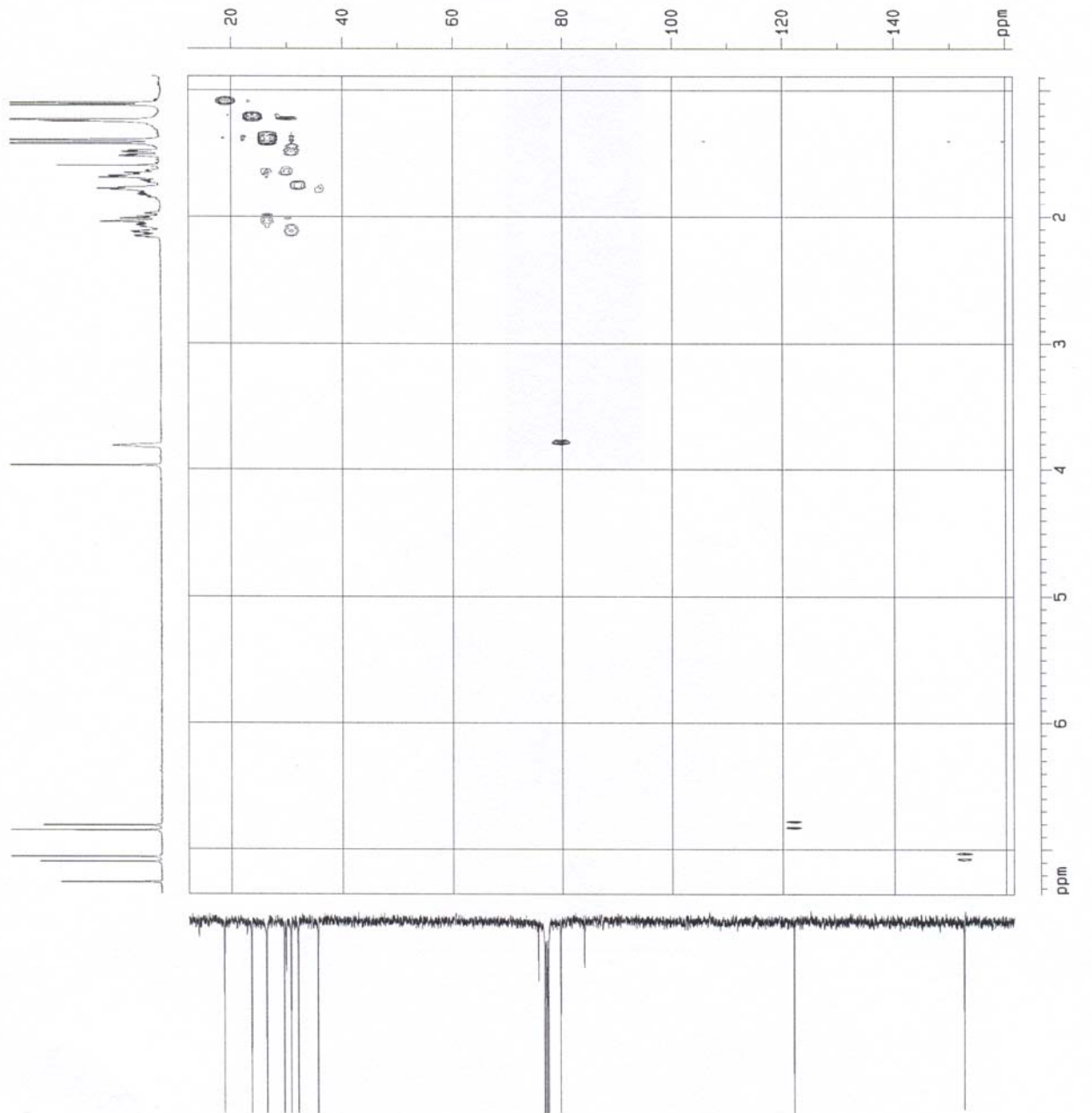
SI 32  
 MC2 0  
 SF 400.1343957 MHz  
 N0 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0

F1 - Processing Parameters

SI 32  
 MC2 0  
 SF 400.1343957 MHz  
 N0 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters

CX2 15.00 cm  
 CX1 15.00 cm  
 F2PL0 7.450 ppm  
 F2L0 2980.59 Hz  
 F2PHI 0.792 ppm  
 F2H1 317.08 Hz  
 F1PL0 7.481 ppm  
 F1L0 2993.35 Hz  
 F1PHI 0.808 ppm  
 F1H1 323.26 Hz  
 F2PPMCM 0.44384 ppm/cm  
 F2HZCM 177.59427 Hz/cm  
 F1PPMCM 0.44487 ppm/cm  
 F1HZCM 178.00623 Hz/cm



Current Data Parameters  
NAME e922.2.2541CH  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 980222  
Time 21:30  
PULPROG invetd  
SOLVENT CDCl3  
AQ 0.3280340 sec  
FIDRES 1.545134 Hz  
DM 158.0 ussec  
RG 2048  
HACQUIS 1H  
AL1 2.0000000 sec  
P1 12.0000000 sec  
P2 0.0050000 sec  
P3 25.5 ussec  
P4 14.0 ussec  
SF02 100.624514 MHz  
D7 0.6000000 sec  
D0 0.0000000 sec  
D13 0.0000000 sec  
DLO 20.0000000 sec  
SF01 400.1361425 MHz  
SFO 3164.55 Hz  
TD 2048  
P31 80.0 ussec  
HS 24  
DS 4  
TH0 0.0000110 sec

F1 - Acquisition Parameters

RG0 128  
TD 128  
SF01 100.6245 MHz  
FIDRES 177.566934 Hz  
SFO 225.862 ppm

F1 - Processing Parameters

SF 1024  
MC2 GF  
SF 400.1344651 MHz  
KCN 051HC  
SSB 0  
LB 0.00 Hz  
GB 0

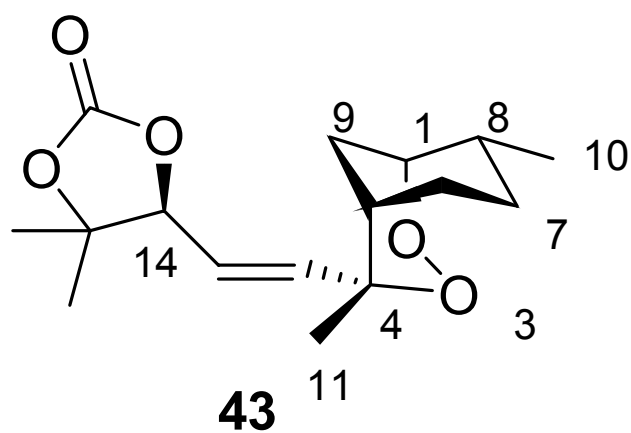
F1 - Processing Parameters

SF 256  
MC2 1991  
SF 100.6138602 MHz  
KCN 0M  
SSB 0  
LB -10.00 Hz  
GB 0.33

2D NMR plot parameters

D2 15.00 cm  
D1 15.00 cm  
F2P0 7.347 ppm  
F2L0 2339.62 Hz  
F2PH 0.890 ppm  
F2H1 356.06 Hz  
F1P0 161.451 ppm  
F1L0 16244.23 Hz  
F1PH 12.301 ppm  
F1H1 12.301 Hz/cm  
F2PDM 9.94134 ppm/cm  
F2LDM 1000.23662 Hz/cm

Carbonate **43**:



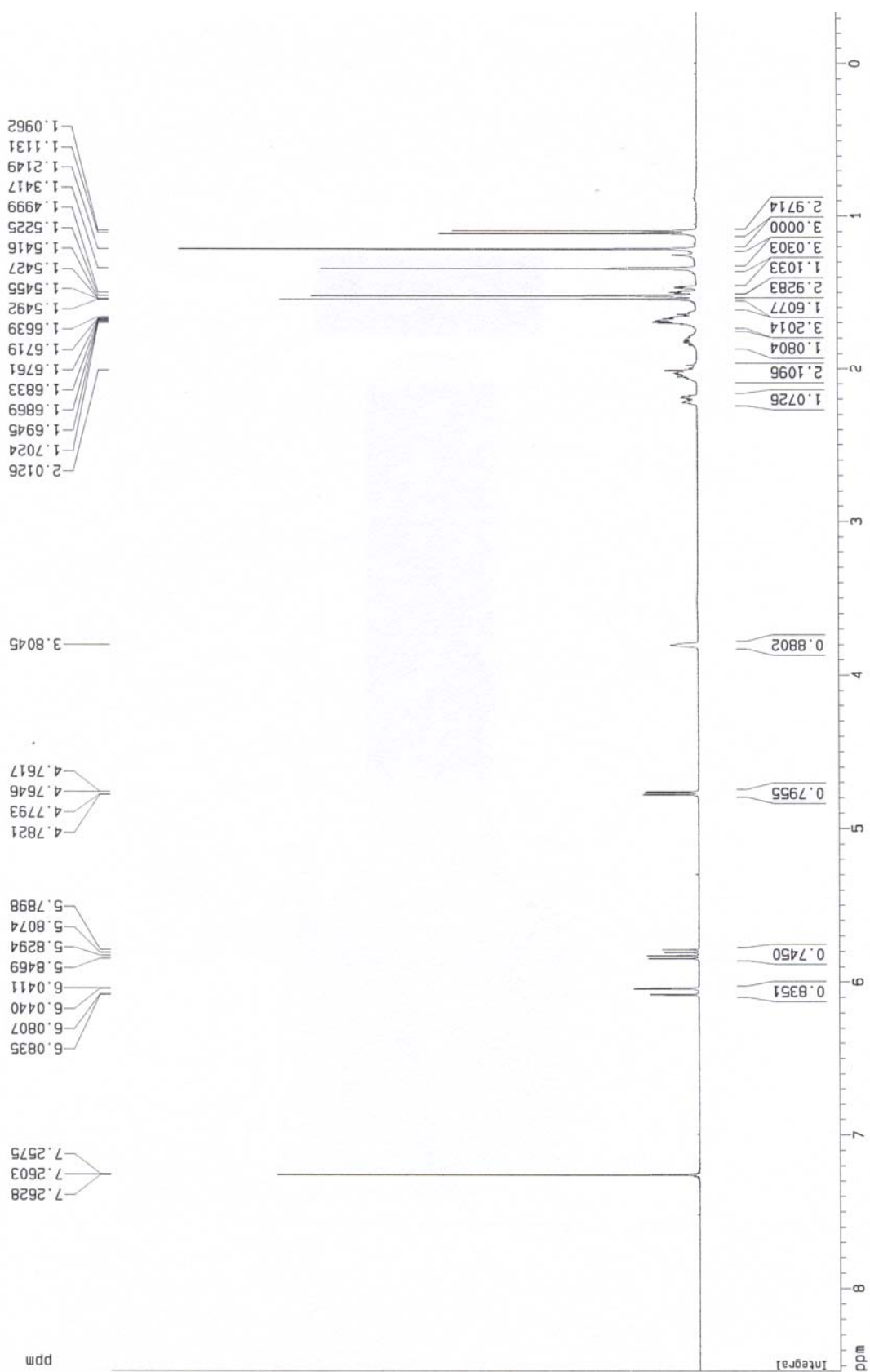
Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

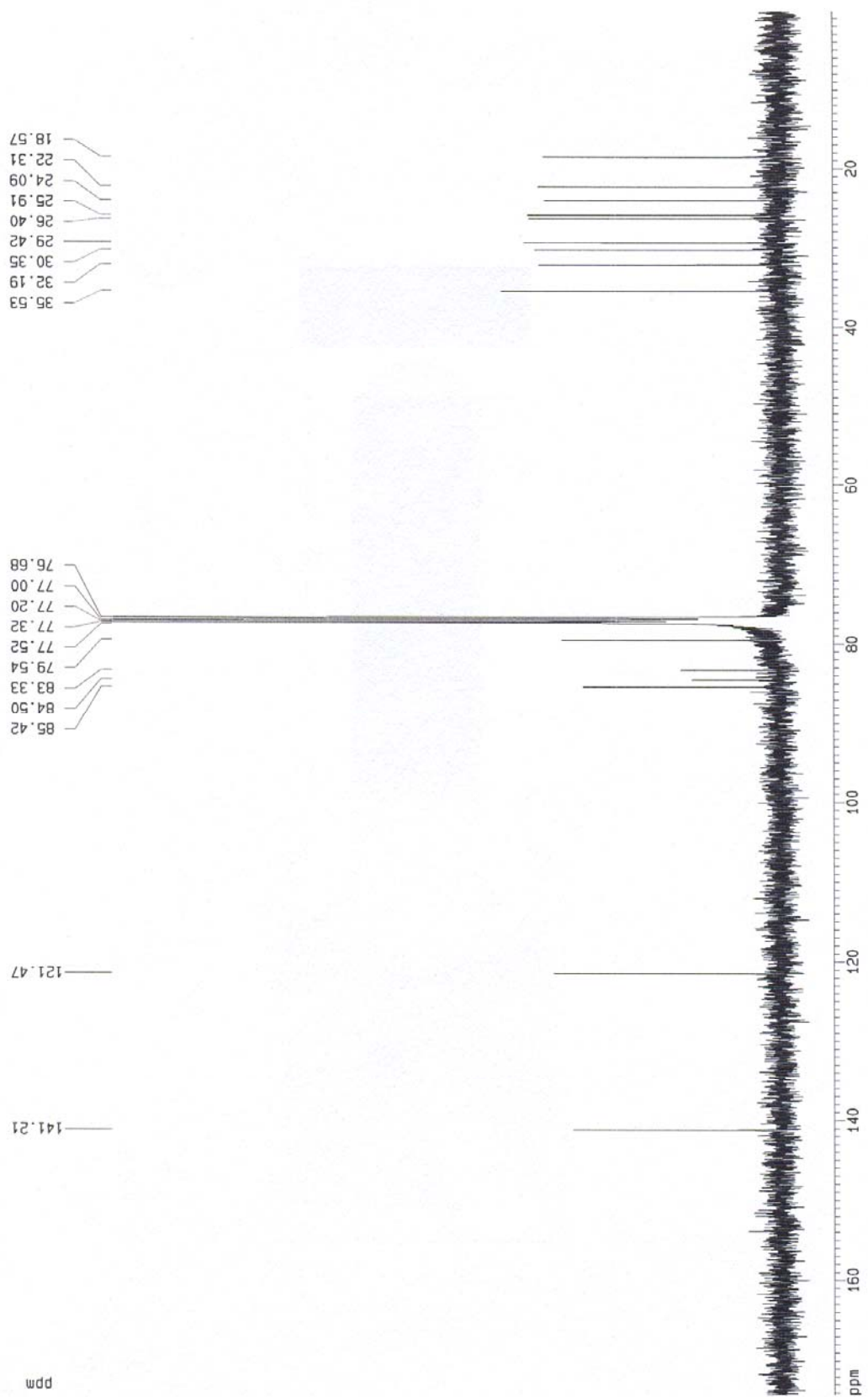
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra







Current Data Parameters  
 NAME ed29.3.261.20  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 960329  
 Time 23:28  
 PULPROG dept135  
 SOLVENT CDCl3  
 AQ 1.0485960 sec  
 FIDRES 0.476637 Hz  
 DM 16.0 usec  
 RG 32768  
 NUCLEUS 13C

H4.1 1 dB  
 D1 2.0000000 sec  
 S1 1 dB  
 P3 10.3 usec  
 SF02 400.1360000 MHz  
 D2 0.0035714 sec  
 P4 20.6 usec  
 P1 6.2 usec  
 P2 12.4 usec  
 S2 23 dB

DE 22.9 usec  
 SF01 100.6223897 MHz  
 SMH 31250.00 Hz  
 TD 65536  
 P31 100.0 usec  
 NS 1377  
 DS 4

F1 - Processing parameters  
 S1 131072  
 MC2 OF  
 SF 100.6139654 MHz  
 NDM EN  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters  
 CX 23.10 cm  
 FIP 170.516 ppm  
 F1 17156.27 Hz  
 F2 -13.378 ppm  
 F2 -1346.06 Hz  
 PPMCM 7.96080 ppm/cm  
 HZCM 800.96674 Hz/cm

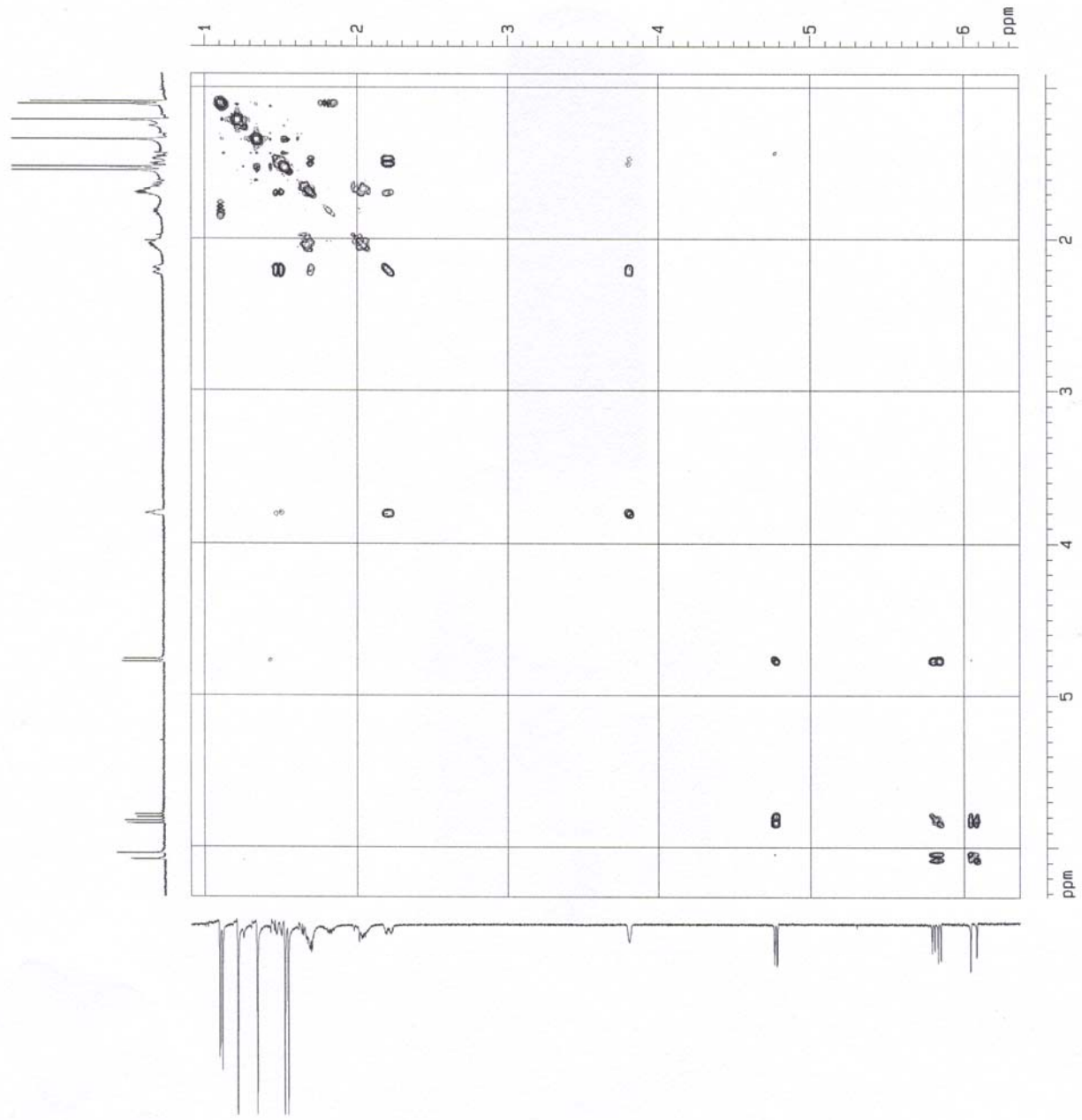
18.66  
 22.38  
 24.17  
 25.95  
 32.19  
 35.55

79.60  
 85.47

121.47

ppm





Current Data Parameters  
 NAME ed29.3.261.20g  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 980330  
 Time 1.14  
 PULPROG cosy45  
 SOLVENT ccd13  
 A0 0.1960280 sec  
 FIDRES 2.54132 Hz  
 DN 192.0 usec  
 RG 2048  
 NUCLEUS 1H  
 H1 1 dB  
 D1 1.500000 sec  
 P1 12.6 usec  
 D0 0.000030 sec  
 DE 274.3 usec  
 SF01 400.1369576 MHz  
 SMH 2604.17 Hz  
 TD 1024  
 NS 4  
 DS 4  
 IN0 0.0003840 sec

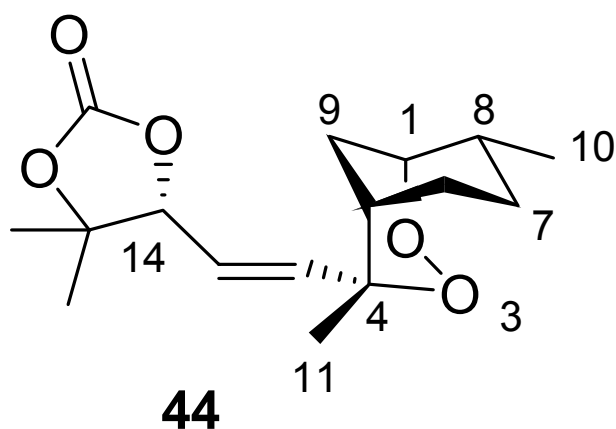
F1 - Acquisition Parameters  
 N00 1  
 TD 256  
 SF01 400.1359 MHz  
 FIDRES 10.172517 Hz  
 SN 6.568 ppm

F1 - Processing Parameters  
 SI 512  
 SF 400.1343959 MHz  
 N00 1  
 N01 1  
 N02 1  
 N03 1  
 N04 1  
 N05 1  
 N06 1  
 N07 1  
 N08 1  
 N09 1  
 N10 1  
 N11 1  
 N12 1  
 N13 1  
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 N19 1  
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 N82 1  
 N83 1  
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 N85 1  
 N86 1  
 N87 1  
 N88 1  
 N89 1  
 N90 1  
 N91 1  
 N92 1  
 N93 1  
 N94 1  
 N95 1  
 N96 1  
 N97 1  
 N98 1  
 N99 1  
 N100 1

F1 - Processing Parameters  
 SI 512  
 SF 400.1343959 MHz  
 N00 1  
 N01 1  
 N02 1  
 N03 1  
 N04 1  
 N05 1  
 N06 1  
 N07 1  
 N08 1  
 N09 1  
 N10 1  
 N11 1  
 N12 1  
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 N52 1  
 N53 1  
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 N80 1  
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 N86 1  
 N87 1  
 N88 1  
 N89 1  
 N90 1  
 N91 1  
 N92 1  
 N93 1  
 N94 1  
 N95 1  
 N96 1  
 N97 1  
 N98 1  
 N99 1  
 N100 1

2D NMR plot parameters  
 Cx2 15.00 cm  
 Cx1 15.00 cm  
 F2PLO 6.327 ppm  
 F2LO 2531.82 Hz  
 F2PHI 0.912 ppm  
 F2HI 365.07 Hz  
 F2LO 6.366 ppm  
 F2LO 2547.08 Hz  
 F2LO 0.912 ppm  
 F2HI 365.07 Hz  
 F2PHI 0.36100 ppm/cm  
 F2H2CM 144.44666 Hz/cm  
 F2P2CM 0.36355 ppm/cm  
 F2H2CM 145.46700 Hz/cm

Carbonate **44**:



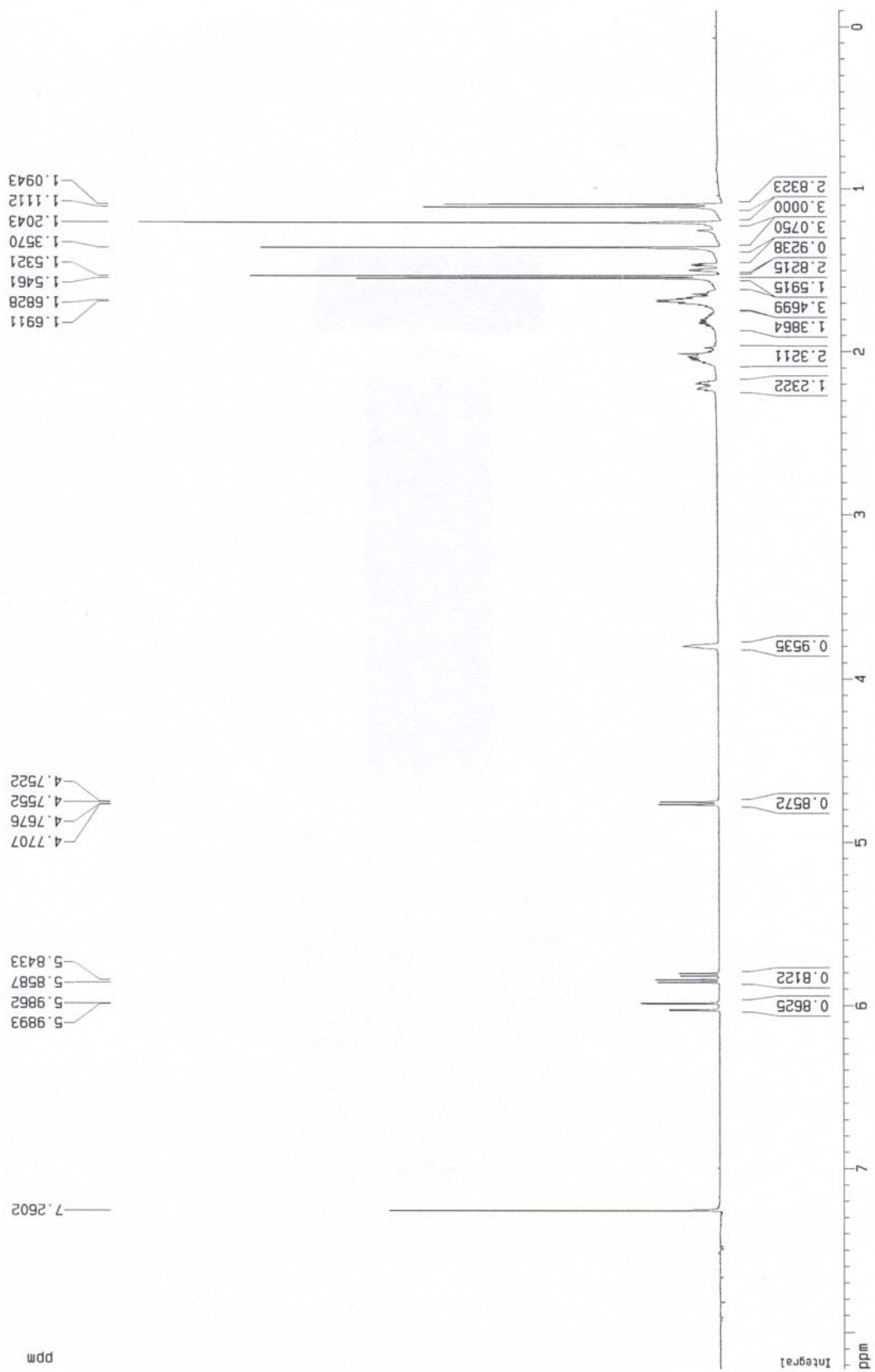
Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

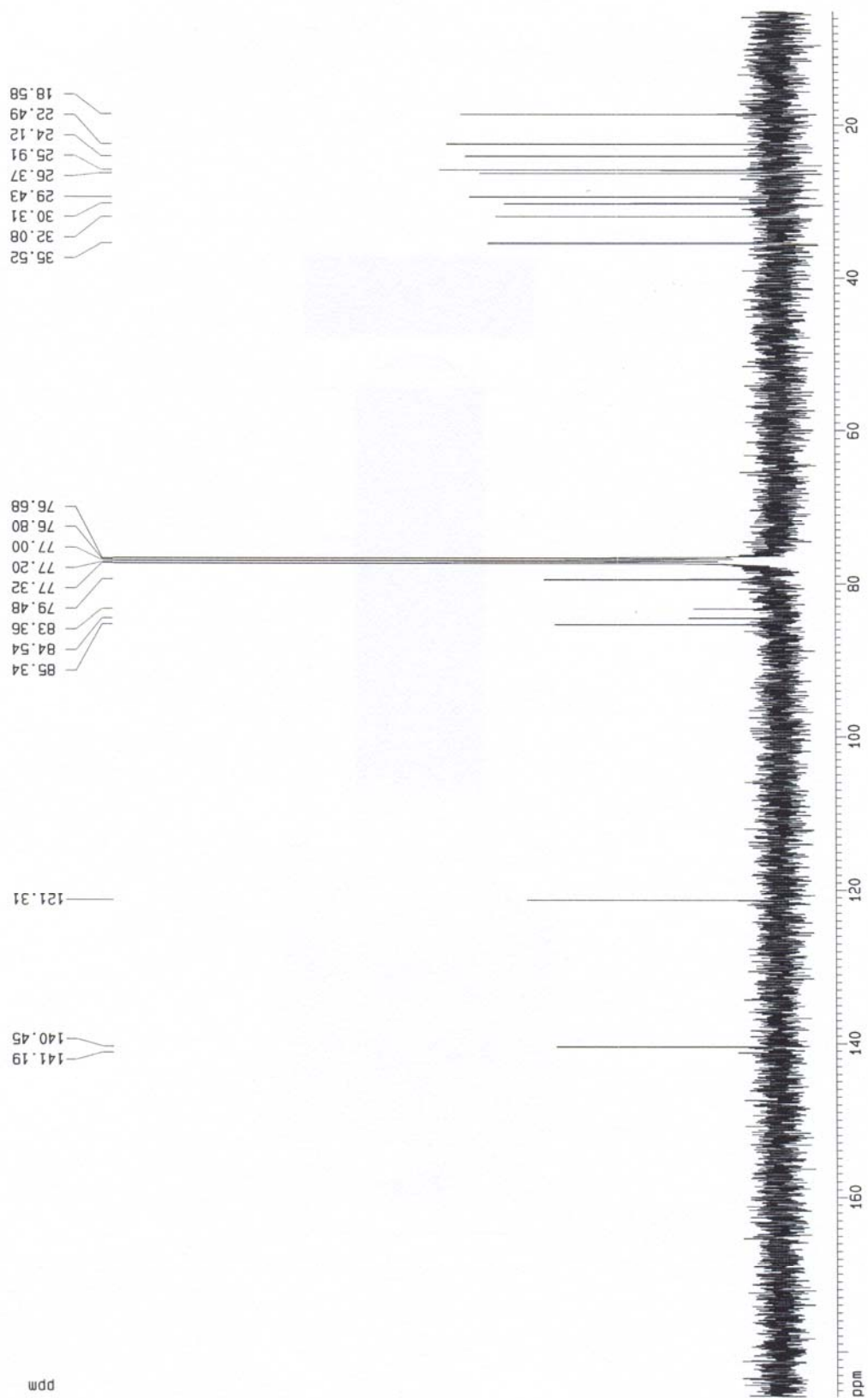
$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra



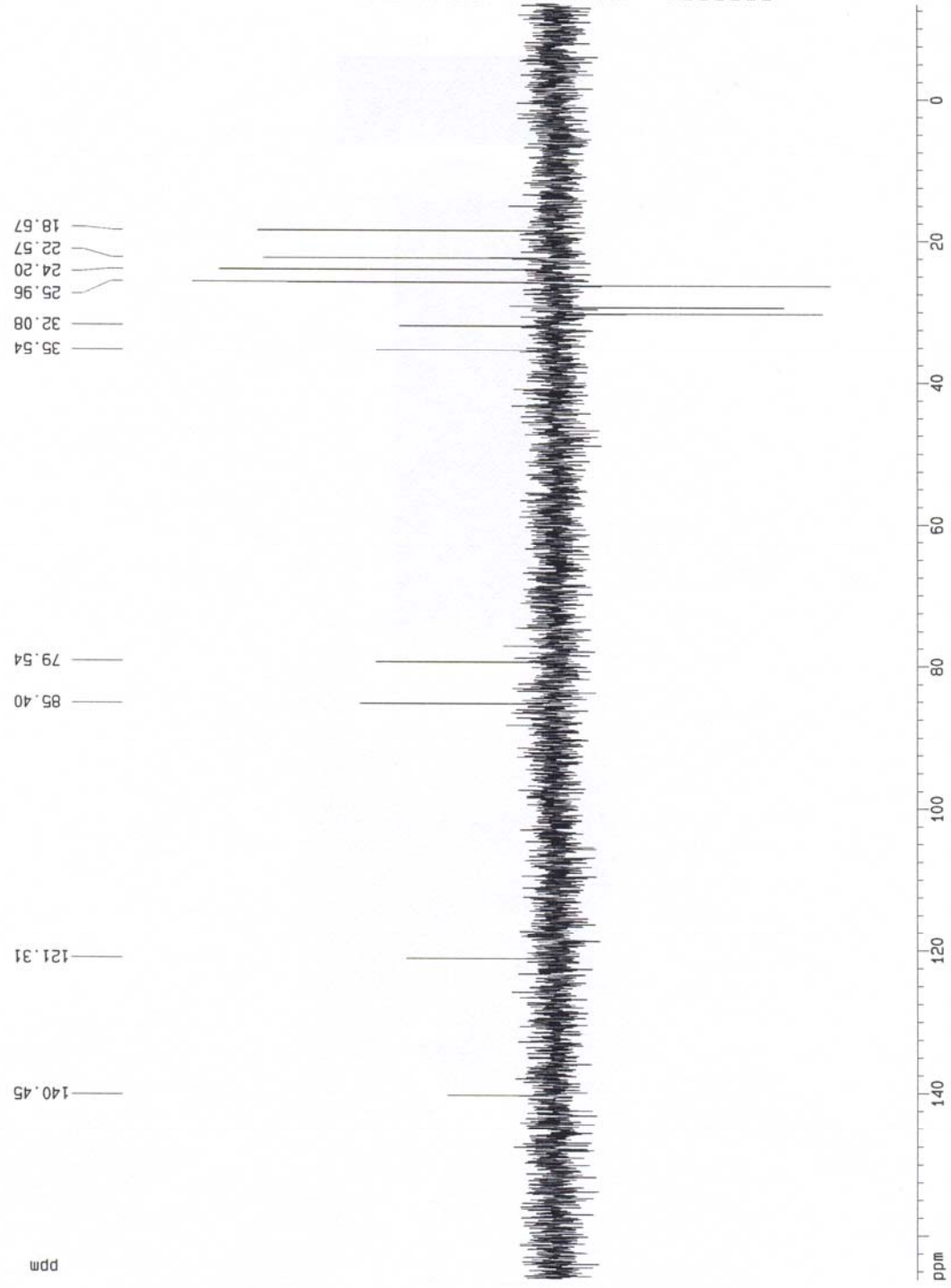


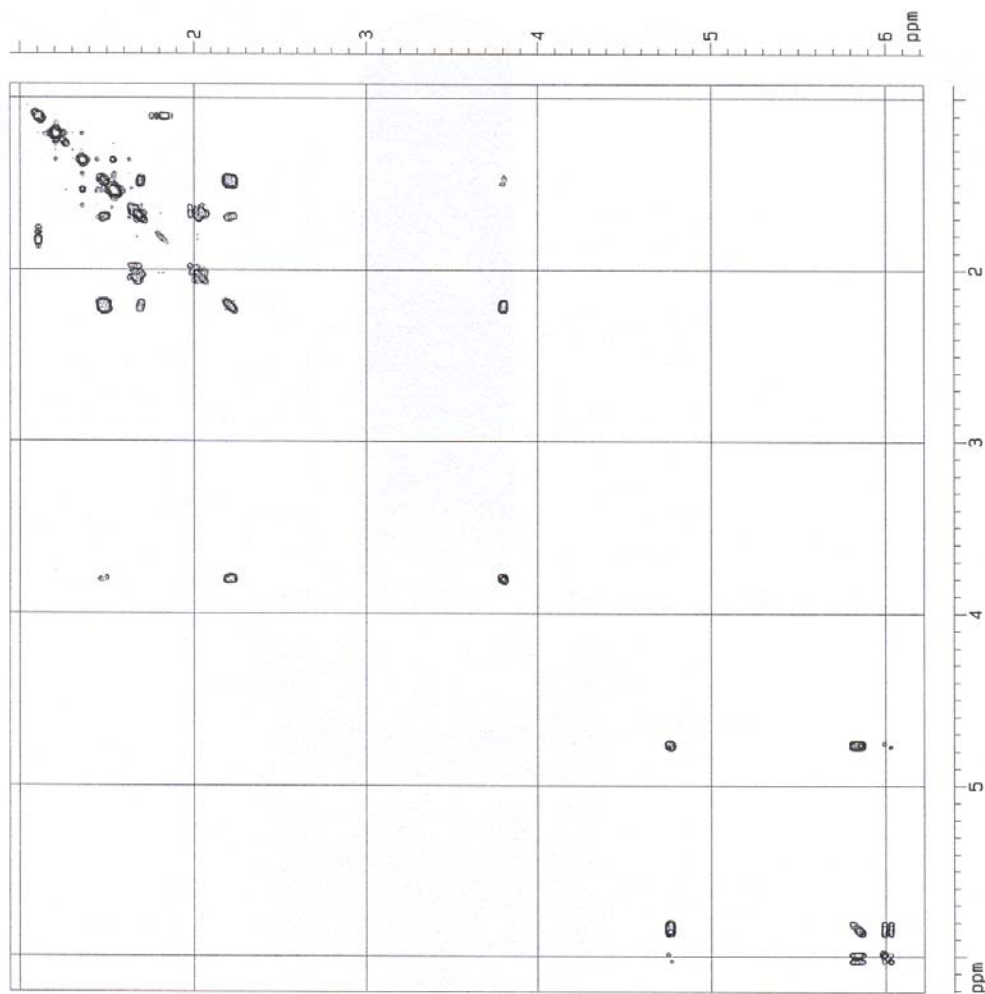
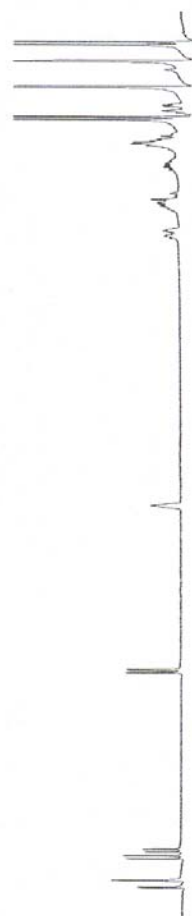
Current Data Parameters  
 NAME ed29\_3.261.D  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 980329  
 Time 18.48  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 AQ 1.0485960 sec  
 FIDRES 0.476637 Hz  
 DQ 16.0 usec  
 RG 32768  
 NUCLEUS 13C  
 RL1 1 dB  
 D1 2.0000000 sec  
 S1 1 dB  
 P3 10.3 usec  
 SF02 400.1360000 MHz  
 D2 0.0035714 sec  
 P4 20.6 usec  
 P1 6.2 usec  
 P2 12.4 usec  
 S2 23 dB  
 DE 22.9 usec  
 SF01 100.622887 MHz  
 SWH 31250.00 Hz  
 TO 65536  
 P31 100.0 usec  
 NS 1574  
 DS 4

F1 - Processing parameters  
 SI 131072  
 MC2 OF  
 SF 100.6139654 MHz  
 NDW EN  
 SSB 0  
 LB 2.00 Hz  
 GB 0

1D NMR plot parameters  
 Cx 23.10 cm  
 FIP 166.172 ppm  
 F1 16719.21 Hz  
 F2P -13.378 ppm  
 F2 -1346.05 Hz  
 PPMCM 7.77275 ppm/cm  
 HZCM 782.04620 Hz/cm





Current Data Parameters  
NAME edes3.261.100  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters

Date 990325  
Time 20.13  
PULPROG zgpg30  
SOLVENT CDCl<sub>3</sub>  
AQ 0.2017480 sec  
FIDRES 2.47655 Hz  
CN 197.0 usec  
RG 2048  
NUCLEUS <sup>1</sup>H  
HL1 1.5000000 sec  
D1 12.0 usec  
P1 0.0000000 sec  
DE 281.4 usec  
SF01 400.136063 MHz  
SWH 2538.107 Hz  
ID 1024  
RS 6  
RG 4  
INQ 0.0002946 sec

F1 - Acquisition Parameters

NEO  
TD 354  
SF01 400.1359 MHz  
FIDRES 9.514395 Hz  
SW 6.343 kHz

F1 - Processing Parameters

SI 512  
MC2 512  
SF 400.1343355 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

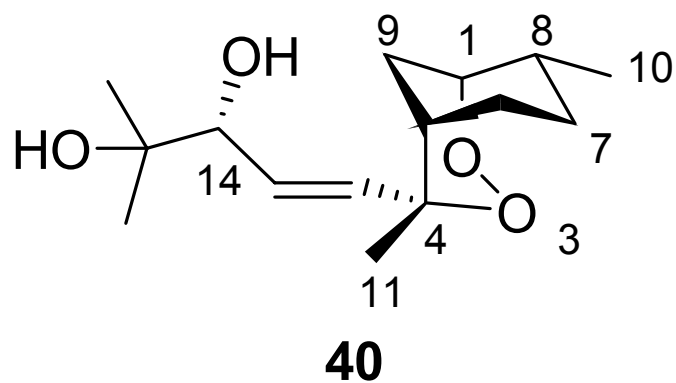
F1 - Processing Parameters

SI 512  
MC2 512  
SF 400.1343355 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

2D NMR plot parameters

CH2 15.00 cm  
CX1 15.00 cm  
F2RLO 6.205 cm  
F2LO 2484.33 Hz  
F2PHI 0.913 deg  
F2U1 367.61 Hz  
F2RLO 6.224 cm  
F2LO 2489.28 Hz  
F2PHI 0.944 deg  
F2U1 377.53 Hz  
F2PHI2M 0.35567 deg/cm  
F2R2CM 141.11415 Hz/cm  
F2PHI2M 0.35184 deg/cm  
F2R2CM 140.78305 Hz/cm

Epimer of yingzhaosu A **40**:



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra





# Current Data Parameters

NAME as2.59a220800C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20000822  
Time 13.35  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpgc  
TD 8192  
SOLVENT CDCl3  
NS 153  
DS 0  
SWH 13089.005 Hz  
FIDRES 1.597779 Hz  
AQ 0.3129844 sec  
RG 6502  
DW 38.200 usec  
DE 6.00 usec  
TE 300.0 K  
D1 4.00000000 sec  
D11 0.03000000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 6.20 usec  
PL1 -6.00 dB  
SFO1 100.6207623 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 120.00 dB  
PL12 14.00 dB  
SFO2 400.1315005 MHz

F2 - Processing parameters  
SI 16384  
SF 100.6127722 MHz  
WDW EM  
SSB 0  
LB 3.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 144.000 ppm  
F1 14488.24 Hz  
F2P 15.000 ppm  
F2 1509.19 Hz  
PPHMM 6.45000 ppm/cm  
HZCM 648.95239 Hz/cm

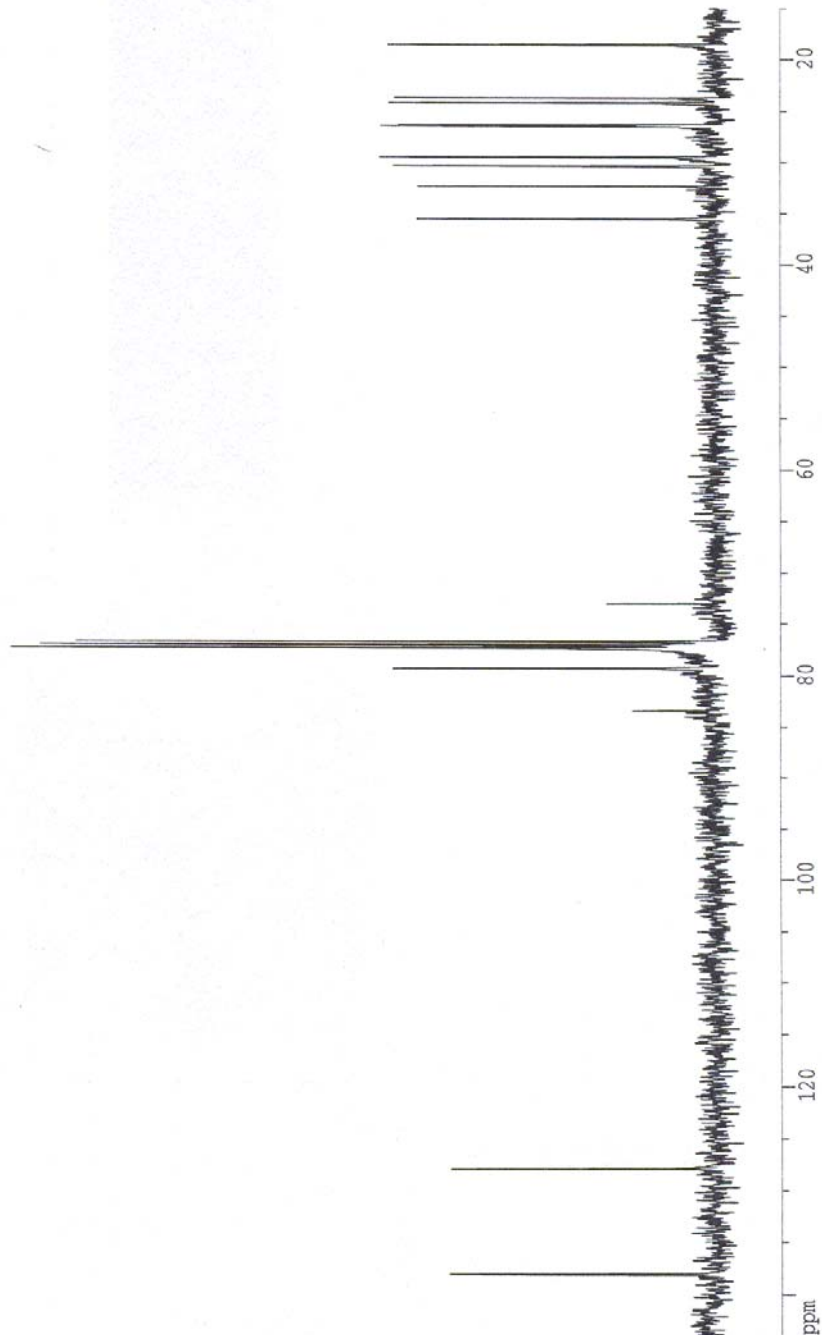
18.617  
23.777  
24.250  
26.409  
26.502  
29.495  
30.303  
32.274  
35.505

72.975  
76.682  
77.000  
77.318  
79.241  
79.327  
83.402

127.839

138.081

ppm



Current Data Parameters  
 NAME as2.59a220800D  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20000822  
 Time 13.50  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 191  
 DS 8  
 SWH 25125.629 Hz  
 FIDRES 0.383387 Hz  
 AQ 1.3042164 sec  
 RG 11585.2  
 DW 19.900 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 D2 0.00345000 sec  
 D12 0.00002000 sec  
 DELTA 6366.18261719 sec

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

NUC1 13C  
 P1 6.20 usec  
 P2 12.40 usec  
 PL1 -6.00 dB  
 SFO1 100.6227903 MHz

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

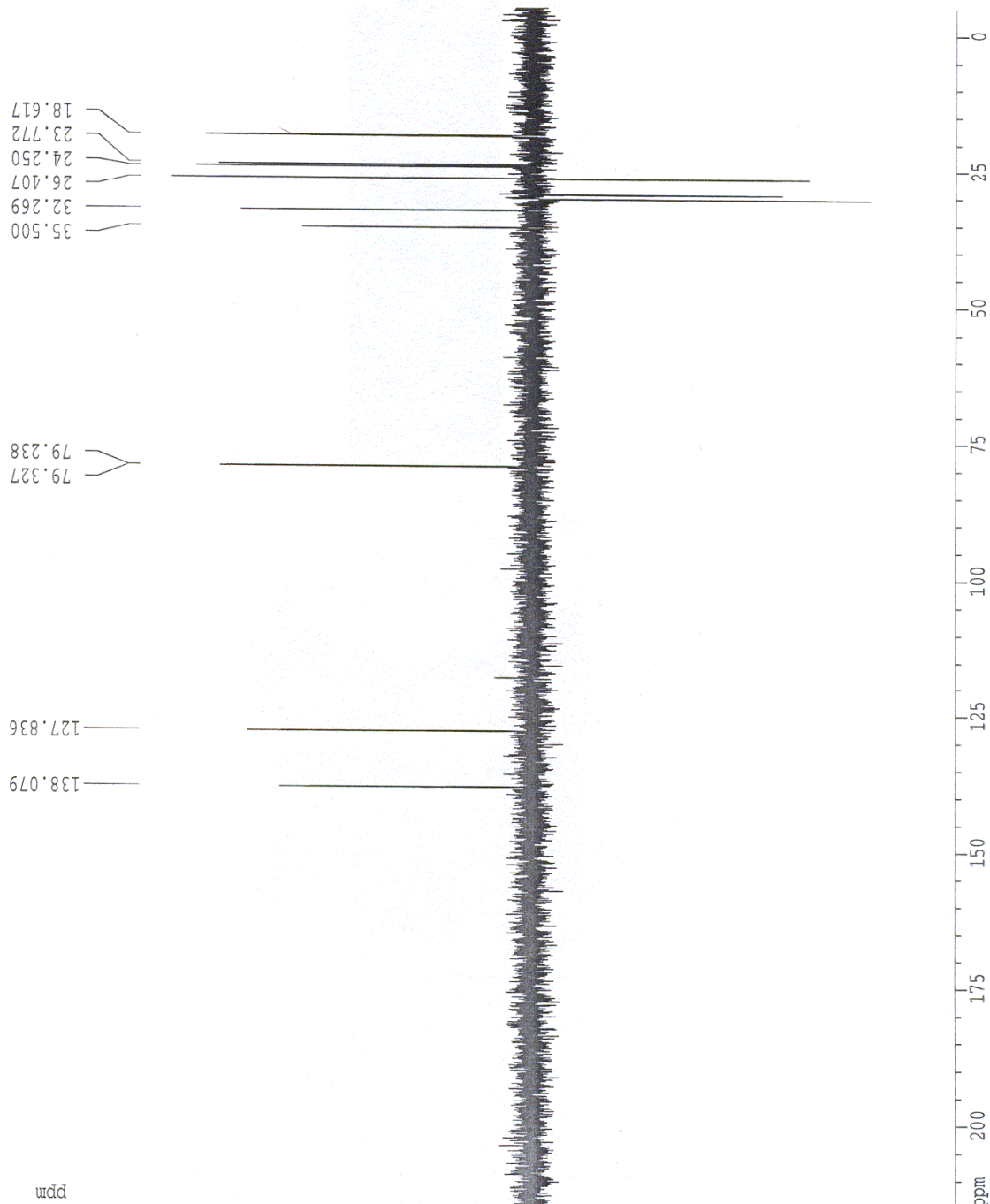
CPDPRG2 waitz16  
 NUC2 1H  
 P3 13.05 usec  
 P4 26.10 usec  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 14.00 dB  
 SFO2 400.1308003 MHz

F2 - Processing parameters

SI 32768  
 SF 100.6127724 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

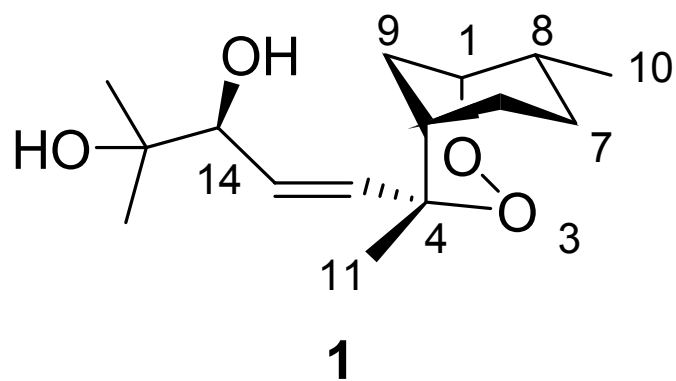
1D NMR plot parameters

CX 20.00 cm  
 F1P 215.000 ppm  
 F1 21631.75 Hz  
 F2P -5.000 ppm  
 F2 -503.06 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 1106.74048 Hz/cm





Yingzhaosu A (**1**):



Recorded at 400 MHz( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ) in  $\text{CDCl}_3$ :

$^1\text{H}$  NMR spectra

$^{13}\text{C}$  NMR spectra

DEPT spectra

COSY spectra

HMQC spectra

Current Data Parameters  
 NAME as2.60a220800H  
 EXPNO 1  
 PROCNO 1

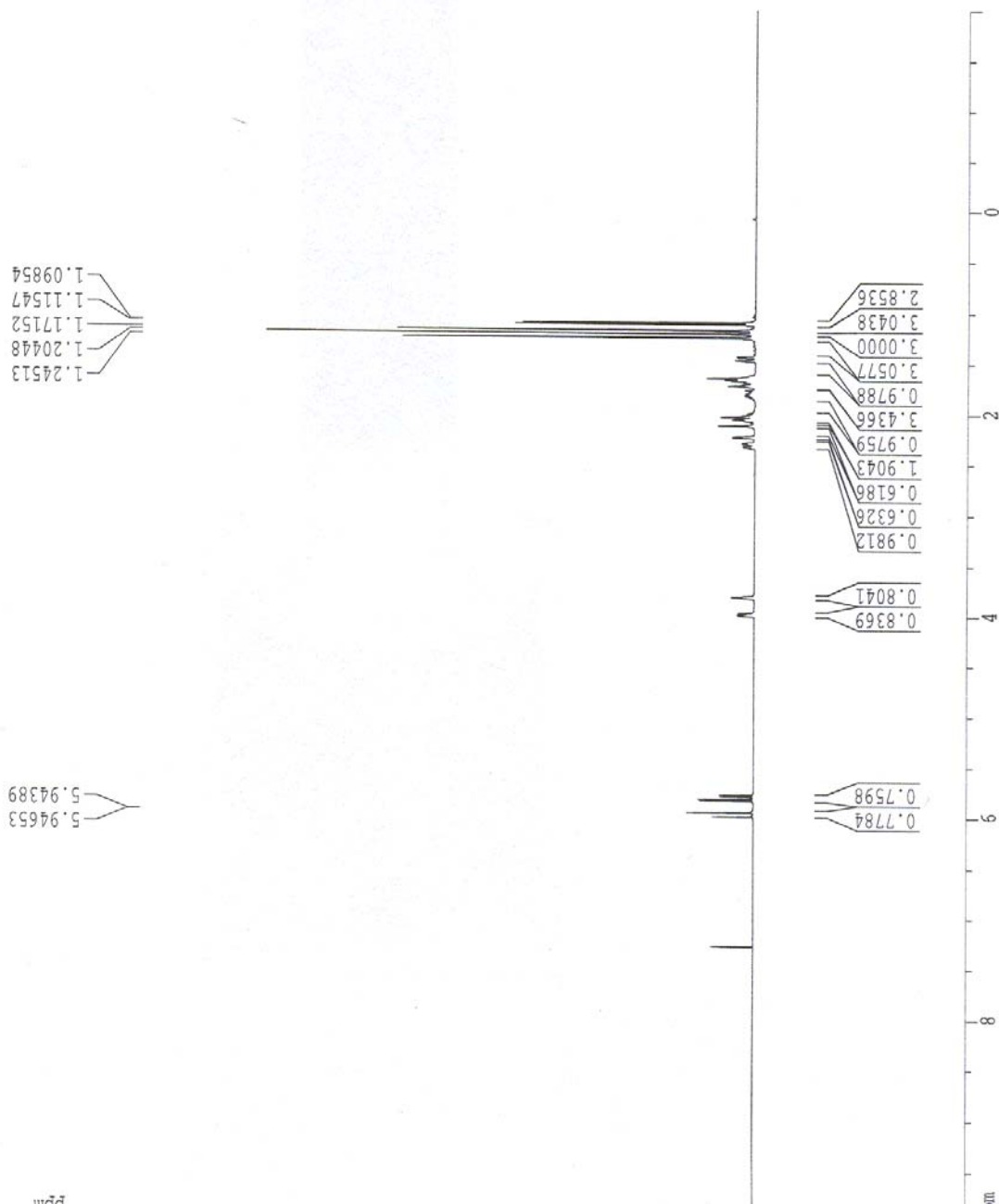
F2 - Acquisition Parameters  
 Date\_ 20000822  
 Time 11.57  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 75  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.244532 Hz  
 AQ 2.0447711 sec  
 RG 181  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 DL 1.50000000 sec

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

NUC1 1H  
 P1 13.00 usec  
 PL1 -4.00 dB  
 SFO1 400.1320007 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300053 MHz  
 WDW EM  
 SSB 0  
 LB 0.01 Hz  
 GB 0  
 PC 0.40

1D NMR plot parameters  
 CX 20.00 cm  
 FIP 10.000 ppm  
 F1 4001.30 Hz  
 F2P -2.000 ppm  
 F2 -800.26 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 240.07800 Hz/cm



Current Data Parameters  
 NAME as2.60a220800C  
 EXPNO 2  
 PROCNO 1

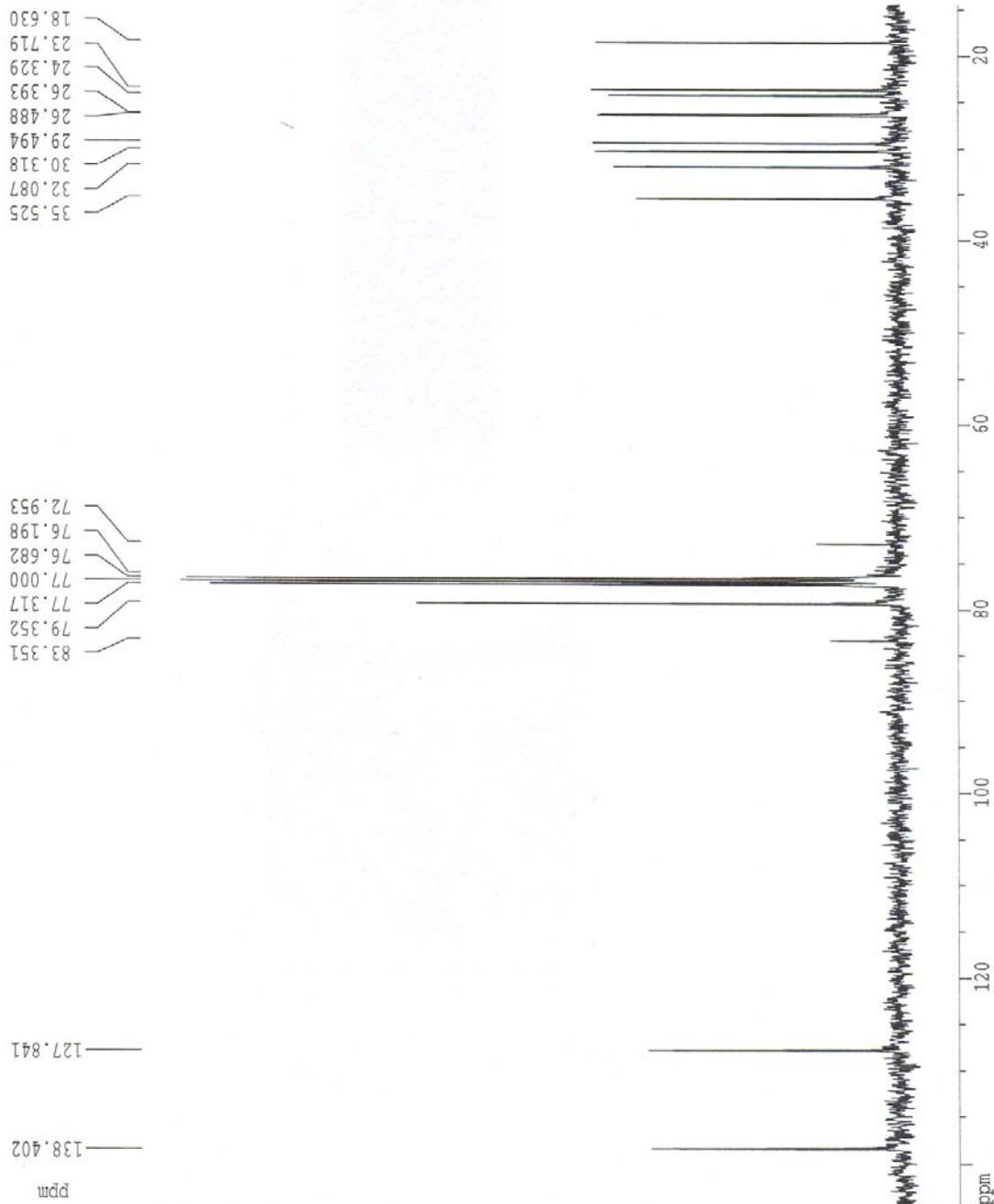
F2 - Acquisition Parameters  
 Date\_ 20000822  
 Time 12.49  
 INSTRUM spect  
 PROHD 5 mm QNP 1H  
 PULPROG zgpg  
 TD 8192  
 SOLVENT CDCl3  
 NS 294  
 DS 0  
 SWH 13089.005 Hz  
 FIDRES 1.597779 Hz  
 AQ 0.3129844 sec  
 RG 1448.2  
 DW 38.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 4.00000000 sec  
 D11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.20 usec  
 PL1 -6.00 dB  
 SFO1 100.6207623 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 120.00 dB  
 PL12 14.00 dB  
 SFO2 400.1316005 MHz

F2 - Processing Parameters  
 SI 16384  
 SF 100.6127714 MHz  
 RMW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 FIP 144.458 ppm  
 F1 14555.35 Hz  
 F2P 14.375 ppm  
 F2 1446.34 Hz  
 PPMCM 6.50464 ppm/cm  
 HZCM 654.45026 Hz/cm

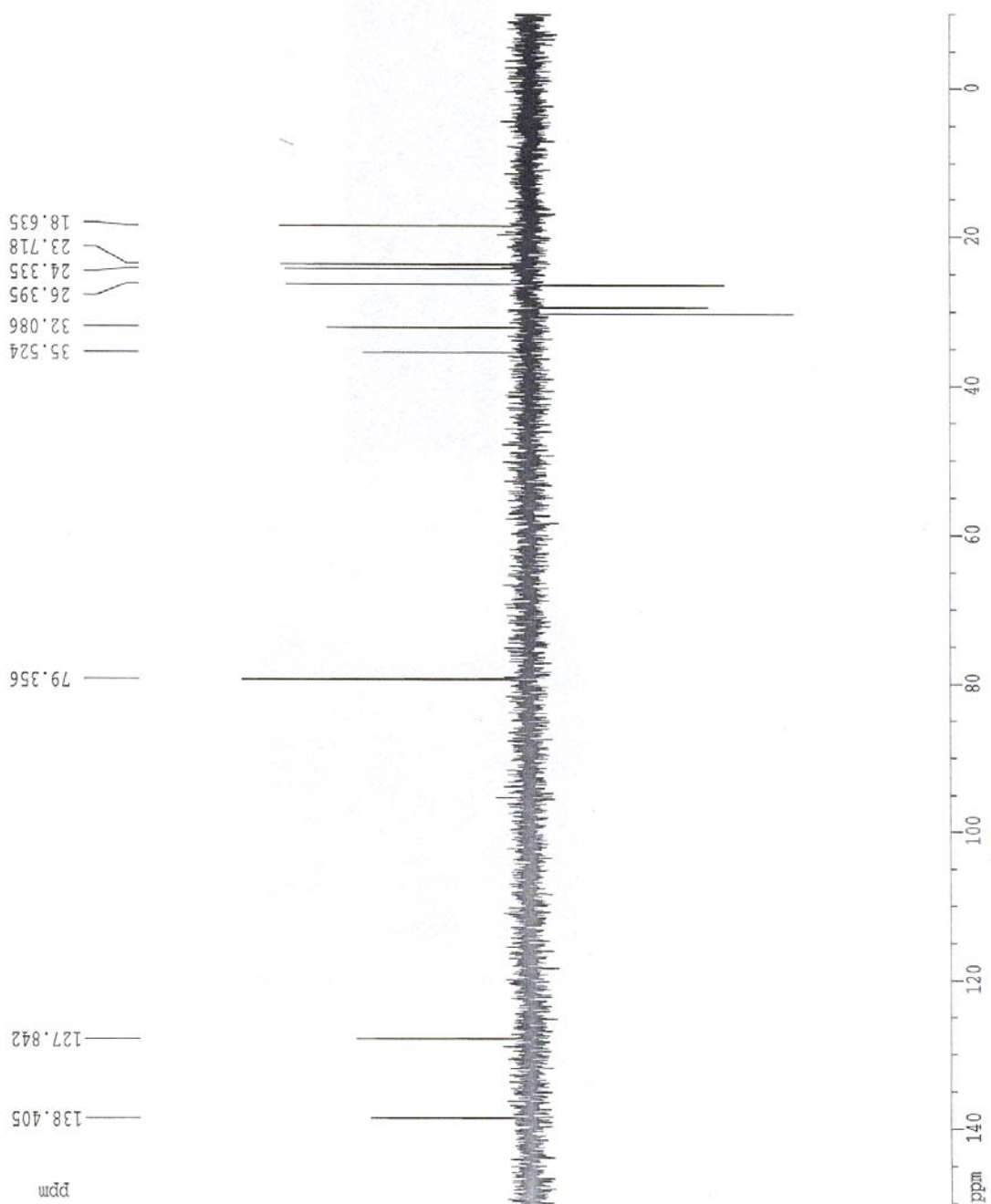


Current Data Parameters  
 NAME as2.60a20800D  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20000822  
 Time 11.13  
 INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 135  
 DS 8  
 SWH 25125.629 Hz  
 FIDRES 0.381387 Hz  
 AQ 1.3042164 sec  
 RG 11585.2  
 DM 19.900 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 D2 0.00345000 sec  
 D12 0.0002000 sec  
 DELTA 6366.1826719 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.20 usec  
 P2 12.40 usec  
 PL1 -6.00 dB  
 SF01 100.627903 MHz  
 ===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P3 13.05 usec  
 P4 26.10 usec  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 14.00 dB  
 SF02 400.1328003 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 100.6127714 MHz  
 MDW EN  
 SSS 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 150.000 ppm  
 F1 15091.92 Hz  
 F2P -10.000 ppm  
 F2 -1006.13 Hz  
 FPCNM 8.00000 ppm/cm  
 HZCM 804.90216 Hz/cm





as2-63A yingzhaosu F

Current Data Parameters  
NAME as2-63A11000  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20011105  
Time 19:02  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 1324  
SOLVENT CDCl3  
NS 4  
DS 16  
SWH 2620.543 Hz  
FIDRES 0.138425 Hz  
AQ 0.158418 sec  
RG 328  
RM 130.800 usec  
DE 6.00 usec  
TE 300.0 K  
D0 0.0000000 sec  
D1 1.0000000 sec  
D2 0.0000000 sec  
D3 0.0000000 sec  
D4 0.0000000 sec  
D5 0.0000000 sec  
D6 0.0000000 sec  
D7 0.0000000 sec  
D8 0.0000000 sec  
D9 0.0000000 sec  
D10 0.0000000 sec  
D11 0.0000000 sec  
D12 0.0000000 sec  
D13 0.0000000 sec  
D14 0.0000000 sec  
D15 0.0000000 sec  
D16 0.0000000 sec  
D17 0.0000000 sec  
D18 0.0000000 sec  
D19 0.0000000 sec  
D20 0.0000000 sec  
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D24 0.0000000 sec  
D25 0.0000000 sec  
D26 0.0000000 sec  
D27 0.0000000 sec  
D28 0.0000000 sec  
D29 0.0000000 sec  
D30 0.0000000 sec  
D31 0.0000000 sec  
D32 0.0000000 sec  
D33 0.0000000 sec  
D34 0.0000000 sec  
D35 0.0000000 sec  
D36 0.0000000 sec  
D37 0.0000000 sec  
D38 0.0000000 sec  
D39 0.0000000 sec  
D40 0.0000000 sec  
D41 0.0000000 sec  
D42 0.0000000 sec  
D43 0.0000000 sec  
D44 0.0000000 sec  
D45 0.0000000 sec  
D46 0.0000000 sec  
D47 0.0000000 sec  
D48 0.0000000 sec  
D49 0.0000000 sec  
D50 0.0000000 sec  
D51 0.0000000 sec  
D52 0.0000000 sec  
D53 0.0000000 sec  
D54 0.0000000 sec  
D55 0.0000000 sec  
D56 0.0000000 sec  
D57 0.0000000 sec  
D58 0.0000000 sec  
D59 0.0000000 sec  
D60 0.0000000 sec  
D61 0.0000000 sec  
D62 0.0000000 sec  
D63 0.0000000 sec  
D64 0.0000000 sec  
D65 0.0000000 sec  
D66 0.0000000 sec  
D67 0.0000000 sec  
D68 0.0000000 sec  
D69 0.0000000 sec  
D70 0.0000000 sec  
D71 0.0000000 sec  
D72 0.0000000 sec  
D73 0.0000000 sec  
D74 0.0000000 sec  
D75 0.0000000 sec  
D76 0.0000000 sec  
D77 0.0000000 sec  
D78 0.0000000 sec  
D79 0.0000000 sec  
D80 0.0000000 sec  
D81 0.0000000 sec  
D82 0.0000000 sec  
D83 0.0000000 sec  
D84 0.0000000 sec  
D85 0.0000000 sec  
D86 0.0000000 sec  
D87 0.0000000 sec  
D88 0.0000000 sec  
D89 0.0000000 sec  
D90 0.0000000 sec  
D91 0.0000000 sec  
D92 0.0000000 sec  
D93 0.0000000 sec  
D94 0.0000000 sec  
D95 0.0000000 sec  
D96 0.0000000 sec  
D97 0.0000000 sec  
D98 0.0000000 sec  
D99 0.0000000 sec  
D100 0.0000000 sec

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

NUC1 1H  
P1 13.00 usec  
PL1 0.00 dB  
SFO1 400.1316413 MHz

F1 - Acquisition Parameters

NUC2 13C  
P2 12.00 usec  
PL2 0.00 dB  
SFO2 100.6281500 MHz

F2 - Processing Parameters

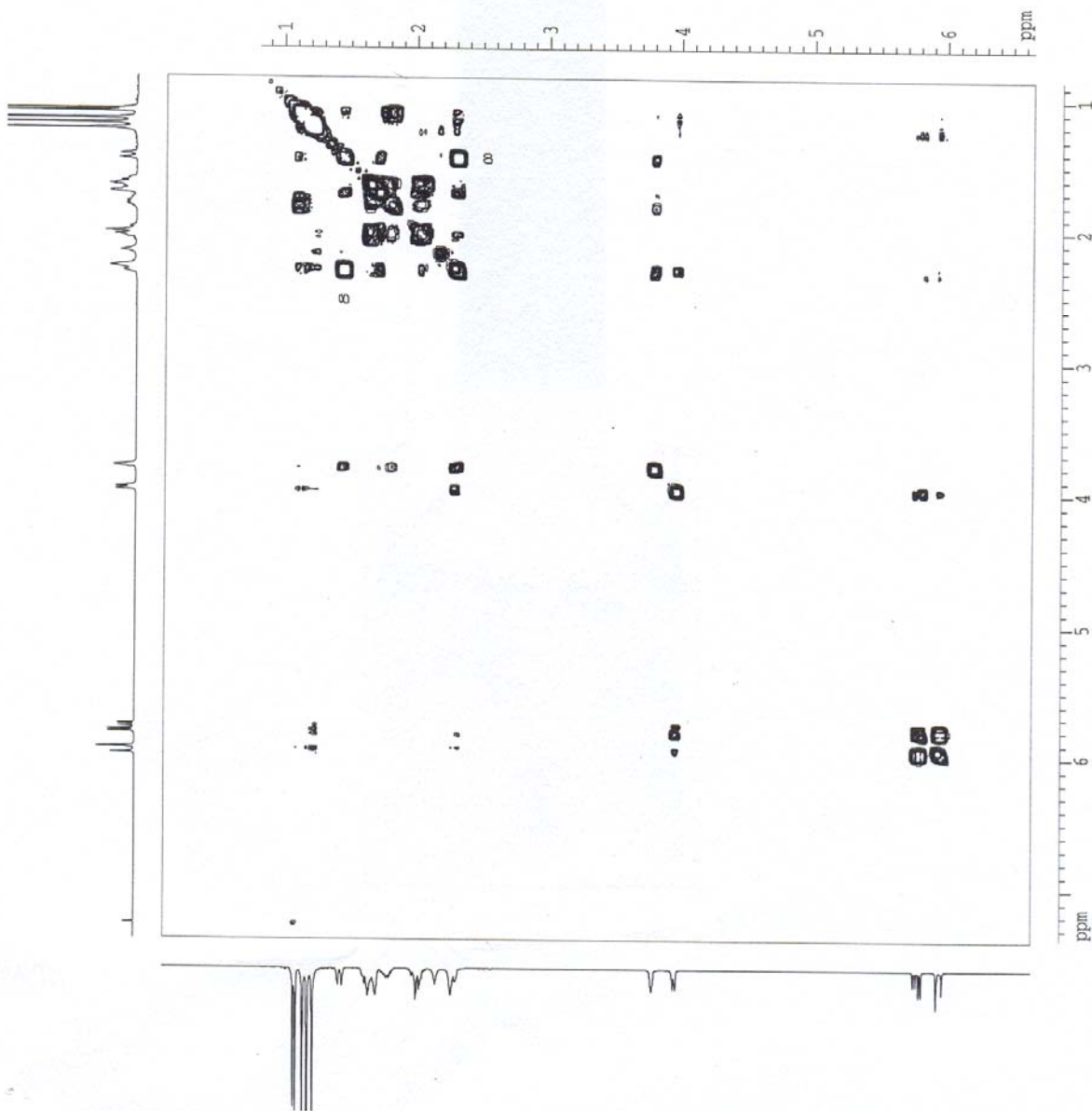
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WDW 0  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

F1 - Processing Parameters

SI 512  
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WDW 0  
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GB 0

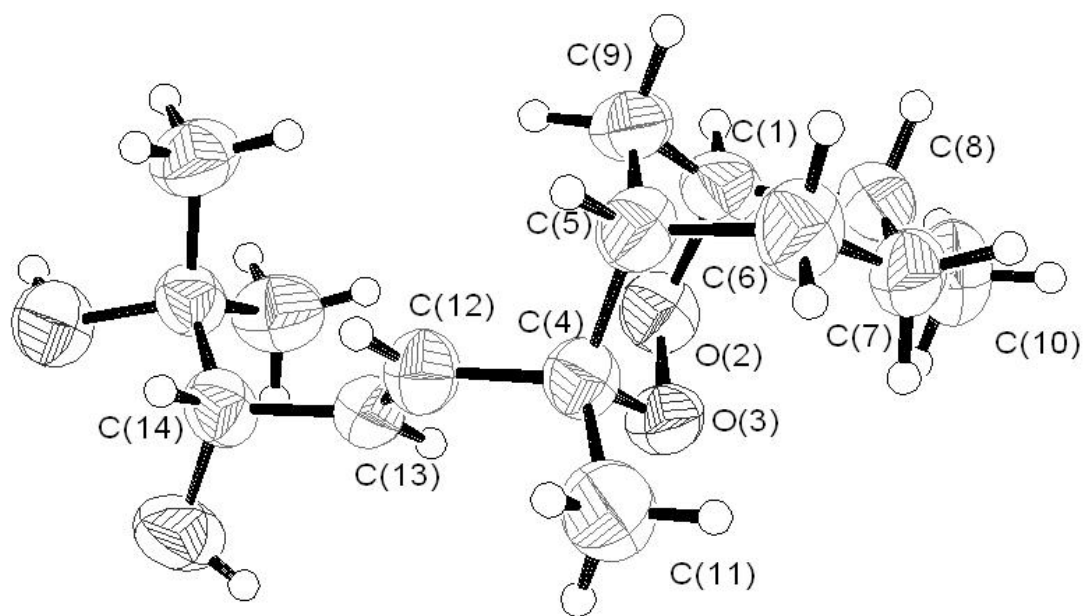
2D NMR plot parameters

CX2 15.00 cm  
CX1 15.00 cm  
F2P2LO 7.391 ppm  
F2LO 2957.43 Hz  
F2PHI 0.842 ppm  
F2P2 336.88 Hz  
F2P1 6.659 ppm  
F2P0 2864.55 Hz  
F2P3 0.111 ppm  
F2P4 44.00 Hz  
F2P5 0.43662 ppm/cm  
F2P6 174.70302 Hz/cm  
F2P7 0.43662 ppm/cm  
F2P8 174.70288 Hz/cm





## X-ray structural data for yingzhaosu A (1)



**Figure S1.** Perspective view (ORTEP) of the X-ray structure of yingzhaosu A (1). Thermal ellipsoids are drawn at the 50% probability level. For more details see the attached CIF file.