Synthesis of silylallene glycosides and diene-diglycosides by *C*-glycosidation of D-glucal with 1,4-bis(trimethylsilyl)-2-butyne

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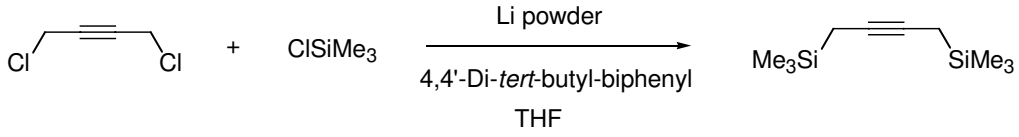
Experimental Section

General Methods:

Proton NMR spectra were recorded on a BRUKER AVANCE-400 (400 MHz). All spectra were measured in $CDCl_3$ solvent and chemical shifts are reported as δ values in parts per million (ppm) relative to tetramethylsilane (δ 0.00) or $CDCl_3$ (δ 7.26) as internal standard. Data are reported as follows; chemical shift (integrate intensity or assignment, multiplicity, coupling constants in Hz, assignment). Carbon NMR spectra were recorded on a BRUKER AVANCE-400 (100 MHz). All spectra were measured in $CDCl_3$ solvent and chemical shifts are reported as δ values in parts per million (ppm) relative to $CDCl_3$ (δ 77.0) as internal standard. Low-resolution FAB mass spectra were obtained with a JEOL JSM-Lcmate MS spectrometer. Infrared spectra were determined on a JASCO FT/IR-8300 spectrophotometer and are reported in wave number (cm-1). Melting points were determined on a Yanaco MP-S3 and are uncorrected. Optical rotation was determined with a JASCO DIP-370 digital polarimeter. Elemental analyses were performed by the Analytical Laboratory of the School of Bioagricultural Sciences.

Analytical thin-layer chromatrography (tlc) was conducted on precoated tlc plates; silica gel 60F-254 [E. Merck , Darmstadt, Germany] using UV light as visualizing agent and p-anisaldehyde solution and heated as developing agent. Silica gel columns for open-column chromatrography utilized silica gel 60 (particle size 0.063-0.2) [E. Merck , Darmstadt, Germany]. Dry CH_2Cl_2 was distilled from CaH_2 under N_2 atmosphere. $BF_3.OEt_2$ was distilled from CaH_2. All other commercially available reagents were used as received.

Preparation of 1,4-Bis(trimethylsilyl)-2-butyne (5)



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To a green suspension of Li powder 1g (140 mmol) and DTBB (0.26 g, 1 mmol) in dry THF 30 ml. at –40ºC was added a solution of 1,4-dichloro-2-butyne 0.98 ml. (10 mmol) and TMSCl 3 ml. (20 mmol) in THF 30 ml. during 1.5 h by using a syringe pump. Then the reaction mixture was carefully hydrolyzed with water and extracted with diethyl ether. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by vacuum distillation to afford the product as colorless liquid (oil bath temp. = 111-128ºC, 0.01 Mpar) 1.49 g (75%)

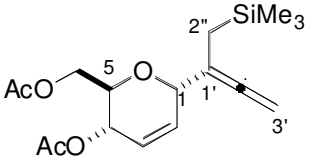
Preparation of the green suspension Li powder and DTBB

Wash Li powder (Lithium dispersion in paraffin liquid, Kanto24244-25) with hexane in the 2 necked flask and decant. The residual mass was evaporated and dried under vacuum. To a suspension of Li in THF (25 ml) was dropwise added a solution of 4,4'-Di-tert-butyl-biphenyl (DTBB) in THF (5 ml) at room temperature. The reaction turned to green color after finish addition.

1,4-Bis(trimethylsilyl)-2-butyne ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 0.09 (18H, s, 2X$SiMe_3$), 1.45 (4H, s, 2XCH_2). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ -2.0 ((CH_3)$_3$Si), 7.2 (CH_2), 75.6 (C≡C)

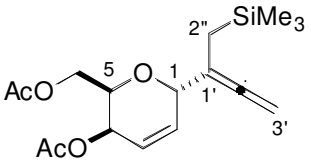
General procedure for the synthesis of alleneglycoside

To a solution of Glycal (0.36 mmol) and the 1,4-Bis(trimethylsilyl)-2-butyne (0.45 mmol) in anhydrous CH_2Cl_2 (35 ml) was added the solution of $BF_3.OEt_2$ (0.36 mmol) in anhydrous CH_2Cl_2 (5 ml) at –20°C. After TLC showed the complete conversion, the reaction was poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (1:2 Et_2O:hexane) to afford alleneglycoside.



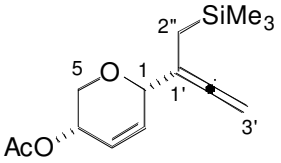
Compound 6. $[\alpha]_D^{27}$ 156.1° (c = 1.3, $CHCl_3$). IR (KBr): 2954, 1951, 1743, 1370, 1239 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 0.08 (9H, s, $SiMe_3$), 1.47 (2H, m, H-2"), 2.10 (3H, s, *Ac*), 2.11 (3H, s, *Ac*), 3.89 (1H, ddd, *J* = 8.5, 5.7, 3.0 Hz, H-5), 4.17 (1H, dd, *J* = 12.0, 3.0 Hz, H-6a), 4.24 (1H, dd, *J* = 12.0, 5.7 Hz, H-6b), 4.60 (1H, d, *J* = 2.5 Hz, H-1), 4.78 (2H, m, H-3'), 5.28 (1H, ddd, *J* = 8.5, 3.5, 2.0 Hz, H-4), 5.79 (1H, dt, *J* = 10.0, 2.0 Hz, H-2), 5.93 (1H, ddd, *J* = 10.0, 3.5, 2.0 Hz, H-3). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ -1.2 , 18.0, 20.8, 21.1, 63.3, 65.5, 68.6, 74.4, 76.8, 99.6, 124.7, 130.9, 170.4, 170.8, 208.0.

Anal. Calcd. For $C_{17}H_{26}O_5Si$: C, 60.32; H, 7.74 Found: C, 60.31; H, 7.69



Compound 8. $[\alpha]_D^{28}$ –403.5° (c = 0.8, $CHCl_3$). IR (KBr): 2955, 1950, 1747, 1371, 1230 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 0.07 (9H, s, $SiMe_3$), 1.44 (1H, dt, *J* = 15.0, 2.5 Hz, H-2"a), 1.50 (1H, dt, *J* = 15.0, 3.0 Hz, H-2"b), 2.07 (3H, s, *Ac*), 2.09 (3H, s, *Ac*), 4.08 (1H, ddd, *J* = 7.0, 5.7, 2.5 Hz, H-5), 4.19 (1H, dd, *J* = 11.4, 7.0 Hz, H-6a), 4.23 (1H, dd, *J* = 11.4, 5.7 Hz, H-6b), 4.64 (1H, m, H-1), 4.72 (2H, m, H-3'), 5.03 (1H, dd, *J* = 5.0, 2.5 Hz, H-4), 5.99 (1H, ddd, *J* = 10.0, 5.0, 2.0 Hz, H-3), 6.10 (1H, dd, *J* = 10.0, 3.5 Hz, H-2). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ -1.2 , 17.8, 20.8, 20.9, 63.1, 63.9, 68.1, 74.1, 76.7, 98.9, 121.9, 133.8, 170.5, 170.6, 207.8.

Anal. Calcd. For $C_{17}H_{26}O_5Si$: C, 60.32; H, 7.74 Found: C, 60.44; H, 7.77

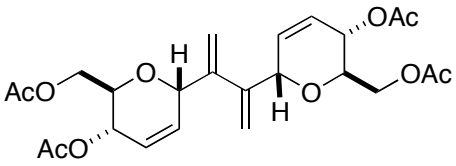


Compound 10. $[\alpha]_D^{26}$ +142.9° (c = 1.3, $CHCl_3$). IR (KBr): 2954, 1950, 1740, 1371, 1244 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 0.08 (9H, s, $SiMe_3$), 1.40 (1H, dt, *J* = 14.5, 2.5 Hz, H-2"a), 1.48 (1H, dt, *J* = 14.5, 2.5 Hz, H-2"b), 2.11 (3H, s, *Ac*), 3.73 (1H dd, *J* = 12.5, 3.0 Hz, H-5a), 4.01 (1H, dd, *J* = 12.5, 3.5 Hz, H-5b), 4.56 (1H, brs, H-1), 4.74 (1H, m, H-3'), 5.06 (1H, dd, *J* = 6.5, 3.0 Hz, H-4), 5.95 (1H, ddd, *J* = 10.0, 4.0, 1.0 Hz, H-2), 6.50 (1H, dd, *J* = 10.0, 3.0 Hz, H-3). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ -1.1, 17.9, 21.2, 63.5, 64.6, 74.2, 76.5, 99.0, 122.7, 133.61, 170.1, 208.2.

Anal. Calcd. For $C_{14}H_{22}O_3Si$: C, 63.12; H, 8.32 Found: C, 63.12; H, 8.32

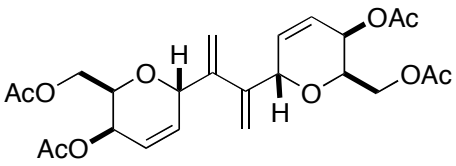
General procedure for the synthesis of symmetric diene glycoside

To a solution of Glycal (0.74 mmol) and the 1,4-Bis(trimethylsilyl)-2-butyne (0.25 mmol) in anhydrous CH_2Cl_2 (35 ml) was added the solution of $SnCl_4$ (0.37 mmol) in anhydrous CH_2Cl_2 (5 ml) at –20°C. After TLC showed the complete conversion, the reaction was poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (2:1 Et_2O:hexane) to afford symmetric diene glycoside.



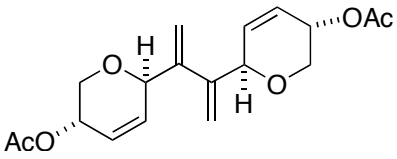
Compound 13. mp. = 125-126°C $[\alpha]_D^{27}$ +23.4° (c = 0.8, $CHCl_3$). IR (KBr): 2952, 1743, 1235, 1048 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 2.07 (6H, s, 2x*Ac*), 2.09 (6H, s, 2x*Ac*), 3.81 (2H, ddd, *J* = 8.5, 6.0, 3.0 Hz, 2xH-5), 4.11 (2H, dd, *J* = 12.5, 3.0 Hz, 2xH-6a), 4.19 (2H, dd, *J* = 12.0, 6.0 Hz, 2xH-6b), 5.07 (2H, d, *J* = 1.5 Hz, 2xH-1), 5.20 (2H, s, 2xH-2'a), 5.28 (2H, ddd, *J* = 8.5, 3.5, 2.0 Hz, 2xH-4), 5.56 (2H, s, H-2'b), 5.91 (2H, dt, *J* = 10.0, 2.0 Hz, 2xH-2), 6.04 (2H, ddd, *J* = 10.0, 3.5, 1.5 Hz, 2xH-3). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ 20.8, 21.0, 63.3, 65.5, 68.5, 72.2, 118.2, 125.9, 130.7, 145.2, 170.3, 170.7.

Anal. Calcd. For $C_{24}H_{30}O_{10}$: C, 60.24; H, 6.32 Found: C, 60.25; H, 6.27



Compound 14. mp. = 157-158°C $[\alpha]_D^{28}$ –349.04° (c = 0.8, $CHCl_3$). IR (KBr): 2927, 1739, 1371, 1227 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 2.01 (6H, s, 2x*Ac*), 2.10 (6H, s, 2x*Ac*), 3.98 (2H, ddd, *J* = 7.5, 5.5, 2.5 Hz, 2xH-5), 4.13 (2H, dd, *J* = 12.0, 7.5 Hz, 2xH-6a), 4.18 (2H, dd, *J* = 12.0, 5.5 Hz, 2xH-6b), 5.06 (2H, dd, *J* = 5.0, 2.5 Hz, 2xH-4), 5.08 (2H, s, 2xH-2'a), 5.16 (2H, brs, 2xH-1), 5.53 (2H, s, 2xH-2'b), 6.12 (2H, ddd, *J* = 10.0, 5.0, 1.5 Hz, 2xH-2), 6.21 (2H, dd, *J* = 10.0, 3.5 Hz, 2xH-3). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ 20.7, 20.9, 62.9, 63.8, 68.1, 72.2, 118.1, 123.6, 133.0, 145.0, 170.5.

Anal. Calcd. For $C_{24}H_{30}O_{10}$: C, 60.24; H, 6.32 Found: C, 60.24; H, 6.24

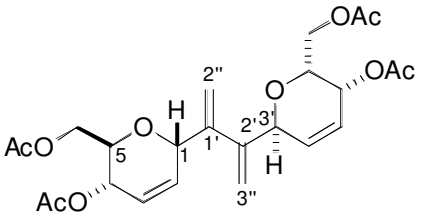


Compound 15 mp. = 87-88°C $[\alpha]_D^{26}$ +290.94° (c = 1.0, $CHCl_3$). IR (KBr): 2861, 1733, 1372, 1237 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 2.08 (6H, s, 2x*Ac*), 3.70 (2H, ddd, *J* = 12.5, 3.0, 0.5 Hz, 2xH-5a), 3.92 (2H, dd, *J* = 12.5, 3.5 Hz, 2xH-5b), 5.02 (2H, brs, 2xH-1), 5.06 (2H, dd, *J* = 7.0, 3.0 Hz, 2xH-4), 5.15 (2H, s, 2xH-2'a), 5.52 (2H, s, 2xH-2'b), 6.00 (2H, dddd, *J* = 10.0, 4.0, 2.0, 0.5 Hz, 2xH-2), 6.10 (2H, ddd, *J* = 10.0, 3.0, 1.0 Hz, 2xH-3). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ 21.1, 63.5, 64.5, 72.4, 117.7, 123.9, 133.1, 144.0, 170.6.

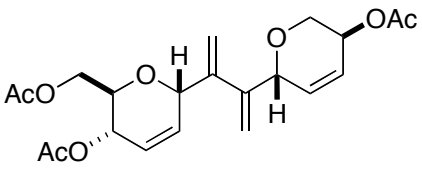
Anal. Calcd. For $C_{18}H_{22}O_6$: C, 64.66; H, 6.63 Found: C, 64.66; H, 6.64

General procedure for the synthesis of asymmetric diene glycoside (1 pot)

To a solution of Glycal (0.37 mmol) and the 1,4-Bis(trimethylsilyl)-2-butyne (0.37 mmol) in anhydrous CH_2Cl_2 (35 ml) was added the solution of $BF_3.OEt_2$ (0.37 mmol) in anhydrous CH_2Cl_2 (5 ml) at –20°C. After TLC showed the complete conversion, another glycal (0.37 mmol) was added to the reaction mixture followed by the solution of $SnCl_4$ (0.37 mmol) in anhydrous CH_2Cl_2 (5 ml). After TLC showed the complete conversion, the reaction was poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (1:1 Et_2O:hexane) to afford asymmetric diene glycoside.

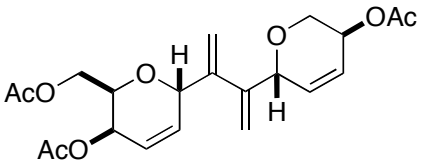


Compound 16. $[\alpha]_D^{28}$ -101.0° (c = 2.0, $CHCl_3$). IR (KBr): 2924, 1740, 1371, 1236, 1048 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 2.01 (3H, s, *Ac*), 2.05 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 2.09 (3H, s, *Ac*), 3.80 (1H, ddd, *J* = 8.5, 6.0, 3.0 Hz, H-5), 3.99 (1H, td, *J* = 6.0, 2.5 Hz, H-7'), 4.11 (1H, dd, *J* = 12.0, 3.0 Hz, H-6a), 4.17 (1H, m, H-8'a), 4.19 (1H, m, H-8'b), 4.26 (1H, dd, *J* = 12.0, 6.0 Hz, H-6b), 5.04-5.09 (2H, m, H-1, H-6'), 5.09 (1H, s, H-3"a), 5.16 (1H, brs, H-3'), 5.19 (1H, s, H-2"a), 5.28 (1H, ddd, *J* = 8.5, 3.5, 2 Hz, H-4), 5.53 (1H, s, H-3"b), 5.56 (1H, s, H-2"b), 5.91 (1H, dt, *J* = 10.0, 2.0 Hz, H-2), 6.04 (1H, ddd, *J* = 10.0, 3.0, 1.5 Hz, H-3), 6.13 (1H, ddd, *J* = 10.0, 5.0, 1.5 Hz, H-4'), 6.21 (1H, dd, *J* = 10.0, 3.5 Hz, H-5'). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ 20.7, 20.8, 20.9, 21.0, 62.9, 63.2, 63.8, 65.4, 68.1, 68.6, 72.1, 72.3, 118.0, 118.2, 123.6, 125.9, 130.6, 133.1, 144.7, 145.4, 170.3, 170.5, 170.6, 170.7. Anal. Calcd. For $C_{24}H_{30}O_{10}$: C, 60.24; H, 6.32 Found: C, 60.25; H, 6.51



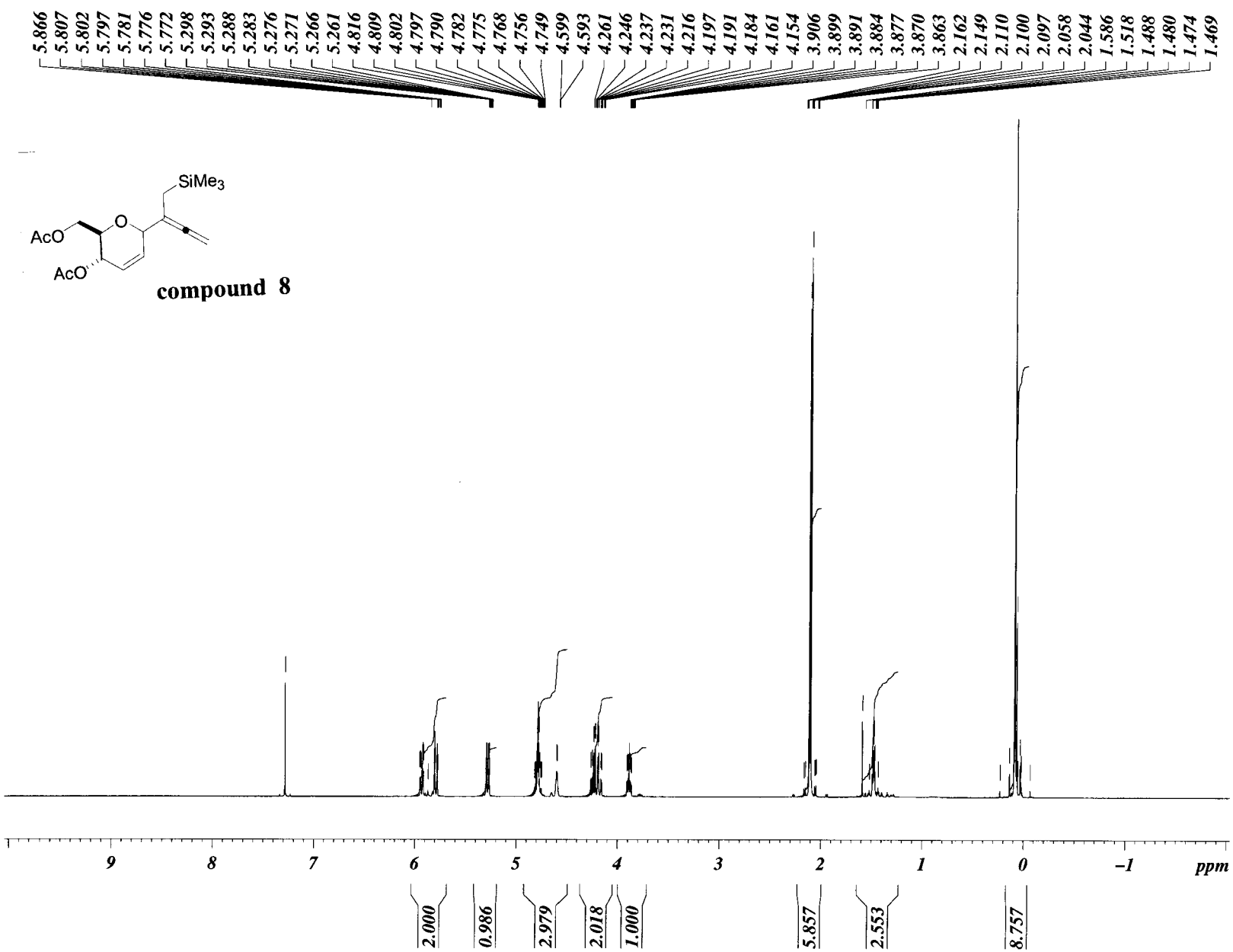
Compound 17 mp. = 77-78°C $[\alpha]_D^{27}$ +156.6° (c = 1.2, $CHCl_3$). IR (KBr): 2936, 1743, 1371, 1236, 1057 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 2.07 (3H, s, *Ac*), 2.09 (3H, s, *Ac*), 2.10 (3H, s, *Ac*), 3.71 (1H, dd, *J* = 12.0, 4.0 Hz, H-7'a), 3.85 (1H, ddd, *J* = 8.0, 6.0, 3.0 Hz, H-5), 4.01 (1H, dd, *J* = 12.0, 4.0 Hz, H-7'b), 4.11 (1H, dd, *J* = 12.0, 3.0 Hz, H-6a), 4.23 (1H, dd, *J* = 12.0, 6.0 Hz, H-6b), 5.01 (1H, d, *J* = 1.5 Hz, H-3'), 5.03 (1H, d, *J* = 2.0 Hz, H-1), 5.13 (1H, dd, *J* = 7.0, 4.0 Hz, H-6'), 5.23 (1H, s, H-3"a), 5.26 (1H, m, H-4), 5.30 (1H, s, H-2"a), 5.55 (1H, s, H-3"b), 5.56 (1H, s, 2xH-2"b), 5.91 (1H, dt, *J* = 10.5, 2.0 Hz, H-2), 6.00 (1H, ddd, *J* = 10.5, 3.0, 1.5 Hz, H-3), 6.01 (1H, m, H-4'), 6.07 (1H, dd, *J* = 10.0, 2.0 Hz, H-5'). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ 20.8, 21.0, 21.1, 63.1, 63.9, 64.6, 65.4, 68.7,

Anal. Calcd. For $C_{21}H_{26}O_8$: C, 62.06; H, 6.45 Found: C, 62.06; H, 6.47



Compound 18 mp. = 78-80°C $[\alpha]_D^{27}$ –47.2° (c = 1.2, $CHCl_3$). IR (KBr): 2936, 1736, 1371, 1236, 1049 cm^{-1}, ^{1}H-NMR (400 MHz, $CDCl_3$) : δ 2.02 (3H, s, *Ac*), 2.09 (3H, s, *Ac*), 2.10 (3H, s, *Ac*), 3.70 (1H, dd, *J* = 12.0, 4.0 Hz, H-7'a), 4.01 (1H, dd, *J* = 12.0, 4.0 Hz, H-7'b), 4.02 (1H, m, H-5), 4.15 (1H, dd, *J* = 11.5, 5.5 Hz, H-6a), 4.19 (1H, dd, *J* = 11.5, 7.5 Hz, H-6b), 4.99 (1H, s, H-6'), 5.06 (1H, dd, *J* = 4.0, 2.5 Hz, H-4), 5.11 (1H, d, *J* = 2.5 Hz, H-3'), 5.13 (1H, d, *J* = 1.0 Hz, H-1), 5.19 (1H, s, H-3"a), 5.25 (1H, s, H-2"a), 5.54 (1H, s, H-3"b), 5.57 (1H, s, H-2"b), 6.01 (1H, m, H-4'), 6.05 (1H, dd, *J* = 10.5, 2.0 Hz, H-5'), 6.12 (1H, dd, *J* = 10.0, 6.0 Hz, H-2), 6.16 (1H, dd, *J* = 10.0, 3.0, 1.5 Hz, H-3). ^{13}C-NMR (100 MHz, $CDCl_3$) : δ 20.8, 20.9, 21.1, 62.8, 63.8, 63.9, 64.6, 67.9, 72.9, 73.3, 117.9, 118.3, 123.6, 124.2, 133.0, 133.2, 142.5, 143.3, 170.4, 170.5, 170.6.

Anal. Calcd. For $C_{21}H_{26}O_8$: C, 62.06; H, 6.45 Found: C, 62.04; H, 6.39



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PROCNO 1

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DS 2
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FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
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PL1 −4.00 dB
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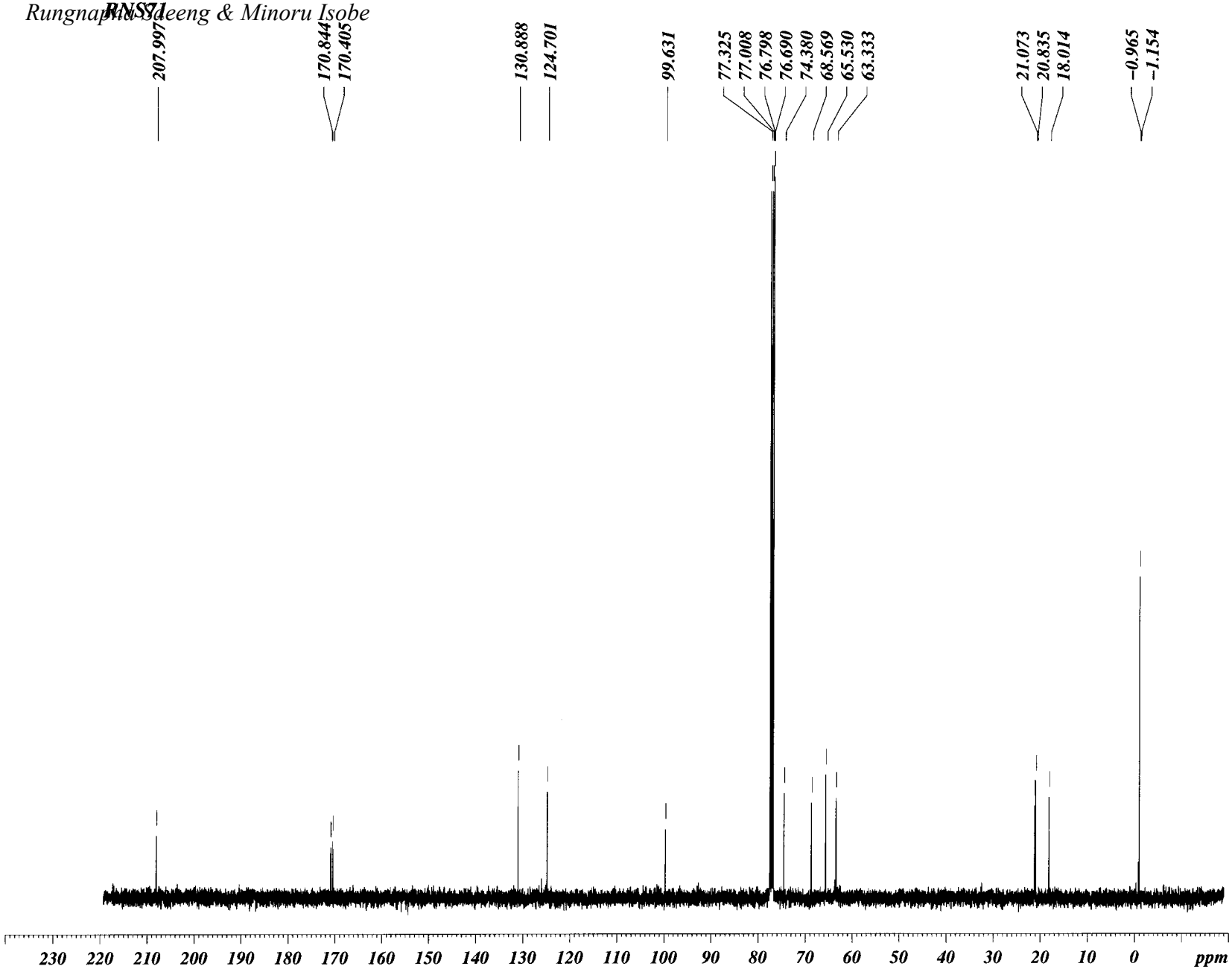

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EXPNO 28
PROCNO 1

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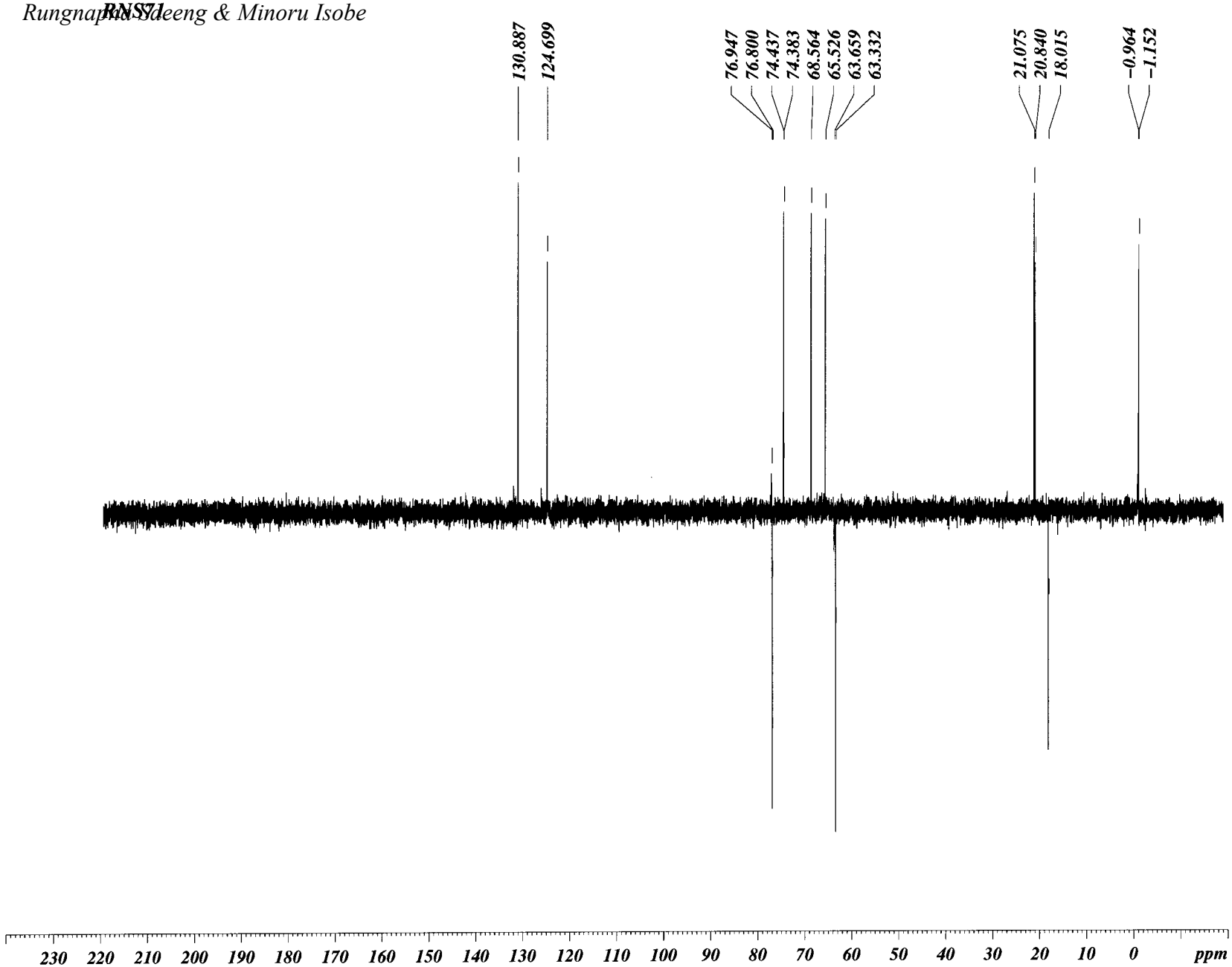
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 −3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 −4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



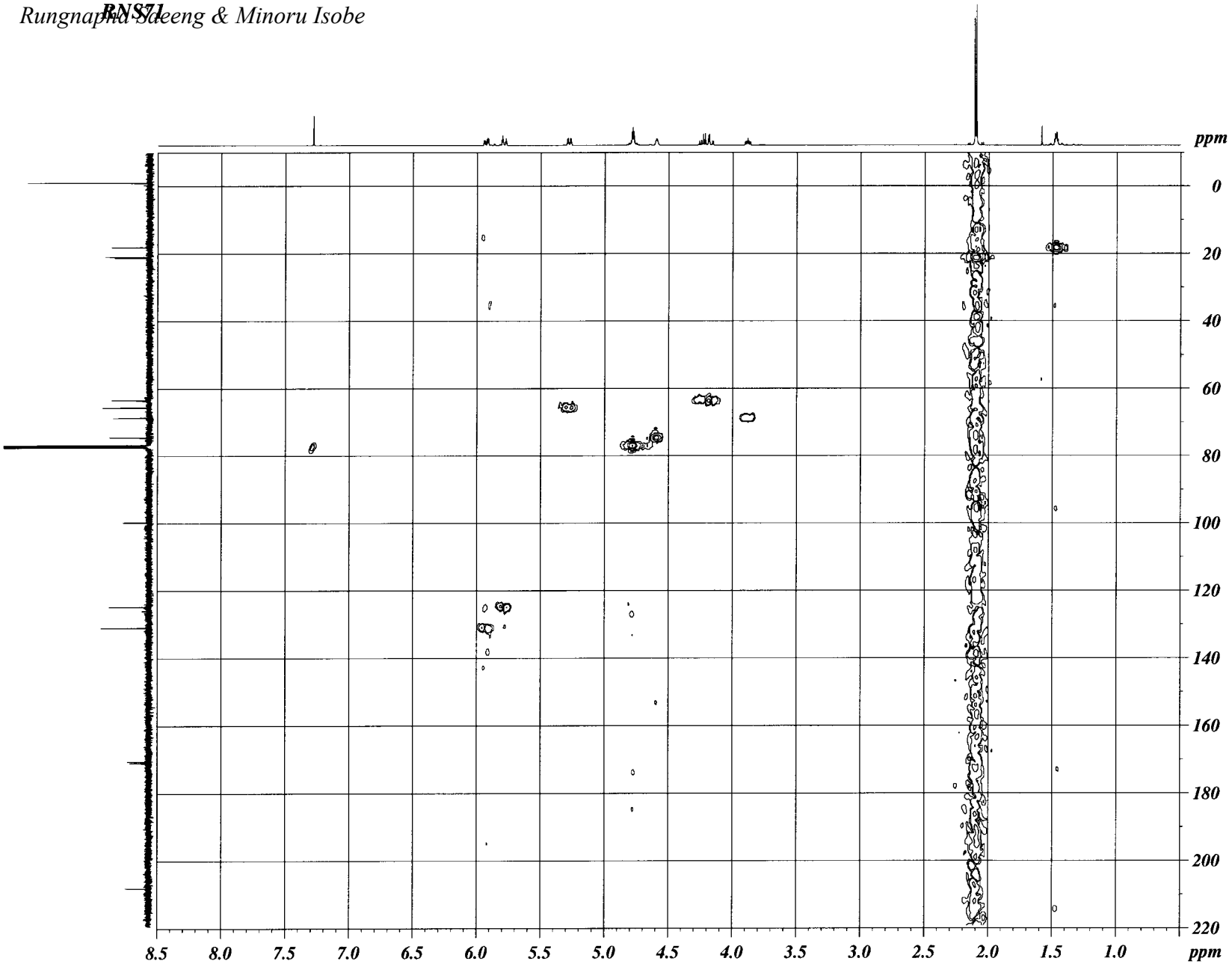
Current Data Parameters
NAME Rungnaphaav400
EXPNO 30
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040421
Time 13.15
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001146 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
p2 18.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
P3 8.60 usec
p4 17.20 usec
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME Rungnaphaav400
EXPNO 27
PROCNO 1

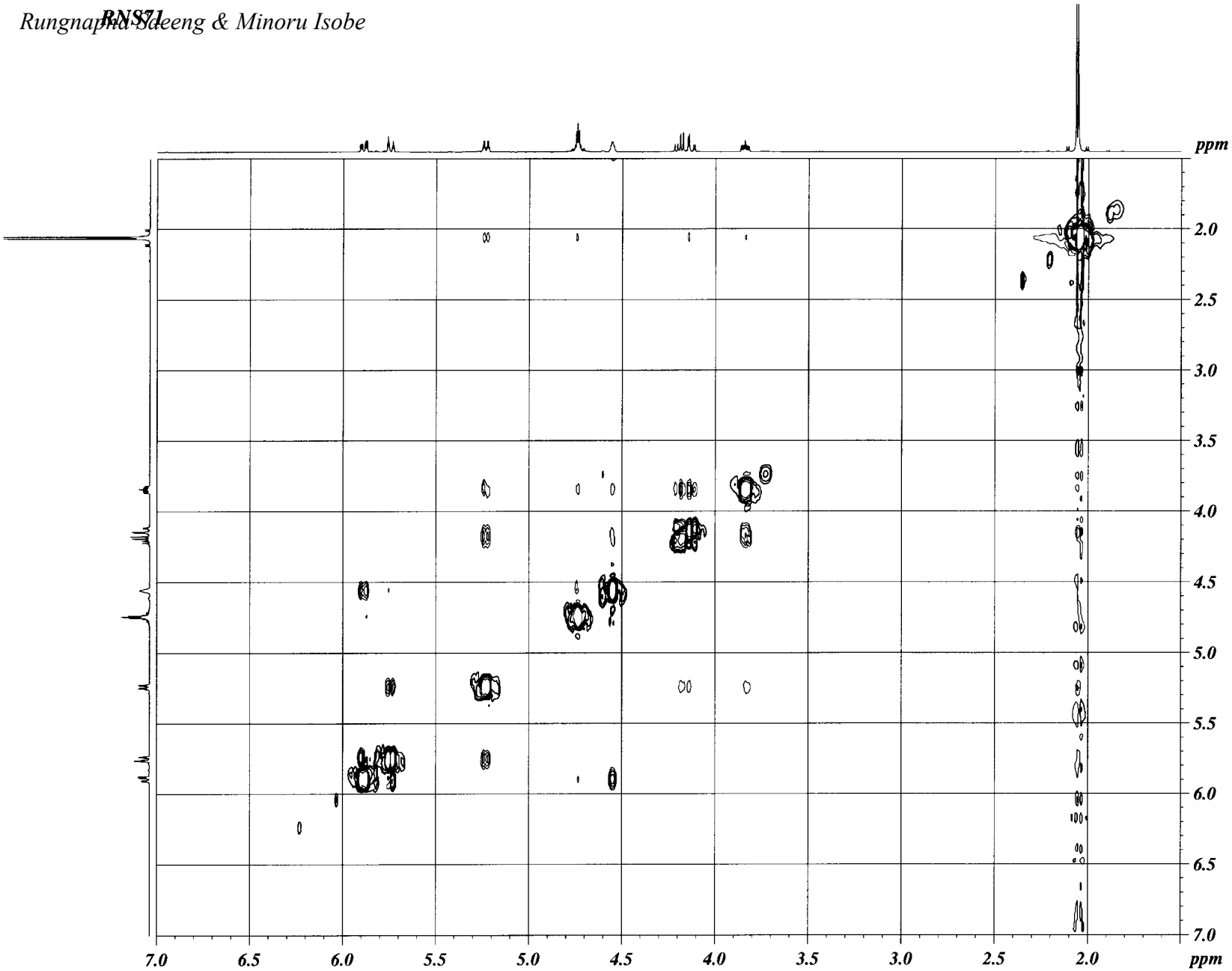
F2 – Acquisition Parameters
Date_ 20040421
Time 11.16
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

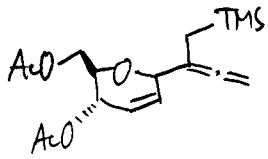
======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

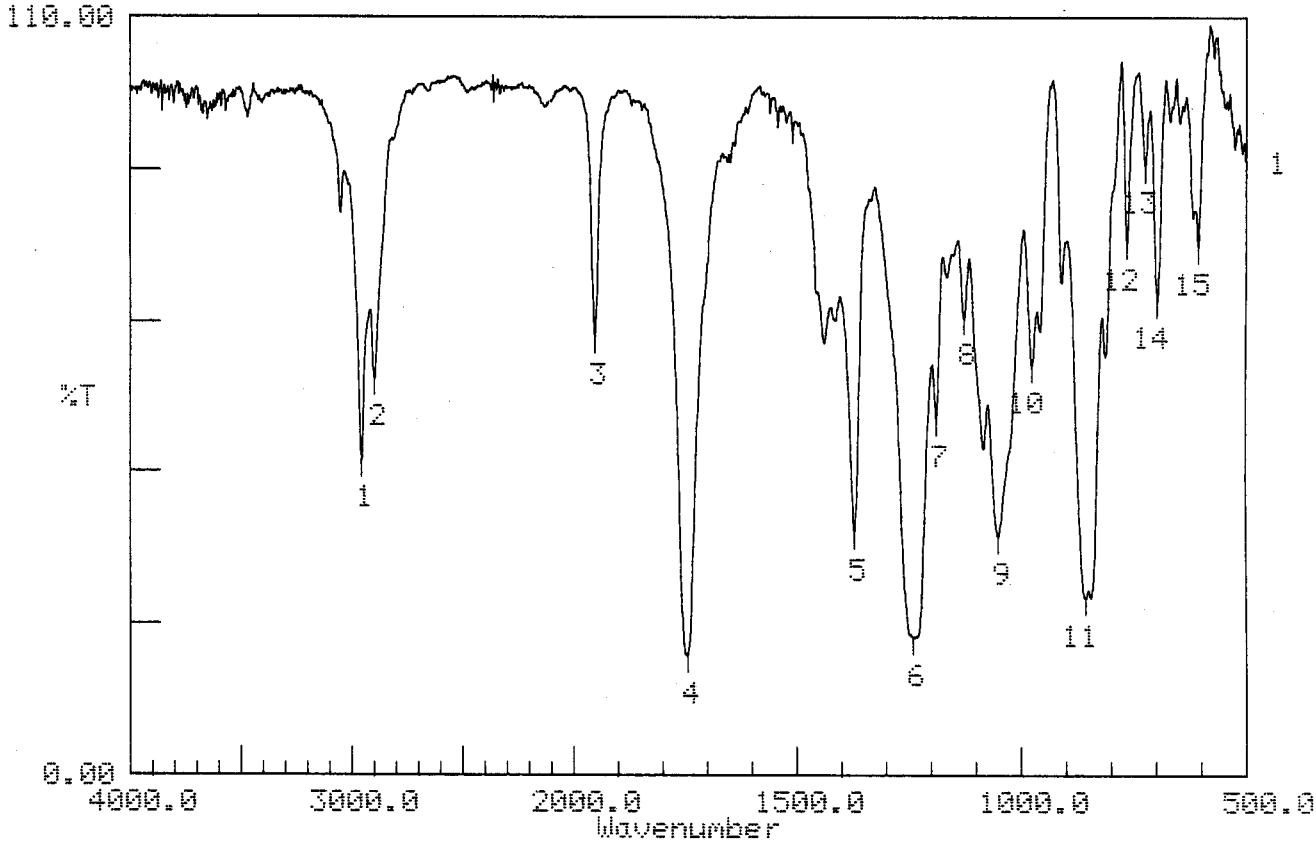
No parameters

NOESY





JASCO FT/IR-8300
Date : 99/04/01 22:18
File Name :
Sample Name: RNS-87-1
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
upper 110.00 lower 0.00 depth 7.00

Peak table

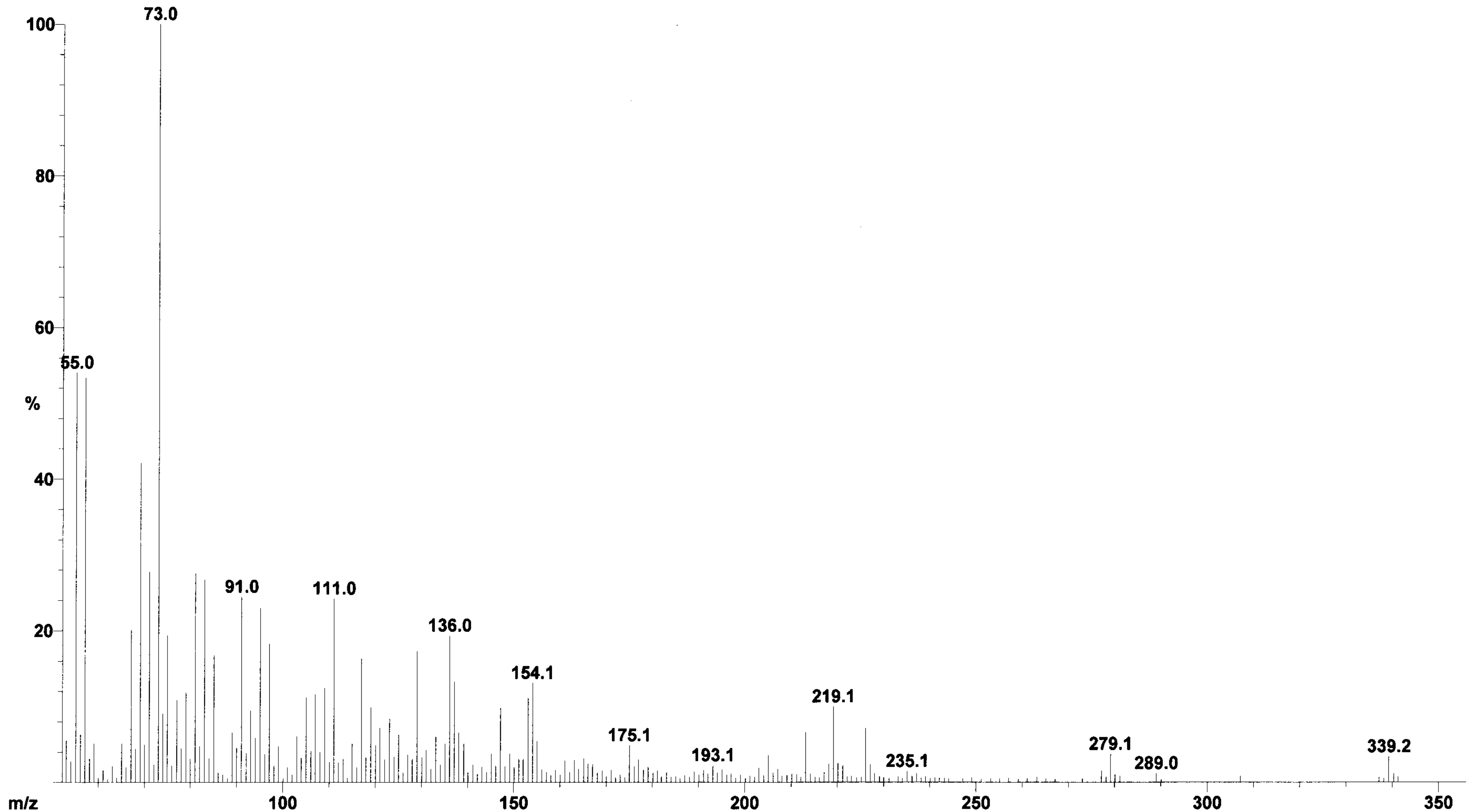
1:	2954.34(45.7)	2:	2896.47(57.7)	3:	1951.23(63.9)
4:	1743.86(17.5)	5:	1370.59(35.2)	6:	1239.41(20.1)
7:	1188.30(51.8)	8:	1125.60(66.8)	9:	1050.37(34.7)
10:	974.17(59.7)	11:	854.57(25.8)	12:	763.90(77.8)
13:	722.43(88.9)	14:	696.39(69.2)	15:	604.76(77.1)

Rungnapha Saeeng & Minoru Isobe

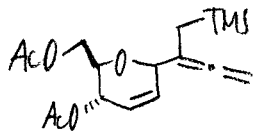
File: RNS-87-2 **Date Run: 2004-06-07 (Time Run: 12:36:22)**
Sample: Description
Instrument: JEOL LCmate

Ionization mode: FAB+

Scan: 9 **R.T.: 1.48**
Base: m/z 73; 13.4%FS TIC: 1516496 **#Ions: 221**



Elemental Analysis Report



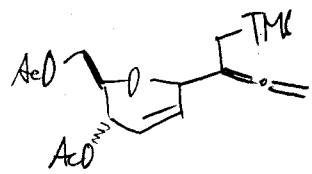
$C_{17}H_{26}O_5Si$

Refference No. : 365
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-87-1
Sample Weight : 2988 micrograms

	N	C	H
Calculated weight %	0.00	60.32	7.74
Found weight %	0.10	60.31	7.69
Element ratio(1)	0	2	3
Element ratio(2)	0	23	35
Element ratio(3)	0	25	38
Element ratio(4)	0	27	41
Element ratio(5)	0	29	44
Element ratio(6)	0	48	73
Element ratio(7)	0	52	79

Remarks :

Date : 2004/06/09 15:54:40 Operator : 繁.Kitamura(789-4169)



DATE 04.06.05
LAMP Na589
TEMP
SAMPLE
CONC. 1.345%
CELL 100mm
INTEG.TIME 20sec
****

SAMPLE NO.	deg
012	
1 α	-2.100
[α]	-156.15
2 α	-2.099
[α]	-156.11
3 α	-2.100
[α]	-156.15
4 α	-2.099
[α]	-156.11
5 α	-2.100
[α]	-156.20
6 α	-2.099
[α]	-156.10
7 α	-2.099
[α]	-156.07
8 α	-2.099
[α]	-156.12
9 α	-2.097
[α]	-155.97
10 α	-2.098
[α]	-156.00

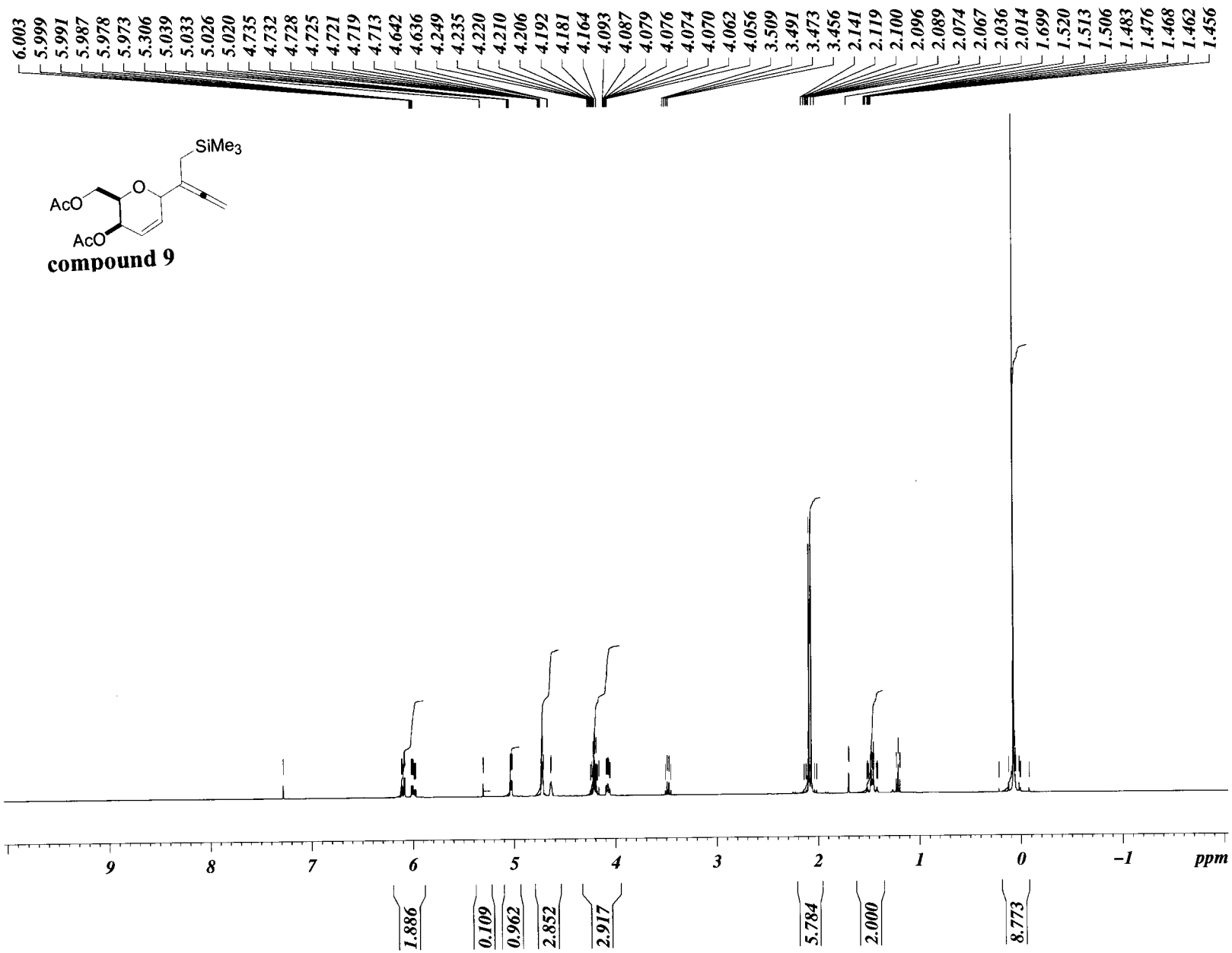
MEAN VALUE N=10

$\bar{\alpha}$	-2.099 deg
σn-1	0.001 deg
RSD	0.047 %
[α]	-156.10 deg
σn-1	0.0702 deg

TEMP 27

Rungnapha Saeeng & Minoru Isobe

RNS85



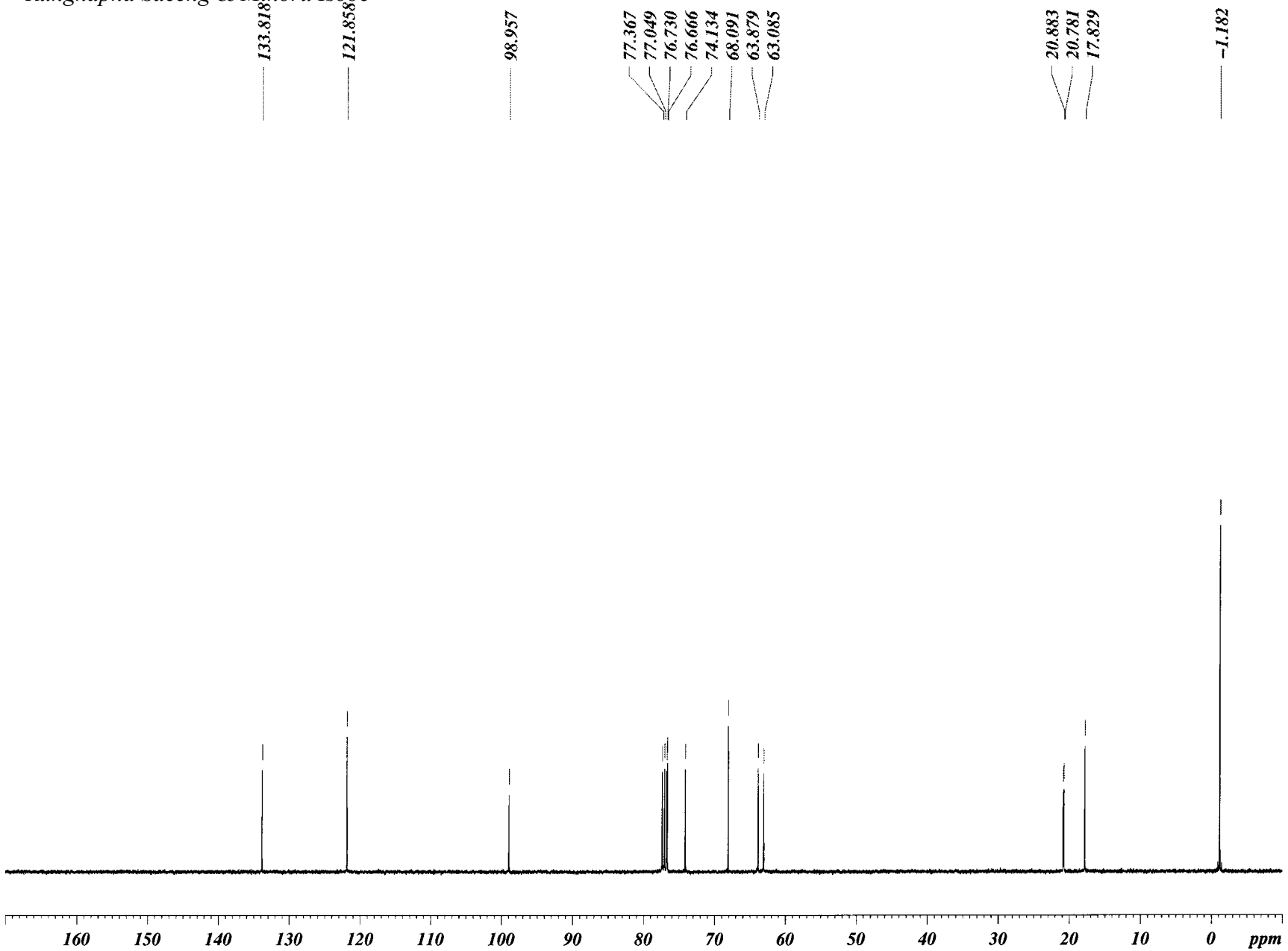
compound 9

Current Data Parameters
NAME Rungnaphaav400
EXPNO 67
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040512
Time 21.13
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

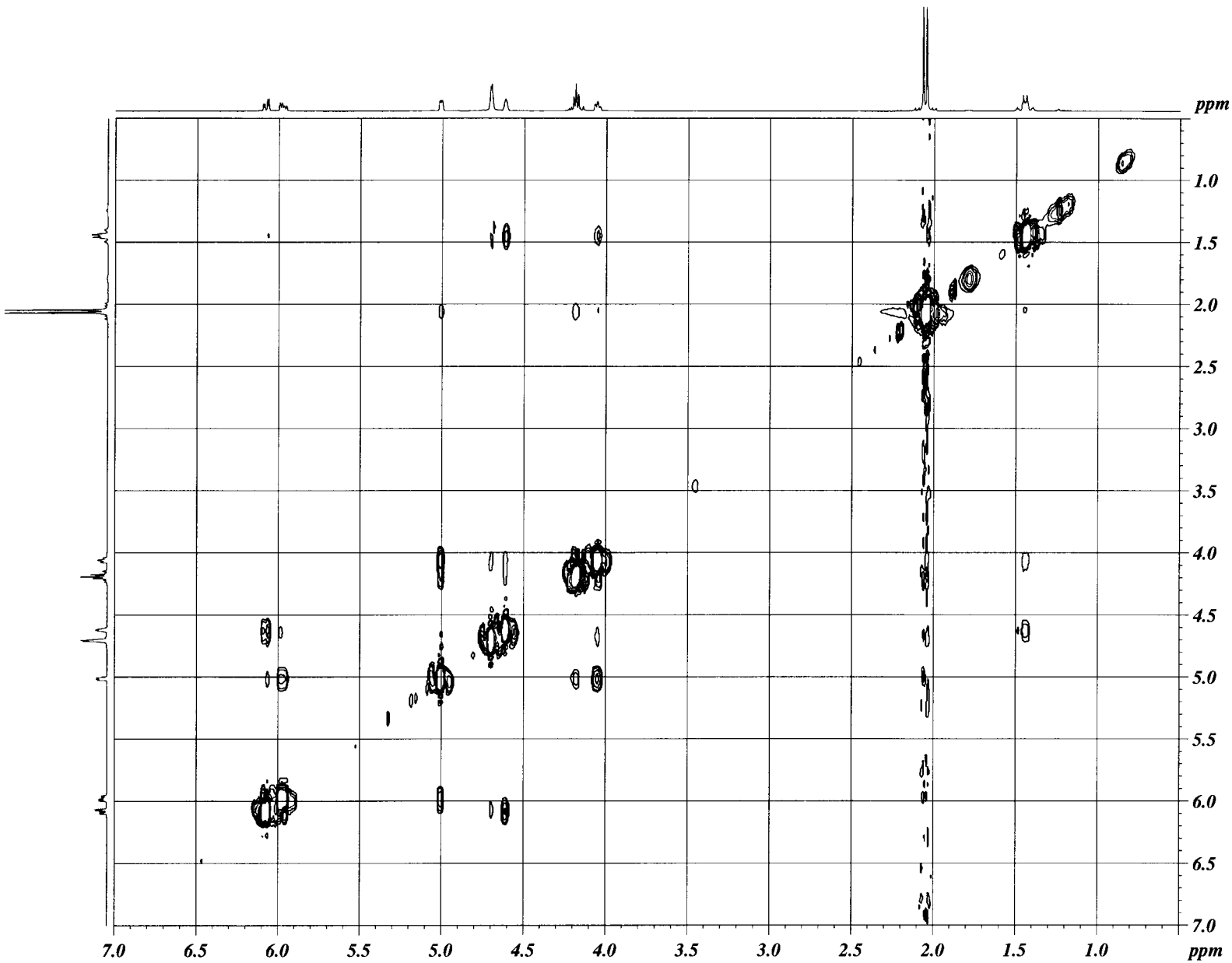
F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

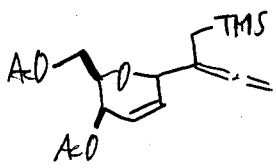

133.818
121.858
98.957
77.367
77.049
76.730
76.666
74.134
68.091
63.879
63.085
20.883
20.781
17.829
-1.182
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

RNS85

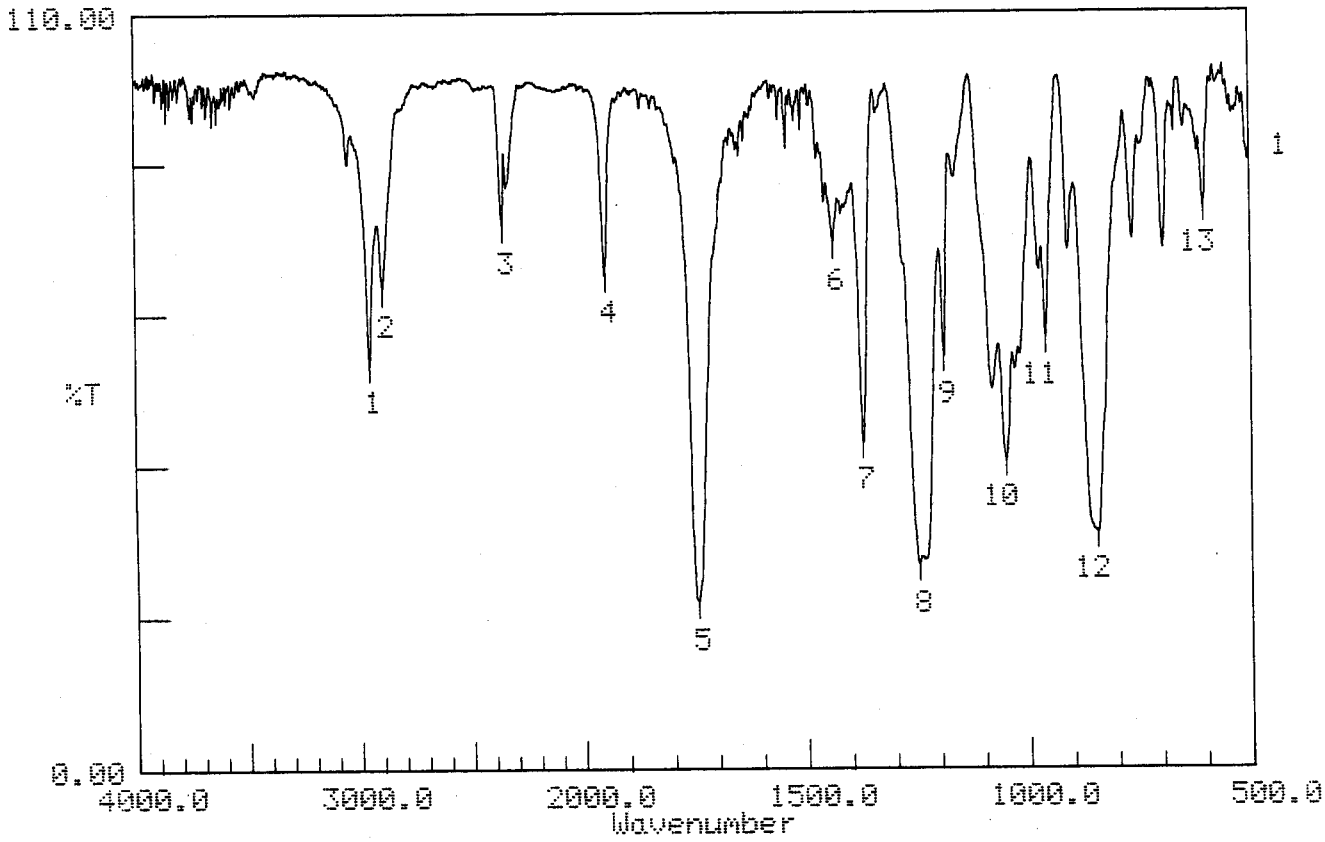
No parameters

NOESY





JASCO FT/IR-8300
Date : 99/04/01 22:46
File Name :
Sample Name: RNS-90-1
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
upper 110.00 lower 0.00 depth 7.00

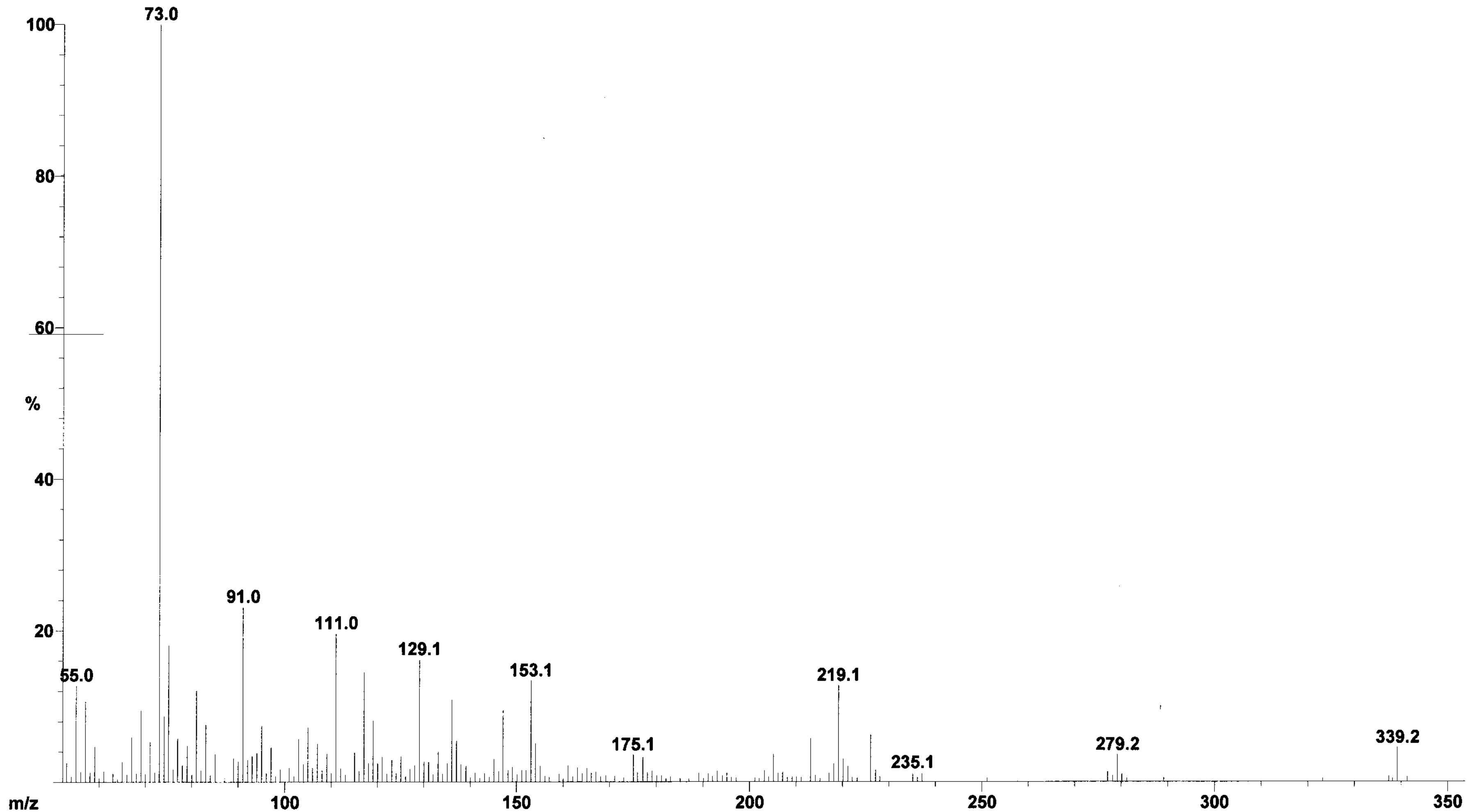
Peak table

1:	2955.31(59.0)	2:	2896.47(70.0)	3:	2359.23(79.0)
4:	1950.27(71.8)	5:	1744.83(24.3)	6:	1437.14(76.4)
7:	1371.55(47.4)	8:	1247.13(29.6)	9:	1189.26(59.8)
10:	1050.37(44.9)	11:	959.70(62.5)	12:	844.92(34.2)
13:	603.79(81.7)				

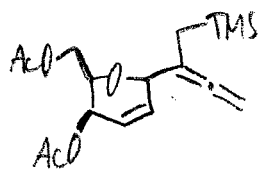
File: RNS-90-1 **Date Run: 2004-06-07 (Time Run: 12:44:09)**
Sample Description
Instrument: JEOL LCmate

Ionization mode: FAB+

Scan: 16 **R.T.: 2.77**
Base: m/z 73; 13.5%FS TIC: 849728 **#Ions: 176**



Elemental Analysis Report



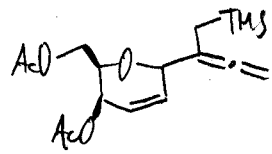
$C_{17}H_{26}O_5Si$

Refference No. : 366
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-90-1
Sample Weight : 2254 micrograms

	N	C	H
Calculated weight %	0.00	60.32	7.74
Found weight %	0.15	60.44	7.77
Element ratio(1)	0	2	3
Element ratio(2)	0	13	20
Element ratio(3)	0	15	23
Element ratio(4)	0	17	26
Element ratio(5)	0	28	43
Element ratio(6)	0	32	49

Remarks :

Date : 2004/06/09 15:54:44 Operator : 繁.Kitamura(789-4169)



DATE 04.06.05
LAMP Na589
TEMP
SAMPLE RNS-90-1
CONC. 0.82%
CELL 100mm
INTEG.TIME 20sec
****

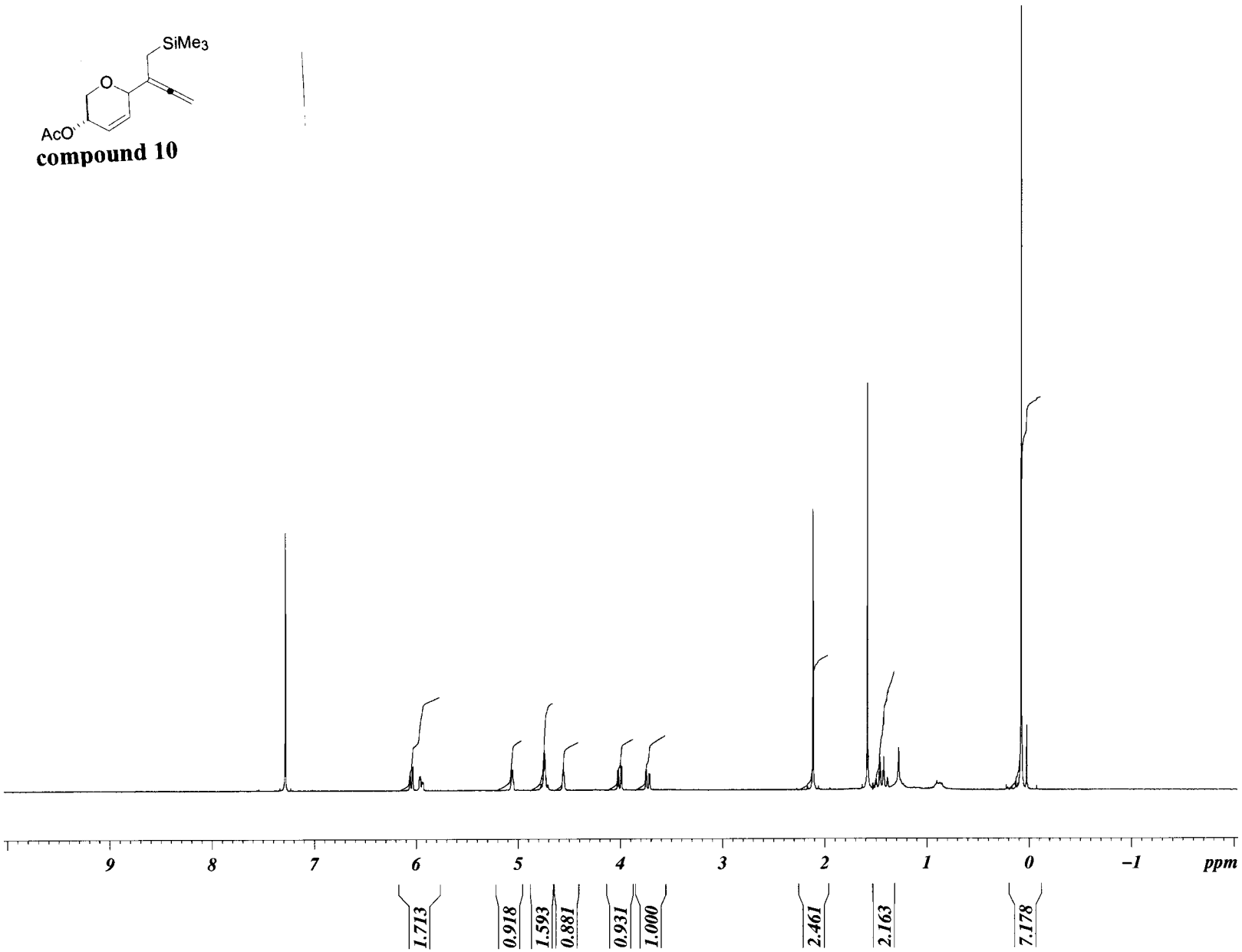
SAMPLE NO.	deg
014	
1 α	-3.308
[α]	-403.42
2 α	-3.307
[α]	-403.37
3 α	-3.311
[α]	-403.79
4 α	-3.309
[α]	-403.63
5 α	-3.310
[α]	-403.71
6 α	-3.308
[α]	-403.49
7 α	-3.307
[α]	-403.33
8 α	-3.310
[α]	-403.74
9 α	-3.306
[α]	-403.26
10 α	-3.309
[α]	-403.62

MEAN VALUE N=10

$\bar{\alpha}$	-3.308 deg
σn-1	0.001 deg
RSD	0.030 %
[α]	-403.54 deg
σn-1	0.1856 deg
TEMP	28

RNS89

compound 10

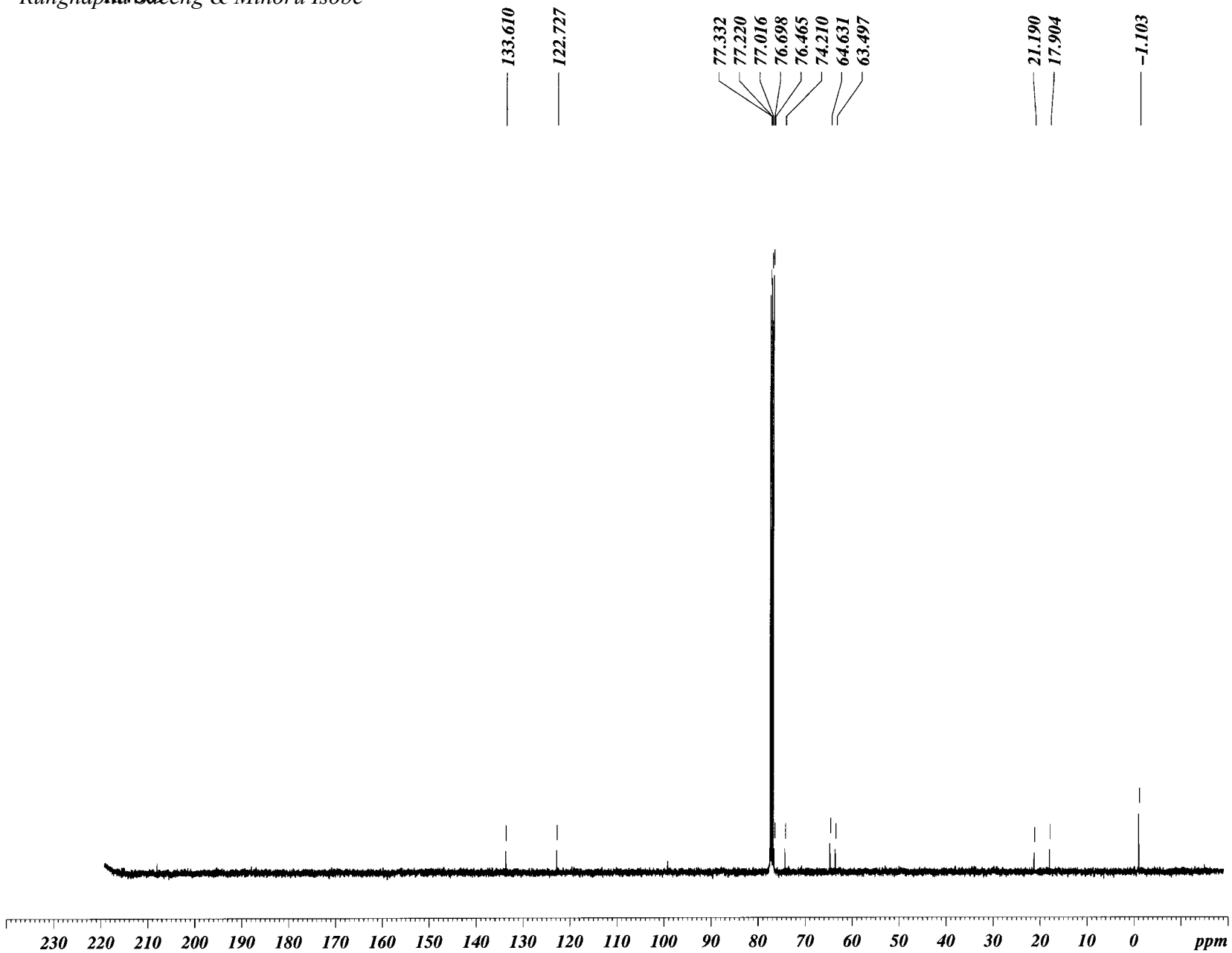



```
Current Data Parameters
NAME      Rungnaphaav400
EXPNO          115
PROCNO         1

F2 - Acquisition Parameters
Date_      20040602
Time       22.40
INSTRUM    av400
PROBHD  5 mm QNP 1H/13
PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         574.7
DW         60.400 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec

======== CHANNEL f1 ===
NUC1       1H
P1         8.60 usec
PL1        -4.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI         32768
SF         400.1300000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```



Current Data Parameters
NAME Rungnaphaav400
EXPNO 116
PROCNO 1

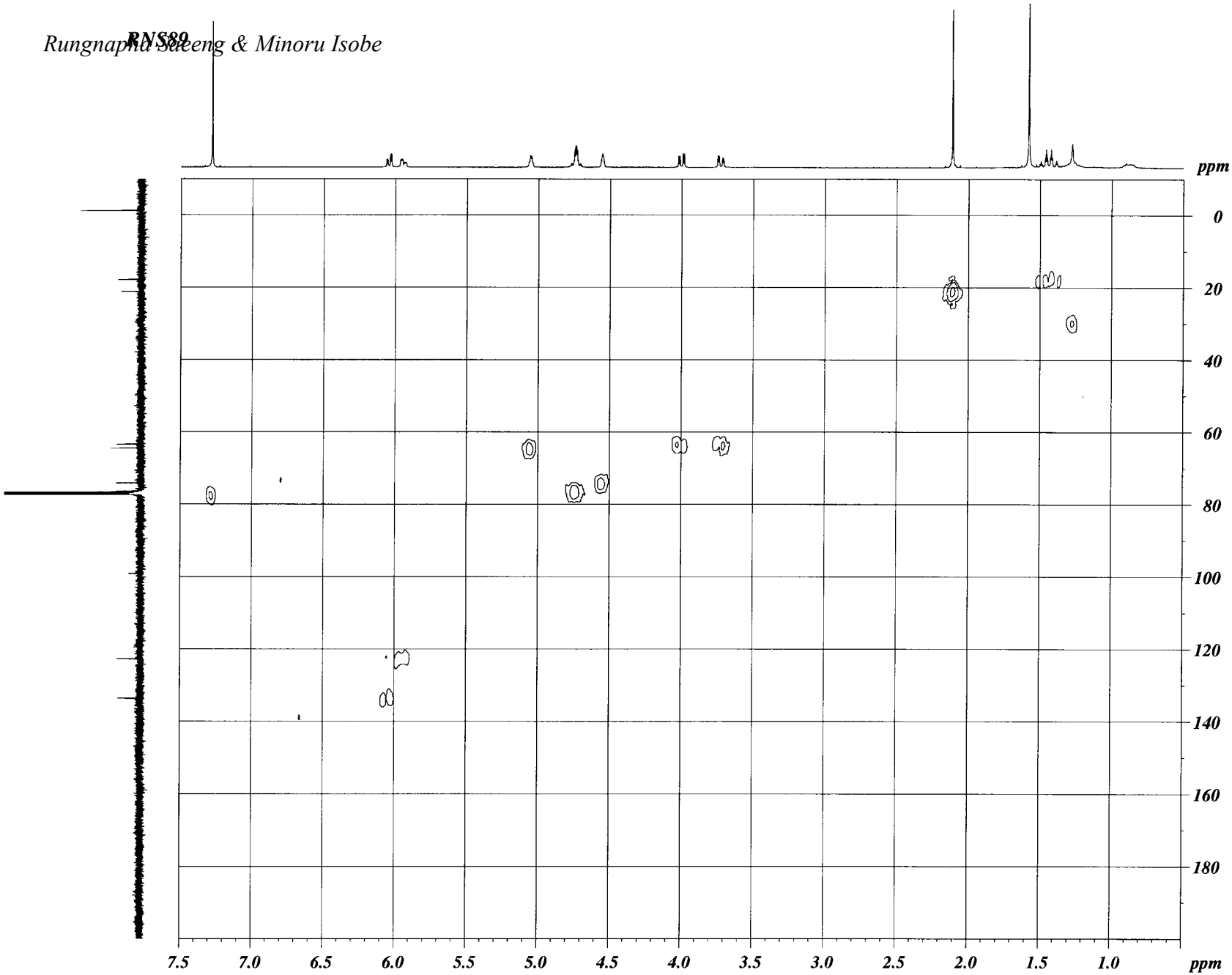
F2 – Acquisition Parameters
Date_ 20040602
Time 23.45
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

RNS89



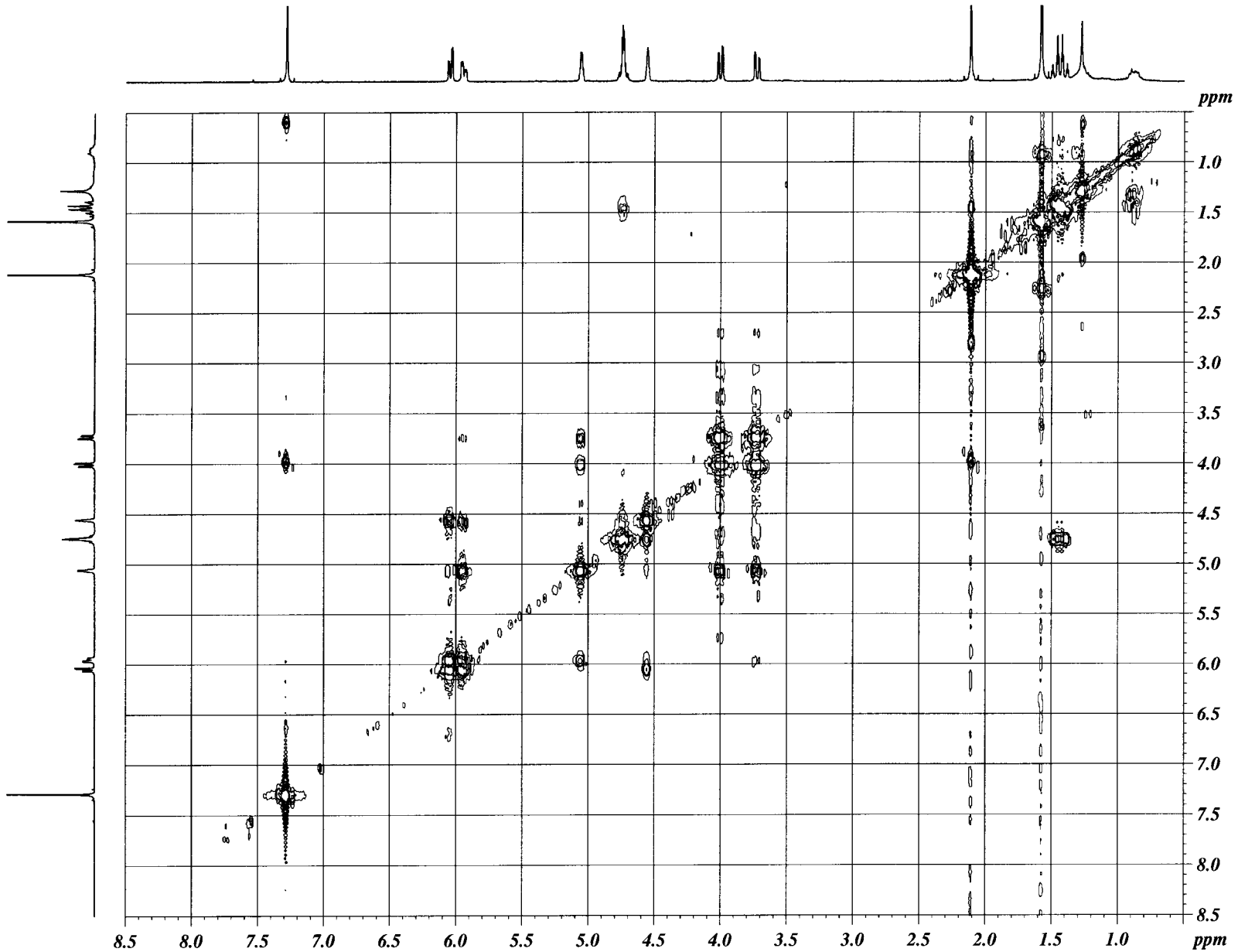
Current Data Parameters
NAME Rungnaphaav400
EXPNO 115
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040602
Time 22.40
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

COSY



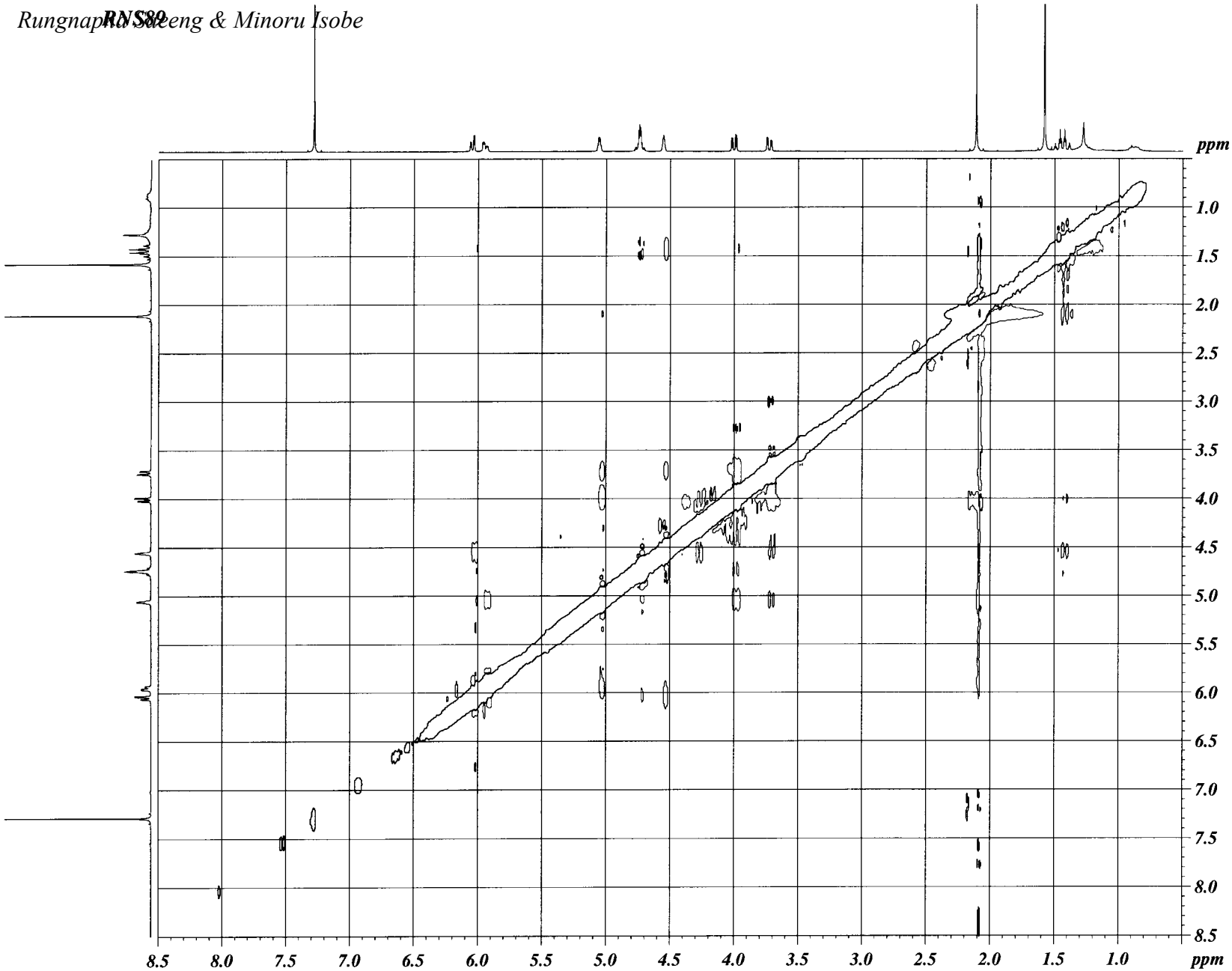
Current Data Parameters
NAME Rungnaphaav400
EXPNO 115
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040602
Time 22.40
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NOESY

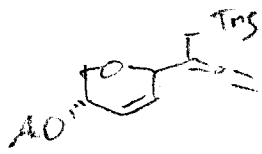


Current Data Parameters
NAME Rungnaphaav400
EXPNO 115
PROCNO 1

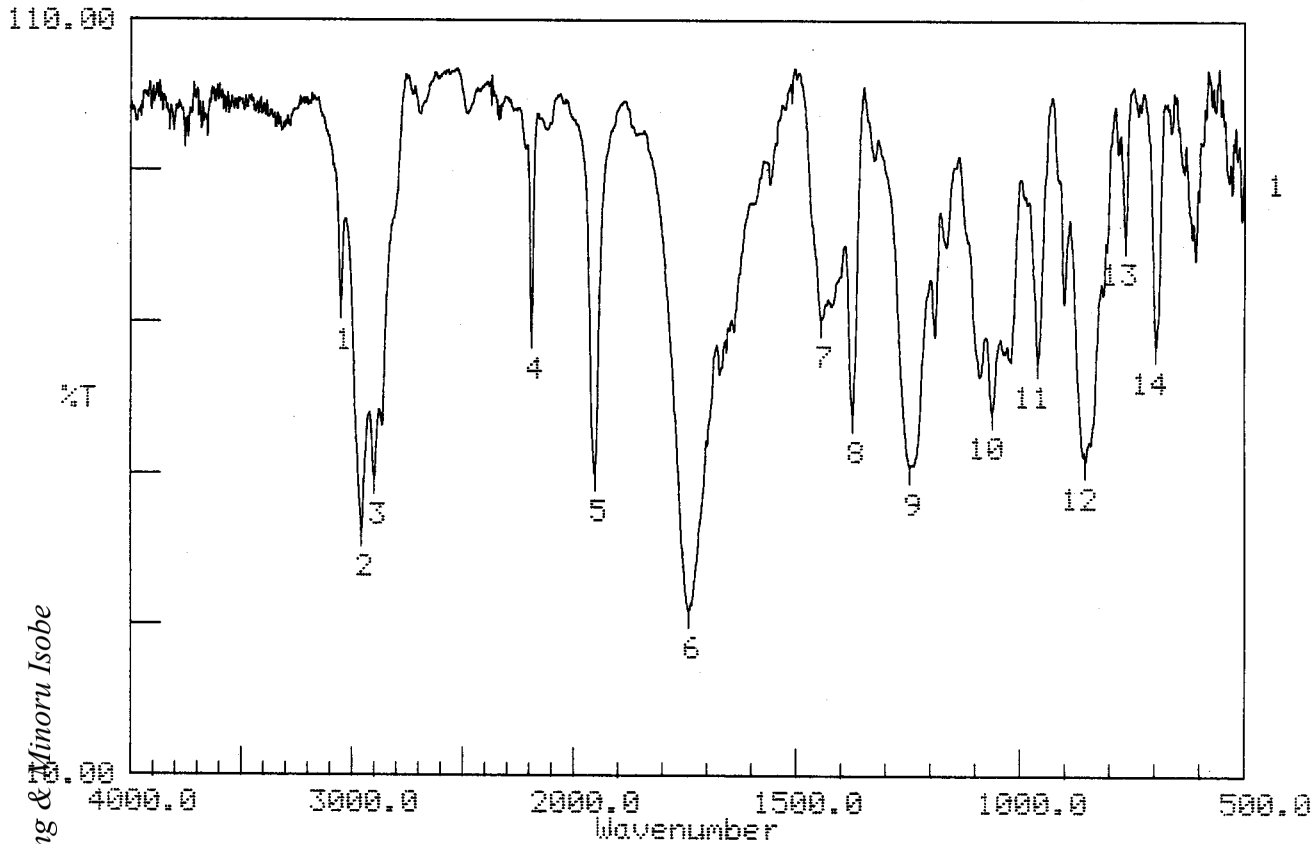
F2 – Acquisition Parameters
Date_ 20040602
Time 22.40
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JASCO FT/IR-8300
Date : 99/04/01 23:24
File Name :
Sample Name: RNS-106
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
Upper 110.00 lower 10.00 depth 7.00

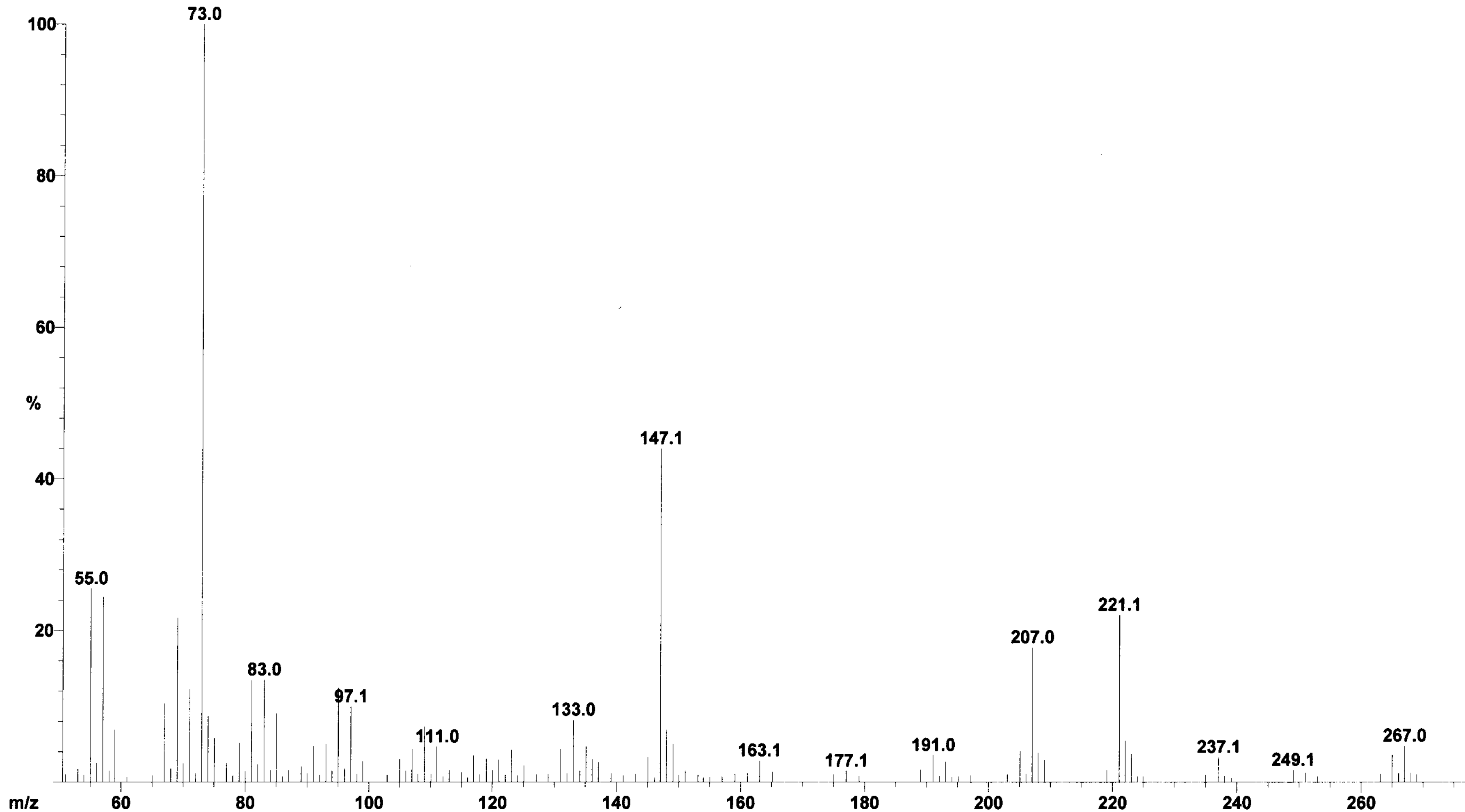
Peak table

1:	3047.90(72.7)	2:	2954.34(42.7)	3:	2897.43(49.6)
4:	2187.54(69.0)	5:	1950.27(50.1)	6:	1740.00(32.0)
7:	1442.93(70.6)	8:	1371.55(58.0)	9:	1244.24(51.0)
10:	1060.98(58.5)	11:	958.74(65.3)	12:	852.64(51.8)
13:	763.90(81.6)	14:	696.39(67.3)		

File: RNS-89-1 **Date Run: 2004-06-07 (Time Run: 12:52:46)**
Sample: Description
Instrument: JEOL LCmate

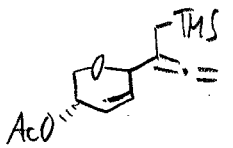
Ionization mode: FAB+

Scan: 1 **R.T.: .02**
Base: m/z 73; 9.2%FS TIC: 634912 **#Ions: 161**



Elemental Analysis Report

Refference No. : 367
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-106
Sample Weight : 1602 micrograms



$C_{14}H_{22}O_3Si$

	N	C	H
Calculated weight %	0.00	63.12	8.32
Found weight %	0.08	63.12	8.32
Element ratio(1)	0	7	11
Element ratio(2)	0	65	102

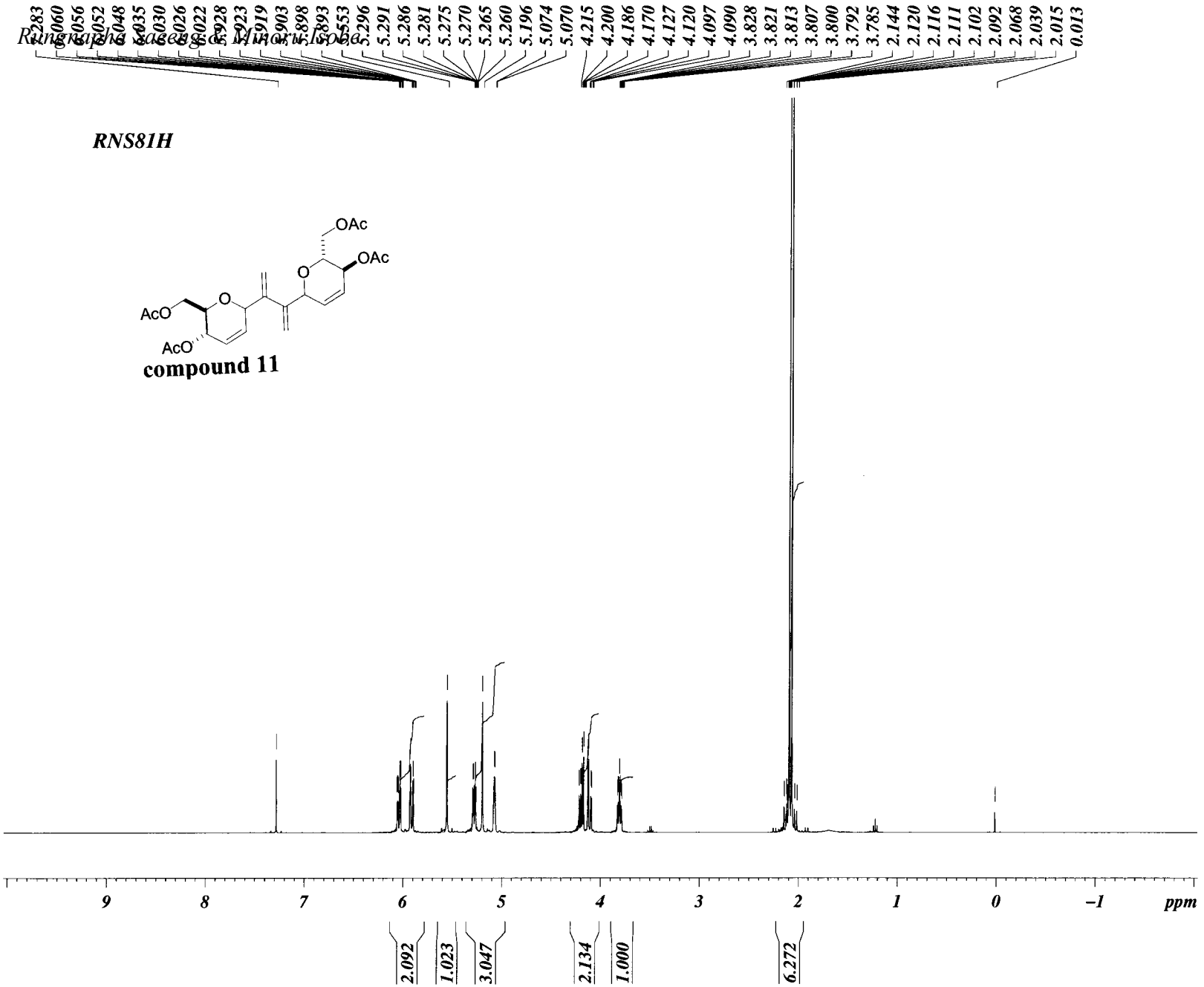
Remarks :

Date : 2004/06/09 15:54:46 Operator : 繁.Kitamura(789-4169)

DATE 04.06.07
LAMP Na589
TEMP
SAMPLE RNS-106
CONC. 1.345%
CELL 100mm
INTEG.TIME 20sec
****
SAMPLE NO. deg
005
1 α 1.921
[α] 142.83
2 α 1.921
[α] 142.85
3 α 1.921
[α] 142.83
4 α 1.922
[α] 142.89
5 α 1.922
[α] 142.95
6 α 1.922
[α] 142.97
7 α 1.923
[α] 143.01
8 α 1.919
[α] 142.74
9 α 1.922
[α] 142.92
10 α 1.924
[α] 143.08

MEAN VALUE N=10
$\bar{\alpha}$ 1.922 deg
σn-1 0.001 deg
RSD 0.052 %

[α] 142.91 deg
σn-1 0.0990 deg
TEMP 26



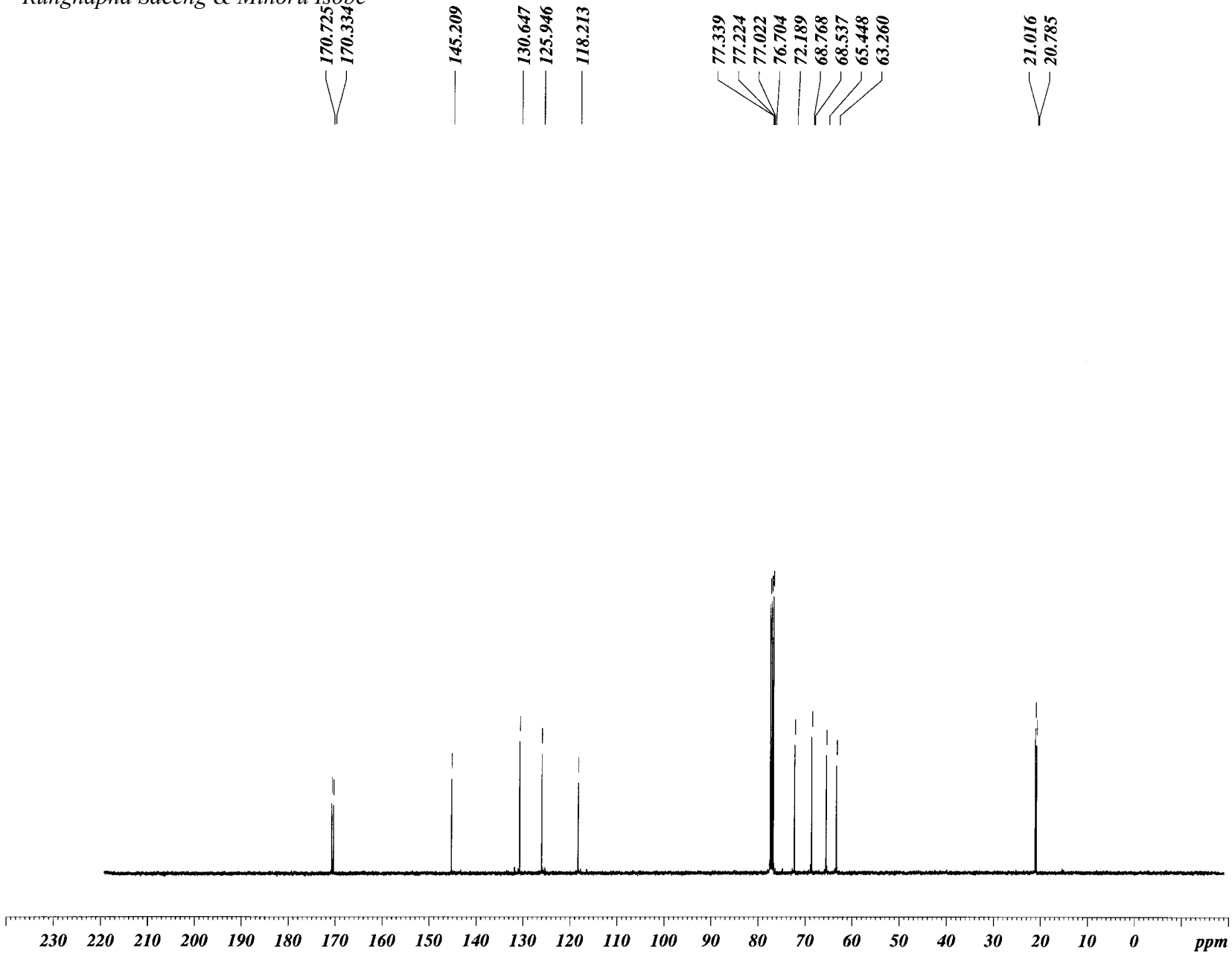
Current Data Parameters
NAME Rungnaphaav400
EXPNO 91
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040523
Time 21.06
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

RNS81



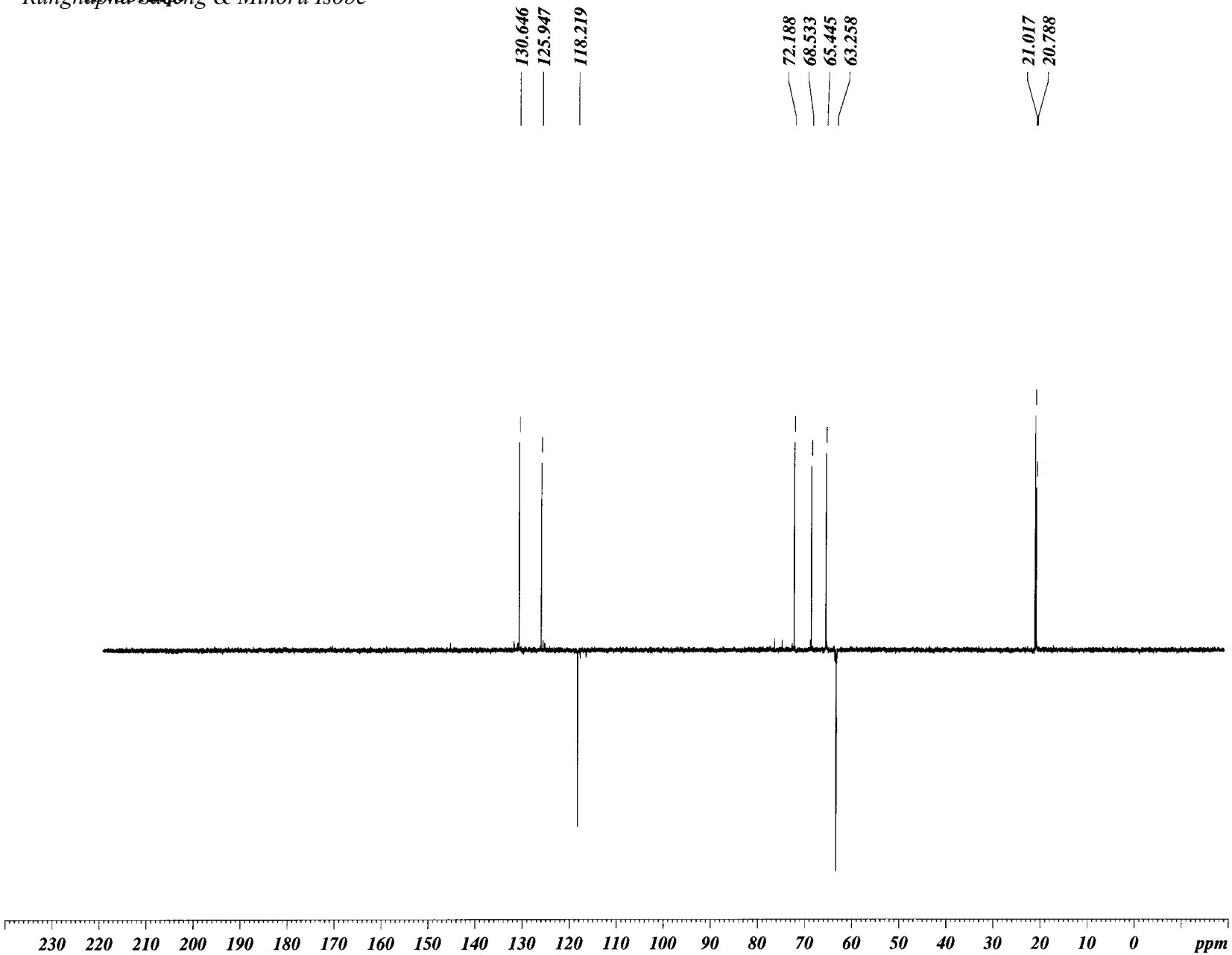
Current Data Parameters
NAME Rungnaphaav400
EXPNO 22
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040419
Time 18.23
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 9195.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



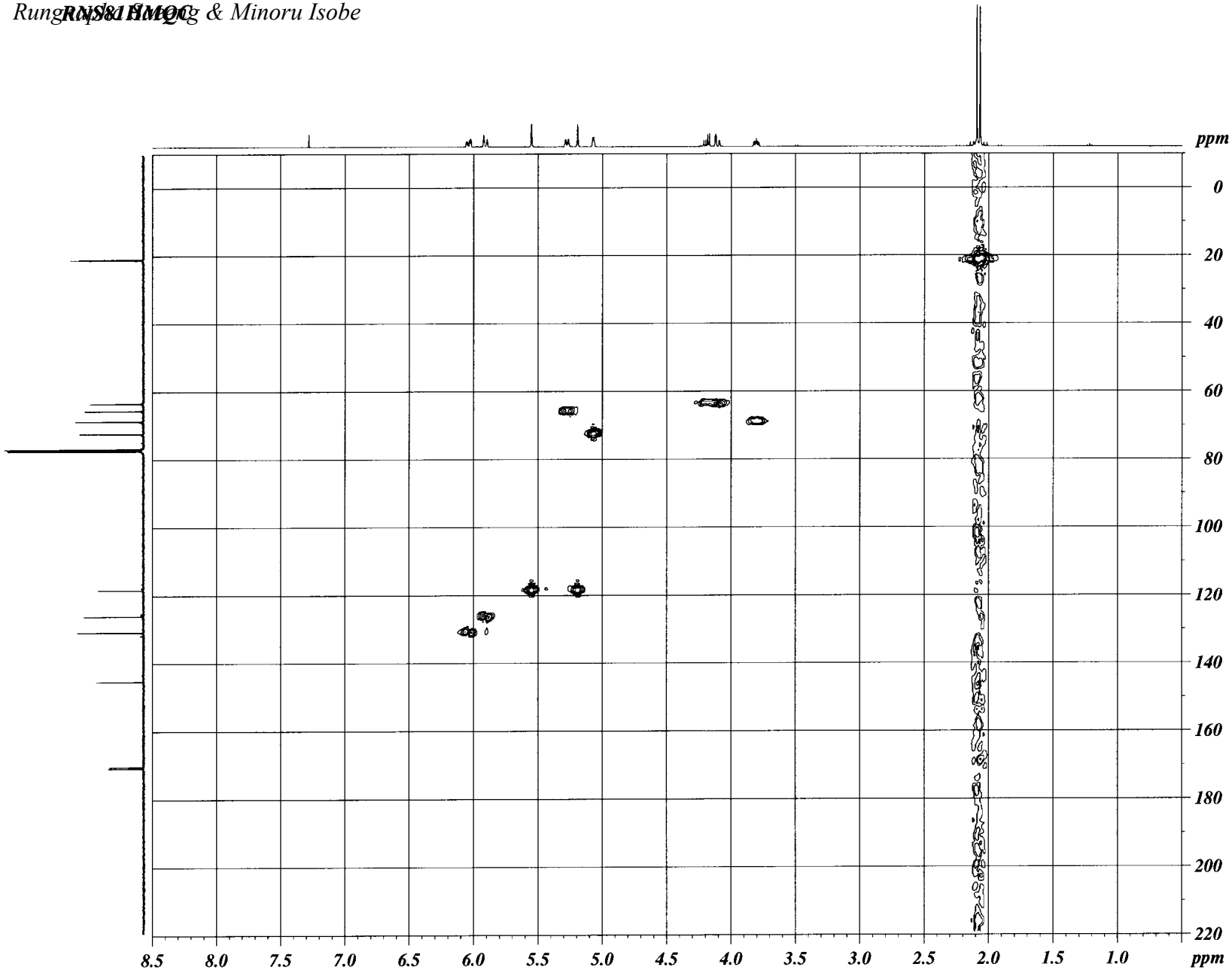

```
Current Data Parameters
NAME      Rungnaphaav400
EXPNO             25
PROCNO             1

F2 - Acquisition Parameters
Date_          20040419
Time             22.05
INSTRUM          av400
PROBHD   5 mm QNP 1H/13
PULPROG        dept135
TD               65536
SOLVENT          CDCl3
NS                 801
DS                   4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                16384
DW             20.850 usec
DE              6.00 usec
TE              300.0 K
CNST2         145.0000000
D1          2.00000000 sec
d2          0.00344828 sec
d12         0.00002000 sec
DELTA       0.00001146 sec

======== CHANNEL f1 ===
NUC1              13C
P1              9.00 usec
p2             18.00 usec
PL1             -3.00 dB
SFO1       100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2       waltz16
NUC2              1H
P3              8.60 usec
p4             17.20 usec
PCPD2          80.00 usec
PL2             -4.00 dB
PL12            14.80 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI             32768
SF         100.6127690 MHz
WDW               EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```

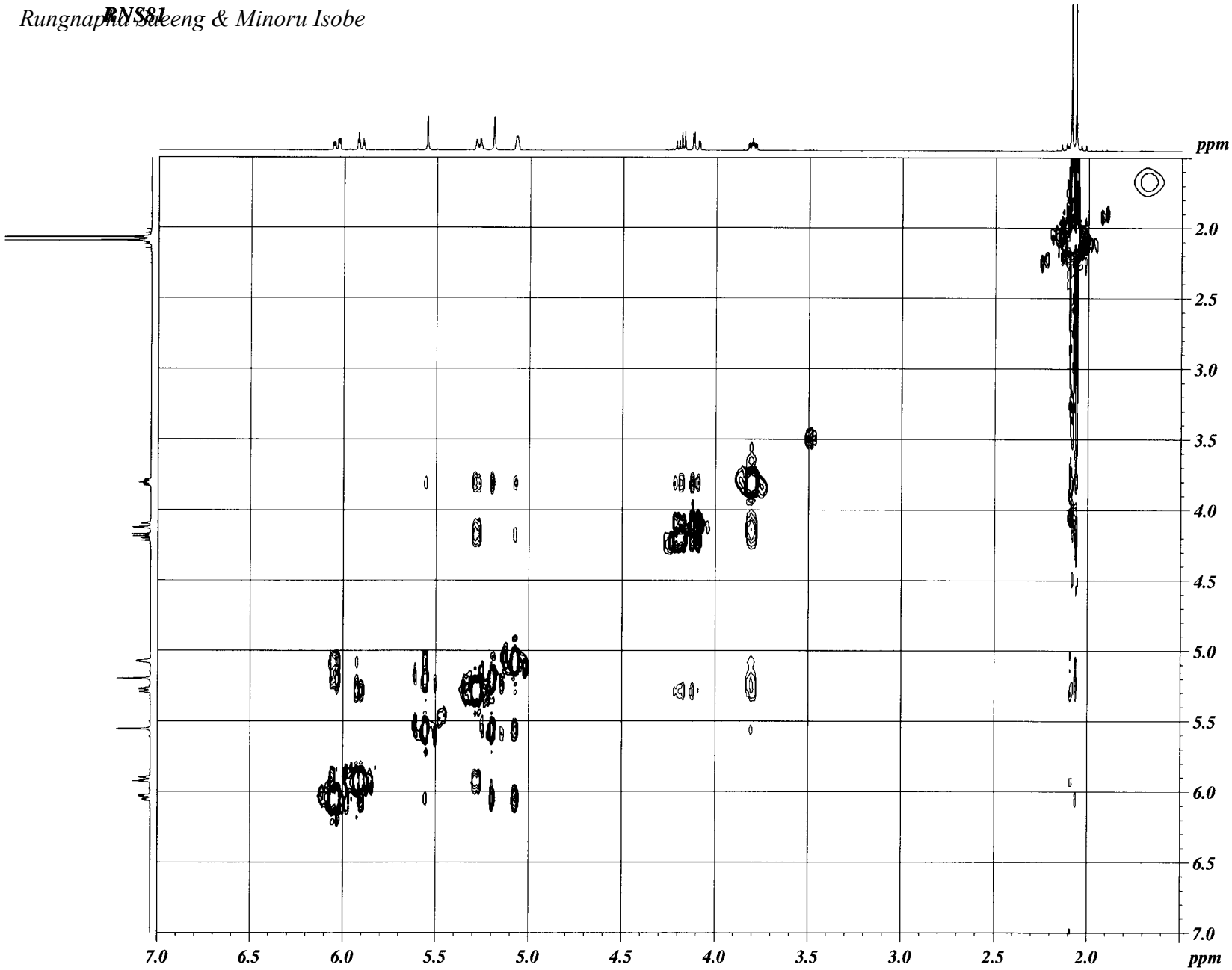


Current Data Parameters
NAME Rungnaphaav400
EXPNO 91
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040523
Time 21.06
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



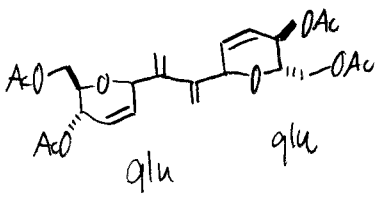
NOESY

Current Data Parameters
NAME Rungnaphaav400
EXPNO 91
PROCNO 1

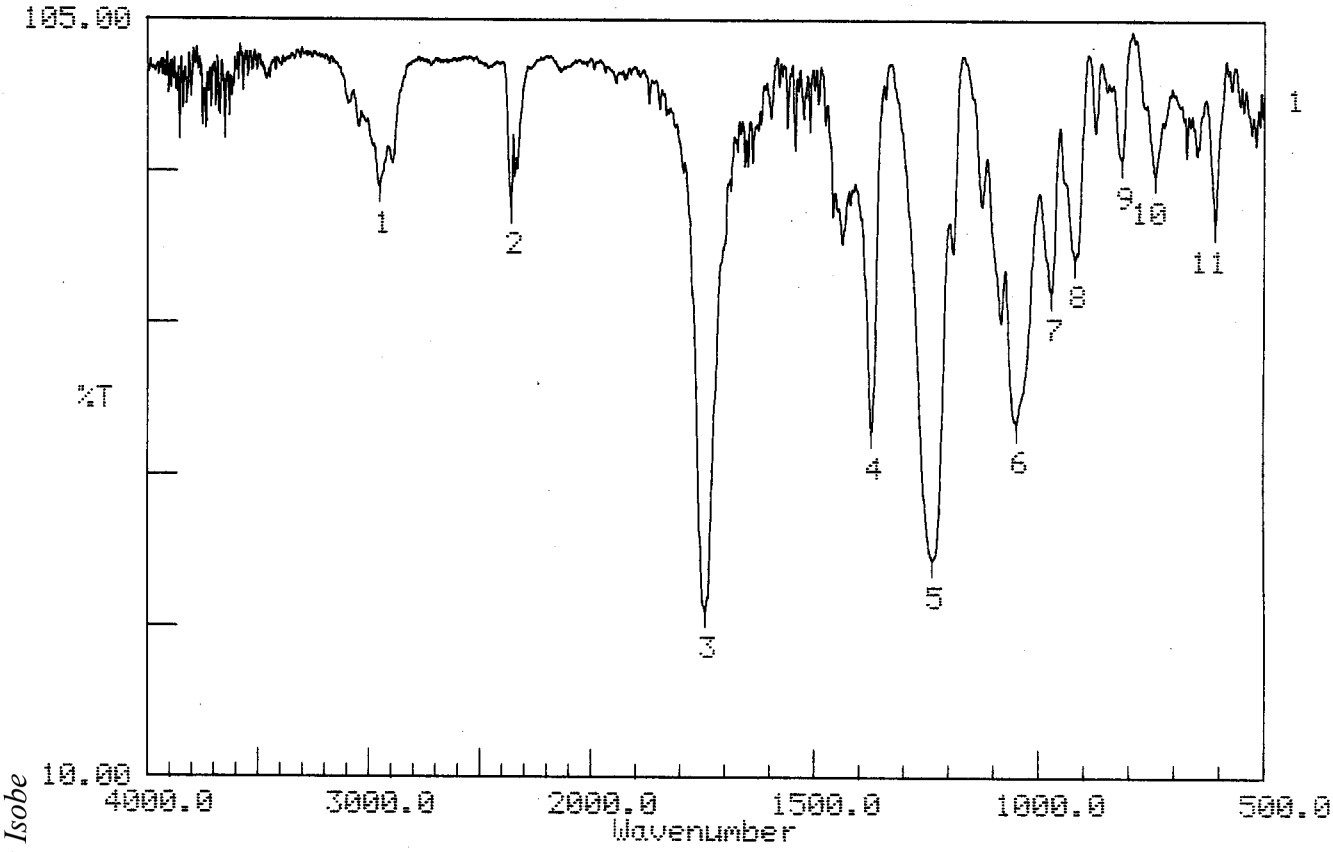
F2 – Acquisition Parameters
Date_ 20040523
Time 21.06
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JASCO FT/IR-8300
Date : 99/04/01 21:15
File Name :
Sample Name: RNS-81
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
upper 105.00 lower 10.00 depth 10.00

Peak table

1:	2952.41(84.2)	2:	2360.19(81.7)	3:	1743.86(31.2)
4:	1371.55(53.6)	5:	1235.56(37.5)	6:	1048.44(54.6)
7:	970.31(71.1)	8:	918.23(75.2)	9:	814.06(87.7)
10:	739.79(85.8)	11:	606.69(79.7)		

Rungnapha Saeeng & Minoru Isobe

File: RNS-81

Sample: Description

Instrument: JEOL LCmate

Date Run: 2004-06-01 (Time Run: 13:25:55)

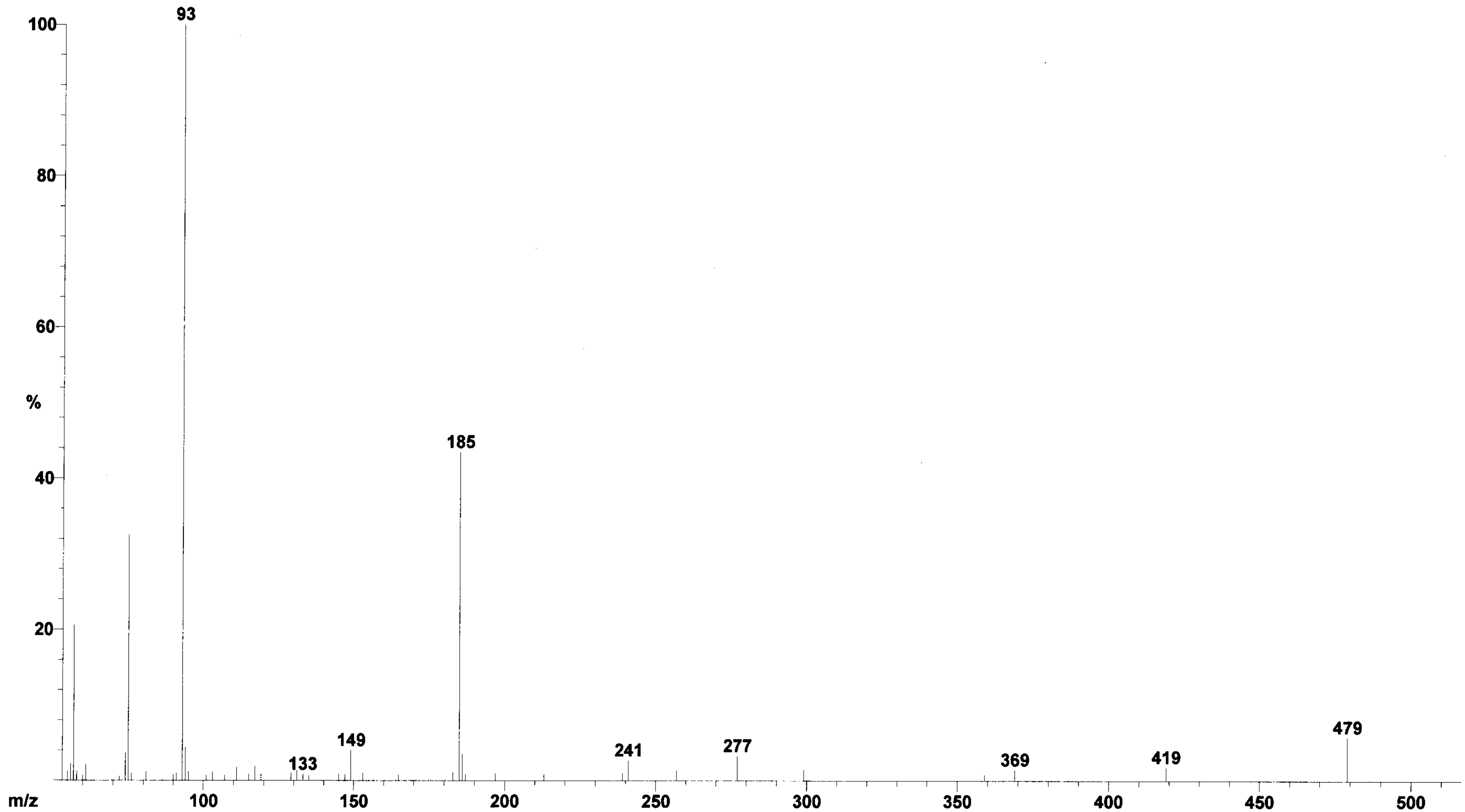
Ionization mode: FAB+

Scan: 1

Base: m/z 93; 9.4%FS TIC: 262688

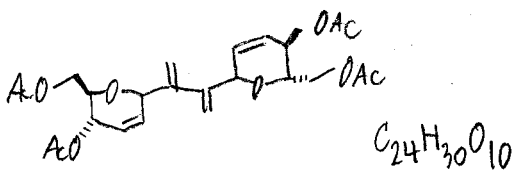
R.T.: .02

#Ions: 48



Elemental Analysis Report

Refference No. : 359
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-81
Sample Weight : 1660 micrograms



$C_{24}H_{30}O_{10}$

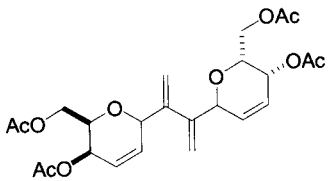
	N	C	H
Calculated weight %	0.00	60.24	6.32
Found weight %	0.16	60.25	6.27
Element ratio(1)	0	4	5
Element ratio(2)	0	17	21
Element ratio(3)	0	21	26
Element ratio(4)	0	25	31
Element ratio(5)	0	29	36

Remarks :

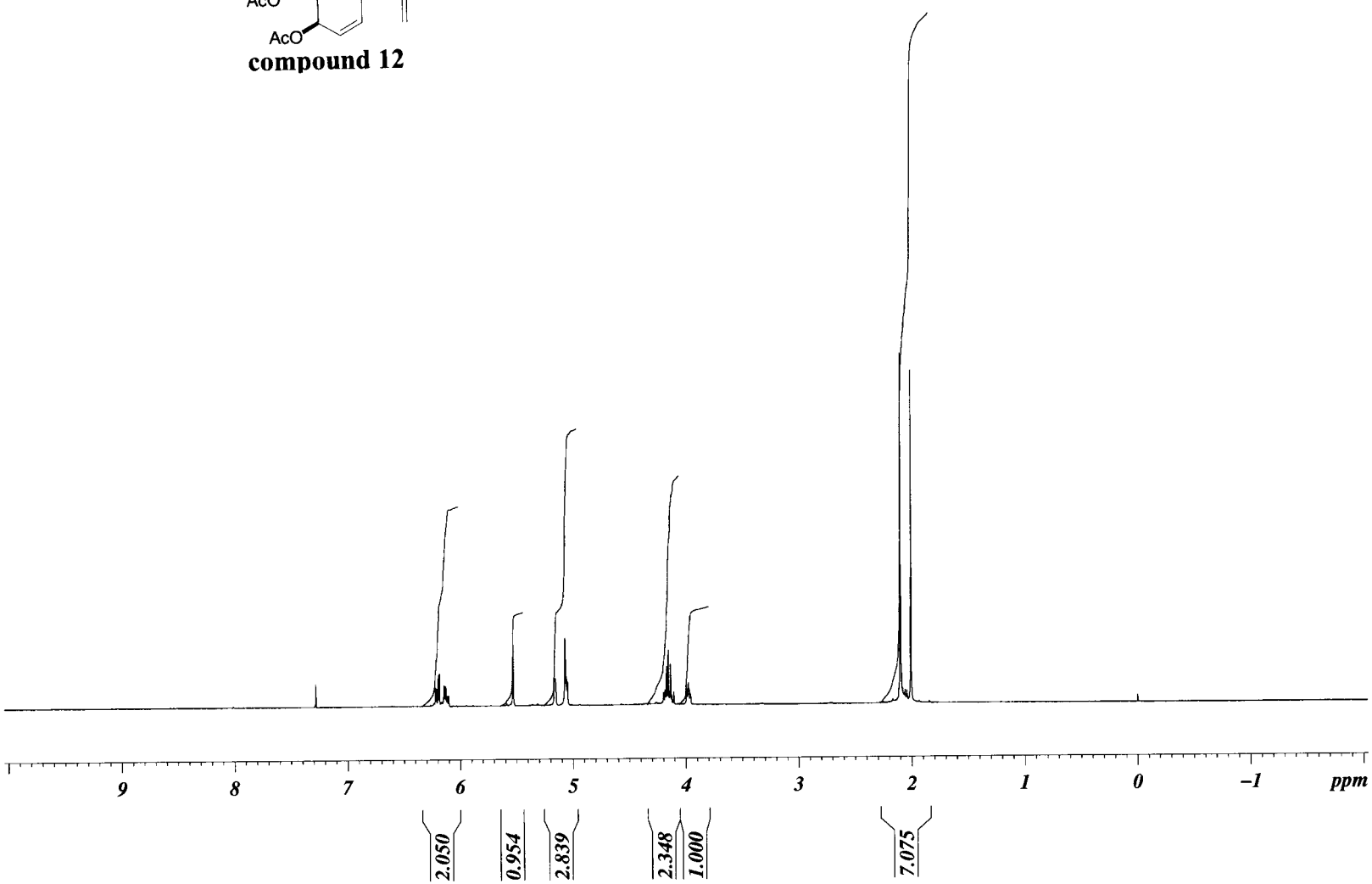
Date : 2004/06/09 15:54:34 Operator : 繁.Kitamura(789-4169)

```
DATE      04.06.06
LAMP      No589
TEMP
SAMPLE    RNS-81
CONC.     0.775%
CELL      100mm
INTEG.TIME 20sec
**********
SAMPLE NO.     deg
019
      1 α   0.180
      [α] 23.354
      2 α   0.183
      [α] 23.671
      3 α   0.182
      [α] 23.560
      4 α   0.181
      [α] 23.433
      5 α   0.180
      [α] 23.247
      6 α   0.182
      [α] 23.494
      7 α   0.178
      [α] 23.030
      8 α   0.180
      [α] 23.276
      9 α   0.180
      [α] 23.249
      10 α   0.181
      [α] 23.435
-------------------------
MEAN VALUE N=10
      ā   0.181 deg
σn-1      0.001 deg
RSD       0.552 %

      [α] 23.375 deg
σn-1  0.1840 deg
TEMP        27
```



compound 12

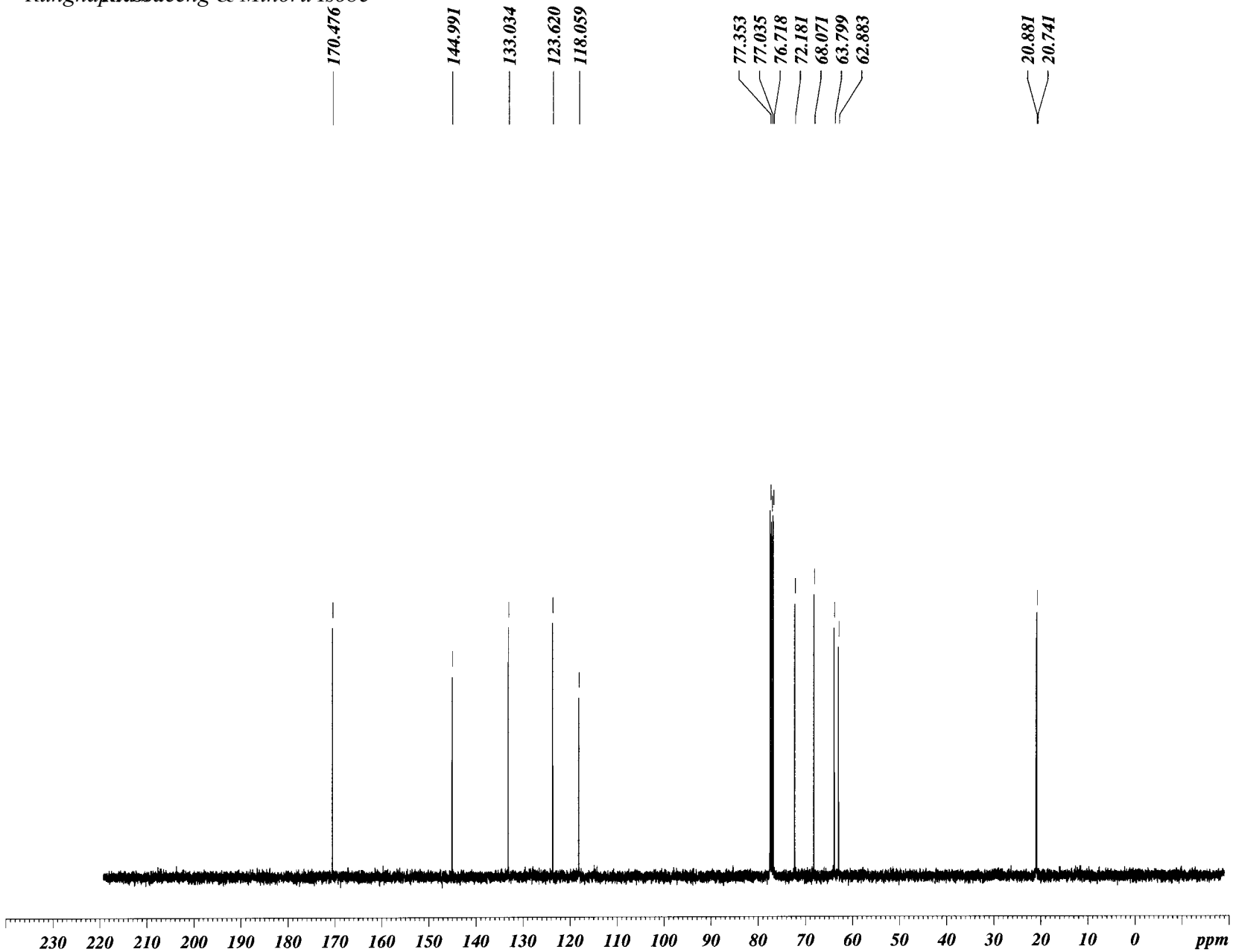


Current Data Parameters
NAME Rungnapha
EXPNO 130
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040611
Time 10.34
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 -4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Rungnapha
EXPNO 86
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040519
Time 15.00
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 124
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

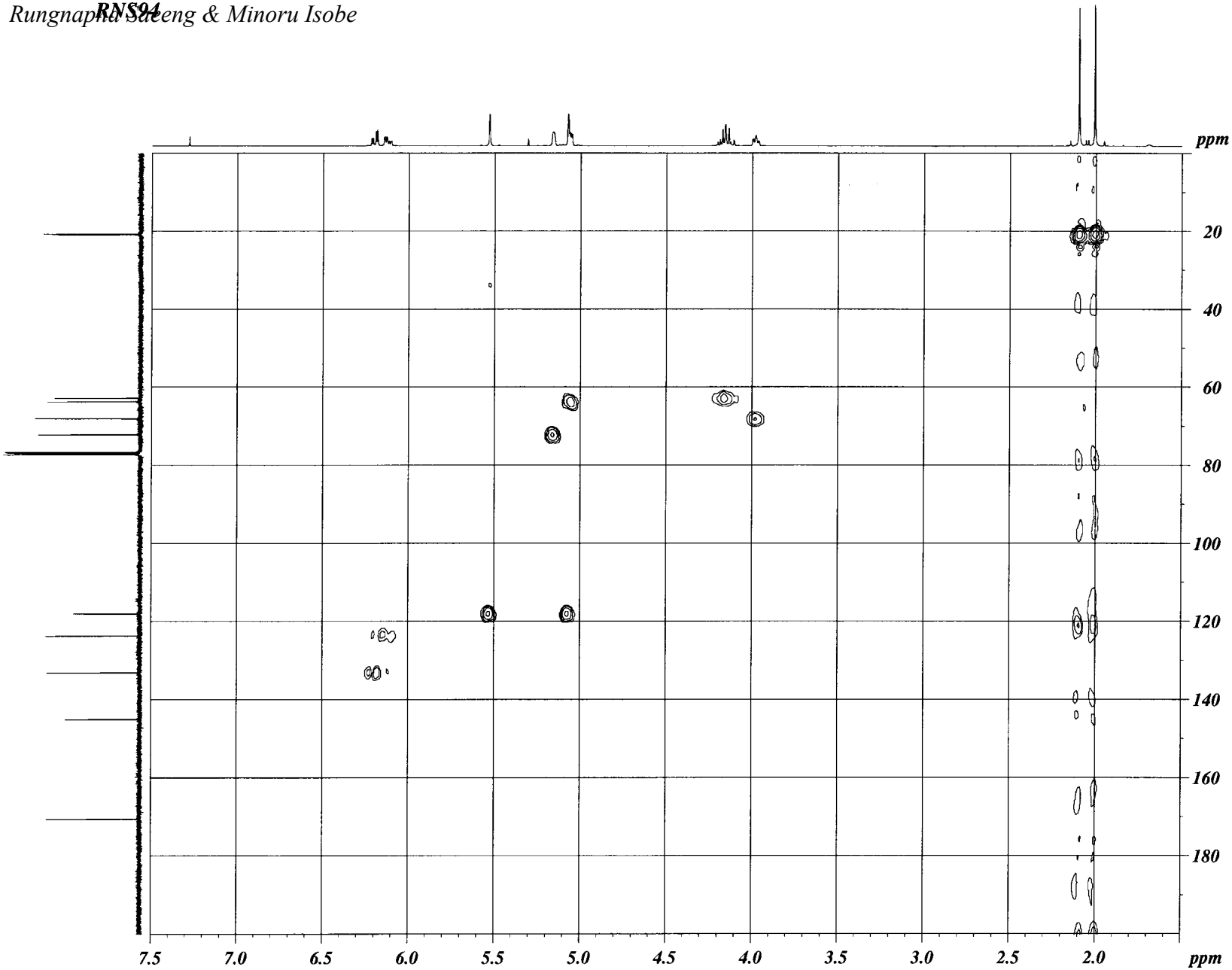
======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P –5.000 ppm
F2 –503.06 Hz
PPMCM 7.33333 ppm/cm
HZCM 737.82697 Hz/cm

RNS94

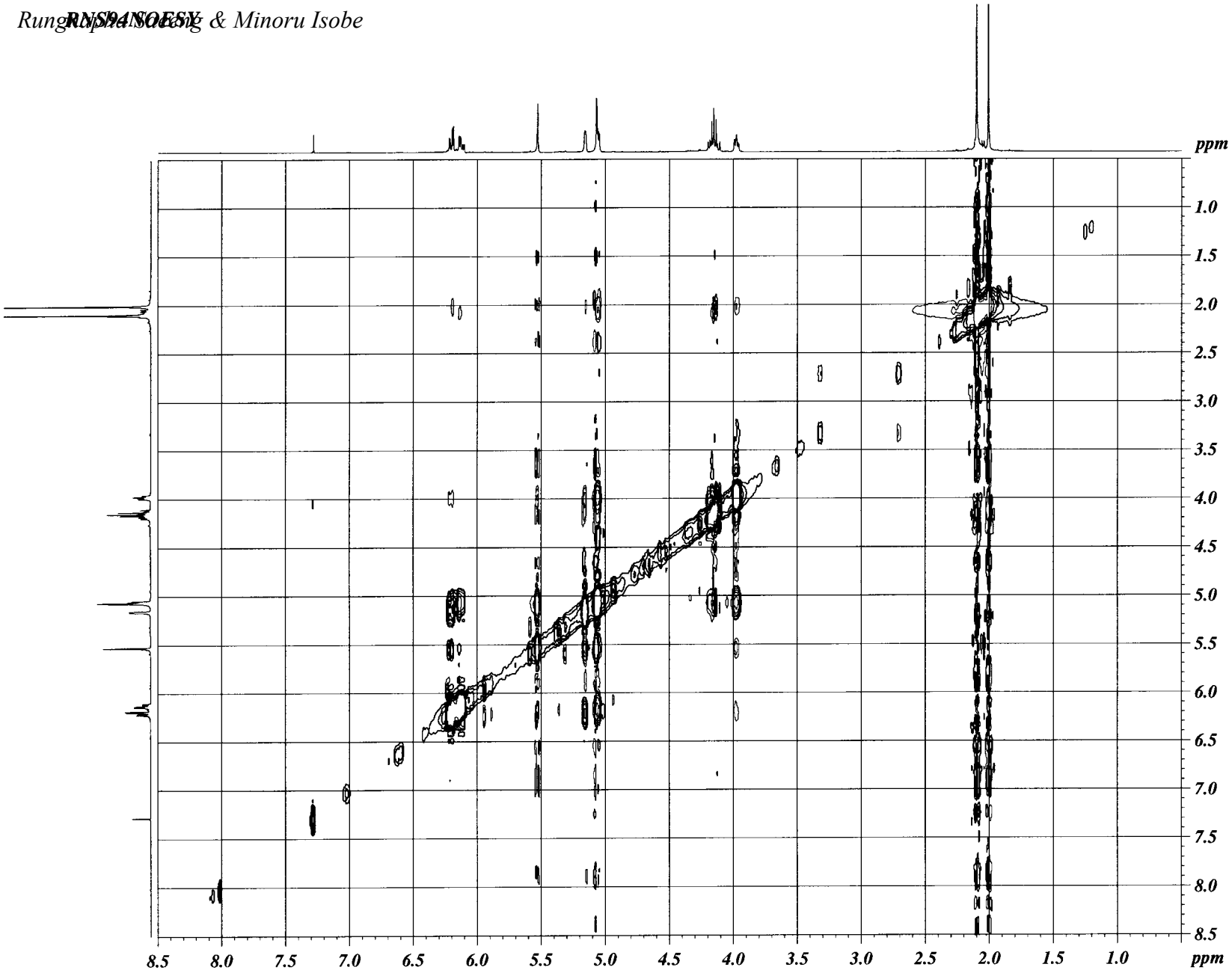


Current Data Parameters
NAME Rungnaphaav400
EXPNO 85
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040519
Time 14.49
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

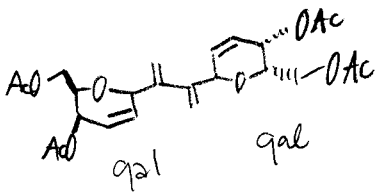


Current Data Parameters
NAME Rungnapha
EXPNO 130
PROCNO 1

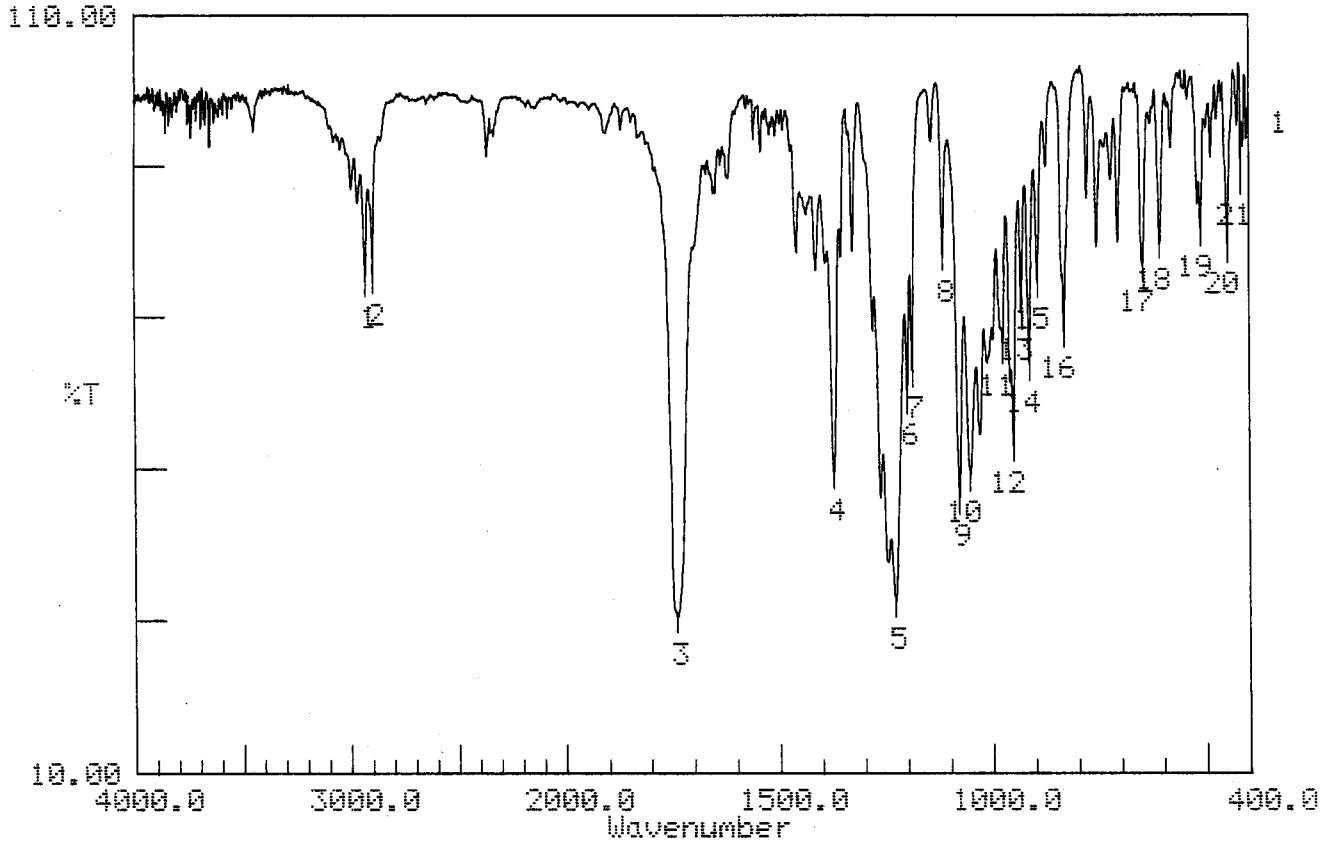
F2 – Acquisition Parameters
Date_ 20040611
Time 10.34
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JASCO FT/IR-8300
Date : 99/04/01 20:54
File Name :
Sample Name: RNS-94-1
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
upper 110.00 lower 10.00 depth 10.00

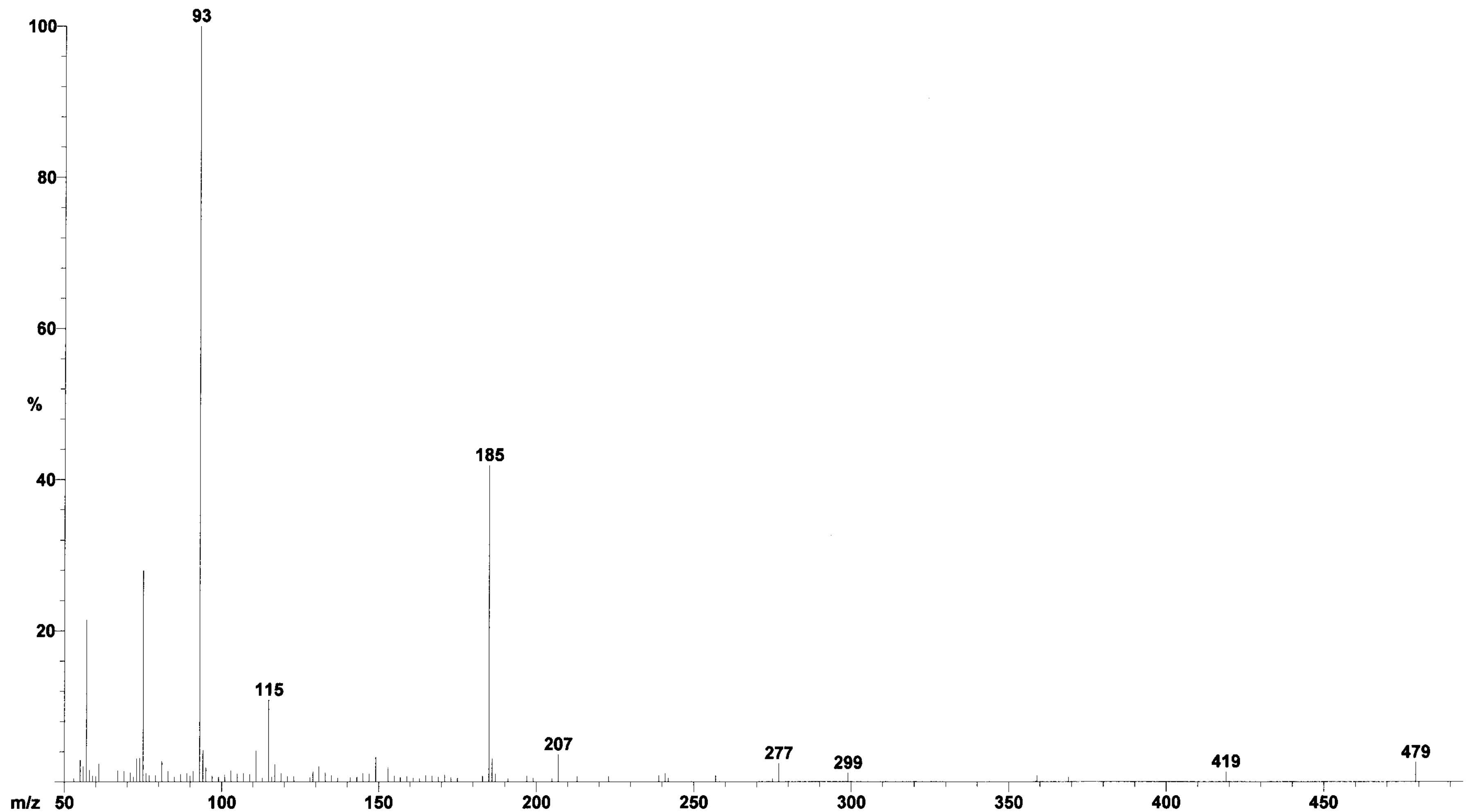
Peak table

1:	2927.33(75.0)	2:	2893.58(75.4)	3:	1739.04(30.5)
4:	1371.55(49.5)	5:	1227.84(32.6)	6:	1199.87(59.5)
7:	1188.30(63.0)	8:	1115.96(78.4)	9:	1077.37(46.2)
10:	1052.30(49.3)	11:	976.10(66.0)	12:	951.02(53.2)
13:	931.73(70.6)	14:	913.41(63.8)	15:	894.11(74.7)
16:	831.42(68.2)	17:	648.16(77.1)	18:	607.65(80.0)
19:	511.20(81.6)	20:	448.50(79.4)	21:	418.60(88.5)

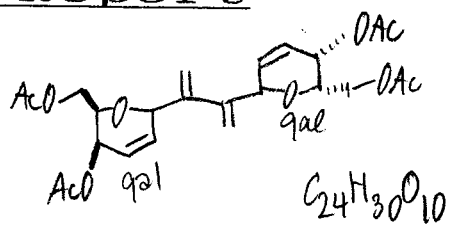
File: RNS-94-1-1 **Date Run: 2004-06-01 (Time Run: 13:35:45)**
Sample: Description
Instrument: JEOL LCmate

Ionization mode: FAB+

Scan: 1 **R.T.: .02**
Base: m/z 93; 12.7%FS TIC: 410528 **#Ions: 89**



Elemental Analysis Report



$C_{24}H_{30}O_{10}$

Refference No. : 361
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-94-1
Sample Weight : 1748 micrograms

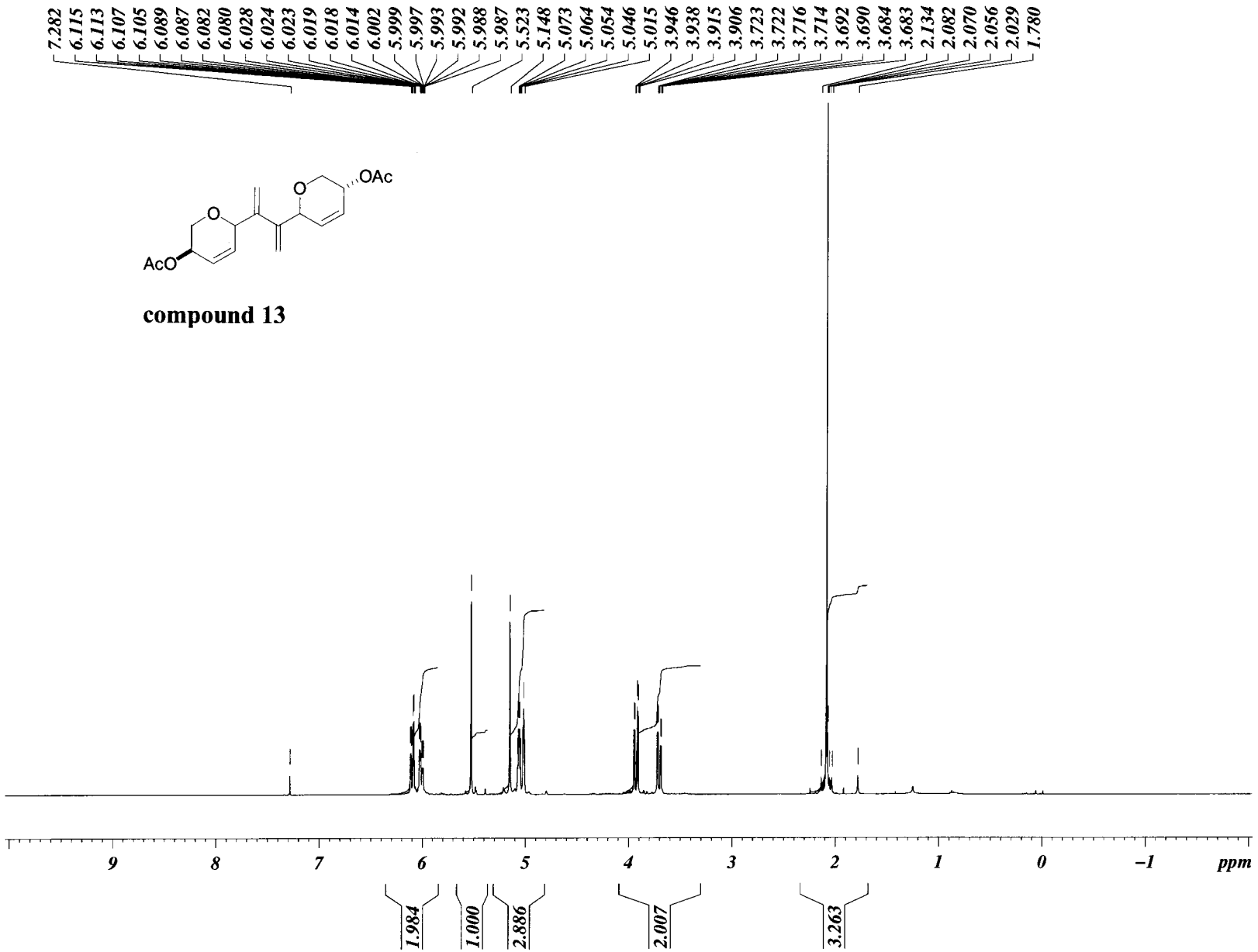
	N	C	H
Calculated weight %	0.00	60.24	6.32
Found weight %	0.00	60.24	6.24
Element ratio(1)	0	13	16
Element ratio(2)	0	17	21
Element ratio(3)	0	30	37
Element ratio(4)	0	47	58
Element ratio(5)	0	64	79
Element ratio(6)	0	77	95
Element ratio(7)	0	81	100

Remarks :

Date : 2004/06/09 15:54:37 Operator : 繁.Kitamura(789-4169)

```
DATE    04.06.06
LAMP    Na589
TEMP
SAMPLE RNS-94
CONC.   0.84%
CELL        100mm
INTEG.TIME 20sec
**********
SAMPLE NO.     deg
017
    1 α  -2.932
      [α]-349.06
    2 α  -2.930
      [α]-348.91
    3 α  -2.934
      [α]-349.31
    4 α  -2.934
      [α]-349.30
    5 α  -2.931
      [α]-348.93
    6 α  -2.930
      [α]-348.87
    7 α  -2.930
      [α]-348.91
    8 α  -2.931
      [α]-348.97
    9 α  -2.931
      [α]-348.94
   10 α  -2.933
      [α]-349.23
-------------------------
MEAN VALUE N=10
      ᾱ  -2.931 deg
   σn-1   0.001 deg
   RSD    0.034 %
      [α]-349.04 deg
   σn-1  0.1720 deg
   TEMP       28
```

RNS93



compound 13

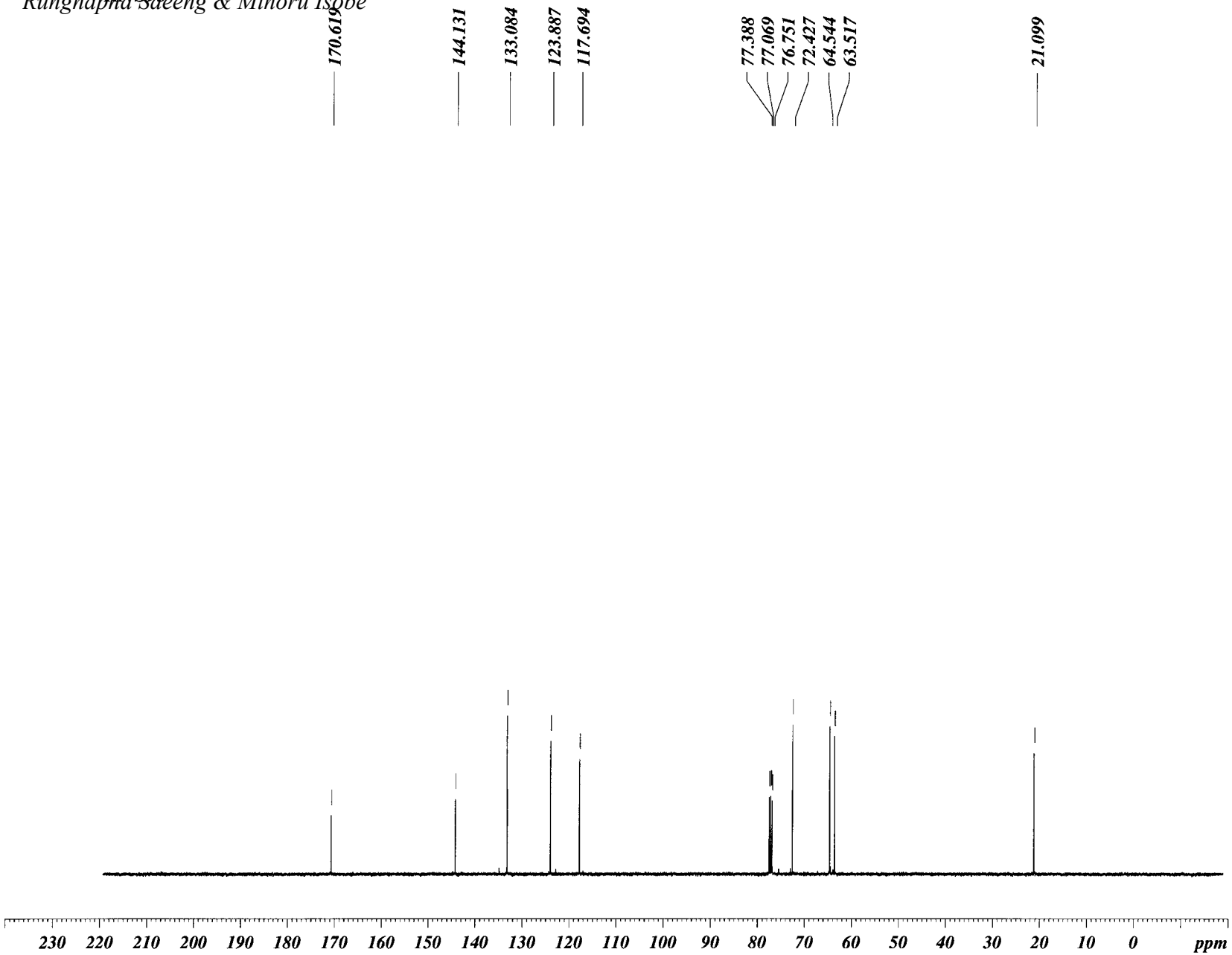
Current Data Parameters
NAME Rungnaphaav400
EXPNO 88
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040519
Time 15.49
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

RNS93



Current Data Parameters
NAME Rungnaphaav400
EXPNO 89
PROCNO 1

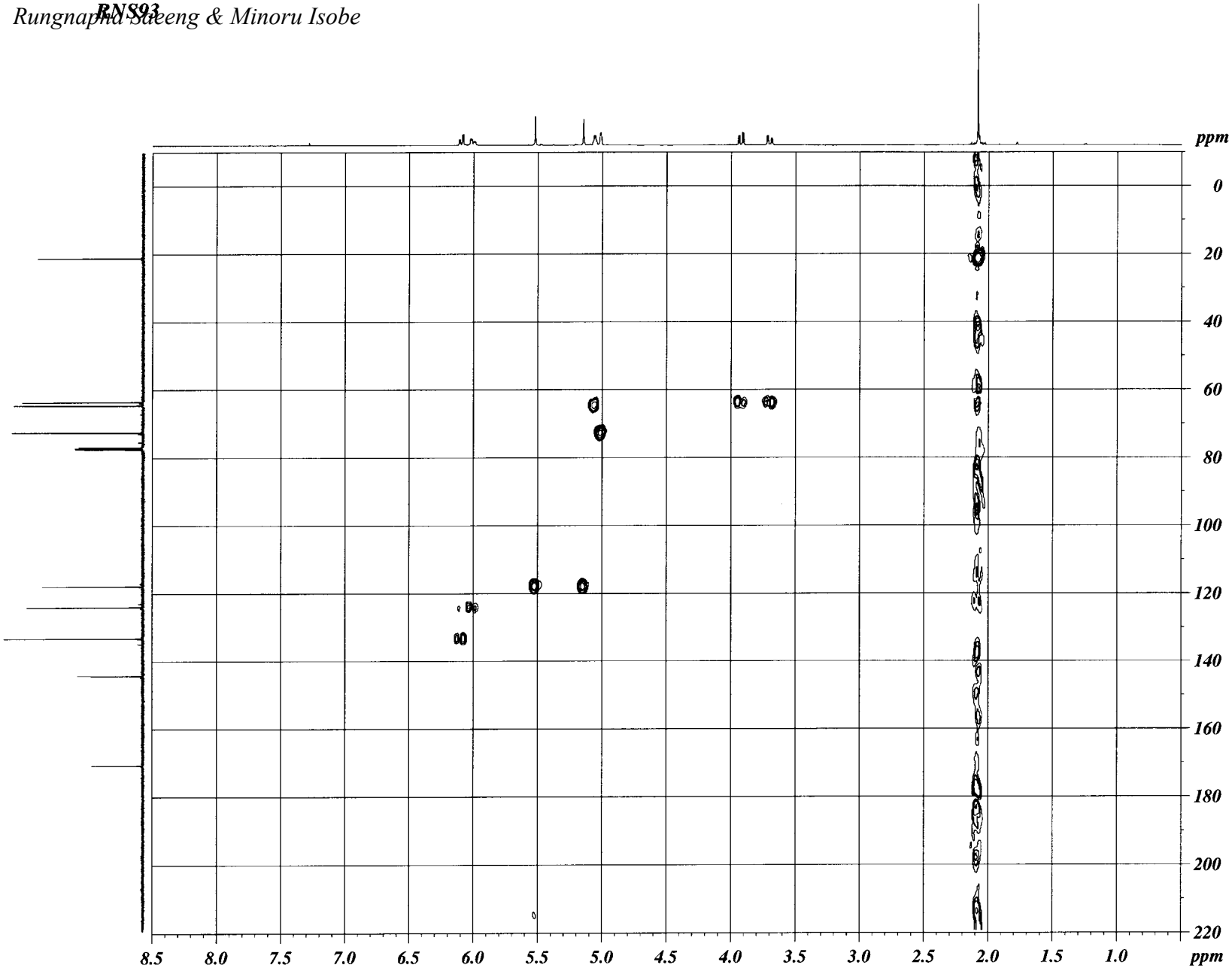
F2 – Acquisition Parameters
Date_ 20040519
Time 15.57
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 109
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

RNS93



Current Data Parameters
NAME Rungnaphaav400
EXPNO 88
PROCNO 1

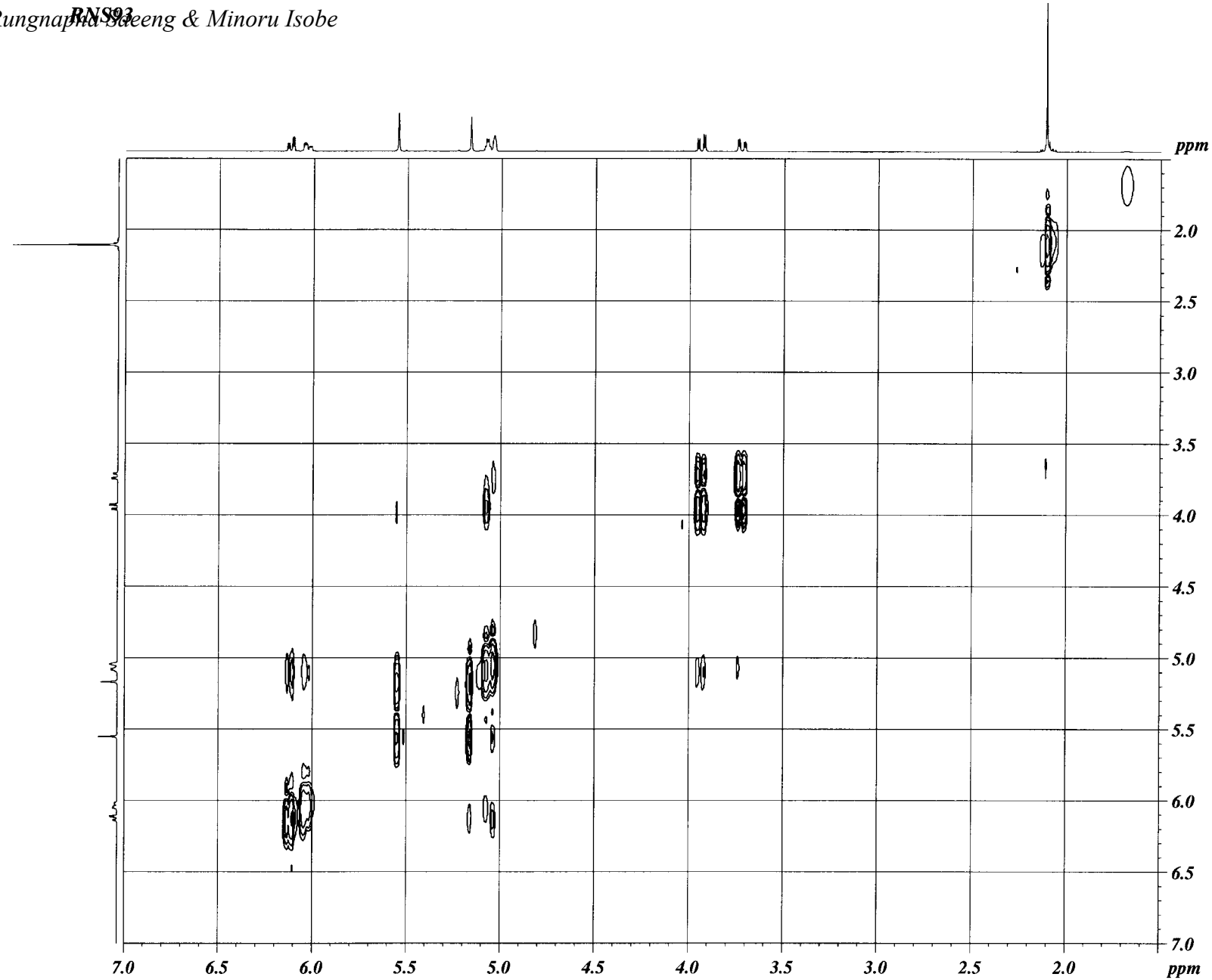
F2 – Acquisition Parameters
Date_ 20040519
Time 15.49
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

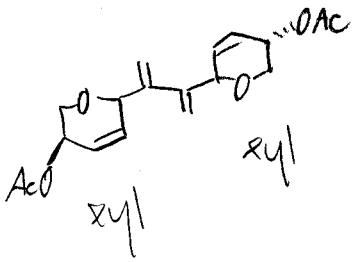
======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

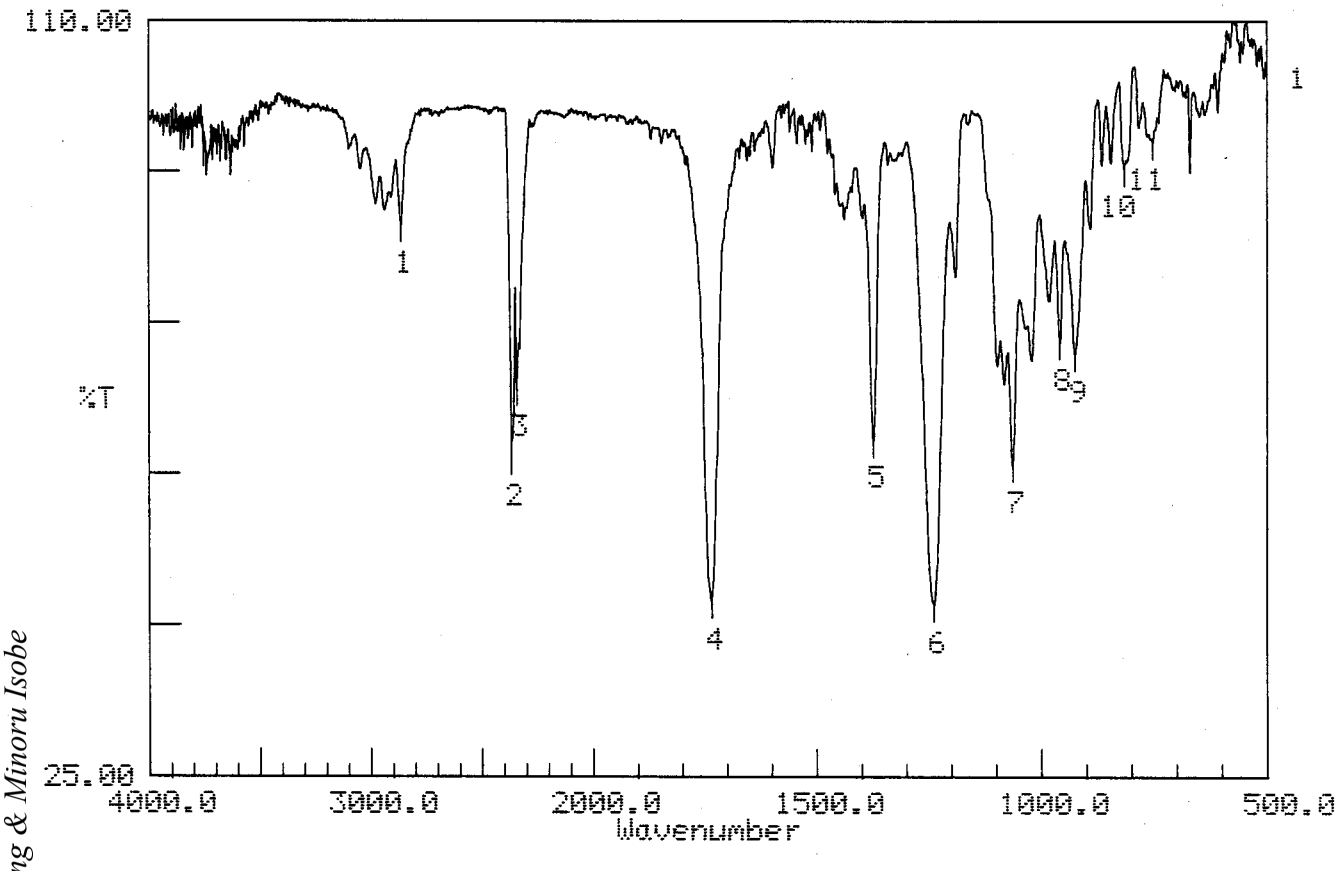
No parameters

NOESY





JASCO FT/IR-8300
Date : 99/04/01 15:37
File Name :
Sample Name: RNS-93
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
upper 110.00 lower 25.00 depth 7.00

Peak table

1:	2861.75(87.0)	2:	2364.05(60.9)	3:	2341.87(68.7)
4:	1733.25(44.7)	5:	1372.52(63.0)	6:	1237.49(44.4)
7:	1062.91(60.2)	8:	957.77(73.9)	9:	924.02(72.6)
10:	814.06(93.4)	11:	751.37(96.4)		

File: RNS-93
Sample: Description
Instrument: JEOL LCmate

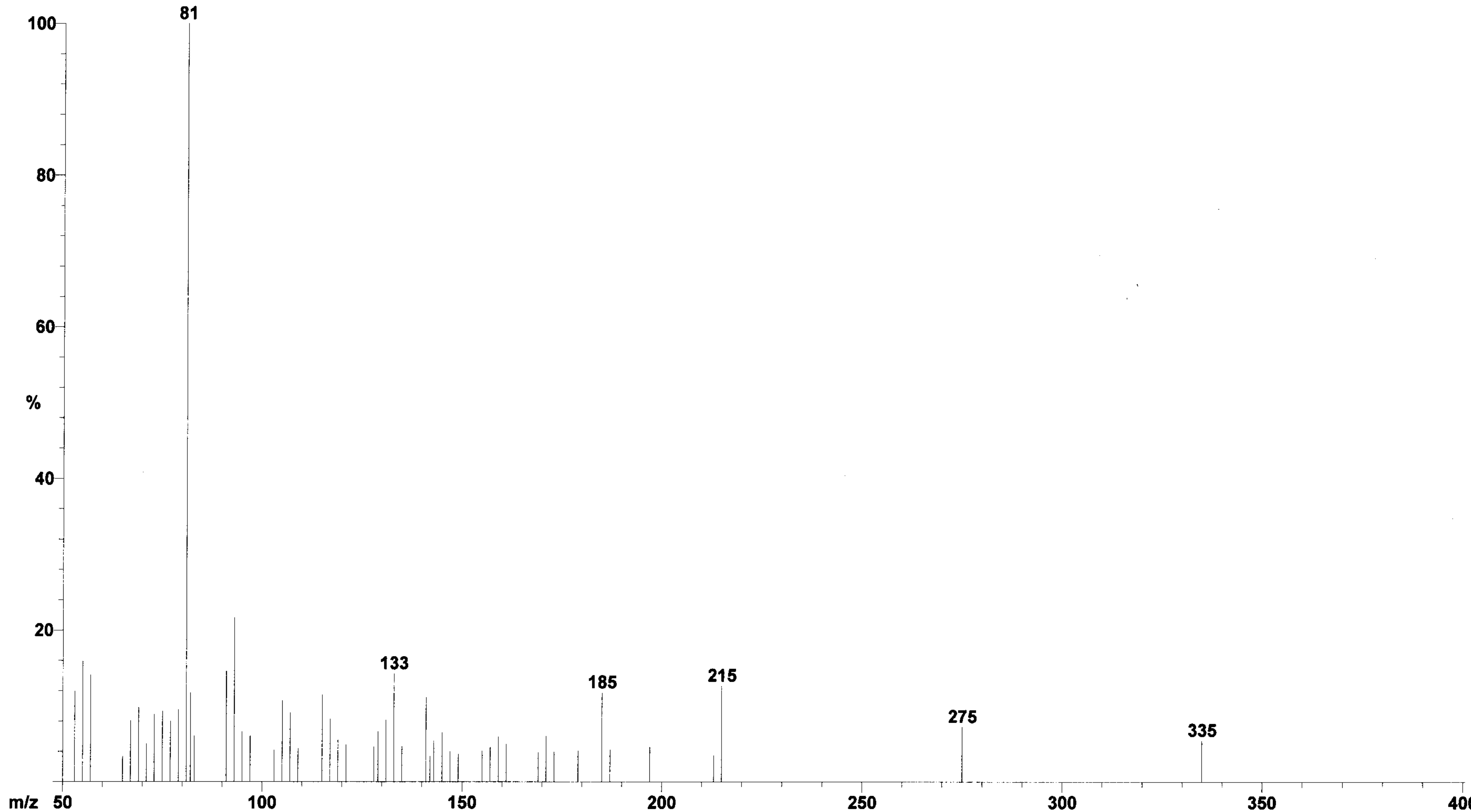
Date Run: 2004-05-31 (Time Run: 19:47:37)

Ionization mode: FAB+

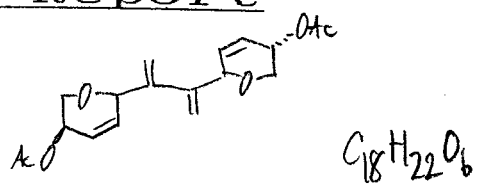
Scan: 2
Base: m/z 81; 1.7%FS TIC: 87904

R.T.: .2

#Ions: 53



Elemental Analysis Report



$C_{18}H_{22}O_6$

Refference No. : 360
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-93-1
Sample Weight : 1695 micrograms

	N	C	H
Calculated weight %	0.00	64.66	6.63
Found weight %	0.00	64.66	6.64
Element ratio(1)	0	9	11

Remarks :

Date : 2004/06/09 15:54:36 Operator : 繁.Kitamura(789-4169)

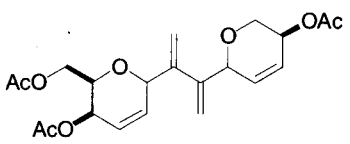
DATE 04.06.06
LAMP Na589
TEMP
SAMPLE ฐNS-93
CONC. 0.98%
CELL 100mm
INTEG.TIME 20sec
****

SAMPLE NO.	deg
013	
1 α	2.852
[α]	291.11
2 α	2.852
[α]	291.05
3 α	2.853
[α]	291.12
4 α	2.850
[α]	290.85
5 α	2.850
[α]	290.85
6 α	2.851
[α]	290.92
7 α	2.851
[α]	290.96
8 α	2.851
[α]	290.98
9 α	2.850
[α]	290.84
10 α	2.849
[α]	290.72

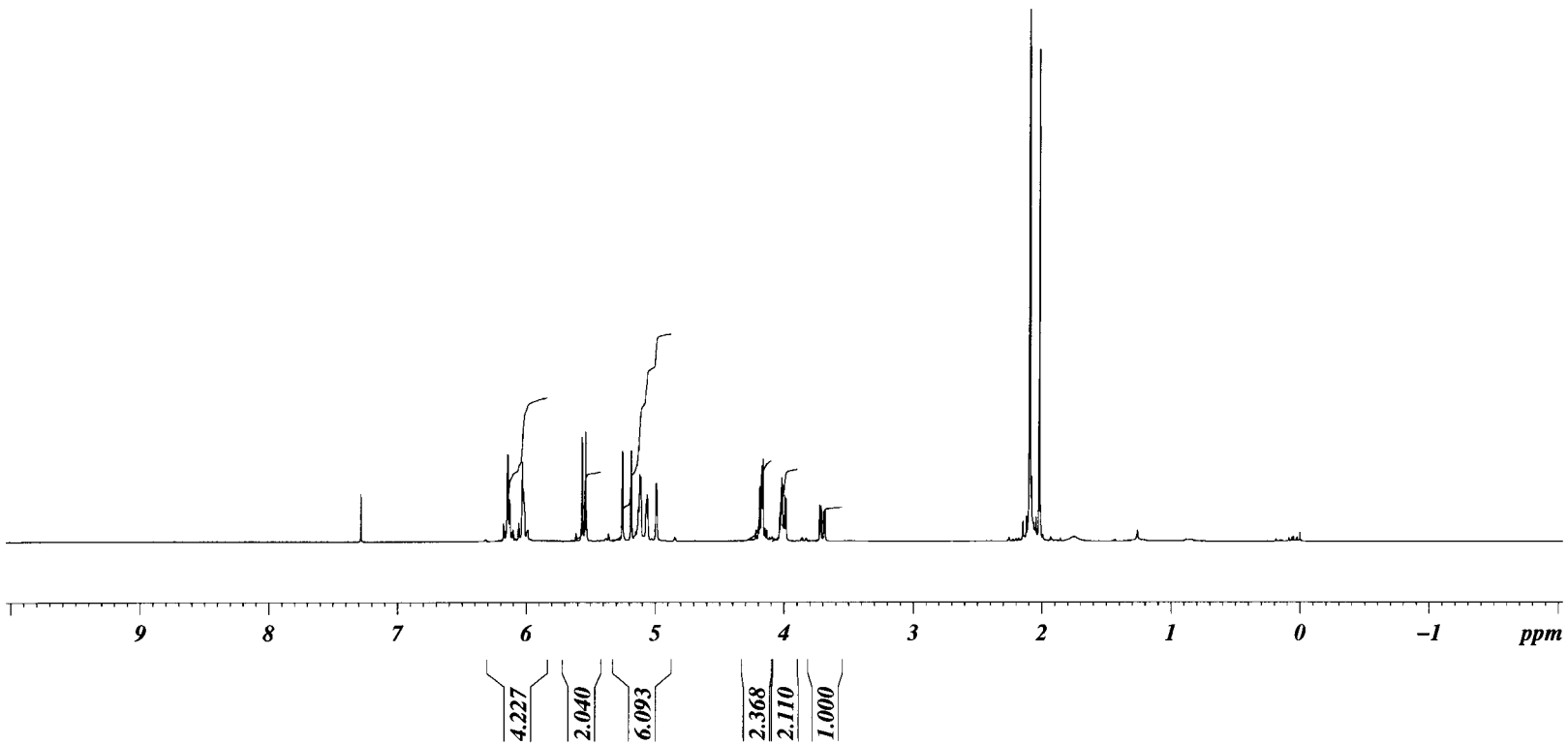
MEAN VALUE N=10	
$\bar{\alpha}$	2.851 deg
σn-1	0.001 deg
RSD	0.035 %
[α]	290.94 deg
σn-1	0.1300 deg
TEMP	26

Rungnapha Saeeng & Minoru Isobe

PNS100



compound 14

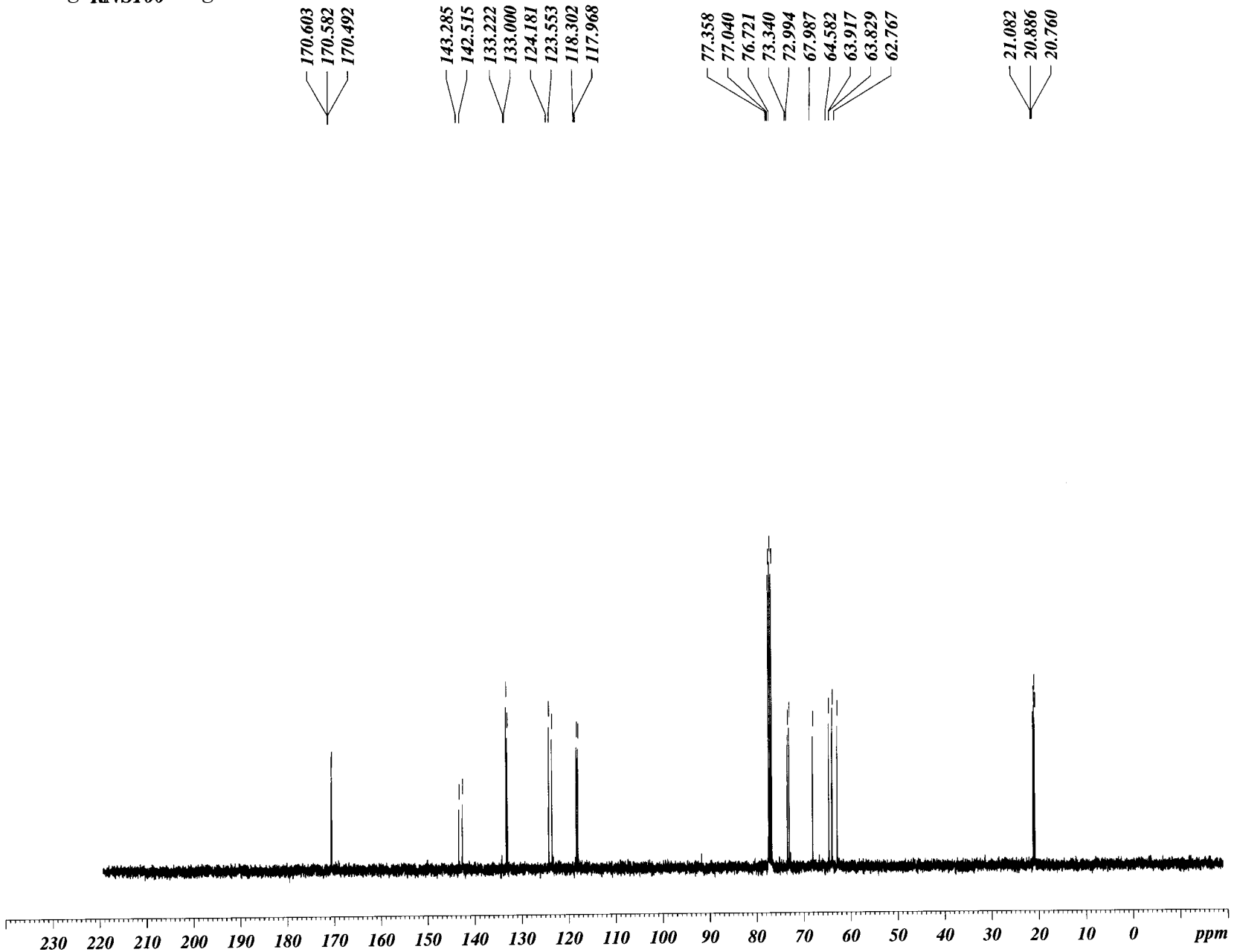


Current Data Parameters
NAME Rungnaphaav400
EXPNO 105
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040530
Time 21.21
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Rungnaphaav400
EXPNO 107
PROCNO 1

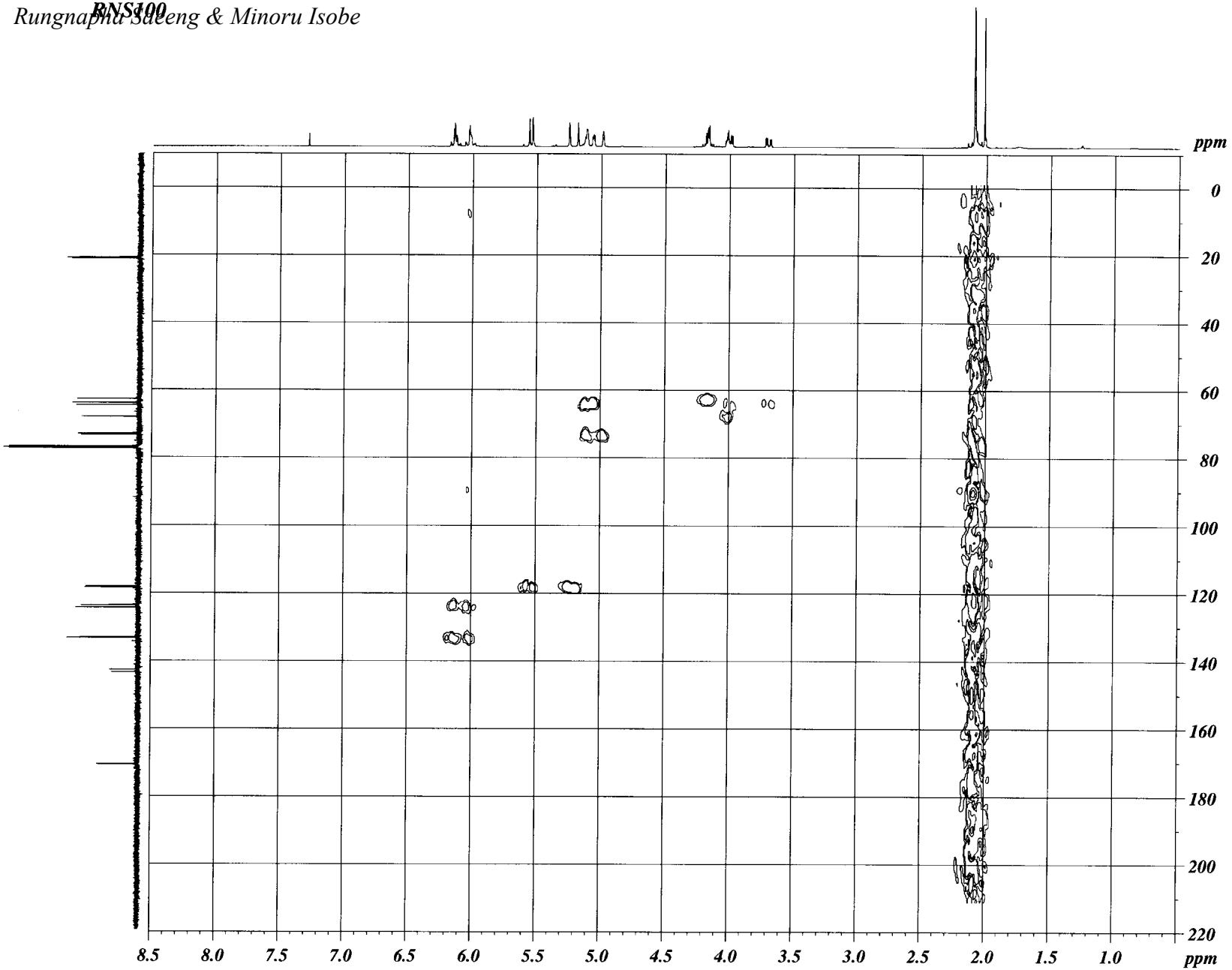
F2 – Acquisition Parameters
Date_ 20040531
Time 10.25
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 132
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

RNS100



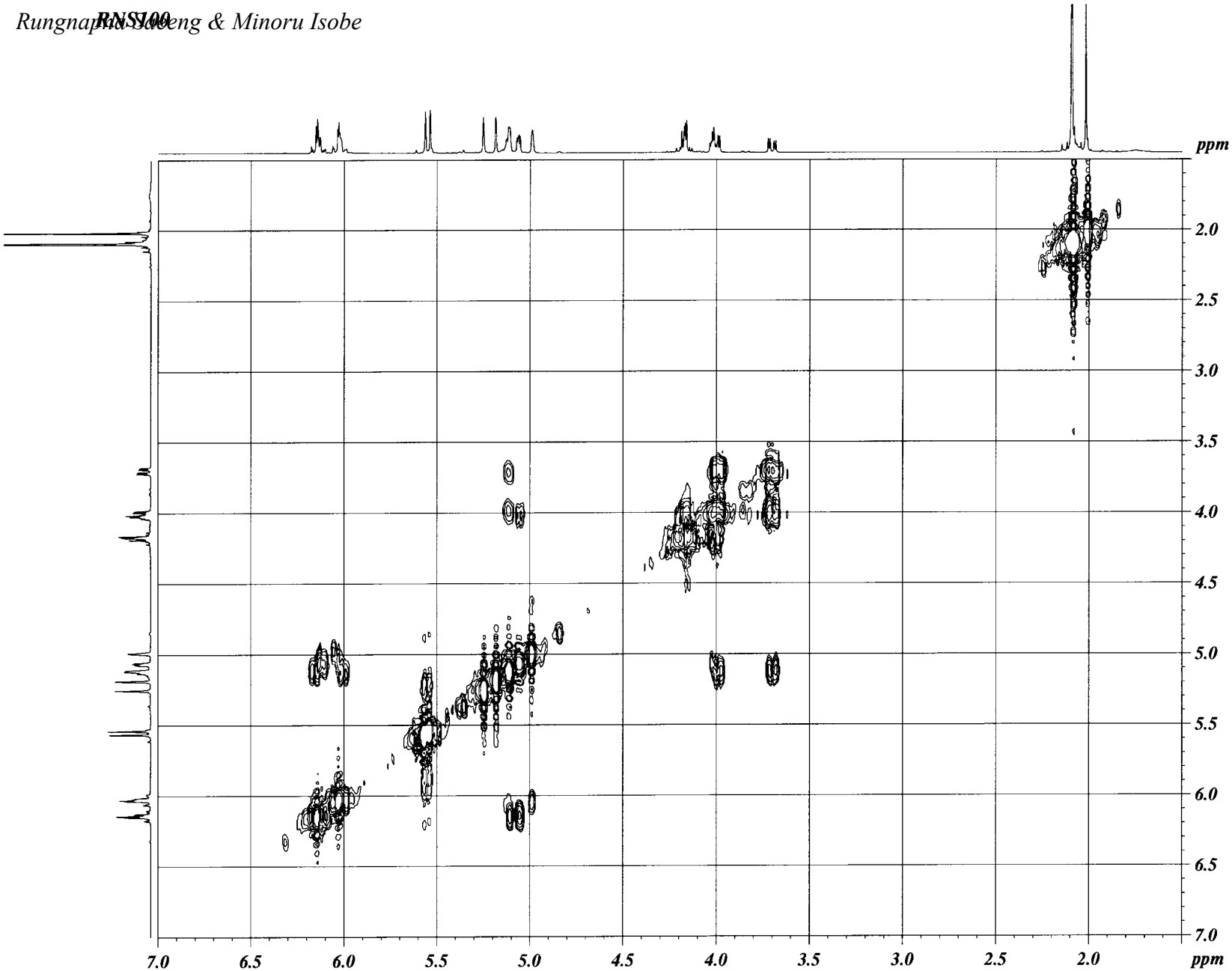
Current Data Parameters
NAME Rungnaphaav400
EXPNO 105
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040530
Time 21.21
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

COSY

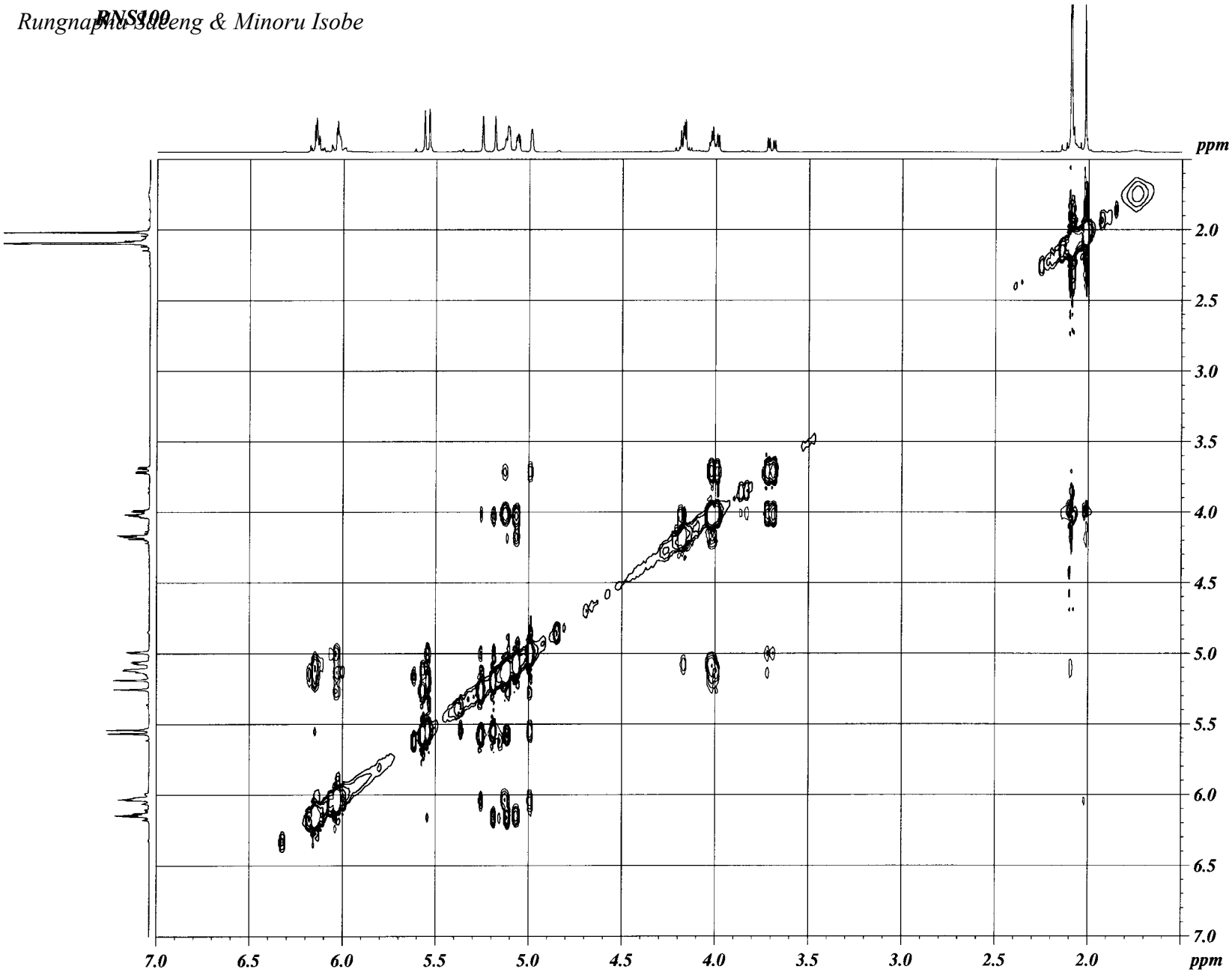


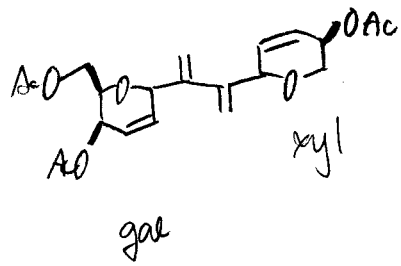
Current Data Parameters
NAME Rungnaphaav400
EXPNO 105
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040530
Time 21.21
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

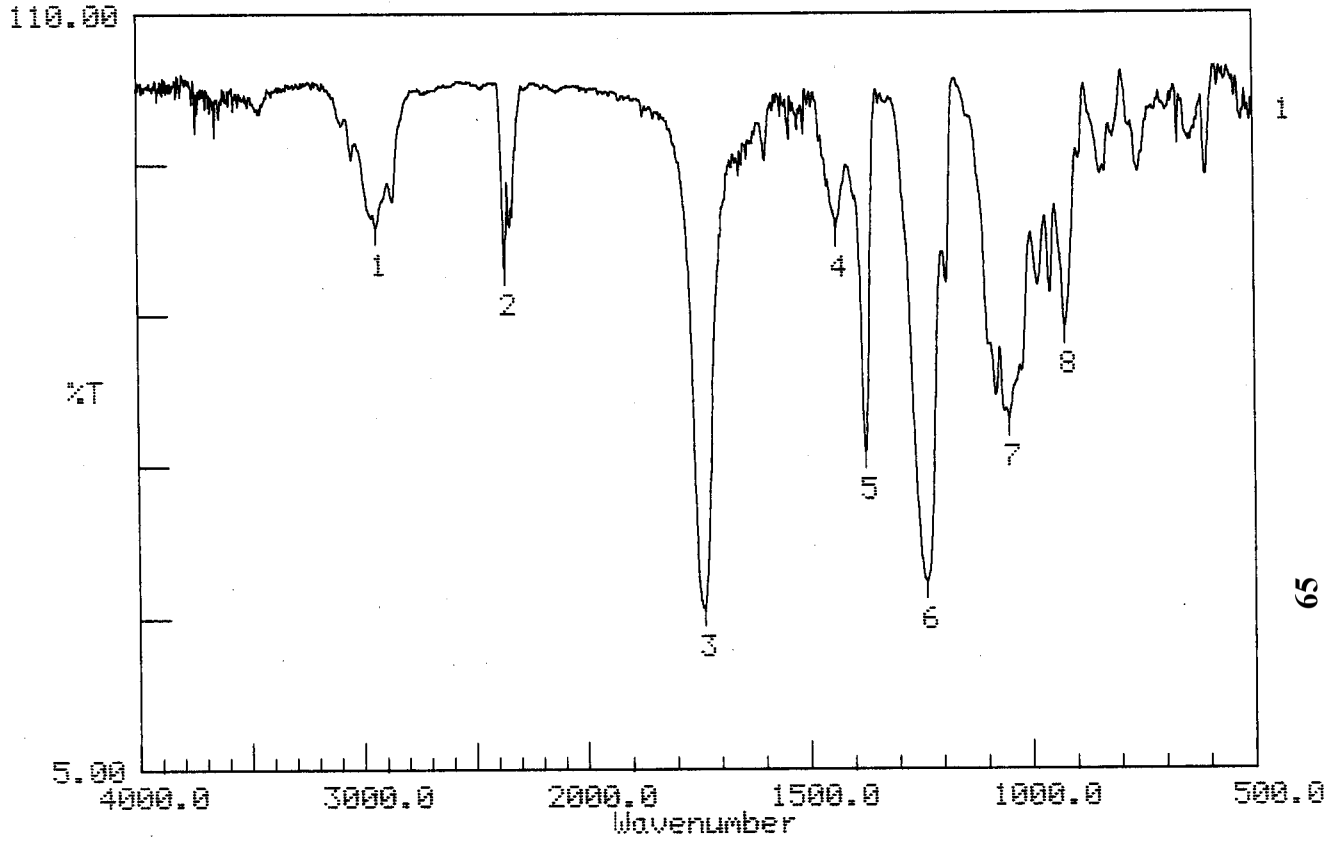
F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


RNS100
NOESY
ppm
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Current Data Parameters
NAME Rungnaphaav400
EXPNO 105
PROCNO 1
F2 – Acquisition Parameters
Date_ 20040530
Time 21.21
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz
F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JASCO FT/IR-8300

Date : 99/04/01 15:53
File Name :
Sample Name: RNS-100
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition

upper 110.00 lower 5.00 depth 7.00

Peak table

1:	2936.98(80.3)	2:	2363.09(74.7)	3:	1736.15(27.4)
4:	1437.14(79.9)	5:	1371.55(49.0)	6:	1236.52(31.2)
7:	1049.40(53.6)	8:	924.02(66.4)		

File: RNS-100
Sample: Description
Instrument: JEOL LCmate

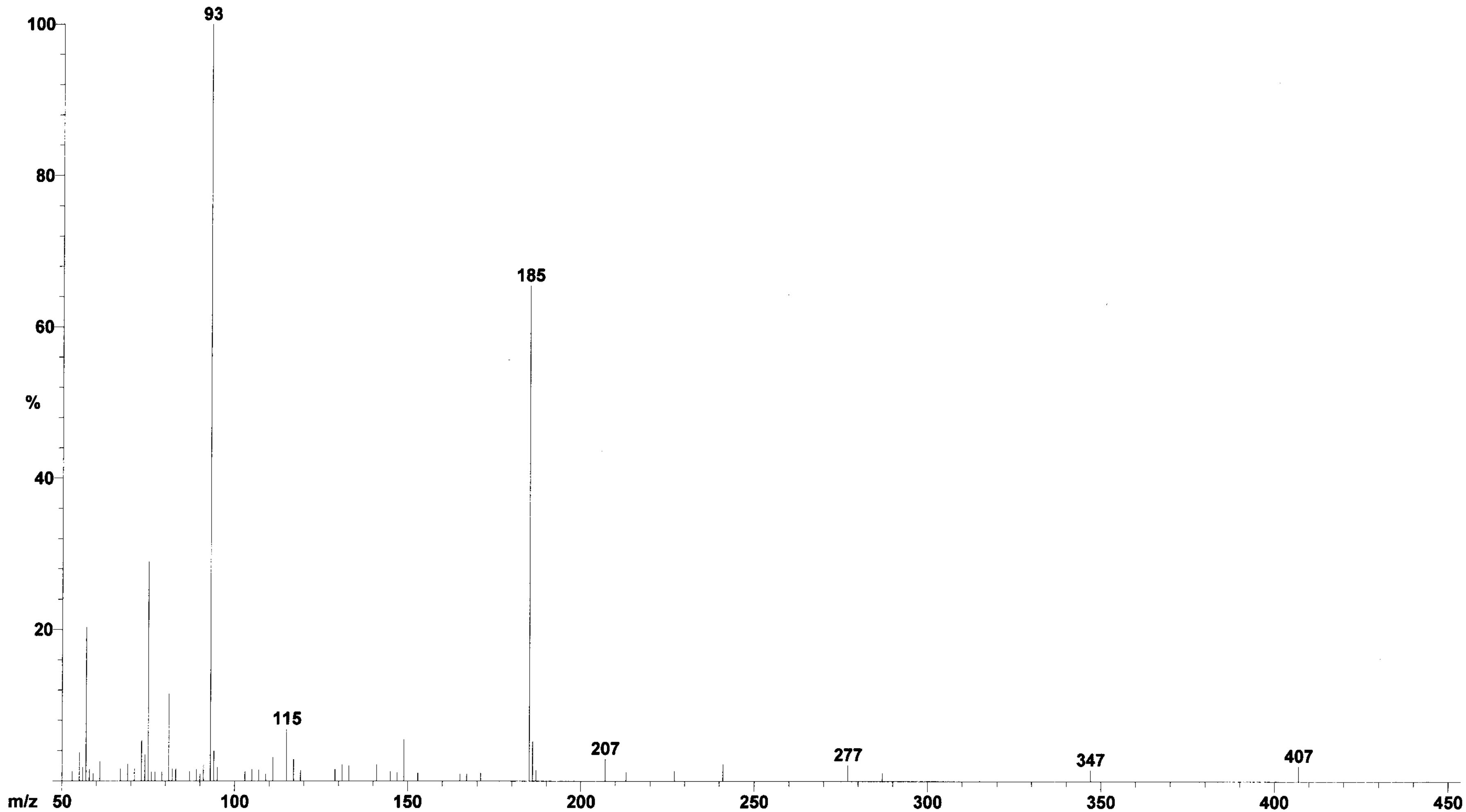
Date Run: 2004-05-31 (Time Run: 20:17:28)

Ionization mode: FAB+

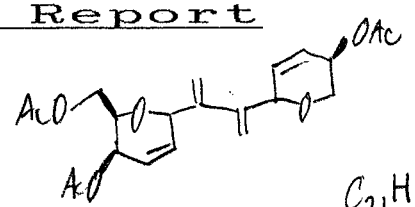
Scan: 3
Base: m/z 93; 6.2%FS TIC: 217088

R.T.: .38

#Ions: 56



Elemental Analysis Report



$C_{21}H_{26}O_8$

Refference No. : 369
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-100
Sample Weight : 829 micrograms

	N	C	H
Calculated weight %	0.00	62.06	6.45
Found weight %	0.00	62.04	6.39
Element ratio(1)	0	9	11
Element ratio(2)	0	13	16
Element ratio(3)	0	22	27
Element ratio(4)	0	35	43
Element ratio(5)	0	57	70
Element ratio(6)	0	79	97

Remarks :

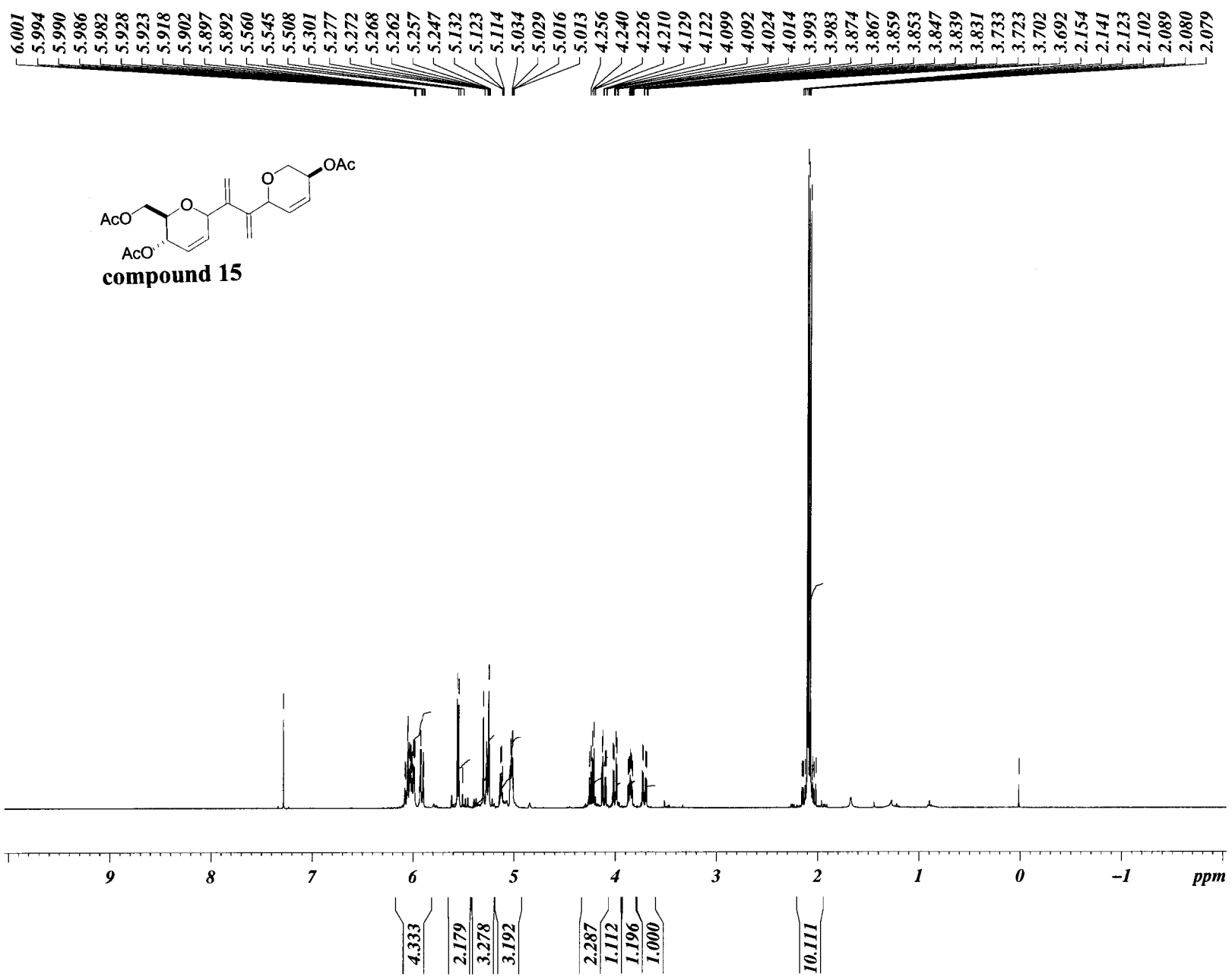
Date : 2004/06/09 15:54:51 Operator : 繁.Kitamura(789-4169)

DATE 04.06.04
LAMP Na589
TEMP
SAMPLE RIS-100
CONC. 1.175%
CELL 100mm
INTEG.TIME 20sec
****
SAMPLE NO. deg
014
1 α -0.550
[α]-46.863
2 α -0.556
[α]-47.378
3 α -0.554
[α]-47.203
4 α -0.554
[α]-47.180
5 α -0.553
[α]-47.098
6 α -0.555
[α]-47.261
7 α -0.556
[α]-47.349
8 α -0.556
[α]-47.389
9 α -0.555
[α]-47.266
10 α -0.554
[α]-47.219

MEAN VALUE N=10
$\bar{\alpha}$ -0.554 deg
σn-1 0.002 deg
RSD 0.360 %

[α]-47.221 deg
σn-1 0.1557 deg
TEMP 27

[α] = -47.22

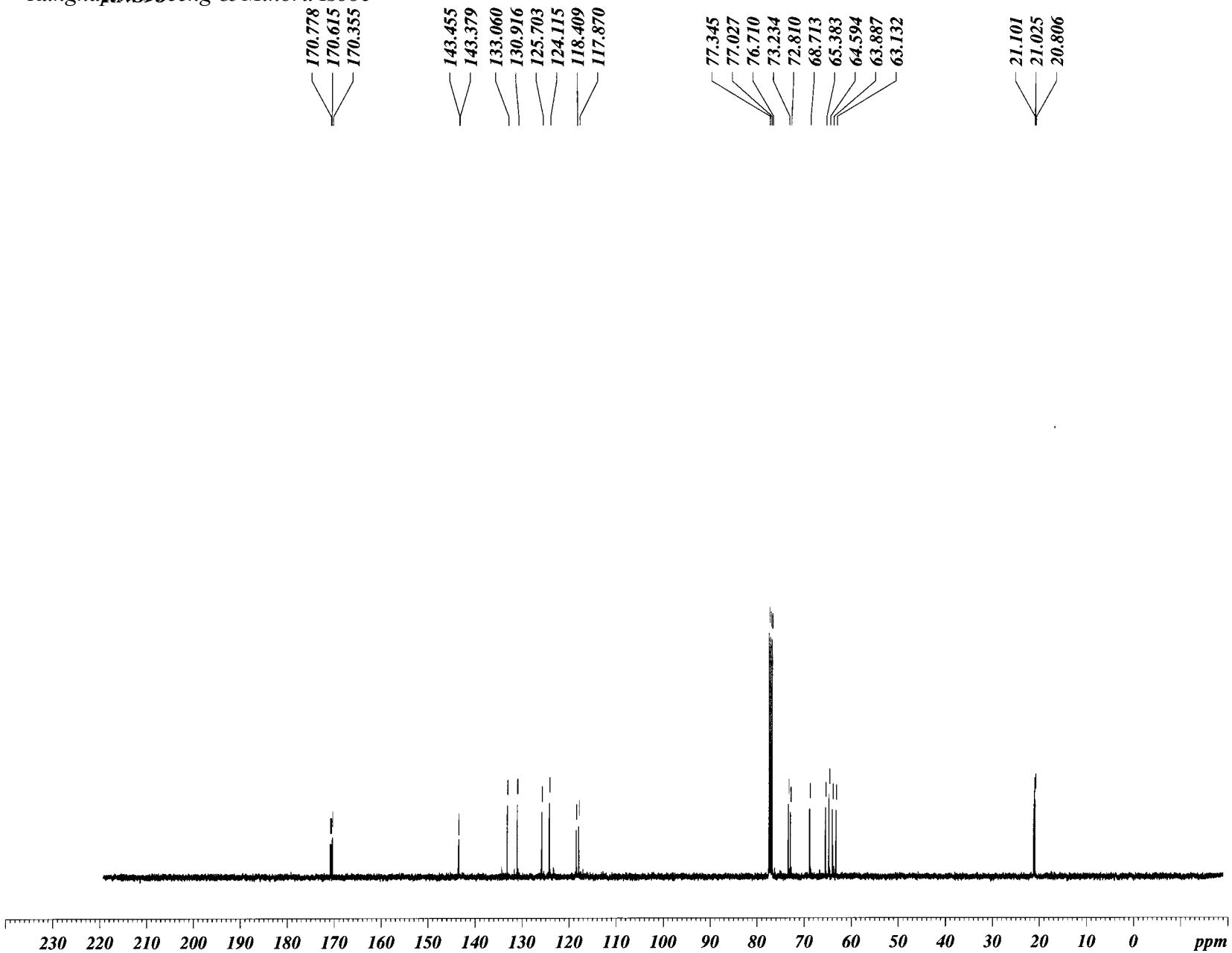


Current Data Parameters
NAME Rungnaphaav400
EXPNO 98
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040526
Time 20.56
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



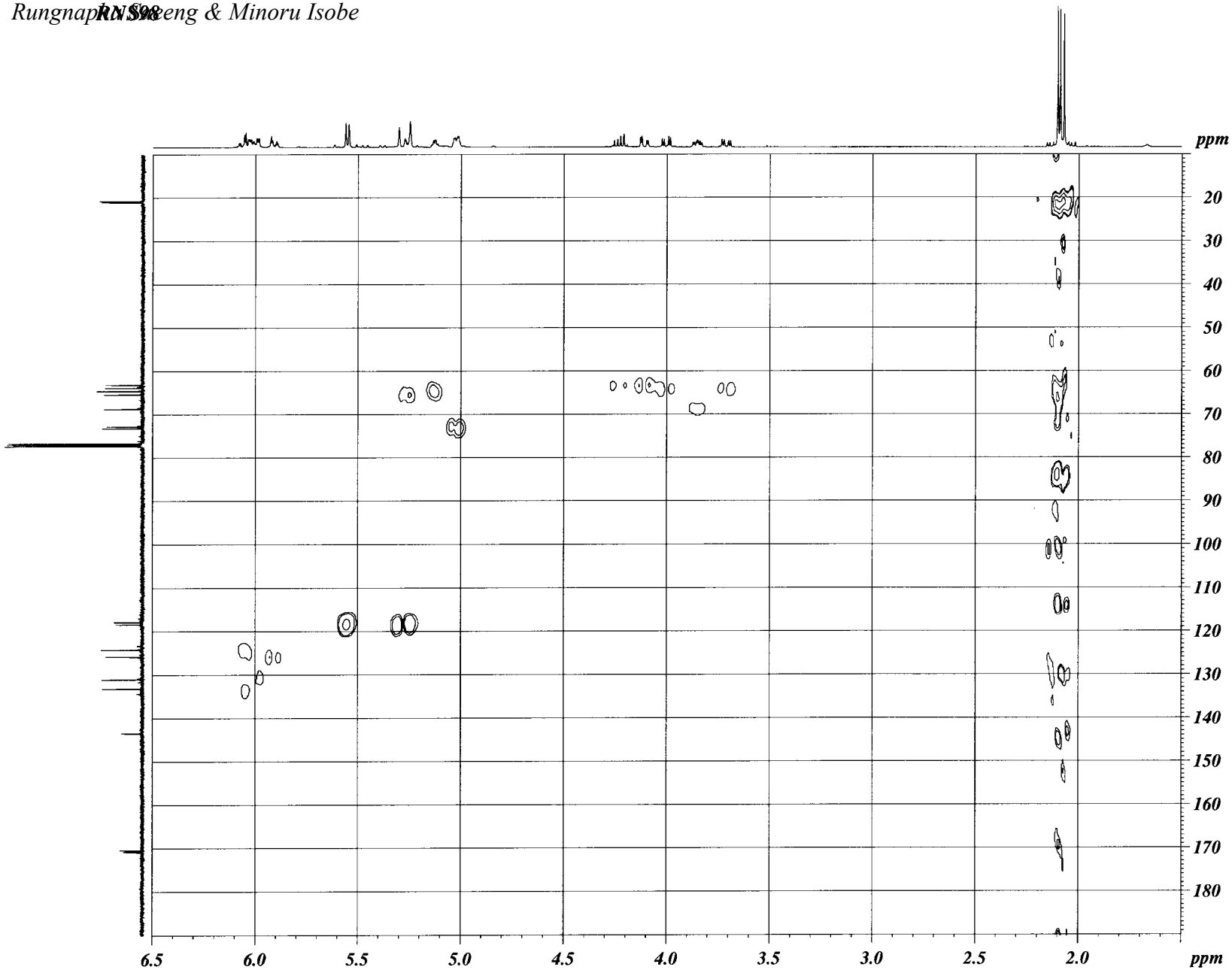
Current Data Parameters
NAME Rungnaphaav400
EXPNO 99
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040526
Time 21.09
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 221
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4597.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 −3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 −4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

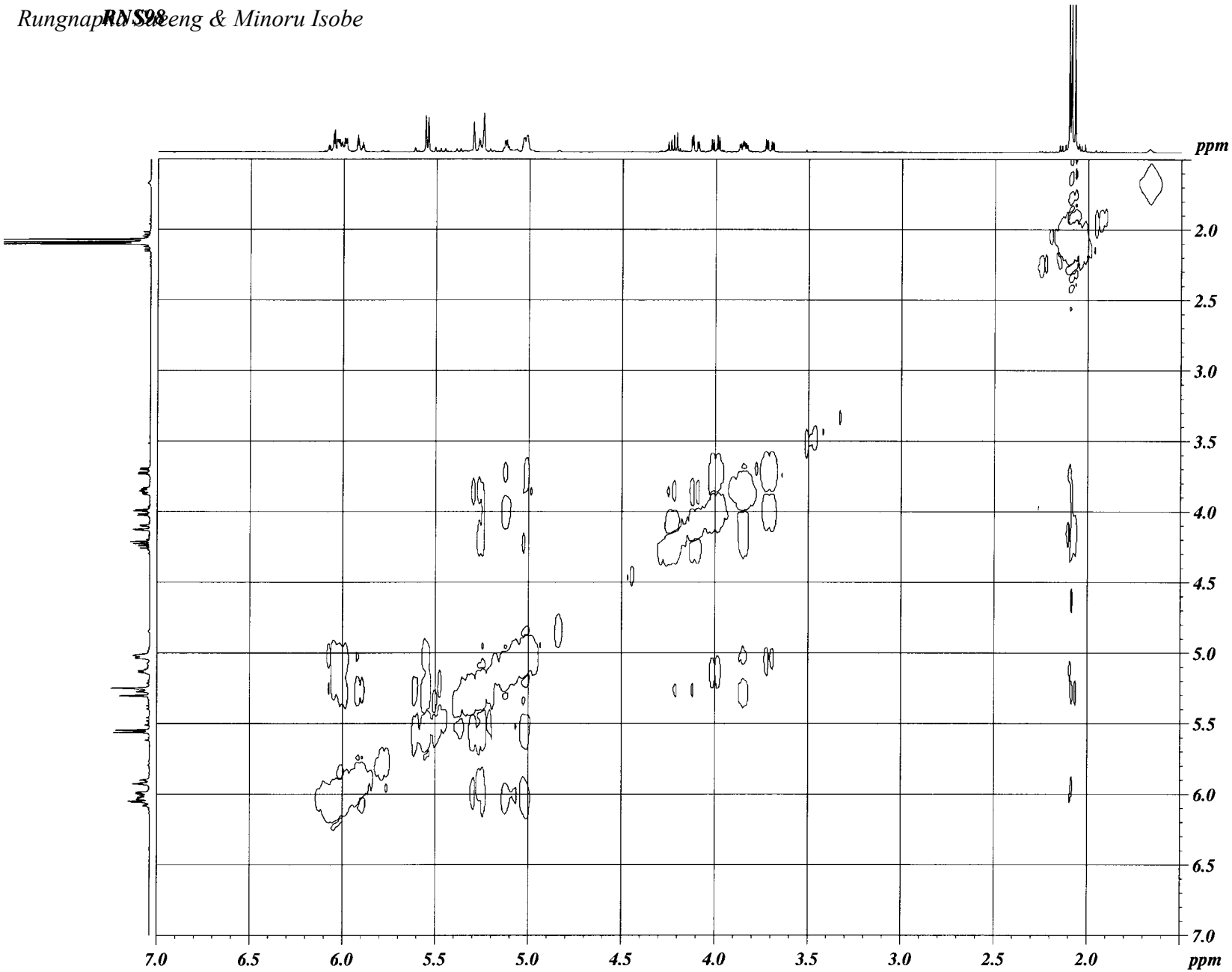


Current Data Parameters
NAME Rungnaphaav400
EXPNO 98
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040526
Time 20.56
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



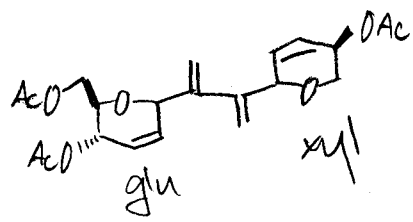
NOESY

Current Data Parameters
NAME Rungnaphaav400
EXPNO 98
PROCNO 1

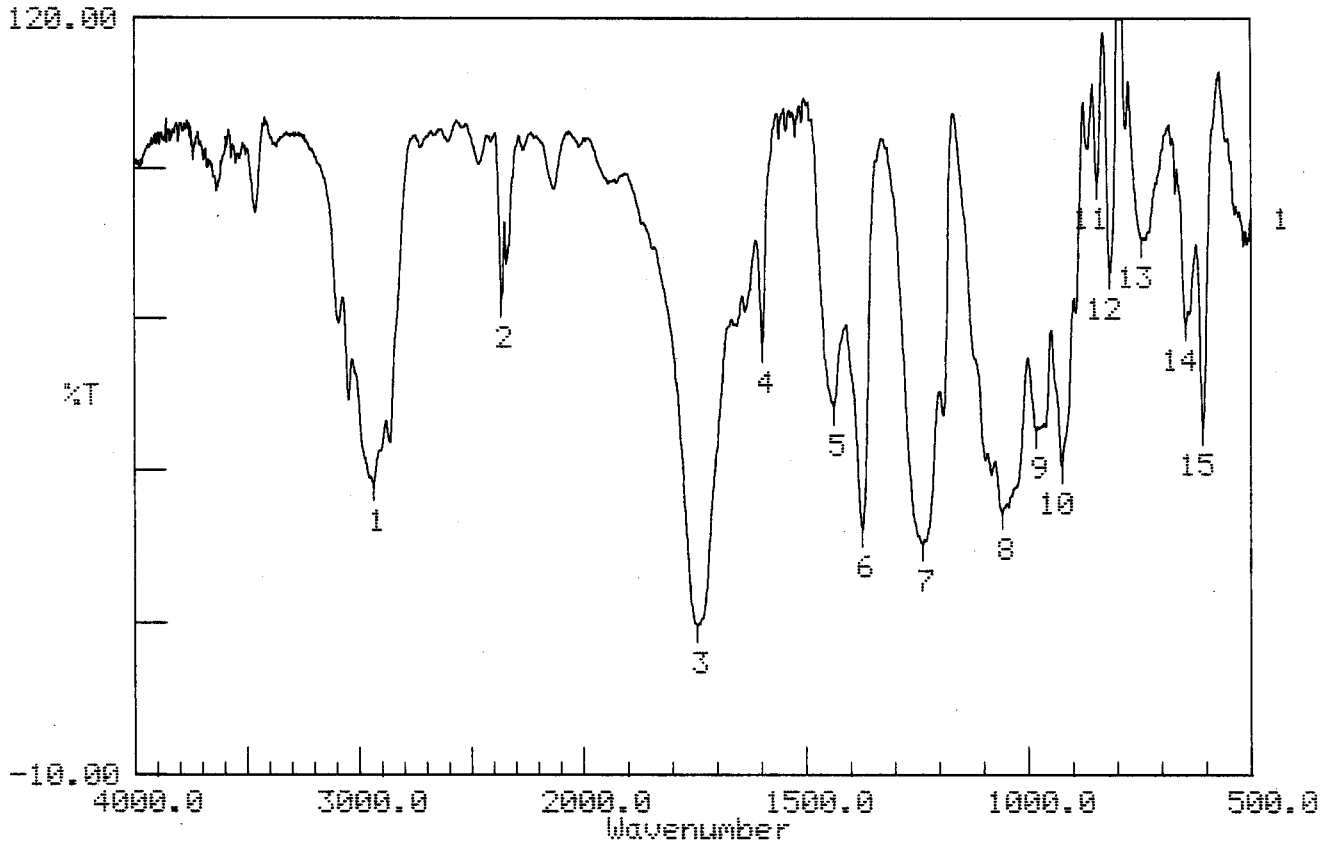
F2 – Acquisition Parameters
Date_ 20040526
Time 20.56
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JASCO FT/IR-8300
Date : 99/04/01 15:45
File Name :
Sample Name: RNS-98-3
Resolution : 4
Scans : 16
Gain : 50
Apodization: CS



Condition
upper 120.00 lower -10.00 depth 10.00

Peak table

1:	2936.01(40.0)	2:	2364.05(71.3)	3:	1743.86(15.6)
4:	1596.29(63.6)	5:	1436.18(52.8)	6:	1371.55(32.2)
7:	1236.52(29.7)	8:	1057.12(35.1)	9:	980.92(49.1)
10:	923.05(43.0)	11:	844.92(92.0)	12:	815.02(76.2)
13:	744.61(81.8)	14:	645.27(67.5)	15:	606.69(49.7)

File: RNS-98 **Date Run: 2004-05-31 (Time Run: 20:08:41)**

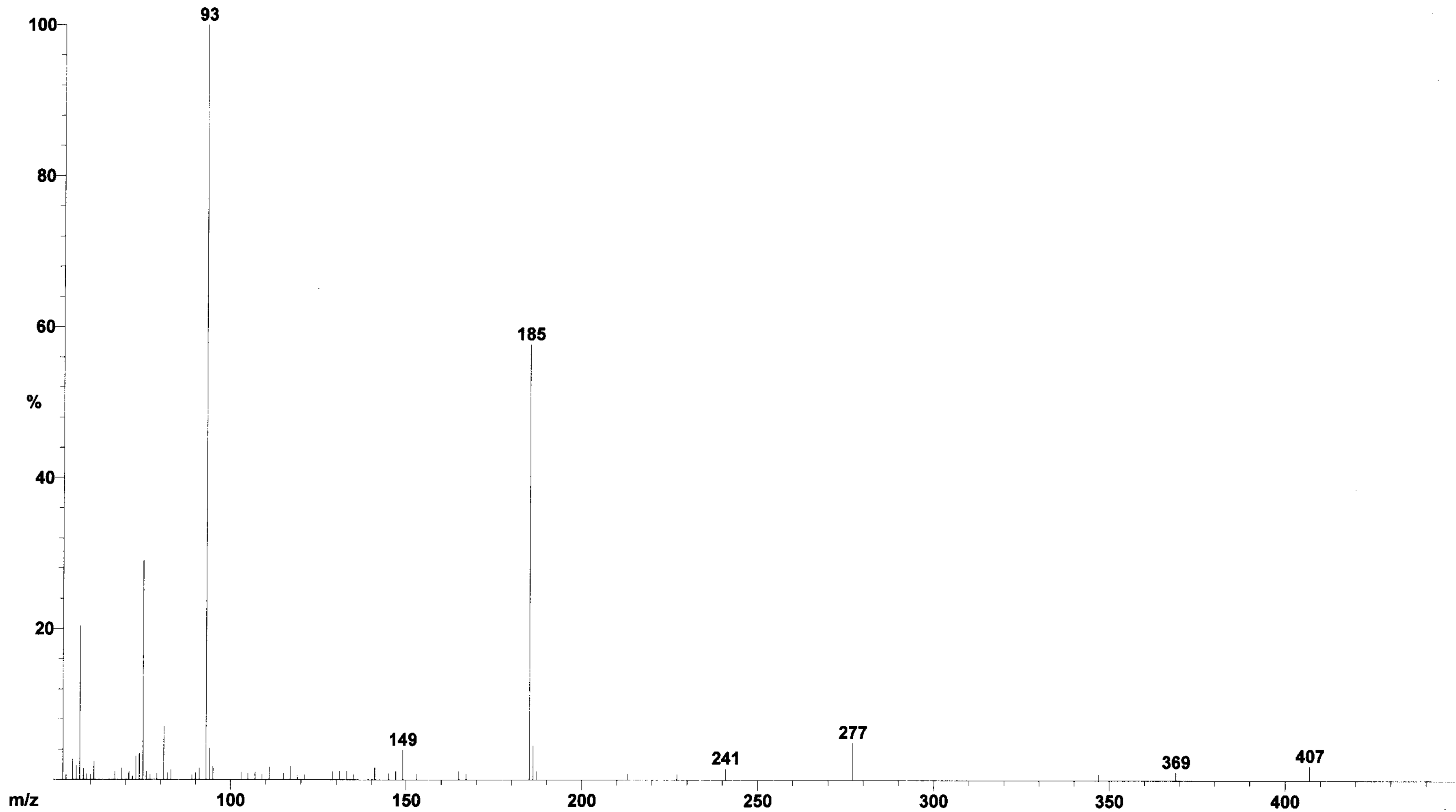
Sample Description

Instrument: JEOL LCmate

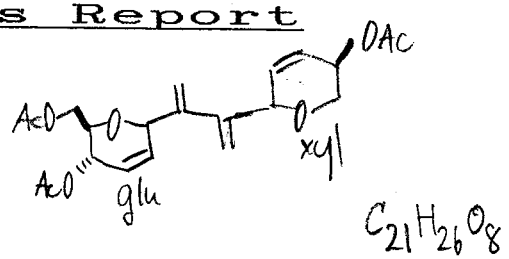
Ionization mode: FAB+

Scan: 1 **R.T.: .02**

Base: m/z 93; 8.9%FS TIC: 272272 **#Ions: 57**



Elemental Analysis Report



$C_{21}H_{26}O_8$

Refference No. : 370
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-98-2
Sample Weight : 2315 micrograms

	N	C	H
Calculated weight %	0.00	62.06	6.45
Found weight %	0.11	62.06	6.47
Element ratio(1)	0	4	5
Element ratio(2)	0	25	31
Element ratio(3)	0	29	36
Element ratio(4)	0	33	41
Element ratio(5)	0	37	46
Element ratio(6)	0	41	51
Element ratio(7)	0	54	67

Remarks :

Date : 2004/06/09 15:54:54 Operator : 繁.Kitamura(789-4169)

DATE 04.06.04
LAMP Na589
TEMP
SAMPLE RNS-97-5
CONC. 1.215%
CELL 100mm
INTEG.TIME 20sec
****

SAMPLE NO.	deg
015	
1 α	1.899
[α]	156.30
~~2~~ α	1.908
[α]	157.09
3 α	1.902
[α]	156.61
4 α	1.901
[α]	156.47
5 α	1.901
[α]	156.51
6 α	1.903
[α]	156.68
7 α	1.902
[α]	156.55
8 α	1.902
[α]	156.55
9 α	1.904
[α]	156.72
10 α	1.903
[α]	156.70

MEAN VALUE N=10	
$\bar{\alpha}$	1.903 deg
σn-1	0.002 deg
RSD	0.105 %
[α]	156.62 deg
σn-1	0.2072 deg
TEMP	27

[α] = 156.57

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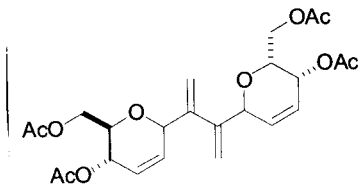
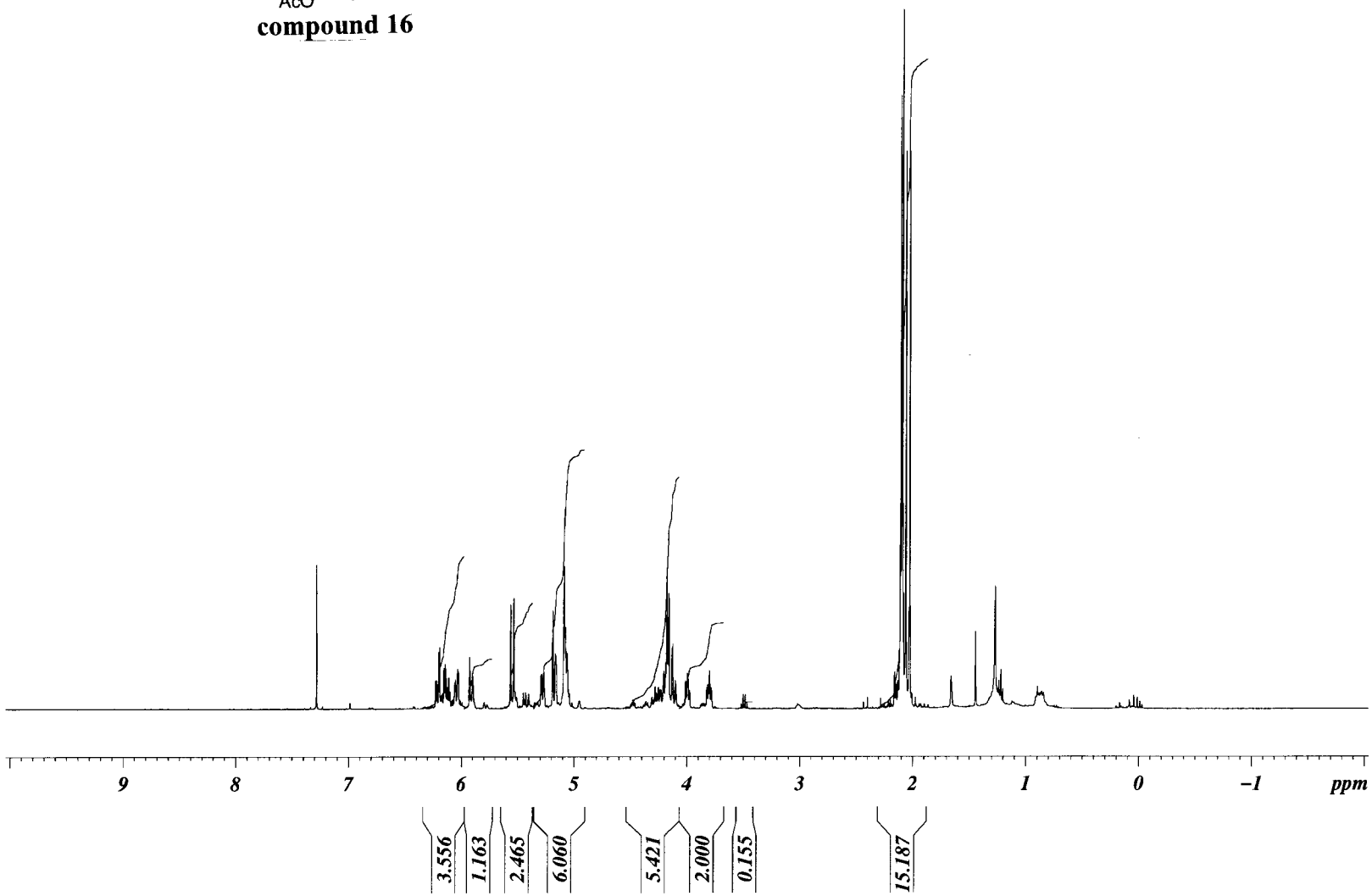
RNS74



compound 16

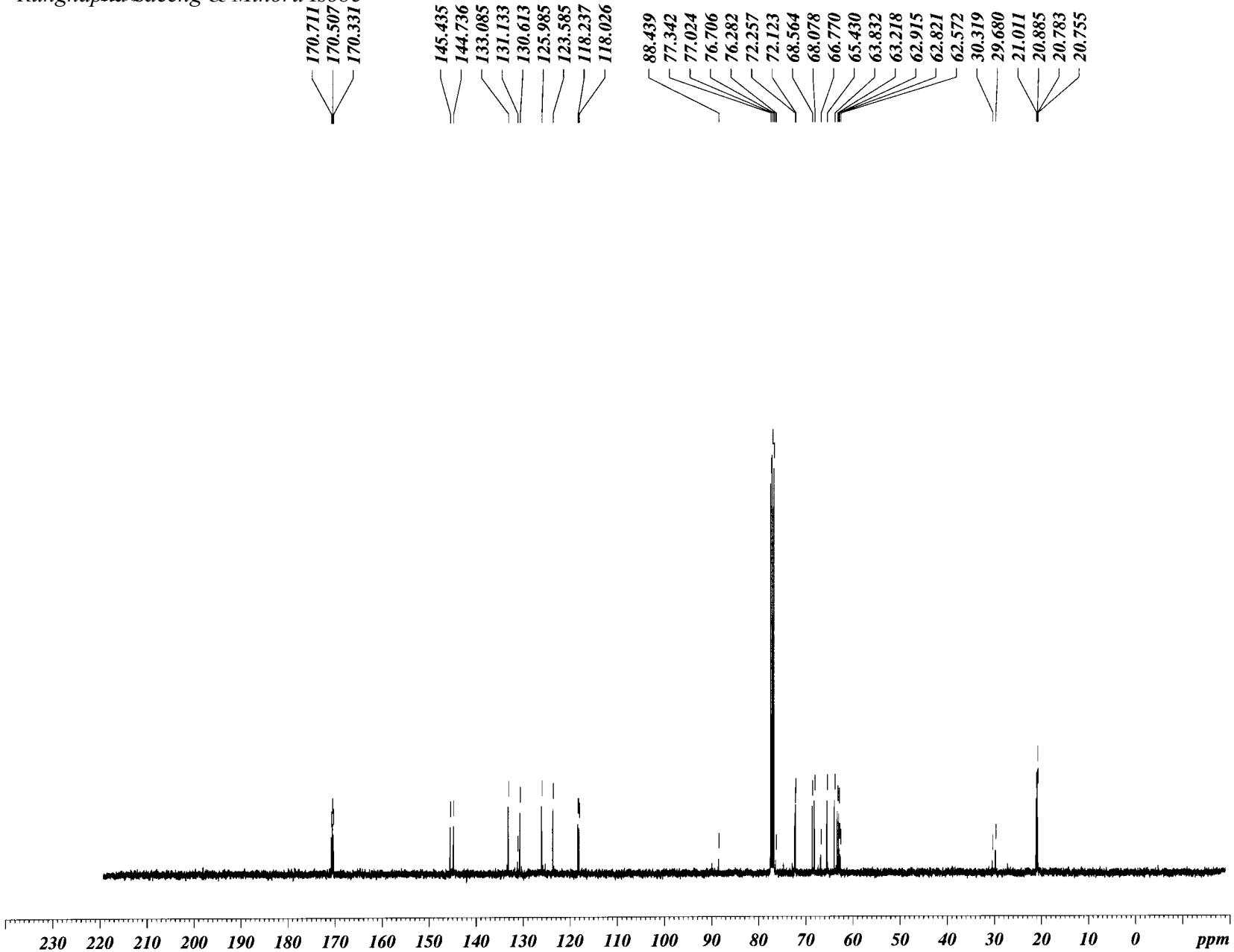


Current Data Parameters
NAME Rungnaphaav400
EXPNO 31
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040426
Time 11.05
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 −4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



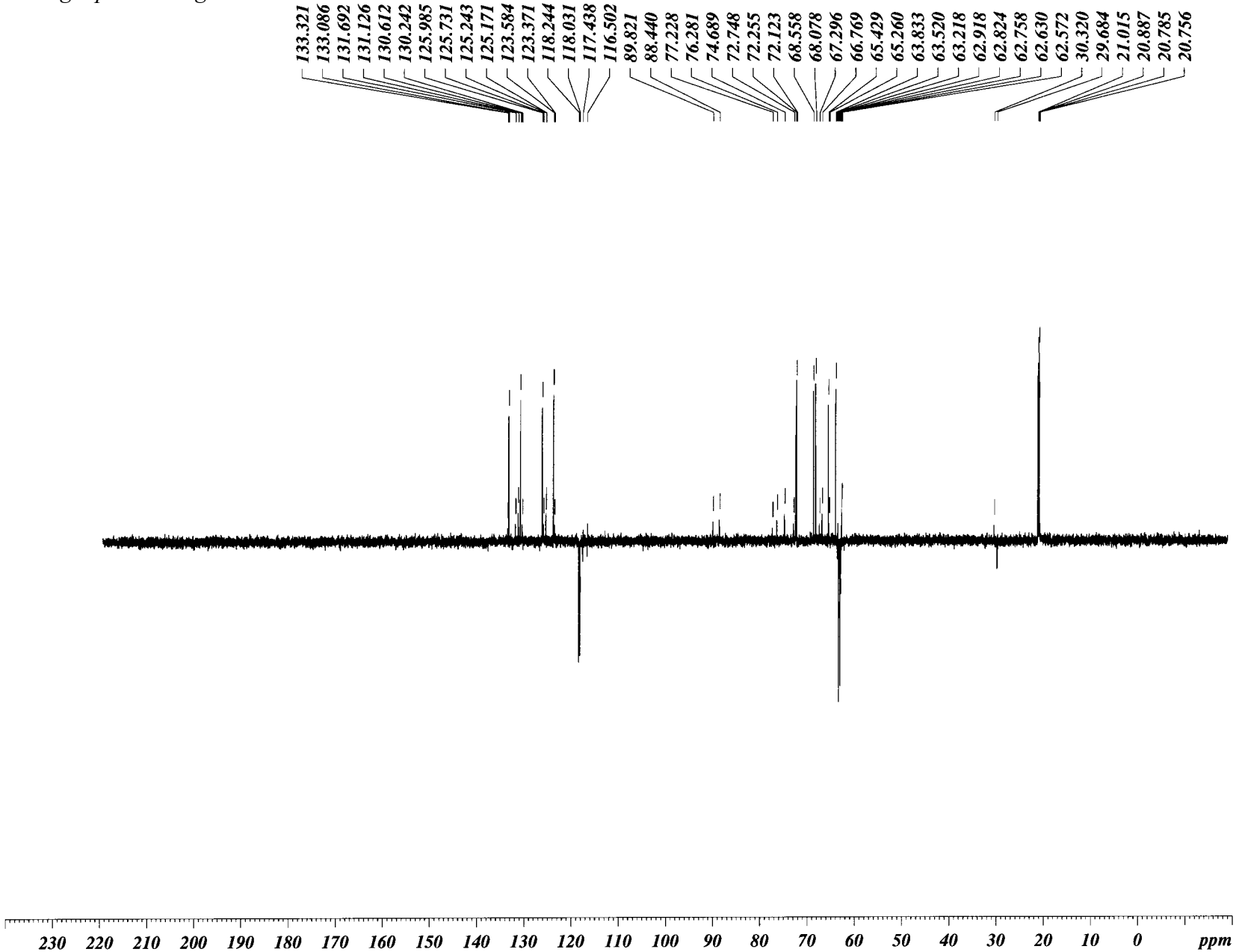
Current Data Parameters
NAME Rungnaphaav400
EXPNO 32
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040426
Time 11.21
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 364
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4597.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



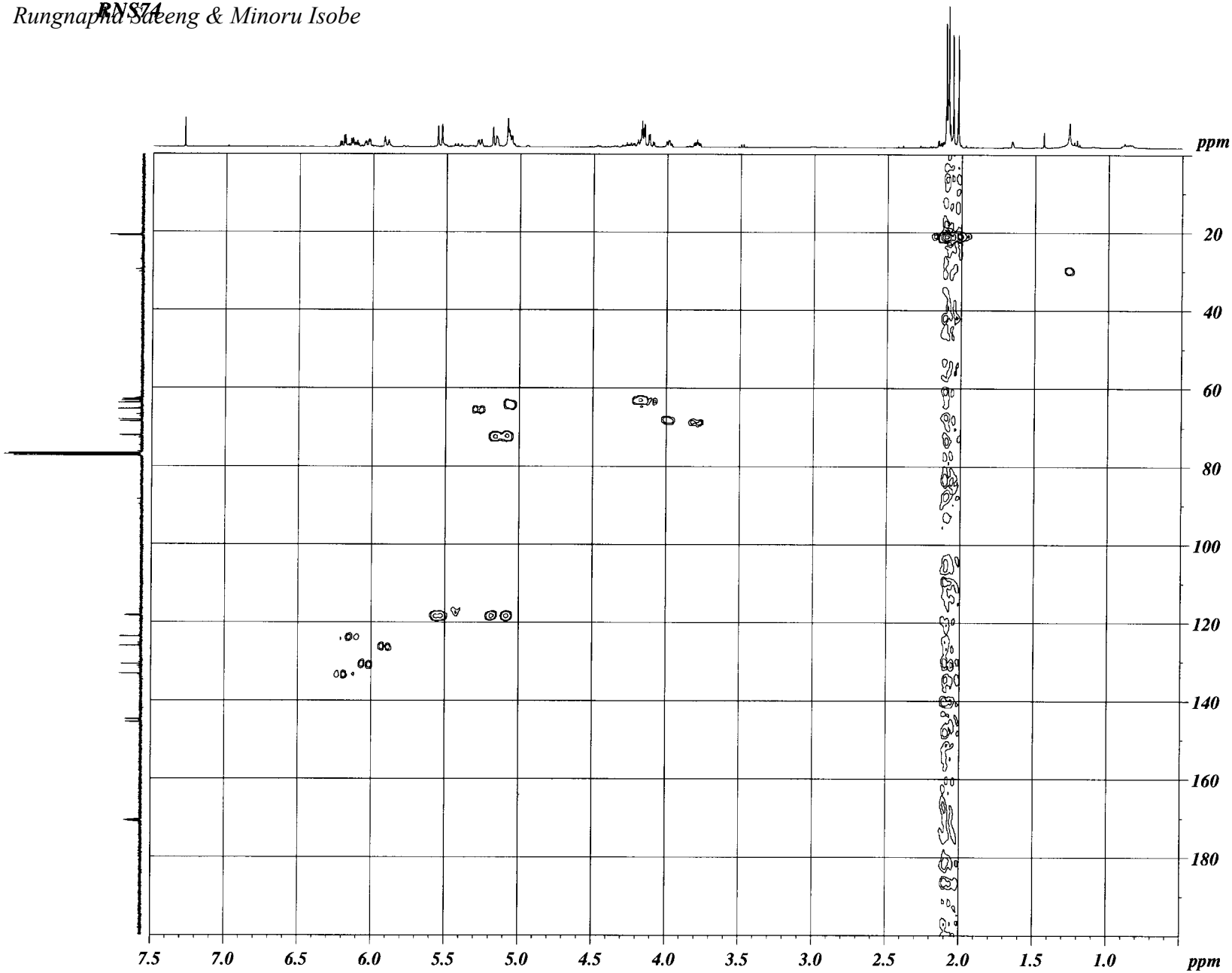
Current Data Parameters
NAME Rungnaphaav400
EXPNO 34
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040426
Time 13.02
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001146 sec

======== CHANNEL f1 ===
NUC1 13C
P1 9.00 usec
p2 18.00 usec
PL1 –3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
P3 8.60 usec
p4 17.20 usec
PCPD2 80.00 usec
PL2 –4.00 dB
PL12 14.80 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



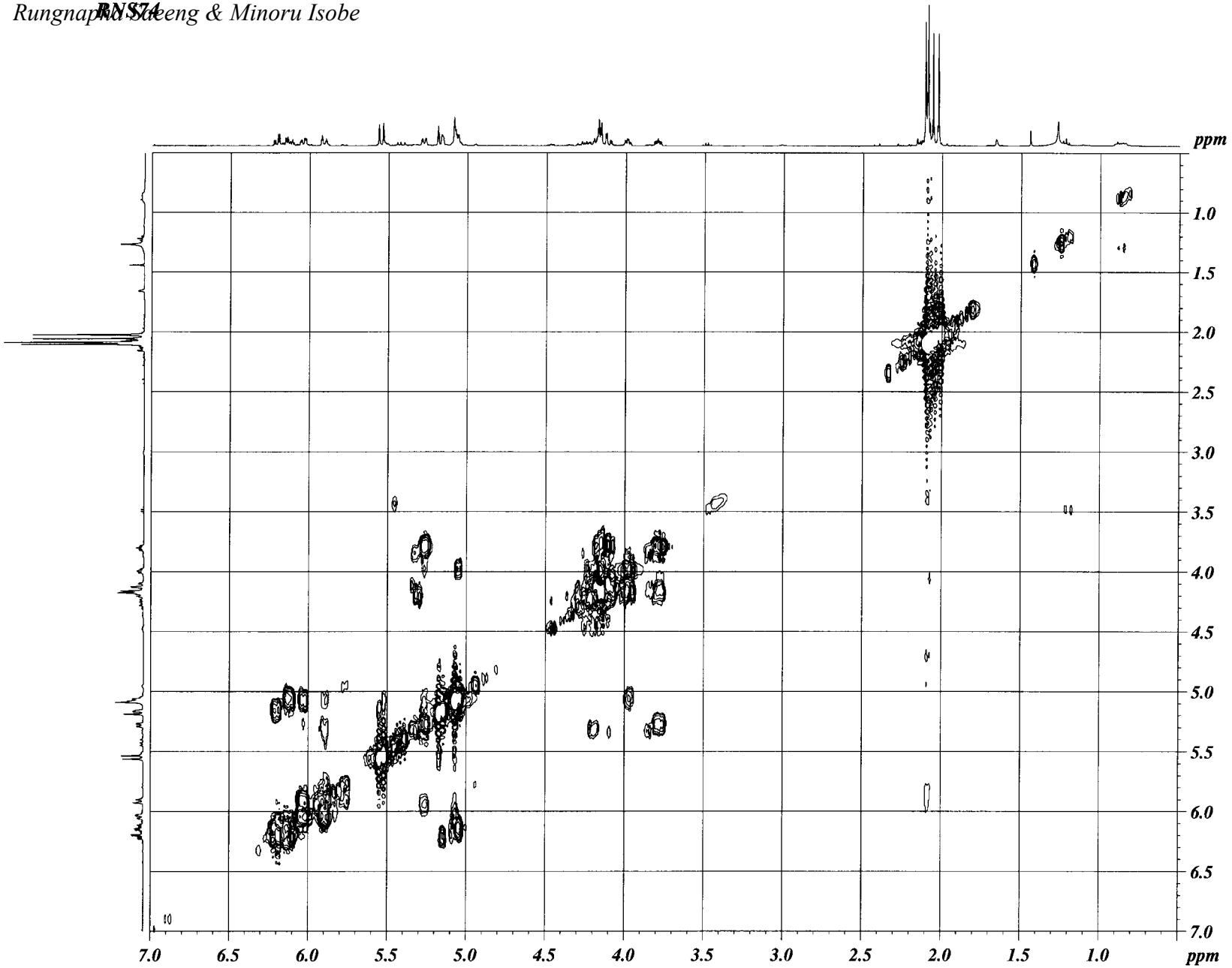
Current Data Parameters
NAME Rungnaphaav400
EXPNO 31
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040426
Time 11.05
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

COSY



Current Data Parameters
NAME Rungnaphaav400
EXPNO 31
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040426
Time 11.05
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 8.60 usec
PL1 –4.00 dB
SFO1 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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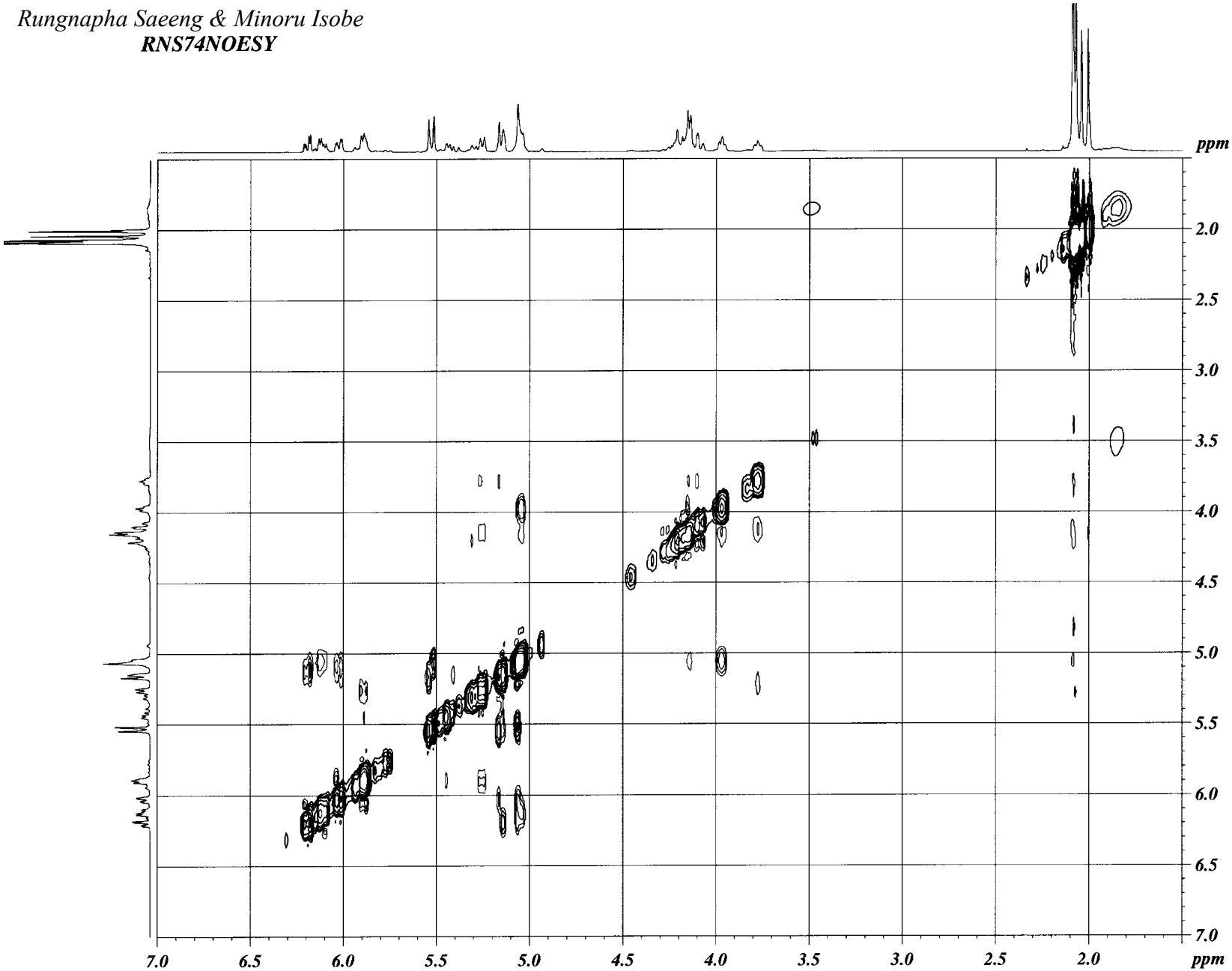
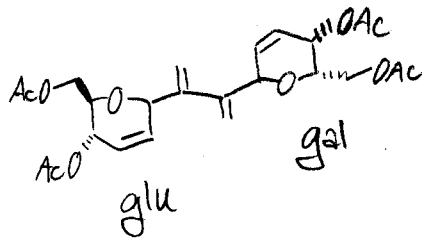
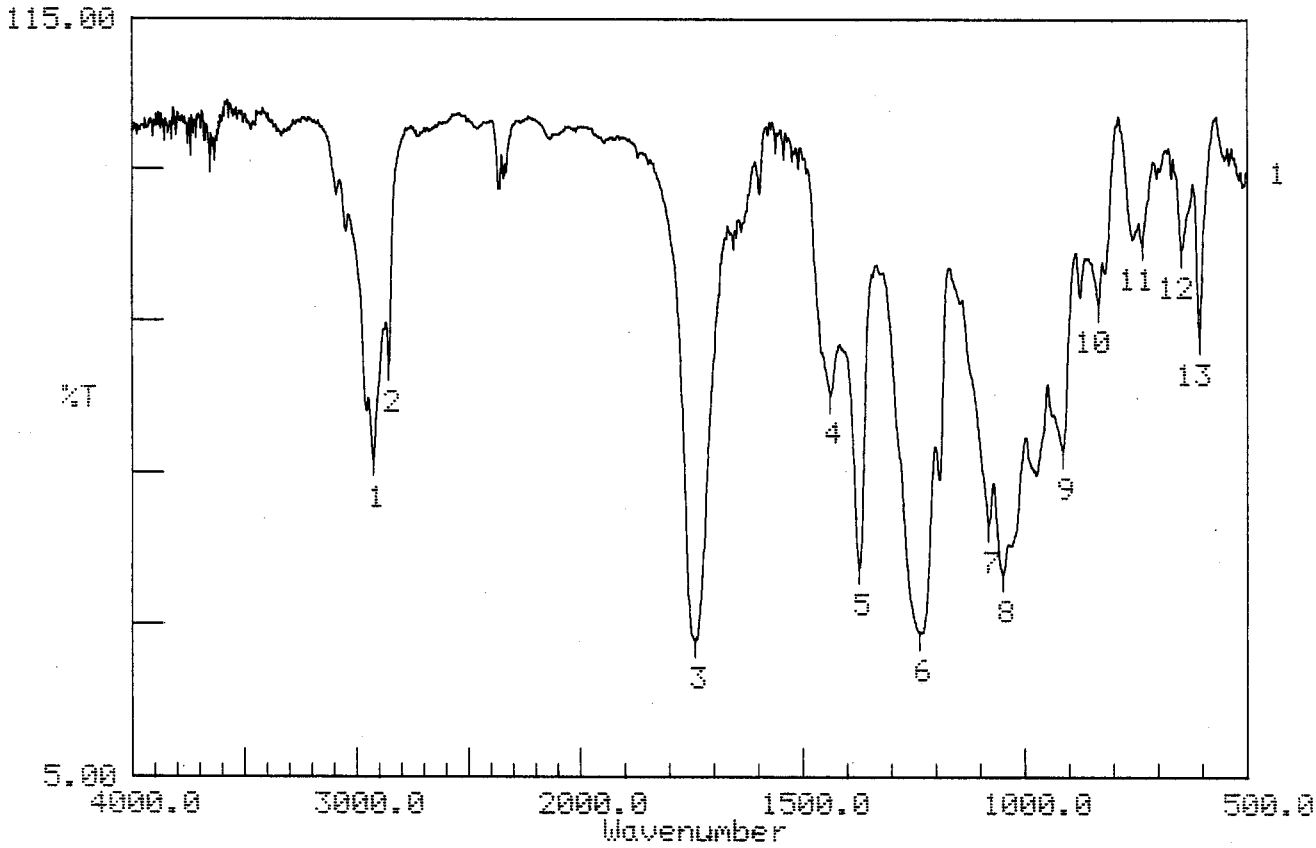
RNS74NOESY





JASCO FT/IR-8300
Date : 99/04/01 14:39
File Name :
Sample Name: RNS-74-1
Resolution : 4
Scans : 16
Gain : 20
Apodization: CS



Condition
upper 115.00 lower 5.00 depth 6.00

Peak table

1:	2924.44(51.0)	2:	2854.03(64.7)	3:	1740.97(24.6)
4:	1437.14(60.3)	5:	1371.55(35.3)	6:	1236.52(25.7)
7:	1080.27(41.5)	8:	1048.44(34.3)	9:	913.41(52.3)
10:	833.35(73.5)	11:	734.97(82.5)	12:	647.20(81.3)
13:	605.72(68.7)				

File: RNS-74
Sample: Description
Instrument: JEOL LCmate

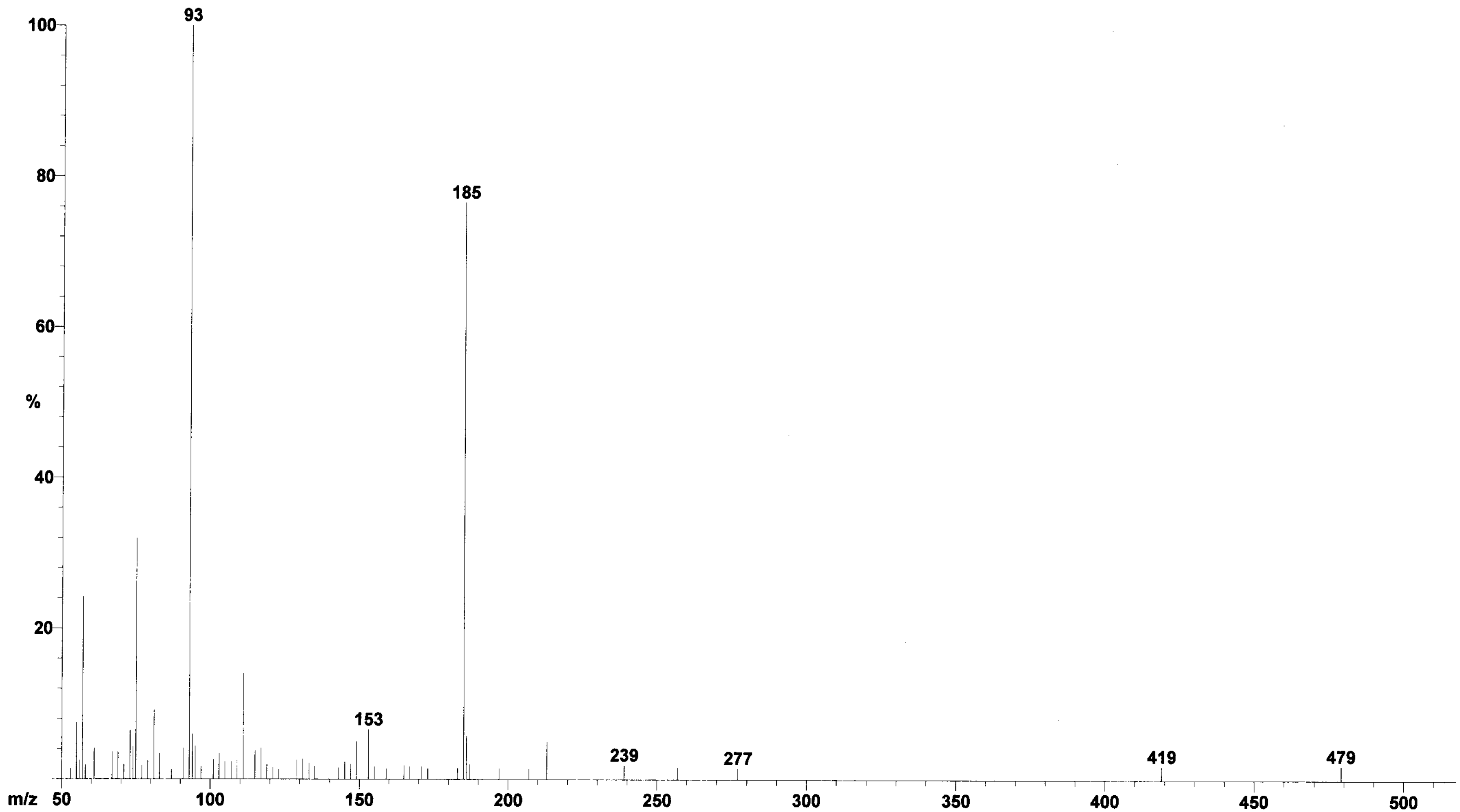
Date Run: 2004-05-31 (Time Run: 20:25:00)

Ionization mode: FAB+

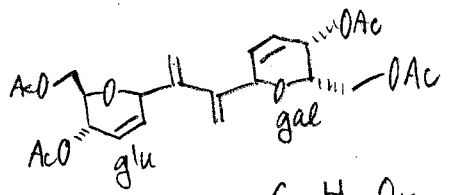
Scan: 2
Base: m/z 93; 4.3%FS TIC: 185296

R.T.: .2

#Ions: 60



Elemental Analysis Report



$C_{24}H_{30}O_{10}$

Refference No. : 368
Laboratory : 生物有機化学
Name : Rungnapha Saeeng
Sample Name : RNS-96-2
Sample Weight : 2559 micrograms

	N	C	H
Calculated weight %	0.00	60.24	6.32
Found weight %	0.00	60.25	6.51
Element ratio(1)	0	7	9
Element ratio(2)	0	45	58
Element ratio(3)	0	52	67
Element ratio(4)	0	59	76
Element ratio(5)	0	66	85
Element ratio(6)	0	73	94
Element ratio(7)	0	80	103

Remarks :

Date : 2004/06/09 15:54:48 Operator : 繁.Kitamura(789-4169)

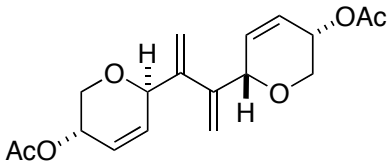
DATE 04.06.04
LAMP Na589
TEMP
SAMPLE RJS-26-2
CONC. 2.01%
CELL 100mm
INTEG.TIME 20sec
****
SAMPLE NO. deg
017
1 α -2.026
[α]-100.84
2 α -2.029
[α]-100.97
3 α -2.027
[α]-100.87
4 α -2.026
[α]-100.83
5 α -2.033
[α]-101.18
6 α -2.032
[α]-101.13
7 α -2.030
[α]-101.00
8 α -2.031
[α]-101.09
9 α -2.033
[α]-101.14
10 α -2.030
[α]-101.03

MEAN VALUE N=10
$\bar{\alpha}$ -2.030 deg
σn-1 0.002 deg
RSD 0.098 %

[α]-101.01 deg
σn-1 0.1294 deg
TEMP 28

[α]= -101.03

Compound 15



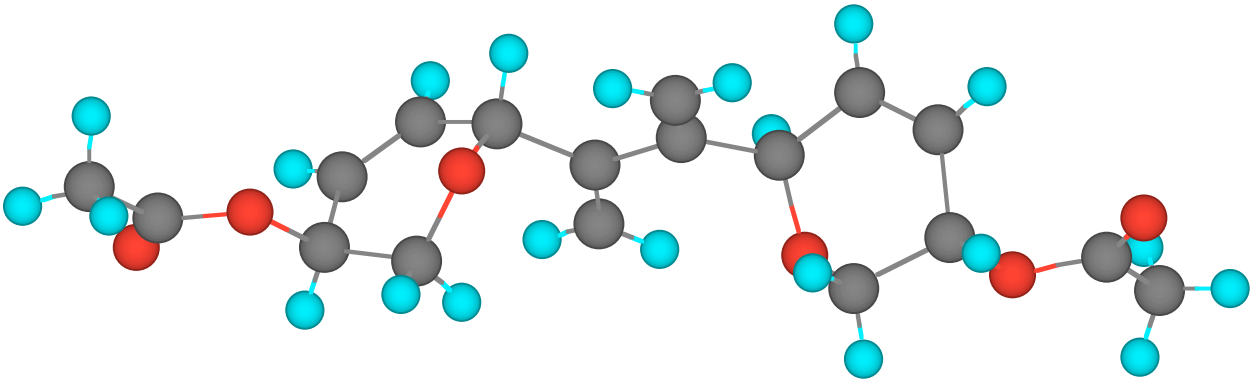


Table 1. Crystal data and structure refinement for isobe2.

Identification code	isobe2	
Empirical formula	C18 H22 O6	
Formula weight	334.36	
Temperature	90 K	
Wavelength	1.54178 ≈	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 9.3507(5) ≈	α= 90∞.
	b = 5.4653(4) ≈	β= 99.110(3)∞.
	c = 16.7583(7) ≈	γ = 90∞.
Volume	845.62(8) ≈^{3}	
Z	2	
Density (calculated)	1.313 Mg/m^3	
Absorption coefficient	0.818 mm^{-1}	
F(000)	356	

Crystal size	0.12 x 0.09 x 0.03 mm^3
Theta range for data collection	2.67 to 68.18°.
Index ranges	-11<=h<=11, -6<=k<=6, -19<=l<=20
Reflections collected	11469
Independent reflections	2606 [R(int) = 0.0276]
Completeness to theta = 68.18°	98.9 %
Absorption correction	Empirical
Max. and min. transmission	0.9759 and 0.9083
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2606 / 1 / 220
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0283, wR2 = 0.0731
R indices (all data)	R1 = 0.0307, wR2 = 0.0770
Absolute structure parameter	-0.08(14)
Extinction coefficient	0.0026(5)
Largest diff. peak and hole	0.187 and -0.130 $e.\AA^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for isobe2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	955(2)	5912(4)	3281(1)	26(1)
C(2)	1016(2)	4057(3)	3944(1)	19(1)
C(3)	2604(2)	4728(3)	6050(1)	20(1)
C(4)	1971(2)	3214(3)	5324(1)	19(1)
C(5)	2897(2)	1024(3)	5241(1)	20(1)
C(6)	4212(2)	815(3)	5670(1)	20(1)
C(7)	4903(2)	2787(3)	6222(1)	17(1)
C(8)	5120(2)	2106(3)	7120(1)	18(1)
C(9)	4343(2)	321(3)	7383(1)	24(1)
C(10)	6232(2)	3502(3)	7672(1)	18(1)
C(11)	6773(2)	5620(3)	7462(1)	22(1)
C(12)	6773(2)	2325(3)	8494(1)	19(1)
C(13)	8236(2)	3249(4)	8885(1)	23(1)
C(14)	8465(2)	4360(4)	9594(1)	23(1)
C(15)	7230(2)	4871(3)	10042(1)	21(1)
C(16)	5806(2)	4725(3)	9474(1)	22(1)
C(17)	8175(2)	3131(3)	11326(1)	21(1)
C(18)	8079(2)	981(4)	11874(1)	25(1)
O(1)	380(1)	2124(2)	3896(1)	27(1)
O(2)	1880(1)	4831(2)	4617(1)	19(1)
O(3)	4124(1)	5058(2)	6089(1)	18(1)
O(4)	5711(1)	2494(2)	9024(1)	21(1)
O(5)	7168(1)	2984(2)	10658(1)	21(1)
O(6)	9028(1)	4785(3)	11452(1)	33(1)

Table 3. Bond lengths [≈] and angles [∞] for isobe2.

C(1)-C(2)	1.498(2)
C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800
C(1)-H(1C)	0.9800
C(2)-O(1)	1.209(2)
C(2)-O(2)	1.3466(19)
C(3)-O(3)	1.4237(19)
C(3)-C(4)	1.513(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(2)	1.4702(18)
C(4)-C(5)	1.497(2)
C(4)-H(4)	1.0000
C(5)-C(6)	1.328(2)
C(5)-H(5)	0.9500
C(6)-C(7)	1.499(2)
C(6)-H(6)	0.9500
C(7)-O(3)	1.4385(19)
C(7)-C(8)	1.533(2)
C(7)-H(7)	1.0000
C(8)-C(9)	1.333(2)
C(8)-C(10)	1.487(2)
C(9)-H(9A)	0.9500
C(9)-H(9B)	0.9500
C(10)-C(11)	1.332(2)
C(10)-C(12)	1.532(2)
C(11)-H(11A)	0.9500
C(11)-H(11B)	0.9500
C(12)-O(4)	1.4358(19)
C(12)-C(13)	1.507(2)
C(12)-H(12)	1.0000

C(13)-C(14)	1.322(2)
C(13)-H(13)	0.9500
C(14)-C(15)	1.500(2)
C(14)-H(14)	0.9500
C(15)-O(5)	1.4669(19)
C(15)-C(16)	1.511(2)
C(15)-H(15)	1.0000
C(16)-O(4)	1.429(2)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(6)	1.201(2)
C(17)-O(5)	1.3464(18)
C(17)-C(18)	1.502(2)
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800

C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
O(1)-C(2)-O(2)	123.63(15)
O(1)-C(2)-C(1)	125.54(14)
O(2)-C(2)-C(1)	110.83(14)
O(3)-C(3)-C(4)	111.53(12)
O(3)-C(3)-H(3A)	109.3
C(4)-C(3)-H(3A)	109.3
O(3)-C(3)-H(3B)	109.3
C(4)-C(3)-H(3B)	109.3
H(3A)-C(3)-H(3B)	108.0
O(2)-C(4)-C(5)	111.48(12)

O(2)-C(4)-C(3)	106.56(13)
C(5)-C(4)-C(3)	110.71(13)
O(2)-C(4)-H(4)	109.3
C(5)-C(4)-H(4)	109.3
C(3)-C(4)-H(4)	109.3
C(6)-C(5)-C(4)	121.22(15)
C(6)-C(5)-H(5)	119.4
C(4)-C(5)-H(5)	119.4
C(5)-C(6)-C(7)	122.97(15)
C(5)-C(6)-H(6)	118.5
C(7)-C(6)-H(6)	118.5
O(3)-C(7)-C(6)	111.53(12)
O(3)-C(7)-C(8)	110.30(13)
C(6)-C(7)-C(8)	114.15(14)
O(3)-C(7)-H(7)	106.8
C(6)-C(7)-H(7)	106.8
C(8)-C(7)-H(7)	106.8
C(9)-C(8)-C(10)	122.27(13)
C(9)-C(8)-C(7)	120.56(14)
C(10)-C(8)-C(7)	117.17(14)
C(8)-C(9)-H(9A)	120.0
C(8)-C(9)-H(9B)	120.0
H(9A)-C(9)-H(9B)	120.0
C(11)-C(10)-C(8)	122.61(14)
C(11)-C(10)-C(12)	121.11(15)
C(8)-C(10)-C(12)	116.24(14)
C(10)-C(11)-H(11A)	120.0
C(10)-C(11)-H(11B)	120.0
H(11A)-C(11)-H(11B)	120.0
O(4)-C(12)-C(13)	112.24(12)
O(4)-C(12)-C(10)	111.54(12)
C(13)-C(12)-C(10)	113.46(14)
O(4)-C(12)-H(12)	106.3

C(13)-C(12)-H(12)	106.3
C(10)-C(12)-H(12)	106.3
C(14)-C(13)-C(12)	123.47(15)
C(14)-C(13)-H(13)	118.3
C(12)-C(13)-H(13)	118.3
C(13)-C(14)-C(15)	120.53(15)
C(13)-C(14)-H(14)	119.7
C(15)-C(14)-H(14)	119.7
O(5)-C(15)-C(14)	109.90(14)
O(5)-C(15)-C(16)	106.13(13)
C(14)-C(15)-C(16)	110.21(12)
O(5)-C(15)-H(15)	110.2
C(14)-C(15)-H(15)	110.2
C(16)-C(15)-H(15)	110.2
O(4)-C(16)-C(15)	110.66(13)
O(4)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16A)	109.5
O(4)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	108.1
O(6)-C(17)-O(5)	123.20(16)
O(6)-C(17)-C(18)	125.74(14)
O(5)-C(17)-C(18)	111.06(14)
C(17)-C(18)-H(18A)	109.5
C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(2)-O(2)-C(4)	115.88(13)
C(3)-O(3)-C(7)	112.03(12)
C(16)-O(4)-C(12)	112.94(12)
C(17)-O(5)-C(15)	116.31(13)

Symmetry transformations used to generate equivalent atoms:

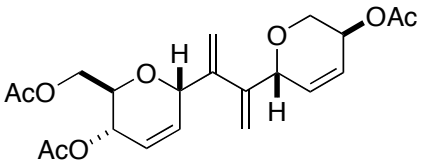
Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for isobe2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	32(1)	27(1)	19(1)	1(1)	0(1)	-5(1)
C(2)	17(1)	22(1)	17(1)	-1(1)	1(1)	-1(1)
C(3)	22(1)	21(1)	16(1)	-3(1)	2(1)	4(1)
C(4)	24(1)	19(1)	14(1)	1(1)	2(1)	-2(1)
C(5)	33(1)	14(1)	14(1)	-3(1)	4(1)	-2(1)
C(6)	32(1)	14(1)	15(1)	1(1)	5(1)	4(1)
C(7)	21(1)	15(1)	16(1)	2(1)	4(1)	4(1)
C(8)	22(1)	16(1)	16(1)	0(1)	2(1)	3(1)
C(9)	34(1)	21(1)	16(1)	2(1)	0(1)	-4(1)
C(10)	20(1)	17(1)	17(1)	-2(1)	4(1)	3(1)
C(11)	24(1)	19(1)	21(1)	-1(1)	1(1)	0(1)
C(12)	23(1)	19(1)	15(1)	0(1)	3(1)	3(1)
C(13)	20(1)	30(1)	21(1)	3(1)	3(1)	4(1)
C(14)	19(1)	29(1)	21(1)	4(1)	-1(1)	-3(1)
C(15)	27(1)	19(1)	15(1)	0(1)	1(1)	-2(1)
C(16)	24(1)	24(1)	17(1)	-2(1)	3(1)	4(1)
C(17)	23(1)	24(1)	16(1)	-1(1)	1(1)	-1(1)
C(18)	29(1)	24(1)	20(1)	3(1)	3(1)	0(1)
O(1)	28(1)	27(1)	23(1)	0(1)	-2(1)	-10(1)
O(2)	22(1)	18(1)	15(1)	1(1)	-1(1)	-3(1)
O(3)	22(1)	13(1)	20(1)	1(1)	0(1)	2(1)
O(4)	23(1)	24(1)	16(1)	-2(1)	3(1)	-3(1)
O(5)	26(1)	22(1)	16(1)	3(1)	-1(1)	-5(1)
O(6)	39(1)	34(1)	22(1)	4(1)	-6(1)	-16(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for isobe2.

	x	y	z	U(eq)
H(1A)	1863	5872	3058	39
H(1B)	818	7545	3498	39
H(1C)	144	5531	2853	39
H(3A)	2124	6347	6020	24
H(3B)	2413	3906	6549	24
H(4)	975	2663	5388	23
H(5)	2537	-241	4874	24
H(6)	4740	-651	5625	24
H(7)	5888	3076	6079	21
H(9A)	4490	-97	7941	29
H(9B)	3642	-532	7014	29
H(11A)	6449	6295	6942	26
H(11B)	7482	6455	7832	26
H(12)	6896	542	8389	23
H(13)	9035	3020	8607	28
H(14)	9419	4839	9822	28
H(15)	7346	6525	10300	25
H(16A)	4996	4815	9788	26
H(16B)	5722	6132	9097	26
H(18A)	8420	-494	11629	37
H(18B)	7071	750	11951	37
H(18C)	8684	1282	12397	37

Compound 17



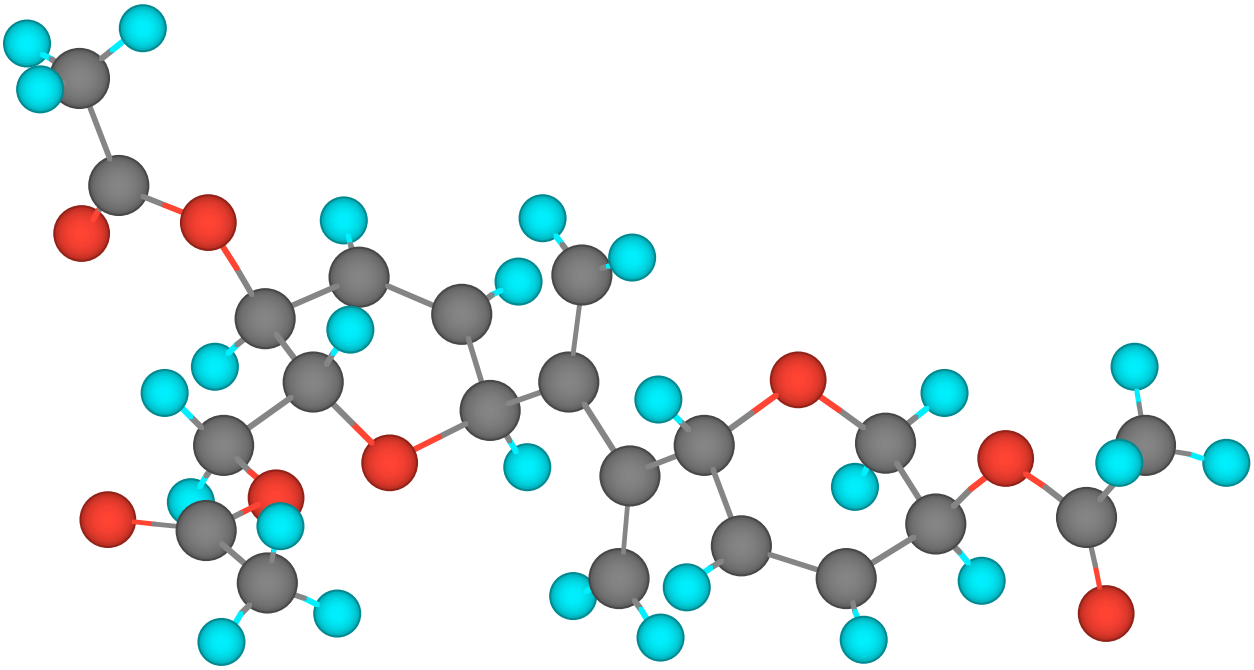


Table 1. Crystal data and structure refinement for isobe3.

Identification code	isobe3	
Empirical formula	C21 H26 O8	
Formula weight	406.42	
Temperature	90 K	
Wavelength	1.54178 ≈	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 13.6210(6) ≈	α= 90∞.
	b = 5.0388(2) ≈	β= 107.222(3)∞.
	c = 15.6367(9) ≈	γ = 90∞.
Volume	1025.08(8) ≈^{3}	
Z	2	

Density (calculated)	1.317 Mg/m^3
Absorption coefficient	0.846 mm^{-1}
F(000)	432
Crystal size	0.32 x 0.10 x 0.02 mm^3
Theta range for data collection	2.96 to 67.39∞.
Index ranges	-16<=h<=16, -5<=k<=5, -18<=l<=18
Reflections collected	8155
Independent reflections	3143 [R(int) = 0.0243]
Completeness to theta = 67.39∞	97.8 %
Absorption correction	Empirical
Max. and min. transmission	0.9833 and 0.7735
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3143 / 1 / 265
Goodness-of-fit on F^2	1.075
Final R indices [I>2sigma(I)]	R1 = 0.0341, wR2 = 0.0925
R indices (all data)	R1 = 0.0350, wR2 = 0.0933
Absolute structure parameter	0.07(15)
Largest diff. peak and hole	0.175 and -0.162 $e.\approx^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for isobe3. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8523(2)	4679(5)	-3029(1)	29(1)
C(2)	8788(1)	2611(4)	-2304(1)	20(1)
C(4)	7323(1)	-54(4)	-908(1)	21(1)
C(5)	8323(1)	719(4)	-1083(1)	20(1)
C(6)	9019(1)	2134(4)	-291(1)	22(1)
C(7)	8691(1)	2954(4)	381(1)	22(1)
C(8)	7589(1)	2685(4)	379(1)	19(1)
C(9)	7445(1)	772(4)	1089(1)	18(1)
C(10)	8198(1)	-865(4)	1516(1)	25(1)
C(11)	6432(1)	848(4)	1270(1)	16(1)
C(12)	5665(1)	2432(4)	821(1)	20(1)
C(13)	6311(1)	-941(4)	2019(1)	17(1)
C(14)	5213(1)	-1480(4)	1975(1)	19(1)
C(15)	4822(1)	-720(4)	2613(1)	21(1)
C(16)	5418(1)	944(4)	3393(1)	18(1)
C(17)	6348(1)	2176(4)	3181(1)	16(1)
C(18)	7082(1)	3440(4)	3993(1)	19(1)
C(19)	8454(1)	6511(4)	4277(1)	20(1)
C(20)	9270(1)	7711(5)	3933(1)	31(1)
C(21)	4193(1)	2709(4)	4059(1)	16(1)
C(22)	3433(1)	4890(4)	3988(1)	19(1)
O(1)	9541(1)	1215(3)	-2130(1)	26(1)
O(2)	8081(1)	2494(3)	-1855(1)	22(1)
O(3)	6930(1)	2051(3)	-499(1)	21(1)
O(4)	6883(1)	110(3)	2883(1)	17(1)
O(5)	7892(1)	4698(3)	3710(1)	20(1)
O(6)	8313(1)	7098(3)	4978(1)	29(1)
O(7)	4760(1)	3112(3)	3492(1)	19(1)

O(8)	4290(1)	801(3)	4542(1)	20(1)

Table 3. Bond lengths [≈] and angles [∞] for isobe3.

C(1)-C(2)	1.503(3)
C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800
C(1)-H(1C)	0.9800
C(2)-O(1)	1.206(2)
C(2)-O(2)	1.350(2)
C(4)-O(3)	1.423(2)
C(4)-C(5)	1.517(2)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(2)	1.460(2)
C(5)-C(6)	1.499(2)
C(5)-H(5)	1.0000
C(6)-C(7)	1.323(3)
C(6)-H(6)	0.9500
C(7)-C(8)	1.507(2)
C(7)-H(7)	0.9500
C(8)-O(3)	1.436(2)
C(8)-C(9)	1.526(3)
C(8)-H(8)	1.0000
C(9)-C(10)	1.332(3)
C(9)-C(11)	1.489(2)
C(10)-H(10A)	0.9500
C(10)-H(10B)	0.9500
C(11)-C(12)	1.337(3)
C(11)-C(13)	1.525(2)
C(12)-H(12A)	0.9500
C(12)-H(12B)	0.9500
C(13)-O(4)	1.446(2)
C(13)-C(14)	1.501(2)
C(13)-H(13)	1.0000

C(14)-C(15)	1.320(3)
C(14)-H(14)	0.9500
C(15)-C(16)	1.504(2)
C(15)-H(15)	0.9500
C(16)-O(7)	1.451(2)
C(16)-C(17)	1.531(2)
C(16)-H(16)	1.0000
C(17)-O(4)	1.426(2)
C(17)-C(18)	1.506(2)
C(17)-H(17)	1.0000
C(18)-O(5)	1.451(2)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-O(6)	1.205(2)
C(19)-O(5)	1.344(2)
C(19)-C(20)	1.498(3)
C(20)-H(20A)	0.9800
C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800
C(21)-O(8)	1.205(2)
C(21)-O(7)	1.353(2)
C(21)-C(22)	1.491(2)
C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800

C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
O(1)-C(2)-O(2)	123.10(17)

O(1)-C(2)-C(1)	124.99(17)
O(2)-C(2)-C(1)	111.92(15)
O(3)-C(4)-C(5)	111.53(16)
O(3)-C(4)-H(4A)	109.3
C(5)-C(4)-H(4A)	109.3
O(3)-C(4)-H(4B)	109.3
C(5)-C(4)-H(4B)	109.3
H(4A)-C(4)-H(4B)	108.0
O(2)-C(5)-C(6)	108.95(16)
O(2)-C(5)-C(4)	108.20(14)
C(6)-C(5)-C(4)	110.81(14)
O(2)-C(5)-H(5)	109.6
C(6)-C(5)-H(5)	109.6
C(4)-C(5)-H(5)	109.6
C(7)-C(6)-C(5)	121.63(16)
C(7)-C(6)-H(6)	119.2
C(5)-C(6)-H(6)	119.2
C(6)-C(7)-C(8)	122.76(16)
C(6)-C(7)-H(7)	118.6
C(8)-C(7)-H(7)	118.6
O(3)-C(8)-C(7)	111.22(14)
O(3)-C(8)-C(9)	112.10(14)
C(7)-C(8)-C(9)	113.47(15)
O(3)-C(8)-H(8)	106.5
C(7)-C(8)-H(8)	106.5
C(9)-C(8)-H(8)	106.5
C(10)-C(9)-C(11)	122.72(16)
C(10)-C(9)-C(8)	120.65(16)
C(11)-C(9)-C(8)	116.63(14)
C(9)-C(10)-H(10A)	120.0
C(9)-C(10)-H(10B)	120.0
H(10A)-C(10)-H(10B)	120.0
C(12)-C(11)-C(9)	122.63(16)

C(12)-C(11)-C(13)	120.74(16)
C(9)-C(11)-C(13)	116.61(14)
C(11)-C(12)-H(12A)	120.0
C(11)-C(12)-H(12B)	120.0
H(12A)-C(12)-H(12B)	120.0
O(4)-C(13)-C(14)	111.30(14)
O(4)-C(13)-C(11)	110.50(14)
C(14)-C(13)-C(11)	113.83(14)
O(4)-C(13)-H(13)	106.9
C(14)-C(13)-H(13)	106.9
C(11)-C(13)-H(13)	106.9
C(15)-C(14)-C(13)	122.14(17)
C(15)-C(14)-H(14)	118.9
C(13)-C(14)-H(14)	118.9
C(14)-C(15)-C(16)	122.21(16)
C(14)-C(15)-H(15)	118.9
C(16)-C(15)-H(15)	118.9
O(7)-C(16)-C(15)	107.65(14)
O(7)-C(16)-C(17)	107.14(15)
C(15)-C(16)-C(17)	109.45(14)
O(7)-C(16)-H(16)	110.8
C(15)-C(16)-H(16)	110.8
C(17)-C(16)-H(16)	110.8
O(4)-C(17)-C(18)	107.94(14)
O(4)-C(17)-C(16)	108.06(14)
C(18)-C(17)-C(16)	111.95(14)
O(4)-C(17)-H(17)	109.6
C(18)-C(17)-H(17)	109.6
C(16)-C(17)-H(17)	109.6
O(5)-C(18)-C(17)	107.57(13)
O(5)-C(18)-H(18A)	110.2
C(17)-C(18)-H(18A)	110.2
O(5)-C(18)-H(18B)	110.2

C(17)-C(18)-H(18B)	110.2
H(18A)-C(18)-H(18B)	108.5
O(6)-C(19)-O(5)	123.73(17)
O(6)-C(19)-C(20)	124.22(18)
O(5)-C(19)-C(20)	112.05(16)
C(19)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
O(8)-C(21)-O(7)	123.52(16)
O(8)-C(21)-C(22)	125.44(15)
O(7)-C(21)-C(22)	111.02(15)
C(21)-C(22)-H(22A)	109.5
C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(2)-O(2)-C(5)	115.86(14)
C(4)-O(3)-C(8)	112.60(13)
C(17)-O(4)-C(13)	112.74(12)
C(19)-O(5)-C(18)	115.53(13)
C(21)-O(7)-C(16)	116.62(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for isobe3. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

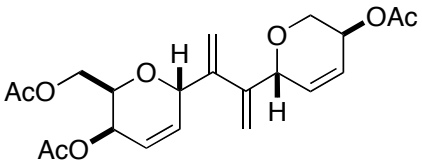
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	26(1)	34(1)	29(1)	9(1)	9(1)	-1(1)
C(2)	17(1)	24(1)	18(1)	-1(1)	4(1)	-3(1)
C(4)	20(1)	27(1)	15(1)	-1(1)	5(1)	-3(1)
C(5)	20(1)	23(1)	17(1)	3(1)	5(1)	1(1)
C(6)	16(1)	28(1)	21(1)	4(1)	5(1)	-1(1)
C(7)	18(1)	26(1)	22(1)	-1(1)	5(1)	-6(1)
C(8)	18(1)	21(1)	18(1)	0(1)	6(1)	1(1)
C(9)	17(1)	20(1)	15(1)	-1(1)	4(1)	1(1)
C(10)	21(1)	32(1)	23(1)	6(1)	8(1)	6(1)
C(11)	18(1)	17(1)	14(1)	-2(1)	5(1)	1(1)
C(12)	20(1)	20(1)	19(1)	1(1)	7(1)	3(1)
C(13)	21(1)	14(1)	17(1)	-1(1)	6(1)	1(1)
C(14)	21(1)	14(1)	23(1)	1(1)	5(1)	-2(1)
C(15)	21(1)	16(1)	28(1)	2(1)	10(1)	-3(1)
C(16)	20(1)	16(1)	22(1)	3(1)	10(1)	3(1)
C(17)	19(1)	16(1)	16(1)	-1(1)	8(1)	1(1)
C(18)	21(1)	20(1)	18(1)	-1(1)	9(1)	-5(1)
C(19)	19(1)	21(1)	18(1)	1(1)	2(1)	0(1)
C(20)	26(1)	39(1)	27(1)	-2(1)	7(1)	-10(1)
C(21)	16(1)	16(1)	15(1)	-3(1)	4(1)	-2(1)
C(22)	19(1)	18(1)	22(1)	0(1)	7(1)	0(1)
O(1)	19(1)	35(1)	26(1)	5(1)	8(1)	4(1)
O(2)	20(1)	27(1)	19(1)	5(1)	8(1)	3(1)
O(3)	17(1)	29(1)	16(1)	3(1)	3(1)	3(1)
O(4)	18(1)	18(1)	16(1)	0(1)	5(1)	3(1)
O(5)	21(1)	22(1)	20(1)	-4(1)	9(1)	-4(1)
O(6)	31(1)	36(1)	20(1)	-8(1)	7(1)	-9(1)
O(7)	23(1)	16(1)	22(1)	5(1)	13(1)	4(1)

O(8)	22(1)	16(1)	23(1)	5(1)	10(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for isobe3.

	x	y	z	U(eq)
H(1A)	8937	6274	-2822	44
H(1B)	7792	5127	-3172	44
H(1C)	8667	3985	-3565	44
H(4A)	6810	-535	-1481	25
H(4B)	7440	-1632	-513	25
H(5)	8673	-906	-1215	24
H(6)	9713	2450	-270	26
H(7)	9170	3746	885	27
H(8)	7371	4474	533	23
H(10A)	8101	-2049	1956	30
H(10B)	8833	-849	1379	30
H(12A)	5755	3569	365	23
H(12B)	5030	2420	957	23
H(13)	6628	-2689	1952	20
H(14)	4785	-2398	1471	23
H(15)	4141	-1241	2579	25
H(16)	5641	-142	3953	22
H(17)	6108	3532	2697	20
H(18A)	6719	4775	4253	23
H(18B)	7376	2077	4454	23
H(20A)	9014	9367	3617	46
H(20B)	9449	6467	3521	46
H(20C)	9881	8083	4437	46
H(22A)	2935	4371	4299	29
H(22B)	3072	5229	3356	29
H(22C)	3793	6503	4263	29

Compound 18



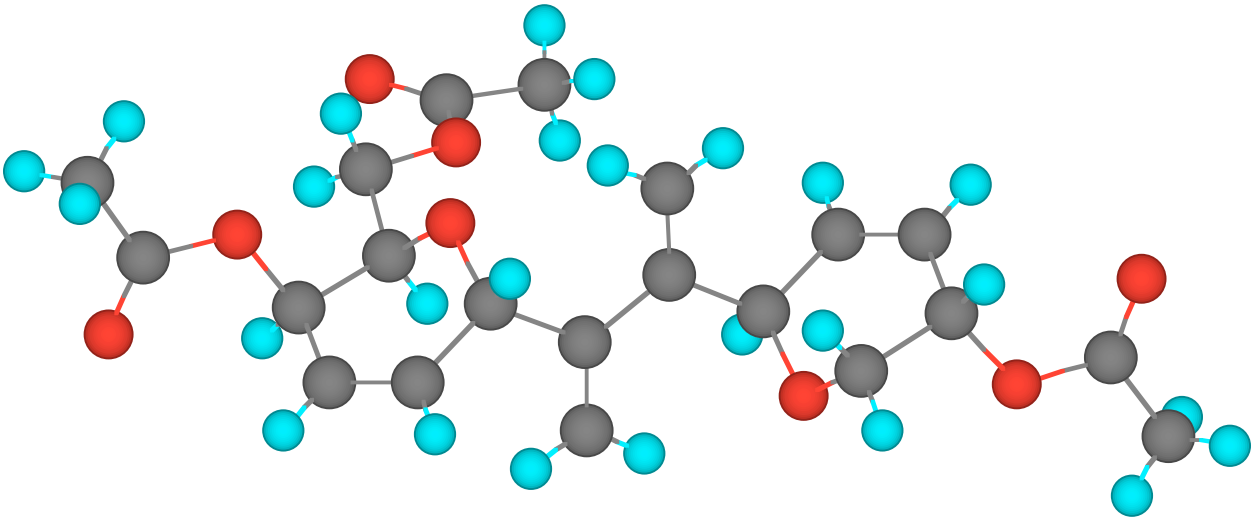


Table 1. Crystal data and structure refinement for isobe4.

Identification code	isobe4	
Empirical formula	C21 H26 O8	
Formula weight	406.42	
Temperature	90 K	
Wavelength	1.54178 ≈	
Crystal system	Monoclinic	
Space group	C2	
Unit cell dimensions	a = 21.213(3) ≈	α= 90∞.
	b = 4.7923(6) ≈	β= 117.332(6)∞.
	c = 22.216(4) ≈	γ = 90∞.
Volume	2006.3(5) ≈^{3}	
Z	4	
Density (calculated)	1.345 Mg/m^3	
Absorption coefficient	0.865 mm^{-1}	
F(000)	864	

Crystal size	0.09 x 0.06 x 0.02 mm^3
Theta range for data collection	4.17 to 67.39∞.
Index ranges	-25<=h<=23, -5<=k<=5, -24<=l<=26
Reflections collected	6276
Independent reflections	2581 [R(int) = 0.0762]
Completeness to theta = 67.39∞	96.6 %
Absorption correction	Empirical
Max. and min. transmission	0.9829 and 0.9262
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2581 / 1 / 266
Goodness-of-fit on F^2	1.373
Final R indices [I>2sigma(I)]	R1 = 0.0548, wR2 = 0.0955
R indices (all data)	R1 = 0.0871, wR2 = 0.1048
Absolute structure parameter	0.0(4)
Extinction coefficient	0.00059(12)
Largest diff. peak and hole	0.289 and -0.322 e.≈$^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for isobe4. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9276(3)	-32(10)	5158(2)	32(1)
C(2)	8844(3)	2026(10)	4625(2)	29(1)
C(3)	7055(3)	4399(9)	4159(2)	27(1)
C(4)	7681(3)	4045(11)	4013(2)	27(1)
C(5)	7451(3)	2831(9)	3323(2)	28(1)
C(6)	6815(3)	1700(11)	2977(2)	30(1)
C(7)	6292(2)	1459(10)	3256(2)	25(1)
C(8)	5615(2)	3150(9)	2869(2)	24(1)
C(9)	5579(3)	5113(10)	2425(2)	30(1)
C(10)	5021(3)	2500(9)	3012(2)	25(1)
C(11)	5050(3)	571(10)	3455(2)	29(1)
C(12)	4328(3)	4148(11)	2608(2)	27(1)
C(13)	3822(2)	4040(10)	2903(2)	28(1)
C(14)	3173(2)	3018(9)	2565(2)	28(1)
C(15)	2920(2)	1705(10)	1879(2)	26(1)
C(16)	3555(2)	1087(10)	1754(2)	26(1)
C(17)	3351(2)	188(10)	1043(2)	28(1)
C(18)	3894(3)	-2956(10)	586(2)	31(1)
C(19)	4597(3)	-4116(11)	682(2)	35(1)
C(20)	1781(2)	3817(11)	1254(2)	28(1)
C(21)	1373(3)	6049(10)	756(2)	33(1)
O(1)	9049(2)	3397(7)	4295(2)	35(1)
O(2)	8173(2)	2101(7)	4538(2)	29(1)
O(3)	6619(2)	1923(6)	3966(2)	27(1)
O(4)	4000(2)	3515(6)	1903(1)	25(1)
O(5)	3995(2)	-919(6)	1046(2)	29(1)
O(6)	3326(2)	-3718(7)	166(2)	36(1)
O(7)	2460(2)	3706(7)	1359(2)	26(1)

O(8)	1547(2)	2323(7)	1543(2)	35(1)

Table 3. Bond lengths [≈] and angles [∞] for isobe4.

C(1)-C(2)	1.489(6)
C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800
C(1)-H(1C)	0.9800
C(2)-O(1)	1.203(6)
C(2)-O(2)	1.347(6)
C(3)-O(3)	1.444(5)
C(3)-C(4)	1.515(6)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(2)	1.483(5)
C(4)-C(5)	1.495(6)
C(4)-H(4)	1.0000
C(5)-C(6)	1.324(7)
C(5)-H(5)	0.9500
C(6)-C(7)	1.503(6)
C(6)-H(6)	0.9500
C(7)-O(3)	1.421(5)
C(7)-C(8)	1.524(6)
C(7)-H(7)	1.0000
C(8)-C(9)	1.339(7)
C(8)-C(10)	1.468(6)
C(9)-H(9A)	0.9500
C(9)-H(9B)	0.9500
C(10)-C(11)	1.332(7)
C(10)-C(12)	1.542(6)
C(11)-H(11A)	0.9500
C(11)-H(11B)	0.9500
C(12)-O(4)	1.424(5)
C(12)-C(13)	1.493(6)
C(12)-H(12)	1.0000

C(13)-C(14)	1.324(6)
C(13)-H(13)	0.9500
C(14)-C(15)	1.500(6)
C(14)-H(14)	0.9500
C(15)-O(7)	1.473(5)
C(15)-C(16)	1.526(6)
C(15)-H(15)	1.0000
C(16)-O(4)	1.439(5)
C(16)-C(17)	1.497(6)
C(16)-H(16)	1.0000
C(17)-O(5)	1.462(5)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-O(6)	1.192(6)
C(18)-O(5)	1.357(5)
C(18)-C(19)	1.513(7)
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800
C(20)-O(8)	1.211(6)
C(20)-O(7)	1.350(5)
C(20)-C(21)	1.496(7)
C(21)-H(21A)	0.9800
C(21)-H(21B)	0.9800
C(21)-H(21C)	0.9800

C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
O(1)-C(2)-O(2)	123.5(4)

O(1)-C(2)-C(1)	125.6(5)
O(2)-C(2)-C(1)	110.8(4)
O(3)-C(3)-C(4)	110.5(4)
O(3)-C(3)-H(3A)	109.6
C(4)-C(3)-H(3A)	109.6
O(3)-C(3)-H(3B)	109.6
C(4)-C(3)-H(3B)	109.6
H(3A)-C(3)-H(3B)	108.1
O(2)-C(4)-C(5)	110.0(4)
O(2)-C(4)-C(3)	105.7(4)
C(5)-C(4)-C(3)	110.9(4)
O(2)-C(4)-H(4)	110.0
C(5)-C(4)-H(4)	110.0
C(3)-C(4)-H(4)	110.0
C(6)-C(5)-C(4)	121.4(5)
C(6)-C(5)-H(5)	119.3
C(4)-C(5)-H(5)	119.3
C(5)-C(6)-C(7)	122.7(5)
C(5)-C(6)-H(6)	118.6
C(7)-C(6)-H(6)	118.6
O(3)-C(7)-C(6)	111.7(4)
O(3)-C(7)-C(8)	113.4(4)
C(6)-C(7)-C(8)	114.1(4)
O(3)-C(7)-H(7)	105.6
C(6)-C(7)-H(7)	105.6
C(8)-C(7)-H(7)	105.6
C(9)-C(8)-C(10)	122.9(4)
C(9)-C(8)-C(7)	120.7(5)
C(10)-C(8)-C(7)	116.4(4)
C(8)-C(9)-H(9A)	120.0
C(8)-C(9)-H(9B)	120.0
H(9A)-C(9)-H(9B)	120.0
C(11)-C(10)-C(8)	123.7(4)

C(11)-C(10)-C(12)	119.6(5)
C(8)-C(10)-C(12)	116.7(4)
C(10)-C(11)-H(11A)	120.0
C(10)-C(11)-H(11B)	120.0
H(11A)-C(11)-H(11B)	120.0
O(4)-C(12)-C(13)	112.9(4)
O(4)-C(12)-C(10)	112.6(4)
C(13)-C(12)-C(10)	114.3(4)
O(4)-C(12)-H(12)	105.3
C(13)-C(12)-H(12)	105.3
C(10)-C(12)-H(12)	105.3
C(14)-C(13)-C(12)	122.3(4)
C(14)-C(13)-H(13)	118.9
C(12)-C(13)-H(13)	118.9
C(13)-C(14)-C(15)	121.7(4)
C(13)-C(14)-H(14)	119.1
C(15)-C(14)-H(14)	119.1
O(7)-C(15)-C(14)	108.8(4)
O(7)-C(15)-C(16)	107.7(4)
C(14)-C(15)-C(16)	109.4(4)
O(7)-C(15)-H(15)	110.3
C(14)-C(15)-H(15)	110.3
C(16)-C(15)-H(15)	110.3
O(4)-C(16)-C(17)	108.5(4)
O(4)-C(16)-C(15)	110.1(4)
C(17)-C(16)-C(15)	113.4(4)
O(4)-C(16)-H(16)	108.3
C(17)-C(16)-H(16)	108.3
C(15)-C(16)-H(16)	108.3
O(5)-C(17)-C(16)	106.4(3)
O(5)-C(17)-H(17A)	110.4
C(16)-C(17)-H(17A)	110.4
O(5)-C(17)-H(17B)	110.4

C(16)-C(17)-H(17B)	110.4
H(17A)-C(17)-H(17B)	108.6
O(6)-C(18)-O(5)	124.2(5)
O(6)-C(18)-C(19)	125.0(4)
O(5)-C(18)-C(19)	110.7(4)
C(18)-C(19)-H(19A)	109.5
C(18)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(18)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
O(8)-C(20)-O(7)	123.4(4)
O(8)-C(20)-C(21)	125.1(4)
O(7)-C(20)-C(21)	111.5(4)
C(20)-C(21)-H(21A)	109.5
C(20)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5
C(20)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5
C(2)-O(2)-C(4)	116.4(4)
C(7)-O(3)-C(3)	112.2(3)
C(12)-O(4)-C(16)	111.3(3)
C(18)-O(5)-C(17)	115.7(4)
C(20)-O(7)-C(15)	115.5(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for isobe4. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	24(3)	39(3)	33(3)	8(2)	13(2)	3(3)
C(2)	24(3)	34(2)	26(2)	-2(2)	8(2)	1(3)
C(3)	26(3)	25(2)	32(3)	-2(2)	15(2)	0(2)
C(4)	23(2)	23(2)	34(3)	6(2)	11(2)	6(3)
C(5)	27(3)	26(2)	37(3)	6(2)	19(2)	4(2)
C(6)	25(3)	29(3)	37(3)	5(2)	16(2)	7(3)
C(7)	22(2)	24(2)	30(3)	0(2)	11(2)	5(2)
C(8)	18(2)	19(2)	31(3)	-3(2)	8(2)	2(2)
C(9)	25(3)	30(2)	34(3)	2(2)	14(2)	-1(3)
C(10)	20(2)	27(2)	24(2)	-4(2)	5(2)	2(2)
C(11)	23(2)	30(3)	35(3)	2(2)	13(2)	-1(3)
C(12)	24(2)	27(2)	30(3)	2(2)	13(2)	4(2)
C(13)	24(2)	26(2)	32(3)	-1(2)	12(2)	8(3)
C(14)	23(2)	30(2)	29(3)	1(2)	12(2)	6(3)
C(15)	20(2)	25(2)	29(3)	4(2)	7(2)	2(2)
C(16)	20(2)	26(2)	33(3)	3(2)	12(2)	3(2)
C(17)	16(2)	31(2)	36(3)	0(2)	11(2)	-5(2)
C(18)	37(3)	27(2)	34(2)	-1(2)	21(2)	-1(3)
C(19)	30(3)	37(3)	43(3)	-3(2)	21(2)	3(3)
C(20)	24(2)	29(2)	30(3)	-5(2)	11(2)	1(3)
C(21)	27(3)	28(2)	40(3)	2(2)	12(2)	-1(3)
O(1)	24(2)	42(2)	41(2)	9(2)	16(2)	-1(2)
O(2)	20(2)	32(2)	35(2)	8(2)	13(2)	4(2)
O(3)	25(2)	24(2)	33(2)	2(1)	13(2)	-2(2)
O(4)	21(2)	26(2)	26(2)	1(1)	9(1)	-5(2)
O(5)	26(2)	28(2)	36(2)	-5(1)	17(2)	-3(2)
O(6)	28(2)	38(2)	38(2)	-7(2)	12(2)	1(2)
O(7)	18(2)	24(2)	36(2)	5(2)	12(1)	-1(2)

O(8)	29(2)	32(2)	49(2)	3(2)	22(2)	-1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for isobe4.

	x	y	z	U(eq)
H(1A)	9359	-1688	4945	48
H(1B)	9021	-572	5413	48
H(1C)	9732	810	5467	48
H(3A)	7229	4764	4648	33
H(3B)	6768	6023	3904	33
H(4)	7919	5884	4050	32
H(5)	7768	2871	3130	34
H(6)	6683	1014	2534	35
H(7)	6139	-539	3192	31
H(9A)	5154	6154	2186	36
H(9B)	5977	5463	2348	36
H(11A)	5473	-473	3700	35
H(11B)	4648	240	3527	35
H(12)	4476	6147	2642	32
H(13)	3972	4728	3349	34
H(14)	2859	3119	2761	33
H(15)	2653	-51	1850	31
H(16)	3838	-446	2067	31
H(17A)	2978	-1266	897	34
H(17B)	3168	1793	729	34
H(19A)	4715	-5787	967	53
H(19B)	4969	-2709	901	53
H(19C)	4563	-4603	240	53
H(21A)	900	5348	446	49
H(21B)	1624	6577	496	49
H(21C)	1328	7682	999	49