

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

Rungnapha Saeeng* and Minoru Isobe*

Department of Chemistry, Faculty of Science, Burapha University, Sangsook, Chonburi 20131, Thailand

*Laboratory of Organic Chemistry, Graduate School of Bioagricultural Sciences,
Nagoya University, Chikusa, Nagoya 464-8601, Japan*

rungnaph@buu.ac.th, isobem@agr.nagoya-u.ac.jp

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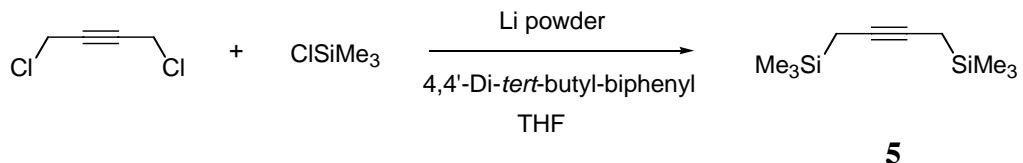
Experimental Section

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 chromatography (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 chromatography 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_3OEt_2 was distilled from CaH_2 . All other commercially available reagents were used as received.

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



To a green suspension of Li powder 1g (140 mmol) and DTBB (0.26 g, 1 mmol) in dry THF 30 ml. at -40°C 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. = 111-128°C, 0.01 Mpar) 1.49 g (75%)

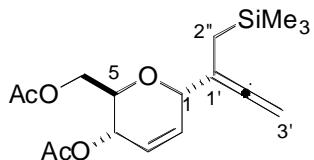
Preparation of the green suspension Li powder and DTBB

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-*t*-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 $^1\text{H-NMR}$ (400 MHz, CDCl_3) : δ 0.09 (18H, s, 2XSiMe_3), 1.45 (4H, s, 2XCH_2). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) : δ -2.0 ((CH_3)₃Si), 7.2 (CH_2), 75.6 ($\text{C}\equiv\text{C}$)

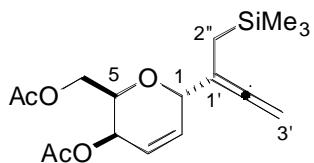
General procedure for the synthesis of alleneglycoside

To a solution of Glycal (0.36 mmol) and the 1,4-Bis(trimethylsilyl)-2-butyne (0.45 mmol) in anhydrous CH₂Cl₂ (35 ml) was added the solution of BF₃.OEt₂ (0.36 mmol) in anhydrous CH₂Cl₂ (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 CH₂Cl₂. 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 Et₂O:hexane) to afford alleneglycoside.



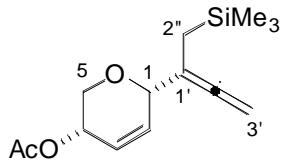
Compound 6. [α]_D²⁷ 156.1° (c = 1.3, CHCl₃). IR (KBr): 2954, 1951, 1743, 1370, 1239 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : δ 0.08 (9H, s, SiMe₃), 1.47 (2H, m, H-2''), 2.10 (3H, s, Ac), 2.11 (3H, s, Ac), 3.89 (1H, ddd, J = 8.5, 5.7, 3.0 Hz, H-5), 4.17 (1H, dd, J = 12.0, 3.0 Hz, H-6a), 4.24 (1H, dd, J = 12.0, 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). ¹³C-NMR (100 MHz, CDCl₃) : 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 C₁₇H₂₆O₅Si: C, 60.32; H, 7.74 Found: C, 60.31; H, 7.69



Compound 8. $[\alpha]_D^{28} -403.5^\circ$ ($c = 0.8$, CHCl_3). IR (KBr): 2955, 1950, 1747, 1371, 1230 cm^{-1} . $^1\text{H-NMR}$ (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). $^{13}\text{C-NMR}$ (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 $\text{C}_{17}\text{H}_{26}\text{O}_5\text{Si}$: C, 60.32; H, 7.74 Found: C, 60.44; H, 7.77

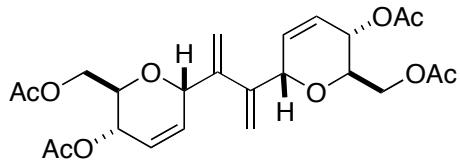


Compound 10. $[\alpha]_D^{26} +142.9^\circ$ ($c = 1.3$, CHCl_3). IR (KBr): 2954, 1950, 1740, 1371, 1244 cm^{-1} . $^1\text{H-NMR}$ (400 MHz, CDCl_3) : δ 0.08 (9H, s, SiMe_3), 1.40 (1H, dt, $J = 14.5, 2.5$ Hz, H-2'a), 1.48 (1H, dt, $J = 14.5, 2.5$ Hz, H-2'b), 2.11 (3H, s, Ac), 3.73 (1H dd, $J = 12.5, 3.0$ 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). $^{13}\text{C-NMR}$ (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.

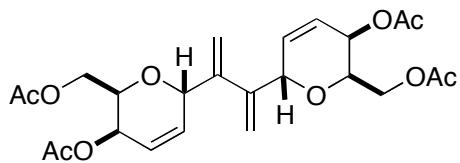
Anal. Calcd. For $\text{C}_{14}\text{H}_{22}\text{O}_3\text{Si}$: C, 63.12; H, 8.32 Found: C, 63.12; H, 8.32

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 CH₂Cl₂ (35 ml) was added the solution of SnCl₄ (0.37 mmol) in anhydrous CH₂Cl₂ (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 CH₂Cl₂. 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 Et₂O:hexane) to afford symmetric diene glycoside.

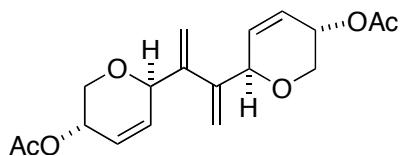


Compound 13. mp. = 125-126°C [α]_D²⁷ +23.4° (c = 0.8, CHCl₃). IR (KBr): 2952, 1743, 1235, 1048 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : δ 2.07 (6H, s, 2xAc), 2.09 (6H, s, 2xAc), 3.81 (2H, ddd, J = 8.5, 6.0, 3.0 Hz, 2xH-5), 4.11 (2H, dd, J = 12.5, 3.0 Hz, 2xH-6a), 4.19 (2H, dd, J = 12.0, 6.0 Hz, 2xH-6b), 5.07 (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). ¹³C-NMR (100 MHz, CDCl₃) : 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 C₂₄H₃₀O₁₀: C, 60.24; H, 6.32 Found: C, 60.25; H, 6.27



Compound 14. mp. = 157-158°C [α]_D²⁸ -349.04° (c = 0.8, CHCl₃). IR (KBr): 2927, 1739, 1371, 1227 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : δ 2.01 (6H, s, 2xAc), 2.10 (6H, s, 2xAc), 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). ¹³C-NMR (100 MHz, CDCl₃) : 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 C₂₄H₃₀O₁₀: C, 60.24; H, 6.32 Found: C, 60.24; H, 6.24

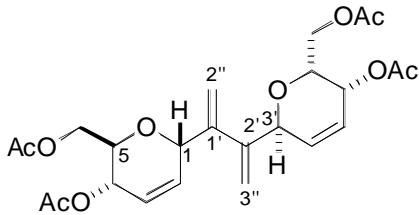


Compound 15 mp. = 87-88°C [α]_D²⁶ +290.94° (c = 1.0, CHCl₃). IR (KBr): 2861, 1733, 1372, 1237 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : δ 2.08 (6H, s, 2xAc), 3.70 (2H, ddd, J = 12.5, 3.0, 0.5 Hz, 2xH-5a), 3.92 (2H, dd, J = 12.5, 3.5 Hz, 2xH-5b), 5.02 (2H, brs, 2xH-1), 5.06 (2H, dd, J = 7.0, 3.0 Hz, 2xH-4), 5.15 (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). ¹³C-NMR (100 MHz, CDCl₃) : δ 21.1, 63.5, 64.5, 72.4, 117.7, 123.9, 133.1, 144.0, 170.6.

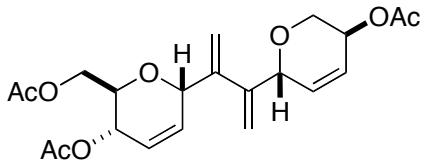
Anal. Calcd. For C₁₈H₂₂O₆: C, 64.66; H, 6.63 Found: C, 64.66; H, 6.64

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

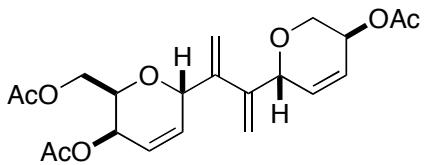
To a solution of Glycal (0.37 mmol) and the 1,4-Bis(trimethylsilyl)-2-butyne (0.37 mmol) in anhydrous CH₂Cl₂ (35 ml) was added the solution of BF₃OEt₂ (0.37 mmol) in anhydrous CH₂Cl₂ (5 ml) at -20°C. After TLC showed the complete conversion, another glycal (0.37 mmol) was added to the reaction mixture followed by the solution of SnCl₄ (0.37 mmol) in anhydrous CH₂Cl₂ (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 (1 vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH₂Cl₂. 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 Et₂O:hexane) to afford asymmetric diene glycoside.



Compound 16. [α]_D²⁸ -101.0° (c = 2.0, CHCl₃). IR (KBr): 2924, 1740, 1371, 1236, 1048 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : 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'). ¹³C-NMR (100 MHz, CDCl₃) : 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 C₂₄H₃₀O₁₀: C, 60.24; H, 6.32 Found: C, 60.25; H, 6.51

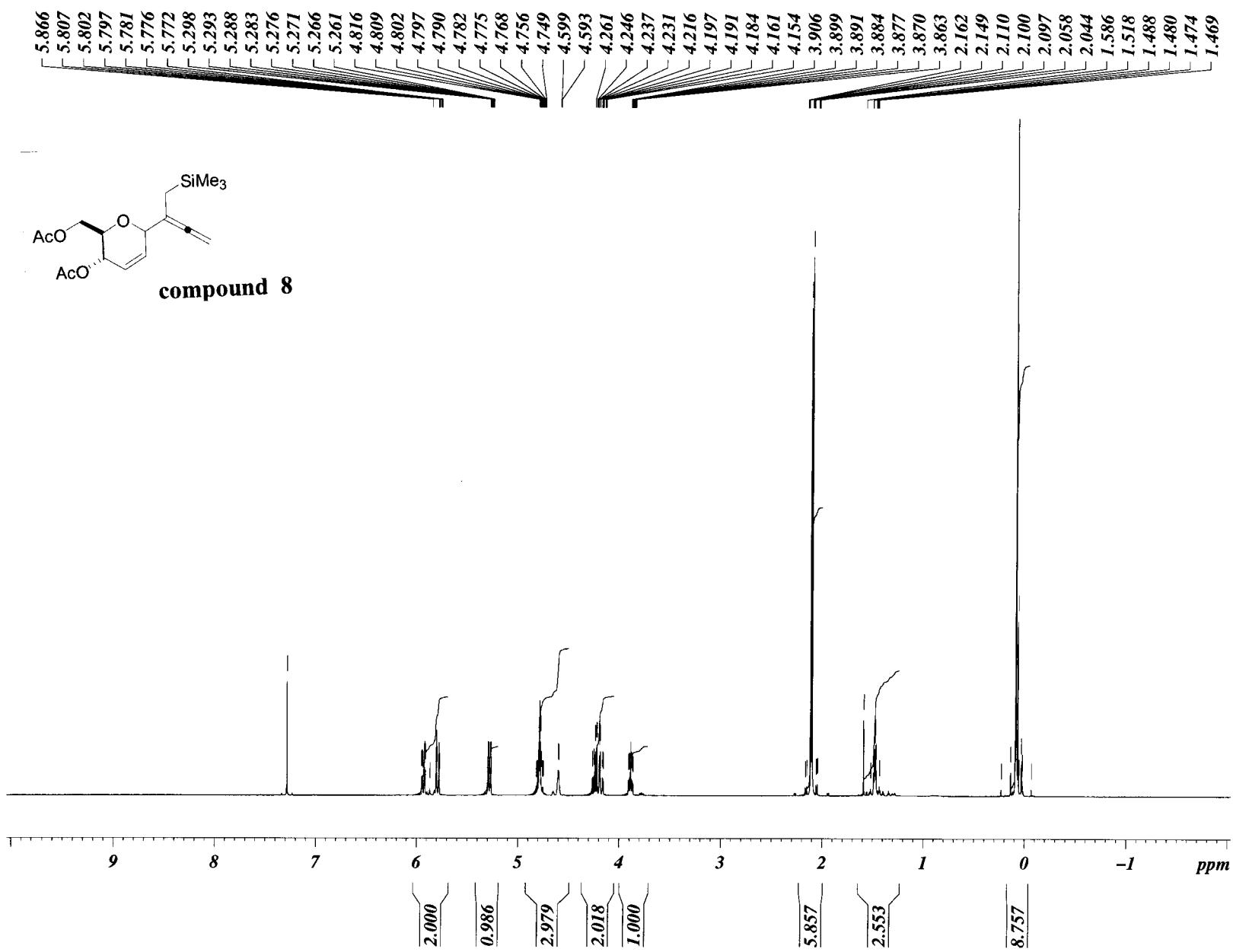


Compound 17 mp. = 77-78°C [α]_D²⁷ +156.6° (c = 1.2, CHCl₃). IR (KBr): 2936, 1743, 1371, 1236, 1057 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : δ 2.07 (3H, s, Ac), 2.09 (3H, s, Ac), 2.10 (3H, s, Ac), 3.71 (1H, dd, J = 12.0, 4.0 Hz, H-7'a), 3.85 (1H, ddd, J = 8.0, 6.0, 3.0 Hz, H-5), 4.01 (1H, dd, J = 12.0, 4.0 Hz, 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'). ¹³C-NMR (100 MHz, CDCl₃) : δ 20.8, 21.0, 21.1, 63.1, 63.9, 64.6, 65.4, 68.7, Anal. Calcd. For C₂₁H₂₆O₈: C, 62.06; H, 6.45 Found: C, 62.06; H, 6.47



Compound 18 mp. = 78-80°C [α]_D²⁷ -47.2° (c = 1.2, CHCl₃). IR (KBr): 2936, 1736, 1371, 1236, 1049 cm⁻¹, ¹H-NMR (400 MHz, CDCl₃) : 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). ¹³C-NMR (100 MHz, CDCl₃) : 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 C₂₁H₂₆O₈: C, 62.06; H, 6.45 Found: C, 62.04; H, 6.39

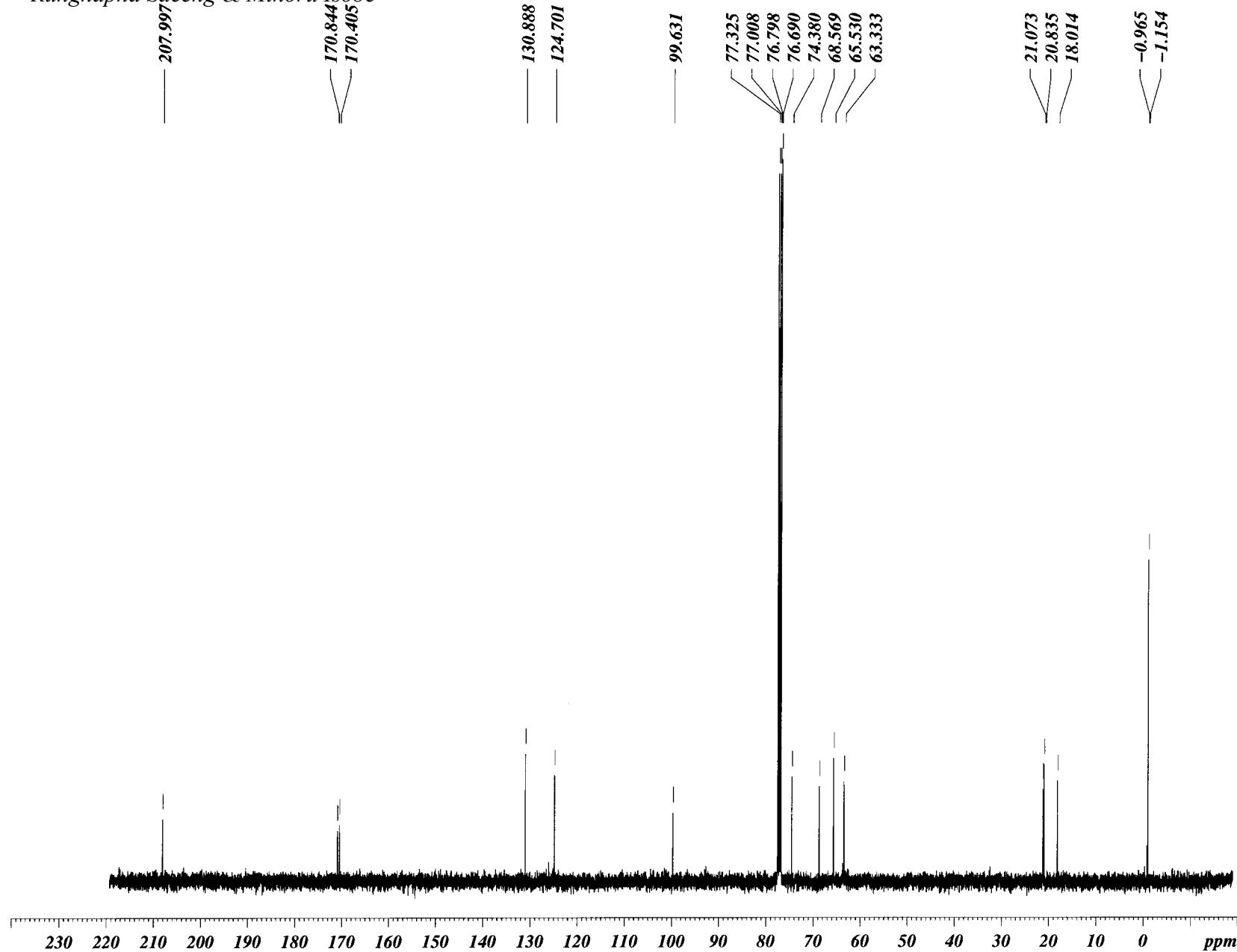


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DS 2
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FIDRES 0.126314 Hz
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DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

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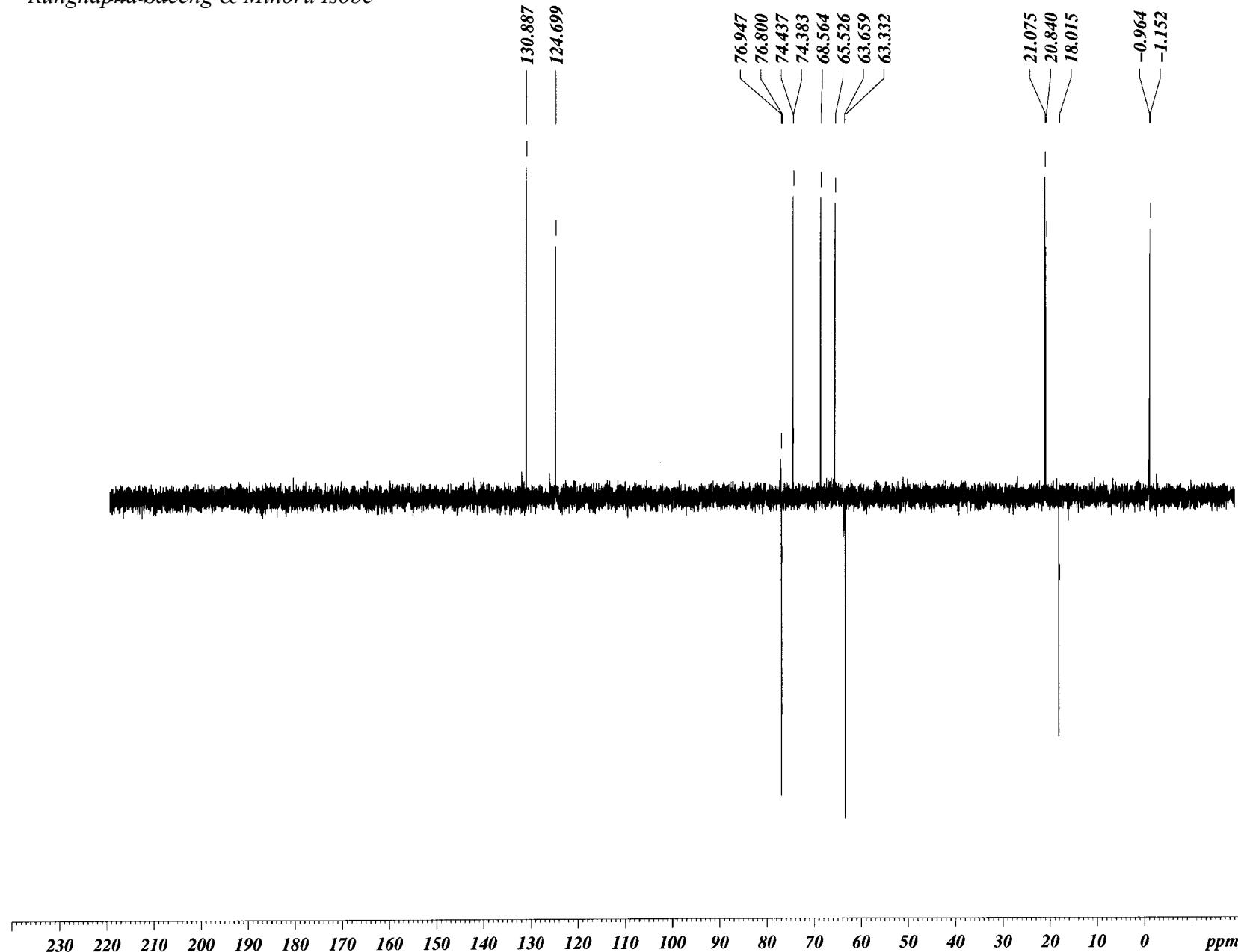
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d11 0.03000000 sec
d12 0.00002000 sec

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PL2 -4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
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SSB 0
LB 1.00 Hz
GB 0
PC 1.00



Current Data Parameters
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 EXPNO 30
 PROCNO 1

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 FIDRES 0.365918 Hz
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 RG 16384
 DW 20.850 usec
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 D1 2.00000000 sec
 d2 0.00344828 sec
 d12 0.00002000 sec
 DELTA 0.00001146 sec

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 p2 18.00 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

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 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 Rungnaphaeng400
 EXPNO 27
 PROCNO 1

F2 - Acquisition Parameters
 Date 20040421
 Time 11.16

INSTRUM av400
 PROBHD 5 mm QNP IH/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
 DI 1.0000000 sec

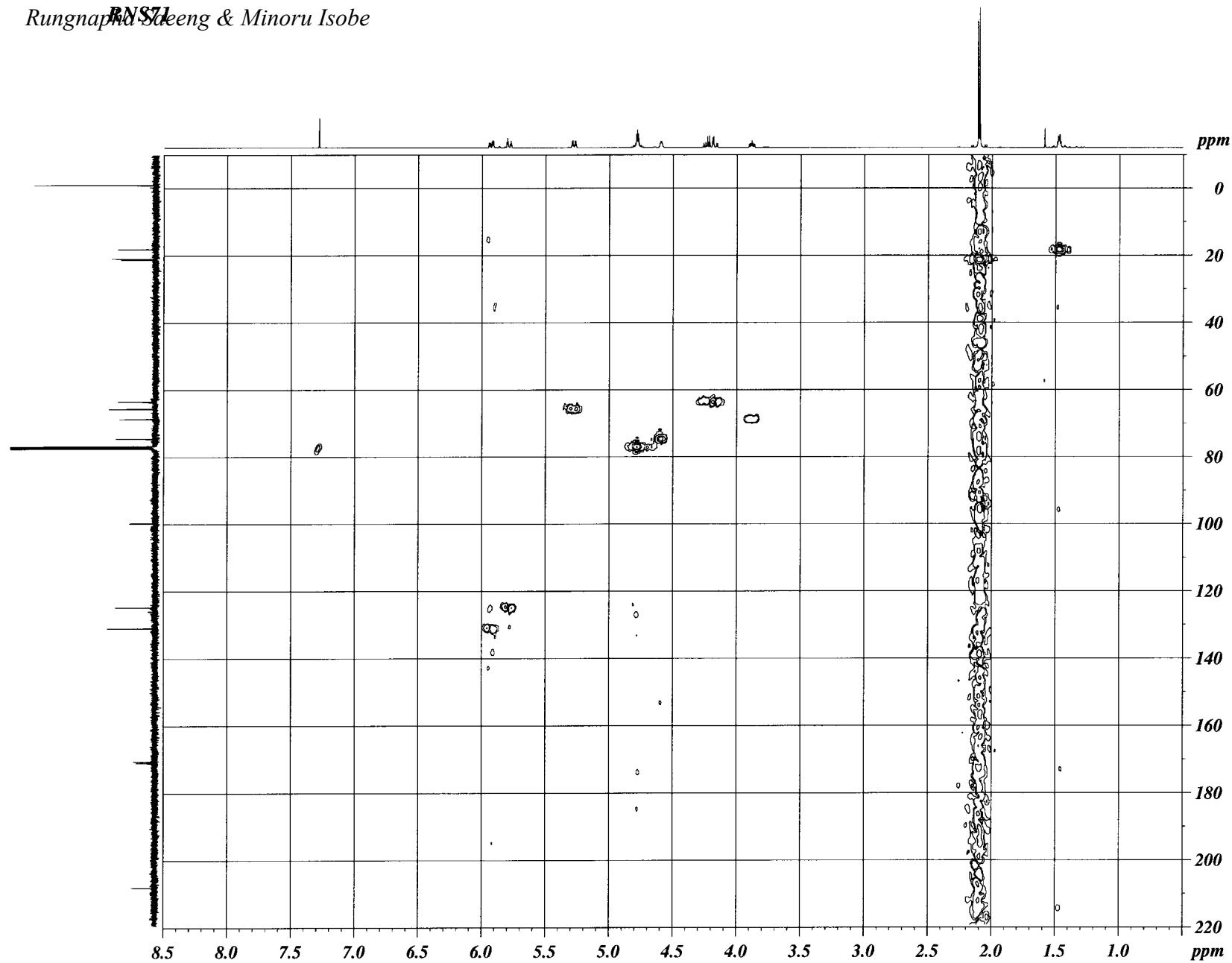
===== CHANNEL f1 =====

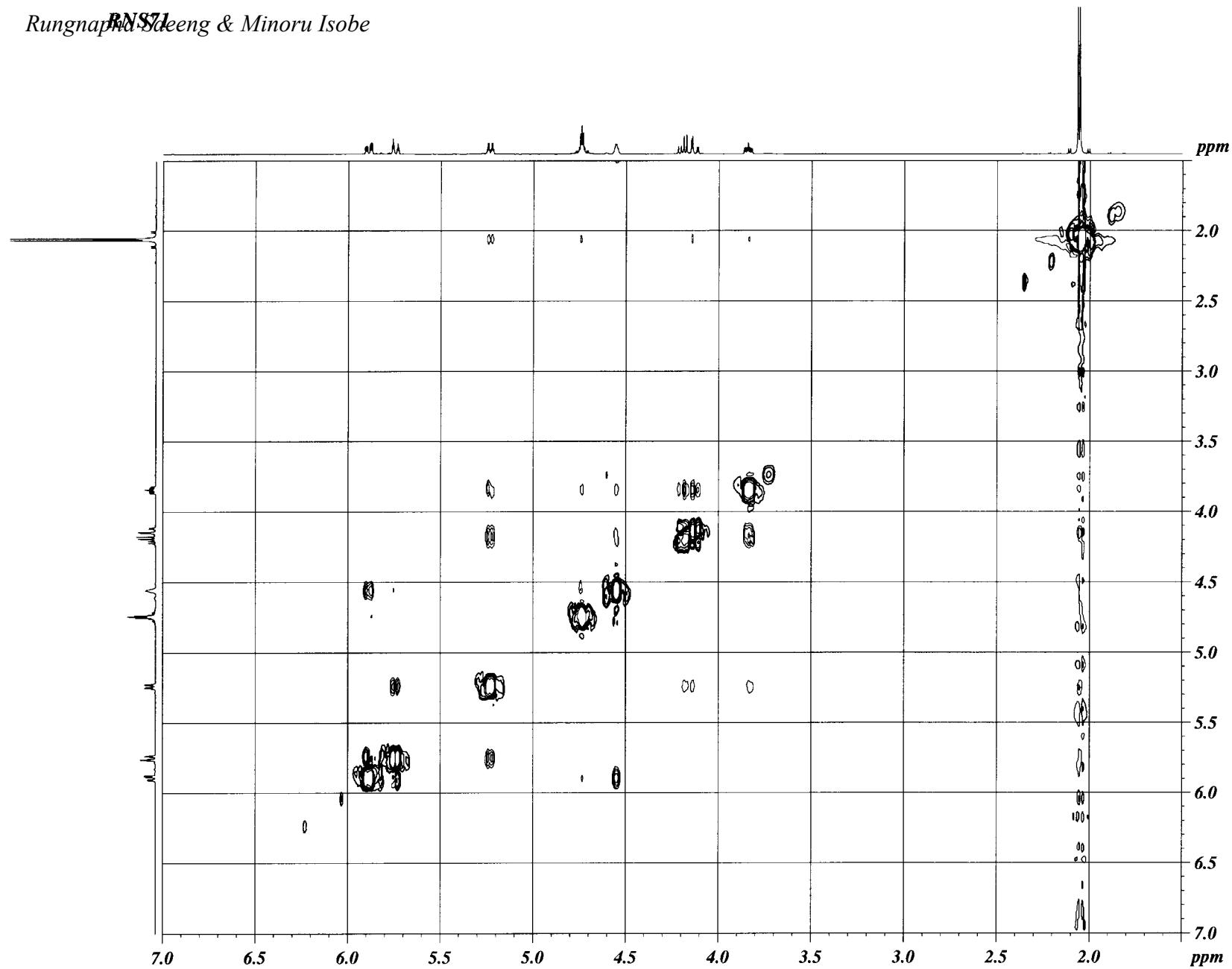
NUC1 IH
 P1 8.60 usec
 PL1 -4.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768

SF 400.1300000 MHz
 WDW EM

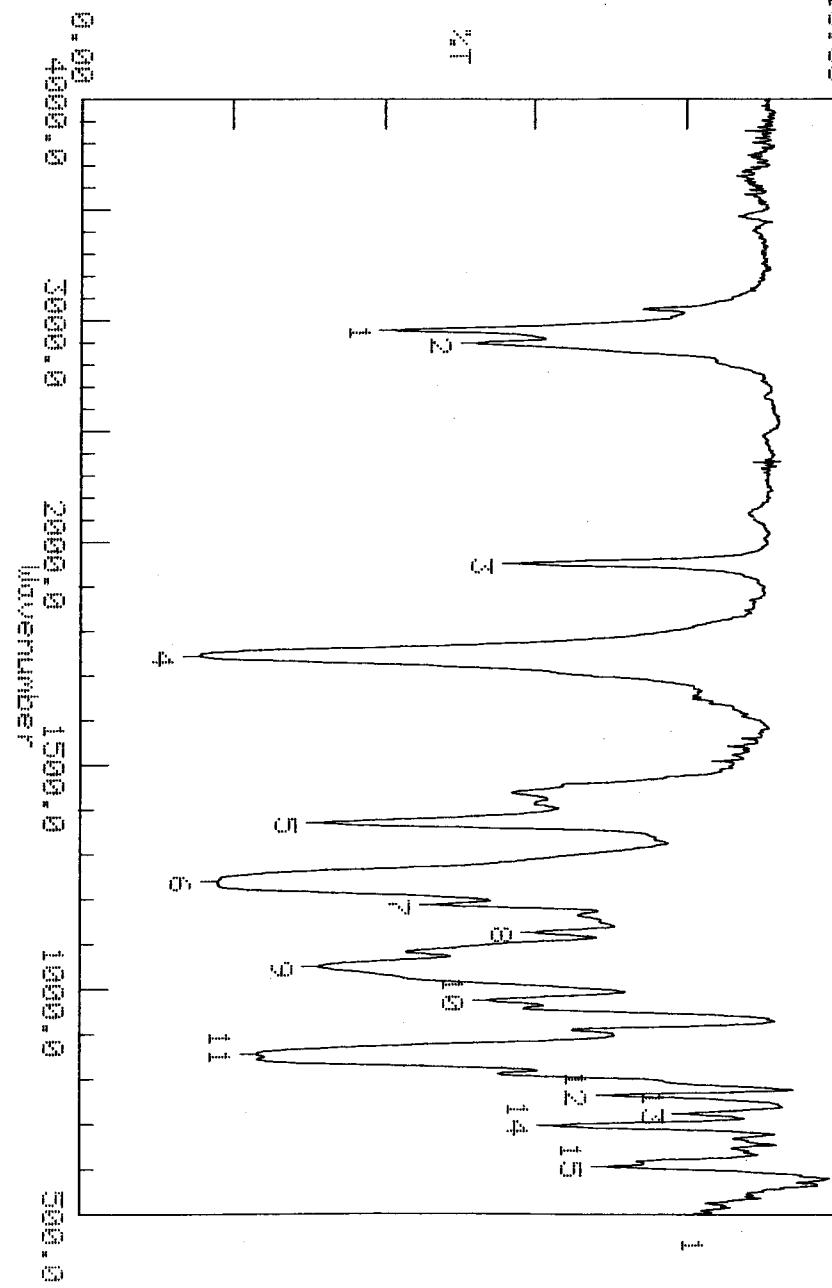
SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





	Condition	Upper 110.00	Lower 0.00	depth 7.00	
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)

Peak table



JASCO FT/IR-8380/01 22:48
 File Name :
 Sample Name : RMS-87-1
 Resolution : 4
 Scans : 16
 Gain : 20
 Apodization: 05



File: RNS-87-2

Date Run: 2004-06-07 (Time Run: 12:36:22)

Run: Sample Description: *u Isobe*

Instrument: JEOL LCmate

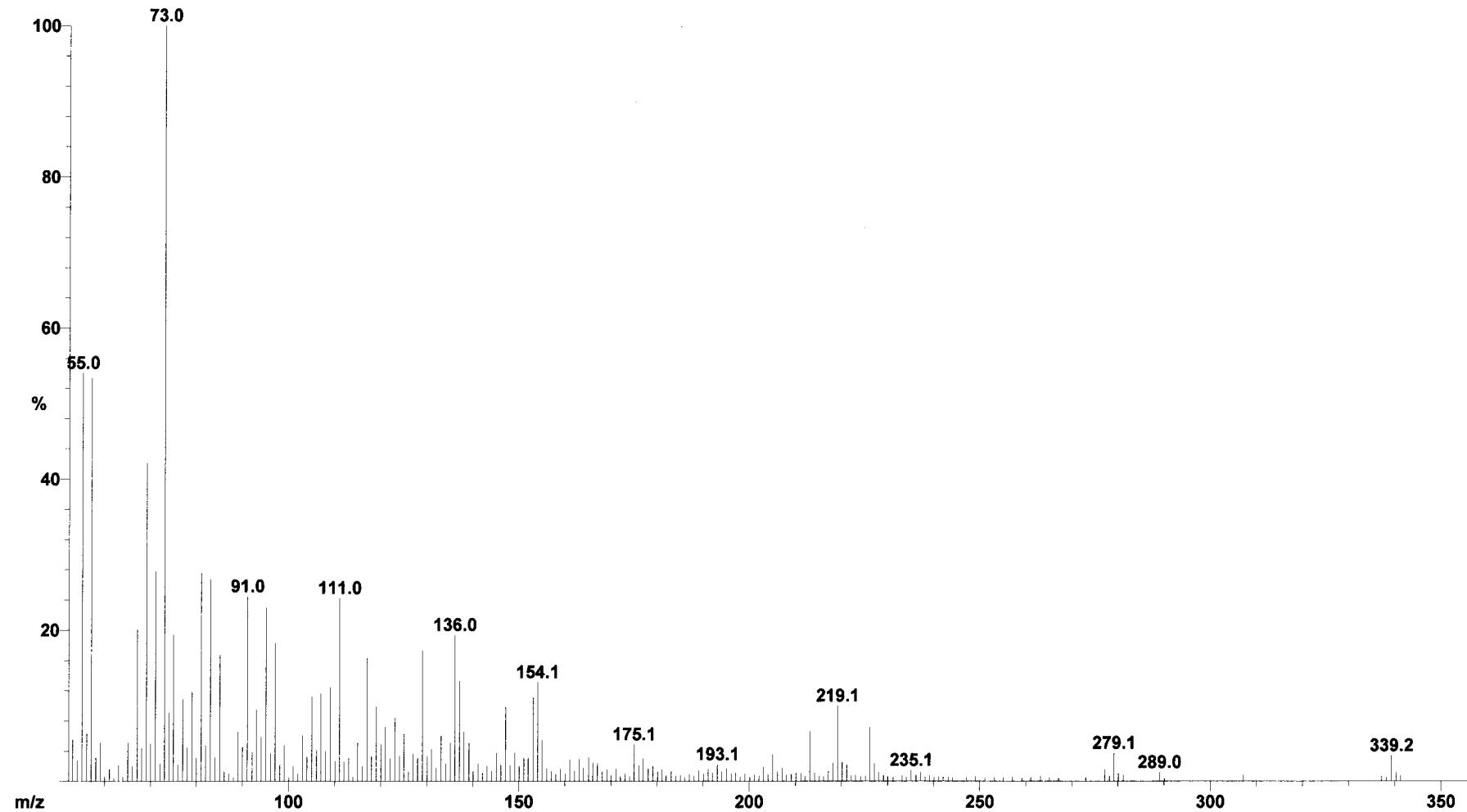
Ionization mode: FAB+

Scan: 9

R.T.: 1.48

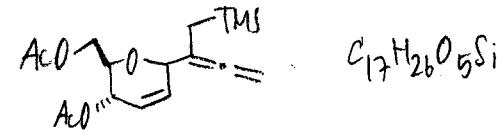
Base: m/z 73; 13.4%FS TIC: 1516496

#Ions: 221



Elemental Analysis Report

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 84.06.05
 LAMP No.589
 TEMP 20°C
 SAMPLE 415-12.1
 CONC. 1.345%
 CELL 100mm
 INTEN. TIME 20sec

 SAMPLE NO. deg 012

1 α -2.100
 $[\alpha] -156.15$
 2 α -2.099
 $[\alpha] -156.11$
 3 α -2.100
 $[\alpha] -156.15$
 4 α -2.099
 $[\alpha] -156.11$
 5 α -2.100
 $[\alpha] -156.20$
 6 α -2.099
 $[\alpha] -156.10$
 7 α -2.099
 $[\alpha] -156.07$
 8 α -2.099
 $[\alpha] -156.12$
 9 α -2.097
 $[\alpha] -155.97$
 10 α -2.098
 $[\alpha] -156.00$

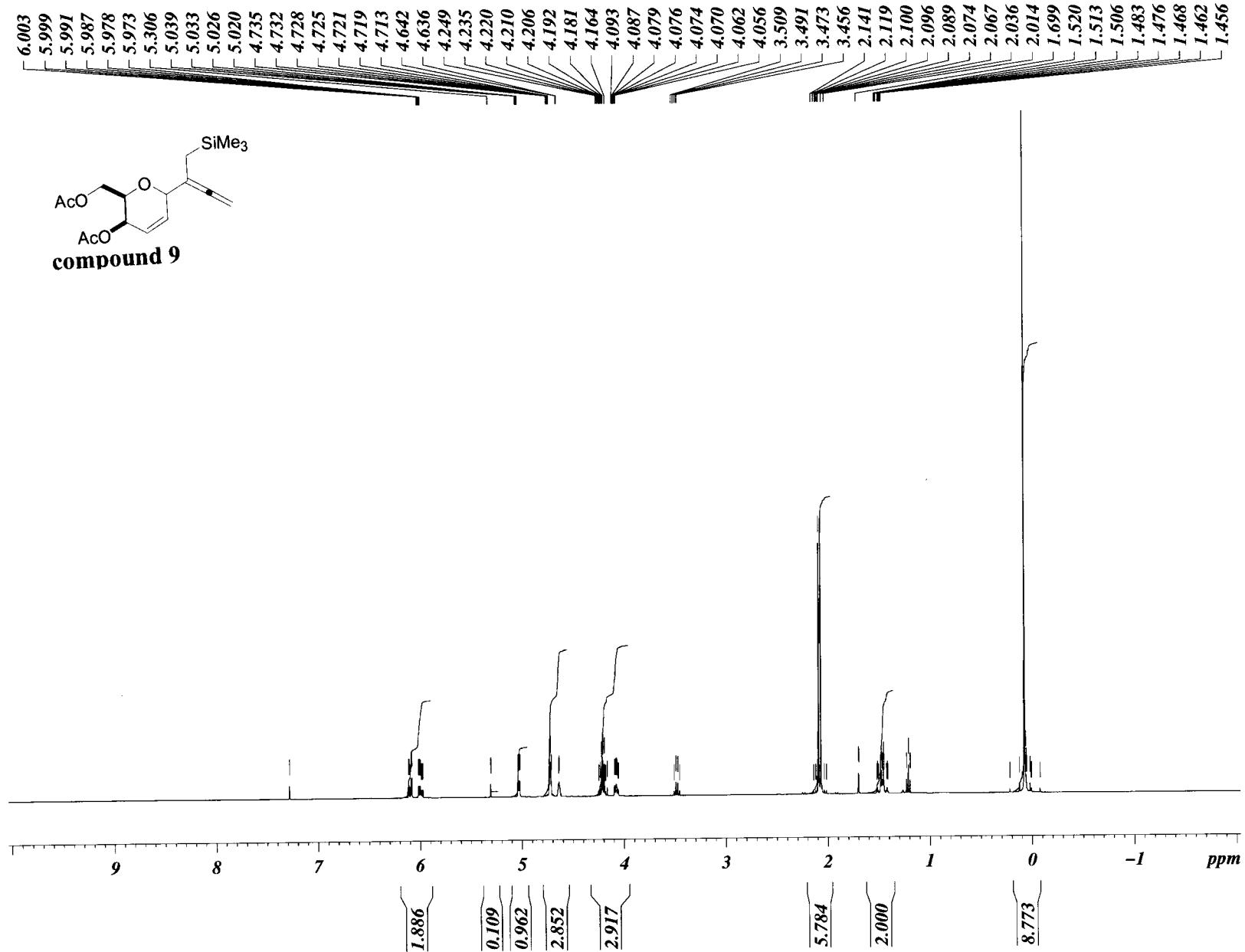
MEAN VALUE N=10

$\bar{\alpha}$ -2.099 deg
 δn^{-1} 0.001 deg
 RSD 0.047 %

$[\alpha] -156.10$ deg
 δn^{-1} 0.0702 deg

TEMP 27



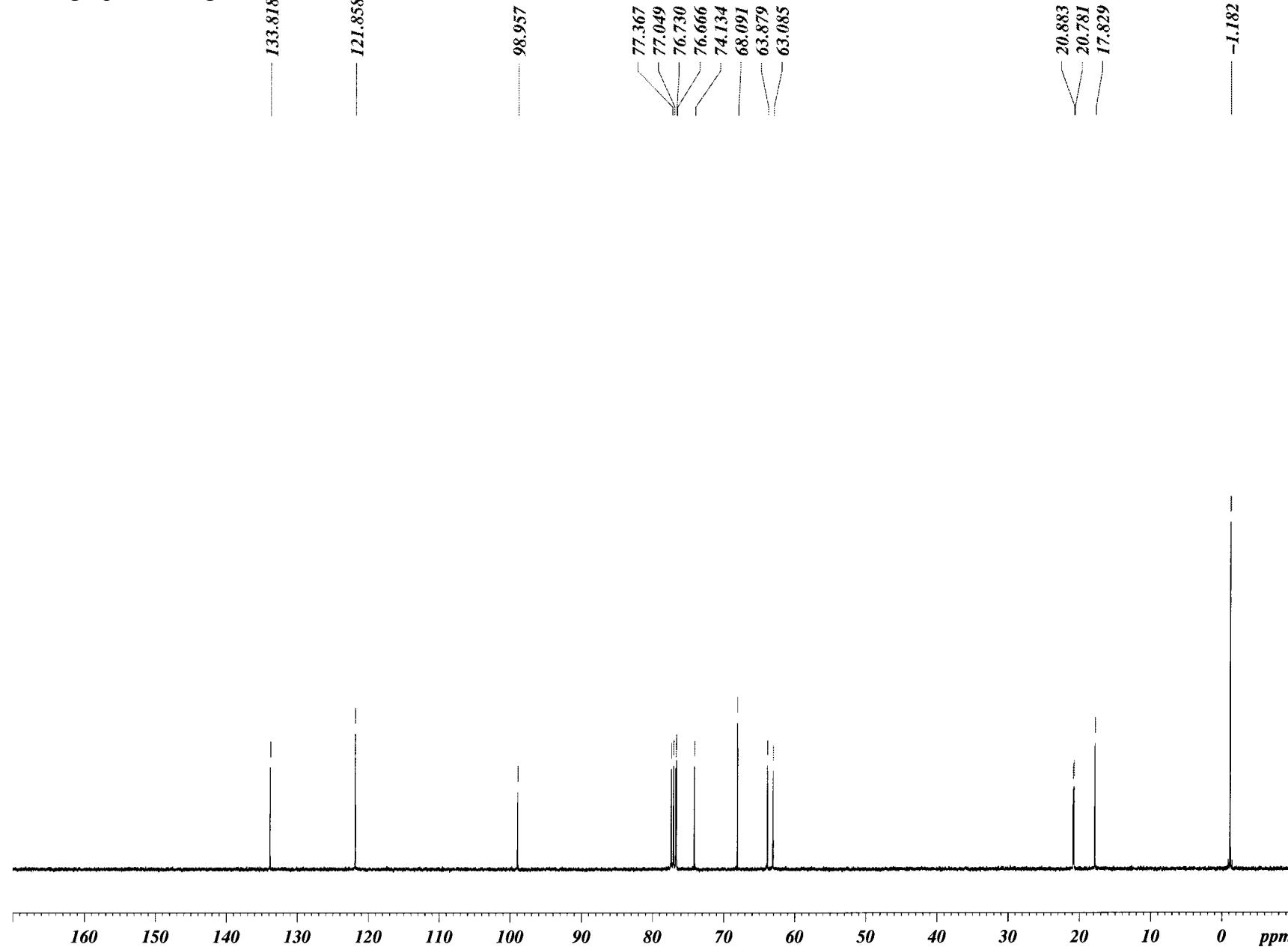


Current Data Parameters
NAME Rungnaphav400
EXPNO 67
PROCNO 1

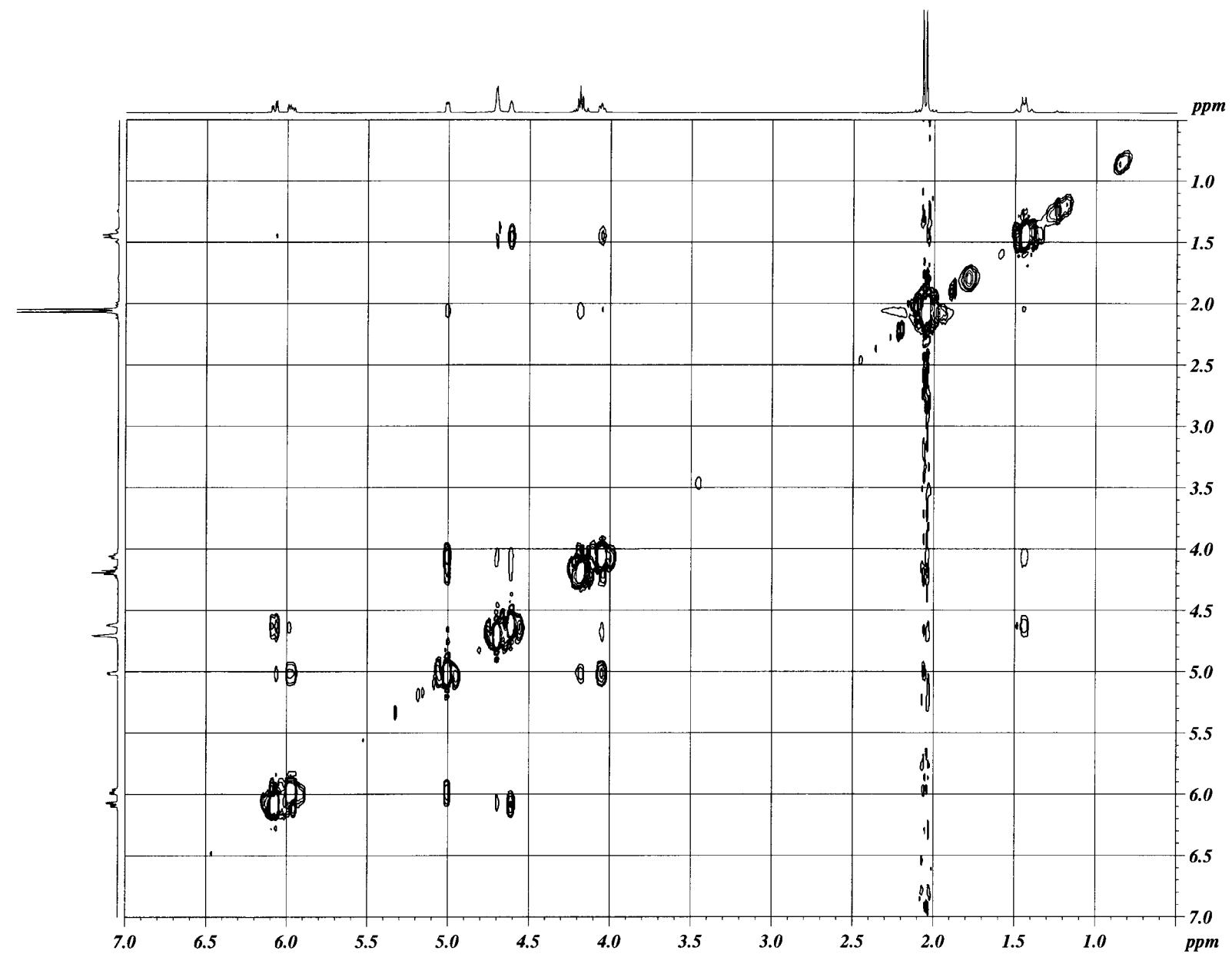
F2 - Acquisition Parameters
Date 20040512
Time 21.13
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
DI 1.0000000 sec

===== CHANNEL,f1 =====
NUCI 1H
PI 8.60 usec
PLI -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

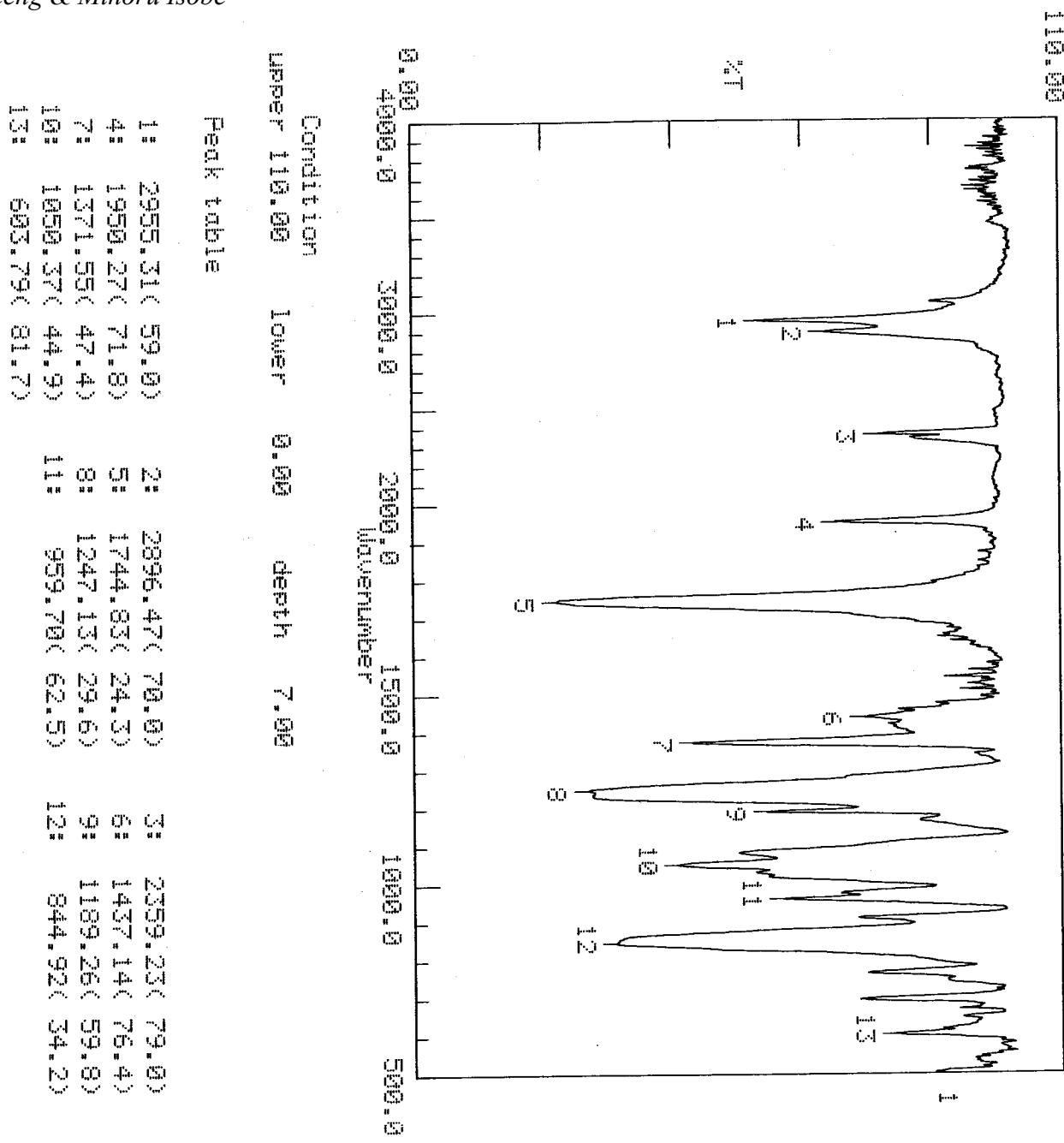


NOESY





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



File: RNS-90-1

Date Run: 2004-06-07 (Time Run: 12:44:09)

Run: Sample Description: *u Isobe*

Instrument: JEOL LCmate

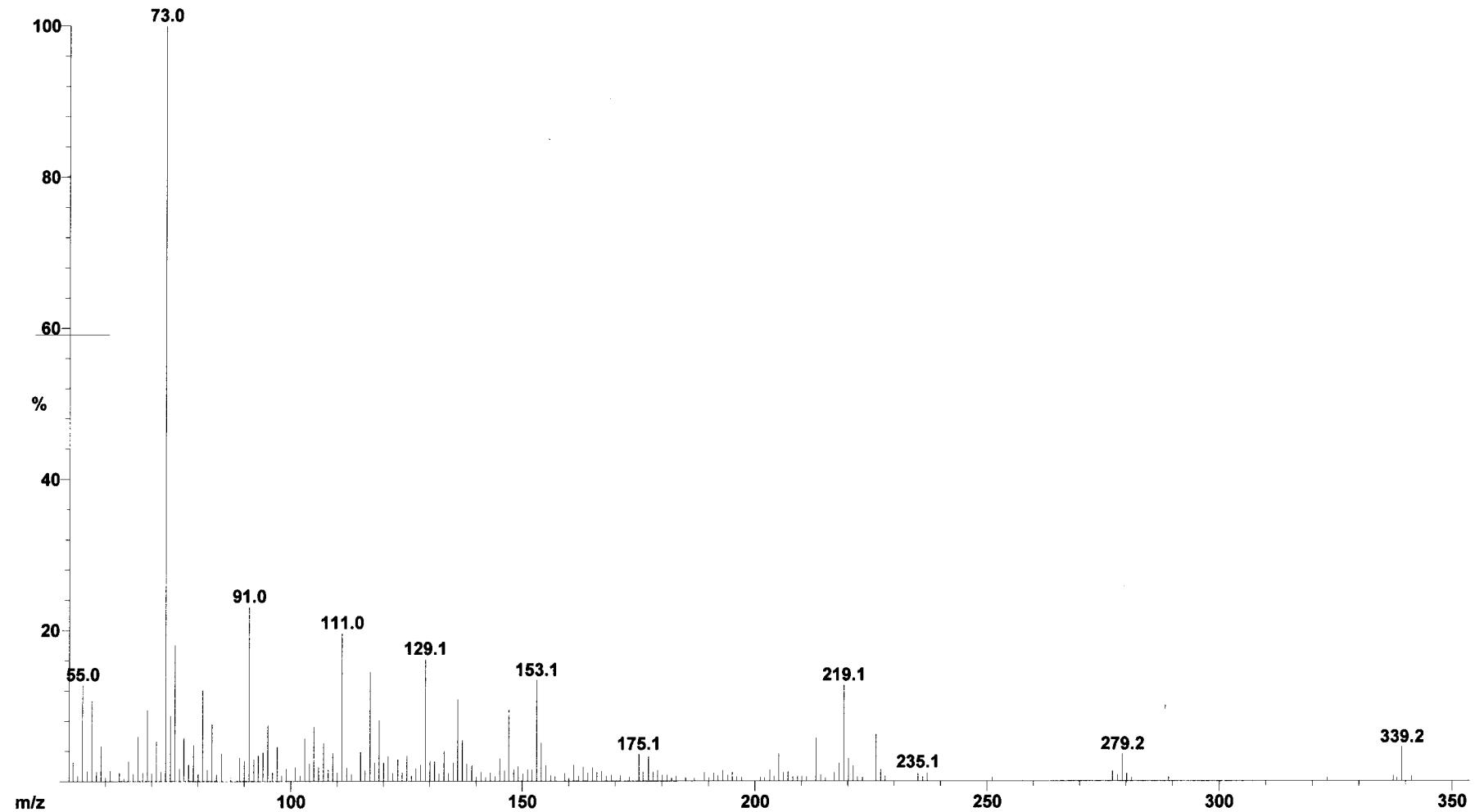
Ionization mode: FAB+

Scan: 16

R.T.: 2.77

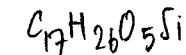
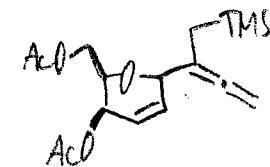
Base: m/z 73; 13.5%FS TIC: 849728

#Ions: 176



Elemental Analysis Report

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



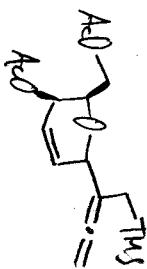
	N	C	H
Calculated weight %	0.00	60.32	7.74
Found weight %	0.15	60.44	7.77
Element ratio(1)	0	2	3
Element ratio(2)	0	13	20
Element ratio(3)	0	15	23
Element ratio(4)	0	17	26
Element ratio(5)	0	28	43
Element ratio(6)	0	32	49

Remarks :

Date : 2004/06/09 15:54:44

Operator : 繁.Kitamura(789-4169)

DATE	04.06.05
LAMP	No.589
TEMP	
SAMPLE	PNS-90-1
CONC.	0.82%
CELL	10mm
INTEG. TIME	20sec



014

1	α	-3.308
2	α	-3.307
	$[\alpha]$	-403.37
3	α	-3.311
	$[\alpha]$	-403.79
4	α	-3.309
	$[\alpha]$	-403.63
5	α	-3.310
	$[\alpha]$	-403.71
6	α	-3.308
	$[\alpha]$	-403.49
7	α	-3.307
	$[\alpha]$	-403.33
8	α	-3.310
	$[\alpha]$	-403.74
9	α	-3.306
	$[\alpha]$	-403.26
10	α	-3.309
	$[\alpha]$	-403.62

MEAN VALUE N=10

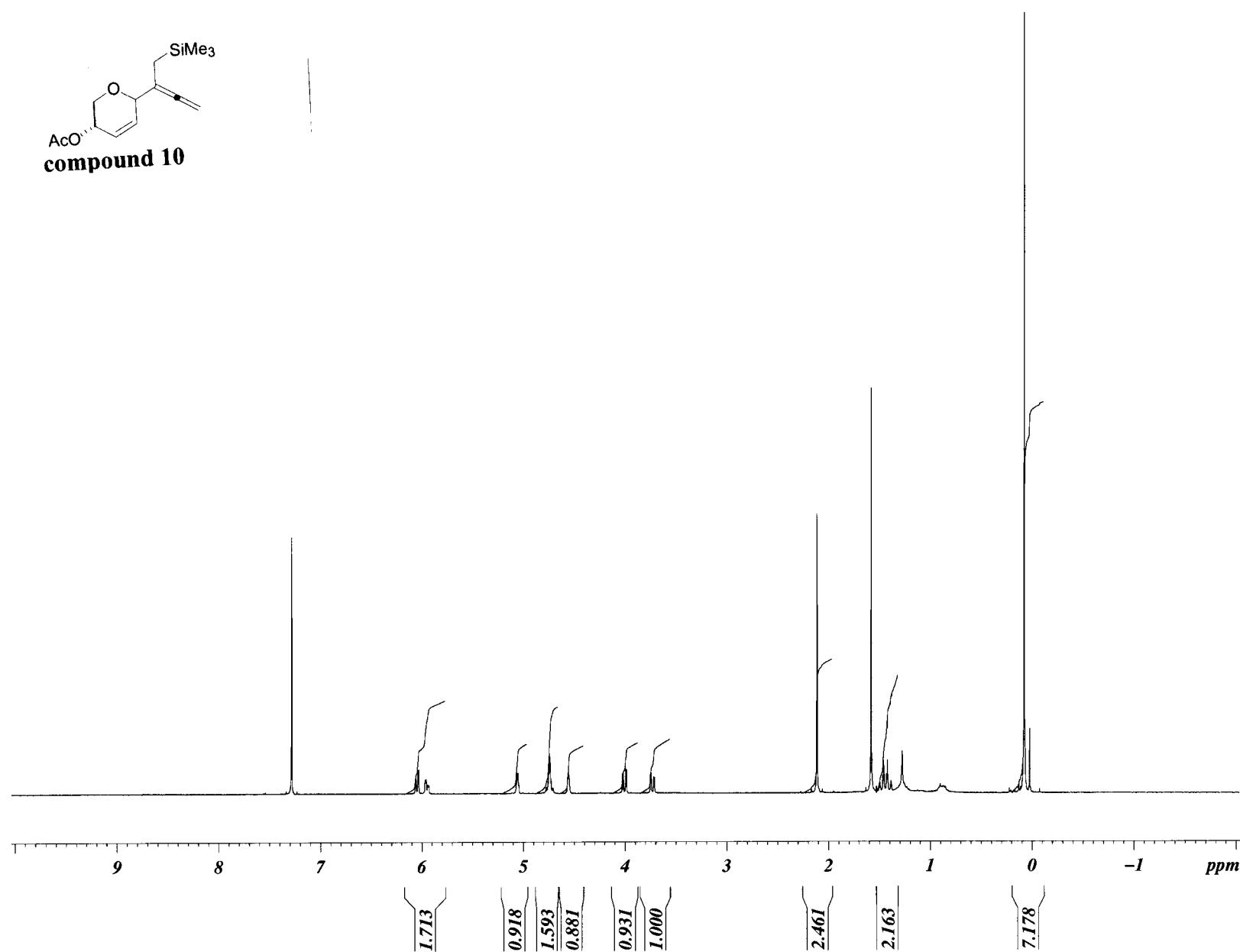
$[\alpha]$ -403.54 deg	6n-1	0.001 deg
6m-1 0.1856 deg	RSD	0.030 %
TEMP		
28		

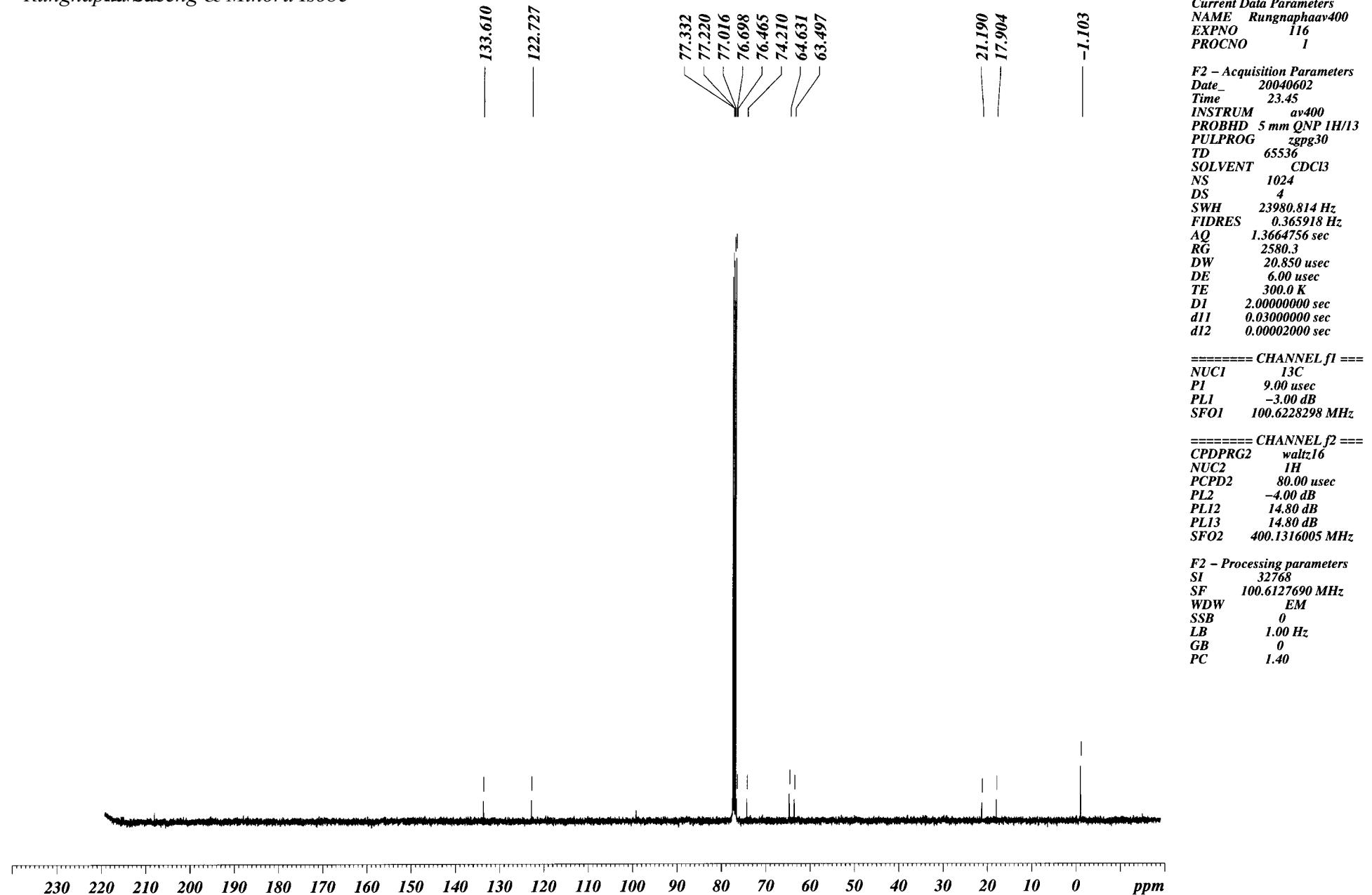
Current Data Parameters
 NAME Rungnaphaav400
 EXPNO 115
 PROCNO 1

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 1.0000000 sec

===== CHANNEL f1 =====
 NUCI 1H
 PI 8.60 usec
 PLI -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 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

DI 1.0000000 sec

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

NUCI 1H

PI 8.60 usec

PLI -4.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.1300000 MHz

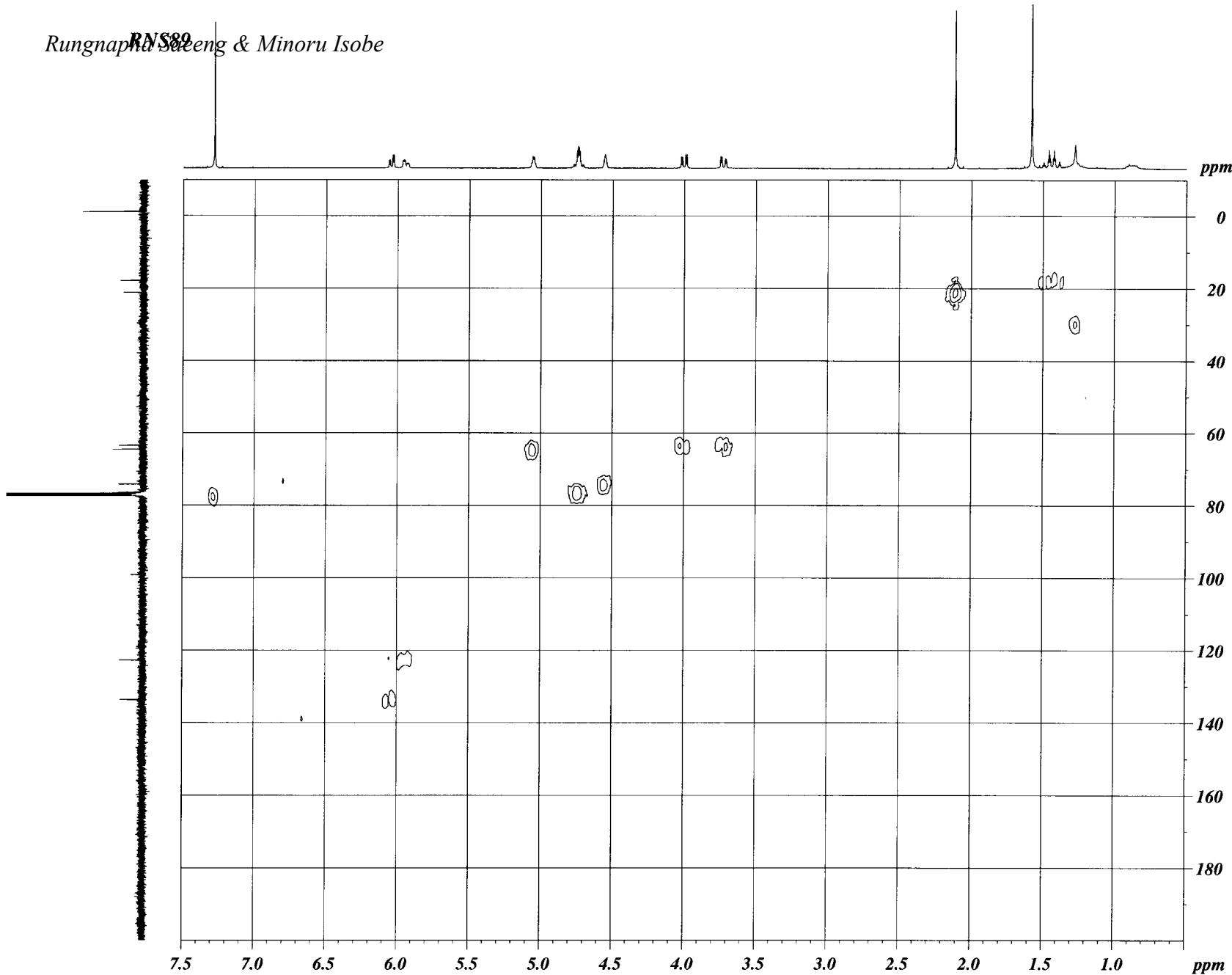
WDW EM

SSB 0

LB 0.30 Hz

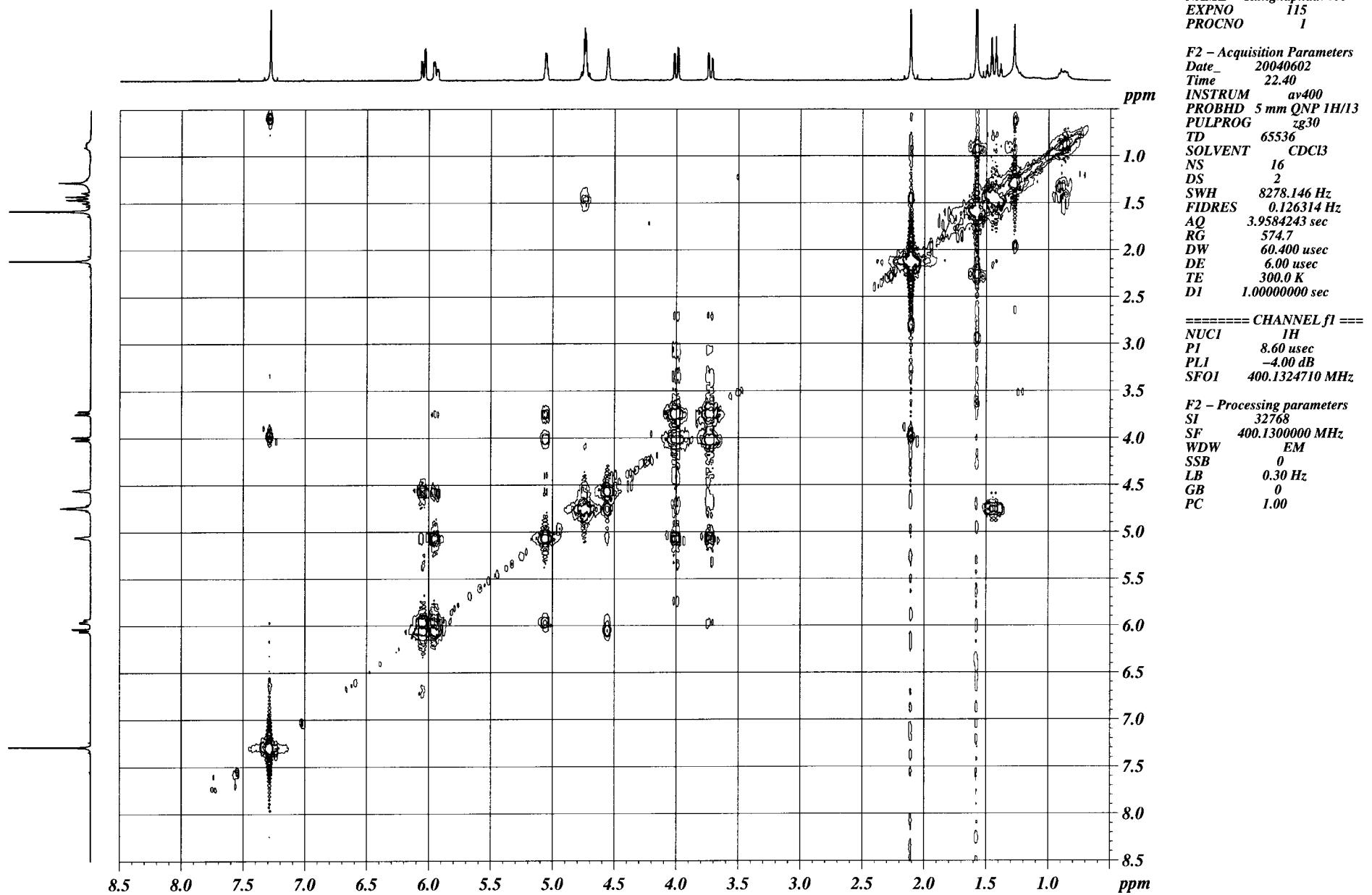
GB 0

PC 1.00



COSY

Rungnaphaveng & Minoru Isobe



NOESY

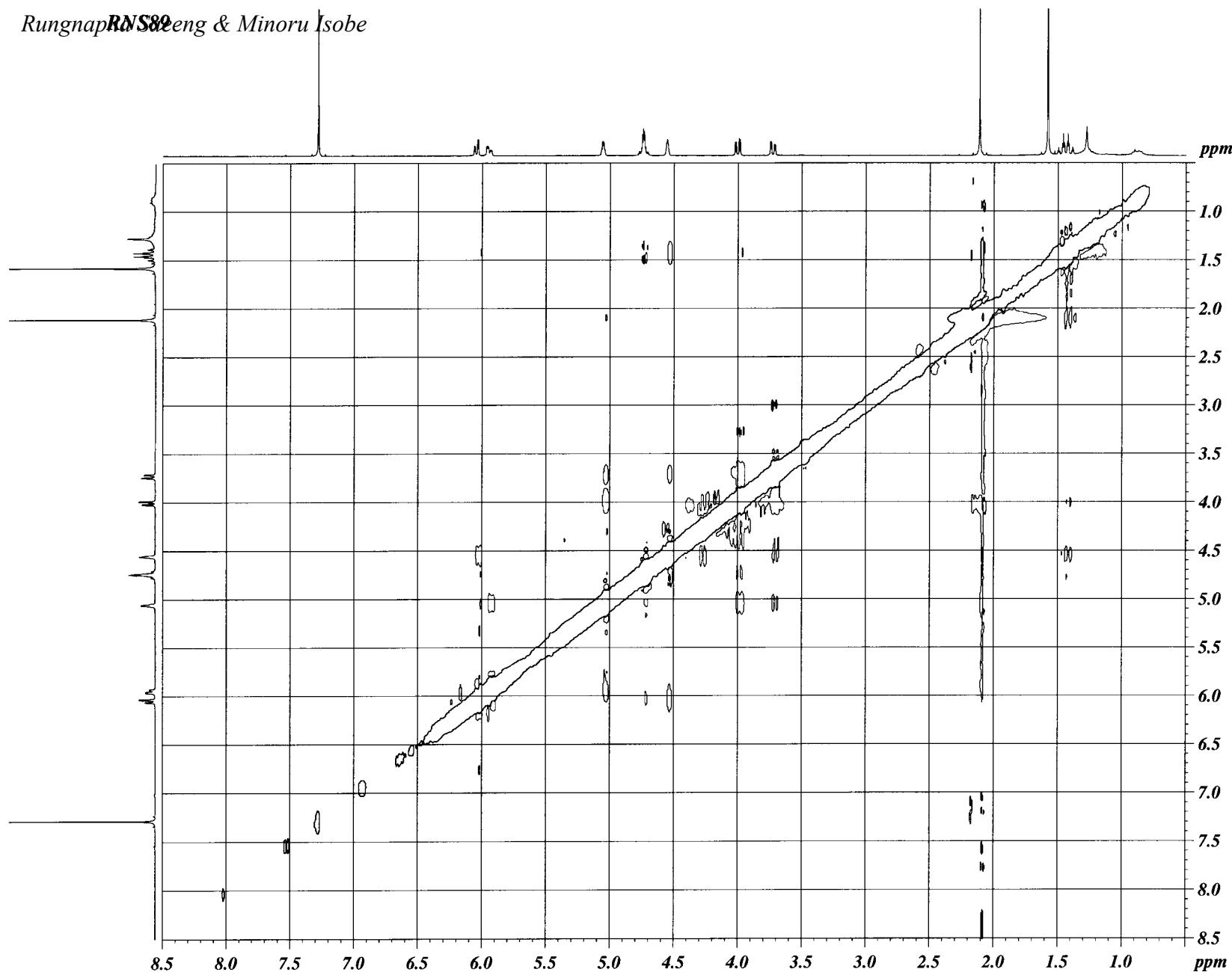
RungnapRNS82eng & Minoru Isobe

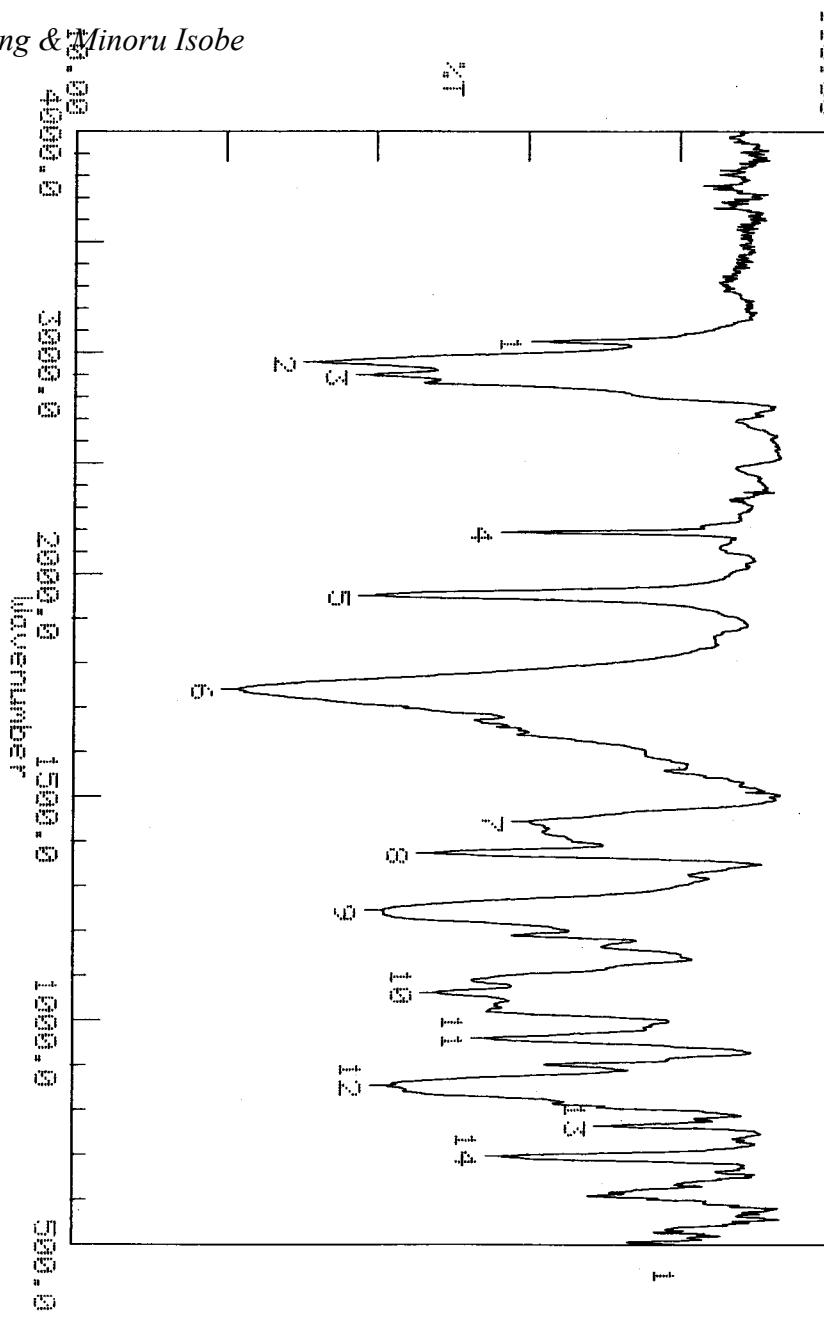
Current Data Parameters
 NAME Rungnaphav400
 EXPNO 115
 PROCNO 1

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 1.0000000 sec

===== CHANNEL f1 =====
 NUCI 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





JR500 FT/IR-8300
 Date : 99/04/01 23:24
 File Name :
 Sample Name: RH5-106
 Resolution : 4
 Scans : 16
 Gain : 20
 Rerodization: 05

Peak table

	Peak No.	Wavenumber (cm⁻¹)	Intensity (%)
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)

Run number: 89
Sample Description: *u Isobe*
Instrument: JEOL LCmate

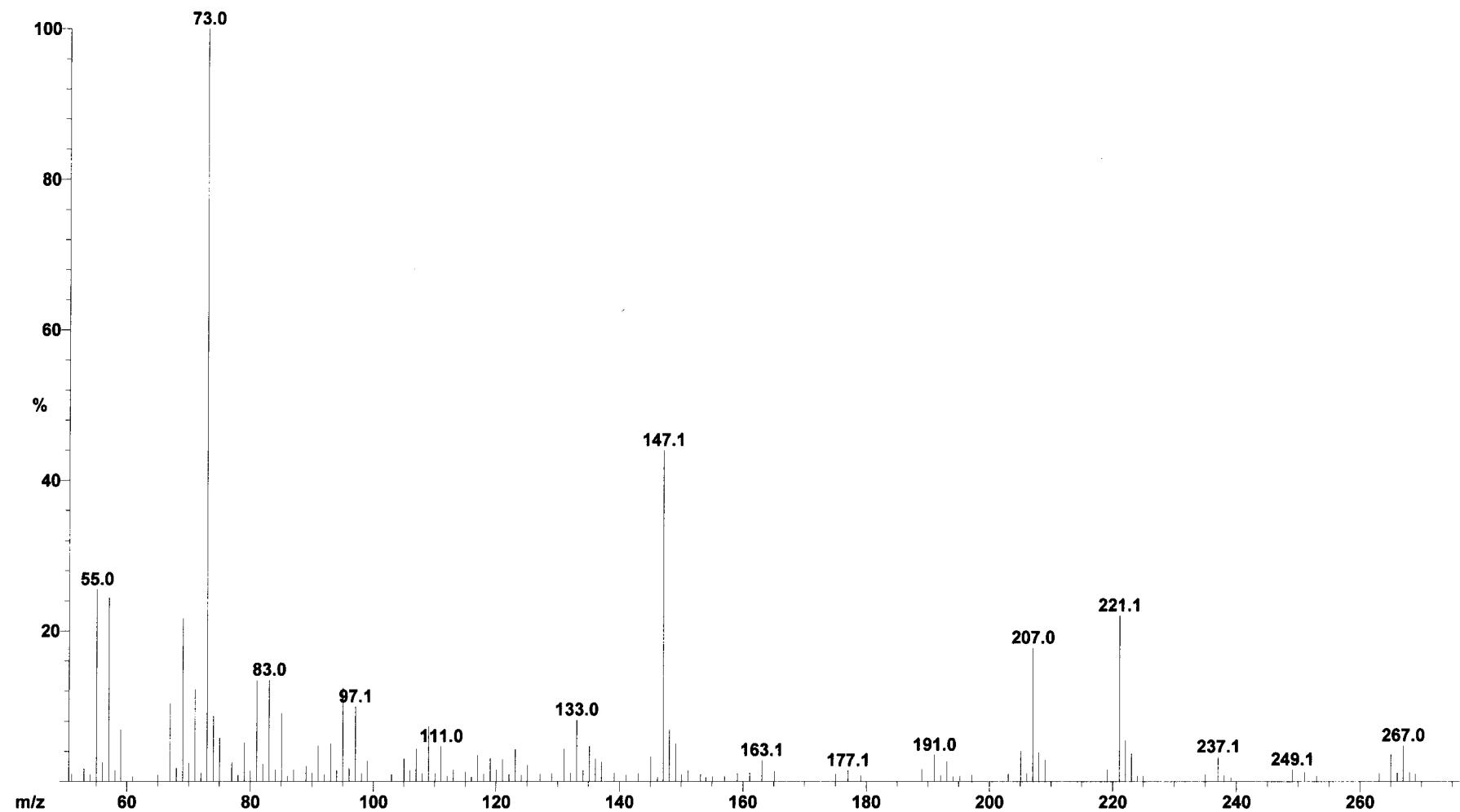
Ionization mode: FAB+

Scan: 1

Base: m/z 73; 9.2%FS TIC: 634912

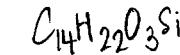
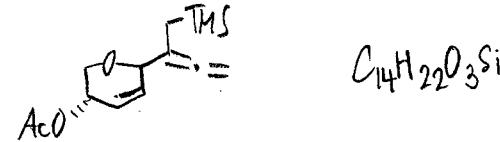
R.T.: .02

#Ions: 161



Elemental Analysis Report

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



	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	No.589
TEMP	
SAMPLE	RNS-10 ^b
CONC.	1.345%
CELL	100nm
INTEG. TIME	20sec

SAMPLE NO.	deg

005

1	δ	1.921
	[α]	142.83
2	δ	1.921
	[α]	142.85
3	δ	1.921
	[α]	142.83
4	δ	1.922
	[α]	142.89
5	δ	1.922
	[α]	142.95
6	δ	1.922
	[α]	142.97
7	δ	1.923
	[α]	143.01
8	δ	1.919
	[α]	142.74
9	δ	1.922
	[α]	142.92
10	δ	1.924
	[α]	143.08

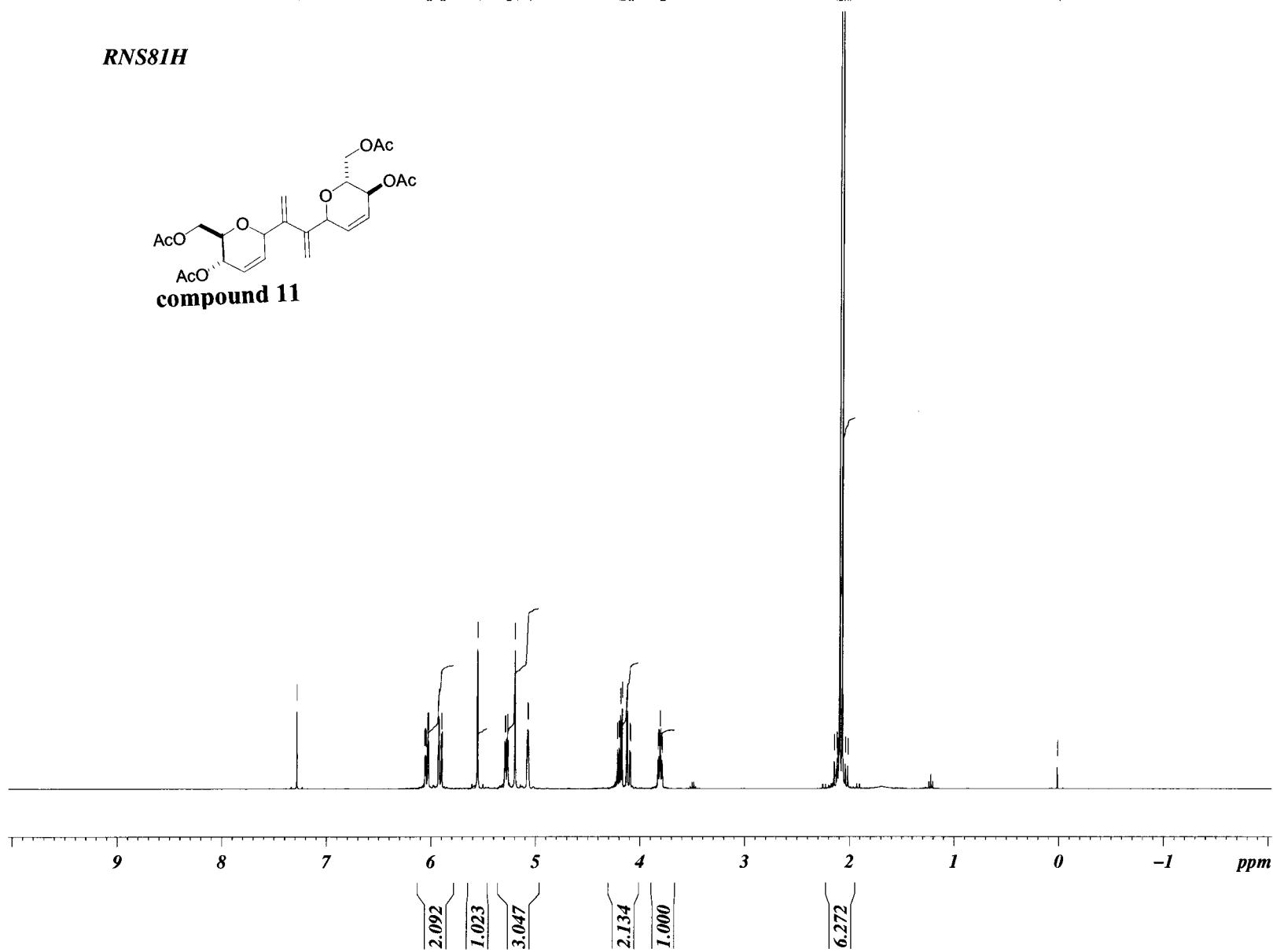
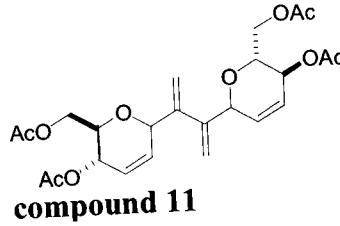
MEAN VALUE N=10

δ	1.922 deg
δn-1	0.001 deg
RSD	0.052 %

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

R283
 Rungnaphaav400
 R056
 R052
 R048
 R035
 R030
 R026
 R022
 R028
 R023
 R019
 R003
 R002
 R001
 R000

RNS81H



Current Data Parameters
 NAME Rungnaphaav400
 EXPNO 91
 PROCNO 1

F2 - Acquisition Parameters

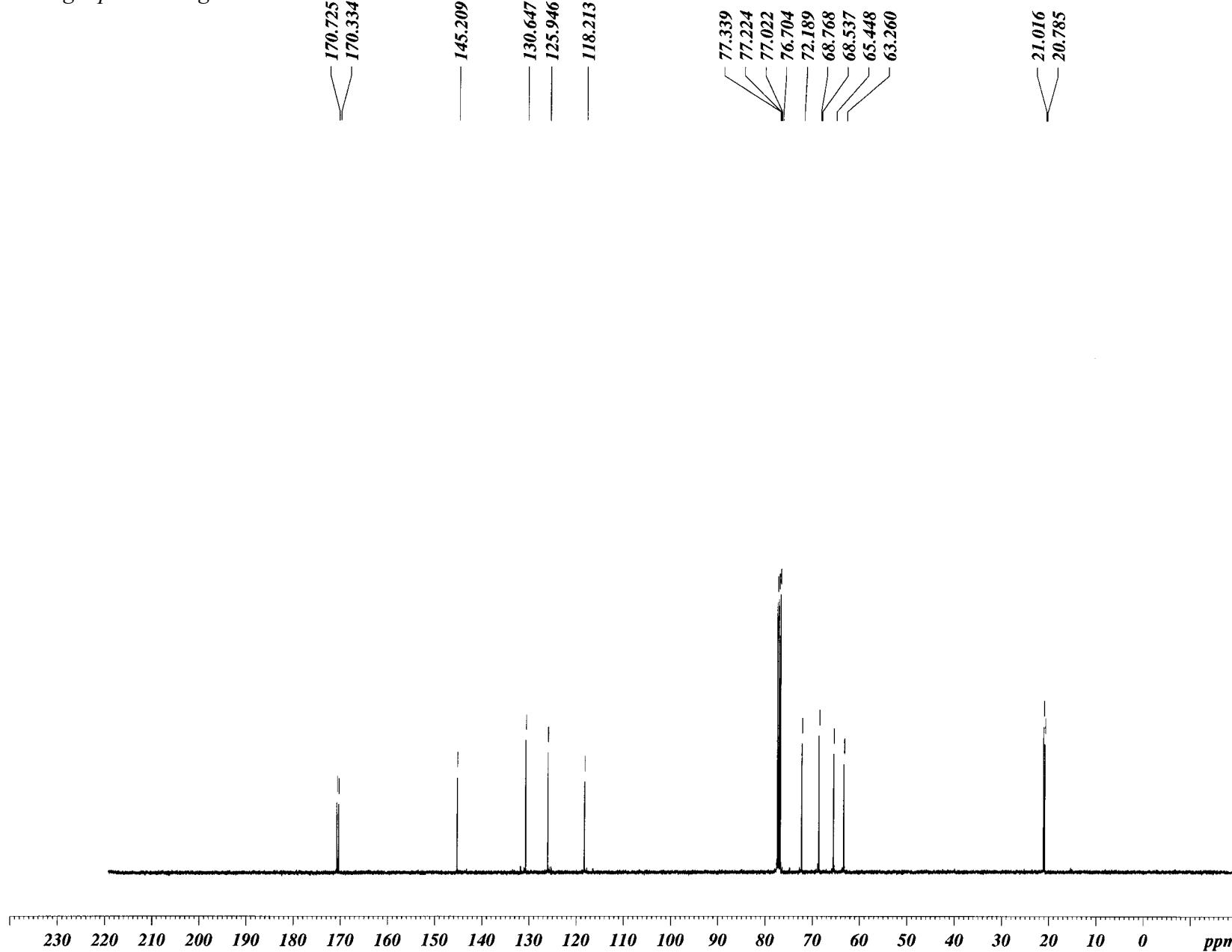
Date_ 20040523
 Time 21.06
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 DI 1.0000000 sec

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

NUC1 1H
 PI 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



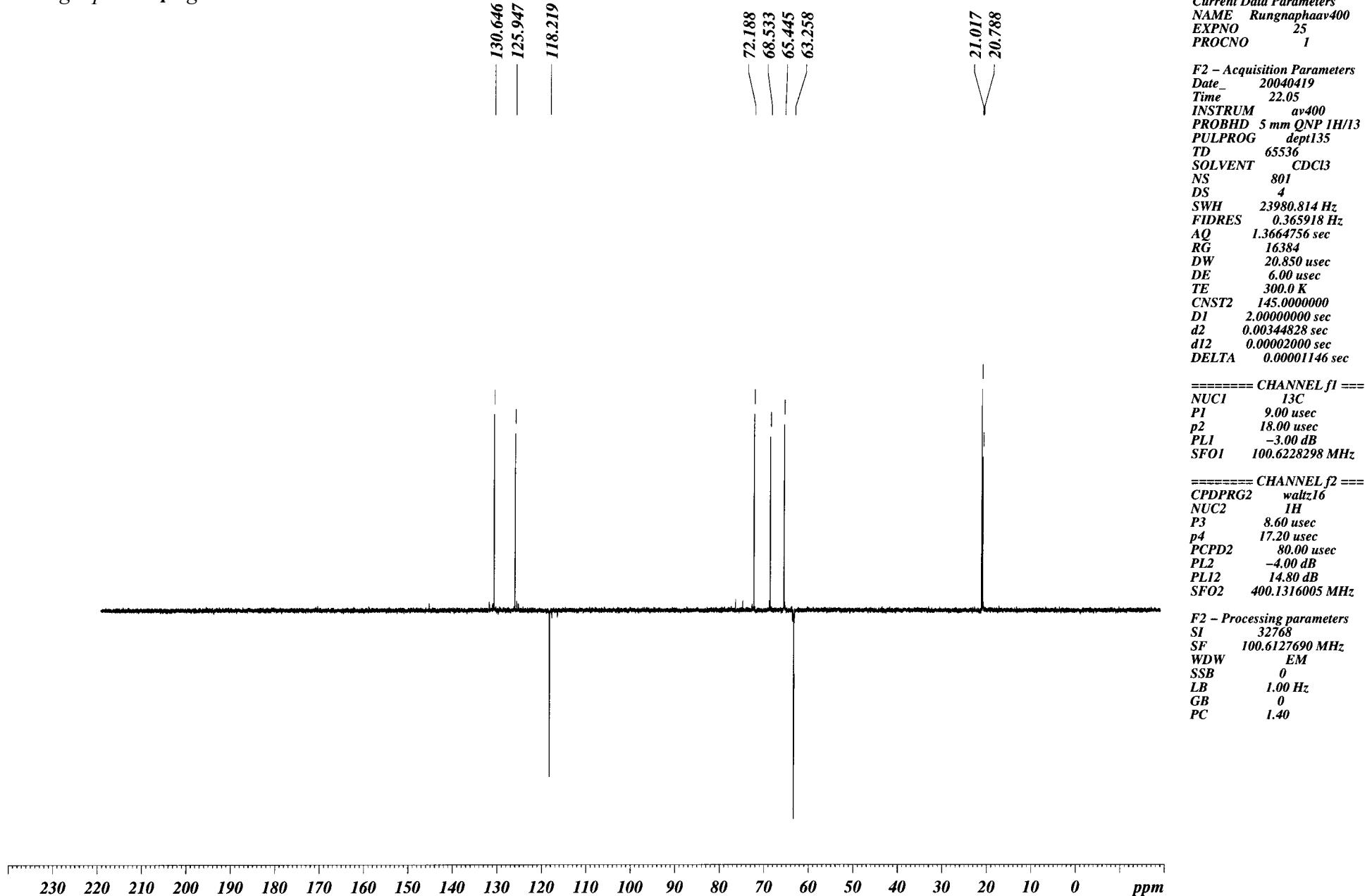
Current Data Parameters
 NAME Rungnaphaavav400
 EXPNO 22
 PROCNO 1

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 2.0000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 ¹³C
 PI 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 Rungnaphaa400
 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

DI 1.0000000 sec

===== CHANNEL f1 ===

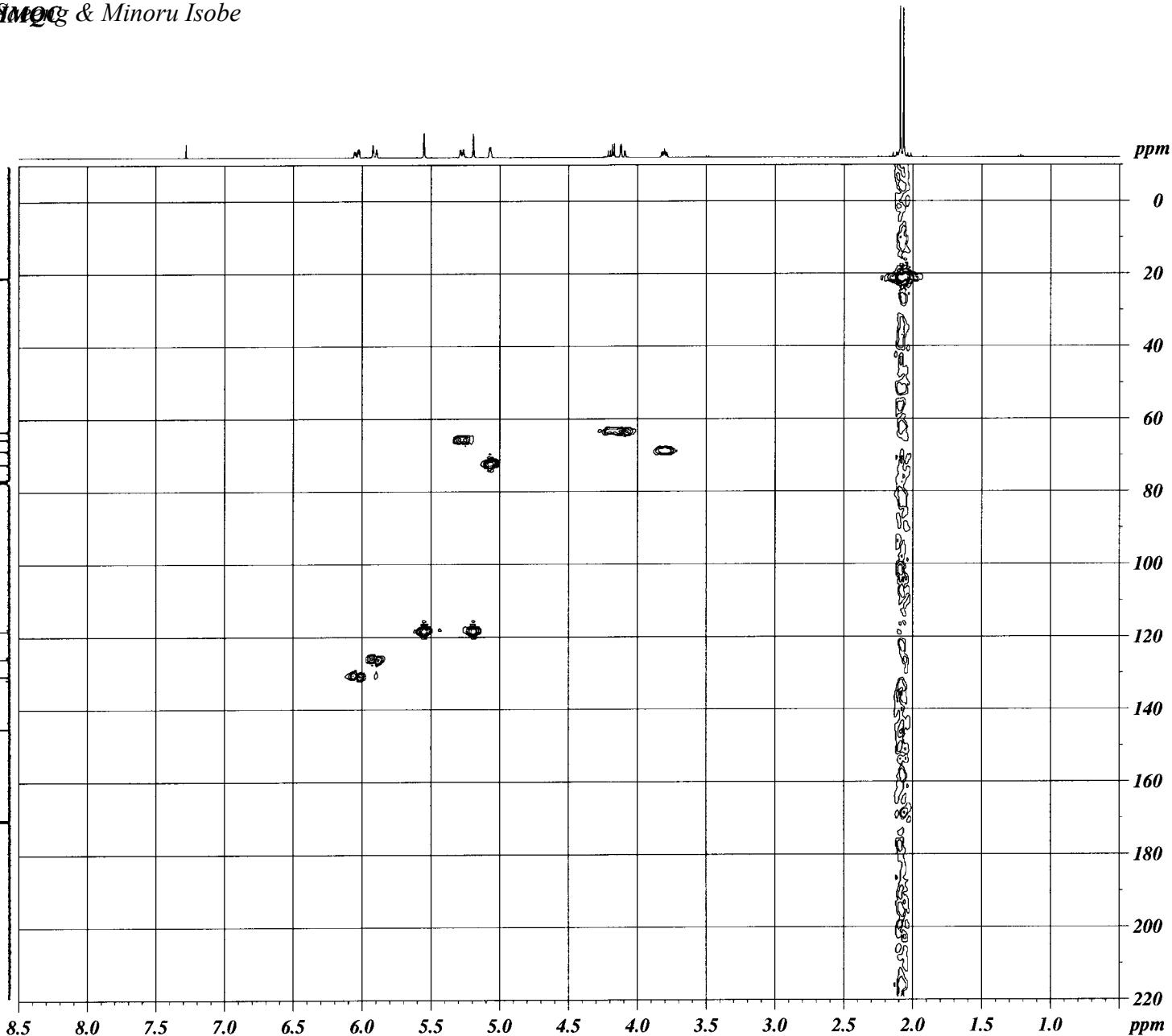
NUCI IH
 PI 8.60 usec
 PLI -4.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300000 MHz

WDW EM
 SSB 0

LB 0.30 Hz
 GB 0
 PC 1.00



NOESY

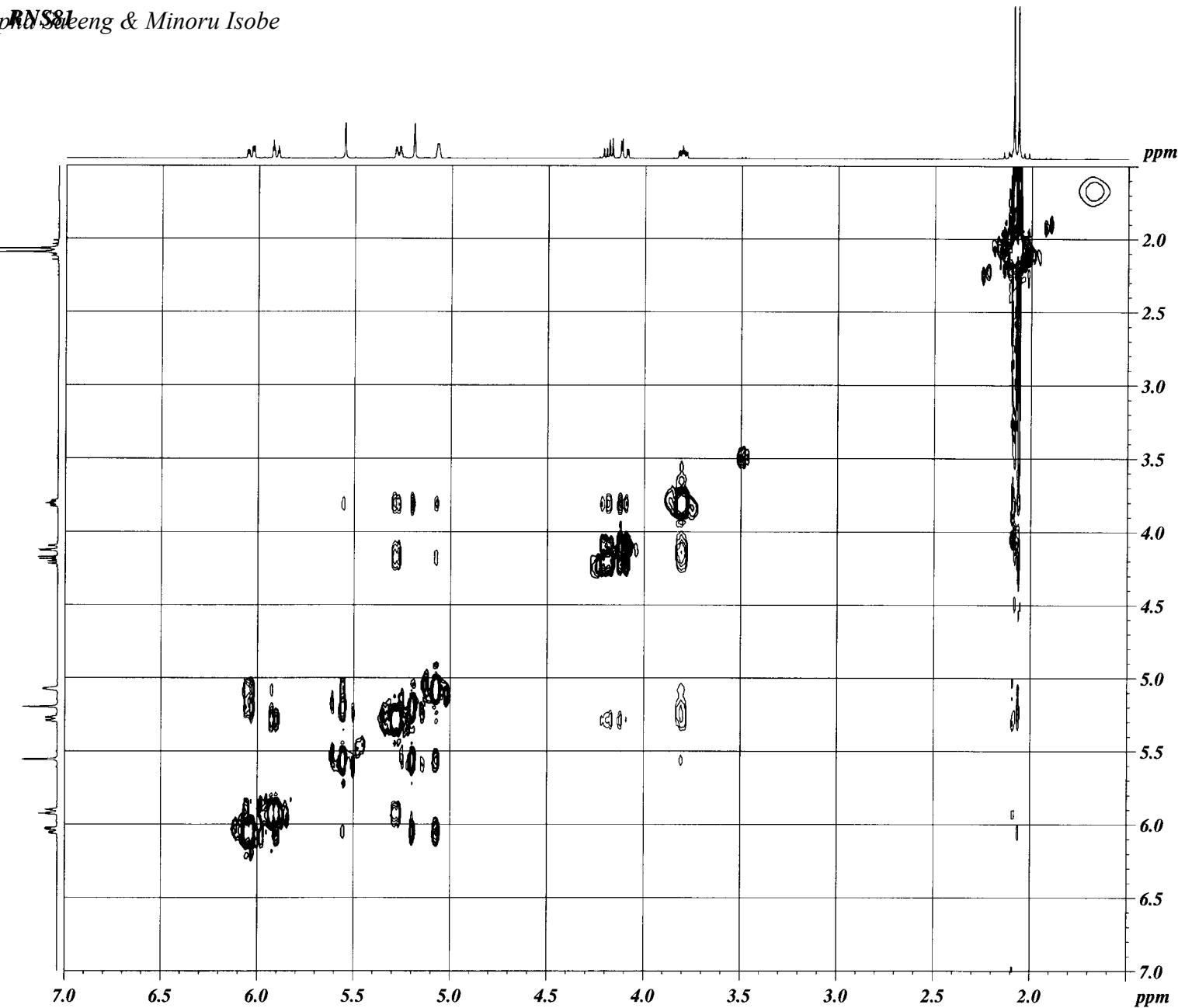
Rungnapa ~~RNS8keng~~ & Minoru Isobe

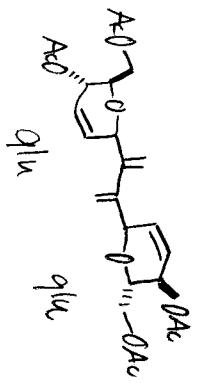
Current Data Parameters
 NAME Rungnaphaav400
 EXPNO 91
 PROCNO 1

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
 DI 1.0000000 sec

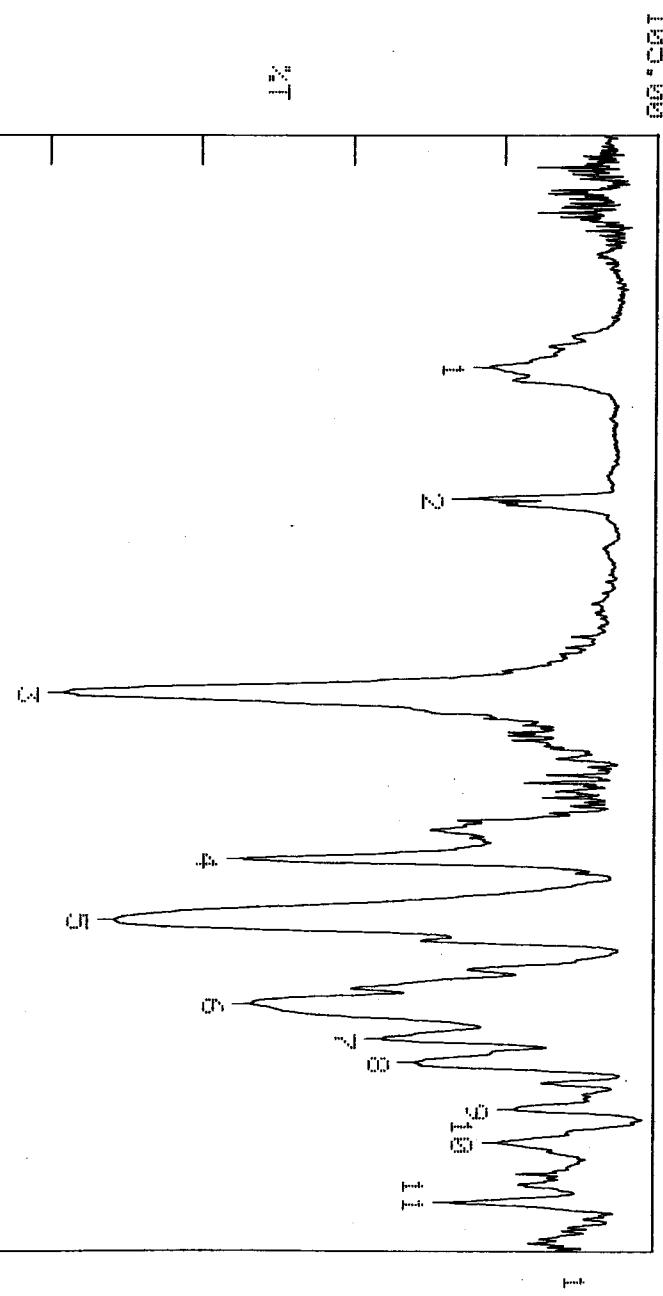
===== CHANNEL f1 =====
 NUCI 1H
 PI 8.60 usec
 PLI -4.00 dB
 SFO1 400.1324710 MHz

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





JR500 FT/IR-8300
 Date : 99/04/01 21:15
 File Name :
 Sample Name: RMS-81
 Resolution : 4
 Scans : 16
 Gain : 20
 Apodization: 05



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:	686.69(79.7)		

File: RNS-81

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

Run # Sample Description: *Isobe*
Instrument: JEOL LCmate

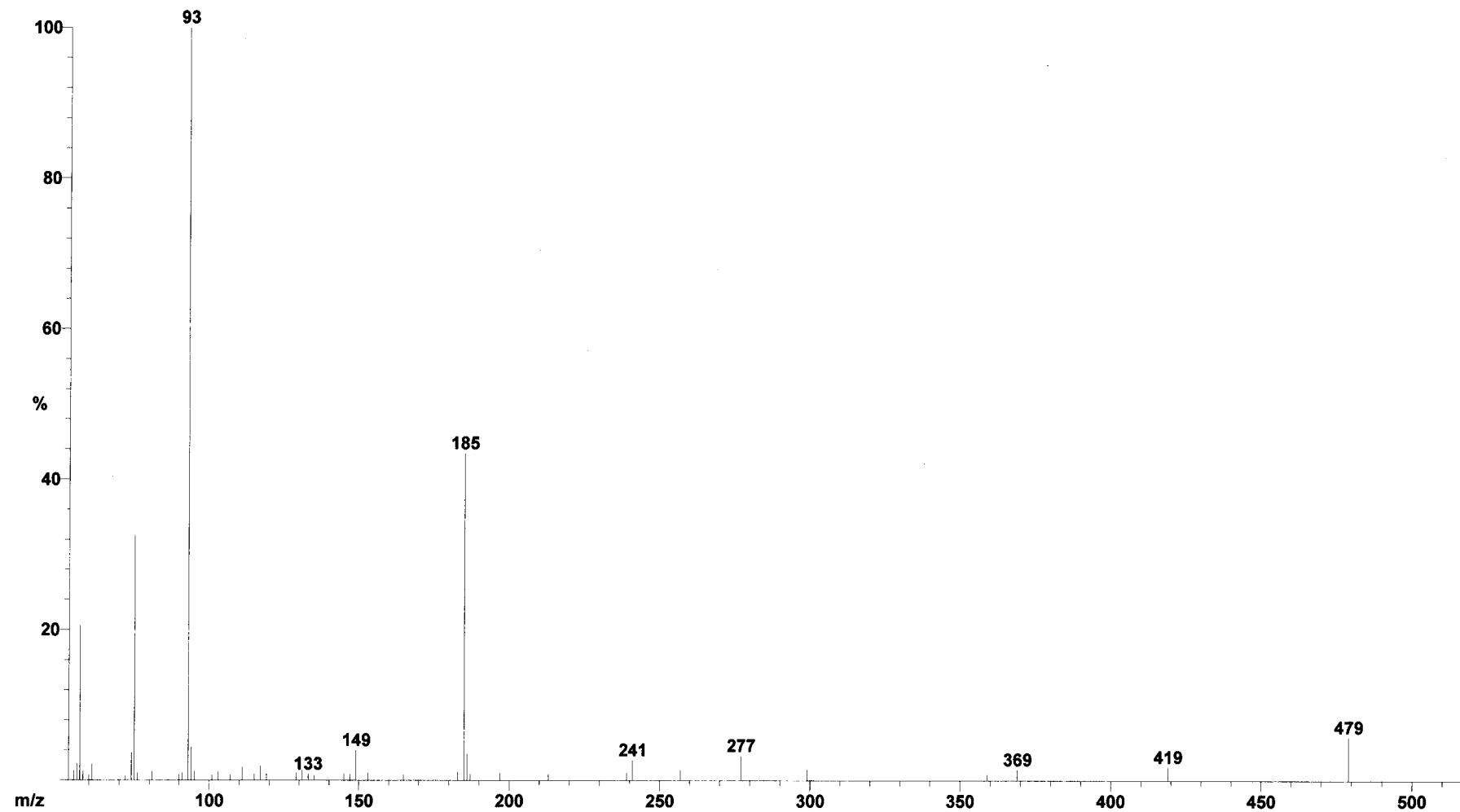
Ionization mode: FAB+

Scan: 1

R.T.: .02

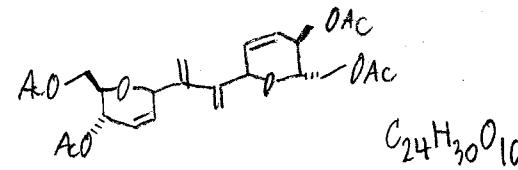
Base: m/z 93; 9.4%FS TIC: 262688

#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



	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

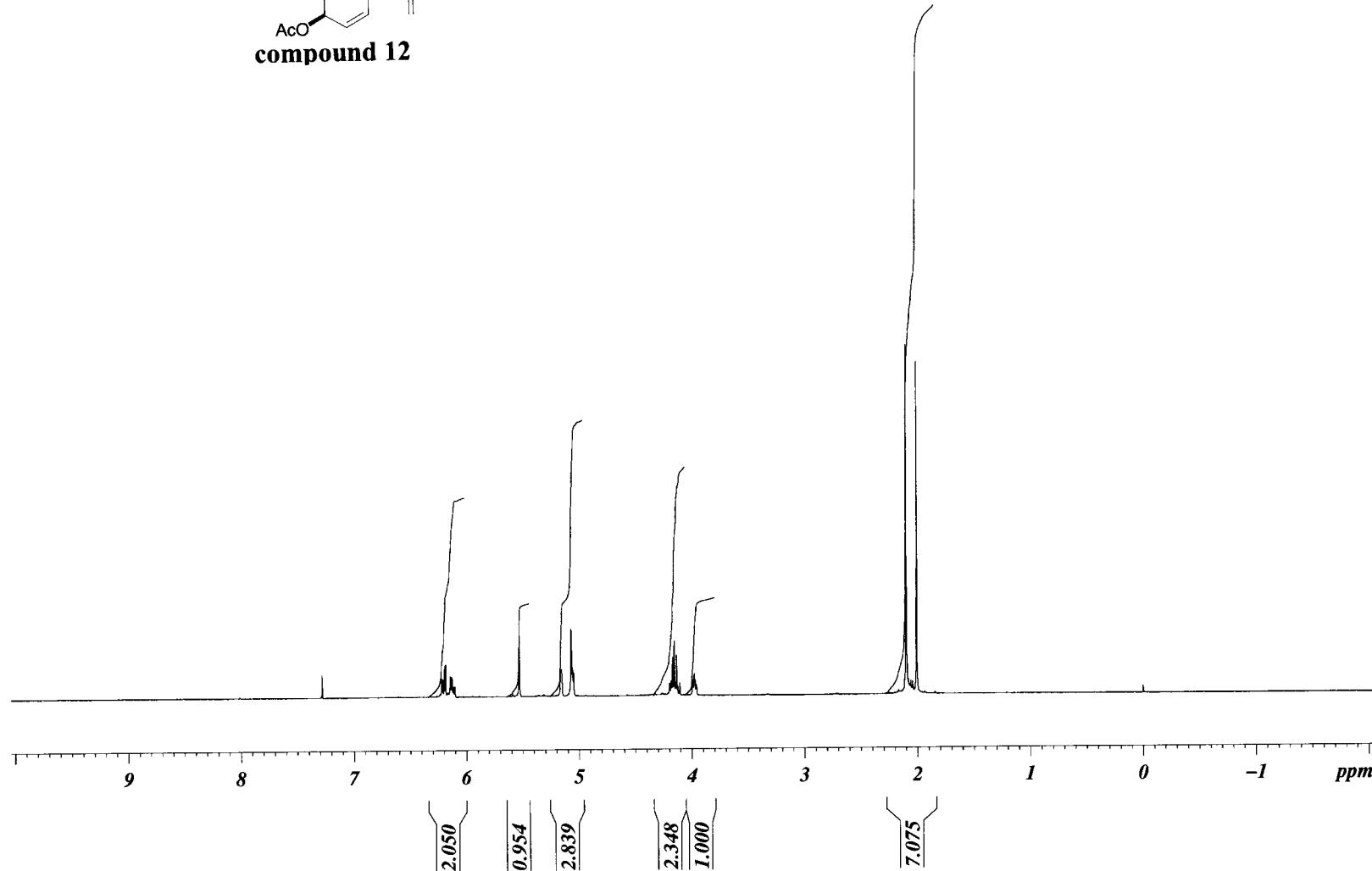
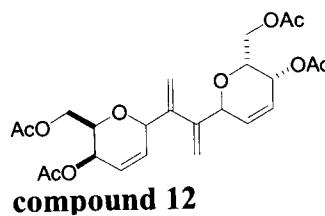
Remarks :

Date : 2004/06/09 15:54:34

Operator : 繁.Kitamura(789-4169)

DATE	04.06.06
LAMP	No.589
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	
\bar{x}	0.181 deg
$\delta n-1$	0.001 deg
RSD	0.552 %
[α]	23.375 deg
$\delta n-1$	0.1840 deg
TEMP	27

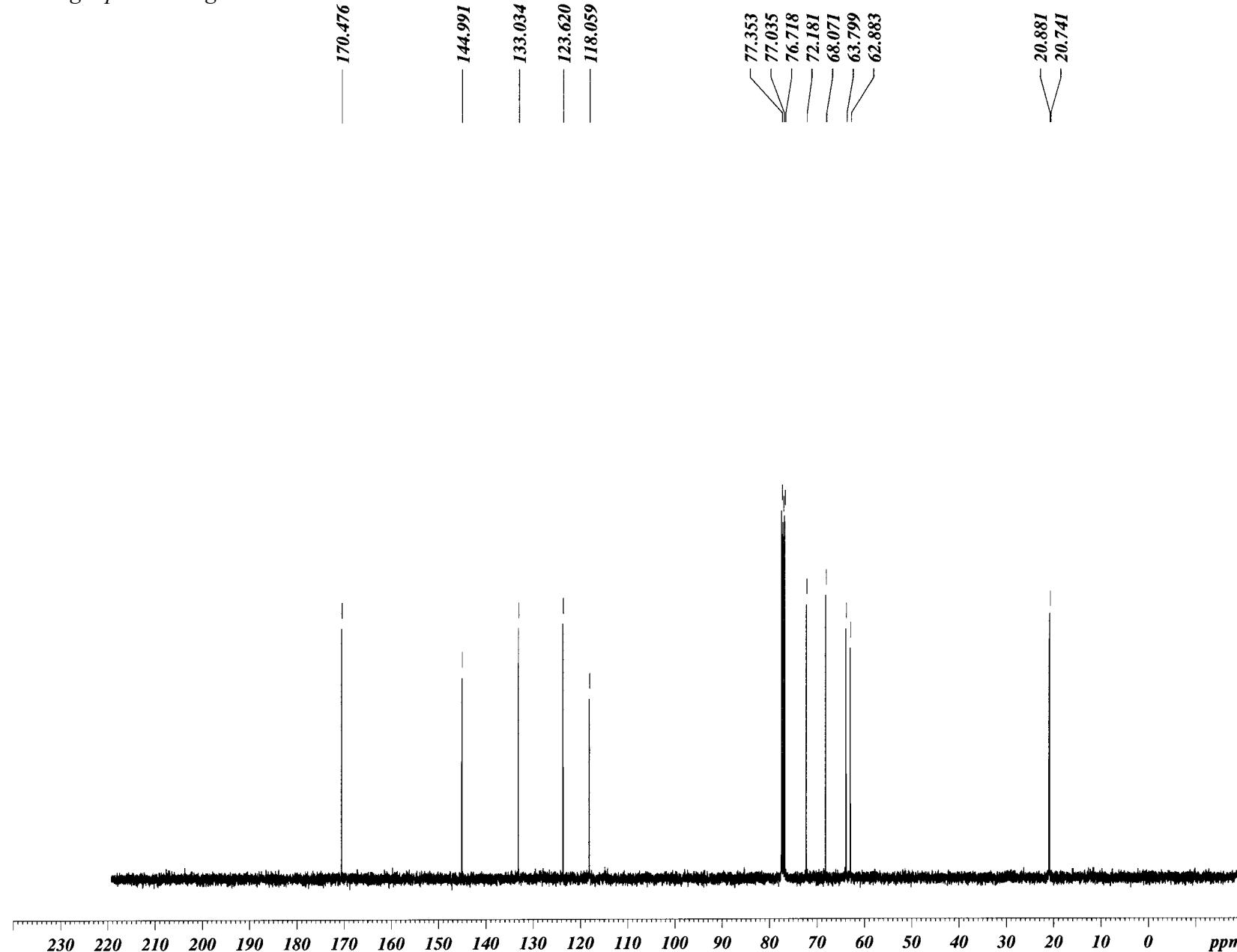
Current Data Parameters
 NAME Rungnapha
 EXPNO 130
 PROCNO 1



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
 DI 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 IH
 PI 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

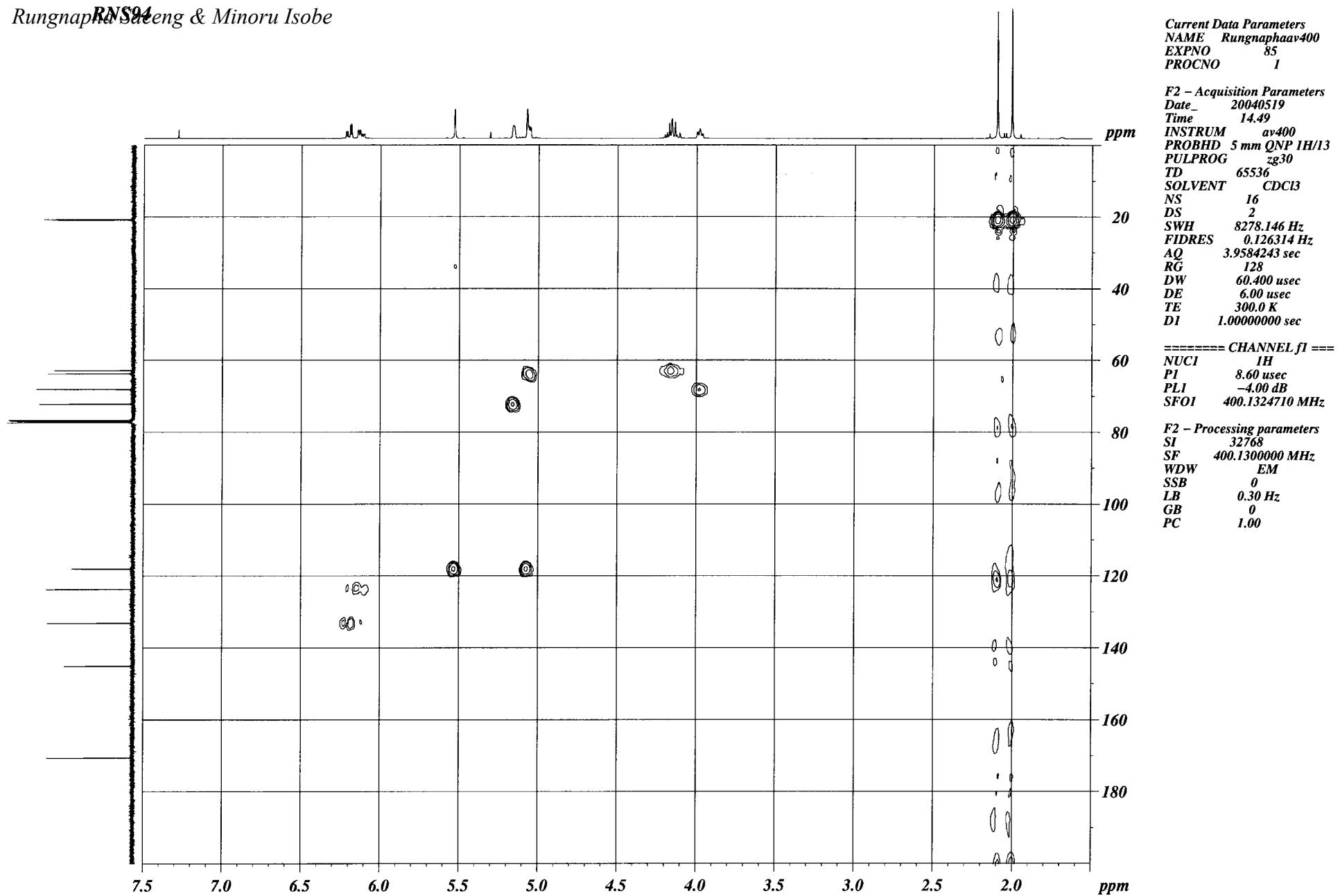
Date 20040519
Time 15.00
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 124
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

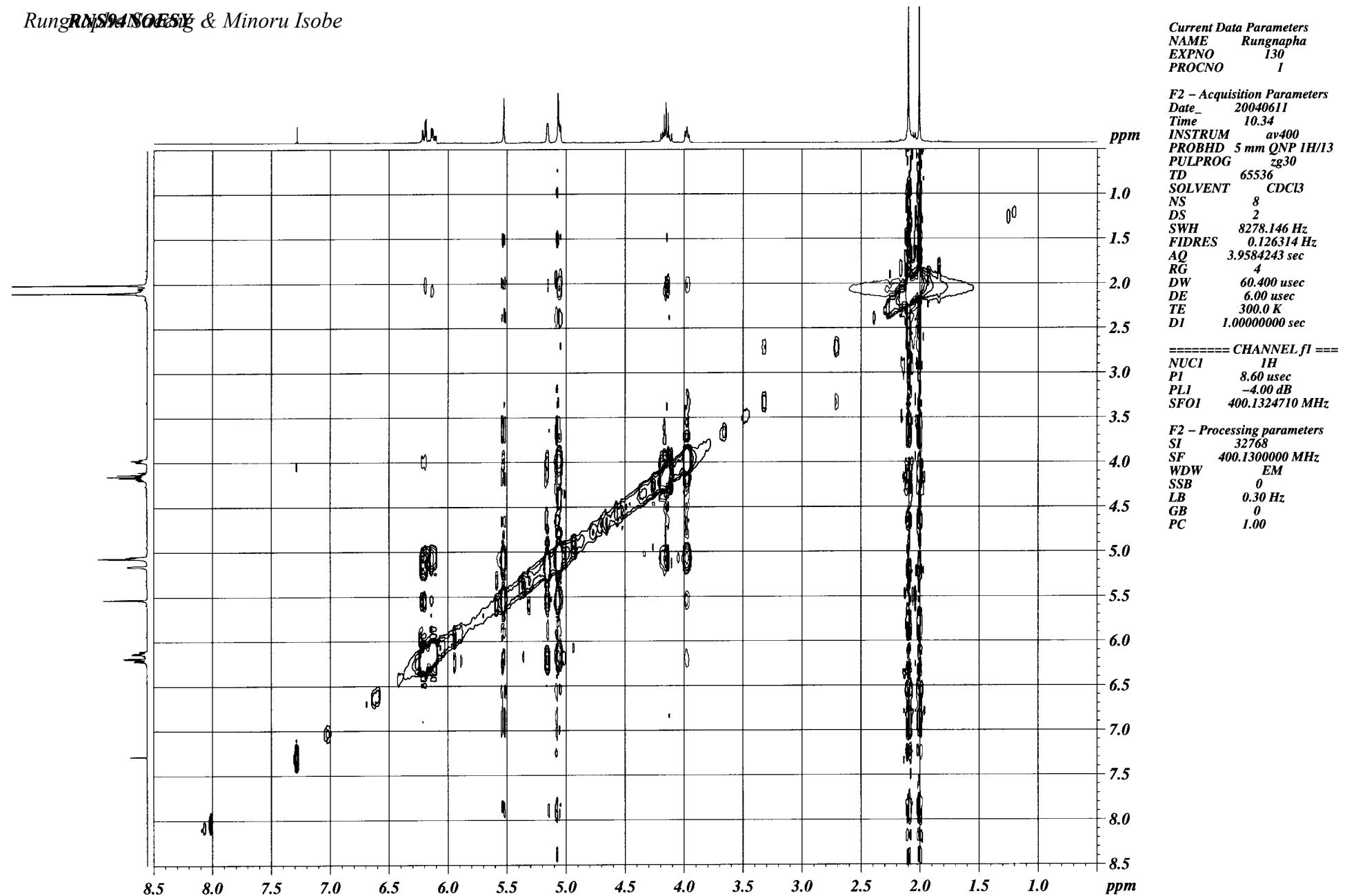
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.80 dB
PL13 14.80 dB
SFO2 400.1316005 MHz

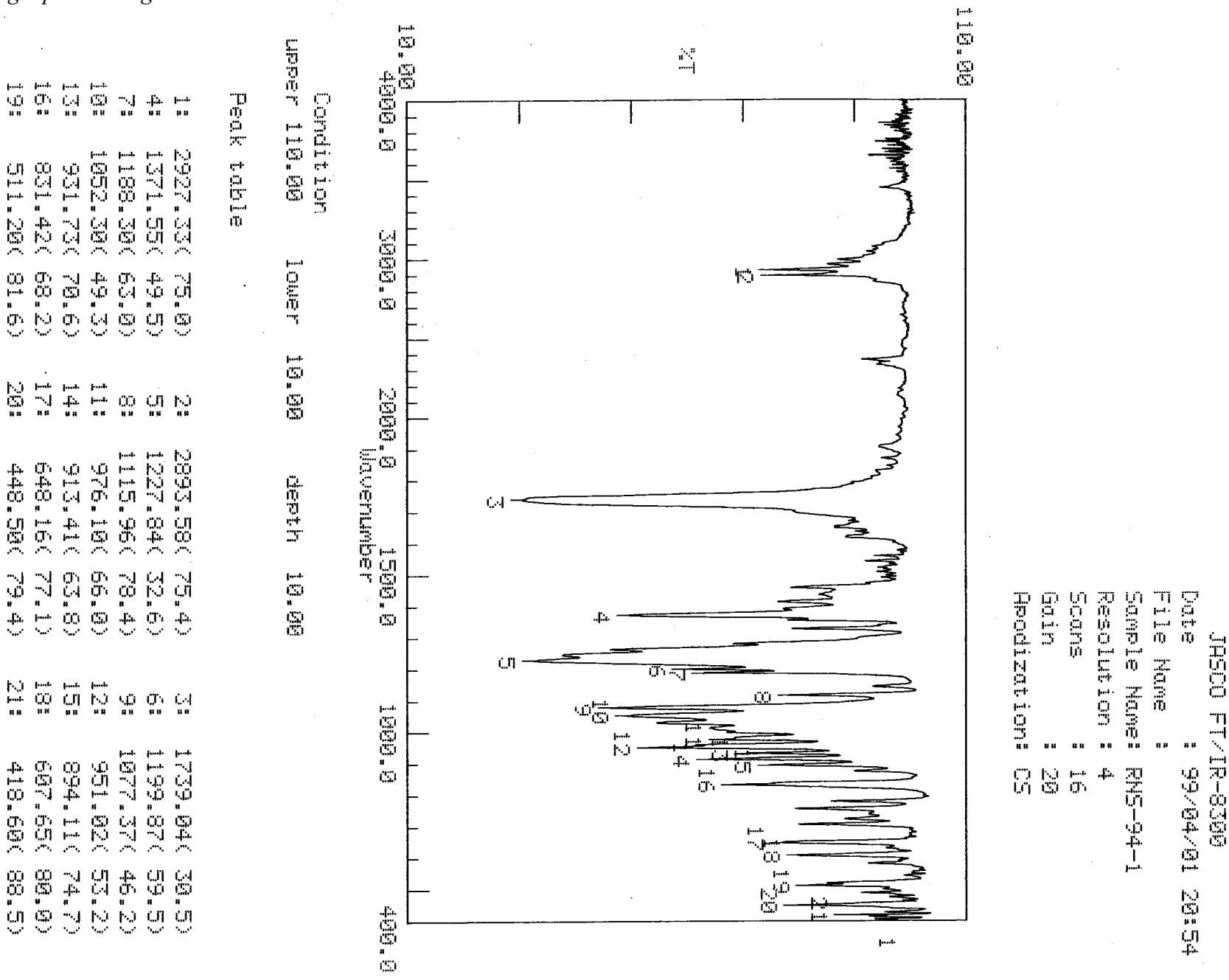
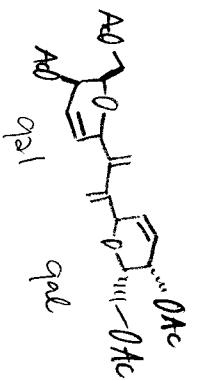
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID 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



Current Data Parameters
 NAME Rungnapha
 EXPNO 130
 PROCNO 1





File: RNS-94-1-1

Date Run: 2004-06-01 (Time Run: 13:35:45)

Run: 1 Sample Description: *mu Isobe*
Instrument: JEOL LCmate

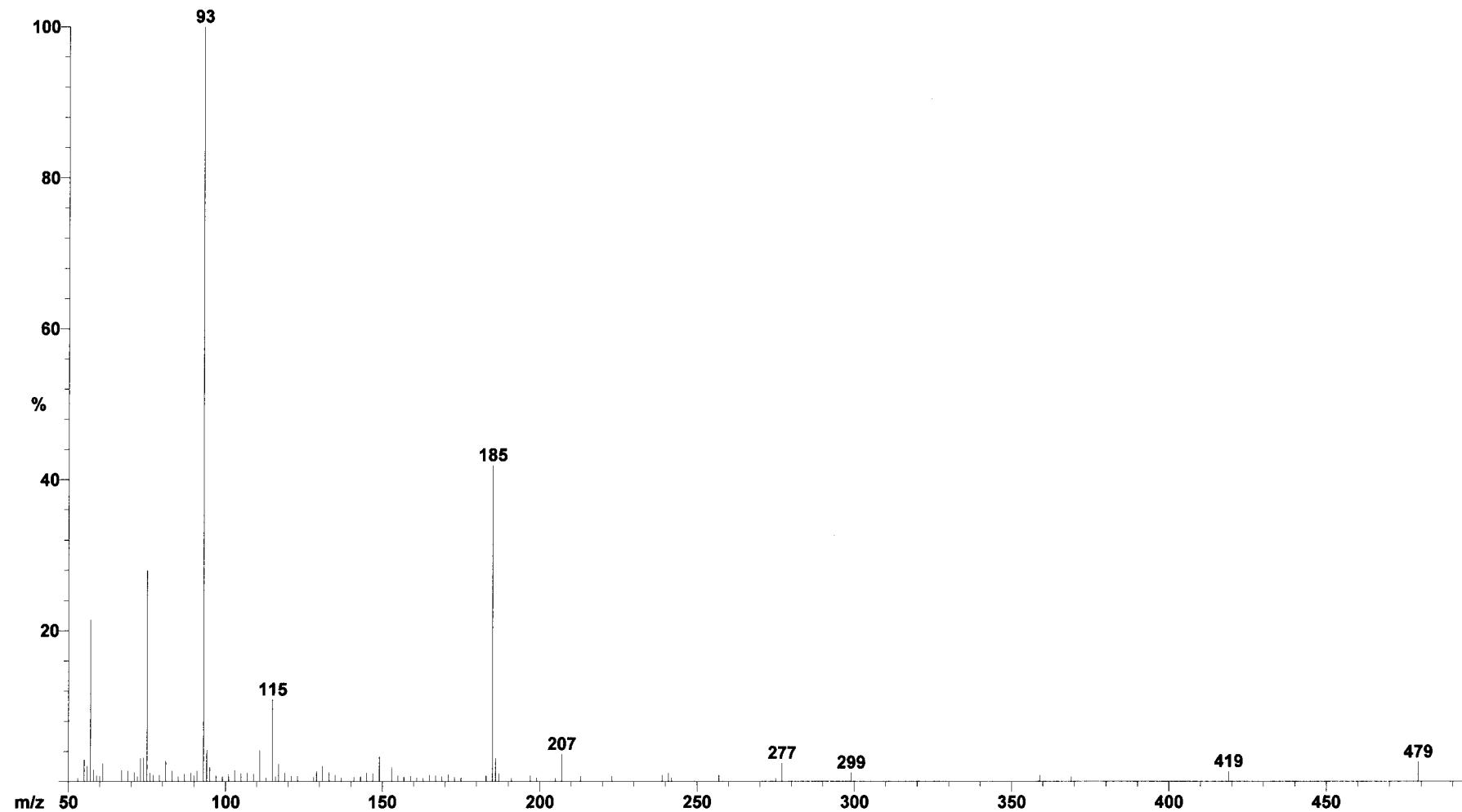
Ionization mode: FAB+

Scan: 1

Base: m/z 93; 12.7%FS TIC: 410528

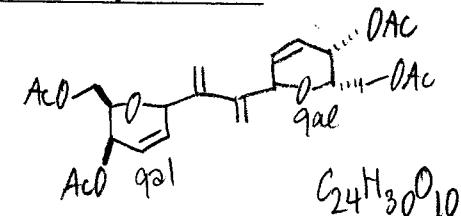
R.T.: .02

#Ions: 89



Elemental Analysis Report

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



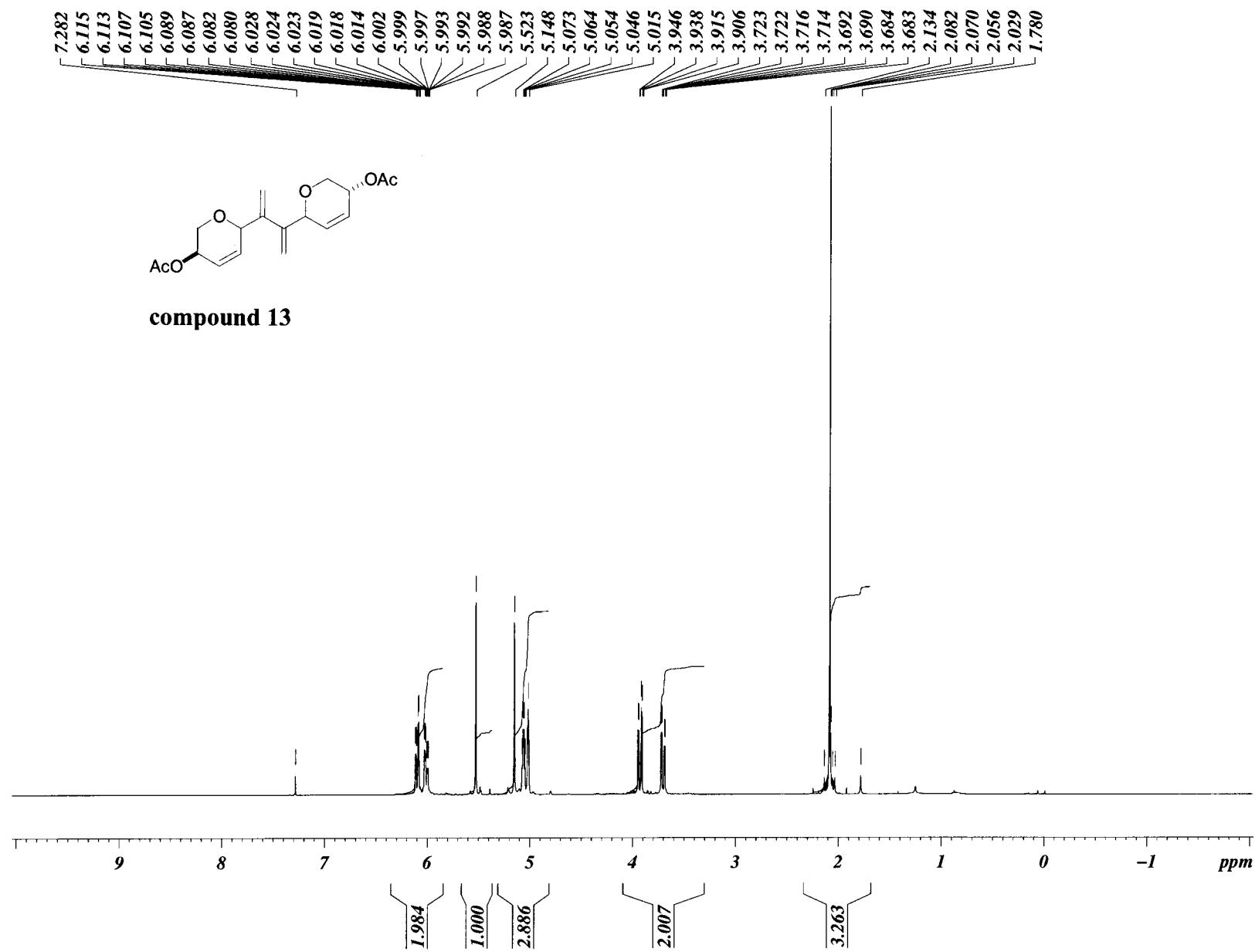
	N	C	H
Calculated weight %	0.00	60.24	6.32
Found weight %	0.00	60.24	6.24
Element ratio(1)	0	13	16
Element ratio(2)	0	17	21
Element ratio(3)	0	30	37
Element ratio(4)	0	47	58
Element ratio(5)	0	64	79
Element ratio(6)	0	77	95
Element ratio(7)	0	81	100

Remarks :

Date : 2004/06/09 15:54:37

Operator : 繁.Kitamura(789-4169)

SAMPLE NO.	DATE	RNS-%
017	04.06.06	0.84%
1 d	LAMP	No.589
2 d	TEMP	
3 d	CELL	100mm
4 d	INTEG. TIME	20sec
5 d	*****	*****
6 d	SAMPLE NO.	deg
7 d		
8 d		
9 d		
10 d		
11 d		
12 d		
13 d		
14 d		
15 d		
16 d		
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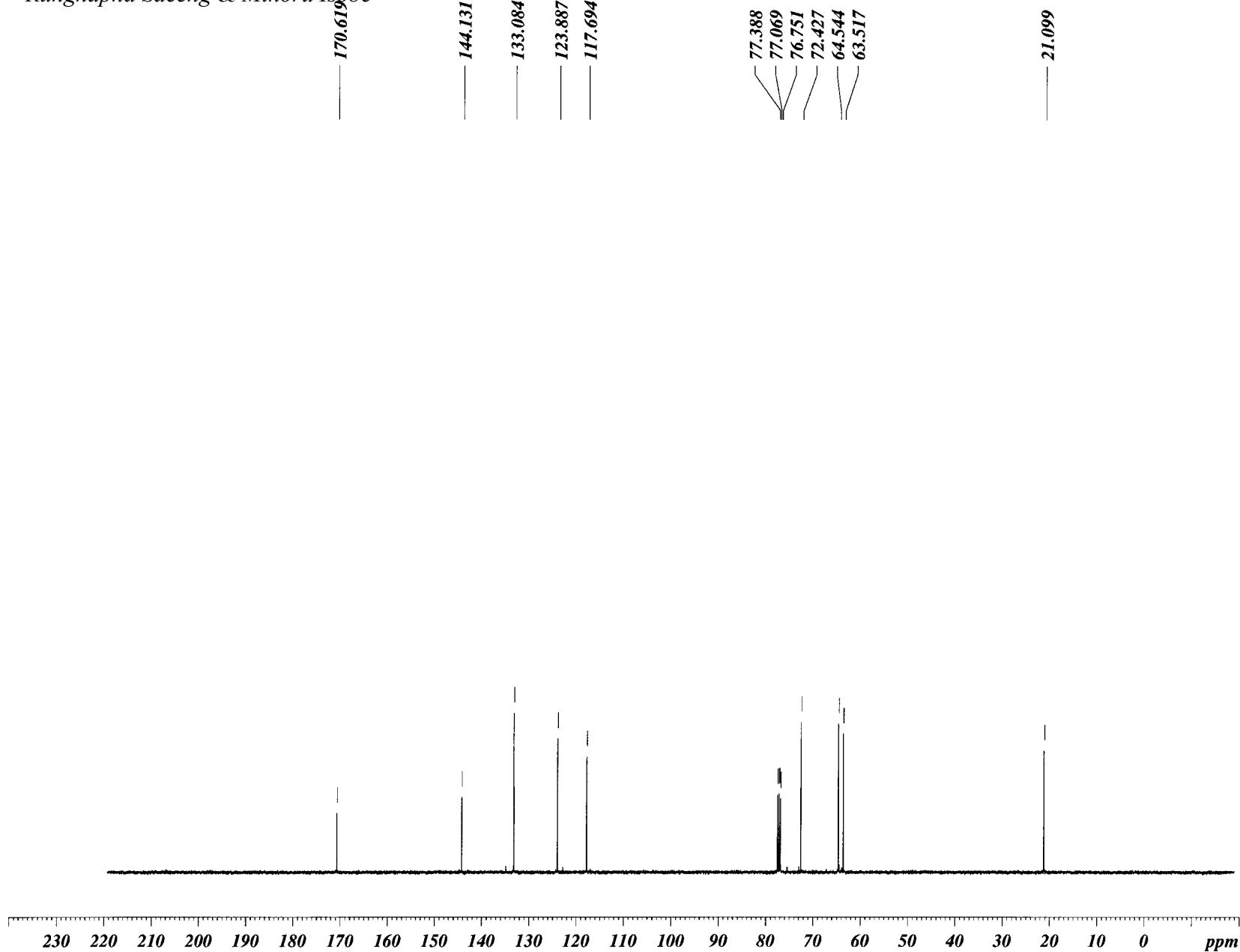


Current Data Parameters
 NAME Rungnaphav400
 EXPNO 88
 PROCNO 1

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 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 PI 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 Rungnapha400
 EXPNO 89
 PROCNO 1

F2 - Acquisition Parameters
 Date 20040519
 Time 15.57
 INSTRUM av400
 PROBHD 5 mm QNP IH/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 2.0000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUCI ¹³C
 PI 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

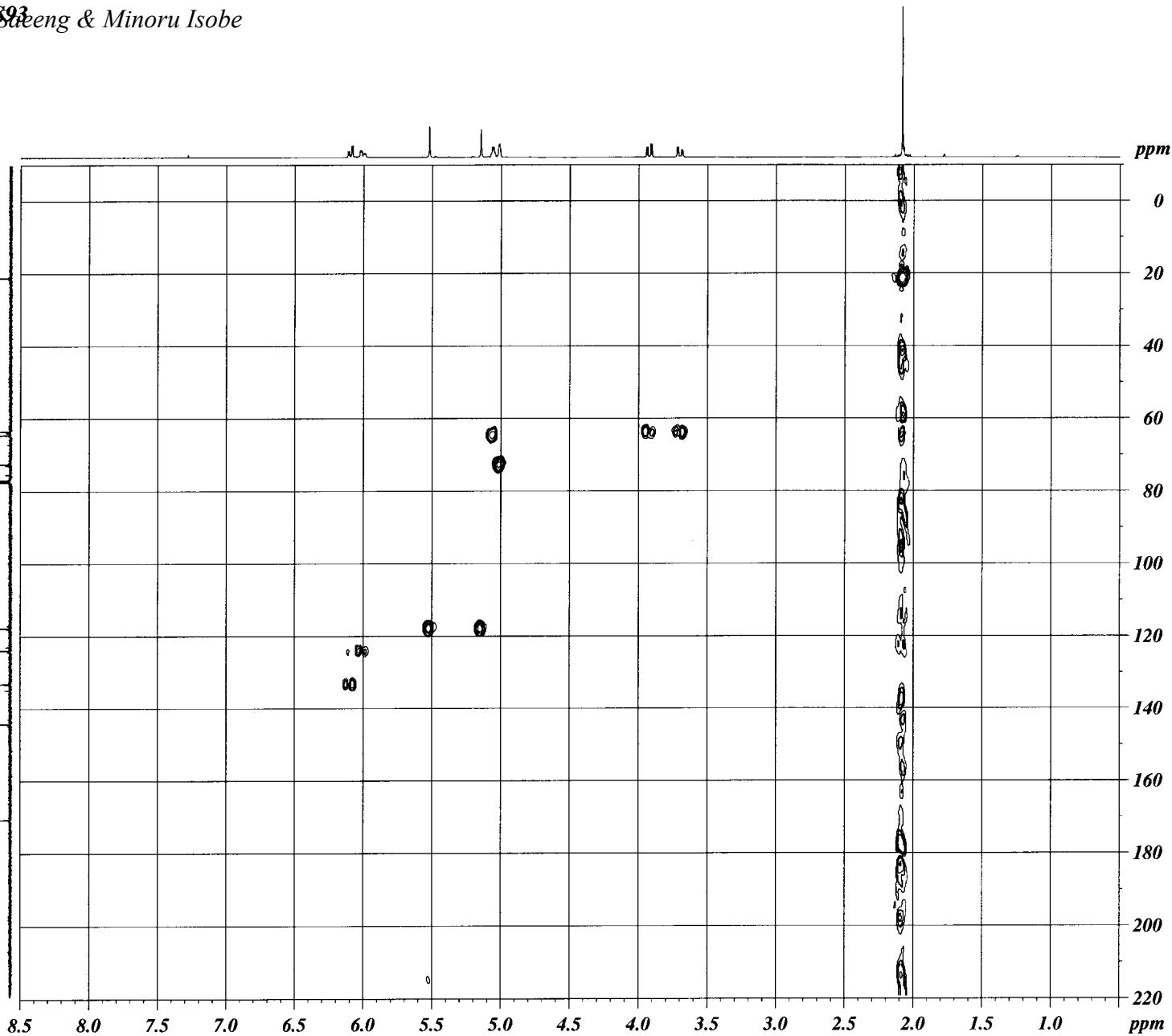
Current Data Parameters
 NAME Rungnapha400
 EXPNO 88
 PROCNO 1

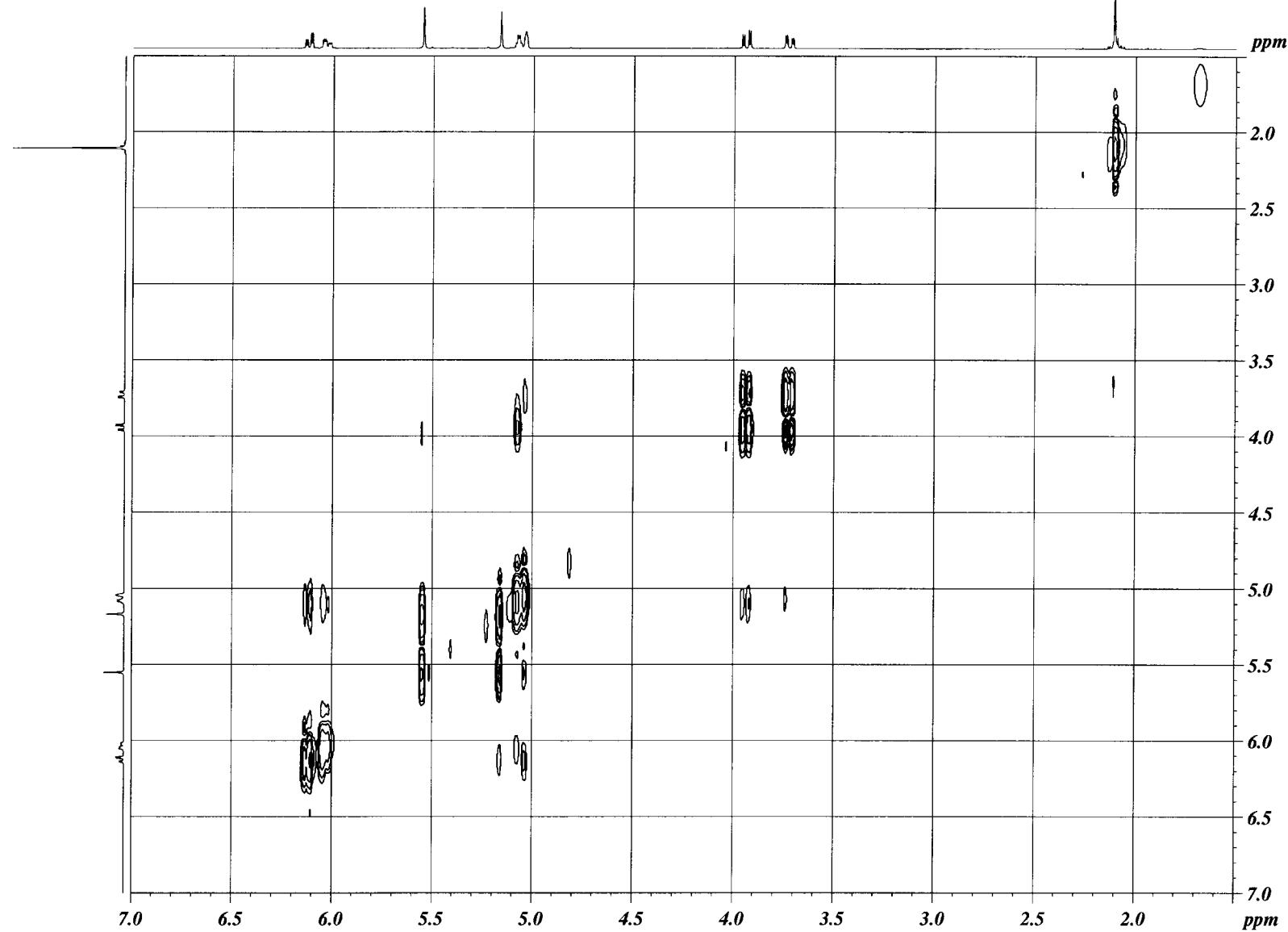
F2 - Acquisition Parameters
 Date 20040519
 Time 15.49

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
 DI 1.0000000 sec

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

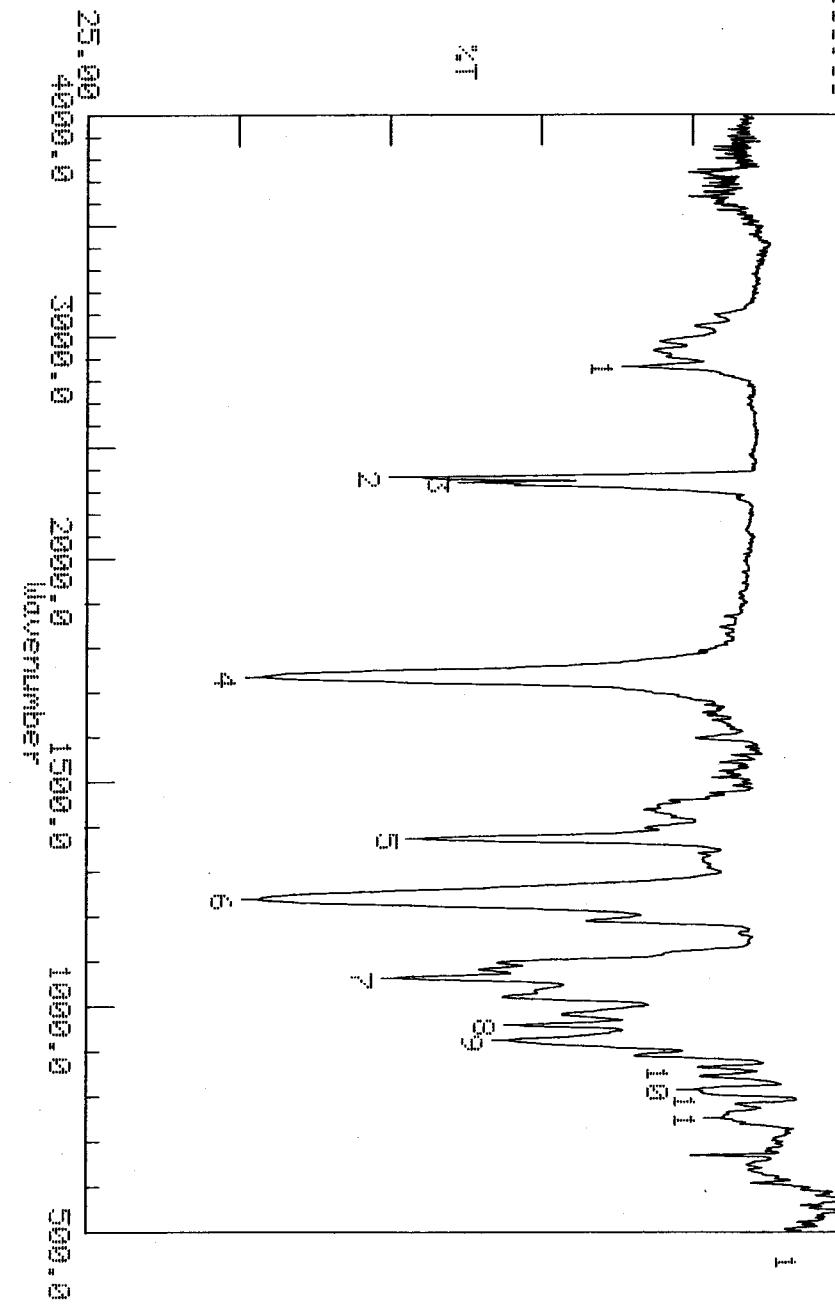
F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



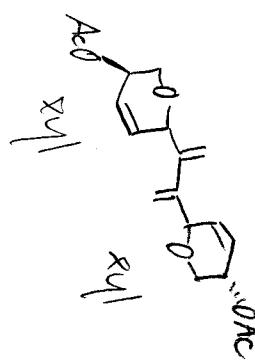


	Condition	Upper 110.00	Lower 25.00	depth 7.00	
1:	2861.75(87.0)	2:	2364.65(60.9)	3:	2341.87(68.7)
4:	1733.25(44.7)	5:	1372.52(63.8)	6:	1237.49(44.4)
7:	1062.91(60.2)	8:	957.77(73.9)	9:	924.62(72.6)
10:	314.06(93.4)	11:	751.37(96.4)		

Peak table



JP500 FT/IR-9300
Date : 99/04/01 15:37
File Name :
Sample Name : RWS-93
Resolution : 4
Scans : 16
Gain : 20
Apoditization: 05



File: RNS-93

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

Run: Sample: Description: *u Isobe*
Instrument: JEOL LCmate

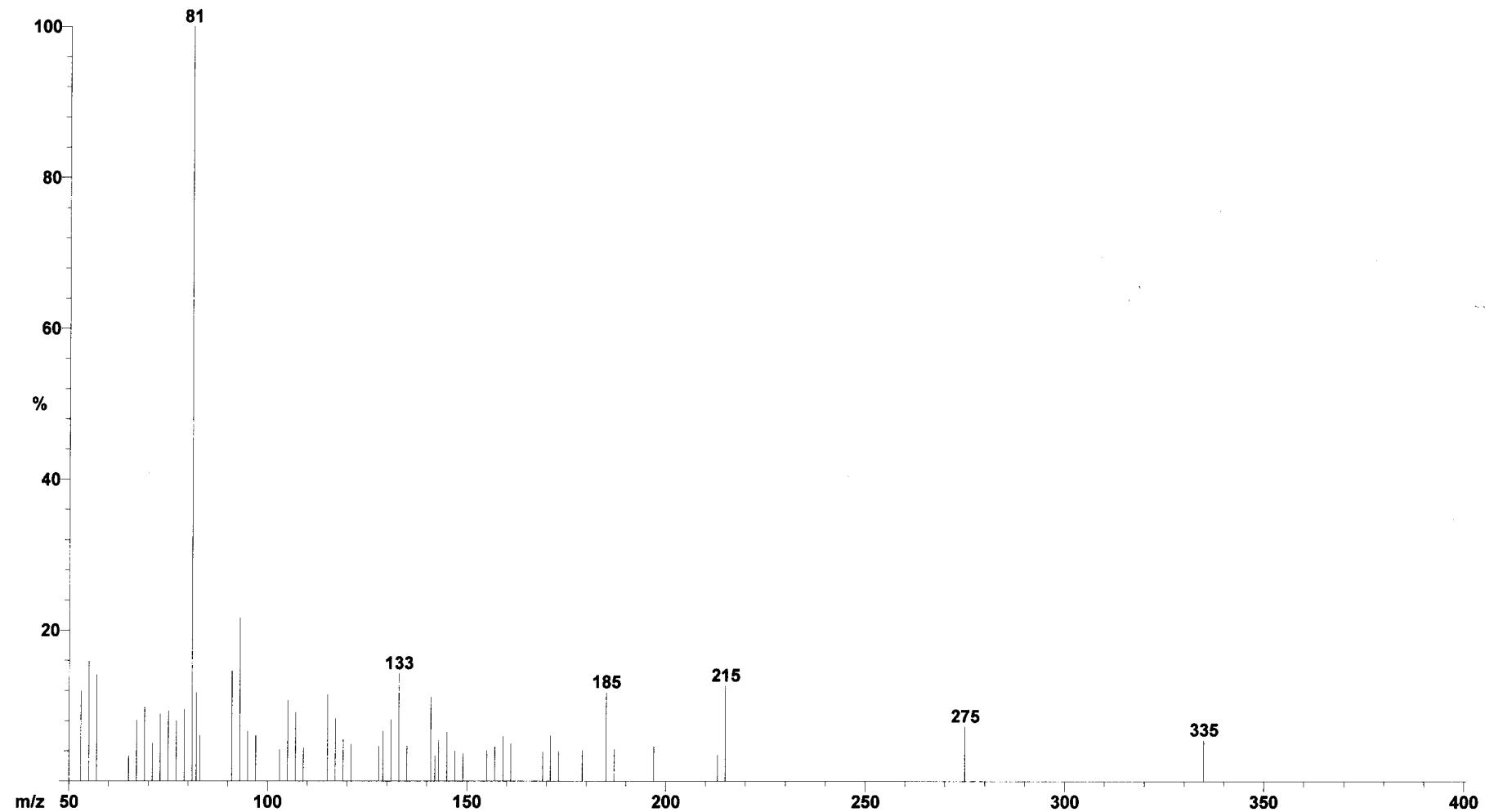
Ionization mode: FAB+

Scan: 2

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

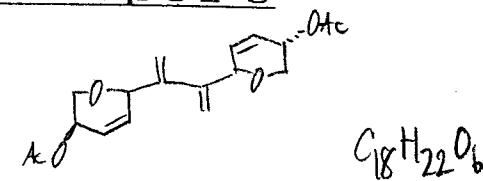
R.T.: .2

#Ions: 53



Elemental Analysis Report

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	04.06.86
LAMP	No.589
TEMP	
SAMPLE	XNS-G3
CONC.	0.98%
CELL	100mm
INTEG. TIME	20sec

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

MEAN VALUE N=10

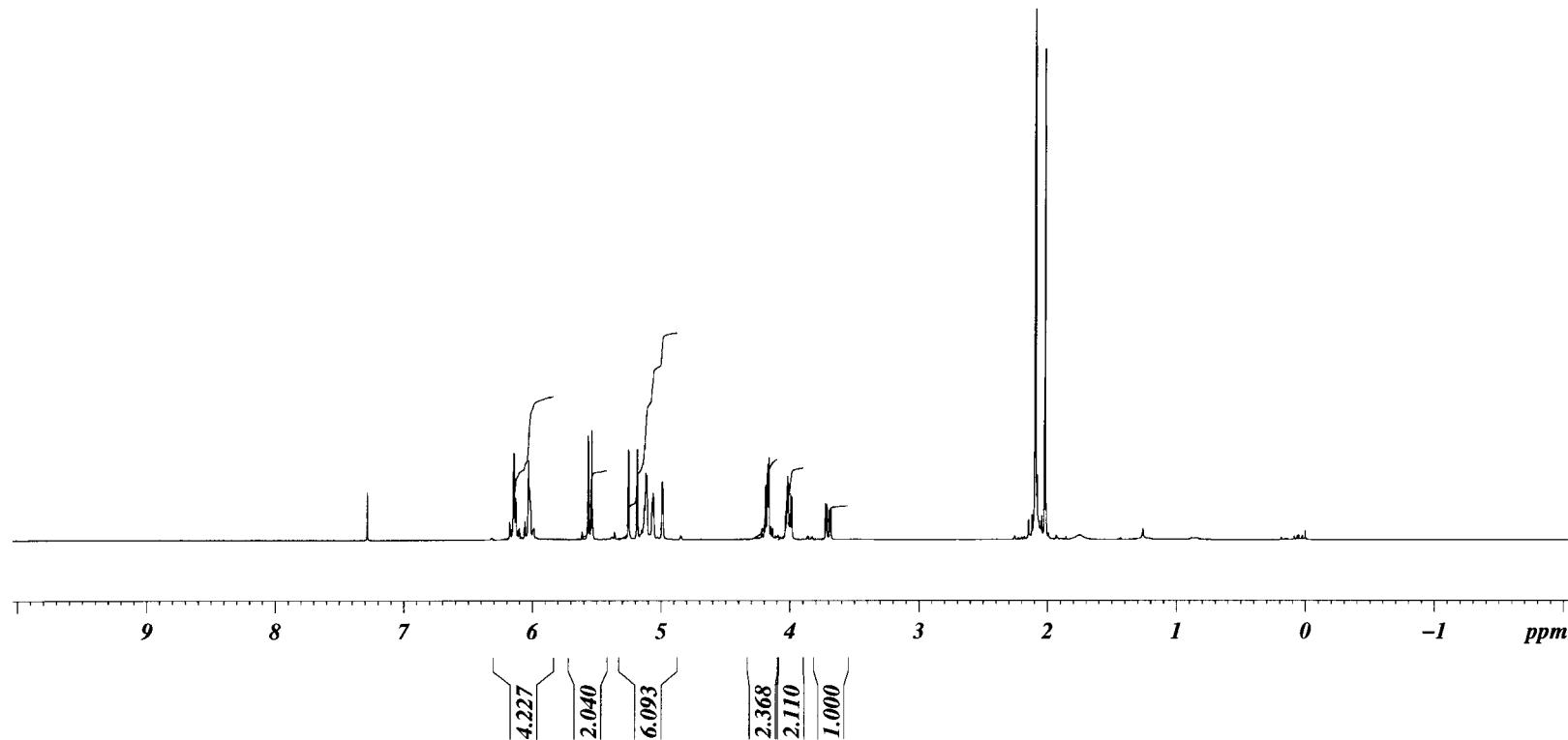
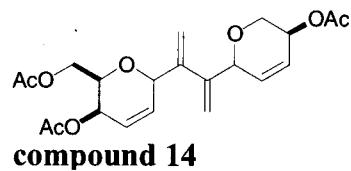
$\bar{\alpha}$ 2.851 deg

$\sigma_{\bar{\alpha}}$ 0.001 deg

RSD 0.035 %

[α] 290.94 deg
 σ_{α} 0.1300 deg

Temp 26

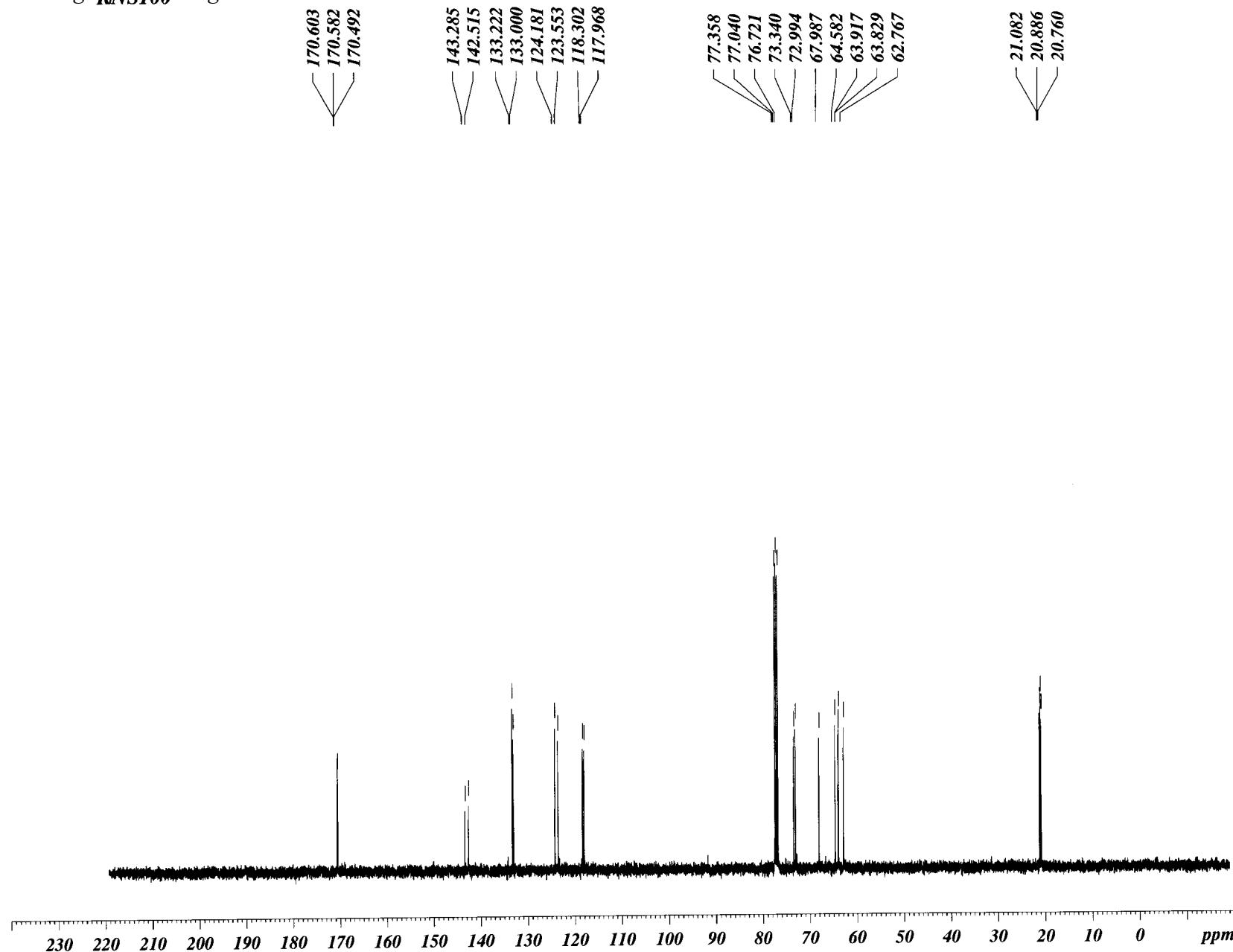


Current Data Parameters
 NAME Rungnaphavav400
 EXPNO 105
 PROCNO 1

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
 DI 1.0000000 sec

===== CHANNEL f1 =====
 NUCI 1H
 PI 8.60 usec
 PLI -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 2.0000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUCI ¹³C
PI 9.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPGR2 waltz16
NUC2 ^{1H}
PCPD2 80.00 usec
PL2 -4.00 dB
PLI2 14.80 dB
PLI3 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 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
 DI 1.0000000 sec

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

NUC1 1H
 PI 8.60 usec
 PL1 -4.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.1300000 MHz

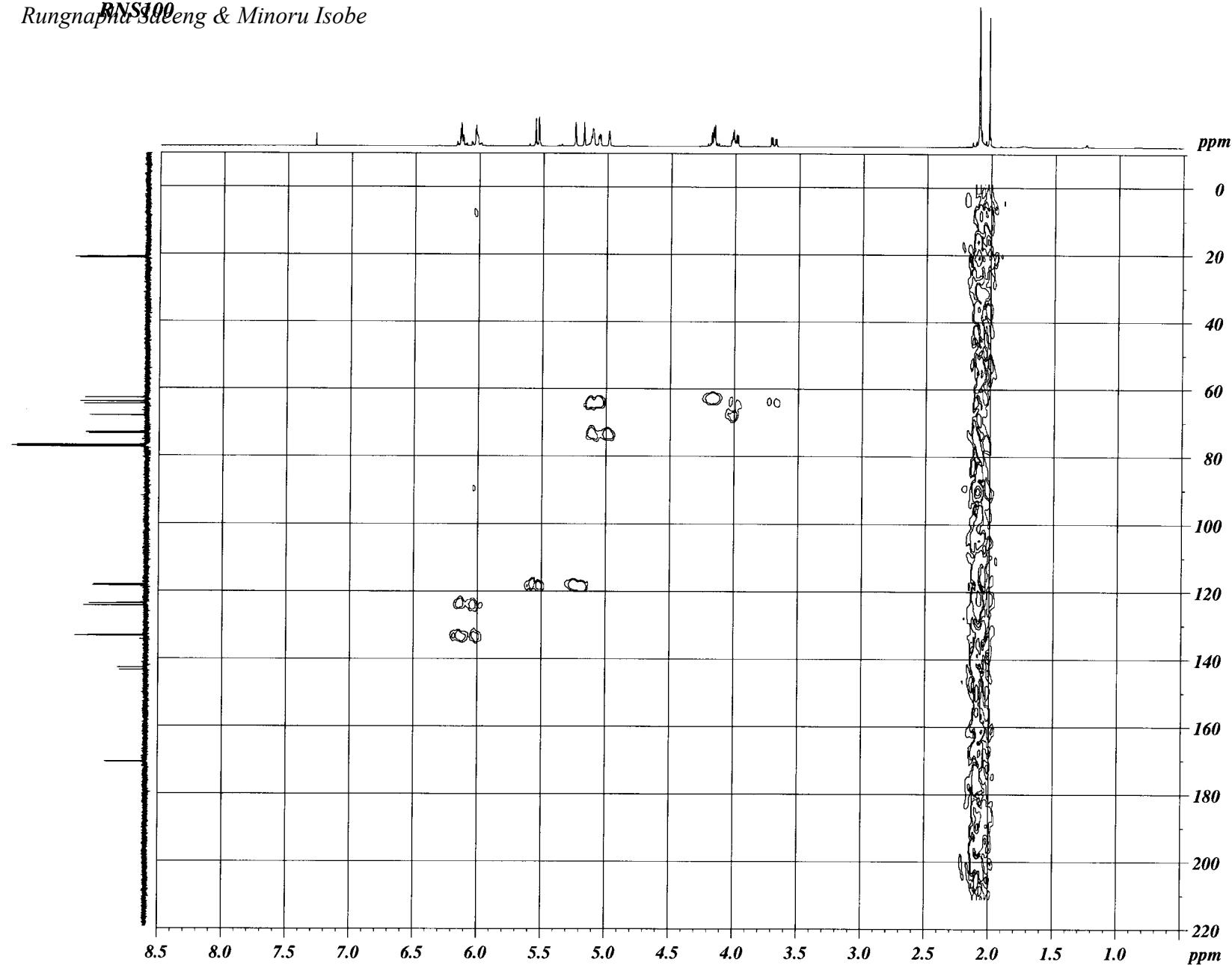
WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00



COSY

Rungnaphavav400eng & Minoru Isobe

Current Data Parameters
NAME Rungnaphav400
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

DI 1.0000000 sec

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

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

F2 - Processing parameters

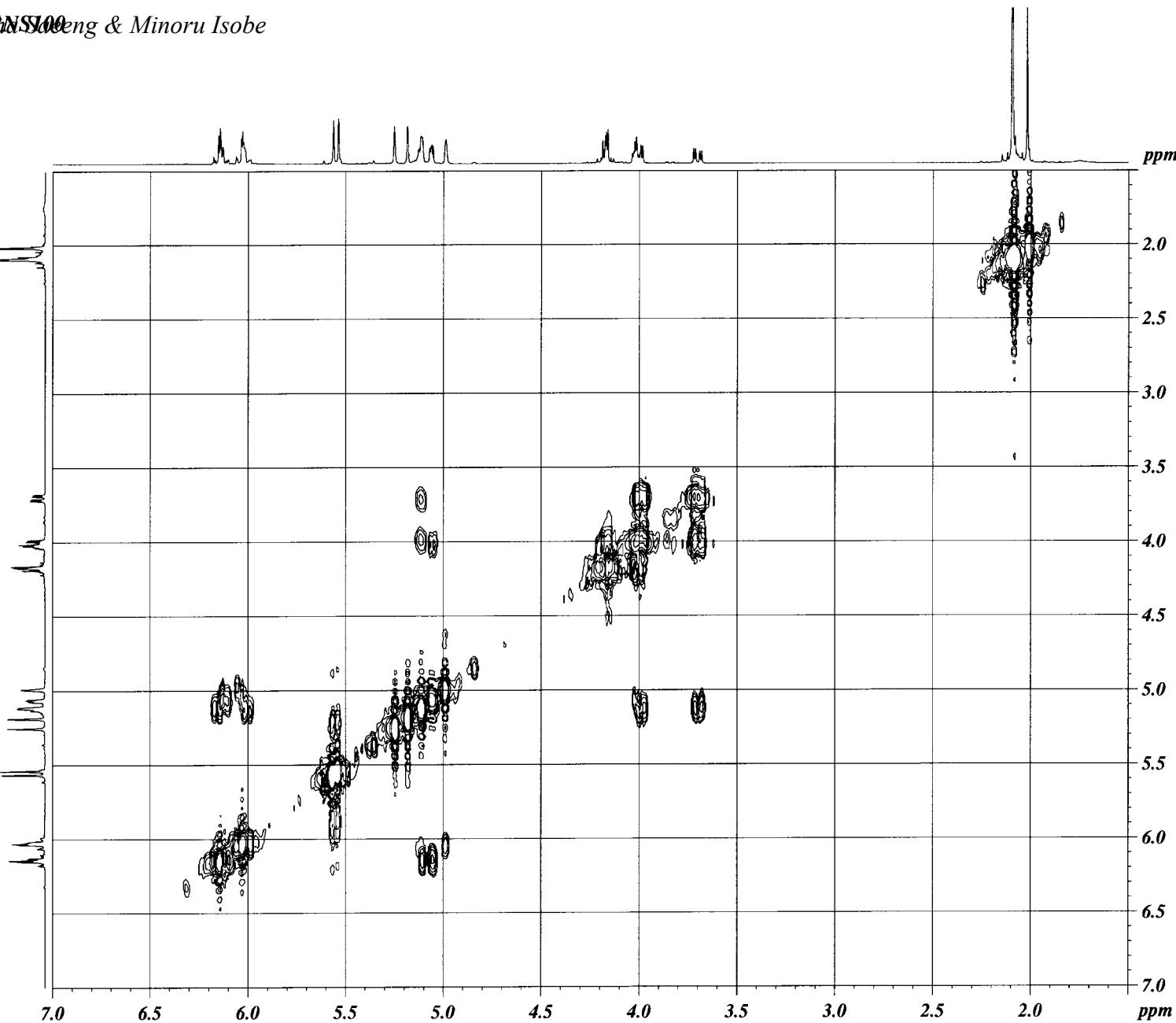
SI 32768

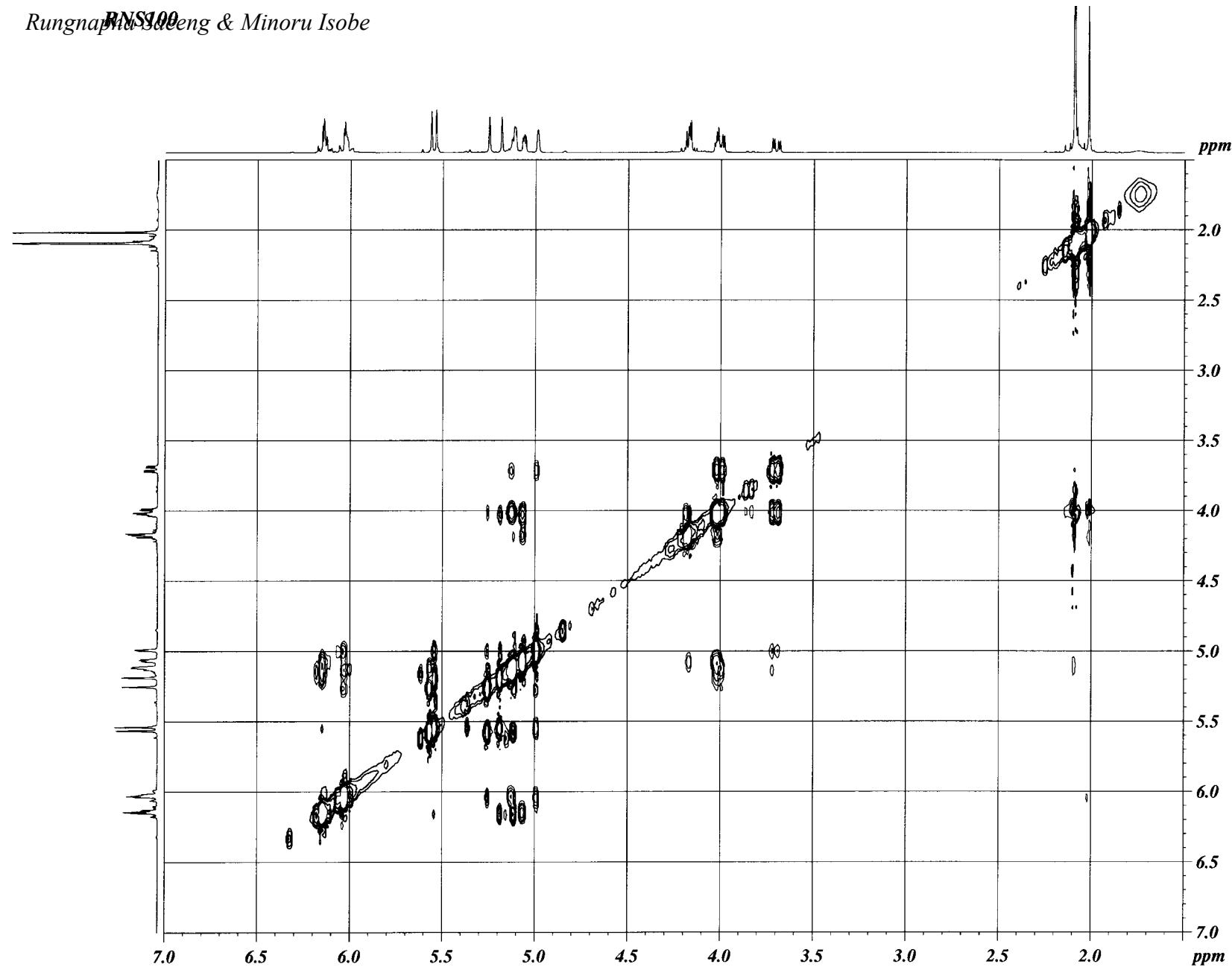
SF 400.1300000 MHz

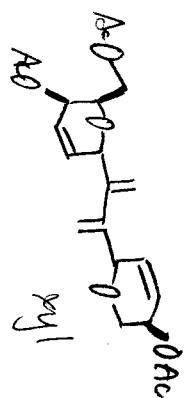
WDW EM
SSB 0

LB 0.30 Hz
GB 0

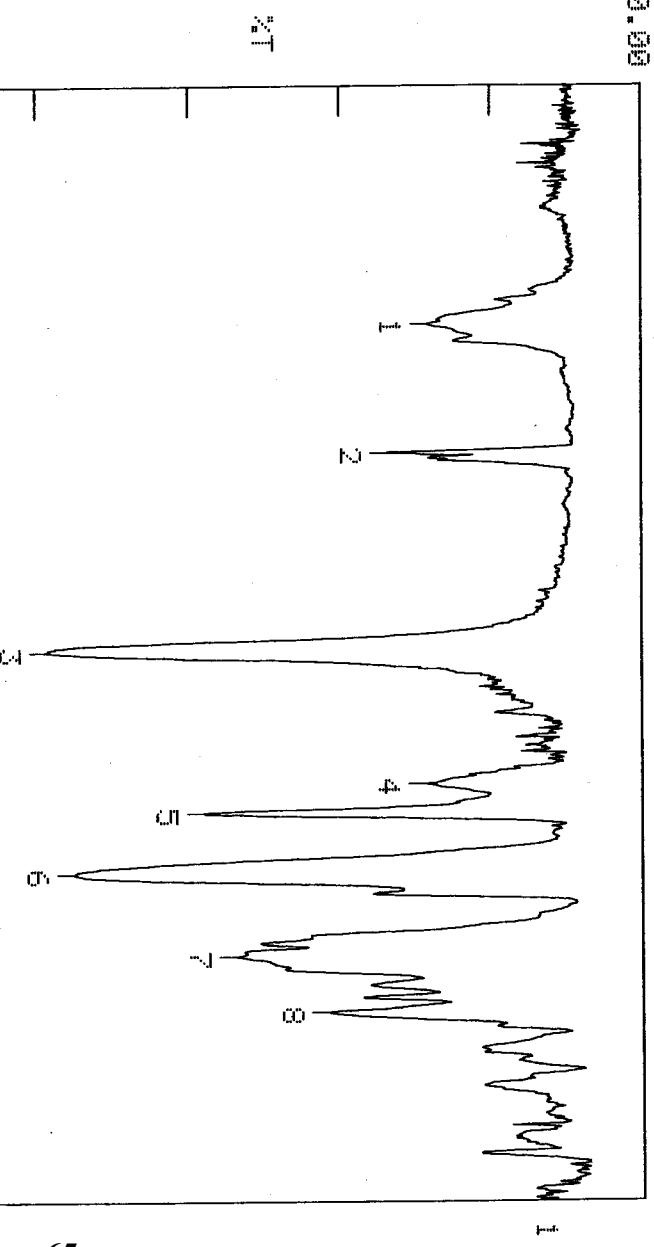
PC 1.00







JASCO FT/IR-8300
Date : 99/04/01 15:53
File Name :
Sample Name: RHS-100
Resolution : 4
Scans : 16
Gain : 20
Averodization: CS



Peak table

	Upper	Lower	5.00	depth	7.00	
1:	2936.98(80.3)		2:	2363.89(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:	924.92(66.4)		

File: RNS-100

Run number: 6
Sample Description: *mu Isobe*
Instrument: JEOL LCmate

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

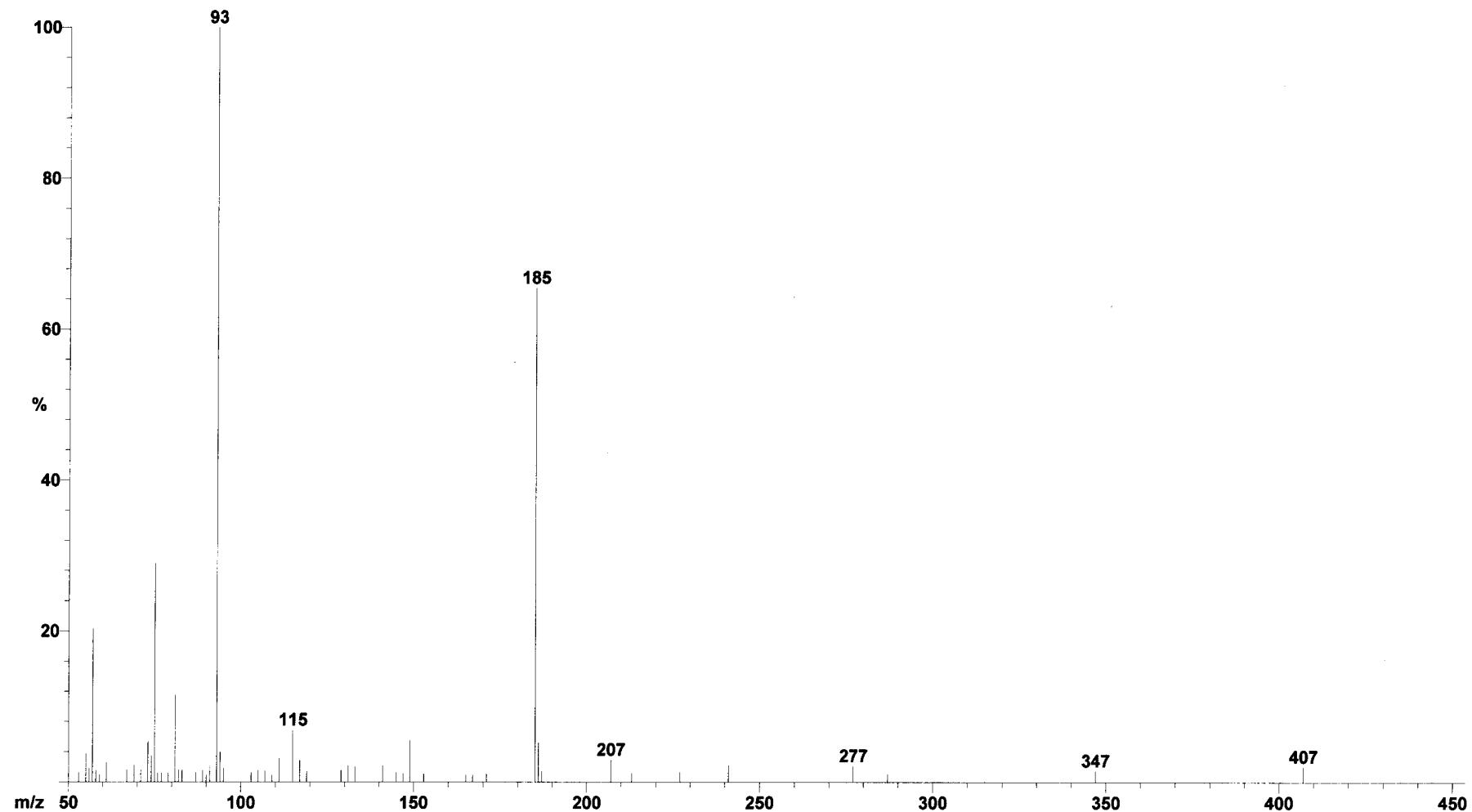
Ionization mode: FAB+

Scan: 3

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

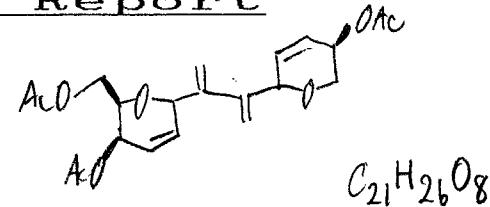
R.T.: .38

#Ions: 56



Elemental Analysis Report

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 No589
TEMP
SAMPLE RIC-100
CONC. 1.175%
CELL 100mm
INTEG. TIME 20sec

SAMPLE NO. deg

014

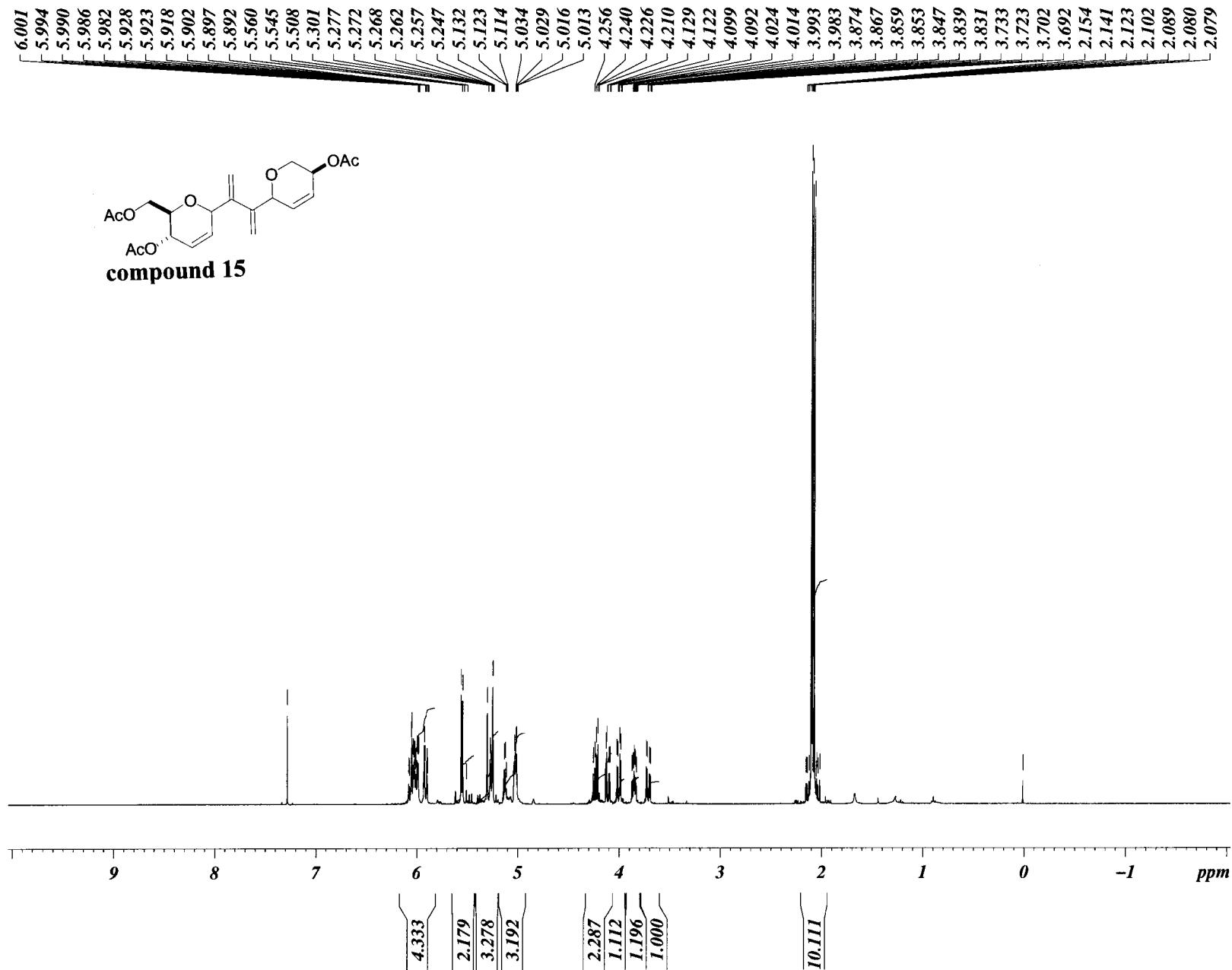
X1 d -0.550
[α]-46.863
2 d -0.556
[α]-47.378
3 d -0.554
[α]-47.203
4 d -0.554
[α]-47.180
5 d -0.553
[α]-47.098
6 d -0.555
[α]-47.261
7 d -0.556
[α]-47.349
8 d -0.556
[α]-47.389
9 d -0.555
[α]-47.266
10 d -0.554
[α]-47.219

MEAN VALUE N=10

$\bar{\alpha}$ -0.554 deg
 σ_{α} 0.002 deg
RSD 0.360 %
[α]-47.221 deg
 σ_{α} 0.1557 deg

$$[\alpha] = -47.22$$

TEMP 27

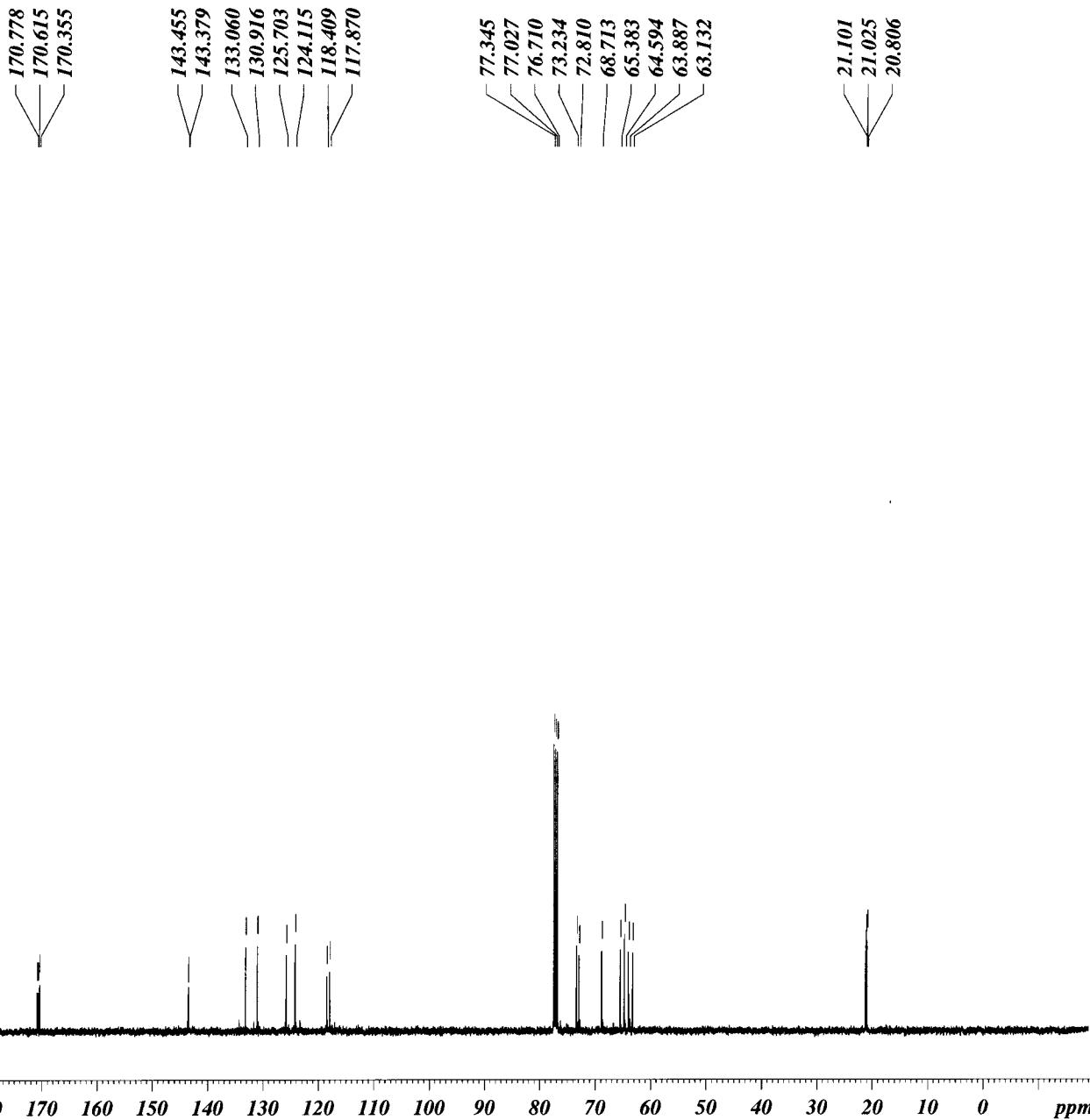


Current Data Parameters
 NAME Rungnaphaeng400
 EXPNO 98
 PROCNO 1

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
 DI 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 IH
 PI 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



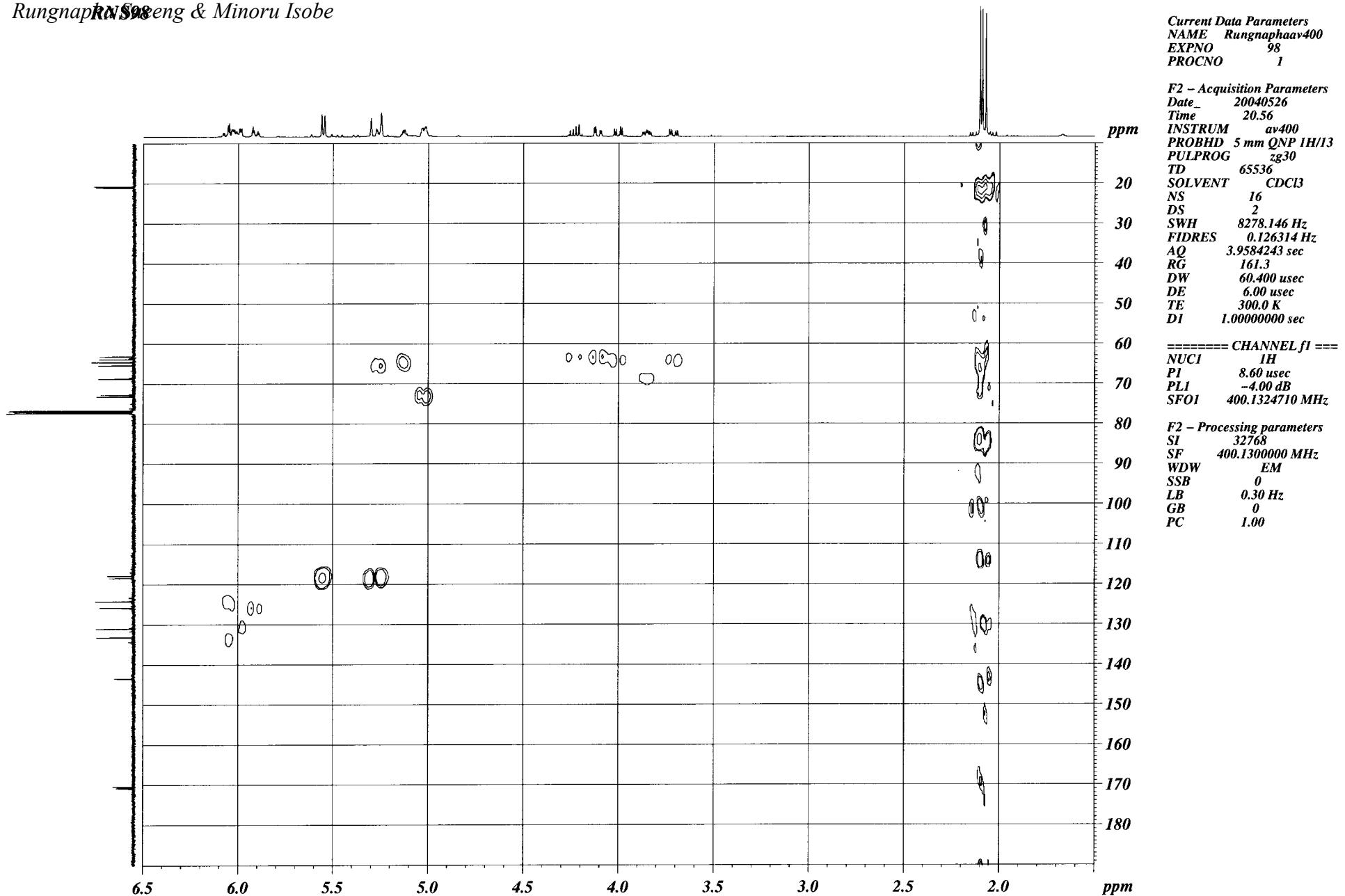
Current Data Parameters
NAME Rungnaphaeng400
EXPNO 99
PROCNO 1

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 2.0000000 sec
d11 0.0300000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
PI 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



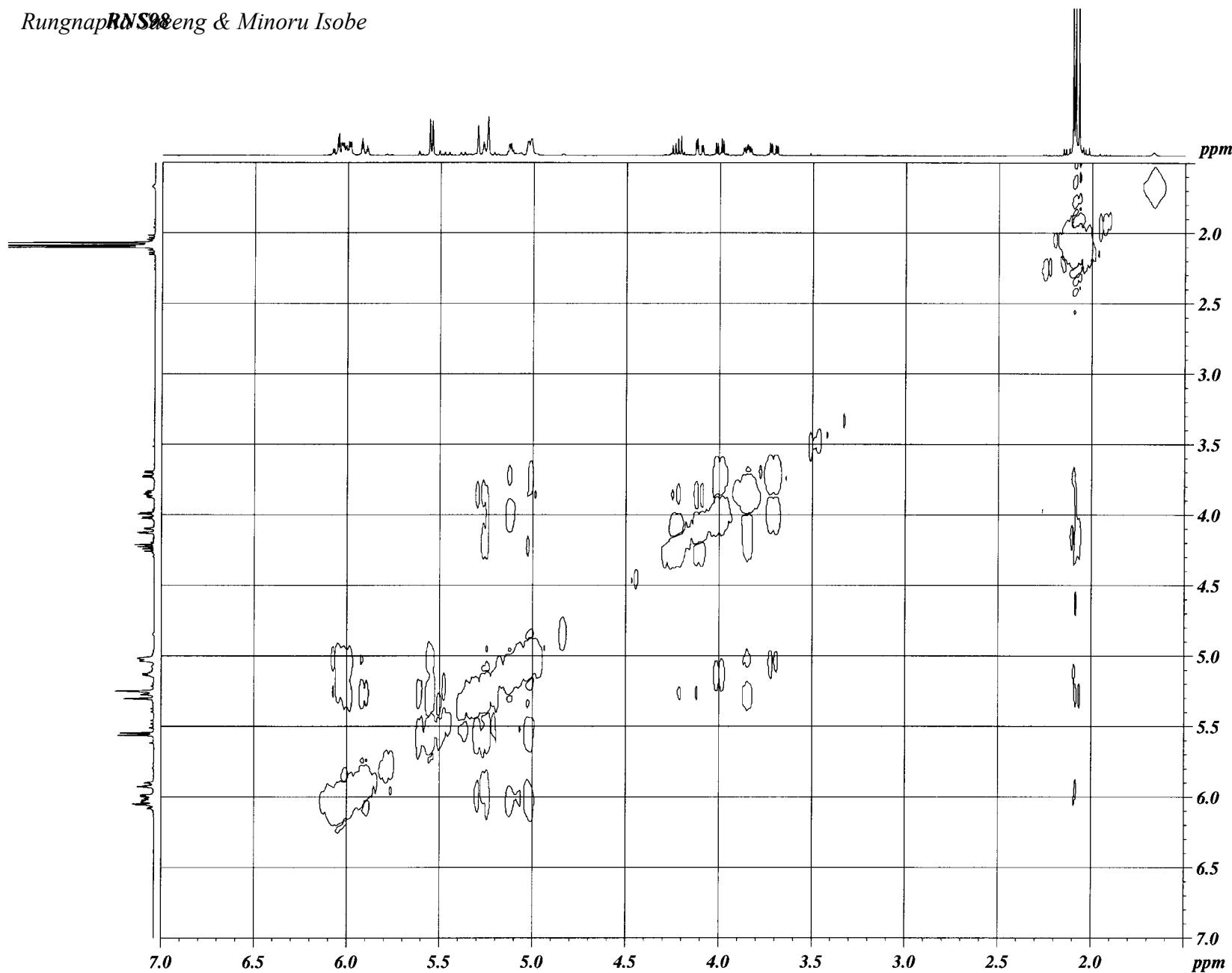
Current Data Parameters
 NAME Rungnaphaaav400
 EXPNO 98
 PROCNO 1

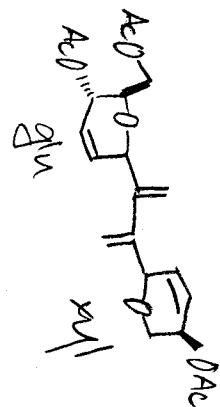
F2 - Acquisition Parameters

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
 DI 1.0000000 sec

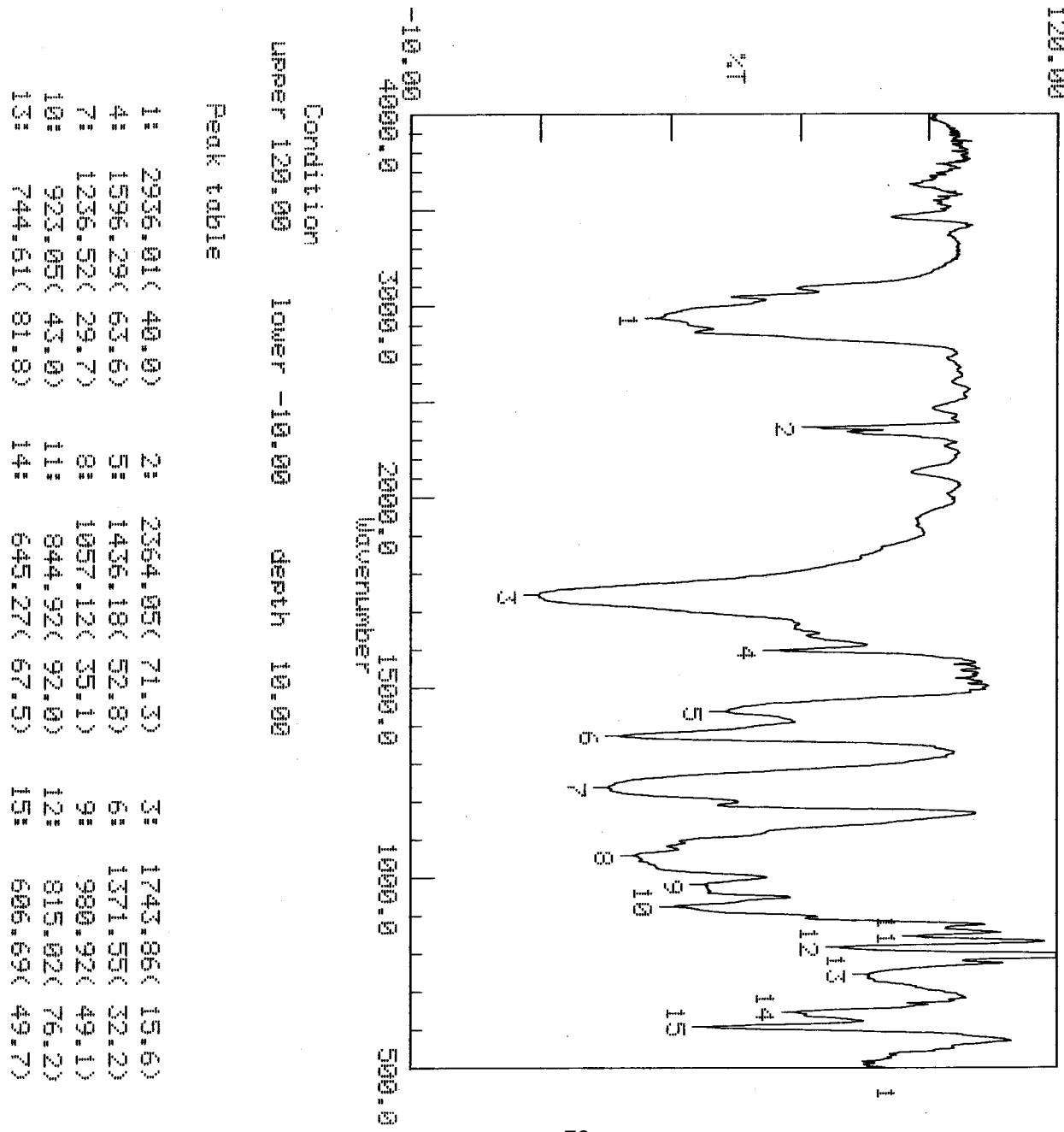
===== CHANNEL f1 =====
 NUCI 1H
 PI 8.60 usec
 PLI -4.00 dB
 SFO1 400.1324710 MHz

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: RHS-98-3
Resolution : 4
Scans : 16
Gain : 50
Apodization: CS



File: RNS-98

Date Run: 2004-05-31 (Time Run: 20:08:41)

Run: Sample Description: *mu Isobe*
Instrument: JEOL LCmate

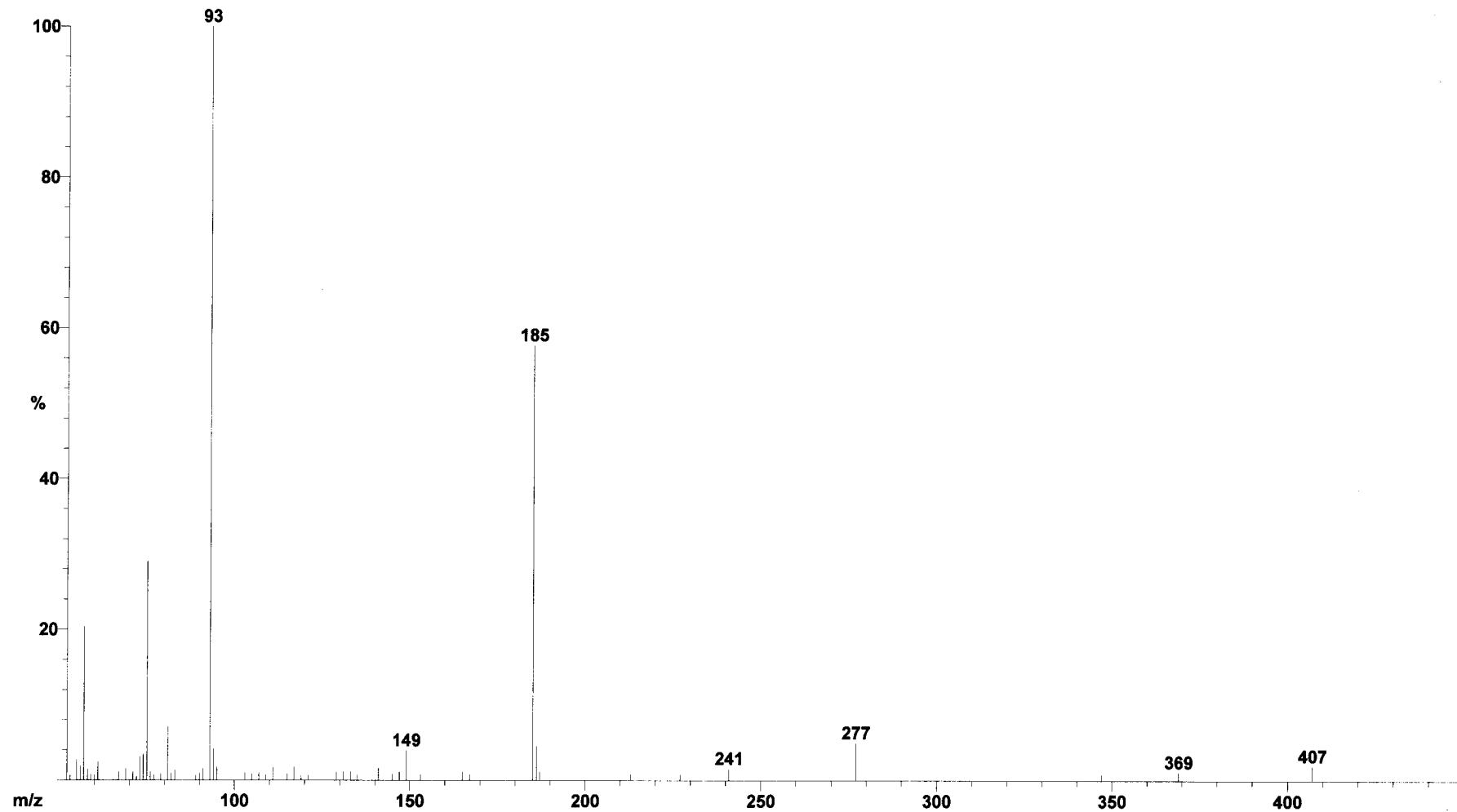
Ionization mode: FAB+

Scan: 1

R.T.: .02

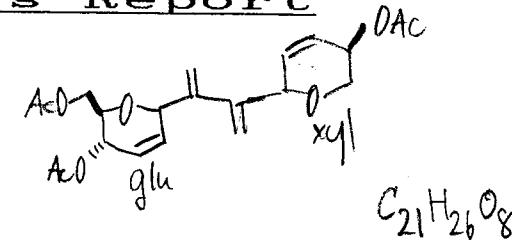
Base: m/z 93; 8.9%FS TIC: 272272

#Ions: 57



Elemental Analysis Report

Reference 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)

SAMPLE NO.	deg	DATE	04.06.04
815		LAMP	No.589
		TEMP	
		SAMPLE	RNS - 97-2
		CONC.	1.215%
		CELL	100mm
		INTEG. TIME	20sec
		*****	*****
		SAMPLE NO.	deg
1	d	1.899	
		[α]	156.38
X	d	1.908	
		[α]	157.09
3	d	1.902	
		[α]	156.61
4	d	1.901	
		[α]	156.47
5	d	1.901	
		[α]	156.51
6	d	1.903	
		[α]	156.68
7	d	1.902	
		[α]	156.55
8	d	1.902	
		[α]	156.55
9	d	1.904	
		[α]	156.72
10	d	1.903	
		[α]	156.70

MEAN VALUE N=10

$\bar{\alpha}$ 1.903 deg

$s_{\bar{\alpha}}$ 0.002 deg

RSD 0.105 %

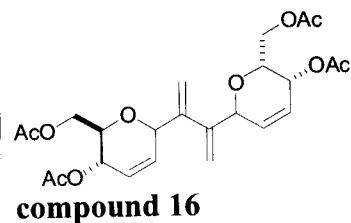
[α] 156.62 deg

s_{α} 0.2072 deg

TEMP 27

$$[\alpha] = 156.57$$

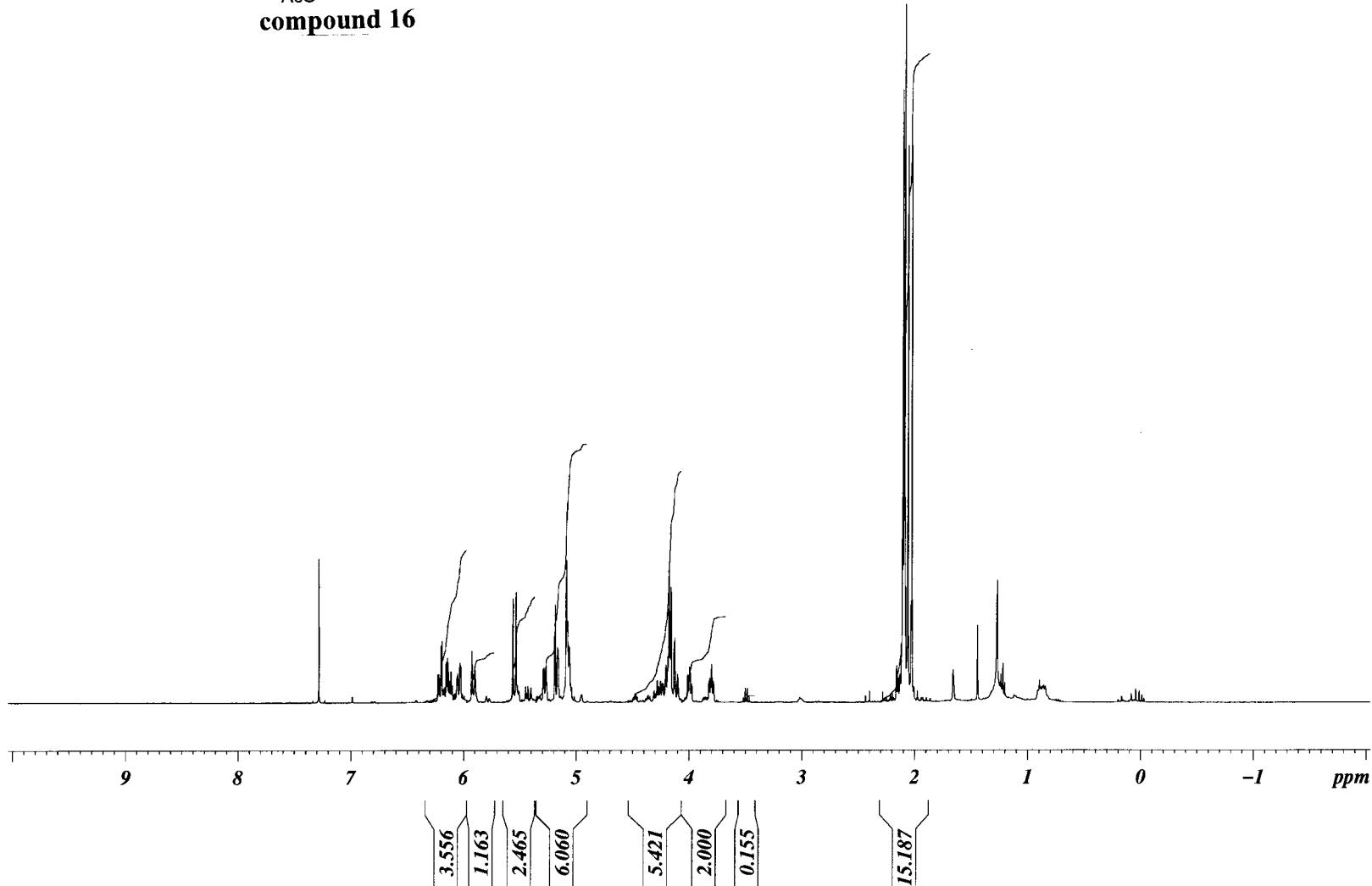
Current Data Parameters
 NAME Rungnaphavay400
 EXPNO 31
 PROCNO 1

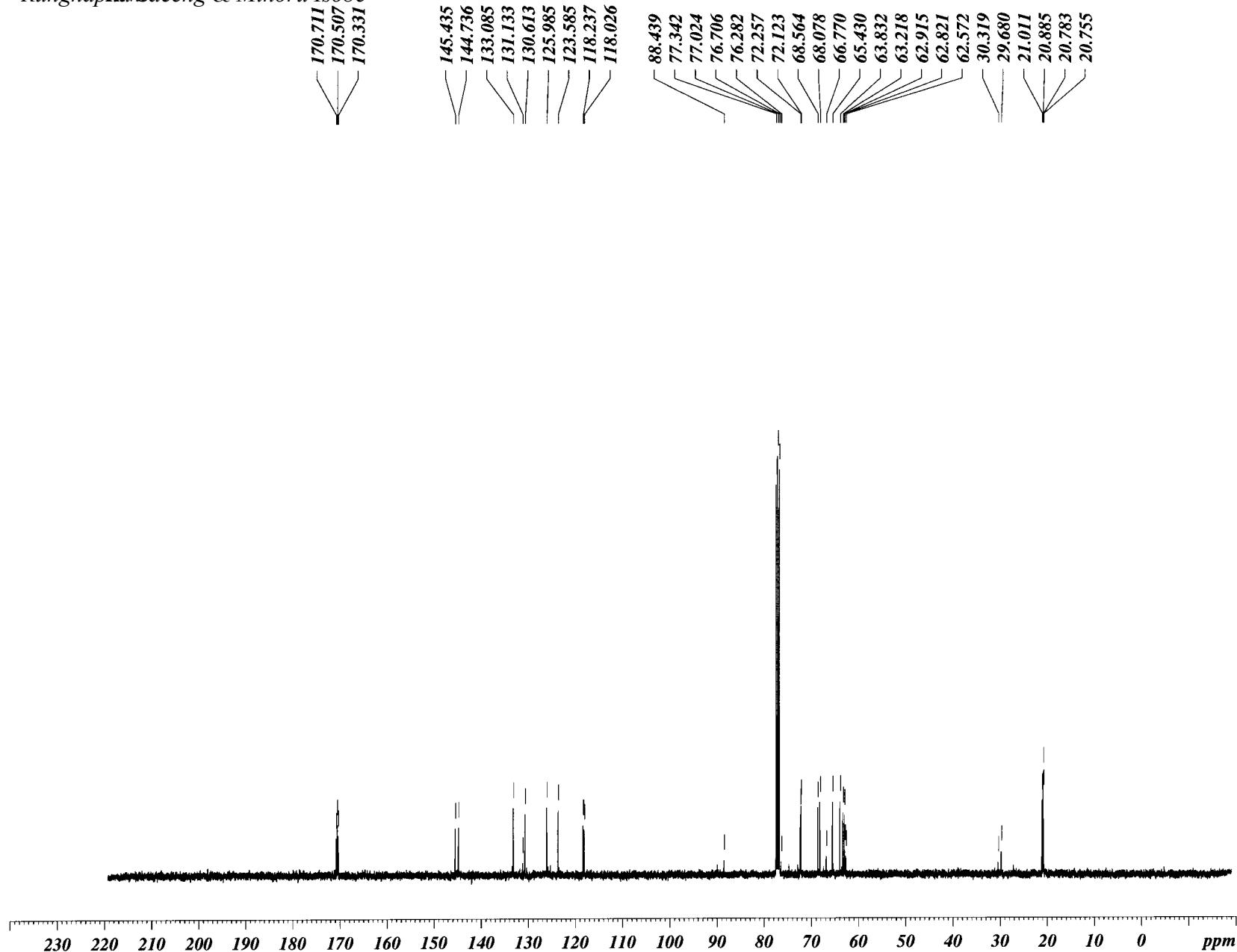


F2 - Acquisition Parameters
 Date 20040426
 Time 11.05
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 181
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 PI 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





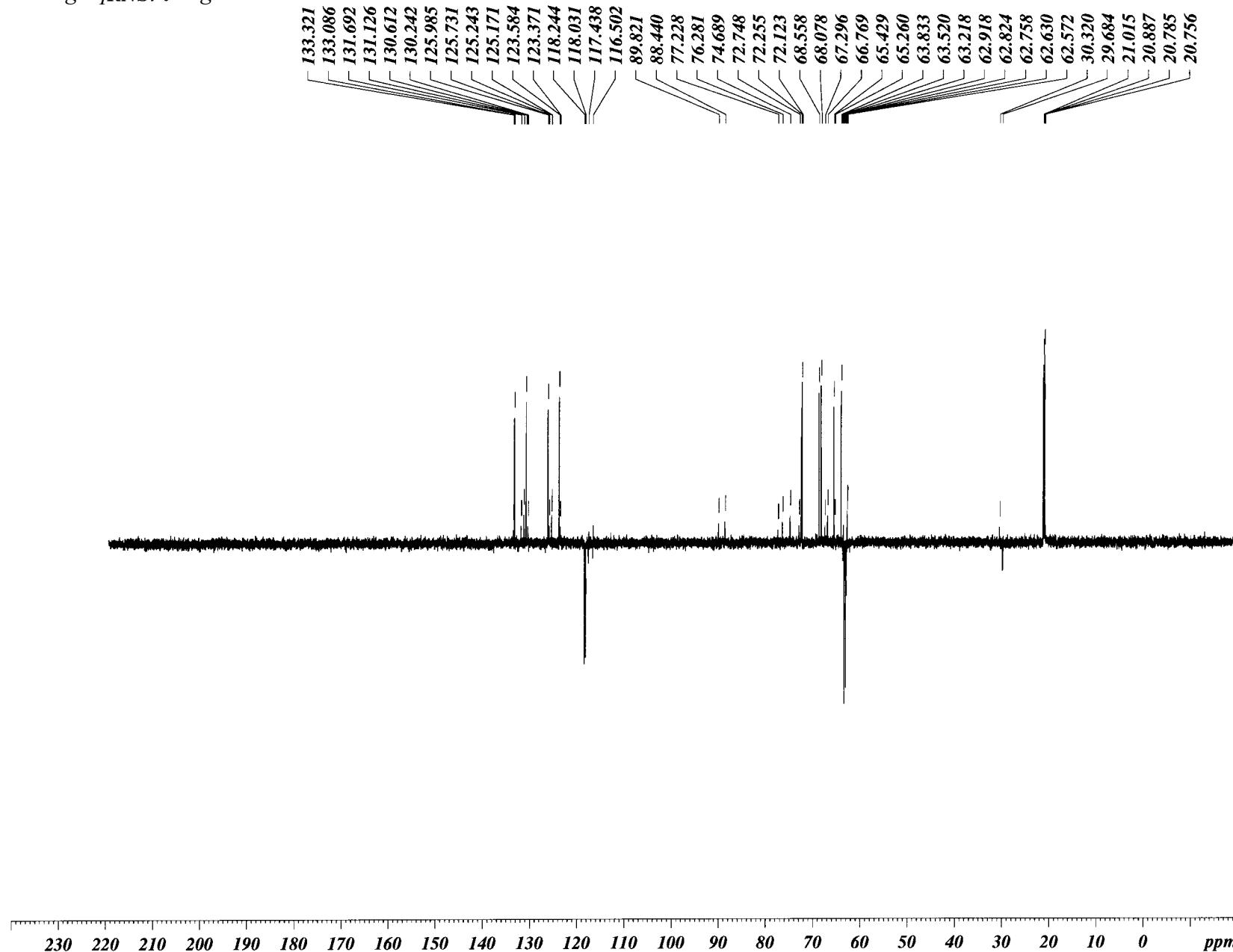
Current Data Parameters
 NAME Rungnaphaav400
 EXPNO 32
 PROCNO 1

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 2.0000000 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 Rungnaphaeng400
 EXPNO 34
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040426
 Time 13.02
 INSTRUM av400
 PROBHD 5 mm QNP JH/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 2.0000000 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
DI 1.0000000 sec

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

NUCI 1H
PI 8.60 usec
PLI -4.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.1300000 MHz

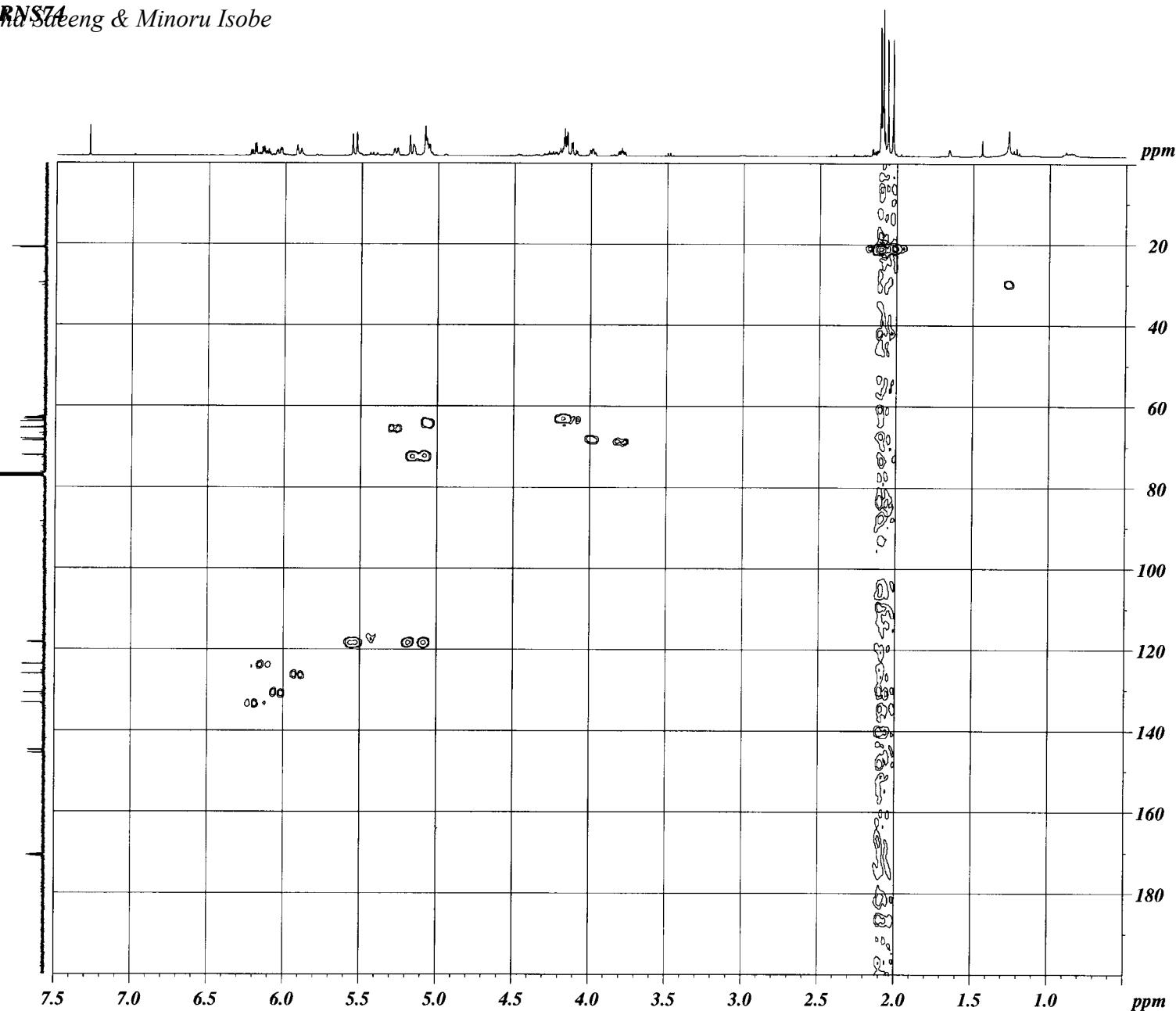
WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00



COSY

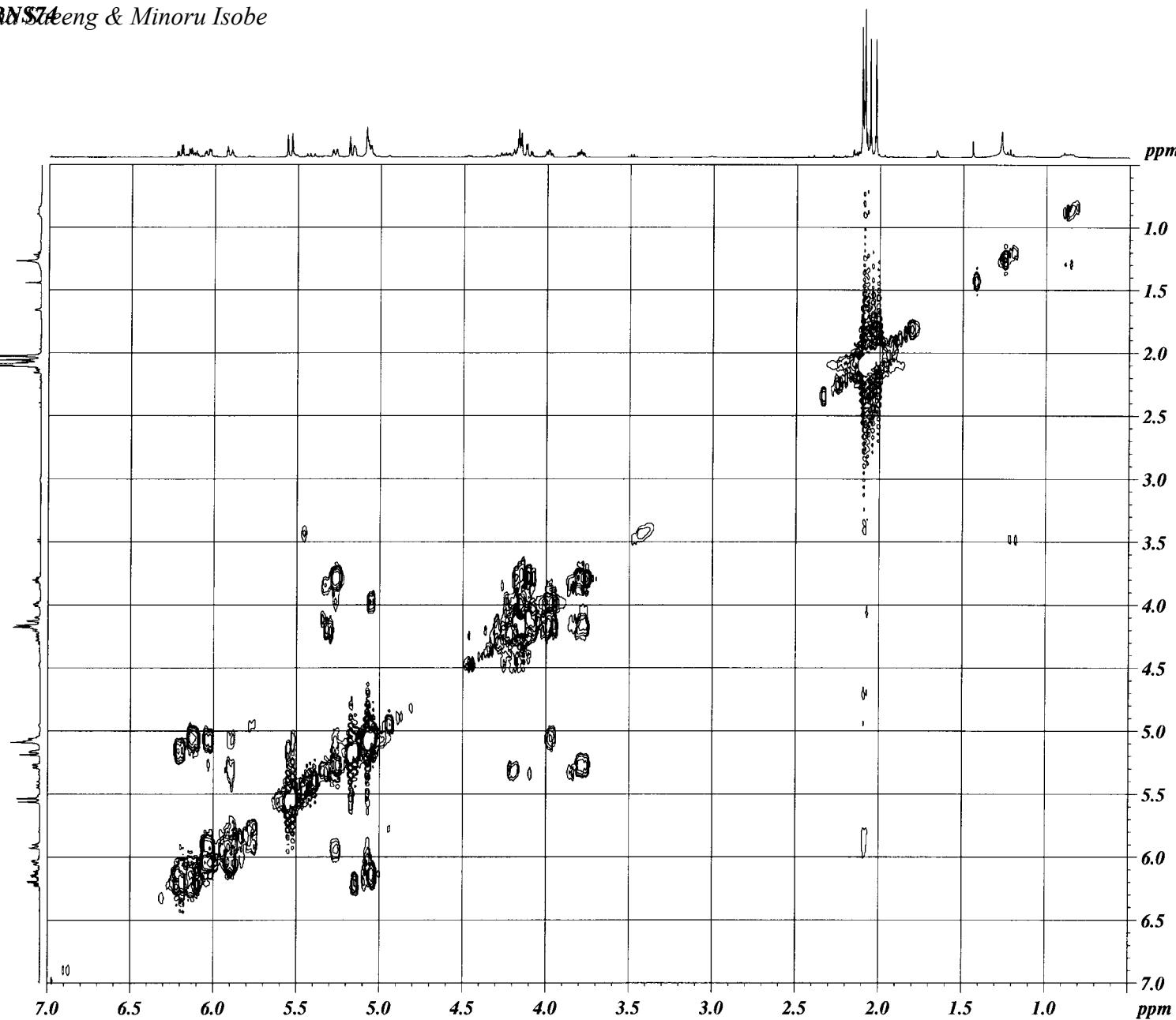
Rungnapaeng & Minoru Isobe

Current Data Parameters
 NAME Rungnaphaa400
 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
 DI 1.0000000 sec

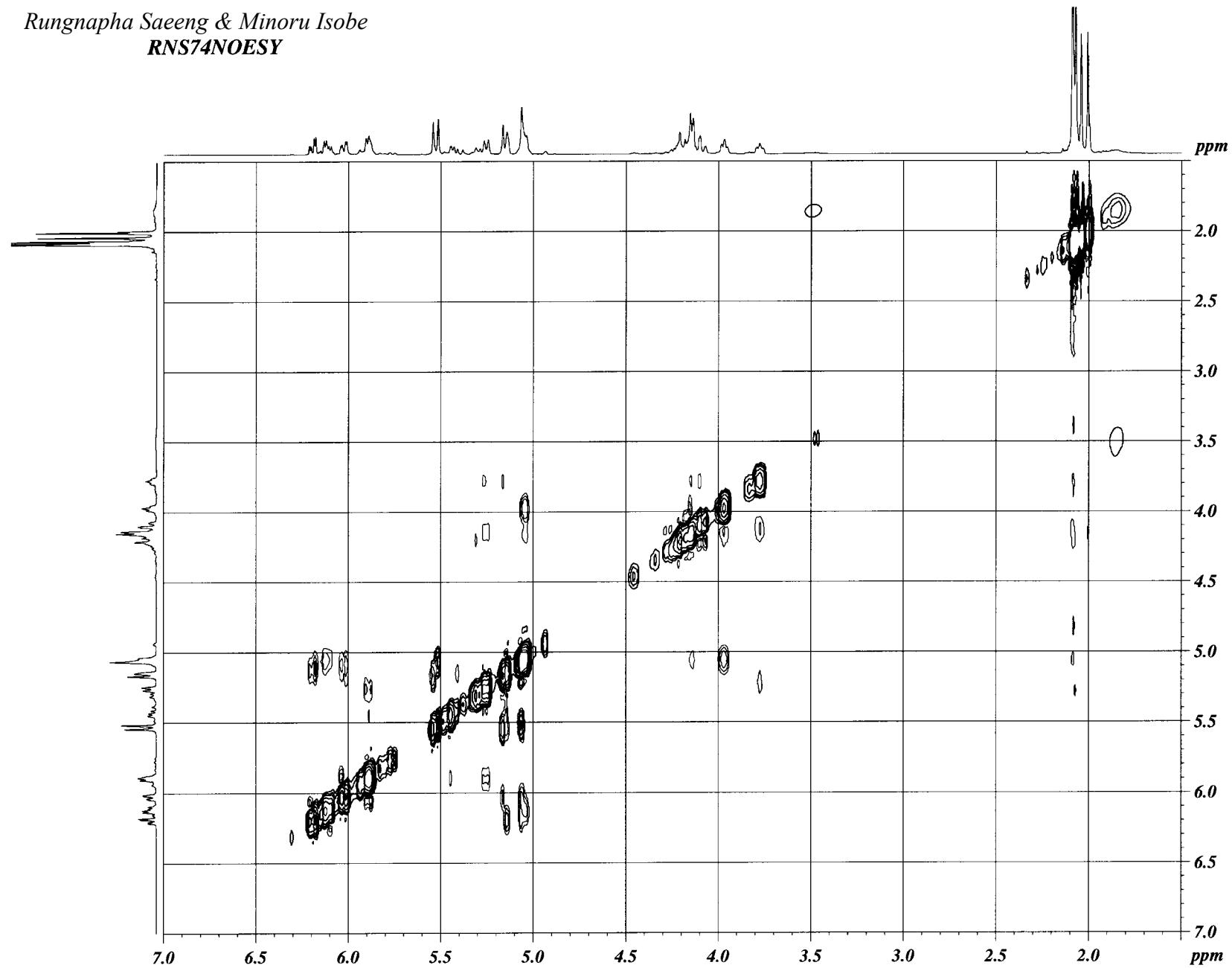
===== CHANNEL f1 =====
 NUCI 1H
 PI 8.60 usec
 PLI -4.00 dB
 SFO1 400.1324710 MHz

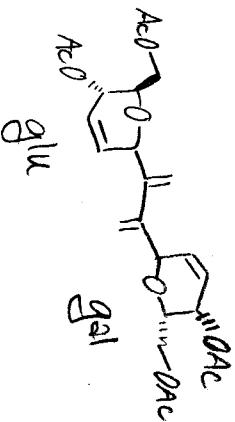
F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



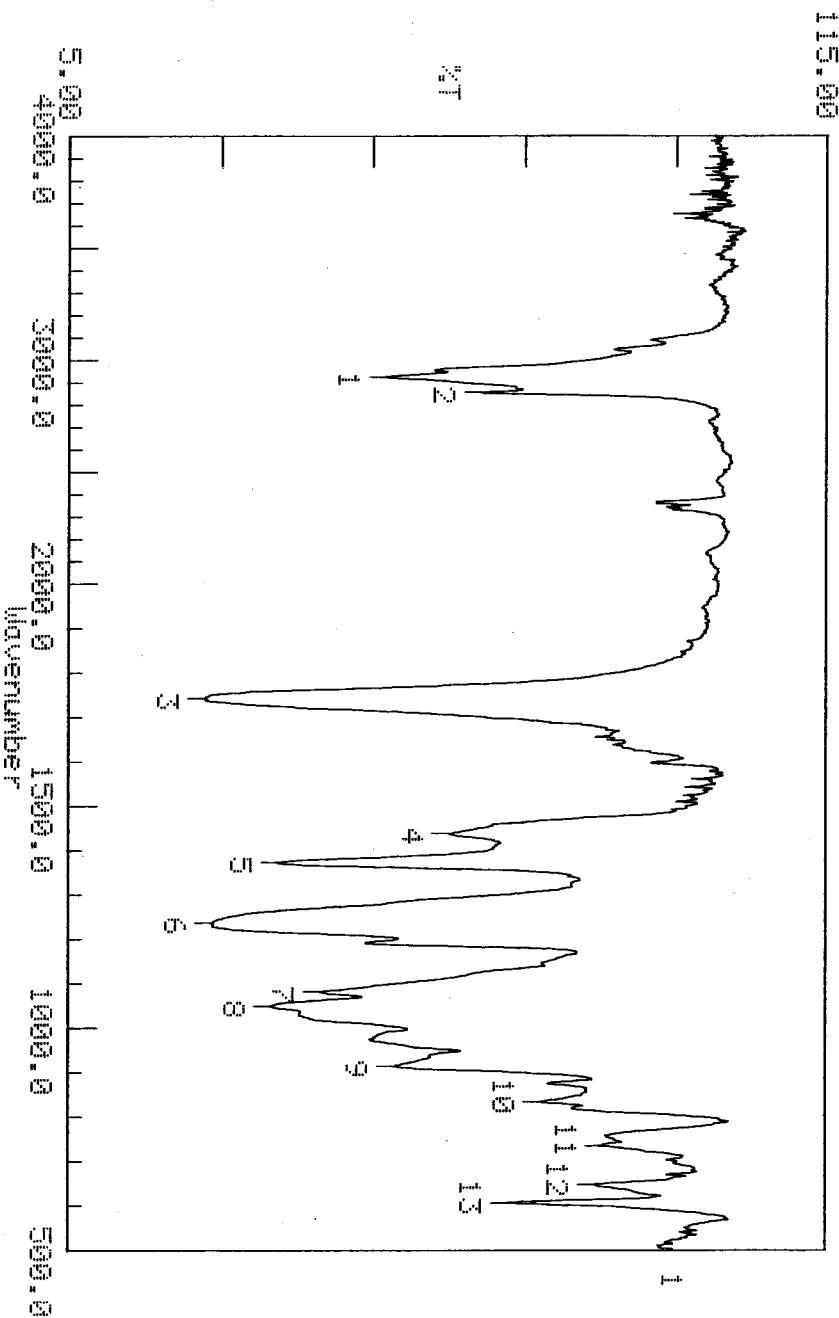
NOESY

Rungnapha Saeeng & Minoru Isobe
RNS74NOESY





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



Condition

UPPER 115.00 LOWER 5.00 depth 6.00

Peak table

	upper	lower	5.00	depth	6.00	
1:	2924.44(51.0)		2:	2854.63(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

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

Run number: 2
Sample Description: Isobe
Instrument: JEOL LCmate

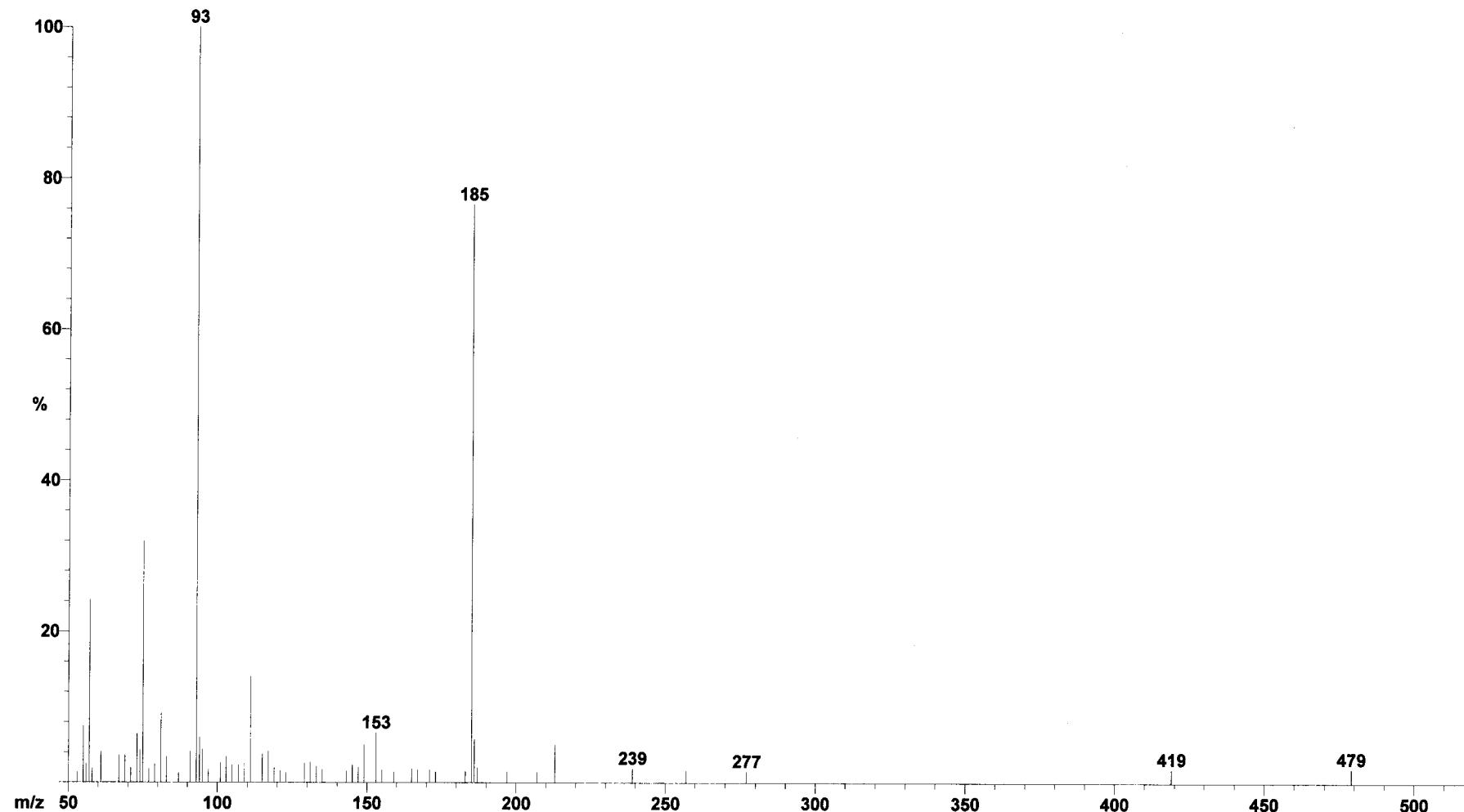
Ionization mode: FAB+

Scan: 2

R.T.: .2

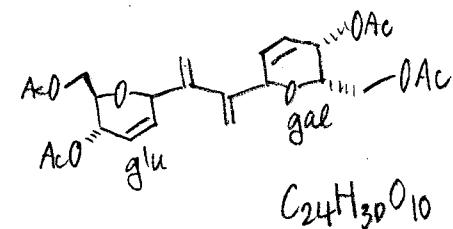
Base: m/z 93; 4.3%FS TIC: 185296

#Ions: 60



Elemental Analysis Report

Reference 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	No.589
TEMP	
SAMPLE	CPG-2 6-2
CONC.	2.01%
CELL	100mm
INTEG. TIME	20sec

SAMPLE NO. deg
017

- 1 d -2.026
[α]-100.84
- 2 d -2.029
[α]-100.97
- 3 d -2.027
[α]-100.87
- X⁴ d -2.026
[α]-100.83
- 5 d -2.033
[α]-101.18
- 6 d -2.032
[α]-101.13
- 7 d -2.030
[α]-101.09
- 8 d -2.031
[α]-101.14
- 9 d -2.033
[α]-101.14
- 10 d -2.030
[α]-101.03

MEAN VALUE N=10

$$\bar{\alpha} = -2.030 \text{ deg}$$

$$6n-1 0.002 \text{ deg}$$

$$RSD 0.098 \%$$

$$[\alpha] = 101.01 \text{ deg} \quad [\alpha] = -101.03$$

$$6n-1 0.1294 \text{ deg}$$

$$TEMP 20$$

Compound 15

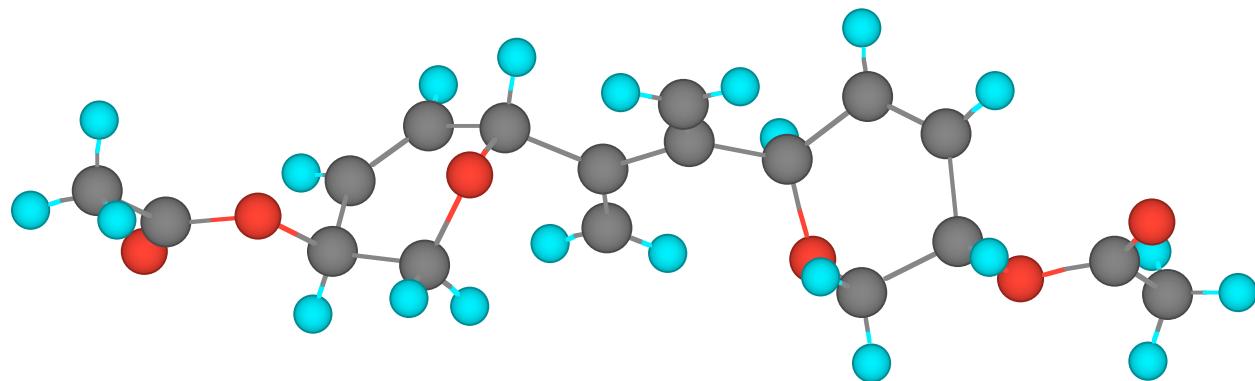
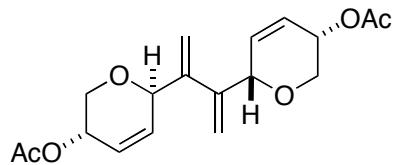


Table 1. Crystal data and structure refinement for isobe2.

Identification code	isobe2		
Empirical formula	C ₁₈ H ₂₂ O ₆		
Formula weight	334.36		
Temperature	90 K		
Wavelength	1.54178 Å		
Crystal system	Monoclinic		
Space group	P2(1)		
Unit cell dimensions	a = 9.3507(5) Å	α = 90°.	
	b = 5.4653(4) Å	β = 99.110(3)°.	
	c = 16.7583(7) Å	γ = 90°.	
Volume	845.62(8) Å 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.Å ⁻³

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

	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 ($\times 10^4$) and isotropic displacement parameters ($\approx^2 \times 10^{-3}$) 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

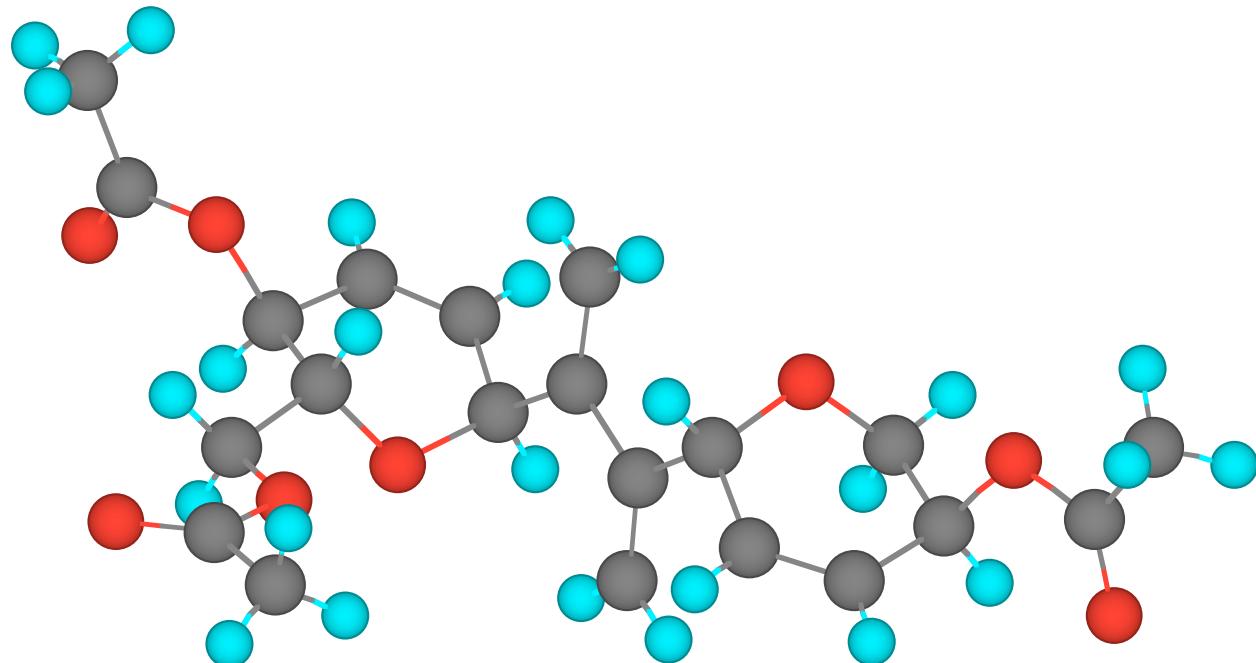
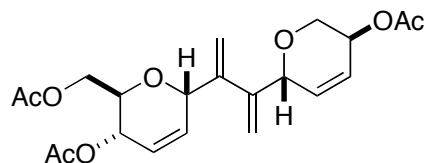


Table 1. Crystal data and structure refinement for isobe3.

Identification code	isobe3	
Empirical formula	C ₂₁ H ₂₆ O ₈	
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. \AA^{-3}

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

	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 ($\times 10^4$) and isotropic displacement parameters ($\approx^2 \times 10^{-3}$) 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

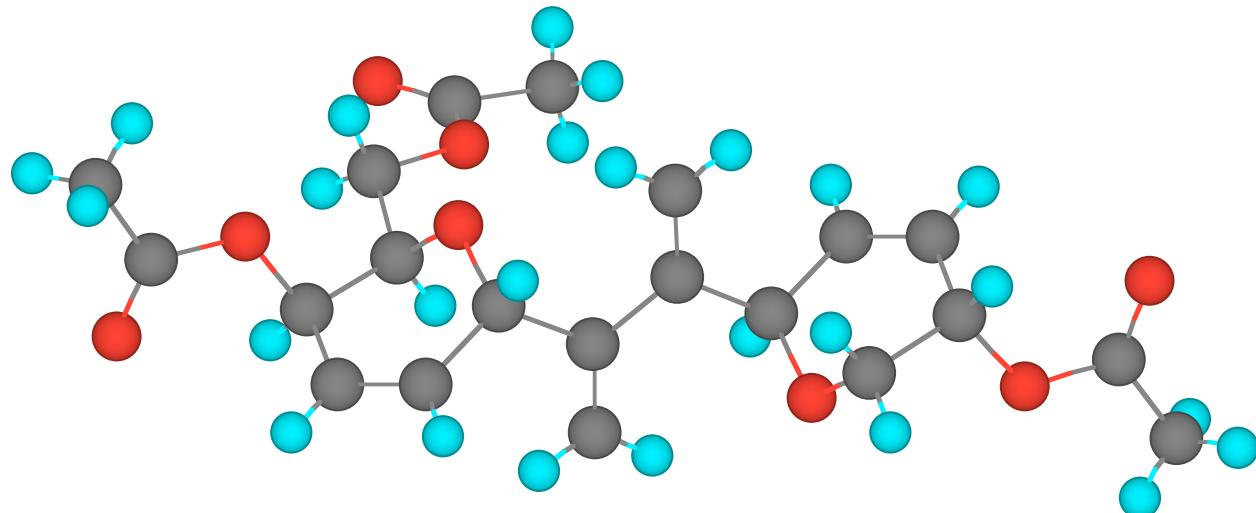
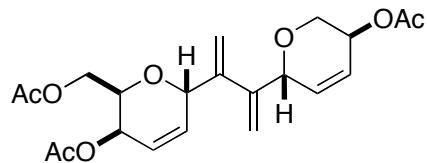


Table 1. Crystal data and structure refinement for isobe4.

Identification code	isobe4		
Empirical formula	C ₂₁ H ₂₆ O ₈		
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 ($\times 10^4$) and equivalent isotropic displacement parameters ($\approx^2 \times 10^3$) 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 [\approx] 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 ($\approx^2 \times 10^3$) for isobe4. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	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 ($\times 10^4$) and isotropic displacement parameters ($\approx^2 \times 10^{-3}$) 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