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Supporting Information

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Determination by asymmetric total synthesis of the absolute configuration of lucilactaene, a cell cycle inhibitor in p53-transfected cancer cells

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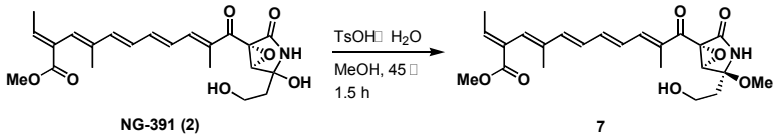
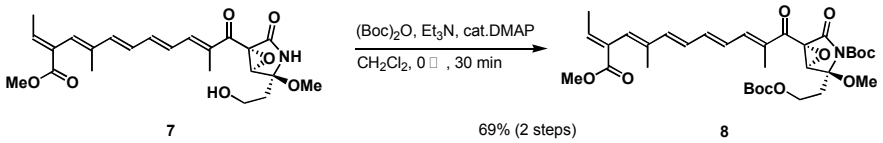
(3*E*, 5*E*, 7*E*, 9*E*)-2-Ethylidene-11-[(1*R*, 4*S*, 5*R*)-4-(2-hydroxy-ethyl)-4-methoxy-2-oxo-6-oxa-3-azabicyclo[3.1.0] hex-1-yl]-4,10-dimethyl-11-oxo-undeca-3,5,7,9-tetraenoic acid methyl ester (**7**)



To a MeOH (25 mL) solution of NG391 (**2**) (358 mg, 0.857 mmol) was added TsOH•H_2O (110 mg, 0.578 mmol), and the reaction mixture was stirred for 1.5 h at 45 °C. After addition of a saturated $NaHCO_3$ solution, the organic materials were extracted with $CHCl_3$ three times, dried over anhydrous Na_2SO_4, concentrated in vacuo after filtration to give 360 mg of crude methylether **7**, which was used in the next reaction without purification.

(3*E*, 5*E*, 7*E*, 9*E*) (1*R*, 4*S*, 5*R*)-4-(2-*tert*-Butoxycarbonyloxy-ethyl)-4-methoxy-1-(10-methoxycarbonyl-2,8-dimethyl-dodeca-2, 4,6,8,10-pentaenoyl)-2-oxo-6-oxa-3-aza-bicyclo[3.1.0]hexane-3-carboxylic acid *tert*-butyl ester (**8**)

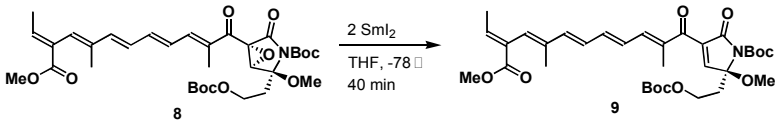


To a CH_2Cl_2 (20 mL) solution of crude methylether **7** (360 mg) was added triethylamine (0.50 mL), Boc_2O (0.7 mL), and dimethylaminopyridine (5.0 mg) at 0 °C, and the reaction mixture was stirred for 30 min at that temperature. The reaction was quenched with pH 7 phosphate buffer solution and the organic materials were extracted with ethyl acetate three times, the combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo after

filtration Purification by flash column chromatography (ethyl acetate/hexane= 1/5-1/3) gave epoxylactam **8** (373 mg, 0.591 mmol) in 69% yield.

^{1}H NMR ($CDCl_3$): □ 1.44 (9H, s), 1.50 (9H, s), 1.65 (3H, s), 1.70 (3H, d, *J*=7.3 Hz), 1.93 (3H, s), 2.25-2.41 (1H, m), 2.70-2.83 (1H, m), 3.40 (3H, s), 3.72 (3H, s), 3.96 (1H, s), 4.18-4.28 (1H, m), 4.30-4.42 (1H, m), 6.20 (1H, s), 6.42 (1H, dd, *J*=15.1, 10.6 Hz), 6.58 (1H, d, *J*=15.1 Hz), 6.65 (1H, dd, *J*=14.5, 11.2 Hz), 6.75 (1H, dd, *J*=14.5, 10.6 Hz), 6.97 (1H, q, *J*=7.3 Hz), 7.42 (1H, d, *J*= 11.2 Hz);
^{13}C NMR ($CDCl_3$): □ 11.1, 14.2, 15.9, 27.7, 27.9, 33.1, 51.0, 51.9, 60.8, 61.6, 62.3, 82.2, 84.4, 91.2, 127.9, 128.0, 128.4, 130.4, 133.7, 138.0, 140.4, 142.2, 143.5, 145.9, 149.3 153.3, 167.0, 167.4, 188.9;
IR (neat) 2923, 2854, 1741, 1722, 1650, 1587, 1097, 862, 836, 802 cm^{-1};
[□]$_D^{22}$ -85.2 (*c* = 0.2, MeOH);
HRMS (FAB) calced for [$C_{33}H_{45}NO_{10}$+H]; 616.3122, found: 616.3141.

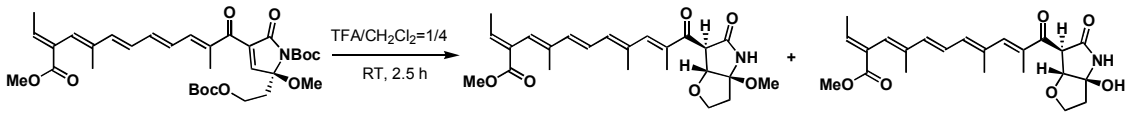
(3*E*, 5*E*, 7*E*, 9*E*, 12 *E*)-(2*R*)-2-(2-*tert*-Butoxycarbonyloxy-ethyl)-2-methoxy-4-(10-methoxycarbonyl-2,8-dimethyl-dodeca-2,4,6,8,10-pentaenoyl)-5-oxo-2,5-dihydro-pyrrole-1-carboxylic acid *tert*-butyl ester (**9**)



8 **9**

To a THF (3.0 mL) solution of epoxylactam **8** (96.0 mg, 0.152 mmol) was added 0.1 M THF solution of SmI_2 (15.0 mL) at -78 °C, and the reaction mixture was stirred for 40 min at -78 °C. After addition of a buffer solution, the organic materials were extracted with ethyl acetate three times, the combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, concentrated in vacuo after filtration. Crude lactam **9** was purified by thin layer chromatography (ethyl acetate/hexane 1/1) to give 70.6 mg lactam **9** in 75%.

(3*E*, 5*E*, 7*E*, 9*E*)-2-Ethylidene-11-(3a-methoxy-5-oxo-hexahydro-furo[3,2-b]pyrrol-6-yl)-4,10-dimethyl-11-oxo-undeca-3,5,7,9-tetraenoic acid methyl ester (**10**), lucilactaene (**1**)



9 **10** 55% **lucilactaene (1)** 28%

To a CH_2Cl_2 (8.0 mL) and H_2O (0.2 mL) solution of lactam **10** (70.6 mg, 0.111 mmol) was added CF_3COOH (2.0 mL) at 0 °C and the reaction mixture was stirred for 2.5 h at room temperature. After the solvent and CF_3COOH were removed in vacuo, purification by preparative thin layer chromatography (Et_2O) gave methylether **10** (25.3 mg, 0.063 mmol) in 55% yield and lucilactaene (**1**) (11.3 mg, 0.027 mmol) in 28% yield over 2 steps.

Methylether **10**

^{1}H NMR ($CDCl_3$): 1.70 (3H, s), 1.73 (3H, d, *J*=7.2 Hz), 1.95 (3H, s), 2.08-2.20 (1H, m), 2.28-2.38 (1H, m), 3.34 (3H, s), 3.72 (3H, m), 3.88-4.00 (1H, m), 4.01-4.09 (1H, m), 4.18 (1H, d, *J*=2.3 Hz), 4.75 (1H, d, *J*=2.3 Hz), 6.18 (1H, s), 6.27 (1H, brs), 6.40-6.76 (4H, m), 6.98 (1H, q, *J*=7.2 Hz), 7.26 (1H, d, *J*=11.2 Hz);

^{13}C NMR ($CDCl_3$): 11.8, 14.3, 15.9, 40.0, 51.9, 52.0, 56.6, 67.3, 83.4, 99.6, 127.5, 128.3, 128.5, 130.4, 134.8, 138.1, 140.4, 141.3, 142.1, 142.8, 167.5, 171.4, 194.6;

IR (neat) 2950, 2925, 1716, 1648, 1585, 1241, 1056, 732 cm^{-1};

$[\alpha]_D^{17}$+36.6 (*c*=0.17, MeOH);

HRMS (FAB) calced for [$C_{23}H_{29}NO_6$+H]; 416.2073, found: 416.2054.

lucilactaene (**1**)

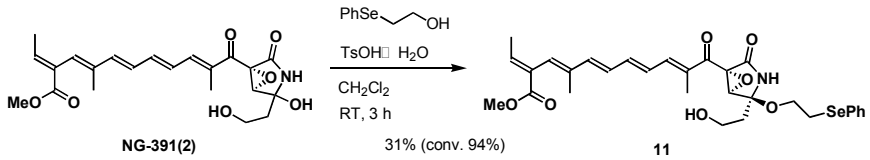
^{1}H NMR ($CDCl_3$): 1.70 (3H, s), 1.73 (1H, d, *J*=7.2 Hz), 1.93 (3H, s), 2.21-2.29 (1H, m), 2.37-2.46 (1H, m), 3.72 (3H, s), 4.21 (1H, brs), 4.30 (1H, brs), 4.97 (1H, brs), 6.20 (1H, s), 6.45 (1H, dd, *J*= 15.2, 10.9 Hz), 6.62 (1H, d, *J*= 15.2 Hz), 6.66 (1H, dd, *J*= 14.7, 11.5 Hz), 6.82 (1H, dd, *J*= 14.7, 11.0 Hz), 6.99 (1H, q, *J*=7.2 Hz), 7.45 (1H, d, *J*=11.5 Hz);

^{13}C NMR ($CDCl_3$): 11.5, 14.2, 15.9, 37.4, 51.9, 56.6, 68.5, 85.7, 94.5, 128.0, 128.1, 128.3, 130.3, 134.1, 137.9, 140.6, 142.3, 143.6, 145.5, 167.5, 170.7, 197.0;

IR (neat) 2948, 1716, 1652, 1585, 1436, 1243, 1118, 1243, 991, 734 cm^{-1};

HRMS (FAB): calced for [$C_{22}H_{27}NO_6$]; 401.1838, found; 401.1838.

(3*E*, 5*E*, 7*E*, 9*E*)-2-Ethylidene-11-[(1*R*, 4*S*, 5*R*)-4-(2-hydroxy-ethyl)-2-oxo-4-(2-phenylselanyl-ethoxy)-6-oxa-3-aza-bicyclo[3.1.0]hex-1-yl]-4,10-dimethyl-11-oxo-undeca-3,5,7,9-tetraenoic acid methyl ester (**11**)

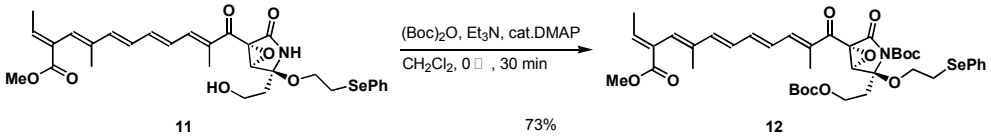


NG-391(2) 31% (conv. 94%) 11

To a CH_2Cl_2 (4.0 mL) solution of NG391 (**2**) (170.0 mg, 0.405 mmol) was added $PhSeCH_2CH_2OH$ (97.8 mg, 0.496 mmol) and $TsOH•H_2O$ (15.4 mg, 0.081 mmol) at room temperature, and the reaction mixture was stirred for 3 h at that temperature. After addition of a saturated $NaHCO_3$ solution, the organic materials were extracted with $CHCl_3$ three times, dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by flash column chromatography (ethyl acetate/hexane=1/3 - MeOH/ $CHCl_3$=1/10) gave selenoethylether **11** (74.8 mg 0.124 mmol) in 31% yield. NG-391 (**2**) (110 mg, 0.26 mmol) was recovered in 65 % yield.

^{1}H NMR ($CDCl_3$): □ 1.69 (3H, s), 1.72 (3H, d, *J*=7.2 Hz), 1.92 (3H, s), 2.03-2.05 (2H, m), 2.07 (1H, brs), 3.09 (2H, t, *J*=6.2 Hz), 3.72 (3H, s), 3.78-3.83 (1H, m), 3.85 (1H, brd), 3.87 (2H, t, *J*=6.2 Hz), 3.93-3.99 (1H, m), 6.18 (1H, s), 6.42 (1H, dd, *J*=10.4, 4.5 Hz), 6.50-6.67 (3H, m), 6.75 (1H, dd, *J*= 14.6, 10.7 Hz), 6.96 (1H, q, *J*=7.2 Hz), 7.16-7.28 (3H, m), 7.44 (1H, d, *J*=10.7 Hz), 7.51 (2H, brd);
^{13}C NMR ($CDCl_3$): □ 11.2, 14.2, 15.9, 27.7, 36.2, 51.9, 56.9, 61.7, 61.9, 62.5, 77.2, 88.2, 127.2, 127.9, 128.0, 128.4, 129.2, 130.3, 132.9, 133.9, 138.0, 140.5, 142.1, 143.2, 145.4, 167.4, 169.7, 189.4;
IR (neat) 3291, 1720, 1641, 1581, 1436, 1261, 1130, 1089, 877, 736 cm^{-1};
$[□]_D^{25}$ =+3.7 (*c*=0.441, MeOH);
HRMS (FAB): calced for [$C_{30}H_{35}NO_7Se$]; 601.1581, found: 601.1575.

(3*E*, 5*E*, 7*E*, 9*E*) -(1*R*, 4*S*, 5*R*)-4-(2-*tert*-Butoxycarbonyloxy-ethyl)-1-(10-methoxycarbonyl-2,8-dimethyl-dodeca-2,4,6,8,10-pentaenoyl)-2-oxo-4-(2-phenylselanyl-ethoxy)-6-oxa-3-aza-bicyclo[3.1.0]hexane-3-carboxylic acid *tert*-butyl ester (**12**)



11 73% **12**

To a CH_2Cl_2 (2.1 mL) solution of selenoethylether **11** (42.0 mg, 0.070 mmol) was added Et_3N (29 □L, 0.21 mmol), Boc_2O (40 □L, 0.175 mmol) and DMAP (2 mg) at 0 °C, and the reaction mixture was stirred for 30 min at that temperature. After addition of a buffer solution, the organic materials were extracted with ethyl acetate three times, the combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate/hexane=1/3) gave epoxylactam **12** (40.8 mg, 0.051 mmol) in 73% yield.

^{1}H NMR ($CDCl_3$): □ 1.45 (9H, s), 1.48 (9H, s), 1.69 (3H, s), 1.73 (3H, d, *J*=7.2 Hz), 2.33 (1H, dt, *J*=

14.4, 7.1 Hz), 2.77 (1H, dt, *J*=14.4, 5.5 Hz), 3.06 (2H, t, *J*=6.3 Hz), 3.73 (3H, s), 3.77-3.83 (1H, m), 3.94 (1H, brs), 3.94-3.40 (1H, m), 4.18-4.24 (1H, m), 4.29-4.35 (1H, m), 6.19 (1H, s), 6.42 (1H, dd, *J*=15.4 Hz), 6.57 (1H, d, *J*=15.4 Hz), 6.64 (1H, dd, *J*= 14.5, 11.3 Hz), 6.76 (1H, dd, *J*=14.5,10.4 Hz), 6.98 (1H, q, *J*=7.2 Hz), 7.16-7.24 (3H, m), 7.39 (1H, d, *J*=11.3 Hz), 7.50 (1H, brd);

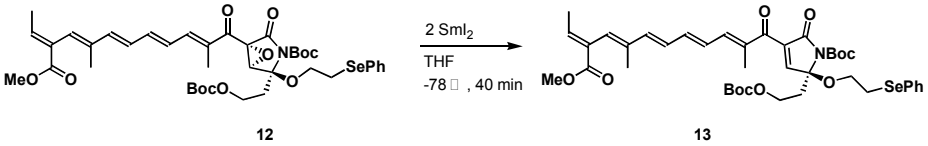
^{13}C NMR ($CDCl_3$): □ 11.1, 14.2, 15.9, 27.1, 27.8, 27.9, 33.1, 51.9. 60.9, 61.7, 62.3, 63.3, 82.2, 84.5, 90.9, 127.1, 127.9, 128.0, 128.4, 129.1, 129.8, 130.3, 132.9, 133.7, 138.0, 140.4, 142.1, 143.4, 145.7, 149.1, 153.2, 166.8, 167.4, 188.6;

IR (neat) 2981, 1789, 1741, 1716, 1644, 1581, 1338, 1278, 1255, 1155, 993, 846 cm^{-1};

$[□]_D^{24}$ -54.8 (*c*=1.03, MeOH);

HRMS (FAB): calced for [$C_{40}H_{51}NO_{11}Se$+H]; 802.2710, found: 802.2730.

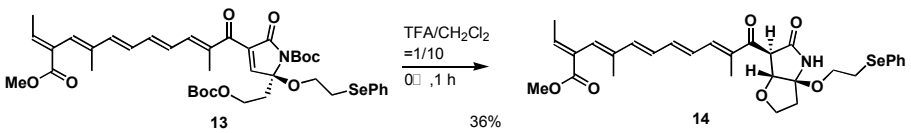
(3*E*, 5*E*, 7*E*, 9*E*)-2-((1*R*, 4*S*, 5*R*)-2-*tert*-Butoxycarbonyloxy-ethyl)-4-(10-methoxycarbonyl-2,8-dimethyl-dodeca-2,4,6,8,10-pentaenoyl)-5-oxo-2-(2-phenylselanyl-ethoxy)-2,5-dihydro-pyrrole-1-carboxylic acid *tert*-butyl ester (**13**)



12 **13**

To a THF (0.4 mL) solution of epoxylactam **12** (22.3 mg, 0.02 mmol) was added 0.1 M THF solution of SmI_2 (0.6 mL) at -78 °C, and the reaction mixture was stirred for 40 min at that temperature. After addition of a buffer solution, the organic materials were extracted with ethyl acetate three times, the combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, concentrated in vacuo after filtration. Crude lactam **13** was purified by thin layer chromatography (ethyl acetate/hexane 1/1) to afford 14.5 mg lactam **13**.

(3*E*, 5*E*, 7*E*, 9*E*)- 2-Ethylidene-4,10-dimethyl-11-oxo-11-[(1*R*, 4*S*, 5*R*)-5-oxo-3a-(2-phenylselanyl-ethoxy)-hexahydro-furo[3,2-b]pyrrol-6-yl]-undeca-3,5,7,9-tetraenoic acid methyl ester (**14**)



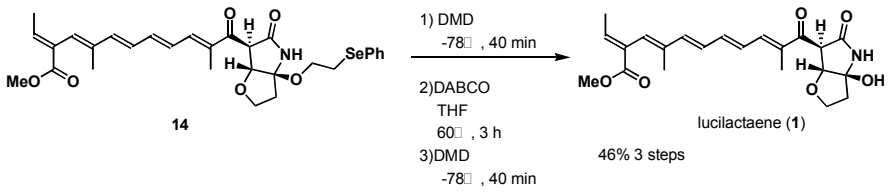
13 36% **14**

To a CH_2Cl_2 (1.0 mL) solution of lactam **13** (14.5 mg) was added CF_3COOH (1.0 mL) at 0 °C and the reaction mixture was stirred for 2.5 h at room temperature. After solvent and CF_3COOH were removed in vacuo, purification by preparative thin layer chromatography (Et_2O/Hexane= 5/1) gave

selenoethylether **14** (5.8 mg, 0.049 mmol) in 36 % yield over 2 steps.

^{1}H NMR ($CDCl_3$): □ 1.64 (3H, s), 1.68 (3H, d, *J*= 7.2 Hz), 1.88 (3H, s), 1.96 (2H, d, *J*=6.1 Hz), 1.97-2.03 (1H, m), 2.19-2.26 (1H, m), 3.00 (2H, t, *J*= 6.1 Hz), 3.74 (3H, s), 3.75-3.94 (2H, m), 4.10 (1H, brs), 4.64 (1H, brs), 4.64 (1H, brs), 6.12 (1H, s), 6.30-6.72 (4H, m), 6.91 (1H, d, *J*= 7.1 Hz), 7.16-7.18 (3H, m), 7.41-7.46 (2H, m);
^{13}C NMR ($CDCl_3$): □11.8, 14.3, 15.9, 27.7, 40.3, 51.9, 56.5, 64.1, 67.2, 83.7, 99.2, 127.0, 127.5, 128.3, 128.5, 129.1, 130.1, 130.4, 132.0, 134.8, 140.4, 141.3, 142.1, 142.9, 167.5, 171.1, 194.7 ;
IR (neat) 2948, 1716, 1652, 1585, 1436, 1243, 1118, 1243, 991, 734 cm^{-1} ;
[□]$_D^{26}$ +28.0 (*c*=0.26, MeOH);
HRMS (FAB): calced for [$C_{30}H_{36}NO_6Se$+H]; 586.1710, found: 586.1712.

<u>lucilactaene (**1**)</u>



To a CH_2Cl_2 (0.2 mL) solution of selenoethylether **14** (3.9 mg, 0.0067 mmol) was added 0.1 M acetone solution of dimethyldioxirane (0.13 mL) at -78 °C, and the reaction mixture was stirred for 40 min at that temperature. The reaction was quenched with Me_2S (0.1 mL), and the mixture was stirred for 10 min at 0 °C. After solvent was removed in vacuo, purification by preparative thin layer chromatography (MeOH/ $CHCl_3$= 1/ 10) gave selenoxide (3.2 mg, 0.0055 mmol) in 82 % yield.

To a THF (0.5 mL) solution of the selenoxide was added DABCO (2.5 mg, 0.023 mmol) at room temperature, and the reaction mixture was stirred for 3 h at 60 °C. The reaction was quenched with pH 7 phosphate buffer solution and the organic materials were extracted with ethyl acetate three times, and the combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration to give 3.3 mg of crude vinylether, which was used in the next reaction without purification.

To a CH_2Cl_2 (0.2 mL) solution of crude vinylether was added 0.1 M acetone solution of dimethyldioxirane (0.10 mL) at -78 °C, and the reaction mixture was stirred for 40 min at that temperature. The reaction was quenched with Me_2S (0.1 mL), and the mixture was stirred for 10 min at 0 °C. After the solvent was removed in vacuo, purification by preparative thin layer chromatography (Et_2O) gave lucilactaene (**1**) (1.2 mg, 0.003 mmol) in 56 % yield over 2 steps.

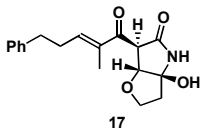
The optical purify of lucilactaene was determined by HPLC analysis with a Chiralcel OD-RH column (CH_3CN/H_2O= 45/100), 1.0 mL/min major enantiomer tr = 40.5 min, minor enantiomer tr = 28.7 min.

$[\alpha]_D^{21}$ +39.5 (*c*=0.1, MeOH).

Production and isolation experiment of lucilactaene (Table 2).

The fungal strain *Fusarium sp*. RK-9794 was inoculated into the medium (70 ml) consisting of 2% glucose, 1% soluble starch, 0.3% meat extract, 2.5% yeast extract, 0.05% NaCl, 0.005% K_2HPO_4, 0.05% $CaCO_3$, and 0.05% $MgSO_4 \cdot 7H_2O$ (adjusted at pH 7.2 before sterilization), and cultured on a rotary shaker (150rpm) at 28 °C. At the indicated times, the fermentation broth (supernatant) and mycelia were separated by centrifugation. The broth was immediately adjusted at pH 7.0 and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was dissolved in methanol and analyzed by the chiral HPLC system. The chiral HPLC conditions as follows: column; CHIRALCEL OD-RH column (150 x 4.6 mm *i.d.*, Daicel Chemical Ind., Tokyo), mobile phase; $CH_3CN:H_2O$=45:55, flow rate; 0.6 ml/min, detection; 360nm. The mycelia were extracted with acetone, which was concentrated in vacuo. The ethyl acetate extract from the mycelia was prepared as described above and analyzed.

Typical procedure of the racemization of lucilactaene-model 17 (Table 1, entry 11).



17

To model **17** (0.7 mg, 0.002 mmol) was added culture medium (2.0 mL) which consists of 2% glucose, 1% soluble starch, 0.3% meat extract, 2.5% yeast extract, 0.05% NaCl, 0.005% K_2HPO_4, 0.05% $CaCO_3$, and 0.05% $MgSO_4 \cdot H_2O$ adjusted pH 7.2. The reaction mixture was for 10 h or 48 h at 28 °C. The organic materials were extracted with ethyl acetate three times, the combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography gave model **17**, and the optical purify of model **17** was determined to be >99% ee by HPLC analysis with a Chiralpak AD-H column.

3a-Hydroxy-6-(2-methyl-5-phenyl-pent-2-enoyl)-hexahydro-furo[3,2-b]pyrrol-5-one (**17**)

^{1}H NMR ($CDCl_3$): δ 1.73 (3H, s), 2.20-2.25 (1H, m), 2.65 (1H, q, *J*=7.5Hz), 2.80-2.86 (1H, m), 3.95-4.04 (1H, m), 4.06-4.14 (1H, m), 4.20 (1H, s), 4.26 (1H, s), 4.81 (1H, s), 6.03 (1H, brs), 7.08 (1H, t, *J*=7.2Hz), 7.18-7.30 (5H, m);

^{13}C NMR ($CDCl_3$): □ 11.1, 31.8, 34.5, 56.7, 68.5, 85.7, 94.4, 126.3, 128.4, 128.6, 136.9, 140.7, 149.9, 170.1, 197.6;

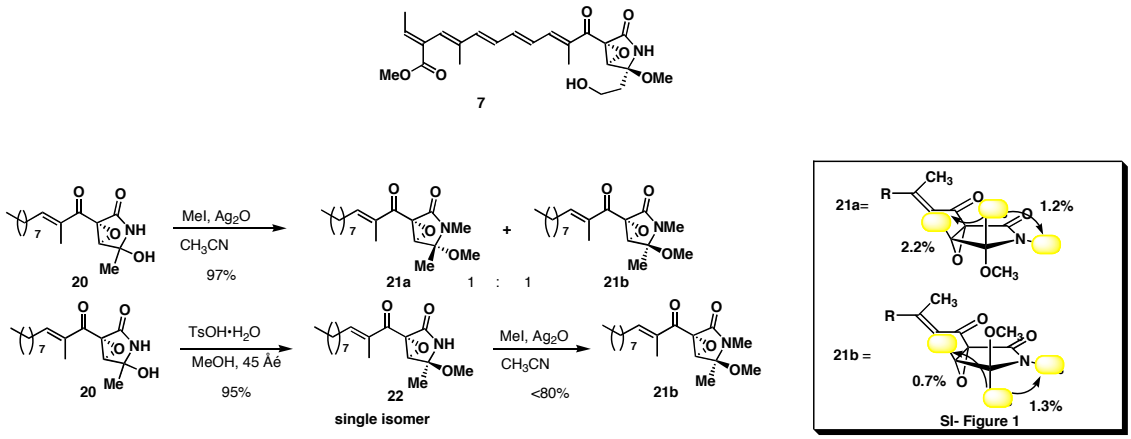
IR (neat) 2925, 2857, 1716, 1658, 1112, 1072, 742, 701 cm^{-1};

HRMS (FAB): calced for [$C_{18}H_{21}NO_4$]; 315.1471, found: 315.1481.

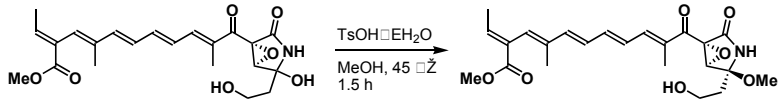
[□]$_D^{28}$ +66.0 (c=0.05, MeOH).

HPLC conditions: Chiralpak AD-H column, *i*-propanol/Hexane = 1/3, 1.0 mL/min, major enantiomer tr = 13.14 min, minor enantiomer tr = 7.7 min.

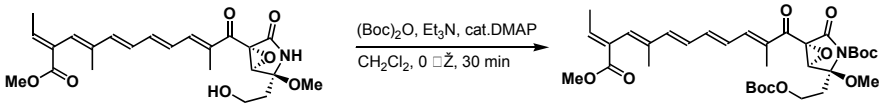
Determination of relative stereochemistry of 7.



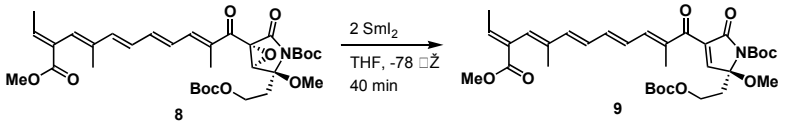
The relative stereochemistry of methyl ether **7** was determined as follows by analogy of the model **20**. When (3*R*,4*R*)-5-hydroxy-5-methyl-3-(2-methyl-2-undecenoyl)-3,4-epoxy-2-pyrrolidone **20** was treated with MeI and Ag_2O, both isomers (3*R*,4*R*,5*R*)-5-methoxy-5-methyl-2-(2-methyl-2-undecenoyl)-2,3-epoxy-2-pyrrolidone **21a** and (3*R*,4*R*,5*S*)-isomer **21b** were obtained in 48% and 48% yield, respectively. By the careful comparison between the difference NOEs of the both diastereomers **21a** and **21b**, the compound in which larger difference NOE between 4-H and 5-Me is assigned to possess *syn* configuration between 4-H and 5-Me. When **20** was treated with TsOH•H_2O in MeOH, followed by MeI and Ag_2O, a single isomer **21b** was obtained stereoselectively, which indicates that the intermediate methyl ether is (3*R*,4*R*,5*S*)-5-hydroxy-5-methyl-3-(2-methyl-2-undecenoyl)-3,4-epoxy-2-pyrrolidone **22**. Under the basic reaction conditions, both □- and □-methoxy derivatives are formed, while only □-methoxy derivative is obtained stereoselectively under the acidic conditions, in which methanol attacks acyliminiun ion intermediate from the opposite side of epoxide. The same sense of attack is expected in the case of NG-391 and the stereochemistry of **7** should be as shown in the above figure.



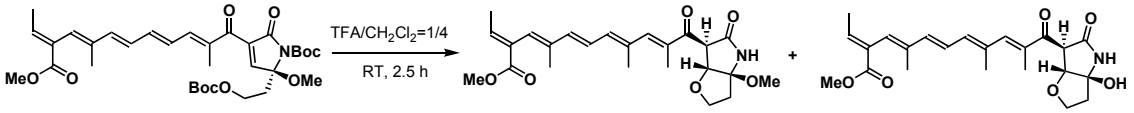
NG-391 (2) **7**



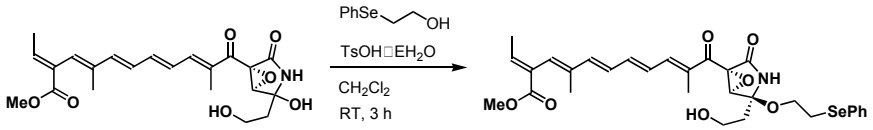
7 69% (2 steps) **8**



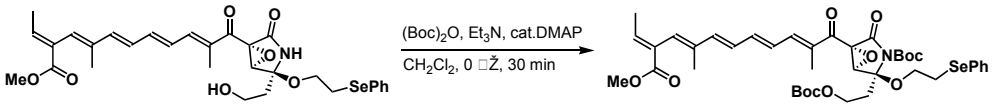
8 **9**



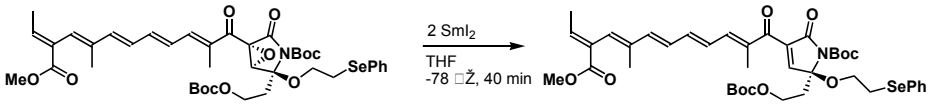
9 **10** 55% **lucilactaene (1)** 28%



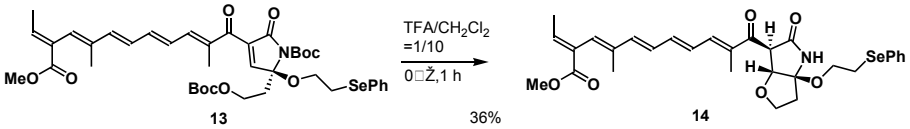
NG-391(2) 31% (conv. 94%) **11**



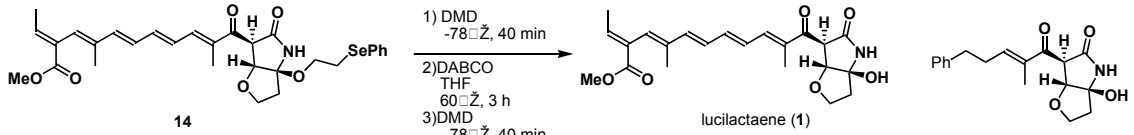
11 73% **12**



12 **13**

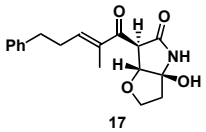


13 36% **14**

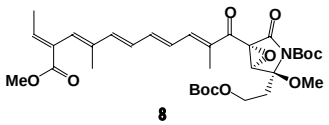


14 lucilactaene (**1**) 46% 3 steps

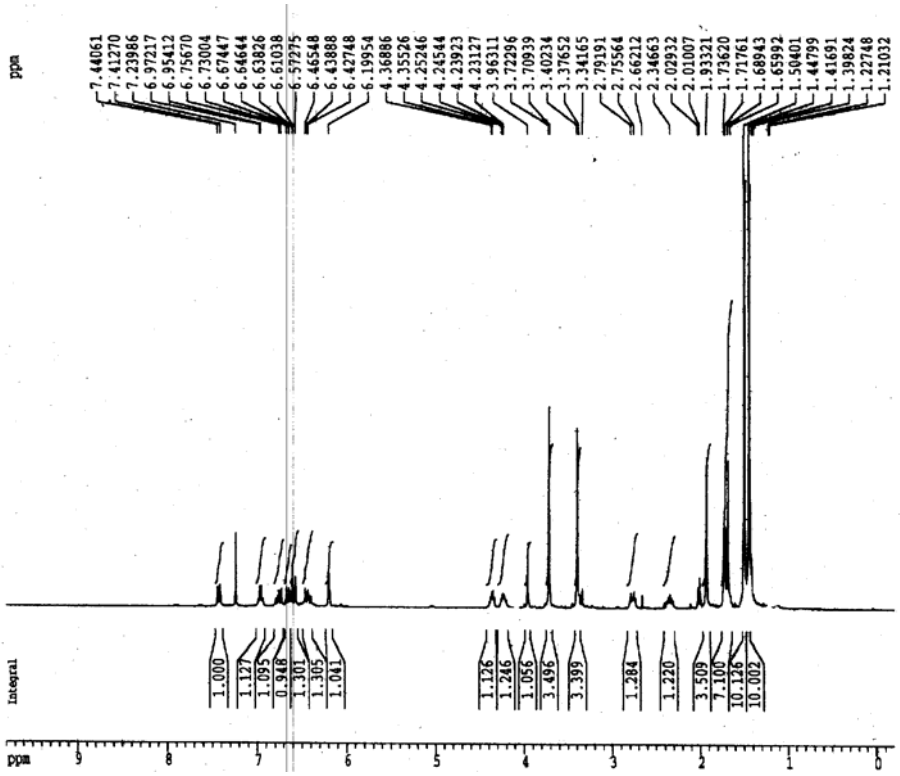
17



17



8

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EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_          20021129
Time               0.28
INSTRUM          dpx400
PROBHD    5 mm BBO 13C-
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   0
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923446 sec
RG                71.8
DW               60.800 usec
DE                6.00 usec
TE               300.0 K
D1            1.00000000 sec
P1                9.00 usec
SFO1        400.1324710 MHz
NUC1                1H
PL1               3.00 dB

F2 - Processing parameters
SI               16384
SF          400.1300172 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
F1P               9.800 ppm
F1             3921.27 Hz
F2P              -0.200 ppm
F2               -80.03 Hz
PPMCM          0.50000 ppm/cm
HZCM         200.06500 Hz/cm
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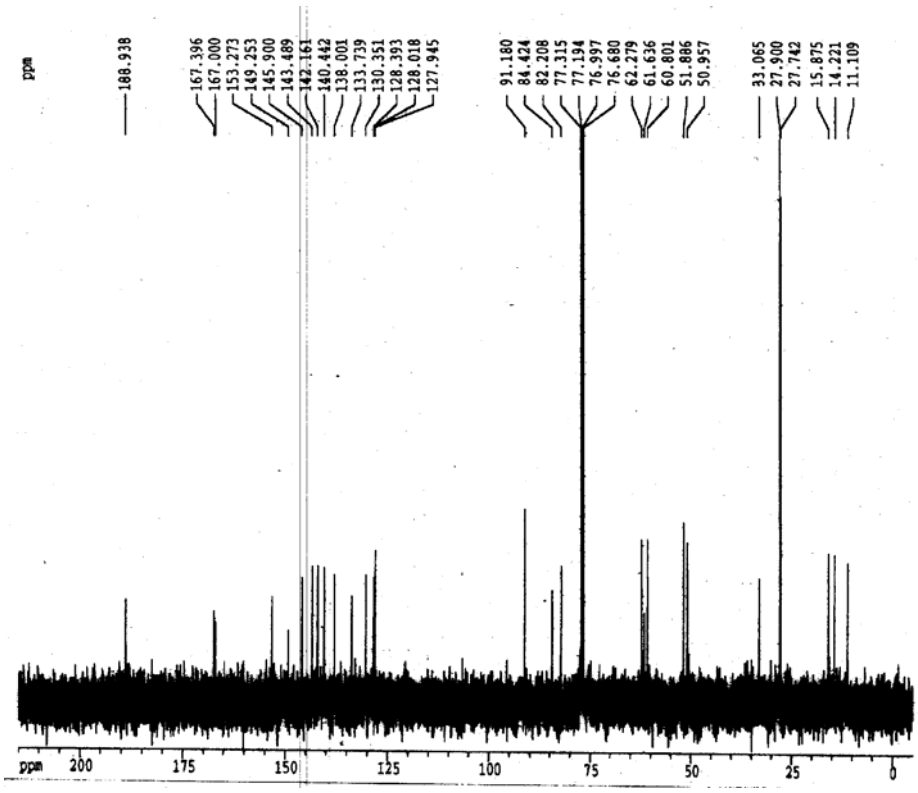


```
Current Data Parameters
NAME      Nov29-02-hayash
EXPNO              12
PROCNO              1

F2 - Acquisition Parameters
Date_          20021129
Time               0.30
INSTRUM          dpx400
PROBHD    5 mm BBO 13C-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  92
DS                   2
SWH          31847.133 Hz
FIDRES        0.485949 Hz
AQ            1.0289652 sec
RG                4096
DW               15.700 usec
DE                6.00 usec
TE               300.0 K
d11           0.03000000 sec
d12           0.00002000 sec
PL13             22.00 dB
D1            2.00000000 sec
CPDPRG2          waltz16
PCPD2             80.00 usec
SFO2        400.1316005 MHz
NUC2                1H
PL2               3.00 dB
PL12             22.00 dB
P1                9.30 usec
SFO1        100.6254358 MHz
NUC1                13C
PL1               2.00 dB

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

1D NMR plot parameters
CX               20.00 cm
F1P             215.000 ppm
F1             21631.75 Hz
F2P              -5.000 ppm
F2               -503.06 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
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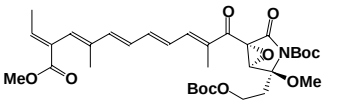

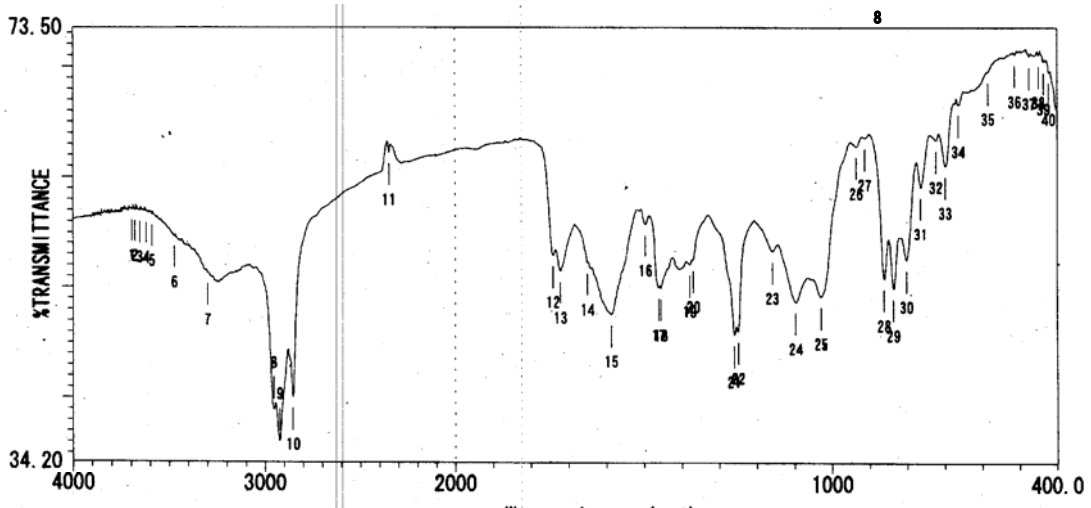

ファイル名	:		ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%
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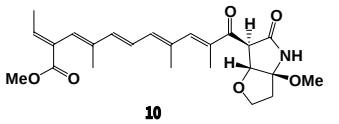
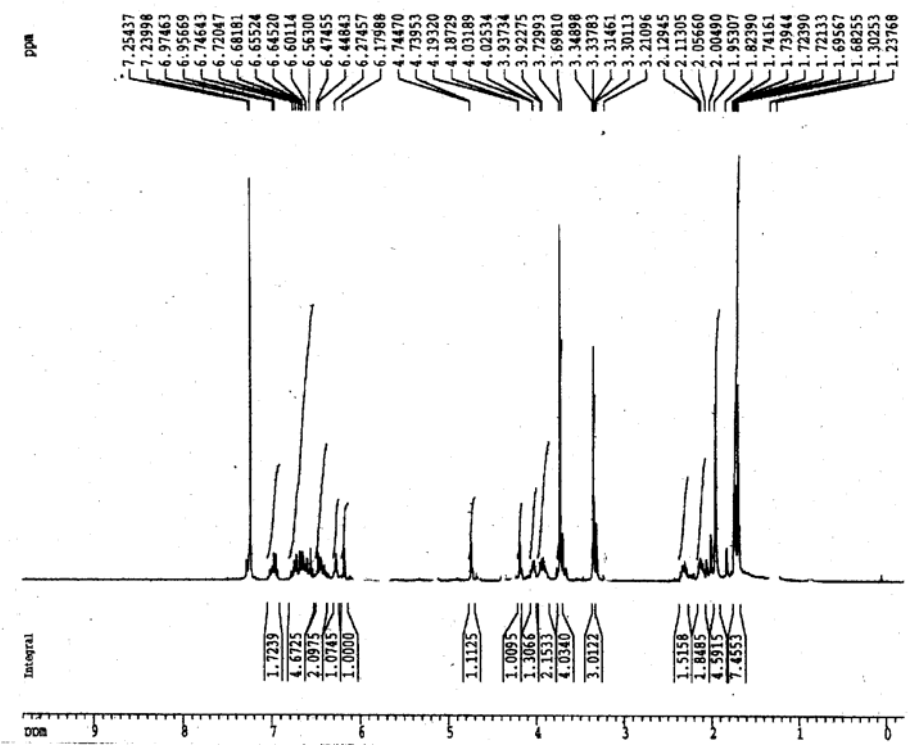
測定日時	:	2003年01月22日 02時5	02	3677.59	57.0306	12	1741.41	52.8742	22	1249.65	46.0069	32	723.175	63.3582
測定分解能	:	4 cm-1	03	3652.52	56.9173	13	1722.12	51.4731	23	1159.01	53.2435	33	698.105	61.0418
スキャン回数	:	10 回	04	3619.73	56.8530	14	1650.77	52.2042	24	1097.30	48.6795	34	663.393	66.5863
測定ゲイン	:	1	05	3588.88	56.5769	15	1587.13	47.5433	25	1029.80	49.1455	35	584.325	69.5487
コメント	:		06	3473.17	54.6572	16	1496.49	55.7503	26	935.306	62.7880	36	512.972	71.1102
			07	3299.61	51.2775	17	1461.78	49.9209	27	912.165	63.5500	37	474.403	70.9261
			08	2954.41	38.9896	18	1455.99	49.9061	28	862.025	50.7744	38	449.333	71.0366
			09	2923.56	36.0689	19	1378.85	52.0392	29	836.955	49.8847	39	435.834	70.4565
			10	2854.13	40.0250	20	1369.21	52.4751	30	802.242	52.4662	40	422.334	69.5268



10

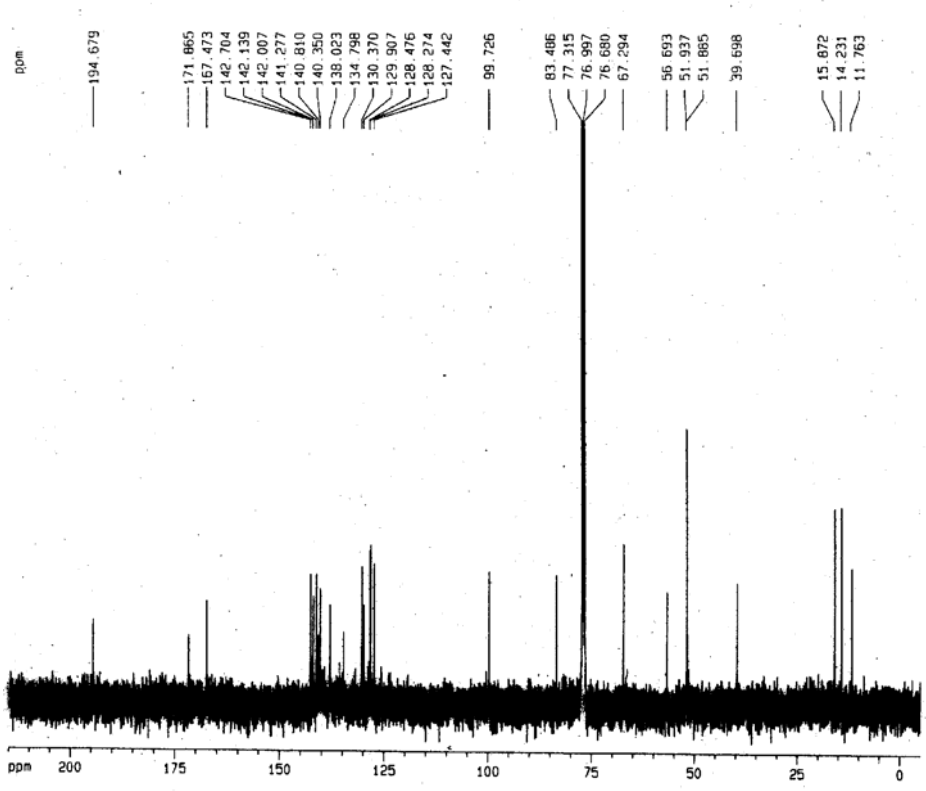



```
Current Data Parameters
NAME      Jan20-03-haysah
EXPNO              21
PROCNO             1

F2 - Acquisition Parameters
Date_          20030120
Time               4.29
INSTRUM          dpx400
PROBHD    5 mm BBO 13C-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   47
DS                    0
SWH           8221.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                 362
DW              60.800 usec
DE               6.00 usec
TE              300.0 K
D1            1.00000000 sec
P1               9.00 usec
SFO1        400.1324710 MHz
NUC1               1H
PL1              0.00 dB

F2 - Processing parameters
SI                16384
SF          400.1300172 MHz
WDW                EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
F1P               9.800 ppm
F1             3921.27 Hz
F2P              -0.200 ppm
F2               -80.03 Hz
PPMCM             0.50000 ppm/cm
HZCM            200.06500 Hz/cm
```




```
Current Data Parameters
NAME      Nov09-2004-haya
EXPNO              143
PROCNO             1

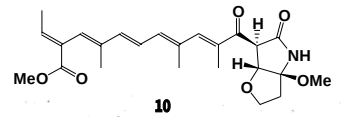
F2 - Acquisition Parameters
Date_          20041109
Time               22.13
INSTRUM          dpx400
PROBHD    5 mm QNP 1H/29
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  548
DS                    2
SWH          31847.133 Hz
FIDRES        0.485846 Hz
AQ            1.0288652 sec
RG              8752.6
DW              15.700 usec
DE               6.00 usec
TE              303.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               9.30 usec
PL1              3.00 dB
SFO1        100.6254358 MHz

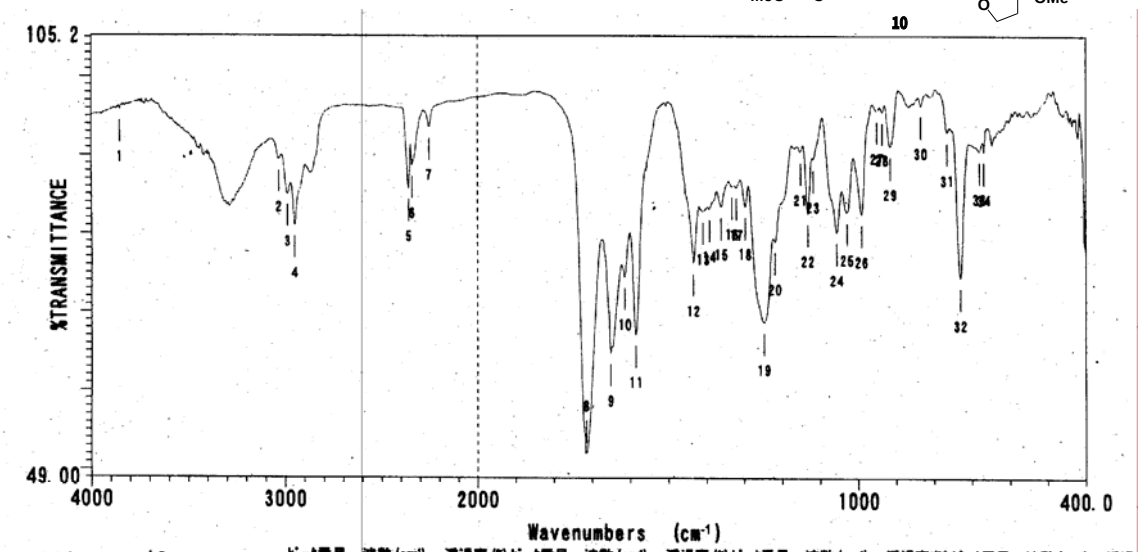
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           80.00 usec
PL2              3.00 dB
PL12            22.00 dB
PL13            22.00 dB
SFO2        400.1318005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127718 MHz
WDW                EM
SSB                   0
LB               1.00 Hz
GB                    0
PC                1.40

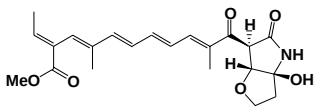
1D NMR plot parameters
CX               20.00 cm
CY                8.00 cm
F1P             215.000 ppm
F1            21631.75 Hz
F2P              -5.000 ppm
F2             -502.96 Hz
PPMCM            11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```



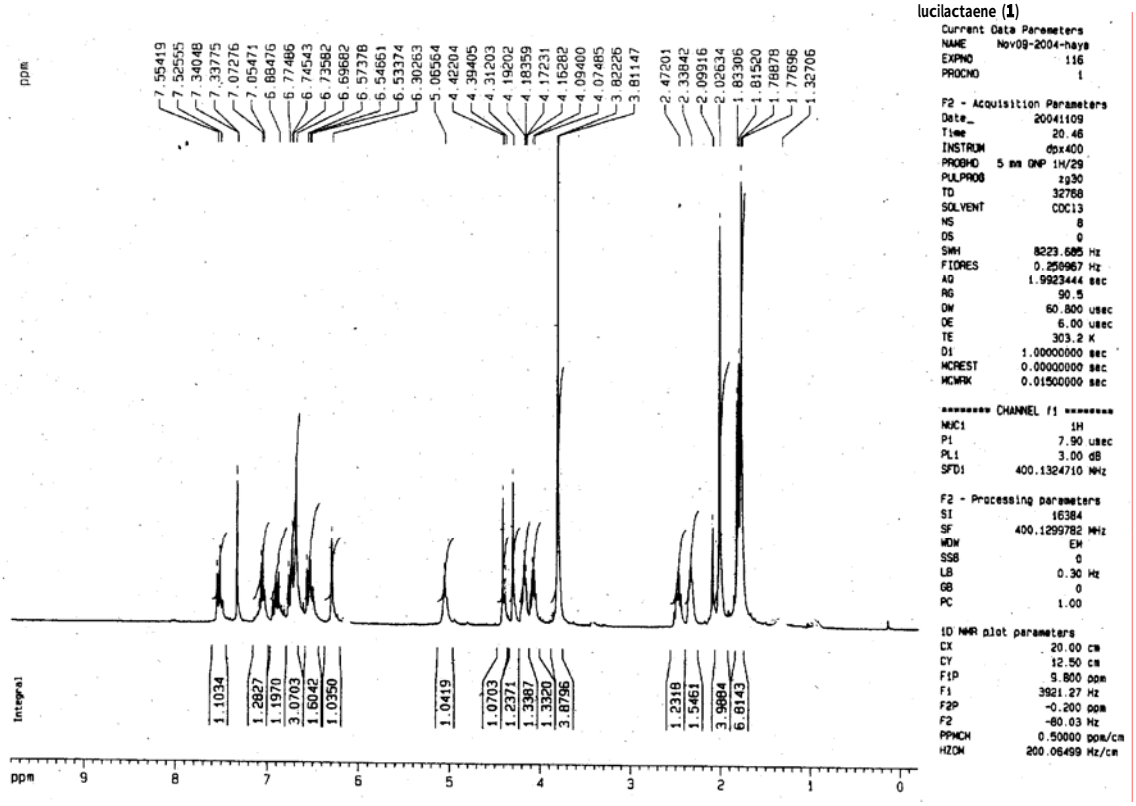
10



ファイル名	:	one	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過
タイトル	:		01	3853.08	95.7706	11	1585.20	67.1016	21	1161.29	90.3776	31	765.801	92.8
測定日時	:	2004年11月09日 23時02	02	3031.55	89.4125	12	1434.78	76.2389	22	1132.01	82.5554	32	730.889	74.4
測定分解能	:	4 cm-1	03	2987.20	85.0338	13	1409.71	82.7728	23	1118.51	89.4470	33	680.749	90.5
スキャン回数	:	10 回	04	2950.55	80.9949	14	1392.35	83.1444	24	1056.80	80.1598	34	669.178	90.4
測定ゲイン	:	1	05	2360.44	85.7131	15	1361.50	83.4041	25	1029.80	82.8943			
コメント	:		06	2341.16	88.8672	16	1332.57	86.0169	26	991.232	82.4611			
			07	2252.45	93.5280	17	1321.00	85.9093	27	950.734	95.8589			
			08	1714.41	52.0545	18	1297.86	83.4380	28	937.235	95.5048			
			09	1650.77	64.7460	19	1247.72	68.6801	29	918.022	91.0835			
			10	1614.13	74.4714	20	1218.79	78.9707	30	835.026	96.3196			



lucilactaene (**1**)

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EXPNO              116
PROCNO             1

F2 - Acquisition Parameters
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Time              20.46
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/29
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                 8
DS                 0
SWH          8223.685 Hz
FIDRES       0.250967 Hz
AQ           1.9923444 sec
RG                90.5
DW              60.800 usec
DE               6.00 usec
TE              303.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              7.90 usec
PL1             3.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                16384
SF         400.1299782 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00

1D NMR plot parameters
CX             20.00 cm
CY             12.50 cm
F1P             9.800 ppm
F1           3921.27 Hz
F2P            -0.200 ppm
F2            -80.03 Hz
PPMCM          0.50000 ppm/cm
HZCM         200.06499 Hz/cm
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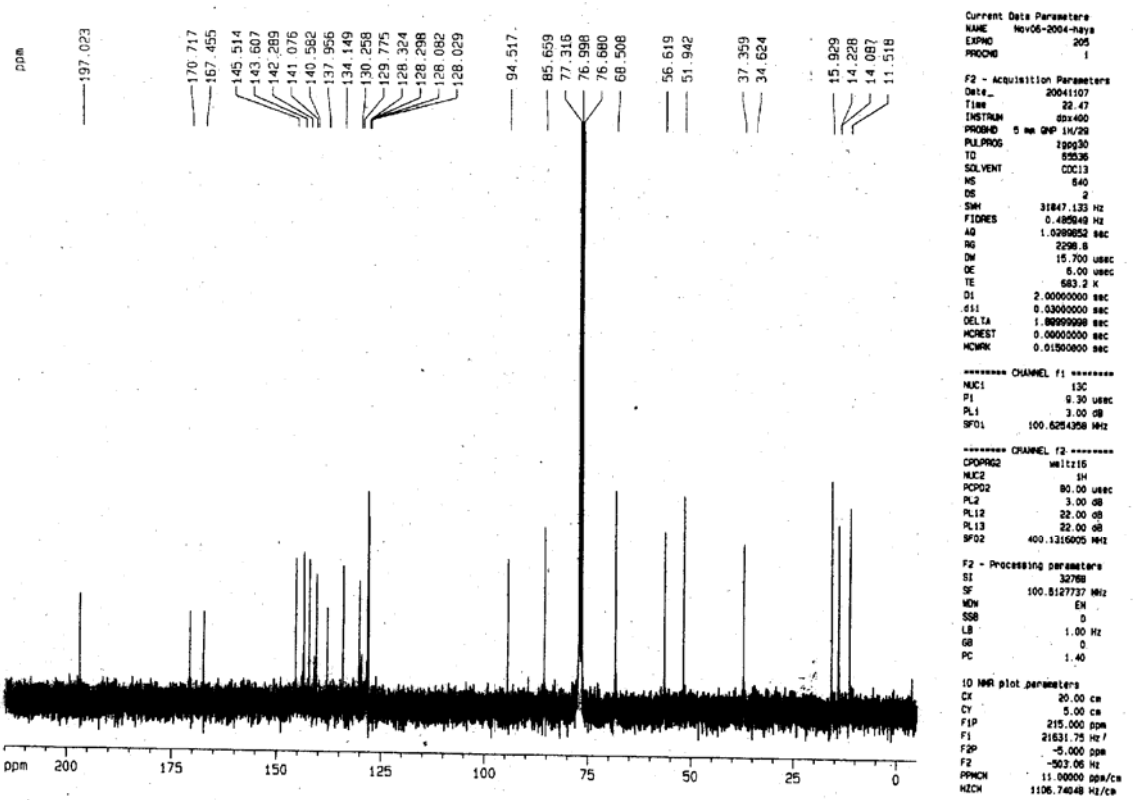


```
Current Data Parameters
NAME        Nov06-2004-haya
EXPNO              205
PROCNO             1

F2 - Acquisition Parameters
Date_          20041107
Time              22.47
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/29
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                640
DS                 2
SWH         31847.133 Hz
FIDRES       0.485948 Hz
AQ           1.0289852 sec
RG              2298.8
DW              15.700 usec
DE               6.00 usec
TE              583.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1              9.30 usec
PL1             3.00 dB
SFO1       100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           80.00 usec
PL2             3.00 dB
PL12           22.00 dB
PL13           22.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127737 MHz
WDW               EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40

1D NMR plot parameters
CX             20.00 cm
CY              5.00 cm
F1P           215.000 ppm
F1          21631.75 Hz
F2P            -5.000 ppm
F2           -503.06 Hz
PPMCM         11.00000 ppm/cm
HZCM       1106.74048 Hz/cm
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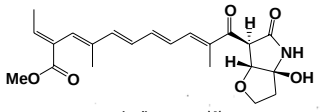
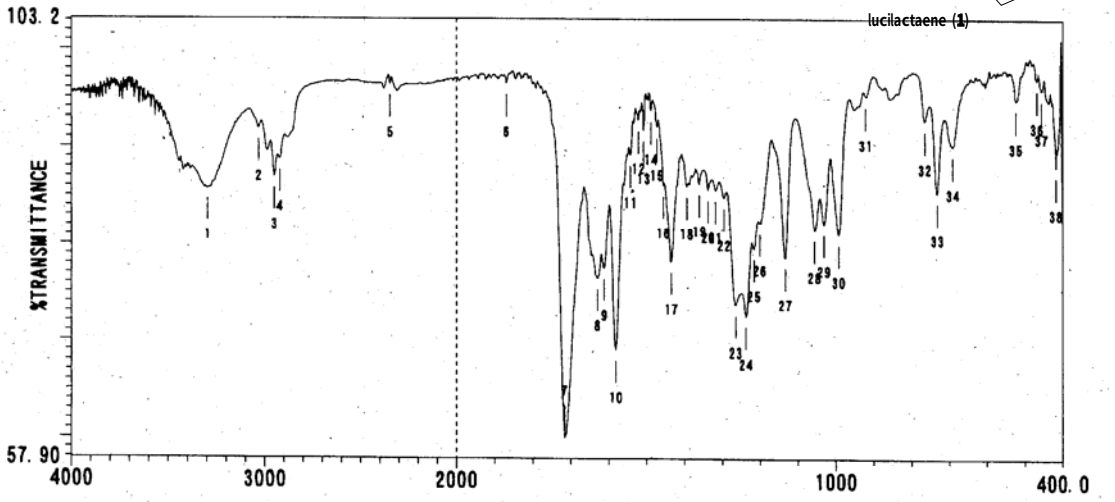
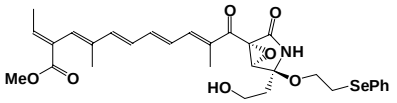


lucilactaene (1)

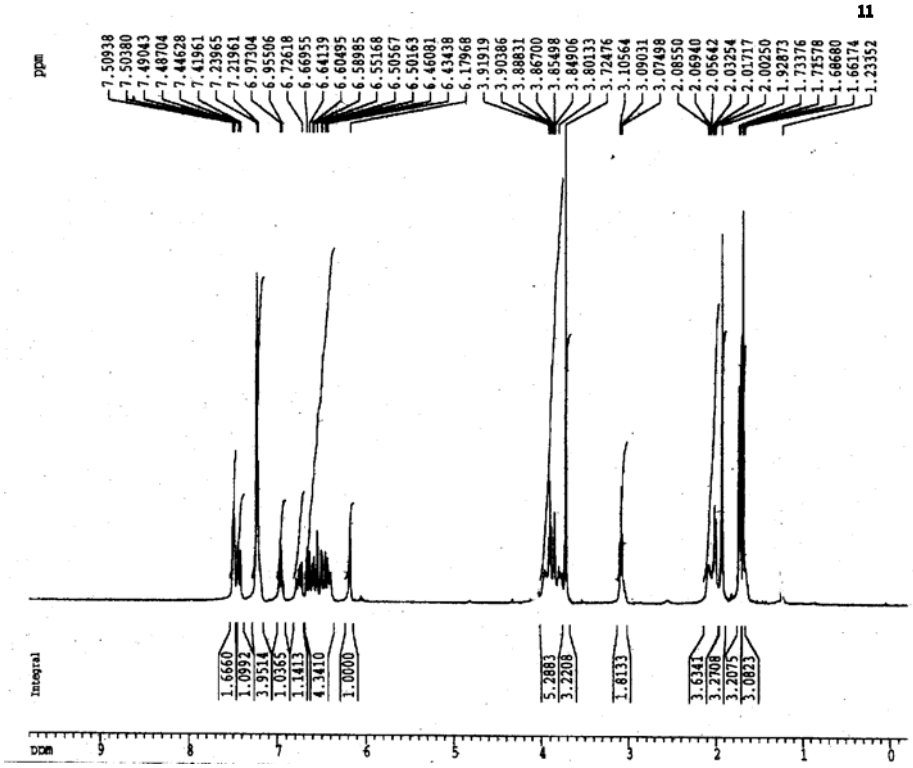


ファイル名 : lucilact
タイトル :
測定日時 : 2004年11月09日 21時47
測定分解能 : 4 cm-1
スキャン回数 : 10 回
測定ゲイン : 1
コメント :

ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過
01	3296.75	85.7958	11	1542.77	89.2476	21	1319.07	85.6085	31	921.807	95.1
02	3031.55	91.8629	12	1521.56	92.7338	22	1295.93	84.8254	32	765.601	92.7
03	2950.55	86.9150	13	1508.06	91.7210	23	1265.07	73.7160	33	732.817	85.3
04	2923.56	88.7297	14	1488.78	93.6746	24	1238.08	72.5292	34	692.320	90.1
05	2346.94	96.3783	15	1473.35	92.0665	25	1216.86	79.5637	35	524.543	94.8
06	1888.68	96.4705	16	1455.99	86.1124	26	1201.43	82.2396	36	488.617	96.9
07	1716.34	60.0226	17	1434.78	78.1981	27	1133.94	78.8190	37	457.047	95.8
08	1629.55	76.4746	18	1394.28	86.0154	28	1056.80	81.5003	38	418.477	88.0
09	1612.20	77.5252	19	1361.50	86.2840	29	1031.73	82.1447			
10	1581.34	69.0803	20	1338.36	85.6347	30	993.160	81.0403			



11

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Current Data Parameters
NAME        Feb18-03-hayash
EXPNO               91
PROCNO               1

F2 - Acquisition Parameters
Date_          20030218
Time              19.23
INSTRUM          dpx400
PROBHD    5 mm QNP 1H/2
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                  128
DW               60.800 usec
DE                6.00 usec
TE               300.0 K
D1            1.00000000 sec
P1                7.90 usec
SFO1        400.1324710 MHz
NUC1                1H
PL1               3.00 dB

F2 - Processing parameters
SI                16384
SF          400.1300177 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.800 ppm
F1              3921.27 Hz
F2P              -0.200 ppm
F2               -80.03 Hz
PPMCM             0.50000 ppm/cm
HZCM            200.06500 Hz/cm
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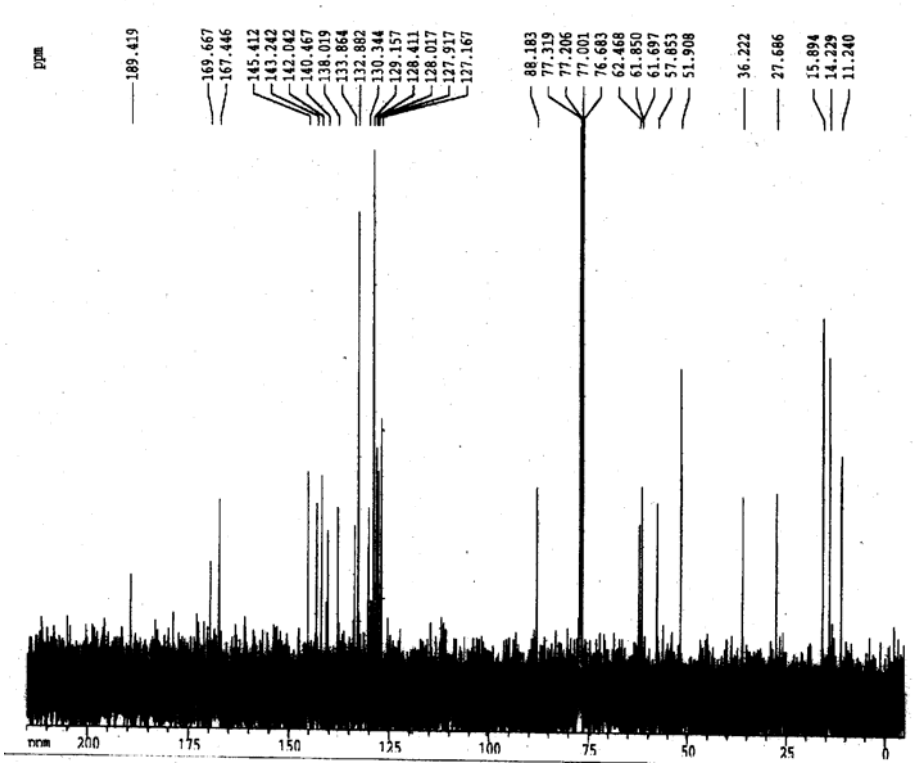


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Current Data Parameters
NAME        Feb18-03-hayash
EXPNO               94
PROCNO               1

F2 - Acquisition Parameters
Date_          20030218
Time              19.49
INSTRUM          dpx400
PROBHD    5 mm QNP 1H/2
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  373
DS                    2
SWH           31847.133 Hz
FIDRES        0.485949 Hz
AQ            1.0289652 sec
RG                812.7
DW               15.700 usec
DE                6.00 usec
TE               300.0 K
d11           0.03000000 sec
d12           0.00002000 sec
PL13             22.00 dB
D1            2.00000000 sec
CPDPRG2          waltz16
PCPD2             80.00 usec
SFO2        400.1316005 MHz
NUC2                1H
PL2               3.00 dB
PL12             22.00 dB
P1                9.30 usec
SFO1        100.6254358 MHz
NUC1                13C
PL1               3.00 dB

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

1D NMR plot parameters
CX                20.00 cm
F1P             215.000 ppm
F1              21631.75 Hz
F2P              -5.000 ppm
F2               -503.06 Hz
PPMCM            11.00000 ppm/cm
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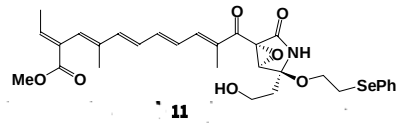
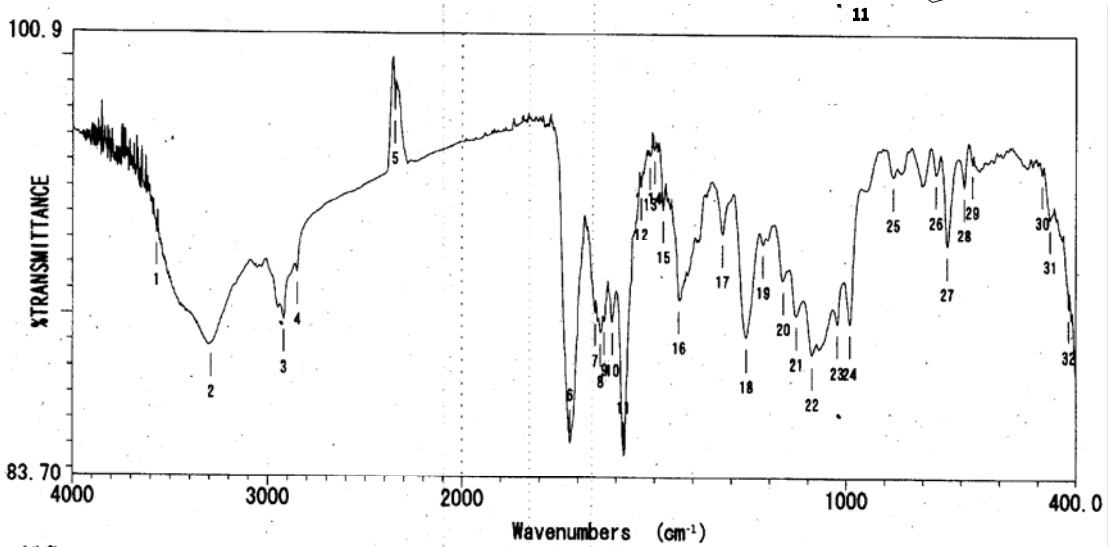
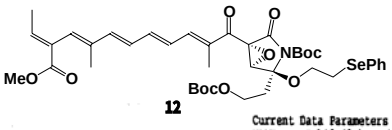


11

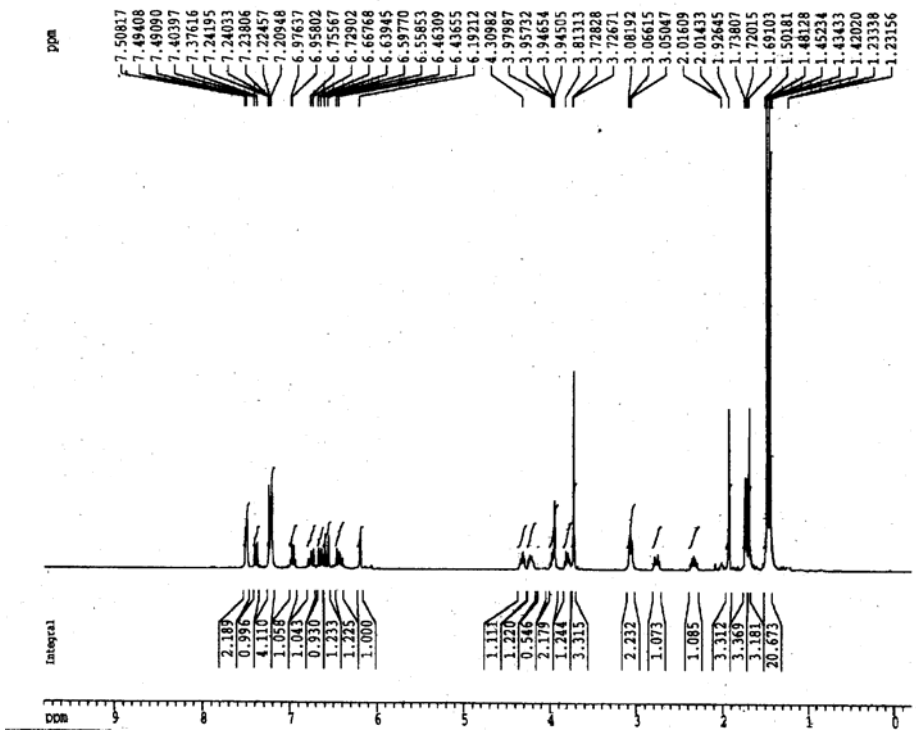


ファイル名 :
タイトル :
測定日時 : 2003年02月18日 23時44分34秒
測定分解能 : 4 cm-1
スキャン回数 : 10 回
測定ゲイン : 1
コメント :

ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号
01	3569.59	93.1211	10	1610.27	89.7342	19	1216.86	92.7504	28
02	3291.89	88.8204	11	1581.34	84.5831	20	1164.79	91.3425	29
03	2919.70	89.7742	12	1535.06	95.0055	21	1130.08	89.9830	30
04	2850.27	91.6203	13	1511.92	96.1929	22	1089.58	88.4529	31
05	2346.94	97.9259	14	1500.35	96.4248	23	1024.02	89.6714	32
06	1720.19	85.0725	15	1477.21	94.1116	24	991.232	89.6625	
07	1654.62	90.0908	16	1436.71	90.5833	25	877.452	95.4303	
08	1641.13	89.3021	17	1322.93	93.1651	26	765.601	95.5216	
09	1631.48	89.7560	18	1261.22	89.1324	27	736.674	92.7227	



12

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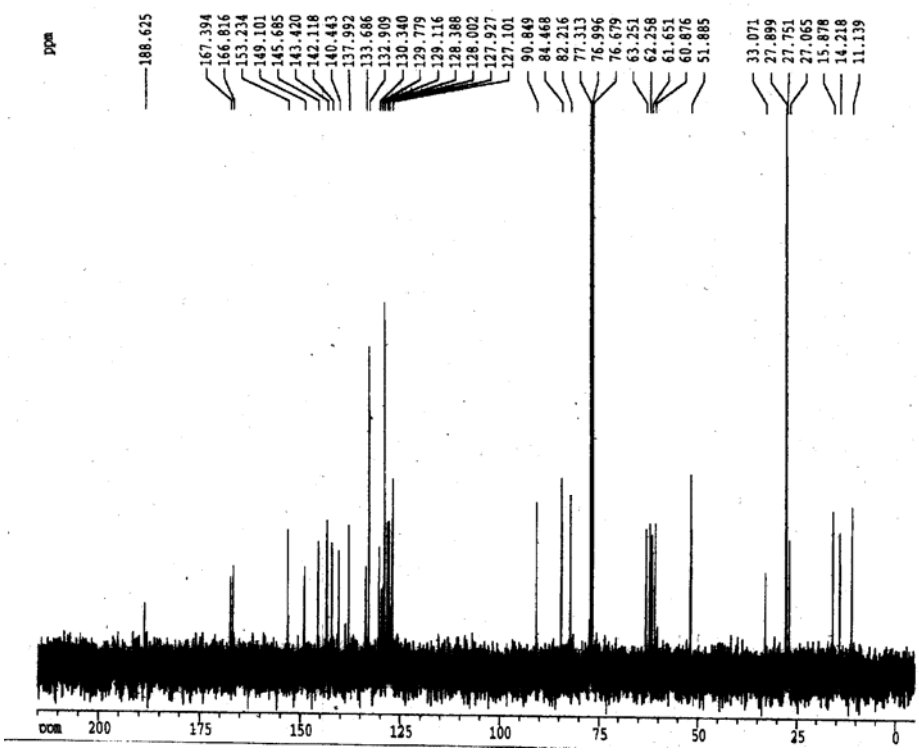
Current Data Parameters
NAME      Feb19-03-hayash
EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_          20030219
Time               5.14
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/2
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             1.9923444 sec
RG                 50.8
DW                60.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
P1                 7.90 usec
SFO1         400.1324710 MHz
NUC1                1H
PL1                3.00 dB

F2 - Processing parameters
SI                16384
SF          400.1300172 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.800 ppm
F1              3921.27 Hz
F2P              -0.200 ppm
F2               -80.03 Hz
PPMCM          0.50000 ppm/cm
HZCM           200.06500 Hz/cm
```

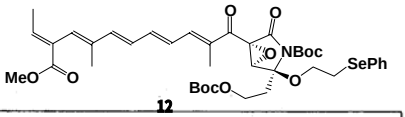



```
Current Data Parameters
NAME      Feb19-03-hayash
EXPNO              12
PROCNO              1

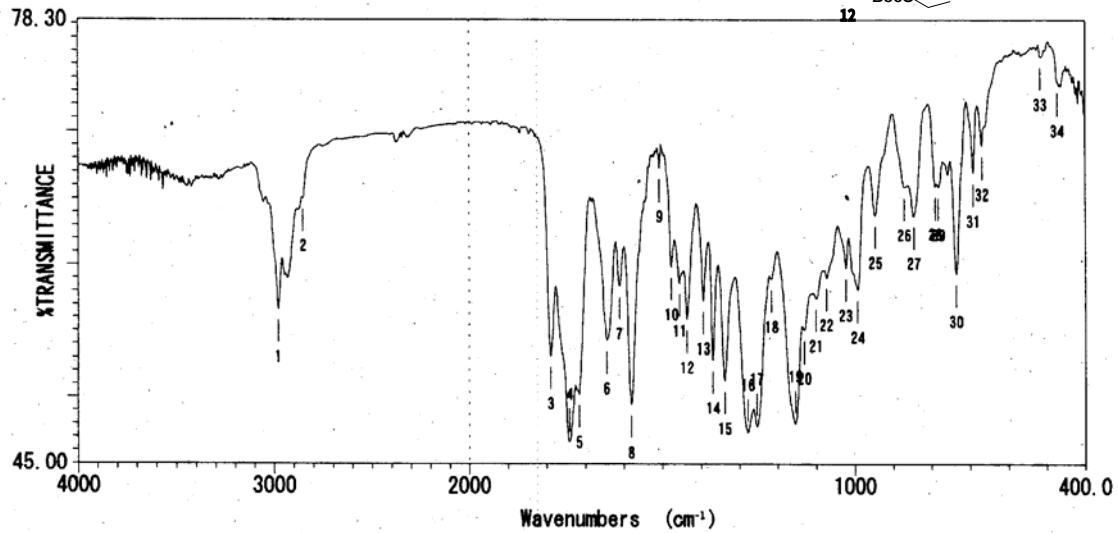
F2 - Acquisition Parameters
Date_          20030219
Time               5.17
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/2
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  193
DS                    2
SWH           31847.133 Hz
FIDRES         0.485949 Hz
AQ             1.0289652 sec
RG                 2048
DW                15.700 usec
DE                 6.00 usec
TE                300.0 K
d11            0.03000000 sec
d12            0.00002000 sec
PL13              22.00 dB
D1             2.00000000 sec
CPDPRG2          waltz16
PCPD2             80.00 usec
SFO2         400.1316005 MHz
NUC2                1H
PL2                3.00 dB
PL12              22.00 dB
P1                 9.30 usec
SFO1         100.6254358 MHz
NUC1                13C
PL1                3.00 dB

F2 - Processing parameters
SI                32768
SF          100.6127718 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.75 Hz
F2P              -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
```

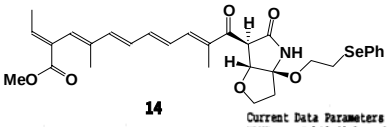


12

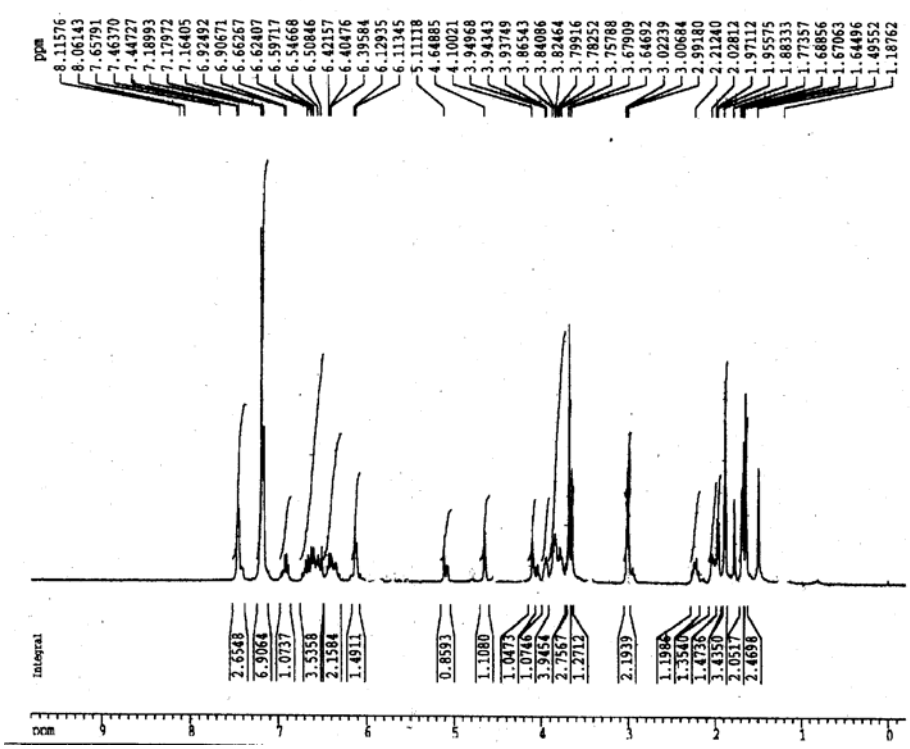


ファイル名 :
タイトル :
測定日時 : 2003年02月19日 22時18分05秒
測定分解能 : 4 cm-1
スキャン回数 : 10 回
測定ゲイン : 1
コメント :

ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号
01	2981.41	56.5869	10	1477.21	59.7999	19	1155.15	47.9651	28
02	2854.13	65.0075	11	1455.99	58.5672	20	1132.01	55.0073	29
03	1789.62	53.0915	12	1436.71	55.8154	21	1101.15	57.4285	30
04	1741.41	46.6308	13	1394.28	57.2174	22	1074.16	58.9991	31
05	1716.34	50.1972	14	1369.21	52.7648	23	1024.02	59.7367	32
06	1644.98	54.2324	15	1338.36	51.2212	24	993.160	58.0745	33
07	1612.20	58.3385	16	1278.57	47.3281	25	946.877	63.7509	34
08	1581.34	49.4237	17	1255.43	47.7692	26	871.667	65.8584	
09	1508.06	57.2371	18	1216.86	58.8818	27	846.597	63.7005	



14

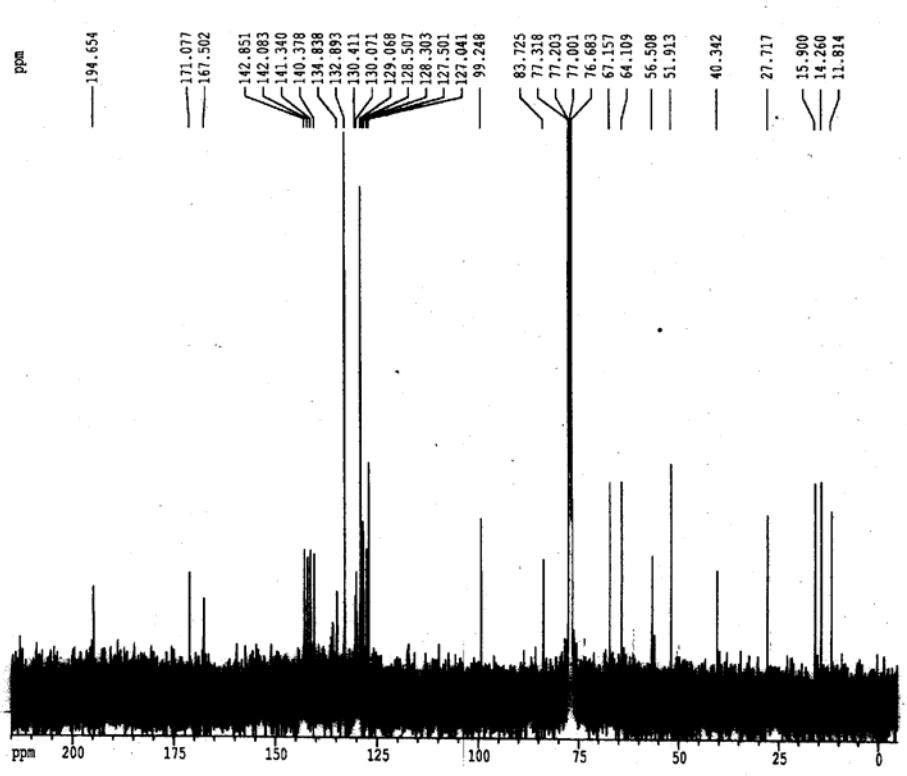


Current Data Parameters
NAME Feb19-03-hayash
EXPNO 72
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030219
Time 21.03
INSTRUM dpx400
PROBHD 5 mm QNP 1H/2
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 203.2
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.90 usec
SFO1 400.1324710 MHz
NUC1 1H
PL1 3.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300378 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.800 ppm
F1 3921.27 Hz
F2P -0.200 ppm
F2 -80.03 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06502 Hz/cm

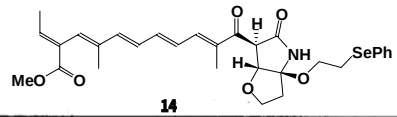


Current Data Parameters
NAME Mar01-03-hayash
EXPNO 13
PROCNO 1

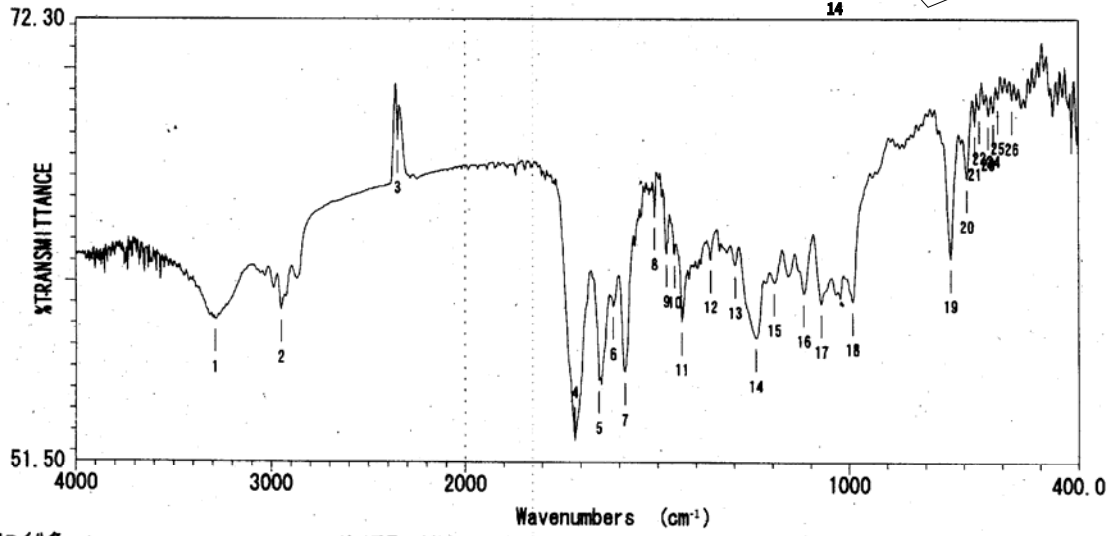
F2 - Acquisition Parameters
Date_ 20030301
Time 7.12
INSTRUM dpx400
PROBHD 5 mm QNP 1H/2
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6548
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 4096
DW 15.700 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.00002000 sec
PL13 22.00 dB
D1 2.00000000 sec
CPDPRG2 waltz16
PCPD2 80.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 3.00 dB
PL12 22.00 dB
P1 9.30 usec
SFO1 100.6254358 MHz
NUC1 13C
PL1 3.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -5.400 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.74048 Hz/cm

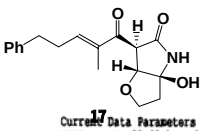


14

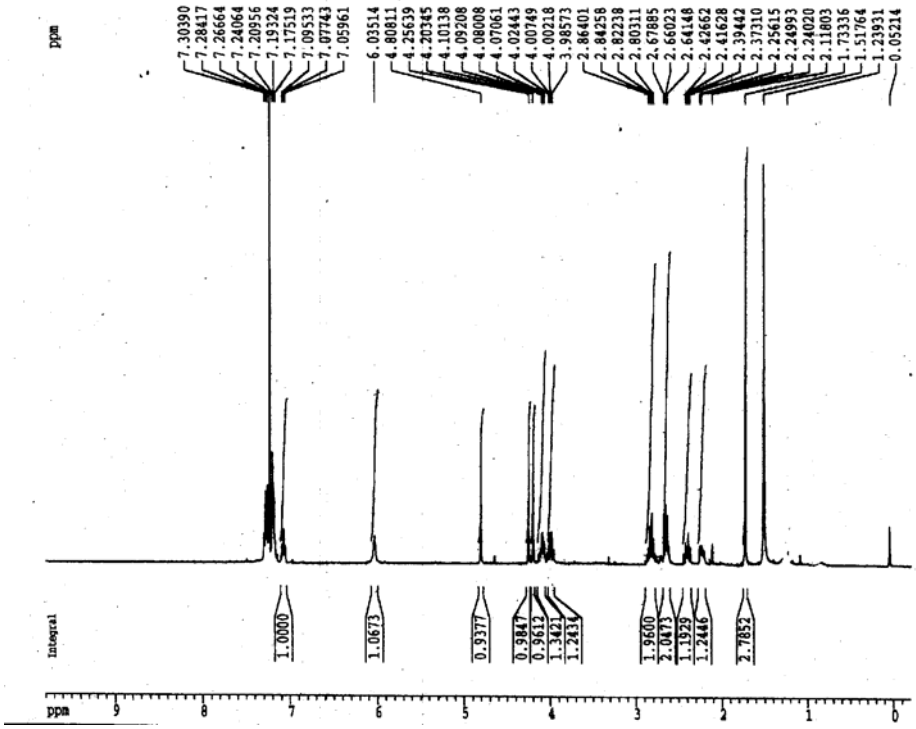


ファイル名 :
タイトル :
測定日時 : 2003年02月19日 22時26分13秒
測定分解能 : 4 cm⁻¹
スキャン回数 : 10 回
測定ゲイン : 1
コメント :

ピーク番号	波数 (cm⁻¹)	透過率 (%)	ピーク番号	波数 (cm⁻¹)	透過率 (%)	ピーク番号	波数 (cm⁻¹)	透過率 (%)
01	3286.11	58.1718	10	1455.99	61.2661	19	734.746	61.1017
02	2948.63	58.6288	11	1436.71	58.0939	20	692.320	64.8968
03	2348.87	66.5758	12	1361.50	61.0443	21	671.106	67.3704
04	1716.34	52.5272	13	1297.86	60.7639	22	659.536	68.1234
05	1652.70	55.3588	14	1243.86	57.3265	23	636.394	67.8301
06	1616.06	58.8114	15	1195.65	59.9453	24	622.895	67.9781
07	1585.20	55.7364	16	1118.51	59.4218	25	611.324	68.5942
08	1508.06	63.0721	17	1072.23	58.9369	26	574.683	68.5542
09	1477.21	61.3095	18	991.232	59.0415			



17

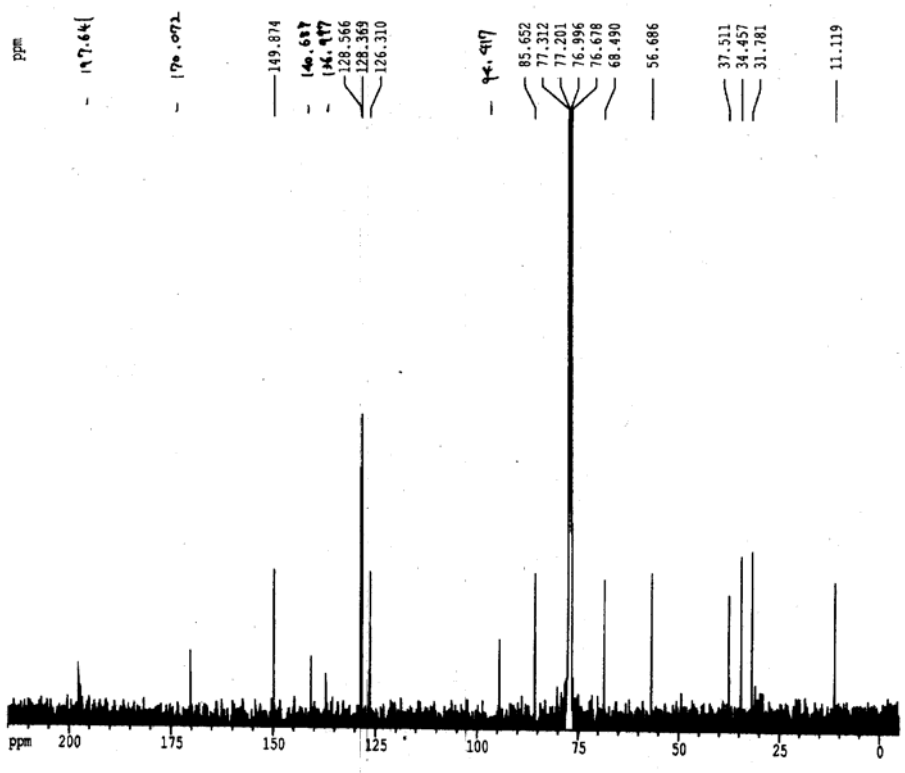


Current Data Parameters
NAME Nov23-02-hayash
EXPNO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021123
Time 18.04
INSTRUM dpx400
PROBHD 5 mm BBO 13C-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 48
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 9.00 usec
SFO1 400.1324710 MHz
NUC1 1H
PL1 3.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.800 ppm
F1 3921.27 Hz
F2P -0.200 ppm
F2 -80.03 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

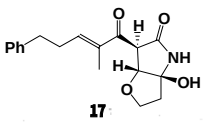


Current Data Parameters
NAME Feb26-03-hayash
EXPNO 94
PROCNO 1

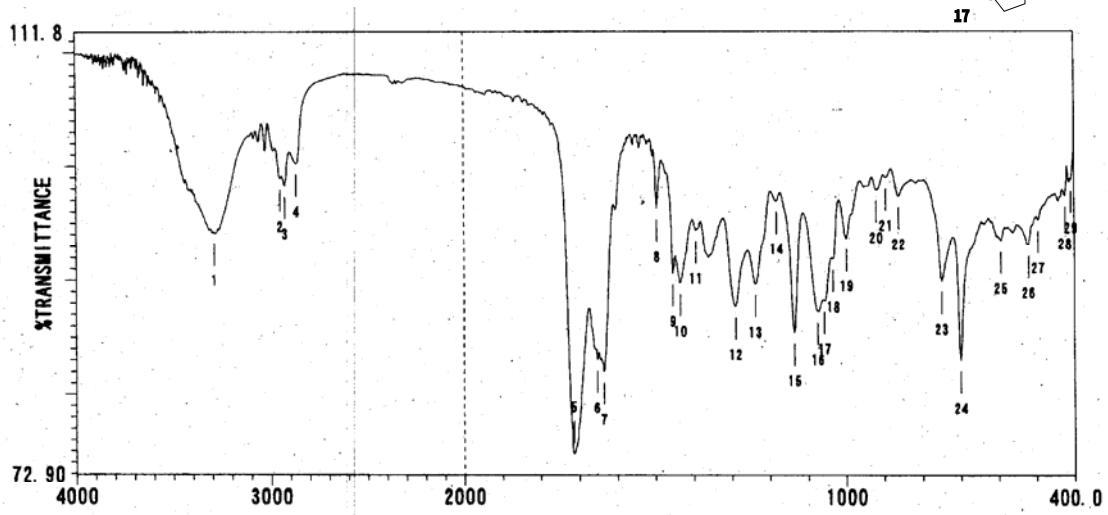
F2 - Acquisition Parameters
Date_ 20030226
Time 21.44
INSTRUM dpx400
PROBHD 5 mm QNP 1H/2
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 14162
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 2048
DW 15.700 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.00002000 sec
PL13 22.00 dB
D1 2.00000000 sec
CPDPRG2 waltz16
PCPD2 80.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 3.00 dB
PL12 22.00 dB
P1 9.30 usec
SFO1 100.6254358 MHz
NUC1 13C
PL1 3.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.74048 Hz/cm



17



ファイル名	: Luci モデル	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)
タイトル	:	01	3289.96	94.1677	11	1394.28	94.3530	21	894.809	98.9859
測定日時	: 2004年11月10日 12時25	02	2950.55	98.8918	12	1290.14	87.6829	22	862.025	97.3457
測定分解能	: 4 cm-1	03	2927.41	98.2601	13	1238.08	89.6489	23	750.174	89.9145
スキャン回数	: 10 回	04	2867.63	100.215	14	1182.15	96.9516	24	700.033	82.9887
測定ゲイン	: 1	05	1714.41	74.7380	15	1135.87	85.3934	25	593.968	93.4931
コメント	:	06	1652.70	83.0181	16	1074.16	87.2381	26	520.686	93.0893
		07	1635.34	81.9820	17	1056.80	88.1683	27	495.616	95.2760
		08	1496.49	96.2877	18	1033.66	91.8522	28	424.263	97.3976
		09	1454.08	90.5979	19	998.946	93.6849	29	408.835	98.7917
		10	1434.78	89.7242	20	919.879	97.9257			