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# Supporting Information

# Highly Diastereo- and Enantioselective, Direct Aldol Reaction of Aldehyde and Ketone Catalyzed by Siloxyproline in the Presence of Water

Seiji Aratake, Takahiko Itoh, Tsubasa Okano, Norio Nagae, Tatsunobu Sumiya, Mitsuru Shoji, Yujiro Hayashi*

# Supporting Information

## Highly Diastereo- and Enantioselective Direct Aldol Reaction in the Presence of Water

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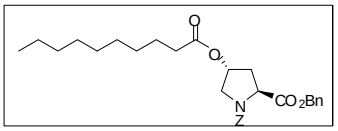
### Typical procedure for a synthesis of (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid

**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester (9c)**



To a solution of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester **8** (2.34 g, 4.50 mmol) and catalytic amount of 4-dimethylaminopyridine in pyridine (9.0 mL) was added *n*-decanoyl chloride (1.37 mL, 6.75 mmol) successively at 0 °C. After stirring the reaction mixture for 18h at that temperature to room temperature, the reaction was quenched with saturated $NaHCO_3$ aqueous solution and the organic materials were extracted with ethyl acetate three times. The combined organic extracts were washed with saturated $NaHCO_3$ aqueous solution and brine each three times, dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel chromatography (ethyl acetate : hexane = 1 : 12) gave (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c** (2.06 g, 3.96 mmol) in 88% yield as a colorless oil.

Data are shown as a mixture of two conformers.

$^1$H NMR ($CDCl_3$, 400MHz): δ 0.81-0.89 (3H, m), 1.24 (14H, br-s), 2.12-2.28 (3H, m), 2.31-2.43 (1H, m), 3.58-3.82 (2H, m), 4.43-4.58 (1H, m), 4.98 (1H, s), 5.03 (1H, s), 5.11-5.27 (3H, m), 7.15-7.37 (10H, m);

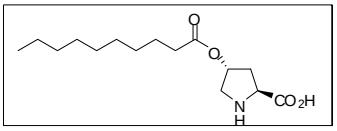
$^{13}$C NMR ($CDCl_3$, 100MHz): δ 14.0, 22.6, 24.7, 29.0, 29.1, 29.14, 29.3, 29.4, 31.8, 34.1, 35.5, 36.6, 52.1, 52.6, 57.7, 58.0, 66.9, 67.0, 67.3, 71.5, 72.2, 127.8, 128.0, 128.001, 128.1, 128.12, 128.2, 128.4, 128.42, 128.5, 135.2, 135.4, 136.2, 136.3, 154.1, 154,7, 171.7, 171.9, 173.0, 173.1;

IR (neat): ν 2927, 1743, 1735, 1455, 1417, 1353, 1162, 1120, 1064 cm$^{-1}$;

HRMS (FAB): [M+H] calcd for [$C_{30}H_{40}O_6N$]: 510.2777, found: 510.2840;

$[\alpha]_D^{22}$-39.5 (*c* = 0.82, $CHCl_3$).

**(2*S*, 4*R*)-4-Decanoyloxypyrrolidine-2-carboxylic acid (4c)**



To a solution of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c** (2.06 g, 3.96 mmol) in ethyl acetate (15.7 mL) was added 20% $Pd(OH)_2$/C (200 mg, 0.29 mmol) at room temperature, and the reaction mixture was stirred for 15h under $H_2$ atomosphere. Warmed MeOH (20 ml) was added, and the reaction mixture was filtered through a pad of Celite, and concentrated in vacuo. The residual solid was washed with ethyl acetate to afford (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid **4c** (1.12 g, 3.96 mmol) in quantitative yield as a white solid.

$^1$H NMR ($CD_3OD$, 600MHz): δ 0.93 (3H, t, *J*=6.8 Hz), 1.28-1.42 (14H, m), 1.63-1.68 (2H, m), 2.26-2.35 (1H, m), 2.37-2.43 (2H, m), 2.49-2.55 (1H, m), 3.37-3.44 (1H, m), 3.61-3.67 (1H, m), 4.19 (1H, dd, *J*=7.8, 10.2 Hz), 5.43 (1H, br-s);

$^{13}$C NMR ($CD_3OD$, 150MHz): δ 15.3, 24.6, 26.7, 31.0, 31.3, 31.4, 33.9, 35.7, 37.5, 52.8, 62.4, 75.5, 173.8, 175.2;

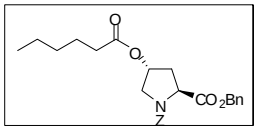
IR (KBr): ν 2852, 1736, 1620, 1579, 1441, 1417, 1383, 1213, 1165, 640 cm$^{-1}$;

HRMS (FAB): [M+H] calcd for [$C_{15}H_{28}O_4N$]: 286.2018, found: 286.2036;

$[\alpha]_D^{22}$-29.4 (*c* = 0.13, MeOH).

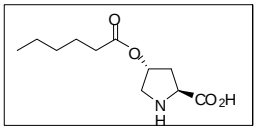
**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-hexanoyloxypyrrolidine-2-carboxylic acid benzyl ester (9a)**

(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-hexanoyloxypyrrolidine-2-carboxylic acid



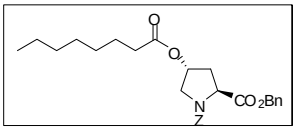
benzyl ester **9a** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester **8** and hexanoyl chloride by the procedure described for the synthesis of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c**.
Data are shown as a mixture of two conformers.
$^1$H NMR ($CDCl_3$, 400 MHz): δ 0.86 (3H, t, *J*=6.2 Hz), 1.15-1.33 (4H, m), 1.42-1.62 (2H, m), 2.11-2.44 (4H, m), 3.59-3.81 (2H, m), 4.43-4.54 (1H, m), 4.94-5.30 (5H, m), 7.14-7.39 (10H, m);
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 13.8, 22.2, 24.4, 31.1, 34.1, 35.5, 36.6, 52.1, 52.6, 57.7, 58.0, 66.9, 67.0, 67.2, 71.5, 72.3, 127.8, 128.0, 128.1, 128.13, 128.2, 128.4, 128.42, 128.5, 135.2, 135.4, 136.2, 136.3, 154.1, 154.7, 171.7, 171.9,173.0, 173.1;
IR (neat): ν 2955, 1736, 1716, 1417, 1354, 1165, 1122, 1066, 750, 698 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{26}H_{32}O_6N$]: 454.2152, found: 454.2230;
$[\alpha]_D^{22}$-45.2 (*c* = 0.88, $CHCl_3$).

**(2*S*, 4*R*)-4-Hexanoyloxypyrrolidine-2-carboxylic acid (4a)**



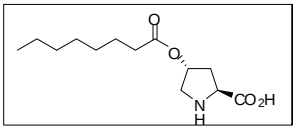
(2*S*, 4*R*)-4-Hexanoyloxypyrrolidine-2-carboxylic acid **4a** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hexanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9a** by the procedure described for the synthesis of (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid **4c**.
$^1$H NMR ($CDCl_3$, 600 MHz): δ 0.83 (3H, t, *J*=7.0 Hz), 1.16-1.31 (4H, m), 1.54-1.57 (2H, m), 2.18-2.27 (3H, m), 2.39-2.43 (1H, m), 3.32-3.34 (1H, m), 3.57-3.59 (1H, m), 4.10 (1H, br-t, *J*=7.4 Hz), 5.29 (1H, br-s);
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 13.8, 22.2, 24.2, 31.1, 33.9, 35.4, 50.4, 60.0, 72.7, 172.4, 173.2;
IR (KBr): ν 2960, 1730, 1624, 1577, 1441, 1419, 1250, 1173, 1038, 640 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{11}H_{20}O_4N$]: 230.1392, found: 230.1389;
$[\alpha]_D^{22}$-25.1 (*c* = 0.11, MeOH).

**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-octanoyloxypyrrolidine-2-carboxylic acid benzyl ester (9b)**



(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-octanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9b** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester **8** and octanoyl chloride by the procedure described for the synthesis of (2*S*, 4*R*)-*N*-benzyloxycarbonyl -4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c**.
Data are shown as a mixture of two conformers.
$^1$H NMR ($CDCl_3$, 400 MHz): δ 0.85 (3H, t, *J*=7.0 Hz), 1.25 (8H, br-s), 1.40-1.62 (2H, m), 2.12-2.42 (4H, m), 3.59-3.80 (2H, m), 4.43-4.54 (1H, m), 4.91-5.30 (5H, m), 7.18-7.38 (10H, m);
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 14.0, 22.5, 24.8, 28.8, 31.5, 34.2, 35.6, 36.6, 52.2, 52.6, 57.7, 58.0, 66.9, 67.0, 67.3, 71.5, 72.3, 127.8, 128.0, 128.1, 128.14, 128.3, 128.4, 128.44, 128.5, 135.2, 135.4, 136.2, 136.3, 154.1, 154.7, 171.7, 172.0, 173.1, 173.12;
IR (neat): ν 2955, 1736, 1716, 1417, 1354, 1165, 1122, 1066, 750, 698 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{28}H_{36}O_6N$]: 481.2464, found: 481.2525;
$[\alpha]_D^{22}$-41.8 (*c* = 0.83, $CHCl_3$).

**(2*S*, 4*R*)-4-Octanoyloxypyrrolidine-2-carboxylic acid (4b)**

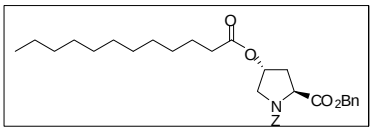


(2*S*, 4*R*)-4-Octanoyloxypyrrolidine-2-carboxylic acid **4b** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-octanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9b** by the procedure described for the synthesis of (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid **4c**.
$^1$H NMR ($CDCl_3$, 600 MHz): δ 0.80-0.88 (3H, m), 1.25 (8H, br-s), 1.56 (2H, br-s), 2.20-2.31 (3H, m), 2.36-2.45 (1H, m), 3.28-3.41 (1H, m), 3.54-3.67 (1H, m), 4.12 (1H, br-s), 5.27 (1H, br-s);
$^{13}$C NMR ($CDCl_3$, 150 MHz): δ 14.0, 22.5, 24.6, 28.8, 29.0, 31.5, 33.9, 35.4, 50.2, 59.9, 72.5, 172.7, 173.2;
IR (KBr): ν 2960, 2929, 1736, 1624, 1577, 1441, 1419, 1383, 1227, 1167 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{13}H_{24}O_4N$]: 258.1705, found: 258.1711;
$[\alpha]_D^{22}$-37.0 (*c* = 0.09, MeOH).

**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-dodecanoyloxypyrrolidine-2-carboxylic acid benzyl ester (9d)**

(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-dodecanoyloxypyrrolidine-2- carboxylic acid benzyl ester **9d** was

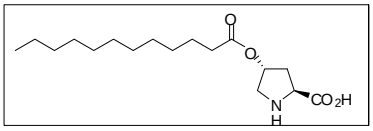
prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine -2-carboxylic acid benzyl ester **8** and dodecanoyl chloride by the procedure described for the synthesis of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c**.



Data are shown as a mixture of two conformers.
$^1$H NMR ($CDCl_3$, 400MHz): δ 0.86 (3H, t, *J*=7.1 Hz), 1.23 (16H, br-s), 1.45-1.65 (2H, m), 2.14-2.28 (2H, m), 2.29-2.42 (2H, m), 3.59-3.80 (2H, m), 4.41-4.55 (1H, m), 4.98 (1H, s), 5.04 (1H, s), 5.11-5.31 (3H, m), 7.17-7.35 (10H, m);
$^{13}$C NMR ($CDCl_3$, 100MHz): δ 14.0, 22.6, 24.8, 28.8, 29.0, 29.1, 29.2, 29.3, 29.4, 29.5, 31.8, 34.2, 35.6, 36.6, 52.2, 52.6, 57.8, 58.0, 67.0, 67.1, 67.3, 71.5, 72.3, 127.8, 128.0, 128.1, 128.15, 128.3, 128.4, 128.45, 128.5, 135.2, 135.4, 136.2, 136.3, 154.1, 154,7, 171.8, 172.0, 173.1, 173.12;
IR (neat): ν 2925, 2360, 1745, 1739, 1714, 1456, 1417, 1165, 1119, 1065, 914 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{32}H_{44}O_6N$]: 538.3090, found: 538.3168;
$[\alpha]_D^{22}$-37.3 (*c* = 0.88, $CHCl_3$).

**(2*S*, 4*R*)-4-Dodecanoyloxypyrrolidine-2-carboxylic acid (4d)**

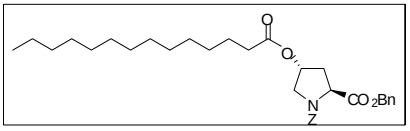
(2*S*, 4*R*)-4-Dodecanoyloxypyrrolidine-2-carboxylic acid **4d** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-dodecanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9d** by the procedure described for the synthesis of (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid **9c**.



$^1$H NMR ($CD_3OD$, 600MHz: δ 0.93 (3H, t, *J*=6.8 Hz), 1.24-1.46 (18H, m), 1.61-1.68 (2H, m), 2.27-2.34 (1H, m), 2.37-2.44 (1H, m), 2.54 (1H, dd, *J*=14.4, 7.7 Hz), 3.41 (1H, d, *J*=13.1 Hz), 3.63 (1H, dd, *J*=4.2, 13.1 Hz), 4.18 (1H, t, *J*=7.7 Hz), 5.42 (1H, br-s);
$^{13}$C NMR ($CD_3OD$, 150MHz): δ 15.4, 24.7, 26.8, 31.0, 31.2, 31.4, 31.5, 31.6, 31.7, 34.1, 35.8, 37.6, 53.0, 62.6, 75.7, 174.0, 175.3;
IR (neat): ν 2920, 2850, 1736, 1616, 1585, 1456, 1417, 1205, 1165, 636 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{17}H_{32}O_4N$]: 314.2331, found: 314.2307;
$[\alpha]_D^{22}$-17.8 (*c* = 0.09, MeOH).

**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-myristoyloxypyrrolidine-2-carboxylic acid benzyl ester (9e)**

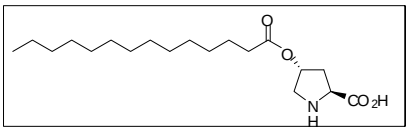
(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-myristoyloxypyrrolidine-2-carboxylic acid benzyl ester **9e** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester **8** and myristoyl chloride by the procedure described for the synthesis of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c**.



Data are shown as a mixture of two conformers.
$^1$H NMR ($CDCl_3$, 400 MHz): δ 0.86 (3H, t, *J*=7.0 Hz), 1.23 (20H, br-s), 1.54 (2H, br-s), 2.10-2.28 (3H, m), 2.31-2.43 (1H, m), 3.58-3.81 (2H, m), 4.39-4.58 (1H, m), 4.88-5.33 (5H, m), 7.16-7.35 (10H, m);
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 14.0, 22.6, 24.8, 29.0, 29.1, 29.3, 29.4, 29.5, 29.57, 29.6, 31.9, 34.2, 35.6, 36.6, 52.6, 57.7, 58.0, 67.0, 67.03, 67.3, 71.5, 72.3, 127.8, 128.0, 128.1, 128.13, 128.3, 128.4, 128.43, 128.5, 135.2, 135.4, 136.2, 136.3, 154.1, 154,7, 171.7, 172.0, 173.1, 173.11;
IR (neat): ν 2925, 1735, 1716, 1456, 1417, 1354, 1167, 1120, 1066, 698 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{34}H_{48}O_6N$]: 566.3403, found: 566.3487;
$[\alpha]_D^{22}$-35.3 (*c* = 1.1, $CHCl_3$).

**(2*S*, 4*R*)-4-Myristoyloxypyrrolidine-2-carboxylic acid (4e)**

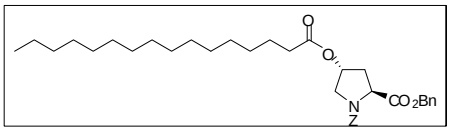
(2*S*, 4*R*)-4-Miristoyloxypyrrolidine-2-carboxylic acid **4e** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-myristoyloxypyrrolidine-2-carboxylic acid benzyl ester **9e** by the procedure described for the synthesis of (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid **4c**.



$^1$H NMR ($CD_3OD$, 600 MHz): δ 0.89-0.99 (3H, m), 1.25-1.46 (20H, m), 1.58-1.70 (2H, m), 2.26-2.34 (1H, m), 2.36-2.44 (2H, m), 2.49-2.56 (1H, m), 3.42 (1H, d, *J*=13.1 Hz), 3.61-3.68 (1H, m), 4.16-4.21 (1H, m), 5.40-5.45 (1H, m);
$^{13}$C NMR ($CD_3OD$, 150 MHz): δ 11.6, 20.9, 22.9, 27.3, 27.5, 27.6, 27.7, 27.8, 27.85, 27.88, 27.91, 30.2,

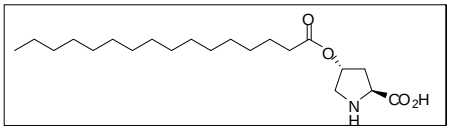
58.71, 71.78, 170.1, 171.4;
IR (KBr): ν 2918, 2850, 1736, 1614, 1587, 1456, 1415, 1220, 1165, 636 $cm^{-1}$;
HRMS (FAB): [M+H] calcd for $[C_{19}H_{36}O_4N]$: 342.2644, found: 342.2619;
$[\alpha]_D^{22}$-20.8 (*c* = 0.07, MeOH).

**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-palmitoyloxypyrrolidine-2-carboxylic acid benzyl ester (9f)**



(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-palmitoyloxypyrrolidine-2-carboxylic acid benzyl ester **9f** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester **8** and palmitoyl chloride by the procedure describeSd for the synthesis of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-decanoyloxypyrrolidine-2-carboxylic acid benzyl ester **9c**.
$^1$H NMR ($CDCl_3$, 400 MHz ): δ 0.86 (3H, t, *J*=6.9 Hz), 1.23 (24H, br-s), 1.53 (2H, br-s), 2.13-2.29 (3H, m), 2.31-2.42 (1H, m), 3.58-3.80 (2H, m), 4.39-4.58 (1H, m), 4.91-5.32 (5H, m), 7.16-7.37 (10H, m);
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 14.0, 22.6, 24.7, 29.0, 29.1, 29.2, 29.3, 29.5, 29.52, 29.53, 29.6, 31.8, 34.1, 35.5, 36.6, 52.5, 57.7, 58.0, 66.8, 67.0, 67.2, 71.4, 72.2, 127.8, 127.9, 128.0, 128.1, 128.2, 128.3, 128.4, 128.5, 135.2, 135.4, 136.1, 136.3, 154.1, 154.6, 171.7, 171.9, 173.0, 173.01;
IR (neat): ν 2854, 1747, 1736, 1716, 1456, 1417, 1354, 1166, 1068, 914 $cm^{-1}$;
HRMS (FAB): [M+H] calcd for $[C_{32}H_{44}O_6N]$: 594.3716, found: 594.3799;
$[\alpha]_D^{22}$-34.0 (*c* = 1.01, MeOH).

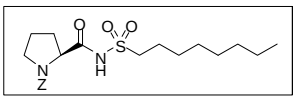
**(2*S*, 4*R*)-4-Palmitoyloxypyrrolidine-2-carboxylic acid (4f)**



(2*S*, 4*R*)-4-Palmitoyloxypyrrolidine-2-carboxylic acid **4f** was prepared from (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-palmitoyloxypyrrolidine-2-carboxylic acid benzyl ester **9f** by the procedure described for the synthesis of (2*S*, 4*R*)-4-decanoyloxypyrrolidine-2-carboxylic acid **9c**.
$^1$H NMR (CDOD, 600 MHz): δ 0.89-0.97 (3H, m), 1.25-1.37 (24H, m), 1.66 (2H, br-t, *J*=6.5 Hz), 2.27-2.35 (1H, m), 2.37-2.44 (2H, m), 2.50-2.56 (1H, m), 3.40 (1H, d, *J*=13.1 Hz), 3.63 (1H, dd, *J*=4.2, 13.1 Hz), 4.12-4.22 (1H, m), 5.43 (1H, br-s);
$^{13}$C NMR ($CD_3OD$, 150 MHz): δ 15.4, 24.7, 26.8, 31.2, 31.4, 31.5, 31.6, 31.72, 31.76, 31.8, 34.1, 35.9, 37.7, 53.0, 62.6, 75.7, 174.0, 175.3;
IR (KBr): ν 2918, 2850, 1736, 1618, 1585, 1441, 1415, 1228, 1167, 721 $cm^{-1}$;
HRMS (FAB): [M+H] calcd for $[C_{21}H_{40}O_4N]$: 370.2957, found: 370.2943;
$[\alpha]_D^{22}$-25.9 (*c* = 0.05, MeOH).

## Typical procedure for a synthesis of sulfonyl pyrrolidine-2-carboxamide

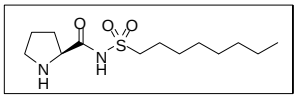
**(2*R*)-*N*-Benzyloxycarbonyl(octanesulfonyl pyrrolidine-2-carboxamide) (11a)**



To a solution of octanesulfonamide (651 mg, 3.37 mmol) in 17 mL of *N*, *N*-dimethylformamide was added sodium hydride (403 mg, 60 wt% dispersion in mineral oil, 10.1 mmol). After stirring for 0.5 hours at room temperature, (2*S*)-*N*-benzyloxycarbonyl-(4-nitrophenyl) pyrrolidine-2-carboxylate **10** (1.87 g, 5.06 mmol) was added. The solution was stirred 5 hours at room temperature and then poured onto crushed ice. The pH was adjusted to 3 by addition of citric acid. The aqueous layer was extracted with ethyl acetate three times. The organic layer was washed with water, dried over sodium sulfate and concentrated in vacue. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:3) gave (2*R*)-*N*-benzyloxycarbonyl(octanesulfonyl pyrrolidine-2-carboxamide) **11a** (909 mg, 2.14 mmol) in 64% yield as a pale yellow oil.
Data are shown as a mixture of two conformers.
$^1$H NMR ($CDCl_3$, 400 MHz): δ 0.85 (3H, t, *J*=7.0 Hz), 1.14-1.43 (11H, m), 1.53-2.07 (5H, m), 2.35 (1H, br-s), 3.03-3.60 (4H, m), 4.31 (1H, br-s), 5.14 (2H, q, *J*=12.0 Hz), 7.25-7.40 (5H, m);
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 14.1, 22.6, 23.3, 24.4, 25.3, 28.2, 29.0, 29.1, 31.7, 34.7, 47.1, 53.2, 61.7, 67.9, 128.0, 128.2, 128.6, 136.0, 156.8;
IR (neat): ν 2925, 1685, 1584, 1456, 1422, 1358, 1124, 1088, 767, 697 $cm^{-1}$;
HRMS (FAB): [M+Na] calcd for $[C_{21}H_{32}N_2O_5SNa]$: 447.1924, found: 447.1917;

$[\alpha]_D^{22}$-132.4 (*c* = 0.95, $CHCl_3$).

**(2*R*)-Octanesulfonyl pyrrolidine-2-carboxamide (6a)**



To a solution of (2*R*)-*N*-benzyloxycarbonyl(octanesulfonyl pyrrolidine-2-carboxamide) **11a** (829 mg, 1.95 mmol) in MeOH (3 mL) was added 10% Pd/C (83 mg, 0.08 mmol) at room temperature. The reaction mixture was stirred for 20 h under $H_2$ atmosphere. The reaction was filtered through a pad of Celite, and concentrated in vacue. The residual solid was washed with $Et_2O$ to afford (2*R*)-octanesulfonyl pyrrolidine-2-carboxamide **6a** in 89% yield as a white solid.

$^1$H NMR ($CDCl_3$, 400 MHz): δ 0.86 (3H, t, *J*=7.0 Hz), 1.15-1.32 (8H, m), 1.30-1.40 (2H, m), 1.68-1.82 (2H, m), 1.88-2.12 (3H, m), 2.30-2.41 (1H, m), 2.97-3.11 (2H, m), 3.54-3.63 (1H, m), 3.51-3.68 (1H, m), 4.19 (1H, t, *J*=7.9 Hz);

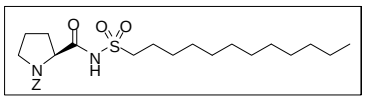
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 14.1, 22.6, 23.7, 24.6, 28.5, 29.1, 29.2, 30.2, 31.8, 46.8, 52.9, 62.6, 173.9;

IR (KBr): ν 3122, 2923, 2853, 1616, 1597, 1564, 1389, 1277, 1125, 854 $cm^{-1}$;

HRMS (FAB): [M+Na] calcd for $[C_{13}H_{26}N_2O_3SNa]$: 313.1556, found: 313.1545;

$[\alpha]_D^{22}$-32.2 (*c* = 1.00, MeOH).

**(2*R*)-*N*-Benzyloxycarbonyl(dodecanesulfonyl pyrrolidine-2-carboxamide) (11b)**



(2*R*)-*N*-Benzyloxycarbonyl(dodecanesulfonyl pyrrolidine-2-carboxamide) **11b** (715 mg, 54%) was prepared from dodecanesulfonamide (687 mg, 2.75 mmol) and (2*S*)-*N*-benzyloxycarbonyl-(4-nitrophenyl)pyrrolidine-2-carboxylate **10** (1.52 g, 4.1 mmol) by the procedure described for the synthesis of (2*R*)-*N*-benzyloxycarbonyl(octanesulfonyl pyrrolidine-2-carboxamide) **11a**. Data are shown as a mixture of two conformers.

$^1$H NMR ($CDCl_3$, 400 MHz): δ 0.86 (3H, t, *J*=6.8 Hz), 1.18-1.42 (19H, m), 1.48-2.00 (5H, m), 2.43 (1H, br-s), 3.00-3.62 (4H, m), 4.35 (1H, br-s), 5.08-5.22 (2H, m), 7.27-7.42 (5H, m);

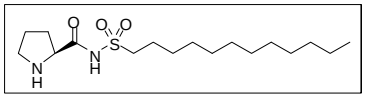
$^{13}$C NMR ($CDCl_3$, 100 MHz): δ 14.1, 22.7, 23.0, 24.5, 27.0, 28.0, 29.0, 29.3, 29.33, 29.5, 29.6, 31.9, 47.3, 53.2, 60.9, 68.2, 128.2, 128.5, 128.7, 135.8, 157.1;

IR (neat): ν 2923, 2852, 2360, 1699, 1457, 1422, 1357, 1338, 1125, 697 $cm^{-1}$;

HRMS (FAB): [M+Na] calcd for $[C_{25}H_{40}N_2O_5SNa]$: 503.2550, found: 503.2542;

$[\alpha]_D^{22}$-83.4 (*c* = 0.95, $CHCl_3$).

**(2*R*)-Dodecanesulfonyl pyrrolidine-2-carboxamide (6b)**



(2*R*)-Dodecanesulfonyl pyrrolidine-2-carboxamide **6b** (434 mg, 98%) was prepared from (2*R*)-*N*-benzyloxycarbonyl-(dodecanesulfonyl pyrrolidine-2-carboxamide) **11b** (595 mg, 1.29 mmol) by the procedure described for the synthesis of (2*R*)-octanesulfonyl pyrrolidine-2-carboxamide **6a**.

$^1$H NMR ($CDCl_3$, 400MHz): δ 0.86 (3H, t, *J*=6.8 Hz), 1.18-1.32 (16H, m), 1.32-1.37 (2H, m), 1.68-1.84 (2H, m), 1.88-2.12 (3H, m), 2.28-2.43 (1H, m), 2.94-3.15 (2H, m), 3.26-3.40 (1H, m), 3.48-3.63 (1H, m), 4.09-4.25 (1H, m);

$^{13}$C NMR ($CDCl_3$, 100MHz): δ 14.1, 22.7, 23.7, 24.5, 28.5, 29.3, 29.31, 29.4, 29.55, 29.6, 30.1, 31.9, 46.5, 52.8, 62.6, 174.0;

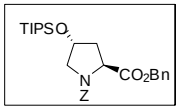
IR (KBr): ν 3124, 2917, 2850, 1597, 1562, 1471, 1387, 1279, 1126, 534 $cm^{-1}$;

HRMS (FAB): [M+Na] calcd for $[C_{17}H_{34}N_2NaO_3S]$: 369.2182, found: 369.2174;

$[\alpha]_D^{22}$-61.6 (*c* = 1.00, $CHCl_3$).

## Typical procedure for the synthesis of (2*S*, 4*R*)-4-triisopropylsiloxypyrrolidine-2-carboxylic acid (7b).

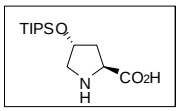
**(2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-triisopropylsiloxypyrrolidine-2-carboxylic acid (12b)**



To a dichloromethane solution (10 mL) of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester **8** (3.55 g, 10.0 mmol) was added 2,6-lutidine (1.90 mL, 13.0 mmol) and TIPSOTf (2.96 mL, 13.0 mmol) at 0 °C. The reaction mixture was stirred for 30 minutes at room temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried ($Na_2SO_4$), concentrated, and purified by column

chromatography (ethyl acetate:hexane = 1:5) to afford (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-triisopropylsiloxypyrrolidine-2-carboxylic acid **12b** (4.8 g, 9.54 mmol, 95%) as a clear viscous oil.
Data are shown as a mixture of two conformers.
$^1$H NMR ($CDCl_3$, 600 MHz): δ 1.01 (21H, d, *J*=4.5 Hz), 2.00-2.12 (1H, m), 2.17-2.31 (1H, m), 3.42-3.59 (1H, m), 3.62-3.76 (1H, m), 4.45-4.60 (2H, m), 4.91-5.26 (4H, m), 7.17-7.37 (10H, m);
$^{13}$C NMR ($CDCl_3$, 150 MHz): δ 12.4, 18.3, 39.5, 40.4, 55.3, 55.7, 58.5, 58.7, 67.1, 67.3, 67.5, 70.3, 71.0, 128.2, 128.3, 128.4, 128.55, 128.61, 128.65, 128.75, 128.8, 128.9, 129.0, 135.9, 136.1, 136.9, 137.1, 154.8, 155.5, 172.8, 173.0;
IR (neat): ν 2943, 2866, 1749, 1712, 1458, 1415, 1117, 1022, 883, 696 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{29}H_{42}NO_5Si$]: 512.2832, found: 512.2809;
$[\alpha]_D^{22}$-35.1 (*c* = 1.00, $CHCl_3$).

**(2*S*, 4*R*)-4-triisopropylsiloxypyrrolidine-2- carboxylic acid (7b)**




To a MeOH solution (10 mL) of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-triisopropylsiloxypyrrolidine-2-carboxylic acid benzyl ester **12b** (4.8 g, 9.54 mmol) was added Pd/C (480 mg, 10 wt%) at room temperature and the reaction mixture was stirred for 20 h at that temperature. The filtration of the inorganic materials and concentration afforded (2*S*, 4*R*)-4-triisopropylsiloxy-pyrrolidine-2-carboxylic acid **7b** in 96% yield (2.8 g, 9.16 mmol) as a white solid.
$^1$H NMR ($CDCl_3$): δ 0.97-1.05 (21H, m), 2.13 (1H, ddd, *J*=12.9, 7.8, 5.4 Hz), 2.27 (1H, ddd, *J*=12.9, 7.8, 4.1 Hz), 3.20 (1H, br-d, *J*=9.0 Hz), 3.46 (1H, br-s), 4.15 (1H, t, *J*=7.8 Hz), 4.51 (1H, quintet, *J*=4.1 Hz);
$^{13}$C NMR ($CDCl_3$): δ 11.9, 17.8, 39.2, 52.5, 59.8, 71.0, 173.7;
IR (KBr): ν 3438, 2942, 1624, 1464, 1400, 1389, 1101, 999, 883, 685 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{14}H_{29}NO_3Si$]: 288.1995, found: 288.2010;
$[\alpha]_D^{22}$-15.9 (*c* = 1.00, $CHCl_3$).

**(2*S*, 4*R*)-4-*tert*-Butyldimethylsiloxypyrrolidine-2-carboxylic acid (7a)**[1]
**(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester (8)**[2]
**(2*S*, 4*R*)-4-*tert*-Butyldiphenylsiloxypyrrolidine-2-carboxylic acid (7c)**[1]
are known compounds.

## Typical procedure of screening of the effect of catalyst on the reaction yield and stereoselectivity in the asymmetric aldol reaction of benzaldehyde with cyclohexanone in the presence of water (Table 1, entry 17).

Catalyst **7c** (14.8 mg, 0.04 mmol) was added to a mixture of benzaldehyde (40.6 μL, 0.4 mmol) and cyclohexanone (207 μL, 2.0 mmol) in water (0.13 mL) at room temperature. The reaction mixture was stirred for 18 h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)cyclohexan-1-one (63.7 mg, 78%) as a clear oil: *anti*:*syn* = 13:1 (by $^1$H NMR spectroscopy of the crude mixture), >99% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, $^i$PrOH/hexane 1/100, 1.0 mL/min; tr=19.4 min (major), 25.9 min (minor)).

**(2*S*, 1'*R*)-2-(Hydroxyphenylmethyl)cyclohexan-1-one**[3]
is known compound. Absolute stereochemistry is determined by the comparison with the literature data[3].
$[\alpha]_D^{14}$+27.7 (*c* = 0.85, $CHCl_3$), >99% ee.
Lit. $[\alpha]_D^{24}$-24.2 (*c* = 1.03, $CHCl_3$). (93% ee, (2*R*, 1'*S*)-2-(Hydroxyphenylmethyl)cyclohexanone).
Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column, λ=213 nm, $^i$PrOH/hexane 1/100, 1.0 mL/min; tr =19.4 min (major), tr =25.9 min (minor).

## Typical procedure of screening of solvent effect on the reaction yield and stereoselectivity of asymmetric aldol reaction of benzaldehyde with cyclohexanone in the presence of water (Table 2, entry 3).

Catalyst **7a** (9.8 mg, 0.04 mmol) was added to a mixture of benzaldehyde (40.6 μL, 0.4 mmol) and cyclohexanone (207 μL, 2.0 mmol) in DMSO (0.4 mL) at room temperature. The reaction mixture was stirred for 18 h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)cyclohexan-1-one (53.9 mg, 66%) as a clear oil: *anti*:*syn* = 1:1 (by $^1$H NMR spectroscopy of the crude mixture), 80% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, $^i$PrOH/hexane 1/100, 1.0 mL/min; tr=19.4 min (major), 25.9 min (minor)).

**Typical procedure of screening of the effect of amount of water on the reaction yield and stereoselectivity in the asymmetric aldol reaction of benzaldehyde with cyclohexanone in the presence of water (Table 3, entry 8).**

Catalyst **7c** (14.8 mg, 0.04 mmol) was added to a mixture of benzaldehyde (40.6 μL, 0.4 mmol) and cyclohexanone (207 μL, 2.0 mmol) in water (0.72 mL) at room temperature. The reaction mixture was stirred for 18 h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)cyclohexan-1-one (68.6 mg, 84%) as a clear oil: *anti*:*syn* = 12:1 (by $^1$H NMR spectroscopy of the crude mixture), >99% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, $^i$PrOH/hexane 1/100, 1.0 mL/min; tr=19.4 min (major), 25.9 min (minor)).

**Typical procedure of screening of the effect of amount of catalyst 7c on the reaction yield and stereoselectivity in the asymmetric aldol reaction of benzaldehyde with cyclohexanone in the presence of water (Table 4, entry 4).**

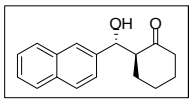
Catalyst **7c** (1.5 mg, 0.004 mmol) was added to a mixture of benzaldehyde (40.6 μL, 0.4 mmol) and cyclohexanone (207 μL, 2.0 mmol) in water (0.72 mL) at room temperature. The reaction mixture was stirred for 49 h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)cyclohexan-1-one (62.1 mg, 76%) as a clear oil: *anti*:*syn* = 10:1 (by $^1$H NMR spectroscopy of the crude mixture), 99% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, $^i$PrOH/hexane 1/100, 1.0 mL/min; tr=19.4 min (major), 25.9 min (minor)).

**Typical procedure of 4-siloxyproline 7c catalyzed asymmetric aldol reactions of aldehydes with ketones in the presence of water (Table 5, entry 5).**

Catalyst **7c** (14.8 mg, 0.04 mmol) was added to a mixture of 2-naphthalene carbaldehyde (62.4 mg, 0.4 mmol) and cyclohexanone (207 μL, 2.0 mmol) in water (0.13 mL) at room temperature. The reaction mixture was stirred for 40h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)cyclohexan-1-one (90.5 mg, 89%) as a clear oil: *anti*:*syn* = 13:1 (by $^1$H NMR spectroscopy of the crude mixture), >99% ee (by HPLC on a chiralcel AS-H column, λ=213 nm, $^i$PrOH/hexane 1/50, 1.0 mL/min; tr=17.6 min (major), 20.5 min (minor).

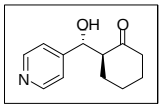
**(2*S*, 1'*R*)-2-(Hydroxy-*p*-nitrophenylmethyl)cyclohexan-1-one**[3]
**(2*S*, 1'*R*)-2-(Hydroxy-*p*-bromophenylmethyl)cyclohexan-1-one**[4]
**(2*S*, 1'*R*)-2-(Hydroxy-*p*-methoxyphenylmethyl)cyclohexan-1-one**[4]
**(2*S*, 1'*R*)-2-(Hydroxy-2-furylmethyl)cyclohexan-1-one**[5]
**(2*S*, 1'*S*)-2-(1'-Hydroxy-3'-methylbutyl)cyclohexan-1-one**[5]
**(2*S*, 1'*S*)-2-(1'-Hydroxyhexyl)cyclohexan-1-one**[6]
**(2*S*, 1'*S*)-2-(Cyclohexylhydroxymethyl)cyclohexan-1-one**[7]
**(2*S*, 1'*S*)-2-(1'-Hydroxy-2'-methylpropyl)cyclohexan-1-one**[8]
**(2*S*, 1'*R*)-2-(Hydroxyphenylmethyl)cyclopentan-1-one**[9]
**(4*S*, 1'*S*)-4-(Hydroxyphenylmethyl)-2,2-dimethyl-1,3-dioxane-5-one**[10]
**(2*S*)-2-(Hydroxymethyl)cyclohexan-1-one**[11]
**(4*R*)-4-Hydroxy-*p*-trifluoromethylphenylbutan-2-one**[12]
**(3*S*, 4*S*)-3,4-Dihydroxy-*o*-chlorophenylbutan-2-one**[13]
**(3*S*, 4*R*)-4-Hydroxy-3-methyl-4-phenylbutan-2-one**[14]
**(*1R*)-1-Hydroxy-1-phenylpentan-3-one**[14]
are known compounds.

**(2*S*, 1'*R*)-2-(Hydroxynaphthalen-2-ylmethyl)cyclohexan-1-one**




$^1$H NMR ($CDCl_3$): δ 1.23-1.40 (1H, m), 1.42-1.61 (2H, m), 1.62-1.79 (2H, m), 2.07 (1H, ddd, *J*=13.2, 6.6, 3.2 Hz), 2.36 (1H, td, *J*=13.2, 5.8 Hz), 2.49 (1H, br-d, *J*= 13.8 Hz), 2.64-2.74 (1H, m), 4.02 (1H, br-s), 4.95 (1H, d, *J*=8.6 Hz), 7.41-7.50 (3H, m), 7.73 (1H, s), 7.77-7.86 (3H, m);
$^{13}$C NMR ($CDCl_3$): δ 24.6, 27.7, 30.8, 42.6, 57.3, 74.8, 124.6, 125.9, 126.1, 126.2, 127.6, 127.9, 128.2, 133.0, 133.1, 138.2, 215.5;
IR (neat): ν 3354, 3055, 2933, 2854, 1695, 1444, 1309, 1122, 1057, 833 cm$^{-1}$;
HRMS (FAB): calcd for [$C_{17}H_{18}O_2$]: 254.1307, found: 254.1311;
$[\alpha]_D^{22}$+7.4 (*c* = 1.07, $CHCl_3$). (mixture of diastereomers, *anti*:*syn*=19:1, 97% ee for *anti*-isomer.)
Enantiomeric excess was determined by HPLC with a Chiralpak AS-H column ($^i$PrOH/hexane 1/50, 1.0 mL/min; major enantiomer tr =17.6 min, minor enantiomer tr =20.5 min).

**(2*S*, 1'*R*)-2-(Hydroxypyridin-4-ylmethyl)cyclohexan-1-one**




$^1$H NMR ($CDCl_3$): δ 1.38 (1H, qd, *J*=12.8, 3.8 Hz), 1.47-1.73 (3H, m), 1.77-1.86 (1H, m), 2.04-2.14 (1H, m), 2.34 (1H, td, *J*=13.3, 6.2 Hz), 2.42-2.50 (1H, m), 2.56 (1H, ddd, *J*=13.5, 8.2, 3.5 Hz), 3.97 (1H, br-s), 4.75 (1H, d, *J*=8.2 Hz), 7.23 (2H, d, *J*=5.7 Hz), 8.56 (2H, d, *J*=5.7 Hz);
$^{13}$C NMR ($CDCl_3$): δ 24.6, 27.7, 30.7, 42.6, 56.8, 73.5, 122.0, 149.7, 149.8, 214.7;
IR (KBr): ν 3140, 2860, 2738, 1711, 1606, 1415, 1300, 1128, 1047, 835 cm$^{-1}$;
HRMS (FAB): [M+H] calcd for [$C_{12}H_{16}NO_2$]: 206.1181, found: 206.1177;
$[\alpha]_D^{21}$+15.8 (*c* = 1.02, $CHCl_3$). (mixture of diastereomers, *anti*:*syn*=12:1, 95% ee for *anti*-isomer .)
Enantiomeric excess was determined by HPLC with a Chiralpak AD-H column ($^i$PrOH/hexane 1/10, 1.0 mL/min; major enantiomer tr = 22.5 min, minor enantiomer tr =20.7 min).

## Typical procedure for the synthesis of (2*R*, 1'*S*)-2-(hydroxyphenylmethyl)cyclohexan-1-one

Catalyst **17** (14.8 mg, 0.04 mmol) was added to a mixture of benzaldehyde (40.6 μL, 0.4 mmol) and cyclohexanone (207 μL, 2.0 mmol) in water (0.13 mL) at room temperature. The reaction mixture was stirred for 18 h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous $Na_2SO_4$, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)cyclohexan-1-one (63.7 mg, 78%) as a clear oil: *anti*:*syn* = >20:1 (by $^1$H NMR spectroscopy of the crude mixture), 98% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, *i*PrOH/hexane 1/100, 1.0 mL/min; tr=25.9 min (major), 19.4 min (minor)).

**Large scale preparation of (2*S*, 1'*R*)-2-(hydroxyphenylmethyl)cyclohexan-1-one. (Used when the product is liquid)**

Catalyst **7c** (259 mg, 0.74 mmol) was added to a mixture of benzaldehyde (7.4 g, 74.4 mmol) and cyclohexanone (13.7 g, 149 mmol) in water (3.8 mL) at room temperature. The reaction mixture was stirred for 48h, then silica gel (2.5 g) was added to the reaction mixture. The mixture was filtered through silica gel using ethyl acetate (60 mL), and the crude organic materials were purified by distillation to afford 2-(hydroxyphenylmethyl)cyclohexan-1-one (10.0 g, 70%) as a colorless oil: *anti*:*syn* = 10:1 (by $^{1}$H NMR spectroscopy of distilled product), >99% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, $^{i}$PrOH/hexane 1/100, 1.0 mL/min; tr=19.4 min (major), 25.9 min (minor))

## References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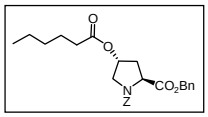


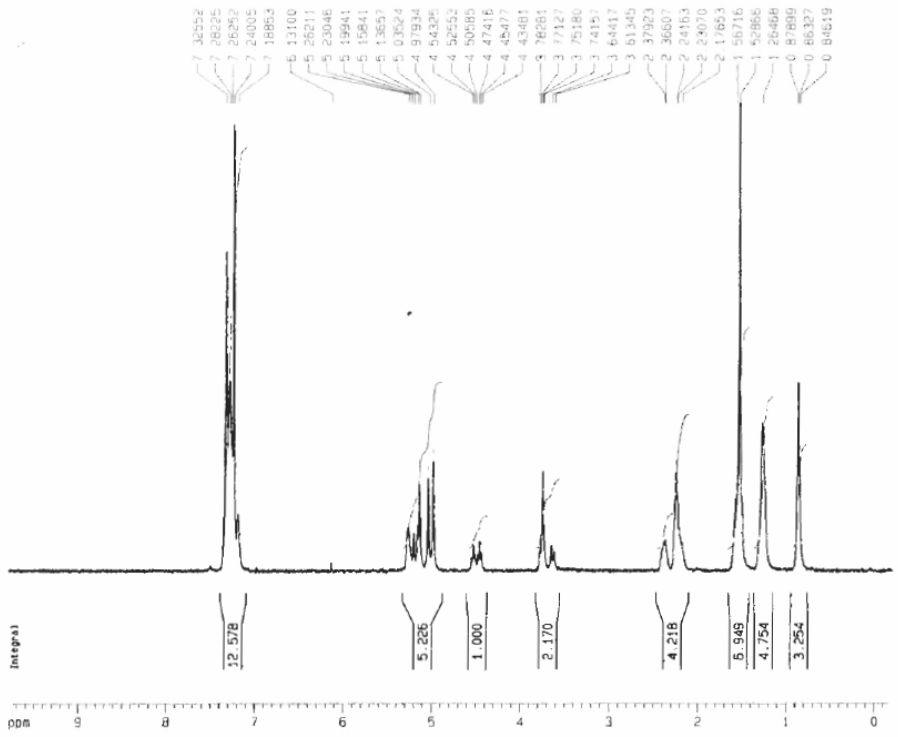


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PROCNO                    1

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PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       0
SWH              8223.685 Hz
FIDRES           0.250967 Hz
AQ               1.9923444 sec
RG                   574.7
DW                  60.800 usec
DE                   6.00 usec
TE                  303.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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PL1                  3.00 dB
SFO1          400.1324710 MHz

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

1D NMR plot parameters
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CY                   12.50 cm
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F1                3921.27 Hz
F2P                 -0.200 ppm
F2                  -80.03 Hz
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HZCM            200.06500 Hz/cm
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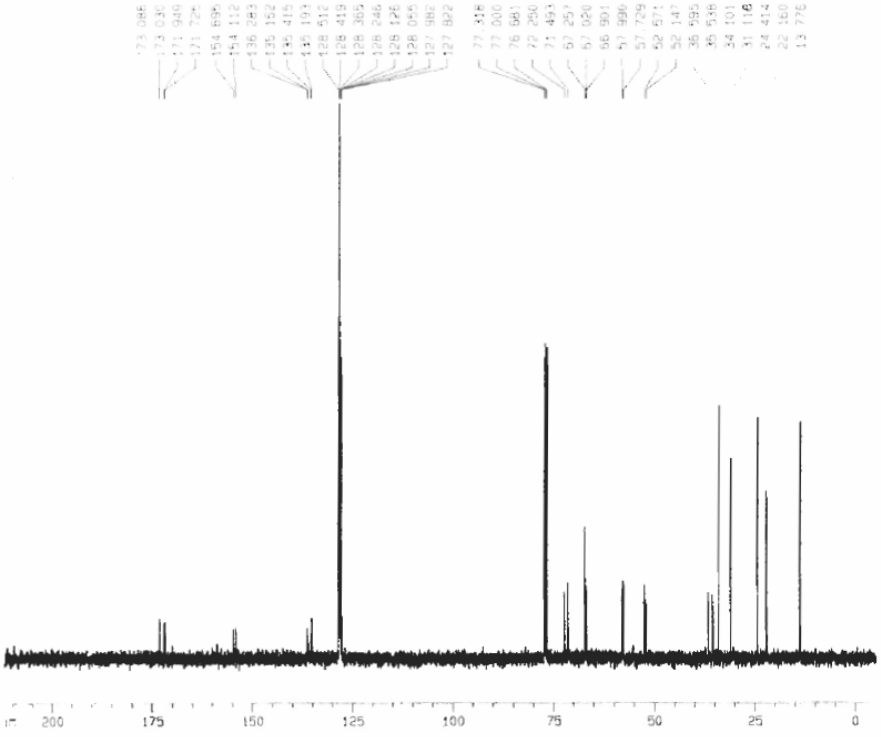


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PROCNO                    1

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PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     116
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                  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          400.1316005 MHz

F2 - Processing parameters
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SF            100.6127686 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
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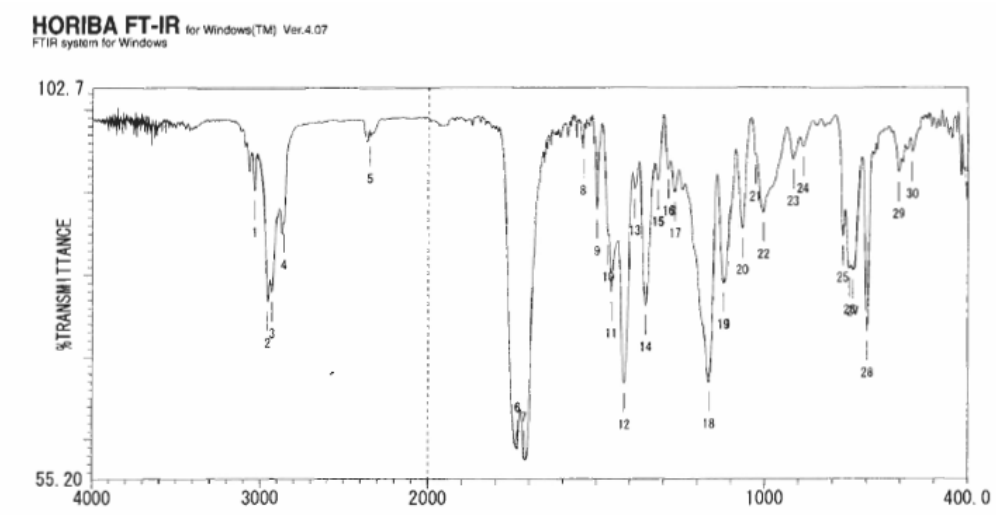


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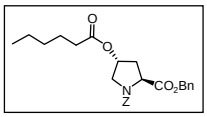
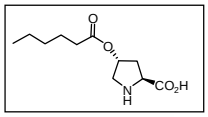
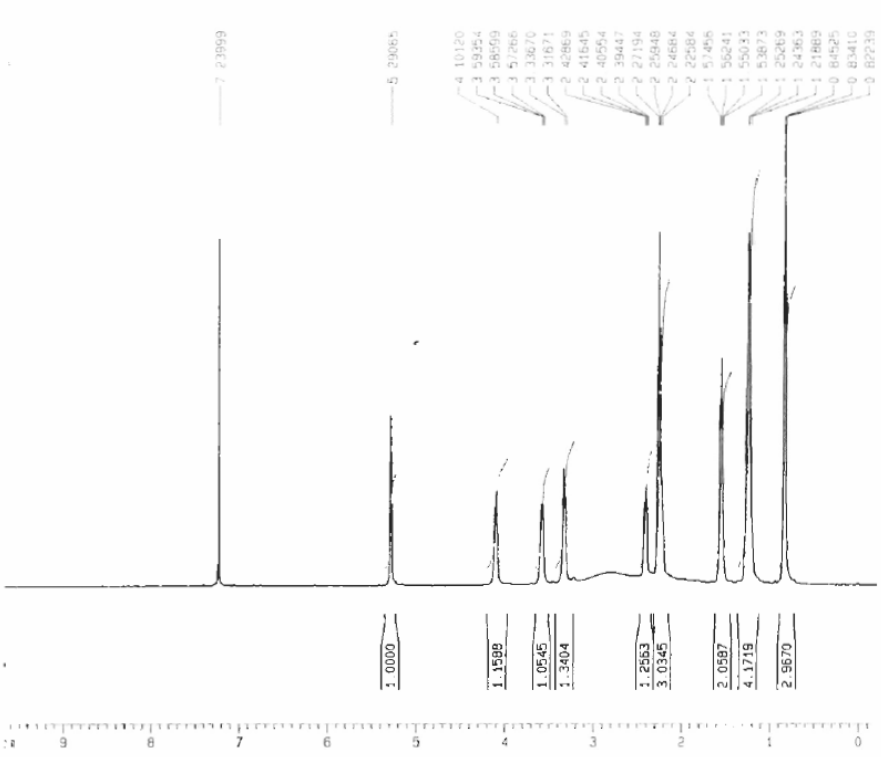
| 測定日時 | : 2005年08月19日 13時23: | 02 | 2956.34 | 76.7712 | 12 | 1417.42 | 66.8130 | 22 | 1002.80 | 87.5365 |
| 測定分解能 | : 4 cm-1 | 03 | 2931.27 | 77.9192 | 13 | 1386.57 | 90.4232 | 23 | 916.022 | 93.9803 |
| スキャン回数 | : 10 回 | 04 | 2861.84 | 86.2761 | 14 | 1353.78 | 76.3009 | 24 | 887.095 | 95.5193 |
| 測定ゲイン | : 1 | 05 | 2345.02 | 96.7159 | 15 | 1317.14 | 91.4703 | 25 | 769.458 | 84.7074 |
| | | 06 | 1735.62 | 58.8219 | 16 | 1286.29 | 92.8097 | 26 | 750.174 | 80.8082 |
| | | 07 | 1716.34 | 57.6848 | 17 | 1267.00 | 90.0860 | 27 | 740.531 | 80.6221 |
| | | 08 | 1540.85 | 95.2722 | 18 | 1164.79 | 66.9629 | 28 | 698.105 | 73.1419 |
| | | 09 | 1498.42 | 87.9464 | 19 | 1122.37 | 79.0282 | 29 | 605.539 | 92.4434 |
| | | 10 | 1465.63 | 84.7899 | 20 | 1066.44 | 85.6405 | 30 | 565.041 | 94.8770 |







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PROCNO 1

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TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 28.5
DW 40.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
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P1 14.40 usec
PL1 -5.80 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
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SF 600.1300236 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
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F2P -0.200 ppm
F2 -120.03 Hz
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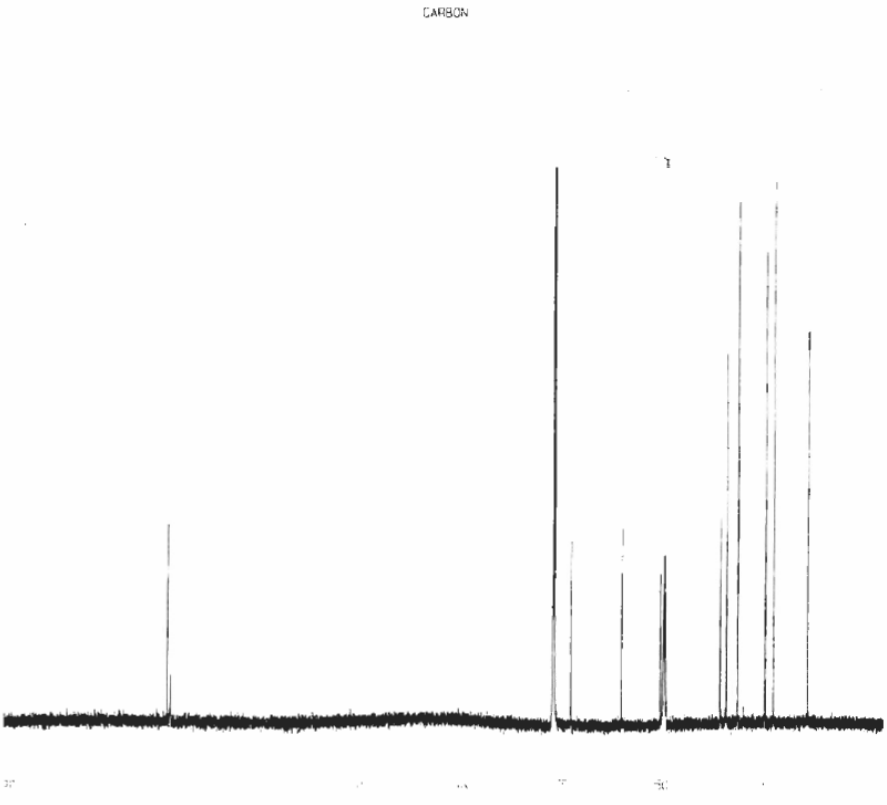
HZCM 300.06500 Hz/cm

CARBON



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EXPNO 19
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 409
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 7298.2
DW 13.900 usec
DE 50.00 usec
TE 298.0 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 10.00 usec
PL1 -4.90 dB
SFO1 150.9178988 MHz

======== CHANNEL f2 ========
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NUC2 1H
PCPD2 100.00 usec
PL2 -5.80 dB
PL12 8.00 dB
PL13 8.00 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
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SF 150.9028181 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
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CY 12.50 cm
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F2 -754.51 Hz
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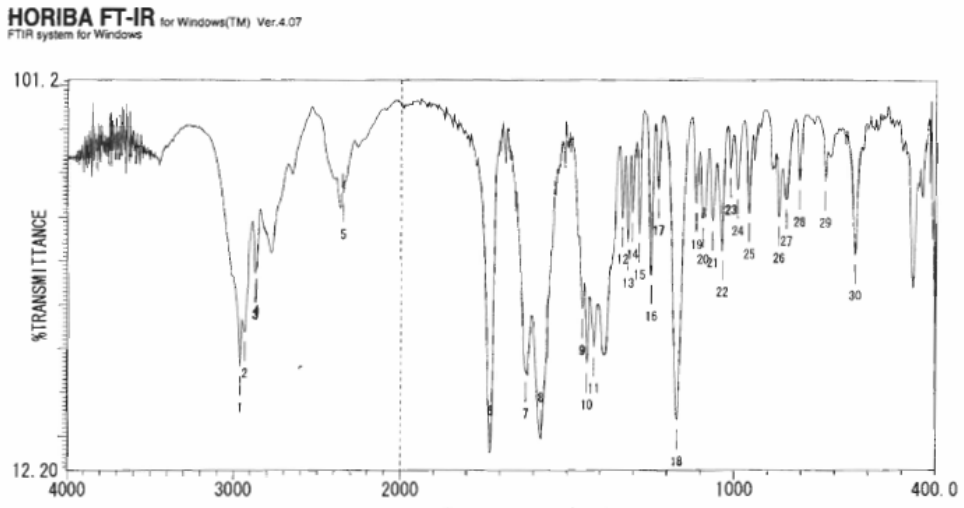
HZCM 1659.93091 Hz/cm

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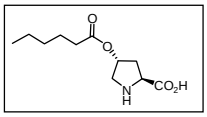
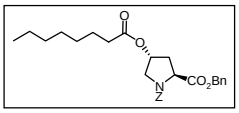
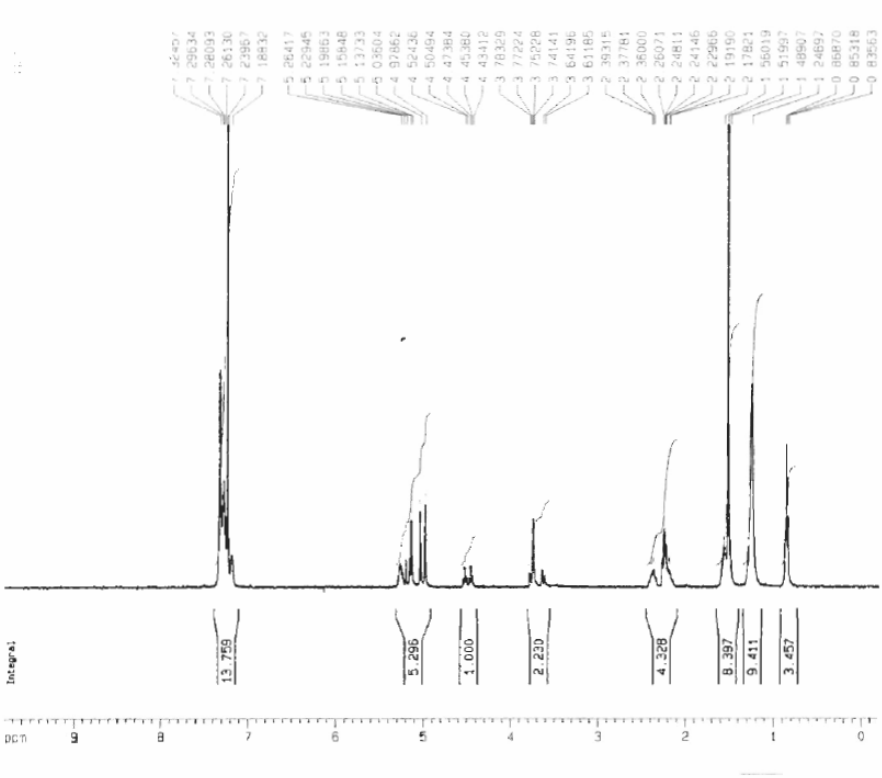
| 測定日時 | : 2005年08月23日 16時24: | 02 | 2933.20 | 43.7516 | 12 | 1334.50 | 69.8640 | 22 | 1037.52 | 62.3196 |
| 測定分解能 | : 4 cm-1 | 03 | 2871.49 | 57.0719 | 13 | 1319.07 | 64.3547 | 23 | 1012.45 | 81.0641 |
| スキャン回数 | : 10 回 | 04 | 2863.77 | 58.1050 | 14 | 1305.57 | 70.9608 | 24 | 991.232 | 76.3001 |
| 測定ゲイン | : 2 | 05 | 2345.02 | 75.4495 | 15 | 1284.36 | 66.2930 | 25 | 956.520 | 70.9854 |
| | | 06 | 1729.83 | 16.2957 | 16 | 1249.65 | 56.7505 | 26 | 867.810 | 70.1541 |
| | | 07 | 1623.77 | 34.5516 | 17 | 1228.43 | 76.3808 | 27 | 844.668 | 74.0484 |
| | | 08 | 1577.49 | 19.3827 | 18 | 1172.51 | 23.6334 | 28 | 806.099 | 78.3941 |
| | | 09 | 1454.06 | 48.9722 | 19 | 1114.65 | 73.2013 | 29 | 728.961 | 78.1764 |
| | | 10 | 1440.56 | 36.5847 | 20 | 1095.37 | 69.7906 | 30 | 640.251 | 61.4574 |



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FIDRES           0.250967 Hz
AQ               1.9923444 sec
RG                   645.1
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DE                   6.00 usec
TE                  303.2 K
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MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
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PL1                   3.00 dB
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F2 - Processing parameters
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WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

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F2P                 -0.200 ppm
F2                  -80.03 Hz
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HZCM            200.06500 Hz/cm
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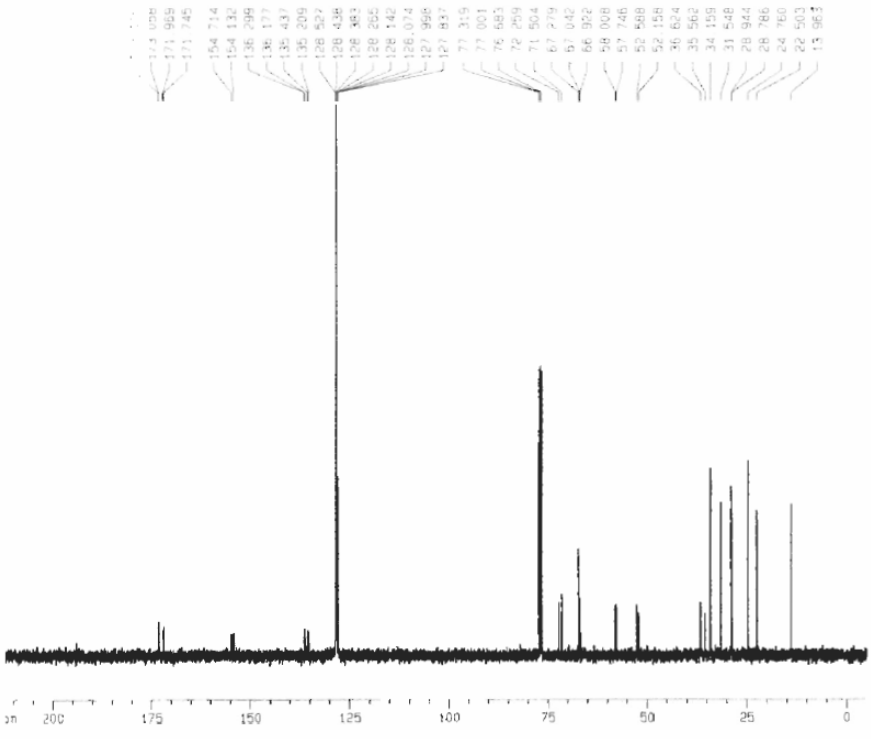


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PROCNO                    1

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TD                   65536
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DS                       2
SWH              31847.133 Hz
FIDRES           0.485949 Hz
AQ               1.0289652 sec
RG                  1448.2
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          400.1316005 MHz

F2 - Processing parameters
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SF            100.6127766 MHz
WDW                     EM
SSB                      0
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GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
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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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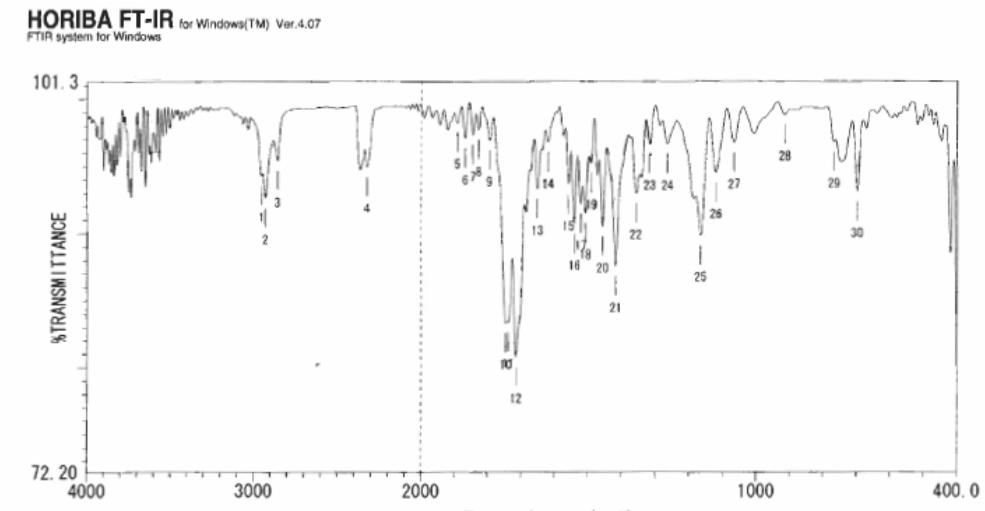


| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
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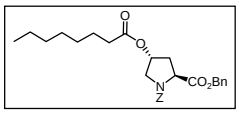
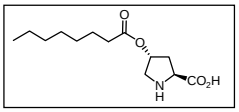
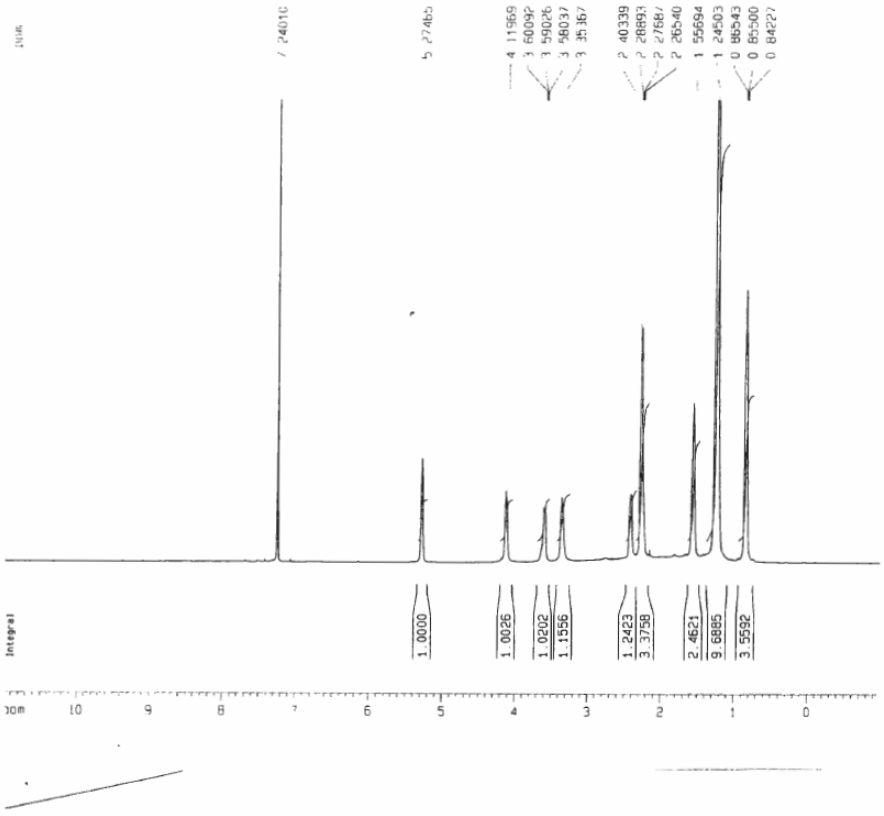
| 測定日時 | : 2005年08月19日 14時05: | 02 | 2929.34 | 92.6314 | 12 | 1714.41 | 80.7664 | 22 | 1357.64 | 92.9896 |
| 測定分解能 | : 4 cm-1 | 03 | 2856.06 | 95.3819 | 13 | 1650.77 | 93.2910 | 23 | 1317.14 | 96.6497 |
| スキャン回数 | : 10 回 | 04 | 2223.80 | 94.9390 | 14 | 1619.91 | 96.7509 | 24 | 1265.07 | 96.6348 |
| 測定ゲイン | : 1 | 05 | 1891.83 | 98.2188 | 15 | 1558.20 | 93.6159 | 25 | 1164.79 | 89.8390 |
| | | 06 | 1868.68 | 97.0506 | 16 | 1540.85 | 90.6975 | 26 | 1120.44 | 94.5001 |
| | | 07 | 1845.54 | 97.2931 | 17 | 1521.56 | 92.1795 | 27 | 1066.44 | 96.7340 |
| | | 08 | 1828.19 | 97.6593 | 18 | 1508.06 | 91.5178 | 28 | 914.093 | 98.8208 |
| | | 09 | 1795.40 | 96.9024 | 19 | 1490.70 | 95.2266 | 29 | 767.530 | 96.7854 |
| | | 10 | 1745.26 | 83.2676 | 20 | 1455.99 | 90.4983 | 30 | 698.105 | 93.1060 |

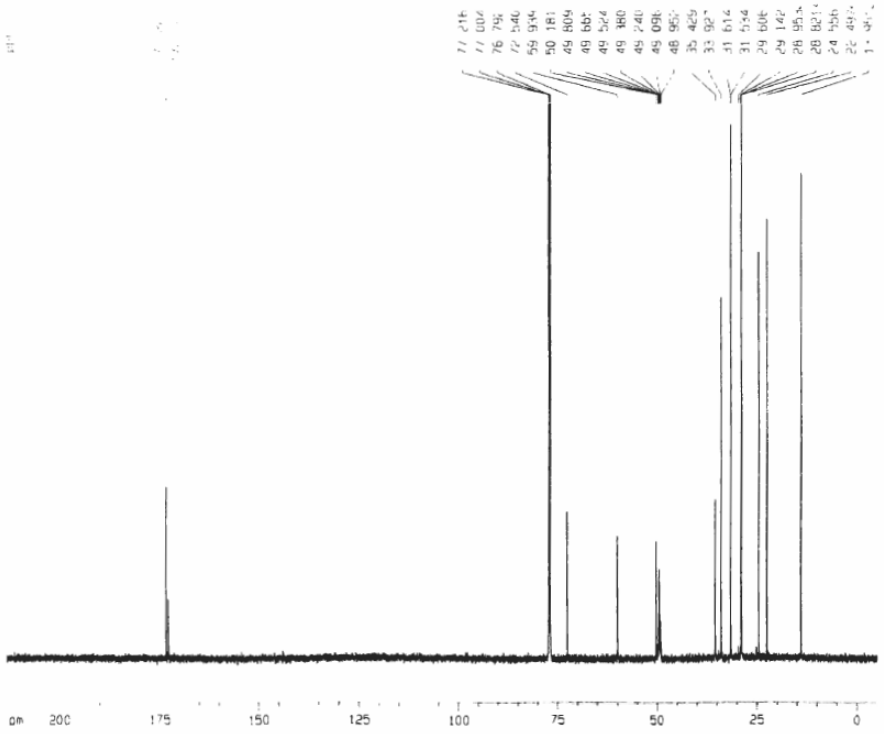





```
Current Data Parameters
NAME            Aug26-2005
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_         20050826
Time             12.59
INSTRUM          av500
PROBHD   5 mm CPDUL 13C
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          12376.237 Hz
FIDRES       0.188846 Hz
AQ           2.6477449 sec
RG                  16
DW             40.400 usec
DE              6.00 usec
TE             298.0 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              14.40 usec
PL1             -5.80 dB
SFO1       600.1337060 MHz

F2 - Processing parameters
SI              32768
SF         600.1300232 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            11.000 ppm
F1           6601.43 Hz
F2P            -1.000 ppm
F2           -600.13 Hz
PPMCM          0.60000 ppm/cm
HZCM         360.07800 Hz/cm
```

CARBON

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Current Data Parameters
NAME            Aug26-2005
EXPNO                   34
PROCNO                   1

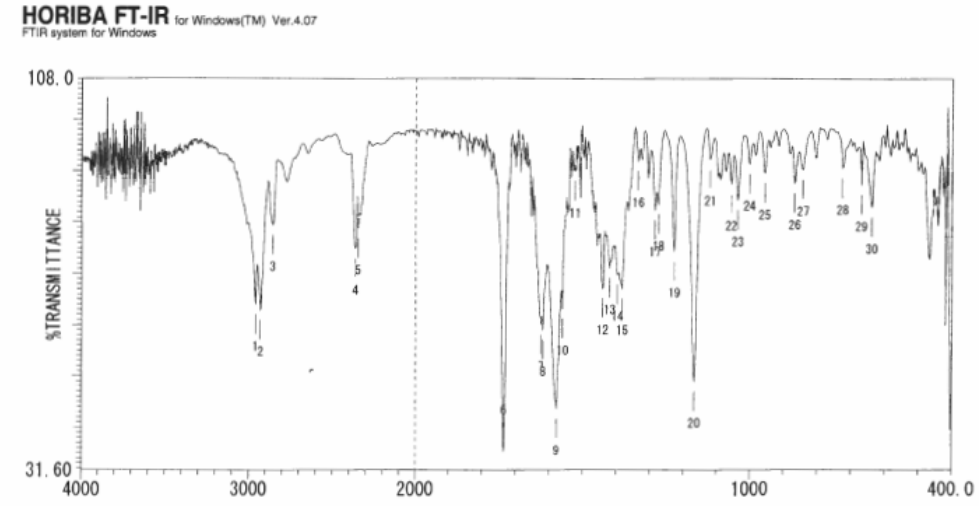
F2 - Acquisition Parameters
Date_         20050826
Time             20.38
INSTRUM          av600
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD              65536
SOLVENT          CDCl3
NS                  64
DS                   4
SWH          35971.223 Hz
FIDRES       0.548877 Hz
AQ           0.9110143 sec
RG                1300.4
DW             13.900 usec
DE              50.00 usec
TE             298.0 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              10.00 usec
PL1             -4.90 dB
SFO1       150.9178988 MHz

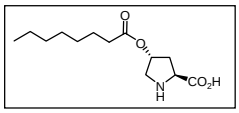
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2             -5.80 dB
PL12             8.00 dB
PL13             8.00 dB
SFO2       600.1324005 MHz

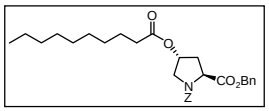
F2 - Processing parameters
SI              32768
SF         150.9027281 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

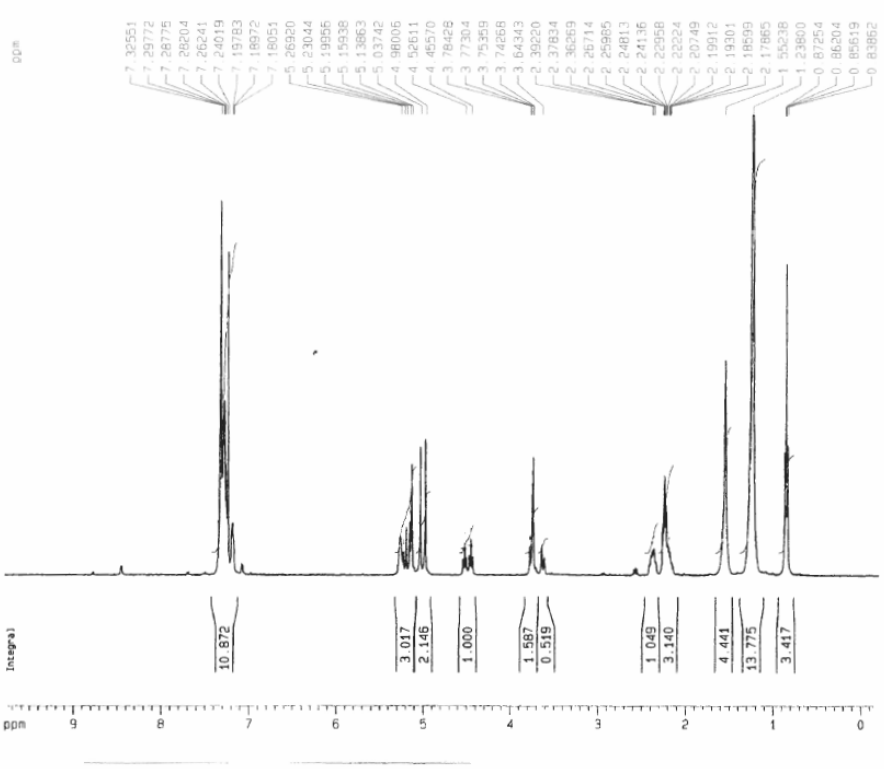
1D NMR plot parameters
CX              20.00 cm
CY              12.50 cm
F1P            215.000 ppm
F1           32444.09 Hz
F2P            -5.000 ppm
F2           -754.51 Hz
PPMCM          11.00000 ppm/cm
HZCM         1659.92993 Hz/cm
```




| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C8 | 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 | 2960.20 | 63.8256 | 11 | 1523.49 | 89.8910 | 21 | 1120.44 | 92.2562 |
| 測定日時 | : 2005年08月23日 16時44: | 02 | 2929.34 | 62.7062 | 12 | 1440.56 | 67.0072 | 22 | 1056.80 | 87.3853 |
| 測定分解能 | : 4 cm-1 | 03 | 2857.99 | 79.3213 | 13 | 1419.35 | 71.0006 | 23 | 1037.52 | 84.3055 |
| スキャン回数 | : 10 回 | 04 | 2360.44 | 74.7761 | 14 | 1396.21 | 69.6653 | 24 | 1002.80 | 91.2856 |
| 測定ゲイン | : 1 | 05 | 2345.02 | 78.6539 | 15 | 1382.71 | 66.9819 | 25 | 956.520 | 89.5164 |
| | | 06 | 1735.62 | 35.1618 | 16 | 1334.50 | 91.9063 | 26 | 867.810 | 87.5541 |
| | | 07 | 1623.77 | 59.9214 | 17 | 1284.36 | 82.2975 | 27 | 842.740 | 90.2551 |
| | | 08 | 1617.98 | 58.9011 | 18 | 1274.72 | 83.4110 | 28 | 725.104 | 90.5148 |
| | | 09 | 1577.49 | 43.5351 | 19 | 1226.50 | 74.2872 | 29 | 669.178 | 87.2533 |
| | | 10 | 1560.13 | 63.0276 | 20 | 1166.72 | 48.8115 | 30 | 638.323 | 82.8969 |





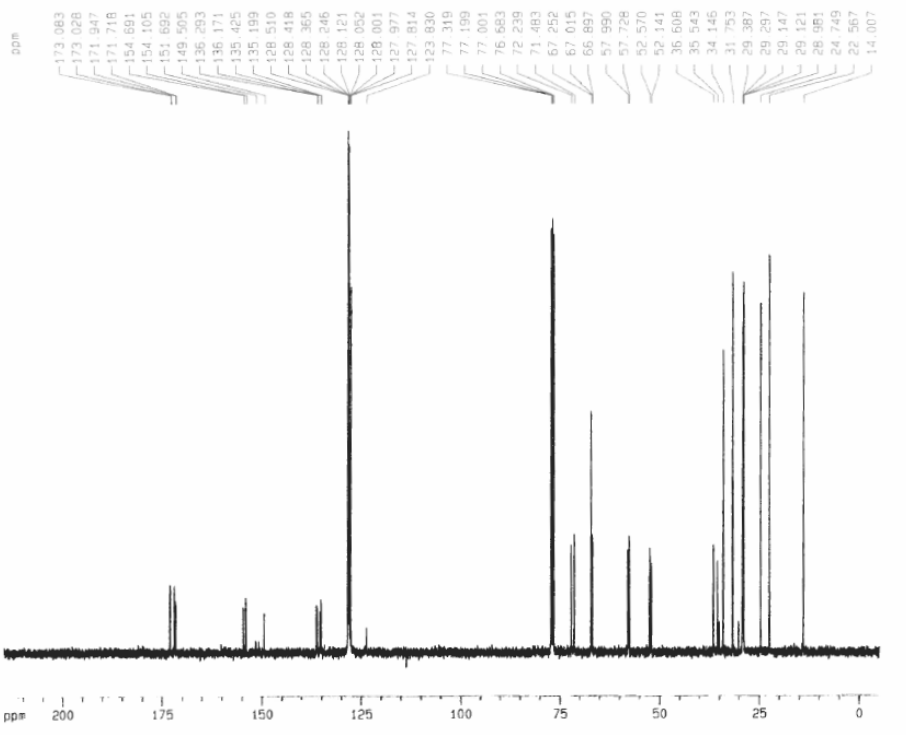

```
Current Data Parameters
NAME        Aug11-2005-haya
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050811
Time                  13.19
INSTRUM              dpx400
PROBHD   5 mm GNP 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                    322.5
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                400.1300172 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                200.06500 Hz/cm
```




```
Current Data Parameters
NAME        Aug14-2005-haya
EXPNO                    51
PROCNO                    1

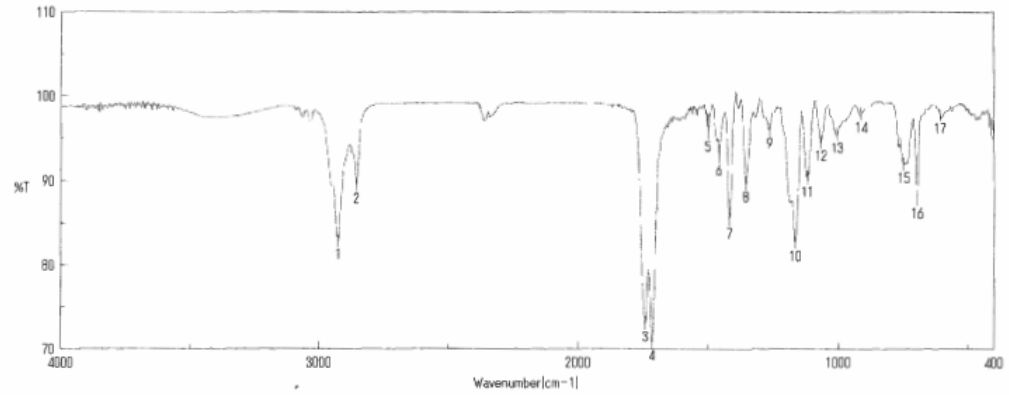
F2 - Acquisition Parameters
Date_              20050814
Time                  16.49
INSTRUM              dpx400
PROBHD   5 mm GNP 1H/29
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      699
DS                        2
SWH              31847.133 Hz
FIDRES            0.485949 Hz
AQ                1.0289652 sec
RG                    724.1
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             400.1316005 MHz

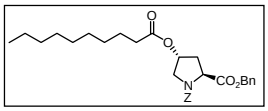
F2 - Processing parameters
SI                        32768
SF               100.6127776 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

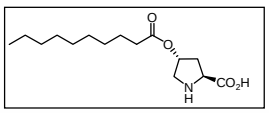
1D NMR plot parameters
CX                        20.00 cm
CY                        12.50 cm
F1P                   215.000 ppm
F1                  21631.75 Hz
F2P                    -5.000 ppm
F2                    -503.06 Hz
PPMCM                11.00000 ppm/cm
HZCM               1106.74048 Hz/cm
```



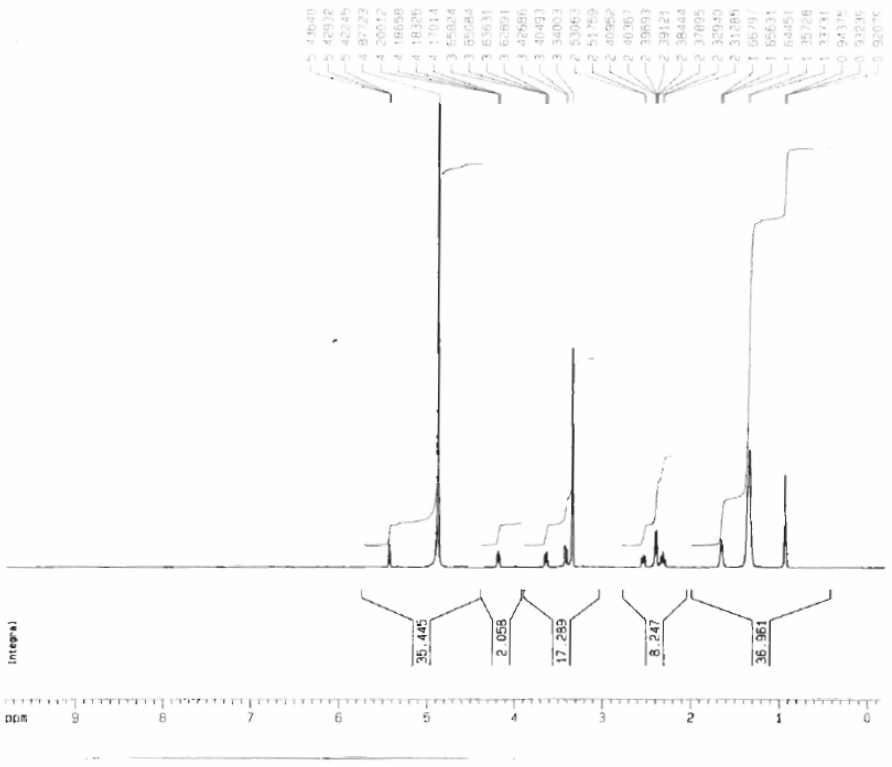

| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/01/23 18:43 | | |
| 測定者 | | | |
| ファイル名 | Memory#5 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | 2926.45, 82.9220 | 2: | 2854.13, 89.5407 | 3: | 1739.48, 73.1552 | 4: | 1714.41, 70.8629 |
| 5 | 1498.42, 95.6313 | 6: | 1455.99, 92.8165 | 7: | 1416.46, 85.3886 | 8: | 1353.78, 89.7088 |
| 9 | 1264.11, 96.0973 | 10: | 1164.79, 82.7263 | 11: | 1119.48, 90.3480 | 12: | 1066.44, 94.5772 |
| 13: | 1004.73, 95.4964 | 14: | 911.20, 97.9362 | 15: | 749.21, 91.9878 | 16: | 697.14, 87.8351 |
| 17: | 609.40, 97.8557 | | | | | | |






CARBON




```
Current Data Parameters
NAME            Oct08-2005
EXPNO                    3
PROCNO                   1

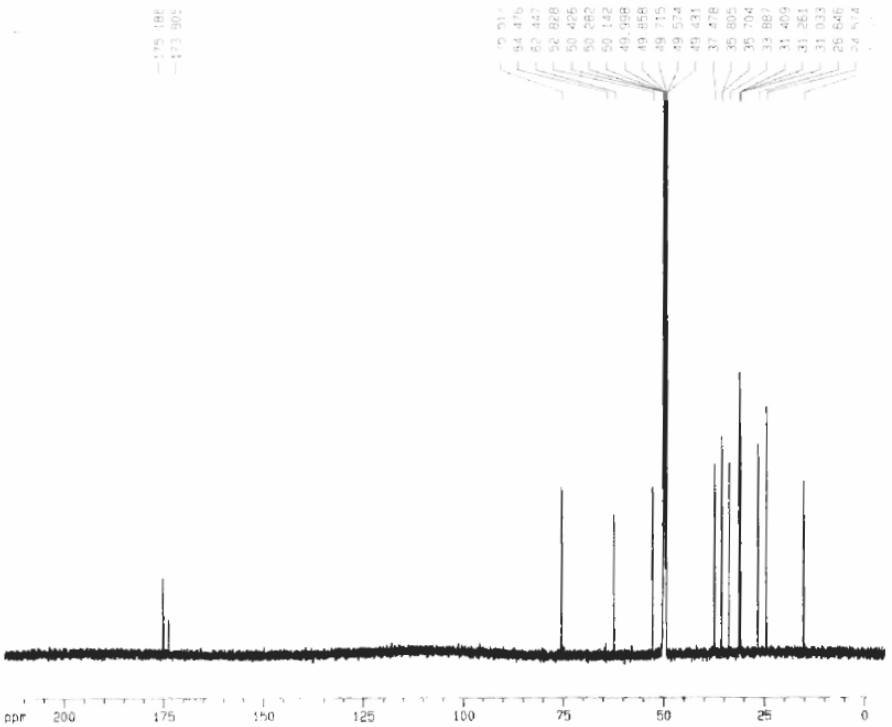
F2 - Acquisition Parameters
Date_             20051008
Time                 14.25
INSTRUM              av600
PROBHD  5 mm CPDUL 13C
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              12376.237 Hz
FIDRES           0.188846 Hz
AQ               2.6477044 sec
RG                    20.2
DW                  40.400 usec
DE                   6.00 usec
TE                  303.0 K
D1               1.00000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.40 usec
PL1                 -5.80 dB
SFO1          600.1337960 MHz

F2 - Processing parameters
SI                    32768
SF               600.1299963 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                  5881.27 Hz
F2P                  -0.200 ppm
F2                  -120.03 Hz
PPMCM                0.50000 ppm/cm
HZCM               300.06500 Hz/cm
```

CARBON




```
Current Data Parameters
NAME            Oct08-2005
EXPNO                    4
PROCNO                   1

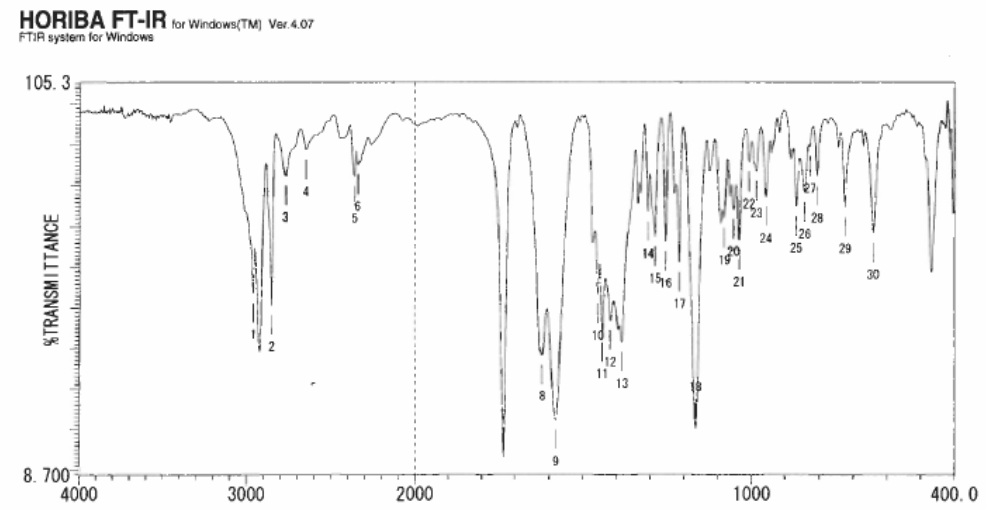
F2 - Acquisition Parameters
Date_             20051008
Time                 14.28
INSTRUM              av500
PROBHD  5 mm CPDUL 13C
PULPROG           zgpg30
TD                  65536
SOLVENT              CDCl3
NS                     207
DS                       4
SWH              35971.223 Hz
FIDRES           0.548877 Hz
AQ               0.9110004 sec
RG                  3649.1
DW                  13.900 usec
DE                   50.00 usec
TE                  303.0 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                  10.00 usec
PL1                  -4.90 dB
SFO1          150.9176988 MHz

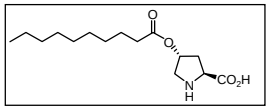
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -5.80 dB
PL12                  8.00 dB
PL13                  8.00 dB
SFO2          600.1324005 MHz

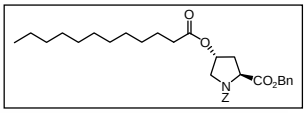
F2 - Processing parameters
SI                    32768
SF               150.9024677 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

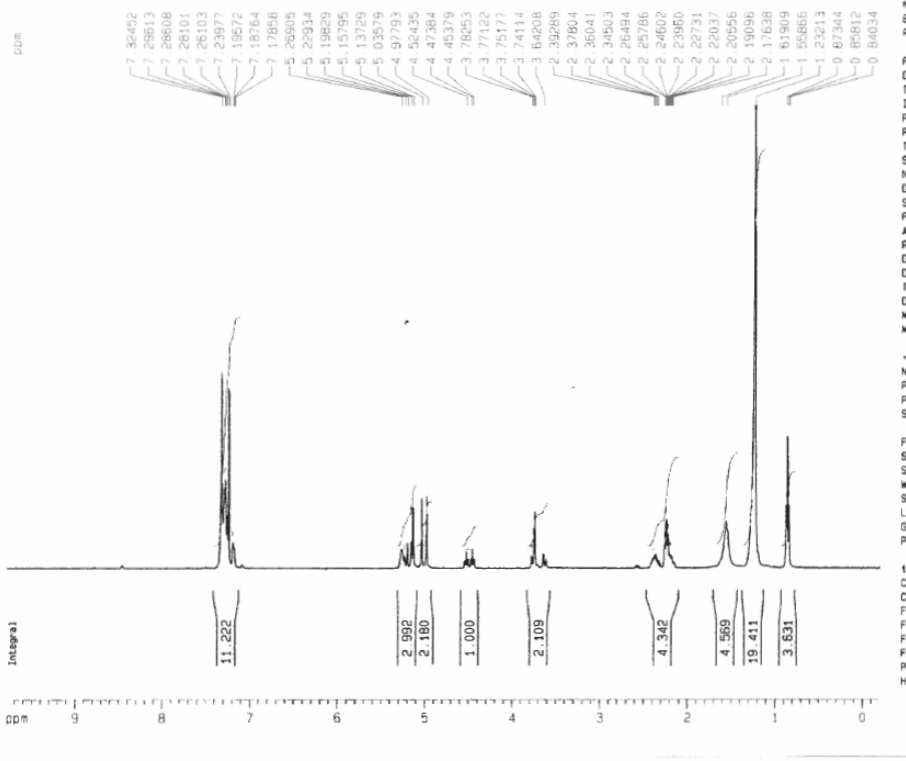
1D NMR plot parameters
CX                    20.00 cm
CY                   200.00 cm
F1P                  215.000 ppm
F1                 32444.03 Hz
F2P                  -5.000 ppm
F2                  -754.51 Hz
PPMCM               11.00000 ppm/cm
HZCM             1659.92712 Hz/cm
```




| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C10 | 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 | 2960.20 | 53.6171 | 11 | 1440.56 | 43.8453 | 21 | 1035.59 | 66.6456 |
| 測定日時 | : 2005年08月14日 15時13: | 02 | 2852.20 | 50.6219 | 12 | 1417.42 | 46.7617 | 22 | 1006.66 | 85.7027 |
| 測定分解能 | : 4 cm-1 | 03 | 2769.28 | 82.4582 | 13 | 1382.71 | 41.4614 | 23 | 985.447 | 83.3992 |
| スキャン回数 | : 10 回 | 04 | 2647.79 | 88.7167 | 14 | 1305.57 | 73.5351 | 24 | 956.520 | 77.3127 |
| 測定ゲイン | : 2 | 05 | 2358.51 | 82.1999 | 15 | 1284.36 | 67.4566 | 25 | 867.810 | 74.9349 |
| | | 06 | 2341.16 | 85.0791 | 16 | 1253.50 | 66.3750 | 26 | 842.740 | 78.3454 |
| | | 07 | 1735.62 | 13.1499 | 17 | 1213.01 | 61.1871 | 27 | 827.312 | 89.4562 |
| | | 08 | 1619.91 | 38.3162 | 18 | 1164.79 | 20.1029 | 28 | 806.099 | 82.2565 |
| | | 09 | 1579.41 | 22.2773 | 19 | 1079.94 | 71.9554 | 29 | 723.175 | 74.8001 |
| | | 10 | 1454.06 | 53.3507 | 20 | 1052.94 | 74.0782 | 30 | 640.251 | 68.4406 |





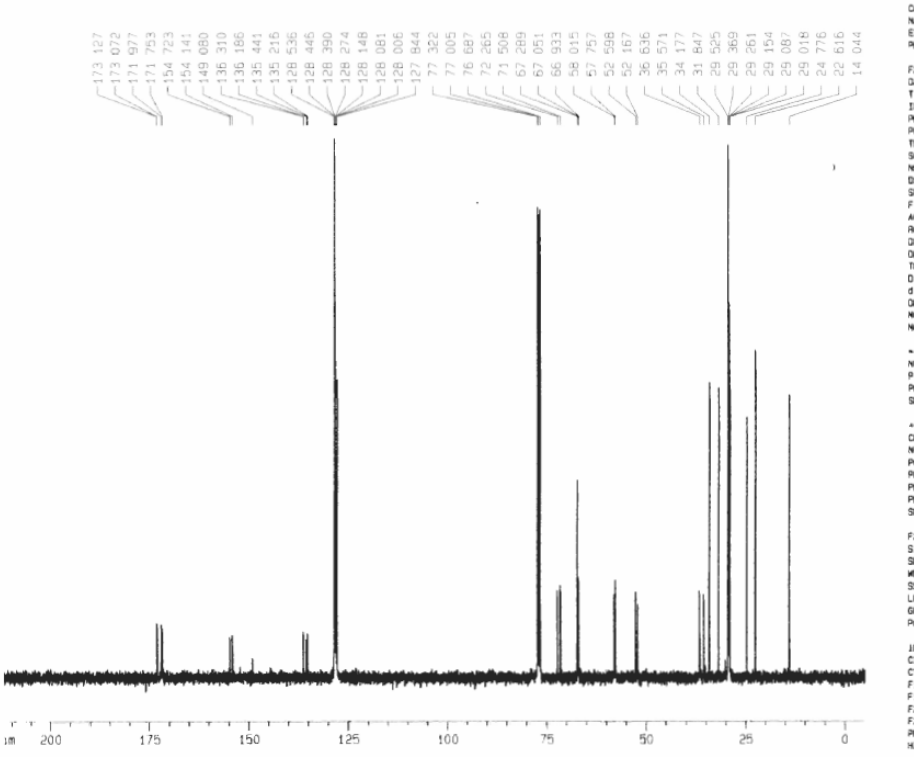

```
Current Data Parameters
NAME      Aug11-2005-haya
EXPNO               12
PROCNO               1

F2 - Acquisition Parameters
Date_          20050811
Time              13.21
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                322.5
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          400.1300177 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          200.06500 Hz/cm
```




```
Current Data Parameters
NAME      Aug14-2005-haya
EXPNO               52
PROCNO               1

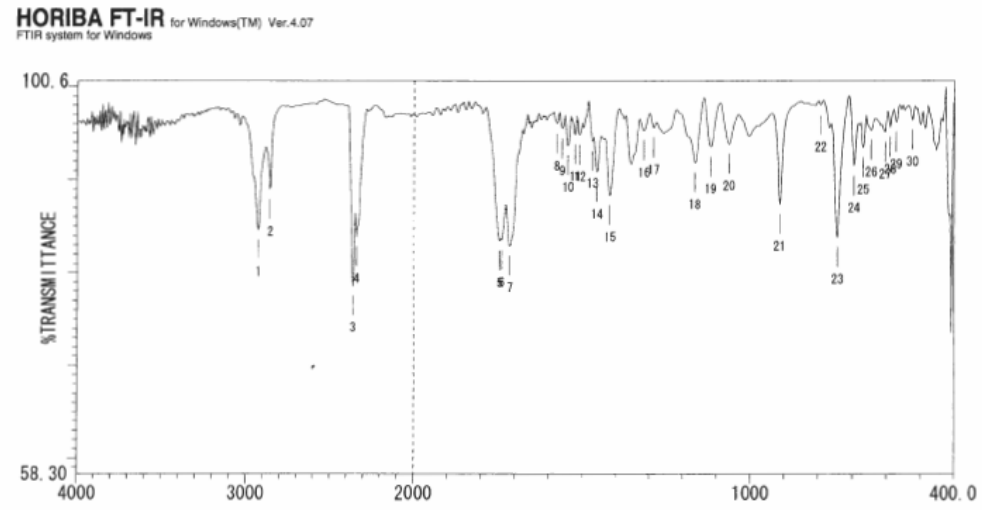
F2 - Acquisition Parameters
Date_          20050814
Time              17.28
INSTRUM          dpx400
PROBHD  5 mm QNP 1H/29
PULPROG         zgpg30
TD               65536
SOLVENT           CDCl3
NS                 584
DS                   2
SWH          31847.133 Hz
FIDRES        0.485849 Hz
AQ            1.0289652 sec
RG                2298.8
DW               15.700 usec
DE                6.00 usec
TE               303.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.68999990 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

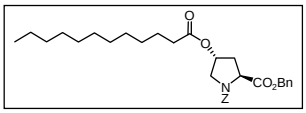
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               3.00 dB
PL12             22.00 dB
PL13             22.00 dB
SFO2        400.1316005 MHz

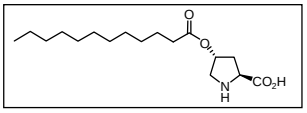
F2 - Processing parameters
SI                32768
SF          100.6127747 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

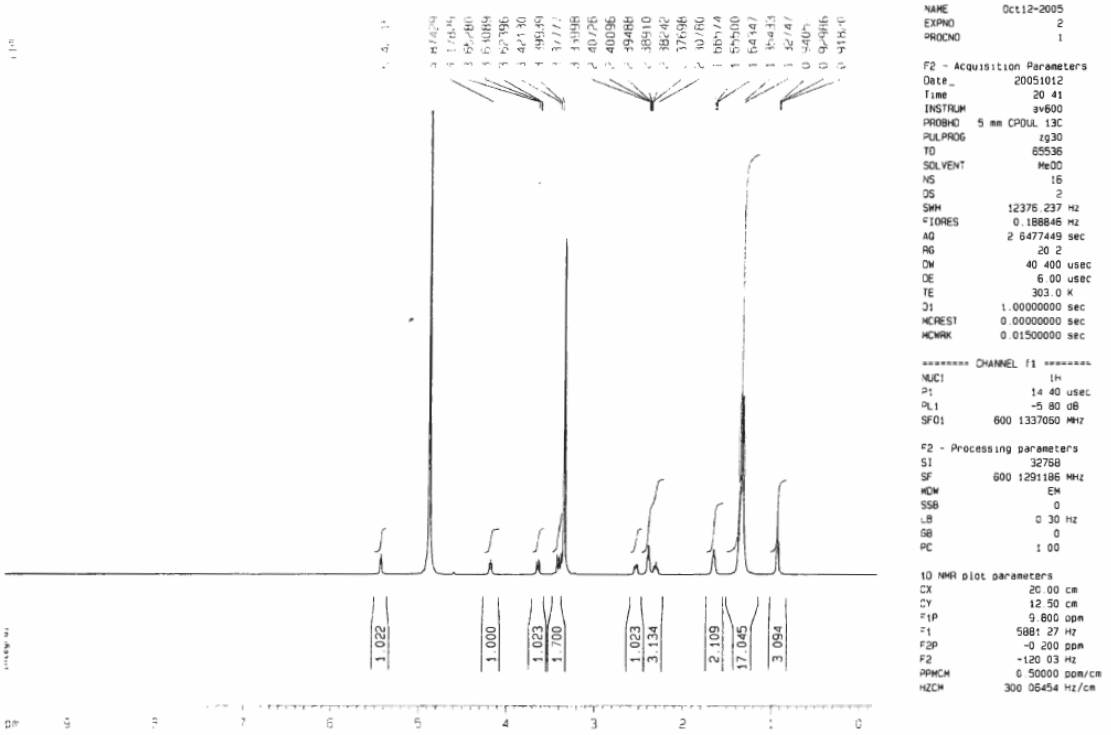
1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P             215.000 ppm
F1             21631.75 Hz
F2P              -5.000 ppm
F2             -503.06 Hz
PPMCM         11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```




| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C12H0G0 | 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 | 2925.48 | 84.5982 | 11 | 1521.56 | 94.8172 | 21 | 914.093 | 87.3375 |
| 測定日時 | : 2005年08月11日 13時51分 | 02 | 2854.13 | 88.9339 | 12 | 1508.06 | 94.7975 | 22 | 794.528 | 98.0034 |
| 測定分解能 | : 4 cm-1 | 03 | 2360.44 | 78.5612 | 13 | 1469.49 | 94.1016 | 23 | 744.388 | 83.7764 |
| スキャン回数 | : 10 回 | 04 | 2341.16 | 83.8667 | 14 | 1455.99 | 90.7779 | 24 | 696.177 | 91.4784 |
| 測定ゲイン | : 1 | 05 | 1745.26 | 83.4083 | 15 | 1417.42 | 88.2830 | 25 | 669.178 | 93.4370 |
| | | 06 | 1739.48 | 83.4426 | 16 | 1317.14 | 95.2348 | 26 | 646.036 | 95.3013 |
| | | 07 | 1714.41 | 82.8546 | 17 | 1288.22 | 95.5551 | 27 | 605.539 | 95.1089 |
| | | 08 | 1575.56 | 95.9499 | 18 | 1164.79 | 91.8092 | 28 | 590.111 | 95.6242 |
| | | 09 | 1560.13 | 95.4188 | 19 | 1118.51 | 93.4863 | 29 | 572.755 | 96.1685 |
| | | 10 | 1542.77 | 93.6509 | 20 | 1064.51 | 93.8132 | 30 | 524.543 | 96.4143 |





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Current Data Parameters
NAME                  Oct12-2005
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20051012
Time                      20 41
INSTRUM                   av600
PROBHD   5 mm CPDUL 13C
PULPROG                   zg30
TD                       65536
SOLVENT                   MeOD
NS                           16
DS                            2
SWH                 12376.237 Hz
FIDRES                0.188846 Hz
AQ                    2.6477449 sec
RG                         20.2
DW                       40.400 usec
DE                        6.00 usec
TE                       303.0 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                         14.40 usec
PL1                        -5.80 dB
SFO1                600.1337050 MHz

F2 - Processing parameters
SI                         32768
SF                  600.1291186 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                       5881.27 Hz
F2P                       -0.200 ppm
F2                      -120.03 Hz
PPMCM                  0.50000 ppm/cm
HZCM                 300.06454 Hz/cm
```
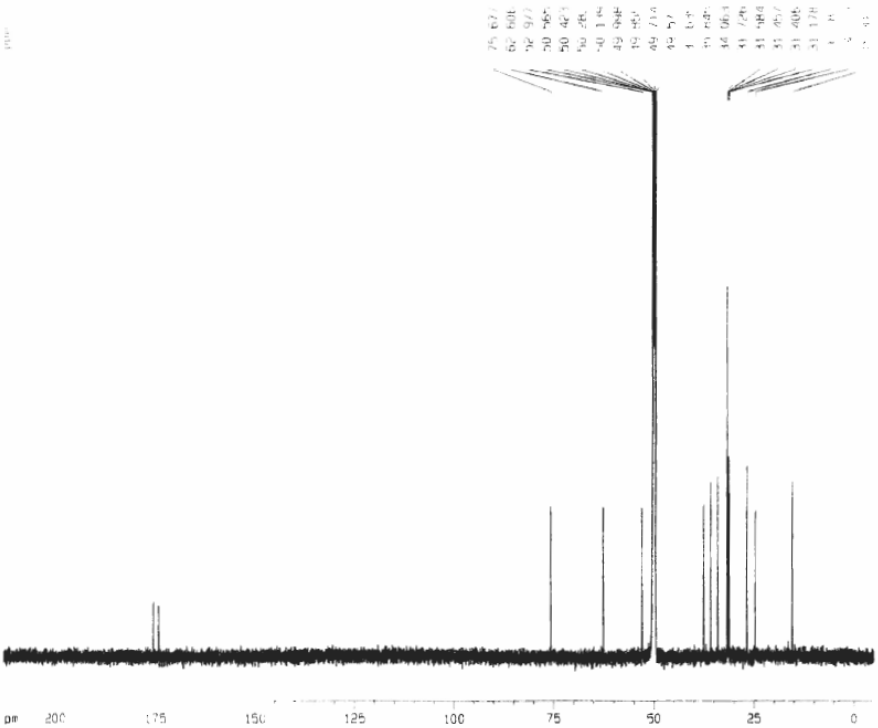

CARBON



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Current Data Parameters
NAME                  Oct12-2005
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20051012
Time                      20.46
INSTRUM                   av600
PROBHD   5 mm CPDUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         282
DS                           4
SWH                 35971.223 Hz
FIDRES                0.548877 Hz
AQ                    0.9110143 sec
RG                        8192
DW                      13.900 usec
DE                       50.00 usec
TE                       303.0 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                         10.00 usec
PL1                        -4.90 dB
SFO1                150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                     100.00 usec
PL2                        -5.80 dB
PL12                        8.00 dB
PL13                        8.00 dB
SFO2                600.1324005 MHz

F2 - Processing parameters
SI                         32768
SF                  150.9022259 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
CY                        300.00 cm
F1P                       215.000 ppm
F1                      32443.98 Hz
F2P                        -5.000 ppm
F2                       -754.51 Hz
PPMCM                  11.00000 ppm/cm
HZCM                 1659.92444 Hz/cm
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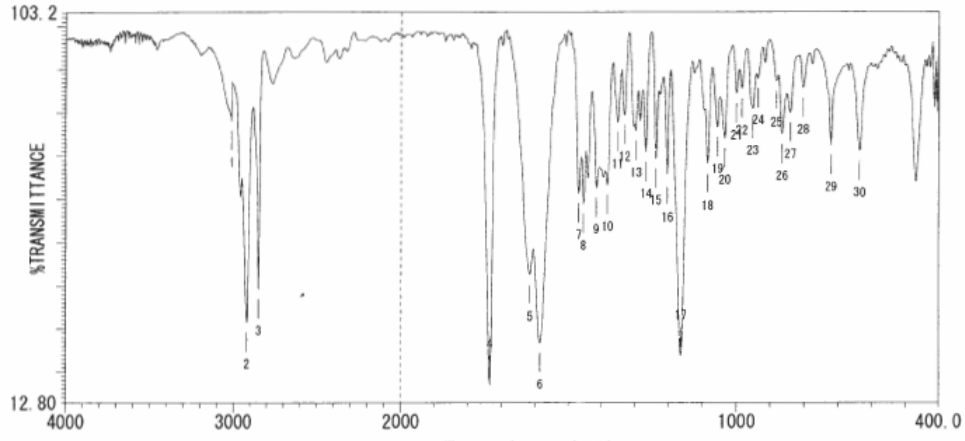
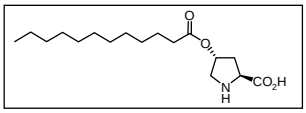
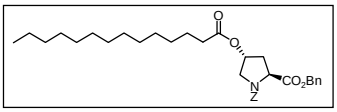
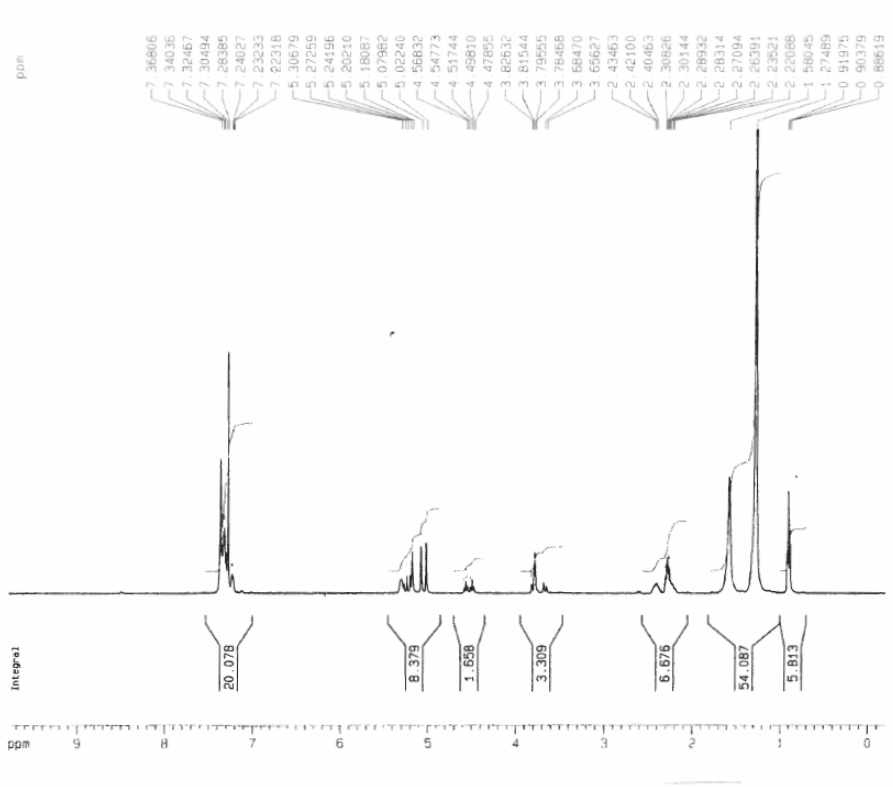
**HORIBA FT-IR** for Windows(TM) Ver.4.07
FTIR system for Windows




| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C12 | 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 | 3012.27 | 78.0972 | 11 | 1353.78 | 77.7170 | 21 | 1000.87 | 84.3388 |
| 測定日時 | : 2005年08月14日 15時31: | 02 | 2919.70 | 31.3808 | 12 | 1332.57 | 79.4081 | 22 | 985.447 | 85.4656 |
| 測定分解能 | 4 cm-1 | 03 | 2850.27 | 39.1254 | 13 | 1299.79 | 75.7146 | 23 | 952.663 | 80.8460 |
| スキャン回数 | 10 回 | 04 | 1735.62 | 16.9540 | 14 | 1268.93 | 70.8401 | 24 | 937.235 | 87.8566 |
| 測定ゲイン | 2 | 05 | 1616.06 | 42.4084 | 15 | 1240.00 | 69.3218 | 25 | 883.238 | 87.3021 |
| | | 06 | 1585.20 | 26.6267 | 16 | 1205.29 | 65.3720 | 26 | 865.882 | 74.9395 |
| | | 07 | 1469.49 | 61.2860 | 17 | 1164.79 | 23.5389 | 27 | 840.812 | 79.9529 |
| | | 08 | 1455.99 | 58.8338 | 18 | 1085.73 | 68.0102 | 28 | 804.171 | 85.6780 |
| | | 09 | 1417.42 | 62.5318 | 19 | 1056.80 | 76.5819 | 29 | 721.247 | 72.4102 |
| | | 10 | 1384.64 | 63.2448 | 20 | 1035.59 | 74.0087 | 30 | 636.394 | 71.0923 |



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Current Data Parameters
NAME        Aug18-2005-haya
EXPNO                    71
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050818
Time                  18.24
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                   456.1
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            400.1300000 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          200.06500 Hz/cm
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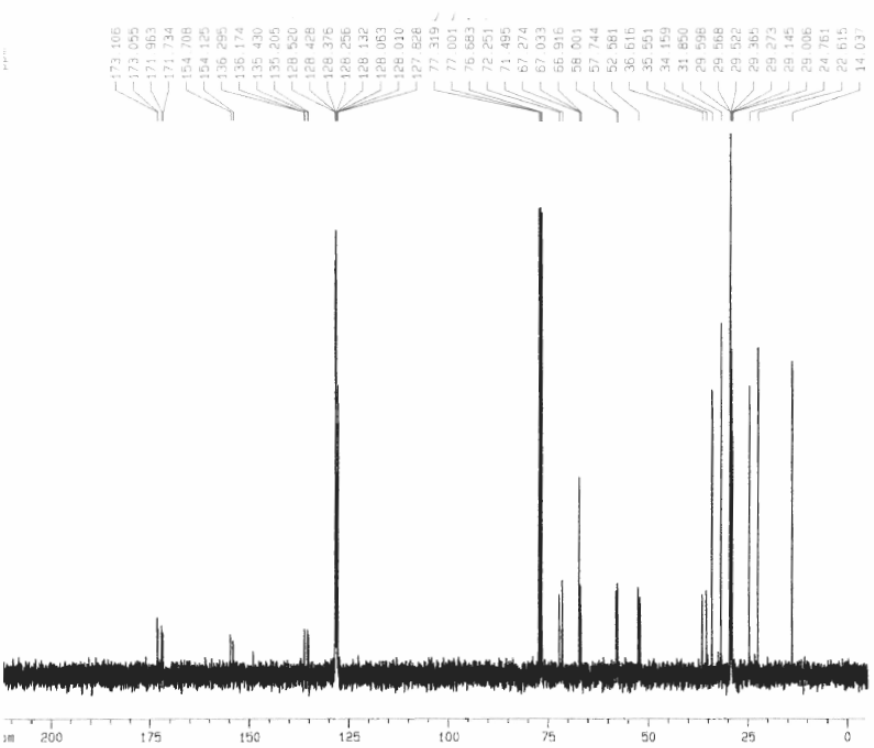
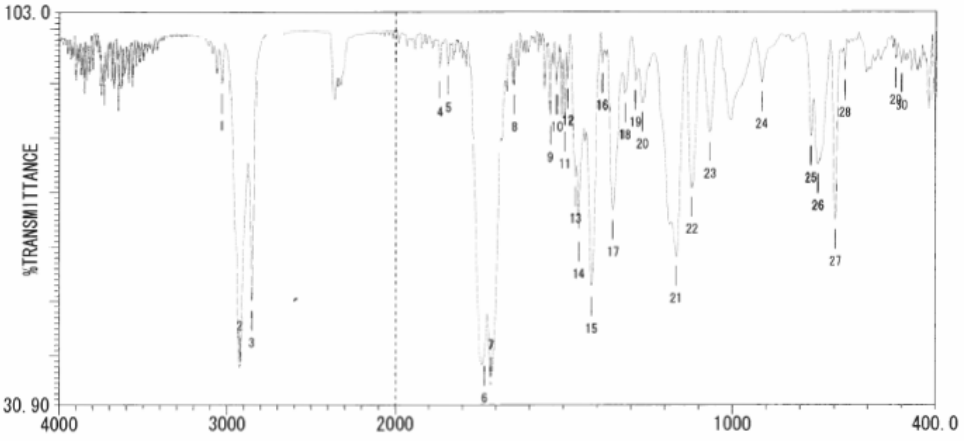
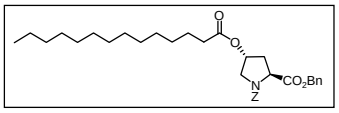


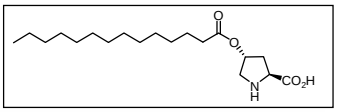

```
Current Data Parameters
NAME        Aug19-2005-haya
EXPNO                    21
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050819
Time                  13.04
INSTRUM              dpx400
PROBHD  5 mm QNP 1H/29
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      81
DS                       2
SWH            31847.133 Hz
FIDRES          0.485949 Hz
AQ              1.0289652 sec
RG                  2580.3
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          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127766 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  12.50 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
```

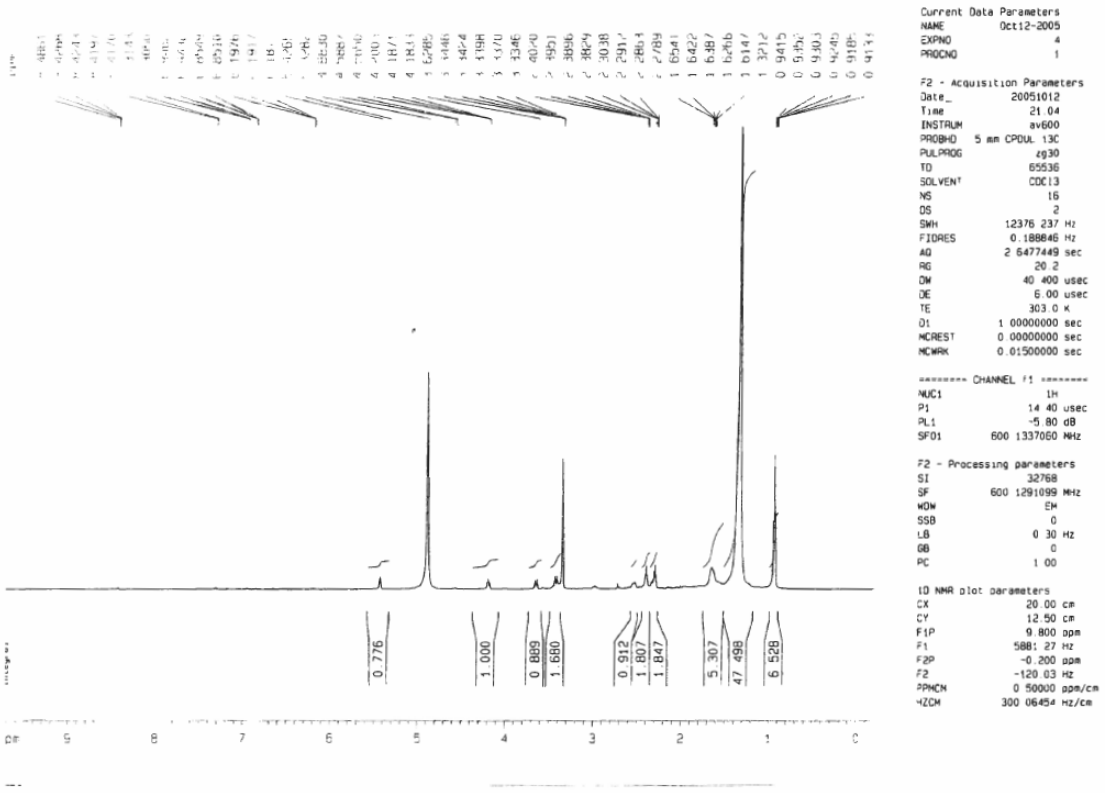
**HORIBA FT-IR** for Windows(TM) Ver.4.07
FTIR system for Windows




| | | | 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(%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C14H0G0 | | 01 | 3033.48 | 89.8256 | 11 | 1498.42 | 82.7801 | 21 | 1166.72 | 58.1070 |
| タイトル | : | | 02 | 2925.48 | 37.8132 | 12 | 1488.78 | 90.6975 | 22 | 1120.44 | 70.8482 |
| 測定日時 | : 2005年08月19日 | 14時34: | 03 | 2854.13 | 49.9966 | 13 | 1465.63 | 72.7715 | 23 | 1066.44 | 80.9960 |
| 測定分解能 | : 4 cm-1 | | 04 | 1868.68 | 92.3511 | 14 | 1455.99 | 62.5934 | 24 | 914.093 | 90.1601 |
| スキャン回数 | : 10 回 | | 05 | 1843.61 | 93.0853 | 15 | 1417.42 | 52.5432 | 25 | 769.458 | 80.2212 |
| 測定ゲイン | : 1 | | 06 | 1735.62 | 39.7956 | 16 | 1386.57 | 93.4879 | 26 | 748.245 | 75.1024 |
| | | | 07 | 1716.34 | 34.2902 | 17 | 1353.78 | 66.6324 | 27 | 698.105 | 64.9272 |
| | | | 08 | 1646.91 | 89.6056 | 18 | 1319.07 | 88.0972 | 28 | 669.178 | 92.2321 |
| | | | 09 | 1540.85 | 83.9992 | 19 | 1288.22 | 90.4285 | 29 | 518.758 | 94.4572 |
| | | | 10 | 1521.56 | 89.6150 | 20 | 1267.00 | 86.4725 | 30 | 501.401 | 93.5020 |






Current Data Parameters
NAME Oct12-2005
EXPNO 4
PROCNO 1

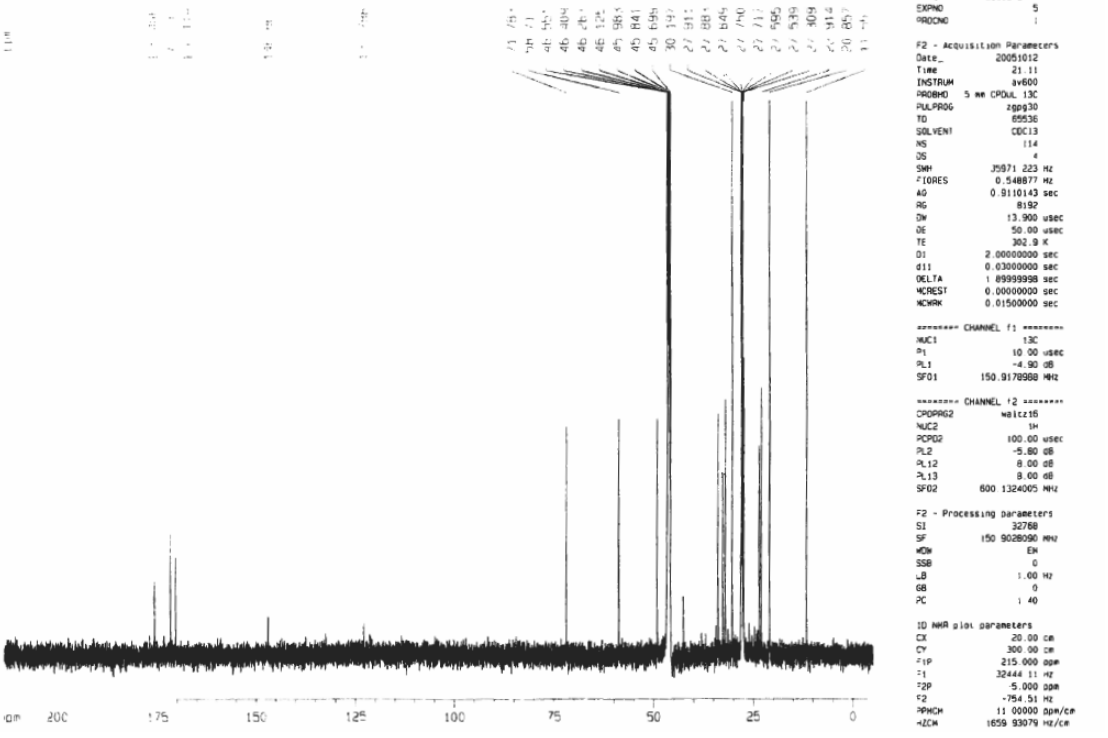
F2 - Acquisition Parameters
Date_ 20051012
Time 21.04
INSTRUM av600
PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477449 sec
RG 20.2
DW 40.400 usec
DE 6.00 usec
TE 303.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.40 usec
PL1 -5.80 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 32768
SF 600.1291099 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 5881.27 Hz
F2P -0.200 ppm
F2 -120.03 Hz
PPMCM 0.50000 ppm/cm
HZCM 300.06454 Hz/cm

CARBON




Current Data Parameters
NAME Oct12-2005
EXPNO 5
PROCNO 1

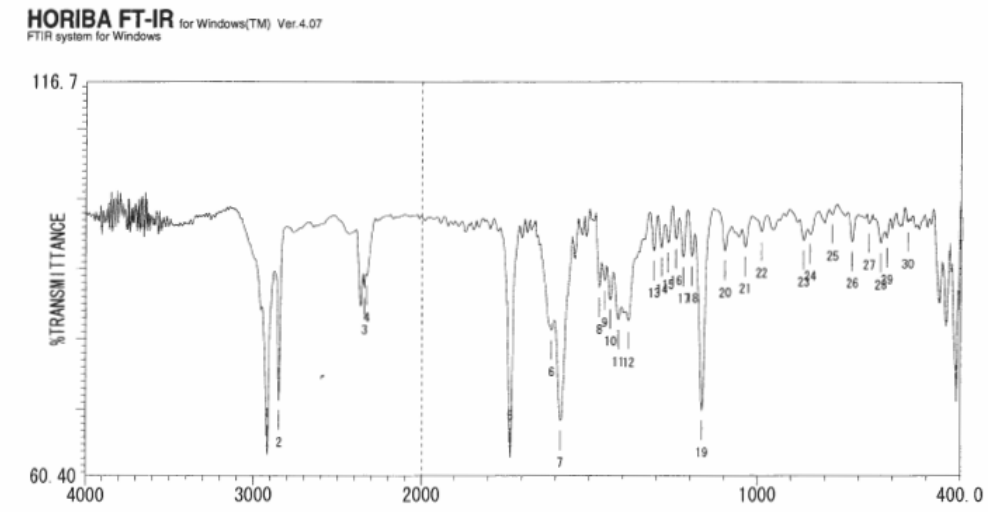
F2 - Acquisition Parameters
Date_ 20051012
Time 21.11
INSTRUM av600
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 114
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 8192
DW 13.900 usec
DE 50.00 usec
TE 302.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -4.90 dB
SFO1 150.9178988 MHz

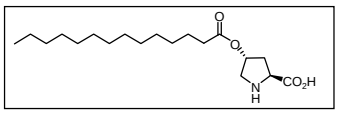
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.80 dB
PL12 8.00 dB
PL13 8.00 dB
SFO2 600.1324005 MHz

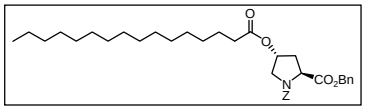
F2 - Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

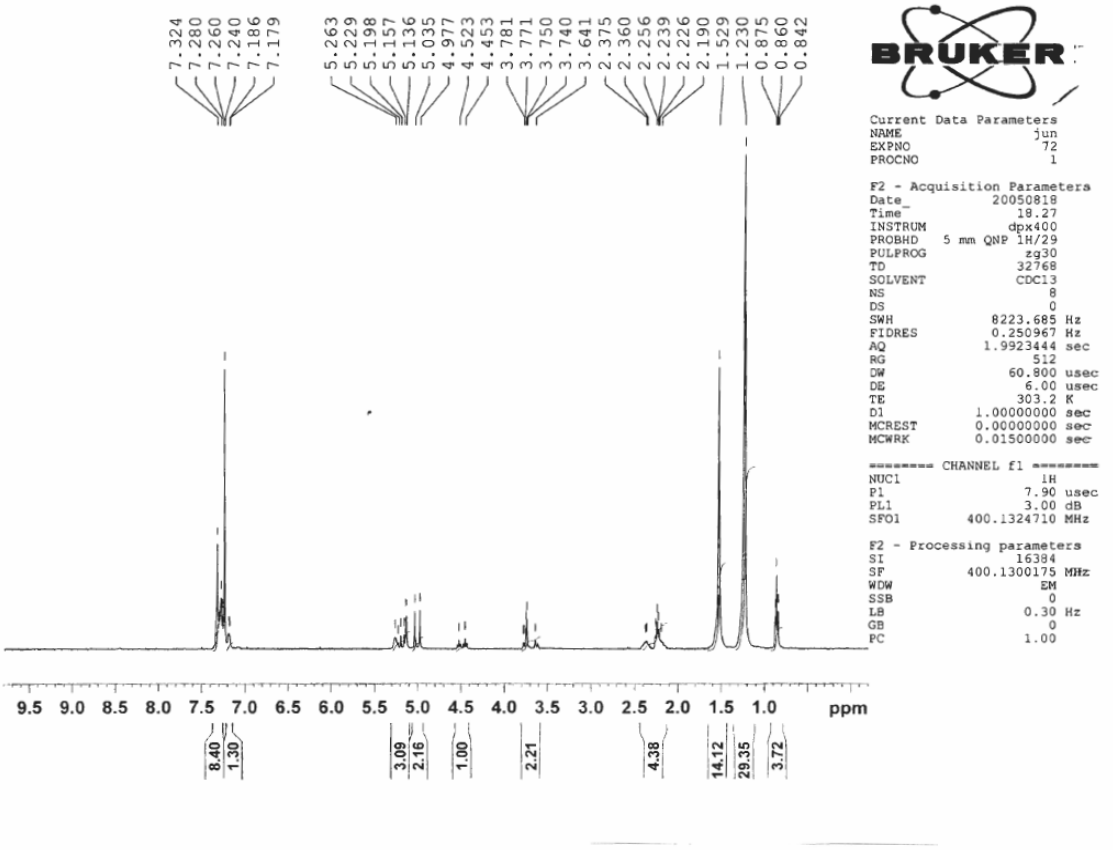
1D NMR plot parameters
CX 20.00 cm
CY 300.00 cm
F1P 215.000 ppm
F1 32444.11 Hz
F2P -5.000 ppm
F2 -754.51 Hz
PPMCM 11.00000 ppm/cm
HZCM 1659.93079 Hz/cm




| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C14 | 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 | 2917.77 | 63.4282 | 11 | 1415.49 | 82.6841 | 21 | 1037.52 | 93.1586 |
| 測定日時 | : 2005年08月23日 16時54: | 02 | 2850.27 | 71.1284 | 12 | 1384.64 | 82.5587 | 22 | 989.304 | 95.3858 |
| 測定分解能 | : 4 cm-1 | 03 | 2343.09 | 87.2925 | 13 | 1309.43 | 92.5379 | 23 | 863.953 | 94.1245 |
| スキャン回数 | : 10 回 | 04 | 2329.59 | 89.0062 | 14 | 1286.29 | 93.0616 | 24 | 844.668 | 95.0060 |
| 測定ゲイン | : 1 | 05 | 1735.62 | 63.0286 | 15 | 1267.00 | 93.7145 | 25 | 779.101 | 97.8447 |
| | | 06 | 1614.13 | 81.2161 | 16 | 1243.86 | 94.2581 | 26 | 721.247 | 93.9527 |
| | | 07 | 1587.13 | 68.2594 | 17 | 1220.72 | 91.7377 | 27 | 671.106 | 96.5908 |
| | | 08 | 1471.42 | 87.3191 | 18 | 1195.65 | 91.7768 | 28 | 636.394 | 93.7491 |
| | | 09 | 1455.99 | 88.3135 | 19 | 1164.79 | 69.6938 | 29 | 619.038 | 94.4989 |
| | | 10 | 1438.64 | 85.6179 | 20 | 1097.30 | 92.5933 | 30 | 557.327 | 96.7545 |




O
O
N
Z
CO2Bn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7.240
7.186
7.179
5.263
5.229
5.198
5.157
5.136
5.035
4.977
4.523
4.453
3.781
3.771
3.750
3.740
3.641
2.375
2.360
2.256
2.239
2.226
2.190
1.529
1.230
0.875
0.860
0.842
BRUKER
Current Data Parameters
NAME jun
EXPNO 72
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050818
Time_ 18.27
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 512
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 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm
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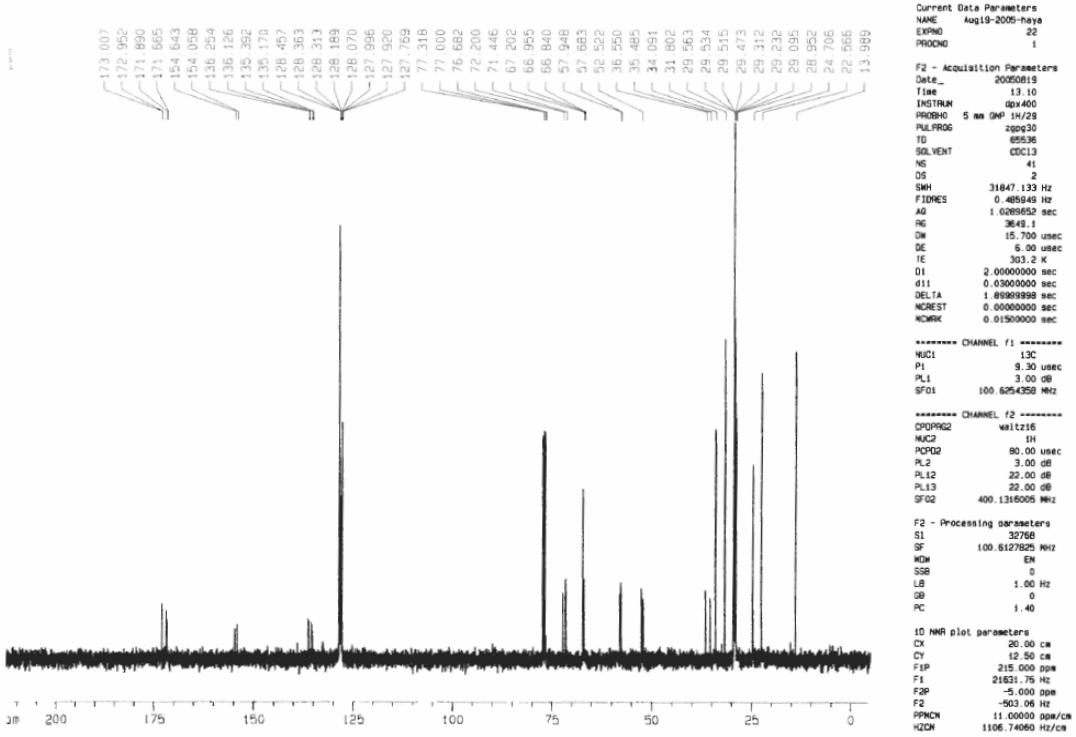
1.30
3.09
2.16
1.00
2.21
4.38
14.12
29.35
3.72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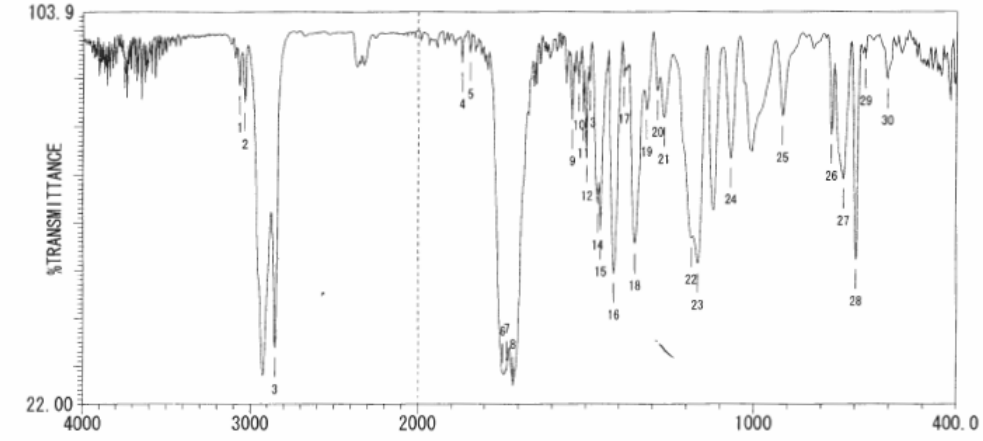
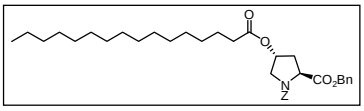
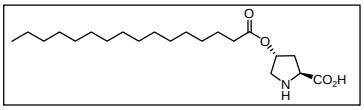
171.665
154.643
154.058
136.254
136.126
135.392
135.170
128.457
128.363
128.313
128.189
128.070
127.996
127.920
127.769
77.318
77.000
76.682
72.200
71.446
67.202
66.955
66.840
57.948
57.683
52.522
36.550
35.485
34.051
31.802
29.563
29.534
29.515
29.473
29.312
29.232
29.095
28.952
24.706
22.566
13.989
200
175
150
125
100
75
50
25
0
Current Data Parameters
NAME Aug19-2005-haya
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050819
Time 13.10
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 41
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 3649.1
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 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127825 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.74060 Hz/cm

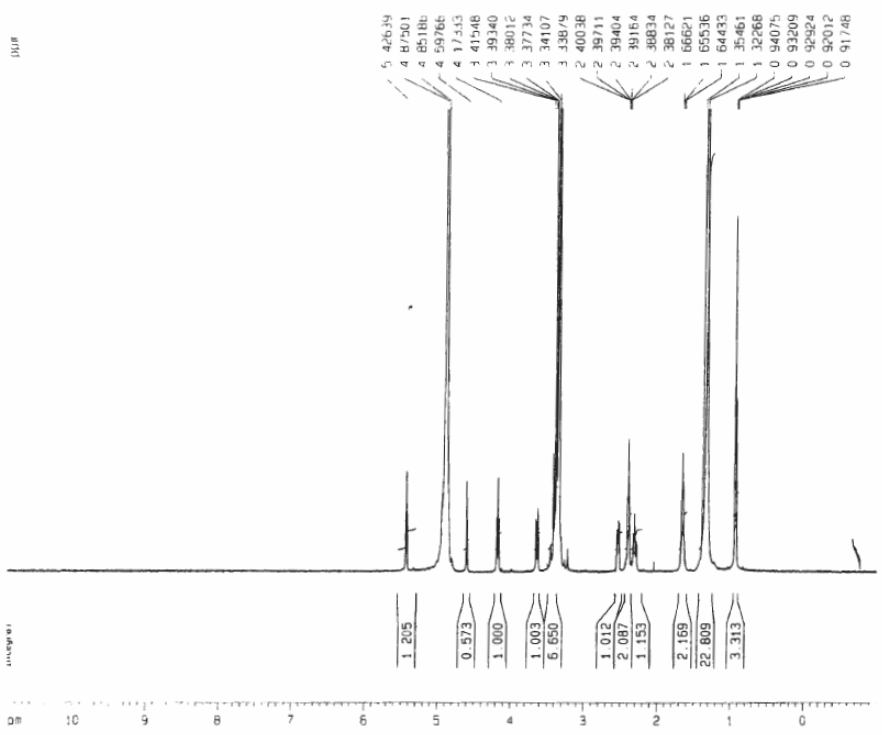
**HORIBA FT-IR** for Windows(TM) Ver.4.07
FTIR system for Windows



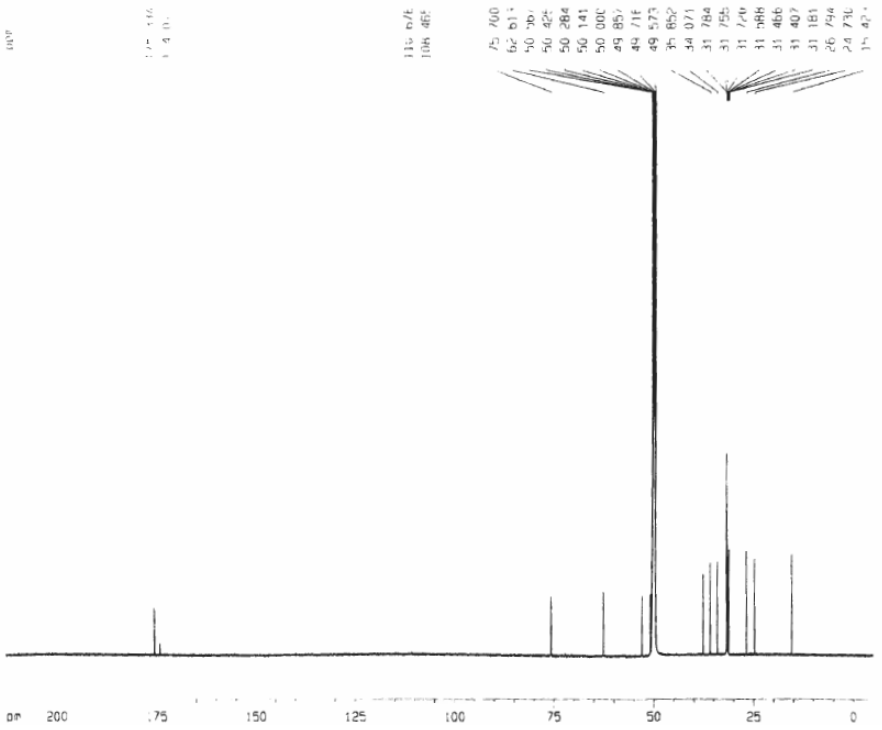

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C16H0G0 | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) |
| タイトル | : | 01 | 3066.26 | 88.4245 | 11 | 1508.06 | 83.0594 | 21 | 1267.00 | 81.8894 |
| 測定日時 | : 2005年08月19日 14時43: | 02 | 3033.48 | 84.9888 | 12 | 1498.42 | 74.2269 | 22 | 1186.01 | 56.7212 |
| 測定分解能 | : 4 cm-1 | 03 | 2854.13 | 33.7720 | 13 | 1488.78 | 89.4615 | 23 | 1166.72 | 51.3869 |
| スキャン回数 | : 10 回 | 04 | 1868.68 | 93.3169 | 14 | 1465.63 | 63.8751 | 24 | 1068.37 | 73.3827 |
| 測定ゲイン | : 1 | 05 | 1843.61 | 95.3807 | 15 | 1455.99 | 58.3427 | 25 | 914.093 | 82.2026 |
| | | 06 | 1747.19 | 28.5184 | 16 | 1417.42 | 49.3204 | 26 | 769.458 | 78.3433 |
| | | 07 | 1735.62 | 29.1129 | 17 | 1386.57 | 90.3116 | 27 | 734.746 | 69.0077 |
| | | 08 | 1716.34 | 25.8248 | 18 | 1353.78 | 55.4425 | 28 | 698.105 | 52.2175 |
| | | 09 | 1540.85 | 81.4253 | 19 | 1319.07 | 83.3933 | 29 | 669.178 | 94.0280 |
| | | 10 | 1521.56 | 88.9645 | 20 | 1286.29 | 87.4768 | 30 | 605.539 | 89.9251 |



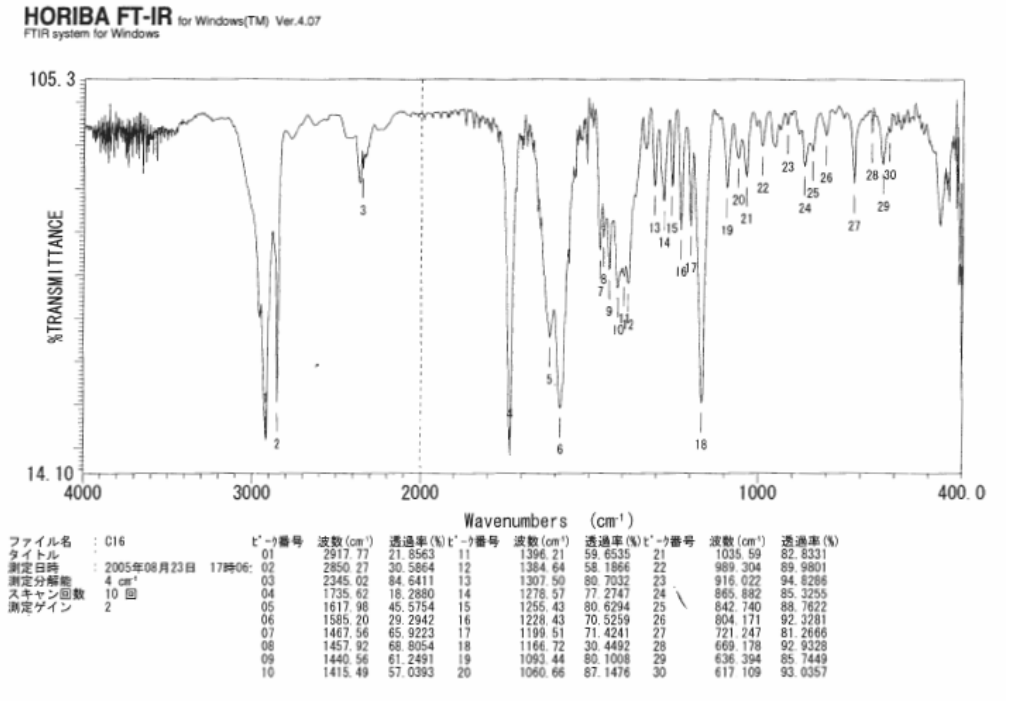


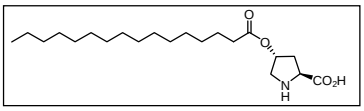

```
Current Data Parameters
NAME            Oct12-2005
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20051012
Time                 21.21
INSTRUM              av600
PROBHD   5 mm CPDUL 13C
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH           12376.237 Hz
FIDRES        0.188846 Hz
AQ            2.6477449 sec
RG                      64
DW               40.400 usec
DE                6.00 usec
TE                303.0 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                 11.40 usec
PL1                -3.00 dB
SFO1          600.1337060 MHz

F2 - Processing parameters
SI                      32768
SF            600.1299944 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                     300.00 cm
F1P                    11.000 ppm
F1                    6601.43 Hz
F2P                    -1.000 ppm
F2                    -600.13 Hz
PPMCM                 0.60000 ppm/cm
HZCM                360.07800 Hz/cm
```

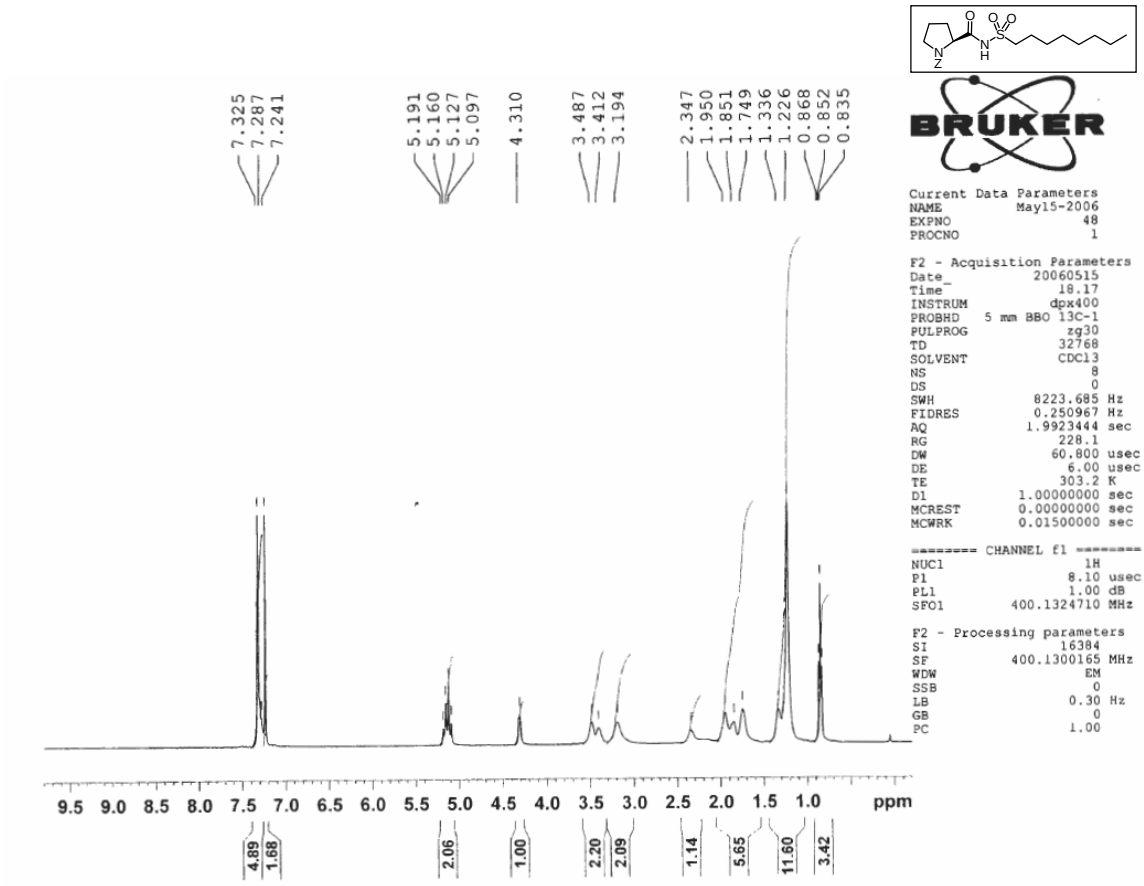


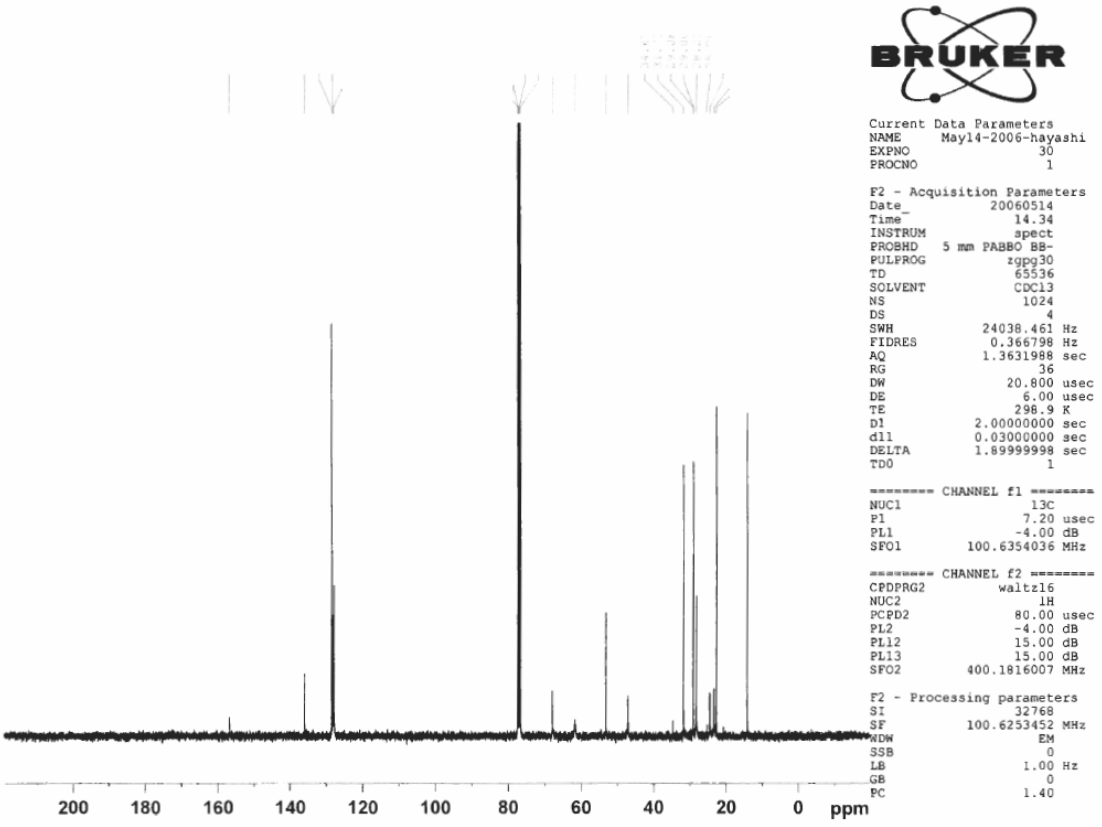

```
Current Data Parameters
NAME            Oct12-2005
EXPNO                    8
PROCNO                   1

F2 - Acquisition Parameters
Date_             20051012
Time                 22.04
INSTRUM              av600
PROBHD   5 mm CPDUL 13C
PULPROG           zgpg30
TD                  65536
SOLVENT              CDCl3
NS                   14135
DS                       4
SWH           35971.223 Hz
FIDRES        0.548877 Hz
AQ            0.9110143 sec
RG                    8192
DW               13.900 usec
DE                50.00 usec
TE                303.0 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                 10.00 usec
PL1                -4.90 dB
SFO1          150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -5.80 dB
PL12                 8.00 dB
PL13                 8.00 dB
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                      32768
SF            150.9024455 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
CY                     500.00 cm
F1P                    215.000 ppm
F1                   32444.03 Hz
F2P                    -5.000 ppm
F2                    -754.51 Hz
PPMCM                 11.00000 ppm/cm
```

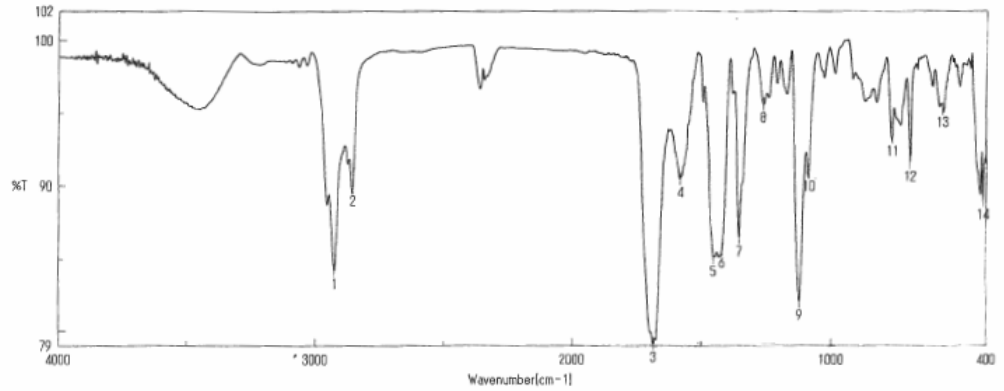



| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ファイル名 | : C16 | | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) | ﾋﾟｰｸ番号 | 波数(cm-1) | 透過率(%) |
| タイトル | : | | 01 | 2917.77 | 21.8563 | 11 | 1396.21 | 59.6535 | 21 | 1035.59 | 82.8331 | | | |
| 測定日時 | : 2005年08月23日 | 17時06: | 02 | 2850.27 | 30.5864 | 12 | 1384.64 | 58.1866 | 22 | 989.304 | 89.9801 | | | |
| 測定分解能 | 4 cm-1 | | 03 | 2345.02 | 84.6411 | 13 | 1307.50 | 80.7032 | 23 | 916.022 | 94.8266 | | | |
| スキャン回数 | 10 回 | | 04 | 1735.62 | 18.2880 | 14 | 1278.57 | 77.2747 | 24 | 865.882 | 85.3255 | | | |
| 測定ゲイン | 2 | | 05 | 1617.98 | 45.5754 | 15 | 1255.43 | 80.6294 | 25 | 842.740 | 88.7622 | | | |
| | | | 06 | 1585.20 | 29.2942 | 16 | 1228.43 | 70.5259 | 26 | 804.171 | 92.3281 | | | |
| | | | 07 | 1467.56 | 65.9223 | 17 | 1199.51 | 71.4241 | 27 | 721.247 | 81.2666 | | | |
| | | | 08 | 1457.92 | 68.8054 | 18 | 1166.72 | 30.4492 | 28 | 669.178 | 92.9328 | | | |
| | | | 09 | 1440.56 | 61.2491 | 19 | 1093.44 | 80.1008 | 29 | 636.394 | 85.7449 | | | |
| | | | 10 | 1415.49 | 57.0393 | 20 | 1060.66 | 87.1476 | 30 | 617.109 | 93.0357 | | | |



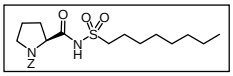

7.325
7.287
7.241
5.191
5.160
5.127
5.097
4.310
3.487
3.412
3.194
2.347
1.950
1.851
1.749
1.336
1.226
0.868
0.852
0.835
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
4.89
1.68
2.06
1.00
2.20
2.09
1.14
5.65
11.60
3.42
BRUKER
Current Data Parameters
NAME May15-2006
EXPNO 48
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060515
Time 18.17
INSTRUM dpx400
PROBHD 5 mm BBO 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 228.1
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 8.10 usec
PL1 1.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300165 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

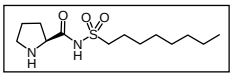

200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME May14-2006-hayashi
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060514
Time 14.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 36
DW 20.800 usec
DE 6.00 usec
TE 298.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 -4.00 dB
SFO1 100.6354036 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1816007 MHz
F2 - Processing parameters
SI 32768
SF 100.6253452 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

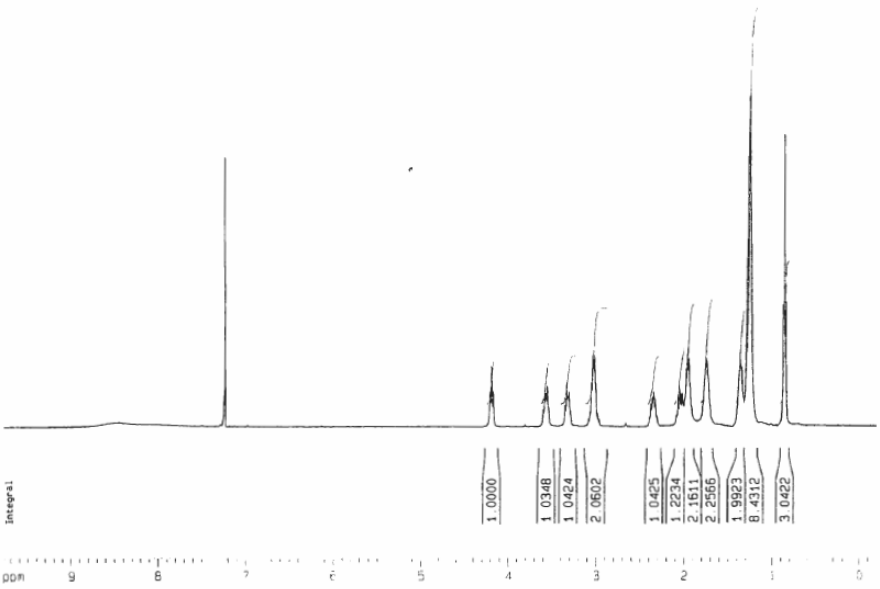



| | | | |
|---|---|---|---|
| 積算回数 | 16 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 106/05/15 16:55 | | |
| 測定者 | | | |
| ファイル名 | Memory#3 | | |
| サンプル名 | | | |
| コメント | コメント | | |

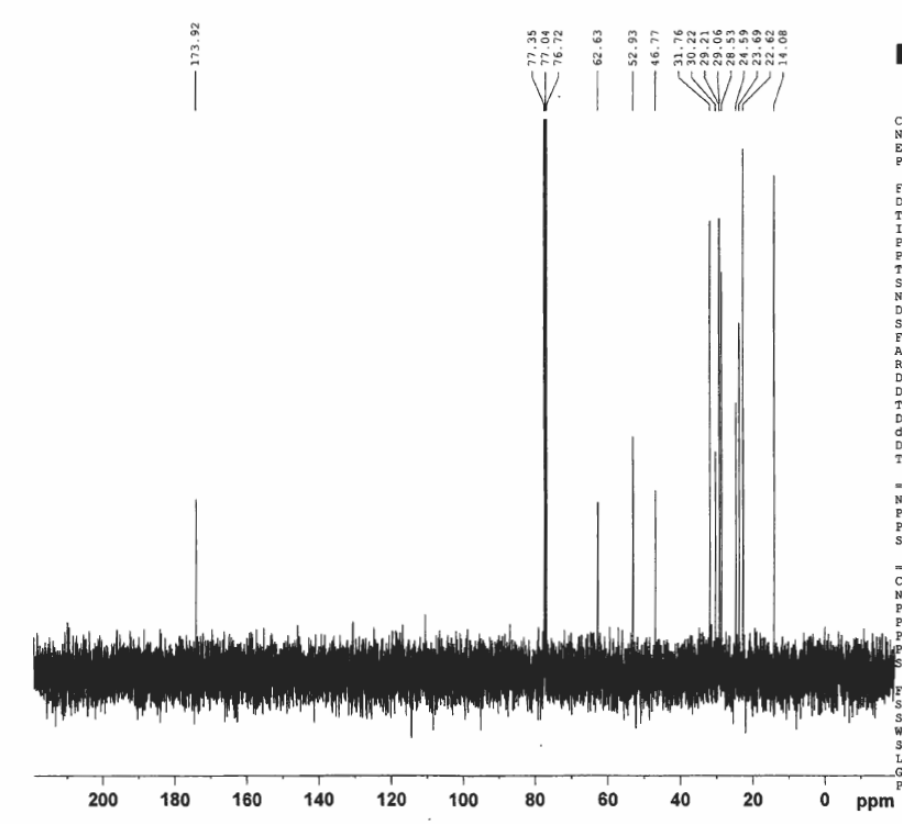
| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 2925.48, | 84.2426 | 2 | 2852.20, | 89.9583 | 3 | 1685.48, | 79.1575 | 4 | 1584.24, | 90.5256 |
| 5 | 1455.99, | 85.0904 | 6 | 1422.24, | 85.6664 | 7 | 1357.64, | 86.4462 | 8 | 1264.11, | 95.6602 |
| 9 | 1124.30, | 82.0730 | 10 | 1087.66, | 90.9633 | 11 | 766.57, | 93.3643 | 12 | 697.14, | 91.6188 |
| 13 | 571.79, | 95.3065 | 14 | 413.66, | 89.0084 | | | | | | |





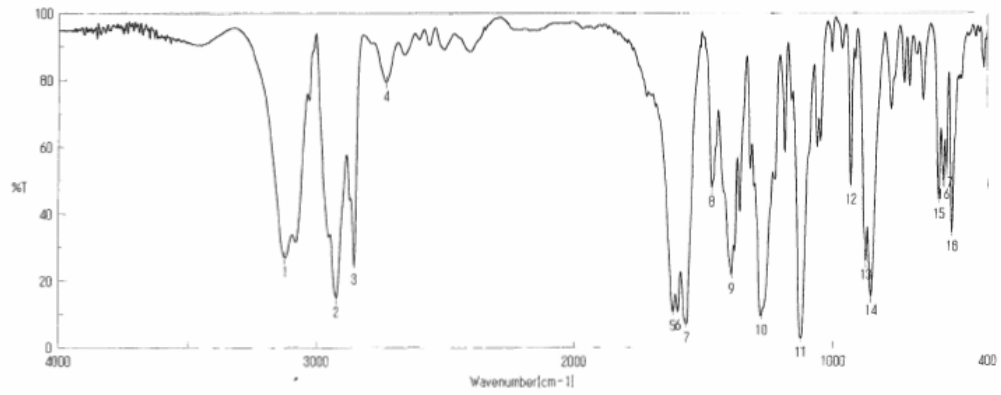



```
Current Data Parameters
NAME          Dec15-2005
EXPNO                 75
PROCNO                 1

F2 - Acquisition Parameters
Date_           20051216
Time               19.37
INSTRUM          dpx400
PROBHD  5 mm BBO 13C-1
PULPROG             zg30
TD                 32768
SOLVENT             CDCl3
NS                     8
DS                     0
SWH             8223.685 Hz
FIDRES          0.250987 Hz
AQ              1.9923444 sec
RG                   256
DW                40.800 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                  8.10 usec
PL1                 1.00 dB
SFO1          400.1324710 MHz

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
CY                  7.50 cm
F1P                9.800 ppm
F1                3921.27 Hz
F2P                0.200 ppm
F2                  80.03 Hz
PPMCM          0.50000 ppm/cm
HZCM           200.06500 Hz/cm
```




BRUKER

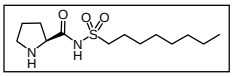
```
Current Data Parameters
NAME      May10-2006-hayashi
EXPNO                     50
PROCNO                     1

F2 - Acquisition Parameters
Date_           20060510
Time               18.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    2
SWH           24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  114
DW                20.800 use
DE                 6.00 use
TE                298.3 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.20 use
PL1                -4.00 dB
SFO1          100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 use
PL2                -4.00 dB
PL12               15.00 dB
PL13               15.00 dB
SFO2          400.1816007 MHz

F2 - Processing parameters
SI                   32768
SF            100.6253410 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

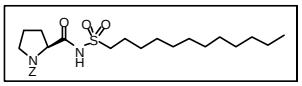


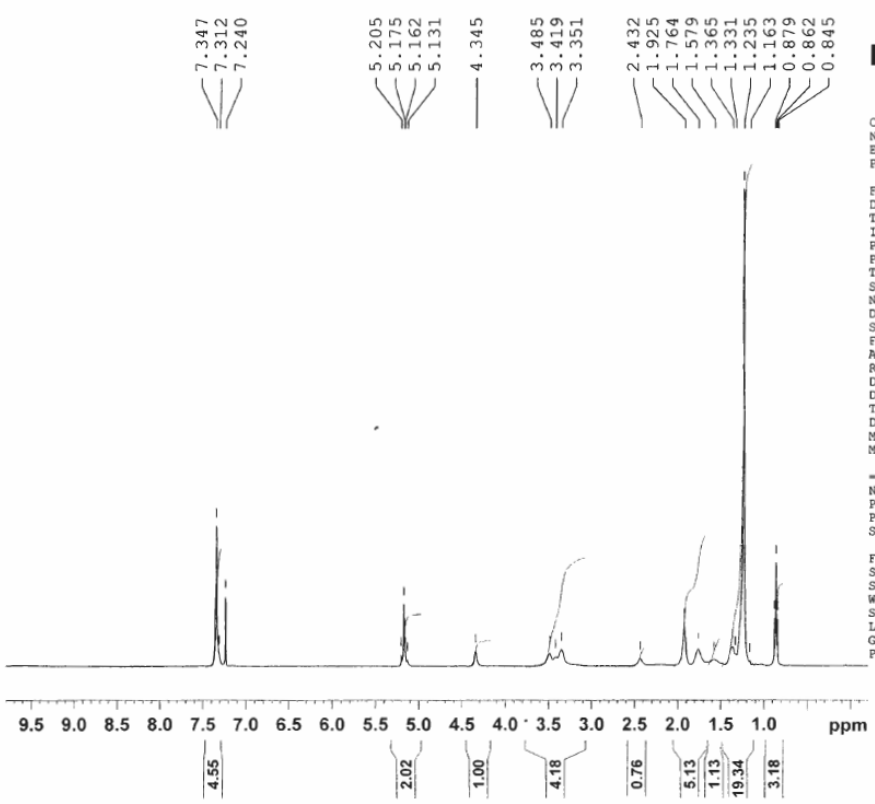

積算回数 16
ゼロフィリング ON
ゲイン 4
日時 105/12/14 22:14
測定者
ファイル名 Memory#13
サンプル名
コメント コメント

分解 4 cm-1
アポダイゼーション Cosine
スキャンスピード 2 mm/sec

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1: | 3122.19, | 26.9267 | 2 | 2923.56, | 14.8422 | 3 | 2853.17, | 24.6351 | 4: | 2728.78, | 79.4310 |
| 5 | 1616.06, | 10.7798 | 6 | 1596.77, | 10.9194 | 7 | 1563.99, | 7.1479 | 8. | 1465.63, | 47.8763 |
| 9: | 1389.46, | 21.7825 | 10 | 1276.65, | 9.3906 | 11 | 1125.26, | 2.6630 | 12: | 930.49, | 48.4796 |
| 13. | 872.63, | 25.9249 | 14 | 854.31, | 15.2961 | 15 | 589.15, | 44.1661 | 16. | 570.83, | 49.8835 |
| 17: | 558.20, | 50.4903 | 18 | 538.04, | 34.3882 | | | | | | |

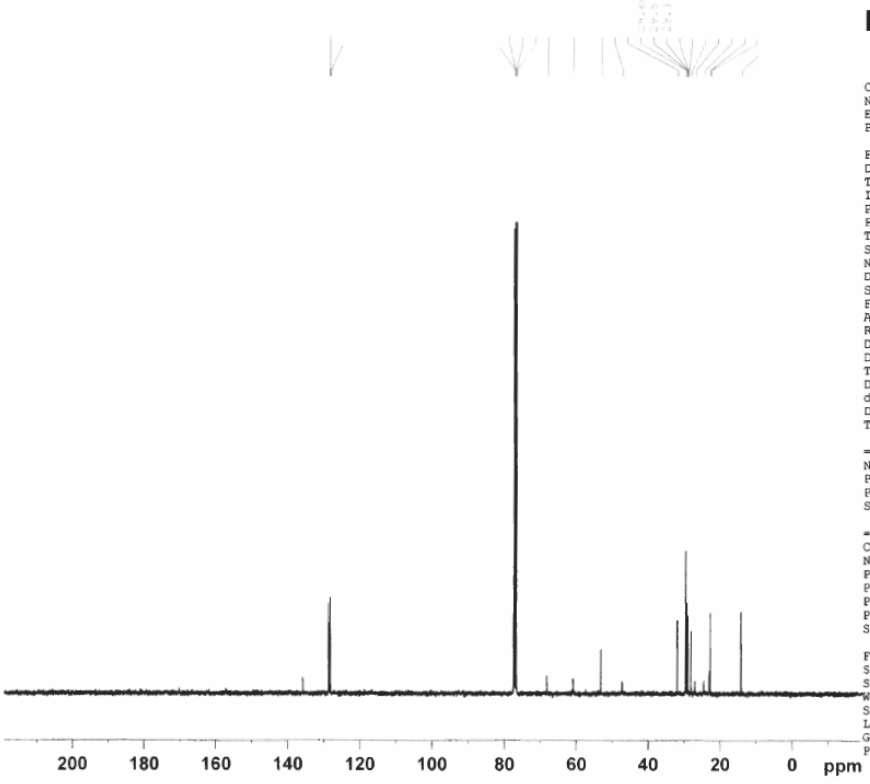






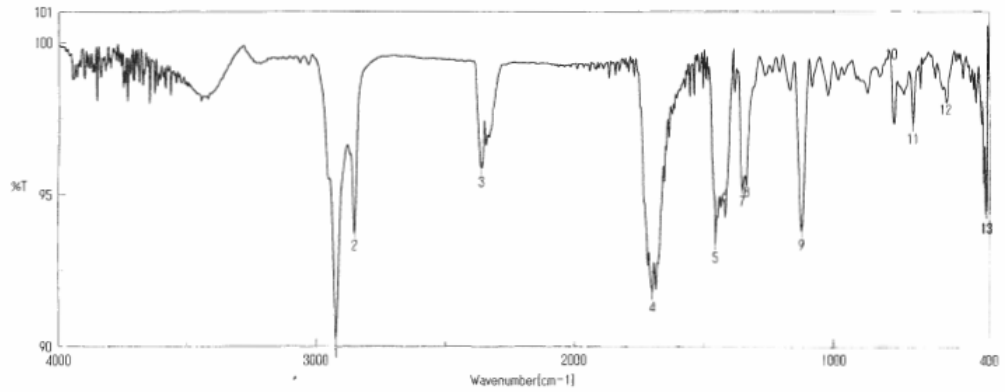

BRUKER

```
Current Data Parameters
NAME          May15-2006
EXPNO                 49
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060515
Time_              18.19
INSTRUM          dpx400
PROBHD   5 mm BBO 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9923444 sec
RG                 228.1
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                  8.10 usec
PL1                 1.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300169 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



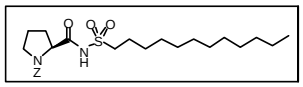

BRUKER

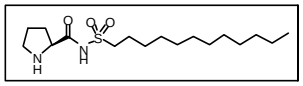
```
Current Data Parameters
NAME      May14-2006-hayashi
EXPNO                     41
PROCNO                     1

F2 - Acquisition Parameters
Date_           20060514
Time_              16.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    32
DW                20.800 usec
DE                 6.00 usec
TE                299.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.20 usec
PL1                -4.00 dB
SFO1         100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               15.00 dB
PL13               15.00 dB
SFO2         400.1816007 MHz

F2 - Processing parameters
SI                 32768
SF          100.6253427 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

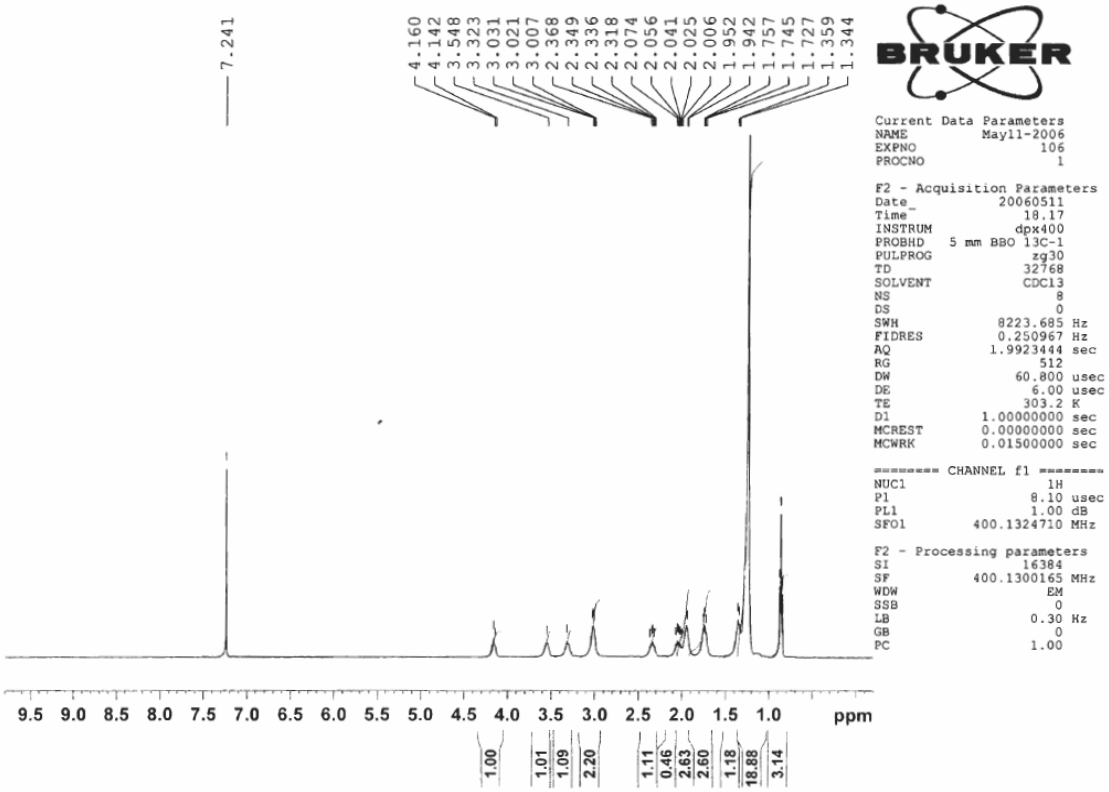



| | | | |
|---|---|---|---|
| 積算回数 | 16 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 106/05/15 17:04 | | |
| 測定者 | | | |
| ファイル名 | Memory#5 | | |
| サンプル名 | | | |
| コメント | コメント | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 2923.56, | 90.2355 | 2 | 2852.20, | 93.7818 | 3 | 2360.44, | 95.8663 | 4 | 1698.98, | 91.7625 |
| 5 | 1456.96, | 93.3915 | 6 | 1422.24, | 94.5240 | 7 | 1356.68, | 95.2308 | 8 | 1338.36, | 95.5344 |
| 9 | 1125.28, | 93.8016 | 10 | 781.03, | 99.1476 | 11 | 697.14, | 97.2661 | 12 | 571.79, | 98.2343 |
| 13 | 411.73, | 94.3371 | | | | | | | | | |

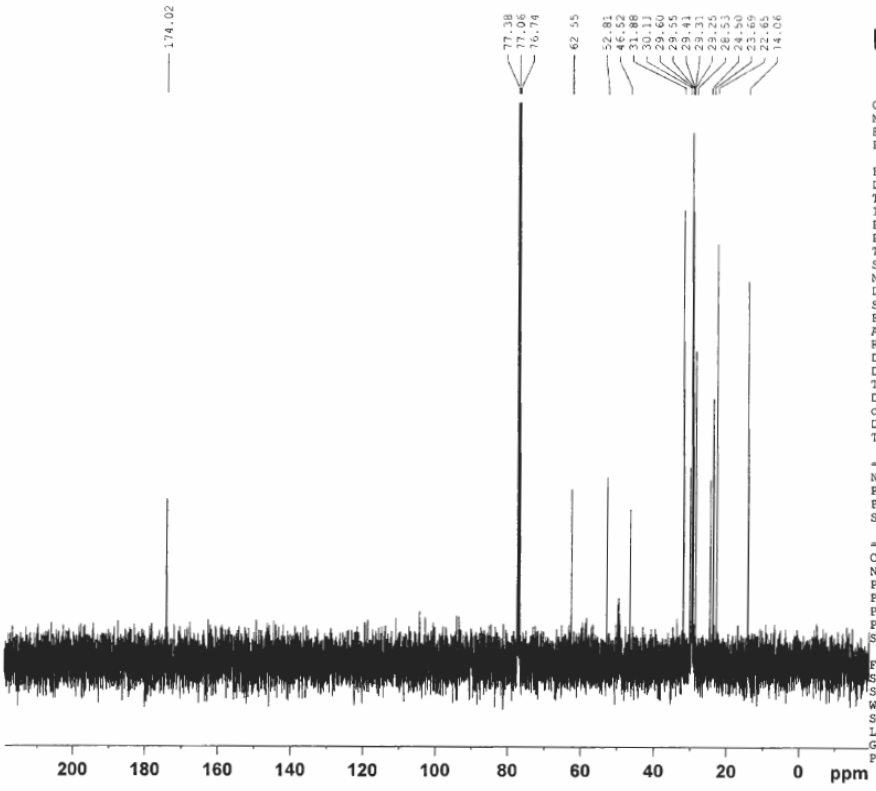






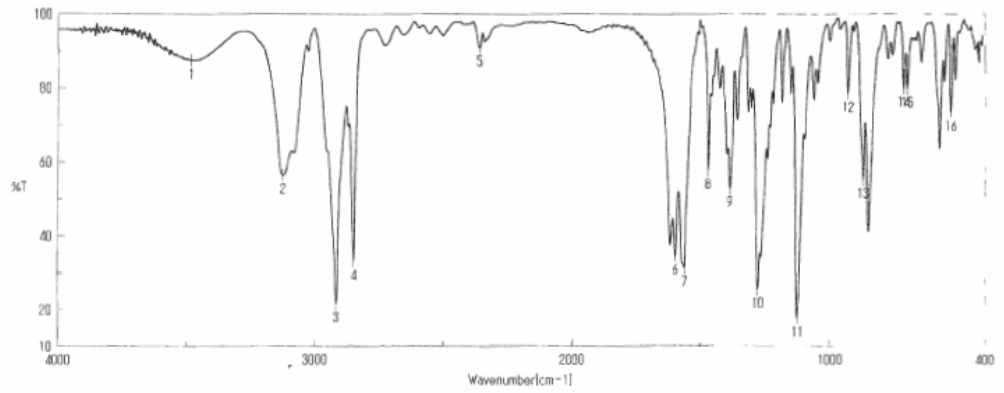

BRUKER

```
Current Data Parameters
NAME          May11-2006
EXPNO                106
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060511
Time               18.17
INSTRUM           dpx400
PROBHD   5 mm BBO 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                  512
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                8.10 usec
PL1               1.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                16384
SF          400.1300165 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```




BRUKER

```
Current Data Parameters
NAME      May10-2006-hayashi
EXPNO                 52
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060510
Time               19.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                  114
DW               20.800 usec
DE                6.00 usec
TE               298.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1               7.20 usec
PL1              -4.00 dB
SFO1       100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -4.00 dB
PL12             15.00 dB
PL13             15.00 dB
SFO2       400.1816007 MHz

F2 - Processing parameters
SI                32768
SF          100.6253410 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                 1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 64 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 2 | スキャンスピード | 2 mm/sec |
| 日時 | 106/05/10 20:45 | | |
| 測定者 | | | |
| ファイル名 | Memory#3 | | |
| サンプル名 | | | |
| コメント | コメント | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3482.81, | 87.4057 | 2 | 3124.12, | 56.4276 | 3 | 2917.77, | 21.5764 | 4 | 2850.27, | 33.0005 |
| 5 | 2360.44, | 90.9796 | 6 | 1597.73, | 34.5923 | 7 | 1562.06, | 31.3863 | 8 | 1471.42, | 57.8547 |
| 9 | 1386.57, | 53.0097 | 10 | 1278.57, | 25.4668 | 11 | 1126.22, | 17.6268 | 12 | 931.45, | 76.6234 |
| 13 | 873.60, | 55.2465 | 14 | 717.39, | 80.0190 | 15 | 702.93, | 79.8932 | 16 | 534.19, | 73.3892 |