Supporting Information

Asymmetric aldol reaction of acetaldehyde and isatin derivatives for the synthesis of convolutamydine E, CPC-1, and a 3a-hydroxyfuroindoline part of madindoline A and B

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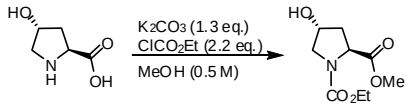
General Remarks

All reactions were carried out under argon atmosphere and monitored by thin-layer chromatography using Merck 60 F254 precoated silica gel plates (0.25 mm thickness). FT-IR spectra were recorded on a JASCO FT/IR-410 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Brucker AM400 (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR) instrument. Data for ^{1}H NMR are reported as chemical shift (δ ppm), coupling constant (Hz), integration, and assignment. Data for ^{13}C NMR are reported as chemical shift. High-resolution mass spectral analyses (HRMS) were carried out using Bruker ESI-TOF MS. Preparative thin layer chromatography was performed using Wakogel B-5F purchased from Wako Pure Chemical Industries, Tokyo, Japan. Flash chromatography was performed using silica gel 60N of Kanto Chemical Co. Int., Tokyo, Japan. GC-MS was performed on Shimazu GC-MS QP2010, equipped with a split-mode capillary injecton system and electron ionization detectors using Bodman Chiraldex Γ-TA (30 m x 0.25 mm). HPLC analysis was performed on a HITACHI Elite LaChrom Series HPLC, UV detection monitered at appropriate wavelength respectively, using Chiralcel OJ-H (0.46 cm × 25 cm).

(2*S*,4*R*)-1-Ethyl 2-methyl 4-hydroxypyrrolidine-1,2-dicarboxylate

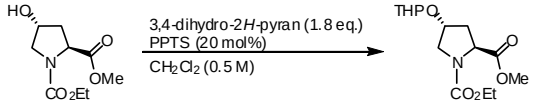


To a solution of (2*S*,4*R*)-4-hydroxypyrrolidine-2-carboxylic acid (15.1 g, 115.7 mmol) and potassium carbonate (20.7 g, 150.4 mmol) in MeOH (230 mL) was added ethyl chloroformate (24.4 mL, 254.5 mmol) at 0 °C. The reaction mixture was stirred for 12 h at room temperature. The resulting mixture was quenched with H_2O (115 mL) and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 1) gave (2*S*,4*R*)-1-Ethyl 2-methyl 4-hydroxypyrrolidine-1,2-dicarboxylate (17.5 g, 80.7 mmol) in 70% yield.

NMR spectra data was observed as a mixture of rotamer.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.15 (1.5H, t, *J* = 7.2 Hz), 1.21 (1.5H, t, *J* = 7.2 Hz), 1.96–2.06 (1H, m), 2.17-2.31 (1H, m), 2.69 (1H, br-s), 3.41–3.62 (2H, m), 3.68 (1.5H, m), 3.70 (1.5H, m), 3.95–4.15 (2H, m), 4.33–4.45 (2H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.5, 14.6, 38.5, 39.2, 52.2, 52.3, 54.6, 55.1, 57.7, 57.8, 61.5, 61.6, 69.4, 70.1, 154.9, 155.3, 173.2, 173.3;
IR (KBr): ν 3439, 2984, 2954, 1749, 1682, 1434, 1383, 1350, 1205, 1174 cm^{-1};

HRMS (ESI): $[M+Na]^+$ calculated for $[C_9H_{15}NO_5Na]$: 240.0842, found: 240.0849;
$[\alpha]_D^{23} = -75.5$ (*c* 1.36, MeOH).

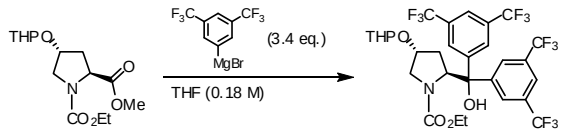
(2*S*,4*R*)-1-Ethyl 2-methyl 4-(tetrahydro-2*H*-pyran-2-yloxy)pyrrolidine-1,2-dicarboxylate



To a solution of (2*S*,4*R*)-1-Ethyl 2-methyl 4-hydroxypyrrolidine-1,2-dicarboxylate (17.5 g, 80.7 mmol) and pyridinium *p*-toluenesulfonate (4.0 g, 15.9 mmol) in methylene chloride (167 mL) was added 3,4-dihydro-2*H*-pyran (11.2 mL, 142.6 mmol) at 0 °C. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 2) gave (2*S*,4*R*)-1-ethyl 2-methyl 4-(tetrahydro-2*H*-pyran-2-yloxy)-pyrrolidine-1,2-dicarboxylate (20.2 g, 67.0 mmol) in 83% yield.

NMR spectra data was observed as a mixture of diastereomer and rotamer.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.06 (1.5H, t, *J* = 7.2 Hz), 1.13 (1.5 H, t, *J* = 7.2 Hz), 1.32–1.50 (4H, m), 1.52–1.76 (2H, m), 1.88–2.07 (1H, m), 2.11–2.38 (1H, m), 3.33–3.64 (6H, m), 3.66–3.76 (1H, m), 3.86–4.09 (2H, m), 4.20–4.39 (2H, m), 4.48–4.58 (1H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.2, 14.3, 18.9, 19.28, 19.34, 25.0, 25.1, 30.33, 30.37, 30.44, 35.1, 35.9, 36.5, 37.4, 38.1, 38.9, 51.2, 51.5, 51.76, 51.82, 51.9, 52.4, 52.7, 54.2, 54.6, 57.3, 57.4, 57.5, 57.6, 57.8, 60.99, 61.06, 61.14, 62.1, 62.2, 62.4, 62.5, 62.6, 68.7, 69.4, 73.1, 74.0, 74.1, 94.2, 97.2, 97.55, 97.63, 97.7, 154.2, 154.4, 154.89, 154.93, 172.8, 172.9, 173.0, 176.2;
IR (KBr): ν 3461, 2952, 2870, 1756, 1681, 1469, 1442, 1270, 1203, 1122, 1022 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{14}H_{23}NO_6Na]$: 324.1418, found: 324.1418;
$[\alpha]_D^{23} = -60.2$ (*c* 1.13, MeOH).

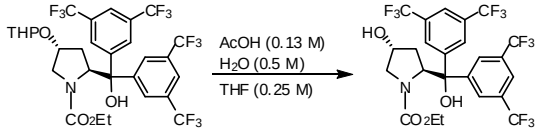
(2*S*,4*R*)-Ethyl 2-(bis(3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)-4-(tetrahydro-2*H*-pyran-2-yloxy)pyrrolidine-1-carboxylate



To a solution of (2*S*,4*R*)-1-ethyl 2-methyl 4-(tetrahydro-2*H*-pyran-2-yloxy)-pyrrolidine-1,2-dicarboxylate (7.6 g, 25.2 mmol) in tetrahydrofuran (30 mL) was added (3,5-bis(trifluoromethyl)phenyl) magnesium bromide tetrahydrofuran solution (0.75 M solution, 114 mL) at 0 °C. The reaction mixture was stirred for 4 h at room temperature. The resulting mixture was quenched with aqueous NH_4Cl solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 2) gave (2*S*,4*R*)-ethyl 2-(bis(3,5-bis(trifluoromethyl) phenyl)(hydroxy)methyl)-4-(tetrahydro-2*H*-pyran-2-yloxy)pyrrolidine-1-carboxylate (16.2 g, 23.0 mmol) in 92% yield.

NMR spectra data was observed as a mixture of diastereomer and rotamer.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.10–1.38 (3H, m), 1.14–1.58 (3H, m), 1.60–1.80 (4H, m), 1.84–1.95 (1H, m), 1.96–2.20 (1H, m), 3.36–3.46 (1H, m), 3.68–3.97 (3H, m), 3.98–4.17 (2H, m), 4.42–4.51 (1H, m), 4.92–5.06 (1H, m), 7.81–7.85 (3H, m), 7.87–7.93 (3H, m);
IR (KBr): ν 2954, 2875, 1735, 1513, 1451, 1247, 1144, 1109, 1005, 736 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{29}H_{27}NO_5F_{12}Na]$: 720.1590, found: 720.1566;
$[\alpha]_D^{23}$ = +48.3 (*c* 1.72, MeOH).

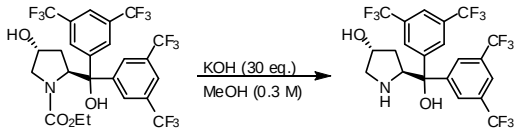
(2*S*,4*R*)-Ethyl 2-(bis(3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)-4-hydroxypyrrolidine-1-carboxylate



To a solution of (2*S*,4*R*)-ethyl 2-(bis(3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)-4-(tetrahydro-2*H*-pyran-2-yloxy)pyrrolidine-1-carboxylate (17.7 g, 25.2 mmol) in tetrahydrofuran (95 mL) and H_2O (47 mL) was added acetic acid (188 mL, 197.4 mmol) at room temperature. The reaction mixture was stirred for 3 h at 60 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 1) gave (2*S*,4*R*)-ethyl 2-(bis(3,5-bis(trifluoromethyl)phenyl) (hydroxy)methyl)-4-hydroxylpyrrolidine-1-carboxylate (14.5 g, 23.4 mmol) in 93% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.13 (3H, t, *J* = 6.8 Hz), 1.77–1.88 (1H, m), 1.90-1.99 (2H, m), 3.04 (1H, dd, *J* = 4.0, 12.8 Hz), 3.70–3.77 (1H, m), 3.91–4.14 (3H, m), 4.15–4.20 (1H, m), 5.04 (1H, t, *J* = 8.4 Hz), 7.81–7.87 (3H, m), 7.88–7.94 (3H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.6, 39.6, 56.7, 62.8, 67.4, 69.0, 80.3, 107.9 (4C), 121.7 (4C), 131.4 (4C), 144.9 (2C), 146.5 (2C), 158.4;
IR (KBr): ν 3393, 2987, 1675, 1429, 1372, 1348, 1279, 1173, 1134, 682 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{24}H_{19}NO_4F_{12}Na]$: 636.1015, found: 636.1020;
$[\alpha]_D^{22}$ = +54.5 (*c* 1.49, MeOH).

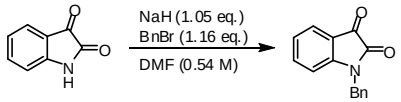
(3*R*,5*S*)-5-(Bis(3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)pyrrolidin-3-ol (6)



To a solution of (2*S*,4*R*)-ethyl 2-(bis(3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)-4-hydroxyl pyrrolidine-1-carboxylate (10.7 g, 17.3 mmol) in MeOH (57 mL) was added potassium hydroxide (29.0 g, 519 mmol) at room temperature. The reaction mixture was stirred for 3 h at 90 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 1) gave (3*R*,5*S*)-5-(bis(3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)pyrrolidin-3-ol (7.1 g, 13.1 mmol) in 88% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42–1.51 (1H, m), 1.68–1.78 (1H, m), 3.08 (1H, dd, *J* = 1.6, 10.0 Hz), 3.17 (1H, dd, *J* = 4.0, 12.0 Hz), 4.41–4.46 (1H, m), 4.72 (1H, dd, *J* = 6.0, 10.0 Hz), 7.77 (2H, d, *J* = 10.8 Hz), 7.94 (2H, s), 8.11 (2H, s);
^{13}C NMR (CD_3OD, 100 MHz): δ 37.1, 56.3, 64.6, 73.0, 78.7, 122.2, 123.4, 126.1, 127.5 (2C), 128.2 (2C), 128.9, 132.8 (2C, d, *J* = 4.0 Hz), 133.1 (2C, d, *J* = 4.0 Hz), 149.3 (2C), 150.2 (2C);
IR (KBr): ν 3365, 1372, 1278, 1174, 1131, 902, 844, 711, 682 cm^{-1};
HRMS (ESI): $[M+H]^+$ calculated for $[C_{21}H_{16}NO_2F_{12}]$: 542.0984, found: 542.0984;
$[\alpha]_D^{23}$ = +27.7 (*c* 1.64, MeOH).

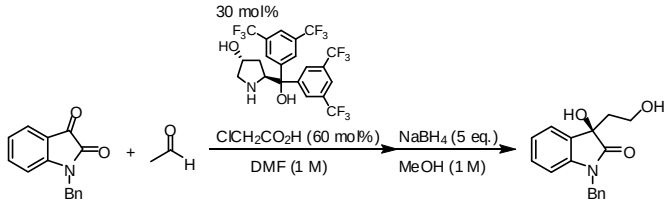
1-Benzylindoline-2,3-dione (7a)



A solution of isatin (5.0 g, 34 mmol) in DMF (62 mL) was cooled to 0 °C (ice bath). NaH (60% dispersion in mineral oil, 1.4 g, 36 mmol) was added portionwise to the orange solution. The color of solution changed to deep purple. When the gas evolution stopped, benzyl bromide (6.7 g, 39 mmol) was added slowly, whereupon the mixture turned red-brown. After the reaction mixture was stirred for 15 min at room temperature, H_2O (300 mL) was introduced to precipitate the product. After filtration, the product was washed with hexane to afford1-benzylindoline-2,3-dione (7.6 g, 95%) after drying.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.85 (2H, s), 6.70 (1H, d, *J* = 8.0 Hz), 7.01 (1H, dt, *J* = 0.4, 7.2 Hz), 7.19–7.31 (5H, m), 7.40 (1H, dt, *J* = 1.2, 8.0 Hz), 7.53 (1H, dd, *J* = 0.4, 7.2 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 44.1, 111.0, 117.7, 123.9, 125.4, 127.4 (2C), 128.2, 129.1 (2C), 134.5, 138.3, 150.8, 158.3, 183.2;
IR (KBr): ν 1731, 1613, 1471, 1349, 1177, 1078, 1004, 766, 754, 694 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{15}H_{11}NO_2Na]$: 260.0682, found: 260.0681;

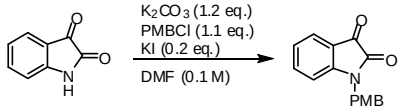
(*R*)-1-Benzyl-3-hydroxy-3-(2-hydroxyethyl)indolin-2-one (8a) (Table 1, entry 13)



To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (48 mg, 0.090 mmol), chloroacetic acid (17 mg) and 1-benzylindoline-2,3-dione (71 mg, 0.30 mmol) in DMF (0.30 mL) was added acetaldehyde (84 μL, 1.50 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 48 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (56 mg, 1.5 mmol) were added, and the mixture was stirred for 1 h at –20 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and the organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-1-benzyl-3-hydroxy-3-(2-hydroxyethyl)indolin-2-one (47 mg, 0.17 mmol) in 55% yield with 86% ee.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.01–2.10 (1H, m), 2.23–2.32 (1H, m), 3.90–4.01 (2H, m), 4.75 (1H, d, *J* = 15.6 Hz), 4.94 (1H, d, *J* = 15.6 Hz), 6.68 (1H, d, *J* = 8.0 Hz), 7.04 (1H, t, *J* = 7.6 Hz), 7.17 (1H, dt, *J* = 1.2, 7.6 Hz), 7.20–7.31 (5H, m), 7.38 (1H, dd, *J* = 0.8, 8.0 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 39.4, 43.9, 58.6, 76.1, 109.6, 123.3, 123.9, 127.2 (2C), 127.8, 128.9 (2C), 129.7, 130.7, 135.4, 142.0, 178.5;
IR (KBr): ν 3393, 2946, 1705, 1614, 1489, 1468, 1368, 1174, 1080, 753 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{17}H_{17}NO_3Na]$: 306.1101, found: 306.1106;
$[\alpha]_D^{22}$ = +23.4 (*c* 0.95, MeOH).
Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 10 : 1, λ = 254 nm), 1.0 mL / min; major enantiomer t_R = 24.3 min, minor enantiomer t_R = 19.3 min.

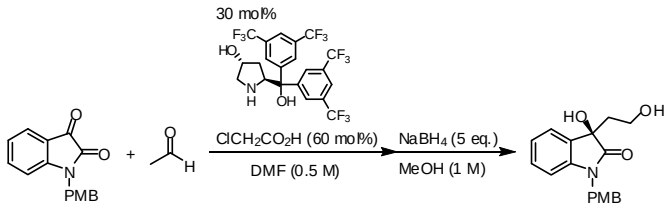
1-(4-Methoxybenzyl)indoline-2,3-dione (7c)



A solution of isatin (10.2 g, 69 mmol) in DMF (690 mL) was cooled to 0 °C (ice bath). Potassium carbonate (11.5 g, 83 mmol) and potassium iodide (2.3 g, 13.9 mmol) were added to the orange solution. The color of solution changed to deep purple. When the gas evolution stopped, *p*-methoxybenzyl chloride (11.2 mL, 76 mmol) was added slowly. The reaction mixture was stirred for 3 h at 110 °C. The reaction was quenched with aqueous 1N-HCl and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. The product was washed with hexane to afford 1-(4-methoxybenzyl)indoline-2,3-dione (17.5 g, 96%) after drying.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.77 (3H, s), 4.85 (2H, s), 6.79 (1H, d, *J* = 8.0 Hz), 6.82–6.88 (2H, m), 7.06 (1H, dt, *J* = 0.4, 7.6 Hz), 7.22-7.29 (2H, m), 7.47 (1H, dt, *J* = 1.2, 7.6 Hz), 7.57 (1H, dd, *J* = 0.8, 6.8 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 43.6, 55.3, 111.0, 114.4 (2C), 117.7, 123.8, 125.4, 126.5, 128.9 (2C), 138.3, 150.8, 158.3, 159.5, 183.4;
IR (KBr): ν 1735, 1610, 1513, 1467, 1353, 1248, 1182, 1021, 856, 762 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{16}H_{13}NO_3Na]$: 290.0788, found: 290.0782;

(*R*)-3-Hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one (8c)

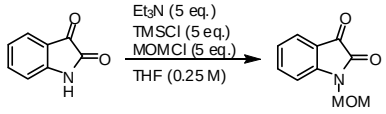


To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (48 mg, 0.090 mmol), chloroacetic acid (17 mg) and 1-(4-methoxybenzyl)indoline-2,3-dione (80 mg, 0.30 mmol) in DMF (0.60 mL) was added acetaldehyde (84 μL, 1.50 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 48 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (56 mg, 1.5 mmol) were added, and the reaction mixture was stirred for 1 h at –20 °C. The

resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-3-hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one (68 mg, 0.22 mmol) in 73% yield with 86% ee.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.03–2.12 (1H, m), 2.23–2.32 (1H, m), 3.75 (3H, s), 3.92–3.99 (2H, m), 4.71 (1H, d, *J* = 15.2 Hz), 4.88 (1H, d, *J* = 15.2 Hz), 6.73 (1H, d, *J* = 4.8 Hz), 6.79–6.86 (2H, m), 7.05 (1H, dt, *J* = 0.8, 7.6 Hz), 7.20 (3H, dt, *J* = 0.6, 7.6 Hz), 7.39 (1H, dd, *J* = 0.8, 7.2 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 39.5, 43.3, 55.3, 58.6, 76.1, 109.7, 114.2 (2C), 123.7, 123.9, 127.5, 128.6 (2C), 129.6, 130.7, 142.0, 159.2, 178.5;
IR (KBr): ν 3395, 1704, 1614, 1513, 1468, 1367, 1249, 1176, 1033, 751 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{18}H_{19}NO_4Na]$: 336.1206, found: 336.1206;
$[\alpha]_D^{22}$ = +21.5 (*c* 1.53, MeOH).
Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 10 : 1, λ = 254 nm), 1.0 mL / min; major enantiomer t_R = 29.7 min, minor enantiomer t_R = 27.9 min.

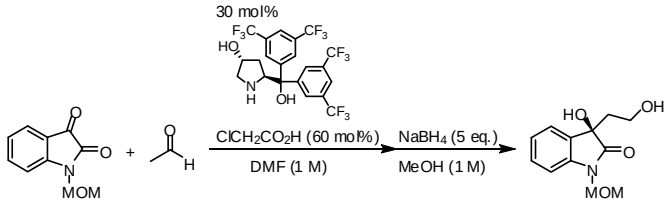
1-(Methoxymethyl)indoline-2,3-dione (7d)



A solution of isatin (1.0 g, 6.8 mmol) in THF (27 mL) was cooled to 0 °C (ice bath). Triethylamine (4.7 mL, 34 mmol) and trimethylsilyl chloride (4.3 mL, 34 mmol) were added to the orange solution. The reaction mixture was stirred for 2 h at 80 °C. After cooled the reaction mixture to 0 °C, chloro(methoxy)methane (3.0 mL, 34 mmol) was added slowly. The reaction mixture was stirred for 6 h at 80 °C, then the resulting mixture was quenched with pH 7.0 phosphate buffer solution. Organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 9) gave 1-(methoxymethyl)indoline-2,3-dione (694 mg, 3.6 mmol) in 53% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.37 (3H, s), 5.15 (2H, s), 7.11 (1H, d, *J* = 8.0 Hz), 7.18 (1H, dt, *J* = 0.8, 8.0 Hz), 7.58–7.67 (2H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 56.6, 71.7, 111.6, 117.5, 124.3, 125.4, 138.6, 150.1, 158.4, 182.8;
IR (KBr): ν 2944, 1726, 1605, 1467, 1346, 1287, 1183, 1076, 910, 755 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{10}H_9NO_3Na]$: 214.0475, found: 214.0480;

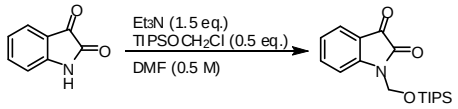
(*R*)-3-Hydroxy-3-(2-hydroxyethyl)-1-(methoxymethyl)indolin-2-one (8d)



To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (48 mg, 0.090 mmol), chloroacetic acid (17 mg) and 1-(methoxymethyl)indoline-2,3-dione (58 mg, 0.30 mmol) in DMF (60 mL) was added acetaldehyde (84 μL, 1.50 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 48 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (56 mg, 1.5 mmol) were added, and the reaction mixture was stirred for 1 h at –20 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-3-Hydroxy-3-(2-hydroxyethyl)-1-(methoxymethyl)indolin-2-one (52 mg, 0.22 mmol) in 72% yield with 86% ee.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.01–2.09 (1H, m), 2.25–2.35 (1H, m), 3.34 (3H, s), 3.90–4.03 (2H, m), 5.05 (1H, d, *J* = 14.0 Hz), 5.13 (1H, d, *J* = 14.0 Hz), 7.03 (1H, d, *J* = 6.8 Hz), 7.14 (1H, dt, *J* = 0.8, 8.4 Hz), 7.32 (1H, dt, *J* = 0.8, 8.4 Hz), 7.41 (1H, dd, *J* = 0.8, 6.8 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 39.4, 56.4, 58.6, 71.6, 76.3, 110.0, 123.8, 124.0, 129.9, 130.0, 141.2, 178.8;
IR (KBr): ν 3399, 1718, 1614, 1487, 1468, 1350, 1182, 1094, 913, 755 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{12}H_{15}NO_4Na]$: 260.0893, found: 260.0891;
$[\alpha]_D^{22}$ = +12.5 (*c* 0.44, MeOH).
Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 30 : 1, λ = 215 nm), 1.0 mL / min; major enantiomer t_R = 49.1 min, minor enantiomer t_R = 53.2 min.

1-[(Triisopropylsilyloxy)methyl]indoline-2,3-dione (7e)

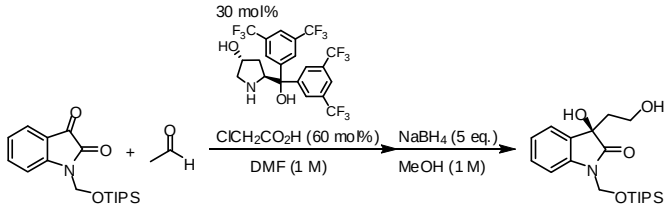


A solution of isatin (7.3 g, 49.8 mmol) in DMF (100 mL) was cooled to 0 °C (ice bath). Triethylamine (10.4 mL, 75 mmol) and (chloromethoxy)triisopropylsilane (5.6 g, 24.9 mmol) was added to the orange solution. The reaction mixture was stirred for 24 h at room temperature, then the resulting mixture was quenched with pH 7.0 phosphate buffer solution. Organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 10) gave 1-[(triisopropylsilyloxy)methyl]indoline-2,3-dione (2.1 g, 6.3 mmol) in 25% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.06 (18H, d, *J* = 6.8 Hz), 1.12–1.22 (3H, m), 5.41 (2H, s), 7.12–7.19 (2H, m), 7.62 (2H, dt, *J* = 1.6, 7.6 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.9 (3C), 17.7 (6C), 64.8, 111.8, 117.5, 124.0, 125.3, 138.4, 150.5,

157.2, 183.3;
IR (KBr): ν 2941, 2865, 1746, 1600, 1452, 1311, 1274, 1226, 1159, 1092, cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{18}H_{27}NO_3SiNa]$: 356.1652, found: 356.1660;

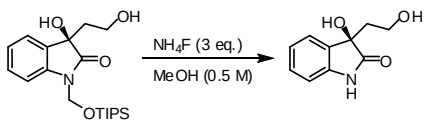
(*R*)-3-Hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (8e)



To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (71 mg, 0.13 mmol), chloroacetic acid (25 mg, 0.26) and 1-[(triisopropylsilyloxy)methyl]indoline-2,3-dione (146 mg, 0.44 mmol) in DMF (0.44 mL) was added acetaldehyde (123 μL, 2.2 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 72 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (82 mg, 2.2 mmol) were added, and the reaction mixture was stirred for 1 h at −20 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropyl silyloxy)methyl)indolin-2-one (121 mg, 0.31 mmol) in 73% yield with 85% ee.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.06 (18H, d, *J* = 8.8 Hz), 1.11–1.22 (3H, m), 1.94–2.04 (1H, m), 2.22–2.32 (1H, m), 2.60-2.75 (1H, m), 3.90-4.08 (3H, m), 5.30 (1H, d, *J* = 7.6 Hz), 5.42 (1H, d, *J* = 7.6 Hz), 7.09 (1H, d, *J* = 11.6 Hz), 7.13 (1H, dt, *J* = 0.8, 7.6 Hz), 7.33 (1H, dt, *J* = 0.8, 7.6 Hz), 7.41 (1H, dd, *J* = 11.6 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.9 (3C), 17.8 (6C), 39.2, 58.6, 64.9, 76.4, 110.3, 123.4, 123.7, 129.7, 130.2, 141.4, 177.3;
IR (KBr): ν 3392, 2944, 2866, 1718, 1615, 1469, 1364, 1279, 1092, 751 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{20}H_{33}NO_4SiNa]$: 402.2071, found: 402.2078;
$[\alpha]_D^{20}$ = +12.8 (*c* 0.78, MeOH).
Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 10 : 1, λ = 254 nm), 1.0 mL / min; major enantiomer t_R = 13.1 min, minor enantiomer t_R = 13.7 min.

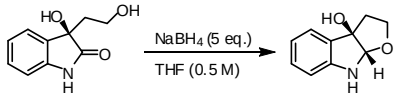
(*R*)-3-Hydroxy-3-(2-hydroxyethyl)indolin-2-one (14)



To a solution of (*R*)-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (498 mg, 1.3 mmol) in MeOH (2.6 mL) was added ammonium fluoride (147 mg, 3.9 mmol) at room temperature. The reaction mixture was stirred for 12 h at 70 °C. After that reaction mixture was concentrated in vacuo, purification by preparative thin layer chromatography (ethyl acetate) gave (*R*)-3-hydroxy-3-(2-hydroxyethyl)indolin-2-one (225 mg, 1.2 mmol) in 88% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.00–2.09 (1H, m), 2.22–2.31 (1H, m), 3.90–4.03 (2H, m), 6.86 (1H, d, *J* = 7.6 Hz), 7.08 (1H, t, *J* = 7.6 Hz), 7.25 (1H, dt, *J* = 0.8, 7.6 Hz), 7.38 (1H, d, *J* = 7.6 Hz), 7.93 (1H, br-s);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 39.2, 58.6, 76.3, 110.3, 123.3, 124.3, 129.8, 131.0, 139.9, 180.2;
IR (KBr): ν 3284, 2923, 1358, 1715, 1621, 1471, 1279, 1178, 753, 584 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{10}H_{11}NO_3Na]$: 216.0631, found: 216.0631;
$[\alpha]_D^{20}$ = +21.6 (*c* 0.23, MeOH).

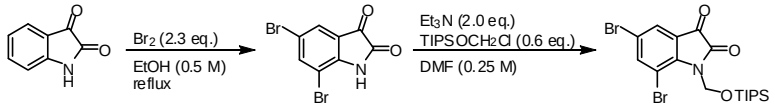
(3a*R*,8a*S*)-3,3a,8,8a-Tetrahydro-2*H*-furo[2,3-b]indol-3a-ol (15)



To a solution of (*R*)-3-hydroxy-3-(2-hydroxyethyl)indolin-2-one (118 mg, 0.61 mmol) in tetrahydrofuran (1.2 mL) was added $NaBH_4$ (116 mg, 3.1 mmol) at 0 °C. The reaction mixture was stirred for 48 h at room temperature. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (3a*R*,8a*S*)-3,3a,8,8a-tetrahydro-2*H*-furo[2,3-b] indol-3a-ol (51 mg, 0.28 mmol) in 47% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.36 (1H, ddd, *J* = 2.2, 5.4, 12.0 Hz), 2.48 (1H, ddd, *J* = 7.6, 11.0, 12.0 Hz), 3.69 (1H, ddd, *J* = 5.4, 9.0, 11.0 Hz), 4.08 (1H, ddd, *J* = 2.2, 7.6, 9.0 Hz), 4.57 (1H, br-s), 5.41 (1H, s), 6.62 (1H, d, *J* = 7.6 Hz), 6.82 (1H, t, *J* = 7.6 Hz), 7.18 (1H, dt, *J* = 1.2, 7.6 Hz), 7.33 (1H, dt, *J* = 1.5, 7.6 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 40.9, 67.4, 89.5, 99.4, 109.5, 119.5, 124.1, 130.1, 130.3, 149.6;
IR (KBr): ν 3382, 2952, 2873, 1668, 1613, 1471, 1313, 1111, 1021, 949, 747 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{10}H_{11}NO_2Na]$: 200.0682, found: 200.0681;
$[\alpha]_D^{22}$ = −114.8 (*c* 0.77, $CHCl_3$);
lit.[S1] $[\alpha]_D$ = −144 (*c* 0.84, $CHCl_3$).

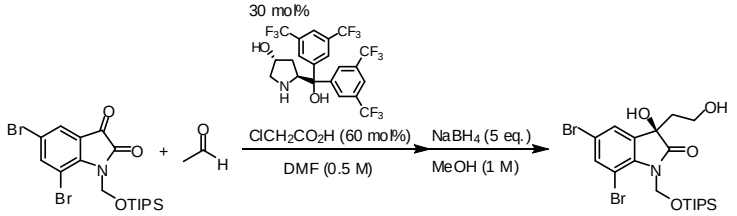
5,7-Dibromo-1-((triisopropylsilyloxy)methyl)indoline-2,3-dione (9)



5,7-Dibromoindoline-2,3-dione was prepared by known method[S2]. A solution of 5,7-dibromoindoline-2,3-dione (9.9 g, 32.5 mmol) in DMF (130 mL) was cooled to 0 °C (ice bath). Triethylamine (9.0 mL, 65 mmol) and (chloromethoxy)triisopropylsilane (4.8 g, 21.6 mmol) were added to the orange solution. The reaction mixture was stirred for 24 h at room temperature, then the resulting mixture was quenched with pH 7.0 phosphate buffer solution. Organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 10) gave 5,7-dibromo-1-((triisopropylsilyloxy)methyl)indoline-2,3-dione (4.5 g, 9.1 mmol) in 42% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.02–1.08 (18H, m), 1.10–1.22 (3H, m), 5.73 (2H, s), 7.72 (1H, d, *J* = 2.0 Hz), 7.91 (1H, d, *J* = 2.0 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.2 (3C), 17.1 (6C), 63.7, 105.3, 116.4, 120.6, 126.5, 144.4, 145.9, 156.8, 180.6;
IR (KBr): ν 2941, 2468, 1746, 1600, 1452, 1313, 1274, 1227, 1160, 1092 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{18}H_{25}Br_2NO_3SiNa]$: 513.9843, found: 513.9864;

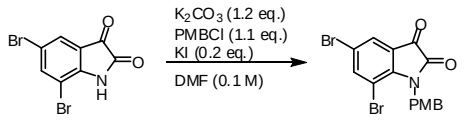
(*R*)-5,7-Dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (11)



To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (71 mg, 0.13 mmol), chloroacetic acid (25 mg, 0.26) and 5,7-dibromo-1-((triisopropylsilyloxy)methyl) indoline-2,3-dione (220 mg, 0.44 mmol) in DMF (0.89 mL) was added acetaldehyde (123 μL, 2.2 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 48 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (82 mg, 2.2 mmol) were added, and the reaction mixture was stirred for 1 h at –20 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-5,7-Dibromo-3-hydroxy-3-(2-hydroxy ethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (200 mg, 0.83 mmol) in 83% yield with 81% ee.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.06 (18H, dd, *J* = 2.0, 9.2 Hz), 1.10–1.20 (3H, m), 1.89 (1H, ddd, *J* = 4.0, 4.8, 15.2 Hz), 2.27 (1H, ddd, *J* = 4.8, 9.2, 15.2 Hz), 3.85–3.96 (1H, m), 4.06–4.18 (1H, m), 5.66 (2H, s), 7.46 (1H, d, *J* = 2.0 Hz), 7.63 (1H, d, *J* = 2.0 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.8 (3C), 17.8 (6C), 36.4, 58.3, 65.1, 77.5, 113.1, 119.8, 123.8, 126.9, 129.7, 144.4, 176.3;
IR (KBr): ν 3356, 2941, 2864, 1725, 1598, 1464, 1278, 1174, 1093, 881 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{20}H_{31}Br_2NO_4SiNa]$: 535.0389, found: 535.0394;
$[\alpha]_D^{22}$ = +14.2 (*c* 0.85, MeOH).
Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 30 : 1, λ = 298 nm), 1.0 mL / min; major enantiomer t_R = 6.2 min, minor enantiomer t_R = 7.2 min.

5,7-Dibromo-1-(4-methoxybenzyl)indoline-2,3-dione



A solution of 5,7-dibromoindoline-2,3-dione (2.3 g, 7.5 mmol) in DMF (75 mL) was cooled to 0 °C (ice bath). Potassium carbonate (1.3 g, 9.1 mmol) and potassium iodide (0.25 g, 1.5 mmol) were added to the orange solution. The color of solution changed to deep purple. When the gas evolution stopped, *p*-methoxybenzyl chloride (1.2 mL, 8.3 mmol) was added slowly. The reaction mixture was stirred for 4 h

at 110 °C. The resulting mixture was quenched with aqueous 1N-HCl and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. The product was washed with hexane to afford 5,7-dibromo-1-(4-methoxybenzyl)indoline-2,3-dione (3.0 g, 95%) after drying.

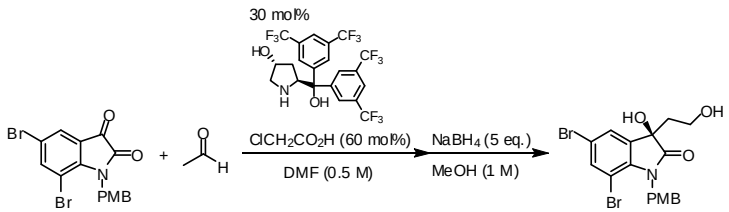
^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.77 (3H, s), 5.35 (2H, s), 6.85 (2H, d, *J* = 8.4 Hz), 7.20 (2H, d, *J* = 8.4 Hz), 7.70 (1H, d, *J* = 2.0 Hz), 7.82 (1H, d, *J* = 2.0 Hz);

^{13}C NMR ($CDCl_3$, 100 MHz): δ 44.2, 55.3, 105.2, 114.1, 114.3, 117.1, 121.5, 127.5, 127.6, 128.0, 129.2, 145.3, 146.8, 158.4, 159.3, 181.4;

IR (KBr): ν 1744, 1600, 1513, 1447, 1315, 1247, 1178, 1144, 1031, 724 cm^{-1};

HRMS (ESI): $[M+Na]^+$ calculated for $[C_{16}H_{11}Br_2NO_3Na]$: 445.8998, found: 445.8985;

(*R*)-5,7-Dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one



To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (63 mg, 0.12 mmol), chloroacetic acid (22 mg, 0.23 mmol) and 5,7-dibromo-1-(4-methoxybenzyl) indoline-2,3-dione (163 mg, 0.38 mmol) in DMF (0.77 mL) was added acetaldehyde (108 μL, 1.93 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 48 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (56 mg, 1.5 mmol) were added, and the mixture stirred for 1 h at –20 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-5,7-dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one (153 mg, 0.32 mmol) in 85% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.92–2.01 (1H, m), 2.24–2.35 (1H, m), 3.76 (3H, s), 3.88–3.96 (1H, m), 4.01–4.16 (1H, m), 5.20–5.33 (2H, m), 6.80 (2H, d, *J* = 12.0 Hz), 7.13 (2H, d, *J* = 8.4 Hz), 7.48 (1H, d, 1.6 Hz), 7.52–7.56 (1H, m);

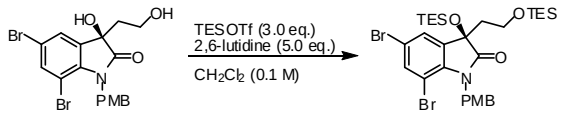
^{13}C NMR ($CDCl_3$, 100 MHz): δ 39.2, 44.0, 55.2, 58.5, 75.5, 103.4, 114.1 (2C), 116.4, 126.5, 127.7 (2C), 128.6, 135.6, 137.3, 138.8, 158.9, 178.7;

IR (KBr): ν 3393, 2362, 2330, 1716, 1513, 1452, 1247, 1177, 1144, 1033 cm^{-1};

HRMS (ESI): $[M+Na]^+$ calculated for $[C_{18}H_{17}Br_2NO_4Na]$: 491.9417, found: 491.9423;

$[\alpha]_D^{22}$ = +15.5 (*c* 1.53, MeOH).

(*R*)-5,7-Dibromo-1-(4-methoxybenzyl)-3-(triethylsilyloxy)-3-(2-(triethylsilyloxy)ethyl)indolin-2-one

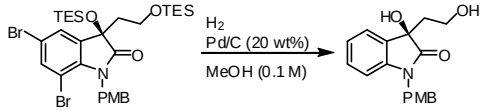


To a solution of (*R*)-5,7-dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one (238 mg, 0.51 mmol), 2,6-lutidine (380 μL, 2.55 mmol) in methylene chloride (5.1 mL) were added triethylsilyl trifluoromethanesulfonate (354 μL, 1.53 mmol). The reaction mixture was stirred for 1 h at 0 ^{o}C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution. Organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 7) gave (*R*)-5,7-dibromo-1-(4-methoxybenzyl)-3-(triethylsilyloxy)-3-(2-(triethyl silyloxy)ethyl)indolin-2-one (320 mg, 0.46 mmol) in 90% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.35–0.54 (12H, m), 0.83 (9H, t, *J* = 8.0 Hz), 0.88 (9H, t, *J* = 8.0 Hz), 2.04–2.13 (1H, m), 2.18–2.27 (1H, m), 3.54-3.67 (2H, m), 3.77 (3H, s), 5.21 (1H, d, *J* = 16.0 Hz), 5.24 (1H, d, *J* = 16.0 Hz), 6.80–6.85 (2H, m), 7.19–7.25 (1H, m), 7.36 (1H, d, *J* = 2.0 Hz), 7.36 (1H, d, *J* = 2.0 Hz), 7.54 (1H, d, *J* = 2.0 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 4.3 (3C), 5.7 (3C), 6.7 (6C), 43.1, 43.9, 55.2, 57.8, 75.9, 77.3, 102.7, 113.9 (2C), 115.6, 126.7, 128.4 (2C), 129.1, 136.8, 138.8, 158.9, 177.3;
IR (KBr): ν 2954, 2875, 1735, 1513, 1450, 1247, 1144, 1110, 1005, 736 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{30}H_{45}Br_2NO_4Si_2Na]$: 720.1146, found: 720.1120;
$[\alpha]_D^{21} = -15.3$ (*c* 1.36, MeOH).

The absolute configuration of (*R*)-5,7-Dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy) methyl)indolin-2-one (**11**) was determined after debromination of (*R*)-5,7-dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one;

(*R*)-3-Hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one

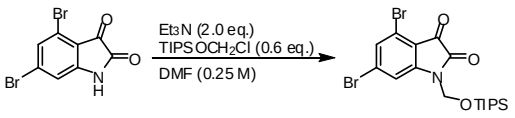


To a solution of (*R*)-5,7-dibromo-1-(4-methoxybenzyl)-3-(triethylsilyloxy)-3-(2-(triethylsilyloxy)ethyl) indolin-2-one (27.0 mg, 0.039 mmol) in MeOH (0.39 mL) was added 20% Pd/C (5.4 mg, 0.0078 mmol) at room temperature. The reaction mixture was stirred for 1h under H_2 atomosphere. Warmed MeOH (5 ml) was added, and the resulting mixture was filtered through a pad of celite, and concentrated in vacuo. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-3-Hydroxy-3-(2-hydroxyethyl)-1-(4-methoxybenzyl)indolin-2-one (10.7 mg, 0.019 mmol) in 88% yield. In this reaction, the deprotection of TES group proceeded in addition to debromination.

Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 10 : 1, λ = 254 nm), 1.0 mL / min; major enantiomer t_R = 28.2 min, minor enantiomer t_R = 26.8 min.

The absolute configuration was determined by the comparison with retention time of previous synthetic compound **7c**; see page S5.

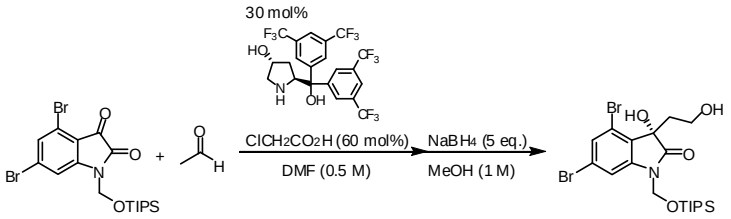
4,6-Dibromo-1-((triisopropylsilyloxy)methyl)indoline-2,3-dione (10)



4,6-Dibromoindoline-2,3-dione was prepared from *p*-nitroaniline by known method[S3]. A solution of 4,6-dibromoindoline-2,3-dione (6.1 g, 20.1 mmol) in DMF (80 mL) was cooled to 0 °C (ice bath). Triethylamine (5.6 mL, 40 mmol) and (chloromethoxy)triisopropylsilane (3.0 g, 13.4 mmol) were added to the orange solution. The reaction mixture was stirred for 24 h at room temperature, then the resulting mixture was quenched with pH 7.0 phosphate buffer solution. Organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 10) gave 4,6-dibromo-1-((triisopropylsilyloxy)methyl)indoline-2,3-dione (1.6 g, 3.2 mmol) in 24% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.04–1.09 (18H, m), 1.01–1.23 (3H, m), 5.40 (2H, s), 7.31 (1H, d, *J* = 1.6 Hz), 7.49 (1H, d, *J* = 1.6 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.2 (3C), 17.1 (6C), 63.7, 105.3, 116.4, 120.6, 126.5, 144.4, 145.9, 156.8, 180.6;
IR (KBr): ν 2942, 2865, 1745, 1591, 1393, 1321, 1239, 1096, 883, 689 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{18}H_{25}Br_2NO_3SiNa]$: 513.9843, found: 513.9861;

(*S*)-4,6-Dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (12)

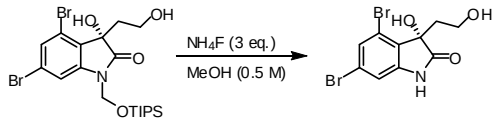


To a solution of (2*S*,4*R*)-4-hydroxy-2-(bis-[3,5-bis(trifluoromethyl)phenyl]hydroxymethyl)pyrrolidine (71 mg, 0.13 mmol), chloroacetic acid (25 mg, 0.26) and 4,6-dibromo-1-((triisopropylsilyloxy)methyl) indoline-2,3-dione (220 mg, 0.44 mmol) in DMF (0.89 mL) was added acetaldehyde (123 μL, 2.2 mmol) in the sealed tube (ACE GLASS, product number 5027-05) at 4 °C. After the reaction mixture was stirred for 48 h at 4 °C, MeOH (0.5 mL) and $NaBH_4$ (82 mg, 2.2 mmol) were added, and the reaction mixture was stirred for 1 h at –20 °C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*S*)-4,6-dibromo-3-hydroxy-3-(2-hydroxy ethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (207 mg, 0.86 mmol) in 86% yield with 82% ee.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.03–1.09 (18H, m), 1.10–1.20 (3H, m), 2.01–2.14 (1H, m), 2.75–2.86 (1H, m), 3.79–4.00 (3H, m), 5.30 (1H, d, *J* = 9.6 Hz), 5.35 (1H, d, *J* = 9.6 Hz), 7.20 (1H, d, *J* = 0.8 Hz), 7.40 (1H, d, *J* = 0.8 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.8 (3C), 17.8 (6C), 36.4, 58.3, 65.1, 77.5, 113.1, 119.8, 123.8, 126.9, 129.7, 144.4, 176.3;
IR (KBr): ν 3356, 2941, 2864, 1725, 1598, 1464, 1278, 1174, 1093, 881 cm^{-1};

HRMS (ESI): $[M+Na]^+$ calculated for $[C_{20}H_{31}Br_2NO_4SiNa]$: 535.0389, found: 535.0391;
$[\alpha]_D^{17} = -11.2$ (*c* 0.76, MeOH).
Enantiometric excess was determined by HPLC with a Chiralcel OJ-H column (hexane : 2-propanol = 50 : 1, λ = 222 nm), 0.5 mL / min; major enantiomer t_R = 22.9 min, minor enantiomer t_R = 21.1 min.

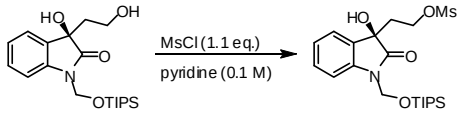
<u>*ent*-Convolutamydine E (13)</u>



To a solution of (*S*)-4,6-dibromo-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (40 mg, 0.082 mmol) in MeOH (0.16 mL) was added ammonium fluoride (9.1 mg, 0.25 mmol) at room temperature. The reaction mixture was stirred for 12 h at 70 °C. After that reaction was concentrated in vacuo, purification by preparative thin layer chromatography (ethyl acetate) gave *ent*-Convolutamydine E (19.0 mg, 0.062 mmol) in 76% yield.

^{1}H NMR (pyridine-d_5, 400 MHz): δ 3.20 (2H, t, *J* = 6.8 Hz), 3.62 (1H, s), 4.00–4.04 (2H, m), 7.12 (1H, s), 7.49 (1H, s);
^{13}C NMR (pyridine-d_5, 100 MHz): δ 39.2, 58.0, 77.4, 112.5, 120.8, 128.1, 130.1, 130.1, 146.7, 180.6;
IR (KBr): ν 3275, 2923, 2380, 2348, 1729, 1607, 1574, 1424, 1168, 1082 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{10}H_9Br_2NO_3Na]$: 371.8841, found: 371.8832;
$[\alpha]_D^{20} = -7.8$ (*c* 0.67, MeOH, *ent*-Convolutamydine E).
lit.[S4] $[\alpha]_D = +12.6$ (*c* 1.00, MeOH, Convolutamydine E).

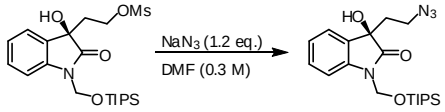
<u>(*R*)-2-(3-Hydroxy-2-oxo-1-((triisopropylsilyloxy)methyl)indolin-3-yl)ethyl methanesulfonate (16)</u>



To a solution of (*R*)-3-hydroxy-3-(2-hydroxyethyl)-1-((triisopropylsilyloxy)methyl)indolin-2-one (492 mg, 1.30 mmol) in pyridine (13 mL) was added methanesulfonyl chloride (111 μL, 1.43 mmol) at 0 °C. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was quenched with aqueous 1N-HCl and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 3) gave (*R*)-2-(3-hydroxy-2-oxo-1-((triisopropylsilyloxy)methyl)indolin-3-yl)ethyl methanesulfonate (445 mg, 0.97 mmol) in 75% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.03–1.09 (18H, m), 1.11–1.24 (3H, m), 2.33 (1H, dt, *J* = 6.4, 14.4 Hz), 2.41–2.51 (1H, m), 2.90 (3H, s), 4.32–4.38 (2H, m), 5.36 (2H, d, *J* = 1.2 Hz), 7.09–7.18 (2H, m), 7.33–7.43 (2H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.9 (3C), 17.8 (6C), 37.2, 37.3, 65.0, 74.8, 77.3, 110.6, 123.6, 124.0, 128.7, 130.3, 141.8, 176.1;
IR (KBr): ν 3437, 2944, 2866, 1730, 1615, 1469, 1359, 1175, 1094, 752 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{21}H_{35}NSO_6SiNa]$: 480.1847, found: 480.1835;
$[\alpha]_D^{21} = +7.8$ (*c* 0.80, MeOH).

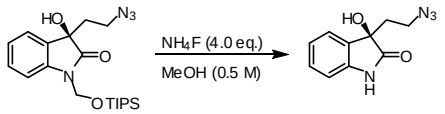
(*R*)-3-(2-Azidoethyl)-3-hydroxy-1-((triisopropylsilyloxy)methyl)indolin-2-one (17)



To a solution of (*R*)-2-(3-hydroxy-2-oxo-1-((triisopropylsilyloxy)methyl)indolin-3-yl)ethyl methane sulfonate (572 mg, 1.25 mmol) in DMF (4.2 mL) was added sodium azide (98 mg, 1.50 mmol) at room temperature. The reaction mixture was stirred for 6 h at 60 ^{o}C. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 3) gave (*R*)-2-(3-hydroxy-2-oxo-1-((triisopropylsilyloxy)methyl)indolin-3-yl)ethyl methanesulfonate (419 mg, 1.04 mmol) in 83% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.06 (18H, dd, *J* = 2.4, 7.2 Hz), 1.10–1.23 (3H, m), 2.09–2.20 (1H, m), 2.28 (1H, dt, *J* = 8.0, 13.6 Hz), 3.34–3.45 (2H, m), 5.31 (1H, d, *J* = 9.6 Hz), 5.41 (1H, d, *J* = 9.6 Hz), 7.08–7.16 (2H, m), 7.32–7.41 (2H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.9 (3C), 17.8 (6C), 37.0, 46.2, 64.9, 75.2, 110.5, 123.4, 123.8, 128.9, 130.1, 141.7, 176.3;
IR (KBr): ν 3402, 2943, 2866, 2097, 1715, 1616, 1469, 1366, 1092, 751 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{20}H_{32}N_4O_3SiNa]$: 427.2136, found: 427.2133;
$[\alpha]_D^{20}$ = +12.5 (*c* 1.20, MeOH).

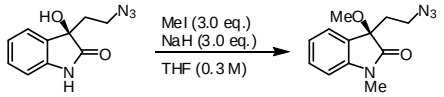
(*R*)-3-(2-Azidoethyl)-3-hydroxyindolin-2-one (18)



To a solution of (*R*)-3-(2-azidoethyl)-3-hydroxy-1-((triisopropylsilyloxy)methyl)indolin-2-one (390 mg, 0.97 mmol) in MeOH (2.0 mL) was added ammonium fluoride (144 mg, 3.8 mmol) at room temperature. The reaction mixture was stirred for 3 h at 70 ^{o}C. The resulting mixture was concentrated in vacuo. Purification by preparative thin layer chromatography (ethyl acetate : hexane = 1:1) gave (*R*)-3-(2-azidoethyl)-3-hydroxylindolin-2-one (207 mg, 0.97 mmol) in 98% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.14–2.23 (1H, m), 2.28 (1H, dt, *J* = 7.6, 14.0 Hz), 3.41 (2H, dt, *J* = 2.4, 7.6 Hz), 6.89 (1H, d, *J* = 8.0 Hz), 7.18 (1H, dt, *J* = 0.8, 7.6 Hz), 7.29 (1H, dt, *J* = 1.6, 8.0 Hz), 7.36 (1H, d, *J* = 7.6 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 36.9, 46.3, 75.3, 110.4, 123.4, 124.4, 129.6, 130.1, 140.1, 178.9;
IR (KBr): ν 3313, 2358, 2098, 1706, 1471, 1333, 1264, 1212, 1183, 753 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{10}H_{10}N_4O_2Na]$: 241.0696, found: 241.0692;
$[\alpha]_D^{24}$ = +2.8 (*c* 0.31, MeOH).

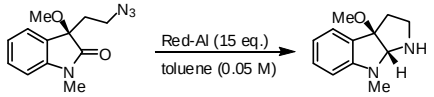
(*R*)-3-(2-Azidoethyl)-3-methoxy-1-methylindolin-2-one (19)



To a solution of (*R*)-3-(2-azidoethyl)-3-hydroxyindolin-2-one (218 mg, 0.99 mmol) in THF (3.3 mL) was cooled to 0 °C (ice bath). Then NaH (60% dispersion in mineral oil, 129 mg, 2.97 mmol) was added portionwise. When the gas evolution stopped, methyl iodide (0.18 mL, 2.97 mmol) was added slowly. The reaction mixture was stirred for 4 h at room temperature. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by column chromatography (ethyl acetate : hexane = 1 : 3) gave (*R*)-3-(2-azidoethyl)-3-methoxy-1-methylindolin-2-one (207 mg, 0.84 mmol) in 85% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.06-2.16 (1H, m), 2.20–2.31 (1H, m), 2.99 (3H, s), 3.22 (3H, s), 3.27–3.45 (2H, m), 6.87 (1H, d, *J* = 8.0 Hz), 7.11–7.16 (1H, m), 7.29 (1H, d, *J* = 7.2 Hz), 7.34–7.41 (1H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.1, 36.6, 45.9, 52.8, 80.9, 108.5, 123.1, 124.1, 126.3, 130.2, 143.9, 175.3;
IR (KBr): ν 2934, 2098, 1725, 1613, 1492, 1469, 1372, 1348, 1105, 754 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{12}H_{14}N_4O_2Na]$: 269.1009, found: 269.1005;
$[\alpha]_D^{23} = -18.8$ (*c* 1.20, MeOH).

(3a*R*,8a*R*)-3a-Methoxy-8-methyl-1,2,3,3a,8,8a-hexahydropyrrolo[2,3-b]indole (20)

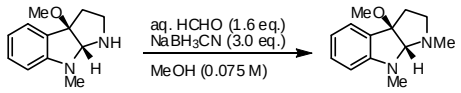


To a solution of (*R*)-3-(2-azidoethyl)-3-methoxy-1-methylindolin-2-one (14 mg, 0.058 mmol) in toluene (1.2 mL) was added sodium bis(2-methoxyethoxy)aluminum hydride (65 wt% in toluene; 0.26 mL, 0.87 mmol) at 0 °C. The reaction mixture was stirred for 1.5 h at room temperature. After that the solution was heated to 80 °C and maintained at 80 °C for 8 h. After cooling to room temperature, the resulting mixture was quenched with saturated aqueous sodium potassium tartrate (7 mL), diluted with ethyl acetate (5 mL), and stirred vigorously for 45 min. Organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (methanol : chloroform = 1 : 10) gave (3a*R*,8a*R*)-3a-methoxy-8-methyl-1,2,3,3a,8,8a-hexahydropyrrolo[2,3-b]indole (7.2 mg, 0.035 mmol) in 61% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.17–2.23 (2H, m), 2.75 (1H, dt, *J* = 8.4, 11.2 Hz), 2.86 (3H, s), 3.09 (3H, s), 3.11–3.17 (1H, m), 4.78 (1H, s), 6.41 (1H, d, *J* = 8.0 Hz), 6.69 (1H, dt, *J* = 0.8, 7.6 Hz), 7.14–7.21 (2H, m);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 31.8, 41.9, 45.4, 52.9, 86.4, 94.7, 106.0, 117.1, 124.4, 126.7, 129.9, 152.3;
IR (KBr): ν 2937, 2360, 2341, 1609, 1490, 1294, 1099, 938, 743 cm^{-1};

HRMS (ESI): $[M+Na]^+$ calculated for $[C_{12}H_{16}N_2ONa]$: 227.1155, found: 227.1164;
$[\alpha]_D^{19} = -79.1$ (*c* 0.64, MeOH).

CPC-1 (21)



To a solution of (3a*R*,8a*R*)-3a-methoxy-8-methyl-1,2,3,3a,8,8a-hexahydropyrrolo[2,3-b]indole (37 mg, 0.18 mmol) in MeOH (2.4 mL) was added formaldehyde (37% water solution, 23 μL, 0.29 mmol) at 0 °C. The reaction mixture was stirred for 1 h at room temperature. Then, $NaBH_3CN$ (34 mg, 0.54 mmol) was added and the reaction mixture was stirred for 1 h at room temperature. The resulting mixture was quenched with pH 7.0 phosphate buffer solution and organic materials were extracted with ethyl acetate three times, and then combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by preparative thin layer chromatography (methanol : chloroform = 1 : 10) gave CPC-1 (27 mg, 0.12 mmol) in 67% yield.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.13 (1H, ddd, *J* = 4.5, 6.0, 12.3 Hz), 2.35 (1H, ddd, *J* = 6.8, 8.3, 12.3 Hz), 2.58 (3H, s), 2.62 (1H, dt, *J* = 6.0, 8.8 Hz), 2.80 (1H, ddd, *J* = 4.4, 6.8, 9.2 Hz), 2.97 (3H, s), 3.04 (3H, s), 4.36 (1H, s), 6.51 (1H, d, *J* = 7.6 H), 6.75 (1H, dt, *J* = 1.2, 7.6 Hz), 7.16 (1H, dd, *J* = 1.2, 7.6 Hz), 7.20 (1H, dt, *J* = 1.2, 7.6 Hz);
^{13}C NMR ($CDCl_3$, 100 MHz): 36.2, 38.6, 39.3, 52.4, 52.5, 91.7, 94.1, 107.8, 117.9, 124.1, 128.1, 129.7, 153.1
IR (KBr): ν 2938, 2791, 1608, 1489, 1162, 1098, 1041, 933, 742 cm^{-1};
HRMS (ESI): $[M+Na]^+$ calculated for $[C_{13}H_{18}N_2ONa]$: 241.1311, found: 241.1302;
$[\alpha]_D^{19} = -83.0$ (*c* 0.73, MeOH).
lit.[S5] $[\alpha]_D^{26} = -88$ (*c* 0.1, MeOH).

References;

S1) T. Sunazuka, T. Hirose, T. Shirahata, Y. Harigaya, M. Hayashi, K. Komiyama, S. Omura, A. B. Smith III, *J. Am. Chem. Soc*. **2000**, *122*, 2122.
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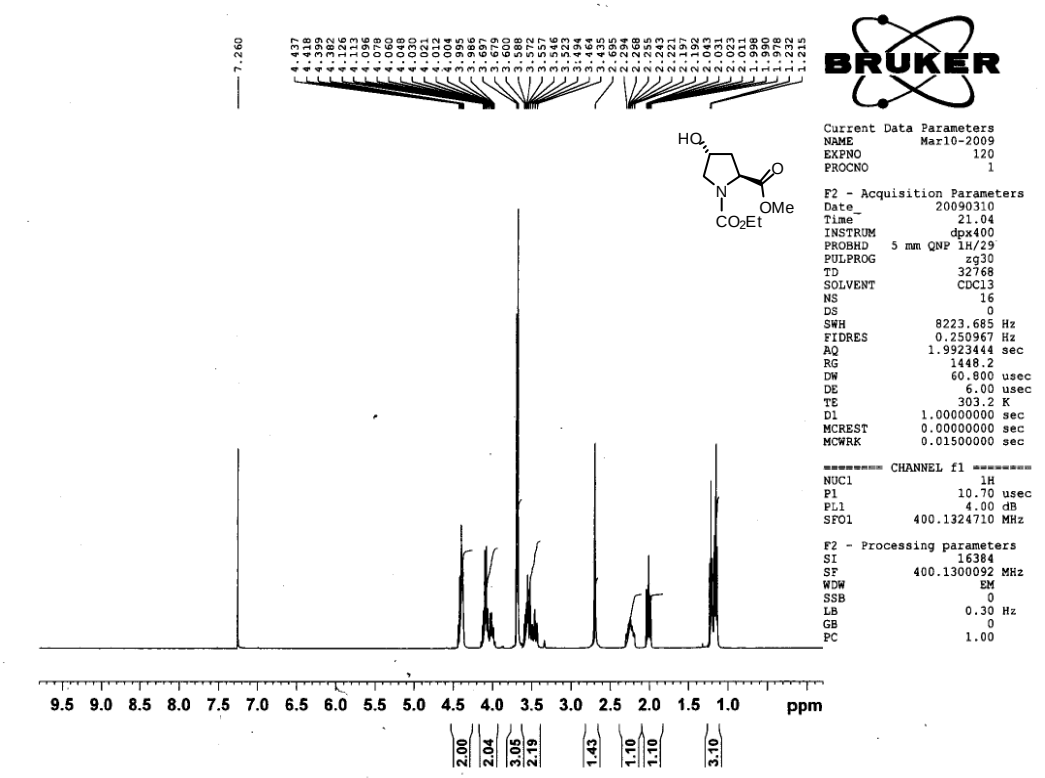
S3) S. J. Garden, J. C. Torres, A. A. Ferreira, R. B. Silva, A. C. Pinto, *Tetrahedron Lett*. **1997**, *38*, 1501.
S4) T. Nakamura, S. Shirokawa, S. Hosokawa, A. Nakazaki, S. Kobayashi, *Org. Lett*. **2006**, *8*, 677.
S5) M. Kitajima, I. Mori, K. Arai, N. Kogure, H. Takayama, *Tetrahedron Lett*. **2006**, *47*, 3199.

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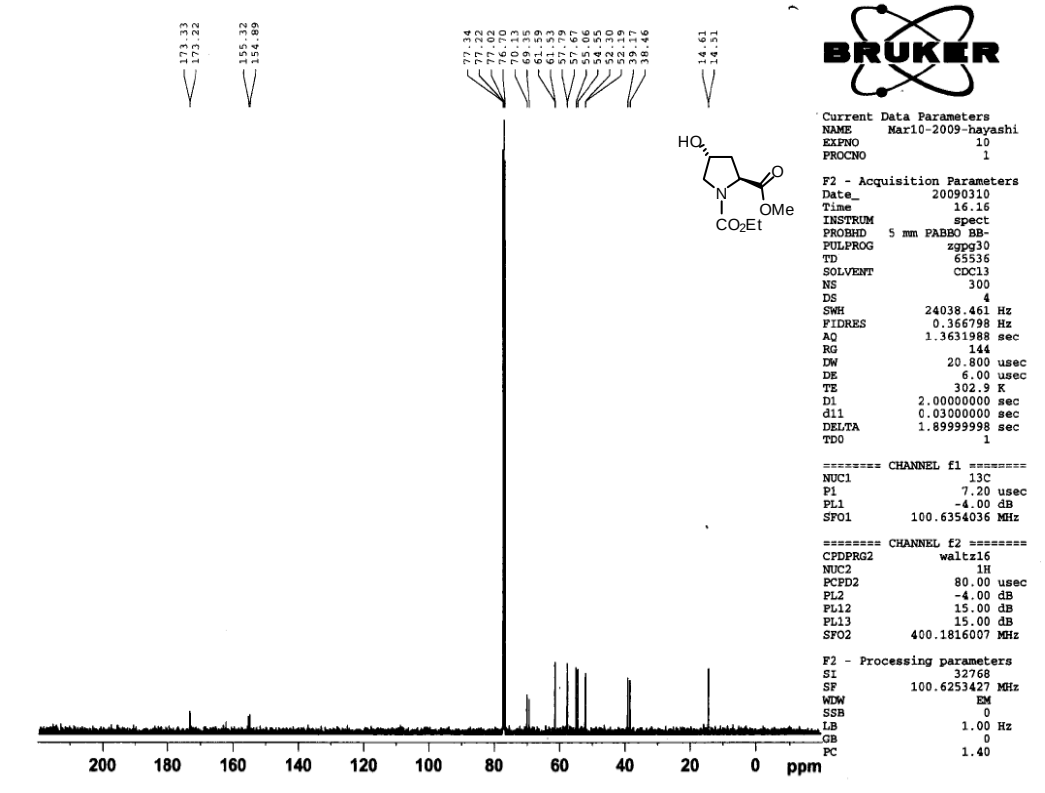


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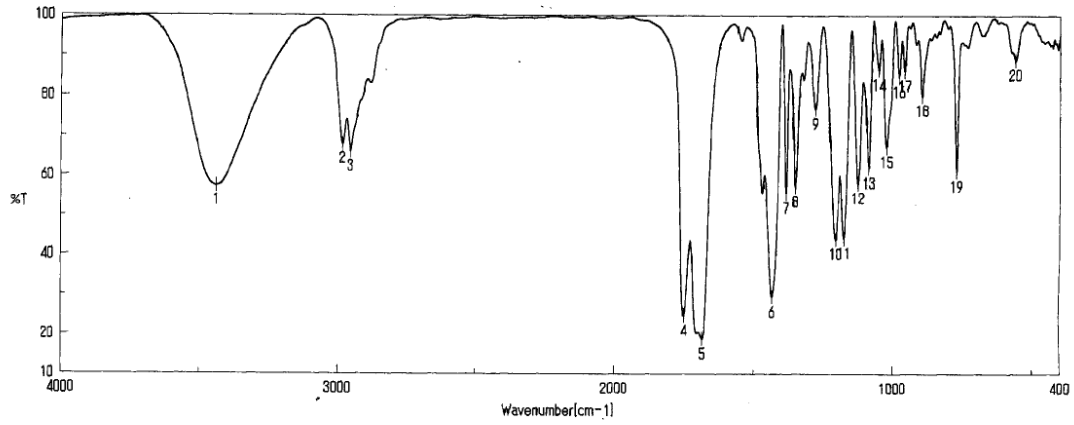
DS                    4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631988 sec
RG                  144
DW              20.800 usec
DE               6.00 usec
TE              302.9 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

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

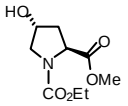
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1816007 MHz

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



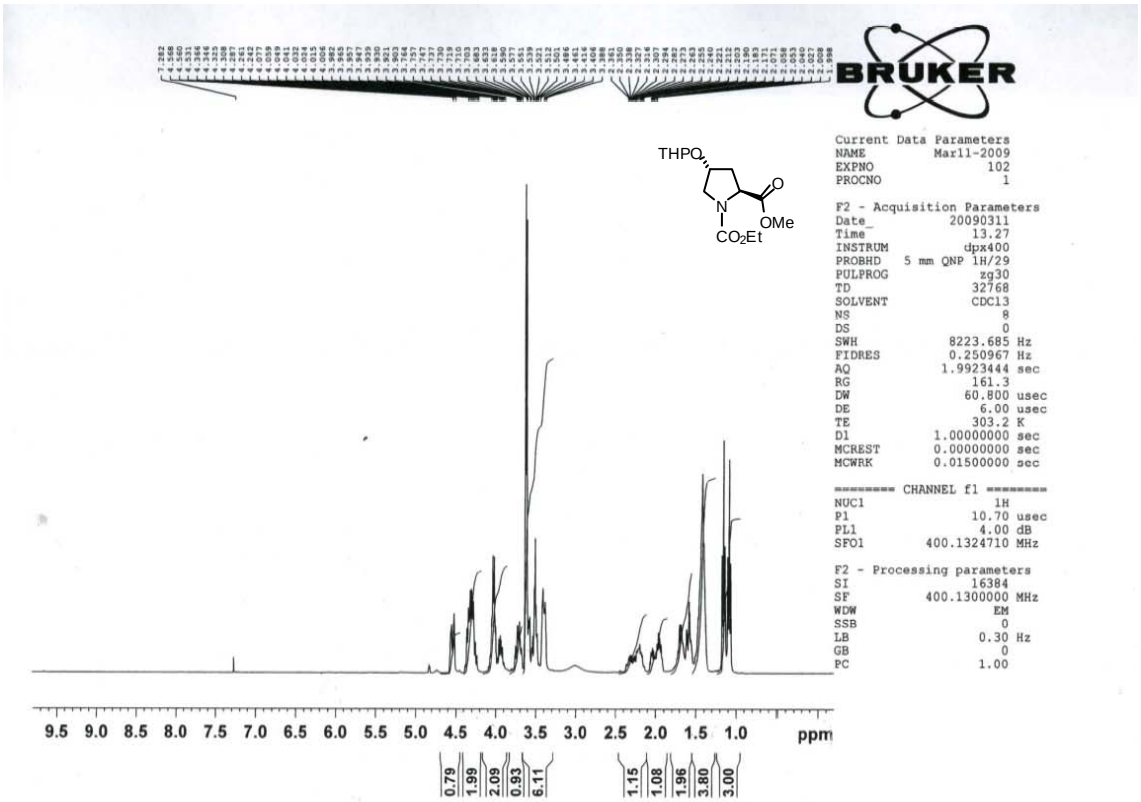
積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/10 18:40
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

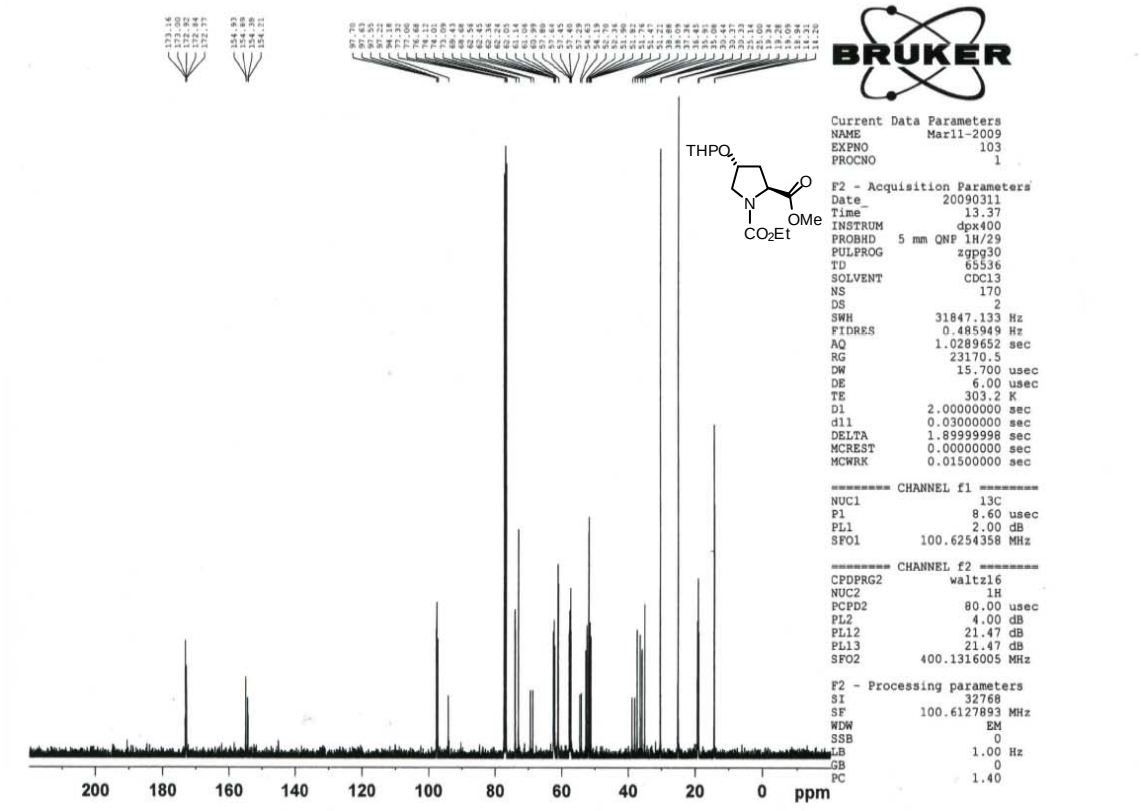
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec

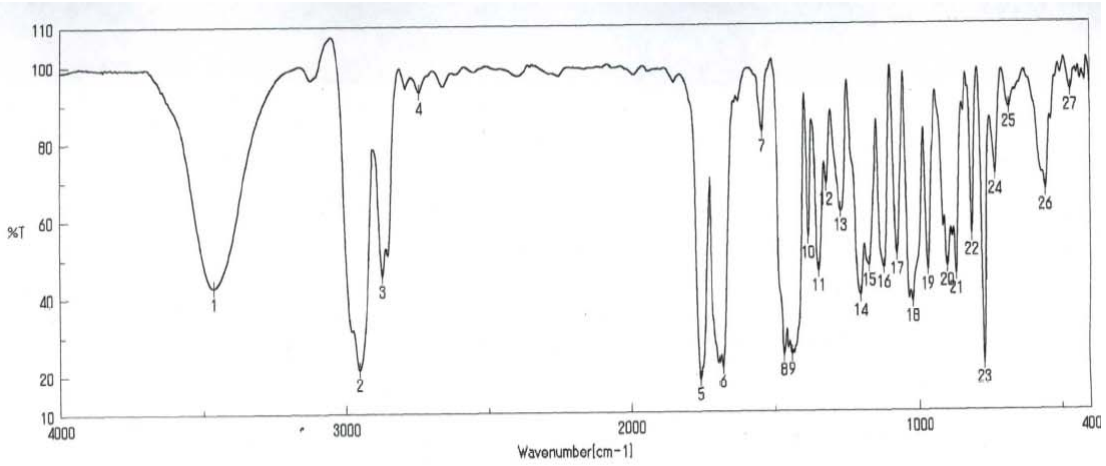


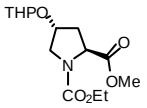
1:	3439.42,	57.3560	2:	2984.30,	67.8789	3:	2954.41,	65.9904	4:	1749.12,	24.7730
5:	1682.59,	18.8285	6:	1434.78,	29.3656	7:	1383.68,	55.5817	8:	1350.89,	57.1559
9:	1277.61,	76.6739	10:	1205.29,	43.9232	11:	1174.44,	44.1738	12:	1126.22,	58.0948
13:	1086.69,	81.8590	14:	1050.05,	86.5472	15:	1024.02,	67.1091	16:	978.70,	84.7850
17:	957.48,	86.1910	18:	896.74,	80.0592	19:	773.32,	61.0680	20:	562.15,	88.9576

積算回数＝the number of accumulation	分解＝resolution
ゼロフィリング＝zero filling	アポダイゼーション＝apodization
ゲイン＝gain	スキャンスピード＝scan speed
日時＝date	
測定者＝user name	
ファイル名＝file name	
サンプル名＝sample name	
コメント＝comment	


BRUKER
THPO
N
O
OMe
CO2Et
Current Data Parameters
NAME Mar11-2009
EXPNO 102
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090311
Time 13.27
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 161.3
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 10.70 usec
PL1 4.00 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
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
0.79 1.99 2.09 0.93 6.11 1.15 1.08 1.96 3.80 3.00


BRUKER
THPO
N
O
OMe
CO2Et
Current Data Parameters
NAME Mar11-2009
EXPNO 103
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090311
Time 13.37
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 170
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 23170.5
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 8.60 usec
PL1 2.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 21.47 dB
PL13 21.47 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127893 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



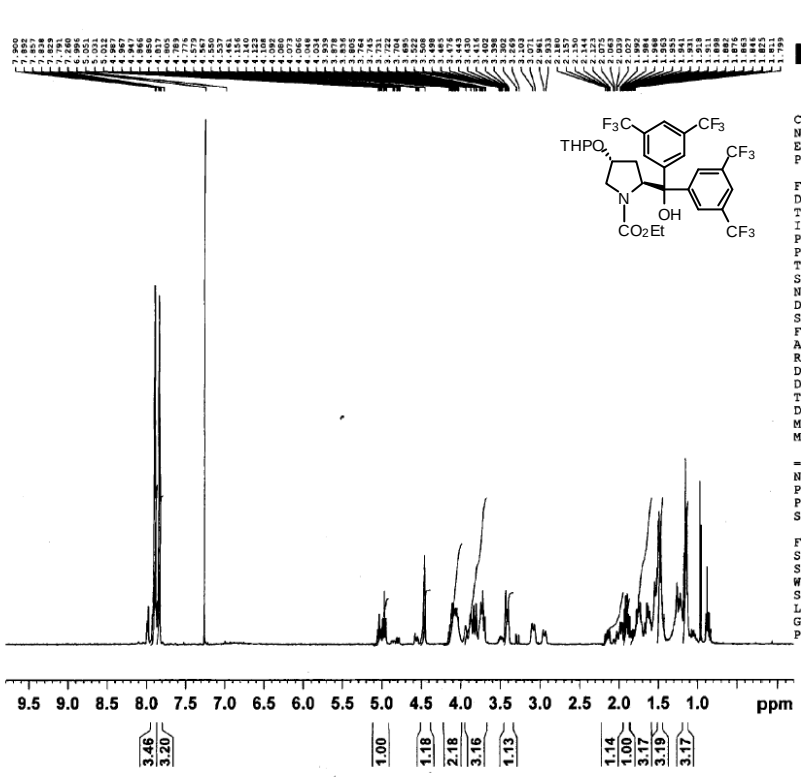


積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/11 19:48
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

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:	3461.60, 42.8242	2:	2952.48, 21.6119	3:	2870.52, 45.6707	4:	2741.32, 93.0954
5:	1756.83, 18.5669	6:	1681.62, 21.7069	7:	1543.74, 82.5993	8:	1469.49, 24.7528
9:	1442.49, 24.9478	10:	1383.68, 55.1146	11:	1347.03, 46.4233	12:	1321.00, 68.6932
13:	1270.86, 62.0144	14:	1203.35, 40.0492	15:	1173.47, 47.6789	16:	1122.37, 47.2491
17:	1077.05, 50.6431	18:	1022.09, 38.4555	19:	969.05, 46.7079	20:	900.59, 47.5202

21:	869.74, 45.5165	22:	813.81, 55.4627	23:	772.35, 22.4313	24:	733.78, 71.2444
25:	684.61, 88.4793	26:	557.33, 67.0409	27:	469.58, 92.8297		

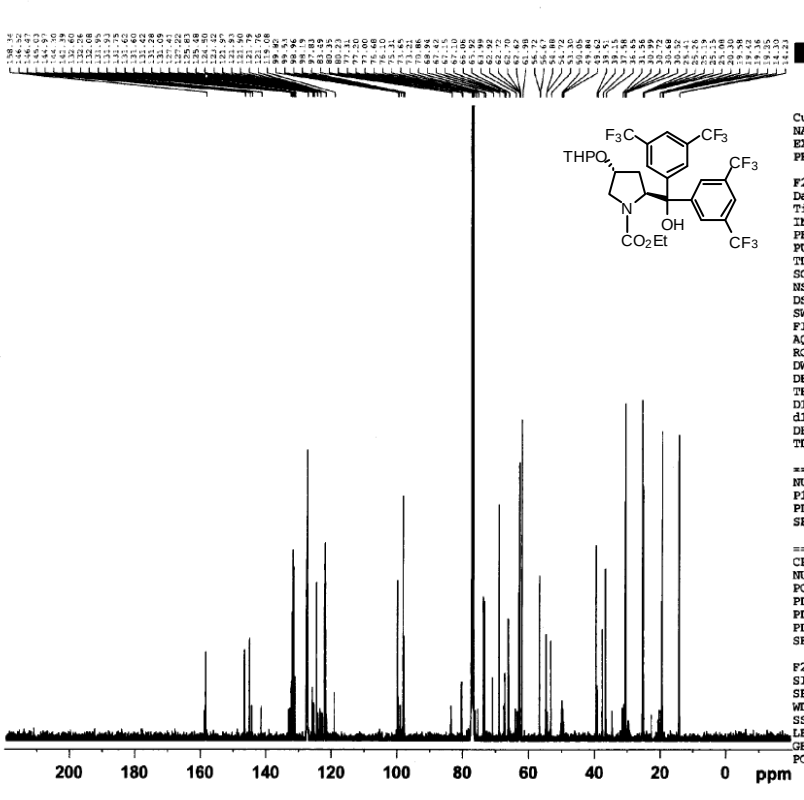



```
Current Data Parameters
NAME          Mar11-2009
EXPNO                 34
PROCNO                 1

F2 - Acquisition Parameters
Date_          20090311
Time               2.49
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                 3251
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                10.70 usec
PL1               4.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                16384
SF          400.1300092 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



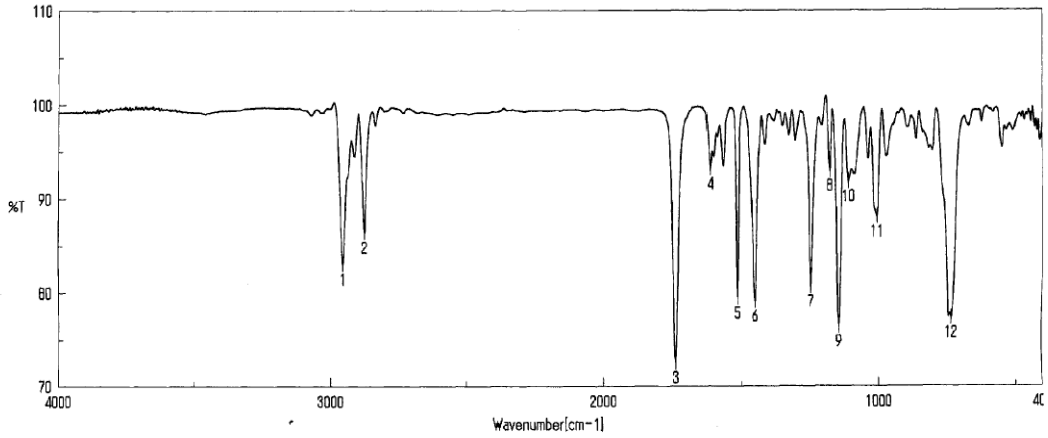

```
Current Data Parameters
NAME      Mar11-2009-hayashi
EXPNO                  180
PROCNO                   1

F2 - Acquisition Parameters
Date_          20090312
Time               1.59
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4000
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                  128
DW               20.800 usec
DE                6.00 usec
TE               302.9 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

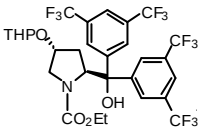
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2              -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1816007 MHz

F2 - Processing parameters
SI                32768
SF          100.6253403 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

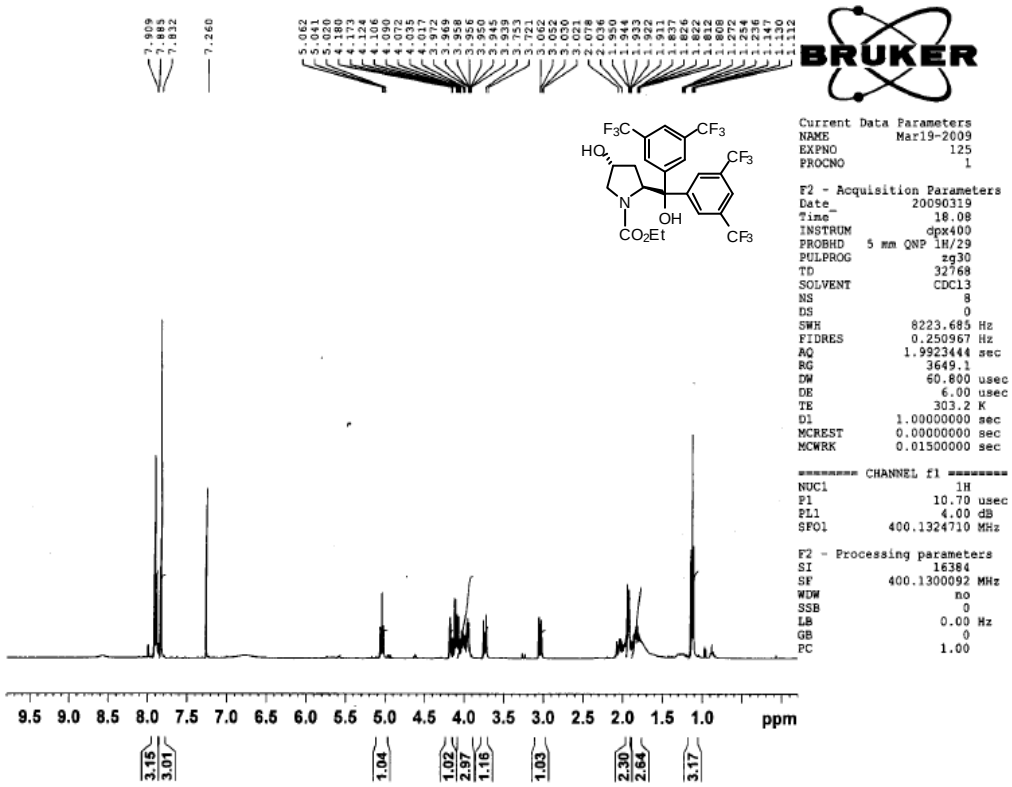


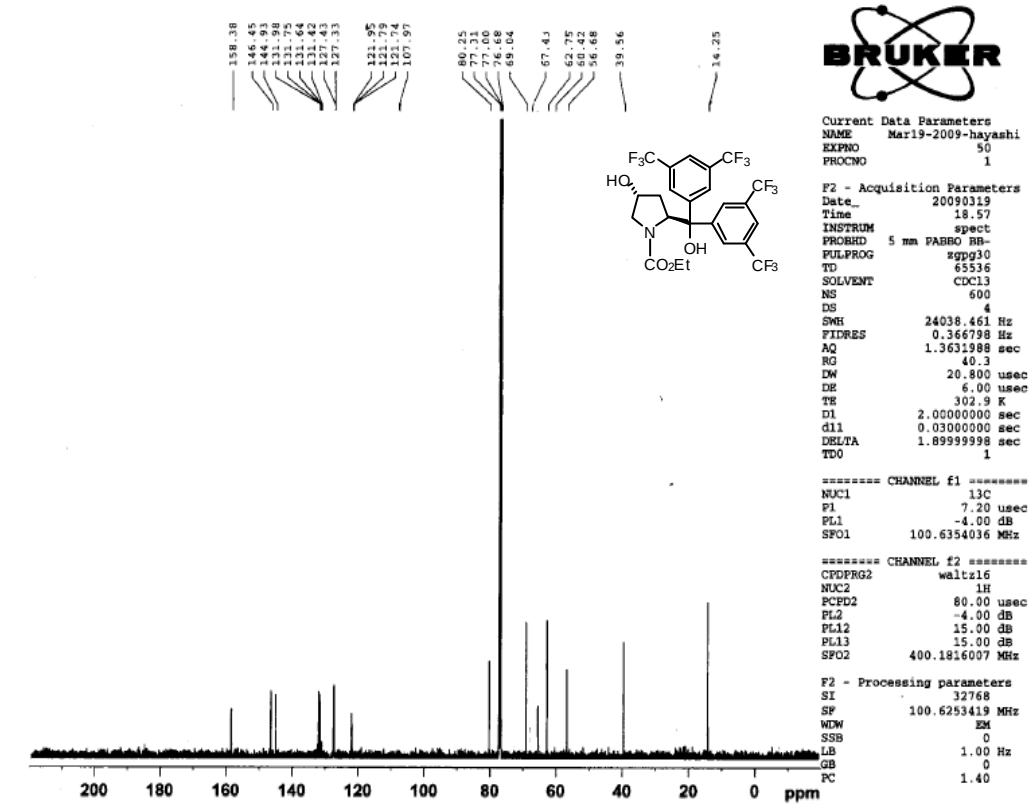
積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/11 20:19
測定者	
ファイル名	Memory#5
サンプル名	buckground
コメント	

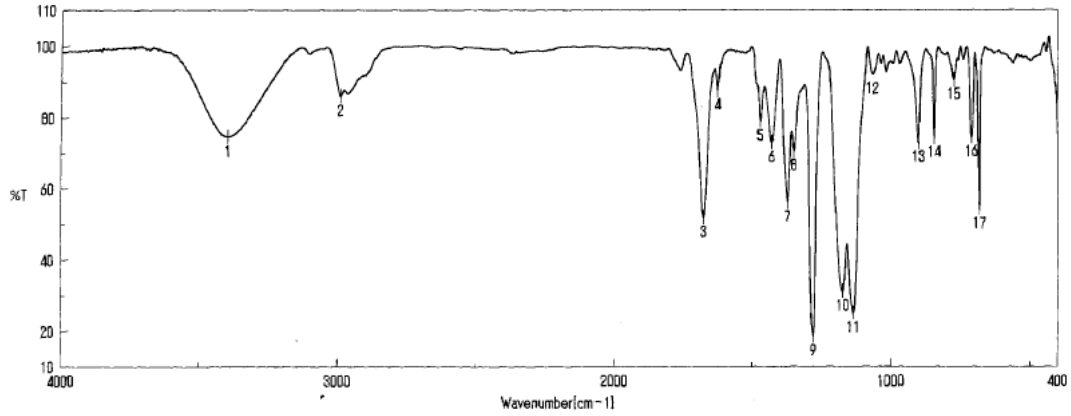
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec



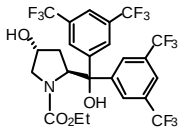
1:	2954.41,	83.0789	2:	2875.34,	86.4379	3:	1735.62,	72.5595	4:	1613.16,	93.2559
5:	1513.85,	79.4440	6:	1451.17,	79.1066	7:	1247.72,	80.7302	8:	1177.33,	93.0283
9:	1144.55,	78.4633	10:	1109.83,	91.9178	11:	1005.70,	88.1501	12:	736.67,	77.3761


7.909
7.885
7.832
7.260
5.062
5.041
5.020
4.110
4.133
4.124
4.106
4.090
4.072
4.035
4.017
3.972
3.969
3.958
3.956
3.950
3.945
3.939
3.753
3.721
3.062
3.052
3.030
3.021
3.008
2.036
1.950
1.944
1.933
1.922
1.831
1.826
1.822
1.812
1.808
1.272
1.264
1.236
1.147
1.130
1.112
BRUKER
F3C
CF3
HO
CF3
N
OH
CO2Et
CF3
Current Data Parameters
NAME Mar19-2009
EXPNO 125
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090319
Time 18.08
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 3649.1
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW no
SSB 0
LB 0.00 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
3.15
3.01
1.04
1.02
2.97
1.16
1.03
2.30
2.64
3.17

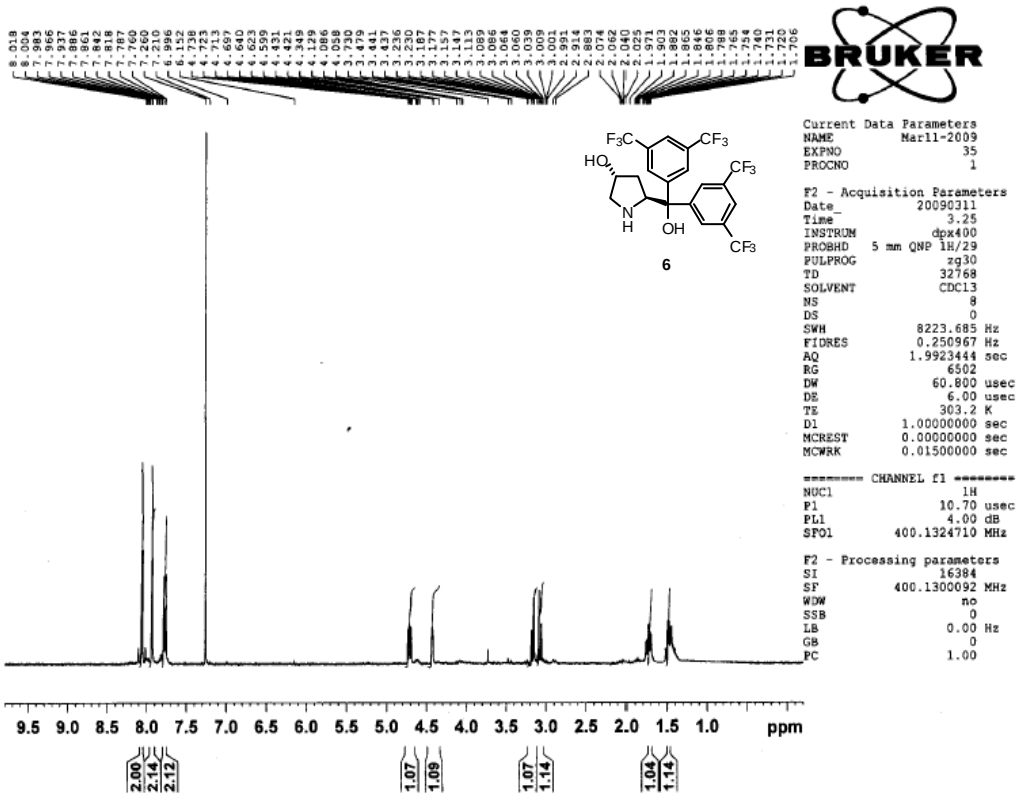

158.38
146.45
146.93
131.98
131.75
131.64
131.42
127.43
127.33
121.95
121.79
121.74
107.97
80.25
77.31
77.00
76.68
69.04
67.43
62.75
60.42
56.68
39.56
14.25
BRUKER
F3C
CF3
HO
CF3
N
OH
CO2Et
CF3
Current Data Parameters
NAME Mar19-2009-hayashi
EXPNO 50
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090319
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 40.3
DW 20.800 usec
DE 6.00 usec
TE 302.9 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 100.6253419 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

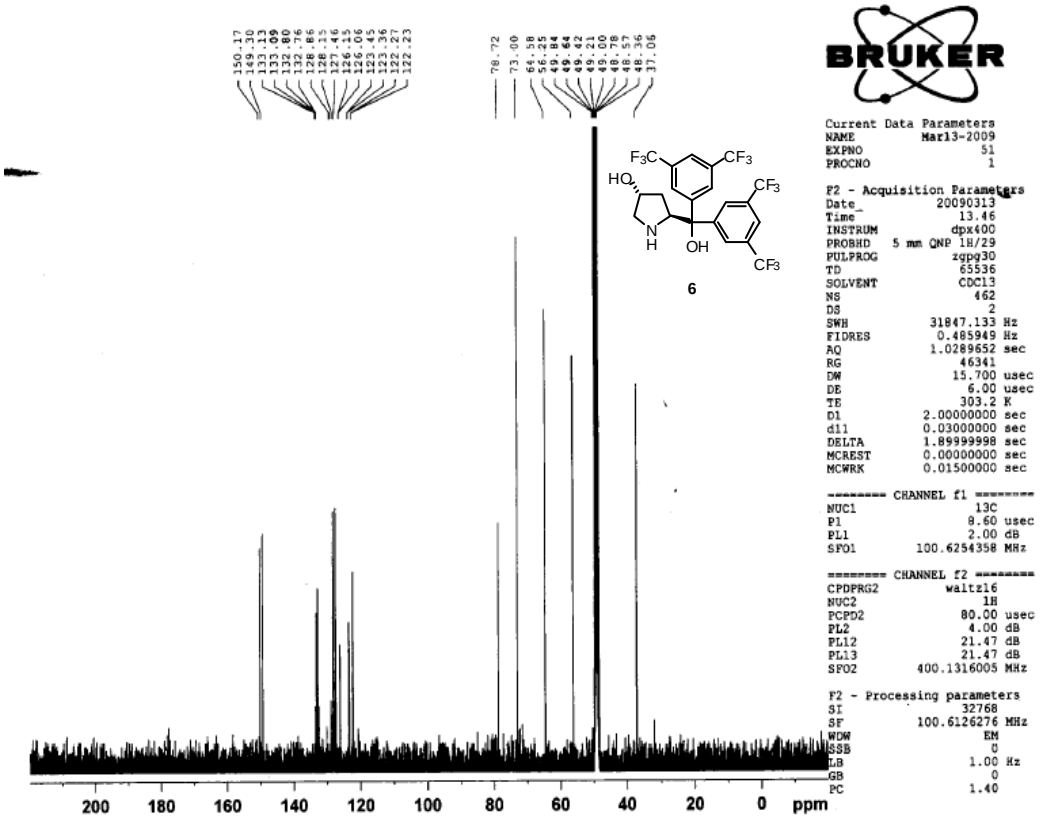


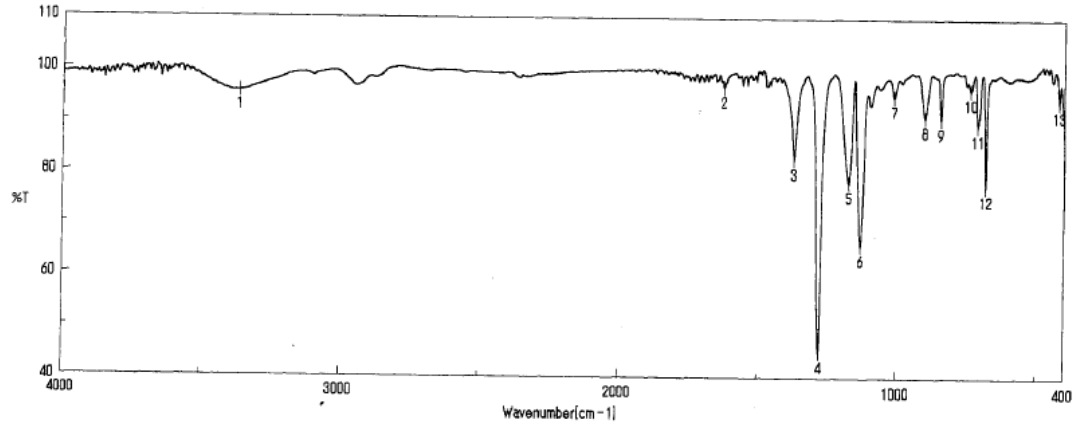
積算回数	32	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/04/24 11:39		
測定者			
ファイル名	Memory#3		
サンプル名	buckground		
コメント			



1:	3393.14,	74.6334	2:	2987.20,	86.1250	3:	1675.84,	51.9780	4:	1624.73,	87.7940
5:	1470.46,	79.0937	6:	1429.96,	73.1706	7:	1372.10,	56.2901	8:	1348.96,	70.7330
9:	1279.54,	18.7468	10:	1173.47,	31.4150	11:	1134.90,	25.2293	12:	1066.44,	92.3618
13:	901.56,	73.1558	14:	844.67,	74.4791	15:	773.32,	90.7548	16:	710.64,	74.6391
17:	682.68,	54.4788									

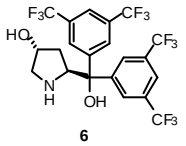

BRUKER
F3C
CF3
HO
CF3
N
H
OH
CF3
6
Current Data Parameters
NAME Mar11-2009
EXPNO 35
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090311
Time 3.25
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 6502
DW 60.800 usec
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW no
SSB 0
LB 0.00 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
2.00 2.14 2.12 1.07 1.09 1.07 1.14 1.04 1.14


BRUKER
F3C
CF3
HO
CF3
N
H
OH
CF3
6
Current Data Parameters
NAME Mar13-2009
EXPNO 51
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090313
Time 13.46
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 462
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 46341
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 8.60 usec
PL1 2.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 21.47 dB
PL13 21.47 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6126276 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/11 20:09
測定者	
ファイル名	Memory#2
サンプル名	buckground
コメント	

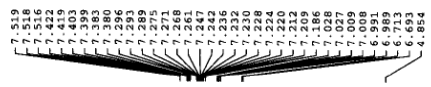
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec

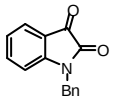


6

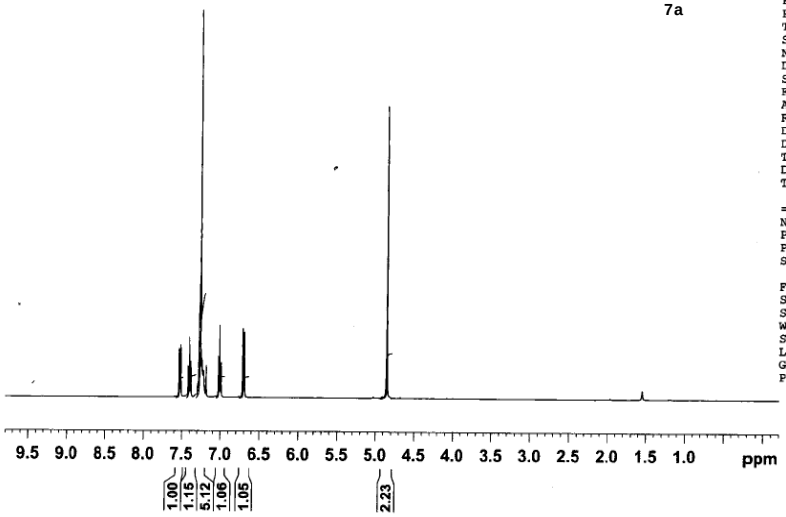
1:	3365.17,	95.7224	2:	1624.73,	96.6898	3:	1372.10,	82.6768	4:	1278.57,	45.1105
5:	1174.44,	78.2102	6:	1131.05,	65.8814	7:	1013.41,	95.0227	8:	902.52,	90.9982
9:	844.67,	90.6281	10:	737.64,	96.4286	11:	711.60,	89.3870	12:	682.66,	77.4696
13:	418.48,	93.9506									

Bn-isatin





7a



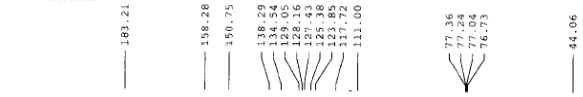

```
Current Data Parameters
NAME      Dec06-2008-hayashi
EXPNO                   80
PROCNO                   1

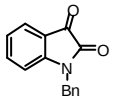
F2 - Acquisition Parameters
Date_           20081206
Time               11.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  181
DW               60.800 usec
DE                6.00 usec
TE               303.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              12.00 usec
PL1             -4.00 dB
SFO1        400.1824713 MHz

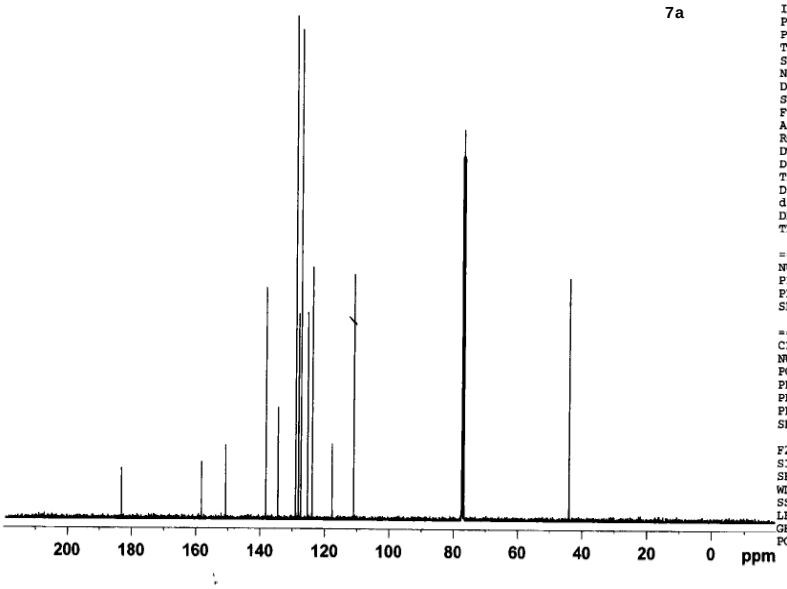
F2 - Processing parameters
SI                32768
SF          400.1800369 MHz
WDW                  EM
SSB                   0
LB             0.30 Hz
GB                    0
PC                 1.00
```

Bn-isatin





7a



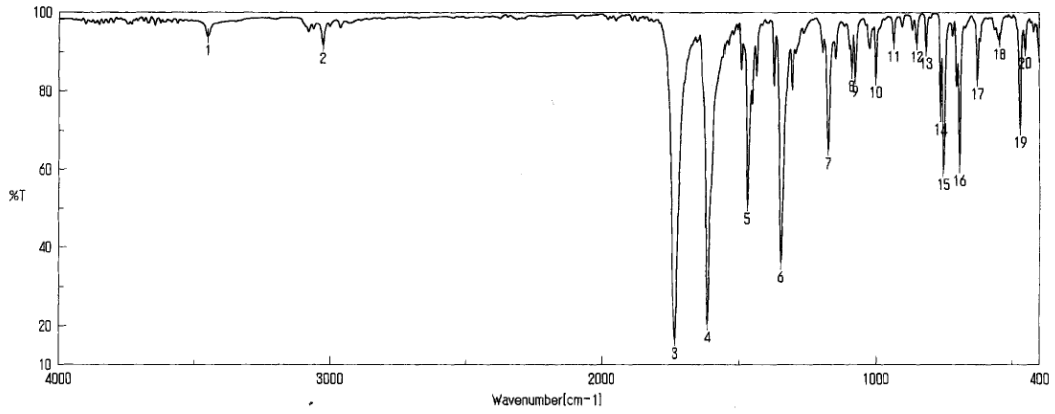

```
Current Data Parameters
NAME      Dec06-2008-hayashi
EXPNO                  120
PROCNO                   1

F2 - Acquisition Parameters
Date_           20081206
Time               16.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 80.6
DW               20.800 usec
DE                6.00 usec
TE               302.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                  1

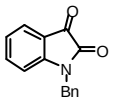
======== CHANNEL f1 ========
NUC1               13C
P1              7.20 usec
PL1             -4.00 dB
SFO1        100.6354036 MHz

======== 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          100.6253448 MHz
WDW                  EM
SSB                   0
LB             1.00 Hz
GB                    0
PC                 1.40
```

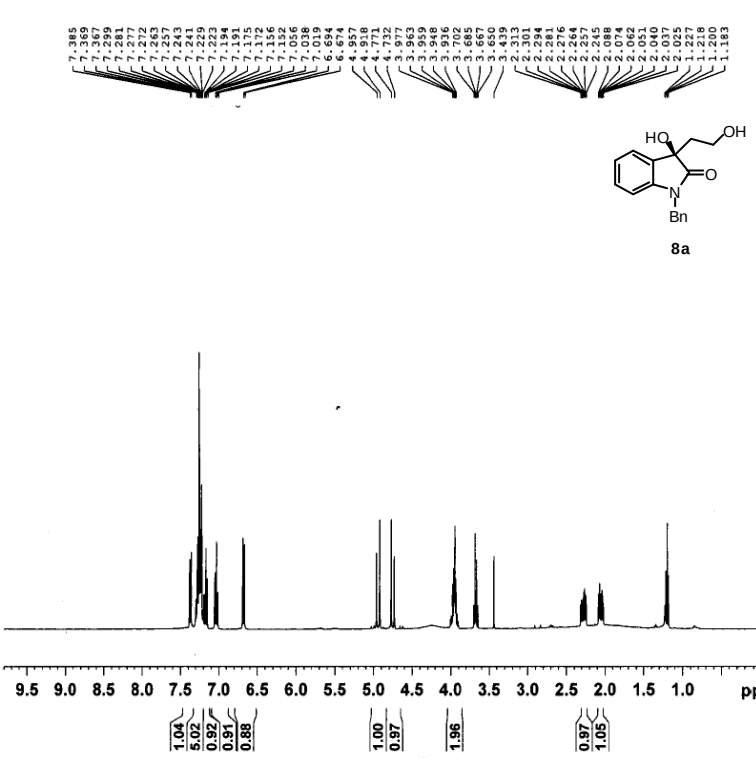


積算回数	64	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/01/03 15:25		
測定者			
ファイル名	Bnisatin.JWS		
サンプル名	buckground		
コメント			



7a

1:	3453.88, 94.0413	2:	3029.62, 91.9169	3:	1731.76, 16.4638	4:	1613.16, 20.6778
5:	1471.42, 50.9012	6:	1348.96, 36.1033	7:	1177.33, 65.0943	8:	1091.51, 84.8911
9:	1078.01, 83.6301	10:	1003.77, 83.4161	11:	938.20, 92.4271	12:	853.35, 92.3515
13:	818.63, 90.5934	14:	765.60, 73.8559	15:	754.03, 59.8688	16:	694.25, 60.7783
17:	627.72, 82.9286	18:	547.68, 93.0124	19:	471.51, 70.6046	20:	453.19, 90.6829



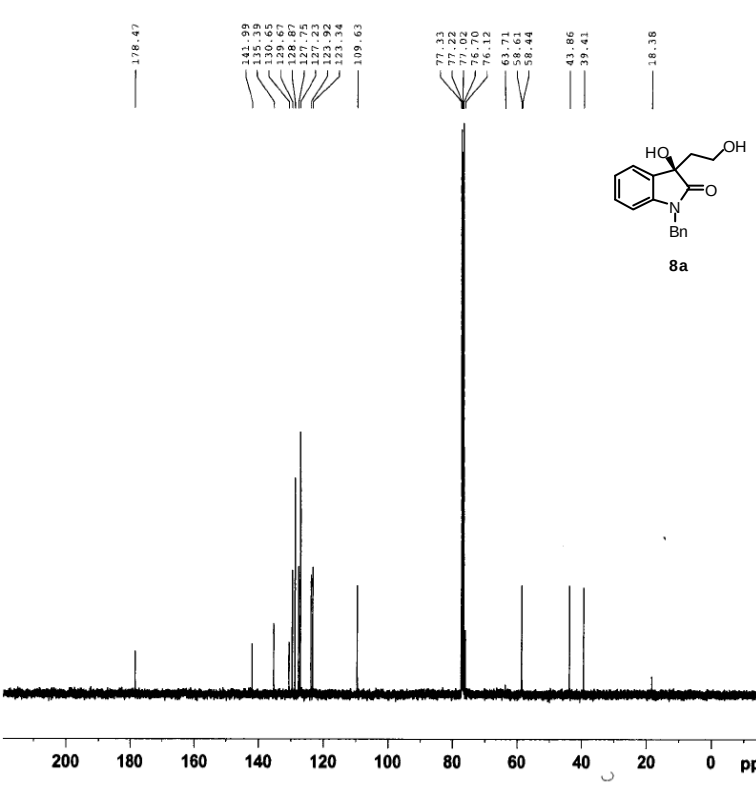
BRUKER

```
Current Data Parameters
NAME          Dec24-2008
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081224
Time               16.26
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                  2048
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                10.70 usec
PL1                4.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300218 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



BRUKER

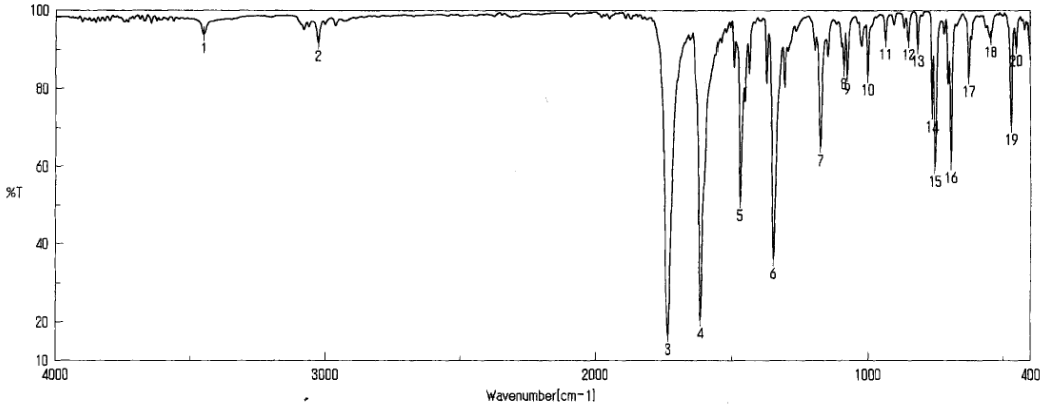
```
Current Data Parameters
NAME      Dec24-2008-hayashi
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081224
Time               18.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                  40.3
DW                20.800 usec
DE                 6.00 usec
TE                302.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

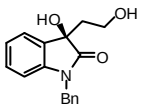
======== CHANNEL f1 ========
NUC1               13C
P1                7.20 usec
PL1               -4.00 dB
SFO1         100.6354036 MHz

======== 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          100.6253440 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



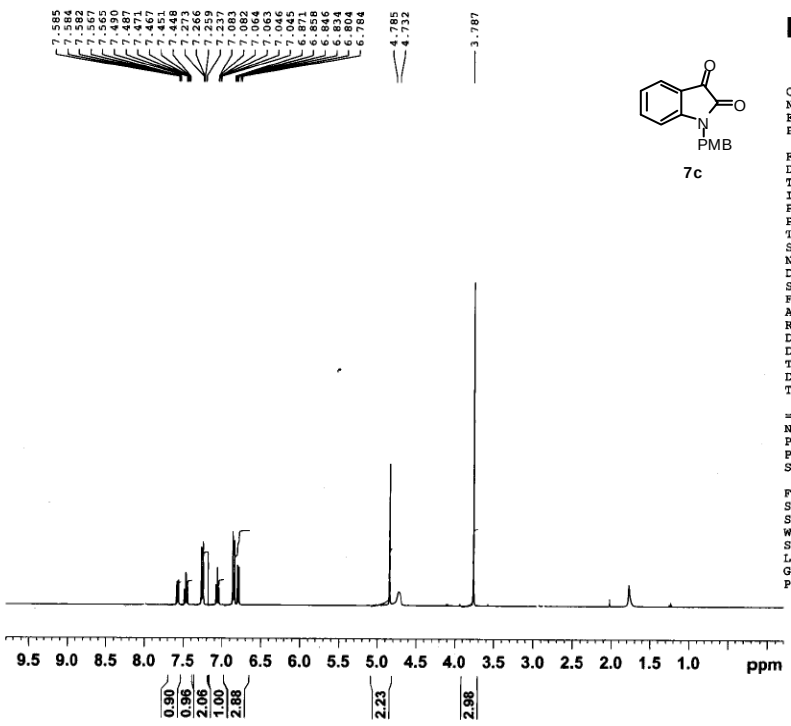
積算回数	64	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/01/03 15:25		
測定者			
ファイル名	Bnisatin.JWS		
サンプル名	buckground		
コメント			



8a

1:	3453.88,	94.0413	2:	3029.62,	91.9169	3:	1731.76,	16.4638	4:	1613.16,	20.6778
5:	1471.42,	50.9012	6:	1348.96,	36.1033	7:	1177.33,	65.0943	8:	1091.51,	84.8911
9:	1078.01,	83.6301	10:	1003.77,	83.4161	11:	938.20,	92.4271	12:	853.35,	92.3515
13:	818.63,	90.5934	14:	765.60,	73.8559	15:	754.03,	59.8688	16:	694.25,	60.7783
17:	627.72,	82.9286	18:	547.68,	93.0124	19:	471.51,	70.6046	20:	453.19,	90.6829

PMB-isatin



7c

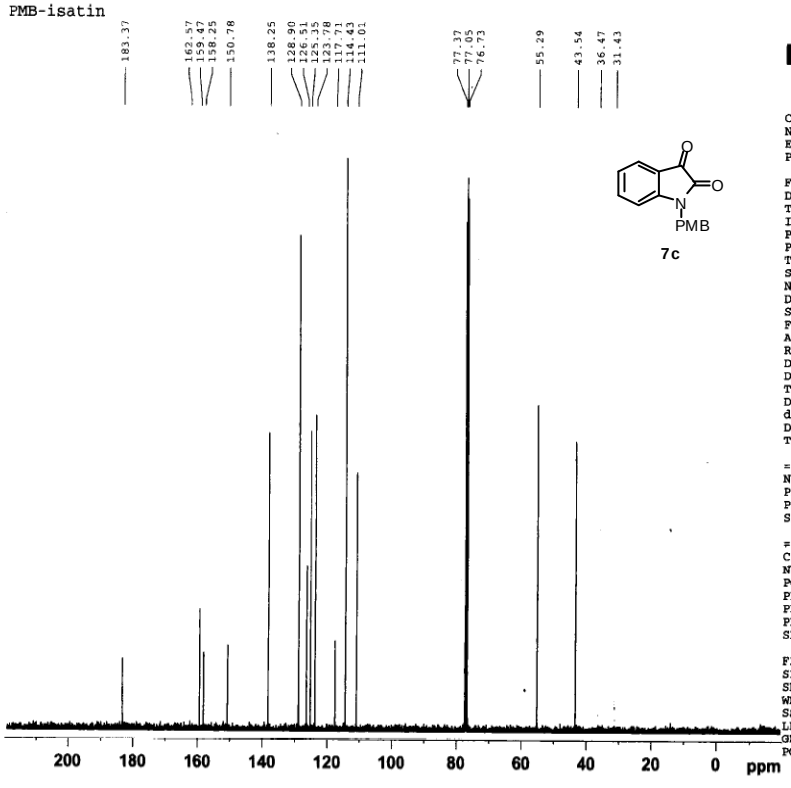
```
Current Data Parameters
NAME      Dec06-2008-hayashi
EXPNO                 90
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081206
Time               11.52
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                 80.6
DW               60.800 usec
DE                6.00 usec
TE               303.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -4.00 dB
SFO1         400.1824713 MHz

F2 - Processing parameters
SI                 32768
SF           400.1800078 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

PMB-isatin



7c

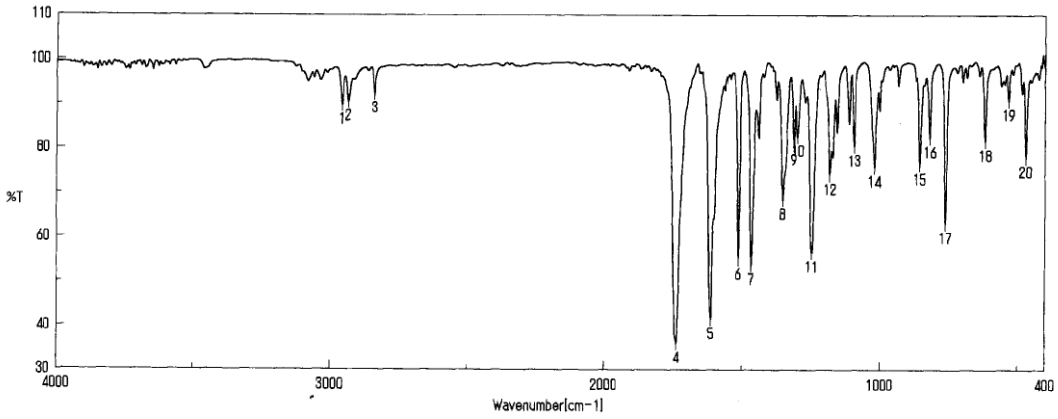
```
Current Data Parameters
NAME      Dec06-2008-hayashi
EXPNO                130
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081206
Time               17.05
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 40.3
DW               20.800 usec
DE                6.00 usec
TE               302.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

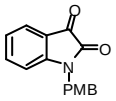
======== CHANNEL f1 ========
NUC1                 13C
P1                7.20 usec
PL1               -4.00 dB
SFO1        100.6354036 MHz

======== 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          100.6253410 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

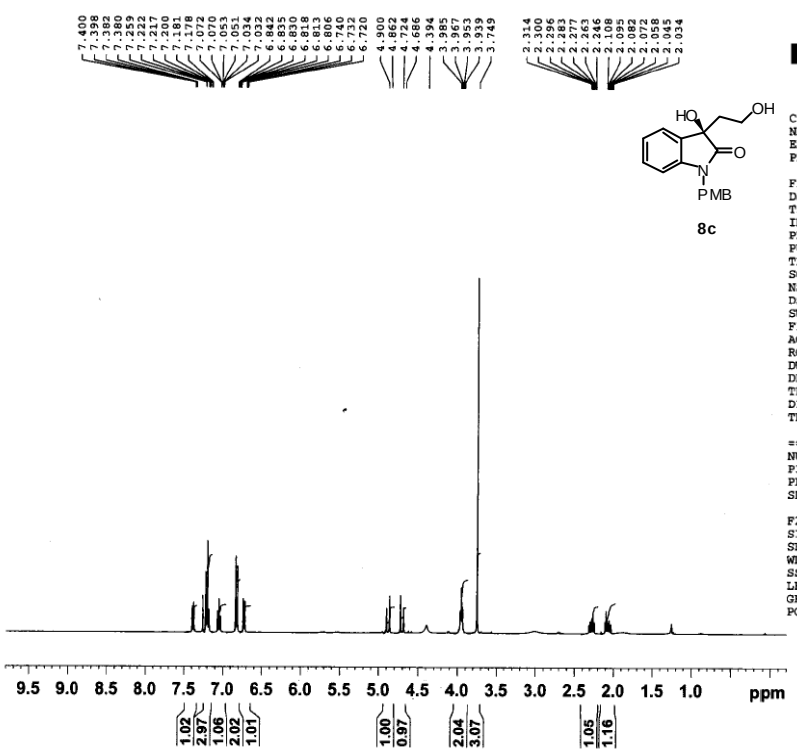


積算回数	64	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/01/03 15:22		
測定者			
ファイル名	PMBisatin.JWS		
サンプル名	buckground		
コメント			



7c

1:	2960.20,	89.8313	2:	2936.09,	90.5909	3:	2840.63,	92.4719	4:	1735.62,	36.1058
5:	1610.27,	41.5858	6:	1513.85,	55.0553	7:	1467.56,	53.8085	8:	1353.78,	98.4944
9:	1314.25,	80.8667	10:	1299.79,	82.8880	11:	1248.68,	56.6697	12:	1182.15,	74.1755
13:	1094.41,	80.6432	14:	1021.12,	75.9802	15:	856.24,	76.5139	16:	819.60,	82.5785
17:	762.71,	63.2523	18:	618.07,	81.7643	19:	533.22,	90.9339	20:	470.55,	78.0972

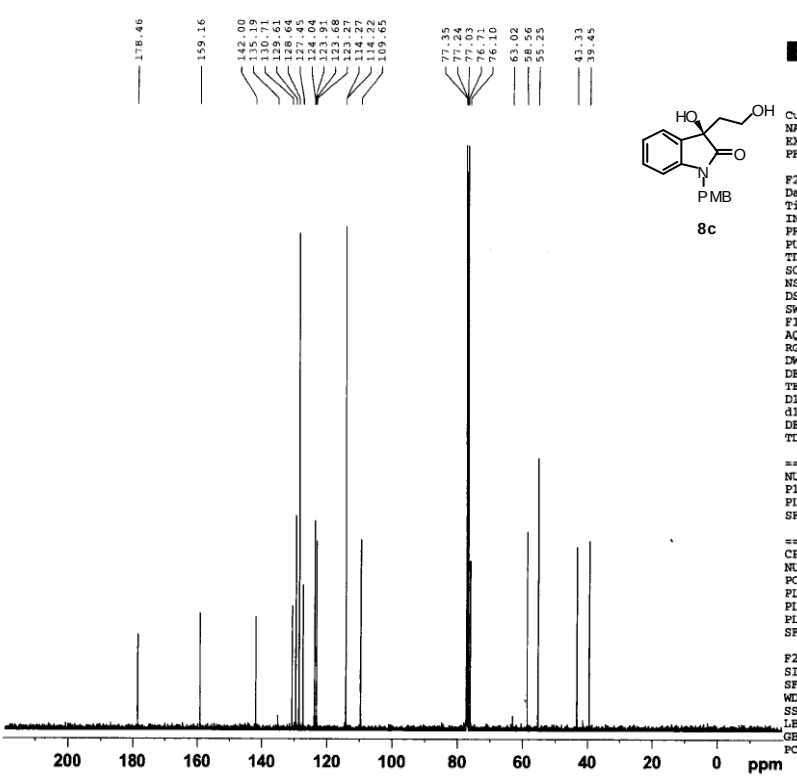



```
Current Data Parameters
NAME      Dec18-2008-hayashi
EXPNO                     50
PROCNO                     1

F2 - Acquisition Parameters
Date_               20081218
Time                   16.01
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      144
DW                 60.800 usec
DE                  6.00 usec
TE                  303.0 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                  12.00 usec
PL1                 -4.00 dB
SFO1          400.1824713 MHz

F2 - Processing parameters
SI                     32768
SF            400.1800079 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00
```



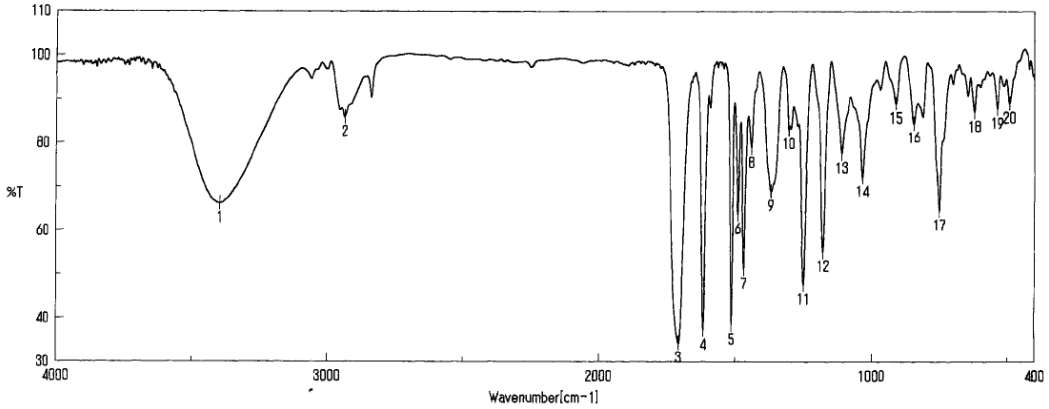

```
Current Data Parameters
NAME      Dec18-2008-hayashi
EXPNO                     51
PROCNO                     1

F2 - Acquisition Parameters
Date_               20081218
Time                   16.32
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      500
DS                        4
SWH               24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                      40.3
DW                 20.800 usec
DE                  6.00 usec
TE                  302.9 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

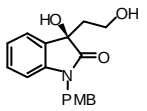
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                15.00 dB
PL13                15.00 dB
SFO2          400.1816007 MHz

F2 - Processing parameters
SI                     32768
SF            100.6253437 MHz
WDW                       EM
SSB                        0
LB                  1.00 Hz
GB                         0
PC                      1.40
```



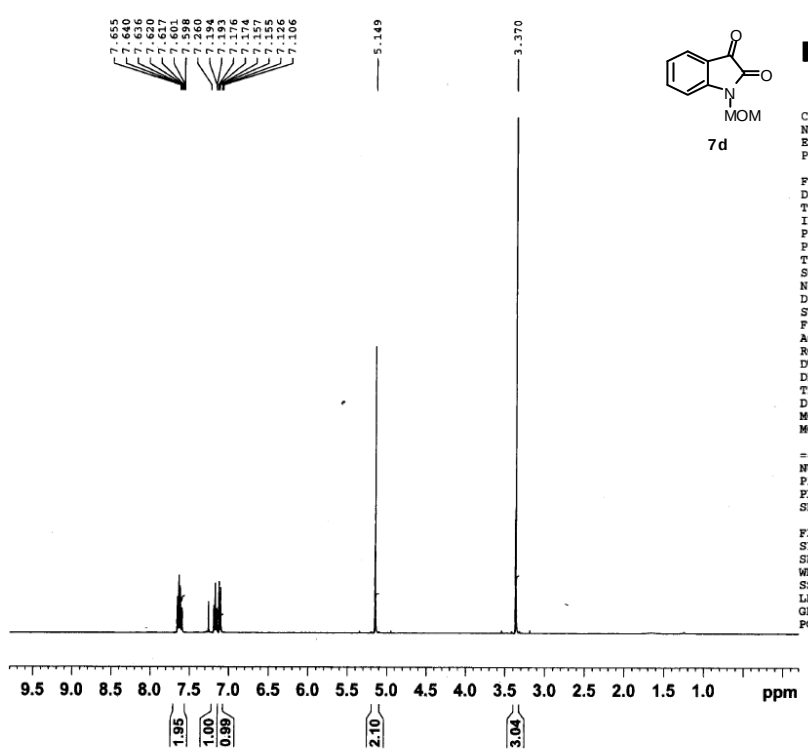
積算回数	23
ゼロフィリング	ON
ゲイン	2
日時	108/12/24 18:53
測定者	
ファイル名	Memory#7
サンプル名	buckground
コメント	

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



8c

1:	3395.07,	66.2394	2:	2933.20,	86.0409	3:	1704.76,	34.3665	4:	1614.13,	37.2696
5:	1513.85,	38.4710	6:	1487.81,	63.7787	7:	1467.56,	51.2527	8:	1439.60,	78.9403
9:	1367.28,	69.1035	10:	1302.68,	83.2122	11:	1248.68,	47.7100	12:	1176.36,	55.1991
13:	1108.87,	77.7164	14:	1032.69,	72.3730	15:	910.24,	89.0888	16:	843.70,	84.6343
17:	751.14,	64.6205	18:	620.00,	87.1548	19:	537.08,	87.8828	20:	490.79,	89.1436

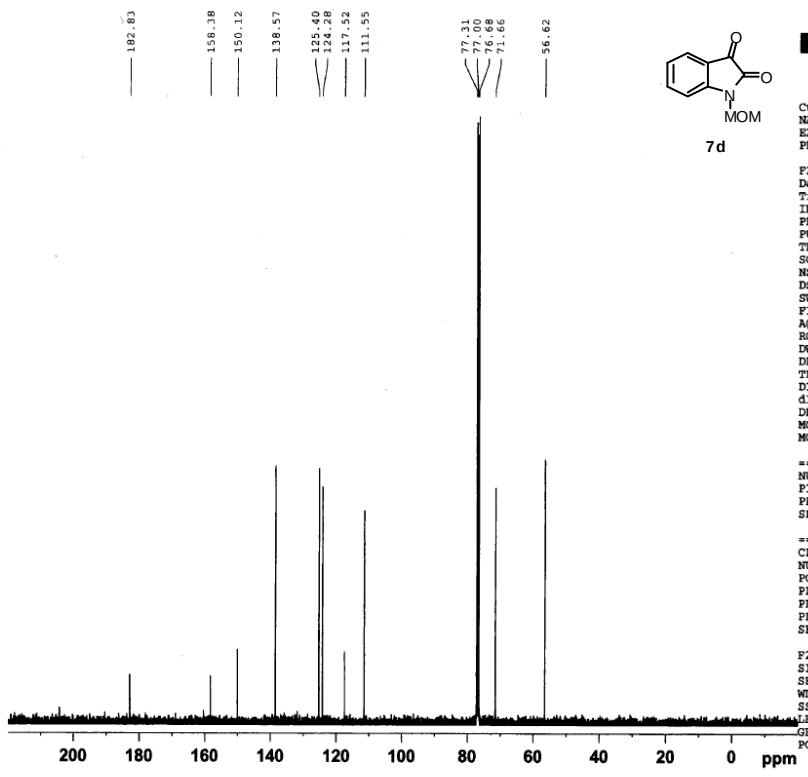



```
Current Data Parameters
NAME          Dec06-2008
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081206
Time               20.39
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                2580.3
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                 10.70 usec
PL1                4.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300092 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



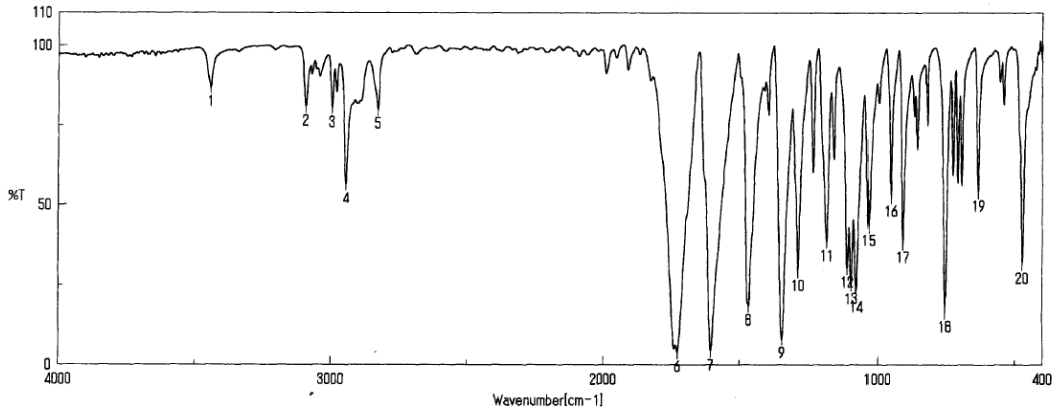

```
Current Data Parameters
NAME          Dec06-2008
EXPNO                102
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081206
Time               20.51
INSTRUM           dpx400
PROBHD   5 mm QNP 1H/29
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   327
DS                     2
SWH            31847.133 Hz
FIDRES          0.485949 Hz
AQ             1.0289652 sec
RG                4597.6
DW                15.700 usec
DE                 6.00 usec
TE                303.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

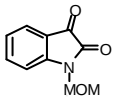
======== CHANNEL f1 ========
NUC1                 13C
P1                 8.60 usec
PL1                2.00 dB
SFO1         100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                4.00 dB
PL12              21.47 dB
PL13              21.47 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127821 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

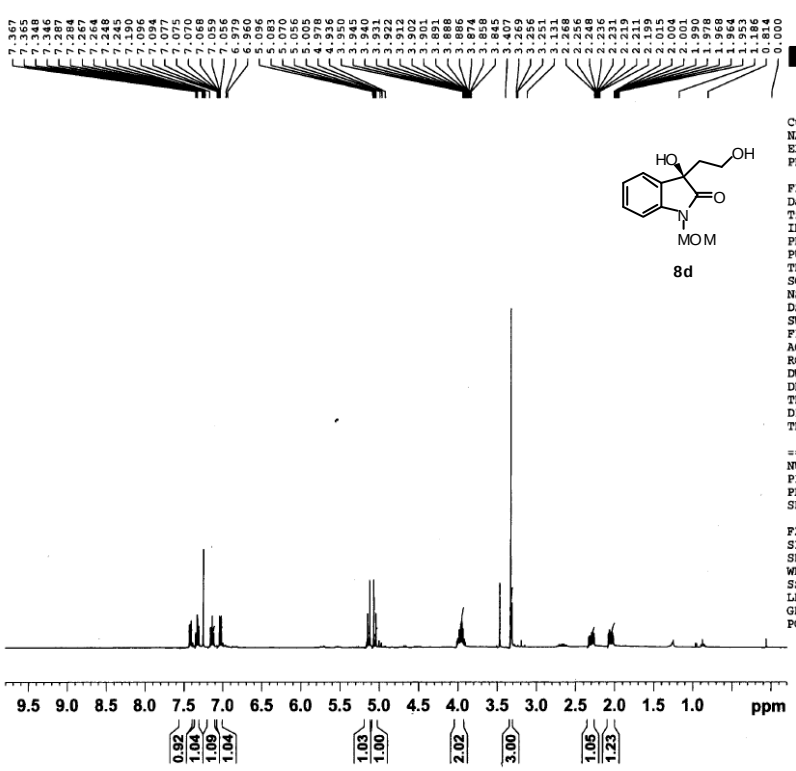


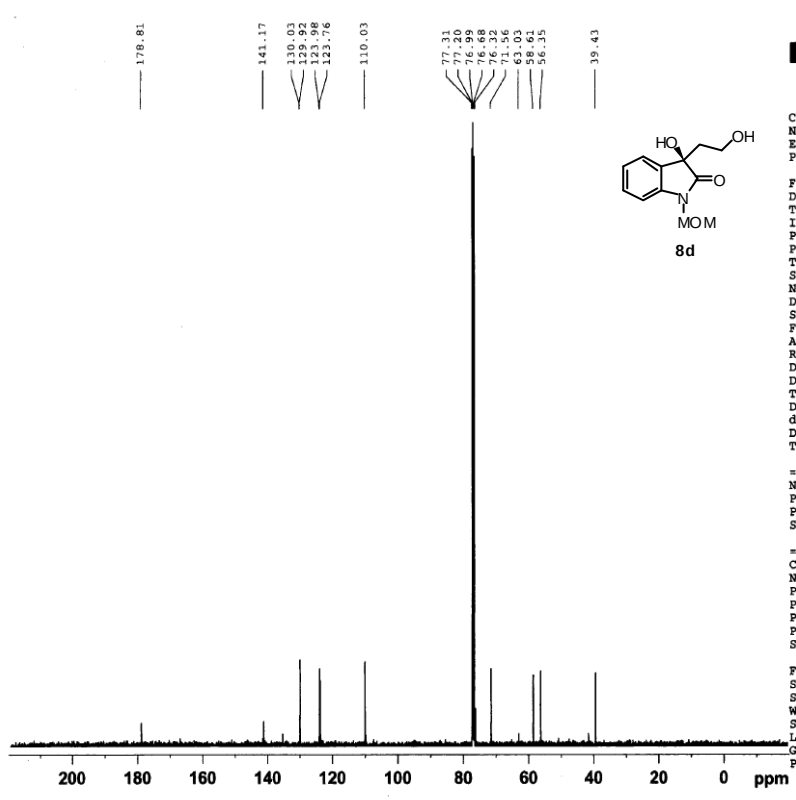
積算回数	64	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	4	スキャンスピード	2 mm/sec
日時	109/01/03 15:11		
測定者			
ファイル名	MOMisatin.JWS		
サンプル名	buckground		
コメント			

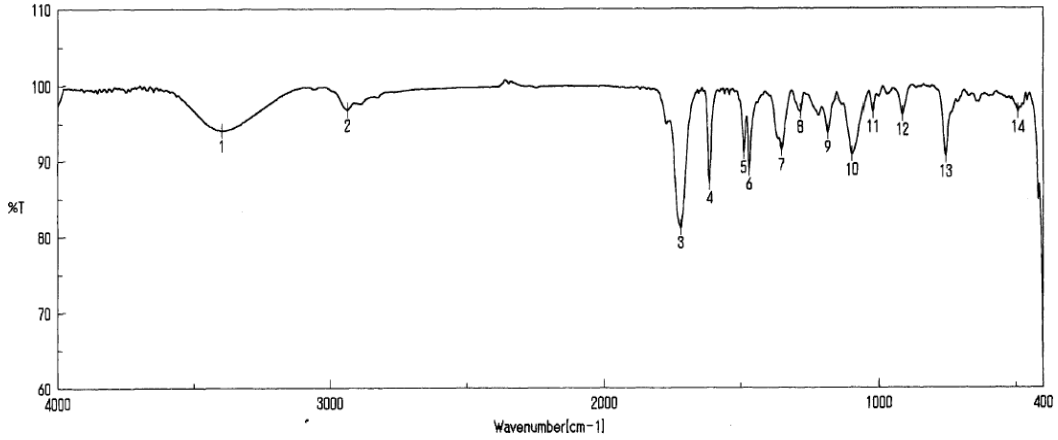


7d

1:	3443.28,	86.8512	2:	3094.23,	80.9017	3:	2996.84,	80.4734	4:	2944.77,	56.6455
5:	2828.10,	80.0723	6:	1726.94,	4.0359	7:	1605.45,	4.4528	8:	1467.56,	18.5314
9:	1346.07,	8.2906	10:	1287.25,	29.5032	11:	1183.11,	38.7052	12:	1108.87,	30.5332
13:	1094.41,	25.3041	14:	1076.08,	22.6360	15:	1031.73,	43.4538	16:	952.66,	52.6006
17:	910.24,	38.1401	18:	755.96,	16.5387	19:	634.47,	54.1929	20:	472.47,	31.7685

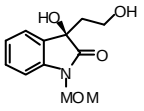

7.367
7.365
7.348
7.346
7.287
7.284
7.267
7.264
7.248
7.245
7.190
7.096
7.094
7.077
7.075
7.070
7.068
7.059
7.056
6.979
6.960
6.956
5.083
5.070
5.056
5.005
4.978
4.935
3.950
3.945
3.940
3.931
3.922
3.913
3.902
3.901
3.891
3.888
3.886
3.874
3.858
3.845
3.407
3.269
3.256
3.251
3.231
3.168
2.256
2.248
2.236
2.231
2.219
2.211
2.199
2.015
2.004
2.001
1.990
1.978
1.968
1.964
1.953
1.186
0.814
0.000
BRUKER
HO
OH
O
N
MOM
8d
Current Data Parameters
NAME Dec17-2008-hayashi
EXPNO 53
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081217
Time 11.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.00 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 400.1824713 MHz
F2 - Processing parameters
SI 32768
SF 400.1800092 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
0.92
1.04
1.09
1.04
1.03
1.00
2.02
3.00
1.05
1.23


178.81
141.17
130.03
129.92
123.98
123.76
110.03
77.31
77.20
76.99
76.68
76.32
71.56
63.03
58.61
56.35
39.43
BRUKER
HO
OH
O
N
MOM
8d
Current Data Parameters
NAME Dec17-2008-hayashi
EXPNO 54
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081217
Time 12.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 302.9 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 100.6253424 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



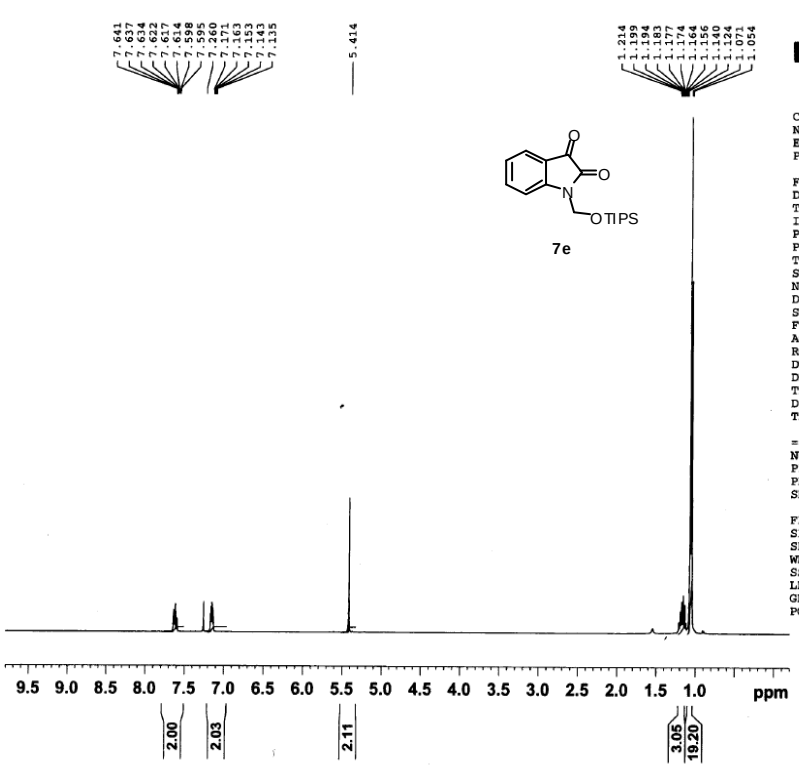
積算回数	21
ゼロフィリング	ON
ゲイン	2
日時	108/12/24 18:34
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

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



8d

1:	3399.89,	94.0082	2:	2936.09,	96.7471	3:	1718.26,	81.1851	4:	1614.13,	87.1222
5:	1487.81,	91.1412	6:	1468.53,	88.9544	7:	1350.89,	91.4164	8:	1281.47,	96.4765
9:	1182.15,	93.7924	10:	1094.41,	90.8186	11:	1021.12,	96.5099	12:	913.13,	96.1381
13:	755.96,	90.5991	14:	492.72,	96.4407						

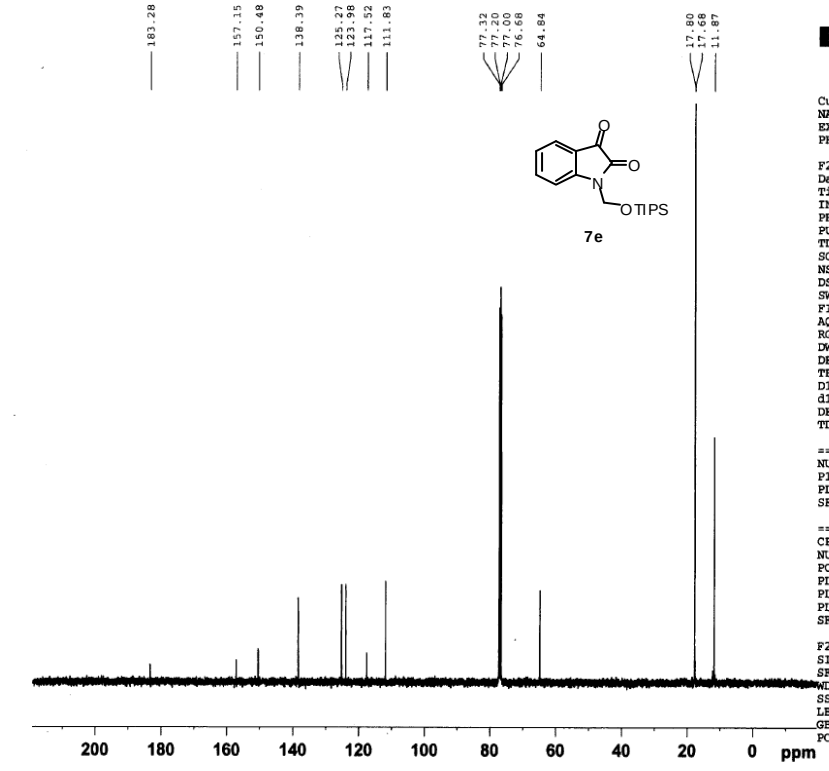


Current Data Parameters
NAME Dec17-2008-hayashi
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081217
Time 10.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.00 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 400.1824713 MHz

F2 - Processing parameters
SI 32768
SF 400.1800075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



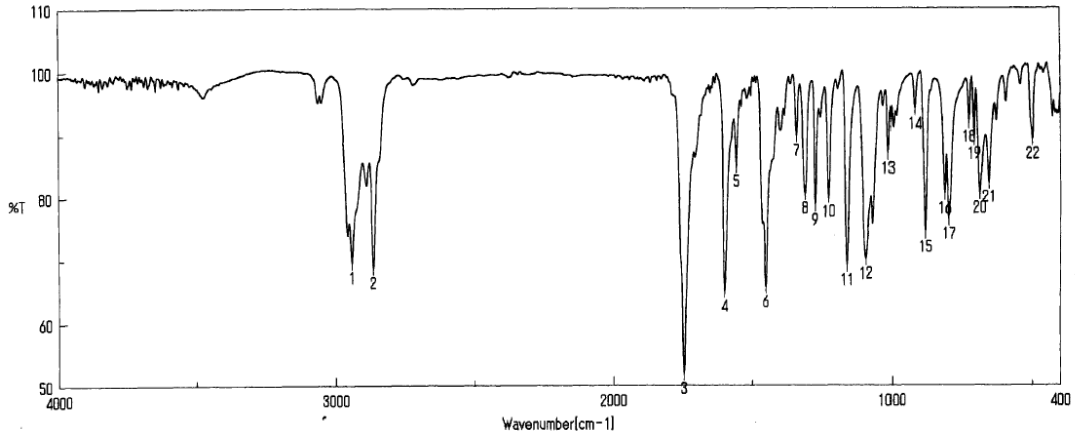
Current Data Parameters
NAME Dec17-2008-hayashi
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081217
Time 10.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.00 usec
TE 302.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

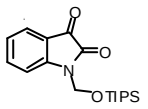
======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 -4.00 dB
SFO1 100.6354036 MHz

======== 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 100.6253420 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

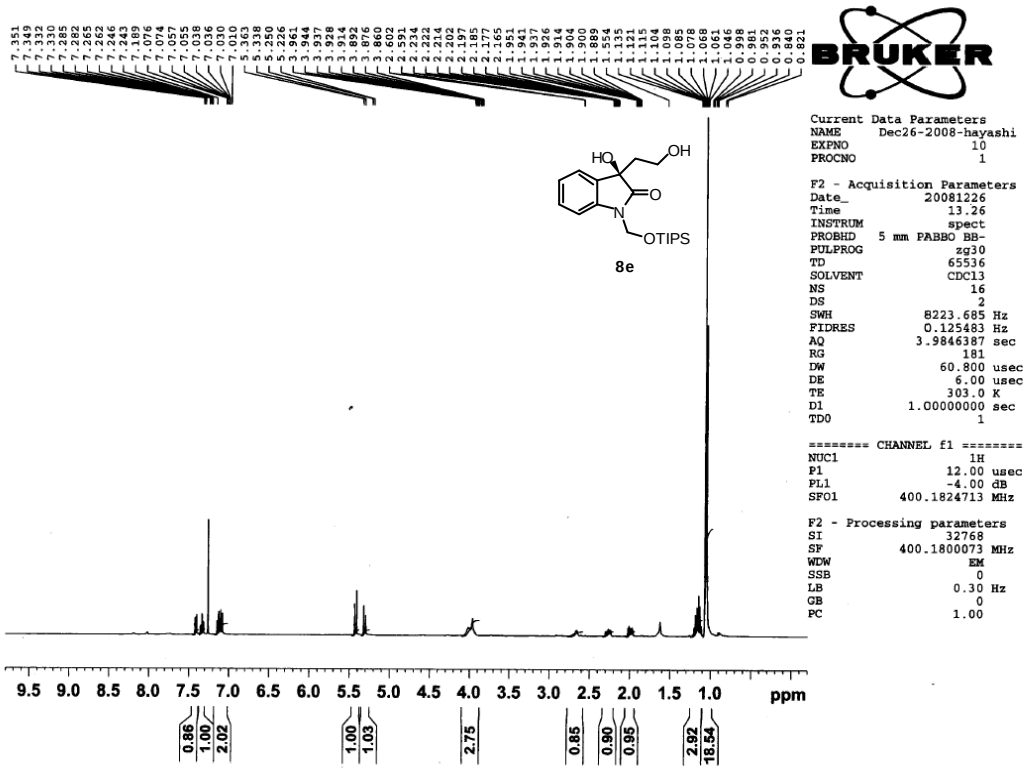


積算回数	23	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	108/12/24 19:06		
測定者			
ファイル名	Memory#11		
サンプル名	buckground		
コメント			



7e

1:	2941.88,	69.6814	2:	2865.70,	68.8611	3:	1746.23,	51.9457	4:	1600.63,	65.0892
5:	1559.17,	85.0539	6:	1452.14,	65.7717	7:	1343.18,	89.8150	8:	1311.36,	80.6676
9:	1274.72,	78.8715	10:	1226.51,	80.1445	11:	1159.97,	69.2797	12:	1092.48,	70.2863
13:	1013.41,	87.0269	14:	919.88,	94.1106	15:	883.24,	74.5372	16:	812.85,	80.6181
17:	799.35,	76.6129	18:	726.07,	92.0852	19:	707.75,	89.3292	20:	688.46,	80.7240

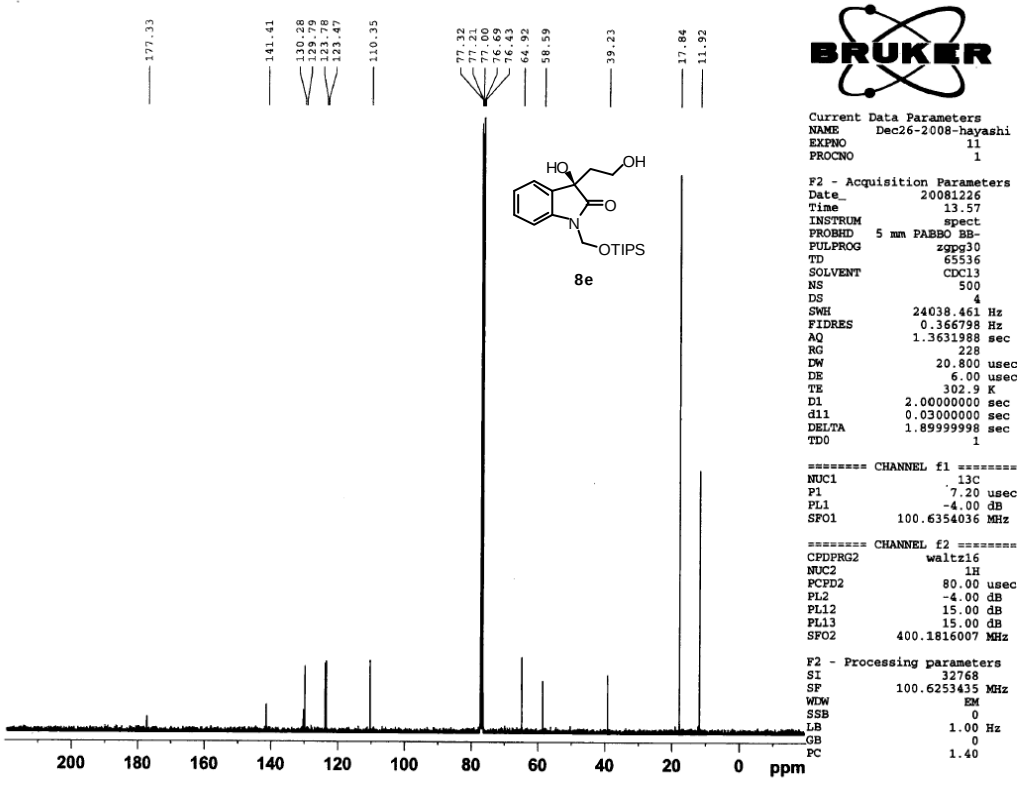



```
Current Data Parameters
NAME      Dec26-2008-hayashi
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20081226
Time                  13.26
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      181
DW                   60.800 usec
DE                    6.00 usec
TE                   303.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     12.00 usec
PL1                    -4.00 dB
SFO1           400.1824713 MHz

F2 - Processing parameters
SI                        32768
SF                 400.1800073 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



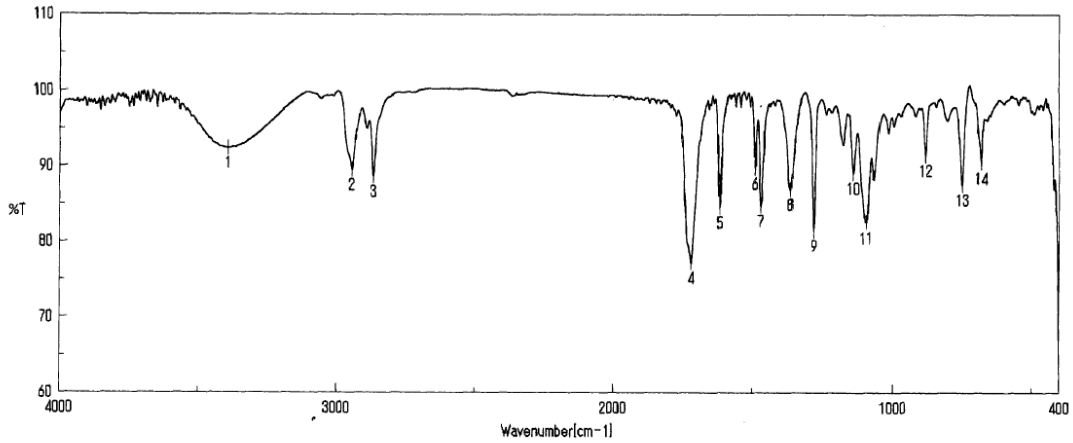

```
Current Data Parameters
NAME      Dec26-2008-hayashi
EXPNO                     11
PROCNO                     1

F2 - Acquisition Parameters
Date_              20081226
Time                  13.57
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      500
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      228
DW                   20.800 usec
DE                    6.00 usec
TE                   302.9 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

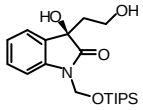
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    -4.00 dB
PL12                   15.00 dB
PL13                   15.00 dB
SFO2           400.1816007 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6253435 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```



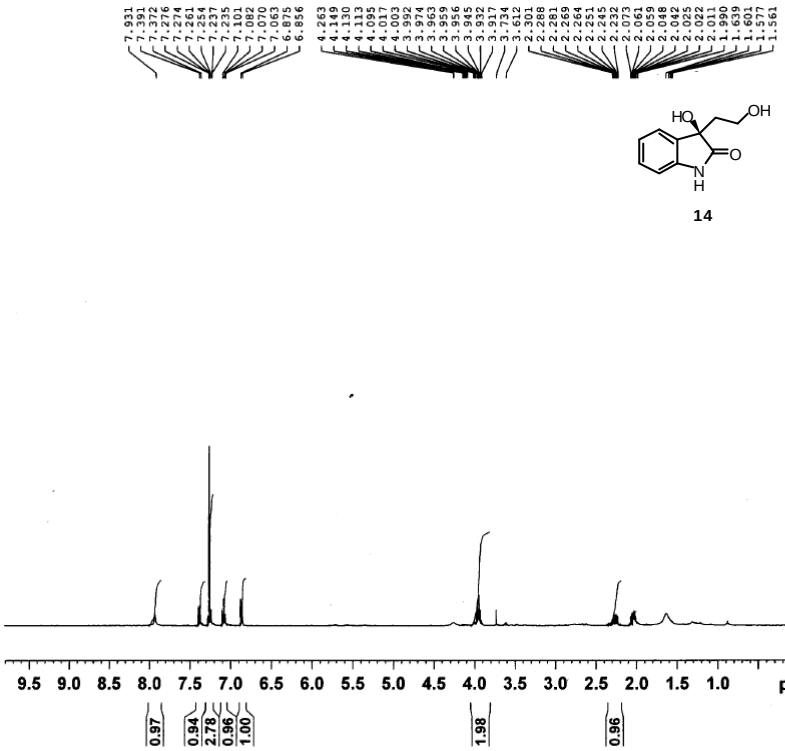
積算回数	16
ゼロフィリング	ON
ゲイン	1
日時	109/03/09 20:44
測定者	
ファイル名	Memory#5
サンプル名	buckground
コメント	

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



8e

1:	3392.17, 92.4051	2:	2944.77, 89.5829	3:	2866.67, 88.6778	4:	1718.26, 77.2375
5:	1615.09, 84.5267	6:	1488.78, 89.7725	7:	1469.49, 84.7470	8:	1364.39, 86.9990
9:	1279.54, 81.5174	10:	1139.72, 89.1043	11:	1092.48, 82.5320	12:	882.27, 91.4709
13:	751.14, 87.6203	14:	682.68, 90.4104				

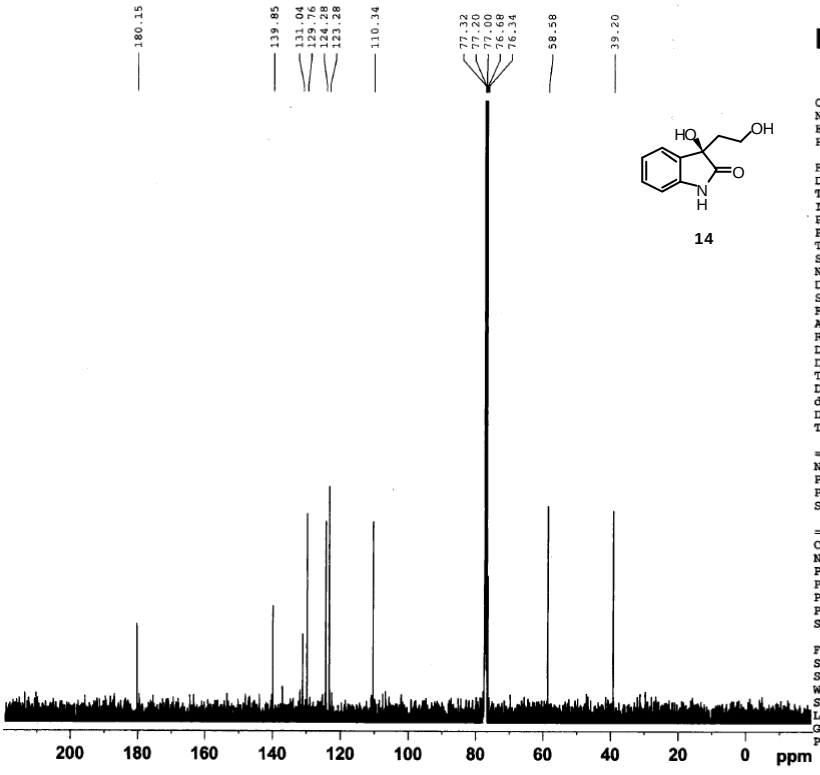



```
Current Data Parameters
NAME          Mar21-2009
EXPNO                 81
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090321
Time               19.48
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                5160.6
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                10.70 usec
PL1                4.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300092 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```



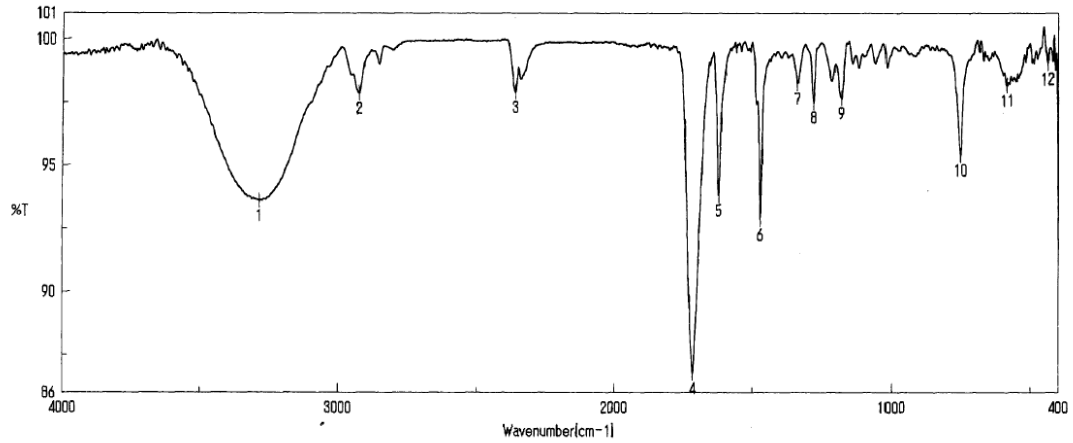

```
Current Data Parameters
NAME      Mar21-2009-hayashi
EXPNO                    110
PROCNO                     1

F2 - Acquisition Parameters
Date_           20090321
Time               21.52
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2000
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   114
DW                20.800 usec
DE                 6.00 usec
TE                302.9 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

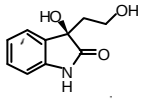
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1816007 MHz

F2 - Processing parameters
SI                 32768
SF          100.6253405 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



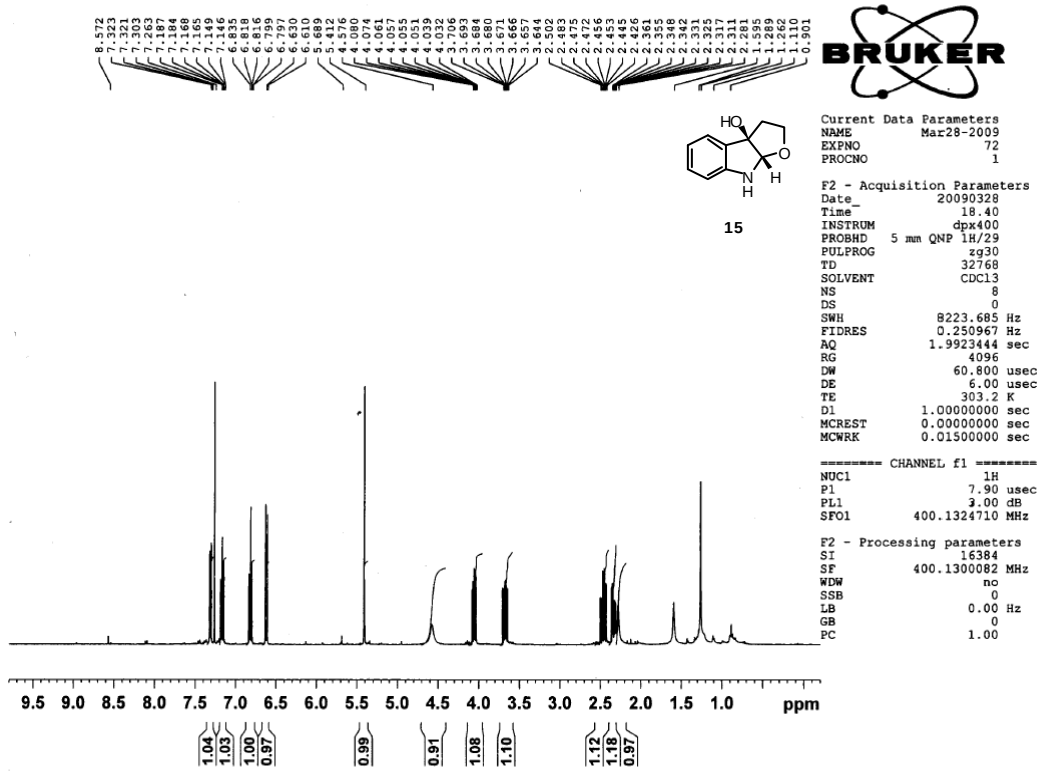
積算回数	16
ゼロフィリング	ON
ゲイン	1
日時	109/03/23 14:57
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

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



14

1:	3284.18,	93.6011	2:	2923.56,	97.8678	3:	2358.52,	97.9053	4:	1715.37,	86.7861
5:	1621.84,	93.8252	6:	1471.42,	92.8601	7:	1337.39,	98.2566	8:	1279.54,	97.5109
9:	1178.29,	97.6699	10:	753.07,	95.4080	11:	584.33,	98.1779	12:	437.76,	99.0196



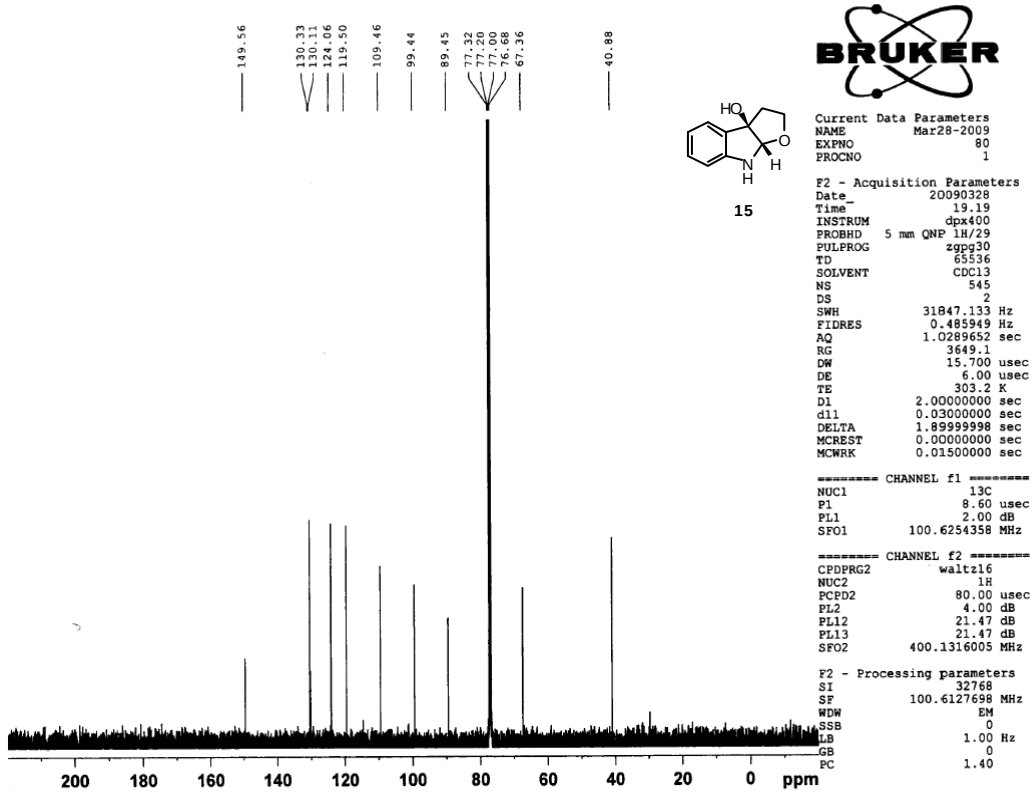
BRUKER

```
Current Data Parameters
NAME                  Mar28-2009
EXPNO                         72
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20090328
Time                       18.40
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                          4096
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                  400.1300082 MHz
WDW                          no
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.00
```



BRUKER

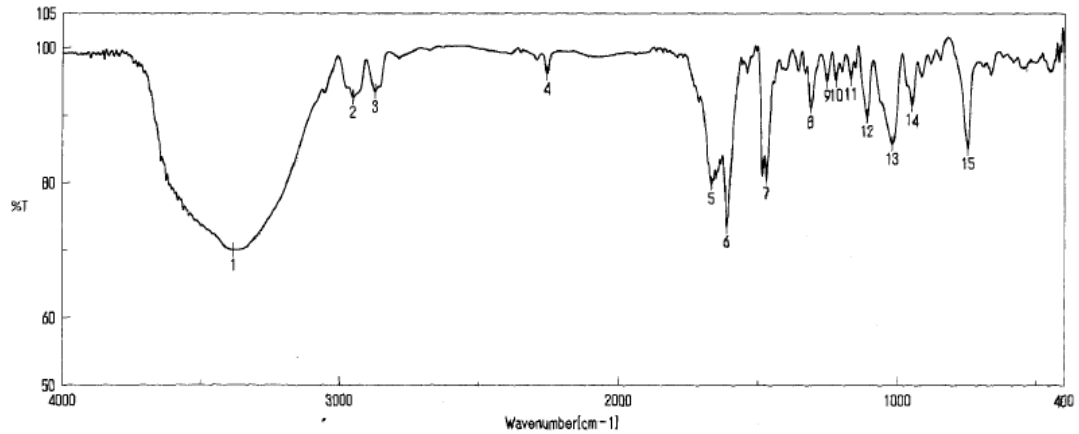
```
Current Data Parameters
NAME                  Mar28-2009
EXPNO                         80
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20090328
Time                       19.19
INSTRUM                   dpx400
PROBHD    5 mm QNP 1H/29
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           545
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

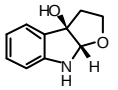
======== CHANNEL f1 ========
NUC1                         13C
P1                         8.60 usec
PL1                        2.00 dB
SFO1                100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                        4.00 dB
PL12                      21.47 dB
PL13                      21.47 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127698 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

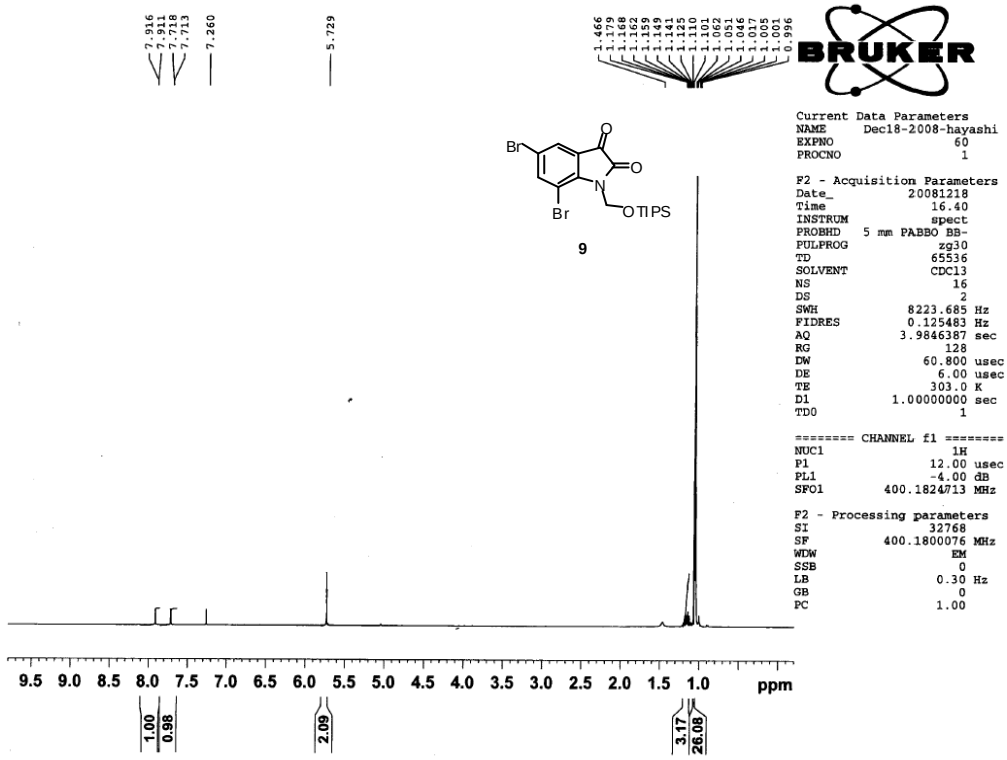


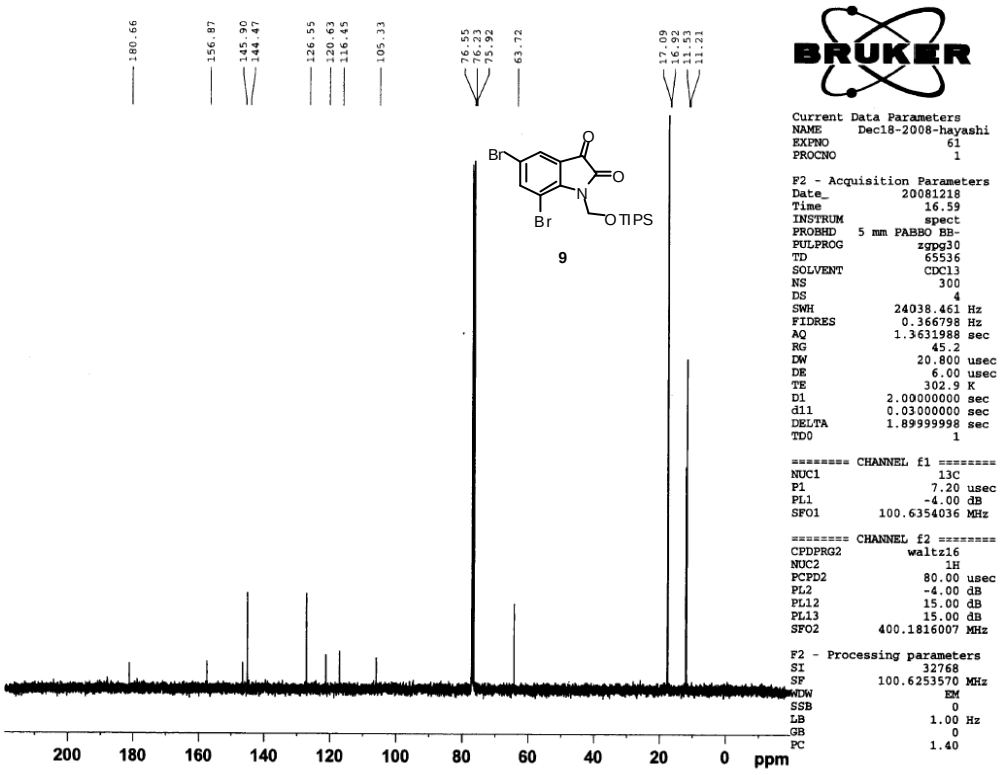
積算回数	32	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/04/23 18:43		
測定者			
ファイル名	Memory#5		
サンプル名	buckground		
コメント			

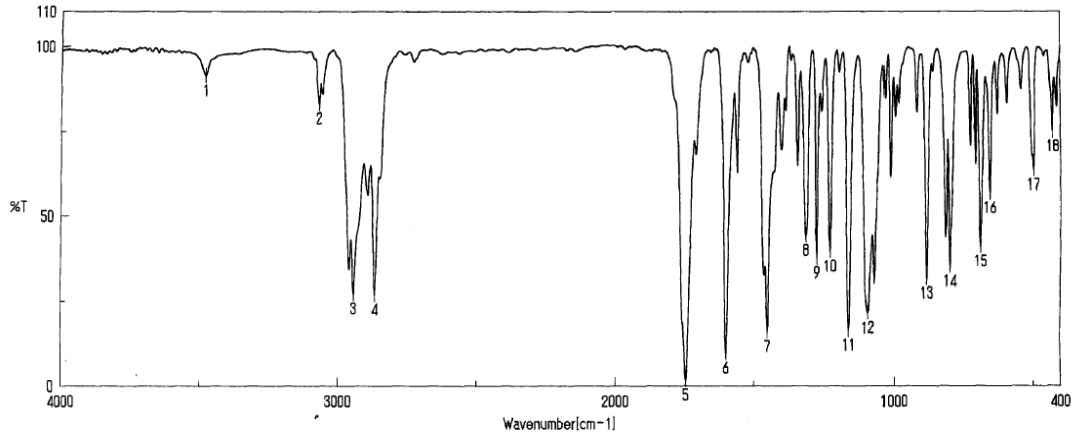


15

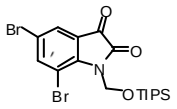
1:	3382.53,	70.0549	2:	2952.48,	92.7123	3:	2873.42,	93.5309	4:	2255.34,	96.2442
5:	1698.12,	79.9558	6:	1613.16,	73.5297	7:	1471.42,	80.7052	8:	1313.29,	91.2658
9:	1255.43,	95.1091	10:	1223.61,	95.2541	11:	1169.62,	95.4512	12:	1111.76,	89.8878
13:	1021.12,	85.8415	14:	949.77,	91.5903	15:	747.28,	85.1480			


7.916
7.911
7.718
7.713
7.260
5.729
1.466
1.179
1.168
1.162
1.159
1.149
1.141
1.125
1.110
1.101
1.062
1.051
1.046
1.017
1.005
1.001
0.996
BRUKER
Br
O
O
N
Br
OTIPS
9
Current Data Parameters
NAME Dec18-2008-hayashi
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081218
Time 16.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.00 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 400.1824713 MHz
F2 - Processing parameters
SI 32768
SF 400.1800076 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
1.00
0.98
2.09
3.17
26.08


180.66
156.87
145.90
144.47
126.55
120.63
116.45
105.33
76.55
76.23
75.92
63.72
17.09
16.92
11.53
11.21
BRUKER
Br
O
O
N
Br
OTIPS
9
Current Data Parameters
NAME Dec18-2008-hayashi
EXPNO 61
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081218
Time 16.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 45.2
DW 20.800 usec
DE 6.00 usec
TE 302.9 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 100.6253570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0
ppm

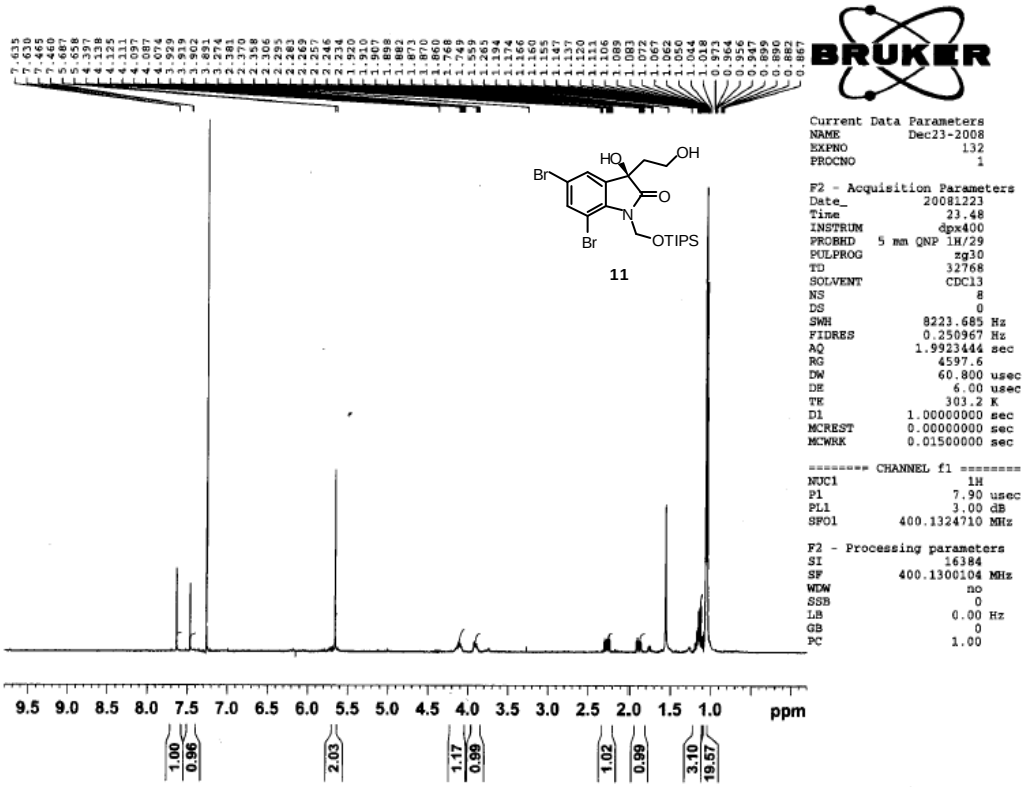


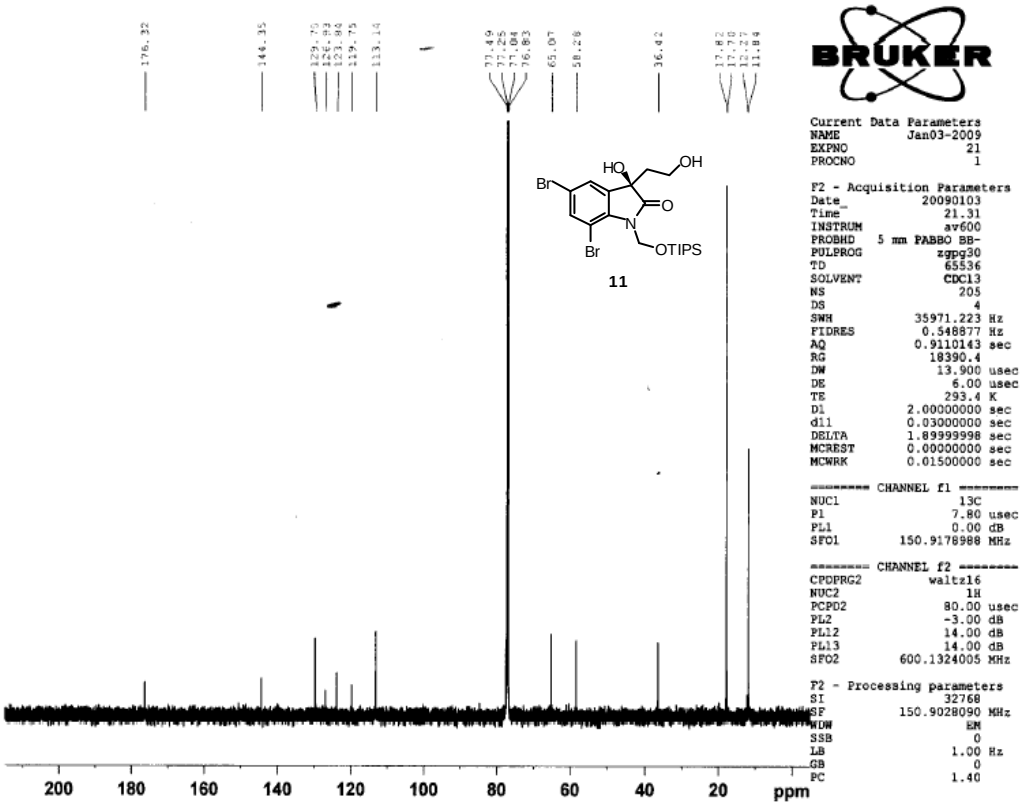
積算回数	64	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/01/03 15:06		
測定者			
ファイル名	diBrisatin(OTIPS) not natural.JWS		
サンプル名	buckground		
コメント			

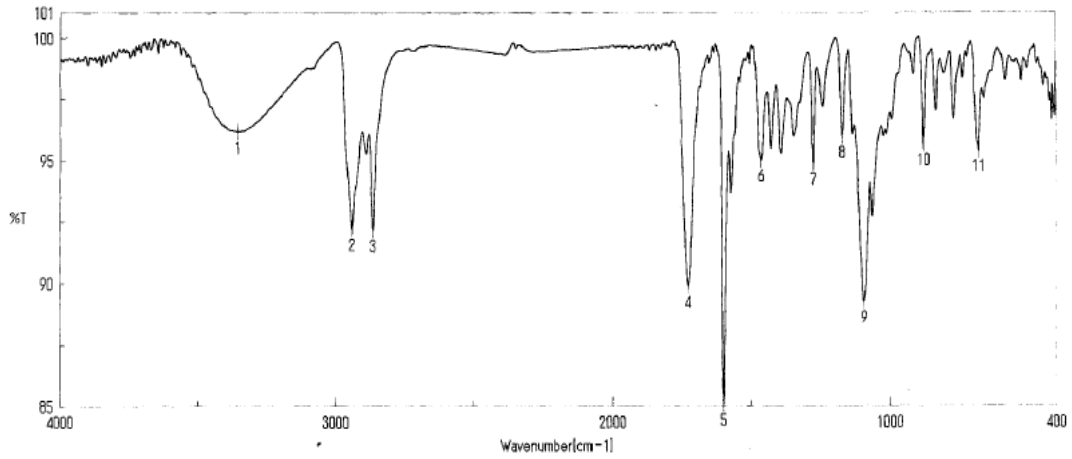


9

1:	3476.06,	91.4517	2:	3066.26,	83.2333	3:	2941.88,	27.3585	4:	2864.74,	27.0436
5:	1746.23,	1.8945	6:	1600.63,	9.7920	7:	1452.14,	16.2026	8:	1313.29,	44.5592
9:	1274.72,	37.7887	10:	1227.47,	39.7451	11:	1160.94,	16.6128	12:	1092.48,	21.6006
13:	884.20,	31.9686	14:	800.31,	35.3972	15:	689.43,	41.2033	16:	656.64,	56.9665
17:	499.47,	63.8390	18:	429.08,	75.1549						

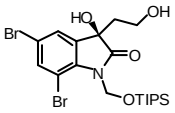

BRUKER
Current Data Parameters
NAME Dec23-2008
EXPNO 132
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081223
Time 23.48
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 4597.6
DW 60.800 usec
DE 6.00 usec
TE 303.2 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 400.1300104 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO
OH
Br
O
N
Br
OTIPS
11
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
1.00
0.96
2.03
1.17
0.99
1.02
0.99
3.10
19.57


BRUKER
Current Data Parameters
NAME Jan03-2009
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090103
Time 21.31
INSTRUM av600
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 18390.4
DW 13.900 usec
DE 6.00 usec
TE 293.4 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 7.80 usec
PL1 0.00 dB
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 14.00 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
176.32
144.35
129.73
126.83
123.96
119.75
113.14
77.49
77.25
77.04
76.83
65.07
58.28
36.42
17.82
17.70
17.27
11.84
HO
OH
Br
O
N
Br
OTIPS
11
200 180 160 140 120 100 80 60 40 20
ppm



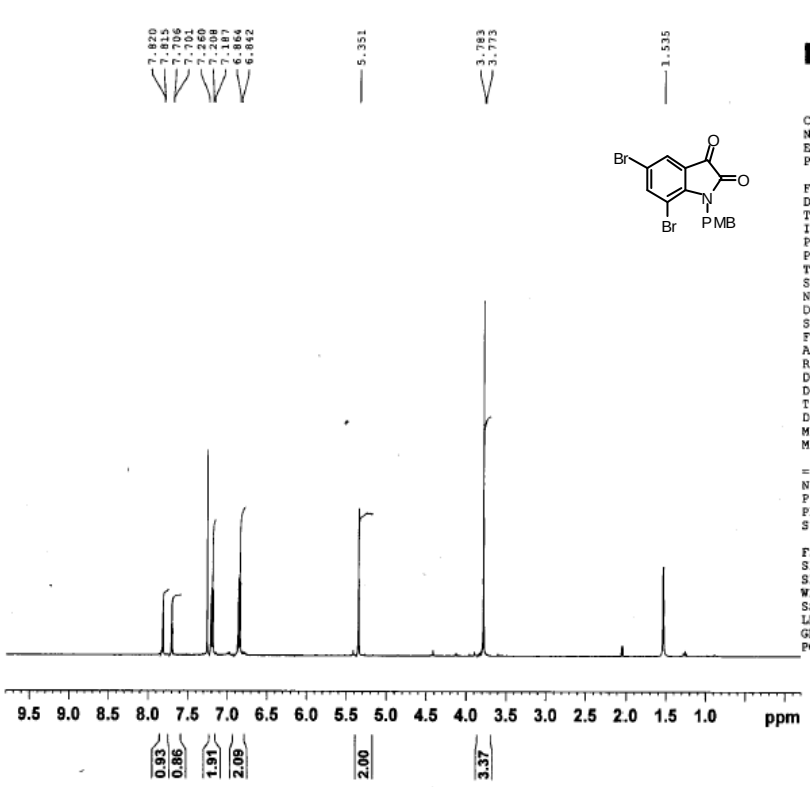
積算回数	64
ゼロフィリング	ON
ゲイン	2
日時	109/01/03 23:06
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

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



11

1:	3356.50,	96.1773	2:	2941.88,	92.2225	3:	2864.74,	92.1813	4:	1725.98,	89.8775
5:	1598.70,	85.0725	6:	1464.67,	95.0076	7:	1278.57,	94.8629	8:	1174.44,	95.9840
9:	1093.44,	89.2615	10:	881.31,	95.6806	11:	683.64,	95.3996			



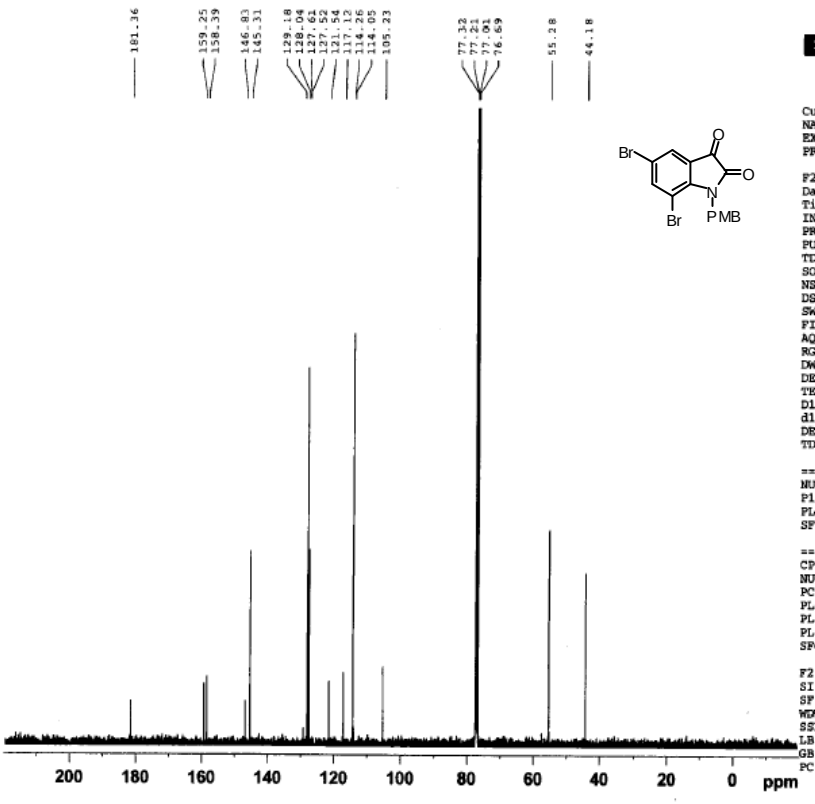
BRUKER

```
Current Data Parameters
NAME                  Apr23-2009
EXPNO                        114
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20090423
Time                     18.02
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                     10321.3
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                    10.70 usec
PL1                    4.00 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                       16384
SF                400.1300092 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                     1.00
```



BRUKER

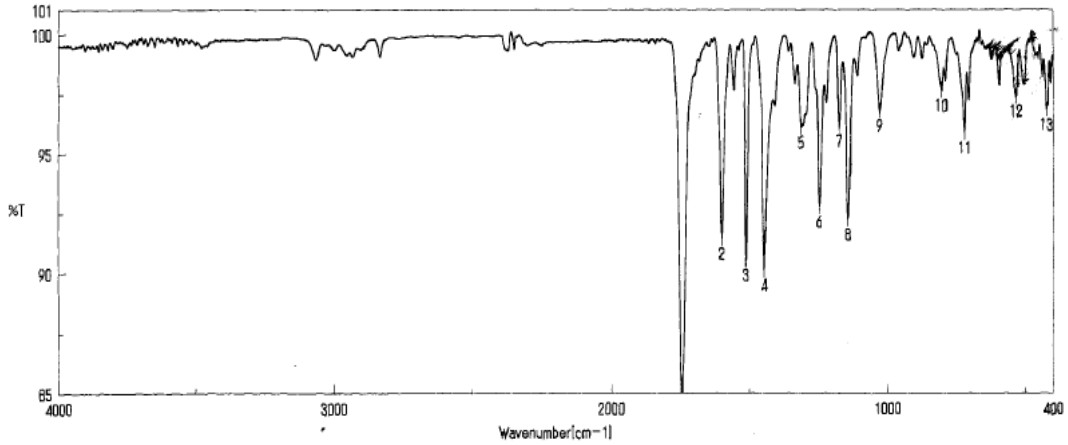
```
Current Data Parameters
NAME          Apr23-2009-hayashi
EXPNO                        150
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20090423
Time                     19.20
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                         228
DW                    20.800 usec
DE                     6.00 usec
TE                     302.9 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                            1

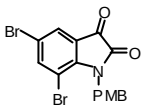
======== CHANNEL f1 ========
NUC1                      13C
P1                     7.20 usec
PL1                   -3.50 dB
SFO1             100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.50 dB
PL13                  14.50 dB
SFO2             400.1816007 MHz

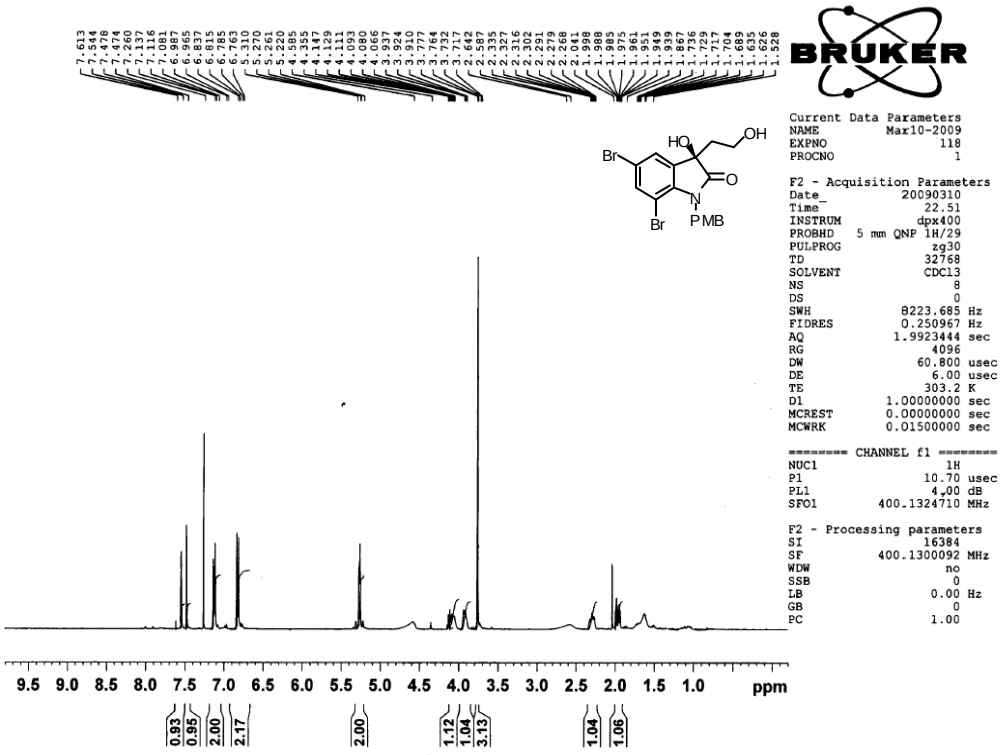
F2 - Processing parameters
SI                       32768
SF                100.6253411 MHz
WDW                         EM
SSB                          0
LB                     1.00 Hz
GB                           0
PC                     1.40
```



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	2	スキャンスピード	2 mm/sec
日時	109/04/23 18:36		
測定者			
ファイル名	Memory#3		
サンプル名	buckground		
コメント			



1:	1744.30,	85.0069	2:	1600.63,	91.4791	3:	1513.85,	90.5878	4:	1447.31,	90.1976
5:	1315.21,	96.1531	6:	1247.72,	92.8201	7:	1178.29,	96.2131	8:	1144.55,	92.2825
9:	1031.73,	96.8260	10:	808.03,	97.6855	11:	724.14,	95.9124	12:	537.06,	97.4495
13:	425.23,	96.8853									



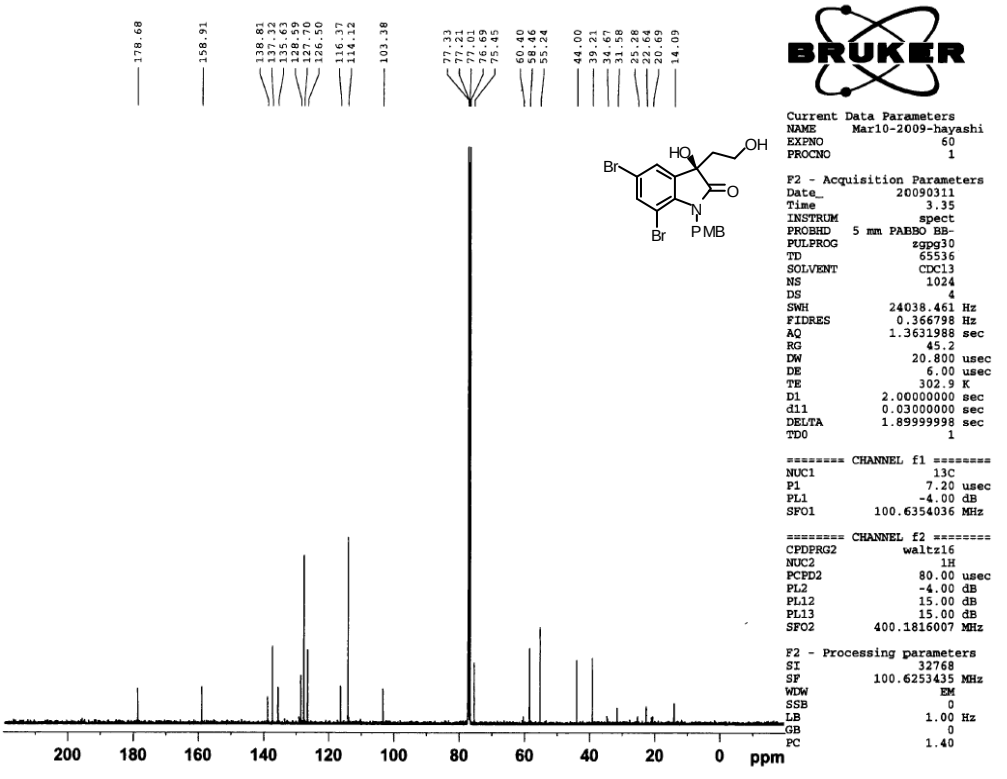
BRUKER

```
Current Data Parameters
NAME                  Mar10-2009
EXPNO                        118
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20090310
Time                     22.51
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                        4096
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                       10.70 usec
PL1                       4.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                       16384
SF                  400.1300092 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00
```



BRUKER

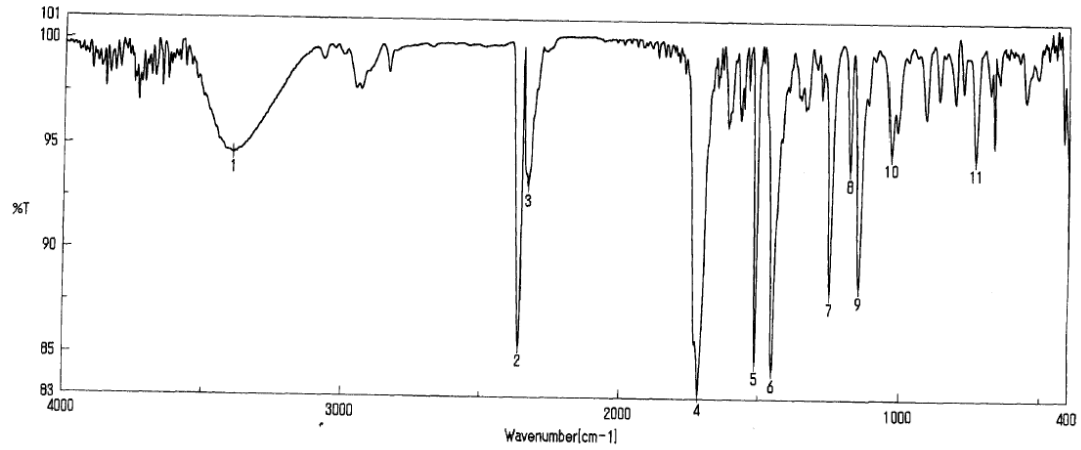
```
Current Data Parameters
NAME            Mar10-2009-hayashi
EXPNO                           60
PROCNO                           1

F2 - Acquisition Parameters
Date_                 20090311
Time                      3.35
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                         45.2
DW                      20.800 usec
DE                       6.00 usec
TE                      302.9 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

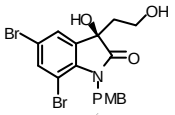
======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                      -4.00 dB
PL12                     15.00 dB
PL13                     15.00 dB
SFO2               400.1816007 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6253435 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

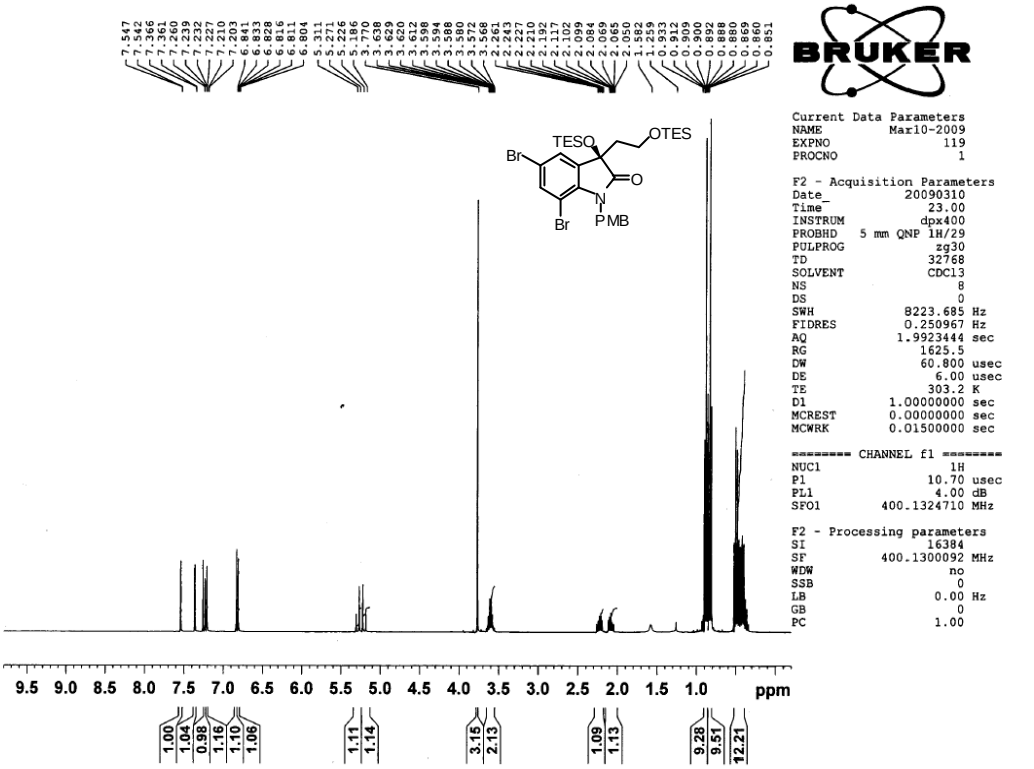


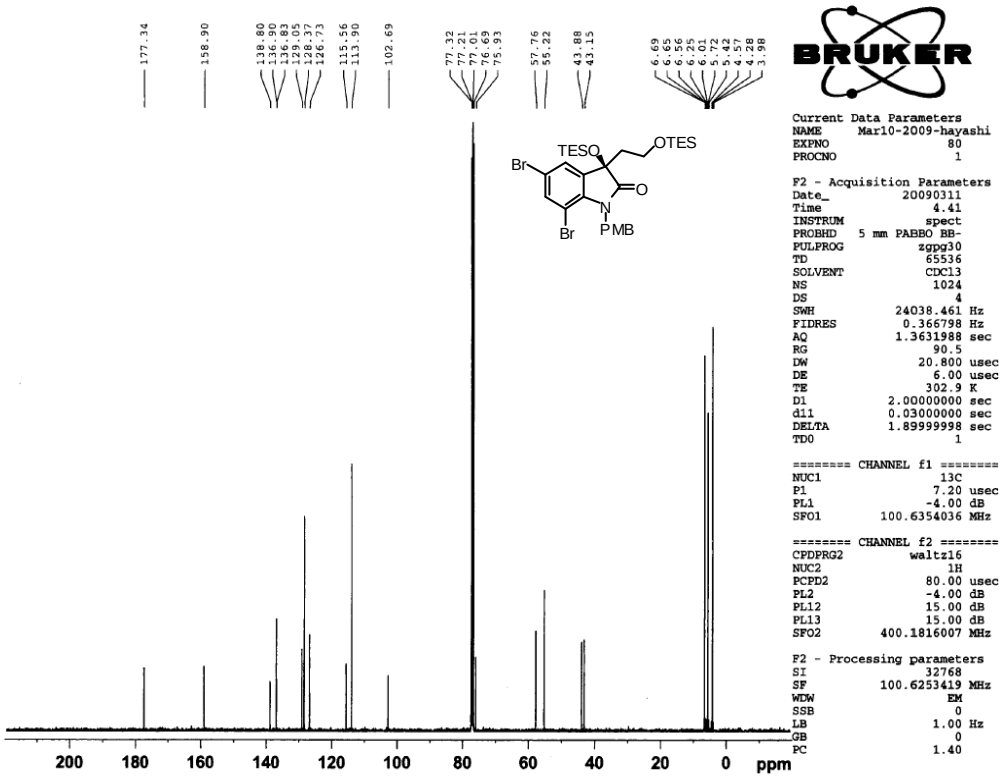
積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/11 20:03
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

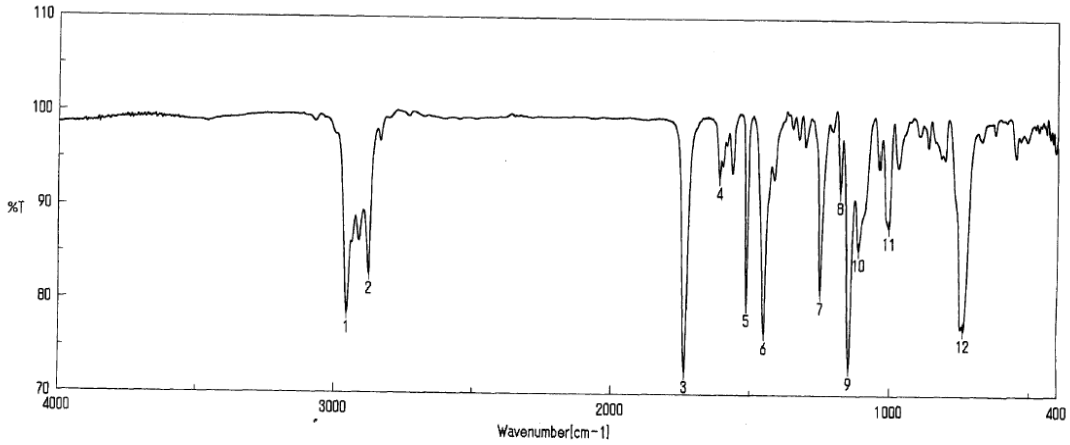
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec



1:	3393.14,	94.5952	2:	2362.37,	85.4855	3:	2330.55,	93.1385	4:	1716.34,	83.2270
5:	1513.85,	84.7556	6:	1452.14,	84.3838	7:	1247.72,	88.0958	8:	1177.33,	93.9836
9:	1144.55,	88.3948	10:	1033.66,	94.7545	11:	735.71,	94.5325			

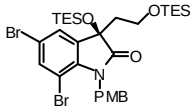

7.547 7.542 7.366 7.361 7.260 7.239 7.232 7.227 7.210 7.203 6.841 6.833 6.828 6.816 6.811 6.804 5.311 5.271 5.226 5.186 3.770 3.638 3.629 3.620 3.612 3.598 3.594 3.588 3.580 3.572 3.568 2.261 2.243 2.227 2.210 2.192 2.117 2.102 2.099 2.084 2.069 2.065 2.050 1.582 1.259 0.933 0.912 0.909 0.900 0.892 0.888 0.880 0.869 0.860 0.851
TESO
OTES
Br
O
N
Br
PMB
Current Data Parameters
NAME Mar10-2009
EXPNO 119
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090310
Time 23.00
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 1625.5
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW no
SSB 0
LB 0.00 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
1.00 1.04 0.98 1.16 1.10 1.06 1.11 1.14 3.15 2.13 1.09 1.13 9.28 9.51 12.21


177.34 158.90 138.80 136.90 136.83 130.05 128.37 126.73 115.56 113.90 102.69 77.32 77.21 77.01 76.69 75.93 57.76 55.22 43.88 43.15 6.69 6.65 6.56 6.25 6.01 5.72 5.42 4.57 4.28 3.98
TESO
OTES
Br
O
N
Br
PMB
Current Data Parameters
NAME Mar10-2009-hayashi
EXPNO 80
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090311
Time 4.41
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 90.5
DW 20.800 usec
DE 6.00 usec
TE 302.9 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 100.6253419 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

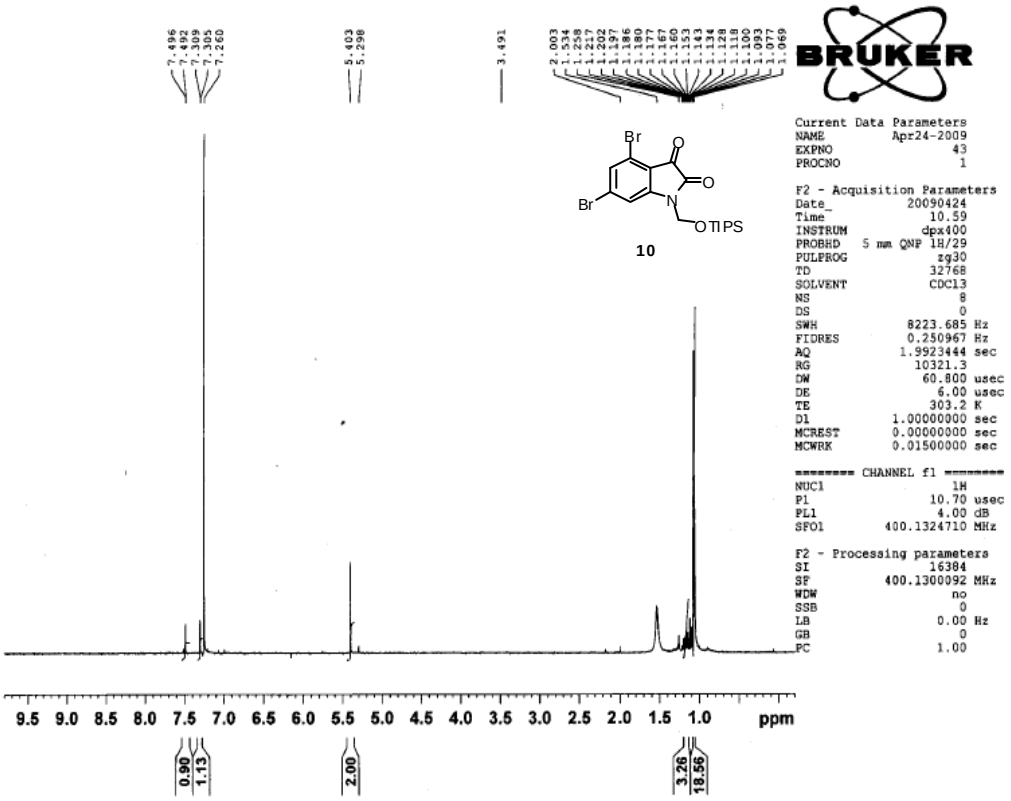


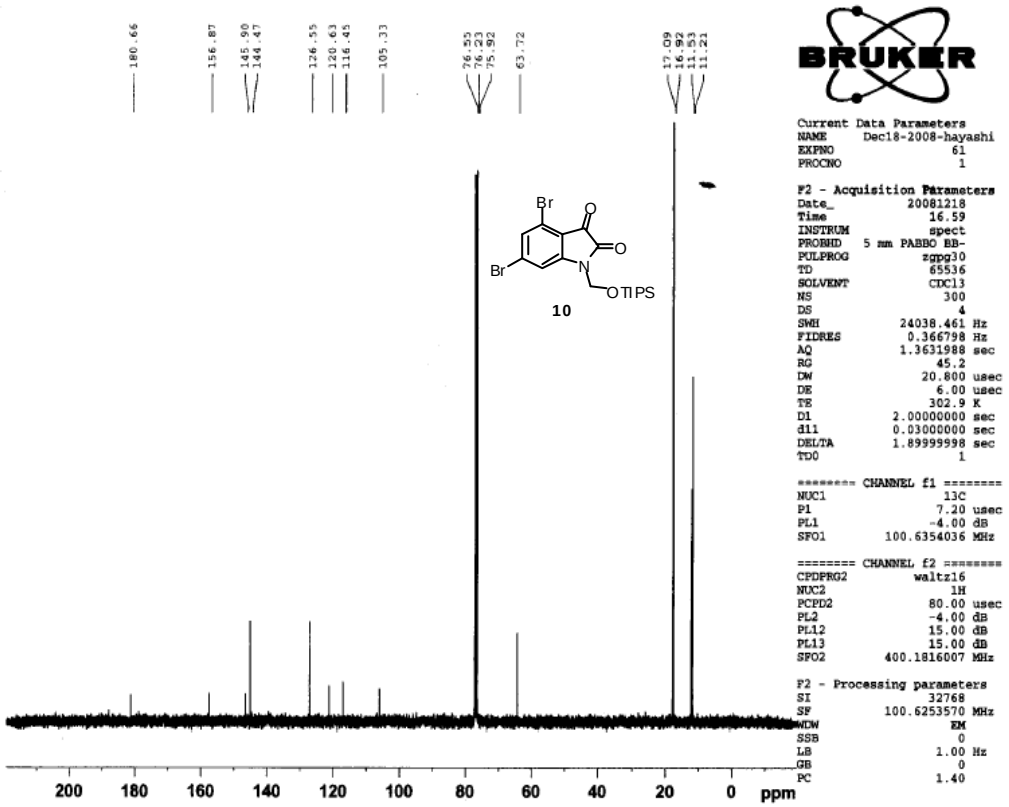
積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/11 20:19
測定者	
ファイル名	Memory#4
サンプル名	buckground
コメント	

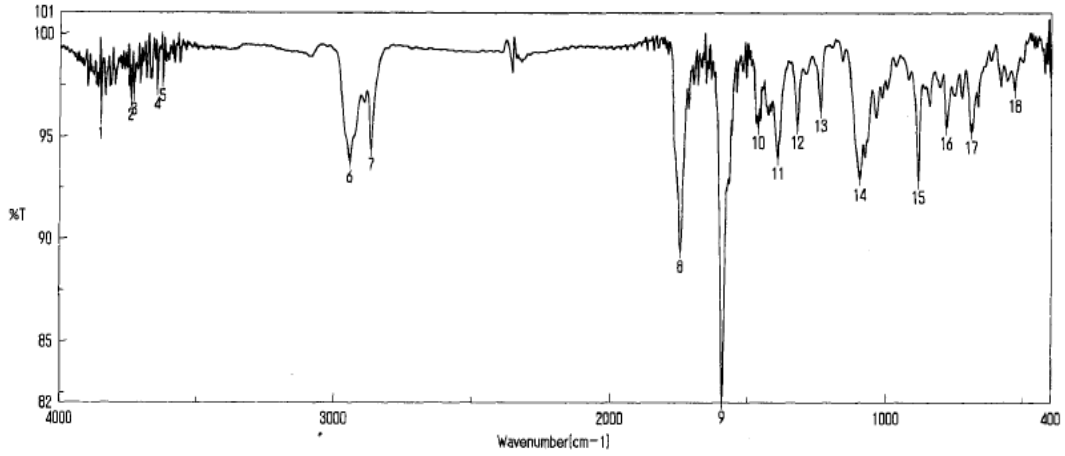
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec



1:	2954.41, 78.5649	2:	2875.34, 82.7350	3:	1735.62, 72.4620	4:	1613.16, 93.1573
5:	1513.85, 79.6046	6:	1450.21, 76.6440	7:	1247.72, 80.9638	8:	1177.33, 91.6334
9:	1144.55, 72.9468	10:	1110.80, 85.6001	11:	1005.70, 87.8985	12:	736.67, 77.0784

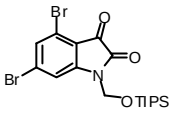

7.496
7.492
7.309
7.305
7.260
5.403
5.298
3.491
2.003
1.534
1.258
1.217
1.202
1.197
1.186
1.180
1.177
1.167
1.160
1.153
1.143
1.134
1.124
1.118
1.100
1.093
1.077
1.069
Br
O
O
N
Br
OTIPS
10
BRUKER
Current Data Parameters
NAME Apr24-2009
EXPNO 43
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090424
Time 10.59
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 10321.3
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW no
SSB 0
LB 0.00 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
0.90
1.13
2.00
3.26
18.56


180.66
156.87
145.90
144.47
126.55
120.63
116.45
105.33
76.55
76.23
75.92
63.72
17.09
16.92
11.53
11.21
Br
O
O
N
Br
OTIPS
10
BRUKER
Current Data Parameters
NAME Dec18-2008-hayashi
EXPNO 61
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081218
Time 16.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 45.2
DW 20.800 usec
DE 6.00 usec
TE 302.9 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 100.6253570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



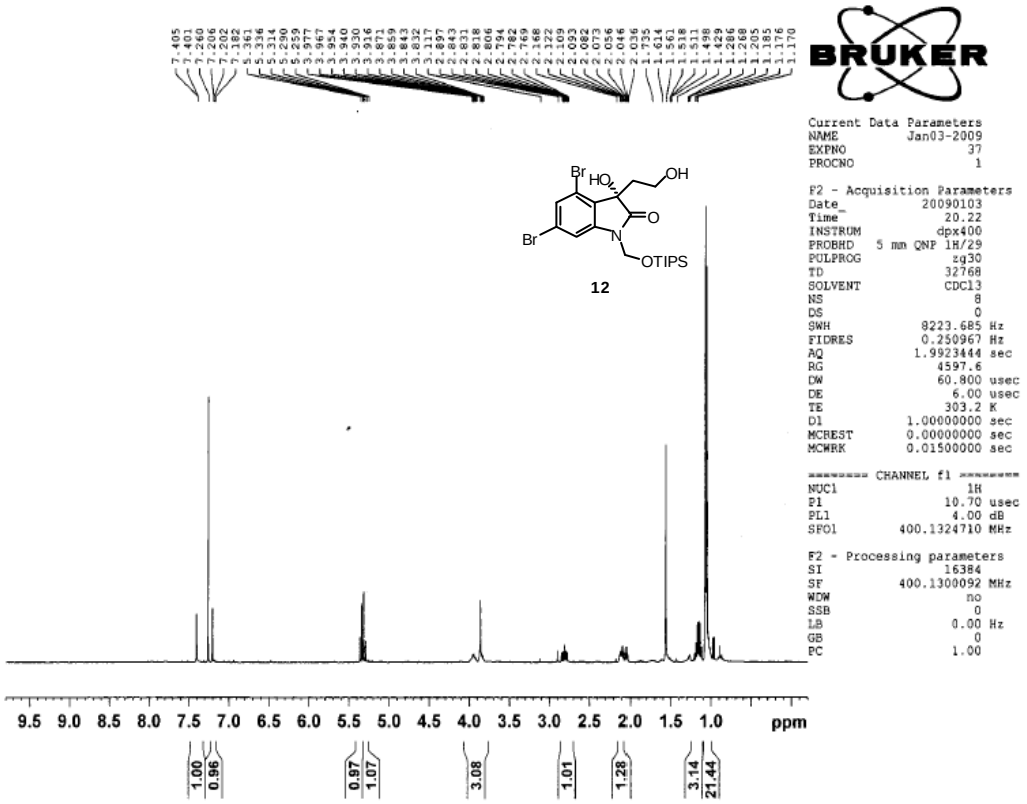
積算回数	64
ゼロフィリング	ON
ゲイン	2
日時	109/01/03 14:49
測定者	
ファイル名	diBrisatin(OTIPS) natural.JWS
サンプル名	buckground
コメント	

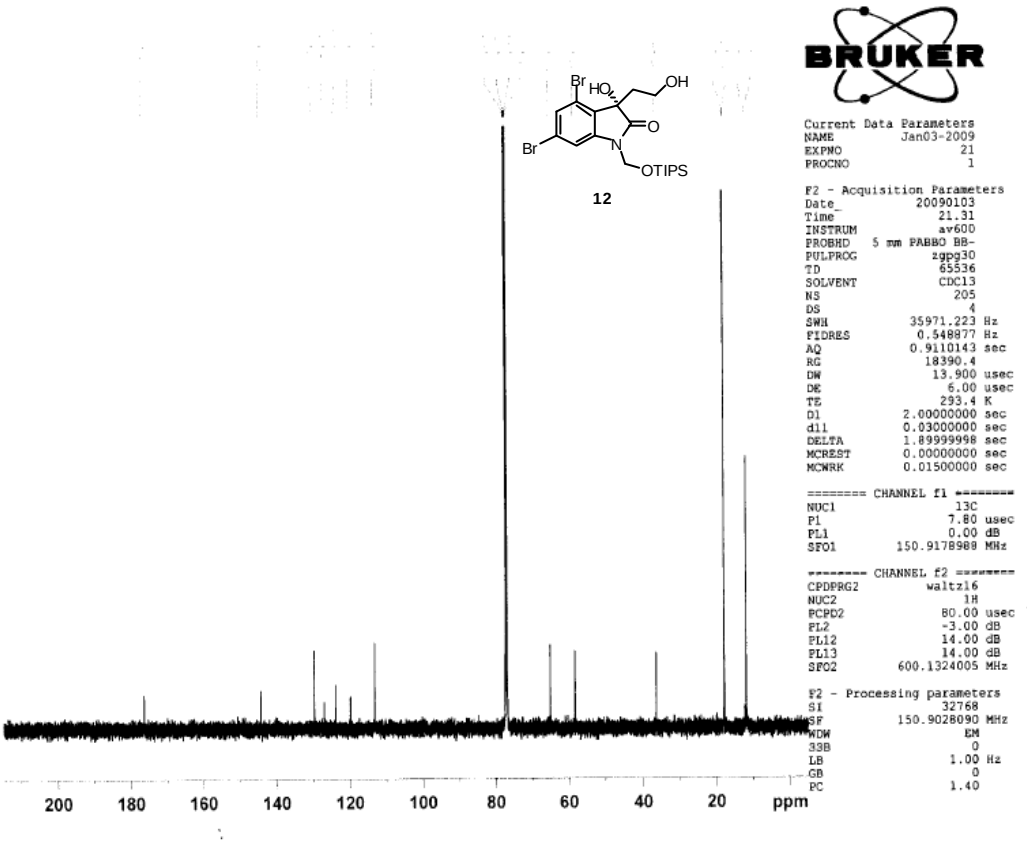
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec

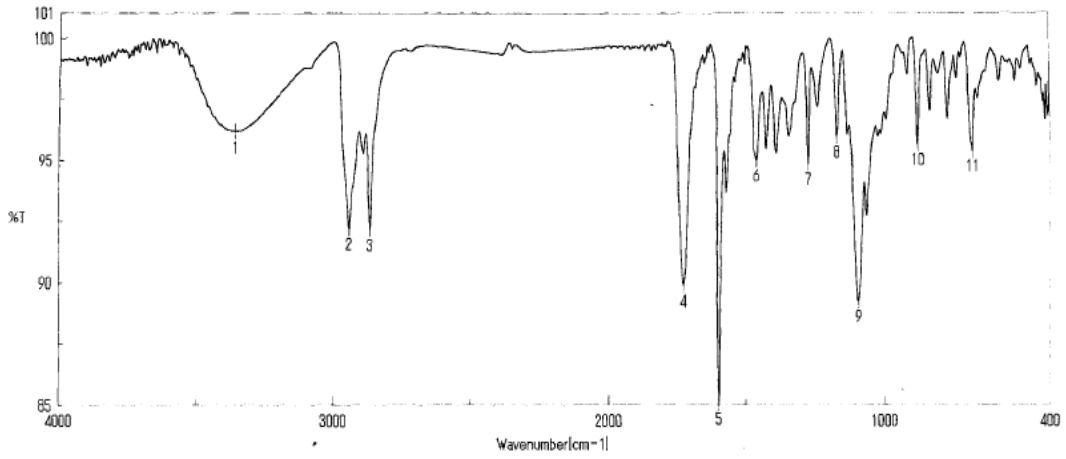


10

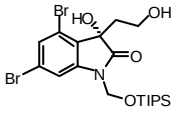
1:	3850.18,	95.8923	2:	3741.23,	96.8167	3:	3730.62,	97.0873	4:	3645.77,	97.3724
5:	3625.52,	97.7652	6:	2942.84,	93.8090	7:	2865.70,	94.3987	8:	1745.26,	89.4255
9:	1591.95,	82.0631	10:	1462.74,	95.4954	11:	1393.32,	93.9870	12:	1321.96,	95.5032
13:	1239.04,	96.3097	14:	1096.33,	92.9626	15:	883.24,	92.8413	16:	780.06,	95.4653
17:	689.43,	95.2644	18:	532.26,	97.2834						


7.405 7.401 7.260 7.206 7.202 7.182 5.361 5.336 5.314 5.290 5.259 5.237 3.967 3.954 3.940 3.930 3.916 3.871 3.859 3.845 3.832 3.117 2.897 2.843 2.831 2.818 2.806 2.794 2.782 2.769 2.168 2.122 2.109 2.093 2.082 2.073 2.056 2.046 2.036 1.735 1.614 1.601 1.518 1.511 1.498 1.429 1.286 1.268 1.205 1.185 1.176 1.170
Br HO OH O N OTIPS Br
12
Current Data Parameters
NAME Jan03-2009
EXPNO 37
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090103
Time 20.22
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 4597.6
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW no
SSB 0
LB 0.00 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
1.00 0.96 0.97 1.07 3.08 1.01 1.28 3.14 21.44


Br HO OH O N OTIPS Br
12
Current Data Parameters
NAME Jan03-2009
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090103
Time 21.31
INSTRUM av600
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 18390.4
DW 13.900 usec
DE 6.00 usec
TE 293.4 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 7.80 usec
PL1 0.00 dB
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 14.00 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
200 180 160 140 120 100 80 60 40 20 ppm

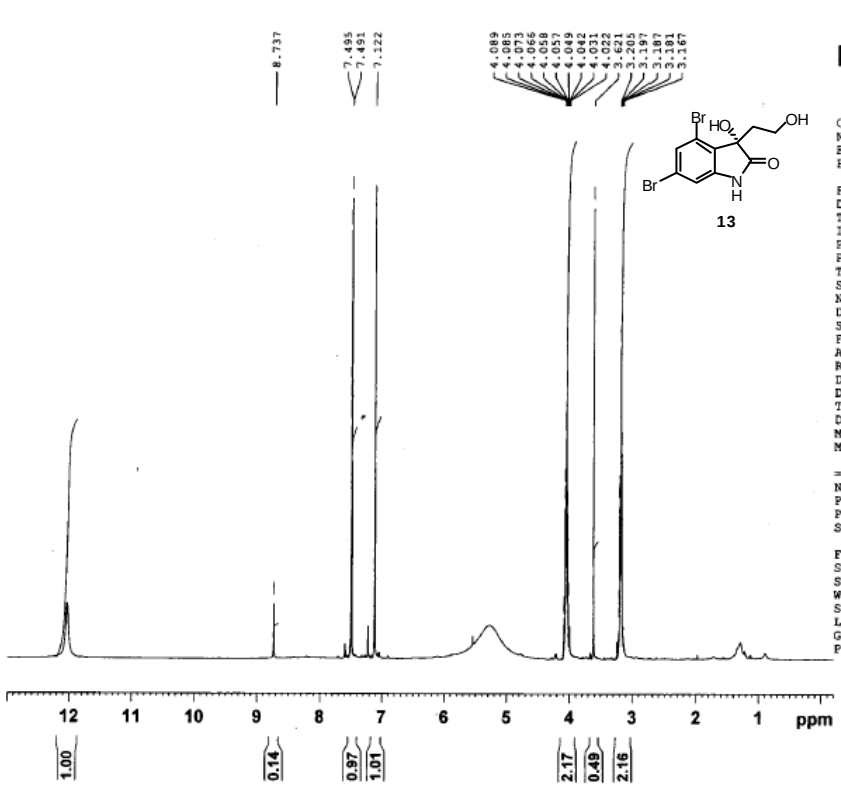


積算回数	64	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/01/03 23:06		
測定者			
ファイル名	Memory#3		
サンプル名	buckground		
コメント			



12

1:	3356.50,	96.1773	2:	2941.88,	92.2225	3:	2864.74,	92.1813	4:	1725.98,	89.8775
5:	1598.70,	85.0725	6:	1464.67,	95.0076	7:	1278.57,	94.8629	8:	1174.44,	95.9840
9:	1093.44,	89.2615	10:	881.31,	95.6806	11:	683.84,	95.3996			



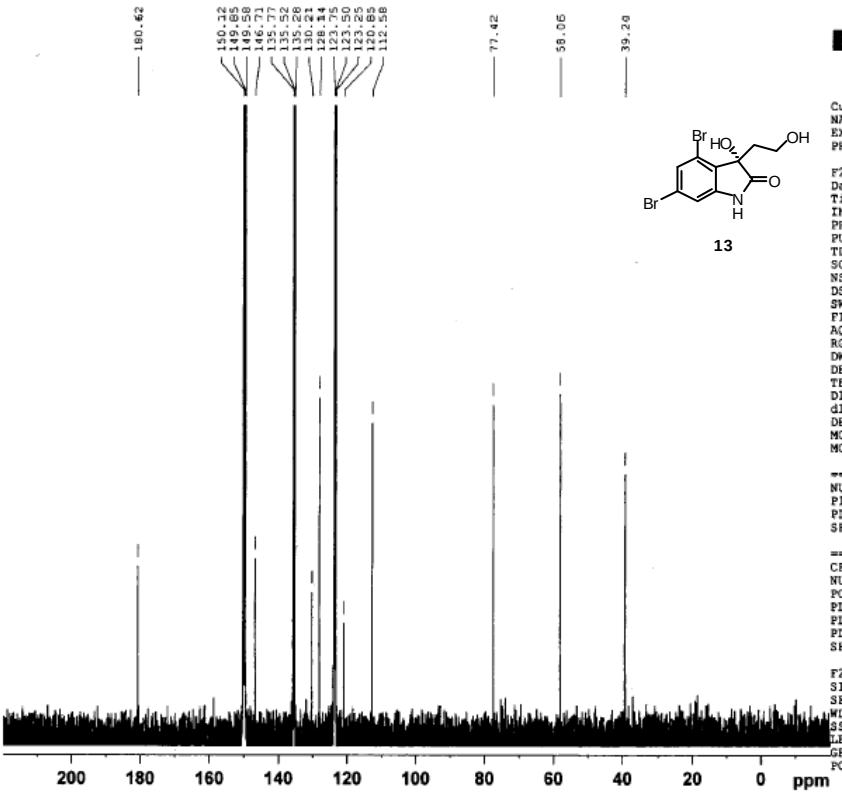
BRUKER

```
Current Data Parameters
NAME                  Mar31-2009
EXPNO                         30
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20090331
Time                      15.58
INSTRUM                  dpx400
PROBHD    5 mm QNP 1H/29
PULPROG                   zg30
TD                       32768
SOLVENT                    Pyr
NS                          32
DS                           0
SWH                   8223.685 Hz
FIDRES                0.250967 Hz
AQ                    1.9923444 sec
RG                       2580.3
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                       10.70 usec
PL1                       4.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                       16384
SF                  400.1305874 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



BRUKER

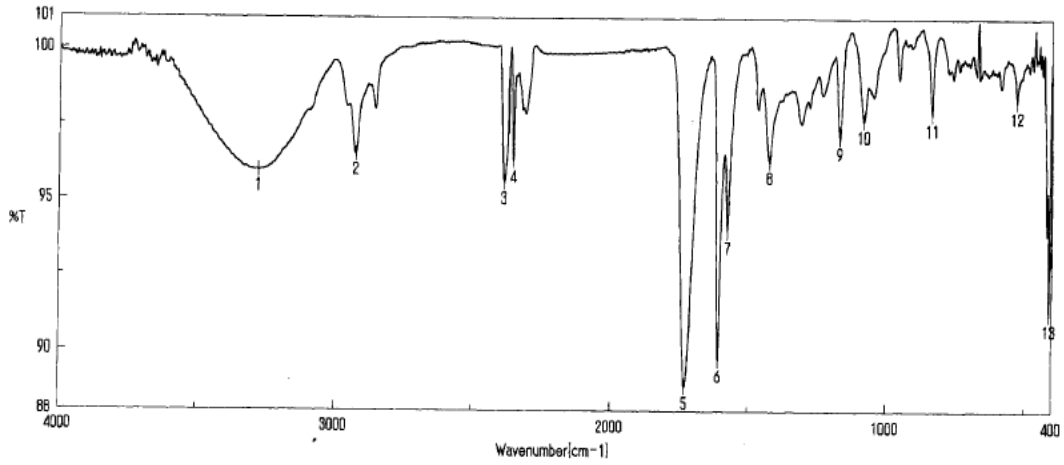
```
Current Data Parameters
NAME                  Mar31-2009
EXPNO                         31
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20090331
Time                      16.11
INSTRUM                  dpx400
PROBHD    5 mm QNP 1H/29
PULPROG                 zgpg30
TD                       65536
SOLVENT                    Pyr
NS                         180
DS                           2
SWH                  31847.133 Hz
FIDRES                0.485949 Hz
AQ                    1.0289652 sec
RG                        7298.2
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

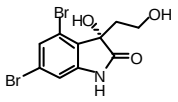
======== CHANNEL f1 ========
NUC1                        13C
P1                        8.60 usec
PL1                       2.00 dB
SFO1                100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       4.00 dB
PL12                     21.47 dB
PL13                     21.47 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                  100.6128973 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

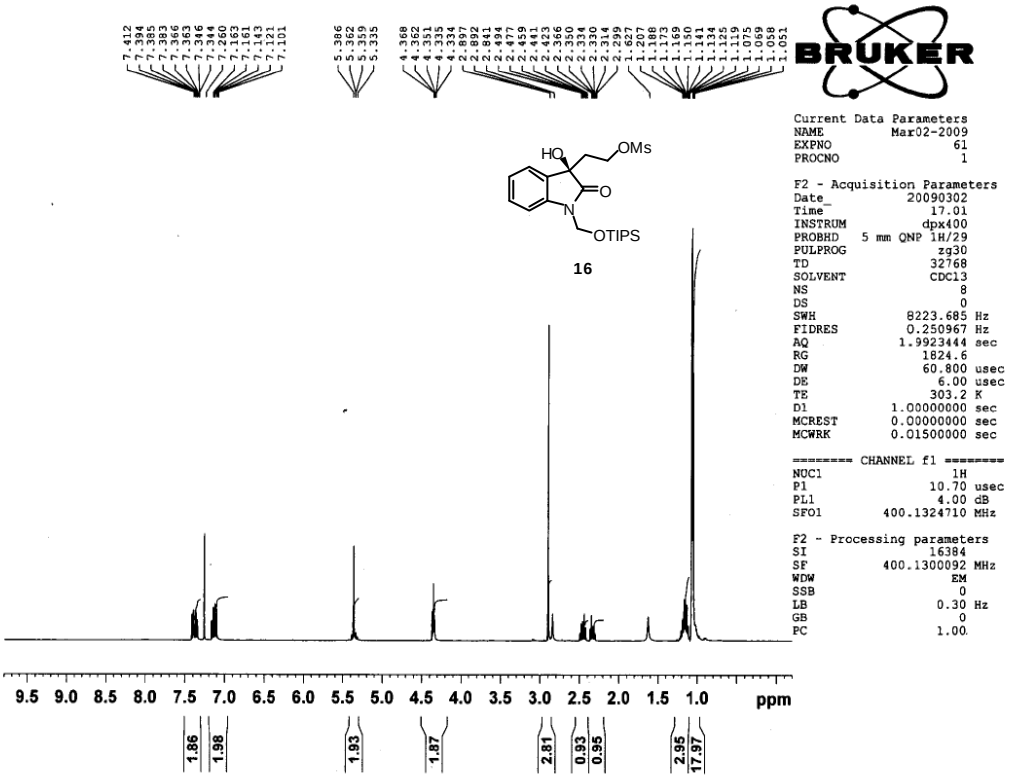


積算回数	16	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	1	スキャンスピード	2 mm/sec
日時	109/03/31 15:35		
測定者			
ファイル名	Memory#4		
サンプル名	buckground		
コメント			



13

1:	3275.50,	95.9556	2:	2923.56,	96.5016	3:	2380.89,	95.5681	4:	2348.87,	96.2823
5:	1729.83,	88.8083	6:	1607.38,	89.6934	7:	1574.59,	93.9574	8:	1424.17,	96.2745
9:	1168.65,	97.0914	10:	1082.83,	97.6470	11:	836.95,	97.8771	12:	527.44,	96.2881
13:	498.83,	91.2279									



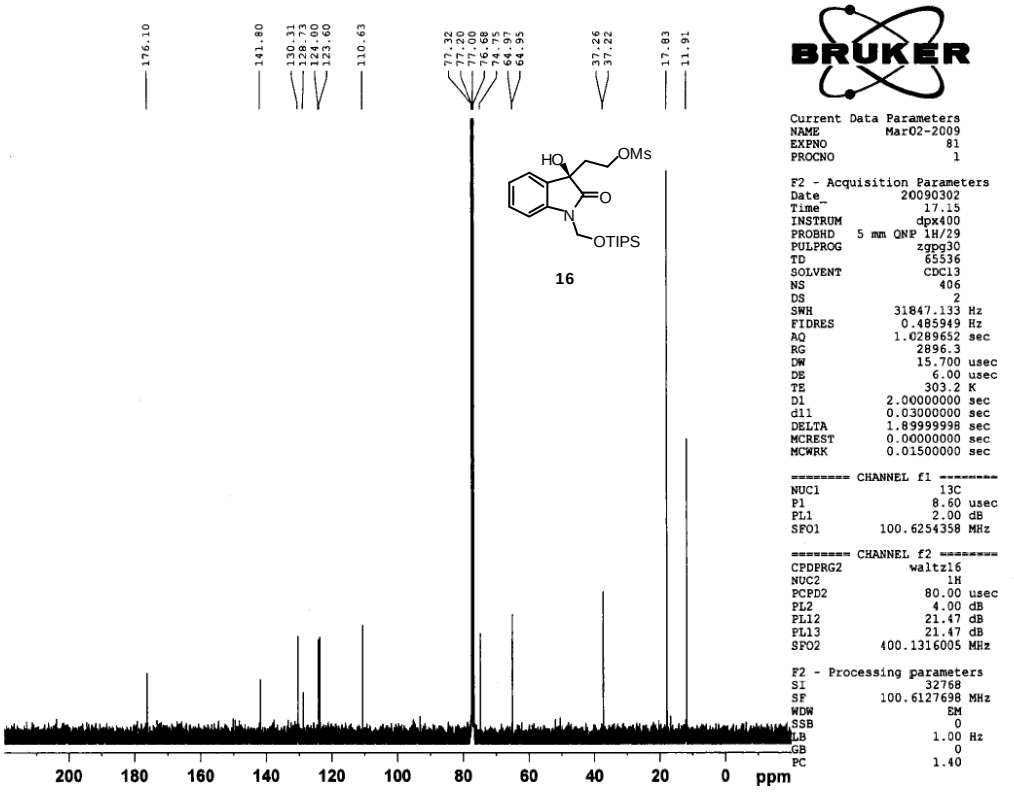
BRUKER

```
Current Data Parameters
NAME                  Mar02-2009
EXPNO                         61
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20090302
Time                       17.01
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                         1824.6
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                        10.70 usec
PL1                        4.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                         16384
SF                  400.1300092 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



BRUKER

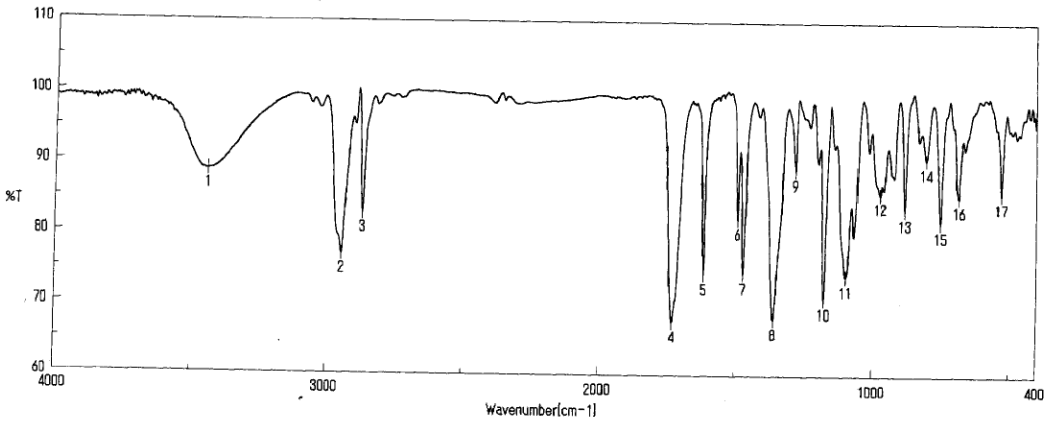
```
Current Data Parameters
NAME                  Mar02-2009
EXPNO                         81
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20090302
Time                       17.15
INSTRUM                  dpx400
PROBHD     5 mm QNP 1H/29
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           406
DS                             2
SWH                    31847.133 Hz
FIDRES                  0.485949 Hz
AQ                      1.0289652 sec
RG                         2896.3
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                         8.60 usec
PL1                        2.00 dB
SFO1                100.6254358 MHz

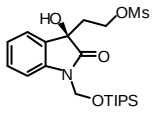
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                        4.00 dB
PL12                      21.47 dB
PL13                      21.47 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127698 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



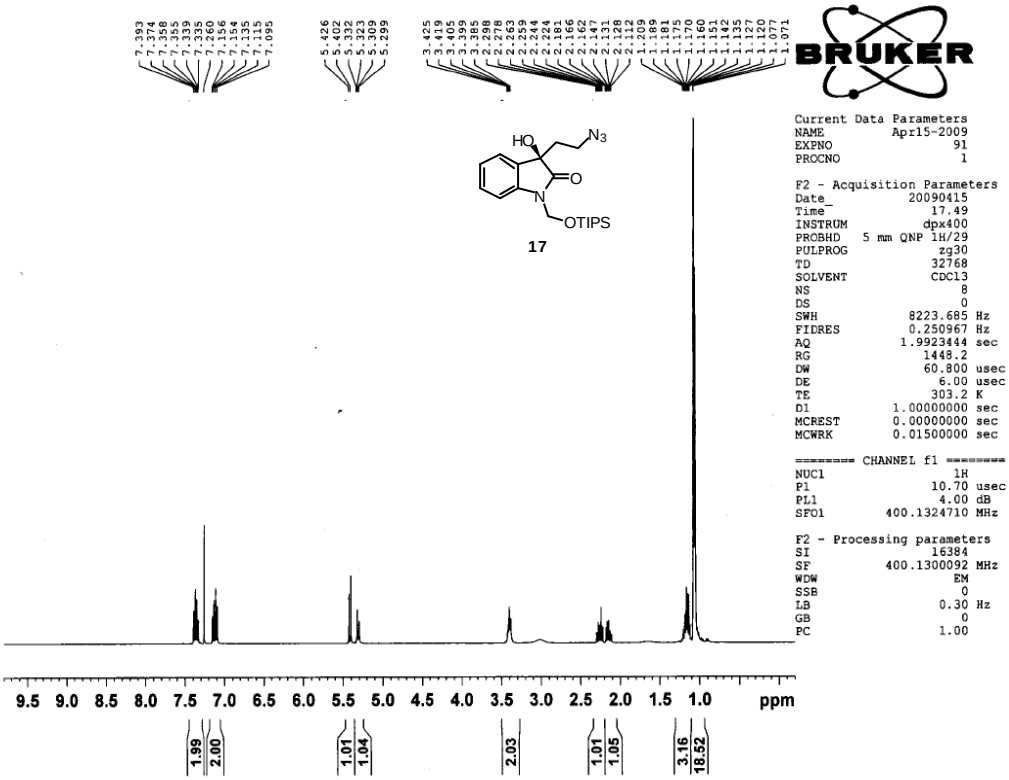
積算回数	16
ゼロフィリング	ON
ゲイン	1
日時	109/03/09 20:36
測定者	
ファイル名	Memory#3
サンプル名	background
コメント	

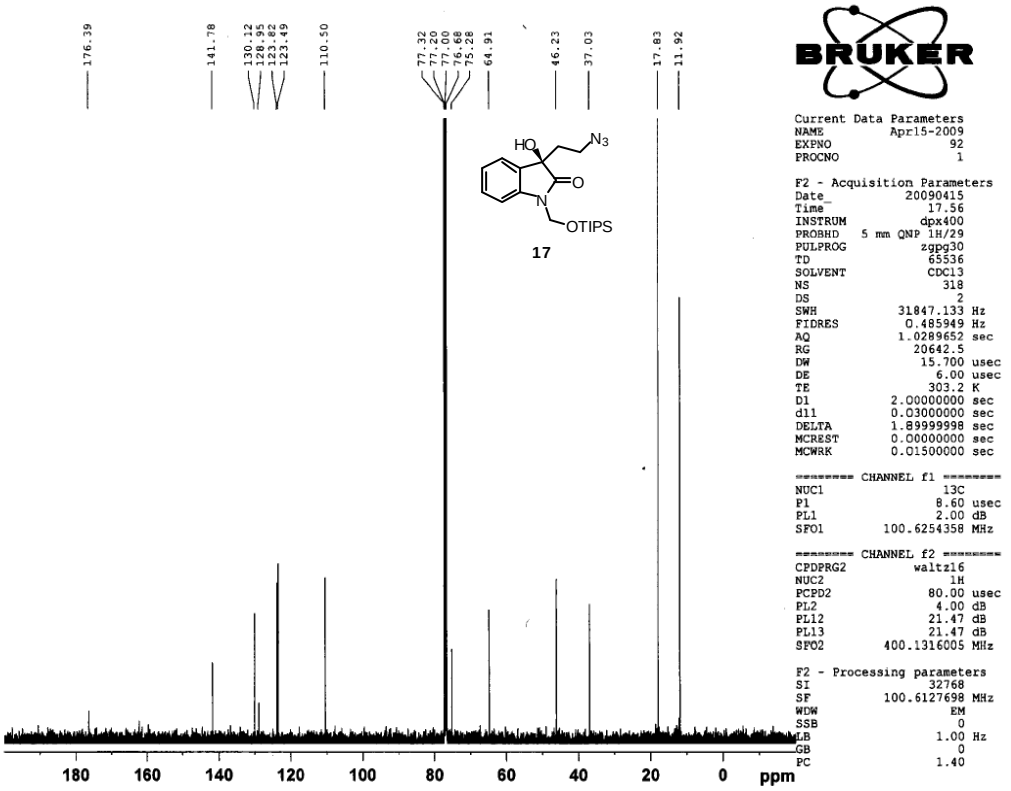
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec

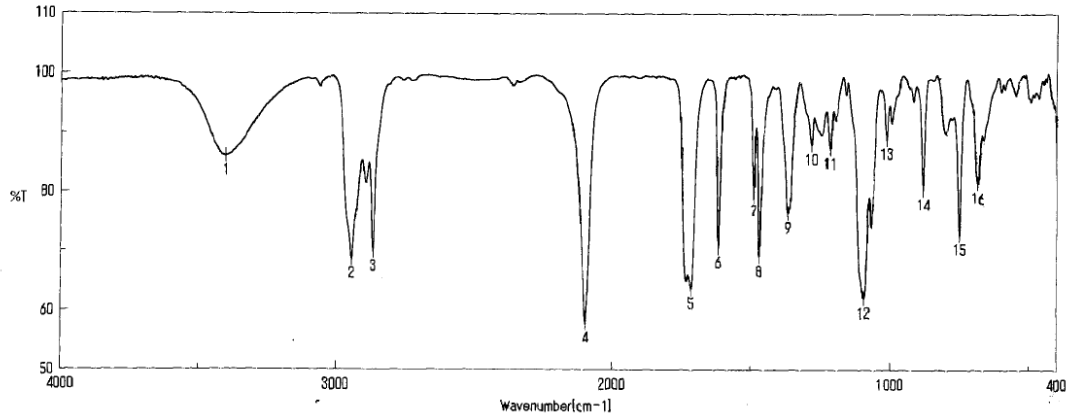


16

1:	3437.49,	88.7907	2:	2944.77,	76.9865	3:	2866.67,	82.8222	4:	1730.80,	67.6003
5:	1615.09,	74.3235	6:	1489.74,	82.1824	7:	1469.49,	74.4732	8:	1359.57,	68.0199
9:	1280.50,	89.1570	10:	1175.40,	70.8730	11:	1094.41,	74.1168	12:	973.88,	85.8708
13:	883.24,	83.5410	14:	806.10,	90.7340	15:	752.10,	81.8160	16:	684.61,	85.3872
17:	528.40,	85.9095									

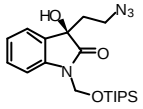

7.393 7.374 7.368 7.355 7.339 7.335 7.260 7.156 7.154 7.135 7.115 7.095
5.426 5.402 5.332 5.323 5.309 5.299
3.425 3.419 3.405 3.399 3.385 2.298 2.278 2.263 2.259 2.244 2.224 2.181 2.166 2.162 2.147 2.131 2.128 2.112 2.109 1.189 1.181 1.175 1.170 1.160 1.151 1.142 1.135 1.127 1.120 1.077 1.071
HO
N3
O
N
OTIPS
17
BRUKER
Current Data Parameters
NAME Apr15-2009
EXPNO 91
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090415
Time 17.49
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 1448.2
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 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
1.99 2.00 1.01 1.04 2.03 1.01 1.05 3.16 18.52


176.39
141.78
130.12
128.95
123.62
123.49
110.50
77.32
77.20
77.00
76.68
75.28
64.91
46.23
37.03
17.83
11.92
HO
N3
O
N
OTIPS
17
BRUKER
Current Data Parameters
NAME Apr15-2009
EXPNO 92
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090415
Time 17.56
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 318
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 20642.5
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 8.60 usec
PL1 2.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 21.47 dB
PL13 21.47 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127698 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 160 140 120 100 80 60 40 20 0 ppm



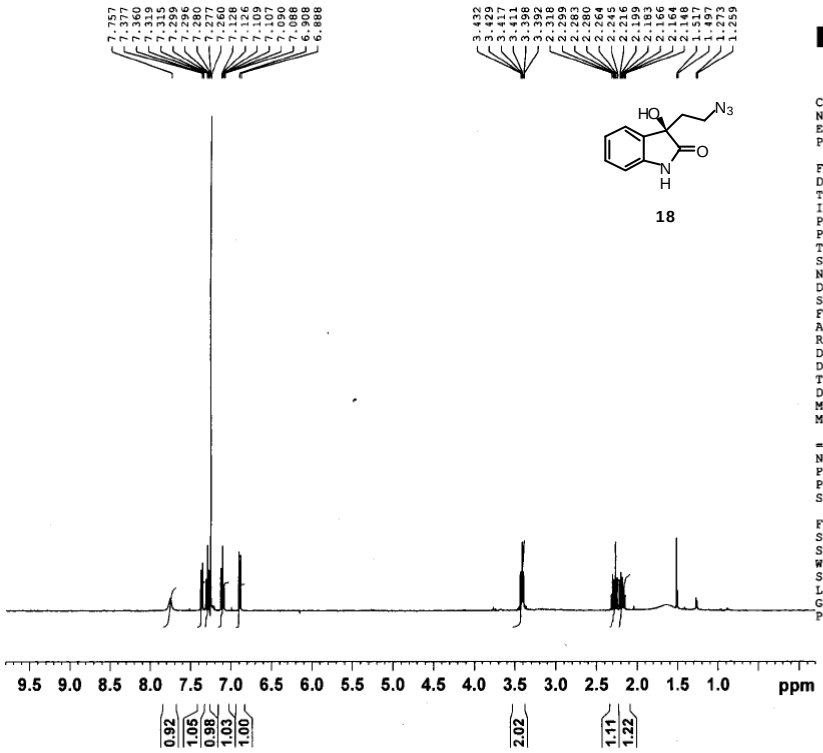
積算回数	16
ゼロフィリング	ON
ゲイン	1
日時	109/04/15 20:44
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

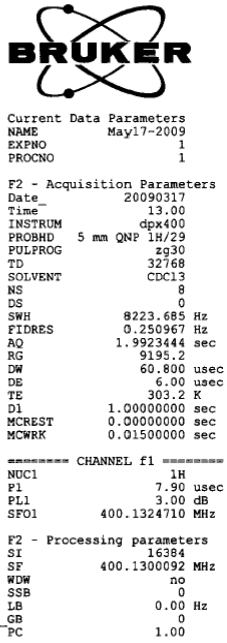
分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec



17

1:	3402.78,	86.0810	2:	2943.80,	68.7794	3:	2866.67,	69.9037	4:	2097.21,	57.9311
5:	1715.37,	63.7242	6:	1616.06,	70.5728	7:	1488.78,	79.2320	8:	1469.49,	69.1118
9:	1366.32,	76.5065	10:	1281.47,	87.9909	11:	1213.97,	87.2796	12:	1092.48,	62.0546
13:	1015.34,	88.9378	14:	882.27,	80.4073	15:	751.14,	72.8242	16:	685.57,	81.5139



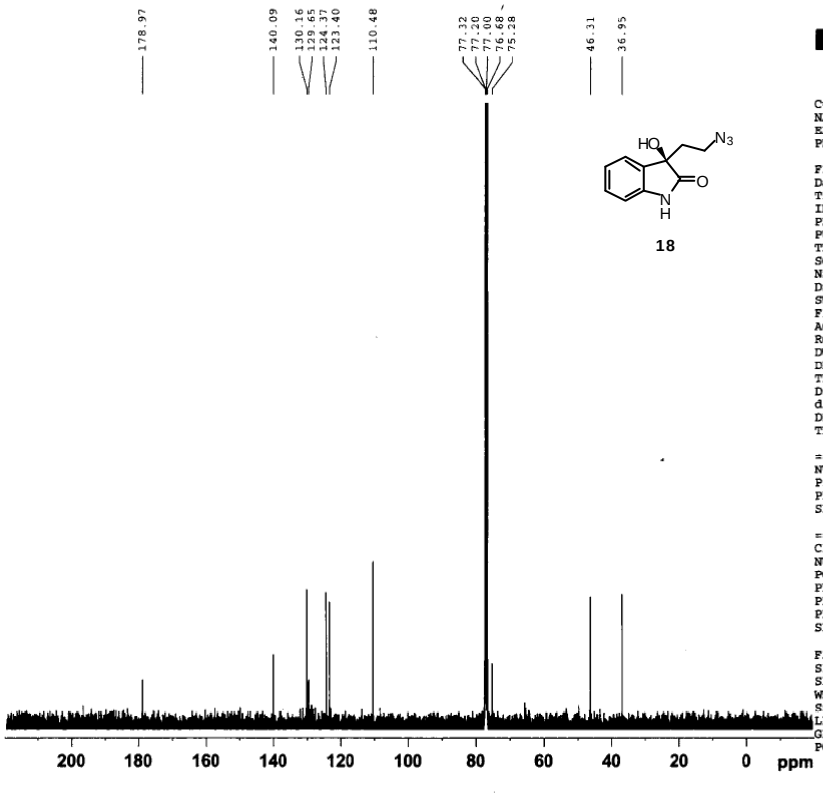



```
Current Data Parameters
NAME          May17-2009
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090317
Time               13.00
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                9195.2
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          400.1300092 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.00
```



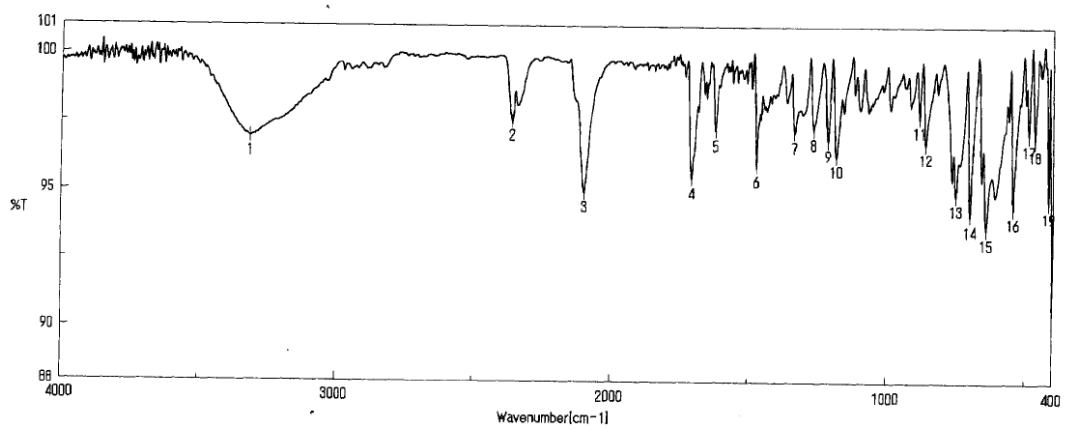

```
Current Data Parameters
NAME      Mar17-2009-hayashi
EXPNO                     20
PROCNO                     1

F2 - Acquisition Parameters
Date_           20090317
Time               13.41
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   600
DS                     4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                   128
DW               20.800 usec
DE                6.00 usec
TE                302.9 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

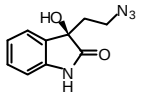
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1816007 MHz

F2 - Processing parameters
SI                 32768
SF          100.6253449 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```



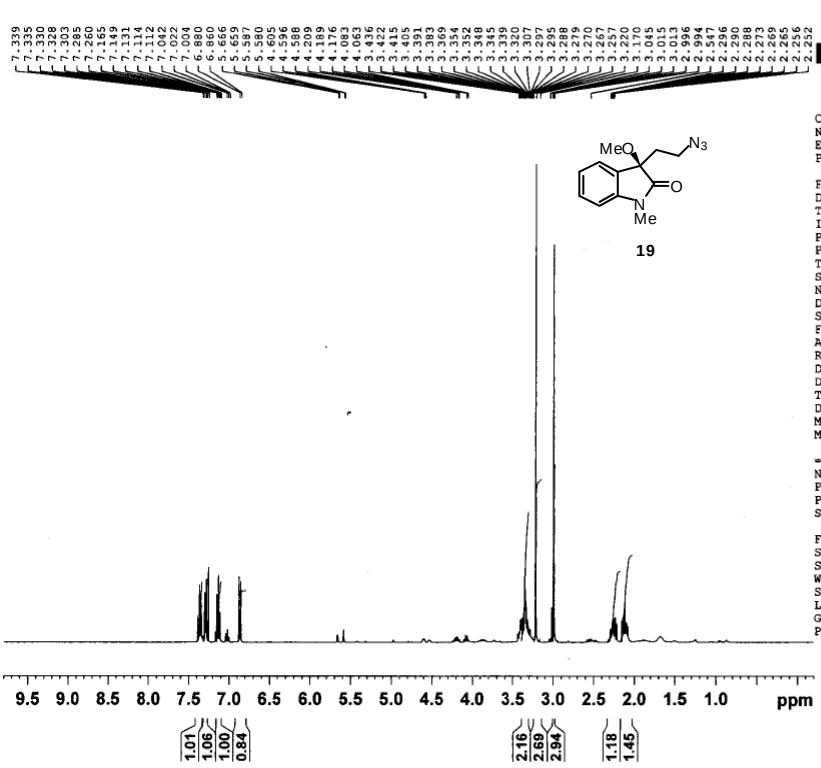
積算回数	16
ゼロフィリング	ON
ゲイン	2
日時	109/03/17 14:08
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

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



18

1:	3313.11, 96.9334	2:	2358.52, 97.5331	3:	2098.17, 94.9395	4:	1706.69, 95.4665
5:	1620.88, 97.1981	6:	1471.42, 95.8913	7:	1333.53, 97.1408	8:	1264.11, 97.2578
9:	1212.04, 96.8497	10:	1183.11, 96.2697	11:	886.13, 97.6995	12:	862.99, 96.7435
13:	753.07, 94.8560	14:	702.93, 94.0977	15:	643.14, 93.6280	16:	545.76, 94.3675
17:	488.87, 97.0611	18:	466.69, 96.8830	19:	417.51, 94.5844		



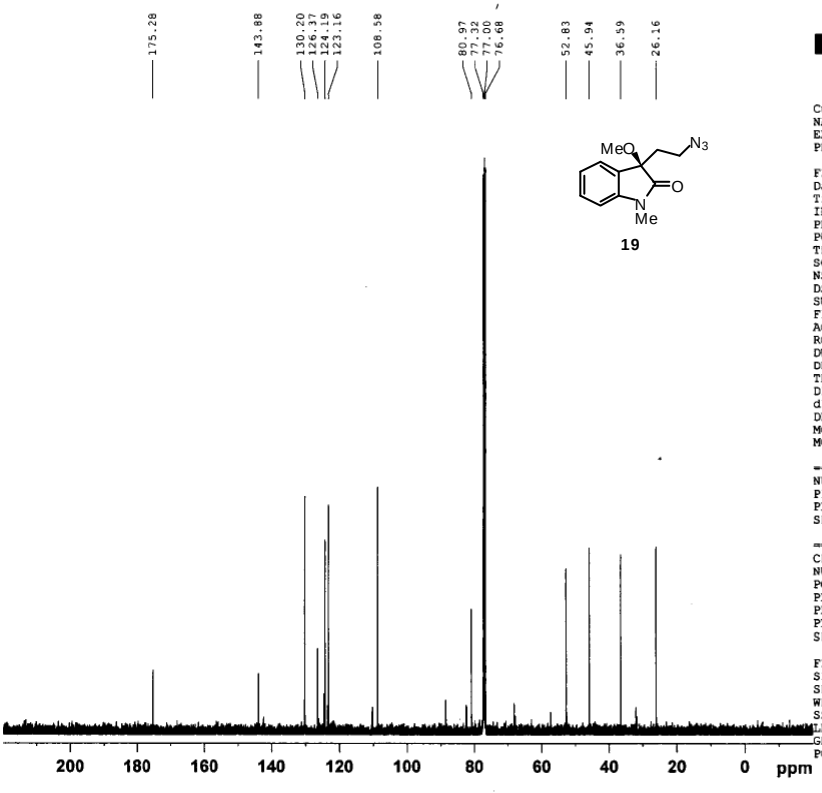
BRUKER

```
Current Data Parameters
NAME            Mar20-2009
EXPNO                   32
PROCNO                   1

F2 - Acquisition Parameters
Date_             20090320
Time                 14.13
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                   1824.6
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                   10.70 usec
PL1                   4.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                      16384
SF            400.1300092 MHz
WDW                        no
SSB                         0
LB                   0.00 Hz
GB                          0
PC                       1.00
```



BRUKER

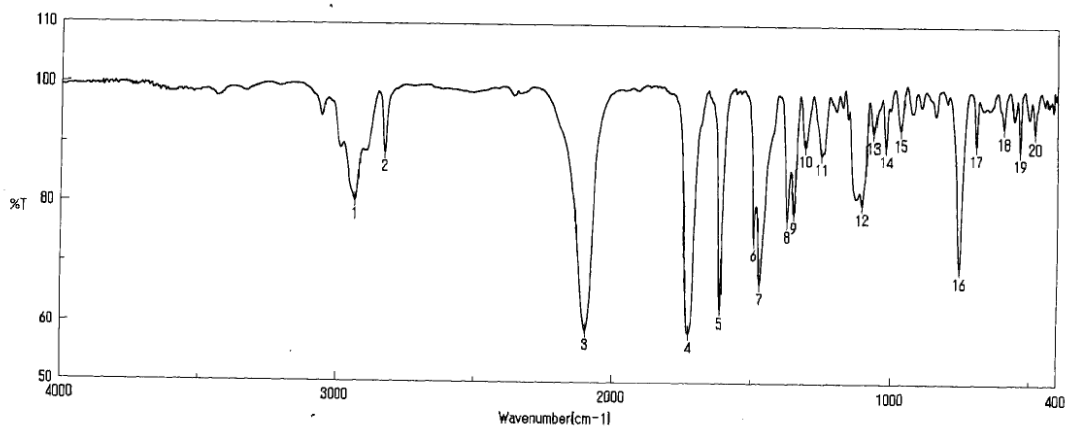
```
Current Data Parameters
NAME            Mar20-2009
EXPNO                   41
PROCNO                   1

F2 - Acquisition Parameters
Date_             20090320
Time                 14.38
INSTRUM              dpx400
PROBHD   5 mm QNP 1H/29
PULPROG             zgpg30
TD                  65536
SOLVENT              CDCl3
NS                     335
DS                       2
SWH              31847.133 Hz
FIDRES            0.485949 Hz
AQ                1.0289652 sec
RG                    6502
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                    8.60 usec
PL1                   2.00 dB
SFO1          100.6254358 MHz

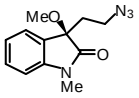
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                   4.00 dB
PL12                 21.47 dB
PL13                 21.47 dB
SFO2          400.1316005 MHz

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



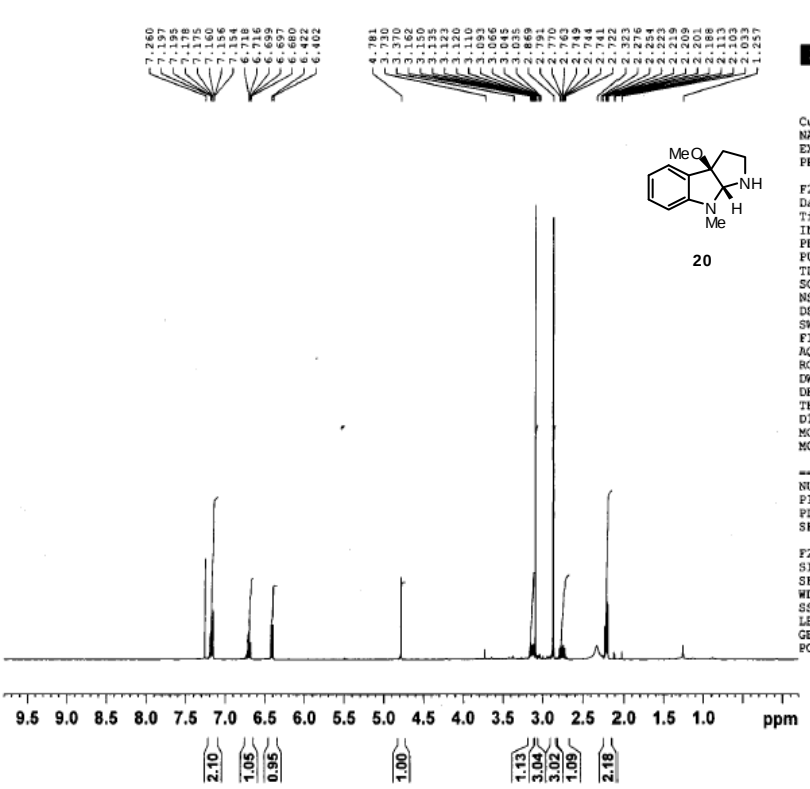
積算回数	16
ゼロフィリング	ON
ゲイン	1
日時	109/03/20 15:18
測定者	
ファイル名	Memory#3
サンプル名	buckground
コメント	

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



19

1:	2934.16,	80.4971	2:	2827.13,	88.5275	3:	2098.17,	58.8342	4:	1725.01,	58.4829
5:	1613.16,	62.5833	6:	1492.63,	74.0805	7:	1469.49,	66.7502	8:	1372.10,	77.2481
9:	1348.96,	78.6395	10:	1309.43,	89.9629	11:	1250.61,	88.5760	12:	1105.01,	80.1304
13:	1065.48,	92.3101	14:	1023.05,	89.9927	15:	970.02,	92.7791	16:	754.99,	69.5357
17:	698.11,	90.2584	18:	597.82,	93.1563	19:	538.04,	89.4276	20:	484.05,	92.3390

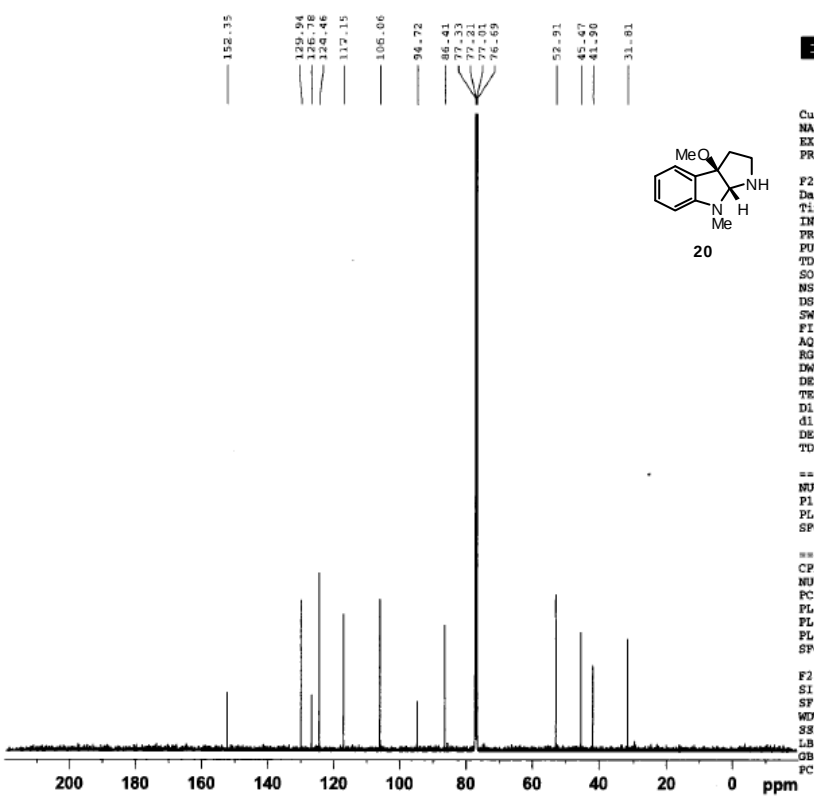



```
Current Data Parameters
NAME          Apr16-2009
EXPNO                164
PROCNO                 1

F2 - Acquisition Parameters
Date_          20090416
Time              21.11
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               3649.1
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                10.70 usec
PL1                4.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                16384
SF         400.1300092 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



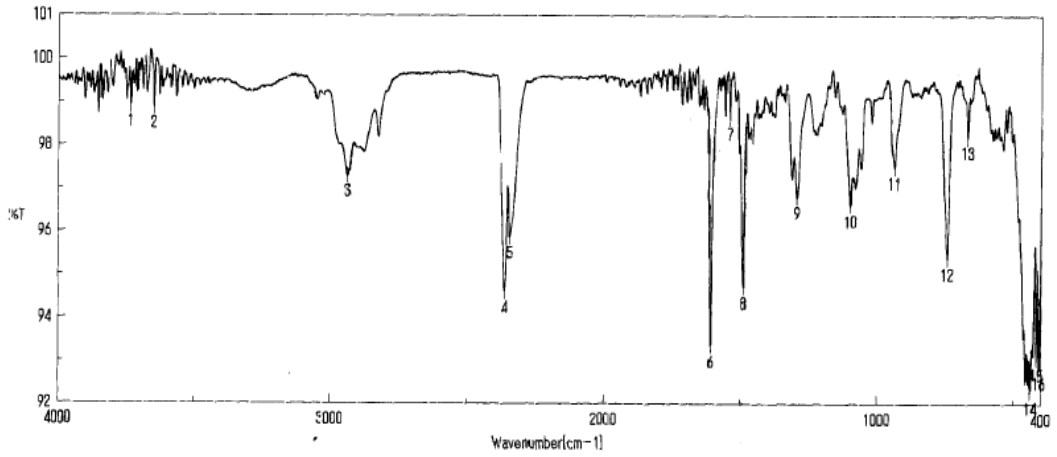

```
Current Data Parameters
NAME      Apr16-2009-hayashi
EXPNO                    70
PROCNO                    1

F2 - Acquisition Parameters
Date_          20090416
Time              22.16
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                 203
DW               20.800 usec
DE                6.00 usec
TE               296.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

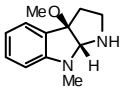
======== CHANNEL f1 ========
NUC1                13C
P1                7.20 usec
PL1               -3.50 dB
SFO1        100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               -4.00 dB
PL12              14.50 dB
PL13              14.50 dB
SFO2        400.1816007 MHz

F2 - Processing parameters
SI                32768
SF         100.6253433 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

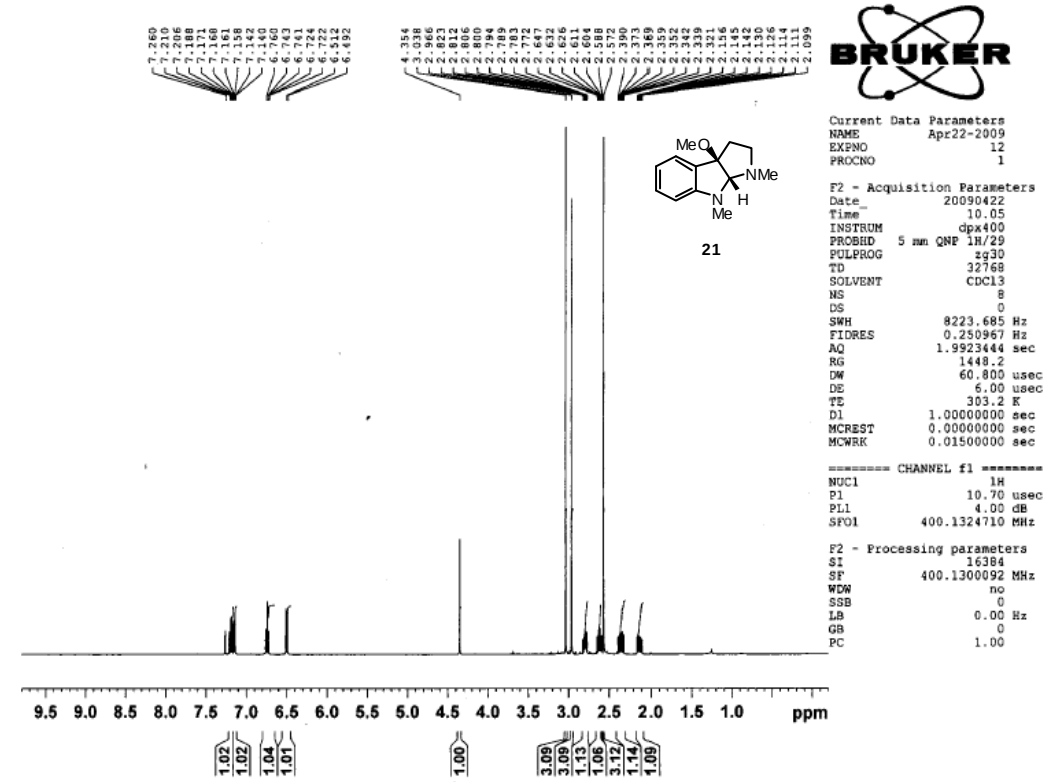


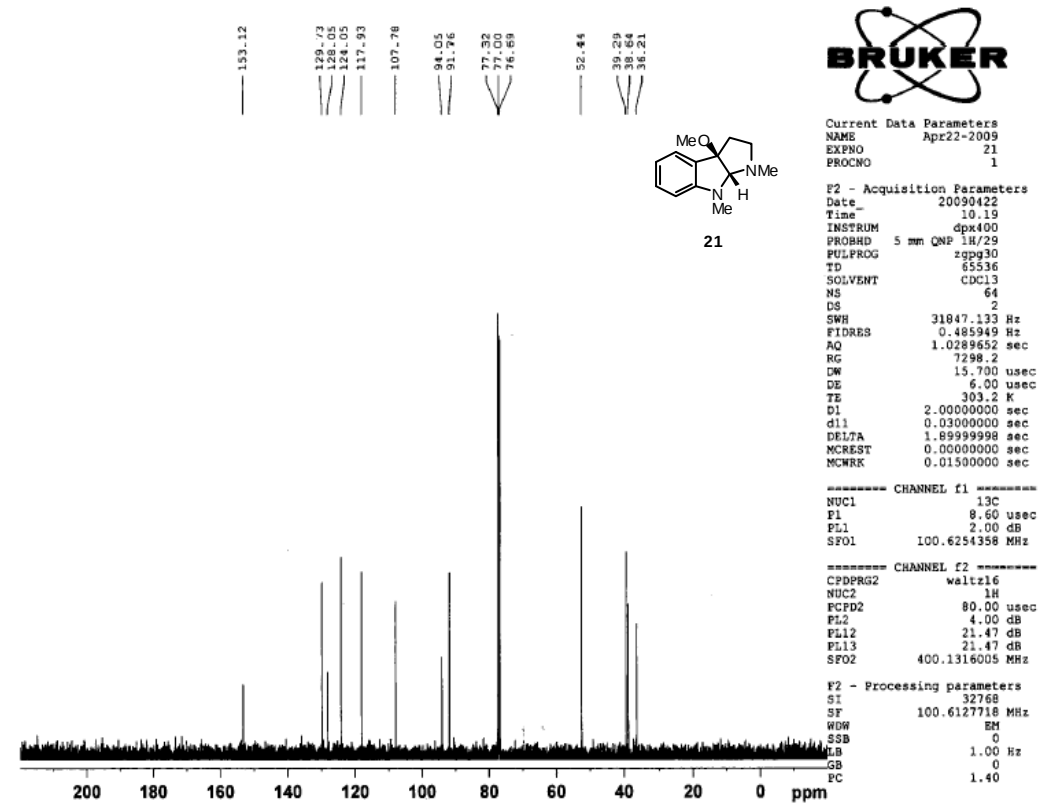
積算回数	16	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	2	スキャンスピード	2 mm/sec
日時	109/04/18 15:26		
測定者			
ファイル名	Memory#3		
サンプル名	background		
コメント			

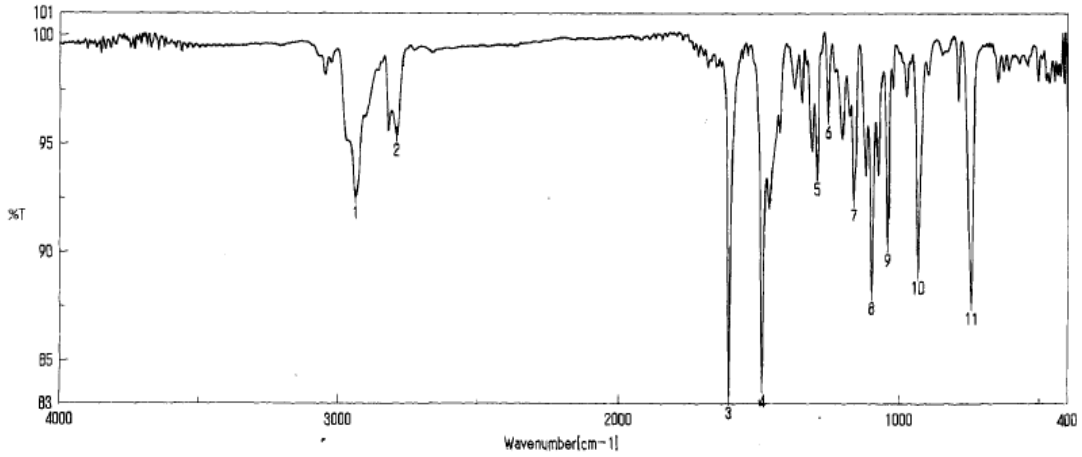


20

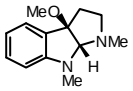
1:	3734.48,	96.0191	2:	3648.66,	98.8743	3:	2937.08,	97.3060	4:	2360.44,	94.6147
5:	2341.16,	95.8702	6:	1609.31,	93.3449	7:	1540.85,	96.6429	8:	1490.70,	94.7051
9:	1294.00,	96.8044	10:	1099.23,	96.5995	11:	938.20,	97.4886	12:	743.42,	95.3726
13:	669.18,	98.2113	14:	437.76,	92.2808	15:	416.55,	93.0498	16:	406.91,	92.8490


7.260
7.210
7.206
7.188
7.171
7.160
7.161
7.158
7.142
7.140
6.760
6.743
6.741
6.724
6.722
6.512
6.492
4.354
3.038
2.966
2.823
2.812
2.806
2.800
2.784
2.769
2.703
2.772
2.647
2.632
2.626
2.611
2.604
2.588
2.572
2.390
2.373
2.369
2.359
2.352
2.342
2.339
2.321
2.156
2.145
2.142
2.130
2.126
2.114
2.111
2.099
MeO
NMe
N H
Me
21
BRUKER
Current Data Parameters
NAME Apr22-2009
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090422
Time 10.05
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 1448.2
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 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW no
SSB 0
LB 0.00 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
1.02 1.02 1.04 1.01 1.00 3.09 3.09 1.13 1.06 3.12 1.14 1.09


153.12
129.73
128.05
124.05
117.93
107.78
94.05
91.96
77.32
77.00
76.69
52.44
39.29
38.64
36.21
MeO
NMe
N H
Me
21
BRUKER
Current Data Parameters
NAME Apr22-2009
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090422
Time 10.19
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 7298.2
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 8.60 usec
PL1 2.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 21.47 dB
PL13 21.47 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



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	109/04/24 10:15		
測定者			
ファイル名	Memory#3		
サンプル名	buckground		
コメント			



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1:	2938.02,	92.5473	2:	2791.46,	95.4216	3:	1608.34,	83.3025	4:	1489.74,	83.8498
5:	1294.00,	93.6511	6:	1254.47,	96.2803	7:	1162.87,	92.4345	8:	1098.26,	88.1443
9:	1041.37,	90.3637	10:	933.38,	89.0991	11:	742.46,	87.6738			