Supporting Information

Synthesis of [^{123}I]-Iodometomidate from a Polymer-Supported Precursor with a Large Excluded Volume.

Chika Nakagawa, Masahito Toyama, Ryota Takeuchi, Takashi Takahashi, and Hiroshi Tanaka,*

Experimental Section

General Methods

NMR spectra were recorded on a JEOL Model EX-270 (270 MHz for ^{1}H, 67.8 MHz for ^{13}C) or a JEOL Model ECP-400 (400 MHz for ^{1}H, 100 MHz for ^{13}C, 373 MHz for ^{19}F) in the indicated solvent. Chemical shifts were reported in parts per million (ppm) relative to the signal (0.00 ppm) for internal tetramethylsilane solutions in $CDCl_3$. ^{1}H NMR spectral data are reported as follows: $CDCl_3$ (7.26 ppm), CD_2Cl_2 (5.32 ppm), Acetone-d_6 (2.04 ppm), DMSO-d_6 (2.50 ppm), CD_3OD (3.30 ppm) ^{13}C NMR spectral data are reported as follows: $CDCl_3$ (77.0 ppm), CD_2Cl_2 (53.8 ppm), Acetone-d_6 (29.8 ppm), DMSO-d_6 (39.5 ppm), CD_3OD (49.8 ppm) or D_2O (Acetone-d_6 (206.0 ppm) as an internal standard). ^{19}F NMR spectral data are reported as follows: CF_3COOH (-79.0 ppm) as an external standard. NMR multiplicities are reported using the following abbreviations.
(s: singlet, d: doublet, t: triplet, q: quartet, m: multiplet, br: broad, *J*: coupling constants in Hertz.)

Infrared spectra (IR) were recorded on a Perkin-Elmer Spectrum 1. Only the strongest and/or structurally important absorbances are reported as the IR data given in cm^{-1}.

All reactions were monitored by thin layer chromatography carried out on Merck precoated TLC plates (60F-254) using UV light and *p*-anisaldehyde H_2SO_4 ethanol solution or 10% ethanolic phosphomolybdic acid.

Column chromatography separations were performed using silica gel (KANTO, silica gel 60 N, spherical, neutral, 40-100 um).

High performance liquid chromatography (HPLC) for qualitative and quantitative analysis were performed on a Gilson 506C system using a SHODEX ODS column (4.6 × 250 mm).Asahipak GF-7M HQ (7.5 × 300 mm), GPC K-804L (8.0 × 300 mm),

ESI-TOF Mass spectra were measured with AppliedBioSystems Mariner TK-3500 Biospectrometry Workstation mass spectrometers and Waters LCT Premier^{TM} XE. HRMS(ESI-TOF) were calibrated with angiotensin I (SIGMA), bradykinin (SIGMA), and neurotensin (SIGMA) as an internal standard.

Dry THF, dry hexane and dry toluene were distilled from sodium wire with a catalytic amount of benzophenone. Dry CH_2Cl_2 was distilled from P_2O_5. Dry DMF was distilled from CaH_2. Dry MeOH and dry EtOH were distilled from magnesium turning with a catalytic amount of iodine.

Preparation of the polymer-supported IMTO precursor 2.

1-((4-(4-Bromobutyl)dimethylsilyl)phenyl)-1-ethanol (5)

To a stirred solution of 1-(4-bromophenyl)ethyl-1-(2-tetrahydropiranyl)ether (**3**) (4.70 g, 16.5 mmol) in THF (49.5 mL), ^{n}BuLi (1.64 mol/L n-hexyl solution, 12.1 mL, 19.8 mmol) was added dropwise at -78 °C. After 1 h at the same temperature, the mixture was treated with (4-bromobutyl)chlorodimethylsilane **4** (4.95 g, 21.5 mmol, 1.30 eq.) was added dropwise to the reaction mixture at the same temperature. After stirring the mixture at 1 h, the mixture was gradually warmed to room temperature. The reaction mixture was poured into NH_4Cl aq. with cooling. The aqueous layer was extracted by diethyl ether twice. The combined extract was washed with saturated $NaHCO_3$, brine, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The mixture was used in the next reaction without further purification.

To a stirred solution of the above residue in MeOH (165 mL) was added PPTS (415 mg, 1.65 mmol) at 0 °C, and stirred at room temperature. The reaction mixture was quenched by NEt_3 and was concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane: ethyl acetate 92:8 to give **5** (4.60 g, 14.6 mmol, 88% in 2 steps).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.50 (d, 2H, *J* = 8.2 Hz), 7.37 (d, 2H, *J* = 7.7 Hz), 4.88 (q, 1H, *J* = 6.6 Hz), 3.39 (t, 2H, *J* = 6.8 Hz), 1.90-1.83 (m, 2H), 1.50 (d, 3H, *J* = 6.7 Hz), 1.50-1.43 (m, 3H), 0.77-0.73 (m, 2H), 0.28 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.44, 138.23, 133.76, 124.80, 70.31, 36.19, 33.42, 25.00, 22.44, 14.81, -3.10 ; FT-IR (neat): 3371, 2958, 2930, 1602, 1391, 1271, 1249, 1111, 1083, 1007, 821839, 552 (cm^{-1})

Methyl 1-(1-(4-bromobutyl)dimethylsilylphenyl)ethyl)-1*H*-imidazole-5-carboxylate (7)

To a stirred solution of 4-methoxycarbonyl-1*H*-imidazole (**6**) (1.68 g, 13.3 mmol) and triphenylphosphine (4.32 g, 16.6 mmol) in dry THF (40.0 mL) was added a solution of the alcohol **6** (4.60 g, 14.6 mmol) in dry THF (15.0 mL) at -40 °C, dropwise under argon. After a solution of di-*tert*-butyl azodicarboxyate (3.82 g, 16.6 mmol) in dry THF (25.0 mL) was added dropwise to the reaction mixture at the same temperature, the mixture was warmed from -40 °C to 0 °C within 2 h. After removal of the solvents *in vacuo,* the residue was solved with diethyl ether and stirred for 10 min. The mixture was filtered through a pad of Celite® with diethyl ether. After the filtrate was concentrated in vacuo, the residue was purified by column chromatography on silica gel with hexane: ethyl acetate 70:30 to give **7** (3.70 g, 8.74 mmol, 66%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.82 (s, 1H), 749 (d, 2H, *J* = 7.7 Hz), 7.20 (d, 2H, *J* = 7.7 Hz), 6.40 (q, 1H, *J* = 6.9 Hz) , 3.86 (s, 3H), 3.39 (t, 2H, *J* = 6.8 Hz), 1.89-1.82 (m, 5H), 1.49-1.43 (m, 2H), 0.76-0.72 (m, 2H), 0.26 (s, 6H),; ^{19}C NMR (100 MHz, $CDCl_3$) δ 160.27, 141.40, 139.56, 138.63, 137.80, 125.29, 54.88, 51.04, 35.80, 33.03, 22.06, 21.76, 14.37, -3.43 ; FT-IR (neat): 2953, 1715, 1601, 1539, 1436, 1362, 1213, 1113, 1052, 954, 840 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{19}H_{27}BrN_2O_2Si$ $[M+H]^+$ 423.1103, found 423.1135.

Methyl 1-(4-((4-azidobutyl)dimethylsilyl)phenyl)ethyl)-1*H*-imidazole-5-carboxylate (8)

To a solution of **7** (1.68 g, 3.97 mmol) in DMF (27.8 mL), sodium azide (1.29 g, 19.8 mmol) and a catalytic amount of sodium iodide was added. After stirring the mixture for 6 h at 80 °C, the mixture was cooled down and washed with H_2O and the aqueous layer was extracted by diethyl ether, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The residue was purified by short pass of column chromatography on silica gel with dichloromethane : methanol 98 : 2 to give **8** (1.35 g, 3.50 mmol, 89%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.79 (s, 1H), 7.48 (d, 2H, *J* = 8.0 Hz), 7.18 (d, 2H, *J* = 8.0 Hz), 6.38 (q, 1H, *J* = 7.2 Hz), 3.83 (s, 3H), 3.22 (t, 2H, *J* = 6.8 Hz), 1.87 (d, 3H, *J* = 7.2 Hz), 1.60 (tt, 2H, *J* = 7.2, 7.2 Hz), 1.42-1.34 (m, 2H), 0.76-0.72 (m, 2H), 0.24 (s, 6H),; ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.41, 141.46, 139.65, 138.80, 137.83, 134,33, 133.84, 125.41, 122.11, 55.03, 51.17, 50.75, 32.19, 21.84, 20.8, 15.03, -3.38 ; FT-IR (neat): 3785, 2952, 2315, 2097, 1714, 1437, 1362, 1256, 1218, 1133, 1113, 1050, 821 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{23}H_{33}N_3O_3Si$ $[M+H]^+$ 428.2369, found 428.2392.

Methyl 1-(4-(4-(*N*-(2-methylprop-2-enoyl)amino)butyl)dimethylsilyl)phenyl)ethyl)-1*H*-imidazole-5-carboxylate (10)

To a stirred solution of **8** (648 mg, 1.68 mmol, 1.00 eq.) in THF/H_2O (1:1) (6.72 mL), NH_4Cl (270 mg, 5.04 mmol, 3.00 eq.) and activated Zn (446 mg, 6.82 mmol, 4.06 eq.) was added at 0 °C. After assuring no azide **8** was detected by TLC, the mixture was filtered through a pad of Celite®, and the filtrate was evaporated in vacuo. The residue was used in the next reaction without further purification.

To a stirred solution of the above residue and $NaHCO_3$ (998 mg, 11.8 mmol) in THF/H_2O (1:1) (6.72 mL), methacryloyl chloride (**9**) (522 mg, 5.04 mmol) was added dropwise at 0 °C. After no amine was detected, the reaction mixture was washed with saturated $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated *in vacuo.* Before evaporation, the filtrate was added polymerization inhibitor. The residue was purified by column chromatography on silica gel with 98:2 dichloromethane: methanol to give **10** (590 mg, 1.38 mmol, 83%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.80 (s, 1H), 7.48-7.46 (d, 2H, *J* = 7.2 Hz), 7.19-7.16 (d, 2H, *J* = 7.2 Hz), 6.40-6.35 (q, 1H, *J* = 7.3 Hz), 5.71 (m, 1H), 5.62 (s, 1H), 5.28 (s, 1H), 3.83 (s, 3H), 3.30-3.25 (dt, 2H, *J* = 6.6 Hz), 1.94 (s, 3H), 1.53-1.51 (tt, 2H, *J* = 7.2 Hz), 1.36-1.30 (m, 2H, *J* = 3.9 Hz), 0.77-0.73 (m, 2H), 0.24 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168,24, 160.38, 141.32, 140.13, 138.99, 137.73, 133.84, 125.34, 122.19, 118.64, 55.04, 51.17, 38.96, 32.92, 21.82, 18.46, 15.04, -3.35 ; FT-IR (neat): 3324, 2953, 1714, 1660, 1622, 1539, 1437, 1362, 1248, 1219, 1133, 1113, 1052, 922, 838, 767, 733, 673, 538 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{23}H_{33}N_3O_3Si$ $[M+H]^+$ 428.2369, found 428.2392.

Polymer 2

The solution of monomer in reaction mixture of monomer in 40wt% toluene was passed through a column of alumina without dilution to remove the polymerization inhibitor. To the solution of the monomer **10** (580 mg, 1.36 mmol) in toluene (40 wt%, 1.45 g/ 1.24mL), AIBN (11.2 mg,

0.0670 mmol) was added. The mixture was frozen by liq. N_2, then degassered, and risen to room temperature under reduced pressure. The same operation were done about three times, the mixture was stirred at 65 °C for 24 h. The reaction mixture was cooled to room temperature, the solvent was removed *in vacuo*, and the residue was poured into hexane. The insoluble materials was separated by the filter paper. The residue was purified by column chromatography on silica gel eluted with CH_2Cl_2. The residue was precipitated from hexane to provide polymer **2**. (282 mg, 0.661 mmol, 49%). The number-averaged molecular weight of the purified polymer **2** was estimated by a size-exclusion chromatography (SEC: Asahipak GF-7M HQ) eluted with 1.0 M LiCl DMF solution to be $1.02x10^4$.

^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.91-7.76 (br, 1H), 7.71 (s, 1H), 7.47-7.38 (br, 2H), 7.18-7.10 (br, 2H), 6.35-6.27 (br, 1H), 5.07 (m, 1H), 3.72 (s, 3H), 3.14-2.90 (br, 2H), 1.87-1.77 (br, 3H), 1.64-1.36 (br, 2H), 1.36-1.20 (br, 5H), 0.90-0.89 (br, 2H), 0.73-0.62 (br, 2H), 0.23-0.13 (br, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 142.2, 140.2, 139.2, 137.9, 134.1, 128.2, 125.6, 122.5, 77.6, 45.3, 39.8, 31.7, 22.7, 22.1, 21.7, 15.3, 13.9, -3.15 ; FT-IR (neat): 2945, 2328, 1705, 1456, 1360, 1225, 1114, 1003, 918, 659, 558 (cm^{-1}).

Preparation of standard samples

Methyl 1-[1-(4-iodophenyl)methyl]-1*H*-imidazole-5-carboxylate 1

To a stirred solution of 4-methoxy-imidazole-carbonic acid **18** (694 mg, 1.00 eq. 5.50 mmol) and Triphenylphosphine (1.79 mg, 6.88 mmol, 1.25 eq.) in dry THF (14.0 mL) was added a solution of 1-(4-iodophenyl)ethanol **42** (1.50g, 6.05 mmol,1.10 eq.) in dry THF (5.50 mL) at -30 °C, dropwise under argon. Then, a solution of di-*tert*-butyl azodicarboxyate (1.58 mg, 6.88 mmol, 1.25 eq.) in THF (8.00 mL) was added dropwise and the mixture was warmed from -30 °C to 0 °C within 2 h. After no alcohol was detected by TLC, the solution was evaporated *in vacuo*. The residue was mixed with diethyl ether and stirred for 10 min. The crystal of Ph_3PO was filtered through a pad of Celite®, and washed with diethyl ether. The filtrate was evaporated *in vacuo* and the residue was purified by column chromatography silica gel with hexane: ethyl acetate 65:35 to give methyl 1-[1-(4-iodophenyl)ethyl]-1*H*-imidazole-5-carboxylate **1** (1.03 g, 2.89 mmol, 53%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.86 (m, 1H, H-a), 7.80 (s, 1H, H-b), 7.69-7.66 (d, 2H, H-g, J = 8.3 Hz), 6.93-6.91 (d, 2H, H-f, J = 8.3 Hz), 6.33-6.30 (q, 1H, H-d, J = 7.1 Hz) , 3.81 (s, 3H, H-c), 1.86-1.84 (d, 3H, H-e J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 140.9, 139.5, 138.1, 127.9, 122.2, 93.4, 54.8, 51.3, 21.9; FT-IR (neat): 3419, 3126, 2984, 2950, 1715, 1538, 1488, 1436, 1362, 1221, 1134, 1112, 1064, 1007, 660, 525 (cm^{-1}); mp: 69-71℃; HRMS (ESI-TOF) calcd. for $C_{13}H_{13}IN_2O_2$ $[M+H]^+$ 357.0100, found 357.0093.

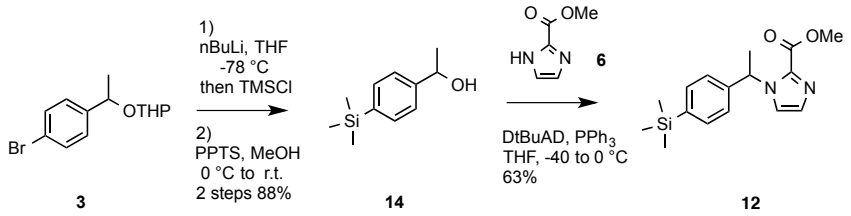
Methyl 1-[1-(4-chlorophenyl)methyl]-1*H*-imidazole-5-carboxylate (11)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (s, 1H, H-a), 7.76 (s, 1H, H-b), 7.30 (d, 2H, H-f, J = 8.7 Hz), 7.10 (d, 2H, H-g, J = 8.7 Hz), 6.31 (q, 1H, H-d, J = 7.4 Hz) , 3.80 (s, 3H, H-c), 1.85 (d, 3H, H-e J = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6, 139.8, 139.5, 138.3, 133.8, 129.0, 127.5, 126.2, 54.8, 51.5, 22.2; FT-IR (neat): 3713, 2952, 1715, 1538, 1494, 1437, 1363, 1219, 1133, 1112, 1053, 1015, 919,655 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{13}H_{13}ClN_2O_2$ $[M+H]^+$ 256.0744, found 256.0753.

Methyl 1-[1-(phenyl)methyl]-1*H*-imidazole-5-carboxylate (13)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (s, 1H, H-a), 7.74 (s, 1H, H-b), 7.37-7.27 (m, 3H, H-g,h), 7.19 (d, 2H, H-f, *J* = 7.3 Hz), 6.36 (q, 1H, H-d, *J* = 7.2 Hz) , 3.81 (s, 3H, H-c), 1.86 (d, 3H, H-e *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.39, 140.89, 137.87, 128.60, 126.02, 122.13, 55.13, 51.15, 21.92; FT-IR (neat): 3419, 2986, 1715, 1538, 1495, 1455, 1438, 1363, 1217, 1133, 765, 702, 660 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{13}H_{14}N_2O_2$ $[M+H]^+$ 231.1134, found 231.1136.

Preparation of the silylated IMTO



1-(4-Trimethylsilylphenyl)-1-ethanol (14)

To a stirred solution of **3** (4.00 g, 14.0 mmol) in THF (76.5 mL), nBuLi (1.59 mol/L n-hexane solution, 9.69 mL, 15.4 mmol) was added dropwise at -78 °C. After stirring for 1 h at the same temperature, the mixture was treated with distilled trimethylsilylchloride (1.83 g, 16.8 mmol), dropwise. The temperature was warmed to room temperature gradually. The reaction mixture was poured into NH_4Cl aq. with cooling. The aqueous layer was extracted by diethyl ether and the combined extract was washed with saturated $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated in vacuo. The mixture was used in the next reaction without further purification.
To a stirred solution of the above residue in MeOH (140 mL) PPTS (352 mg, 1.40 mmol) was added, at 0 °C and stirred at room temperature. After no ether x is detected on TLC, the reaction was quenched by adding NEt_3 until the pH becomes basic. The mixture was evaporated in vacuo, and the residue was purified by column chromatography on silica gel with hexane: ethyl acetate 94:6 to give **14** (1.59 g, 8.18 mmol, 84% in 2 steps).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, 2H, *J* = 7.7 Hz), 7.37 (d, 2H, *J* = 7.9 Hz), 4.89 (q, 1H, *J* = 6.6 Hz), 1.63 (m, 1H), 1.50 (d, 3H), 0.26 (s, 9H,); ^{13}C NMR(100 MHz, $CDCl_3$) δ 146.3, 139.5, 133.5, 124.8, 70.2, 24.9, -1.2; FT-IR (neat): 3368, 2957, 1602, 1393, 1249, 1113, 1085, 1008, 840, 821, 758, 551 (cm^{-1})

Methyl 1-[1-(4-trimethylsilylphenyl)ethyl]-1H-imidazole-5-carboxylate (12)

To a stirred solution of **6** (177 mg, 1.40 mmol) and triphenylphosphine (473 mg, 1.82 mmol) in dry THF (3.50 mL) was added a solution of **14** (300 mg, 1.54 mmol) in dry THF (3.00 mL) at -30 °C, dropwise under argon. Then, a solution of di-*tert*-butyl azodicarboxyate (387 mg, 1.68 mmol) in THF (4.00 mL) was added dropwise to the reaction mixture at the same temperature. The reaction mixture was warmed from -30 °C to 0 °C within 2 h. After no alcohol was detected by TLC, the solution was evaporated in vacuo. After removal of the solvents *in vacuo,* the residue was solved with diethyl ether and stirred for 10 min. The mixture was filtered through a pad of Celite® with diethyl ether. After the

filtrate was concentrated in vacuo, the residue was purified by column chromatography on silica gel with hexane:ethyl acetate, 68:32 to give **11** (225 mg, 0.784 mmol, 56%).

1H NMR (400 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.80 (s, 1H), 7.50 (d, 2H, *J* = 7.8 Hz), 7.18 (d, 2H, *J* = 7.8 Hz), 6.38 (q, 1H, J = 7.1 Hz), 3.84 (s, 3H), 1.87 (d, 3H, J = 6.8 Hz), 0.25 (s, 9H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.62, 141.40, 140.42, 139.79, 133.83, 125.53, 122.33, 55.29, 51.39, 22.08, -1.24; FT-IR (neat): 2955, 1715, 1539, 1438, 1362, 1249, 1219, 1134, 1114, 841.8, 766, 671 (cm^{-1}); mp: 142-143 °C; HRMS (ESI-TOF) calcd. for $C_{16}H_{22}N_2O_2Si$ [M+H]+ 303.1529, found 303.1556.

Reaction of 11 in the presence of NCS under acidic conditions.

To a solution of methyl 1-[1-(4-trimethylsilylphenyl)ethyl]-1*H*-imidazole-5-carboxylate **10** (20.0 mg, 0.661 mmol) in TFA (500 μL) was added a solution of NCS (26.5 mg, 0.173 mmol) in TFA (661 μL). The reaction mixture was stirred at 40 or 90 ^oC for 60 min. Then, 100 μL of the reaction mixture was quenched by 10% $Na_2S_2O_3$ aq. and saturated $NaHCO_3$, and extracted by ethyl acetate. The organic layer was evaporated *in vacuo*, The residue was analyzed by HPLC based on a photo diode-array detector.

Iodination using 2

To a stirred solution of **2** (20.0 mg, 0.0469mmol, 1.00 eq.) in TFA (469 μL), NaI (7.03 mg, 0.0469 mmol, 1.00 eq.) and NCS (18.8 mg, 0.141 mmol, 3.00 eq.) was added. The time was counted from the time as NCS was added. After stirring the mixture for 60 min at 40 °C, the reaction was quenched by 10% $Na_2S_2O_3$ aq. and saturated $NaHCO_3$, and extracted by ether acetate. The organic layer was evaporated in vacuo, and the residue was passed through a short pass of column chromatography on silica gel with dichloromethane: methanol 98:2 to give a mixture of iodide **1** (13.0 mg, 0.0366 mmol, 78%) and the chloride **11** (0.4 mg, 0.00094 mmol, 2.0%). The yields were estimated by ^1H NMR spectra of the mixture.

Synthesis of [^{123}I]IMTO from monomer 12

Methyl 1-[1-(4-trimethylsilylphenyl)ethyl]-1*H*-imidazole-5-carboxylate **11** (1.4-3.2 mg, 4.6-10.6 μmol, 1.00 eq.) was added to [^{123}]NaI in 1.0 mol/L NaOH(10-15μL, 60-145MBq), 14mg/mL NCS in solvent (138-320μL, 14- 32μmol, 3.00 eq.) and solvent (80-262 μL, total volume is 410-415μL). After being stirred at the indicated temperature for 60 min, the reaction was quenched by 30% $Na_2S_2O_3$ aq.. The mixture was diluted with water(10mL) and unreacted [^{123}]NaI was removed by passage through a C18 Sep-Pak. The Sep-Pak was rinsed with 10mL water and the [^{123}I]IMTO was eluted with 2mL EtOH. The eluant was analyzed by Radio-TLC and HPLC.

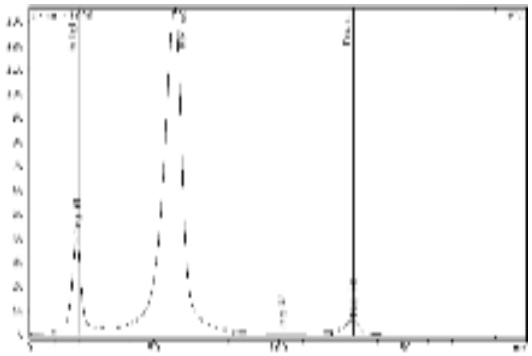
TLC : The radioactive spots are detected and recorded by Rita Star

High performance liquid chromatography (HPLC) for qualitative and quantitative analysis were performed on a 2695 Alliance HPLC Separation Module using a Inertsil WP300 C18 column (4.6 × 250 mm, 5μm).

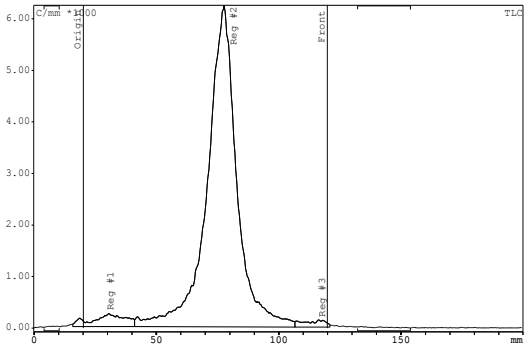
Synthesis of [^{123}I]IMTO from the polymer precursor 2.

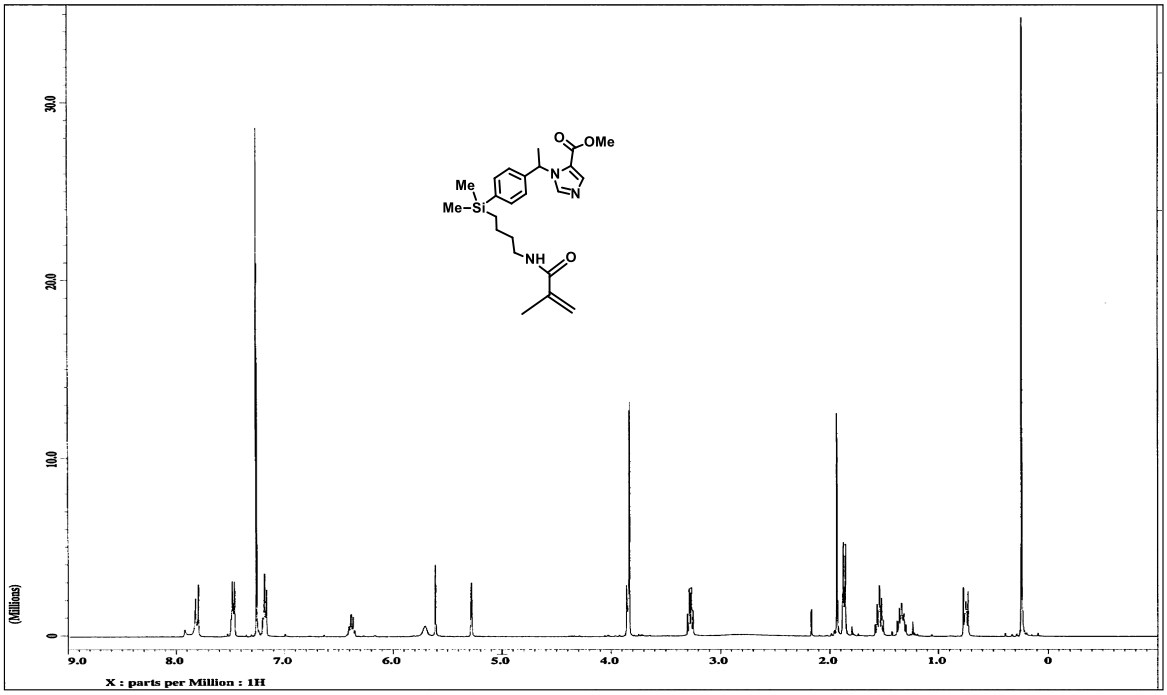
To a stirred solution of Polymer precursor **2** (1 mg) in TFA(50µL), [^{123}]NaI in 1.0 mol/L NaOH (10µL, 147MBq) and then 5mg/mL NCS in TFA (109µL) was added at room temperature . After being stirred at 40ºC for 60 min (TLC01), the mixture was diluted with methylene chloride(15mL) and then was added to PS-DIEA(548mg). After being stirred at room temperature for 5 min, the mixture was passed through a Silicagel Sep-pak and was rinsed with 10mL of methylene chloride. After being connected another Silicagel Sep-pak, [^{123}I]IMTO was eluted with 15mL methylene chloride/methanol (1:9). The eluant was heated at 40ºC and the solvent was evaporated with the aid of Argon gas flow. Acetonitrile (2mL) was added to the residue and [^{123}I]IMTO was obtained with radiochemical purity over 94% and radiochemical yield was 85%(decay collect) (TLC02),

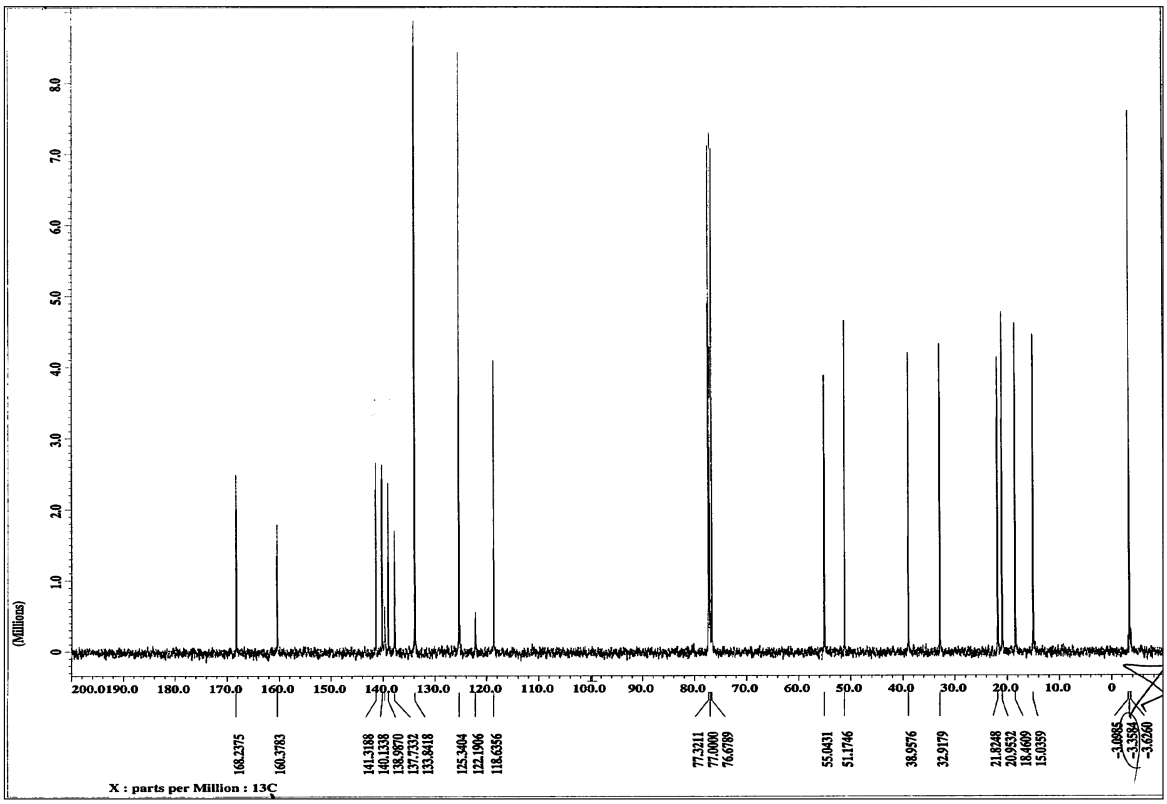
Radioactivity analysis by a TLC Scanner
TLC 01

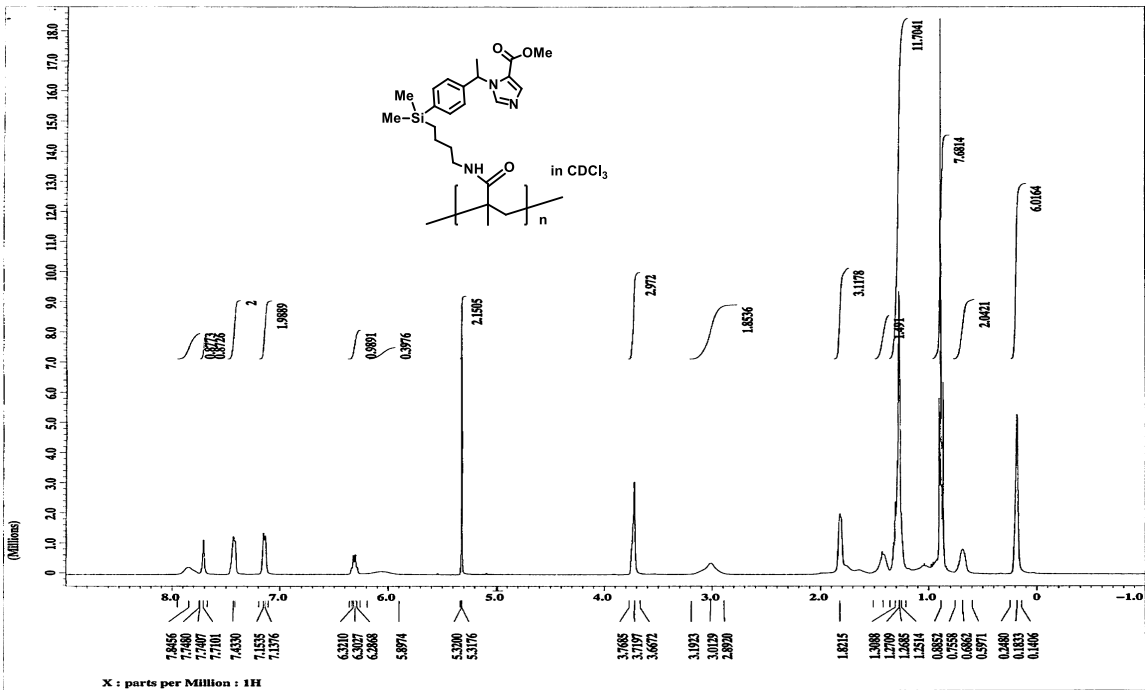


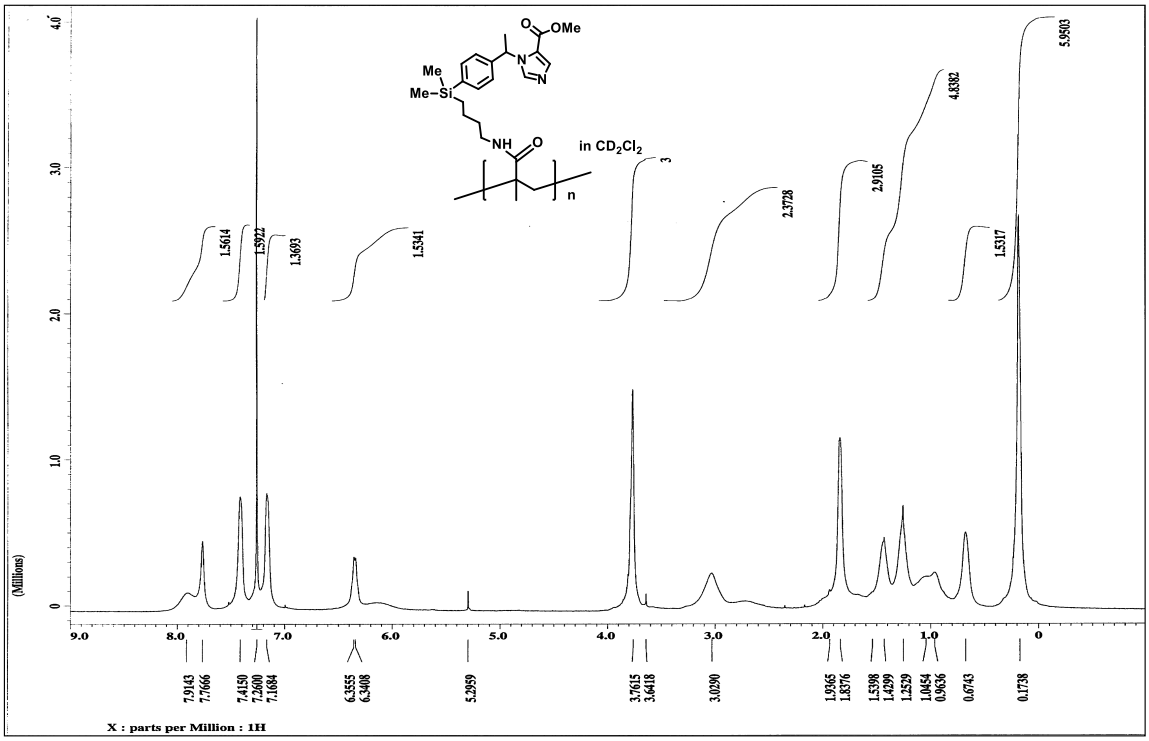
TLC02

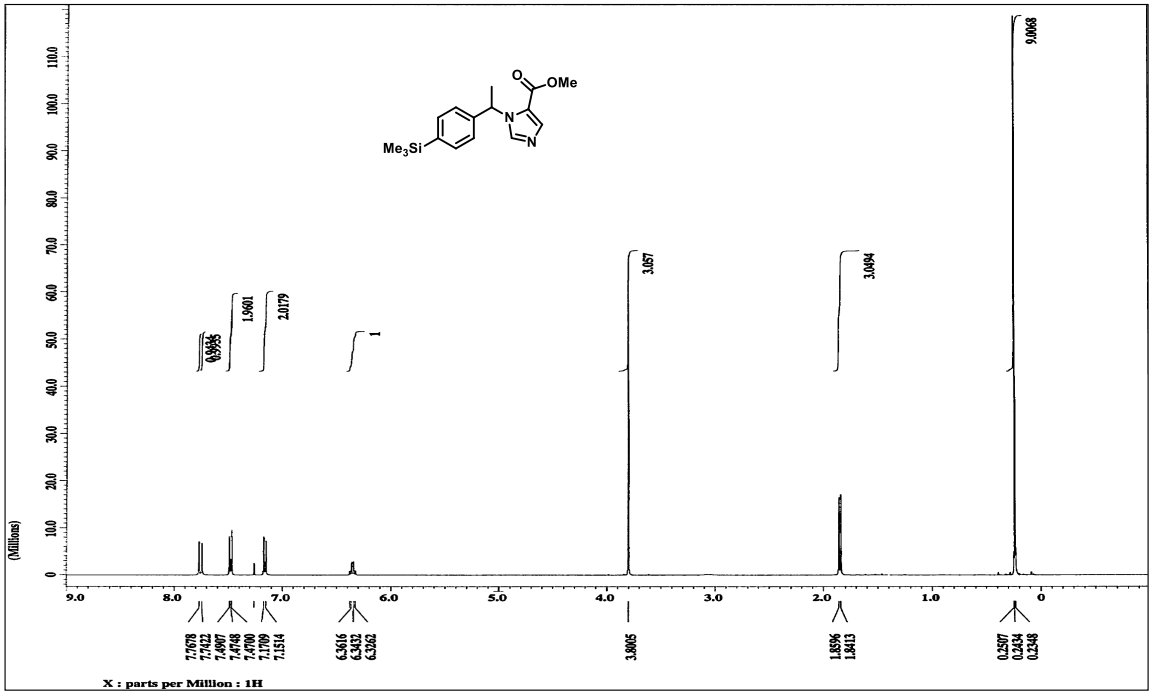


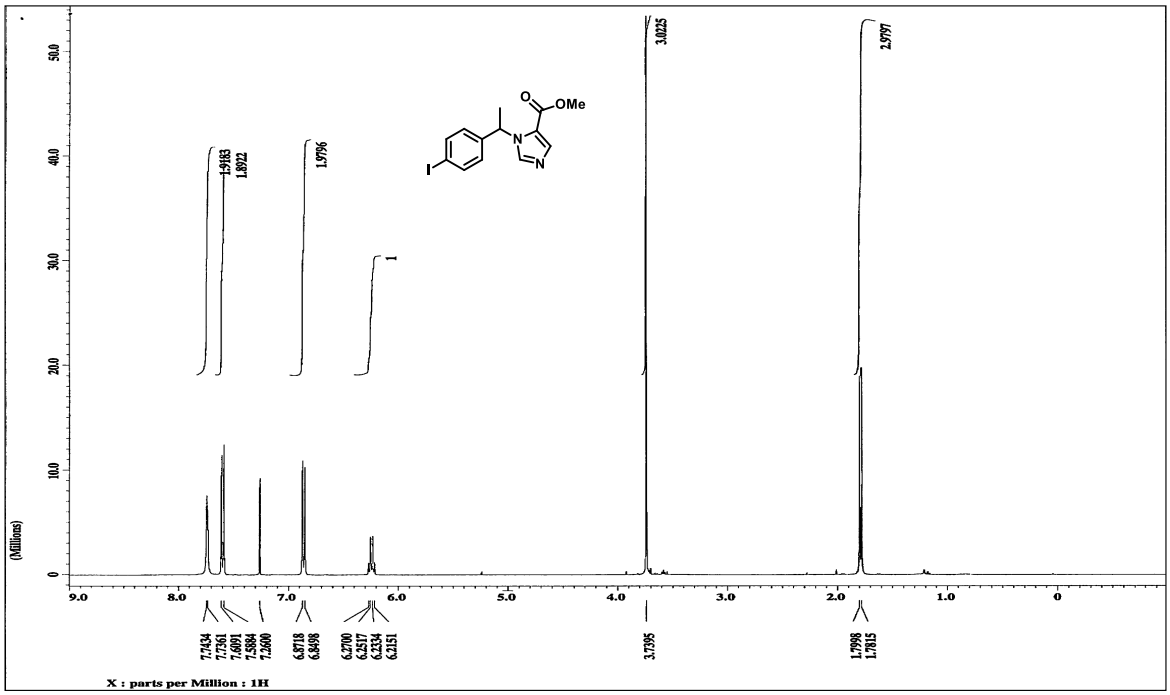

O
OMe
Me
Me–Si
N
N
NH
O
(Millions)
30.0
20.0
10.0
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H

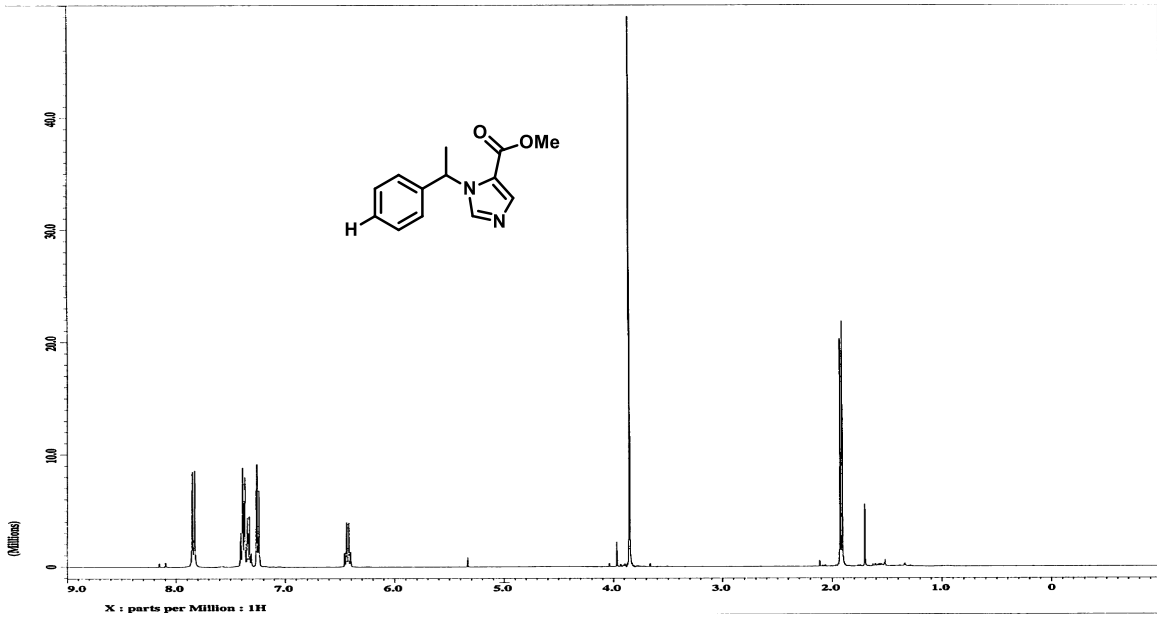

(Millions)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
168.2375
160.3783
141.3188
140.1338
138.9870
137.7332
133.8418
125.3404
122.1906
118.6356
77.3211
77.0000
76.6789
55.0431
51.1746
38.9576
32.9179
21.8248
20.9532
18.4609
15.0359
-3.0985
-3.3584
-3.6260
X : parts per Million : 13C

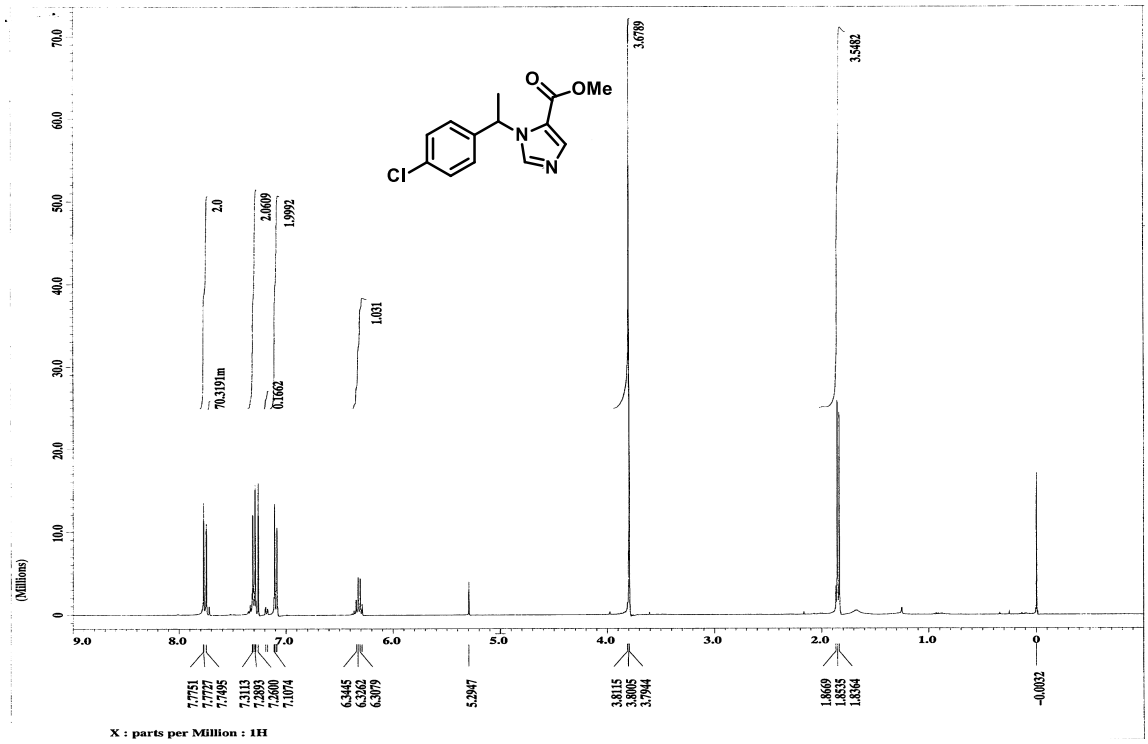

in $CDCl_3$
(Millions)
X : parts per Million : 1H


in CD_2Cl_2
(Millions)
X : parts per Million : 1H


(Millions)
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
O
OMe
N
N
Me3Si
0.9935
1.9601
2.0179
1
3.057
3.0494
9.0068
7.7678
7.7422
7.4967
7.4748
7.4700
7.1709
7.1514
6.3616
6.3432
6.3262
3.8005
1.8696
1.8413
0.2597
0.2434
0.2348
X : parts per Million : 1H


(Millions)
50.0
40.0
30.0
20.0
10.0
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
O
OMe
N
N
I
1.9183
1.8922
1.9796
1
3.0225
2.9797
7.7434
7.7361
7.6091
7.5084
7.2600
6.8718
6.8498
6.2700
6.2517
6.2334
6.2151
3.7395
1.7998
1.7815
X : parts per Million : 1H


O
OMe
N
N
H
(Millions)
40.0
30.0
20.0
10.0
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H


O
OMe
N
N
Cl
3.6789
3.5482
2.0
2.0609
1.9992
1.031
70.3191m
0.1662
(Millions)
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.7751
7.7727
7.7495
7.3113
7.2893
7.2600
7.1074
6.3445
6.3262
6.3079
5.2947
3.8115
3.8005
3.7944
1.8669
1.8535
1.8364
-0.0032
X : parts per Million : 1H