Experimental Section

General procedures. All reagents were commercial products and were used without further purification. Flash column chromatography was performed on 230-400 mesh silica gel. Low and high resolution mass analyses were performed by Taejon Analytical Laboratory of Korea Basic Science Institute. ^{1}H NMR spectra were recorded on 300 MHz NMR spectrometer, and chemical shifts (δ) are in part per million relative to TMS.

(*S*)-2-*O*-(2-Allyl)-1-*O*-benzyl-3-*O*-(*tert*-butyldiphenylsilyl)glycerol (2) : A solution of *tert*-butyldiphenylsilyl chloride (10.4 g, 38 mmol) in anhydrous DMF (20 mL) was added to a stirred mixture of imidazole (26 g, 38.2 mmol) and 1-*O*-benzylglycerol **1** (5.76 g, 31.7 mmol) in dry DMF (30mL). After being stirred overnight, the reaction mixture was poured into water and extracted with EtOAc (3 x 50 mL). The extracts were combined and washed with brine, dried over $MgSO_4$, and concentrated. Purification of the residue by flash chromatography (9:1, hexanes-ethyl acetate) afforded (S)-1-*O*-benzyl-3-*O*-(*tert*-butyldiphenylsilyl)glycerol (11.7 g, 88%) as colorless oil. $[\alpha]^{23}_D$ -1.73 (*c* 10.5, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 9H), 3.57 (m, 2H), 3.72 (br s, 1H), 3.74 (br s, 1H), 3.92 (m, 1H), 4.54 (s, 2H), 7.25-7.46 (m, 11H), 7.64-7.66 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 19.2, 26.8, 64.7, 70.79, 70.94, 73.4, 127.6, 127.7, 128.4, 129.8, 133.1, 135.5, 138.0; FAB-MS m/z 421.1 ($[M + H]^+$, calcd 421.2).
To a suspension of NaH (1.4 g, 41.7 mmol) in THF (40 mL) at room temperature was added dropwise 1.7 g of the above alcohol, and the resulting solution was stirred for 45 min. To this alkoxide solution was added allyl bromide (2.6 mL, 30 mmol), and the resulting mixture was stirred for 4 h. It was diluted with Et_2O and washed with water,

and the extract was dried over $MgSO_4$ and concentrated. Purification of the residue by flash chromatography (20:1, hexanes-ethyl acetate) afforded **2** (11.9 g, 93%) as colorless oil. $[\alpha]^{23}_D$ -7.65 (*c* 10.09, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.05 (s, 9H), 3.43-3.75 (m, 5H), 4.08 (br s, 1H), 4.09 (br s, 1H), 4.54 (s, 2H), 5.11-5.27 (m, 2H), 5.84-5.88 (m, 1H), 7.26-7.42 (m, 11H), 7.65-7.68 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 19.1, 26.7, 63.4, 70.1, 71.2, 73.3, 78.6, 116.5, 127.4, 127.5, 127.6, 128.2, 129.6, 133.3, 133.4, 135.1, 135.5, 138.3; FAB-MS m/z 461.2 ($[M + H]^+$, calcd 461.2).

***(S)*-1-*O*-Benzyl-3-*O*-(*tert*-butyldiphenylsilyl)-2-*O*-(2-hydroxyethyl)glycerol (3a).** To a solution of **2** (11.9 g, 25.9 mmol), *N*-methylmorpholine *N*-oxide (4.7 g, 40 mmol), THF (40 mL), *t*-BuOH (15 mL), and water (7 mL) at 0 °C was added OsO_4 (2.6 g, 2.5 wt % solution in *t*-BuOH, 0.26 mmol, 1 mol %), and the resulting mixture was stirred for 24 h at room temperature. The reaction mixture was treated with a saturated aqueous $NaHSO_3$, solution and it was vigorously stirred for 1 h. The mixture was poured into water, and then extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over $MgSO_4$, and concentrated to give the corresponding diol, which was subjected to the next step without purification. To a THF (40 mL) solution of the above diol was added portionwise dry $Pb(OAc)_4$ (11.5 g, 25.9 mmol) while the temperature was kept below 10 °C. The reaction mixture was stirred for 30 min with an ice-water bath and additional 30 min without. After filtering through Celite and cooling in an ice bath, $NaBH_4$ (1.9 g, 50 mmol) in 4% aqueous NaOH (40 mL) solution was added dropwise with vigorous stirring while the temperature was kept below 10 °C. The reaction mixture was stirred 0 °C for 30 min and then at room temperature for 90 min. The pH of the reaction mixture was adjusted to about 8 by adding solid ammonium chloride, and it was extracted with ethyl acetate. The organic layer was washed with 5%

aqueous NaOH solution, and then brine. Drying over $MgSO_4$, concentration, and purification by flash chromatography (4:1, hexanes-ethyl acetate) afforded **3a** (9.8 g, 81%) as colorless oil. $[\alpha]^{23}_D$ -10.94 (*c* 10.05, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 9H), 3.60-3.74 (m, 9H), 4.55 (s, 2H), 7.33-7.45 (m, 11H), 7.66-7.70 (m, 4H); ^{13}CNMR ($CDCl_3$) δ 19.1, 26.7, 62.0, 63.7, 70.3, 71.9, 73.4, 80.0, 127.6, 127.7, 128.3, 129.7, 133.1, 133.2, 135.5, 137.7; FAB-MS m/z 465.1 ($[M + H]^+$, calcd 465.2).

(*S*)-1-*O*-Benzyl-3-*O*-*tert*-butyldiphenylsilyl-2-*O*-[2-(*p*-toluenesulfonyloxy)ethyl]glycerol (3b). To a solution of **3a** (9.8 g, 21 mmol) in pyridine (30 mL) at 0 °C, was added portionwise *p*-toluenesulfonyl chloride (4 g, 21 mmol) with stirring, and the resulting mixture was kept in a refrigerator (0-5 °C) for 16 h. The reaction mixture was diluted with ether (50 ml), washed successively with 1 *N* HCl and a saturated aqueous $NaHCO_3$ solution. The organic layer was dried over $MgSO_4$, concentrated, and purified by flash chromatography (9:1, hexanes-ethyl acetate) to afford **3b** (11.3 g, 87%) as colorless oil. $[\alpha]^{23}_D$ -5.65 (*c* 11.92, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.15 (s, 9H), 2.43 (s, 3H), 3.59-3.88 (m, 7H), 4.22 (m, 2H), 4.59 (s, 2H), 7.29-7.51 (m, 13H), 7.76-7.86 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 19.0, 21.3, 26.6, 63.4, 67.7, 69.3, 69.9, 73.1, 80.0, 127.4, 127.5, 127.7, 128.1, 129.5, 132.9, 133.1, 133.2, 135.3, 135.4, 138.0, 144.4; FAB-MS m/z 619.2 ($[M + H]^+$, calcd 619.3).

***N*-2-{[(2-Hydroxy)ethoxy]ethyl}-*p*-toluenesulfonamide (4)**. To a solution of 2-[(2-amino)ethoxy]ethanol (5.35 g 50 mmol) in water (50 mL) at room temperature was added Na_2CO_3 (12.2 g, 115 mmol), and the resulting suspension was stirred until it became solution. To this alkoxide solution was added *p*-toluenesulfonyl chloride (10.5 g, 55 mmol) dissolved in benzene (25 mL). The reaction mixture was stirred at room temperature overnight. It was diluted with benzene (100 ml), and the organic layer was

separated and washed with brine. Drying over $MgSO_4$, concentration, and purification by flash chromatography (1:1, hexanes-ethyl acetate) afforded **4** (8.8 g, 68%) as colorless oil. ^{1}H NMR ($CDCl_3$) δ 2.44 (s, 3H), 3.11 (m, 2H), 3.46-3.51 (m, 4H), 3.67-3.70 (m, 2H), 7.26-7.30 (m, 2H), 7.73-7.76 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.5, 42.9, 61.6, 69.2, 72.2, 127.1, 129.7, 136.9, 143.4; FAB-MS m/z 260.0 ($[M + H]^+$, calcd 260.1).

(*S*)-1-Benzyloxy-2-[(*tert*-butyldiphenylsilyloxy)methyl]-6-*N*-(*p*-toluenesulfonyl)-6-aza-3,9-dioxa-11-undecanol (5a) : A mixture of sulfonamide **4** (4.8 g, 18.3 mmol), tosylate **3b** (11.3 g, 18.3 mmol), and anhydrous K_2CO_3 (7.6 g, 55 mmol) in DMF (100 mL) was refluxed for 2 days with vigorous stirring. After cooling to room temperature, the solid was removed by filtration, and the filtered solution was concentrated. The residue was purified by column chromatography (1:1, hexanes-ethyl acetate) to give **5a** (8.4 g, 65%) as colorless oil. $[\alpha]^{23}_D$ -3.93 (*c* 11.2, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.07 (s, 9H), 2.41 (s, 3H), 3.38-3.76 (m, 17H), 4.54 (s, 2H), 7.25-7.46 (m, 13H), 7.67-7.73 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 19.6, 21.9, 27.2, 49.0, 49.2, 62.1, 63.7, 69.8, 70.1, 70.4, 72.7, 73.8, 80.4, 127.6, 128.00, 128.04, 128.1, 128.8, 130.0, 130.1, 133.75, 133.79, 136.0, 137.3, 138.6, 143.6; FAB-MS m/z 706.3 ($[M + H]^+$, calcd 706.3); HRMS calcd 706.3234 for $C_{39}H_{52}NO_7SSi$ ($[M + H]^+$), found 706.3259.

(*S*)-2-[(*tert*-Butyldiphenylsilyloxy)methyl]-6-*N*-(*p*-toluenesulfonyl)-6-aza-3,9-dioxa-1,11-undecanediol (5b) : An ethanol (50 mL) solution of tosylate **5a** (8.4 g, 11.9 mmol) was subjected to hydrogenolysis in the presence of 10% Pd/C (1.5 g) for 12 h at room temperature under balloon-filled hydrogen atmosphere. Filtration of the catalyst and evaporation of the solvent gave **5b** in quantitative yield. $[\alpha]^{23}_D$ -10.87 (*c* 7.56, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 9H), 2.42 (s, 3H), 3.16-3.76 (m, 17H), 7.28-7.31 (m, 2H), 7.38-7.45 (m, 6H), 7.66-7.71 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 19.6, 21.9, 27.2, 49.4, 49.9,

62.0, 62.9, 63.6, 69.7, 70.3, 73.1, 81.8, 127.7, 128.2, 130.1, 130.2, 133.5, 133.6, 136.0, 136.5, 143.9; FAB-MS m/z 616.2 ($[M + H]^+$, calcd 616.3).

(*R*)-1,11-Bis(methansulfonyloxy)-2-[(*tert*-butyldiphenylsilyloxy)methyl]-6-*N*-(*p*-toluenesulfonyl)-6-aza-3,9-dioxaundecane (6). To a pyridine (20 mL) solution of **5b** (7.3 g, 11.9 mmol) at 0 °C was added methanesulfonyl chloride (2.8 g, 24 mmol), and the resulting mixture was stirred overnight. The reaction mixture was poured into a mixture of ice (50 g) and 2 *N* HCl (25 mL), and then it was extracted with CH_2Cl_2 (3 x 40 mL). The organic layer was washed with brine, dried over $MgSO_4$, and concentrated. Purification of the residue by flash chromatography (4:1, hexanes-ethyl acetate) gave **6** (7.4 g, 81%). $[\alpha]^{23}_D$ -8.07 (*c* 9.00, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.07 (s, 9H), 2.41 (s, 3H), 3.018 (s, 3H), 3.021 (s, 3H), 3.30-3.37 (m, 4H), 3.60-3.71 (m, 9H), 4.27-4.30 (m, 3H), 4.41-4.46 (m, 1H), 7.27-7.30 (m, 2H), 7.39-7.47 (m, 6H), 7.65-7.70 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 19.6, 21.9, 27.2, 37.8, 38.0, 49.2, 49.4, 62.3, 69.1, 69.3, 69.7, 70.4, 78.7, 127.5, 128.3, 130.1, 130.4, 133.2, 133.3, 135.9, 136.9, 143.9; FAB-MS m/z 772.2 ($[M + H]^+$, calcd 772.2).

(*S*)-2-[(*tert*-Butyldiphenylsilyloxy)methyl]-4,10,16-tris-(*p*-toluenesulfonyl)-4,10,16-triaza-1,7,13-trioxacyclooctadecane (8): Bis-sulfonamide **7**(0.1 g, 0.24 mmol) and dimesylate **6** (0.18 g, 0.24 mmol) in benzene (1.4 ml) are added to a refluxing mixture of tetra-*n*-butylammonium iodide (25% mol), 7 % aqueous lithium hydroxide (1.4 ml), and benzene (9 ml). The vigorously stirred mixture is heated under reflux for 7 days, the organic layer is separated and the solvent is removed under reduced pressure. The solid is washed with methanol and filtered off. Purification by flash chromatography on silica gel (2:1, hexane-ethyl acetate) afforded **8** (0.1 g, 45 %). $[\alpha]^{23}_D$ -15.7 (c 0.25, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 1.0 (s, 9 H), 2.3 (s, 3H), 2.42 (d, 6H), 2.9-3.0 (m, 1H), 3.2-

3.6 (m, 23 H), 3.7 (t, 1H) 7.3-7.4 (m, 12 H), 7.6 (q, 10 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 19.1, 21.4, 26.7, 45.9, 49.3, 49.5, 49.7, 51.4, 63.5, 66.0, 69.5, 70.0, 70.5, 70.6, 70.8, 77.4, 80.0, 126.9, 127.1, 127.7, 129.6, 129.7, 129.8, 133.0, 135.4, 136.0, 136.3, 136.5, 143.2, 143.3 ; FAB-MS *m/z* 991.39 ($[M+H]^+$, calcd 991.35).

[4,10,16-Tris-(*p*-toluenesulfonyl)-4,10,16-triaza-1,7,13-trioxa-cyclooctadec-2-yl]-methanol (9) : A solution 58 mg (0.06 mmol) of **8** in 5 ml of THF was treated with 0.03 ml of a 1.0 M solution of tetrabutylammonium fluoride in tetrahydrofuran at room temperature for 3 h. The reaction mixture was dried over $MgSO_4$ and concentrated. Purification by flash chromatography on silica gel (1:1, hexane-ethyl acetate) afforded **9** (40 mg, 91%). mp 69.0-70.3 °C; $[\alpha]^{23}_D$ -7.57 (c 0.81, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.44 (s, 9H), 3.24-3.40 (m, 12H), 3.53-3.80 (m, 13 H), 7.32 (t, 6H), 7.68 (q, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.4, 29.6, 49.4, 49.6, 49.8, 50.0, 50.1, 60.6, 68.5, 70.4, 70.5, 70.6, 70.9, 79.1, 126.9, 127.0, 127.1, 129.7, 129.8, 135.6, 135.8, 136.3, 143.4, 143.5, 143.6 ; FAB-MS *m/z* 754.25 ($[M+H]^+$, calcd 753.25) ; HRMS cacld 754.2502 for $C_{34}H_{47}N_3O_{10}S_3$ ($[M+H]$)$^+$, found 753.2511.

(*S*)-1-*O*-(*tert*-Butyldiphenylsilyl)-3-*O*-(*p*-toluenesulfonyl)2-*O*-[2-(*p*-toluenesulfonyloxy)ethyl]- glycerol (10)

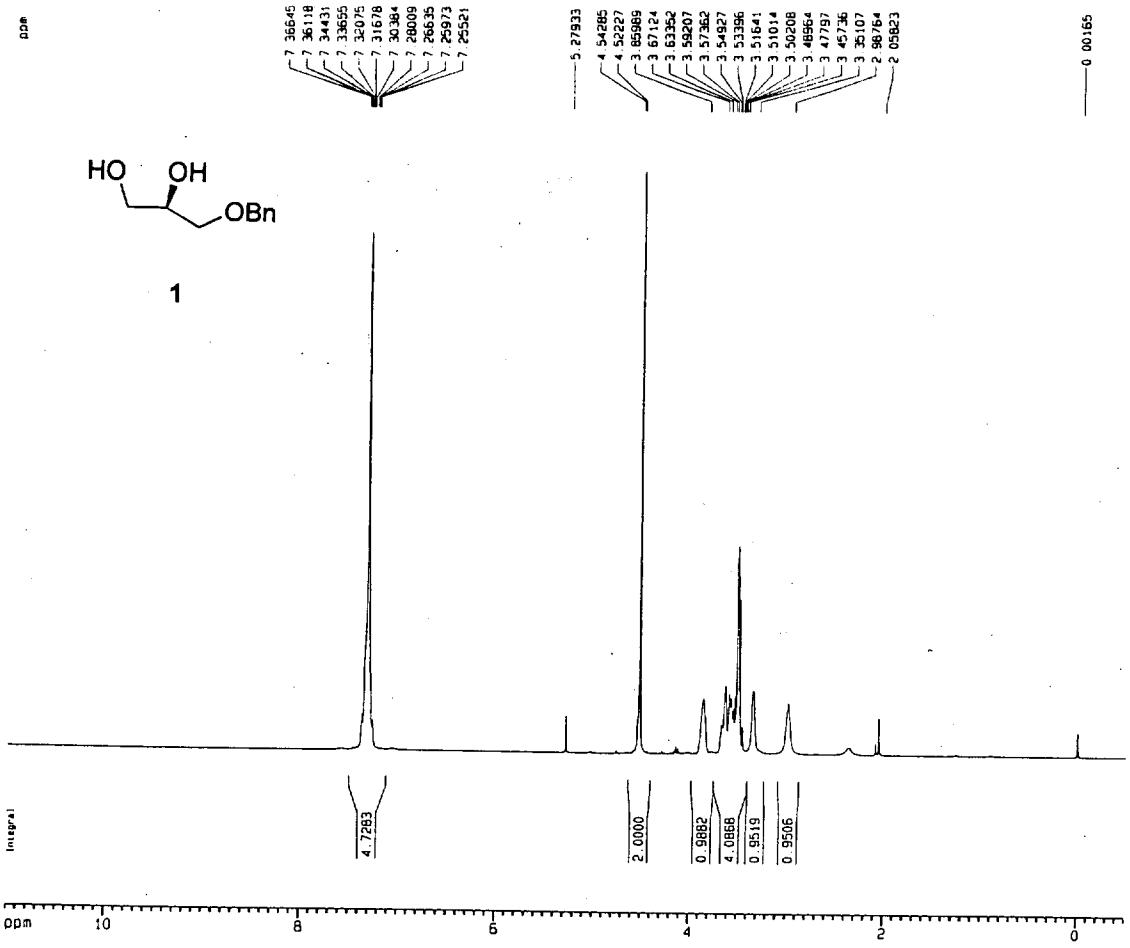
To the solution of 0.8 g (1.8 mmol) of **3a** in 10 ml of EtOH was added 0.25 g of 10 % Pd/C. The atmosphere of the reaction was exchanged for hydrogen gas and the reaction mixture was stirred at room temperature for 24 h. The dispersion was filtered off and washed with CH_2Cl_2 several times. The resulting solution was concentrated under reduced pressure. Purification by flash chromatography on silica gel (2:1, hexane-ethyl acetate) afforded diol (0.6 g, 94%). $[\alpha]^{23}_D$ -17.6 (c 2.10 : $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.05 (s, 9H), 3.52-3.73 (m, 11H), 7.37 (q, 6H), 7.67 (d, 4H); ^{13}C NMR

($CDCl_3$, 75 MHz) δ 19.0, 26.7, 61.8, 62.6, 63.4, 71.5, 81.1, 127.6, 129.7, 133.0, 133.1, 135.5 ; FAB-MS *m/z* 375.12 ($[M+H]^+$, calcd 374.55). To an ice-cooled solution of 0.9 g (2.58 mmol) of diol in 10 ml of pyridine was added portionwise with stirring 1.2 g (6.2 mmol) of *p*-toluenesulfonyl chloride. After standing in a refrigerator for 24 h, the reaction mixture was diluted with ether, saturated with NaCl, dried ($MgSO_4$), and concentrated. Purification by flash chromatography on silica gel (3:1, hexane-ethyl acetate) afforded **10** (1.5 g, 83%). $[\alpha]^{23}_D$ -9.35 (c 4.28, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 1.02 (s, 9H), 2.40 (d, 6H), 3.54-3.60 (m, 5H), 4.02 (t, 3H), 4.16-4.20 (dd, 1H), 7.25-7.44 (m, 10H), 7.59 (d, 4H), 7.76 (t, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.8, 21.3, 21.4, 26.5, 61.9, 67.7, 68.9, 69.2, 78.0, 127.6, 127.70, 127.74, 129.6, 129.7, 132.4, 132.5, 132.6, 132.7, 135.2, 135.3, 144.6, 144.7; FAB-MS *m/z* 682.89 ($[M+H]^+$, calcd 682.92).

(*S*)-2-[(*tert*-Butyldiphenylsilyloxy)methyl]-4,10-bis(*p*-toluenesulfonyl)-4,10-diaza-1,7-dioxacyclododecane (11). Bis-sulfonamide **7** (1.0 g, 4 mmol) and ditosylate **10** (1.7 g, 25 mmol) in benzene (15 ml) are added to a refluxing mixture of tetra-*n*-butylammonium iodide (25% mol), 7 % aqueous lithium hydroxide (15 ml), and benzene (90 ml). The vigorously stirred mixture is heated under reflux for 7 days, the organic layer is separated and the solvent is removed under reduced pressure. The solid is washed with methanol and filtered off. Purification by flash chromatography on silica gel (3:1, hexane-ethyl acetate) afforded **11** (1.3 g, 70%). mp 65.3-67.3 °C; $[\alpha]^{23}_D$ -16.7 (c 2.10, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 1.05 (s, 9H), 2.43 (s, 6H), 2.99-3.02 (m, 2H), 3.16-3.24 (m, 2H), 3.31-3.37 (m, 3H), 3.47-3.72 (m, 10H), 7.63-7.72 (m, 8H); ^{13}C NMR ($CDCl_3$, 75 MHz) 19.0, 21.2, 26.6, 49.7, 50.3, 51.3, 52.7, 58.0, 64.0, 69.3, 69.7, 70.6, 78.0, 78.8, 127.1, 127.3, 127.5, 127.6, 129.5, 129.6, 133.0, 133.1, 134.7, 135.4,

135.6, 143.1, 143.2 ; FAB-MS *m/z* 751.2 ($[M+H]^+$, calcd 751.04).

[4,10-Bis(*p*-toluenesulfonyl)-4,10-diaza-1,7-dioxacyclododec-2-yl]methanol (12) : A solution 0.4 g (0.5 mmol) of **11** in 15 ml of THF was treated with 0.3 ml of a 1.0 M solution of tetrabutylammonium fluoride in tetrahydrofuran at room temperature for 3 h. The reaction mixture was dried over $MgSO_4$ and concentrated. Purification by flash chromatography on silica gel (1:4, hexane-ethyl acetate) afforded **12** (0.27 g, 99%): mp 175.2-177.0 °C; $[\alpha]^{23}_D$ +15.6 (c 0.83, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 2.44 (s, 6H), 3.04-3.36 (m, 7H), 3.53-4.09 (m, 10 H), 7.29-7.34 (m, 4H), 67.69 (d, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.4, 50.7, 51.4, 51.5, 51.9, 62.7, 67.3, 70.5, 71.0, 78.3, 127.31, 127.33, 129.7, 129.8, 134.8, 134.9, 143.5, 143.6 ; FAB-MS *m/z* 513.17 ($[M+H]^+$, calcd 513.17) ; HRMS cacld 513.1729 for $C_{23}H_{32}N_2O_7S_2$ $([M+H])^+$, found 513.1744. Crystal Data: M_r = 512.63, monoclinic, $P2_1$, a = 6.0853(4) Å, α = 90°, b = 13.7666(9) Å, β = 95.5070(10)°, c = 14.4567(10) Å, γ = 90°, V = 1205.50(14) $Å^3$, Z = 2, D_{calcd} = 1.412 Mg/m^3, F(000) = 544, μ(Mo Kα) = 3.2 cm^{-1}, λ = 0.71073 Å, T = 243 K, R1 = 0.0417 and wR2 = 0.1121 for 2816 absorption corrected reflections with $I>2\sigma(I)$.



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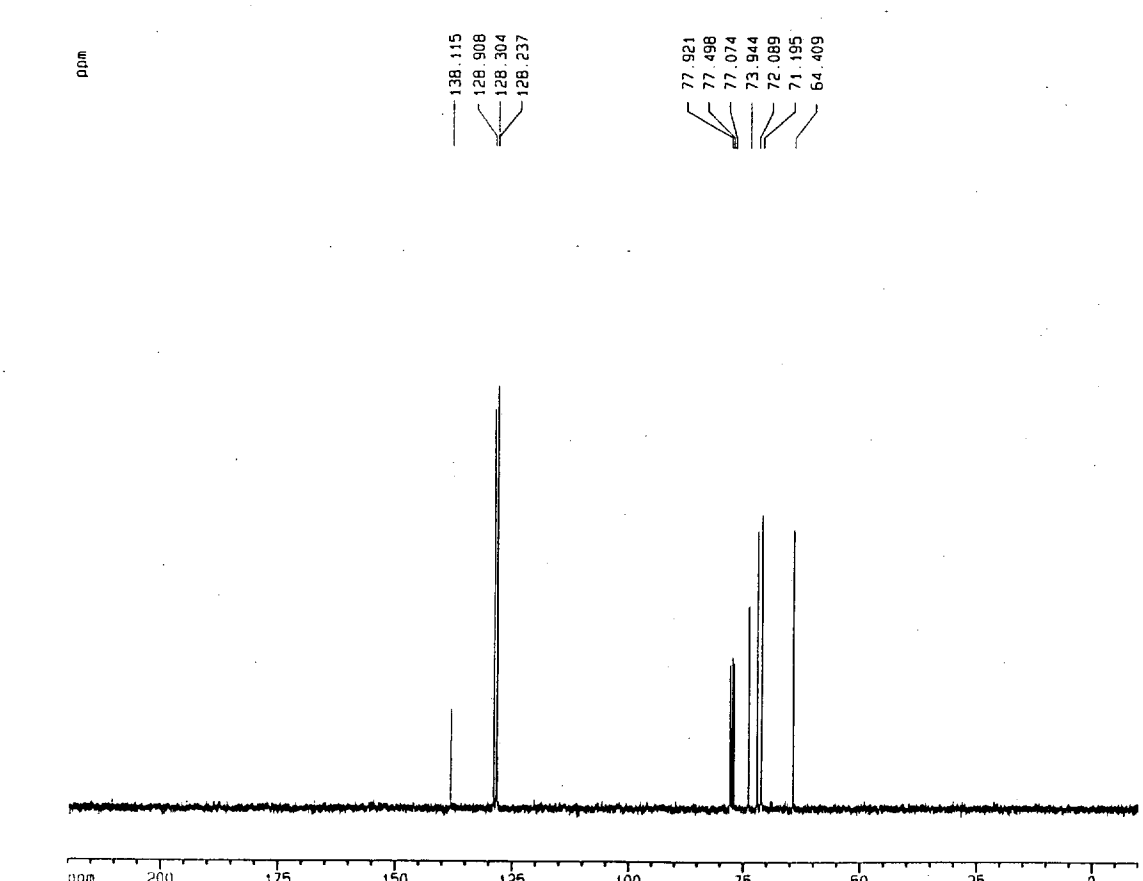

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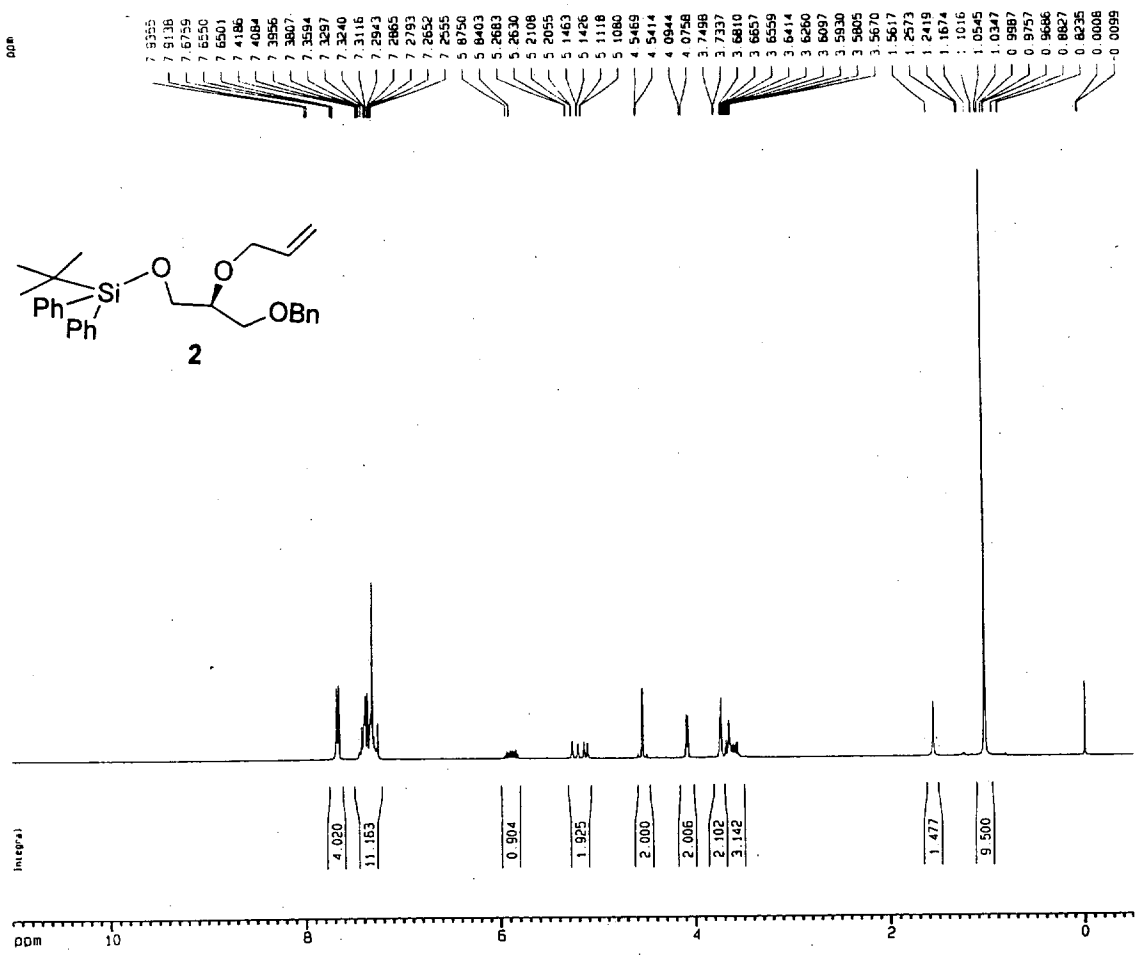

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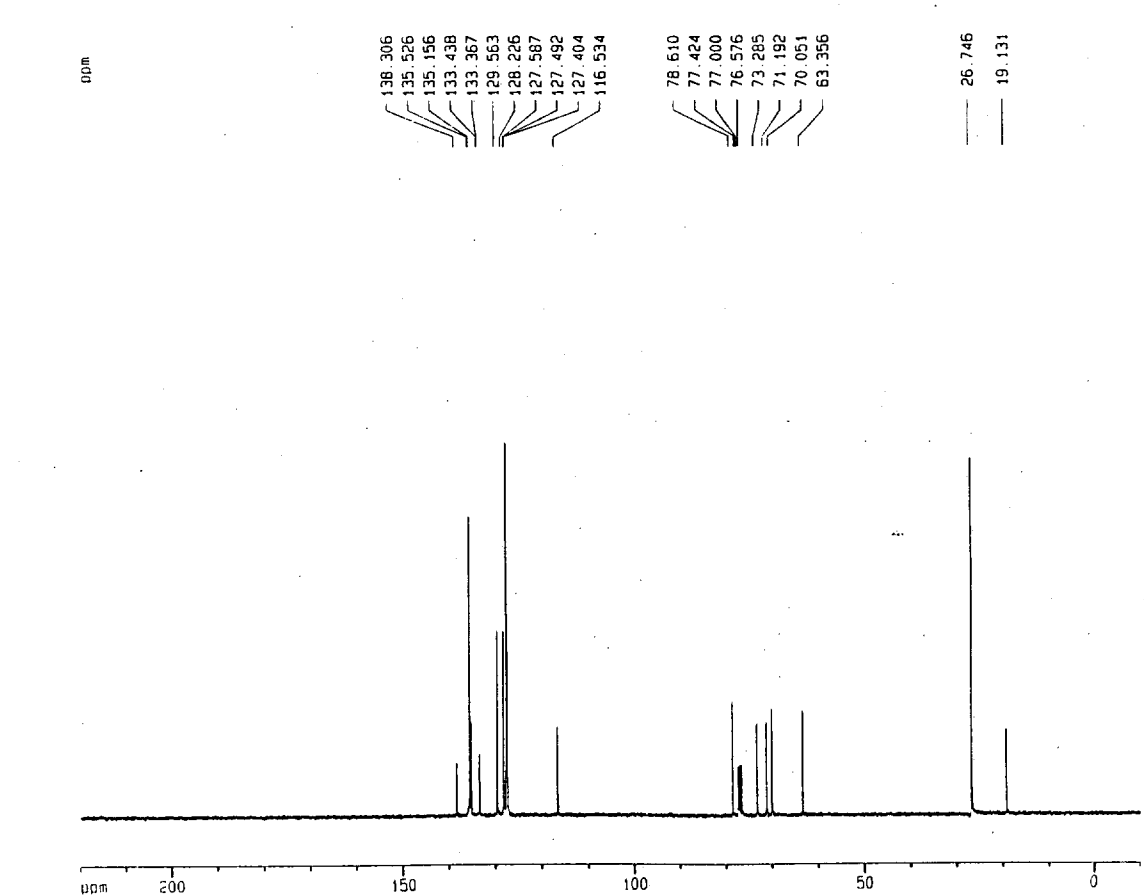

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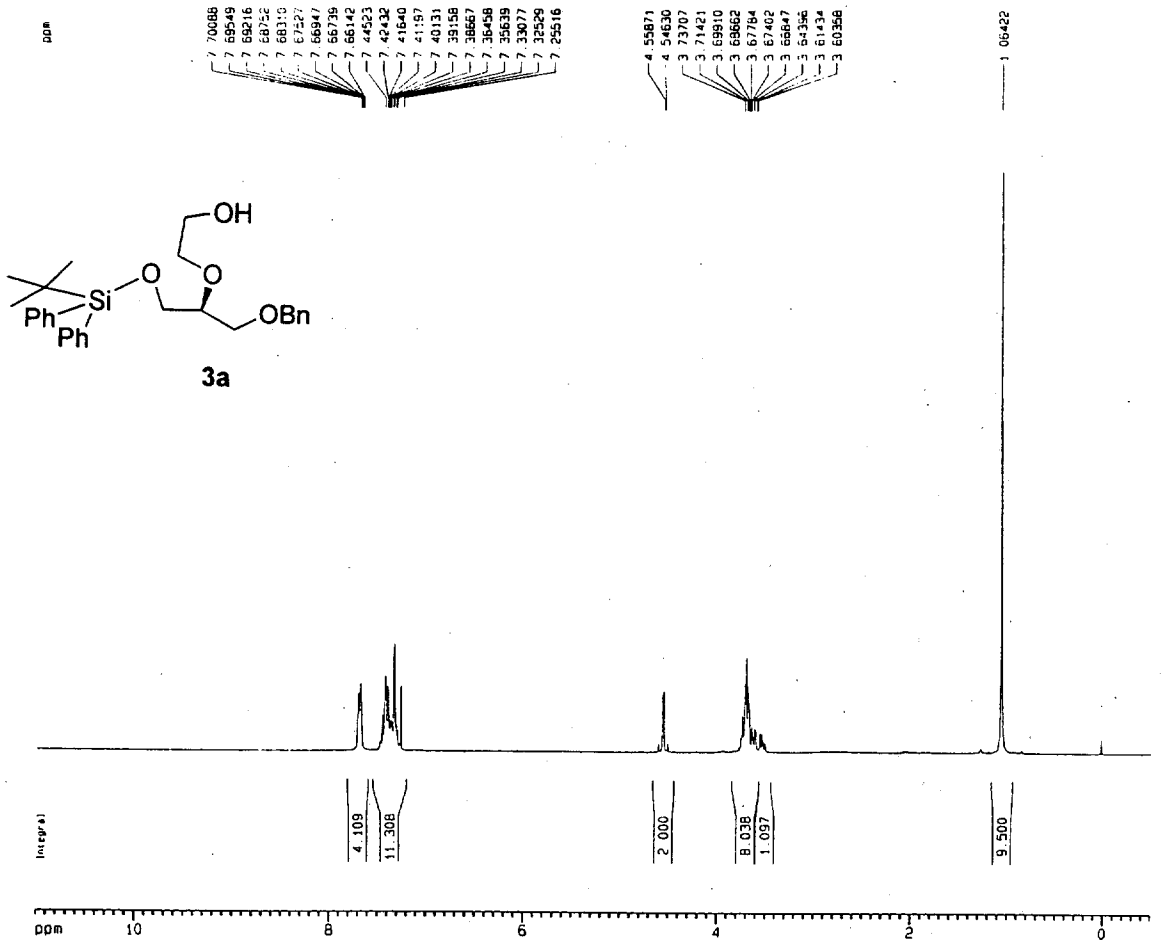
AQ 0.8356340 sec
RG 1149.4
DW 25.500 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -3.00 dB
PL12 16.00 dB
P1 6.10 usec
DE 4.50 usec
SFO1 75.4760988 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677650 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16602.91 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.87933 Hz/cm

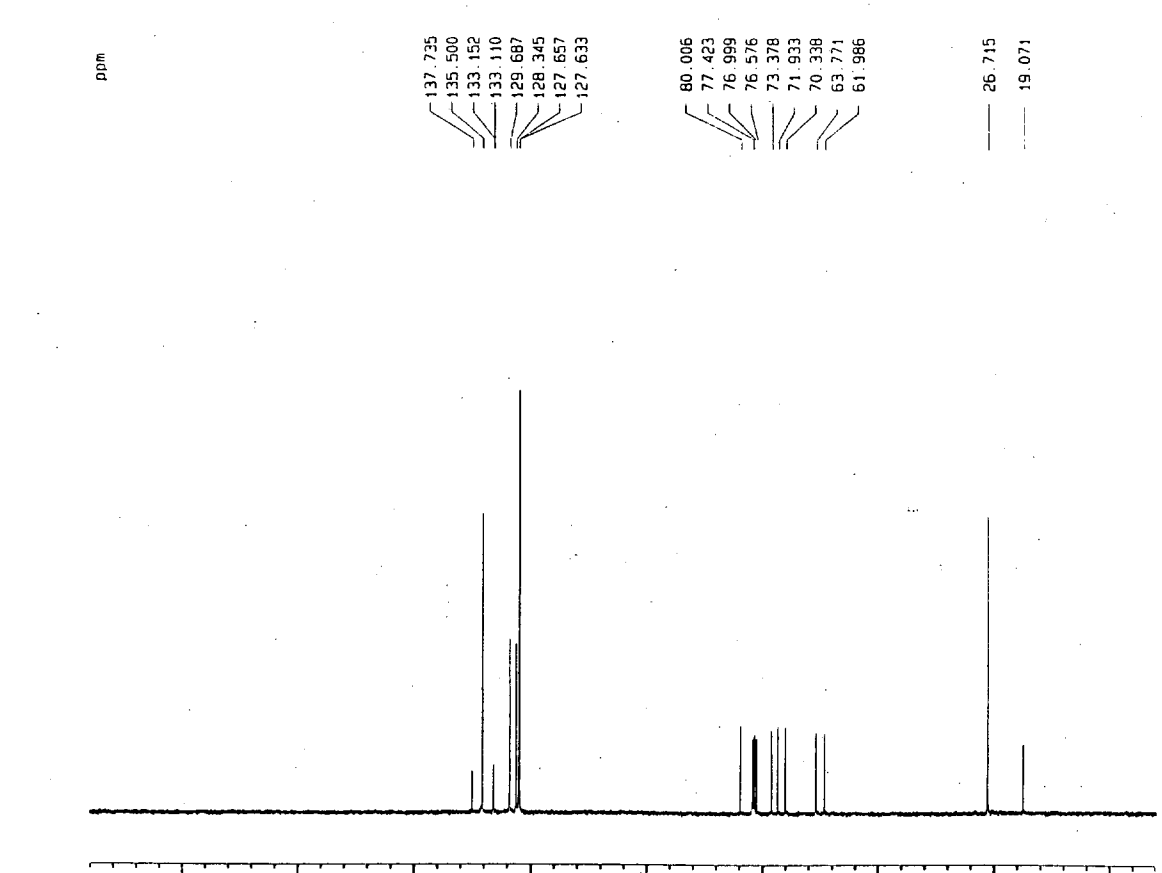


Current Data Parameters
NAME alcohol
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980501
Time 14 04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5787 037 Hz
FIDRES 0 353213 Hz
AQ 1.4156276 sec
RG 64
DW 86 400 usec
DE 4 50 usec
TE 300 0 K
D1 1 00000000 sec
P1 12 60 usec
DE 4 50 usec
SFO1 300 1319886 MHz
NUC1 1H
PL1 -3 00 dB

F2 - Processing parameters
SI 16384
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0 20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 11.000 ppm
F1 3301 43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0 54762 ppm/cm
HZCM 164.35690 Hz/cm

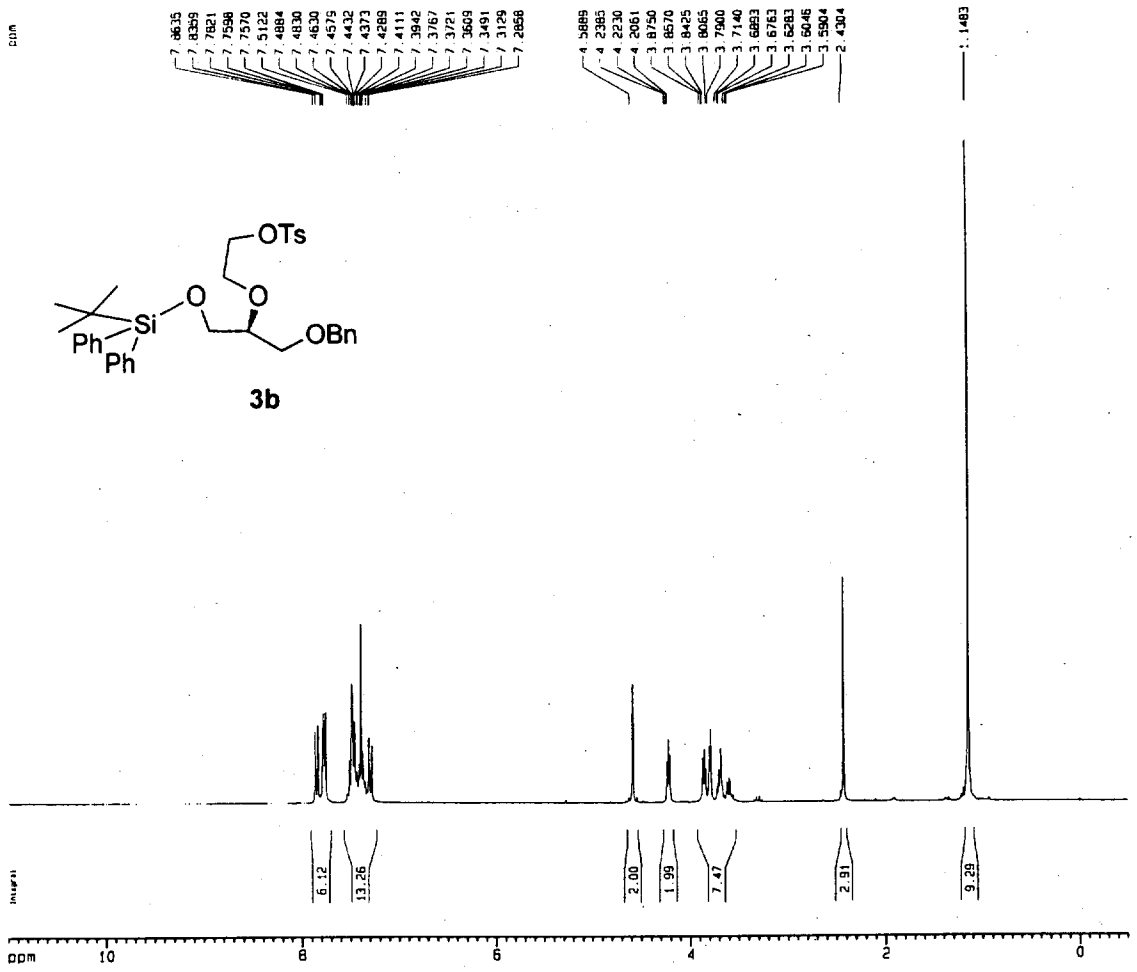


Current Data Parameters
NAME alcohol
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980430
Time 21 16
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 240
DS 2
SWH 19607 844 Hz
FIDRES 0 598384 Hz
AQ 0 8356340 sec
RG 8192
DW 25 500 usec
DE 4 50 usec
TE 300 0 K
D12 0.00002000 sec
PL13 17 00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300 1301000 MHz
NUC2 1H
PL2 -3 00 dB
PL12 16.00 dB
P1 6 10 usec
DE 4 50 usec
SFO1 75.4760988 MHz
NUC1 13C
PL1 -3 00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75 4677590 MHz
WDW EM
SSB 0
LB 1 20 Hz
GB 0
PC 1 40

1D NMR plot parameters
CX 20 00 cm
F1P 220 000 ppm
F1 16602 91 Hz
F2P -10 000 ppm
F2 754 68 Hz

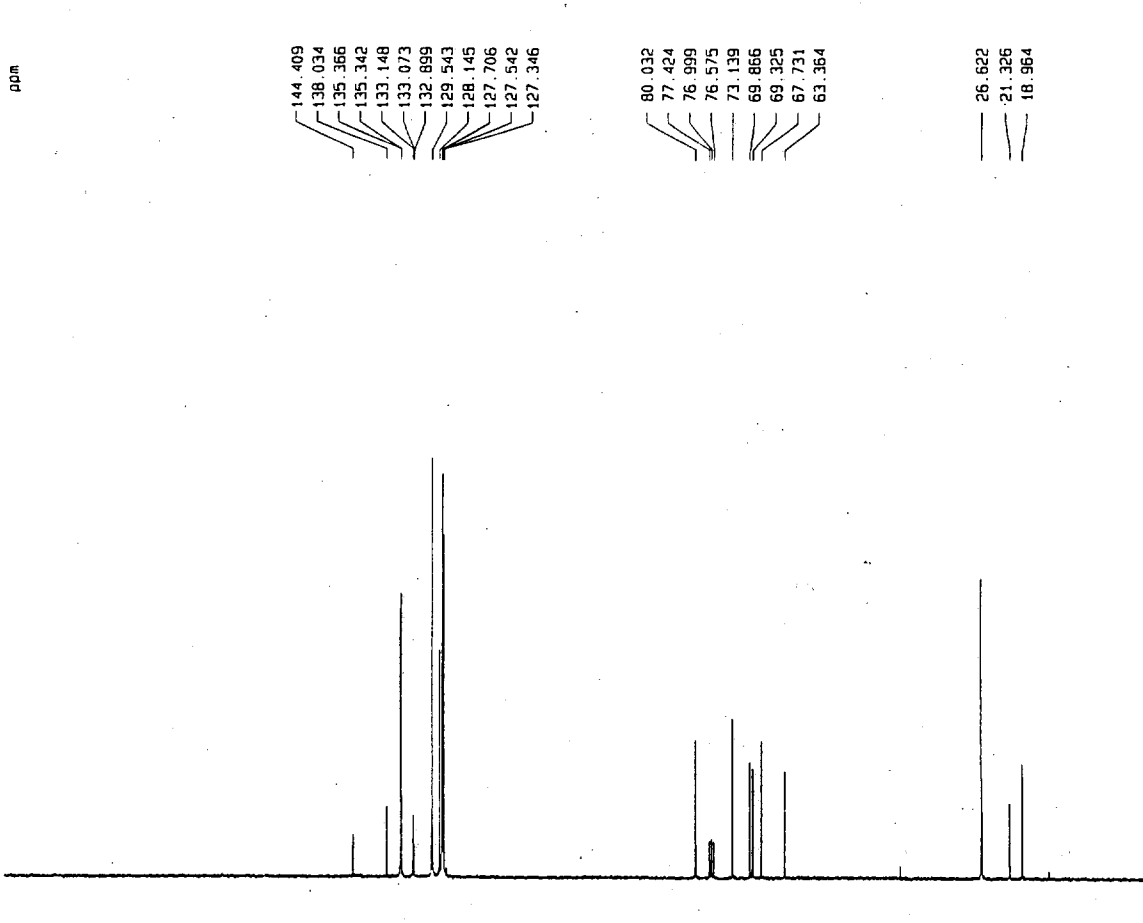


Current Data Parameters
NAME 980706
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980706
Time 15.11
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 35.9
DW 81.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 13.00 usec
DE 4.50 usec
SFO1 300.1318534 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.54762 ppm/cm
HZCM 164.35690 Hz/cm

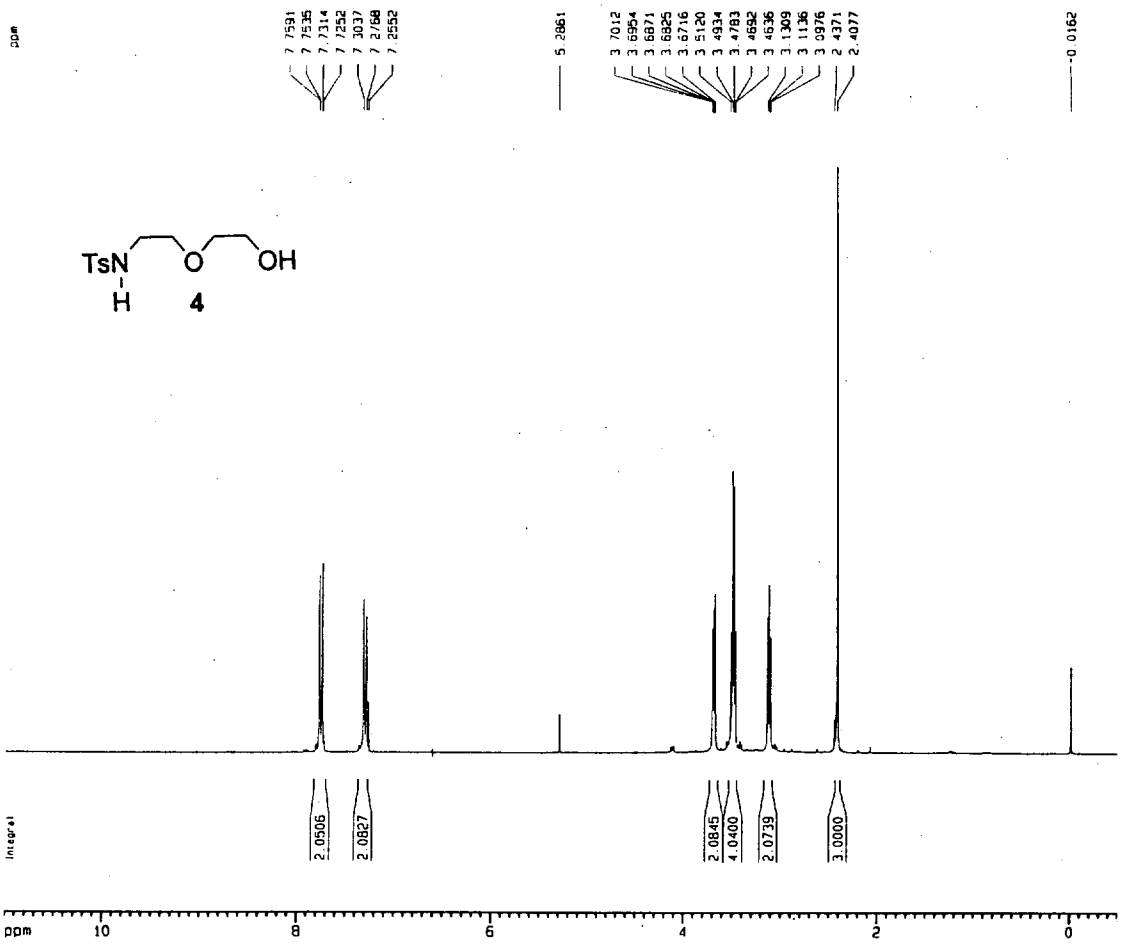


Current Data Parameters
NAME 980706
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 980706
Time 15.15
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 151
DS 2
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 0.6881780 sec
RG 4096
DW 21.000 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 13.50 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 12.00 dB
P1 5.40 usec
DE 4.50 usec
SFO1 75.4772501 MHz
NUC1 13C
PL1 -6.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677734 MHz
WDW EM
SSB 0
LB 1.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 210.000 ppm
F1 15848.23 Hz
F2P -10.000 ppm
F2 -754.68 Hz

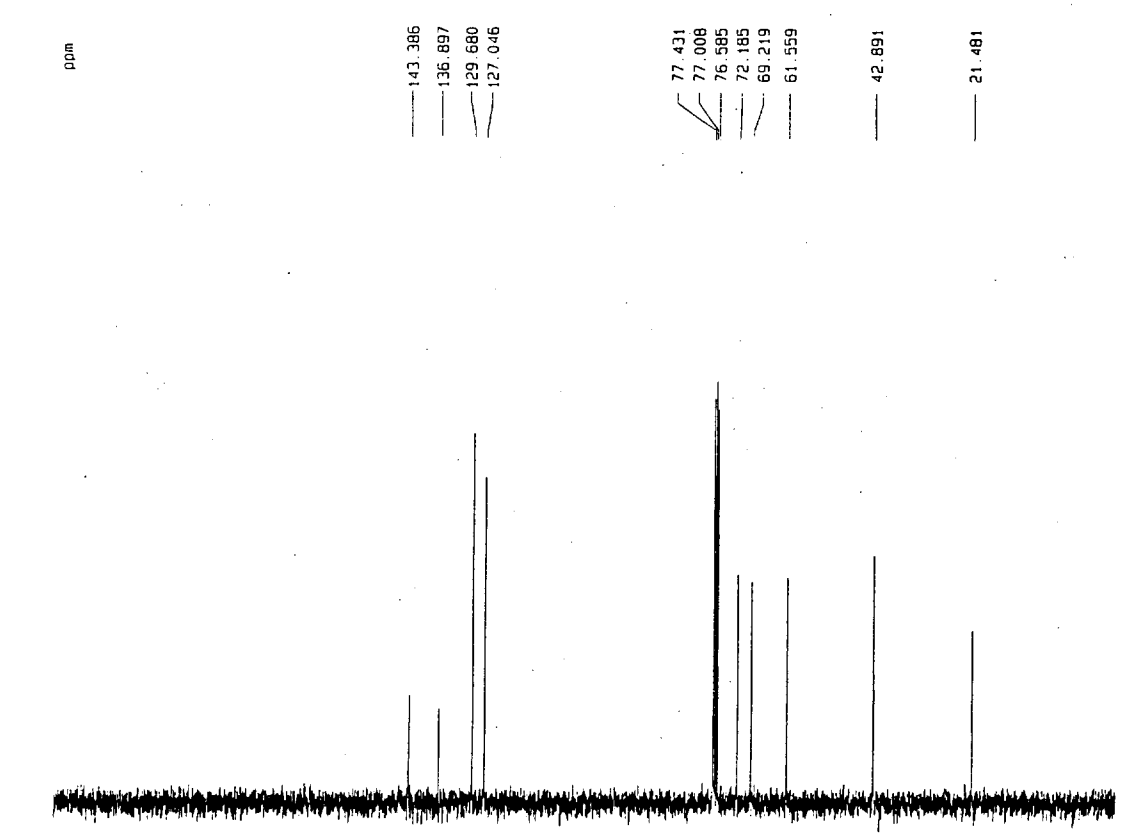


Current Data Parameters
NAME 980603
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980603
Time 13.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 22
DS 0
SWH 5787.037 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 181
DW 86.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 12.60 usec
DE 4.50 usec
SFO1 300.1319886 MHz
NUC1 1H
PL1 -3.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.54762 ppm/cm
HZCM 164.35690 Hz/cm

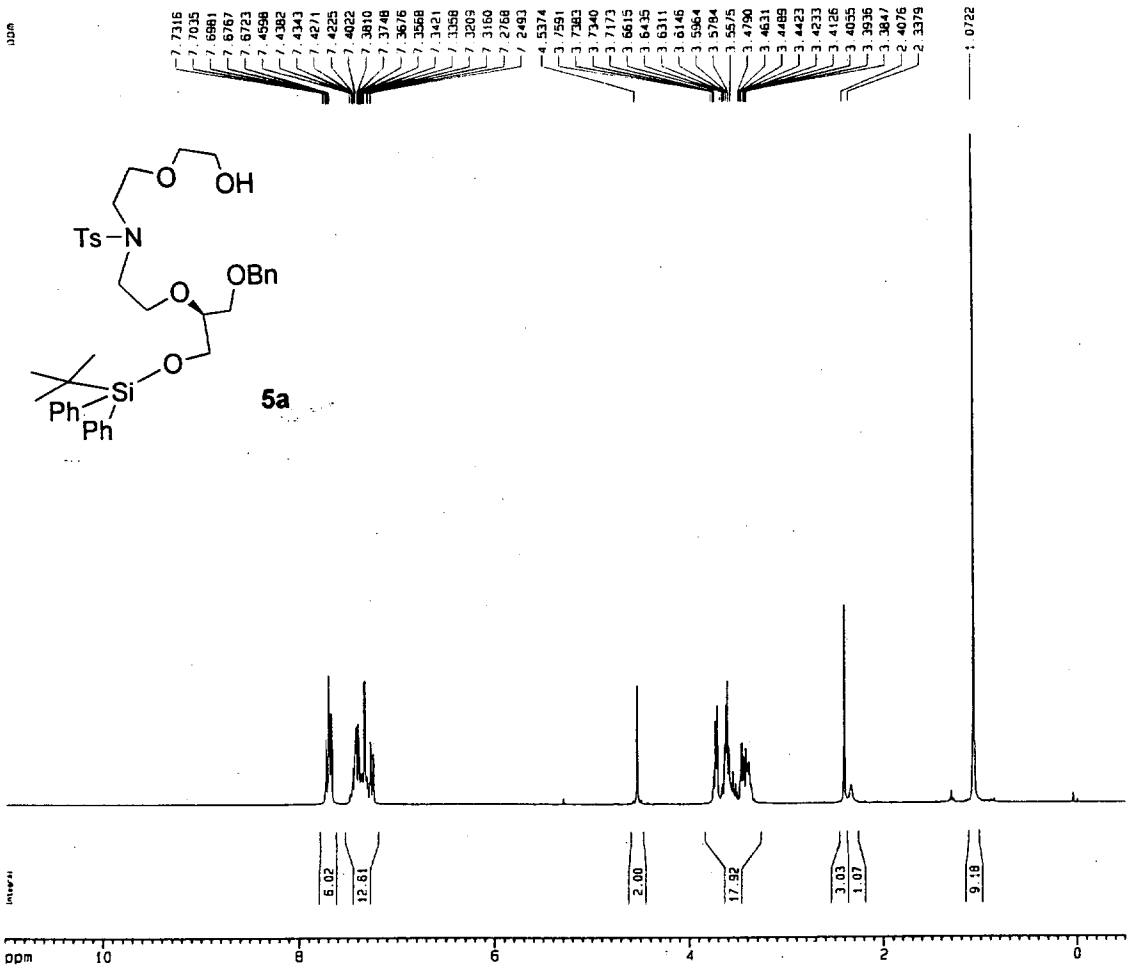


Current Data Parameters
NAME 980603
EXPNO 113
PROCNO 1

F2 - Acquisition Parameters
Date_ 980603
Time 13.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 121
DS 2
SWH 19607.844 Hz
FIDRES 0.598384 Hz
AQ 0.8356340 sec
RG 2298.8
DW 25.500 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -3.00 dB
PL12 16.00 dB
P1 6.10 usec
DE 4.50 usec
SFO1 75.4760988 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677525 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm

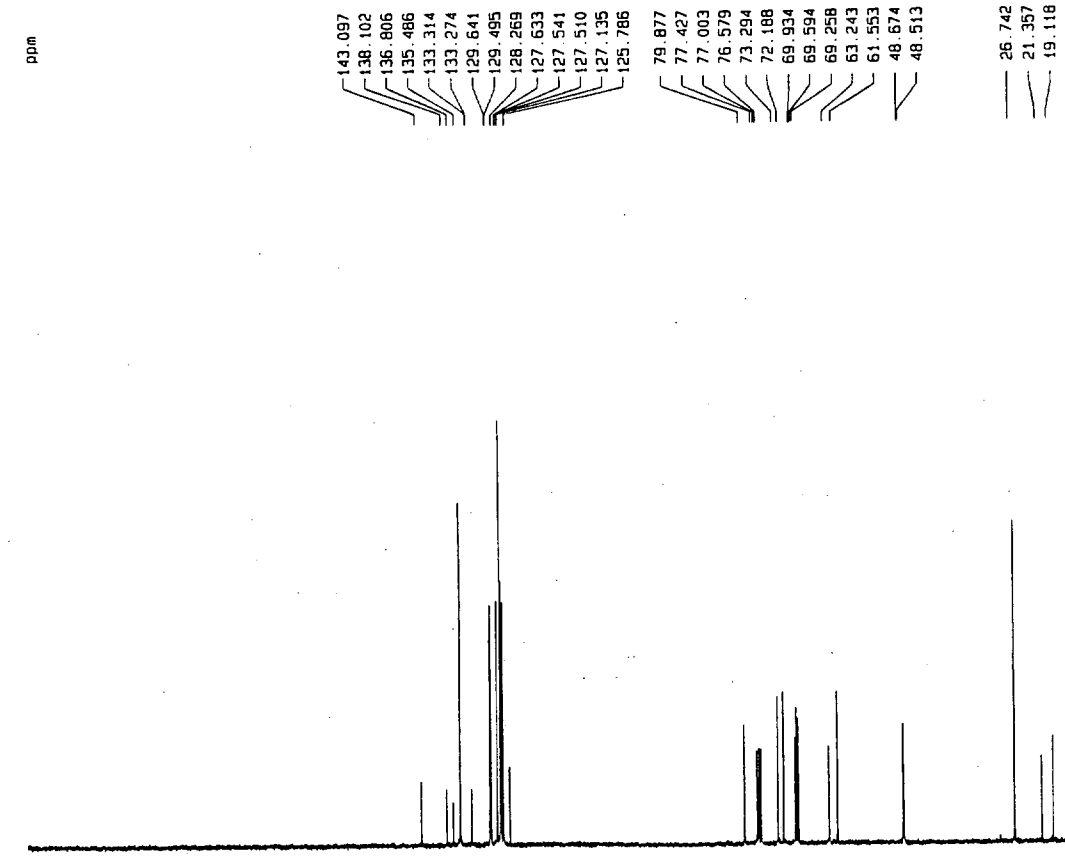


Current Data Parameters
NAME 980722
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 980722
Time 15.43
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 90.5
DW 81.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 13.00 usec
DE 4.50 usec
SFO1 300.1318534 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.54762 ppm/cm
HZCM 164.35690 Hz/cm

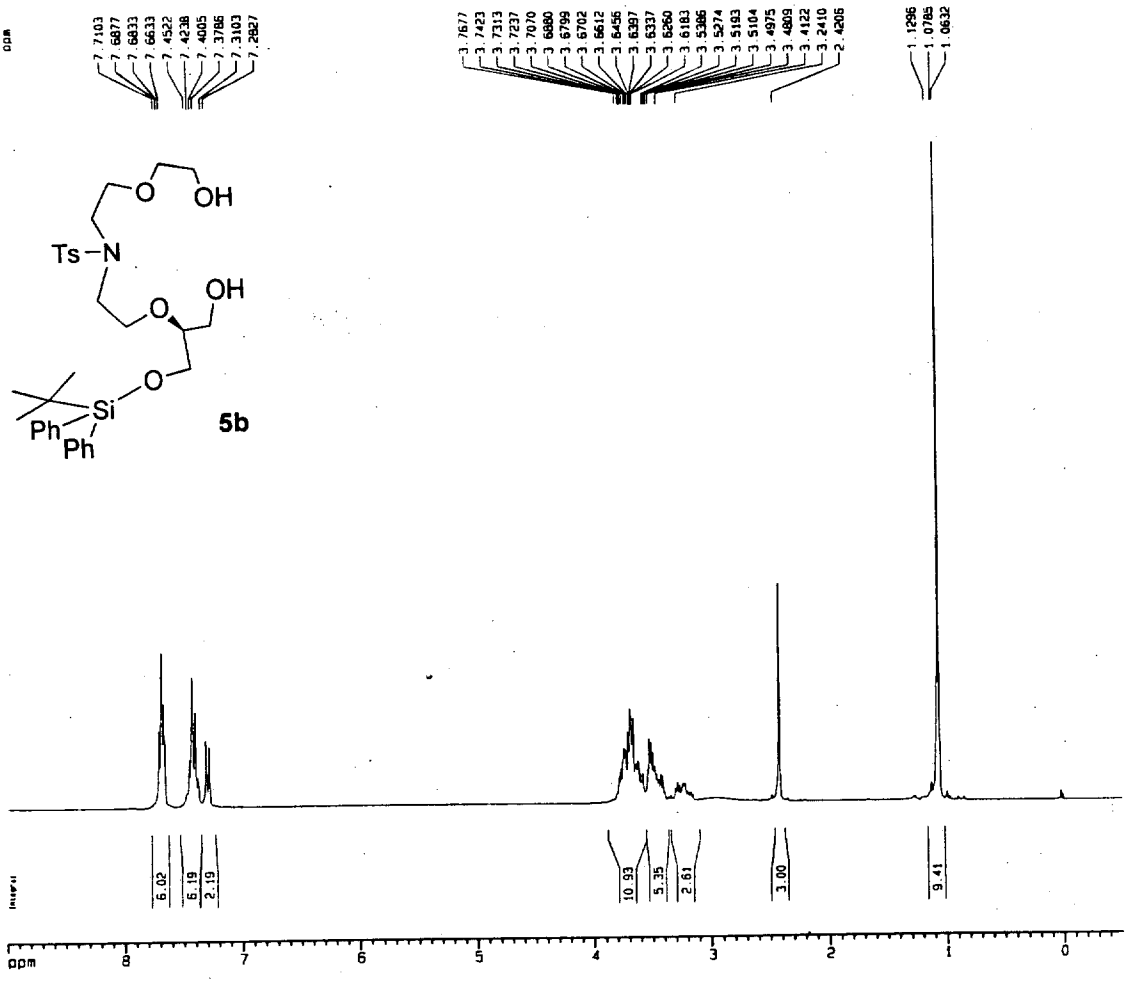


Current Data Parameters
NAME 980722
EXPNO 313
PROCNO 1

F2 - Acquisition Parameters
Date_ 980722
Time 15.47
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 485
DS 2
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 0.6881700 sec
RG 2048
DW 21.000 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 13.50 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 12.00 dB
P1 5.40 usec
DE 4.50 usec
SFO1 75.4772501 MHz
NUC1 13C
PL1 -5.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677574 MHz
WDW EM
SSB 0
LB 1.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 220.000 ppm
F1 16602.91 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.45455 ppm/cm

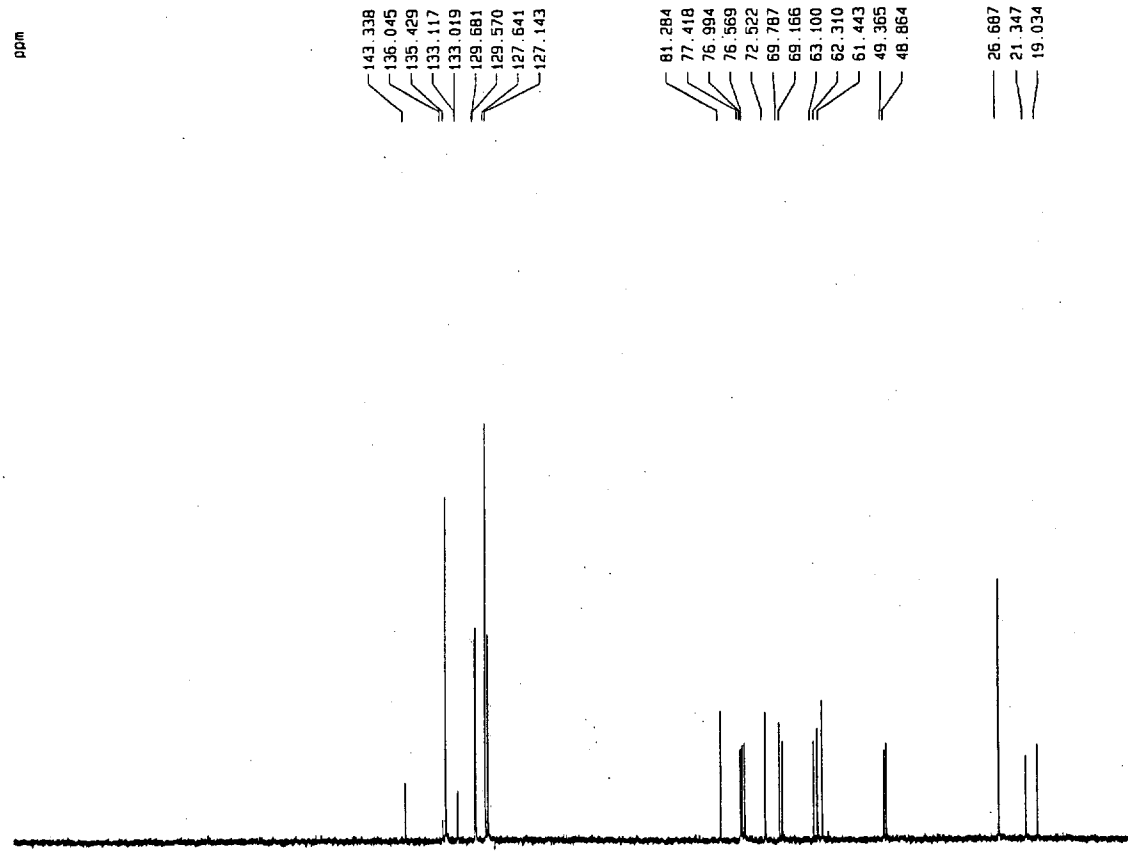


Current Data Parameters
NAME sjong-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980610
Time 14.00
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 90.5
DW 81.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 13.00 usec
DE 4.50 usec
SFO1 300.1318534 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.45238 ppm/cm
HZCM 135.77310 Hz/cm

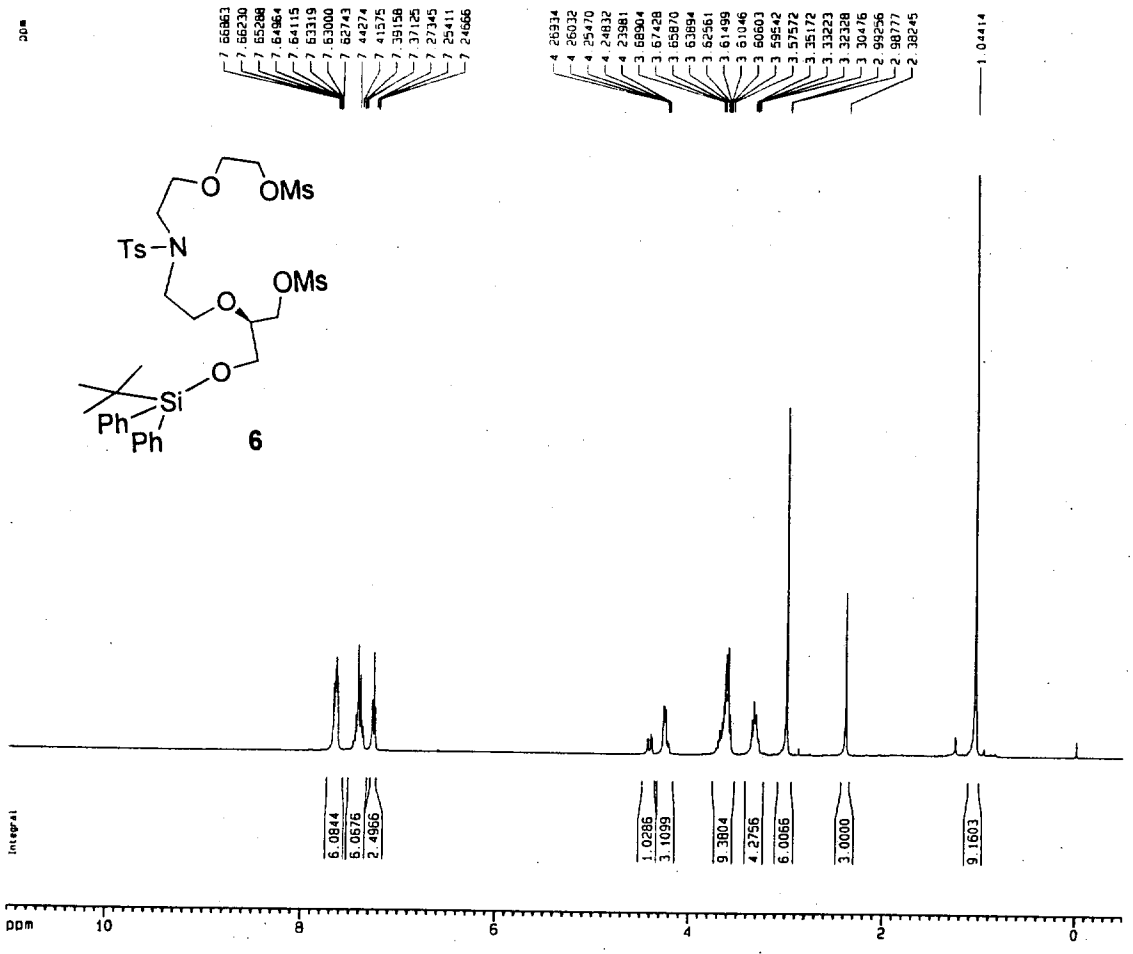


Current Data Parameters
NAME 980723
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 980723
Time 15.42
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 101
DS 2
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 0.6881780 sec
RG 4096
DW 21.000 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 13.50 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 12.00 dB
P1 5.40 usec
DE 4.50 usec
SFO1 75.4772501 MHz
NUC1 13C
PL1 -6.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677595 MHz
WDW EM
SSB 0
LB 1.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 220.000 ppm
F1 16602.91 Hz
F2P -10.000 ppm
F2 -754.68 Hz

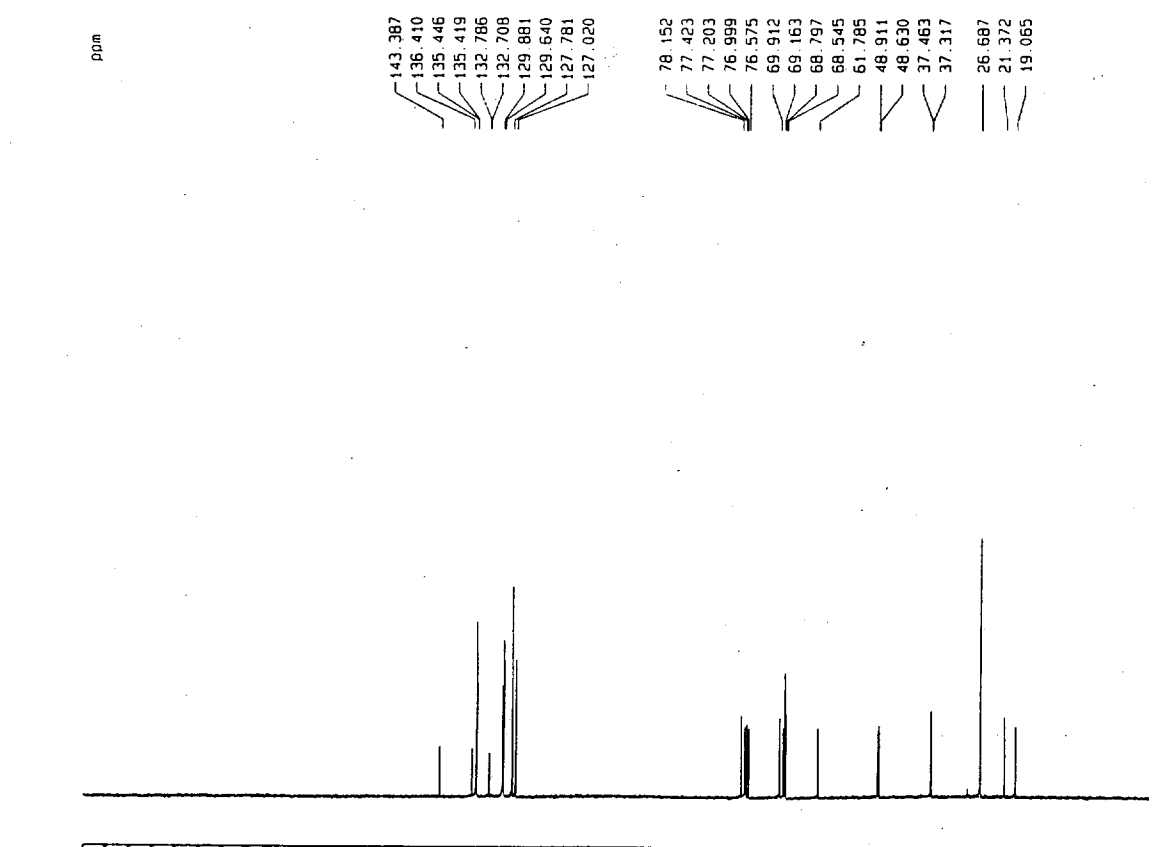


Current Data Parameters
NAME 980725
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980725
Time 10.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 17
DS 0
SWH 5787.037 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 64
DW 86.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 12.50 usec
DE 4.50 usec
SFO1 300.1319886 MHz
NUC1 1H
PL1 -3.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.54762 ppm/cm
HZCM 164.35690 Hz/cm

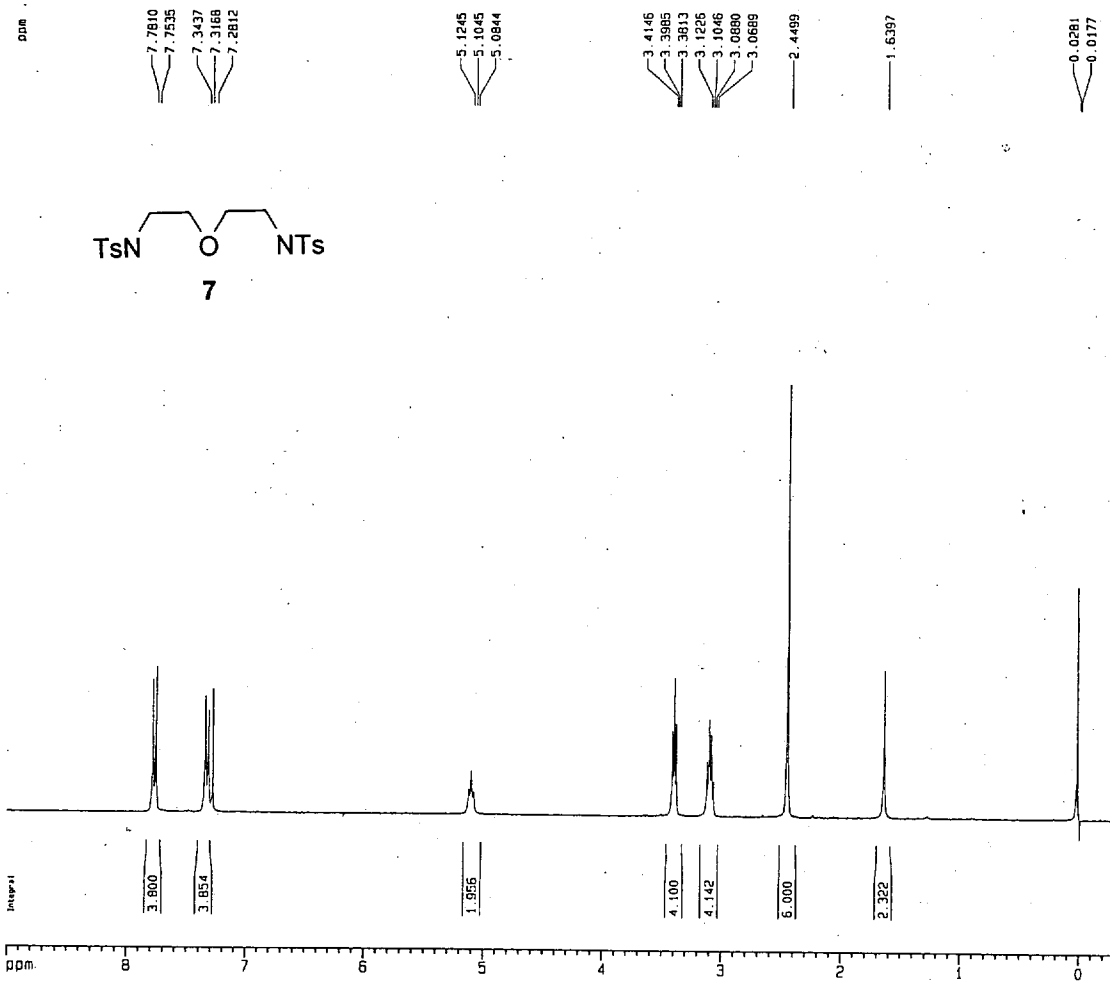


Current Data Parameters
NAME 980725
EXPNO 113
PROCNO 1

F2 - Acquisition Parameters
Date_ 980725
Time 10.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 452
DS 2
SWH 19607.844 Hz
FIDRES 0.598384 Hz
AQ 0.8356340 sec
RG 362
DW 25.500 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -3.00 dB
PL12 16.00 dB
P1 6.10 usec
DE 4.50 usec
SFO1 75.4760988 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677578 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm

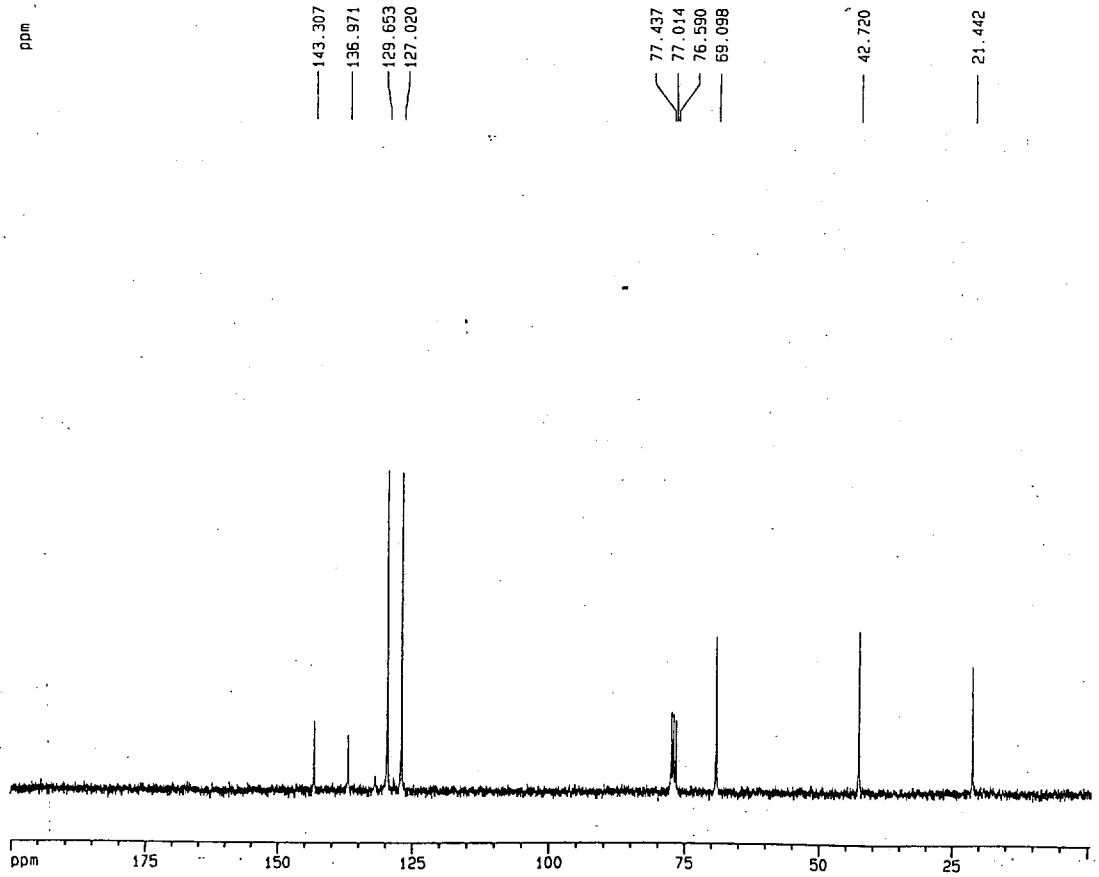


Current Data Parameters
NAME ej-67j-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 990119
Time 14.40
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 1024
DW 81.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 13.00 usec
DE 4.50 usec
SFO1 300.1318534 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.45238 ppm/cm
HZCM 135.77310 Hz/cm

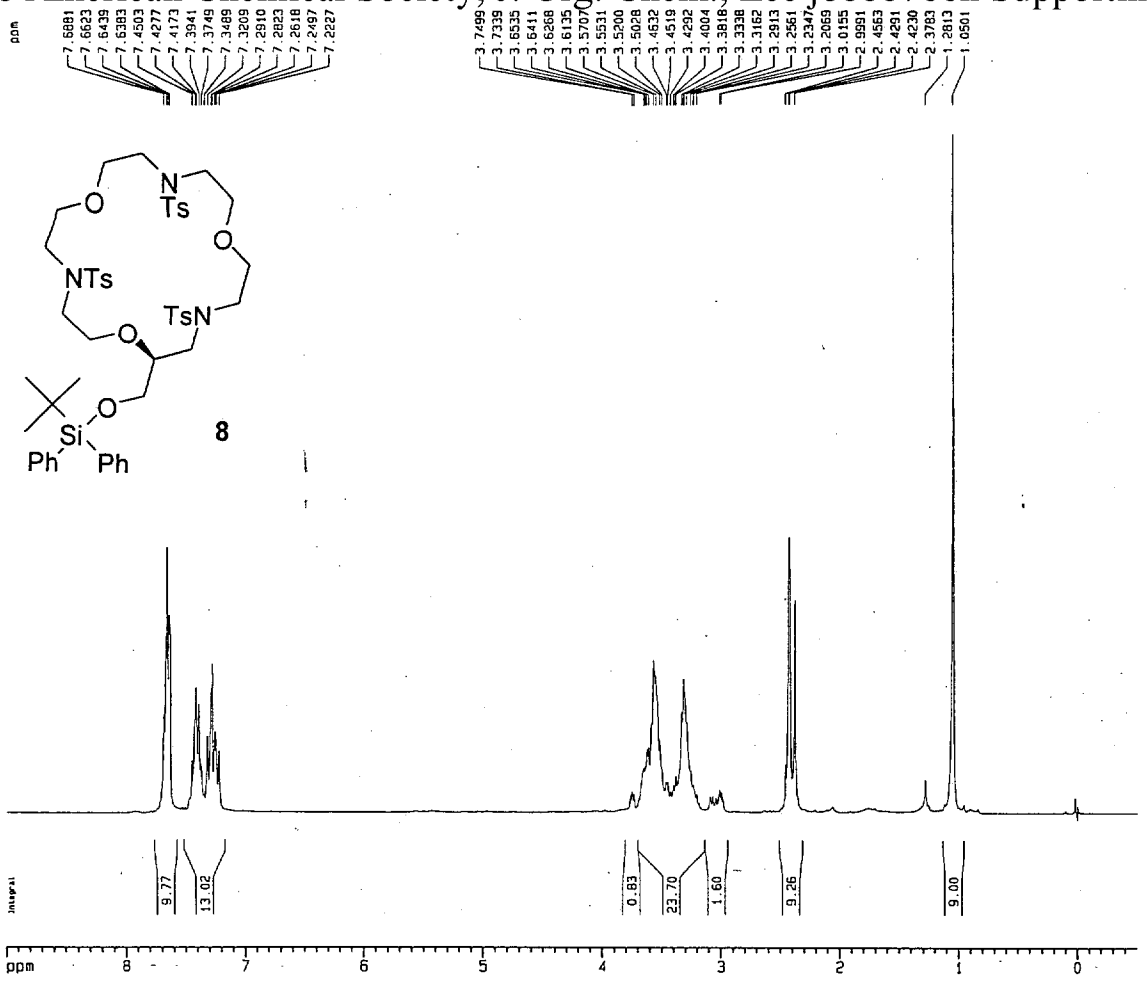


Current Data Parameters
NAME nts
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 16.32
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 300
DS 2
SWH 19607.844 Hz
FIDRES 0.598384 Hz
AQ 0.8356340 sec
RG 5792.6
DW 25.500 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -3.00 dB
PL12 16.00 dB
P1 6.10 usec
DE 4.50 usec
SFO1 75.4760988 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.02500 ppm/cm

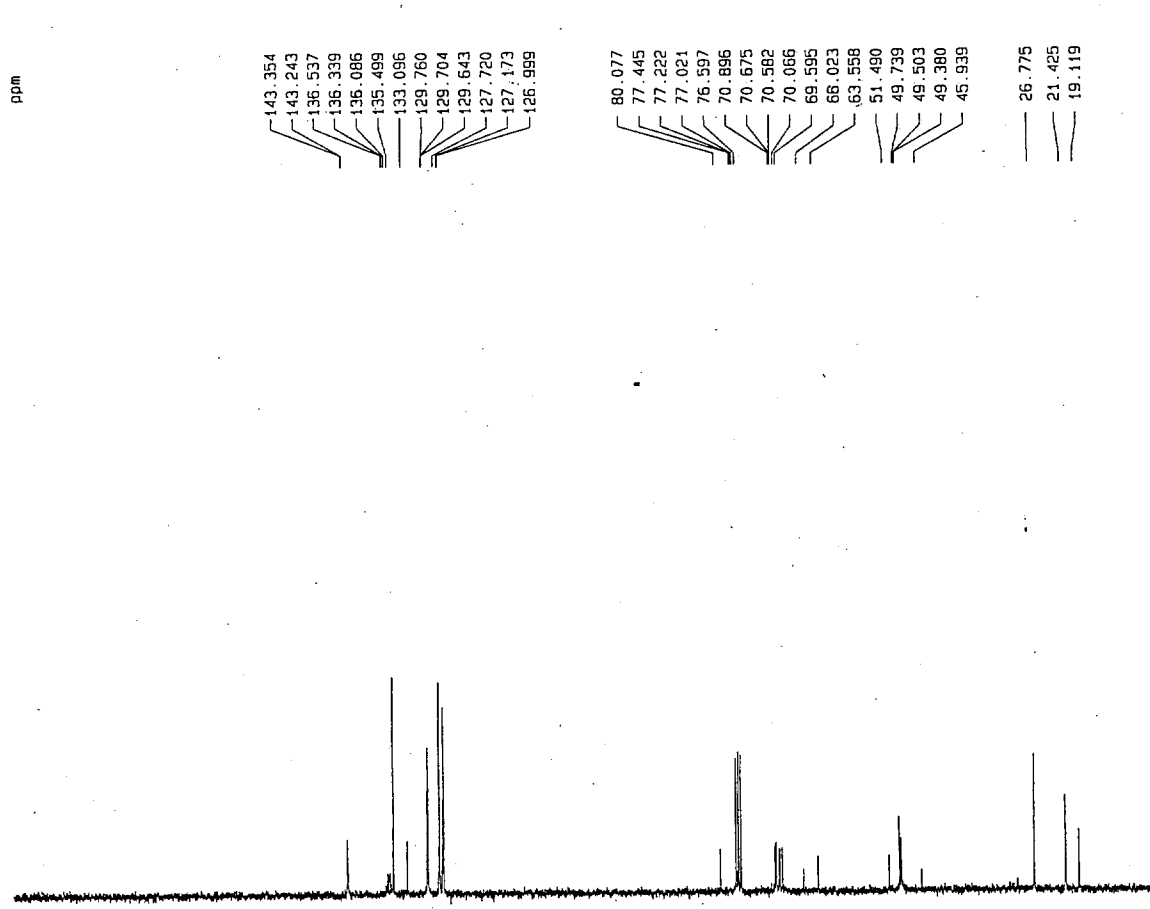


Current Data Parameters
NAME ej2-21-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 990531
Time 15.41
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 90.5
DW 81.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 13.00 usec
OE 4.50 usec
SFO1 300.1318534 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.45238 ppm/cm
HZCM 135.77310 Hz/cm

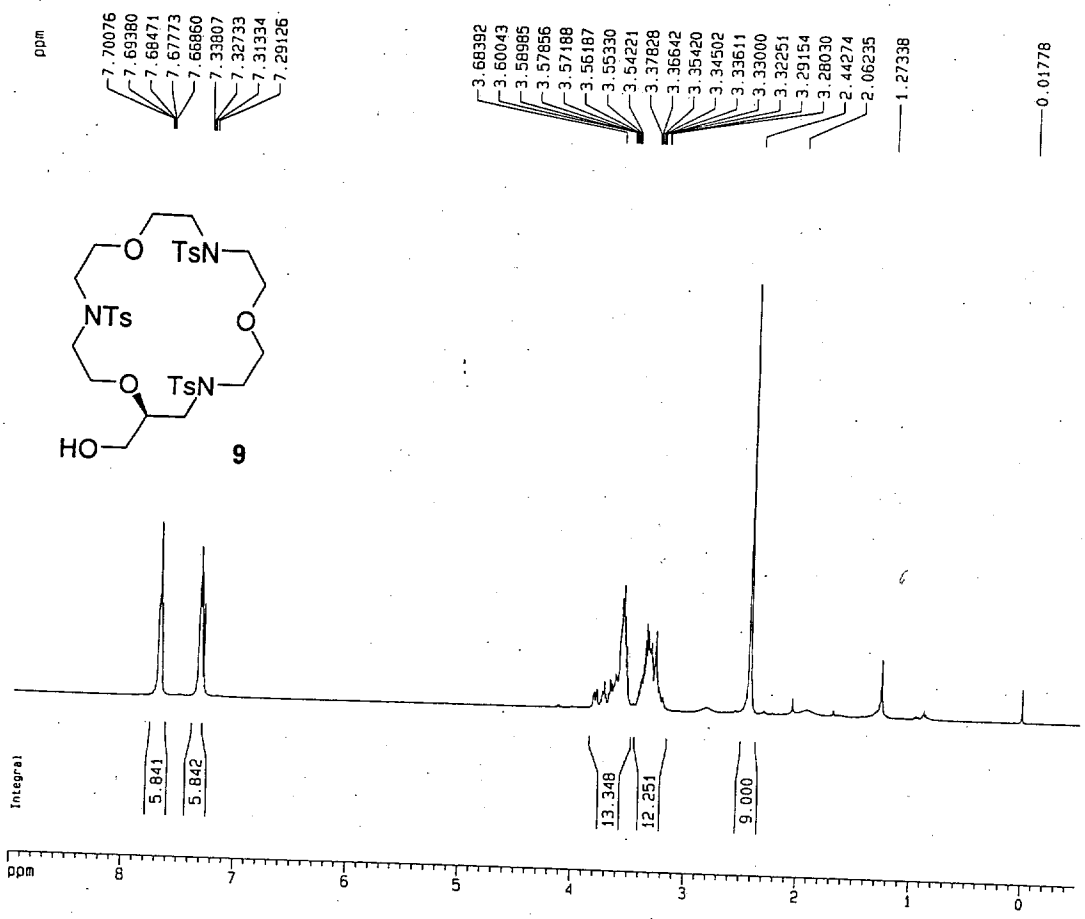


Current Data Parameters
NAME ej2-21-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 990531
Time 15.45
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 241
DS 2
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 0.6881780 sec
RG 8192
DW 21.000 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 13.50 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 12.00 dB
P1 5.40 usec
OE 4.50 usec
SFO1 75.4772501 MHz
NUC1 13C
PL1 -6.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677552 MHz
WDW EM
SSB 0
LB 1.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 9.11364 ppm/cm
HZCM 687.78577 Hz/cm



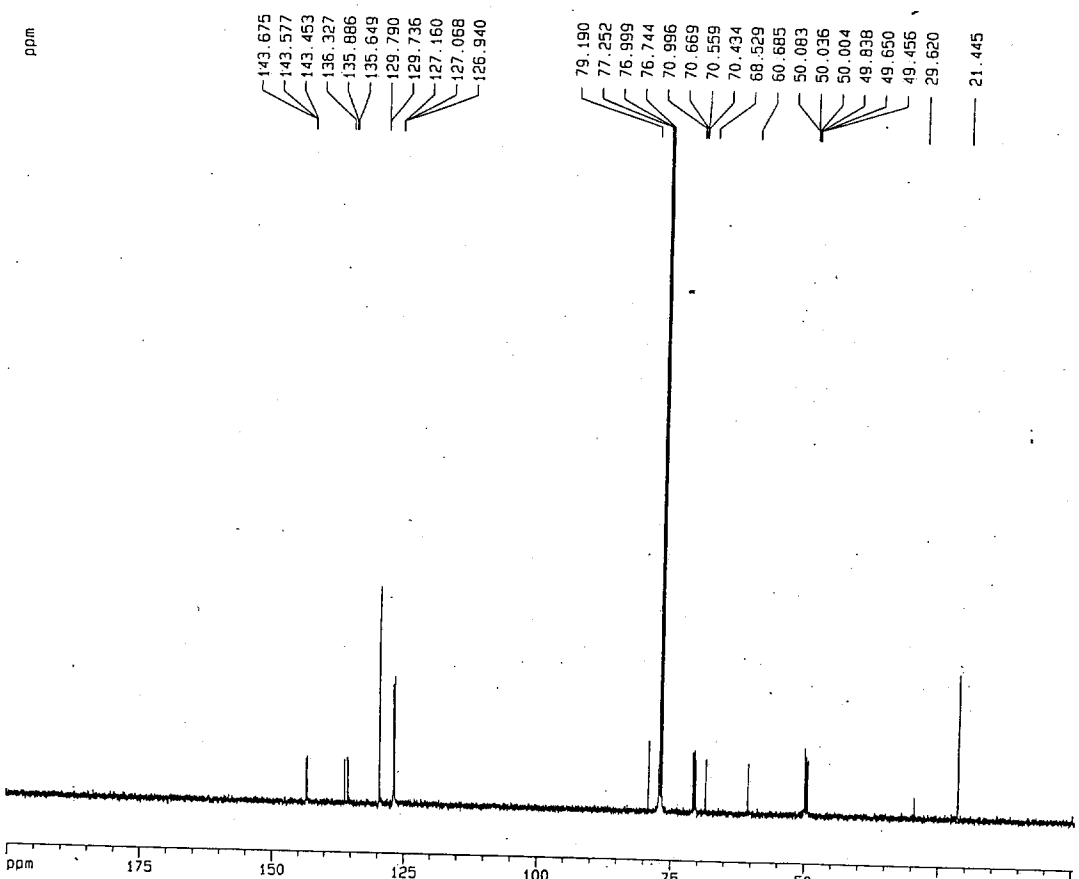
Current Data Parameters
NAME ej2-43-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 990904
Time 10.32
INSTRUM spect
PROBHD 5 mm BBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.576 Hz
FIDRES 0.315264 Hz
AQ 1.5860212 sec
RG 80.6
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

---------- CHANNEL f1 ----------
NUC1 1H
P1 10.00 usec
PL1 5.00 dB
SFO1 500.2330891 MHz

F2 - Processing parameters
SI 32768
SF 500.2300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 4502.07 Hz
F2P -0.500 ppm
F2 -250.12 Hz
PPMCM 0.47500 ppm/cm
HZCM 237.60925 Hz/cm



Current Data Parameters
NAME ej2-43-1
EXPNO 2
PROCNO 1

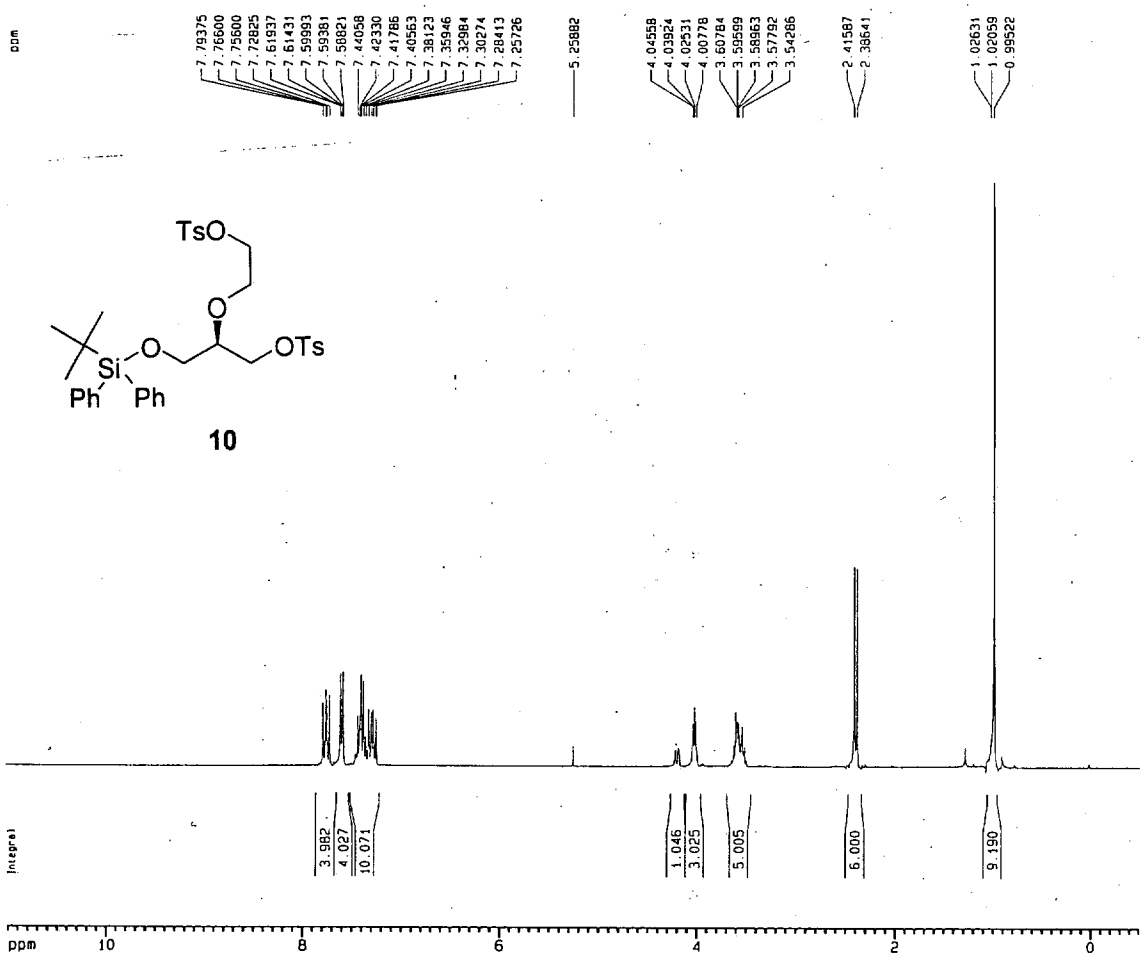
F2 - Acquisition Parameters
Date_ 990904
Time 10.42
INSTRUM spect
PROBHD 5 mm BBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 2
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 3649.1
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

---------- CHANNEL f1 ----------
NUC1 13C
P1 5.40 usec
PL1 2.00 dB
SFO1 125.7957196 MHz

---------- CHANNEL f2 ----------
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 25.00 dB
PL13 28.00 dB
SFO2 500.2320009 MHz

F2 - Processing parameters
SI 32768
SF 125.7829452 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 25156.59 Hz
F2P -0.500 ppm
F2 -62.89 Hz

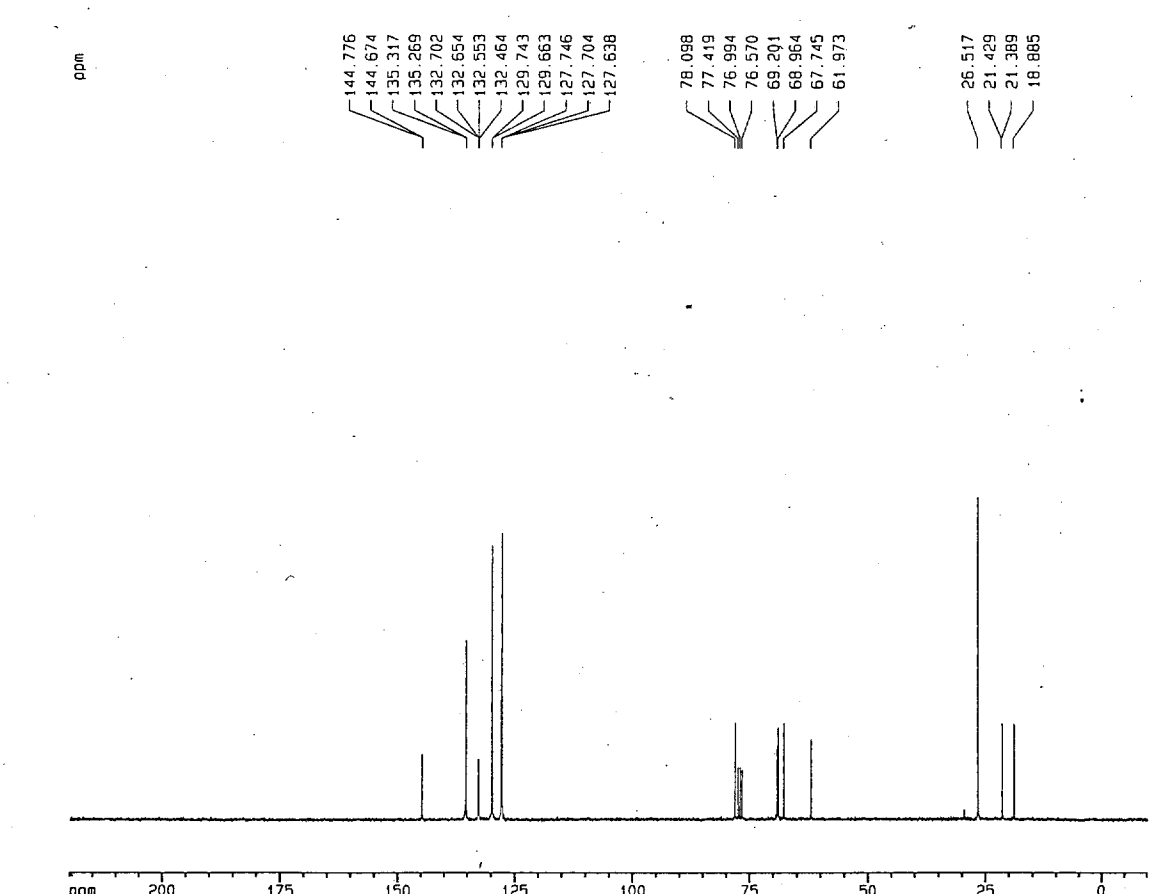


Current Data Parameters
NAME 980727
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 980727
Time 14.32
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 5787.037 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32
DW 86.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 12.50 usec
DE 4.50 usec
SFO1 300.1319886 MHz
NUC1 1H
PL1 -3.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.54762 ppm/cm
HZCM 164.35690 Hz/cm

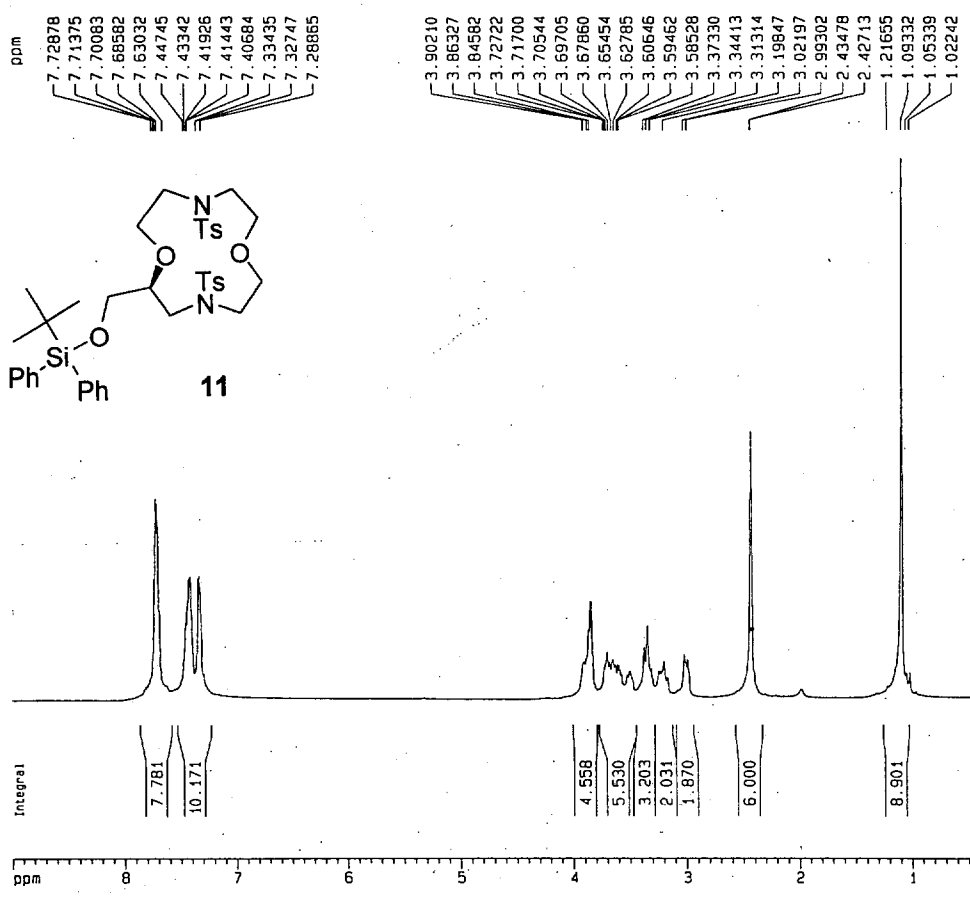


Current Data Parameters
NAME 980727
EXPNO 213
PROCNO 1

F2 - Acquisition Parameters
Date_ 980727
Time 14.37
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 111
DS 2
SWH 19607.844 Hz
FIDRES 0.598384 Hz
AQ 0.8356340 sec
RG 4096
DW 25.500 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -3.00 dB
PL12 16.00 dB
P1 6.10 usec
DE 4.50 usec
SFO1 75.4760988 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677668 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16602.91 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.50000 ppm/cm

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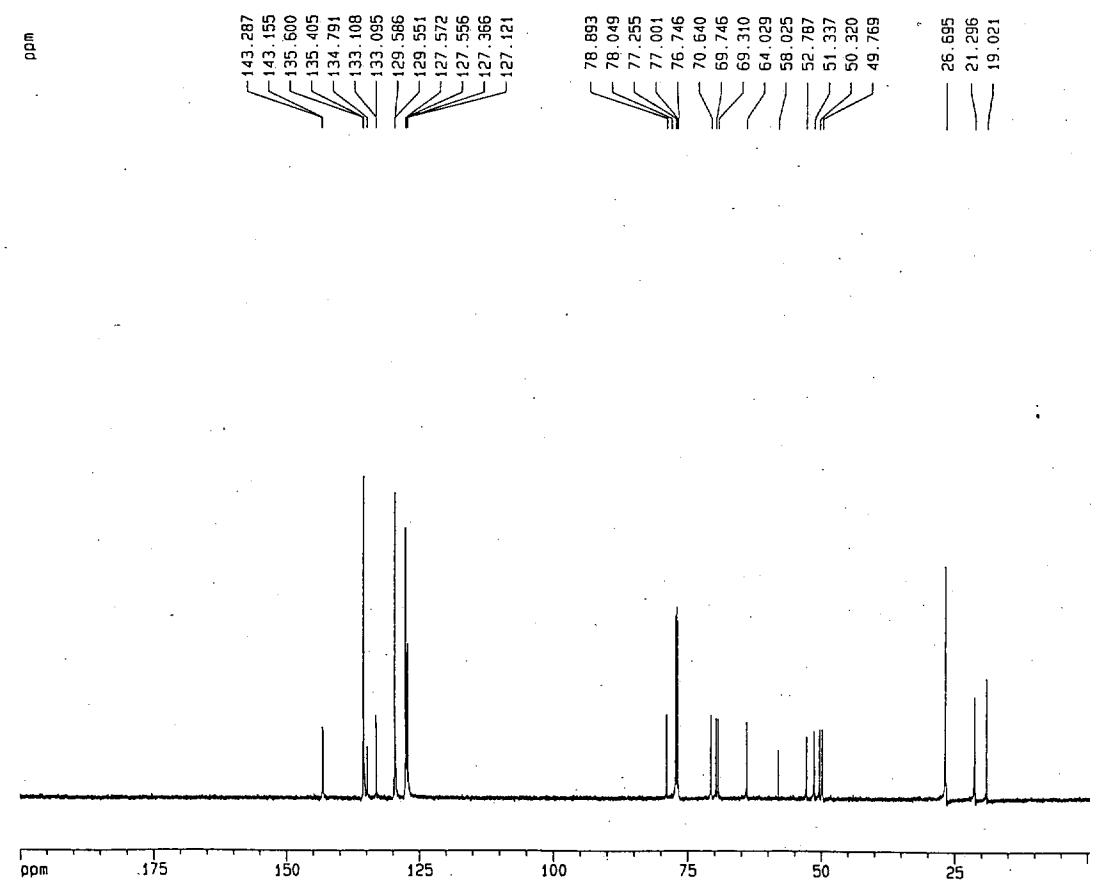
Current Data Parameters
NAME                  tps-4
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_                990907
Time                  14.27
INSTRUM               spect
PROBHD     5 mm QNP 1H/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                       16
DS                        0
SWH              10330.578 Hz
FIDRES           0.315264 Hz
AQ               1.5860212 sec
RG                     25.4
DW                 48.400 usec
DE                  6.00 usec
TE                  300.0 K
D1               1.00000000 sec

========== CHANNEL f1 ==========
NUC1                      1H
P1                     8.40 usec
PL1                    4.00 dB
SFO1           500.2330891 MHz

F2 - Processing parameters
SI                   32768
SF             500.2300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
F1P                 9.000 ppm
F1                4502.07 Hz
F2P                -0.500 ppm
F2                 -250.12 Hz
PPMCM             0.47500 ppm/cm
HZCM            237.60925 Hz/cm
```

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Current Data Parameters
NAME                  tps-4
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_                990907
Time                  14.37
INSTRUM               spect
PROBHD     5 mm QNP 1H/
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      323
DS                        2
SWH              31446.541 Hz
FIDRES           0.479836 Hz
AQ               1.0420724 sec
RG                     8192
DW                 15.900 usec
DE                  6.00 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
D12              0.00002000 sec

========== CHANNEL f1 ==========
NUC1                      13C
P1                     6.70 usec
PL1                    4.00 dB
SFO1           125.7967196 MHz

========== CHANNEL f2 ==========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                80.00 usec
PL2                    3.00 dB
PL12                  17.00 dB
PL13                  27.00 dB
SFO2           500.2320009 MHz

F2 - Processing parameters
SI                   32768
SF             125.7829625 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                 200.000 ppm
F1                25156.59 Hz
F2P                -0.500 ppm
F2                 -62.89 Hz
PPMCM             10.02500 ppm/cm
HZCM            1260.97412 Hz/cm
```
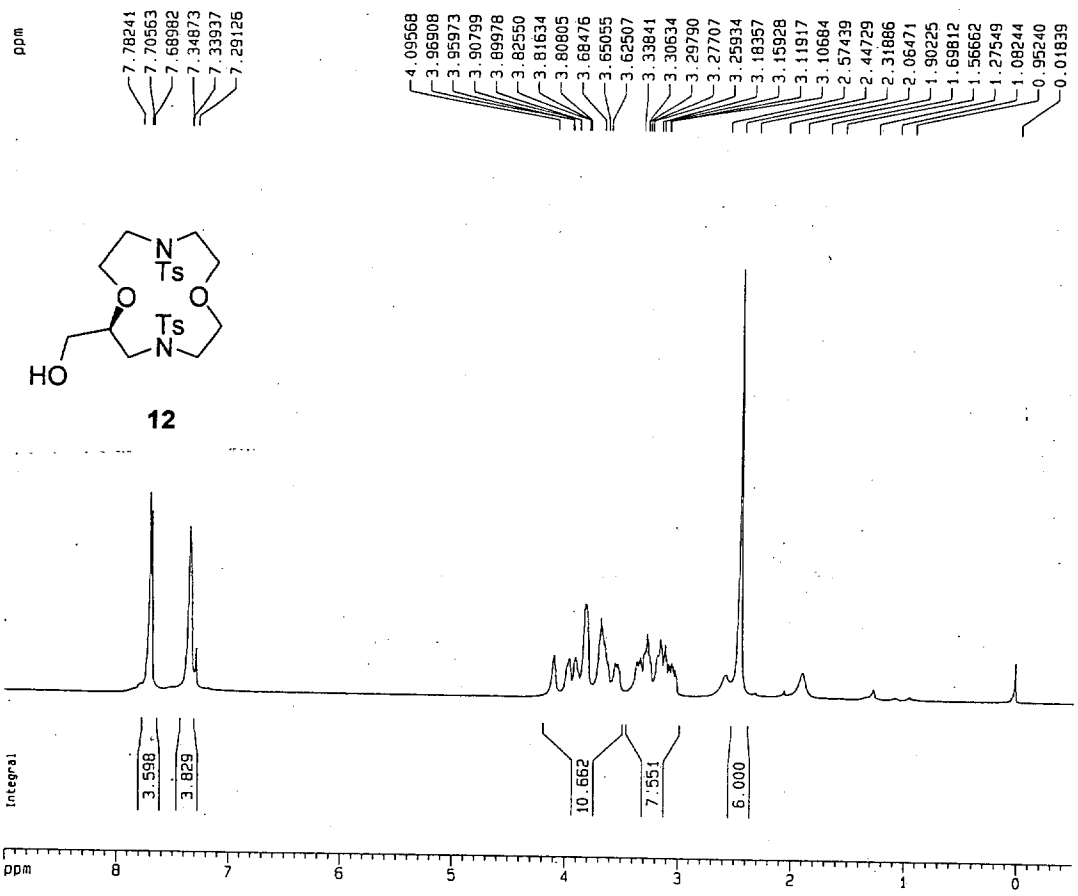



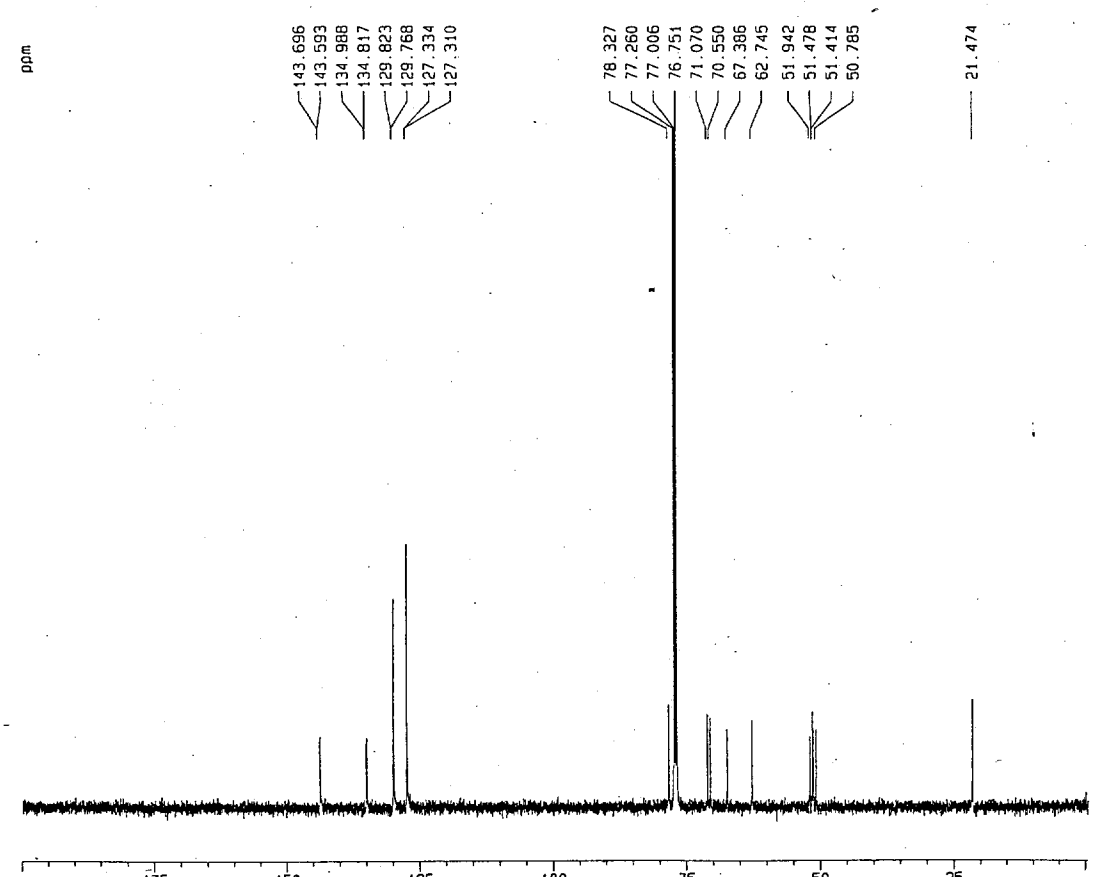
Current Data Parameters
NAME ej2-42j-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 990902
Time 16.28
INSTRUM spect
PROBHD .5 mm QNP 1H/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860212 sec
RG 101.6
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

---------- CHANNEL f1 ----------
NUC1 1H
P1 8.40 usec
PL1 4.00 dB
SFO1 500.2330891 MHz

F2 - Processing parameters
SI 32768
SF 500.2300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 4502.07 Hz
F2P -0.500 ppm
F2 -250.12 Hz
PPMCM 0.47500 ppm/cm
HZCM 237.60925 Hz/cm



Current Data Parameters
NAME ej2-42j-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 990902
Time 16.35
INSTRUM spect
PROBHD 5 mm QNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 2
SWH 31446.541 Hz
FIDRES 0.479835 Hz
AQ 1.0420724 sec
RG 8192
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

---------- CHANNEL f1 ----------
NUC1 13C
P1 6.70 usec
PL1 4.00 dB
SFO1 125.7967196 MHz

---------- CHANNEL f2 ----------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 17.00 dB
PL13 27.00 dB
SFO2 500.2320009 MHz

F2 - Processing parameters
SI 32768
SF 125.7829423 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 25156.59 Hz
F2P -0.500 ppm
F2 -62.89 Hz
PPMCM 10.03500 ppm/cm

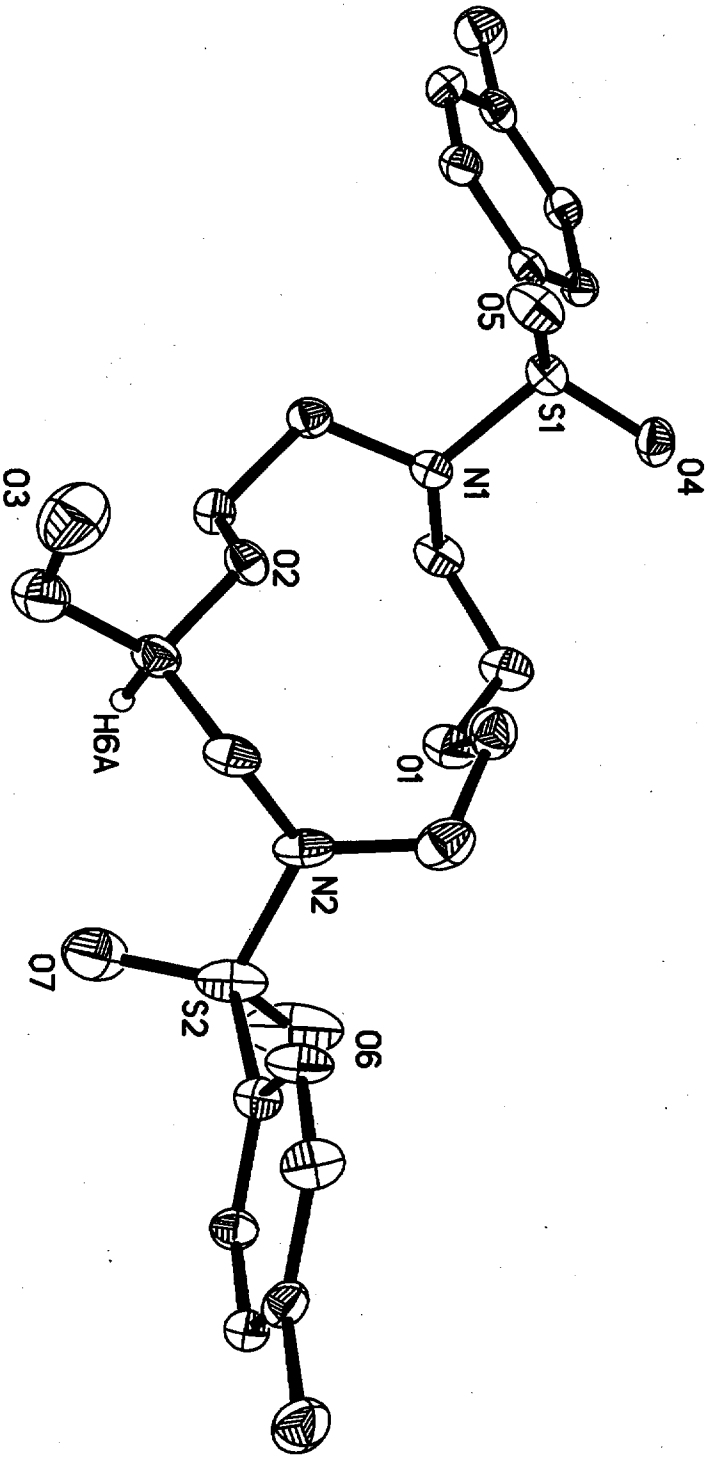

O5
S1
O4
N1
O3
O2
H6A
O1
N2
O7
S2
O6

Table 1. Crystal data and structure refinement for xc414.

Identification code	xc414
Empirical formula	$C_{23}H_{32}N_2O_7S_2$
Formula weight	512.63
Temperature	243(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 6.0853(4) Å alpha = 90° b = 13.7666(9) Å beta = 95.5070(10)° c = 14.4567(10) Å gamma = 90°
Volume, Z	1205.50(14) $Å^3$, 2
Density (calculated)	1.412 Mg/m^3
Absorption coefficient	0.268 mm^{-1}
F(000)	544
Crystal size	0.50 x 0.30 x 0.15 mm
θ range for data collection	2.05 to 24.12°
Limiting indices	$-6 \le h \le 6$, $-15 \le k \le 12$, $-15 \le l \le 16$
Reflections collected	4947
Independent reflections	2816 (R_{int} = 0.0299)
Completeness to θ = 24.12°	98.8 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2816 / 7 / 326
Goodness-of-fit on F^2	1.104
Final R indices [I>2σ(I)]	R1 = 0.0417, wR2 = 0.1092
R indices (all data)	R1 = 0.0441, wR2 = 0.1121
Absolute structure parameter	0.06(10)
Largest diff. peak and hole	0.454 and -0.309 $eÅ^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for xc414. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S(1)	5232(2)	4583(1)	3831(1)	32(1)
S(2)	4987(2)	4950(1)	-1372(1)	51(1)
N(1)	5715(5)	5223(3)	2914(2)	30(1)
N(2)	4010(5)	4696(3)	-389(2)	42(1)
O(1)	7407(4)	4408(3)	1151(2)	44(1)
O(2)	2806(4)	5852(2)	1304(2)	34(1)
O(3)	-1501(8)	6473(4)	536(3)	85(1)
O(4)	6478(5)	3704(2)	3802(2)	41(1)
O(5)	2870(4)	4543(3)	3842(2)	45(1)
O(6)	7253(5)	4668(5)	-1305(3)	94(2)
O(7)	4396(7)	5947(3)	-1600(3)	69(1)
C(1)	8056(6)	5218(4)	2675(3)	37(1)
C(2)	8591(7)	4380(4)	2056(3)	45(1)
C(3)	5435(8)	3864(4)	1072(3)	45(1)
C(4)	4585(9)	3766(4)	62(3)	54(1)
C(5)	1944(7)	5193(4)	-204(3)	39(1)
C(6)	2316(7)	6124(4)	350(3)	38(1)
C(7)	4225(7)	6506(3)	1823(3)	35(1)
C(8)	4579(7)	6160(3)	2815(3)	35(1)
C(9)	394(8)	6810(5)	221(3)	56(2)
C(10)	6288(6)	5241(3)	4826(2)	30(1)
C(11)	4977(7)	5920(3)	5210(3)	37(1)
C(12)	5799(8)	6449(4)	5979(3)	41(1)
C(13)	7946(7)	6299(3)	6388(3)	36(1)
C(14)	9215(7)	5596(3)	6001(3)	35(1)
C(15)	8425(6)	5069(3)	5222(2)	31(1)
C(16)	8823(11)	6875(5)	7211(4)	60(2)
C(17)	3610(6)	4197(3)	-2230(3)	33(1)
C(18)	4482(7)	4091(3)	-3071(3)	34(1)
C(19)	3374(8)	3560(3)	-3769(3)	38(1)
C(20)	1353(7)	3119(3)	-3660(3)	36(1)
C(21)	517(7)	3222(4)	-2810(3)	43(1)
C(22)	1608(7)	3742(4)	-2101(3)	45(1)
C(23)	117(9)	2540(4)	-4426(3)	50(1)

Table 3. Bond lengths [Å] and angles [°] for xc414.

S(1)-O(4)	1.431(3)	S(1)-O(5)	1.440(3)
S(1)-N(1)	1.640(3)	S(1)-C(10)	1.768(4)
S(2)-O(6)	1.427(4)	S(2)-O(7)	1.449(5)
S(2)-N(2)	1.630(3)	S(2)-C(17)	1.765(4)
N(1)-C(8)	1.465(6)	N(1)-C(1)	1.498(5)
N(2)-C(4)	1.464(7)	N(2)-C(5)	1.479(5)
O(1)-C(3)	1.410(6)	O(1)-C(2)	1.432(5)
O(2)-C(7)	1.413(5)	O(2)-C(6)	1.432(5)
O(3)-C(9)	1.361(7)	C(1)-C(2)	1.515(7)
C(3)-C(4)	1.508(7)	C(5)-C(6)	1.517(7)
C(6)-C(9)	1.501(7)	C(7)-C(8)	1.507(6)
C(10)-C(11)	1.380(6)	C(10)-C(15)	1.390(5)
C(11)-C(12)	1.382(6)	C(12)-C(13)	1.397(6)
C(13)-C(14)	1.389(6)	C(13)-C(16)	1.486(7)
C(14)-C(15)	1.387(6)	C(17)-C(18)	1.379(5)
C(17)-C(22)	1.399(6)	C(18)-C(19)	1.371(6)
C(19)-C(20)	1.394(6)	C(20)-C(21)	1.382(6)
C(20)-C(23)	1.505(6)	C(21)-C(22)	1.368(6)

O(4)-S(1)-O(5)	120.0(2)	O(4)-S(1)-N(1)	107.09(18)
O(5)-S(1)-N(1)	106.67(17)	O(4)-S(1)-C(10)	107.73(18)
O(5)-S(1)-C(10)	107.20(18)	N(1)-S(1)-C(10)	107.63(18)
O(6)-S(2)-O(7)	119.4(3)	O(6)-S(2)-N(2)	108.2(2)
O(7)-S(2)-N(2)	107.3(2)	O(6)-S(2)-C(17)	106.2(2)
O(7)-S(2)-C(17)	107.8(2)	N(2)-S(2)-C(17)	107.4(2)
C(8)-N(1)-C(1)	115.5(3)	C(8)-N(1)-S(1)	115.6(2)
C(1)-N(1)-S(1)	115.6(3)	C(4)-N(2)-C(5)	119.7(4)
C(4)-N(2)-S(2)	119.0(3)	C(5)-N(2)-S(2)	116.5(3)
C(3)-O(1)-C(2)	114.2(3)	C(7)-O(2)-C(6)	113.8(3)
N(1)-C(1)-C(2)	113.8(4)	O(1)-C(2)-C(1)	113.7(4)
O(1)-C(3)-C(4)	109.6(4)	N(2)-C(4)-C(3)	113.5(4)
N(2)-C(5)-C(6)	113.6(3)	O(2)-C(6)-C(9)	111.8(3)
O(2)-C(6)-C(5)	107.1(4)	C(9)-C(6)-C(5)	113.0(4)
O(2)-C(7)-C(8)	109.2(3)	N(1)-C(8)-C(7)	113.3(3)
O(3)-C(9)-C(6)	114.8(5)	C(11)-C(10)-C(15)	120.3(4)
C(11)-C(10)-S(1)	119.7(3)	C(15)-C(10)-S(1)	120.0(3)
C(10)-C(11)-C(12)	120.1(4)	C(11)-C(12)-C(13)	121.0(4)
C(14)-C(13)-C(12)	117.7(4)	C(14)-C(13)-C(16)	121.6(5)
C(12)-C(13)-C(16)	120.7(5)	C(15)-C(14)-C(13)	122.0(4)
C(14)-C(15)-C(10)	118.9(4)	C(18)-C(17)-C(22)	118.9(4)
C(18)-C(17)-S(2)	119.0(3)	C(22)-C(17)-S(2)	122.0(3)
C(19)-C(18)-C(17)	120.1(4)	C(18)-C(19)-C(20)	121.7(4)
C(21)-C(20)-C(19)	117.4(4)	C(21)-C(20)-C(23)	120.5(4)
C(19)-C(20)-C(23)	122.1(4)	C(22)-C(21)-C(20)	121.8(4)
C(21)-C(22)-C(17)	120.1(4)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for xc414.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
S(1)	28(1)	38(1)	29(1)	3(1)	-2(1)	-6(1)
S(2)	29(1)	84(1)	40(1)	-22(1)	8(1)	-18(1)
N(1)	24(2)	38(2)	27(2)	0(2)	1(1)	-2(2)
N(2)	26(2)	67(3)	32(2)	-17(2)	-1(1)	0(2)
O(1)	32(2)	65(2)	33(1)	1(2)	-1(1)	4(2)
O(2)	36(2)	40(2)	25(1)	2(1)	-6(1)	-4(1)
O(3)	67(3)	84(4)	105(4)	-5(3)	14(2)	10(3)
O(4)	51(2)	28(2)	41(2)	2(1)	-3(1)	-4(2)
O(5)	25(1)	67(2)	42(2)	7(2)	1(1)	-15(2)
O(6)	19(2)	196(6)	66(2)	-49(3)	3(1)	-15(3)
O(7)	100(3)	53(3)	57(2)	-12(2)	24(2)	-31(2)
C(1)	19(2)	56(3)	36(2)	3(2)	0(1)	-6(2)
C(2)	29(2)	71(4)	36(2)	2(2)	0(2)	10(2)
C(3)	47(3)	42(3)	46(2)	0(2)	-1(2)	1(2)
C(4)	51(3)	58(4)	52(3)	-16(3)	-6(2)	4(3)
C(5)	28(2)	60(3)	27(2)	-4(2)	1(1)	-5(2)
C(6)	38(2)	51(3)	25(2)	1(2)	0(2)	5(2)
C(7)	33(2)	33(2)	37(2)	0(2)	-2(2)	-1(2)
C(8)	37(2)	39(3)	27(2)	-6(2)	-1(2)	-1(2)
C(9)	44(3)	80(4)	43(3)	7(3)	-1(2)	17(3)
C(10)	28(2)	37(3)	24(2)	7(2)	0(1)	-2(2)
C(11)	31(2)	46(3)	33(2)	6(2)	3(2)	7(2)
C(12)	49(3)	35(3)	39(2)	-2(2)	9(2)	8(2)
C(13)	46(3)	32(3)	31(2)	4(2)	3(2)	-11(2)
C(14)	30(2)	38(3)	35(2)	7(2)	-3(2)	-2(2)
C(15)	30(2)	34(2)	28(2)	2(2)	0(1)	3(2)
C(16)	77(4)	57(4)	45(3)	-11(3)	1(3)	-10(3)
C(17)	23(2)	43(3)	32(2)	-2(2)	0(2)	2(2)
C(18)	32(2)	39(3)	33(2)	-1(2)	7(2)	-3(2)
C(19)	50(3)	35(3)	30(2)	1(2)	11(2)	5(2)
C(20)	38(2)	32(3)	37(2)	-6(2)	-5(2)	8(2)
C(21)	28(2)	52(3)	49(2)	-13(2)	4(2)	-12(2)
C(22)	32(2)	71(3)	32(2)	-14(2)	9(2)	-8(2)
C(23)	61(3)	42(3)	46(3)	-12(2)	-7(2)	3(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for xc414.

	x	y	z	U(eq)
H(3)	-1882	5963	259	102
H(1A)	9042	5189	3252	45
H(1B)	8349	5830	2363	45
H(2A)	10178	4387	1988	55
H(2B)	8253	3769	2359	55
H(3A)	4327	4189	1412	54
H(3B)	5710	3218	1345	54
H(4A)	5714	3446	-270	65
H(4B)	3275	3348	13	65
H(5A)	1055	4749	138	46
H(5B)	1098	5343	-798	46
H(6A)	3627	6455	142	46
H(7A)	3568	7157	1801	42
H(7B)	5643	6542	1556	42
H(8A)	5448	6647	3185	42
H(8B)	3144	6105	3065	42
H(9A)	806	7419	543	67
H(9B)	102	6958	-443	67
H(11A)	3521	6023	4948	44
H(12A)	4900	6917	6230	49
H(14A)	10653	5475	6275	42
H(15A)	9319	4602	4966	37
H(16A)	8720(100)	7557(18)	7080(40)	72
H(16B)	10270(60)	6630(50)	7450(40)	72
H(16C)	8040(80)	6640(50)	7710(30)	72
H(18A)	5838	4384	-3164	41
H(19A)	3994	3491	-4337	46
H(21A)	-836	2927	-2716	52
H(22A)	1011	3792	-1527	54
H(23A)	540(90)	2840(40)	-4980(20)	60
H(23B)	620(90)	1880(20)	-4470(40)	60
H(23C)	-1420(40)	2620(40)	-4420(40)	60