Enantioselective Organocatalytic Domino Michael–Acetalization–Henry Reactions of 2-Hydroxynitrostyrene and Aldehyde for the Synthesis of Tetrahydro-6*H*-benzo[*c*]chromenones

Bor-Cherng Hong,* Prakash Kotame, and Ju-Hsiou Liao

Department of Chemistry and Biochemistry, National Chung Cheng University, Chia-Yi, 621, Taiwan, R.O.C.

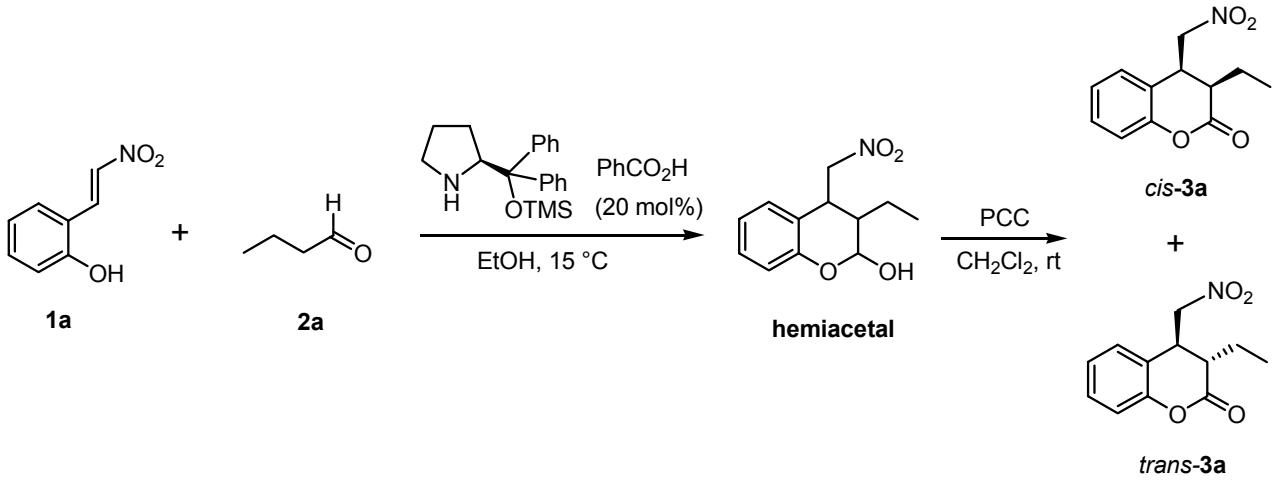
chebch@ccu.edu.tw

SUPPORTING INFORMATION:

Contents: (1) Experimental procedures and characterization data for compounds **3a-3o**.
(2) Spectra data for compounds **3a-3o**.
(3) *Ee* analysis by HPLC with chiral column, in Table 2.

General Procedure. All solvents were reagent grade. L-proline (99+%) was purchased from Bachem. Other chemicals were purchased from Aldrich or Acros Chemical Co. Reactions were normally carried out under argon atmosphere in glassware. Merck silica gel 60 (particle size 0.04-0.063 mm) was employed for flash chromatography. Melting points are uncorrected. ^{1}H NMR spectra were obtained in $CDCl_3$ unless otherwise noted at 400 MHz (Bruker DPX-400) or 500 MHz (Varian-Unity INOVA-500). ^{13}C NMR spectra were obtained at 100 MHz or 125 MHz. *E.e.* values were measured by HPLC on a chiral column (chiralpak IA or chiralcel OD-H, 0.46 cm ID x 25 cm, particle size 5 μ) by elution with IPA-hexane. The flow rate of the indicated elution solvent is maintained at 1 mL/min, and the retention time of a compound is recorded accordingly. HPLC was equipped with the ultraviolet and refractive index detectors. The melting point was recorded on a melting point apparatus (MPA100 – Automated melting point system, Stanford Research Systems, Inc.) and is uncorrected. The optical rotation values were recorded with a Jasco-P-2000 digital polarimeter.

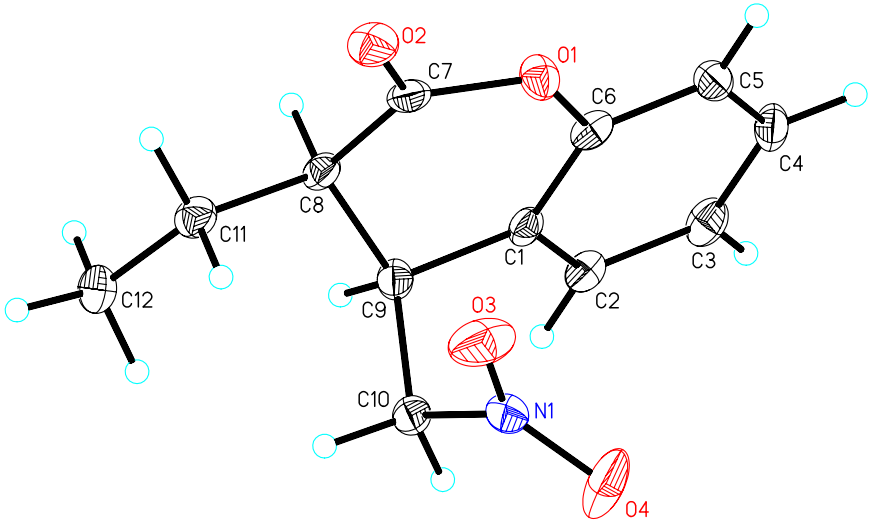
Representative procedure for the preparation of compound 3a in 95% EtOH (Table 2, entry 1).

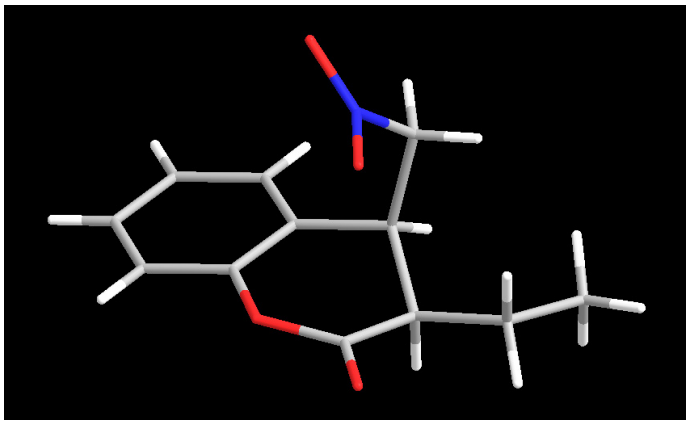


To a solution of *trans*-2-hydroxy-β-nitrostyrene (**1a**, 50 mg, 0.3 mmol), catalyst **I** (20 mg, 0.06 mmol) and benzoic acid (7.3 mg, 0.06 mmol) in 95% EtOH (1.5 mL) was added a solution of butyraldehyde (**2a**, 131 mg, 1.82 mmol) in 95% EtOH (1.5mL). The resulting solution was stirred at 15 °C for 48 h until the completion of reaction, monitored by TLC. The resulting mixture was extracted with EtOAc (20 mL), washed with brine (5 mL), dried over Na_2SO_4, and concentrated *in vacuo* to give the crude product. The residue was purified by flash column chromatography with 12% EtOAc-hexane (R_f = 0.35 for the hemiacetal, in 20% EtOAc-hexane) to give the hemiacetal as a colorless oil (67 mg, 93% yield). A solution of the hemiacetal (55 mg, 0.23 mmol) in CH_2Cl_2 (5 mL) and PCC (150 mg, 0.69 mmol) was stirred at ambient temperature for 20h until the completion of reaction, monitored by TLC. The reaction mixture was diluted with EtOAc (25 mL), and filtered through Celite. The filtrate was concentrated *in vacuo* to give the crude product. The residue was purified by flash column chromatography with 10% EtOAc-hexane (R_f = 0.45 for *cis*-**3a**, R_f = 0.44 for *trans*-**3a** in 20% EtOAc-hexane) to give **3a** as a oil (*cis*-*trans* mixture 92:8, 45 mg, 82% yield). The pure *cis*-**3a** was obtained as a white solid (mp. 91-93 °C) by further purification. For *cis*-**3a**: $[\alpha]_D^{23}$ -80 (c 1.5 $CHCl_3$); IR (neat): 2969, 2880, 1767, 1554, 1378, 1151. 1095, 762 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.33 (d, J = 7.7 Hz, 1 H), 7.14 (d, J = 7.7 Hz, 1 H), 7.11-7.07 (m, 2 H), 4.57 (dd, J = 12.4, 4.7 Hz, 1 H), 4.24 (t, J = 10.5 Hz, 1 H), 3.88 (dt, J = 10.5, 5.1 Hz, 1 H), 2.81 (dd, J = 12.4, 4.7 Hz, 1 H), 2.12-2.04 (m, 1 H), 1.57-1.51 (m, 1 H), 1.12 (t, J = 7.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): 168.8 (C), 150.8 (C), 130.1 (CH), 128.1 (CH), 124.9 (CH), 122.6 (C), 117.3 (CH), 75.4 (CH_2), 43.3 (CH), 37.3 (CH), 19.9 (CH_2), 11.9 (CH_3); MS (*m/z*, relative intensity): 235 (M^+, 23), 188 (100), 173 (39), 160 (66), 145 (44), 131 (63), 91 (64); exact mass calculate for $C_{12}H_{13}NO_4$ (M^+): 235.0845; found (M^+): 235.0842. For *trans*-**3a**: $[\alpha]_D^{23}$ -44.4 (c 2 $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz): δ 7.36 – 7.32 (m, 1 H), 7.21 (dd, J = 7.6, 1.0 Hz, 1 H), 7.14 (ddd, J = 7.5, 7.5, 1.0 Hz, 1 H), 7.07 (dd, J = 7.5, 1.0 Hz, 1H), 4.51-4.42 (m, 2 H), 3.73-3.70 (m, 1 H), 2.79-2.75 (m, 1 H), 1.63-1.49 (m, 2 H), 1.00 (t, J = 7.4 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.0 (C), 150.7 (C), 130.2 (CH), 129.2 (CH), 125.3 (CH), 118.7 (C), 117.2 (CH), 78.1 (CH_2), 44.3 (CH), 39.2 (CH), 23.6 (CH_2), 11.4 (CH_3).

Representative procedure for the preparation of compound 3a on water (Table 2, entry 1).

To a solution of *trans*-2-hydroxy-β-nitrostyrene (**1a**, 50 mg, 0.3 mmol), catalyst **I** (20 mg, 0.06 mmol) and acetic acid (4 mg, 0.06 mmol) in H_2O (0.5 mL) was added a solution of butyraldehyde (**2a**, 131 mg, 1.82 mmol). The resulting solution was stirred at 30 °C for 1 h until the completion of reaction, monitored by TLC. The resulting mixture was extracted with EtOAc (20 mL), washed with brine (5 mL), dried over Na_2SO_4, and concentrated *in vacuo* to give the crude product. The residue was purified by flash column chromatography with 12% EtOAc-hexane (R_f = 0.35 for the hemiacetal, in 20% EtOAc-hexane) to give the hemiacetal as a colorless oil (63 mg, 88% yield). The subsequent oxidation and the purification procedure are the same as the above reaction in 95% EtOH.





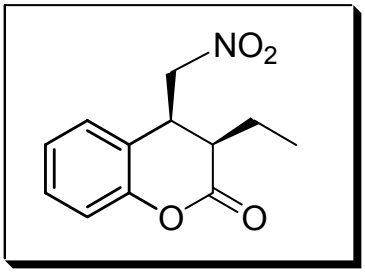
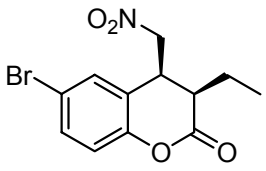


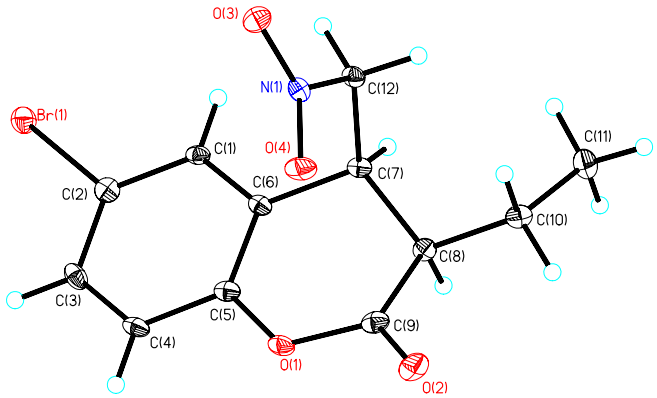
Figure S1. ORTEP and Stereo plots for X-ray crystal structures of (-)-*cis*-**3a**.

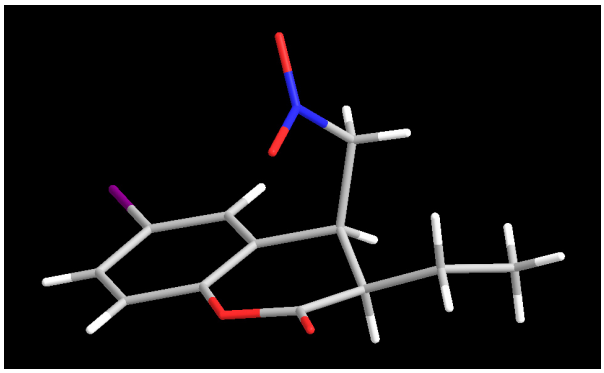
CCDC 794373 contains the supplementary crystallographic data for (-)-*cis*-**3a**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



3b

cis-**3b:** white solid; mp 123-126 °C, R_f = 0.48 for *cis*-**3b** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -42 (c 1.0 $CHCl_3$). IR (neat): 2962, 2926, 1771, 1555, 1413, 1378, 1147. 1096, 818 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.45 (dd, J = 8.7, 2.0 Hz, 1 H), 7.31 (d, J = 2.0 Hz, 1 H), 6.97 (d, J = 8.7 Hz, 1 H), 4.56 (dd, J = 12.8, 5.0 Hz, 1 H), 4.31-4.26 (m, 1 H), 3.85 (dt, J = 10.0, 5.0 Hz, 1 H), 2.78 (dd, J = 12.8, 10.0 Hz, 1 H), 2.09-2.06 (m, 1 H), 1.55-1.51 (m, 1 H), 1.12 (t, J = 7.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.0 (C), 150.0 (C), 133.2 (CH), 130.9 (CH), 124.7 (C), 119.1 (CH), 117.5 (C), 74.9 (CH_2), 43.0 (CH), 37.0 (CH), 19.9 (CH_2), 11.9 (CH_3); MS (*m/z*, relative intensity): 315 (M^++3, 55), 313 (M^++1, 56), 268 (100), 266 (99), 211 (33), 209 (32), 145 (41), 118 (42), 91 (15), 71 (50), 57 (70); exact mass calculate for $C_{12}H_{12}BrNO_4$ (M^+): 312.9950; found (M^+): 312.9947.





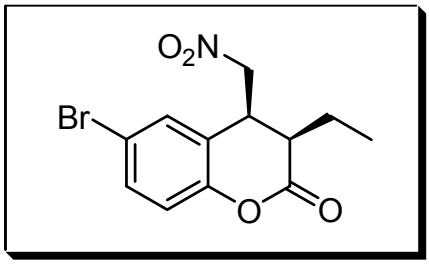
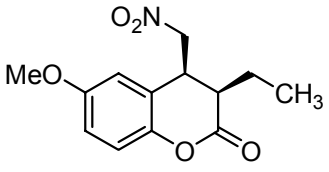


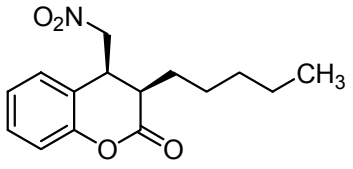
Figure S1. ORTEP and Stereo plots for X-ray crystal structures of (-)-*cis*-**3b**.

CCDC 794374 contains the supplementary crystallographic data for (-)-*cis*-**3b**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



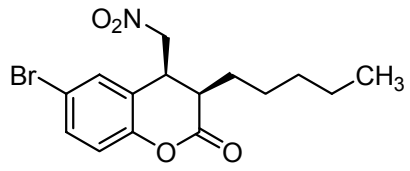
3c

cis-**3c:** yellow solid; mp 85-87 °C, R_f = 0.36 for *cis*-**3c** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -45 (c 1.6 $CHCl_3$). IR (neat): 2964, 2929, 1764, 1555, 1379, 1208, 1034, 817 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.00 (d, J = 8.9 Hz, 1 H), 6.84 (dd, J = 8.9, 2.9 Hz, 1 H), 6.65 (d, J = 2.9 Hz, 1 H), 4.56 (dd, J = 12.5, 5.0 Hz, 1 H), 4.28 (dd, J = 12.5, 10.5 Hz, 1 H), 3.83 (dt, J = 10.5, 5.0 Hz, 1 H), 3.75 (s, 3 H), 2.78-2.76 (m, 1 H), 2.13-2.02 (m, 1 H), 1.57-1.50 (m, 1 H), 1.12 (t, J = 7.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.0 (C), 156.3 (C), 144.6 (C), 123.5 (C), 118.2 (CH), 115.3 (CH), 113.0 (CH), 75.3 (CH_2), 55.7 (CH_3), 43.3 (CH), 37.5 (CH), 19.9 (CH_2), 11.9 (CH_3); MS (*m/z*, relative intensity): 265 (M^+, 100), 218 (76), 204 (19), 189 (18), 175 (41), 161 (45), 149 (38), 121 (44), 91 (31), 77 (99), 55 (17); exact mass calculate for $C_{13}H_{15}NO_5$ (M^+): 265.0950; found (M^+): 265.0948.

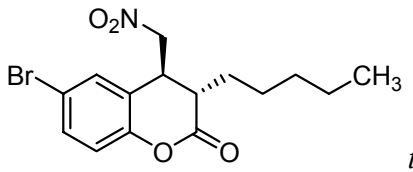


3d

cis-**3d:** white solid; mp 67-69 °C, R_f = 0.50 for *cis*-**3d** in 20% EtOAc-hexane, $[\alpha]_D^{24}$ -85 (c 1.5 $CHCl_3$). IR (neat): 2957, 2929, 2860, 1769, 1556, 1377, 1102. 762 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.34-7.31 (m, 1 H), 7.14-7.06 (m, 3 H), 4.58 (dd, J = 12.5, 5.0 Hz, 1 H), 4.28 (dd, J = 12.5, 10.0 Hz, 1 H), 3.85 (dt, J = 10.0, 5.0 Hz, 1 H), 2.88 (dd, J = 12.5, 6.8 Hz, 1 H), 2.04-2.00 (m, 1 H), 1.51-1.44 (m, 3 H), 1.43-1.34 (m, 4 H), 0.89 (t, J = 6.8 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.9 (C), 150.8 (C), 130.1 (CH), 128.0 (CH), 124.9 (CH), 122.6 (C), 117.3 (CH), 75.5 (CH_2), 41.6 (CH), 37.6 (CH), 31.4 (CH_2), 26.9 (CH_2), 26.5 (CH_2), 22.4 (CH_2), 13.9 (CH_3); MS (*m/z*, relative intensity): 277 (M^+, 7), 161 (21), 160 (100), 131 (19), 107 (20), 91 (16), 77 (7), 55 (13); exact mass calculate for $C_{15}H_{19}NO_4$ (M^+): 277.1314; found (M^+): 277.1315.



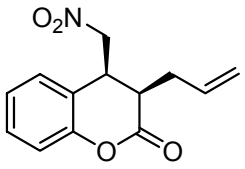
cis-**3e**



trans-**3e**

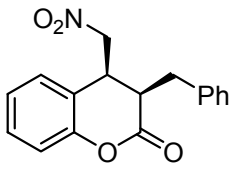
cis-**3e:** colorless oil; R_f = 0.65 for *cis*-**3e** in 20% EtOAc-hexane, $[\alpha]_D^{24}$ -31 (c 1.75 $CHCl_3$). IR (neat): 2956, 2927, 2859, 1773, 1556, 1377, 1105. 1072, 821 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.45 (dd, J = 8.7, 2.2 Hz, 1H), 7.30 (d, J = 2.2 Hz, 1H), 6.97 (d, J = 8.7 Hz, 1H), 4.57 (dd, J = 12.9, 5.0 Hz, 1H), 4.28 (dd, J = 12.9, 10.2 Hz, 1H), 3.82 (dt, J = 10.2, 5.0 Hz, 1H), 2.84 (dd, J = 12.9, 6.8 Hz, 1H), 2.02-2.00 (m, 1H), 1.55-1.42 (m, 3H), 1.33-1.32 (m, 4H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.2 (C), 150.0 (C), 133.2 (CH), 130.9 (CH), 124.7 (C), 119.1 (CH), 117.5 (C), 75.0 (CH_2), 41.4 (CH), 37.3 (CH), 31.4 (CH_2), 26.9 (CH_2), 26.5 (CH_2), 22.4 (CH_2), 13.9 (CH_3); MS (*m/z*, relative intensity): 357 (M^++2, 31), 355 (M^+, 32), 297 (7), 295 (13), 254 (18), 252 (18), 240 (99), 238 (100), 175(22), 161 (22), 149 (20), 118 (22), 105 (23), 97 (20), 91 (24), 85 (27), 71 (43) 57 (51) 55 (46); exact mass calculate for $C_{15}H_{18}BrNO_4$ (M^+): 355.0419; found (M^+): 355.0416.

trans-**3e:** colorless oil; R_f = 0.60 for *trans*-**3e** in 20% EtOAc-hexane, $[\alpha]_D^{24}$ -76.6 (c 1.5 $CHCl_3$). IR (neat): 2956, 2927, 2859, 1773, 1556, 1377, 1105. 1072, 821 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.46 (dd, J = 8.7, 2.2 Hz, 1 H), 7.37 (d, J = 2.2 Hz, 1 H), 6.97 (d, J = 8.7 Hz, 1 H), 4.56-4.43 (m, 2 H), 3.67 (t, J = 7.2 Hz, 1 H), 2.87-2.83 (m, 1 H), 1.54-1.33 (m, 4 H), 1.28-1.20 (m, 4 H), 0.89 (t, J = 6.9 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.4 (C), 149.9 (C), 133.3 (CH), 131.9 (CH), 120.9 (C), 119.0 (CH), 117.8 (C), 76.7 (CH_2), 42.4 (CH), 39.2 (CH), 31.0 (CH_2), 30.1 (CH_2), 26.4 (CH_2), 22.3 (CH_2), 13.9 (CH_3).



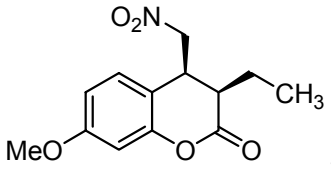
3f

cis-**3f:** white solid; mp 96-98 °C, R_f = 0.36 for *cis*-**3f** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -35 (c 1.0 $CHCl_3$). IR (neat): 2962, 2925, 1769, 1555, 1379, 1119, 1099. 925, 798 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.33 (td, J = 8.1, 1.8 Hz, 1 H), 7.15-7.09 (m, 3 H), 5.85 (dddd, J = 17.0, 10.1, 8.8, 5.0, Hz, 1 H), 5.26 (s, 1H), 5.24-5.22 (m, 1 H), 4.62 (dd, J = 12.4, 4.6 Hz, 1 H), 4.28 (dd, J = 12.4, 10.6 Hz, 1 H), 3.85 (dt, J = 10.6, 5.0 Hz, 1 H), 3.02 (dt, J = 9.0, 5.0 Hz, 1 H), 2.88 (ddd, J = 10.1, 8.8, 4.6 Hz, 1 H), 2.26 (dt, J = 17.0, 9.0 Hz, 1 H) ; ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.3 (C), 150.8 (C), 133.3 (CH), 130.2 (CH), 128.2 (CH), 125.0 (CH), 122.4 (C), 119.0 (CH_2), 117.4 (CH), 75.1 (CH_2), 41.3 (CH), 36.8 (CH), 30.8 (CH_2); MS (*m/z*, relative intensity): 247 (M^+, 6), 217 (42), 200 (90), 199 (28), 186 (32), 185 (55), 172 (34), 144 (41), 131 (100), 107 (52), 77 (29); exact mass calculate for $C_{13}H_{13}NO_4$ (M^+): 247.0845; found (M^+): 247.0845.



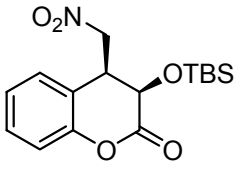
3g

cis-**3g:** colorless oil; R_f = 0.53 for *cis*-**3g** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -51 (c 2.8 $CHCl_3$); IR (neat): 2961, 2925, 1766, 1555, 1377, 1017, 797 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.37-7.27 (m, 4 H), 7.23-7.03 (m, 5 H), 4.72 (dd, *J* = 12.4, 4.4, Hz, 1 H), 4.35 (dd, *J* = 12.4, 10.6 Hz, 1 H), 3.66-3.61 (m, 1 H), 3.52 (dd, *J* = 14.7, 5.7 Hz, 1 H), 3.26 (dt, *J* = 10.6, 4.4 Hz, 1 H), 2.75 (dd, *J* = 14.7, 5.7 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.4 (C), 150.8 (C), 136.9 (C), 130.2 (CH), 129.1 (2 CH), 128.6 (2 CH), 128.2 (CH), 127.3 (CH), 125.0 (CH), 122.6 (C), 117.4 (CH), 75.3 (CH_2), 43.5 (CH), 36.6 (CH), 32.4 (CH_2); MS (*m/z*, relative intensity): 297 (M^+, 9), 250 (18), 161 (23), 131 (41), 107 (12), 91 (100), 71 (19), 57 (24); exact mass calculate for $C_{17}H_{15}NO_4$ (M^+): 297.1001; found (M^+): 297.1001.

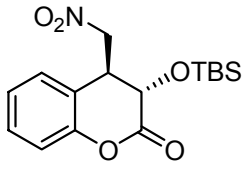


3h

cis-**3h**: white solid; R_f = 0.30 for *cis*-**3h** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -81.9 (c 2.0 $CHCl_3$); IR (neat): 2956, 2862, 1769, 1588, 1376, 1103. 1010, 762 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.03 (d, *J* = 8.3 Hz, 1 H), 6.63 (d, *J* = 2.5 Hz, 1 H), 6.61 (dd, *J* = 8.3, 2.5 Hz, 1 H), 4.54 (dd, *J* = 12.3, 5.1 Hz, 1 H), 4.23 (dd, *J* = 12.3, 10.6 Hz, 1 H), 3.83 (dt, *J* = 10.6, 5.1 Hz, 1 H), 3.77 (s, 3 H), 2.80 (td, *J* = 7.3, 5.1 Hz, 1 H), 2.10-2.05 (m, 1 H), 1.55-1.50 (m, 1 H), 1.12 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.1 (C), 161.2 (C), 152.0 (C), 129.0 (CH), 114.6 (C), 111.0 (CH), 103.2 (CH), 76.0 (CH_2), 55.8 (CH_3), 43.8 (CH), 37.1 (CH), 20.3 (CH_2), 12.2 (CH_3); MS (*m/z*, relative intensity): 265 (M^+, 32), 218 (44), 205 (33), 203 (40), 190 (100), 175 (22), 162 (17), 139 (19), 121 (26), 91 (21); exact mass calculate for $C_{13}H_{15}NO_5$ (M^+): 265.0950; found (M^+): 265.0951.



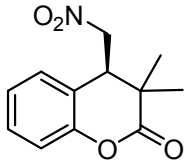
***cis*-3i**



***trans*-3i**

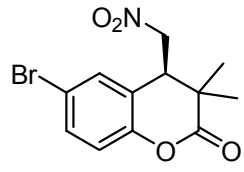
cis-**3i**: white solid; mp 138-140 °C, R_f = 0.46 for *cis*-**3i** in 10% EtOAc-hexane, $[\alpha]_D^{24}$ -57 (c 1.0 $CHCl_3$); IR (neat): 2953, 2929, 2857, 1789, 1558, 1145, 1092, 840 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.35 (td, J = 8.2, 1.5 Hz, 1 H), 7.21 (dd, J = 7.5, 1.5 Hz, 1 H), 7.14 (td, J = 7.5, 1.5 Hz, 1 H), 7.07 (d, J = 8.2 Hz, 1 H), 4.83 (dd, J = 13.1, 5.3 Hz, 1 H), 4.70 (d, J = 5.3 Hz, 1 H), 4.33 (dd, J = 13.1, 5.3 Hz, 1 H), 4.01-3.98 (m, 1 H), 0.92 (s, 9 H), 0.22 (s, 3 H), 0.12 (s, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.1 (C), 150.2 (C), 130.5 (CH), 129.0 (CH), 125.3 (CH), 120.2 (C), 117.4 (CH), 75.1 (CH_2), 68.2 (CH), 41.8 (CH), 25.6 (3 CH_3), 18.3 (C), -4.9 (CH_3), -5.8 (CH_3); MS (*m/z*, relative intensity): 337 (M^+, 2), 280 (24), 131 (13), 113 (13), 111 (10), 107 (25), 99 (19), 97 (21), 99 (19), 97 (21), 91 (15), 85 (57), 83 (24), 71 (77), 57 (100), 55 (23) ; exact mass calculate for $C_{16}H_{23}NO_5Si$ (M^+): 337.1345; found (M^+): 337.1348.

trans-**3i:** colorless oil; R_f = 0.34 for *trans*-**3i** in 10% EtOAc-hexane, $[\alpha]_D^{24}$ -38 (c 1.0 $CHCl_3$). IR (neat): 2953, 2929, 2857, 1789, 1558, 1145, 1092, 840 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.32 (td, J = 8.4, 4.3 Hz, 1 H), 7.15 (d, J = 4.3 Hz, 2 H), 7.09 (d, J = 8.4 Hz, 1 H), 4.86 (dt, J = 10.4, 5.4 Hz, 2 H), 4.59 (d, J = 10.4 Hz, 1 H), 3.84 (dt, J = 10.4, 5.4 Hz, 1 H), 0.87 (s, 9 H), 0.21 (s, 3 H), 0.10 (s, 3 H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.2 (C), 150.2 (C), 129.8 (CH), 125.9 (CH), 125.2 (CH), 120.2 (C), 117.3 (CH), 72.6 (CH_2), 68.0 (CH), 41.1 (CH), 25.6 (3 CH_3), 18.2 (C), -4.6 (CH_3), -5.6 (CH_3).



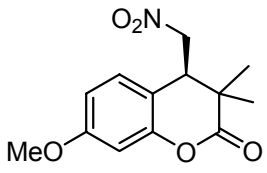
3j

3j: yellow solid; mp 61-63 °C, R_f = 0.30 for **3j** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -109 (c 1.3 $CHCl_3$); IR (neat): 2981, 2928, 1765, 1554, 1379, 1123. 1096, 763 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.33 (ddd, J = 8.0, 6.4, 2.7 Hz, 1 H), 7.13-7.11 (m, 2 H), 7.06 (d, J = 8.0 Hz, 1 H), 4.71 (dd, J = 12.5, 5.2 Hz, 1 H), 4.31 (dd, J = 12.5, 9.7 Hz, 1 H), 3.49 (dd, J = 9.7, 5.2 Hz, 1 H), 1.41 (s, 3 H), 1.24 (s, 3 H), ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.5 (C), 150.5 (C), 130.1 (CH), 128.7 (CH), 125.2 (CH), 121.0 (C), 116.9 (CH), 77.0 (CH_2), 45.7 (CH), 39.8 (C), 25.5 (CH_3), 21.7 (CH_3); MS (*m/z*, relative intensity): 235 (M, 19), 188 (88), 160 (36), 145 (100), 107 (17), 91 (37), 65 (11); exact mass calculate for $C_{12}H_{13}NO_4$ (M^+): 235.0845; found (M^+): 235.0842.



3k

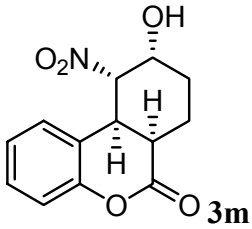
3k: white solid; mp 111-113 °C, R_f = 0.25 for **3k** in 20% EtOAc-hexane, $[\alpha]_D^{24}$ -29 (c 1.3 $CHCl_3$); IR (neat): 2962, 1768, 1554, 1478, 1328, 1177. 1097, 814 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.44 (dd, J = 8.7, 2.3 Hz, 1 H), 7.28 (d, 2.3 Hz, 1 H), 6.95 (d, J = 8.7 Hz, 1 H), 4.69 (dd, J = 12.9, 5.2 Hz, 1 H), 4.32 (dd, J = 12.9, 9.3 Hz, 1 H), 3.46 (dd, J = 9.3, 5.2 Hz, 1 H), 1.40 (s, 3 H), 1.24 (s, 3 H), ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7 (C), 149.7 (C), 133.1 (CH), 131.5 (CH), 123.1 (C), 118.7 (CH), 117.6 (C), 76.5 (CH_2), 45.3 (CH), 39.6 (C), 25.5 (CH_3), 21.6 (CH_3); MS (*m/z*, relative intensity): 315 (M^++3, 59), 313 (M^++1, 59), 268 (98), 266 (100), 240 (56), 238 (58), 225 (93), 223 (90), 160 (96), 145 (64), 70 (53); exact mass calculate for $C_{12}H_{12}BrNO_4$ (M^+): 312.9950; found (M^+): 312.9948.



3l

3l: colorless oil; R_f = 0.36 for **3k** in 20% EtOAc-hexane, $[\alpha]_D^{23}$ -92 (c 1.5 $CHCl_3$); IR (neat): 2961, 1765, 1555, 1435, 1383, 1158. 1029, 809 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.00 (d, J = 8.4 Hz, 1 H), 6.64 (dd, 8.4, 2.5 Hz, 1 H), 6.59 (d, J = 2.5 Hz, 1 H), 4.68 (dd, J = 12.4, 5.2 Hz, 1 H), 4.27 (dd, J = 12.4, 9.8 Hz, 1 H), 3.77 (s, 3 H), 3.42 (dd, J = 9.8, 5.2 Hz, 1 H), 1.39 (s, 3 H), 1.24 (s, 3 H), ^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.6 (C), 160.9 (C), 151.4 (C), 129.4 (CH), 112.7 (C), 110.9 (CH), 102.5 (CH), 76.7 (CH_2), 55.5 (CH_3), 45.2 (CH), 39.9 (C), 25.6 (CH_3), 21.8 (CH_3); MS (*m/z*, relative intensity): 265 (M^+, 50), 218 (100), 205 (38), 191 (26), 175 (94), 150 (31), 137 (28), 121 (29), 91 (23), 83 (40), 70 (33); exact mass calculate for $C_{13}H_{15}NO_5$ (M^+): 265.0950; found (M^+): 265.0950.

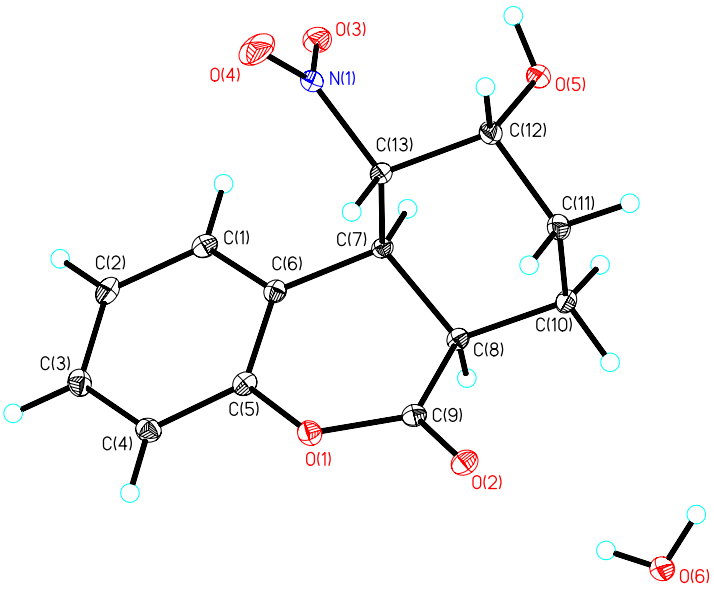
Representative procedure for the preparation of compound 3a on water (Table 2, entry 13).

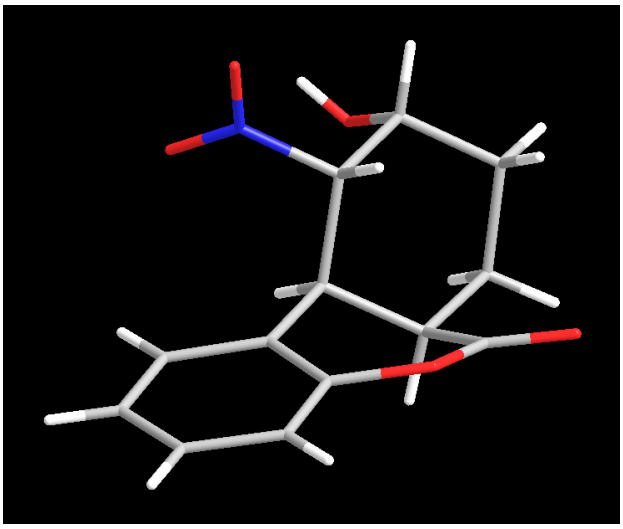


3m

To a solution of *trans*-2-hydroxy-β-nitrostyrene (**1a**, 50 mg, 0.3 mmol), catalyst **I** (20 mg, 0.06 mmol) and acetic acid (4 mg, 0.06 mmol) in H_2O (0.5 mL) was added a solution of glutaraldehyde (61 mg, 0.60 mmol). The resulting solution was stirred at 30 °C for 24 h until the completion of reaction, monitored by TLC. The resulting mixture was extracted with EtOAc (20 mL), washed with brine (5 mL), dried over Na_2SO_4, and concentrated *in vacuo* to give the crude product. The residue was purified by flash column chromatography with 28% EtOAc-hexane (R_f = 0.25 for the hemiacetal, in 30% EtOAc-hexane) to give the hemiacetal as a colorless oil (40 mg, 50% yield). A solution of the hemiacetal (25 mg, 0.09 mmol) in CH_2Cl_2 (4 mL) and PCC (61 mg, 0.28 mmol) was stirred at ambient temperature for 12 h until the completion of reaction, monitored by TLC. The reaction mixture was diluted with EtOAc (25 mL), and filtered through Celite. The filtrate was concentrated *in vacuo* to give the crude product. The residue was purified by flash column chromatography with 22% EtOAc-hexane (R_f = 0.35 for **3m** in 30% EtOAc-hexane) to give **3m** as a white solid (18 mg, 76% yield).

3m: white solid; mp 197-199 °C, R_f = 0.35 for **3m** in 30% EtOAc-hexane, $[\alpha]_D^{23}$ -114 (c 0.2 $CHCl_3$); IR (neat): 3231, 2921, 2857, 1747, 1541, 1373, 1010, 764 cm^{-1}; ^1H NMR (acetone-d_6, 500 MHz): δ 7.38-7.33 (m, 2 H), 7.13 (td, J = 8.1, 1.2 Hz, 1 H), 7.04 (dd, J = 8.1, 1.2 Hz, 1 H), 4.79 (d, J = 4.2, Hz, 1 H), 4.70 (dd, J = 12.2, 2.6 Hz, 1 H), 4.55 (s, 1 H), 4.11 (dd, J = 12.2, 6.0 Hz, 1 H), 3.36-3.33 (m, 1 H), 2.19-2.14 (m, 2 H), 1.97-1.93 (m, 2 H); ^{13}C NMR (acetone-d_6, 125 MHz): δ 169.3 (C), 152.7 (C), 131.1 (CH), 130.4 (CH), 125.1 (CH), 124.0 (C), 117.6 (CH), 90.1 (CH), 68.6 (CH), 39.4 (CH), 34.9 (CH), 29.2 (CH_2), 18.9 (CH_2); MS (*m/z*, relative intensity): 263 (M^+, 41), 216 (100), 197 (31), 188 (43), 171 (29), 160 (33), 147 (43), 131 (30), 111 (27), 97 (38), 91 (26), 77 (24), 71 (41); exact mass calculate for $C_{13}H_{13}NO_5$ (M^+): 263.0794; found (M^+): 263.0797.





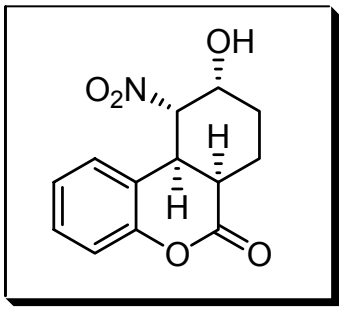
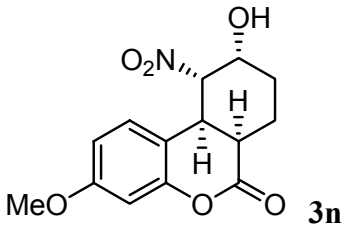


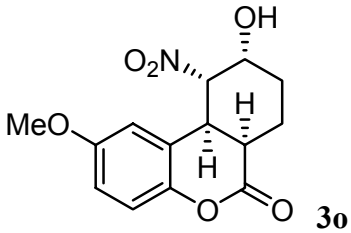
Figure S1. ORTEP and Stereo plots for X-ray crystal structures of (-)-**3m**•H_2O

CCDC 794375 contains the supplementary crystallographic data for (-)-**3m**•H_2O. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



3n

3n: white solid; mp 191-193 °C, R_f = 0.31 for **3n** in 30% EtOAc-hexane, $[\alpha]_D^{23}$ -100 (c 0.25 $CHCl_3$); IR (neat): 3228, 2955, 2909, 1759, 1546, 1373, 1199, 1021, 804 cm^{-1}; ^1H NMR (acetone-d_6, 500 MHz): δ 7.25 (d, J = 8.5 Hz, 1 H), 6.70 (dd, J = 8.5, 2.6 Hz, 1 H), 6.0 (d, J = 2.6 Hz, 1 H), 4.75 (d, J = 4.1, Hz, 1 H), 4.64 (dd, J = 12.1, 2.6 Hz, 1 H), 4.53 (s, 1 H), 4.04 (dd, J = 12.1, 6.0 Hz, 1 H), 3.80 (s, 3 H), 3.33-3.30 (m, 1 H), 2.17-2.12 (m, 2 H), 2.04-1.91 (m, 2 H); ^{13}C NMR (acetone-d_6, 125 MHz): δ 169.3 (C), 161.5 (C), 153.5 (C), 131.7 (CH), 115.7 (C), 111.0 (CH), 103.0 (CH), 90.5 (CH), 68.7 (CH), 55.9 (CH_3), 39.6 (CH), 34.3 (CH), 29.2 (CH_2), 18.8 (CH_2); MS (*m/z*, relative intensity): 293 (M^+, 56), 246 (100), 229 (36), 218 (100), 189 (48), 177 (29), 161 (30), 91 (13), 77 (18), 55 (16); exact mass calculate for $C_{14}H_{15}NO_6$ (M^+): 293.0899; found (M^+): 293.0901.



3o

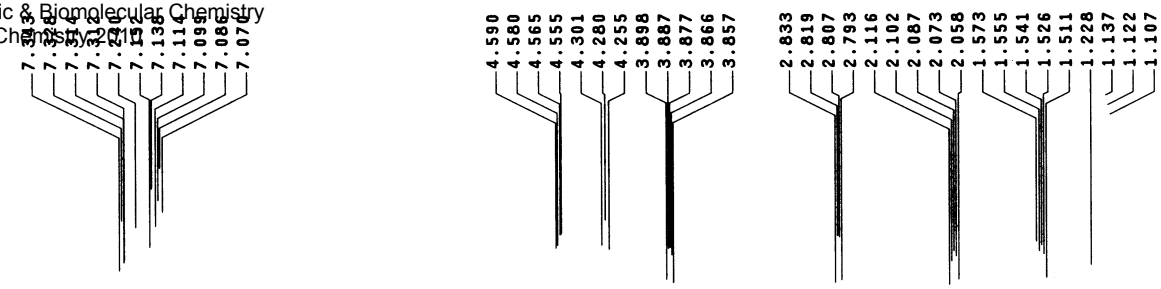
3o: white solid; mp 159-161 °C, R_f = 0.29 for **3o** in 30% EtOAc-hexane, $[\alpha]_D^{23}$ -86 (c 0.22 $CHCl_3$); IR (neat): 3227, 2955, 2909, 1759, 1546, 1339, 1152, 1021, 804 cm^{-1}; ^1H NMR (acetone-d_6, 500 MHz): δ 6.99-6.97 (m, 1 H), 6.92-6.89 (m, 2 H), 4.82 (d, J = 4.4, Hz, 1 H), 4.69 (dd, J = 12.2, 2.6 Hz, 1 H), 4.55 (s, 1 H), 4.06 (dd, J = 12.2, 6.0 Hz, 1 H), 3.78 (s, 3 H), 3.32-3.29 (m, 1 H), 2.17-2.12 (m, 2 H), 1.94-1.91 (m, 2 H); ^{13}C NMR (acetone-d_6, 125 MHz): δ 169.4 (C), 156.9 (C), 146.4 (C), 124.9 (C), 118.4 (CH), 116.1 (CH), 115.3 (CH), 90.1 (CH), 68.6 (CH), 55.9 (CH_3), 39.3 (CH), 35.2 (CH), 29.2 (CH_2), 18.9 (CH_2); MS (*m/z*, relative intensity): 293 (M^+, 100), 246 (55), 229 (18), 227 (60), 189 (19), 174 (26), 161 (44), 105 (54), 91 (62), 77 (28), 57 (87), 55 (62); exact mass calculate for $C_{14}H_{15}NO_6$ (M^+): 293.0899; found (M^+): 293.0901.

Fig S13. 1H NMR (CDCl3, 500 MHz) of compound cis-3a

PMK-02-34-C1p

exp24 s2pul

SAMPLE		DEC. & VT	
date	Mar 8 2010	dfrq	125.693
solvent	cdcl3	dn	C13
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.830	dm	nnn
tn	H1	dmm	c
at	3.000	dmf	200
np	48000	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	PROCESSING	
tpwr	58	wtfile	
pw	4.8	proc	ft
d1	1.000	fn	not used
tof	499.7	math	f
nt	4		
ct	4	werr	react
alock	y	wexp	procplot
gain	not used	wbs	
FLAGS		wnt	wft
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	4998.0		
vs	57		
sc	0		
wc	210		
hzmm	23.80		
is	70.52		
rfl	4638.9		
rfp	3618.7		
th	3		
ins	100.000		
nm	ph		



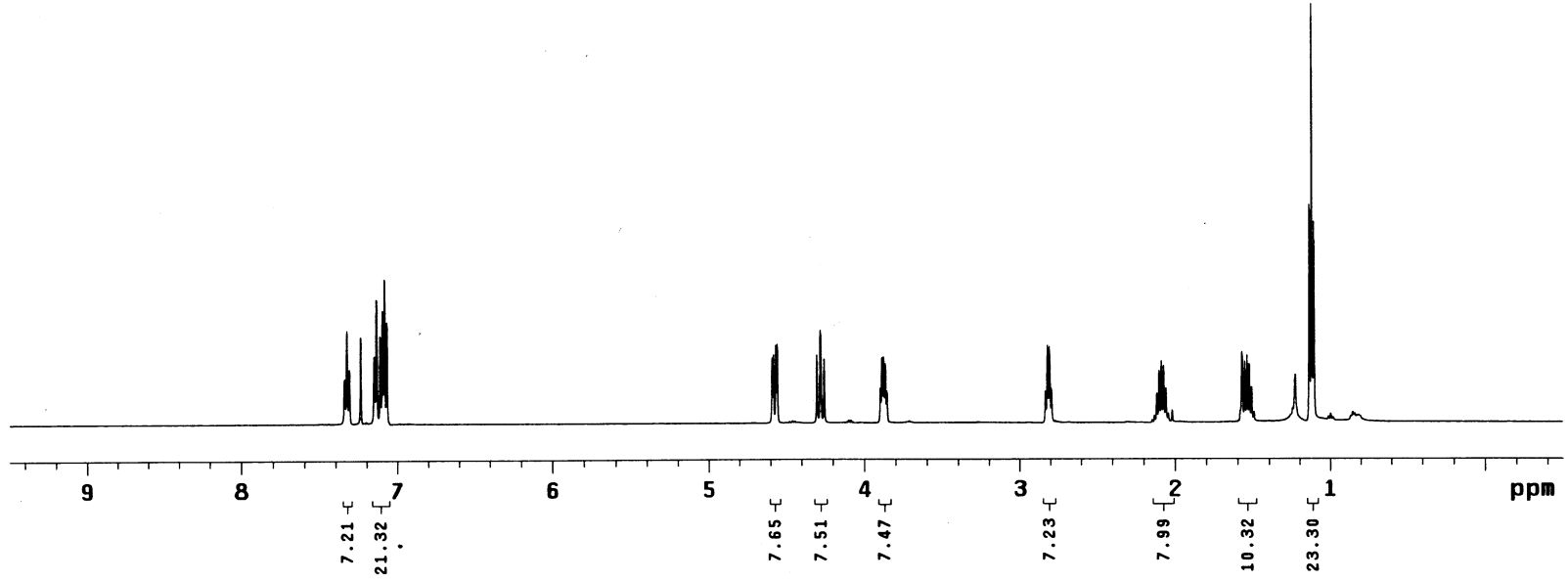


Fig S14. 13C NMR (CDCl3, 125 MHz) of compound cis-3a

PMK-02-334-F1
exp23 s2pul

SAMPLE		DEC. & VT	
date	Mar 8 2010	dfrq	499.829
solvent	cdcl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	125.696	dm	yyy
tn	C13	dmm	w
at	1.000	dmf	11905
np	62894	dseq	
sw	31446.5	dres	1.0
fb	not used	homo	n
bs	16	PROCESSING	
ss	2	lb	1.00
tpwr	54	wtfile	
pw	4.0	proc	ft
d1	1.000	fn	not used
tof	2512.2	math	f
nt	5000		
ct	5000	werr	react
alock	y	wexp	procplot
gain	not used	wbs	testsn
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1257.0		
wp	27649.1		
vs	50		
sc	0		
wc	210		
hzmm	131.67		
is	580.00		
rfl	10984.4		
rfp	9677.5		
th	4		
ins	100.000		
nm	ph		

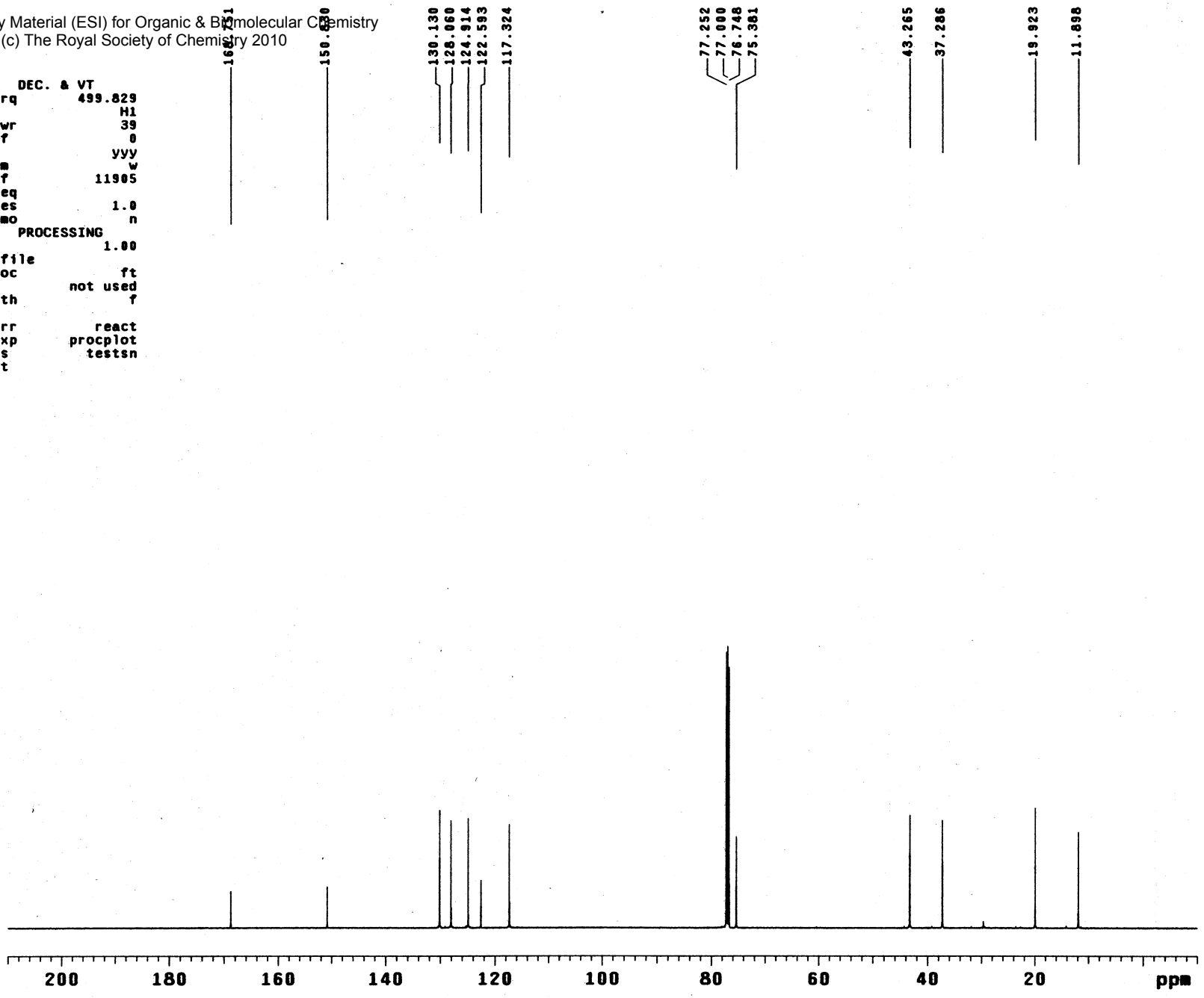


Fig S15. DEPT of compound cis-3a

PMK-02-334-F1
exp25 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Mar 8 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	1	mult
sw	31446.5	gain	24	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	384	wp	27649.1		
ct	384	sp	-1257.4		
TRANSMITTER		rp	131.0		
tn	C13	lp	188.7		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1269.9		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	77		
dmm	ccw	hzmm	131.67		
dmf	11905	th	68		
pplvl	51				
pp	25.600				

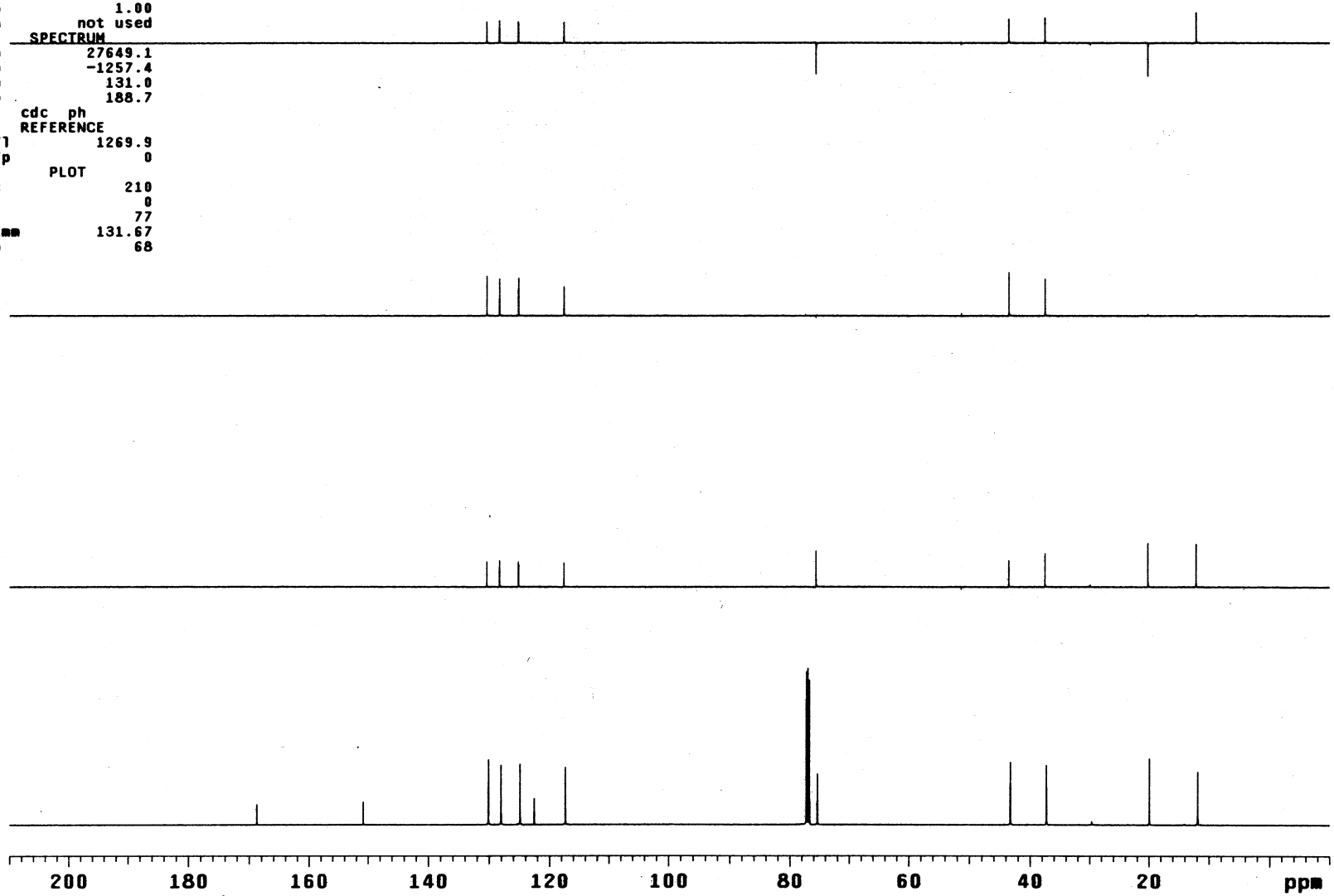


Fig S16. HSQC of compound cis-3a

PMK-02-334-F1

exp27 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Mar 8 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	5006.3	SPECIAL		1	1
at	0.205	temp	not used	2	2
np	2048	gain	24		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	16	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.094		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-499.9	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	104.2		
dmm	ccp	wp	3803.6		
dmf	32258	sp1	806.5		
dpwr	36	wp1	16213.4		
pwxlvl	52	rf1	2660.9		
pwx	14.300	rfp	2139.3		
HSQC		rf1	10775.1		
j1xh	140.0	rfp1	9474.0		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	113		
		th	4		
		ai cdc ph			

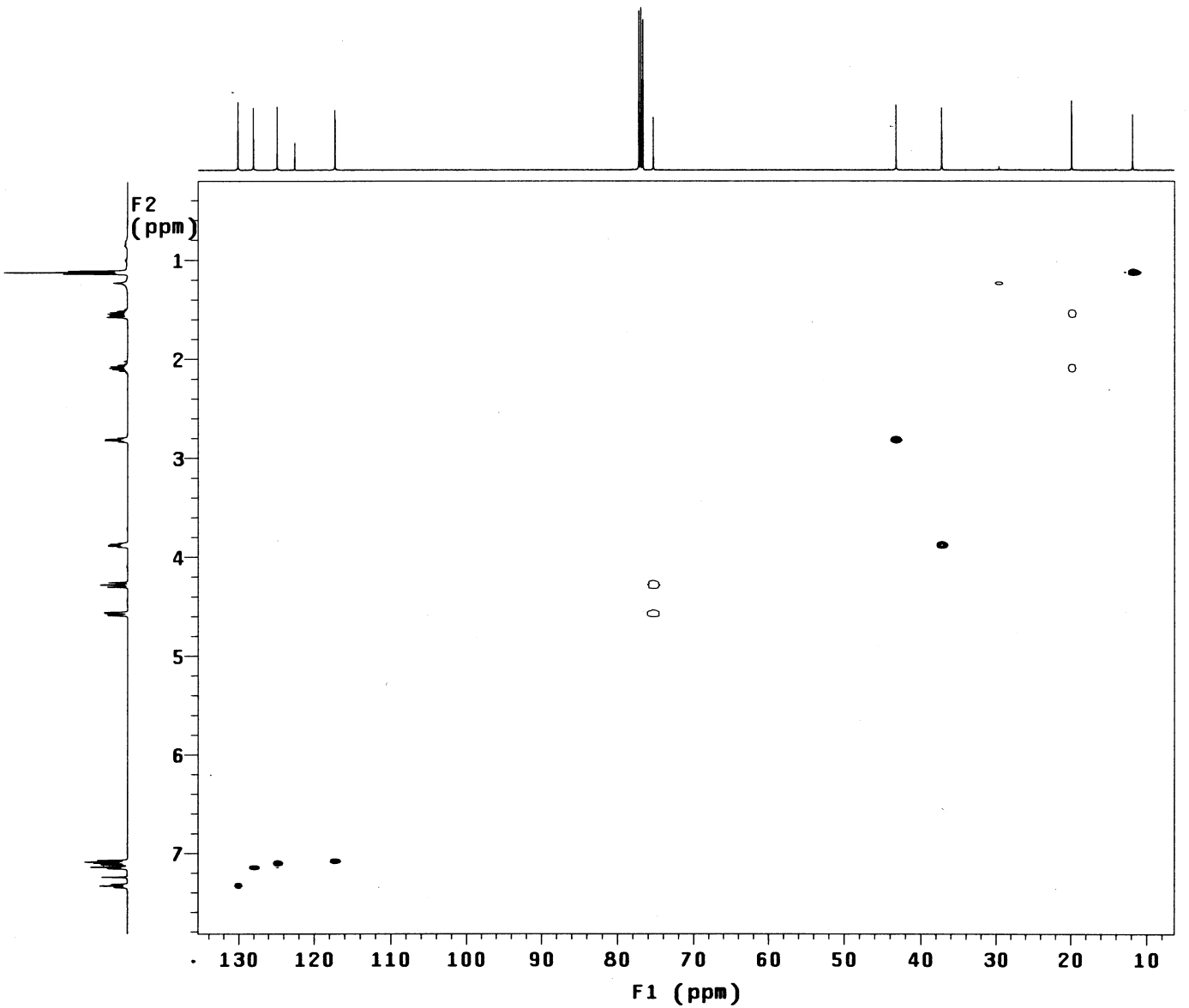


Fig S17. COSY of compound cis-3a

PMK-02-334-F1

exp26 gCOSY

SAMPLE		FLAGS	
date	Mar 8 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	5006.3	temp	not used
at	0.205	gain	24
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.102
d1	1.000	sbs	not used
nt	8	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	5006.3	sb1	-0.026
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-499.9	sp	-519.1
tpwr	58	wp	5001.4
pw	11.100	sp1	-517.0
GRADIENTS		wp1	5001.4
gzlvl1	1003	rfl	2663.2
gt1	0.001000	rfp	2139.3
gstab	0.000500	rfl1	2661.1
DECOUPLER		rfp1	2139.3
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	113
		th	5
		ai cdc av	

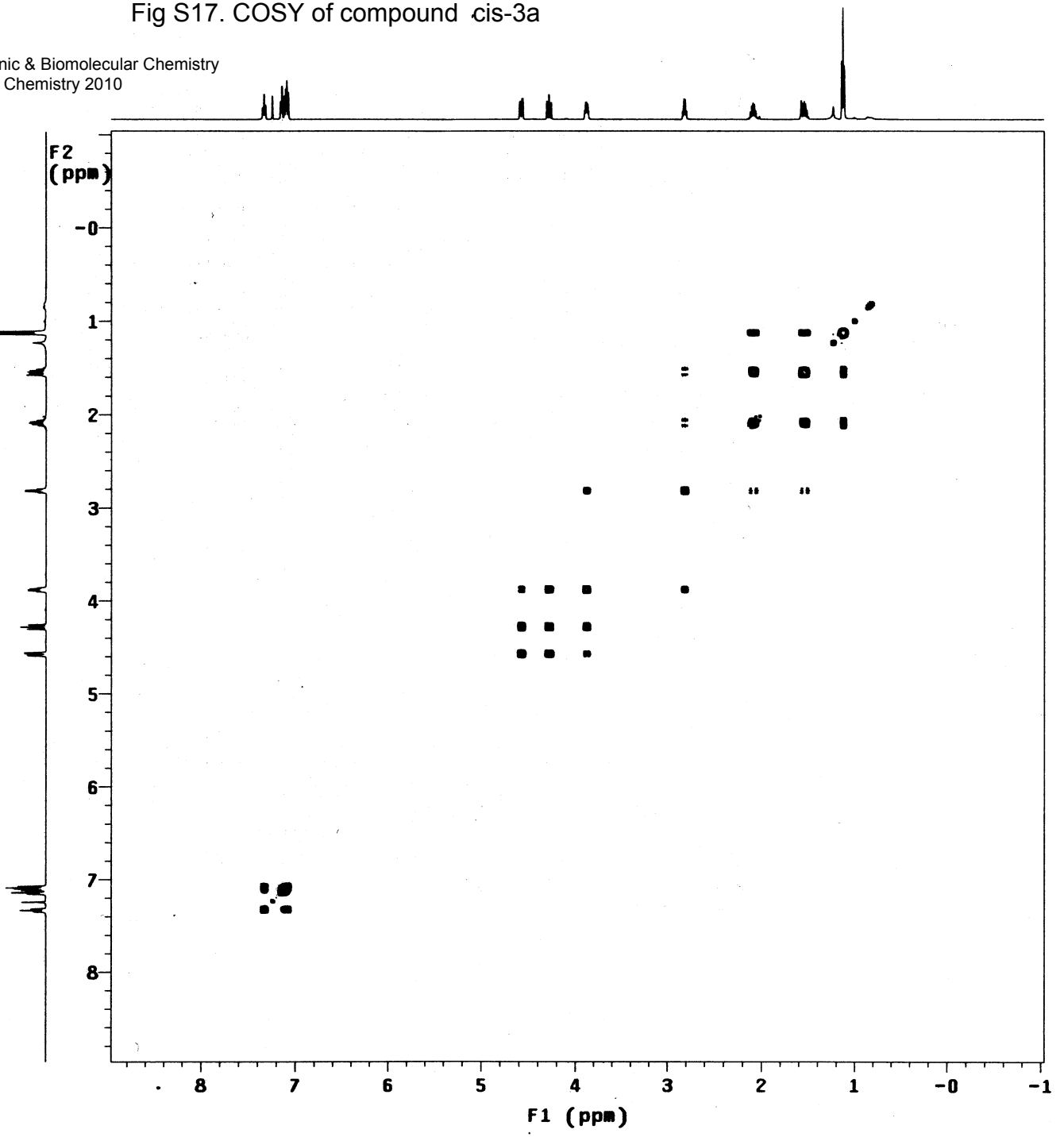


Fig S18. NOESY of compound cis-3a

exp28 NOESY

SAMPLE	
date	Mar 8 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	5006.3
at	0.205
np	2048
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	5006.3
ni	200
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-499.8
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	20
spin	0
F2 PROCESSING	
gf	0.004
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.837
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	-522.1
wp	5001.4
sp1	-520.8
wp1	5001.4
rf1	2666.2
rfp	2139.3
rf11	2664.2
rfp1	2139.3
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	113
th	1
ai ph	

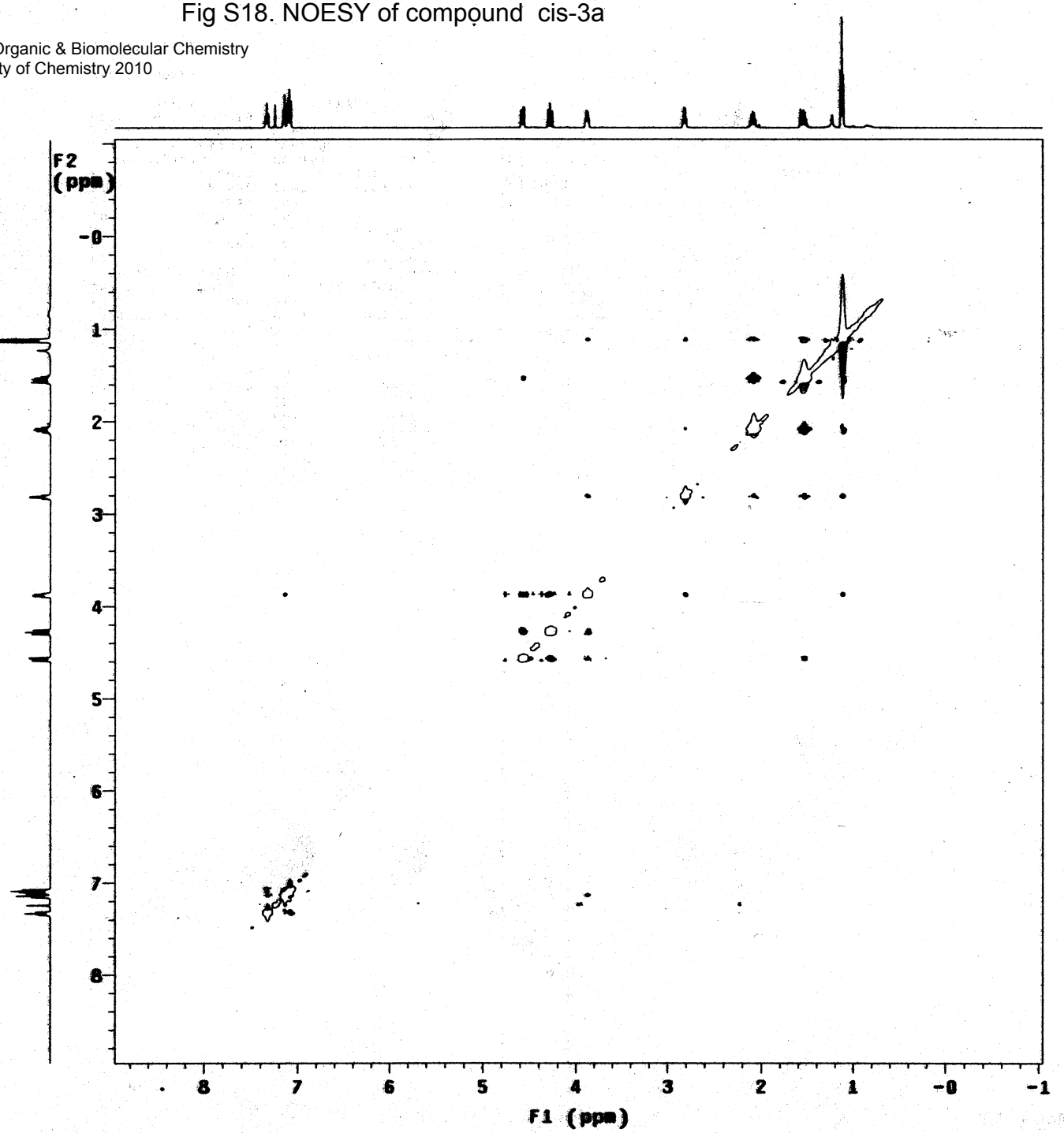


Fig S19. 1H NMR (CDCl3, 500 MHz) of compound trans-3a

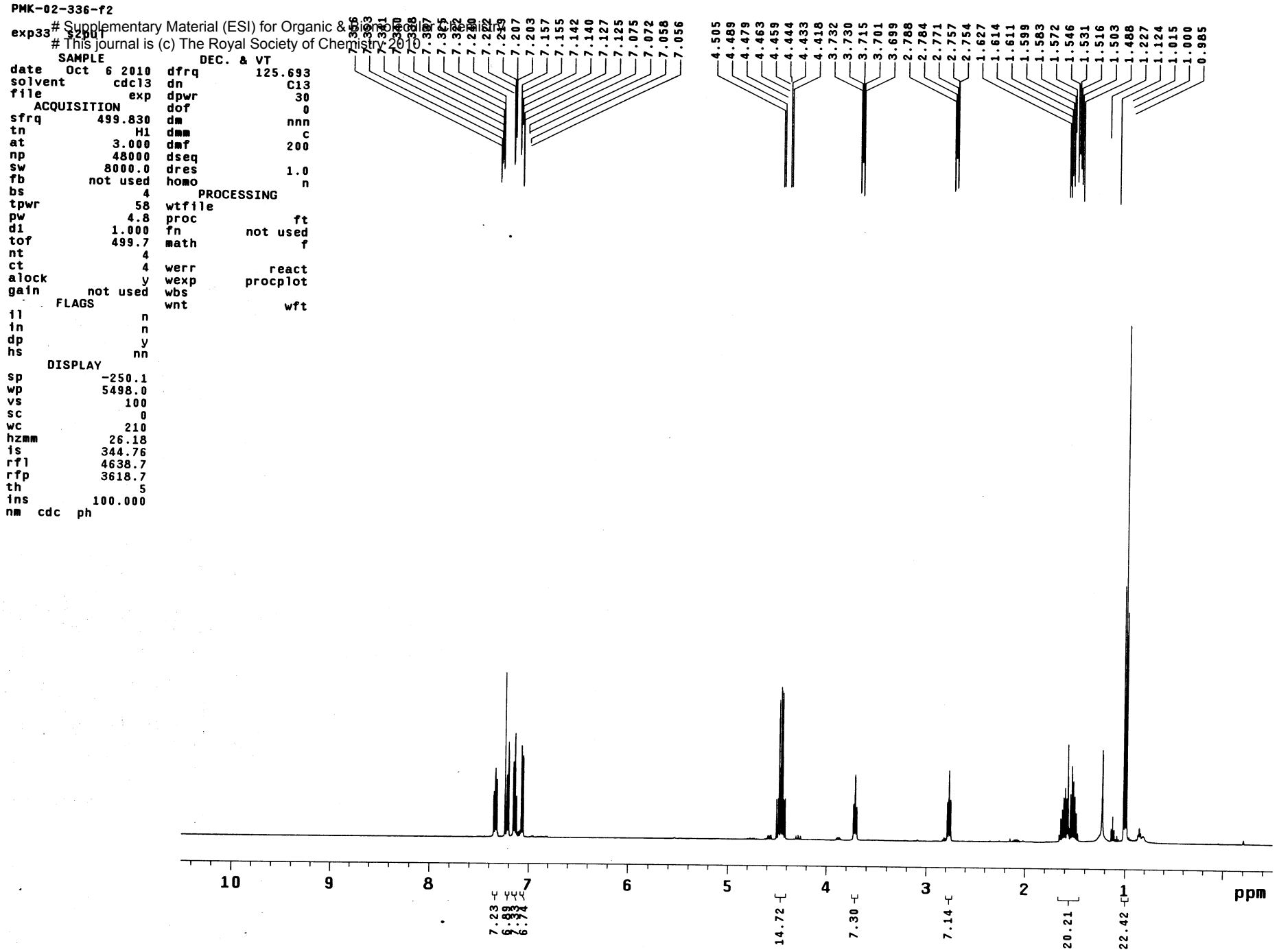


Fig S20. 13C NMR (CDCl3, 125 MHz) of compound trans-3a

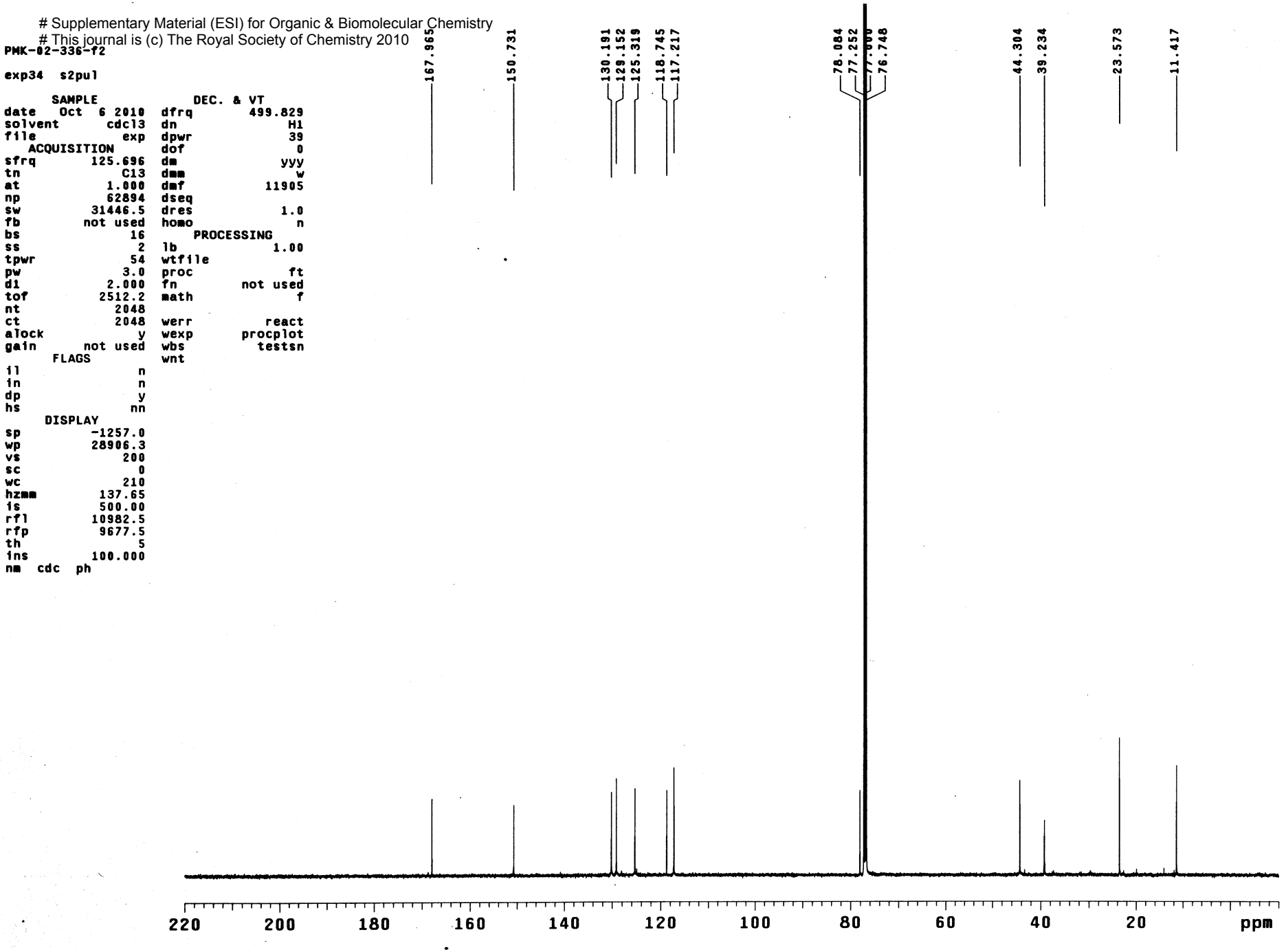


Fig S21. DEPT of compound trans-3a

PMK-02-336-f2

exp35 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Oct 6 2010	jlxh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	1	mult
sw	31446.5	gain	20	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.0		
TRANSMITTER		rp	-128.2		
tn	C13	lp	212.8		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1305.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	200		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	31.000				

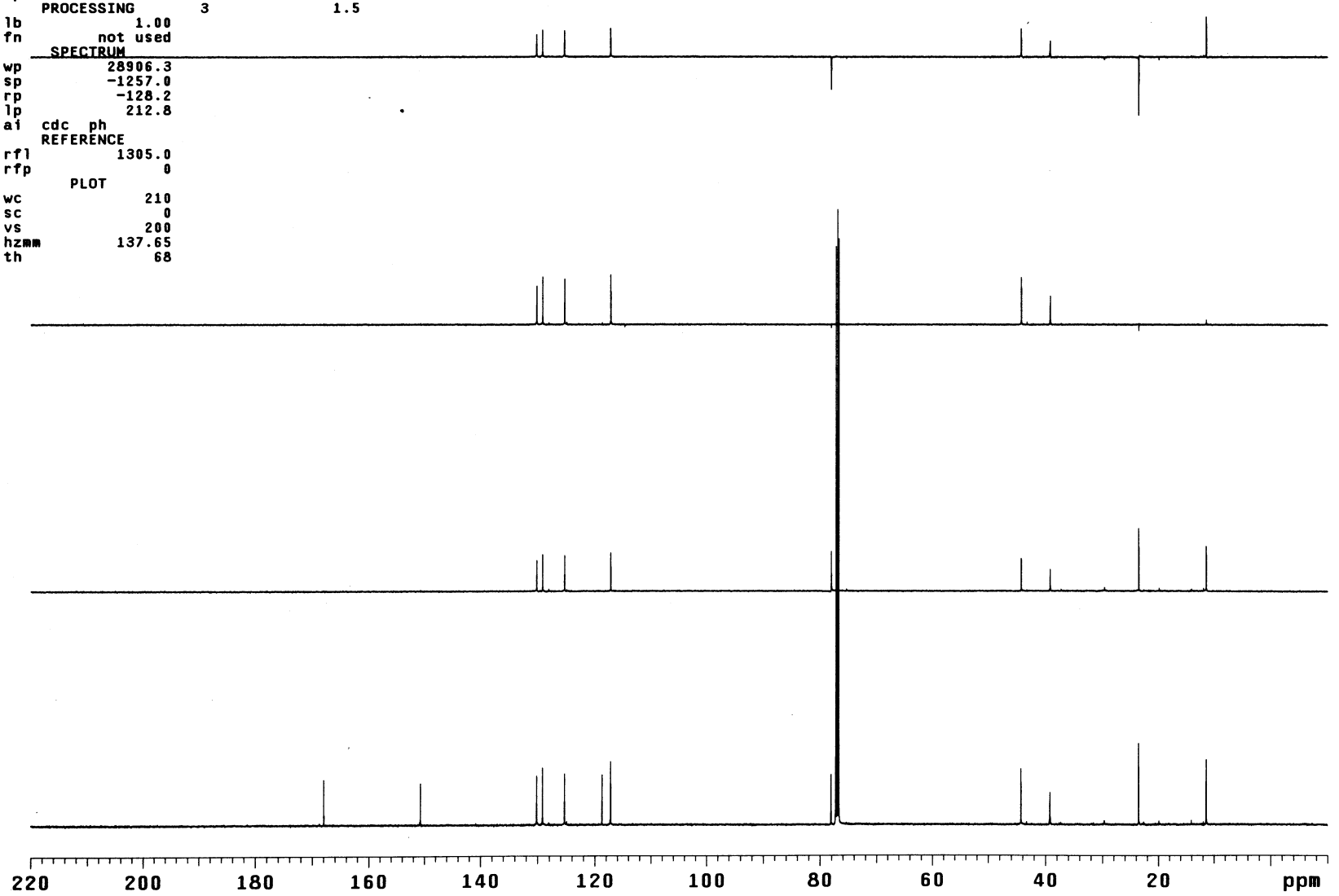


Fig S22. HSQC of compound trans-3a

PMK-02-336-f2

exp38 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Oct 6 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	not used	2	2
np	2048	gain	48		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	301.8		
dmm	ccp	wp	3587.0		
dmf	32258	sp1	738.0		
dpwr	36	wp1	16672.5		
pwxlvl	52	rfl	1870.8		
pwx	14.300	rfp	1856.9		
HSQC		rfl1	6217.1		
jlxh	140.0	rfp1	4931.0		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	6		
		ai cdc ph			

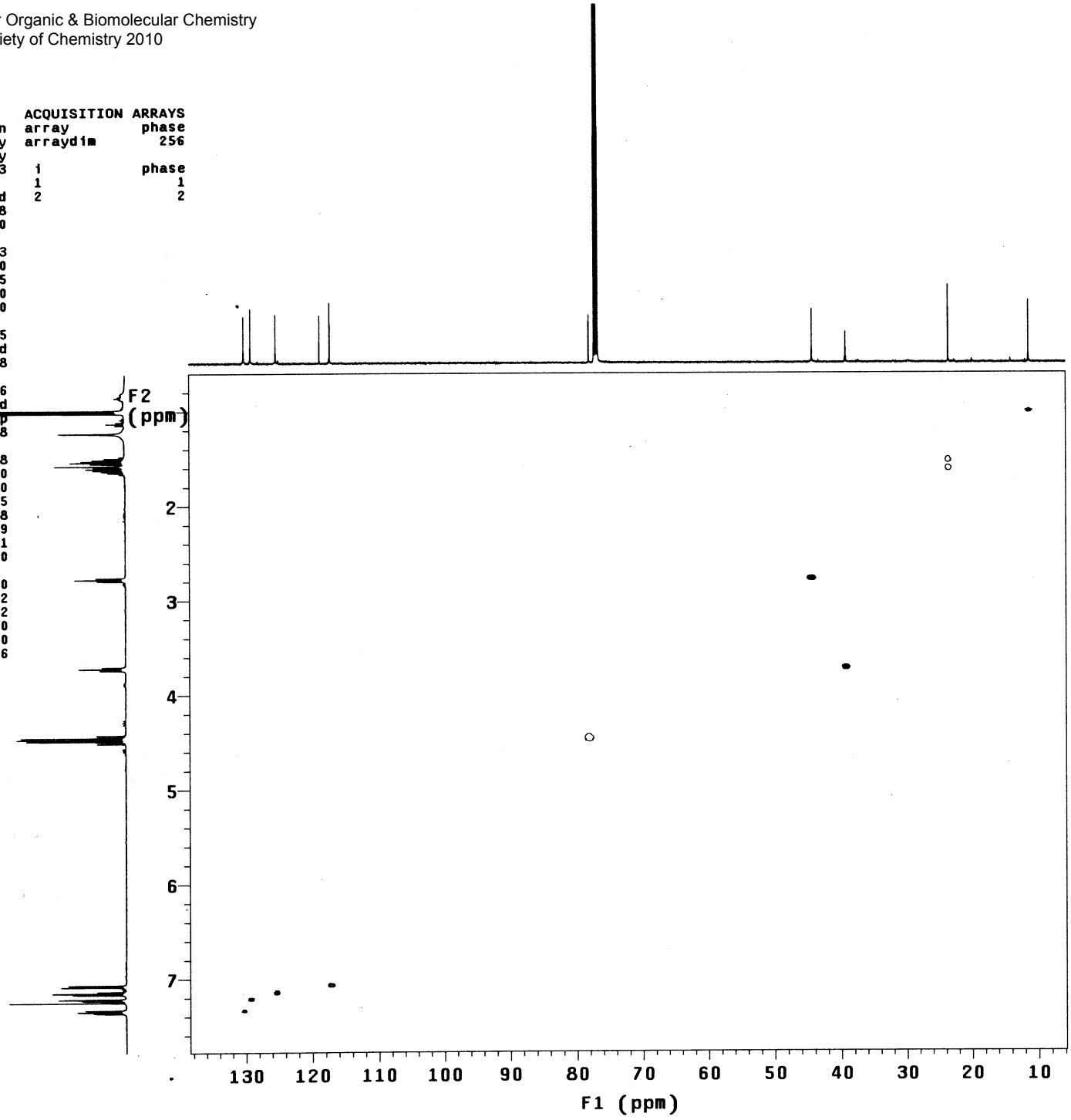


Fig S23.COSY of compound trans-3a

PMK-02-036-f2

exp36 gCOSY

SAMPLE		FLAGS	
date	Oct 6 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4490.3	temp	not used
at	0.228	gain	38
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.114
d1	1.000	sbs	not used
nt	8	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	4490.3	sb1	-0.029
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-250.0	sp	318.0
tpwr	58	wp	3494.9
pw	11.100	sp1	317.7
GRADIENTS		wp1	3499.3
gzlvl1	1003	rfl	1872.1
gt1	0.001000	rfp	1856.9
gstab	0.000500	rfl1	1872.4
DECOUPLER		rfp1	1856.8
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	6
		ai cdc av	

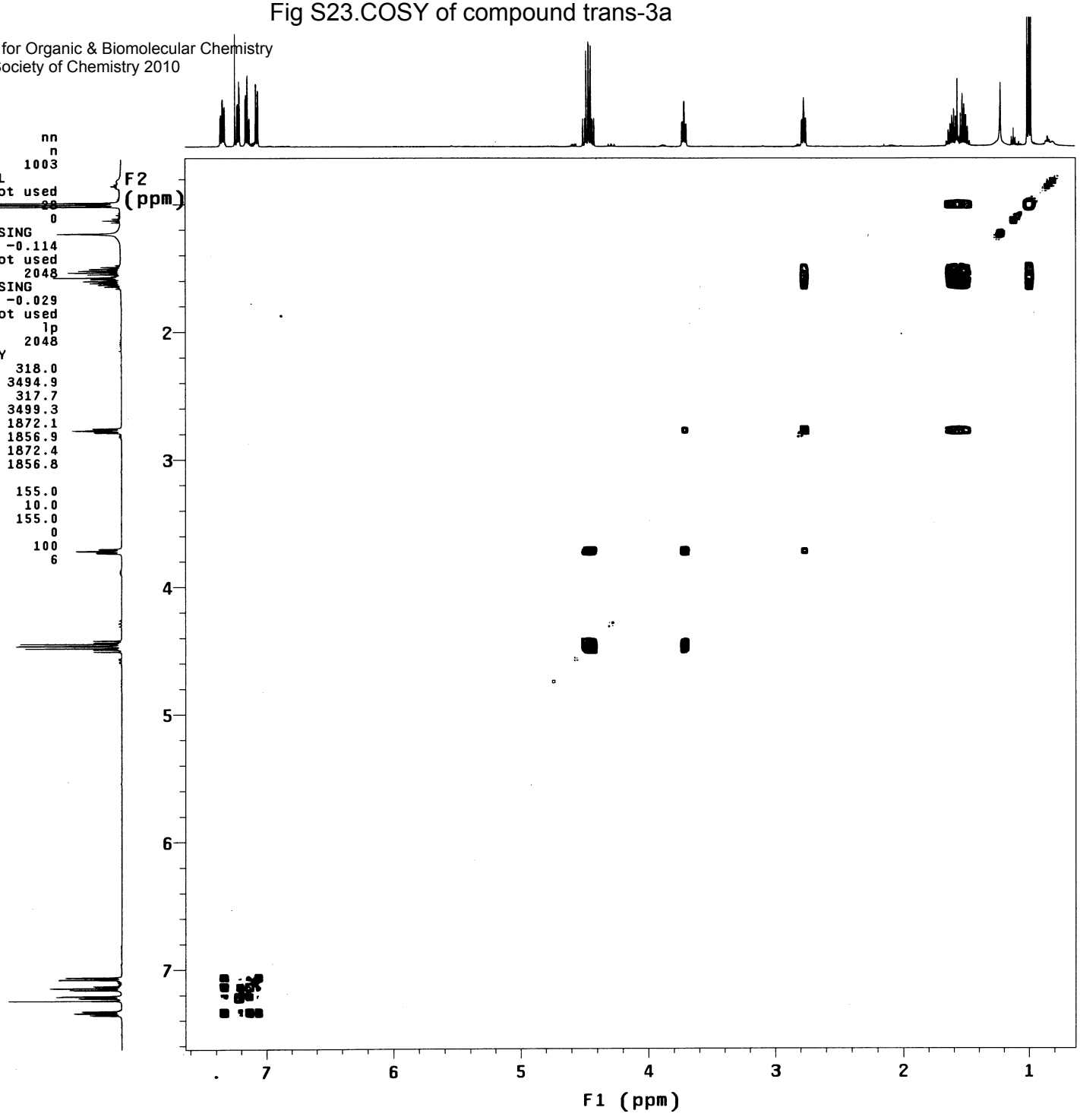


Fig S24. NOESY of compound trans-3a

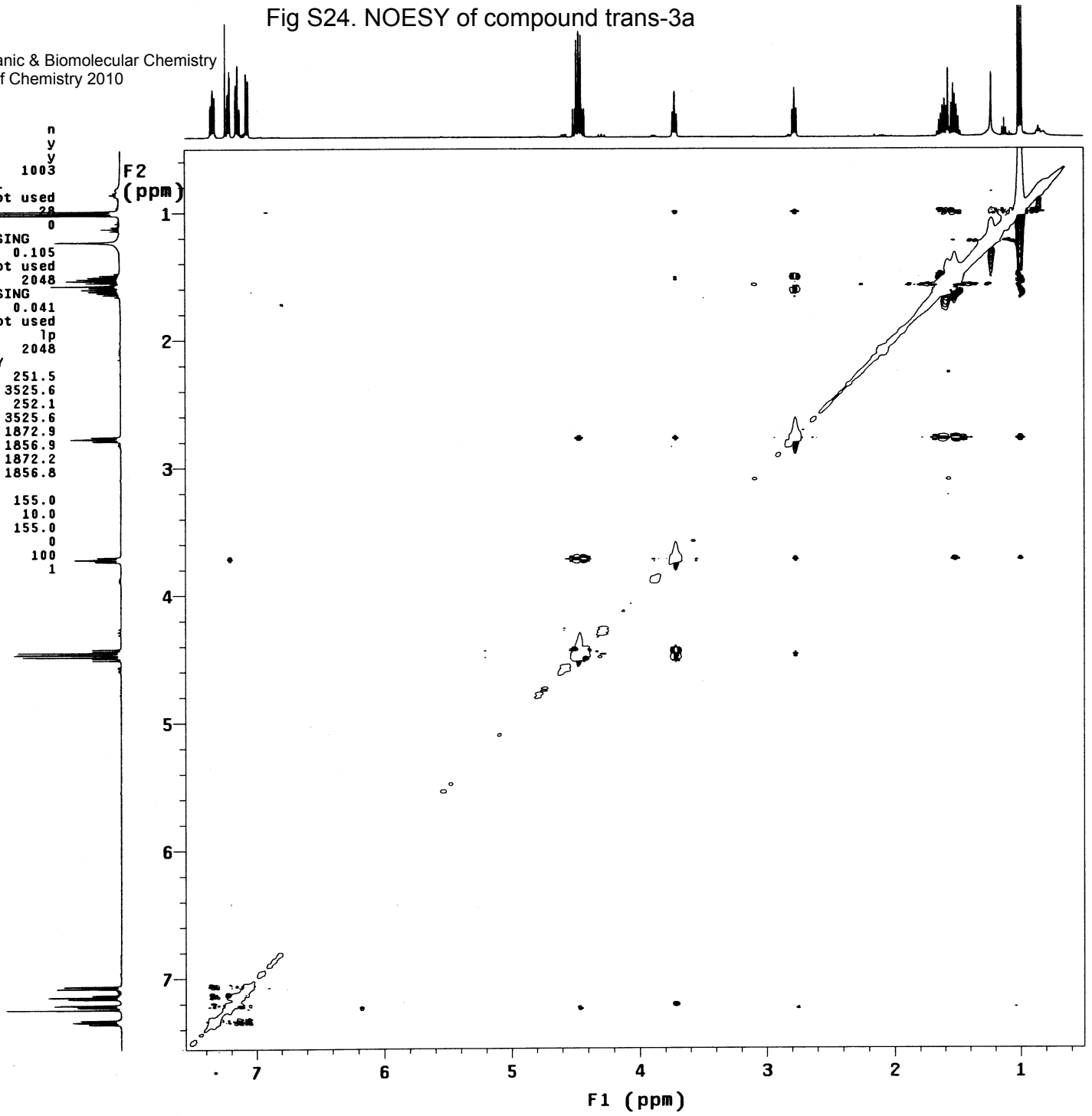
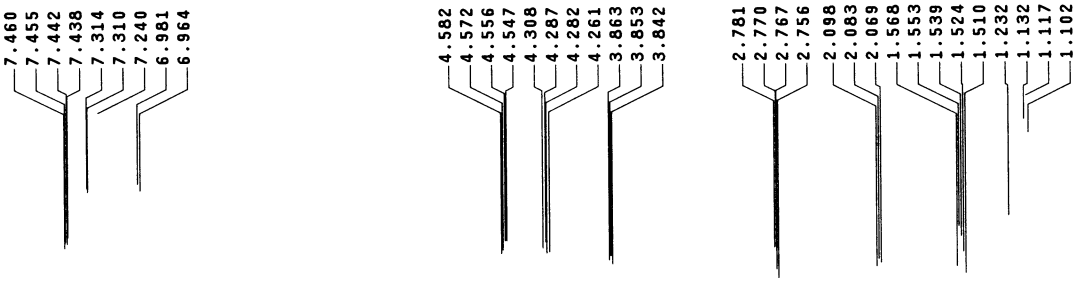


Fig S25. 1H NMR (CDCl3, 500 MHz) of compound 3b

PMK-02-364

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 13 2010	dfrq	125.693
solvent	cdcl3	dn	C13
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.830	dm	nnn
tn	H1	dmm	c
at	3.000	dmf	200
np	48000	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	PROCESSING	
tpwr	58	wtfile	
pw	4.8	proc	ft
d1	1.000	fn	not used
tof	499.7	math	f
nt	4		
ct	4	werr	react
alock	y	wexp	procplot
gain	not used	wbs	
FLAGS		wnt	wft
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5248.0		
vs	50		
sc	0		
wc	210		
hzmm	24.99		
is	274.81		
rfl	4637.9		
rfp	3618.7		
th	4		
ins	100.000		
nm cdc ph			



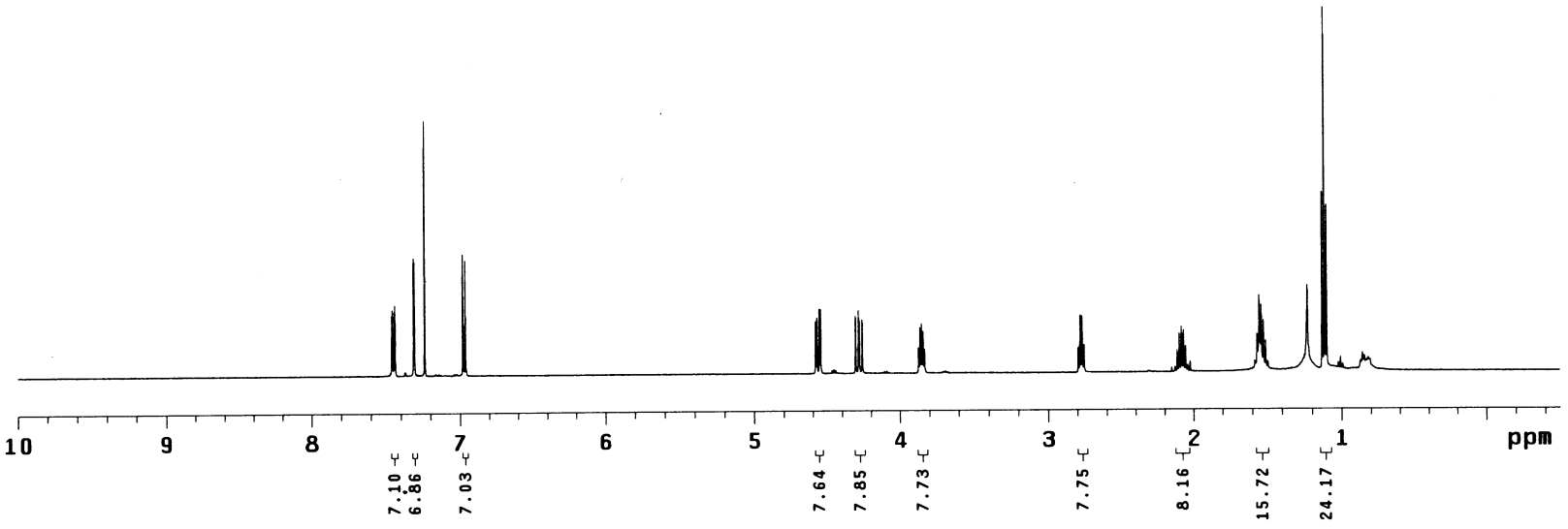


Fig S26. 13C NMR (CDCl3, 125 MHz) of compound 3b


```
PMK-02-364

exp4  s2pul

      SAMPLE          DEC. & VT
date   Apr 13 2010  dfrq        499.829
solvent      cdcl3  dn               H1
file           exp  dpwr             39
   ACQUISITION      dof               0
sfrq       125.696  dm              yyy
tn             C13  dmm               w
at           1.000  dmf           11905
np           62894  dseq
sw         31446.5  dres            1.0
fb       not used  homo              n
bs             16      PROCESSING
ss              2  lb             1.00
tpwr           54  wtfile
pw            4.0  proc              ft
d1          1.000  fn        not used
tof        2512.2  math              f
nt           2048
ct           2048  werr          react
alock           y  wexp       procplot
gain     not used  wbs         testsn
      FLAGS        wnt
il              n
in              n
dp              y
hs             nn
     DISPLAY
sp        -1257.0
wp        28906.3
vs            150
sc              0
wc            210
hzmm       137.65
is         500.00
rfl       10981.5
rfp        9677.5
th              5
ins       100.000
nm  cdc  ph
```

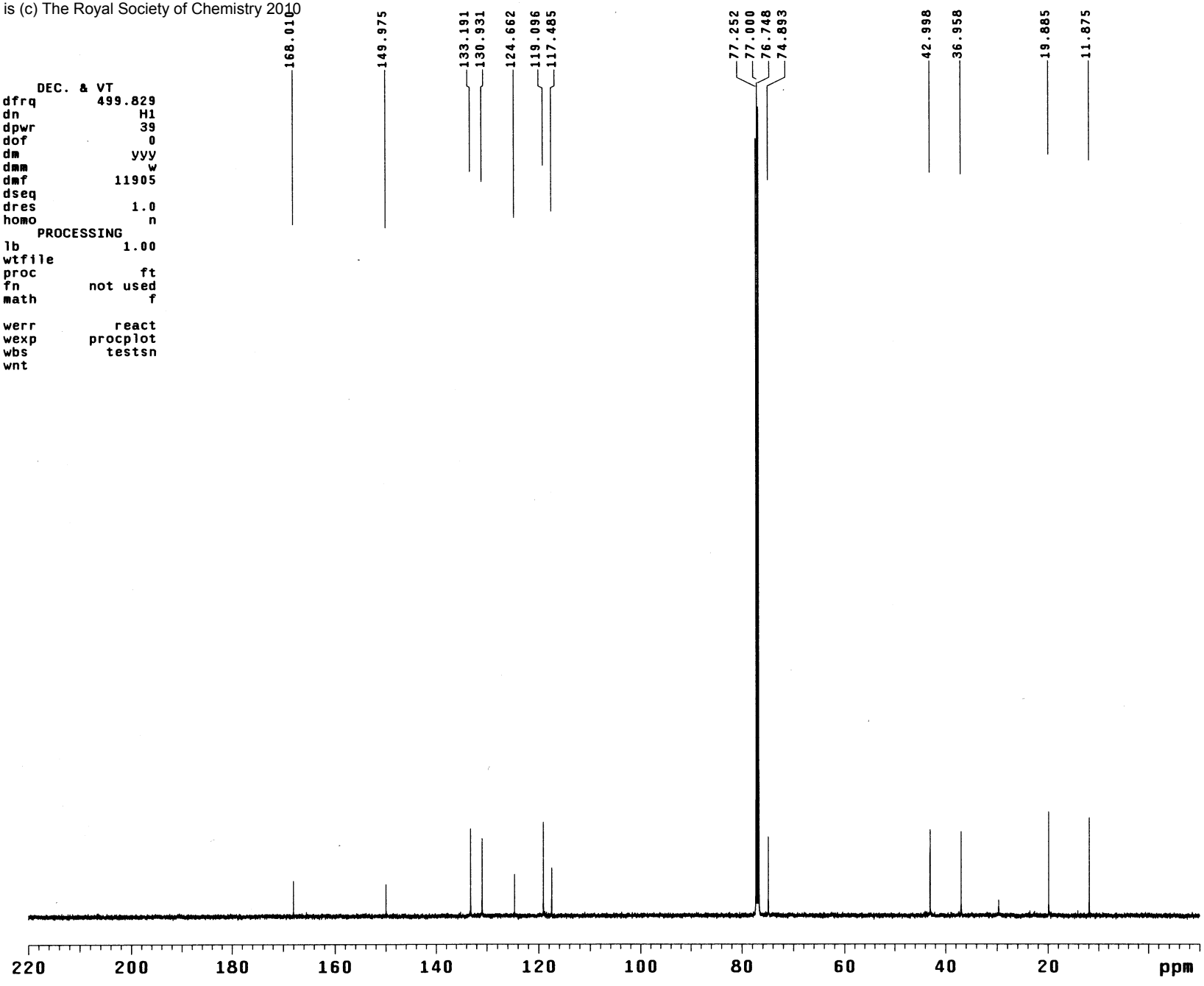


Fig S27. DEPT of compound 3b

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 13 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	34	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	28906.3		
ct	2048	sp	-1257.0		
TRANSMITTER		rp	48.6		
tn	C13	lp	170.9		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1303.1		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	500		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

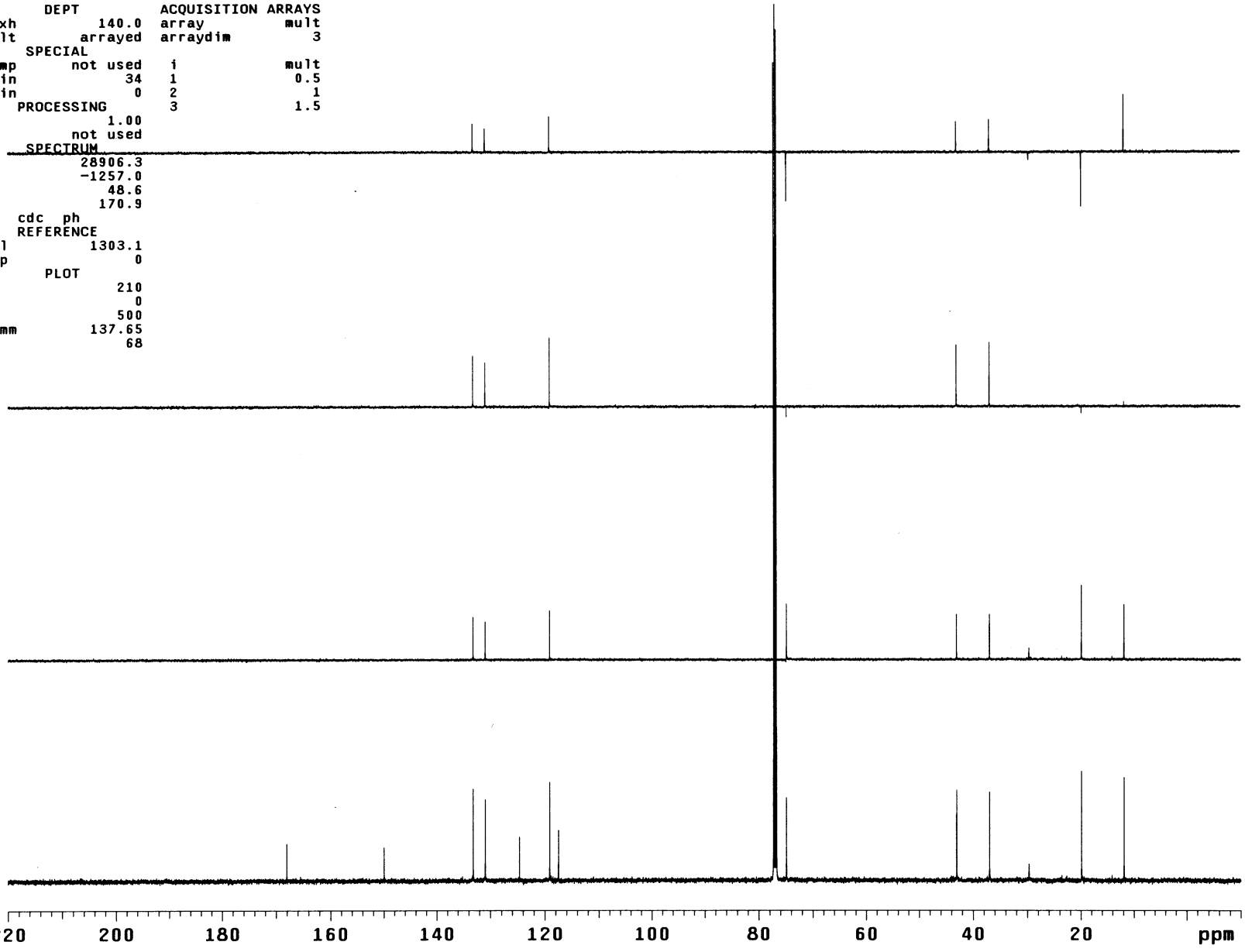


Fig S28. HSQC of compound 3b

PMK-02-364
exp8 gHSQC

SAMPLE	
date	Apr 13 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	32
d1	1.000
nt	16
2D ACQUISITION	
sw1	21367.5
ni	128
phase	arrayed
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
DECOUPLER	
dn	C13
dof	-2515.2
dm	nny
dmm	ccp
dmf	32258
dpwr	36
pwxlvl	52
pwx	14.300
HSQC	
j1xh	140.0
nullflg	y
mult	2

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	20
spin	0
GRADIENTS	
gzlvl1	1003
gt1	0.002000
gzlvl3	505
gt3	0.001000
gstab	0.000500
F2 PROCESSING	
gf	0.105
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.006
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	447.0
wp	3569.5
sp1	792.8
wp1	16776.8
rfl	571.8
rfp	558.3
rfl1	2765.4
rfp1	1492.5
PLOT	
wc	150.0
sc	6.2
wc2	116.2
sc2	0
vs	100
th	6
ai cdc ph	

ACQUISITION ARRAYS	
array	phase
arraydim	256
i	phase
1	1
2	2

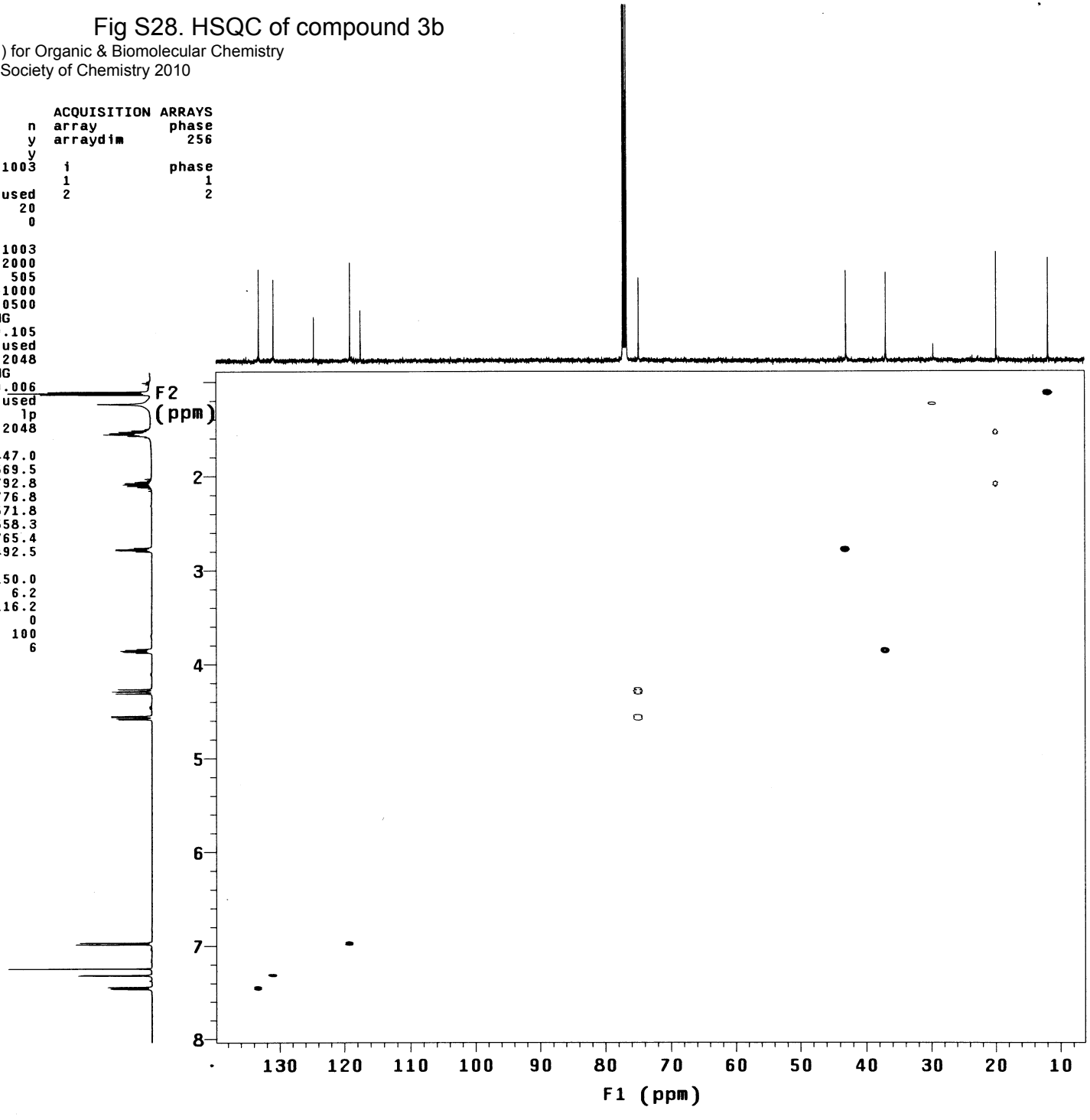


Fig S29. COSY of compound 3b

PMK-02-364

exp6 gCOSY

SAMPLE		FLAGS	
date	Apr 13 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4490.3	temp	not used
at	0.228	gain	34
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.114
d1	1.000	sbs	not used
nt	16	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	4490.3	sb1	-0.029
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-250.0	sp	312.7
tpwr	58	wp	3578.2
pw	11.100	sp1	300.4
GRADIENTS		wp1	3582.6
gzlvl1	1003	rf1	574.5
gt1	0.001000	rfp	558.3
gstab	0.000500	rf11	573.6
DECOUPLER		rfp1	558.3
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	8
		ai cdc av	

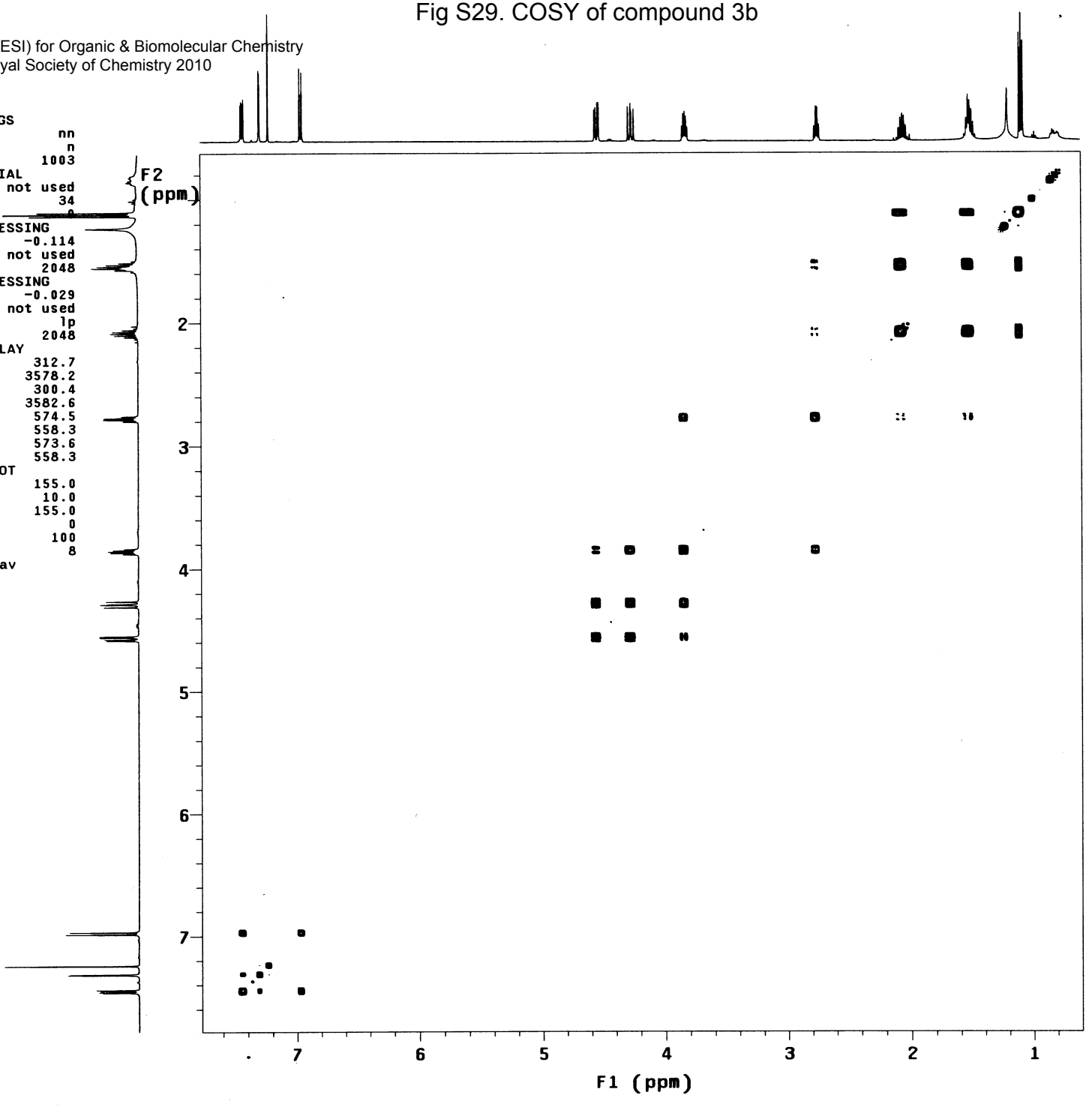


Fig S30. NOESY of compound 3b

PMK-02-364

exp7 NOESY

SAMPLE		FLAGS	
date	Apr 13 2010	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	4490.3	SPECIAL	
at	0.228	temp	not used
np	2048	gain	34
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.105
nt	8	gfs	not used
2D ACQUISITION		fn	2048
sw1	4490.3	F1 PROCESSING	
ni	200	gf1	0.041
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.829	fn1	2048
tof	-250.0	DISPLAY	
tpwr	58	sp	240.0
pw	11.100	wp	3652.8
NOESY		sp1	236.2
mix	0.600	wp1	3657.2
PRESATURATION		rf1	572.6
satmode	nnnn	rfp	558.3
satpwr	0	rfl1	572.0
satdly	0	rfp1	558.3
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	100
		th	2
		ai ph	

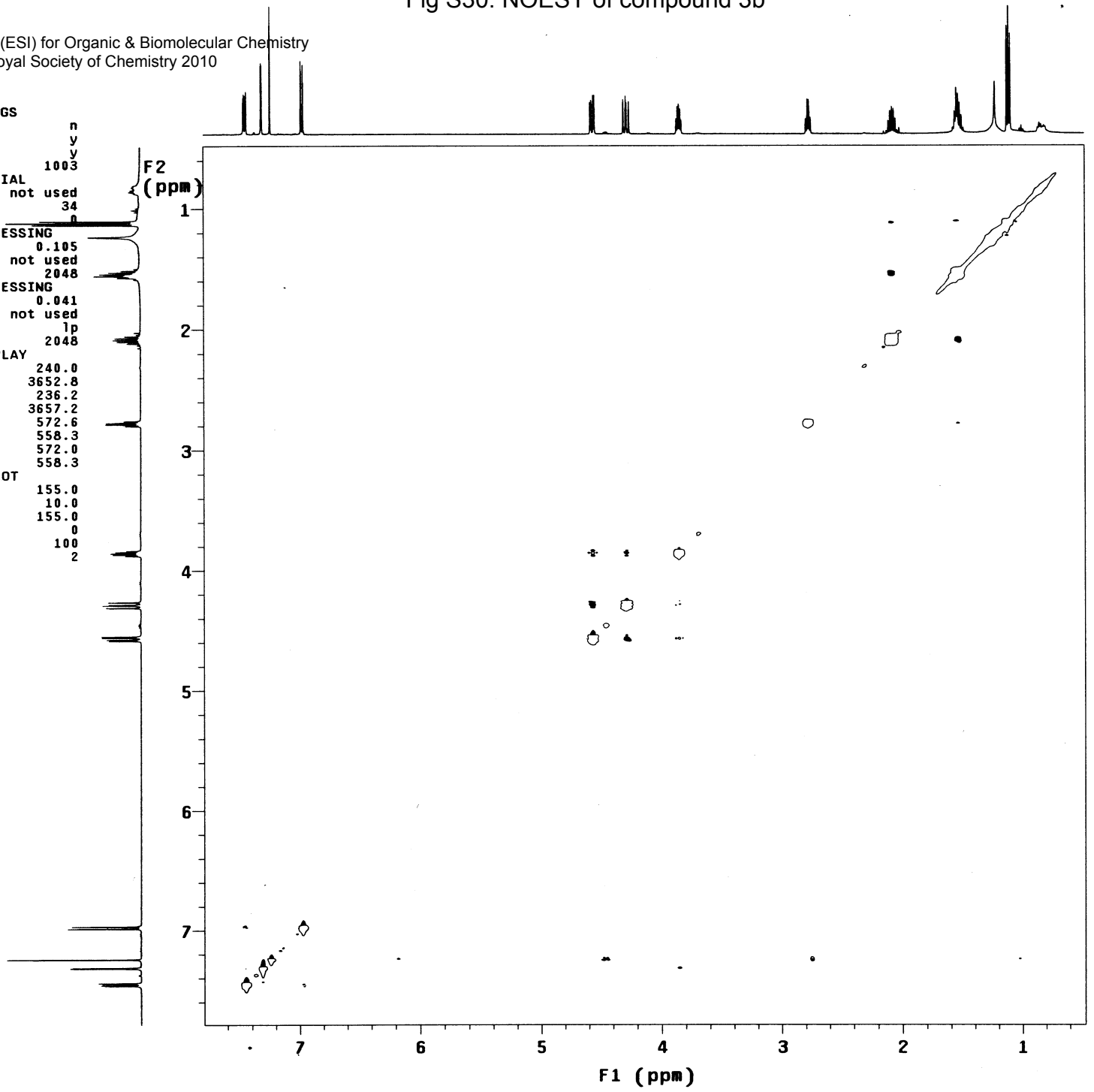


Fig S31. 1H NMR (CDCl3, 500 MHz) of compound 3c


```
PMK-02-365
exp23  s2pul

      SAMPLE                DEC. & VT
date    Apr 16 2010   dfrq        125.693
solvent       cdcl3   dn              C13
file /export/home/~   dpwr             30
vnmr1/vnmrsys/data~   dof               0
/PMK/PMK-02-365/H.~   dm              nnn
                fid   dmm               c
   ACQUISITION        dmf             200
sfrq        499.830   dseq
tn               H1   dres            1.0
at             3.000   homo              n
np             48000       PROCESSING
sw            8000.0   wtfile
fb         not used    proc             ft
bs                4    fn         not used
tpwr             58    math              f
pw              4.8
d1            1.000    werr          react
tof           499.7    wexp       procplot
nt                4    wbs
ct                4    wnt              wft
alock             y
gain       not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -250.1
wp           5248.0
vs              100
sc                0
wc              210
hzmm          24.99
is           360.45
rfl          4637.9
rfp          3618.7
th                2
ins         100.000
nm  cdc  ph
```

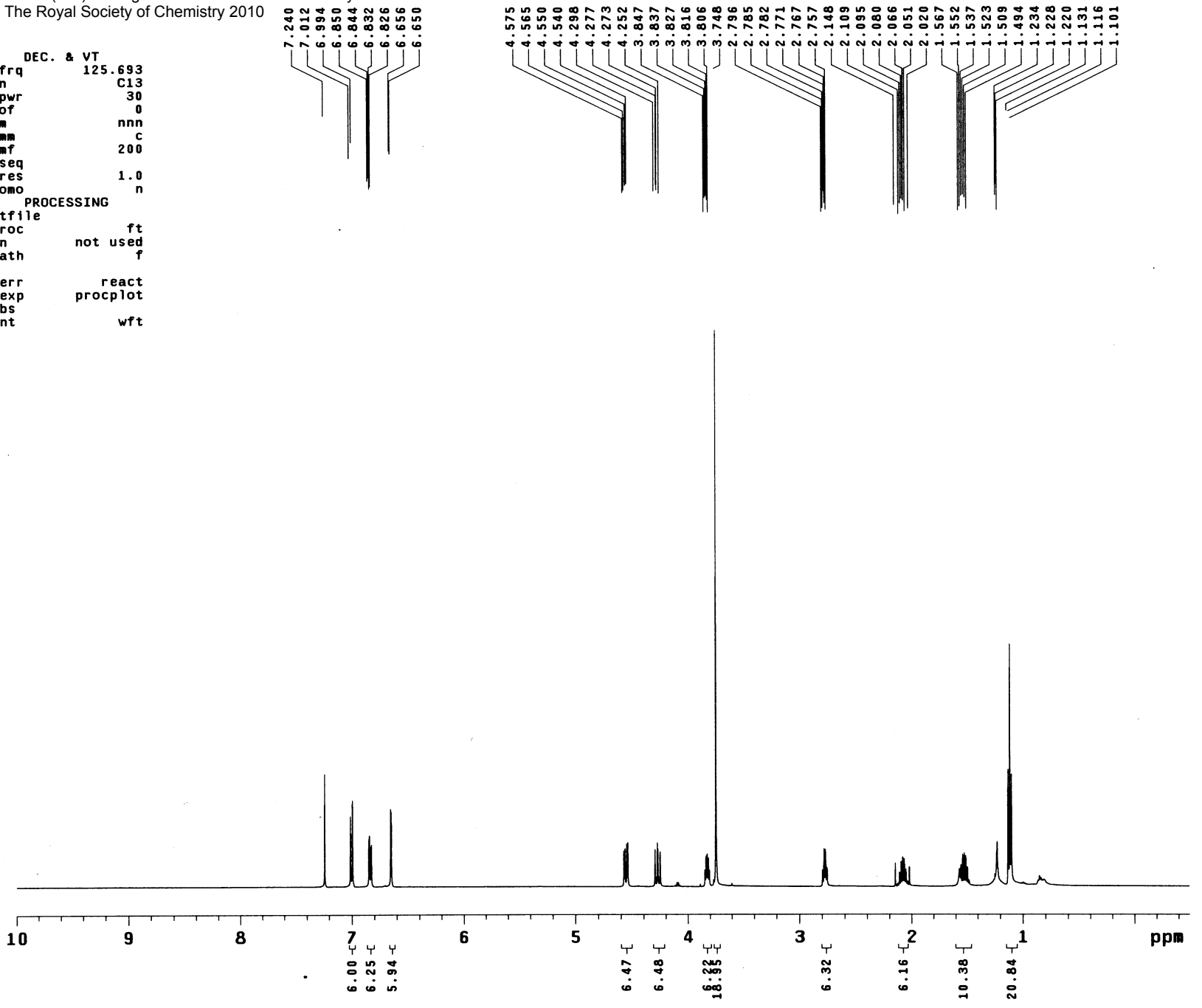


Fig S32. 13C NMR (CDCl3, 125 MHz) of compound 3c

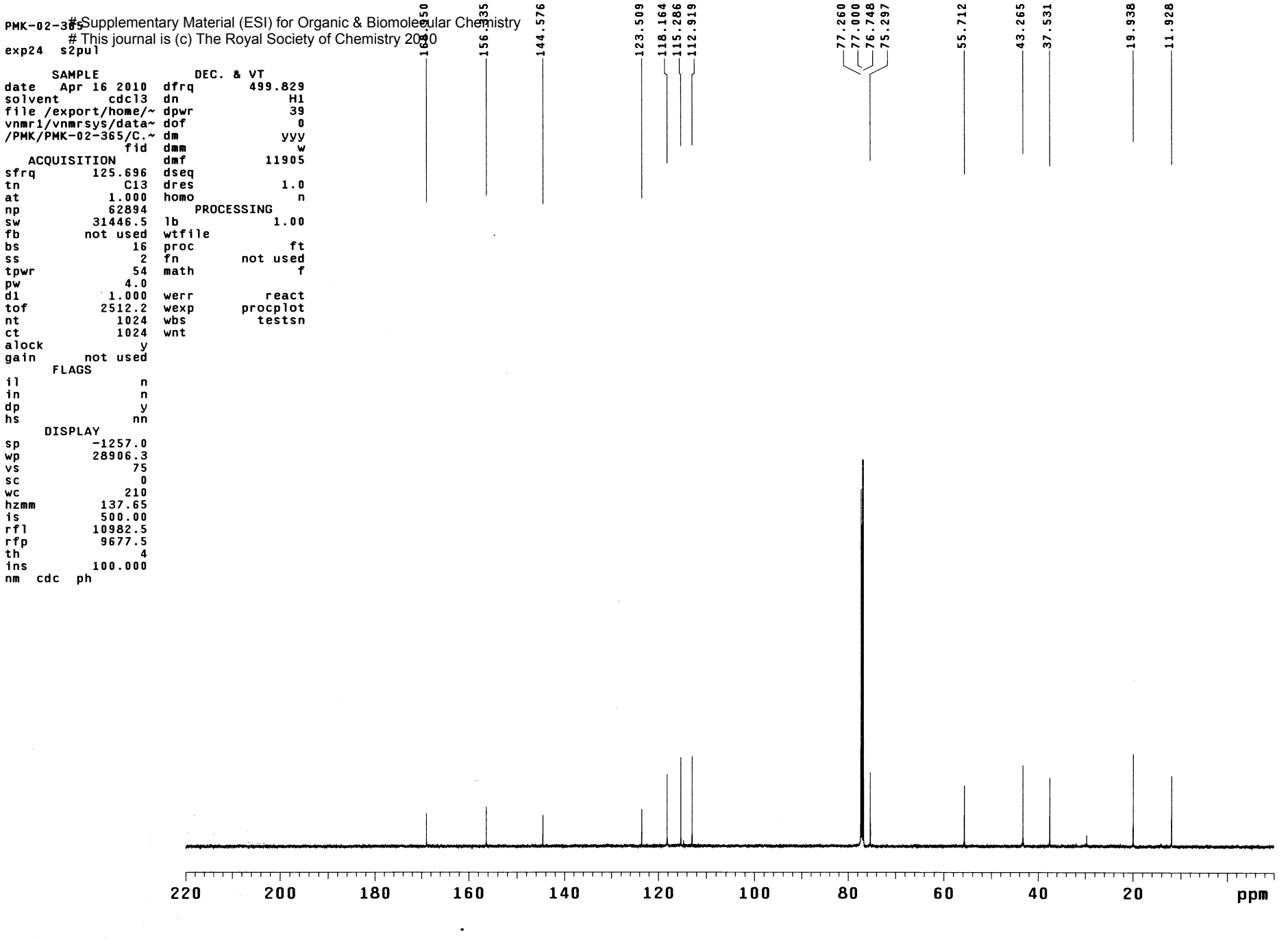


Fig S33. DEPT of compound 3c

PMK-02-365
exp25 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 16 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	4
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1
bs	16	lb	1.00	4	1.5
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.0		
TRANSMITTER		rp	46.1		
tn	C13	lp	206.7		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1305.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	200		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

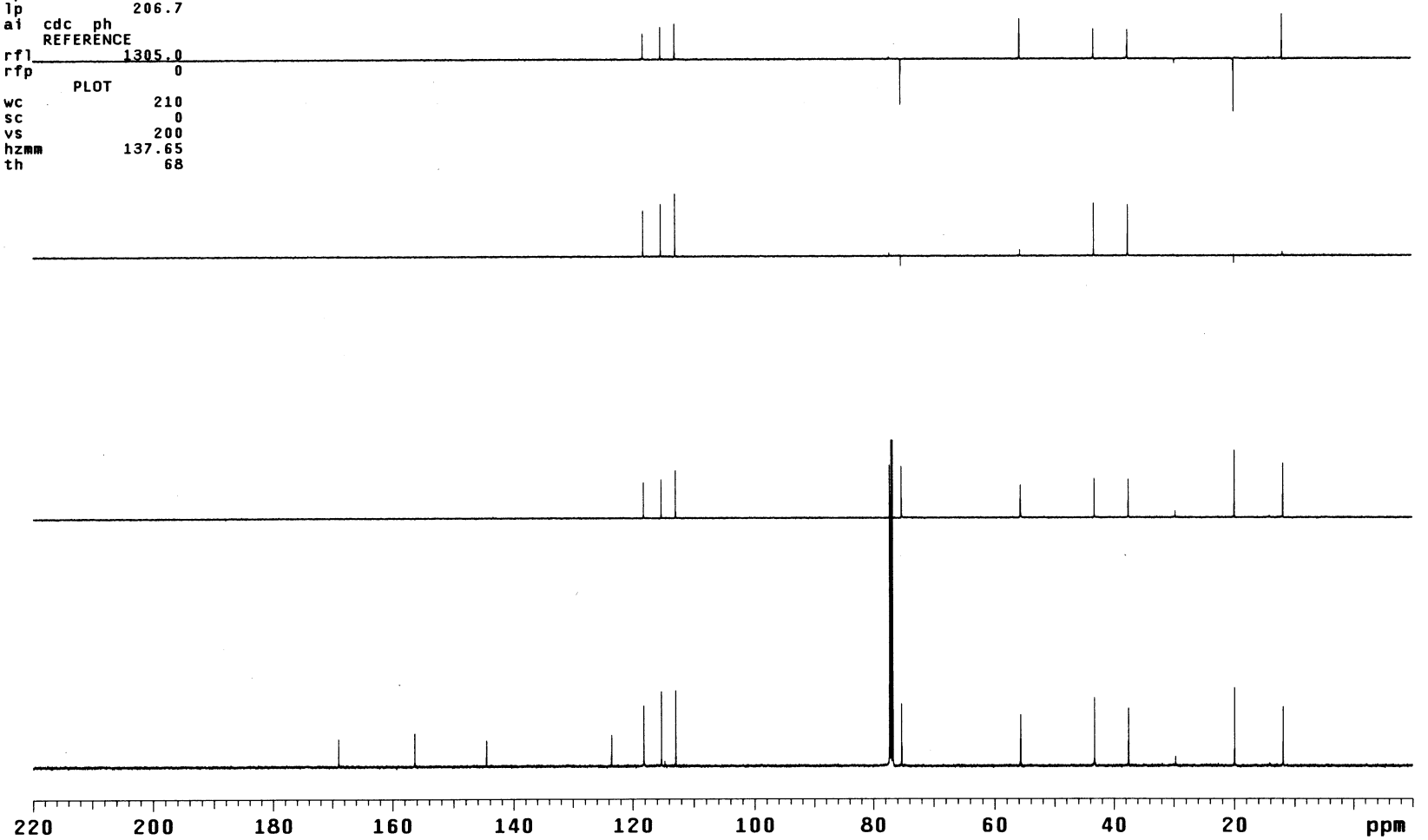


Fig S34. HSQC of compound 3c

PMK-02-365

exp28 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Apr 16 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4001.6	SPECIAL		1	1
at	0.128	temp	not used	2	2
np	1024	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.829	fn	1024		
tof	-499.9	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	tp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	353.8		
dmm	ccp	wp	3431.1		
dmf	32258	sp1	857.7		
dpwr	36	wp1	14585.8		
pwxlvl	52	rf1	1886.8		
pwx	14.300	rfp	1873.4		
HSQC		rfl1	8293.6		
j1xh	140.0	rfp1	7002.0		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	3		
		ai cdc ph			

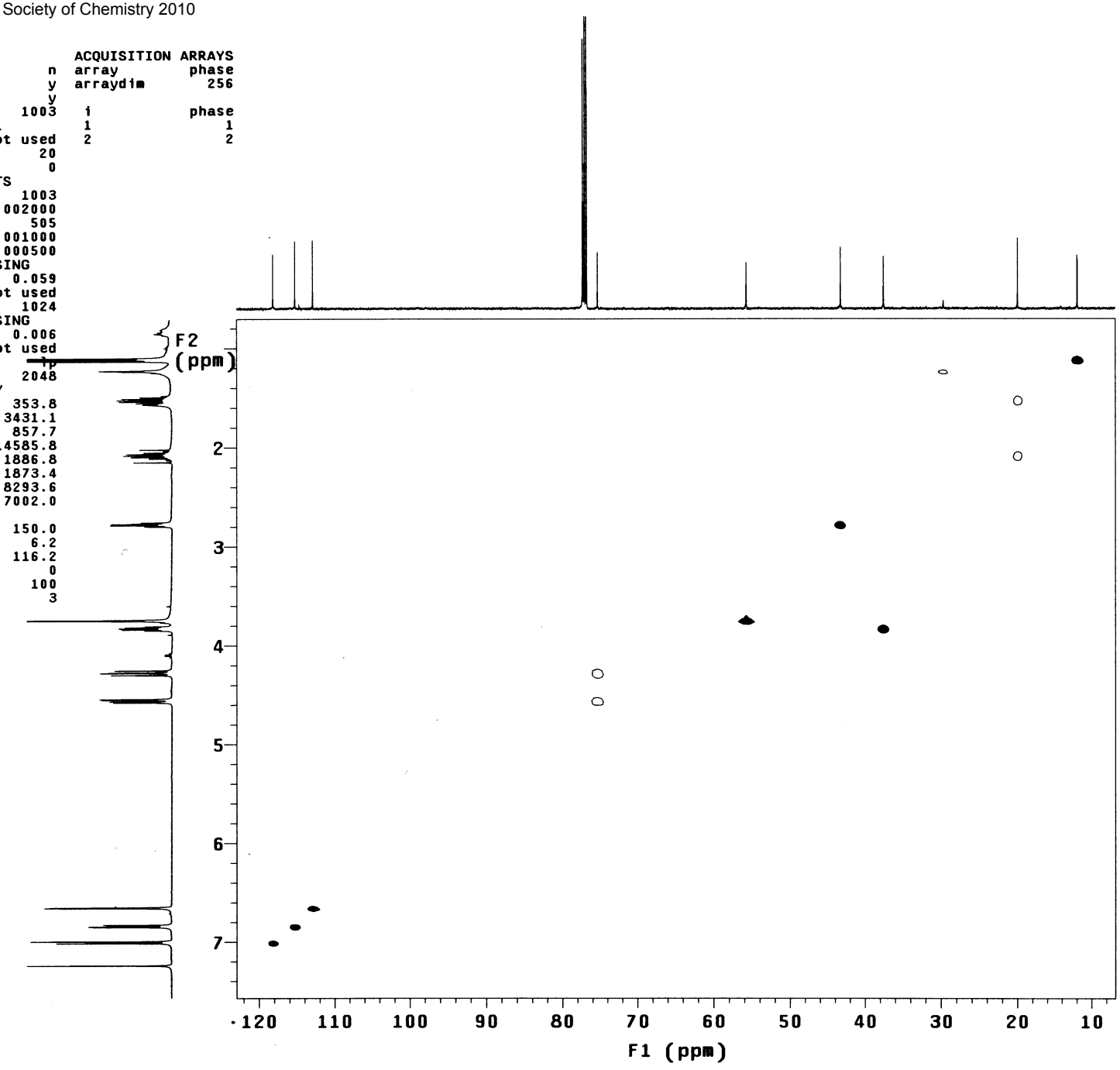


Fig S35. COSY of compound 3c

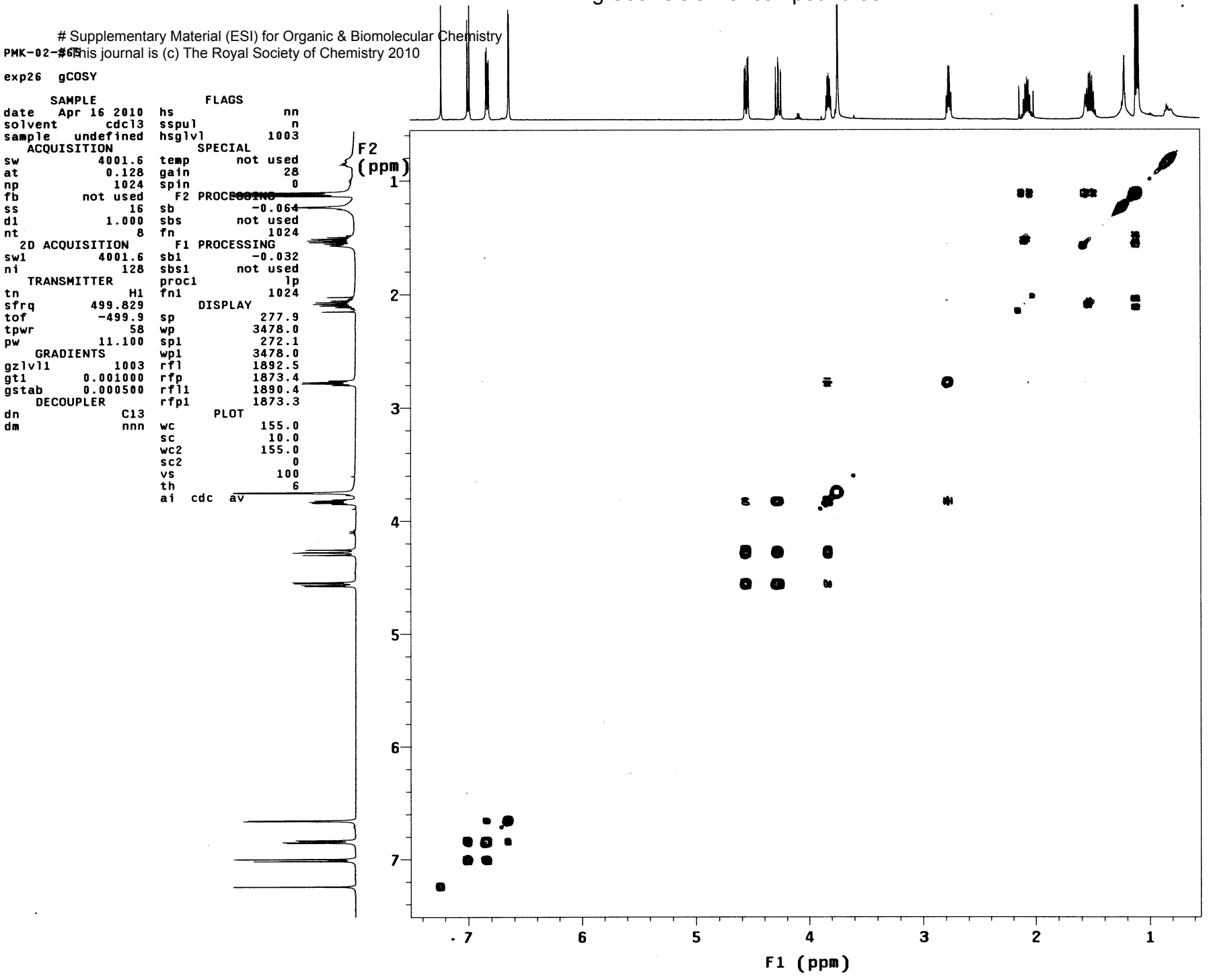


Fig S36. NOESY of compound 3c

PMK-02-365

exp27 NOESY

SAMPLE		FLAGS	
date	Apr 16 2010	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	4001.6	SPECIAL	
at	0.128	temp	not used
np	1024	gain	28
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.059
nt	8	gfs	not used
2D ACQUISITION		fn	1024
sw1	4001.6	F1 PROCESSING	
ni	200	gf1	0.046
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.829	fn1	1024
tof	-499.9	DISPLAY	
tpwr	58	sp	293.0
pw	11.100	wp	3423.2
NOESY		sp1	293.7
mix	0.600	wp1	3423.2
PRESATURATION		rf1	1893.0
satmode	nnnn	rfp	1873.4
satpwr	0	rf11	1892.3
satdly	0	rfp1	1873.3
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	100
		th	2
		ai	ph

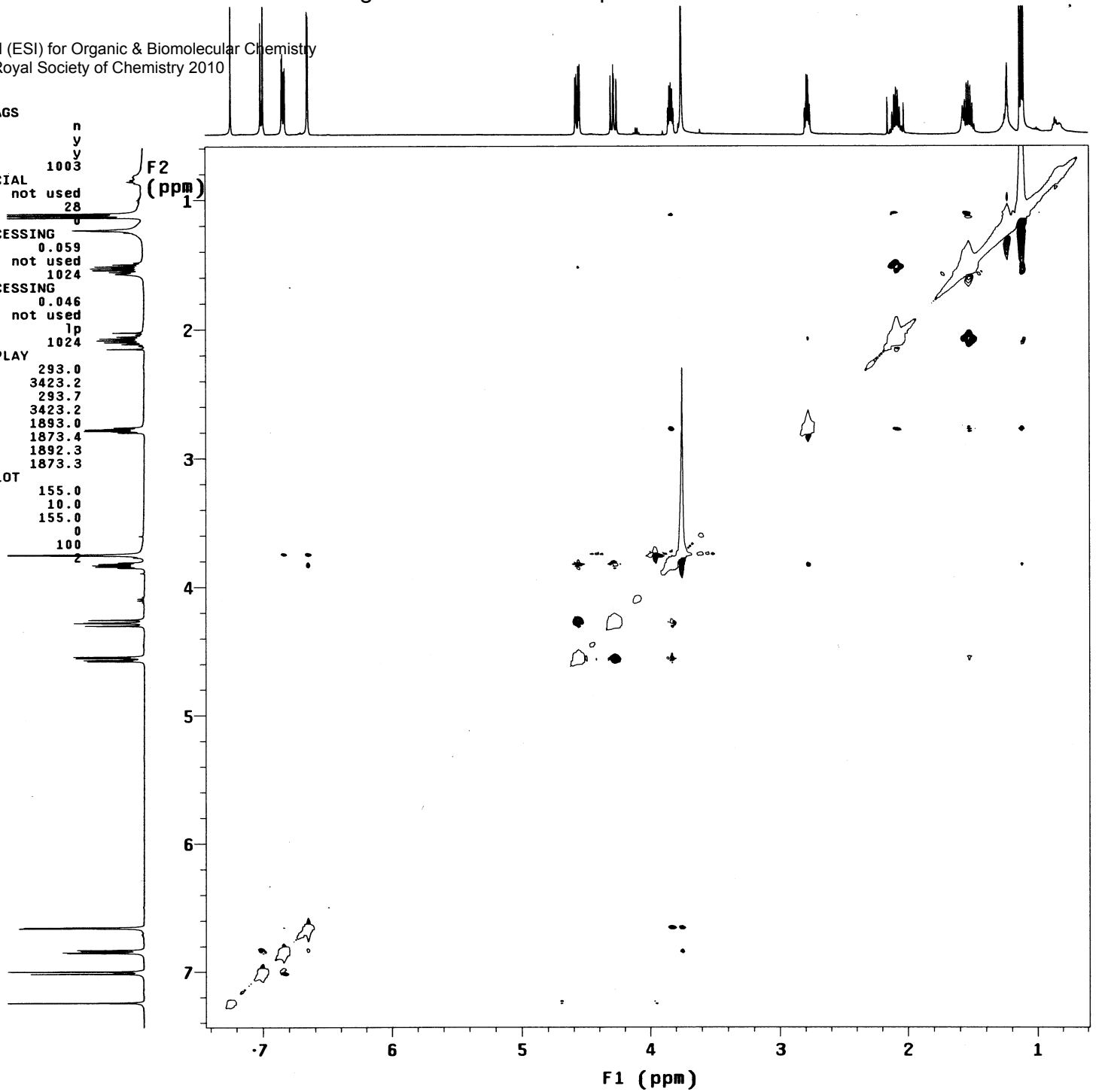
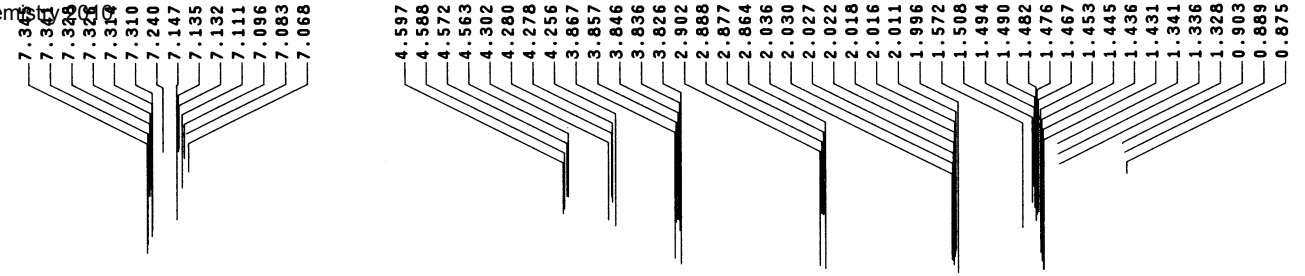


Fig S37. 1H NMR (CDCl3, 500 MHz) of compound 3d

PMK-02-368

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	125.693
solvent	cdcl3	dn	C13
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.830	dm	nnn
tn	H1	dmm	c
at	3.000	dmf	200
np	48000	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	temp	20.0
tpwr	58	PROCESSING	
pw	4.8	wtfile	
d1	1.000	proc	ft
tof	499.7	fn	not used
nt	4	math	f
ct	4		
alock	y	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5248.0		
vs	50		
sc	0		
wc	210		
hzmm	24.99		
is	252.84		
rfl	4637.7		
rfp	3618.7		
th	4		
ins	100.000		
nm	cdc ph		



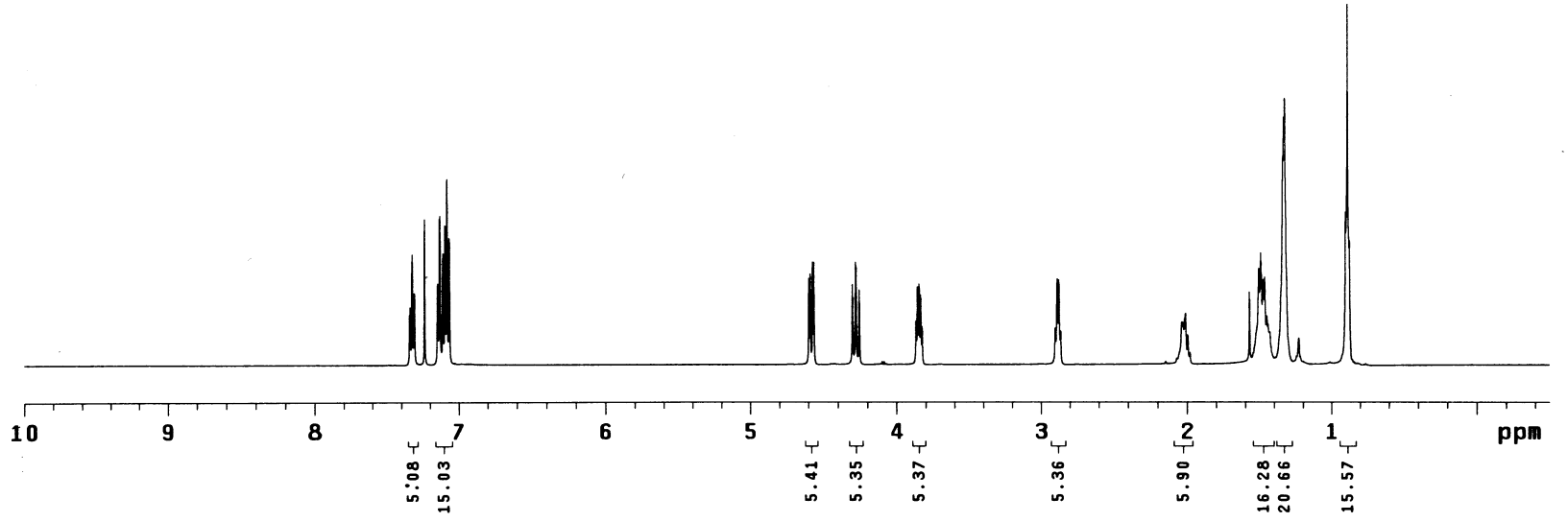


Fig S38. 13C NMR (CDCl3, 125 MHz) of compound 3d

PMK-02-368

exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.829
solvent	cdcl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	125.696	dm	yyy
tn	C13	dmm	w
at	1.000	dmf	11905
np	62894	dseq	
sw	31446.5	dres	1.0
fb	not used	homo	n
bs	16	temp	20.0
ss	2	PROCESSING	
tpwr	54	lb	1.00
pw	4.0	wtfile	
d1	1.000	proc	ft
tof	2512.2	fn	not used
nt	1024	math	f
ct	1024		
alock	y	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1257.0		
wp	28906.3		
vs	50		
sc	0		
wc	210		
hzmm	137.65		
is	500.00		
rfl	10984.4		
rfp	9677.5		
th	7		
ins	100.000		
nm cdc ph			

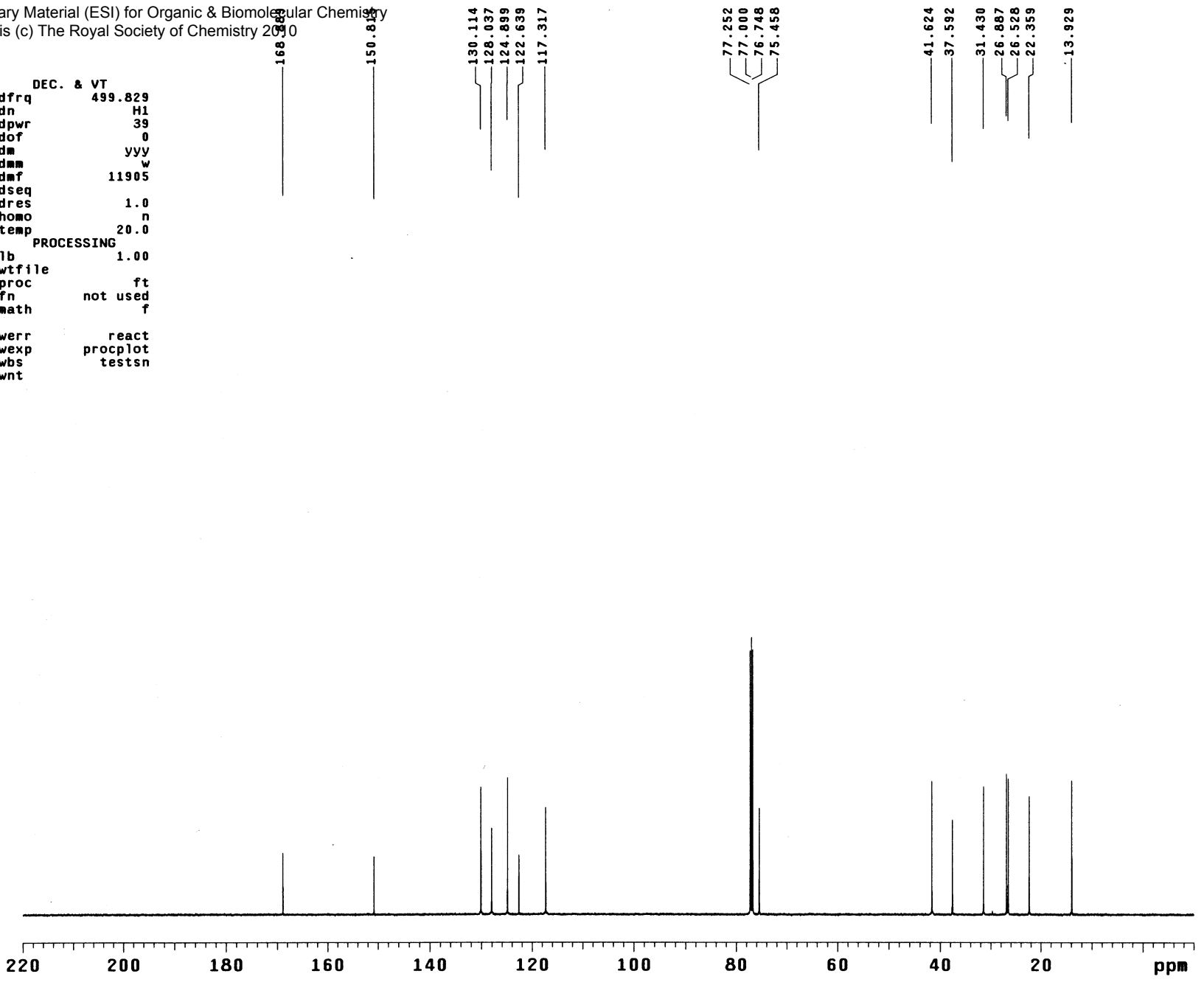


Fig S39. DEPT of compound 3d

Supplementary Material (ESI) for Organic & Biomolecular Chemistry

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 28 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	20.0	i	mult
sw	31446.5	gain	22	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.0		
TRANSMITTER		rp	46.8		
tn	C13	lp	187.2		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1306.9		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	1000		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

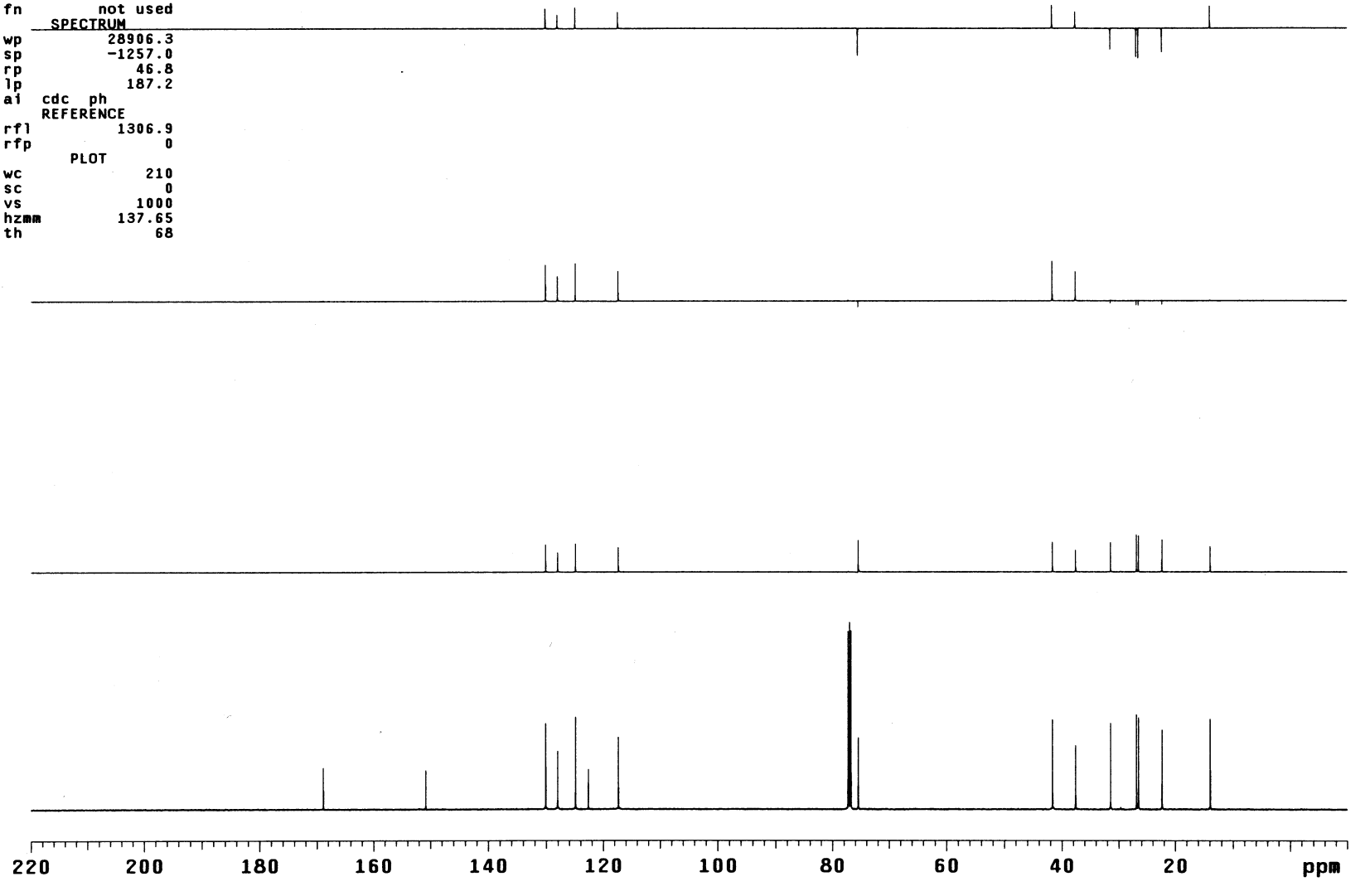


Fig S40. HSQC of compound 3d

exp8 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Apr 28 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	20.0	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	16	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	214.5		
dmm	ccp	wp	3696.6		
dmf	32258	sp1	1102.5		
dpwr	36	wp1	15817.0		
pwxlvl	52	rfl	1935.8		
pwx	14.300	rfp	1922.3		
HSQC		rfl1	6021.8		
j1xh	140.0	rfp1	4724.6		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	4		
		ai cdc ph			

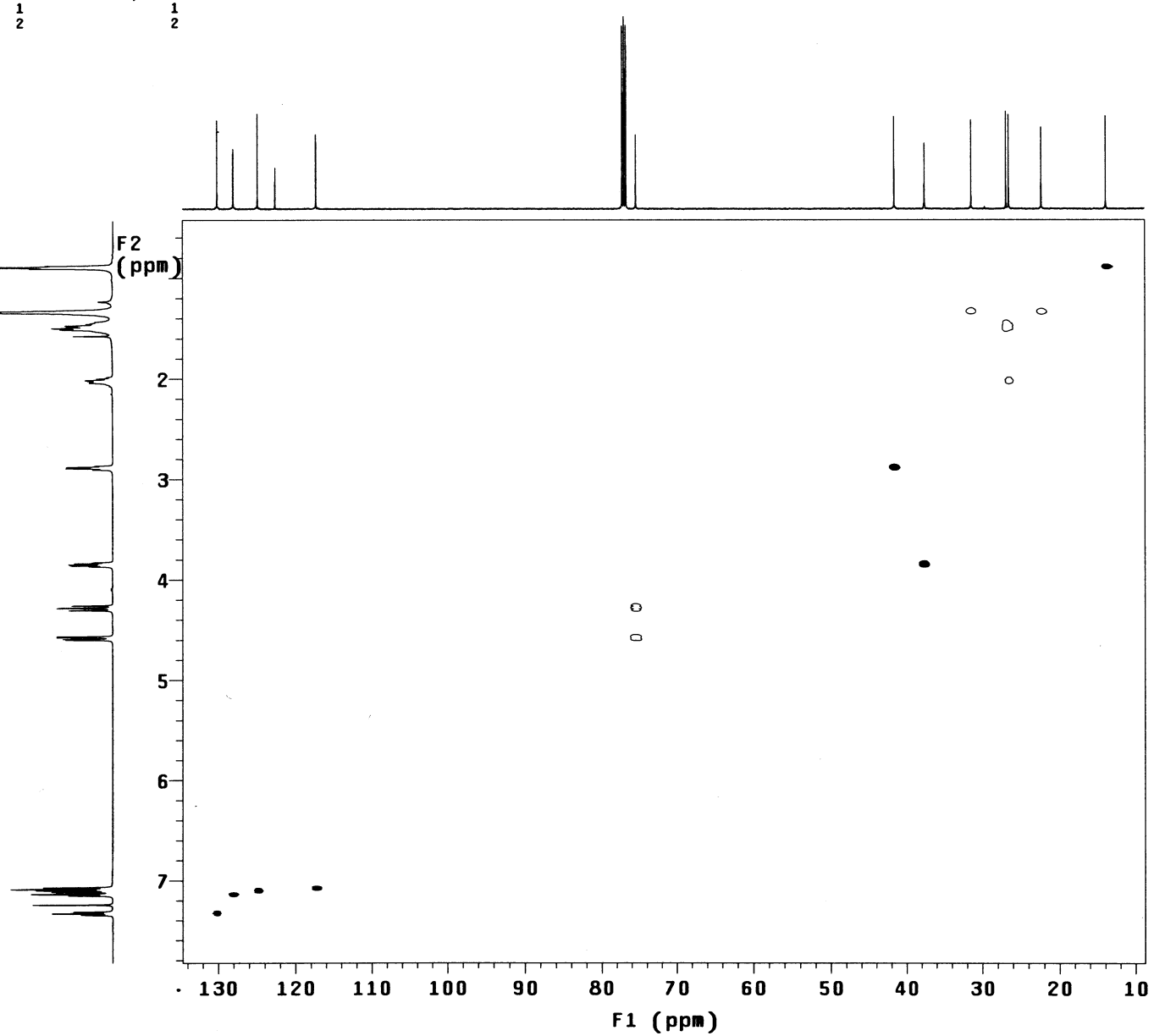


Fig S41. COSY of compound 3d

PMK-02-368

exp6 gCOSY

SAMPLE		FLAGS	
date	Apr 28 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4490.3	temp	20.0
at	0.228	gain	22
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.114
d1	1.000	sbs	not used
nt	8	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	4490.3	sb1	-0.029
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-250.0	sp	265.8
tpwr	58	wp	3587.0
pw	11.100	sp1	271.4
GRADIENTS		wp1	3582.6
gzlvl1	1003	rfl	1937.2
gt1	0.001000	rfp	1922.3
gstab	0.000500	rfl1	1935.9
DECOUPLER		rfp1	1922.3
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	9
		ai cdc av	

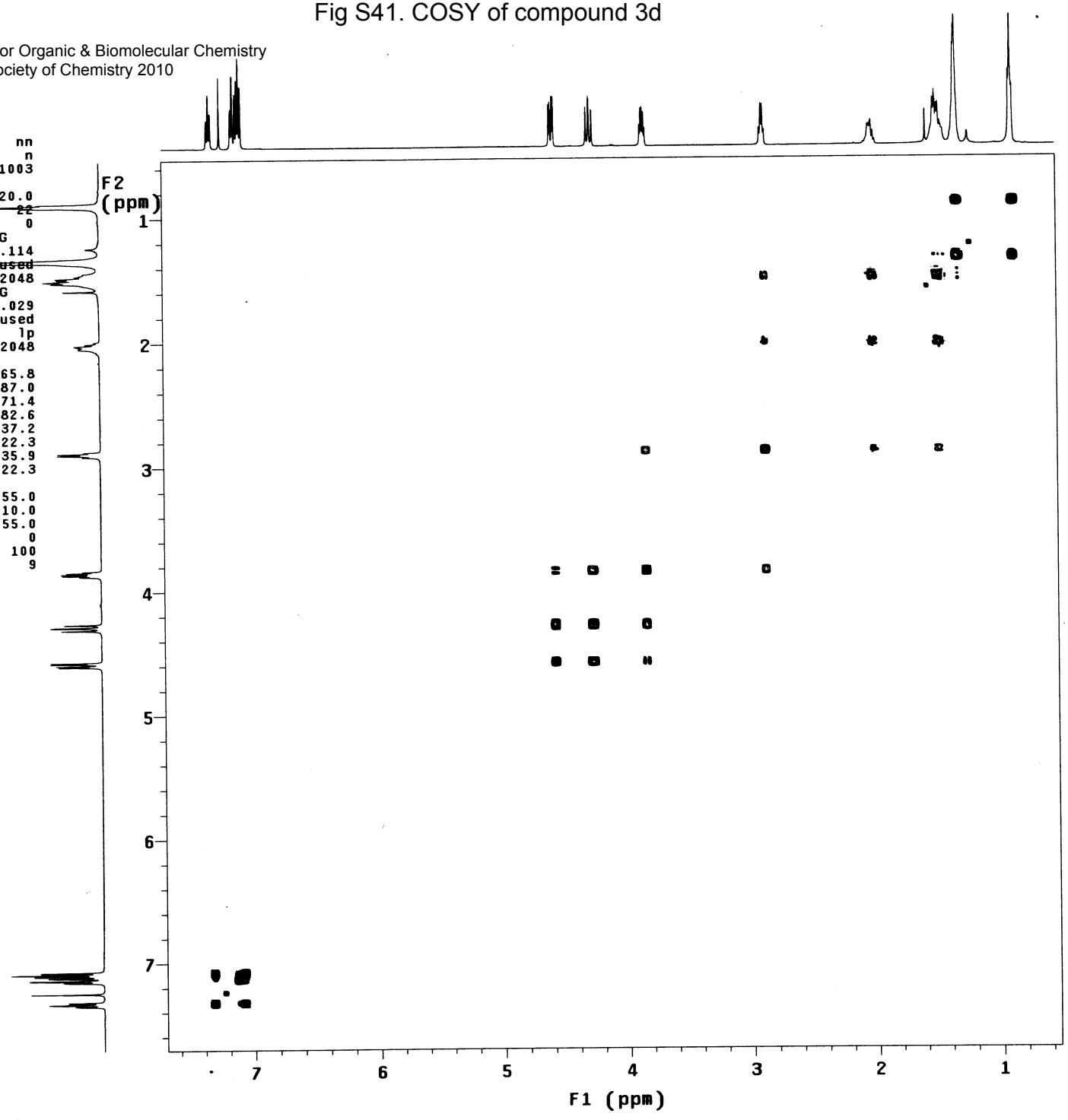


Fig S42. NOESY of compound 3d

PMK-02-368

exp7 NOESY

SAMPLE	
date	Apr 28 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	32
d1	1.000
nt	16
2D ACQUISITION	
sw1	4490.3
ni	200
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	20.0
gain	22
spin	0
F2 PROCESSING	
gf	0.185
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.041
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	267.0
wp	3556.3
sp1	272.3
wp1	3556.3
rfl	1935.9
rfp	1922.3
rfl1	1935.0
rfp1	1922.3
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	1
ai	ph

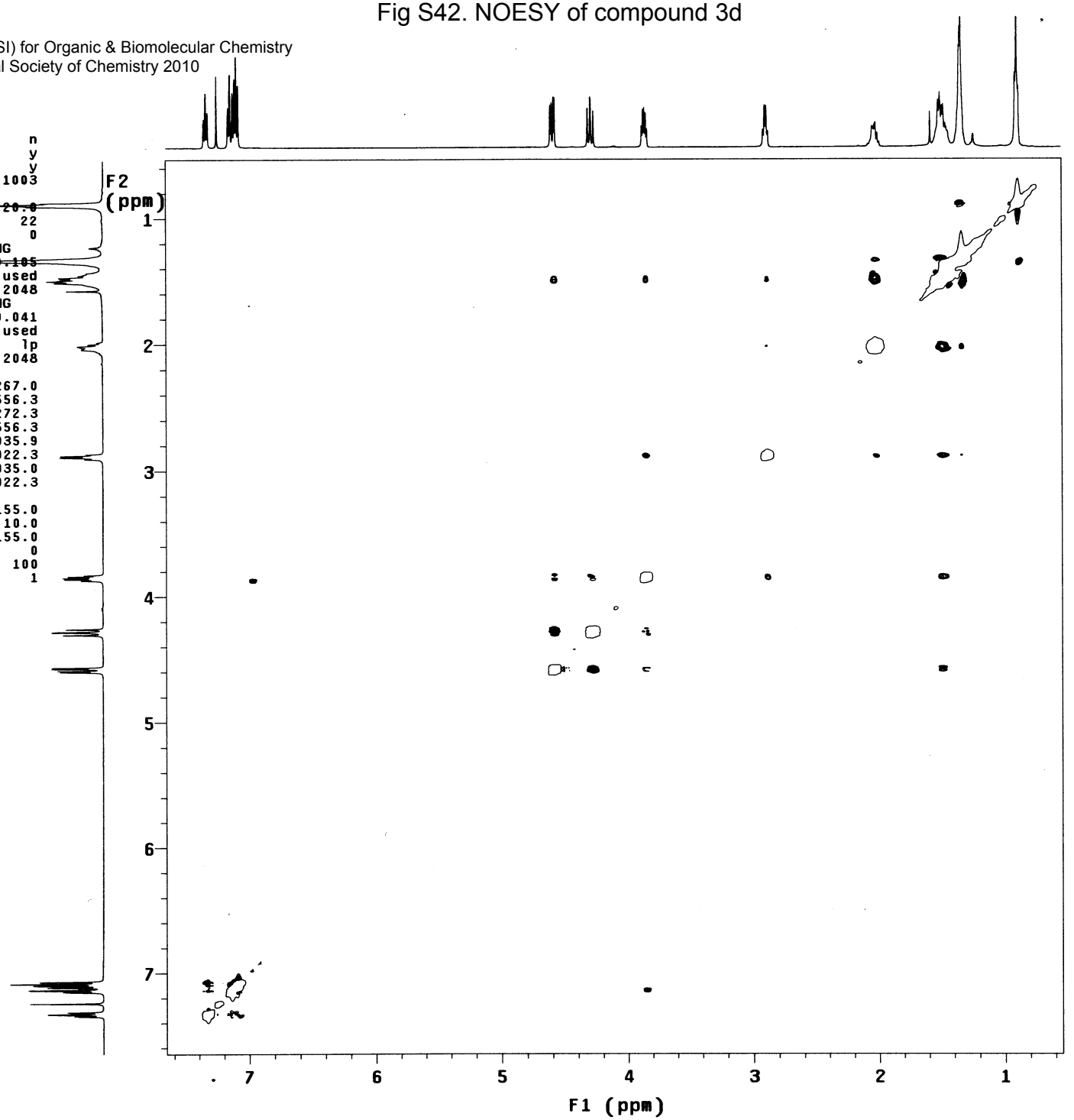
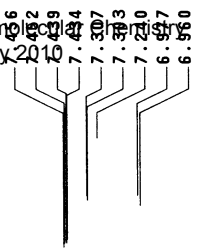
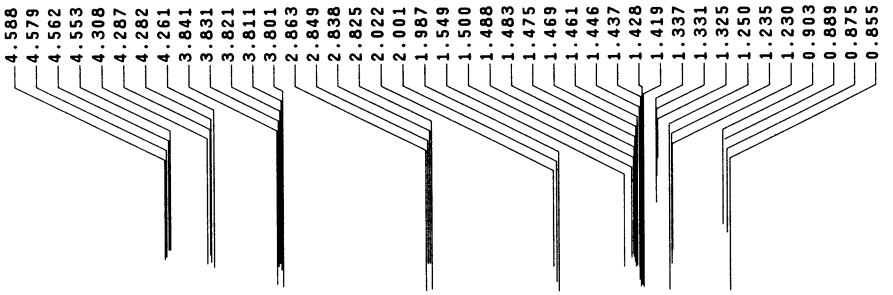


Fig S43. 1H NMR (CDCl3, 500 MHz) of compound cis-3e


```
PMK-02-371-f1
exp12  s2pul
      SAMPLE                DEC. & VT
date    May  7 2010   dfrq       125.693
solvent       cdcl3   dn             C13
file            exp   dpwr            30
   ACQUISITION        dof              0
sfrq        499.830   dm             nnn
tn               H1   dmm              c
at            3.000   dmf            200
np            48000   dseq
sw           8000.0   dres           1.0
fb         not used   homo             n
bs                4        PROCESSING
tpwr             58   wtfile
pw              4.8   proc            ft
d1            1.000   fn       not used
tof           499.7   math             f
nt                4
ct                4   werr         react
alock             y   wexp      procplot
gain       not used   wbs
      FLAGS           wnt            wft
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -250.1
wp           5248.0
vs               75
sc                0
wc              210
hzmm          24.99
is           345.52
rfl          4637.9
rfp          3618.7
th                2
ins          100.000
ai  cdc  ph
```





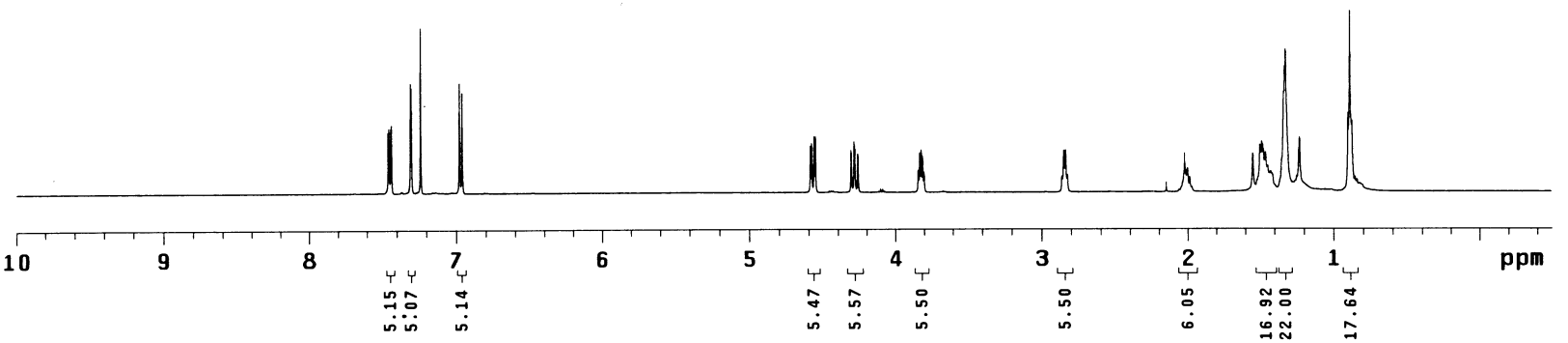


Fig S44. 13C NMR (CDCl3, 125 MHz) of compound cis-3e

PMK-02-371-f1
exp13 s2pul

SAMPLE		DEC. & VT	
date	May 7 2010	dfrq	499.829
solvent	cdcl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	125.696	dm	yyy
tn	C13	dmm	w
at	1.000	dmf	11905
np	62894	dseq	
sw	31446.5	dres	1.0
fb	not used	homo	n
bs	16	PROCESSING	
ss	2	lb	1.00
tpwr	54	wtfile	
pw	4.0	proc	ft
d1	1.000	fn	not used
tof	2512.2	math	f
nt	1024		
ct	1024	werr	react
alock	y	wexp	procplot
gain	not used	wbs	testsn
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1257.0		
wp	28906.3		
vs	100		
sc	0		
wc	210		
hzmm	137.65		
is	500.00		
rfl	10981.5		
rfp	9677.5		
th	4		
ins	100.000		
nm	cdc ph		

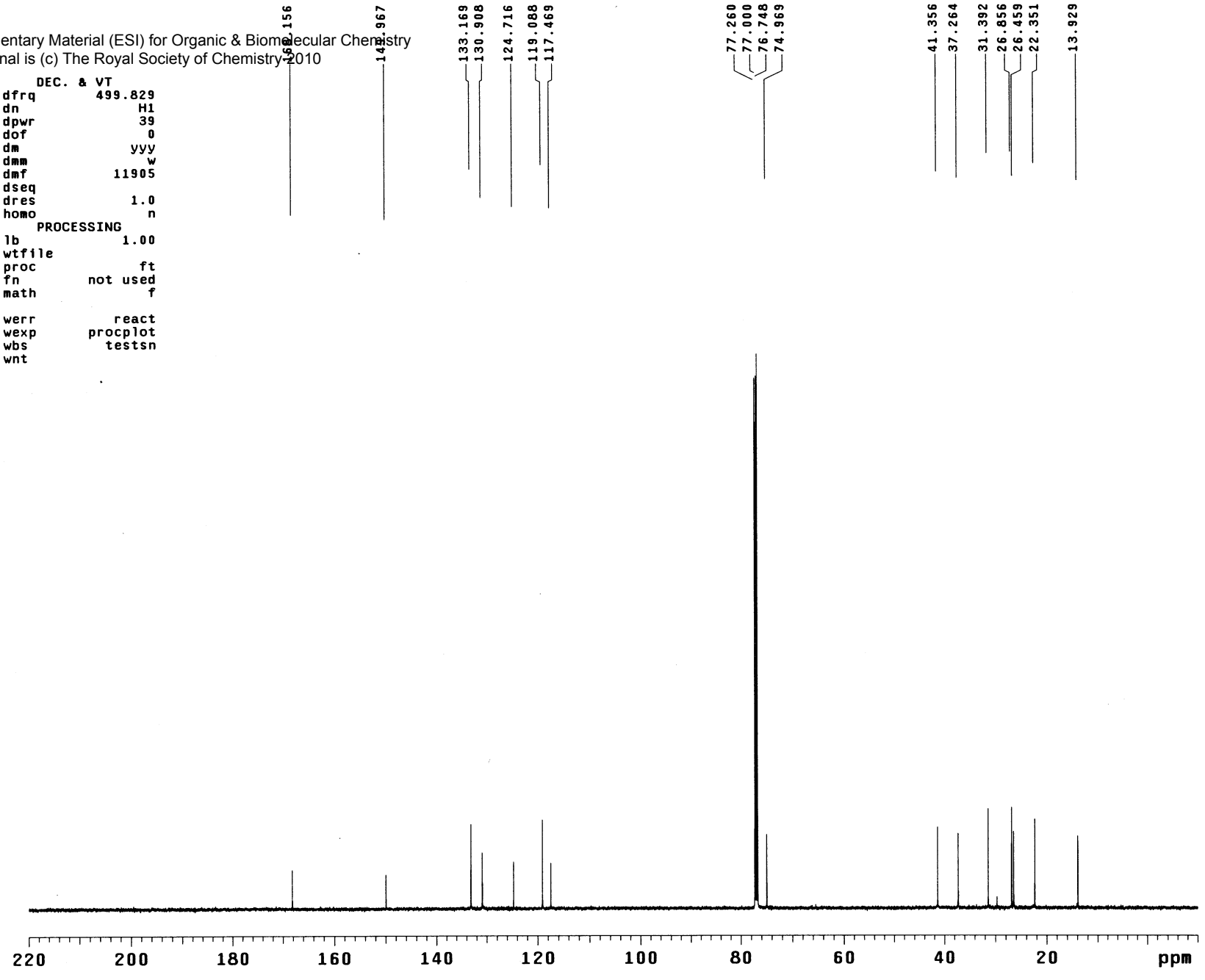


Fig S45. DEPT of compound cis-3e

PMK-02-371-T1

exp14 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	May 7 2010	jlxh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.0		
TRANSMITTER		rp	131.6		
tn	C13	lp	192.7		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1305.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	150		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

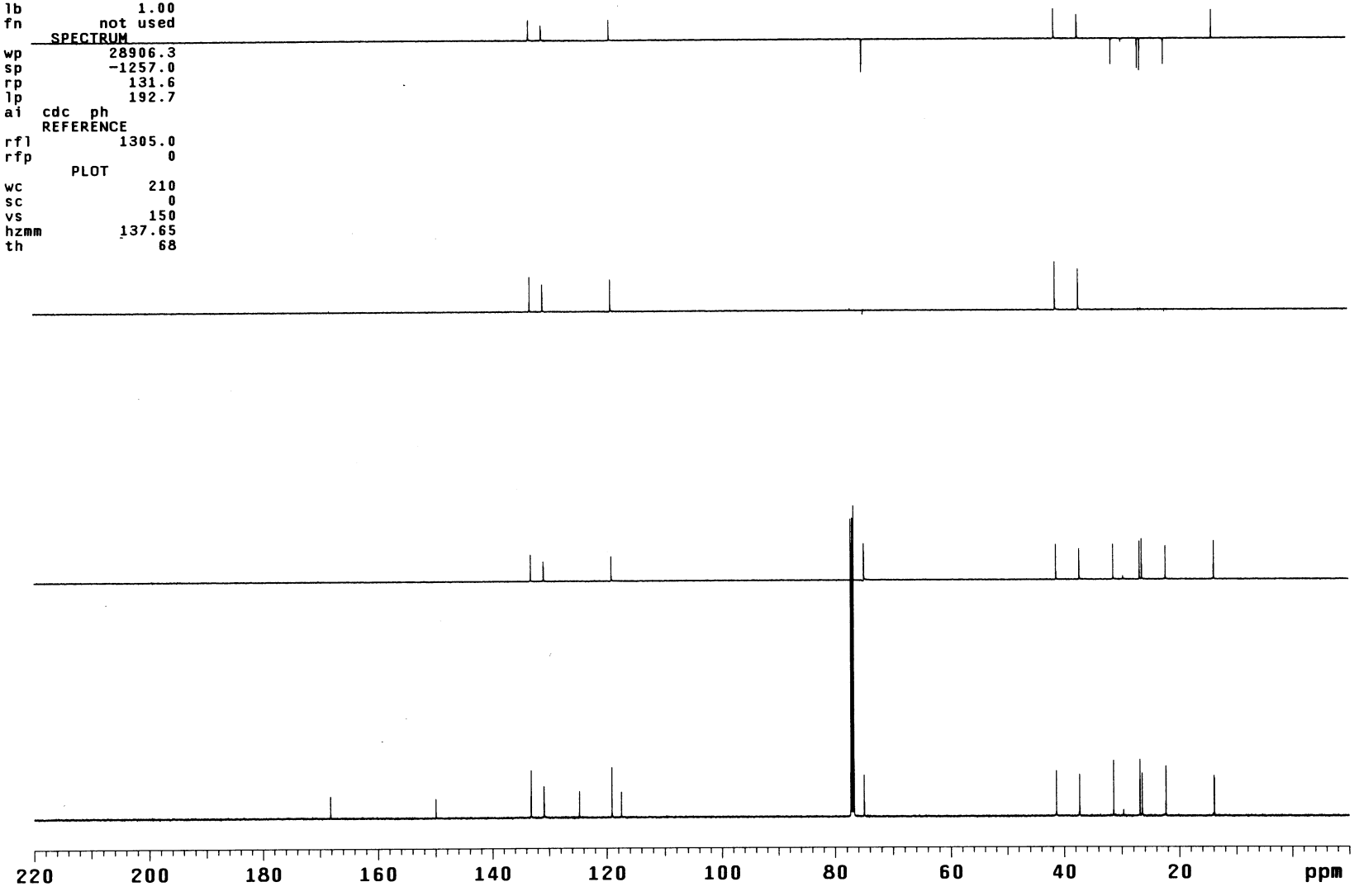


Fig S46. HSQC of compound cis-3e

PMK-02-371-f1
exp17 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	May 7 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	not used	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	296.7		
dmm	ccp	wp	3595.8		
dmf	32258	sp1	1233.0		
dpwr	36	wp1	15963.0		
pwxlvl	52	rfl	2156.4		
pwx	14.300	rfp	2141.8		
HSQC		rfl1	10714.1		
j1xh	140.0	rfp1	9422.2		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	6		
		ai cdc ph			

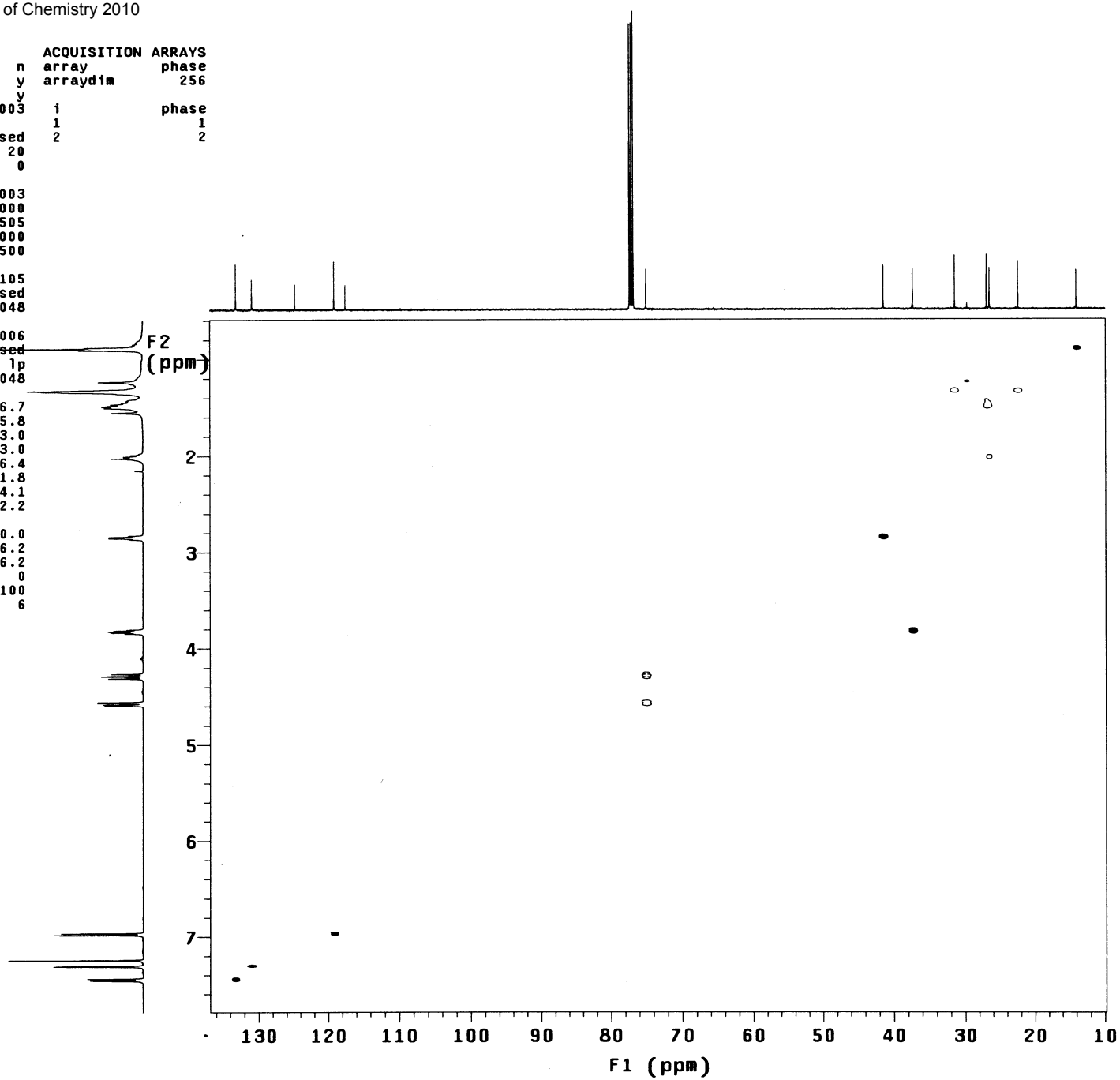


Fig S47. COSY of compound cis-3e

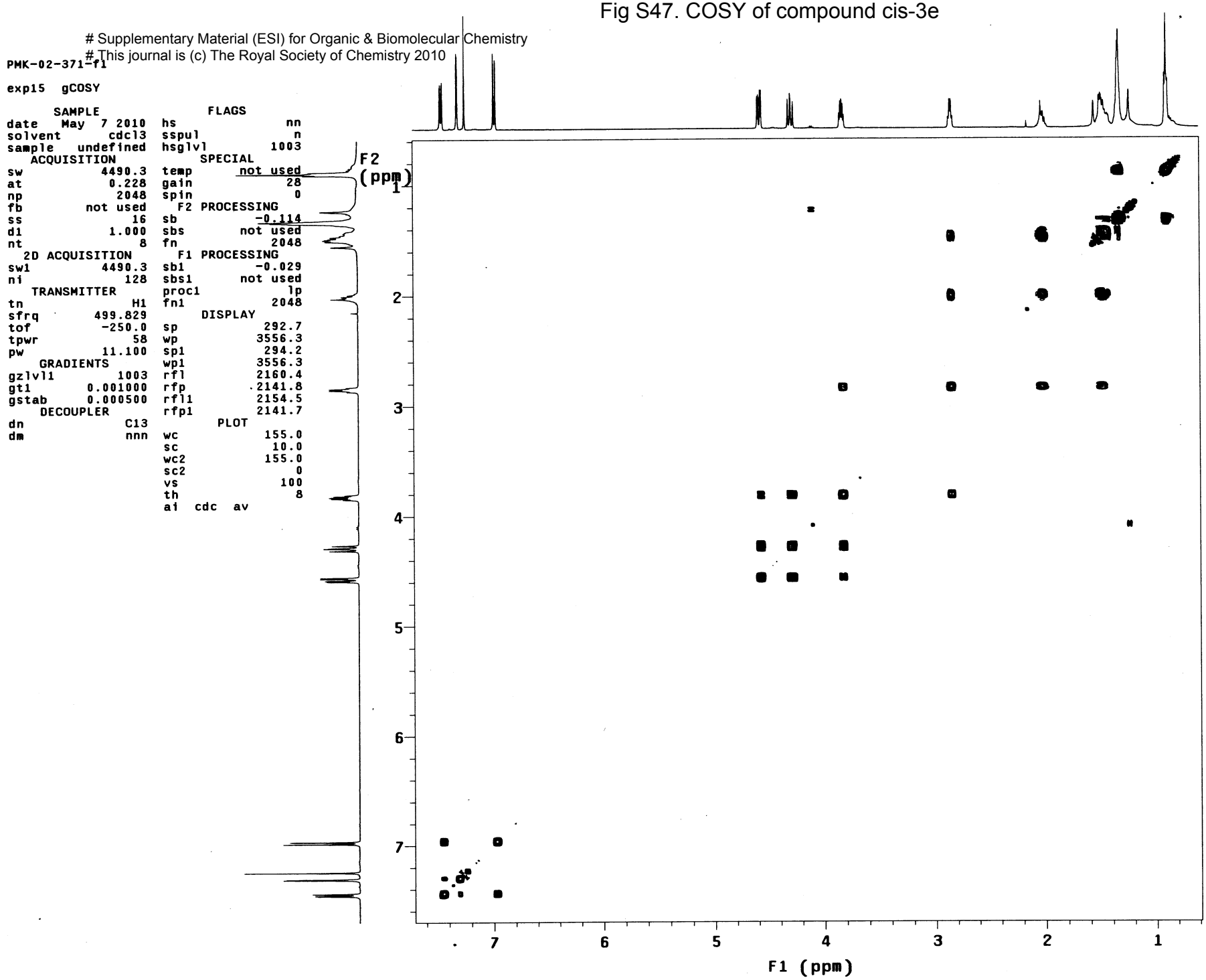


Fig S48. NOESY of compound cis-3e

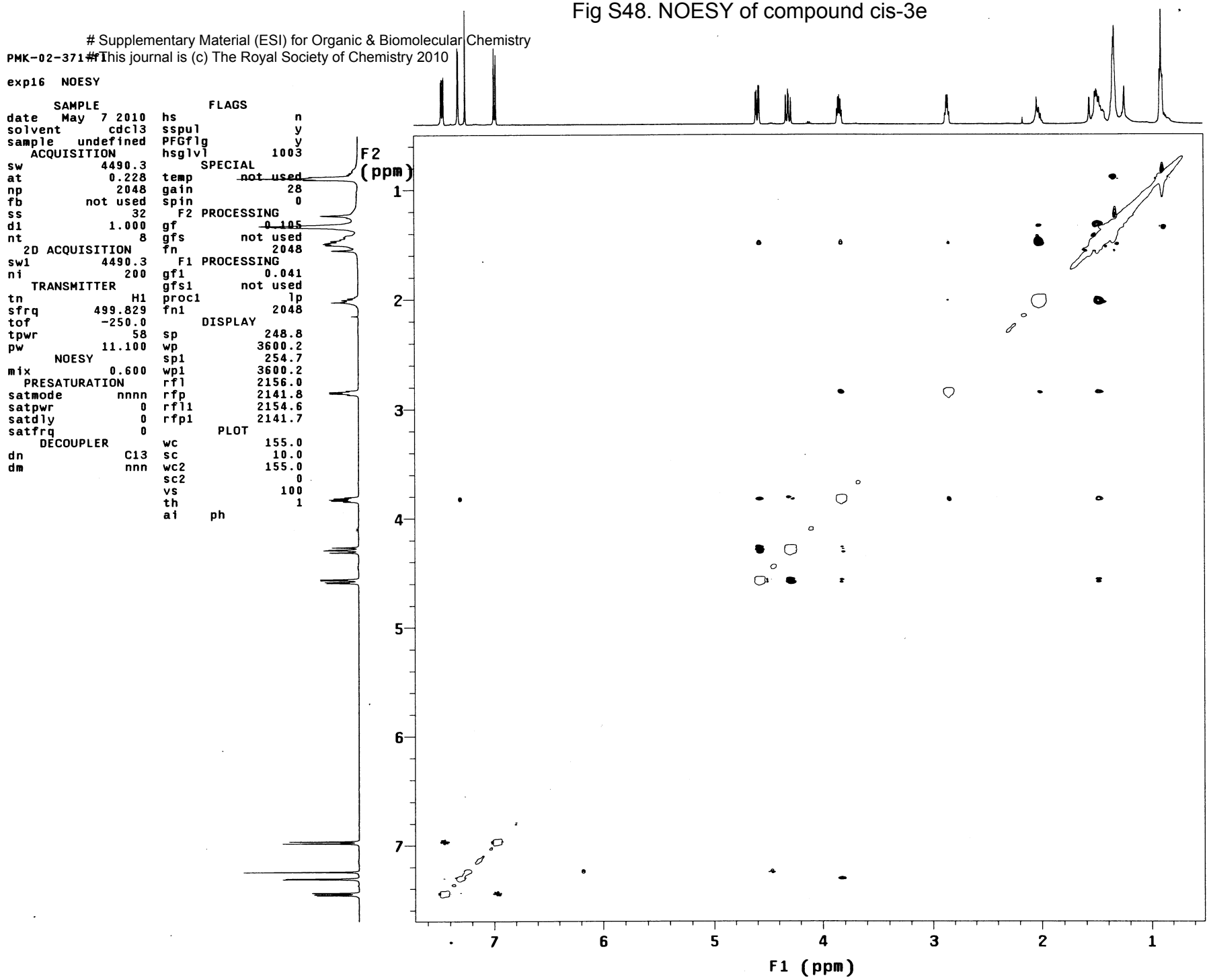
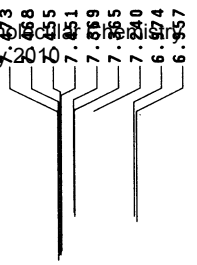
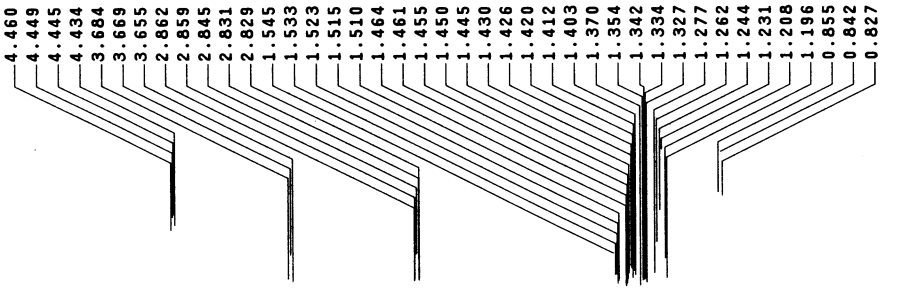


Fig S49. 1H NMR (CDCl3, 500 MHz) of compound trans-3e


```
PMK-02-371-f2
exp43  s2pul
      SAMPLE                DEC. & VT
date    May  6 2010   dfrq        125.693
solvent       cdcl3   dn              C13
file            exp   dpwr             30
   ACQUISITION        dof               0
sfrq        499.830   dm              nnn
tn               H1   dmm               c
at            3.000   dmf             200
np            48000   dseq
sw           8000.0   dres            1.0
fb         not used   homo              n
bs                4       PROCESSING
tpwr             58   wtfile
pw              4.8   proc              ft
d1            1.000   fn         not used
tof           499.7   math              f
nt                4
ct                4   werr          react
alock             y   wexp       procplot
gain       not used   wbs
      FLAGS           wnt             wft
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -250.1
wp           5248.0
vs               75
sc                0
wc              210
hzmm          24.99
is           258.08
rfl          4637.9
rfp          3618.7
th                3
ins          100.000
ai  cdc  ph
```





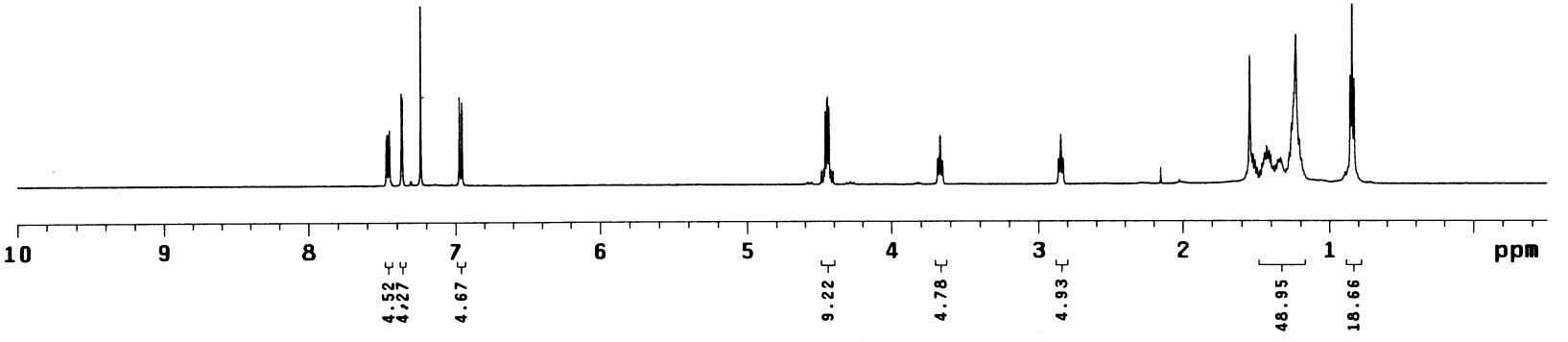


Fig S50. 13C NMR (CDCl3, 125 MHz) of compound trans-3e

PMK-02-371-T2
exp44 s2pul

SAMPLE		DEC. & VT	
date	May 6 2010	dfrq	499.829
solvent	cdcl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	125.696	dm	yyy
tn	C13	dmm	w
at	1.000	dmf	11905
np	62894	dseq	
sw	31446.5	dres	1.0
fb	not used	homo	n
bs	16	PROCESSING	
ss	2	lb	1.00
tpwr	54	wtfile	
pw	4.0	proc	ft
d1	1.000	fn	not used
tof	2512.2	math	f
nt	2048		
ct	2048	werr	react
alock	y	wexp	procplot
gain	not used	wbs	testsn
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1257.0		
wp	28906.3		
vs	200		
sc	0		
wc	210		
hzmm	137.65		
is	500.00		
rfl	10981.5		
rfp	9677.5		
th	6		
ins	100.000		
nm cdc ph			

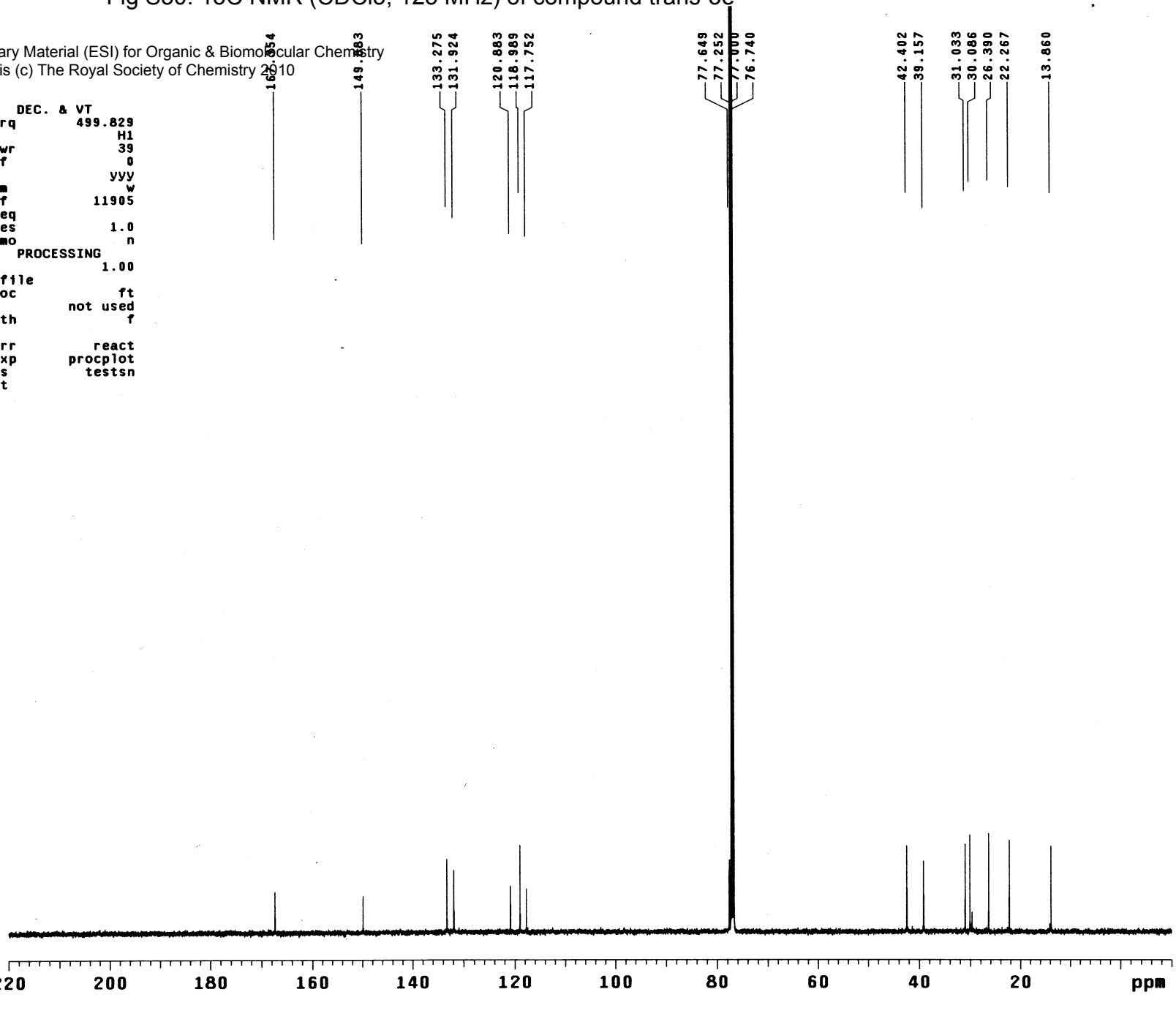


Fig S51. DEPT of compound trans-3e

PMK-02-371-f2

exp45 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	May 6 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	30	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	28906.3		
ct	2048	sp	-1257.0		
TRANSMITTER		rp	134.6		
tn	C13	lp	186.7		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1304.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	7500		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

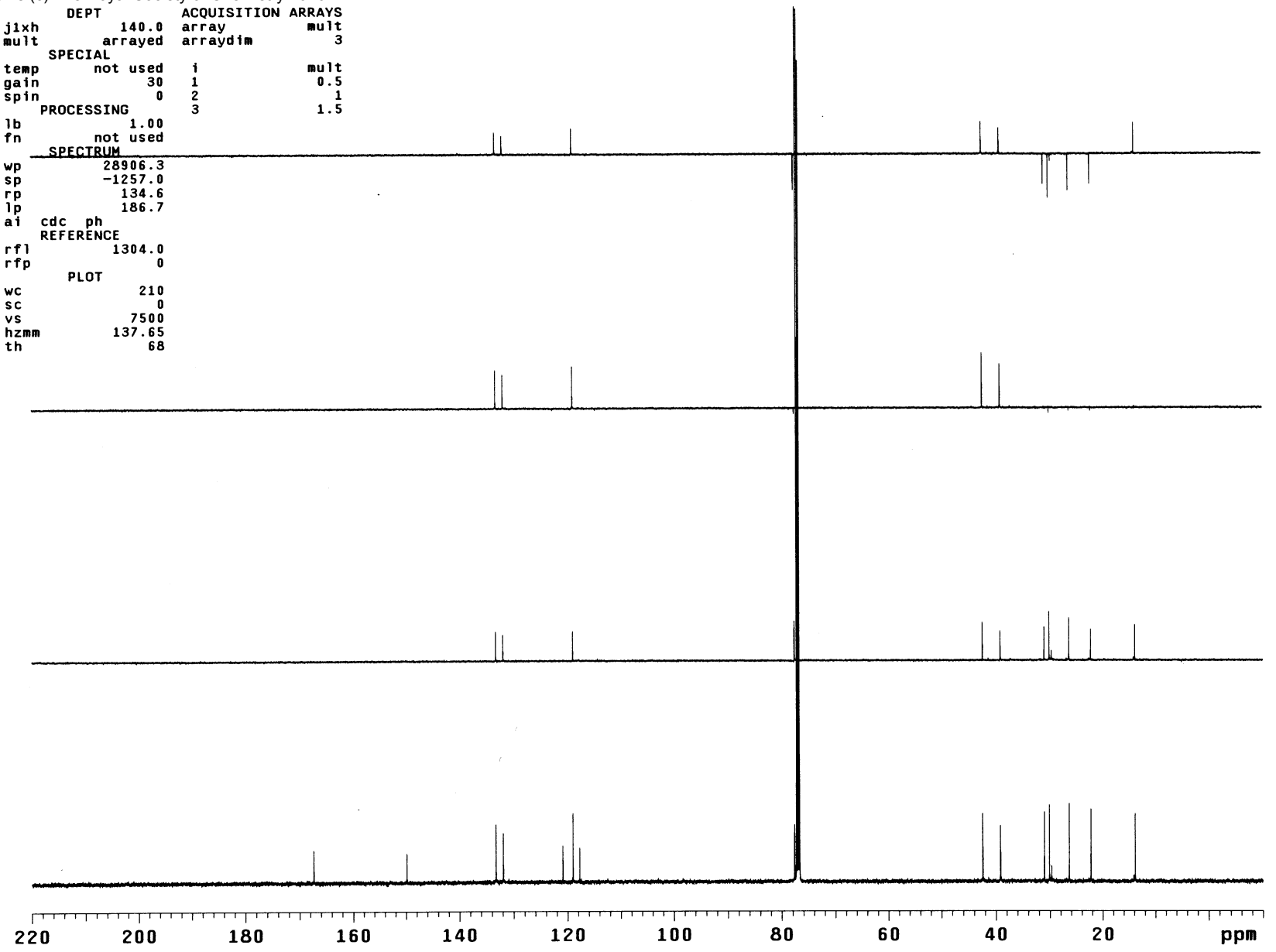


Fig S52. HSQC of compound trans-3e

exp48 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	May 6 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	not used	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	16	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	193.9		
dmm	ccp	wp	3762.4		
dmf	32258	sp1	1053.0		
dpwr	36	wp1	16526.4		
pwxlvl	52	rfl	1850.5		
pwx	14.300	rfp	1833.9		
HSQC		rfl1	6205.4		
j1xh	140.0	rfp1	4921.3		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	5		
		ai cdc ph			

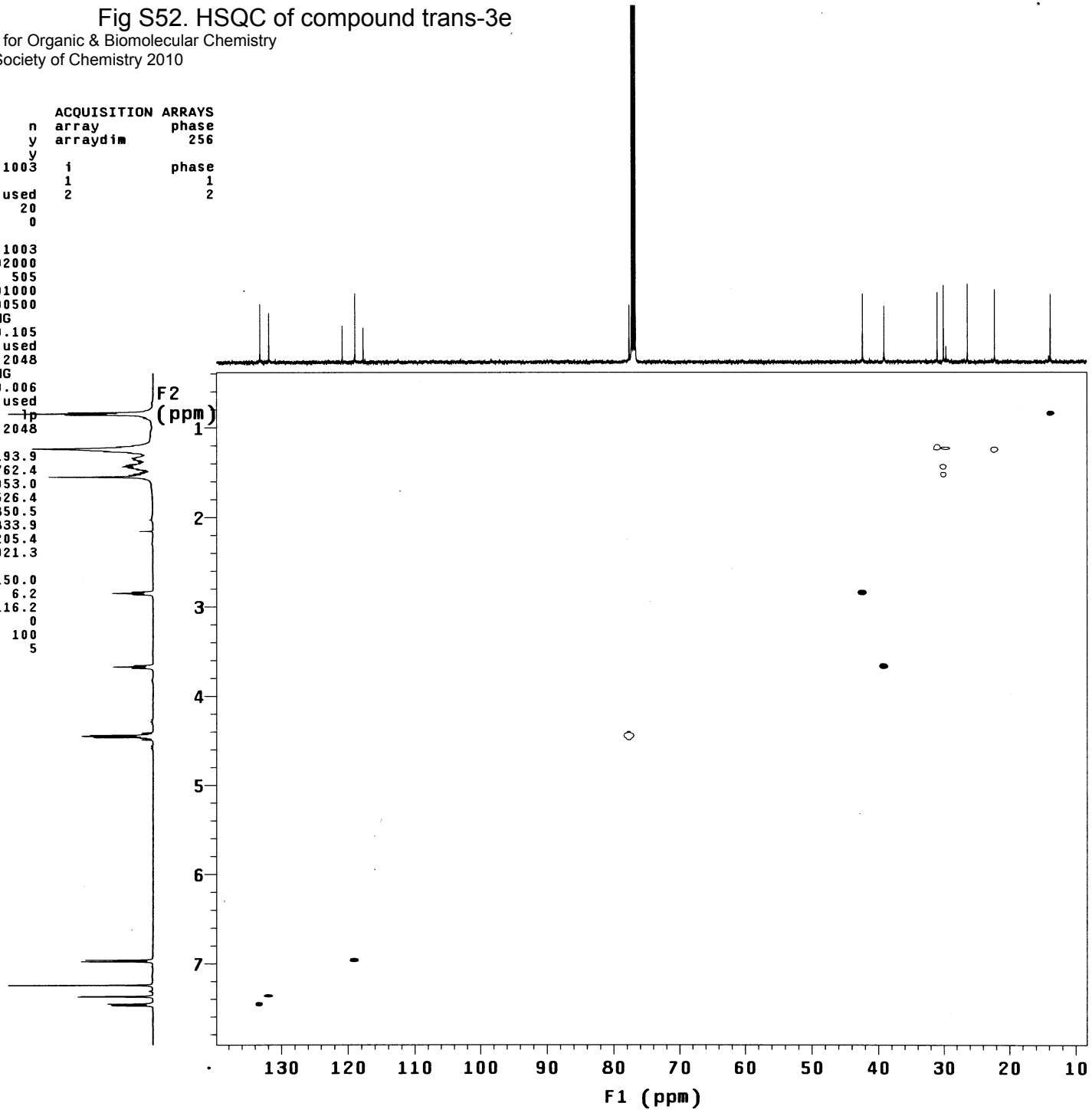


Fig S53. COSY of compound trans-3e

PMK-02-37#1 rs

exp46 gCOSY

SAMPLE		FLAGS	
date	May 6 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4490.3	temp	not used
at	0.228	gain	30
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	0.114
d1	1.000	sbs	not used
nt	16	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	4490.3	sb1	-0.029
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-250.0	sp	265.4
tpwr	58	wp	3630.9
pw	11.100	sp1	262.7
GRADIENTS		wp1	3630.9
gzlvl1	1003	rfl	1849.1
gt1	0.001000	rfp	1833.9
gstab	0.000500	rfl1	1847.4
DECOUPLER		rfp1	1833.9
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	10
		ai cdc av	

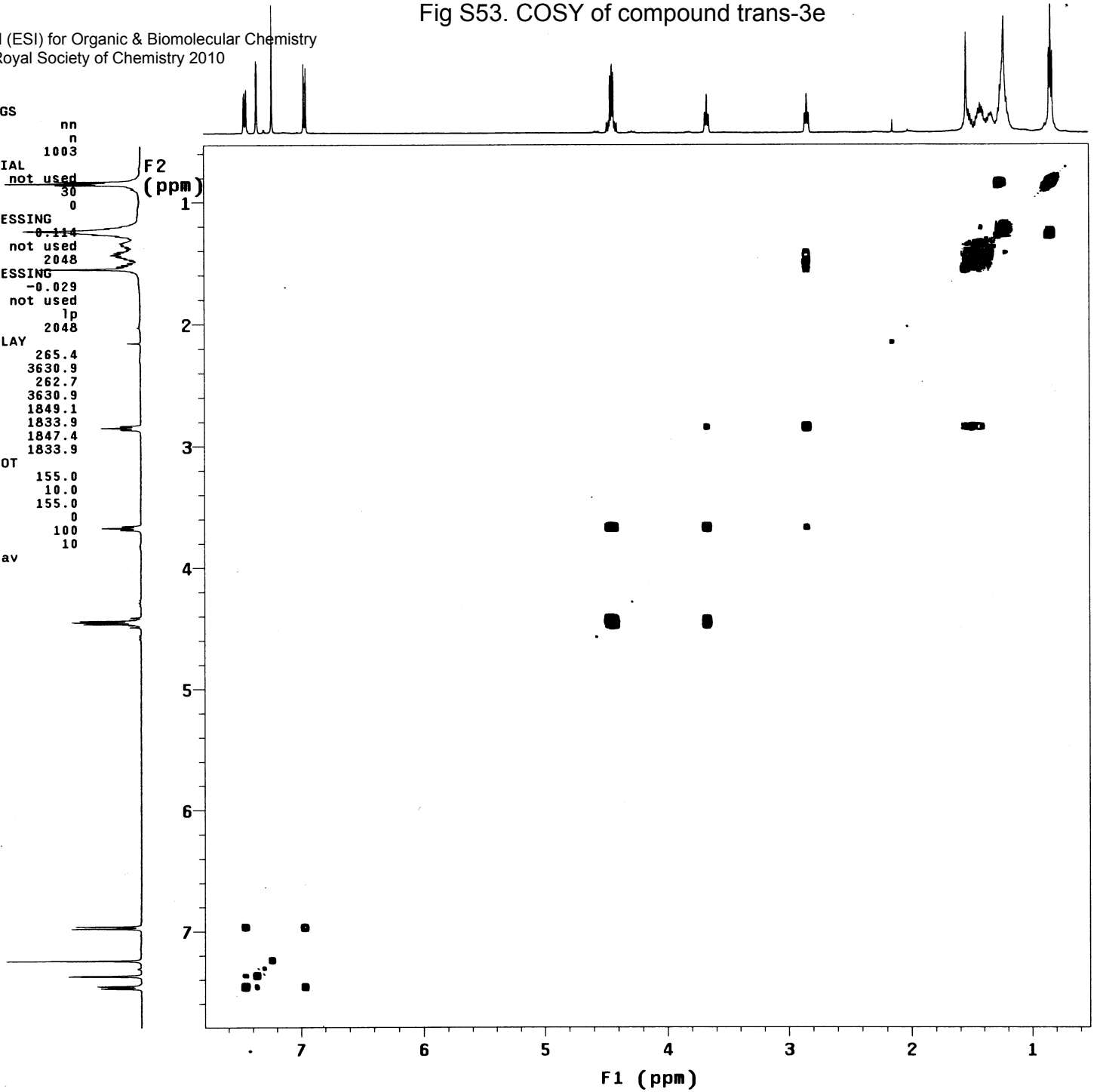


Fig S54. NOESY of compound trans-3e

PMK-02-371-f2
exp47 NOESY

SAMPLE		FLAGS	
date	May 6 2010	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	4490.3	SPECIAL	
at	0.228	temp	not used
np	2048	gain	30
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.105
nt	16	gfs	not used
2D ACQUISITION		fn	2048
sw1	4490.3	F1 PROCESSING	
ni	200	gf1	0.041
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.829	fn1	2048
tof	-250.0	DISPLAY	
tpwr	58	sp	248.2
pw	11.100	wp	3600.2
NOESY		sp1	253.2
mix	0.600	wp1	3600.2
PRESATURATION		rf1	1848.8
satmode	nnnn	rfp	1833.9
satpwr	0	rfl1	1848.1
satdly	0	rfp1	1833.9
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	100
		th	1
		ai	ph

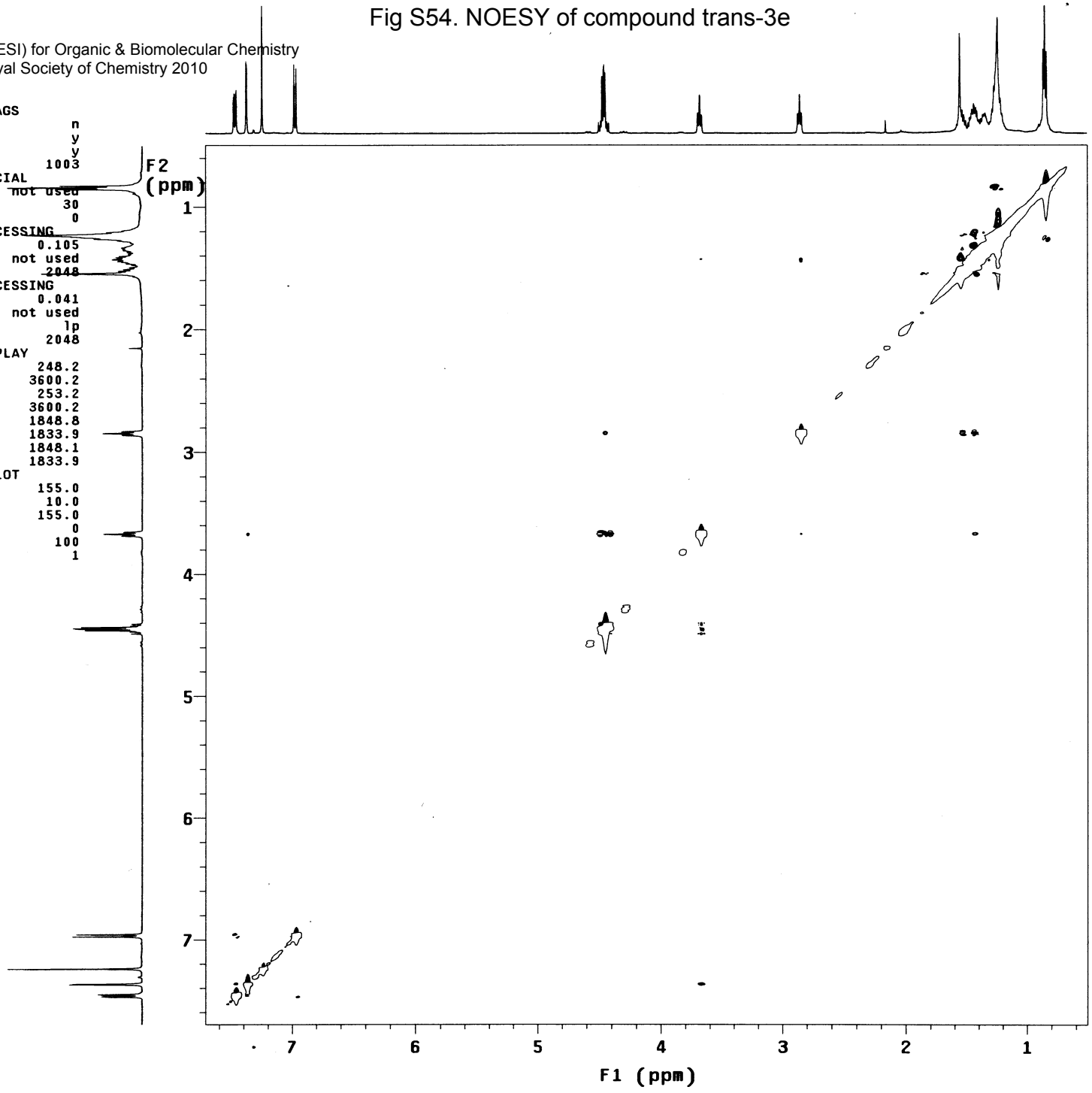


Fig S55. 1H NMR (CDCl3, 500 MHz) of compound 3f

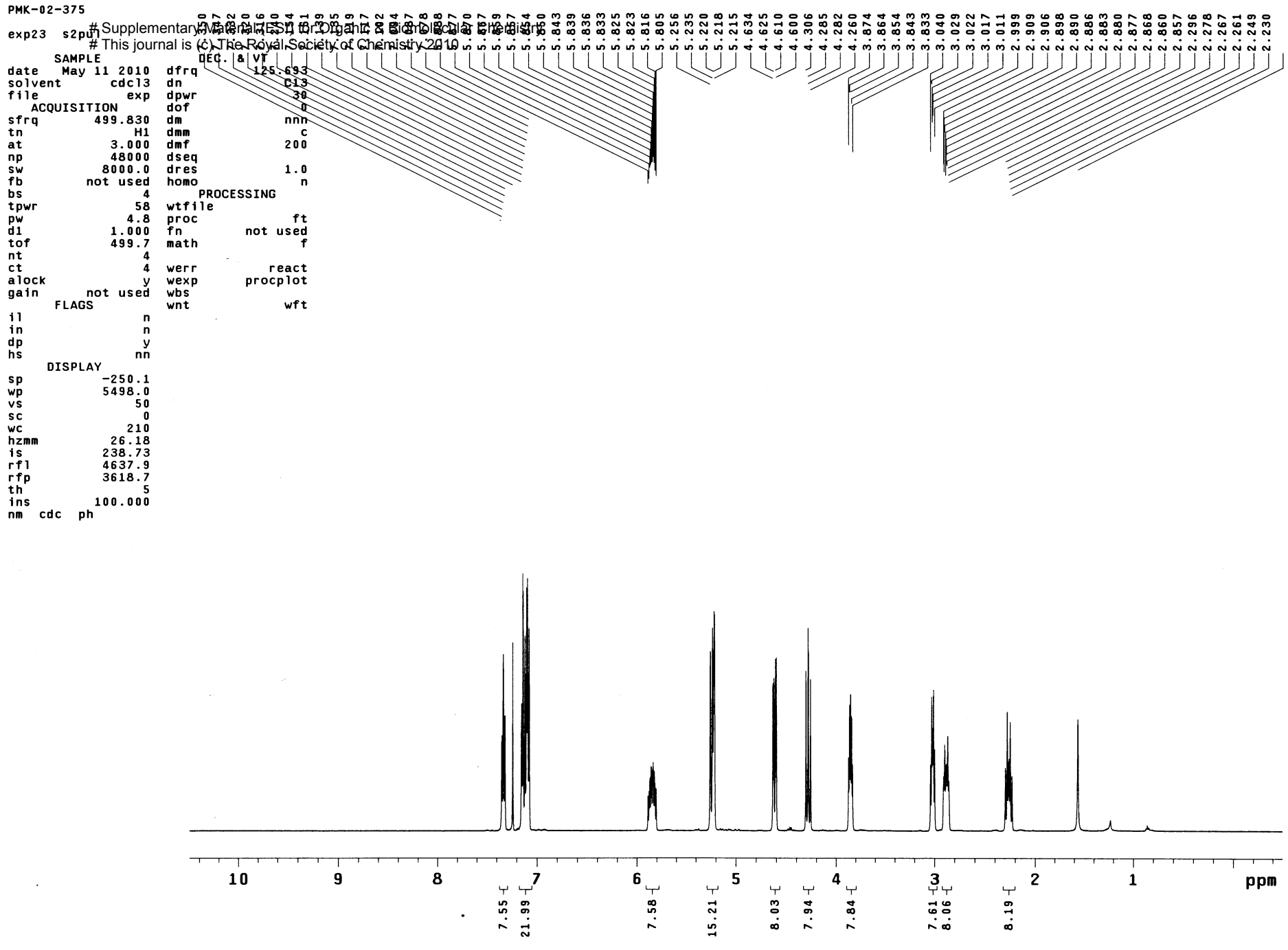
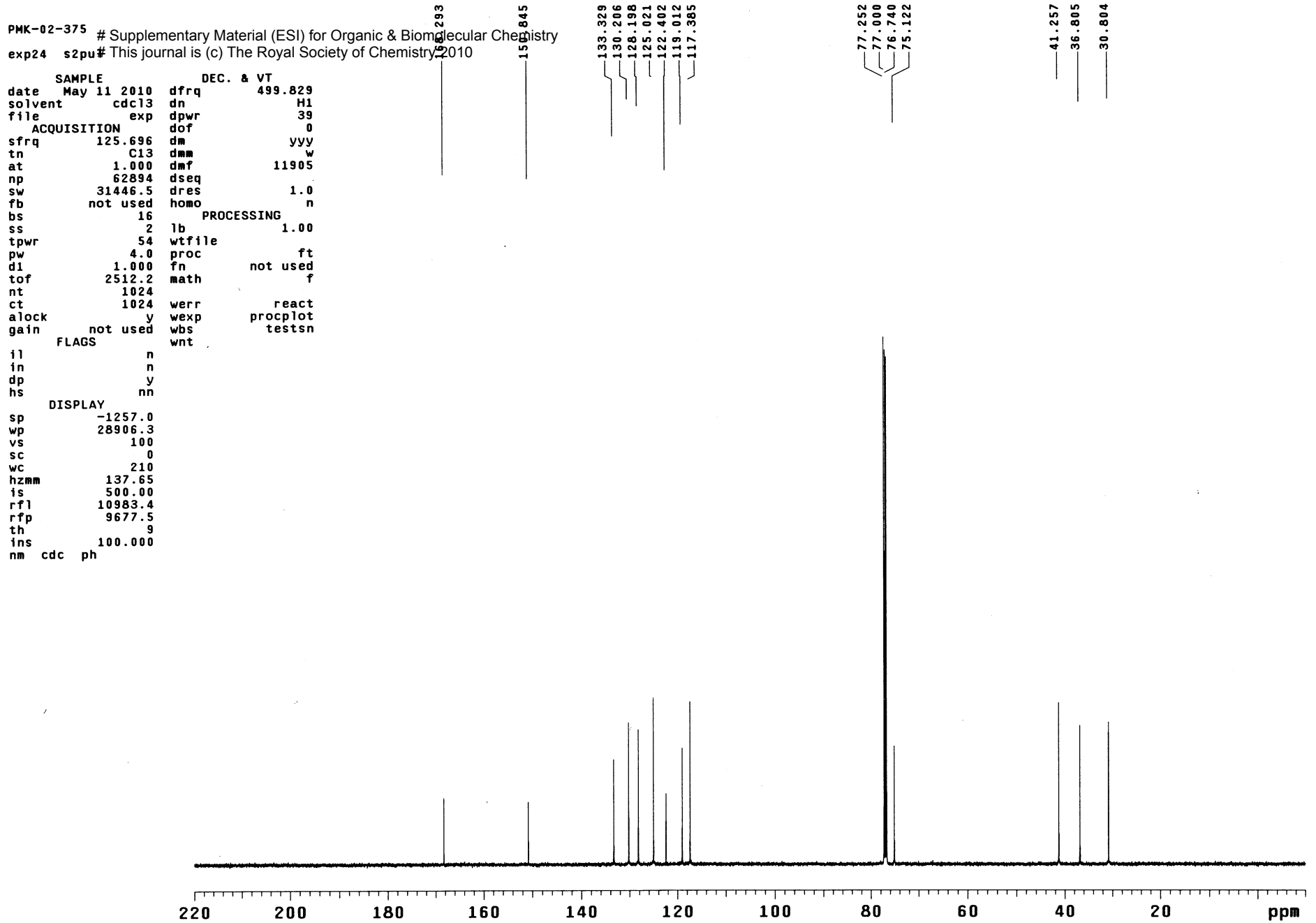


Fig S56. 13C NMR (CDCl3, 125 MHz) of compound 3f



exp25 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	May 11 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.0		
TRANSMITTER		rp	135.9		
tn	C13	lp	204.1		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1305.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	100		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

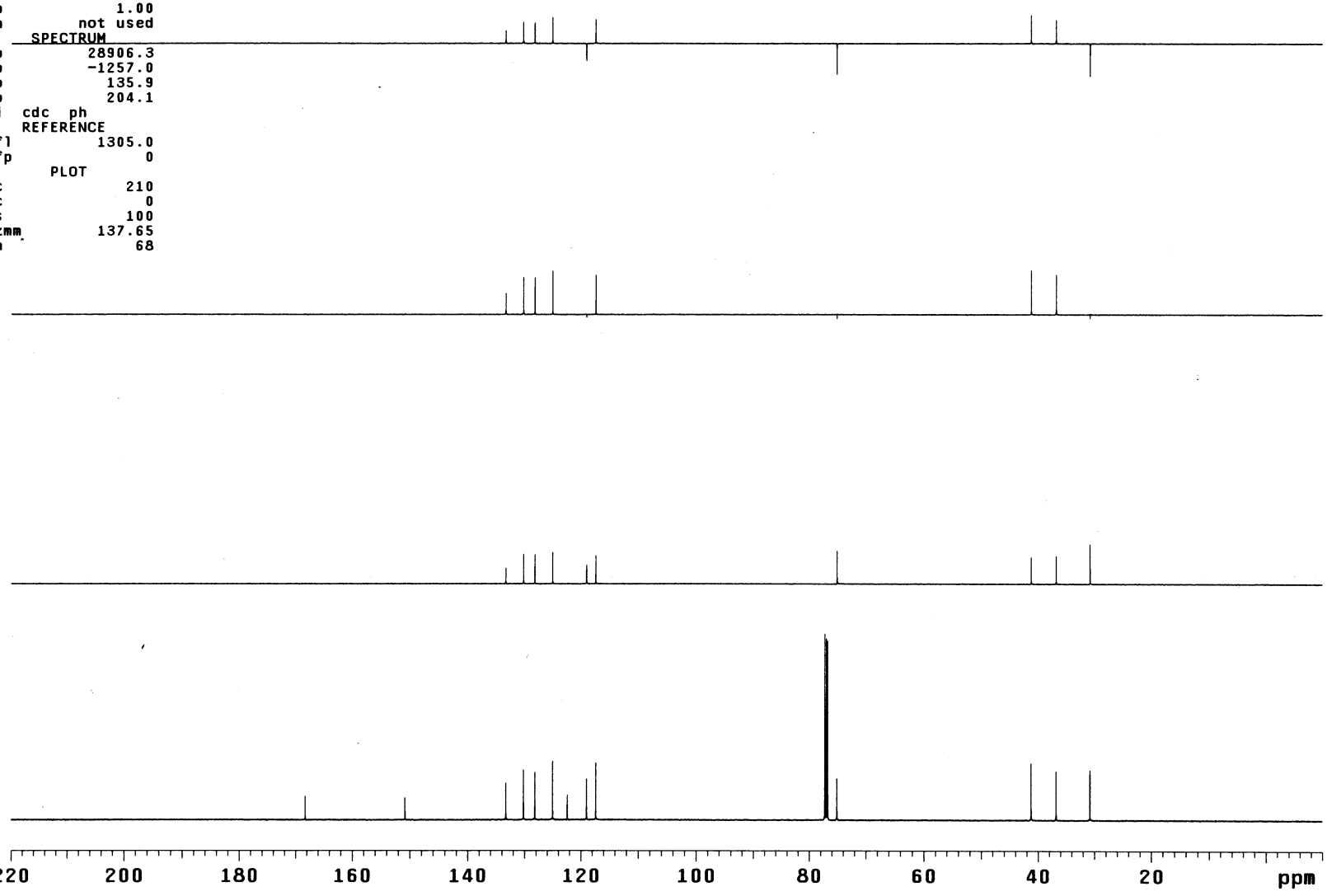


Fig S58. HSQC of compound 3f

exp28 gHSQC

SAMPLE	
date	May 11 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	21367.5
ni	128
phase	arrayed
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
DECOUPLER	
dn	C13
dof	-2515.2
dm	nny
dmm	ccp
dmf	32258
dpwr	36
pwxlvl	52
pwx	14.300
HSQC	
j1xh	140.0
nullflg	y
mult	2

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	20
spin	0
GRADIENTS	
gzlvl1	1003
gt1	0.002000
gzlvl3	505
gt3	0.001000
gstab	0.000500
F2 PROCESSING	
gf	0.105
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.006
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	908.1
wp	3109.0
sp1	3004.7
wp1	14523.2
rfl	2154.1
rfp	2141.3
rfl1	10735.3
rfp1	9441.5
PLOT	
wc	150.0
sc	6.2
wc2	116.2
sc2	0
vs	100
th	5
ai cdc ph	

ACQUISITION ARRAYS	
array	phase
arraydim	256
i	phase
1	1
2	2

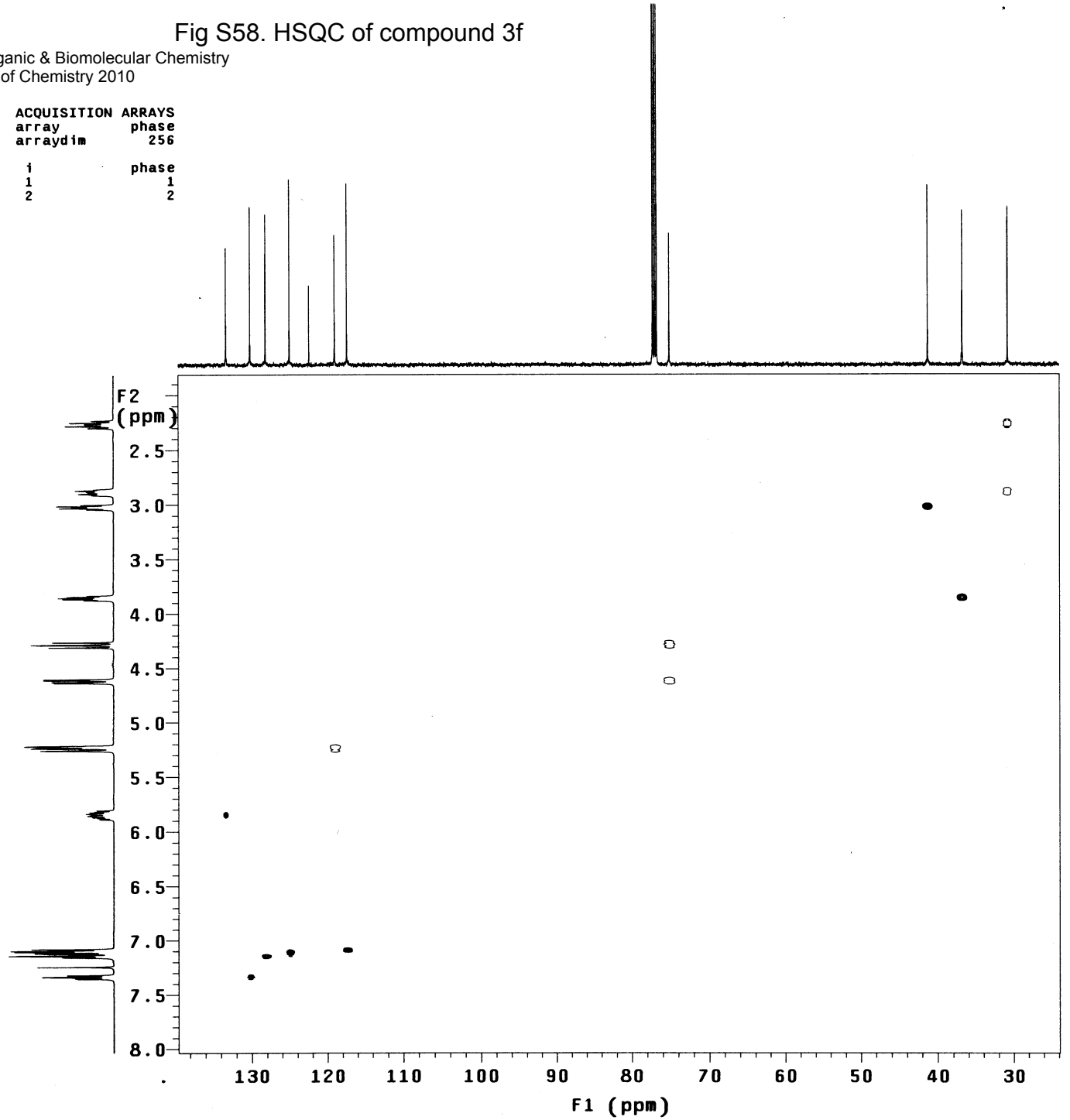


Fig S59. COSY of compound 3f

PMK-02-375
exp26 gCOSY

SAMPLE		FLAGS	
date	May 11 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4490.3	temp	not used
at	0.228	gain	28
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.114
d1	1.000	sbs	not used
nt	8	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	4490.3	sb1	-0.029
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-250.0	sp	927.8
tpwr	58	wp	2911.7
pw	11.100	sp1	929.0
GRADIENTS		wp1	2907.3
gzlvl1	1003	rfl	2156.2
gt1	0.001000	rfp	2141.3
gstab	0.000500	rfl1	2155.1
DECOUPLER		rfp1	2141.2
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	7
		ai cdc av	

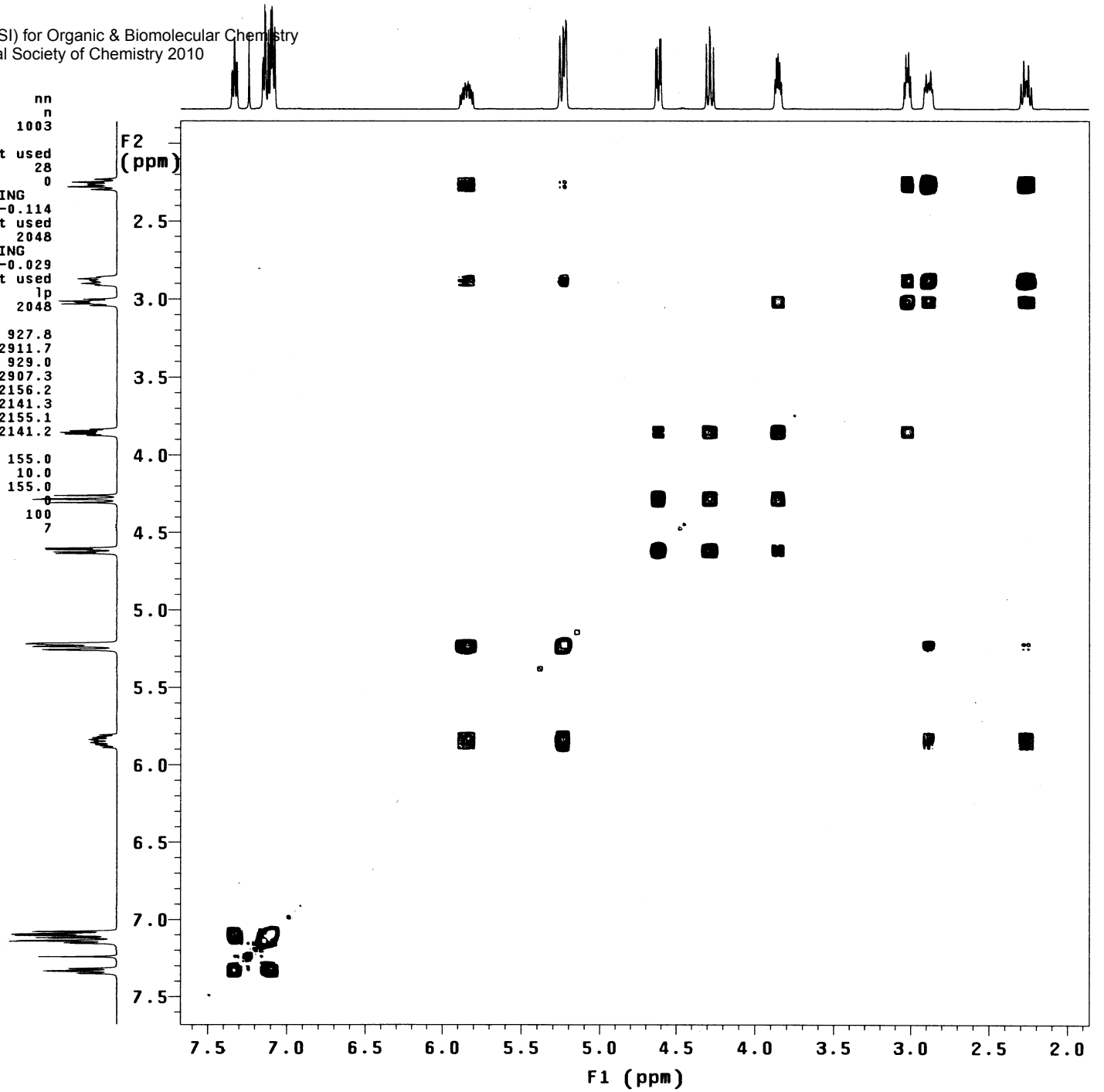


Fig S60. NOESY of compound 3f

exp27 NOESY

SAMPLE	
date	May 11 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	4490.3
ni	200
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	28
spin	0
F2 PROCESSING	
gf	0.105
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.041
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	945.6
wp	2946.8
sp1	946.5
wp1	2946.8
rfl	2156.0
rfp	2141.3
rfl1	2155.1
rfp1	2141.2
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	2
ai	ph

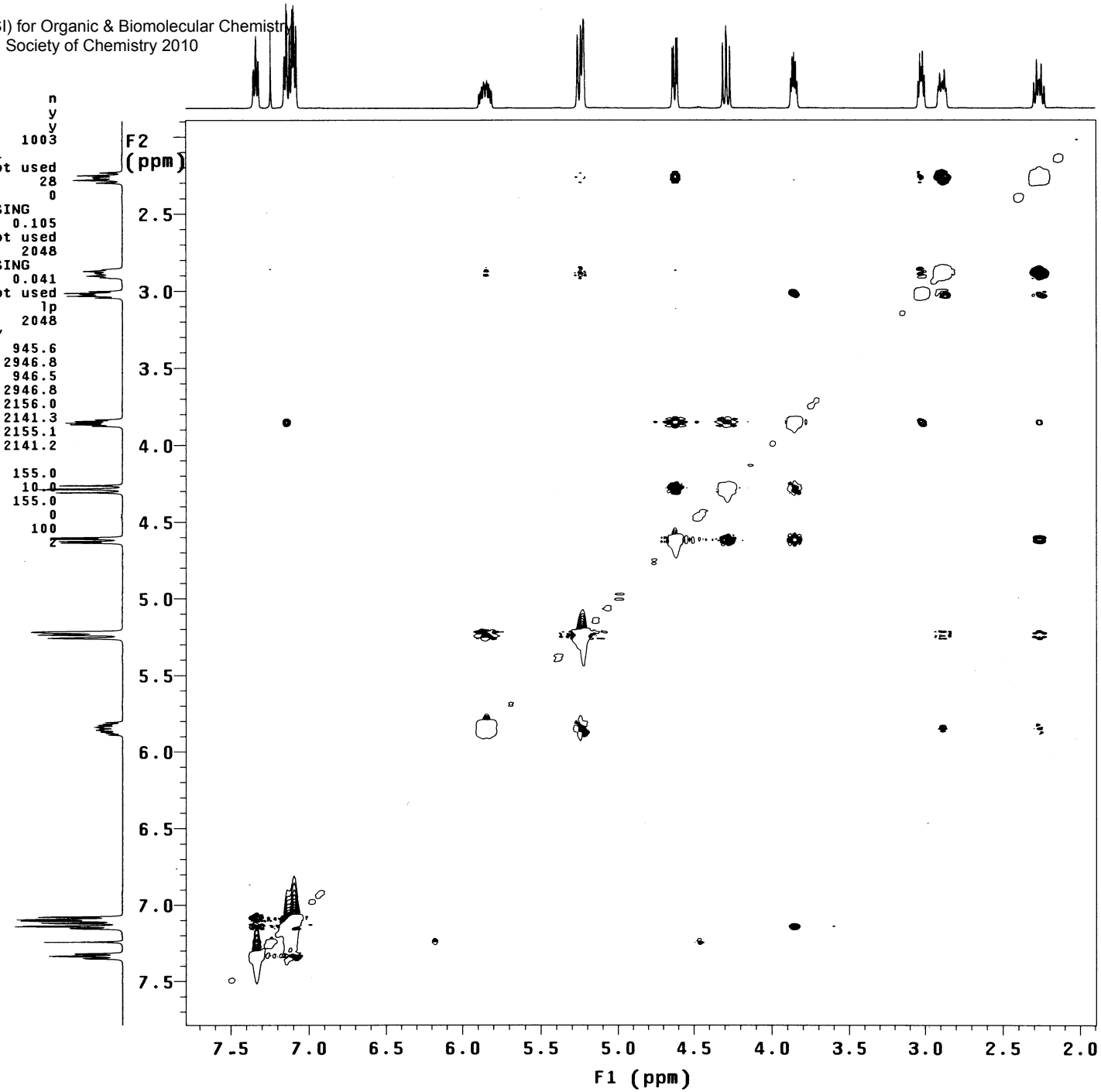


Fig S61. 1H NMR (CDCl3, 500 MHz) of compound 3g

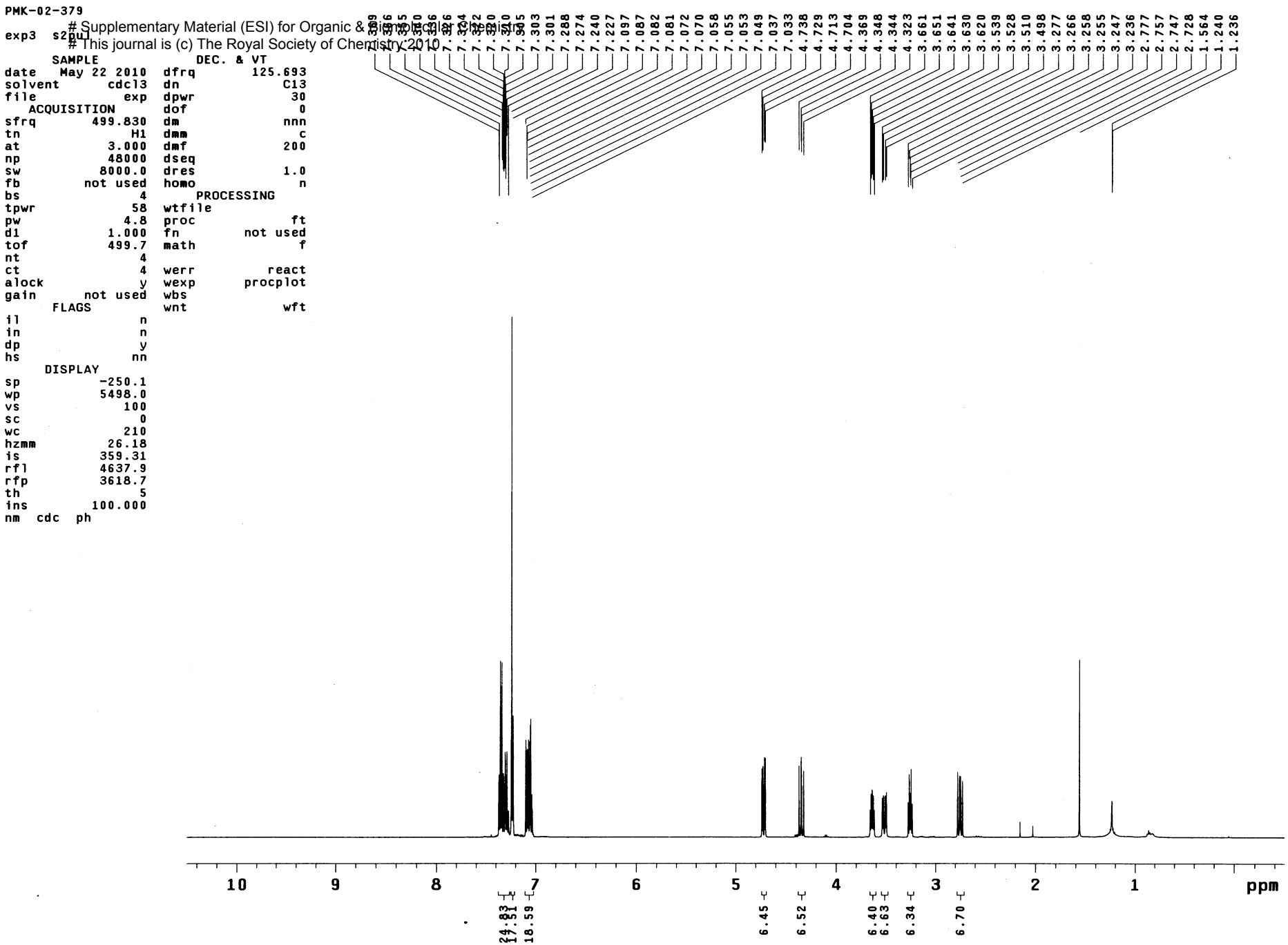


Fig S62. 13C NMR (CDCl3, 125 MHz) of compound 3g

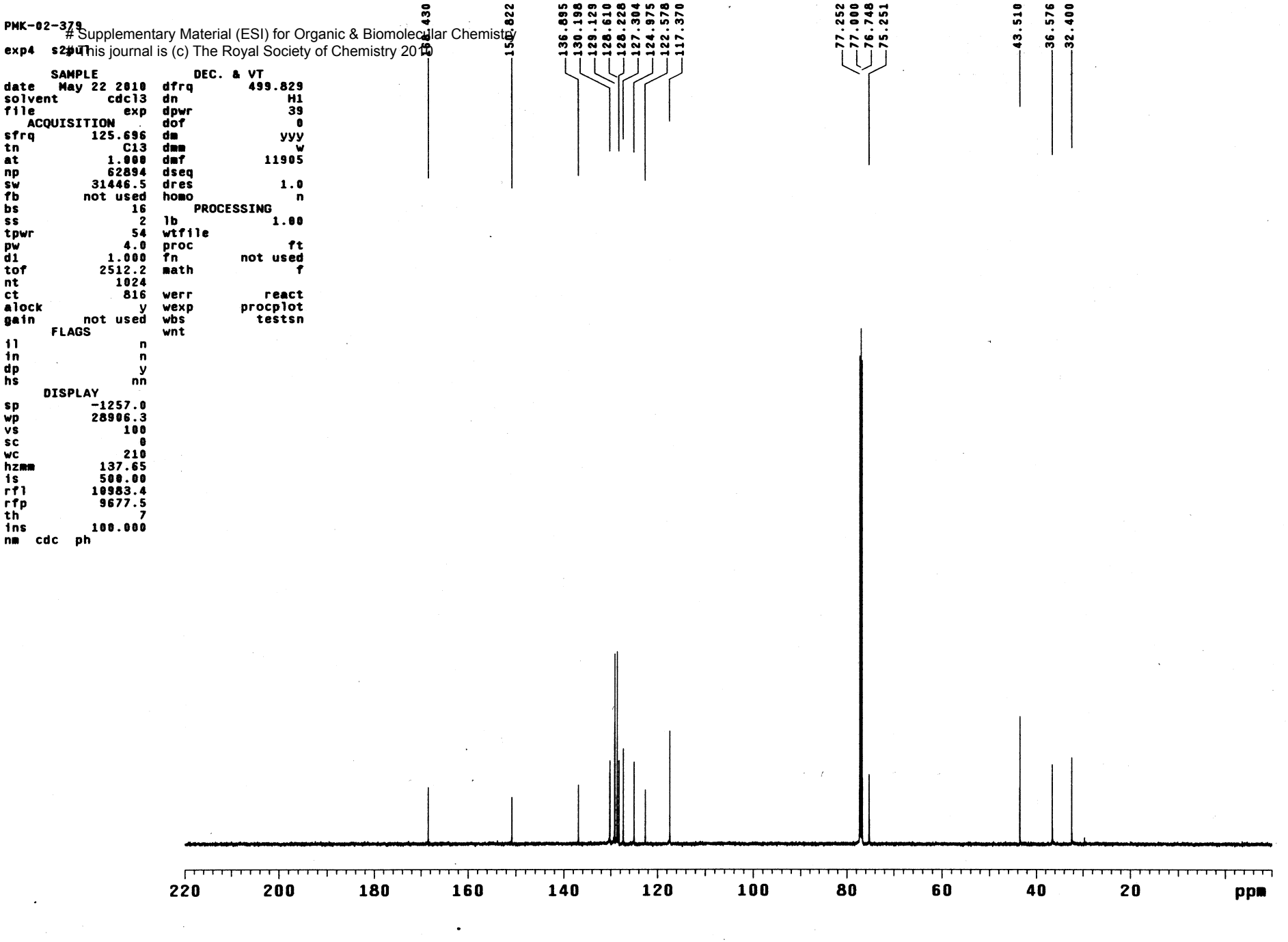


Fig S63. DEPT of compound 3g

SAMPLE	
date	May 22 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	31446.5
at	1.000
np	62894
bs	16
ss	-4
d1	1.000
nt	1024
ct	1024
TRANSMITTER	
tn	C13
tof	2512.2
tpwr	54
pw	11.500
DECOUPLER	
dn	H1
dof	0
dpwr	39
dm	nny
dmm	ccw
dmf	11985
pplvl	51
pp	28.000

DEPT	
jlxh	140.0
mult	arrayed
SPECIAL	
temp	not used
gain	28
spin	0
PROCESSING	
lb	1.00
fn	not used
SPECTRUM	
wp	28906.3
sp	-1257.8
rp	127.5
lp	214.0
ai	cdc ph
REFERENCE	
rfl	1306.8
rfp	0
PLOT	
wc	210
sc	0
vs	100
hzmm	137.65
th	68

ACQUISITION ARRAYS	
array	mult
arraydim	3
1	mult
1	0.5
2	1
3	1.5

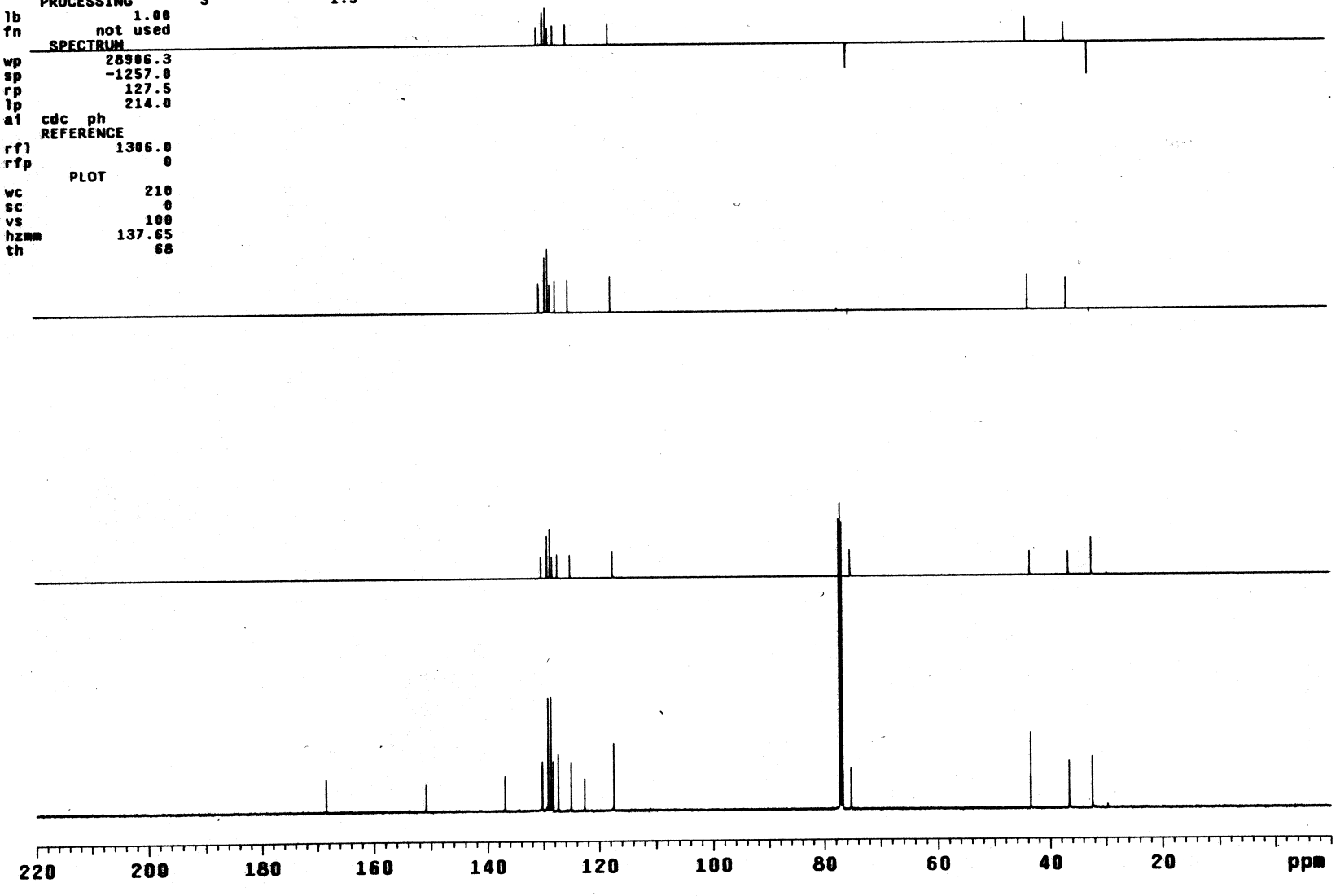


Fig S64. HSQC of compound 3g

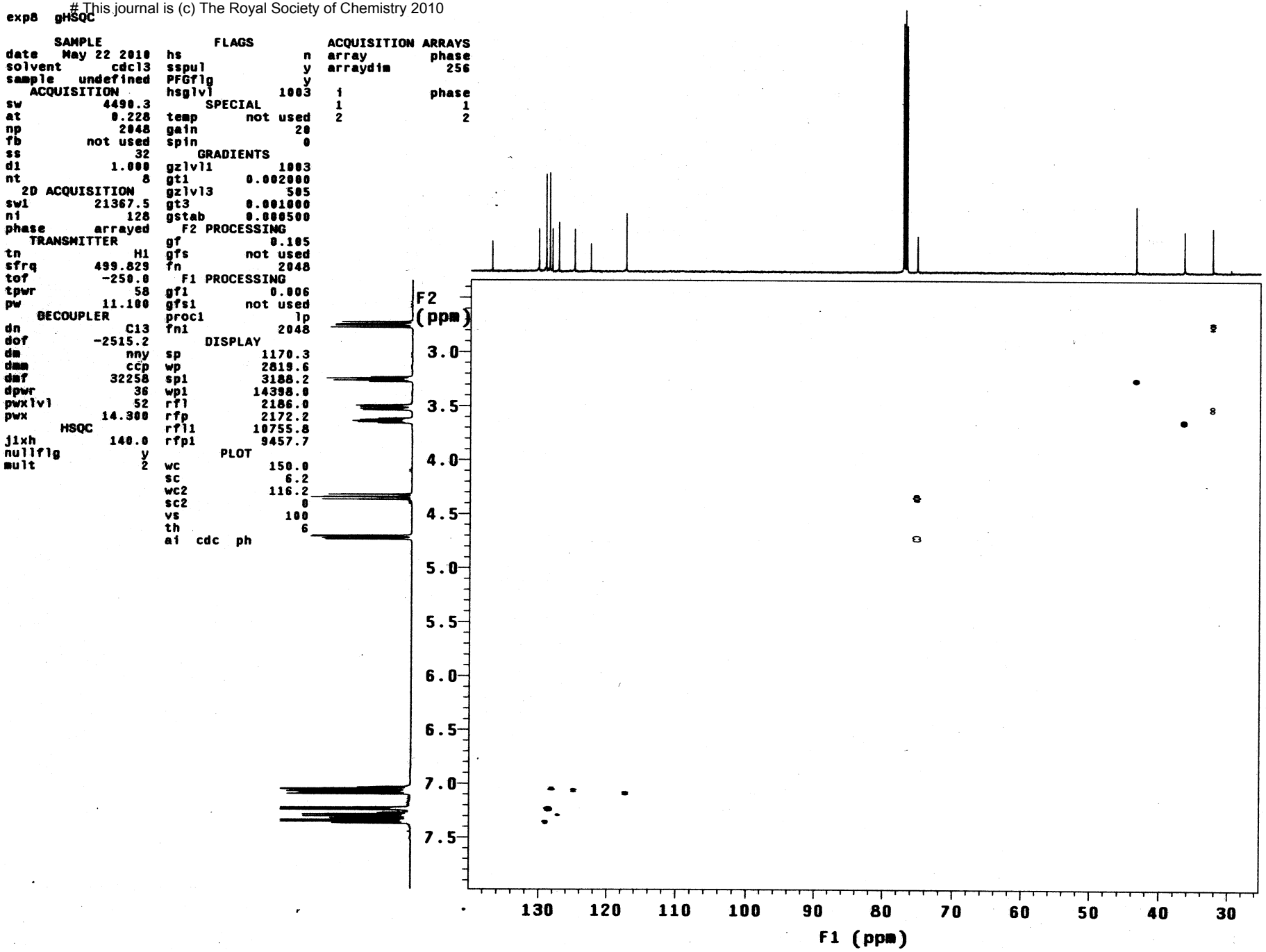


Fig S65. COSY of compound 3g

exp6 gCOSY

SAMPLE	
date	May 22 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	16
d1	1.000
nt	8
2D ACQUISITION	
sw1	4490.3
ni	128
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
GRADIENTS	
gzlvl1	1003
gt1	0.001000
gstab	0.000500
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	nn
sspul	n
hsglvl	1003
SPECIAL	
temp	not used
gain	28
spin	
F2 PROCESSING	
sb	-0.114
sbs	not used
fn	2048
F1 PROCESSING	
sb1	-0.829
sbs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	1200.8
wp	2604.8
sp1	1215.2
wp1	2596.8
rf1	2186.1
rfp	2172.2
rf11	2184.9
rfp1	2172.2
PLOT	
wc	155.8
sc	18.8
wc2	155.8
sc2	0
vs	100
th	3

ai cdc av

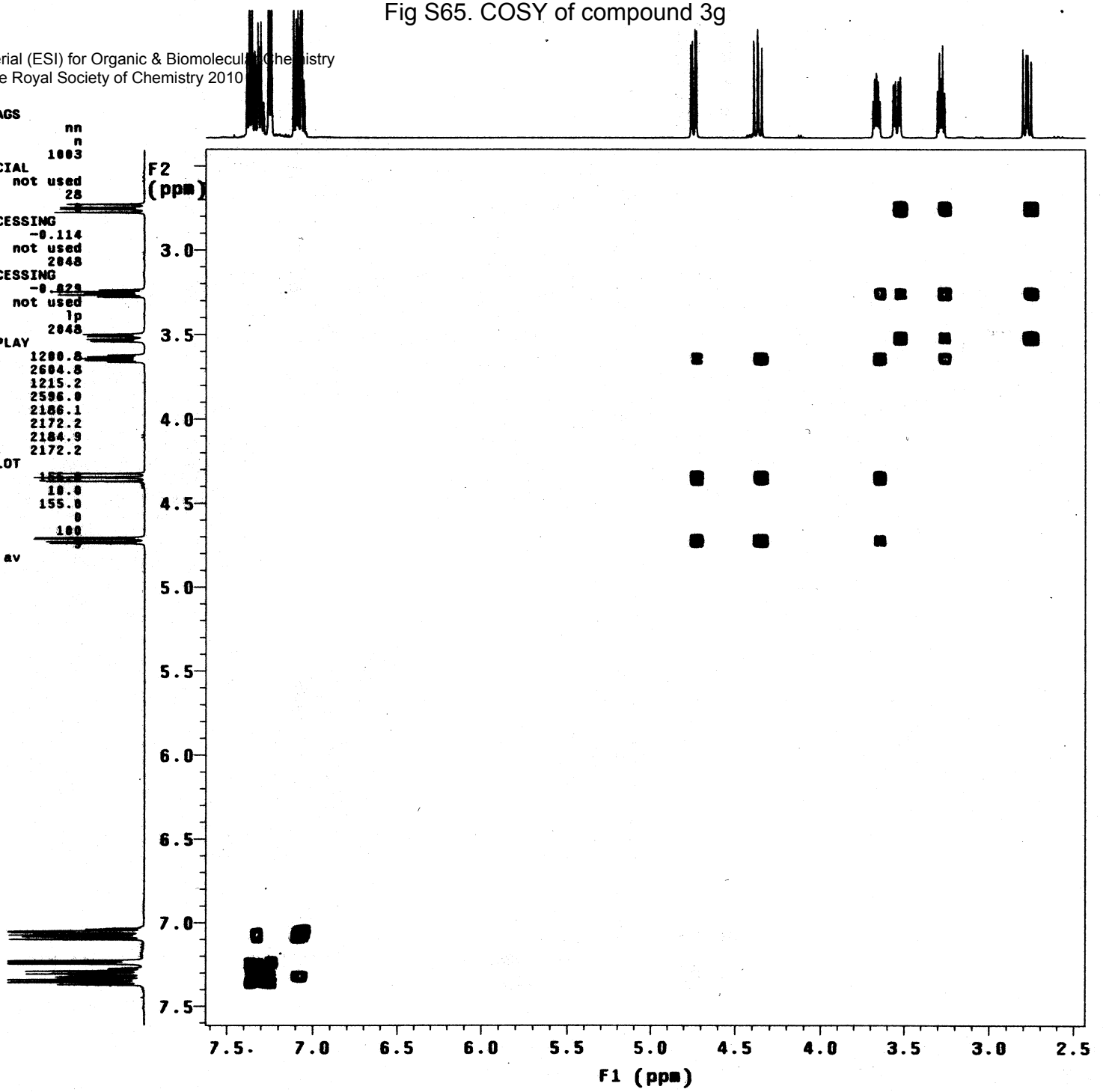
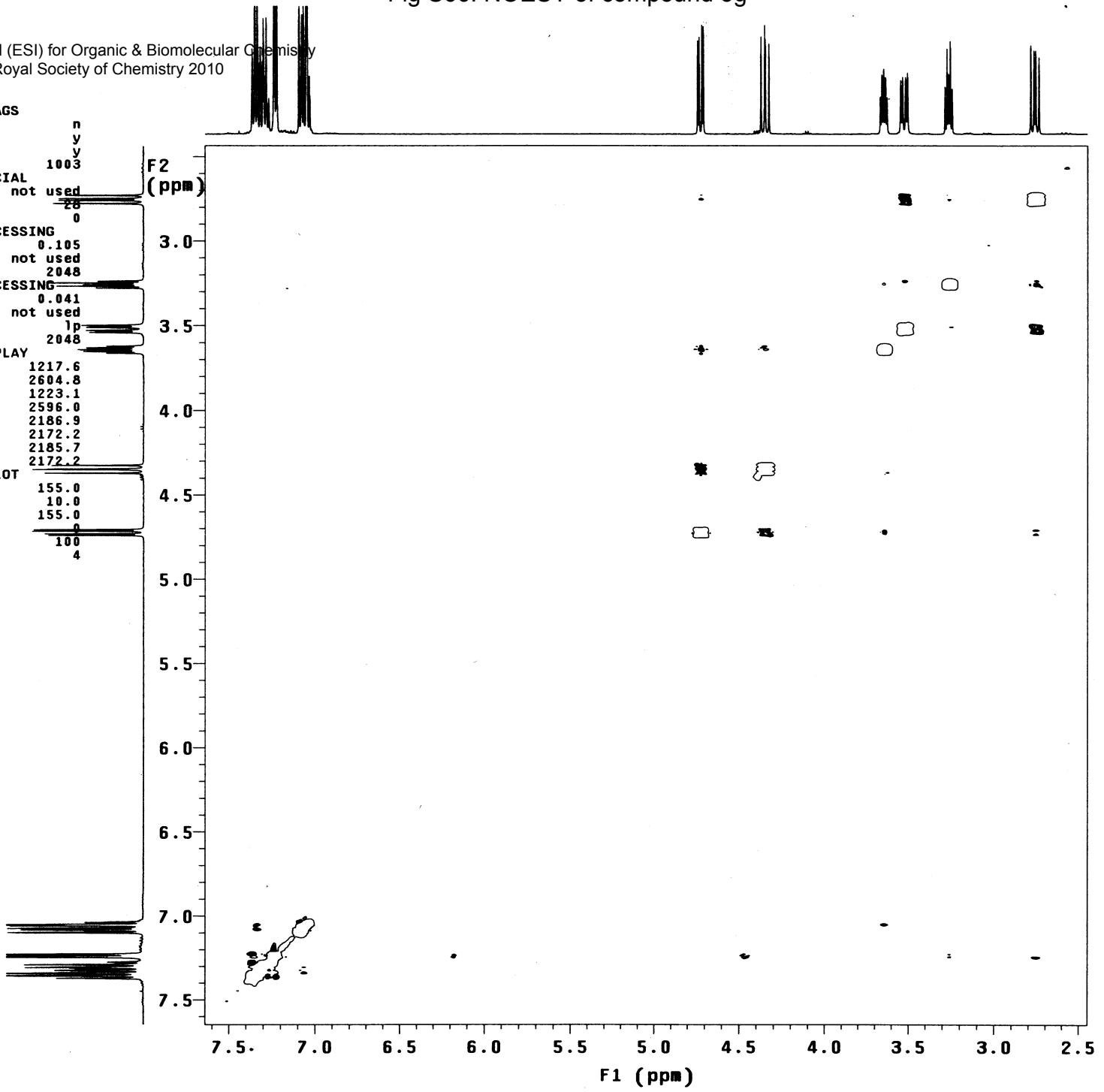


Fig S66. NOESY of compound 3g

PMK-02-379
exp7 NOESY

SAMPLE	
date	May 22 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	4490.3
ni	200
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	28
spin	0
F2 PROCESSING	
gf	0.105
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.041
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	1217.6
wp	2604.8
sp1	1223.1
wp1	2596.0
rfl	2186.9
rfp	2172.2
rfl1	2185.7
rfp1	2172.2
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	4
ai	ph



PMK-02-390

exp13 s2pul

SAMPLE		DEC. at 13	
date	Jul 23 2010	dfrq	125.693
solvent	cdcl3	dn	C13
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.830	dm	nnn
tn	H1	dmm	c
at	3.000	dmf	200
np	48000	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	PROCESSING	
tpwr	58	wtfile	
pw	4.8	proc	ft
d1	1.000	fn	not used
tof	499.7	math	f
nt	4		
ct	4	werr	react
alock	y	wexp	procplot
gain	not used	wbs	
FLAGS		wnt	wft
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5498.0		
vs	100		
sc	0		
wc	210		
hzmm	26.18		
is	345.37		
rfl	4638.7		
rfp	3618.7		
th	2		
ins	100.000		
nm cdc ph			

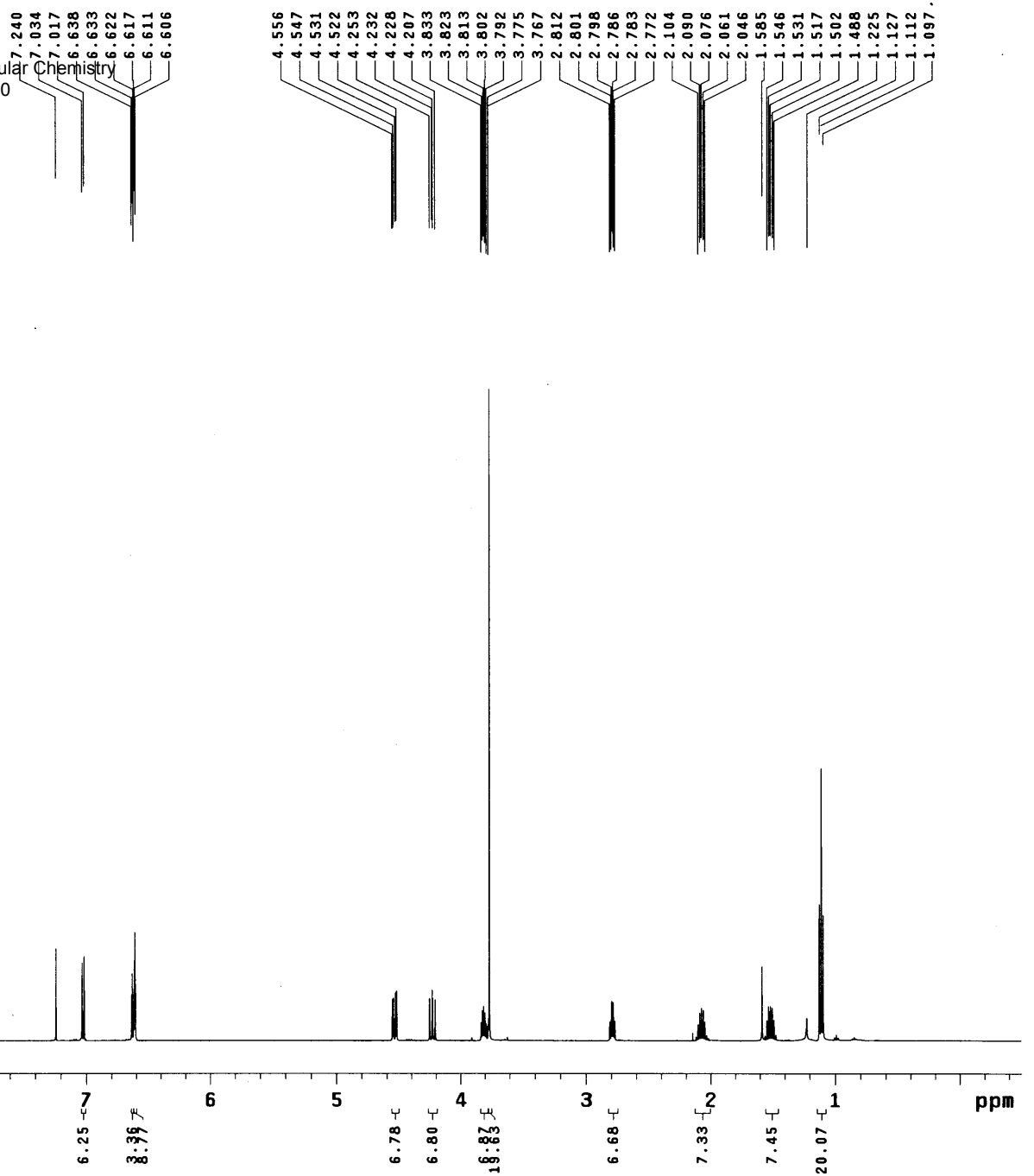
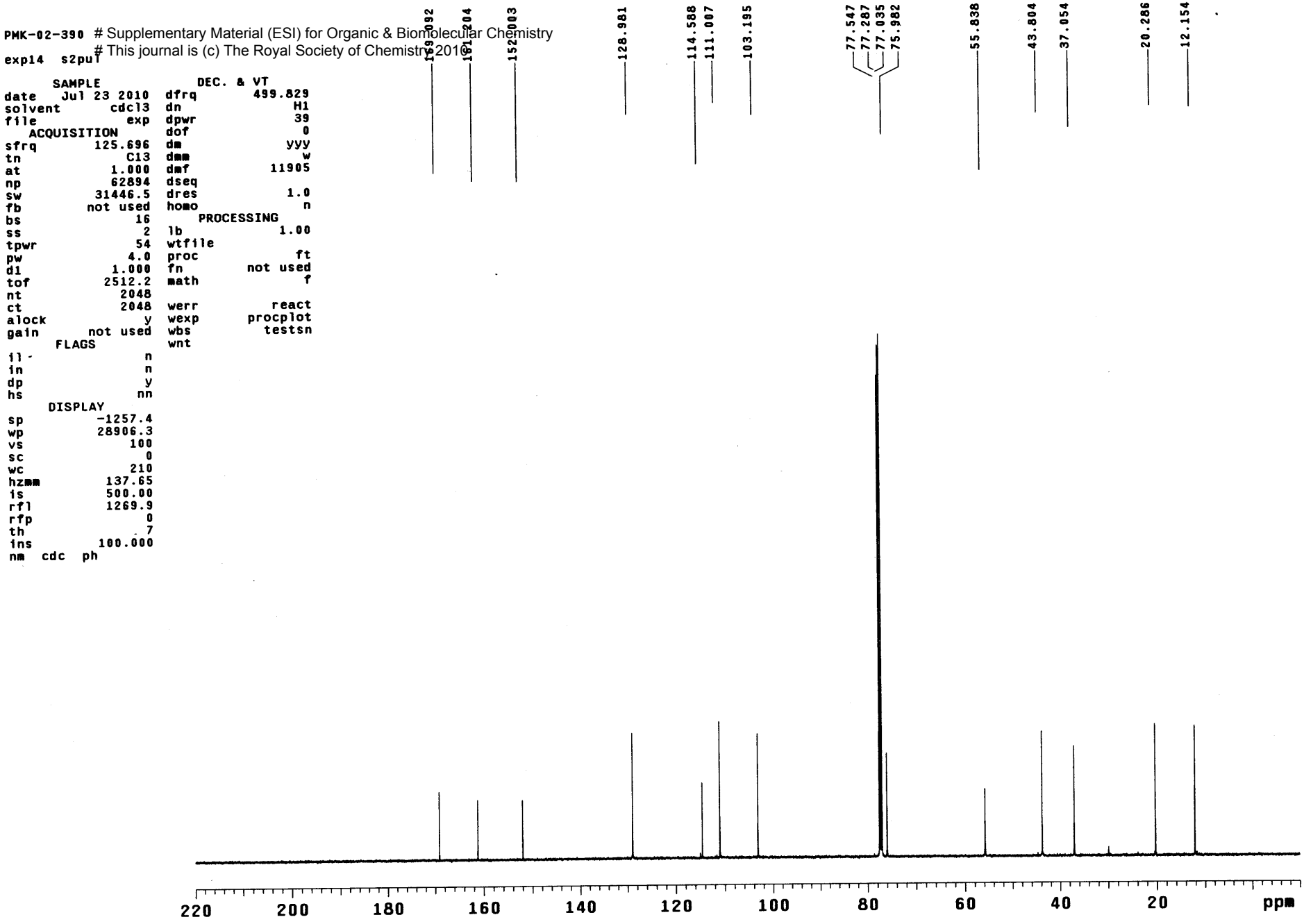


Fig S67. 1H NMR (CDCl3, 500 MHz) of compound 3h

Fig S68. 13C NMR (CDCl3, 125 MHz) of compound 3h



PMK-02-390

exp15 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Jul 23 2010	jlxn	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	20	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.0		
TRANSMITTER		rp	125.8		
tn	C13	lp	225.7		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1306.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	100		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

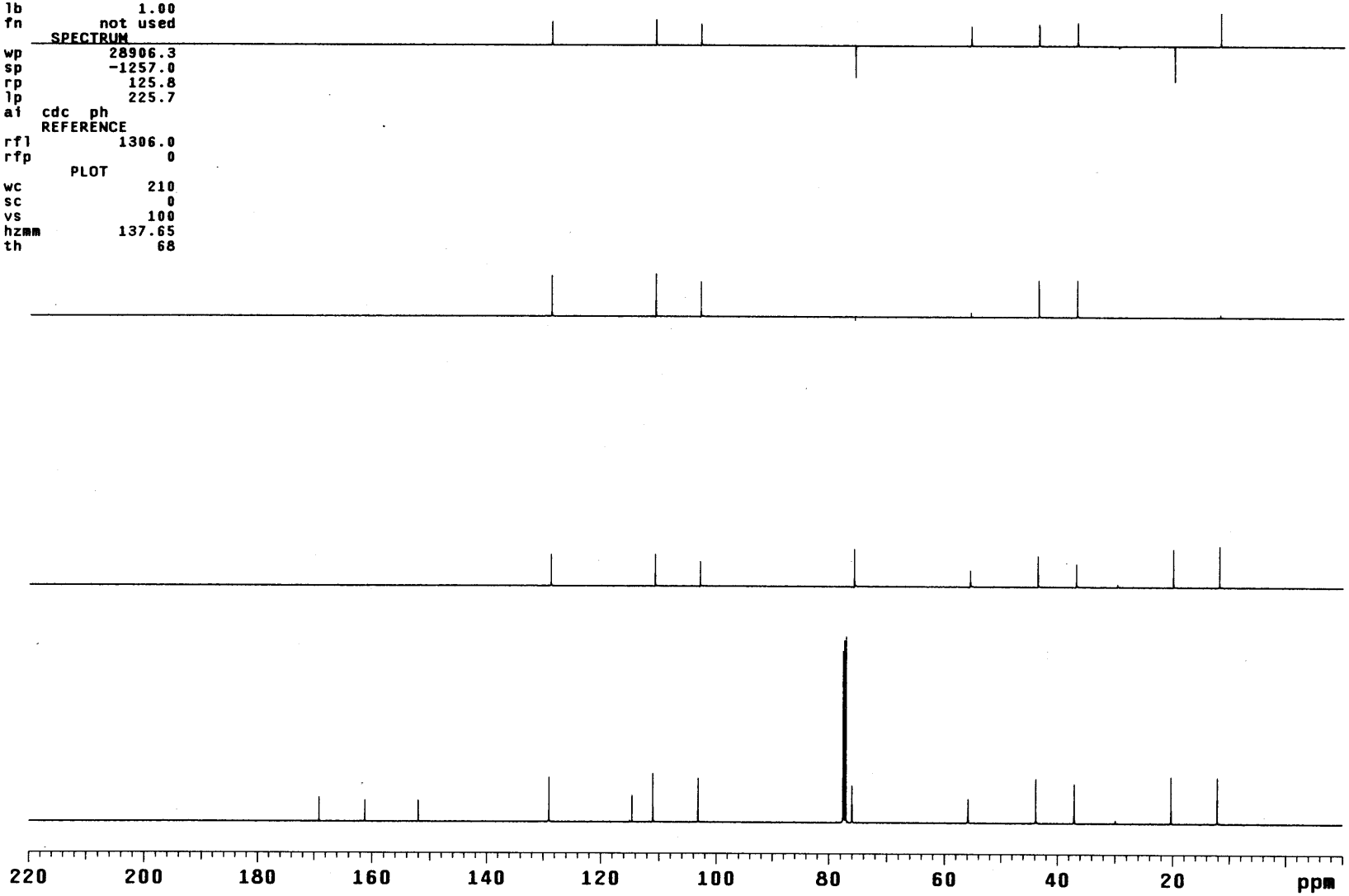


Fig S70. HSQC of compound 3h

PMK-02-390
exp18 gHSQC

SAMPLE	
date	Jul 23 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4001.6
at	0.128
np	1024
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	21367.5
ni	128
phase	arrayed
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-499.9
tpwr	58
pw	11.100
DECOUPLER	
dn	C13
dof	-2515.2
dm	nny
dmm	ccp
dmf	32258
dpwr	36
pwxlvl	52
pwx	14.300
HSQC	
j1xh	140.0
nullflg	y
mult	2

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	44
spin	0
GRADIENTS	
gzlvl1	1003
gt1	0.002000
gzlvl3	505
gt3	0.001000
gstab	0.000500
F2 PROCESSING	
gf	0.059
gfs	not used
fn	1024
F1 PROCESSING	
gf1	0.006
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	315.4
wp	3384.2
sp1	763.5
wp1	16818.6
rfl	2135.0
rfp	2114.3
rfl1	10810.1
rfp1	9549.5
PLOT	
wc	150.0
sc	6.2
wc2	116.2
sc2	0
vs	100
th	4
ai cdc ph	

ACQUISITION ARRAYS	
array	phase
arraydim	256
i	phase
1	1
2	2

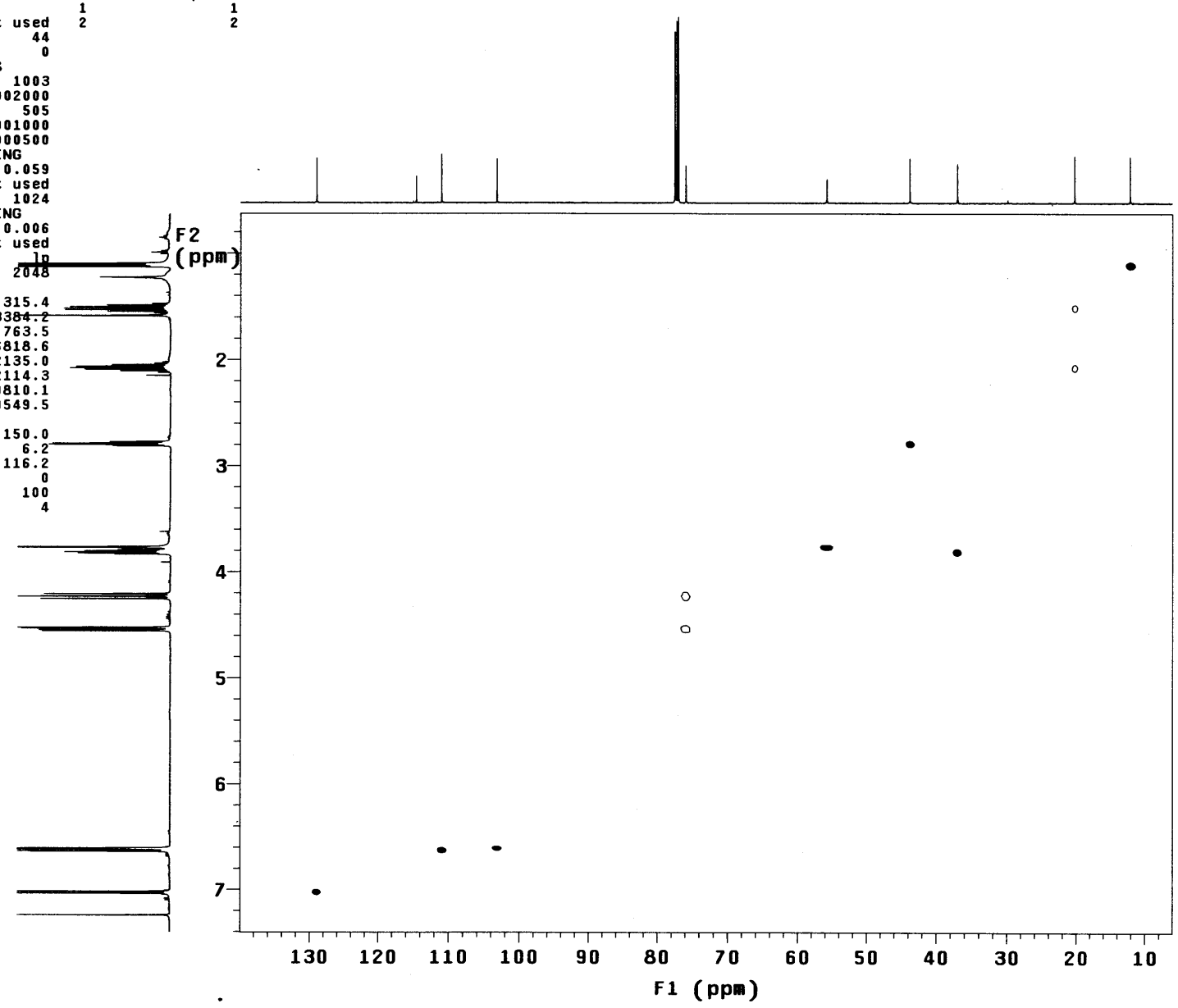


Fig S71. COSY of compound 3h

SAMPLE		FLAGS	
date	Jul 23 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4001.6	temp	not used
at	0.128	gain	24
np	1024	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.064
d1	1.000	sbs	not used
nt	8	fn	1024
2D ACQUISITION		F1 PROCESSING	
sw1	4001.6	sb1	-0.032
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	1024
sfrq	499.829	DISPLAY	
tof	-499.9	sp	301.7
tpwr	58	wp	3399.8
pw	11.100	sp1	293.8
GRADIENTS		wp1	3399.8
gzlvl1	1003	rfl	2133.0
gt1	0.001000	rfp	2114.3
gstab	0.000500	rfl1	2133.0
DECOUPLER		rfp1	2114.3
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	7
		ai cdc av	

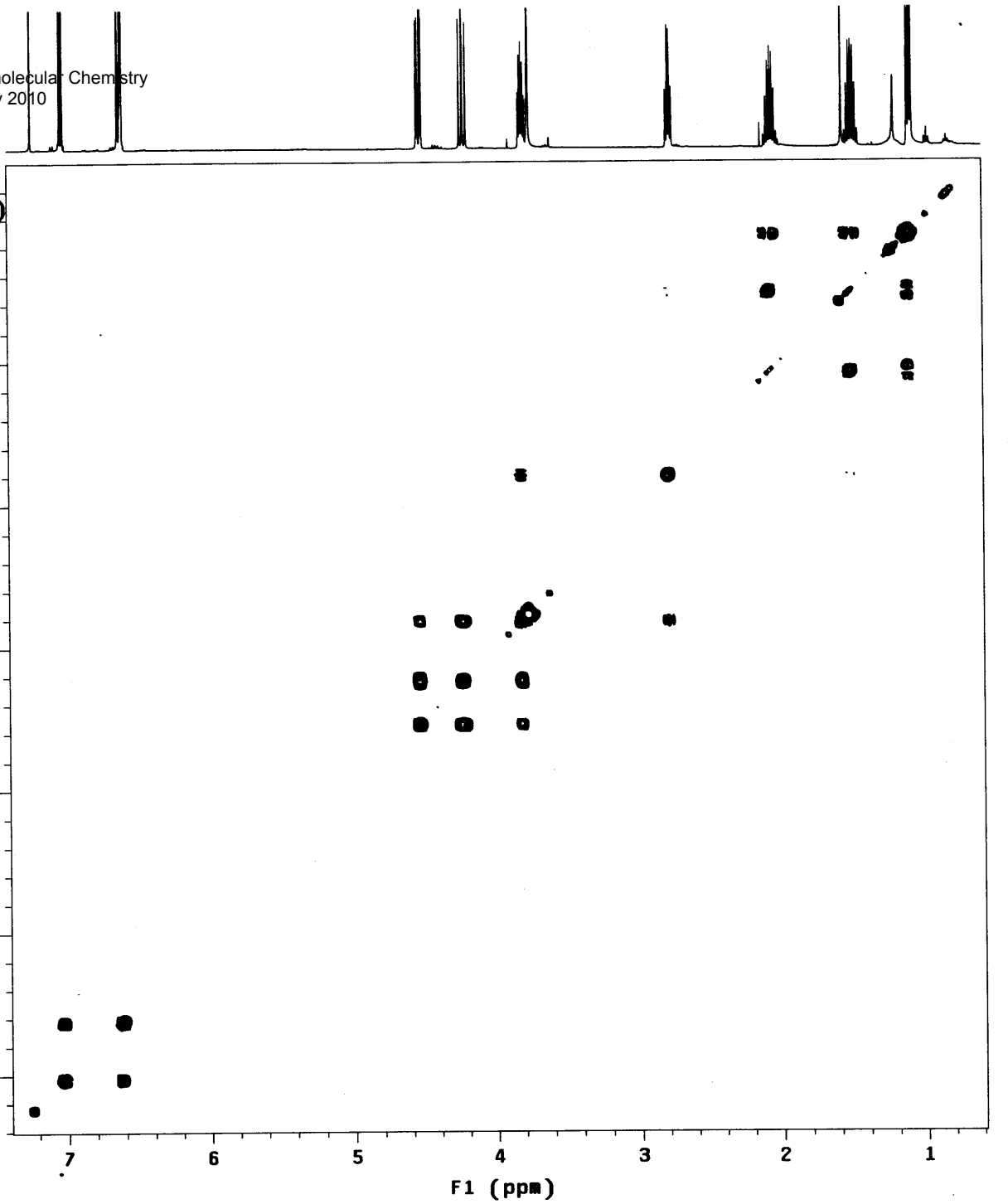


Fig S72. NOESY of compound 3h

PMK-02-390

exp17 NOESY

SAMPLE		FLAGS	
date	Jul 23 2010	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	4001.6	SPECIAL	
at	0.128	temp	not used
np	1024	gain	24
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.059
nt	8	gfs	not used
2D ACQUISITION		fn	1024
swi	4001.6	F1 PROCESSING	
ni	200	gf1	0.046
TRANSMITTER		gfs1	not used
tn	H1	proc1	-1p
sfrq	499.829	fn1	1024
tof	-499.9	DISPLAY	
tpwr	58	sp	348.4
pw	11.100	wp	3360.7
NOESY		sp1	340.3
mix	0.600	wp1	3368.5
PRESATURATION		rfl	2141.0
satmode	nnnn	rfp	2114.3
satpwr	0	rfl1	2133.5
satdly	0	rfp1	2114.3
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	100
		th	1
		ai ph	

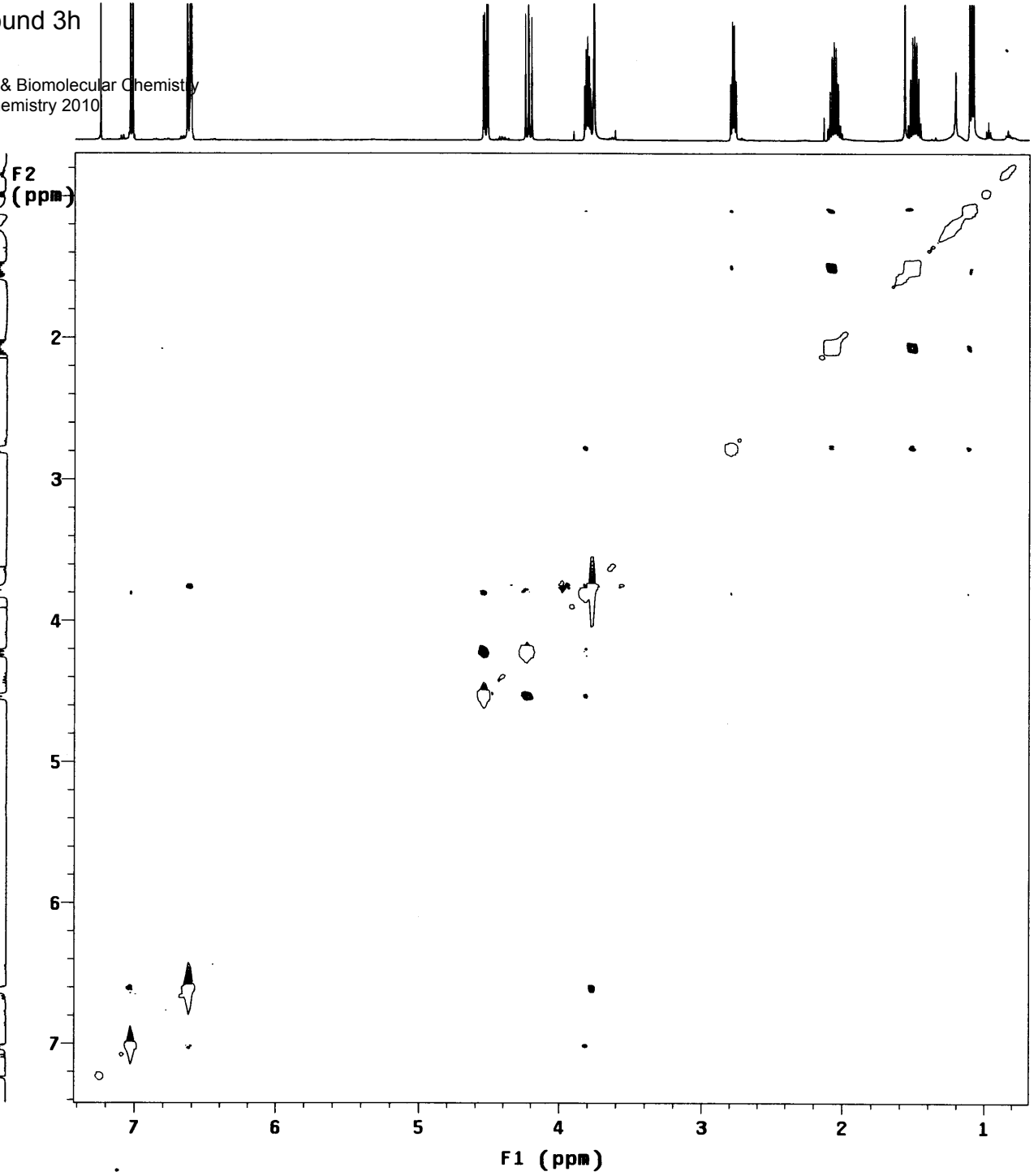


Fig S73. 1H NMR (CDCl3, 500 MHz) of compound cis-3i

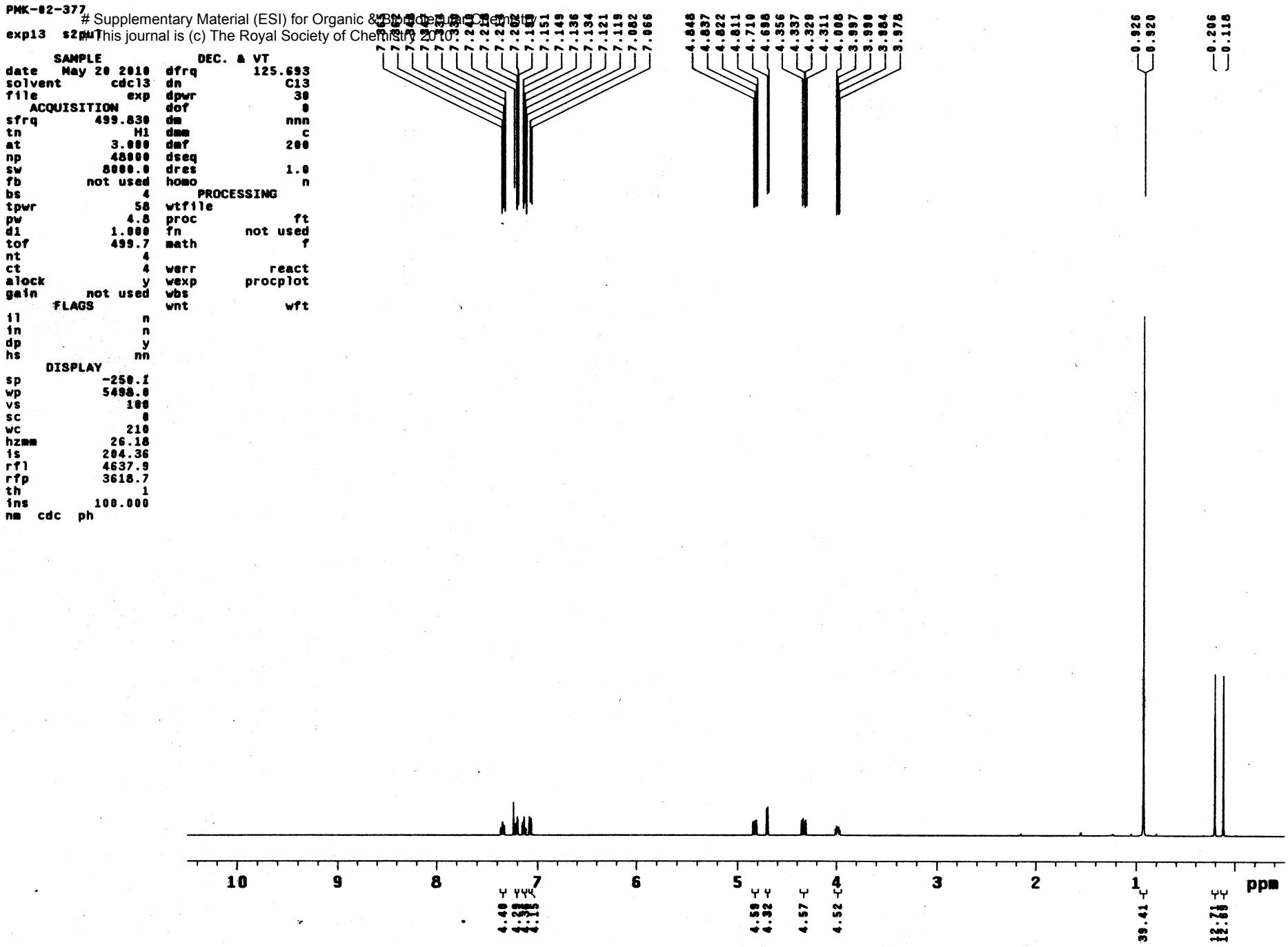


Fig S74. 13C NMR (CDCl3, 125 MHz) of compound cis-3i

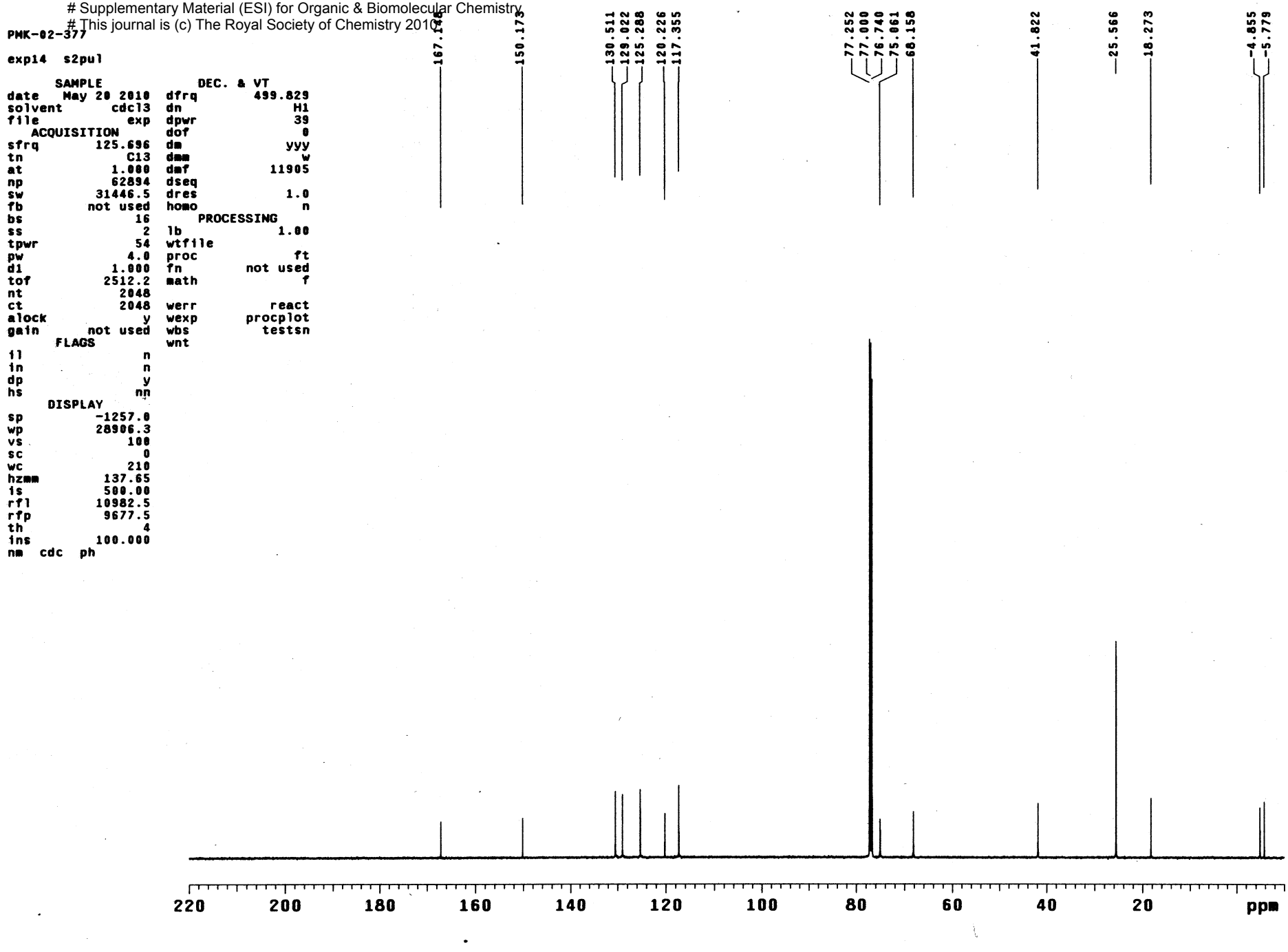


Fig S75. DEPT of compound cis-3i

PMK-02-377

exp15 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	May 20 2010	jixh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	28986.3		
ct	2048	sp	-1257.0		
TRANSMITTER		rp	138.2		
tn	C13	lp	191.8		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1304.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	108		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

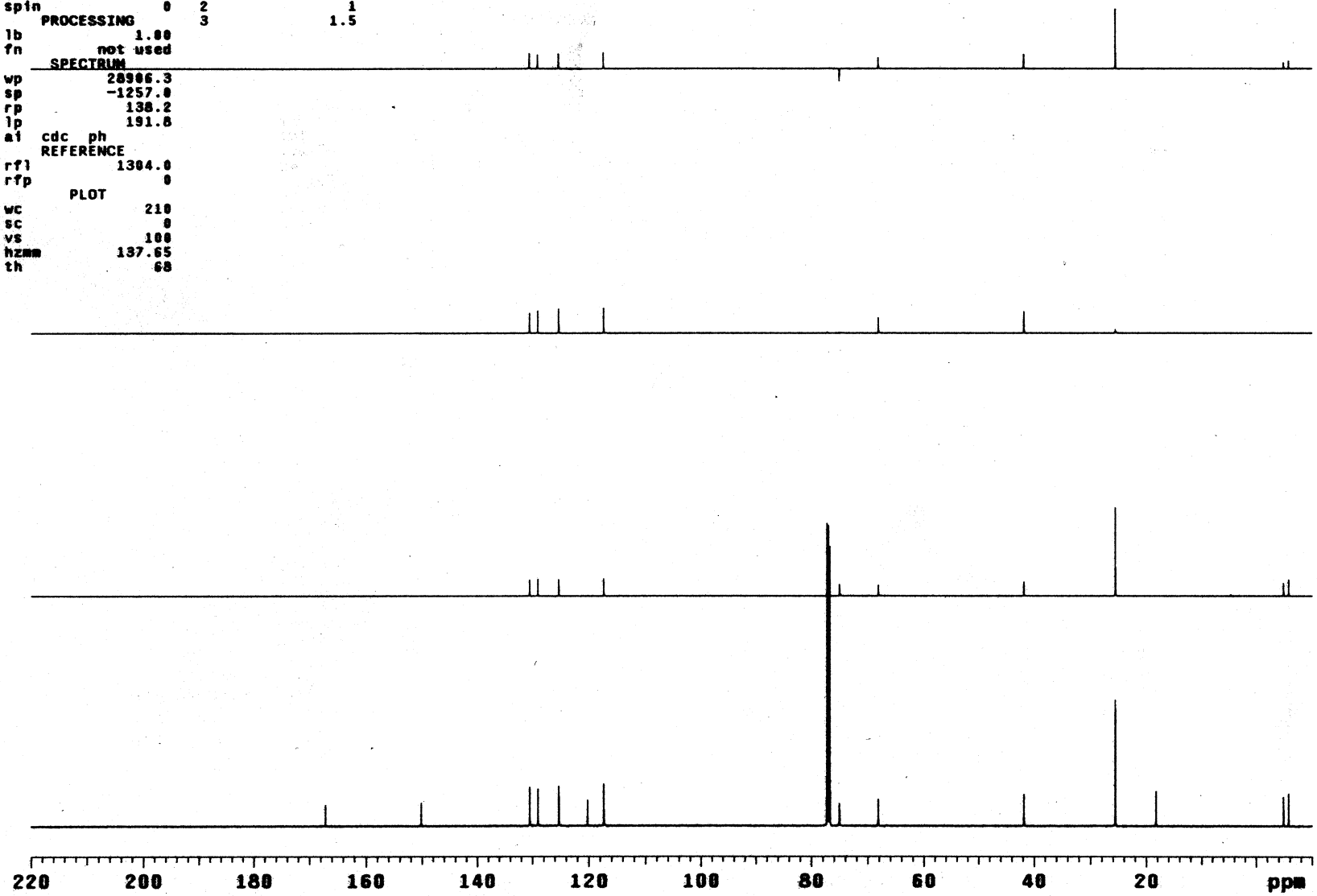


Fig S76. HSQC of compound cis-3i

PMK-02-377

exp18 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	May 20 2010	ss	0	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	5006.3	SPECIAL		1	1
at	0.205	temp	not used	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.094		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-499.9	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	-195.5		
dmm	ccp	wp	4111.6		
dmf	32258	sp1	-1231.1		
dpwr	36	wp1	18821.8		
pwxlvl	52	rfl	2515.9		
pwx	14.300	rfp	1992.8		
HSQC		rfl1	6550.0		
j1xh	140.0	rfp1	5256.3		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	1814		
		th	3		
		ai cdc ph			

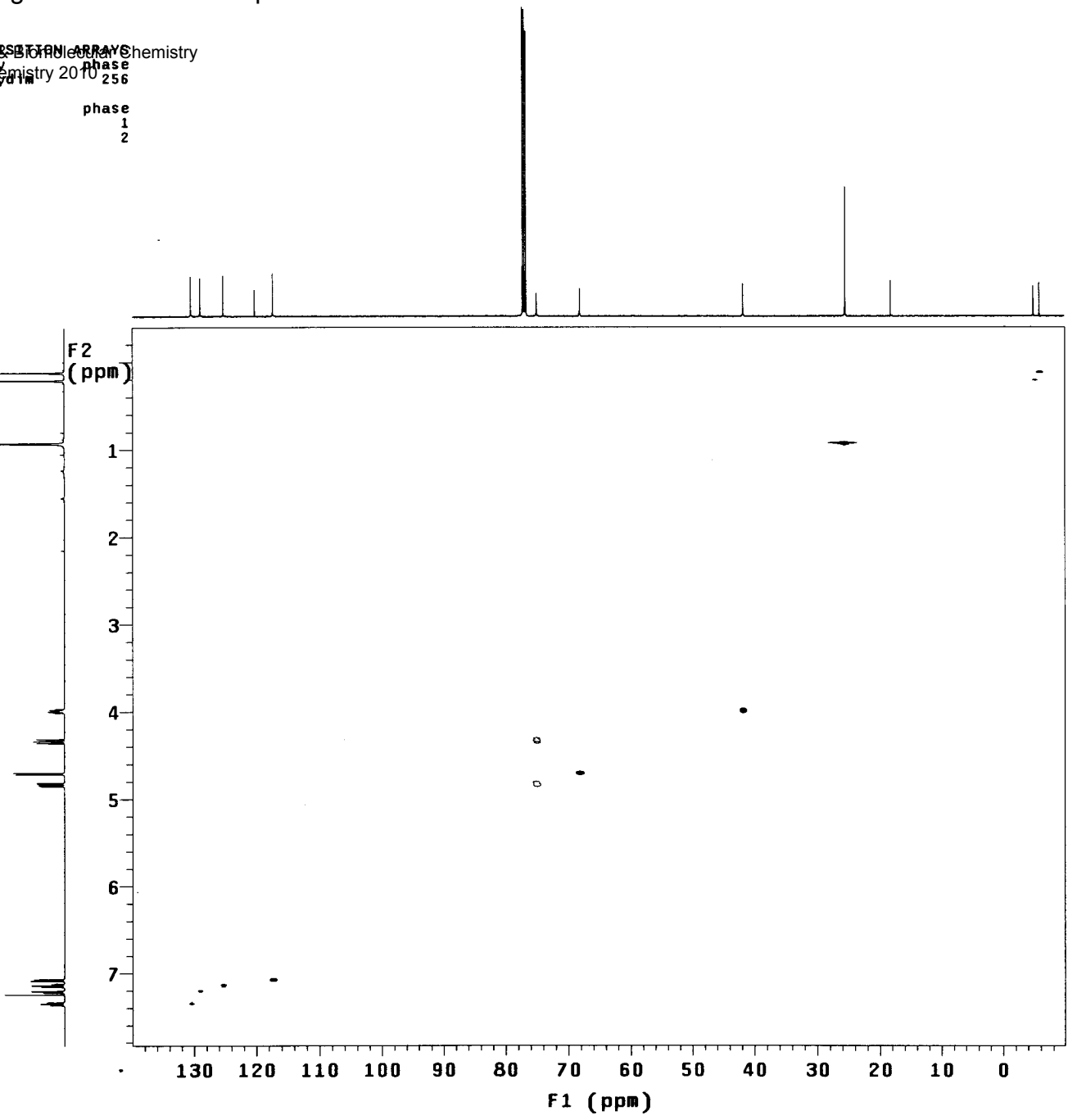


Fig S77. COSY of compound cis-3i

PMK-02-377
exp16 gCOSY

SAMPLE	
date	May 20 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	5006.3
at	0.205
np	2048
fb	not used
ss	16
d1	1.000
nt	16
2D ACQUISITION	
sw1	5006.3
ni	128
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-499.9
tpwr	58
pw	11.100
GRADIENTS	
gzlvl1	1003
gt1	0.001000
gstab	0.000500
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	nn
sspul	n
hsglvl	1003
SPECIAL	
temp	not used
gain	28
spin	0
F2 PROCESSING	
sb	-0.102
sbs	not used
fn	2048
F1 PROCESSING	
sb1	-0.026
sbs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	-136.8
wp	3989.4
sp1	-126.1
wp1	3984.5
rfl	2515.8
rfp	1992.8
rfl1	2514.9
rfp1	1992.8
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	1814
th	5
ai cdc av	

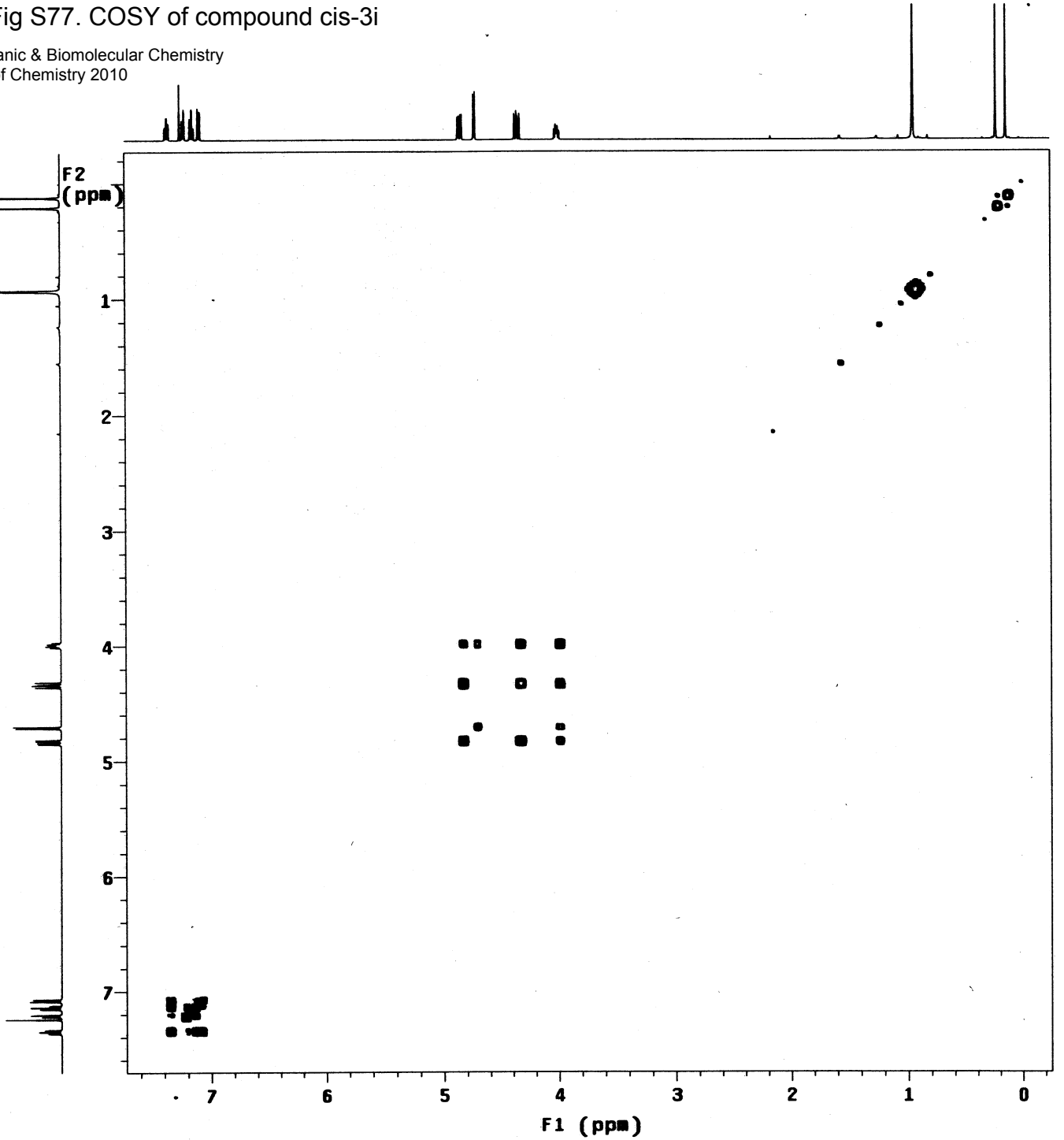


Fig S78. NOESY of compound cis-3i

PMK-02-377

exp17 NOESY

SAMPLE		FLAGS	
date	May 20 2010	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	5006.3	SPECIAL	
at	0.205	temp	not used
np	2048	gain	28
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.094
nt	8	gfs	not used
2D ACQUISITION		fn	2048
sw1	5006.3	F1 PROCESSING	
ni	200	gf1	0.037
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.829	fn1	2048
tof	-499.9	DISPLAY	
tpwr	58	sp	-119.7
pw	11.100	wp	3911.1
NOESY		sp1	-116.3
mix	0.600	wp1	3911.1
PRESATURATION		rf1	2518.3
satmode	nnnn	rfp	1992.8
satpwr	0	rf11	2514.9
satdly	0	rfp1	1992.8
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	1814
		th	6
		ai ph	

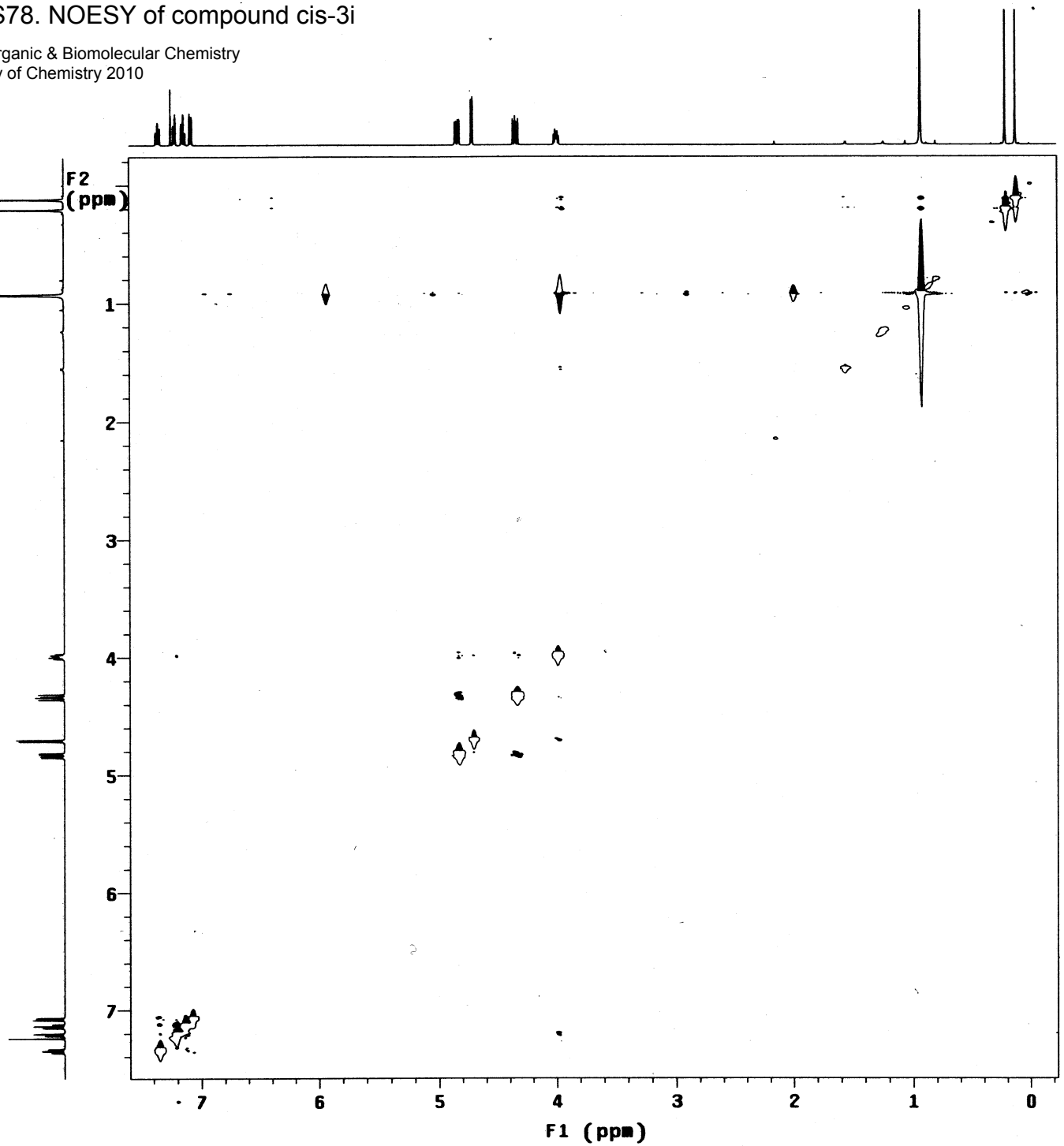


Fig S79. 1H NMR (CDCl3, 500 MHz) of compound trans-3i


```
PMK-02-377-f2
exp23  s2pul
      SAMPLE
date    Aug  4 2010
solvent       cdcl3
file            exp
   ACQUISITION
sfrq         499.830
tn                H1
at            3.000
np            48000
sw           8000.0
fb         not used
bs                4
tpwr             58
pw              4.8
d1            1.000
tof           499.7
nt                4
ct                4
alock             y
gain       not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -250.1
wp           5498.0
vs              150
sc                0
wc              210
hzmm          26.18
is           238.74
rfl          4638.7
rfp          3618.7
th                1
ins          100.000
nm  cdc  ph
      DEC. & VT
dfrq         125.693
dn               C13
dpwr              30
dof                0
dm               nnn
dmm                c
dmf              200
dseq
dres             1.0
homo               n
temp            23.0
     PROCESSING
wtfile
proc              ft
fn         not used
math               f

werr           react
wexp       procplot
wbs
wnt              wft
```

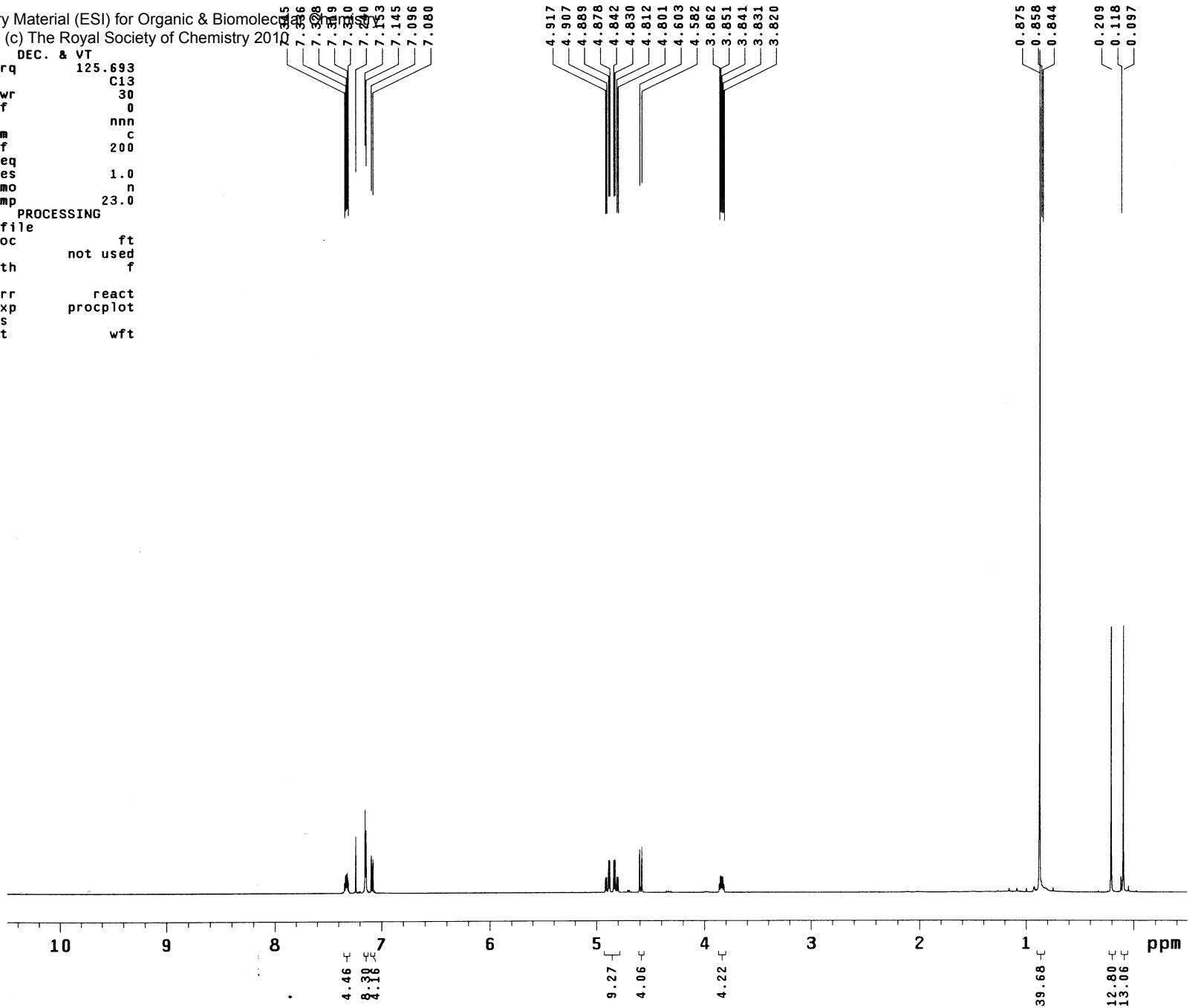


Fig S80. 13C NMR (CDCl3, 125 MHz) of compound trans-3i

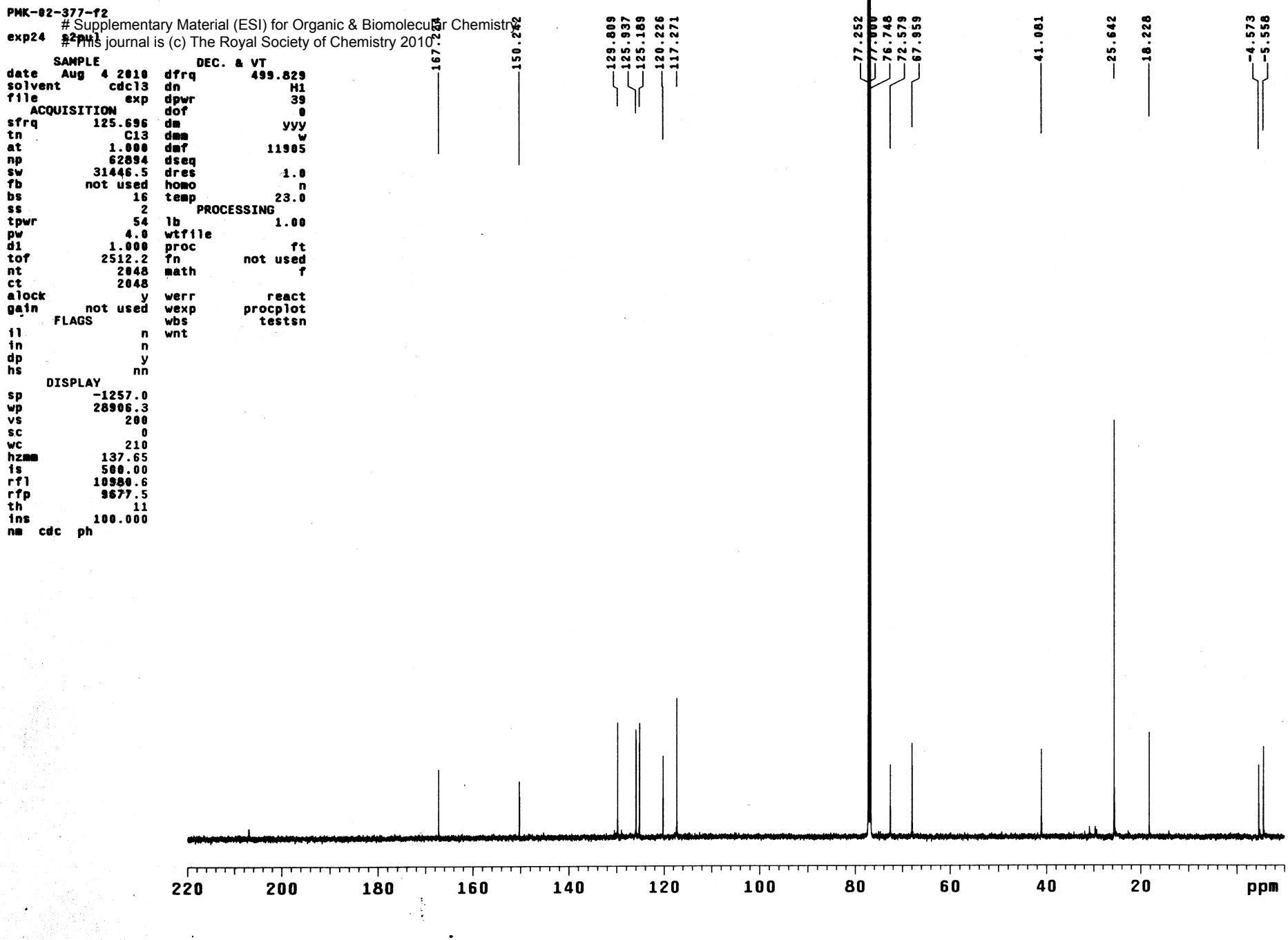


Fig S81. DEPT of compound trans-3i


```
exp25  DEPT
      SAMPLE                    DEPT                 ACQUISITION ARRAYS
date    Aug  4 2010   jlxh          140.0   array          mult
solvent       cdcl3   mult        arrayed   arraydim          3
sample    undefined         SPECIAL
   ACQUISITION          temp          23.0   i              mult
sw          31446.5    gain            54   1               0.5
at            1.000    spin             0   2                 1
np            62894         PROCESSING      3               1.5
bs               16    lb             1.00
ss               -4    fn         not used
d1            1.000         SPECTRUM
nt             1024    wp          28906.3
ct             1024    sp          -1257.0
   TRANSMITTER         rp            129.7
tn               C13   lp            228.6
tof           2512.2   ai  cdc  ph
tpwr              54        REFERENCE
pw            11.500   rfl           1303.1
    DECOUPLER          rfp              0
dn                H1            PLOT
dof               0    wc             210
dpwr-            39    sc               0
dm              nny    vs             200
dmm             ccw    hzmm        137.65
dmf           11905    th               68
pplvl            51
pp            28.000
```

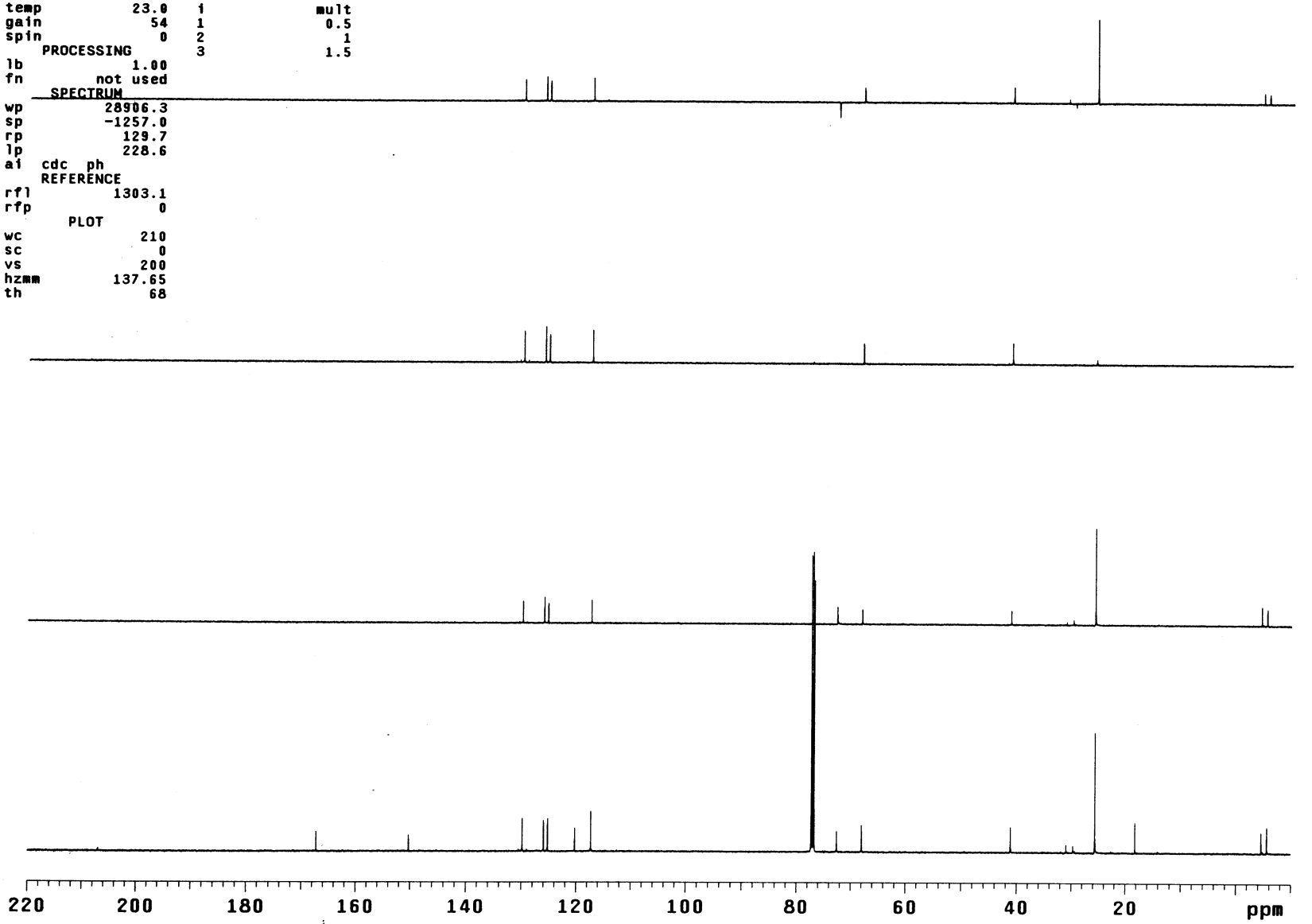


Fig S82. HSQC of compound trans-3i

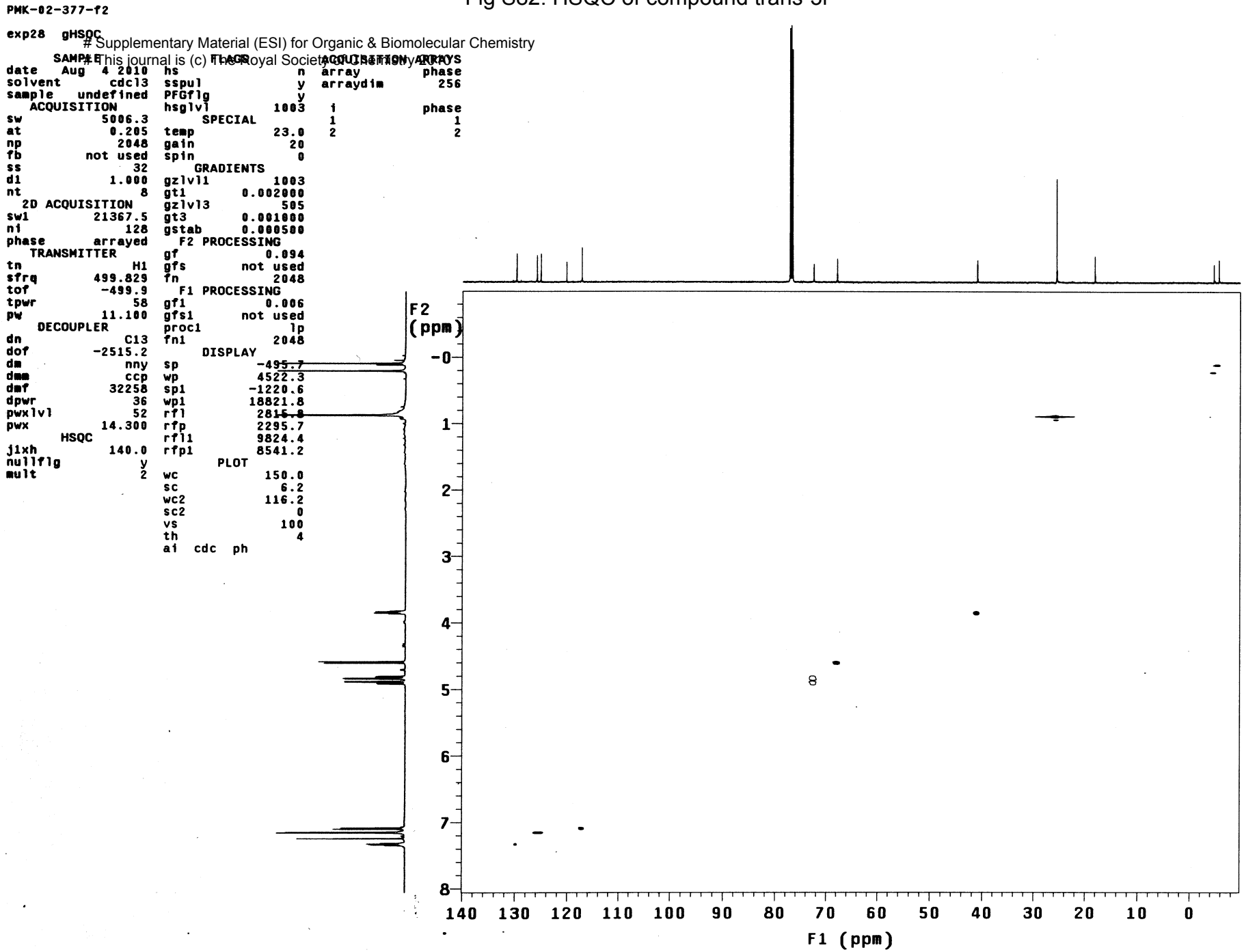


Fig S83. COSY of compound trans-3i

PMK-02-377-f2
exp26 gCOSY

SAMPLE		FLAGS	
date	Aug 4 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	5006.3	temp	23.0
at	0.205	gain	30
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.102
d1	1.000	sbs	not used
nt	8	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	5006.3	sb1	-0.026
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.829	DISPLAY	
tof	-499.9	sp	-131.9
tpwr	58	wp	3916.0
pw	11.100	sp1	-126.3
GRADIENTS		wp1	3911.1
gzlvl1	1003	rfl	2818.8
gt1	0.001000	rfp	2295.7
gstab	0.000500	rfl1	2818.0
DECOUPLER		rfp1	2295.7
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	3
		ai cdc av	

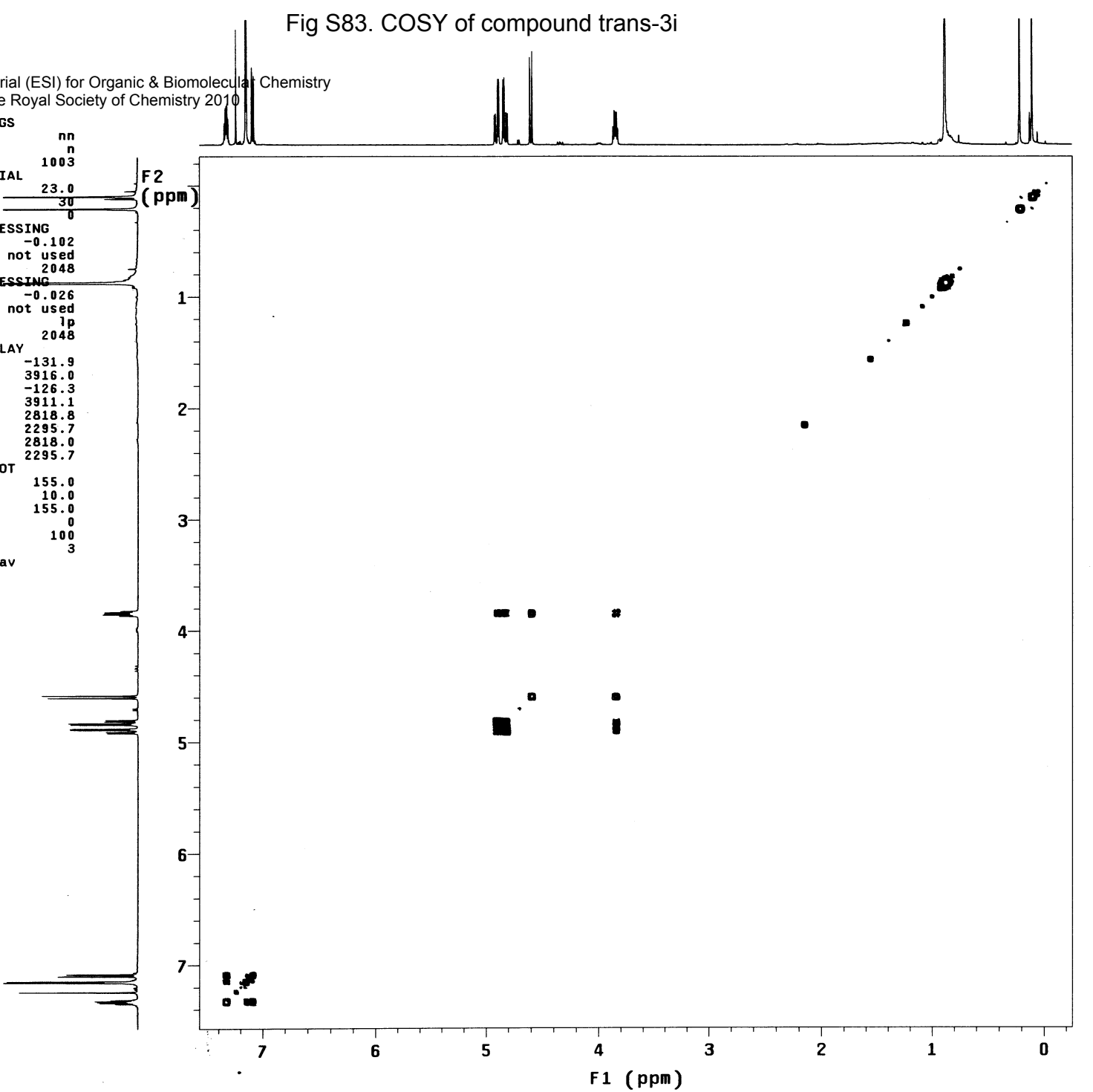


Fig S84. NOESY of compound trans-3i

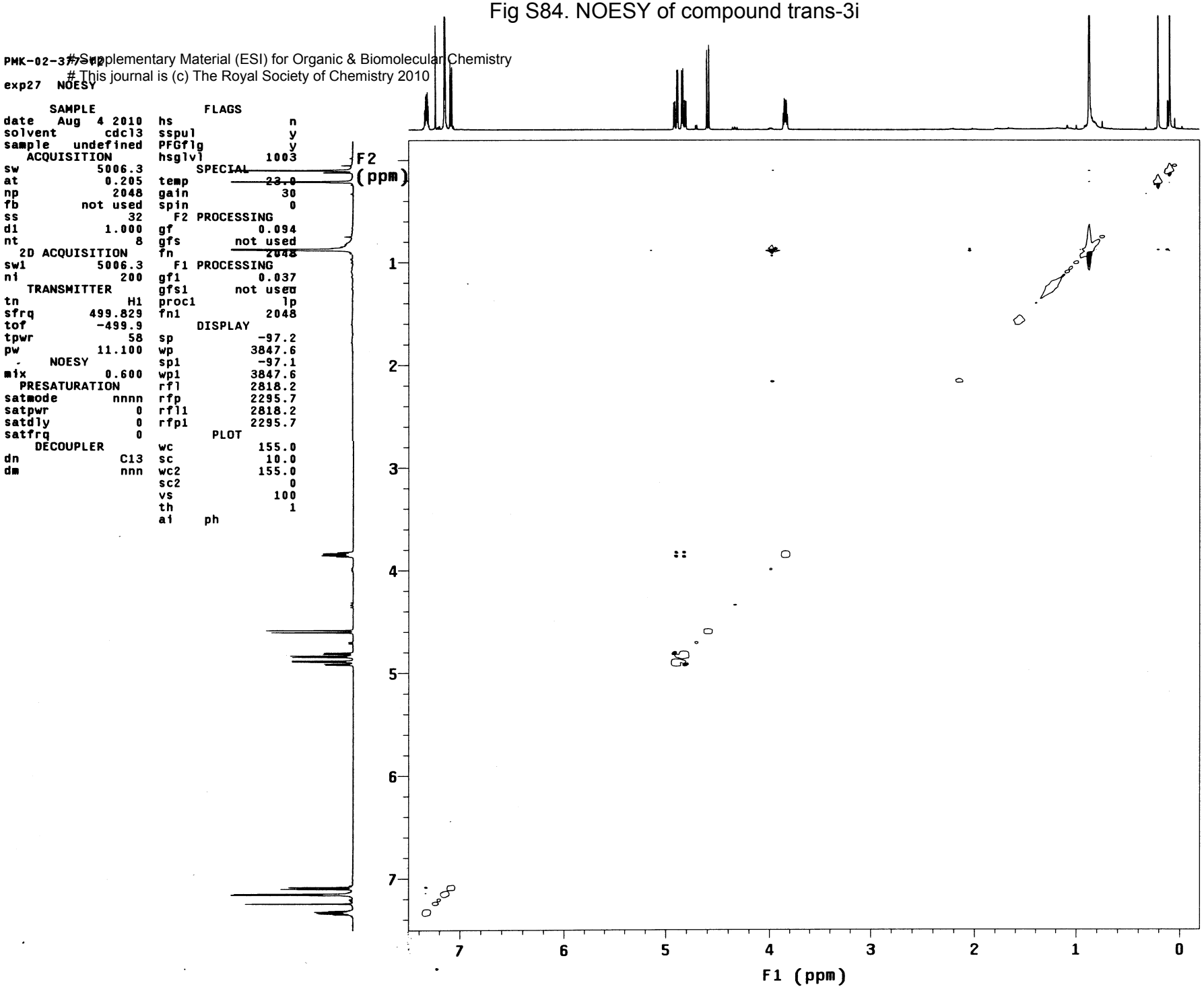


Fig S85. 1H NMR (CDCl3, 500 MHz) of compound 3j

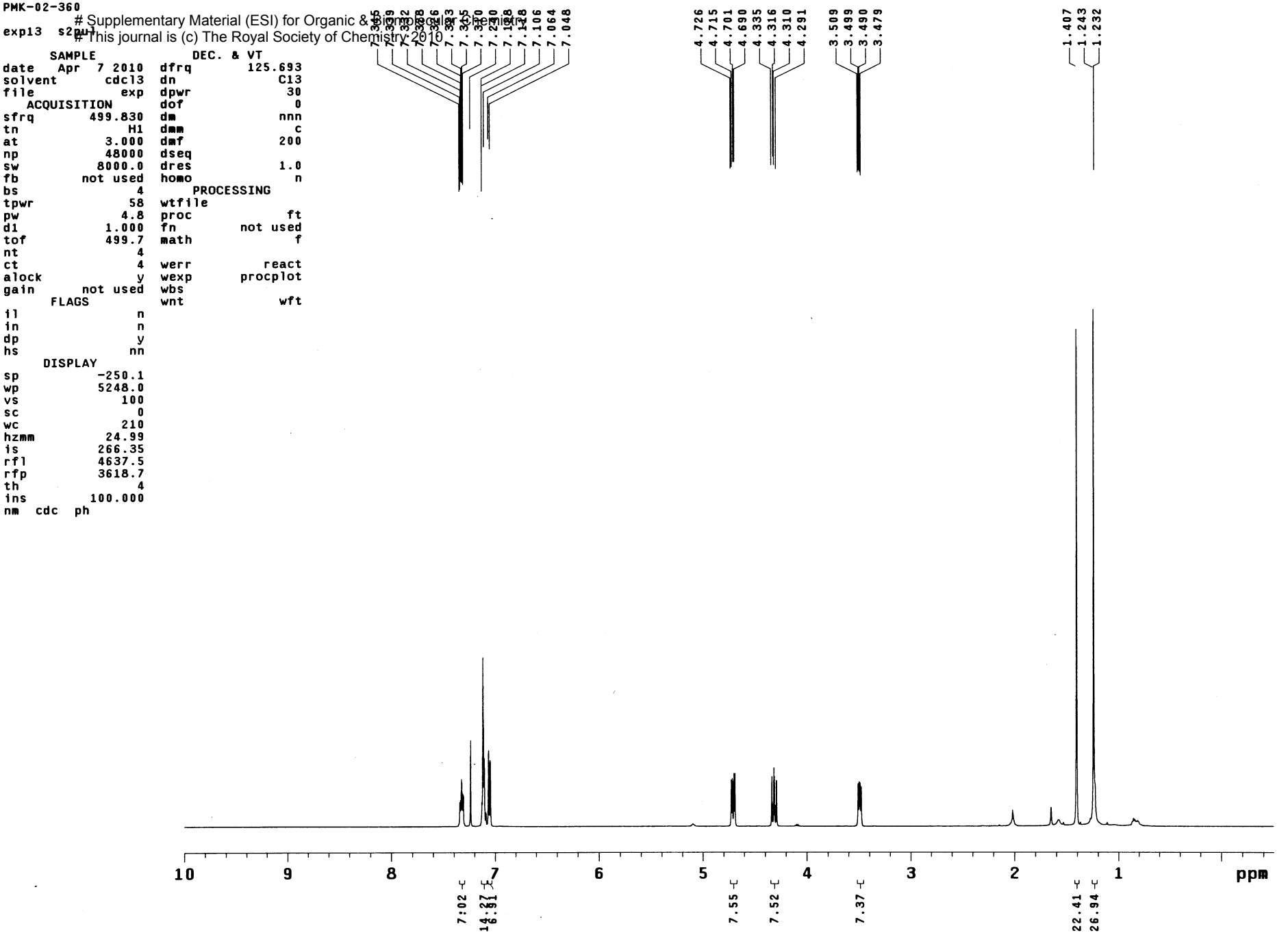


Fig S86. 13C NMR (CDCl3, 125 MHz) of compound 3j

PMK-02-360

exp14 s2pul

SAMPLE		DEC. & VT	
date	Apr 7 2010	dfrq	499.829
solvent	cdcl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	125.696	dm	yyy
tn	C13	dmm	w
at	1.000	dmf	11905
np	62894	dseq	
sw	31446.5	dres	1.0
fb	not used	homo	n
bs	16	PROCESSING	
ss	2	lb	1.00
tpwr	54	wtfile	
pw	4.0	proc	ft
d1	1.000	fn	not used
tof	2512.2	math	f
nt	2048		
ct	2048	werr	react
alock	y	wexp	procplot
gain	not used	wbs	testsn
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1257.0		
wp	28906.3		
vs	100		
sc	0		
wc	210		
hzmm	137.65		
is	500.00		
rfl	10983.4		
rfp	9677.5		
th	6		
ins	100.000		
nm cdc ph			

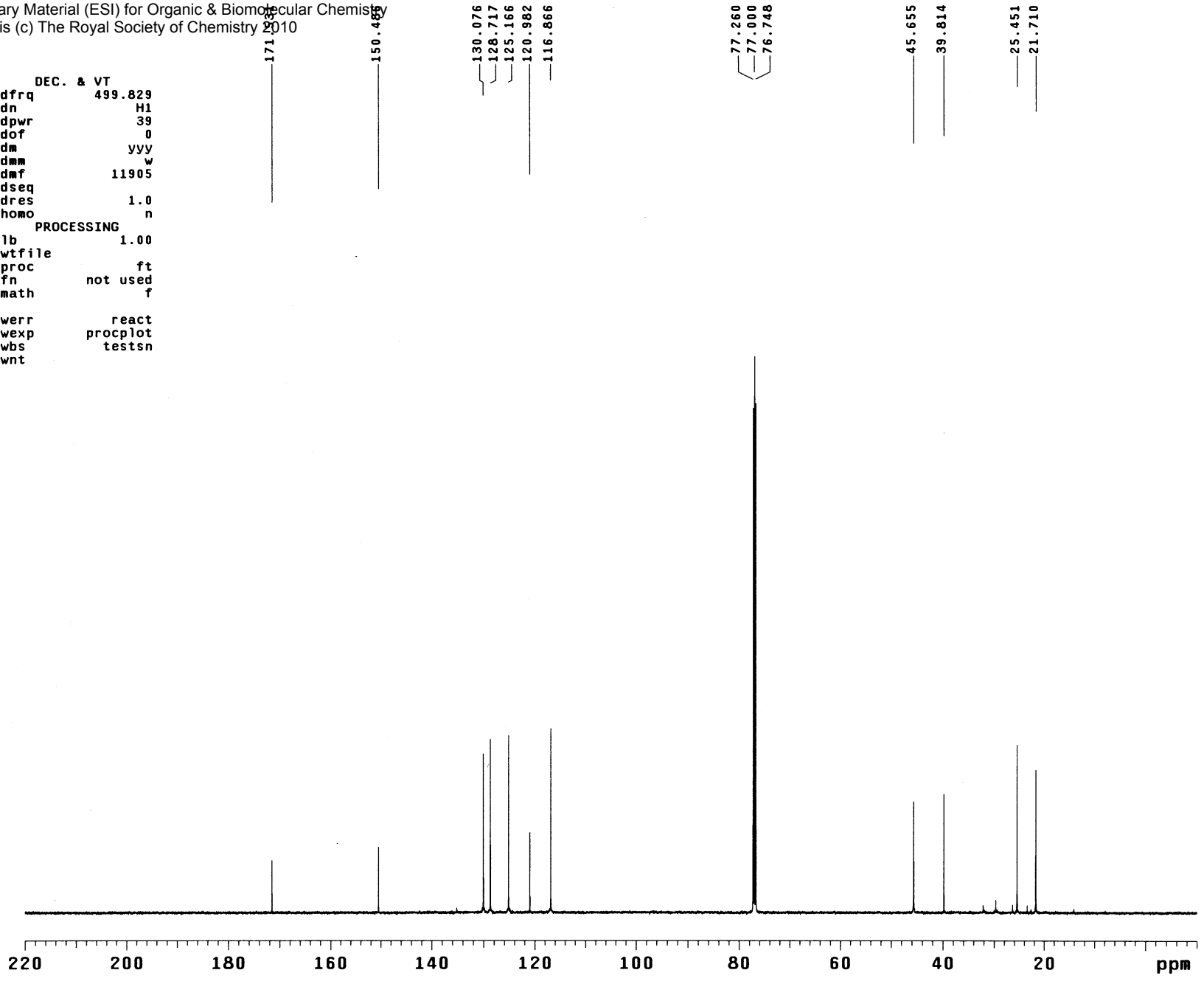


Fig S87. DEPT of compound 3j

PMK-02-360

exp15 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 7 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	28906.3		
ct	2048	sp	-1257.0		
TRANSMITTER		rp	48.8		
tn	C13	lp	201.1		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1306.9		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	100		
dmm	ccw	hzmm	137.65		
dmf	11905	th	7		
pplvl	51				
pp	25.600				

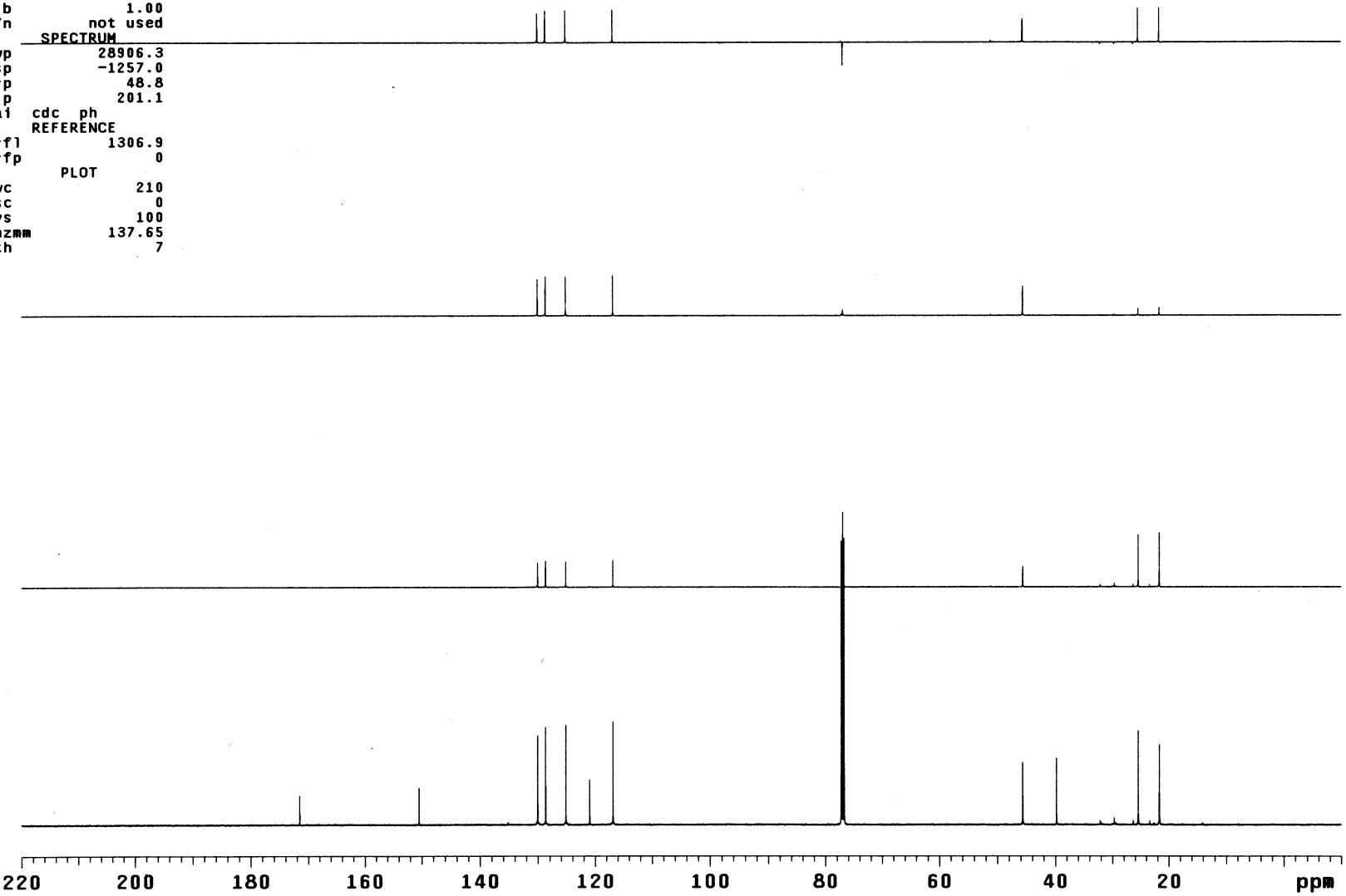


Fig S88. 13C of compound 3j (Extracted from DEPT)

PMK-02-360

exp15 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 7 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	28906.3		
ct	2048	sp	-1257.0		
TRANSMITTER		rp	48.8		
tn	C13	lp	201.1		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1306.9		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	300		
dmm	ccw	hzmm	137.65		
dmf	11905	th	7		
pplvl	51				
pp	25.600				

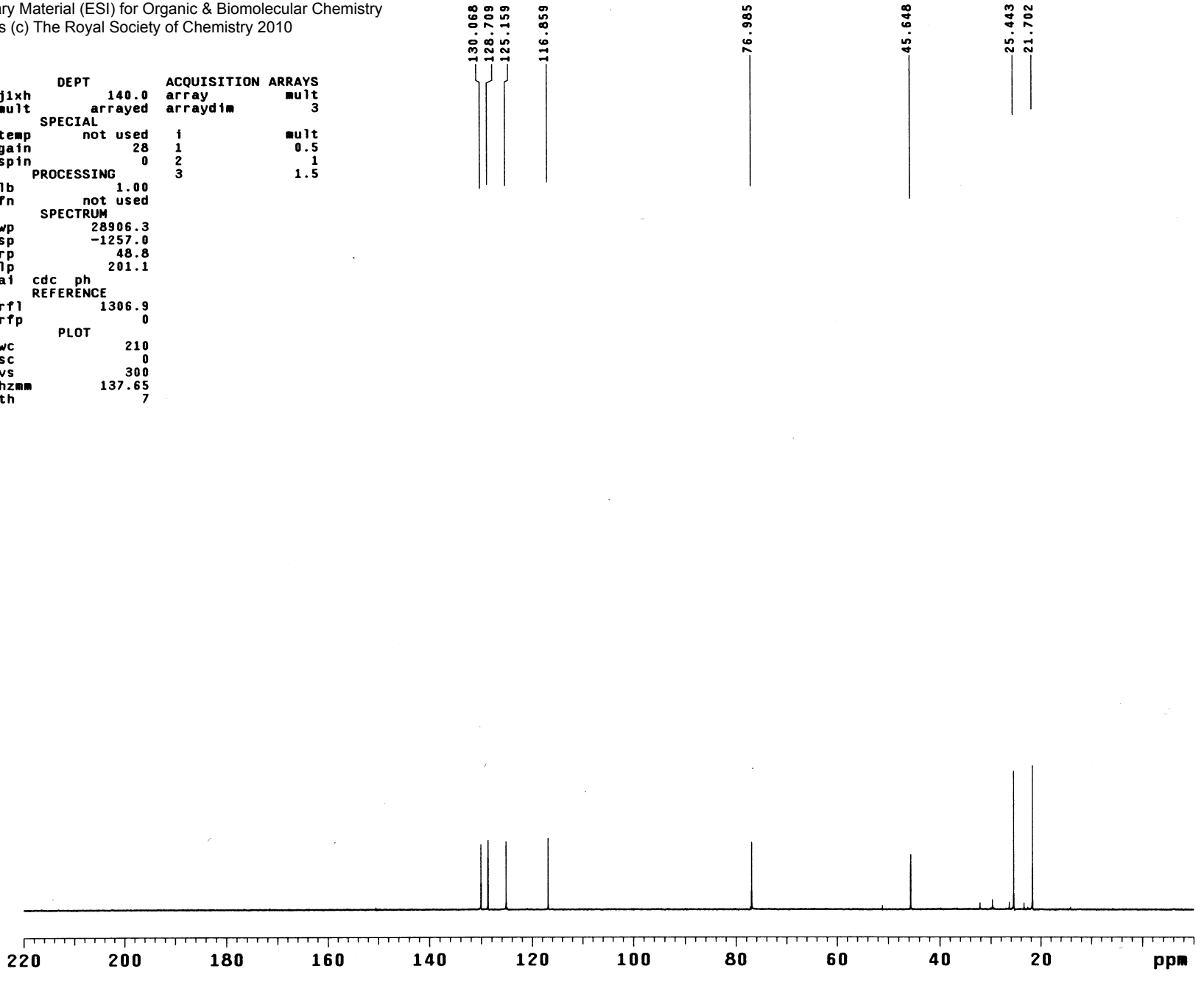


Fig S89. 13C of compound 3j (Extracted from DEPT, Expand.)

exp15 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 7 2010	jlxh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	31446.5	gain	28	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	1759.1		
ct	2048	sp	8797.5		
TRANSMITTER		rp	48.8		
tn	C13	lp	201.1		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1306.9		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	500		
dmm	ccw	hzmm	8.38		
dmf	11905	th	7		
pplvl	51				
pp	25.600				

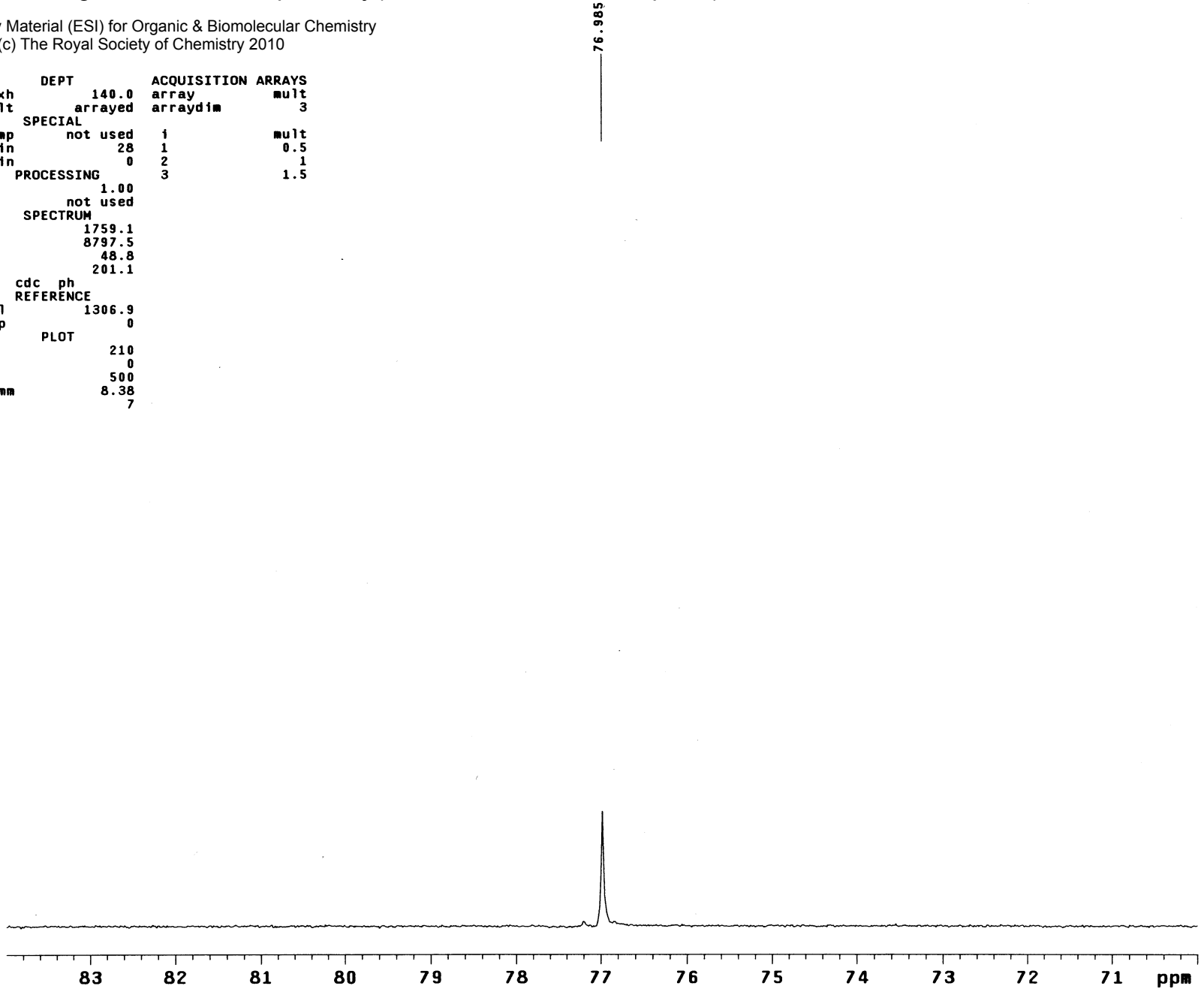


Fig S90. HSQC of compound 3j

PMK-02-360

exp18 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Apr 7 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	not used	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	482.0		
dmm	ccp	wp	3455.5		
dmf	32258	sp1	2085.5		
dpwr	36	wp1	14961.4		
pwxlvl	52	rf1	716.8		
pwx	14.300	rfp	703.3		
HSQC		rfl1	4002.6		
j1xh	140.0	rfp1	2728.5		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	4		
		ai cdc ph			

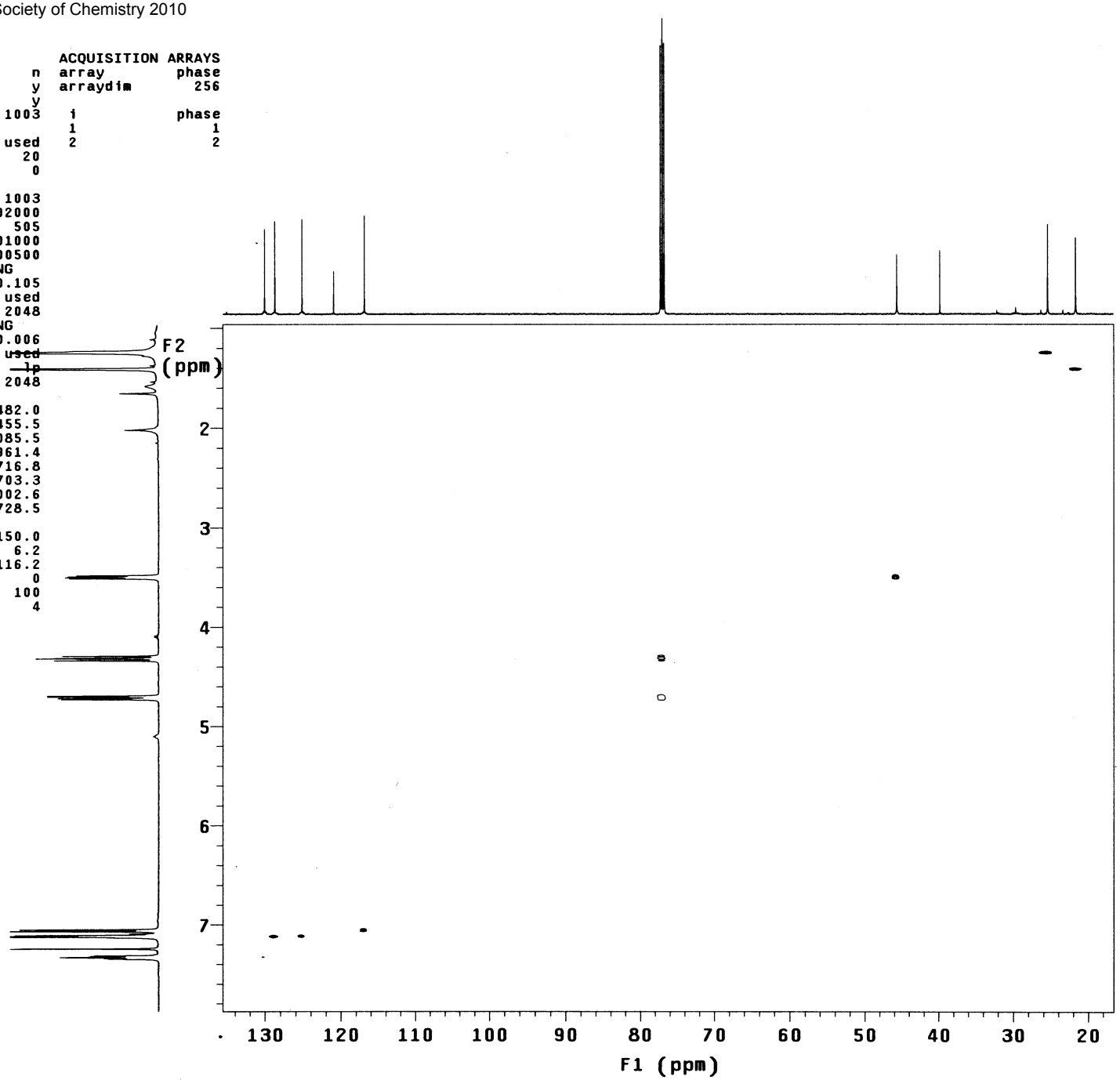


Fig S91. COSY of compound 3j

PMK-02-360
exp16 gCOSY

SAMPLE	
date	Apr 7 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	16
d1	1.000
nt	8
2D ACQUISITION	
sw1	4490.3
ni	128
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
GRADIENTS	
gzlvl1	1003
gt1	0.001000
gstab	0.000500
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	nn
sspul	n
hsglvl	1003
SPECIAL	
temp	not used
gain	28
spin	0
F2 PROCESSING	
sb	-0.114
sbs	not used
fn	2048
F1 PROCESSING	
sb1	-0.029
sbs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	266.5
wp	3543.2
sp1	262.6
wp1	3543.2
rfl	717.4
rfp	703.3
rfl1	716.9
rfp1	703.3
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	
vs	100
th	4
ai cdc av	

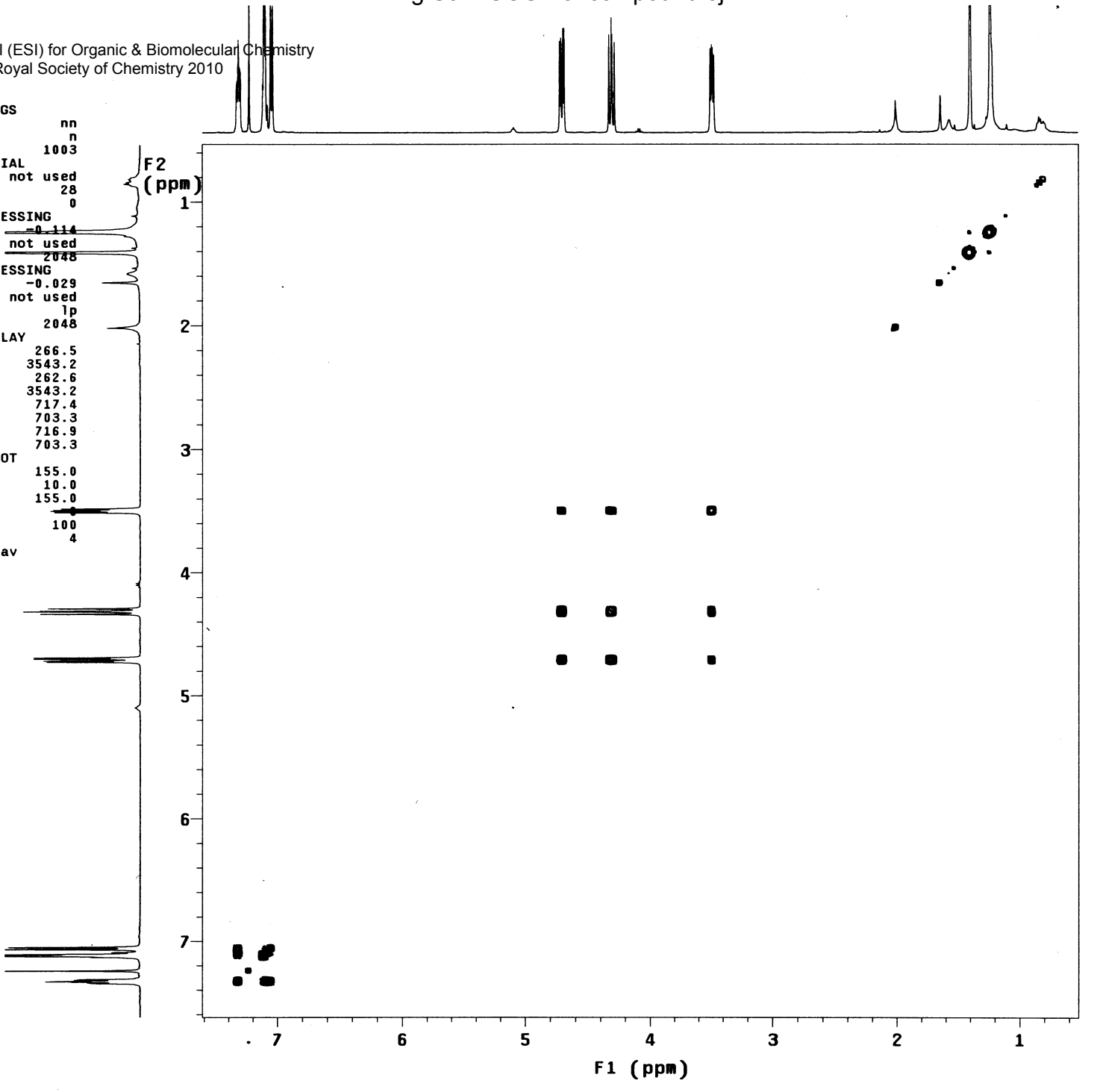


Fig S92. NOESY of compound 3j

PMK-02-360

exp17 NOESY

SAMPLE		FLAGS	
date	Apr 7 2010	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	4490.3	SPECIAL	
at	0.228	temp	not used
np	2048	gain	28
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.105
nt	8	gfs	not used
2D ACQUISITION		fn	2048
sw1	4490.3	F1 PROCESSING	
ni	200	gf1	0.041
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.829	fn1	2048
tof	-250.0	DISPLAY	
tpwr	58	sp	312.7
pw	11.100	wp	3499.3
NOESY		sp1	311.4
mix	0.600	wp1	3499.3
PRESATURATION		rfl	715.0
satmode	nnnn	rfp	703.3
satpwr	0	rfl1	716.3
satdly	0	rfp1	703.3
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	100
		th	1
		ai ph	

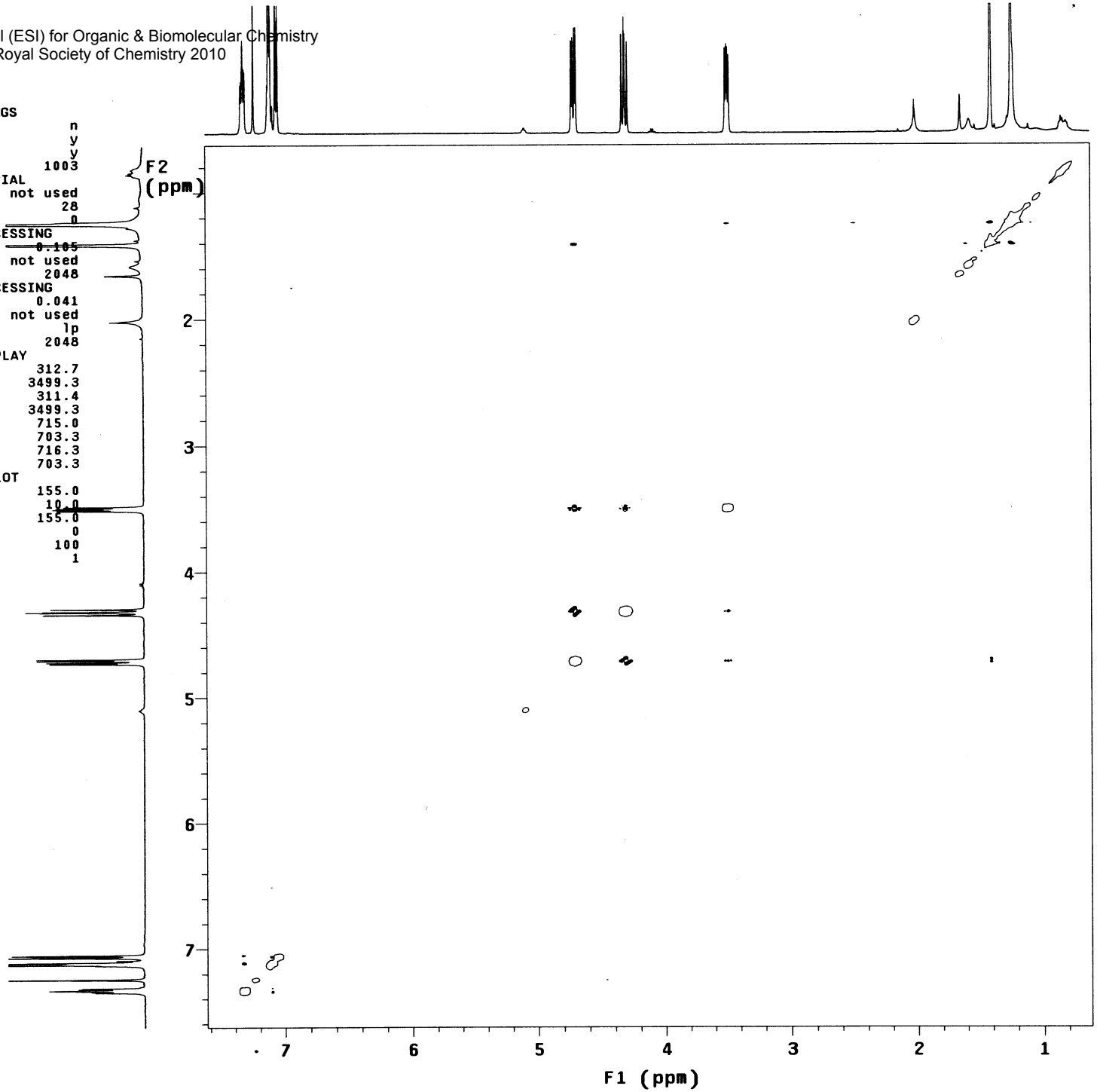
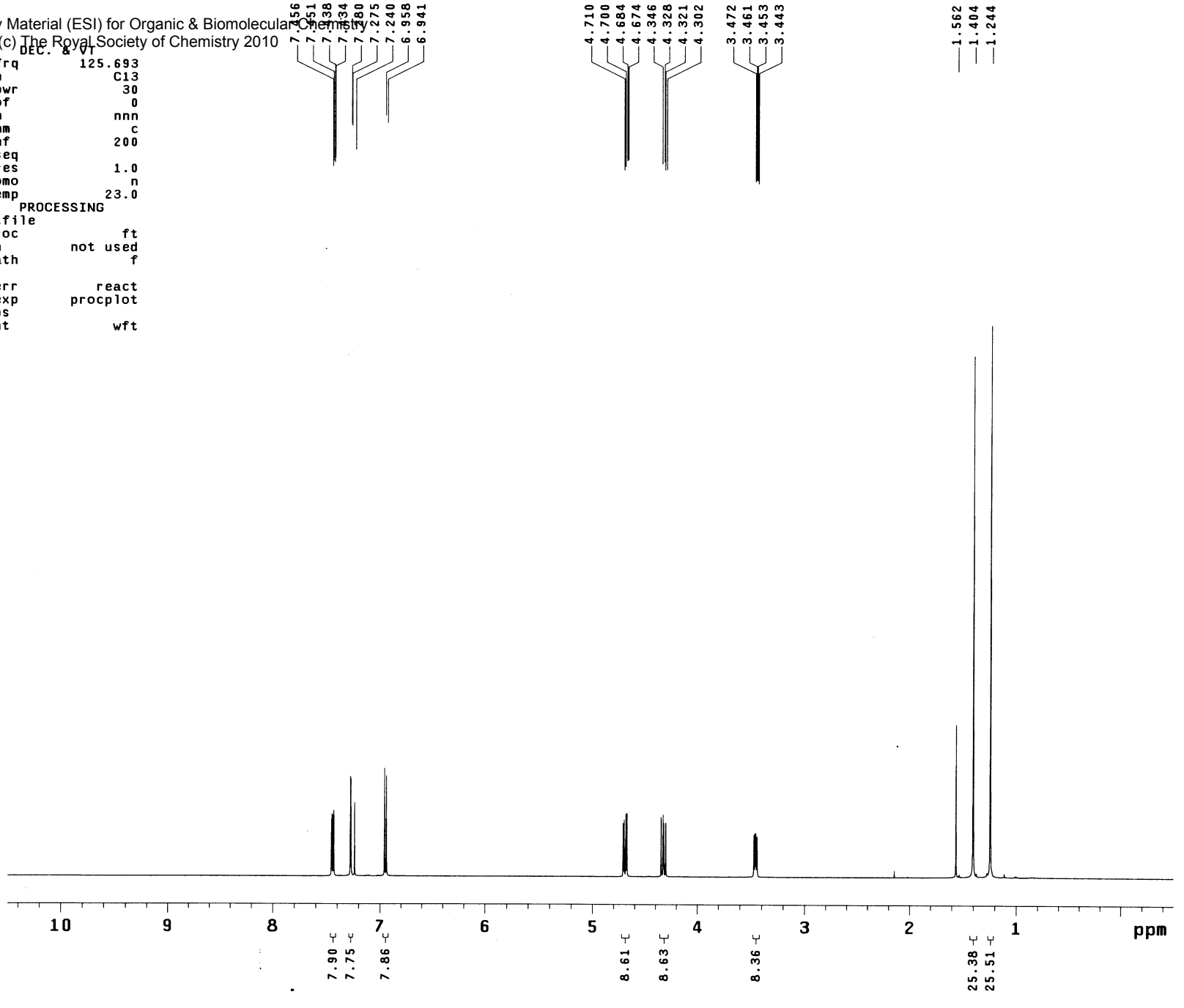


Fig S93. 1H NMR (CDCl3, 500 MHz) of compound 3k


```
PMK-02-394

exp3  s2pul
        SAMPLE
date    Aug  7 2010
solvent       cdcl3
file            exp
    ACQUISITION
sfrq        499.830
tn               H1
at             3.000
np             48000
sw            8000.0
fb         not used
bs                 4
tpwr              58
pw               4.8
d1             1.000
tof            499.7
nt                 4
ct                 4
alock              y
gain       not used
       FLAGS
il                 n
in                 n
dp                 y
hs                nn
      DISPLAY
sp            -250.1
wp            5498.0
vs               100
sc                 0
wc               210
hzmm           26.18
is            274.36
rfl           4638.7
rfp           3618.7
th                 4
ins          100.000
nm   cdc   ph
        DEC. & VT
dfrq        125.693
dn              C13
dpwr             30
dof               0
dm              nnn
dmm               c
dmf             200
dseq
dres            1.0
homo              n
temp           23.0
     PROCESSING
wtfile
proc             ft
fn         not used
math              f

werr          react
wexp       procplot
wbs
wnt             wft
```



PMK-02-394
exp4 s2pul

SAMPLE		DEC. & VT	
date	Aug 7 2010	dfrq	499.829
solvent	cdcl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	125.696	dm	yyy
tn	C13	dmm	w
at	1.000	dmf	11905
np	62894	dseq	
sw	31446.5	dres	1.0
fb	not used	homo	n
bs	16	temp	23.0
ss	2	PROCESSING	
tpwr	54	lb	1.00
pw	4.0	wtfile	
d1	1.000	proc	ft
tof	2512.2	fn	not used
nt	2048	math	f
ct	2048		
alock	y	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1257.0		
wp	28906.3		
vs	200		
sc	0		
wc	210		
hzmm	137.65		
is	500.00		
rfl	10982.5		
rfp	9677.5		
th	10		
ins	100.000		
nm	cdc ph		

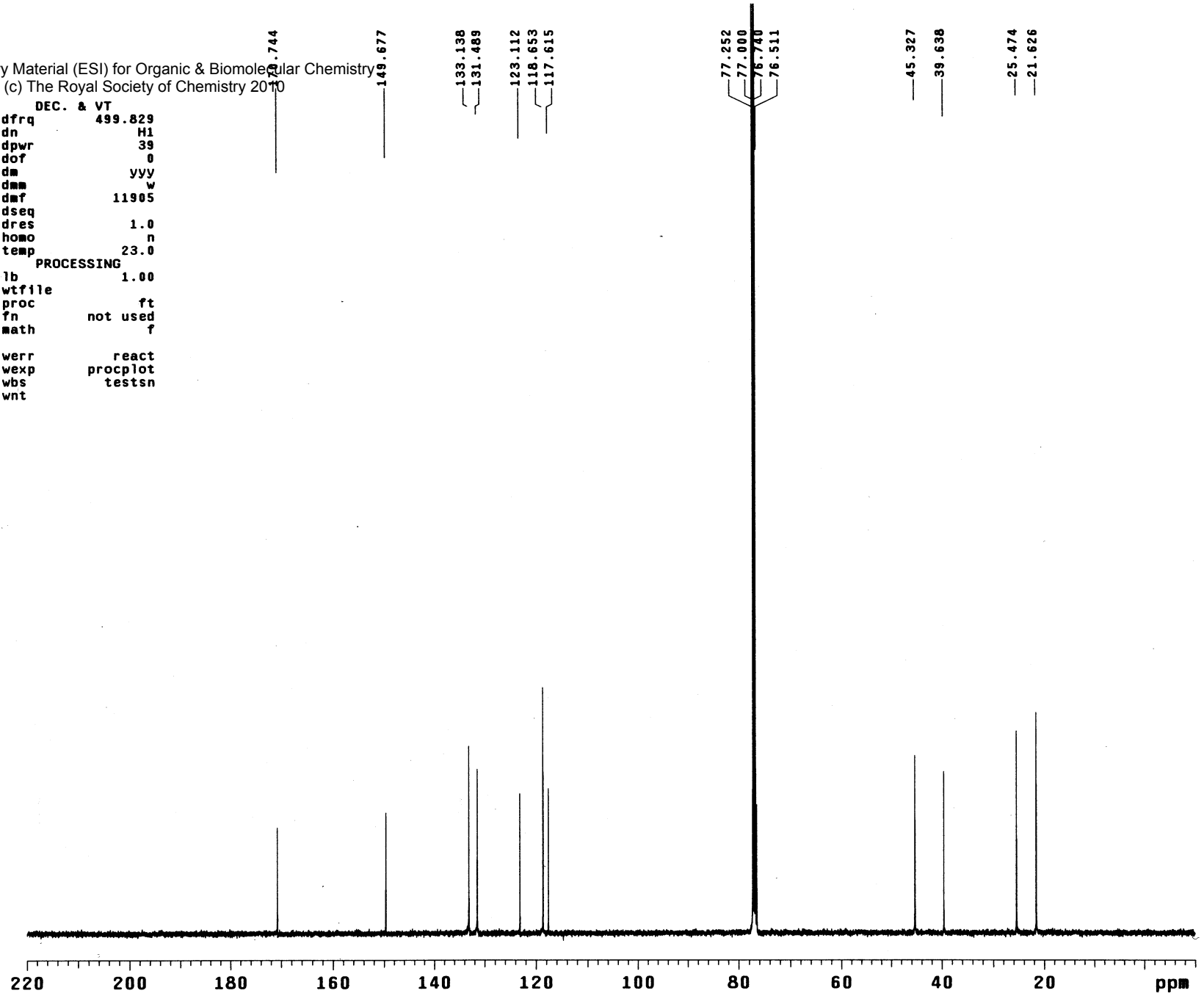


Fig S94. 13C NMR (CDCl3, 125 MHz) of compound 3k

exp5 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Aug 7 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	23.0	i	mult
sw	31446.5	gain	30	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	2048	wp	28906.3		
ct	2048	sp	-1257.0		
TRANSMITTER		rp	129.9		
tn	C13	lp	215.7		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1305.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	150		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

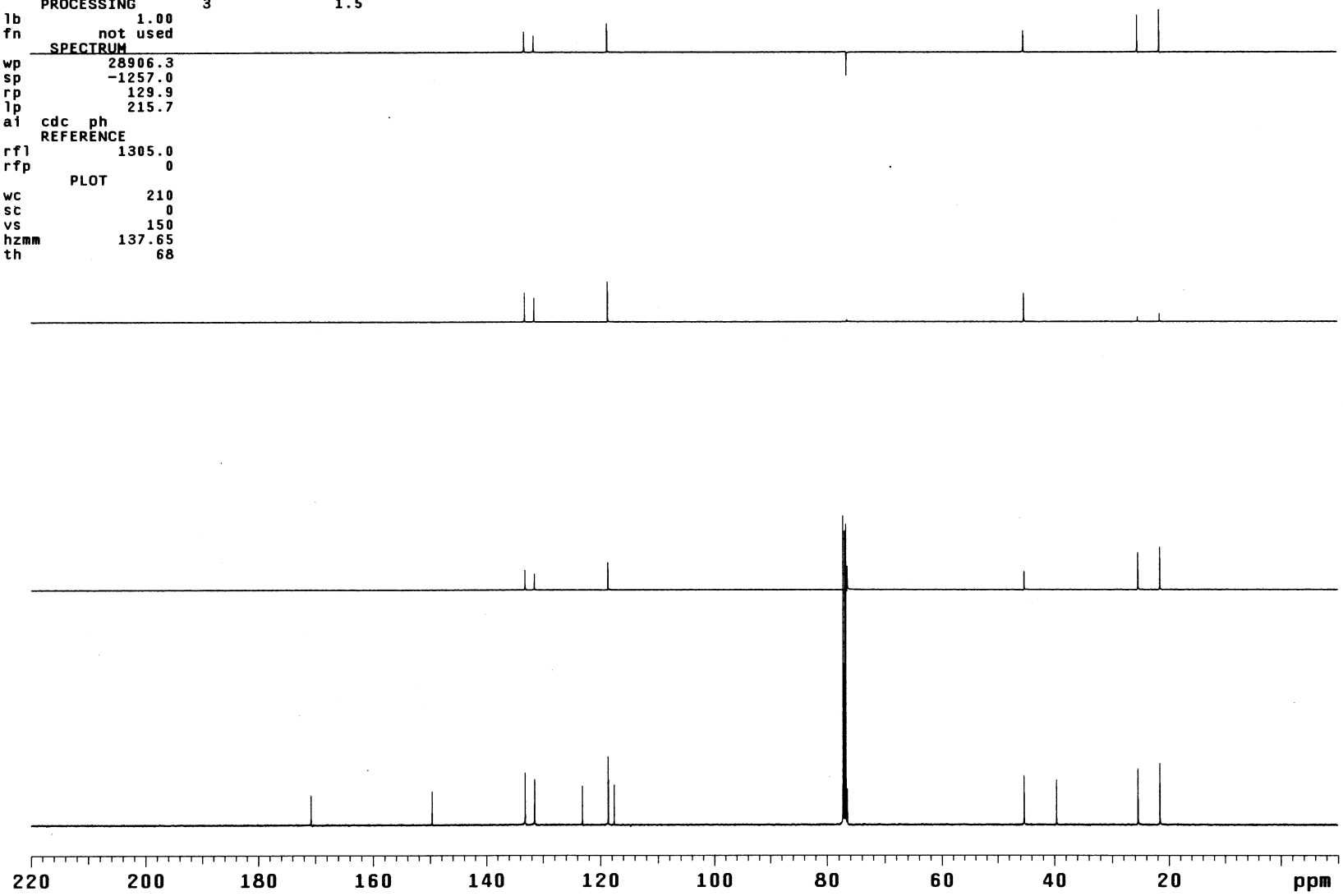


Fig S96. HSQC of compound 3k

PMK-02-394

exp8 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Aug 7 2010	ns	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	23.0	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.829	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	230.9		
dmm	ccp	wp	3753.6		
dmf	32258	sp1	1279.8		
dpwr	36	wp1	18446.2		
pwxlvl	52	rf1	2359.8		
pwx	14.300	rfp	2345.2		
HSQC		rfl1	10902.9		
j1xh	140.0	rfp1	9616.0		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	4		
		ai cdc ph			

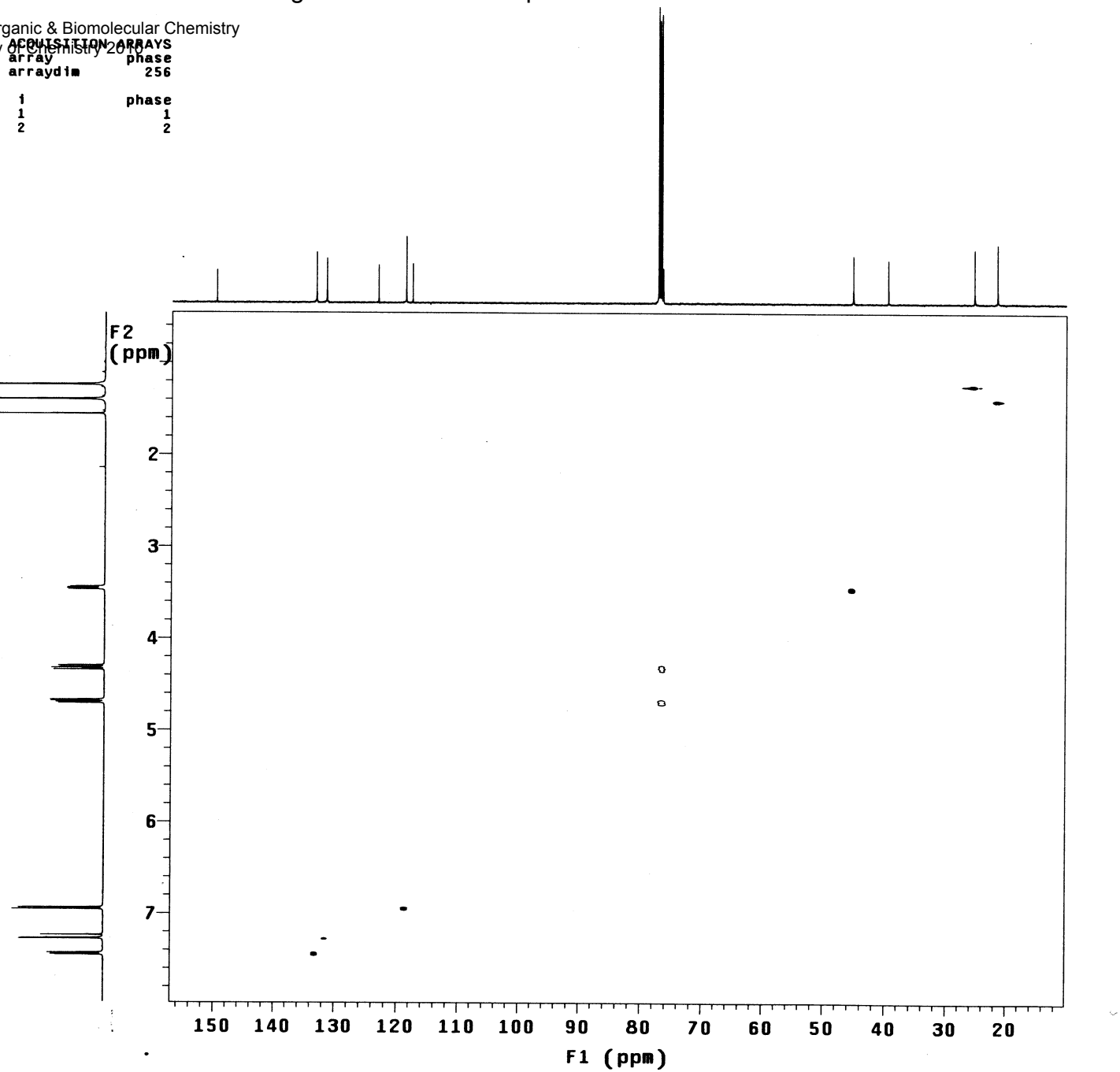


Fig S97. COSY of compound 3k

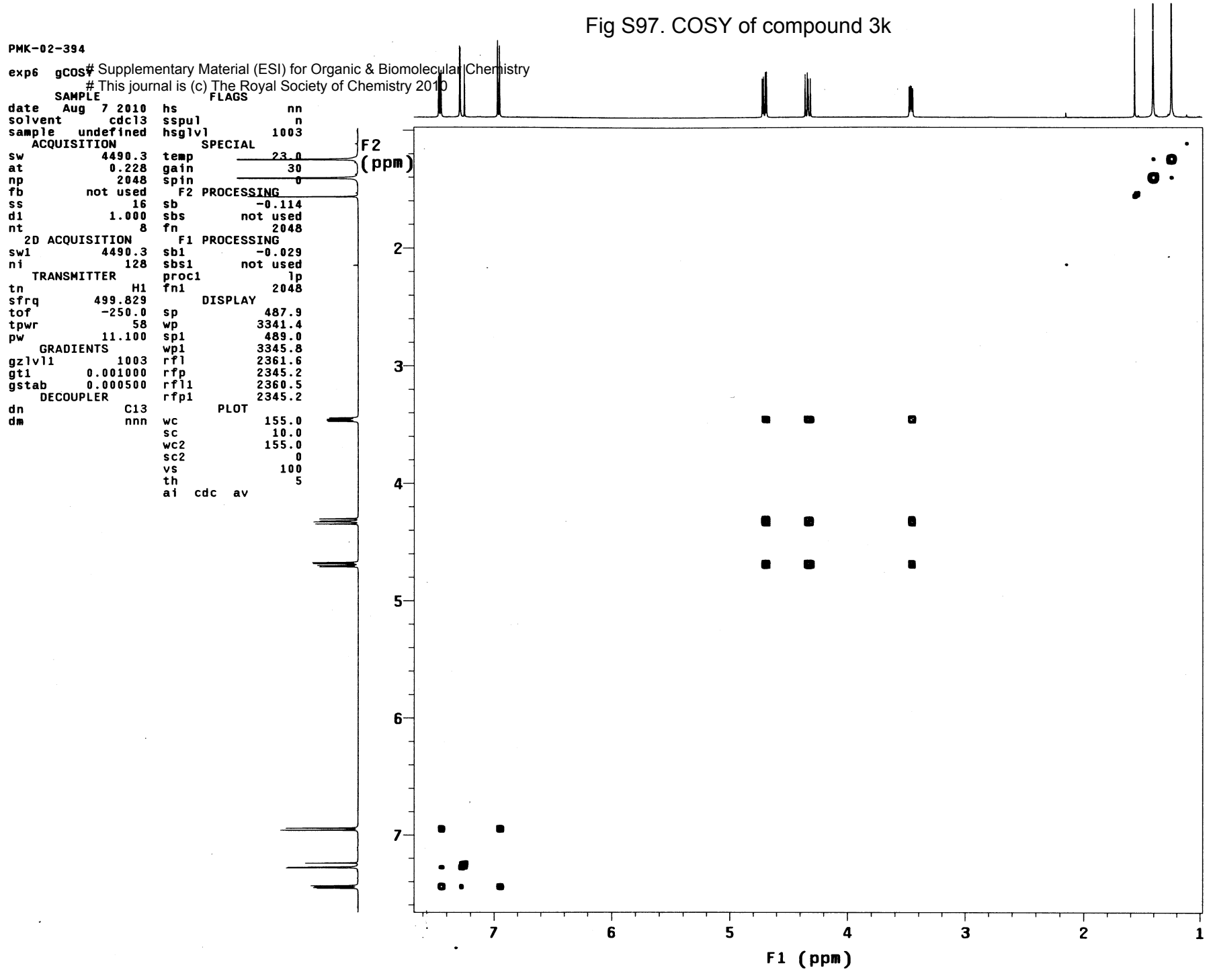


Fig S98. NOESYof compound 3k

PMK-02-394

exp7 NOESY

SAMPLE	
date	Aug 7 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4490.3
at	0.228
np	2048
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	4490.3
ni	200
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-250.0
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	23.0
gain	30
spin	0
F2 PROCESSING	
gf	0.105
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.041
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	472.2
wp	3359.0
sp1	473.0
wp1	3354.6
rfl	2359.7
rfp	2345.2
rfl1	2358.9
rfp1	2345.2
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	1
ai ph	

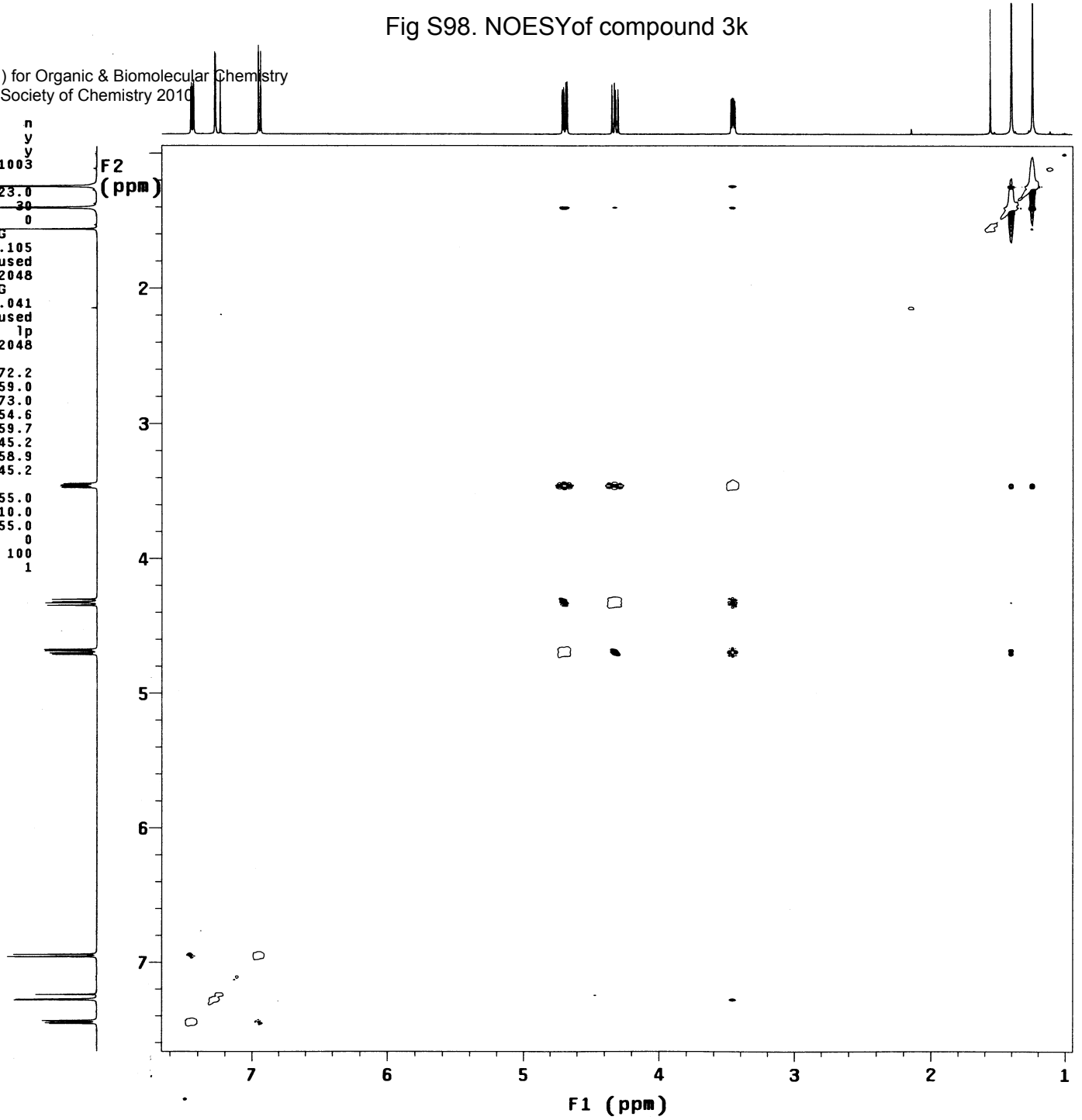
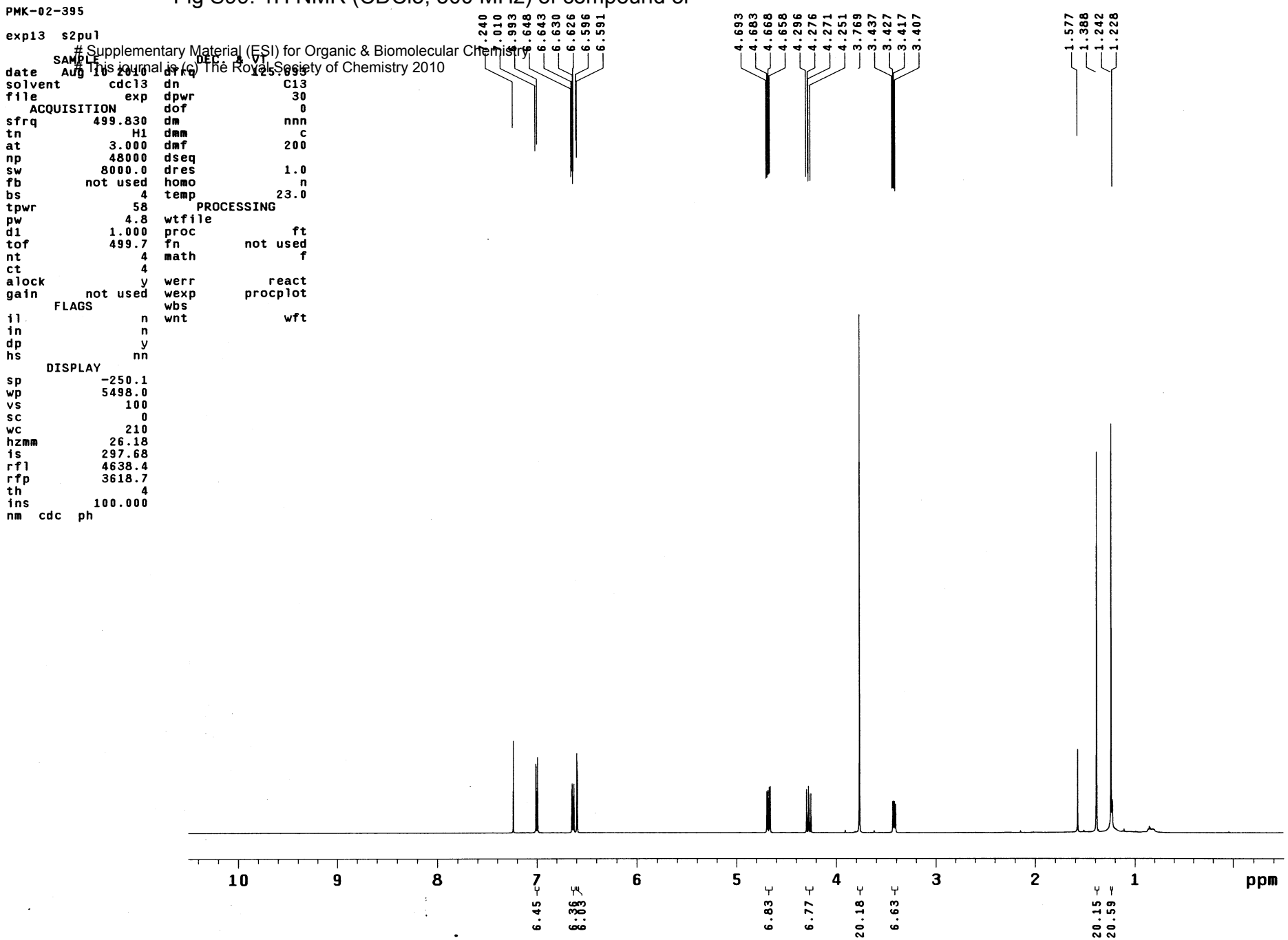
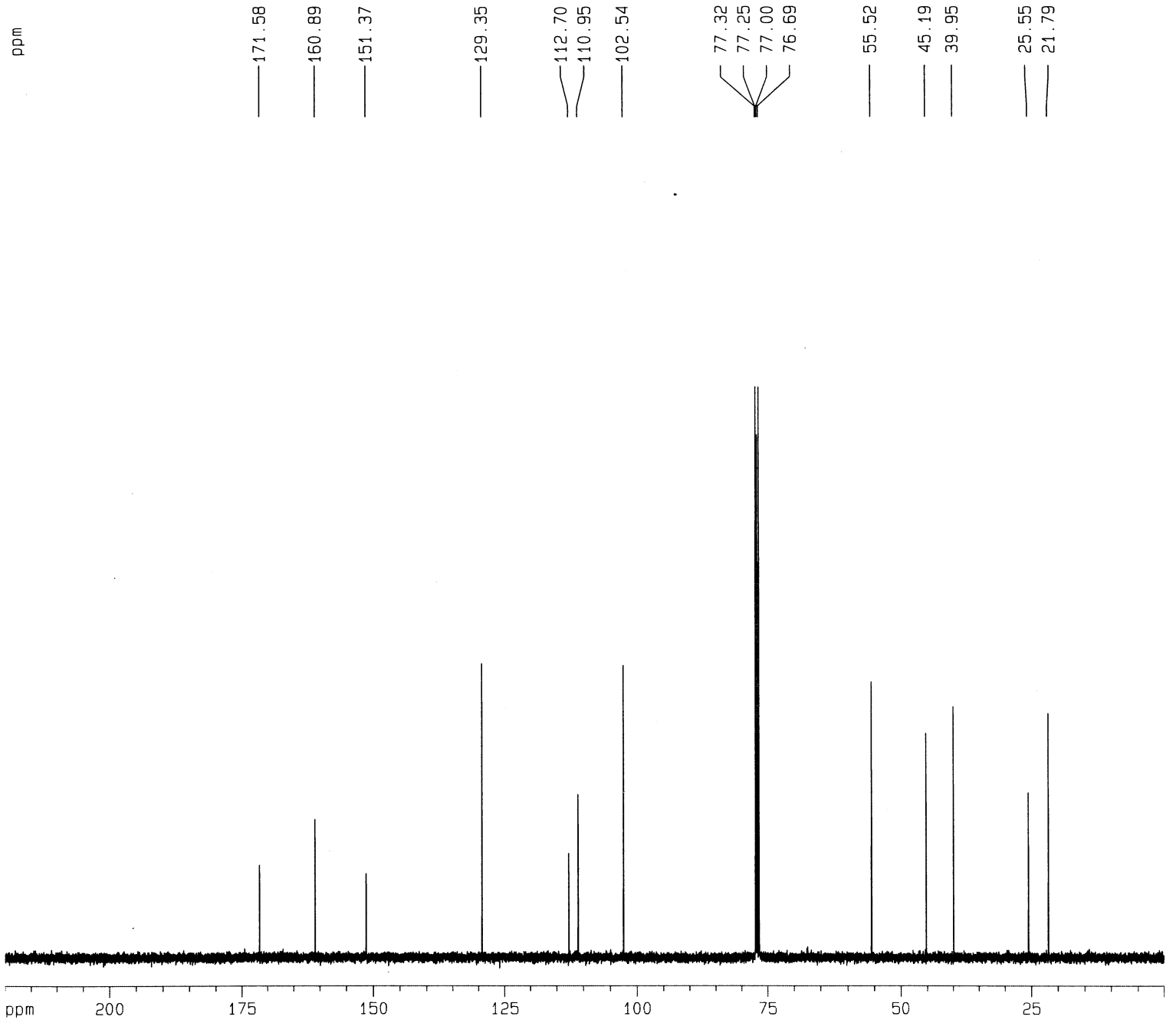


Fig S99. 1H NMR (CDCl3, 500 MHz) of compound 3l



C13 spectrum of

Fig S100. 13C NMR (CDCl3, 100 MHz) of compound 3l

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EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100907
Time               12.51
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               10.00 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             15.60 dB
PL13             18.60 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127731 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
```

Fig S101. DEPT of compound 3l

PMK-02-395
exp15 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Aug 10 2010	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	23.0	i	mult
sw	31446.5	gain	24	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	512	wp	28906.3		
ct	512	sp	-1257.0		
TRANSMITTER		rp	122.7		
tn	C13	lp	233.3		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1305.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	200		
dmm	ccw	hzmm	137.65		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

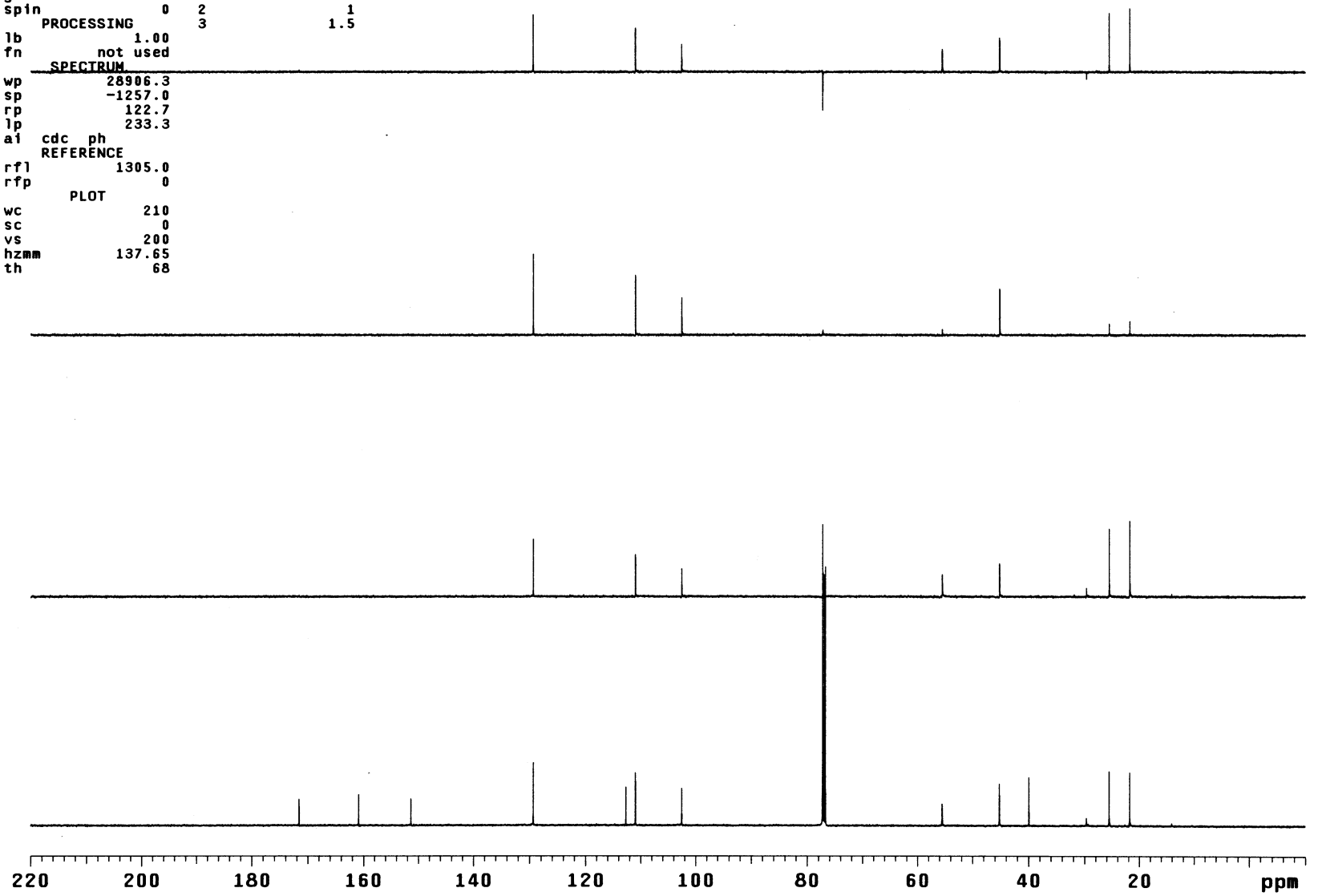


Fig S102. HSQC of compound 3l

PMK-02-395

exp18 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Aug 10 2010	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	8000.0	SPECIAL		1	1
at	0.128	temp	23.0	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.830	fn	2048		
tof	499.7	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.2	DISPLAY			
dm	nny	sp	96.9		
dmm	ccp	wp	3882.8		
dmf	32258	sp1	1186.9		
dpwr	36	wp1	16401.2		
pwx1vl	52	rf1	3156.6		
pwx	14.300	rfp	2136.3		
HSQC		rfl1	11005.4		
j1xh	140.0	rfp1	9709.2		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	4		
		ai cdc ph			

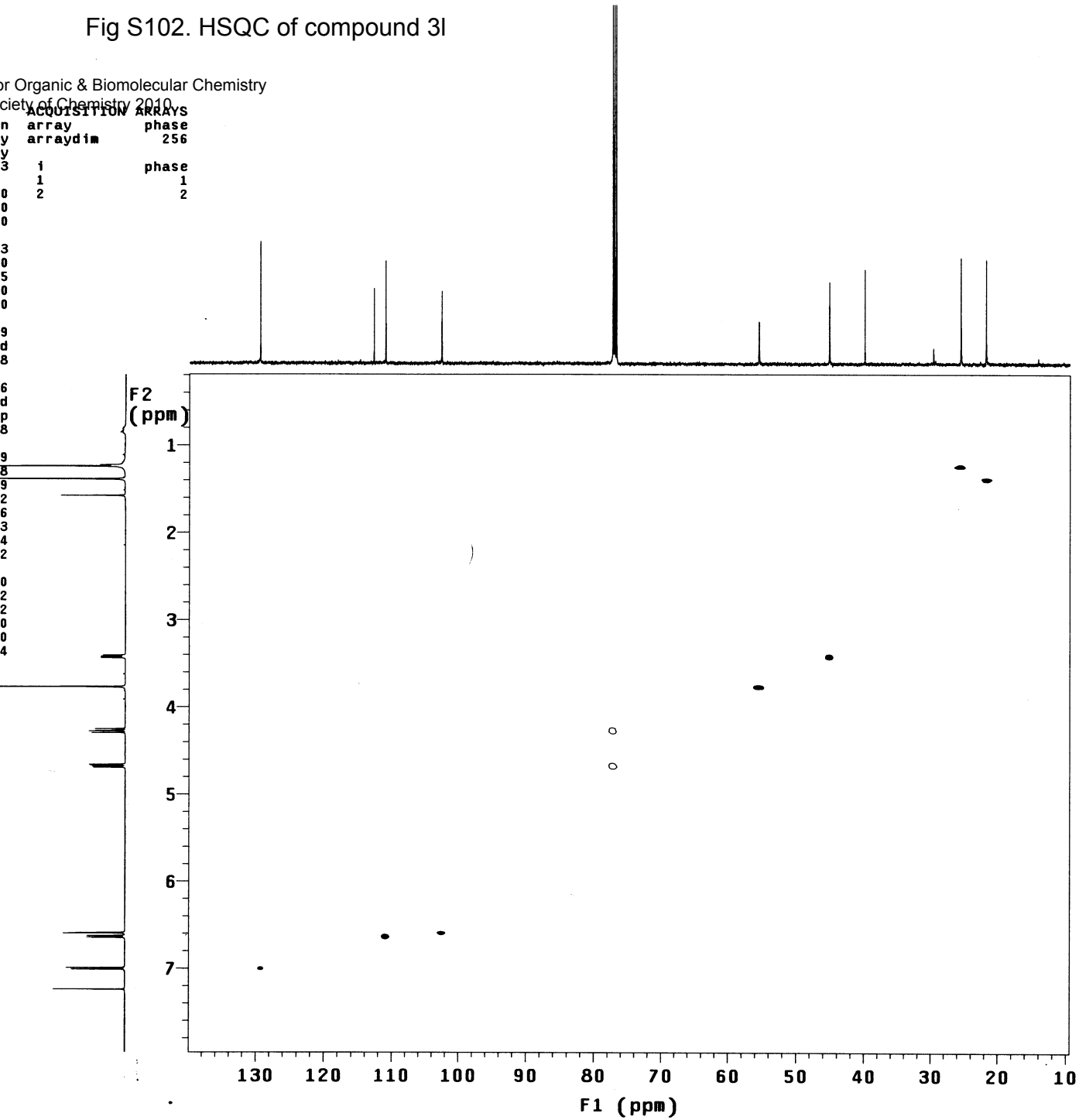


Fig S103. COSY of compound 3l

PMK-02-395
exp16 gCOSY

SAMPLE		FLAGS	
date	Aug 10 2010	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4001.6	temp	23.0
at	0.128	gain	24
np	1024	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.064
d1	1.000	sbs	not used
nt	8	fn	1024
2D ACQUISITION		F1 PROCESSING	
sw1	4001.6	sb1	-0.032
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	1024
sfrq	499.829	DISPLAY	
tof	-499.9	sp	323.4
tpwr	58	wp	3399.8
pw	11.100	sp1	331.6
GRADIENTS		wp1	3392.0
gzlvl1	1003	rfl	2156.8
gt1	0.001000	rfp	2136.3
gstab	0.000500	rfl1	2156.4
DECOUPLER		rfp1	2136.3
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	8
		ai cdc av	

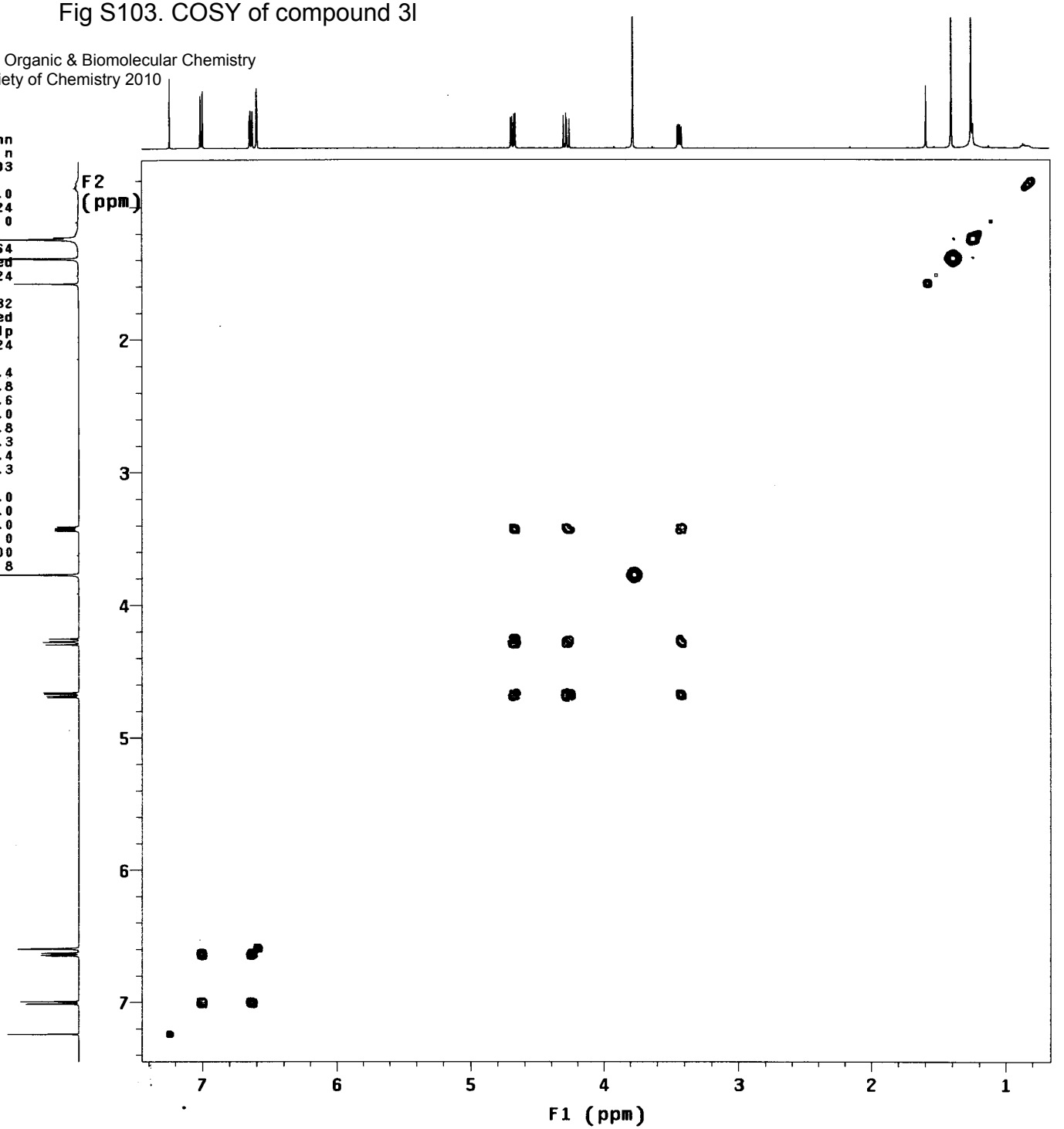


Fig S104. NOESY of compound 3l

SAMPLE	
date	Aug 10 2010
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4001.6
at	0.128
np	1024
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	4001.6
ni	200
TRANSMITTER	
tn	H1
sfrq	499.829
tof	-499.9
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	23.0
gain	24
spin	0
F2 PROCESSING	
gf	0.058
gfs	not used
fn	1024
F1 PROCESSING	
gf1	0.046
gfs1	not used
proc1	lp
fn1	1024
DISPLAY	
sp	307.5
wp	3384.2
sp1	292.5
wp1	3392.0
rf1	2157.0
rfp	2136.3
rf11	2156.4
rfp1	2136.3
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	1
ai ph	

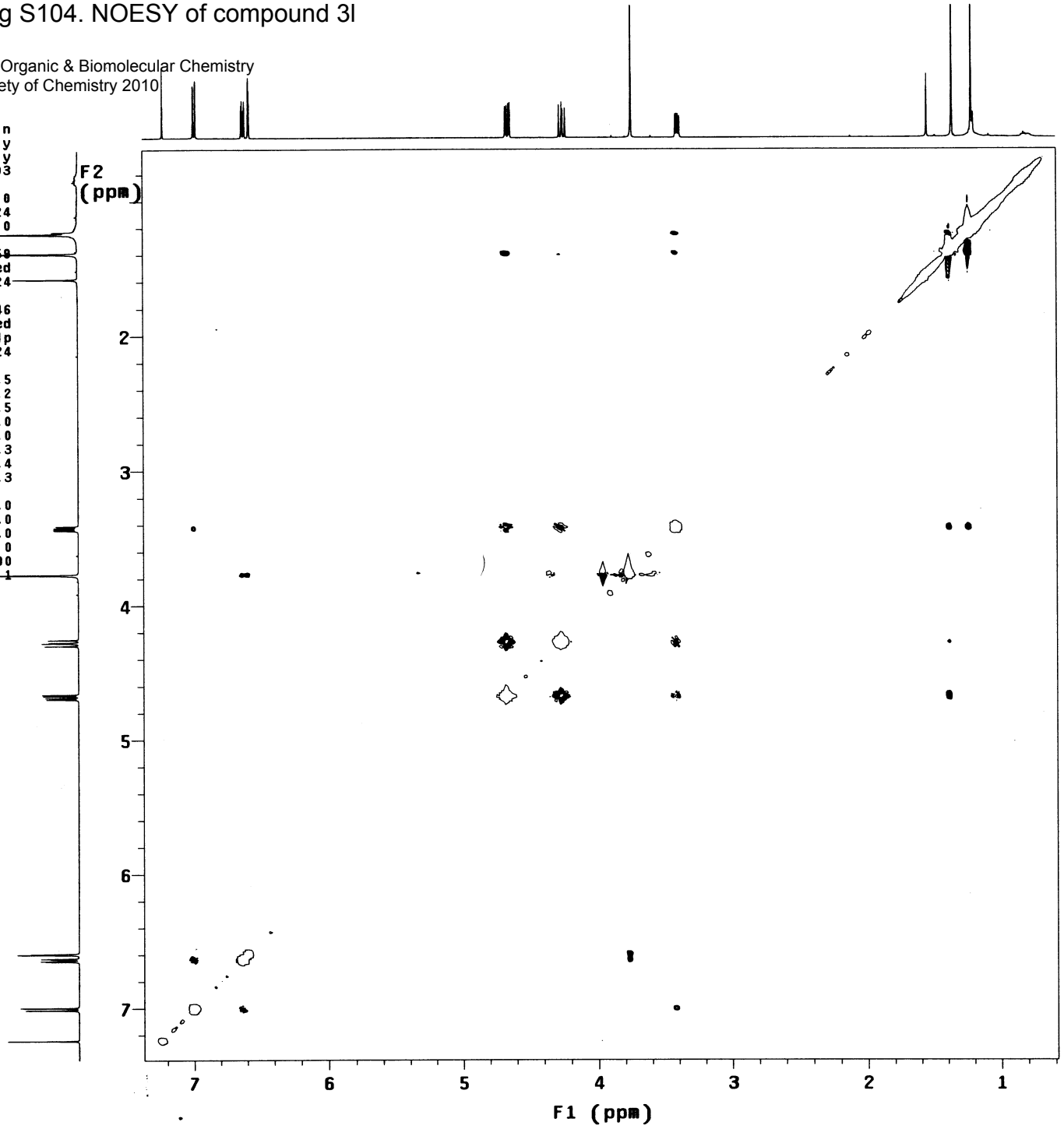
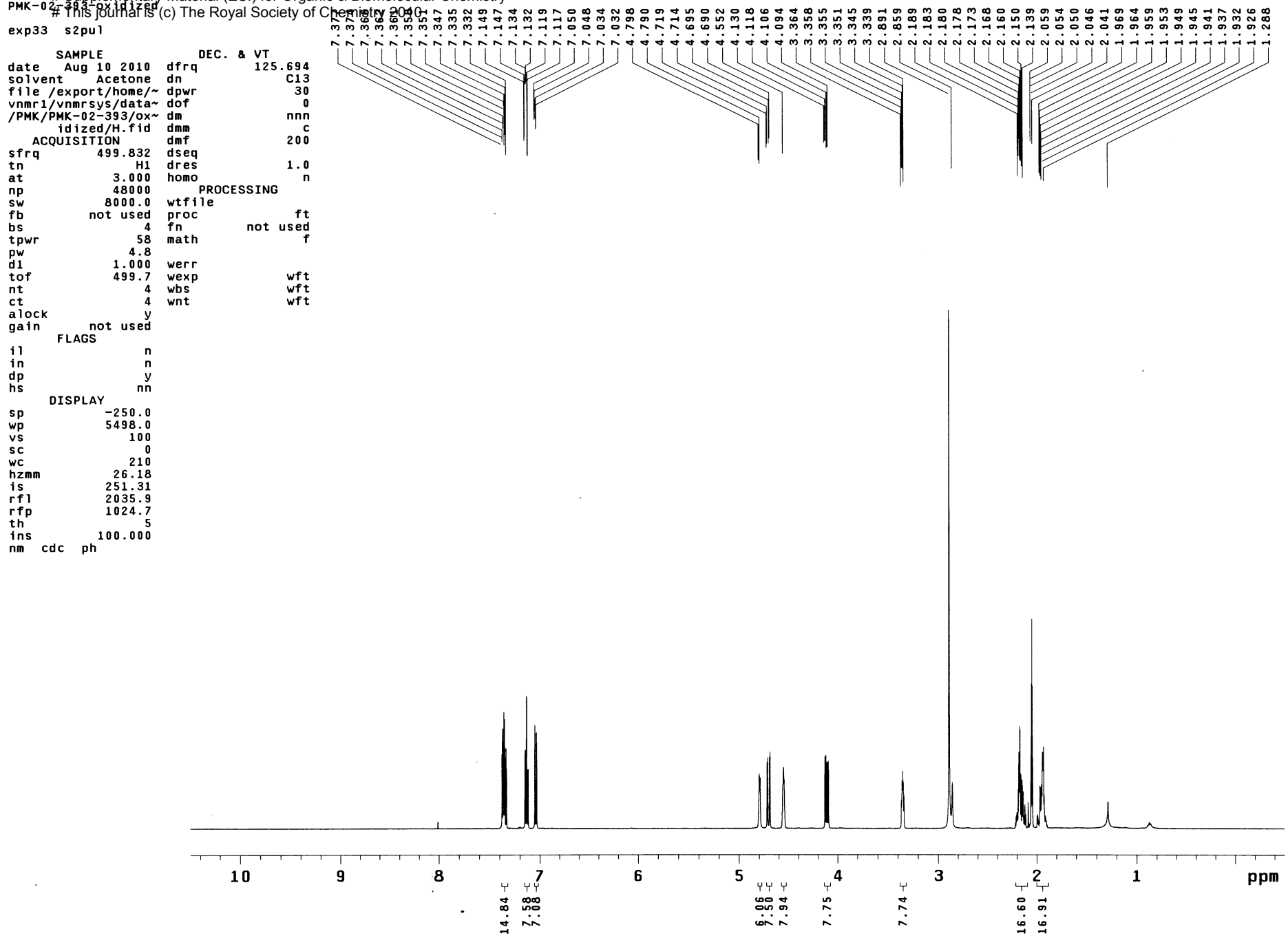
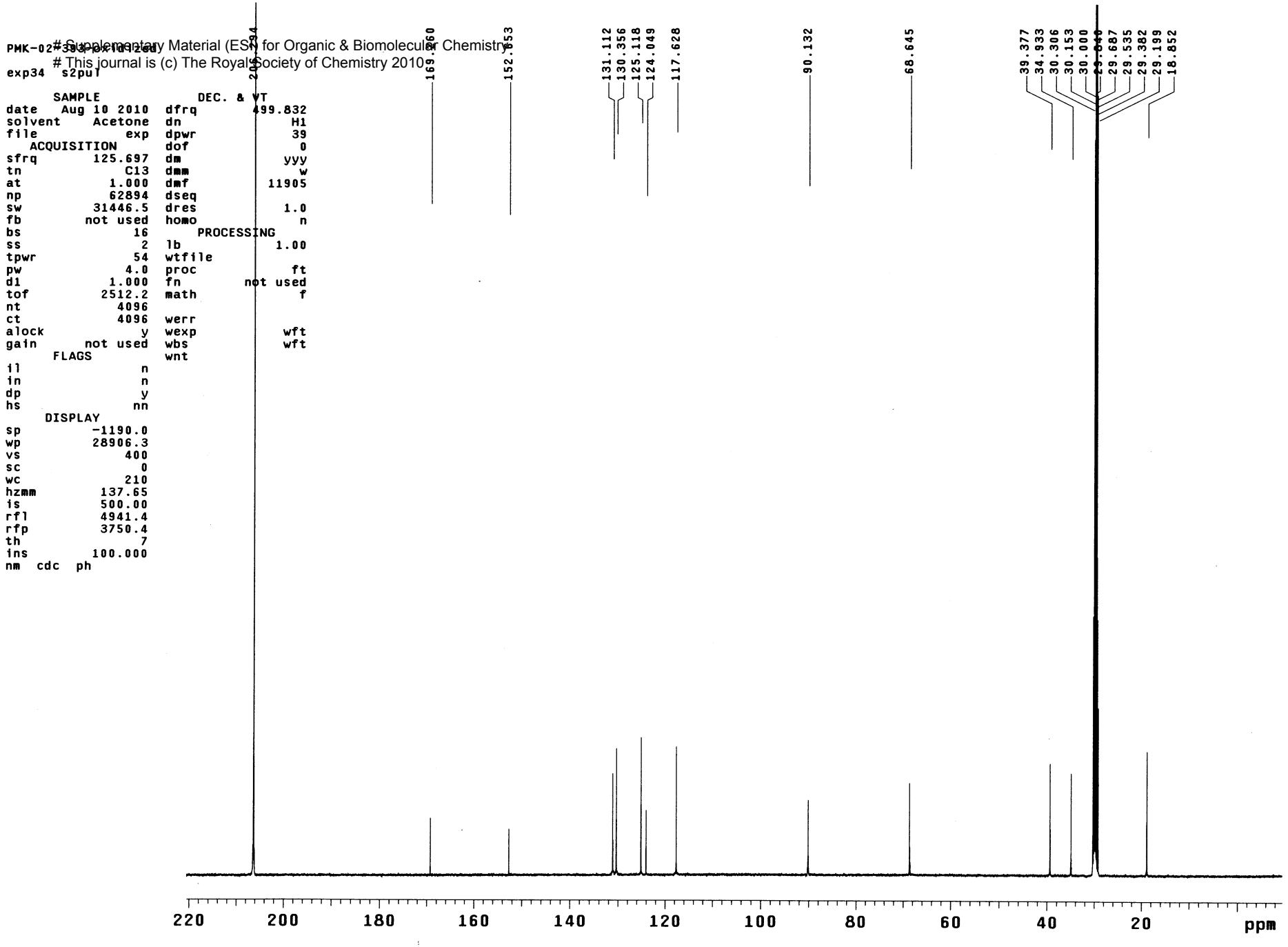


Fig S105. 1H NMR (acetone-d6, 500 MHz) of compound 3m





. Fig S106. 13C NMR (acetone-d6, 125 MHz) of compound 3m

Fig S107. 1H NMR (acetone-d6, 500 MHz) of compound 3m

PMK-02-393-oxidized
exp33 # 1 pulse

SAMPLE		DEC. & VT	
date	Aug 10 2010	dfrq	125.694
solvent	Acetone	dn	C13
file	/export/home/~	dpwr	30
vnmr1/vnmrsys/data~		dof	0
/PMK/PMK-02-393/ox~		dm	nnn
idized/H.fid		dmm	c
ACQUISITION		dmf	200
sfrq	499.832	dseq	
tn	H1	dres	1.0
at	3.000	homo	n
np	48000	PROCESSING	
sw	8000.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58	math	f
pw	4.8		
d1	1.000	werr	
tof	499.7	wexp	wft
nt	4	wbs	wft
ct	4	wnt	wft
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.0		
wp	5498.0		
vs	100		
sc	0		
wc	210		
hzmm	26.18		
is	251.31		
rfl	2035.9		
rfp	1024.7		
th	5		
ins	100.000		
nm cdc ph			

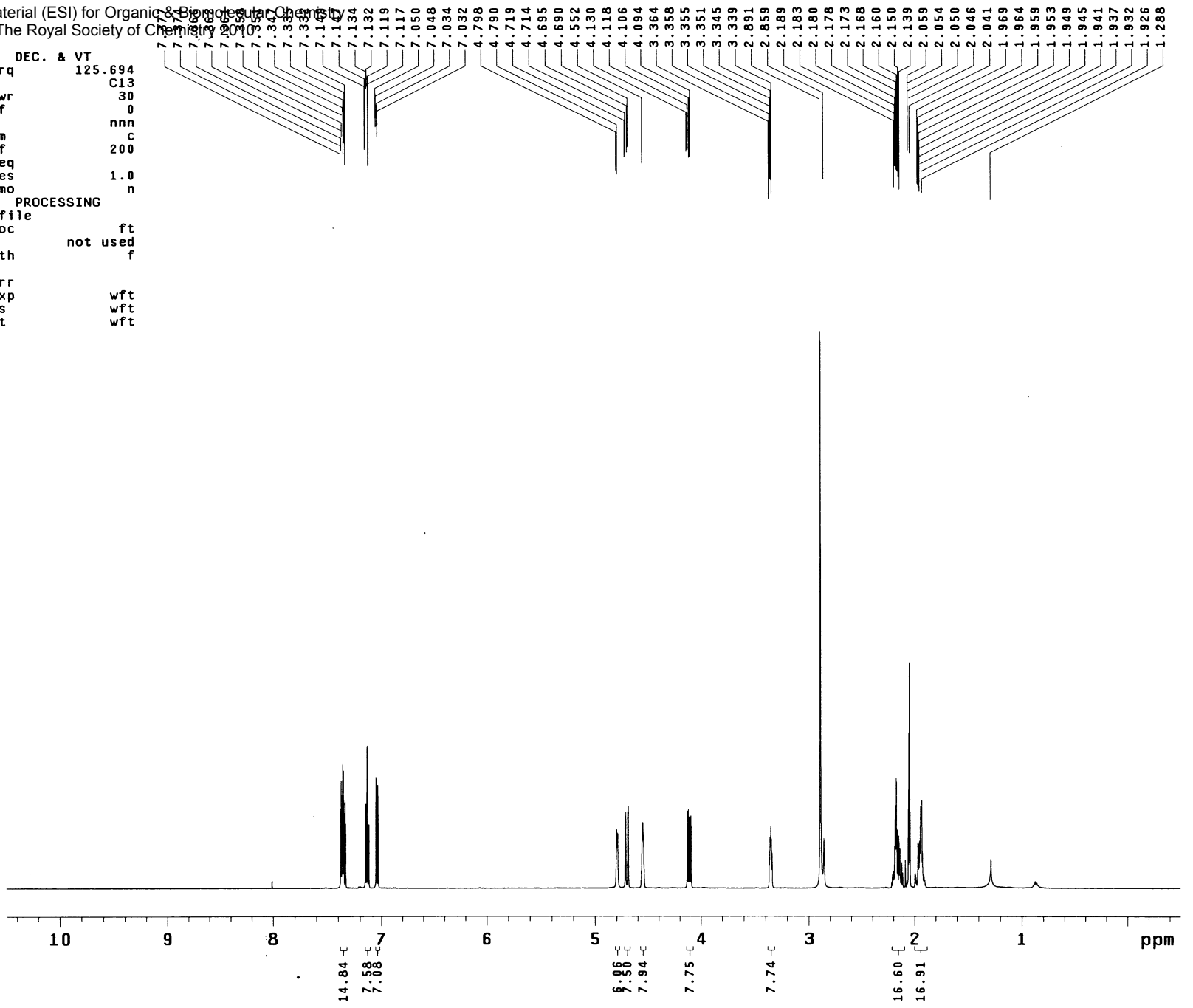


Fig S108. DEPT of compound 3m

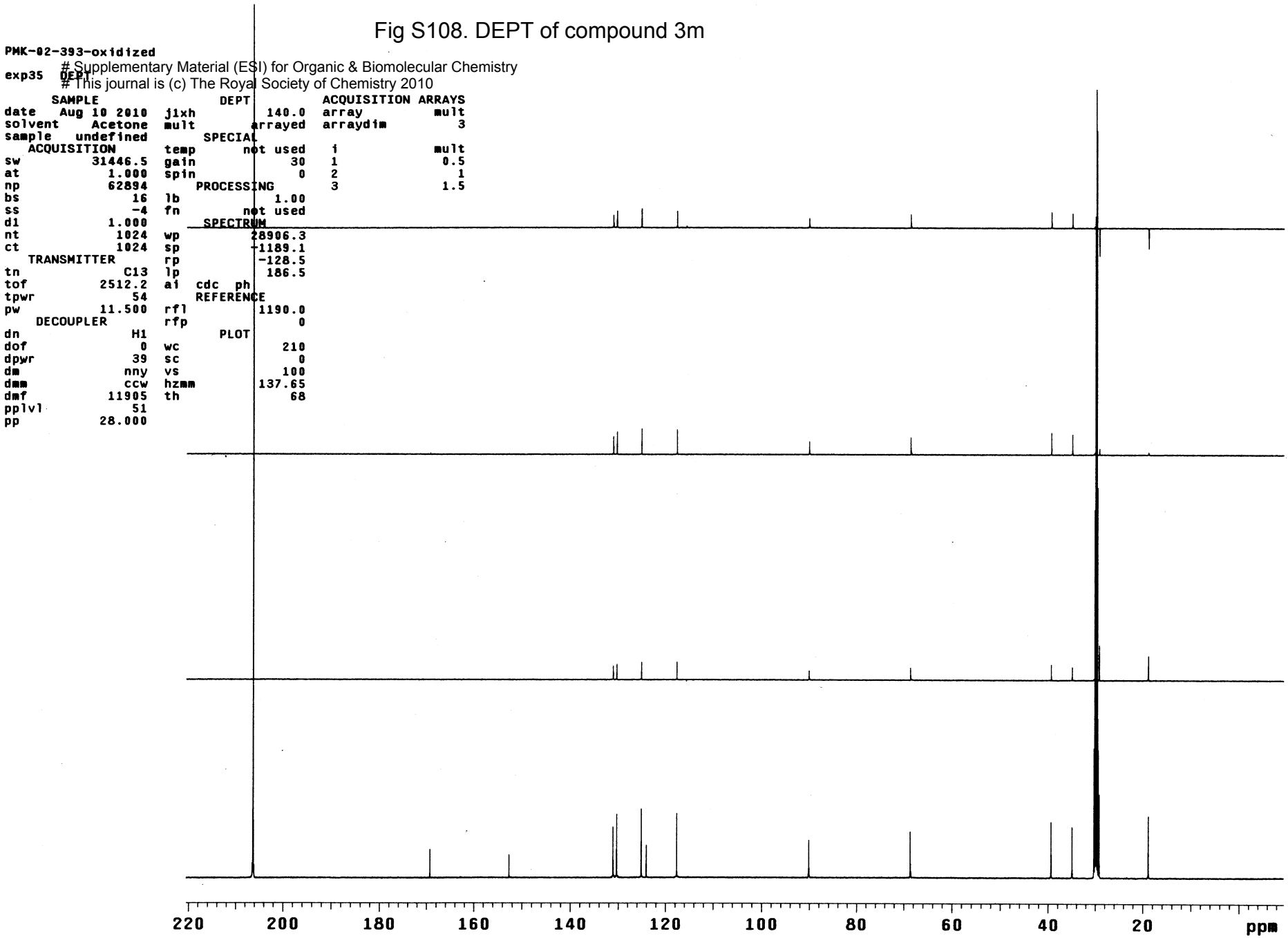


Fig S109. Another DEPT of compound 3m

PMK-02-393-oxidized

exp35 #1 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Aug 10 2010	jlxh	140.0	array	mult
solvent	Acetone	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	1	mult
sw	31446.5	gain	30	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	31445.6		
ct	1024	sp	-1189.1		
TRANSMITTER		rp	-128.5		
tn	C13	lp	186.5		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	1190.0		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	100		
dmm	ccw	hzmm	149.74		
dmf	11905	th	68		
pplvl	51				
pp	28.000				

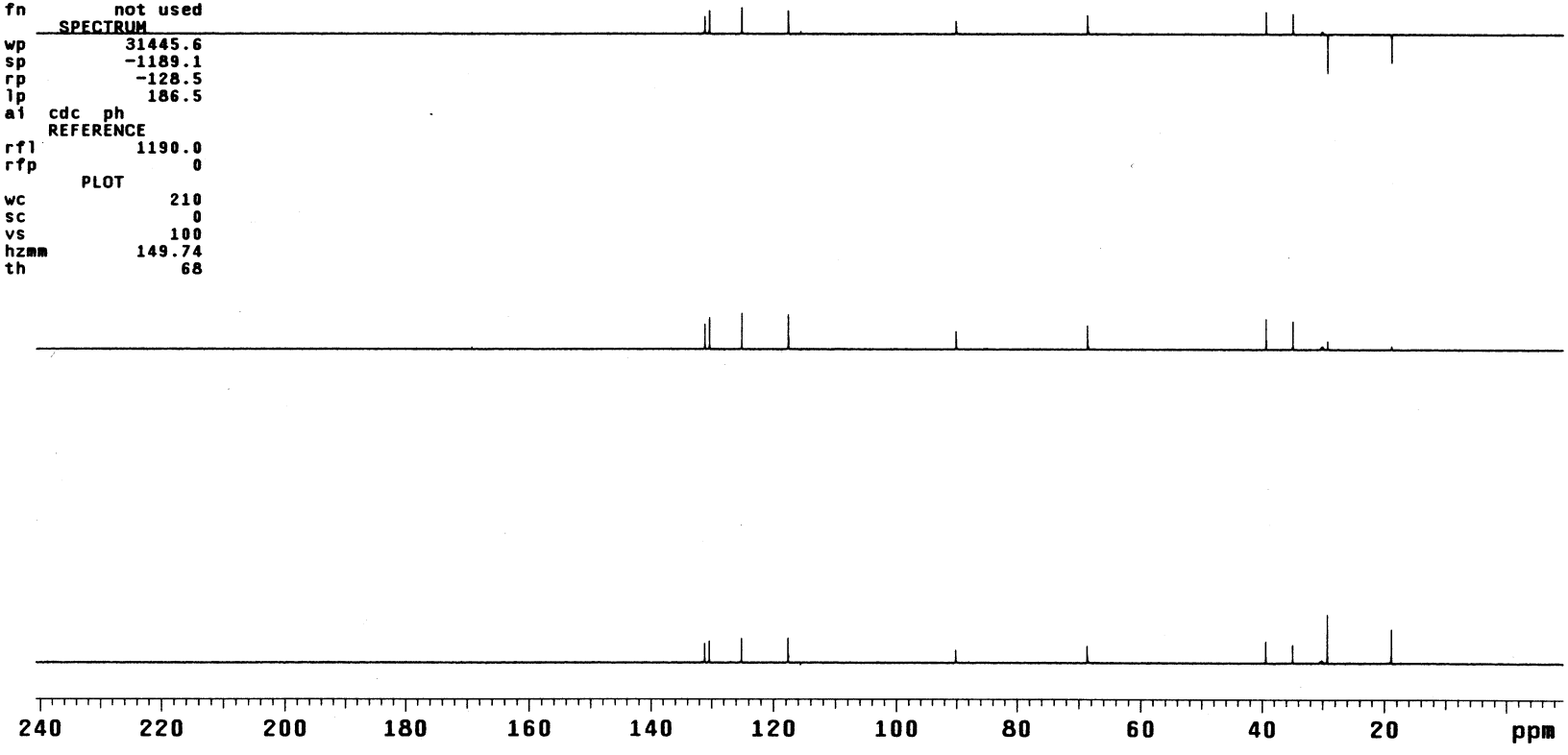


Fig S110. HSQC of compound 3m

PMK-02-393-oxidized
expno gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Aug 10 2010	hs	n	array	phase
solvent	Acetone	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	5006.3	SPECIAL		1	1
at	0.205	temp	not used	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.094		
tn	H1	gfs	not used		
sfrq	499.831	fn	2048		
tof	-499.9	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	796.0		
dmm	ccp	wp	3040.9		
dmf	32258	sp1	1155.9		
dpwr	36	wp1	16255.2		
pwxlvl	52	rfl	4033.5		
pwx	14.300	rfp	3519.3		
HSQC		rfl1	15965.0		
j1xh	140.0	rfp1	14783.7		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	5		
		ai cdc ph			

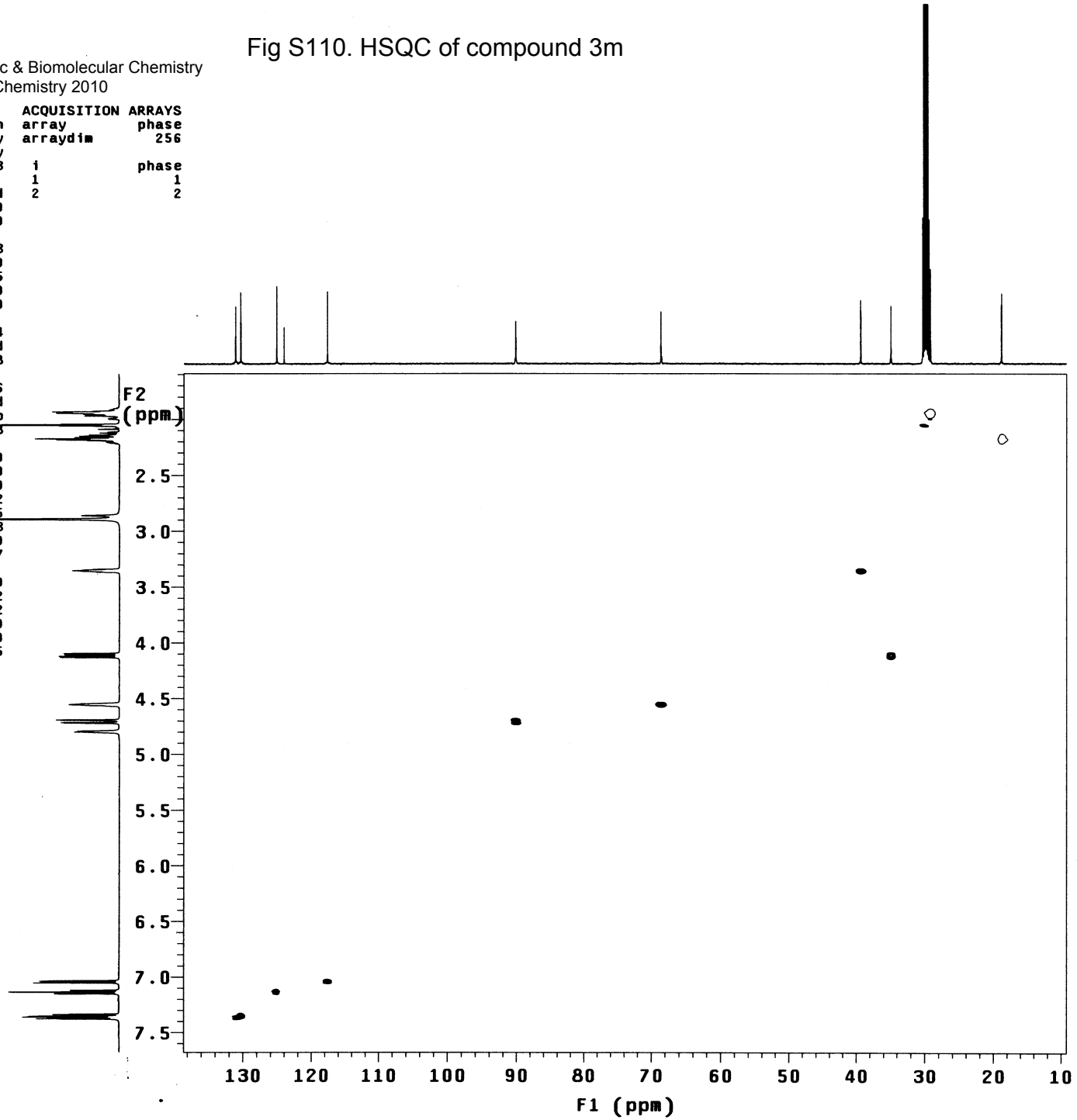


Fig S111. COSY of compound 3m

PMK-02-[illegible]-oxidized
exp36 gCOSY

SAMPLE		FLAGS	
date	Aug 10 2010	hs	nn
solvent	Acetone	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	5006.3	temp	not used
at	0.205	gain	30
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.102
d1	1.000	sbs	not used
nt	8	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	5006.3	sb1	-0.026
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.831	DISPLAY	
tof	-499.9	sp	791.4
tpwr	58	wp	3006.7
pw	11.100	sp1	777.4
GRADIENTS		wp1	3011.6
gzlvl1	1003	rfl	2789.2
gt1	0.001000	rfp	2275.2
gstab	0.000500	rfl1	2788.5
DECOUPLER		rfp1	2275.2
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	9
		ai cdc av	

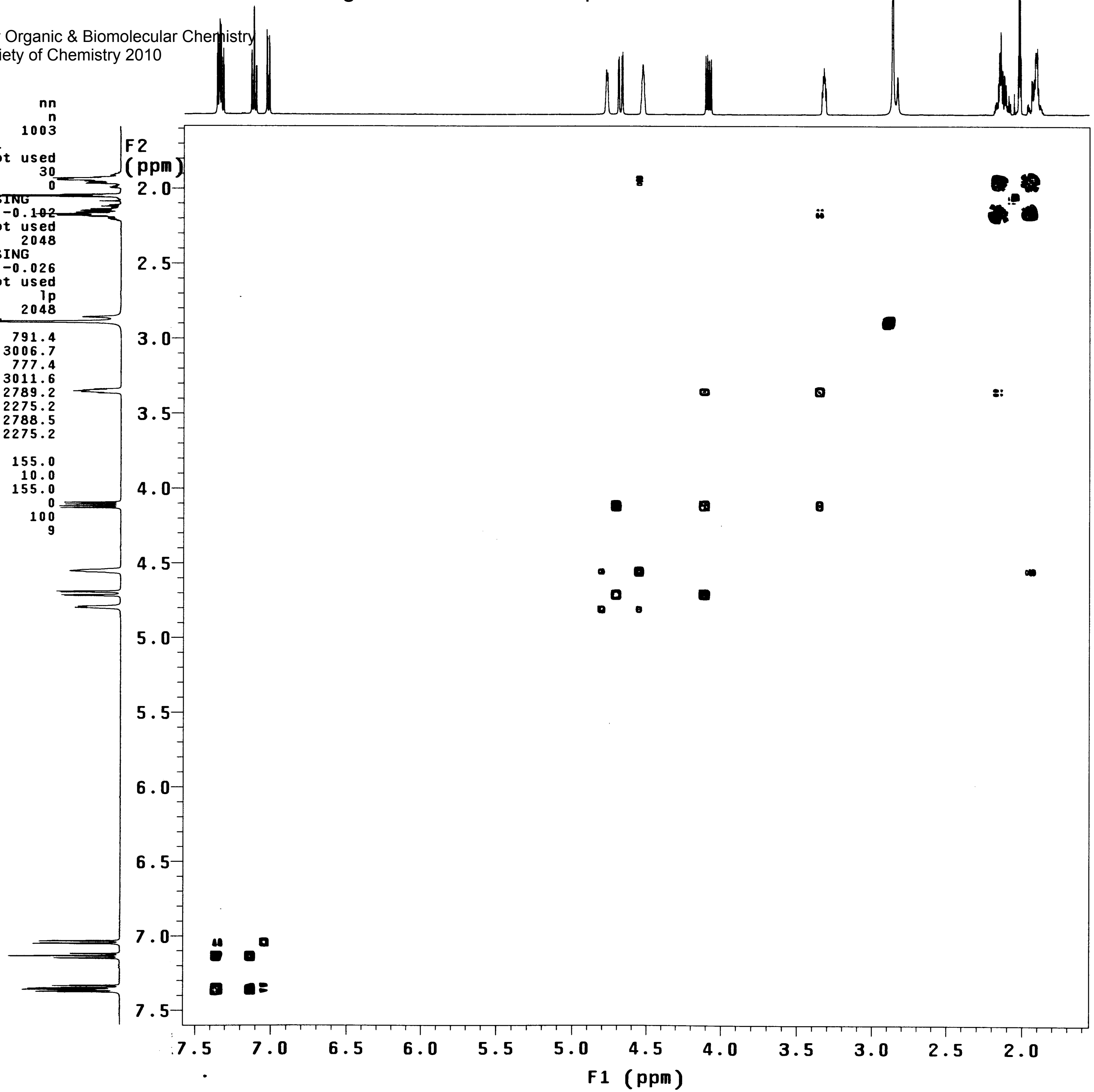


Fig S112. NOESY of compound 3m

PMK-02-393-oxidized

exp37 NOESY

SAMPLE	
date	Aug 10 2010
solvent	Acetone
sample	undefined
ACQUISITION	
sw	5006.3
at	0.205
np	2048
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	5006.3
ni	200
TRANSMITTER	
tn	H1
sfrq	499.831
tof	-499.9
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	30
spin	0
F2 PROCESSING	
gf	0.094
gfs	not used
fn	2048
F1 PROCESSING	
gf1	0.037
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	852.4
wp	2952.9
sp1	855.6
wp1	2957.8
rfl	2791.7
rfp	2275.2
rfl1	2788.5
rfp1	2275.2
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	1
ai ph	

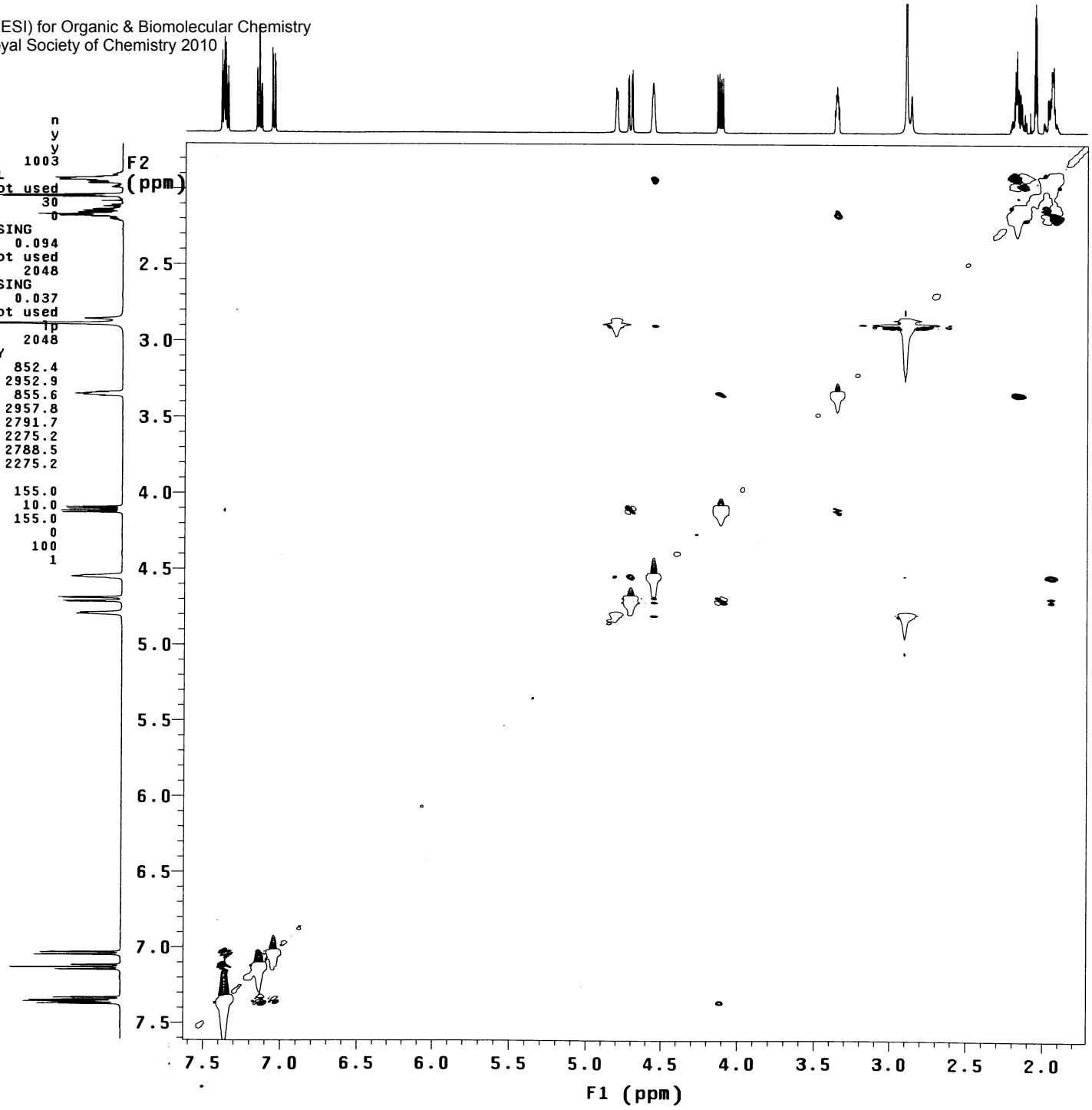


Fig S113. 1H NMR (acetone-d6, 500 MHz) of compound 3n

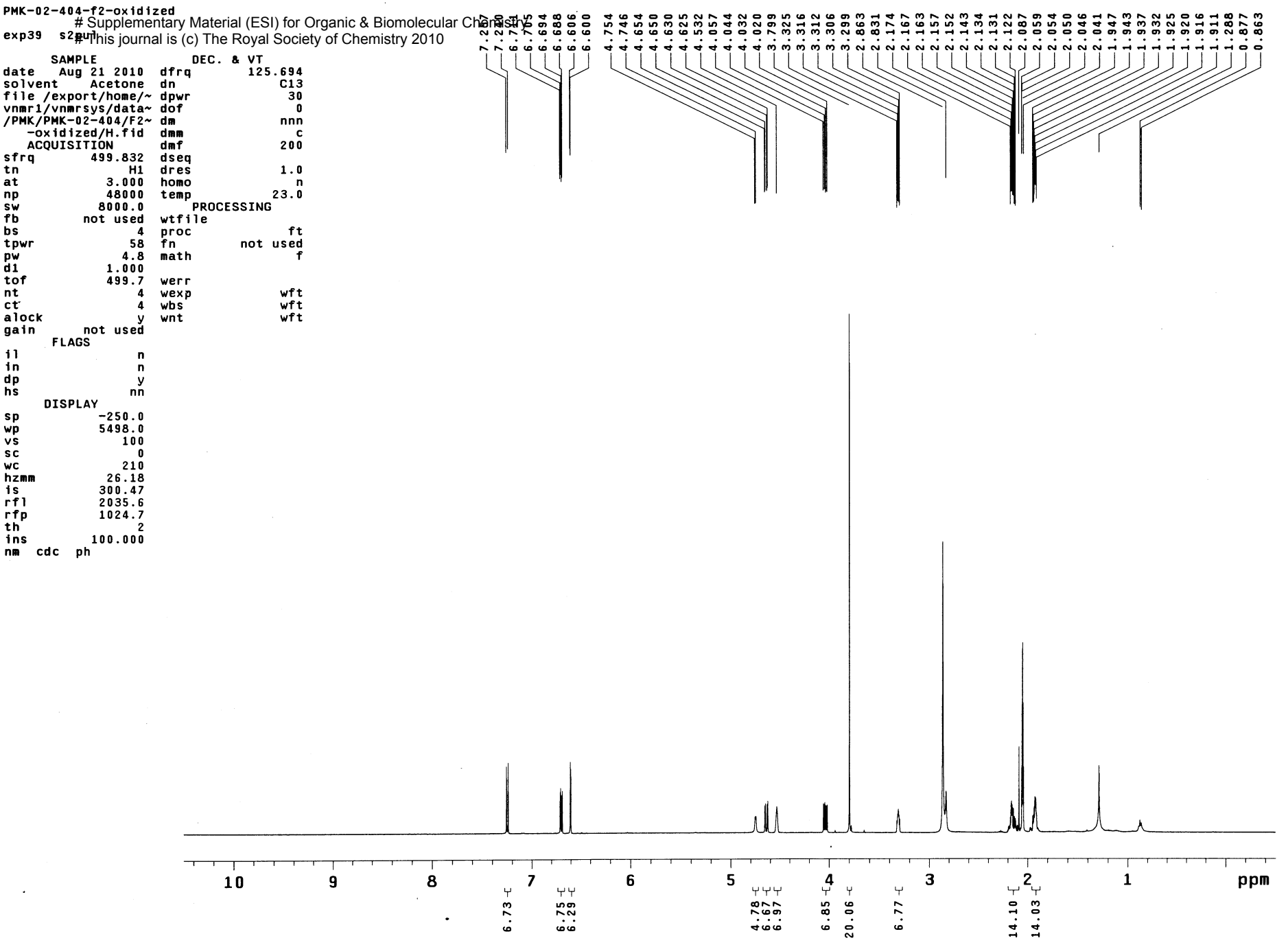
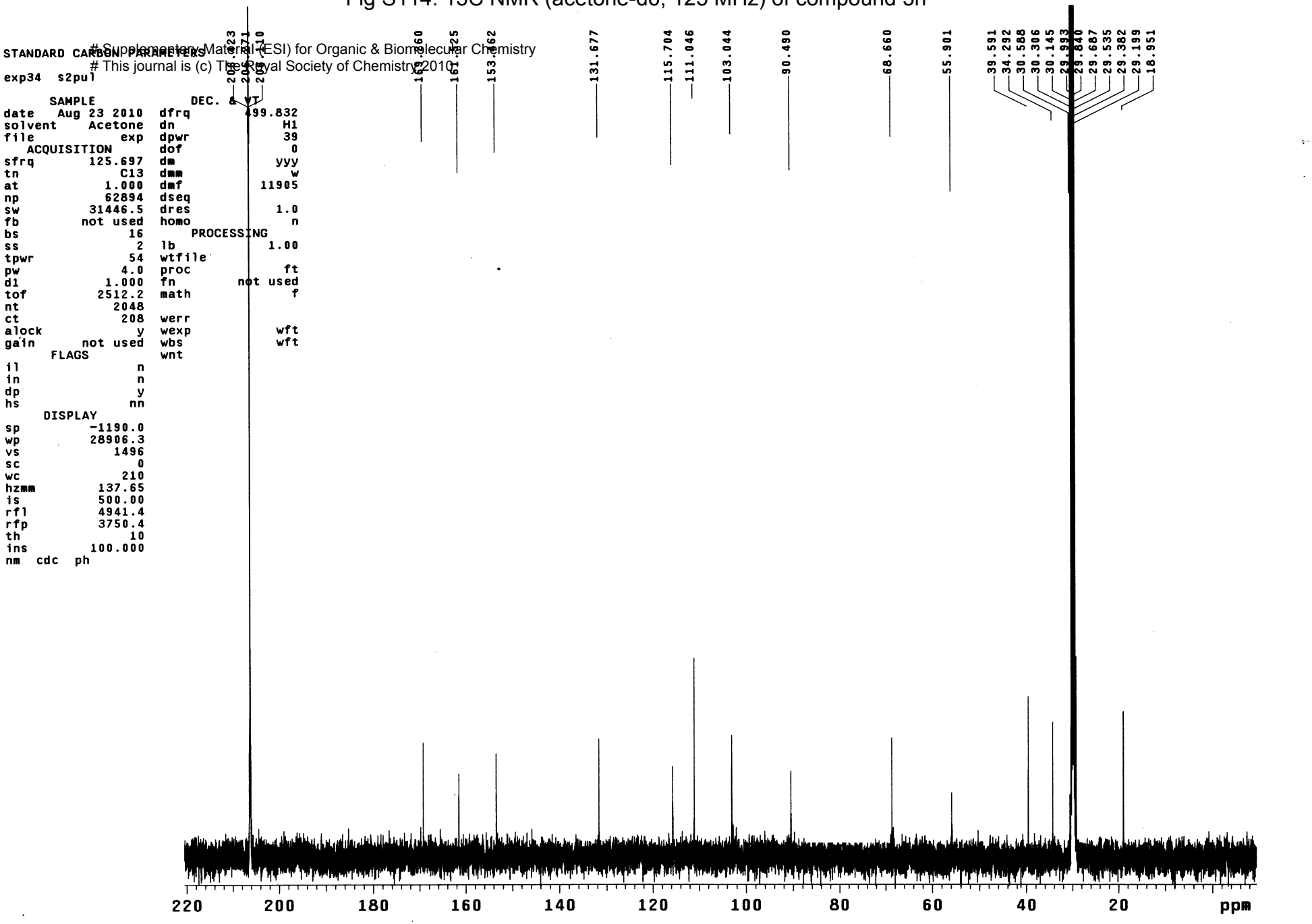


Fig S114. 13C NMR (acetone-d6, 125 MHz) of compound 3n

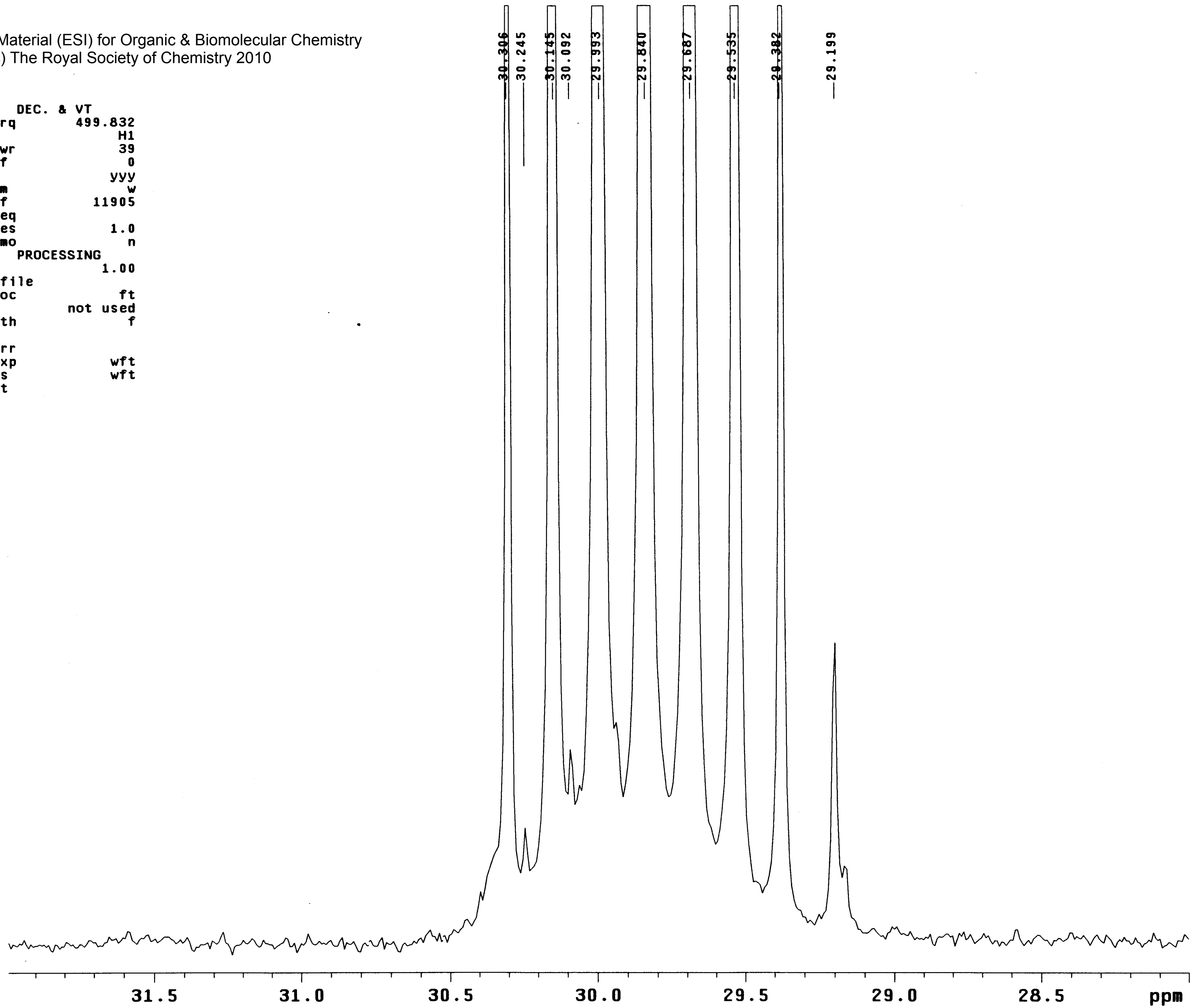


PMK-02-404-C2-oxidized

exp34 s2pul

SAMPLE	
date	Aug 23 2010
solvent	Acetone
file	exp
ACQUISITION	
sfrq	125.697
tn	C13
at	1.000
np	62894
sw	31446.5
fb	not used
bs	16
ss	2
tpwr	54
pw	4.0
d1	1.000
tof	2512.2
nt	2048
ct	2048
alock	y
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	3519.1
wp	501.9
vs	2117
sc	0
wc	210
hzmm	2.39
is	500.00
rfl	4941.4
rfp	3750.4
th	7
ins	100.000
nm cdc ph	

DEC. & VT	
dfrq	499.832
dn	H1
dpwr	39
dof	0
dm	yyy
dmm	w
dmf	11905
dseq	
dres	1.0
homo	n
PROCESSING	
lb	1.00
wtfile	
proc	ft
fn	not used
math	f
werr	
wexp	wft
wbs	wft
wnt	



.Fig S115. 13C NMR (acetone-d6, 125 MHz) of compound 3n (Expand).

Fig S116. DEPT of compound 3n

PMK-02-404-f2-oxidized
exp35 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Aug 21 2010	j1xh	140.0	array	mult
solvent	Acetone	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	23.0	i	mult
sw	31446.5	gain	30	1	0.5
at	1.000	spin	0	2	1
np	62894	PROCESSING		3	1.5
bs	16	lb	1.00		
ss	-4	fn	not used		
d1	1.000	SPECTRUM			
nt	1024	wp	28906.3		
ct	1024	sp	-1257.3		
TRANSMITTER		rp	-33.9		
tn	C13	lp	130.4		
tof	2512.2	ai	cdc ph		
tpwr	54	REFERENCE			
pw	11.500	rfl	3573.8		
DECOUPLER		rfp	2279.1		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	39	sc	0		
dm	nny	vs	400		
dmm	ccw	hzmm	137.65		
dmf	11905	th	14		
pplvl	51				
pp	28.000				

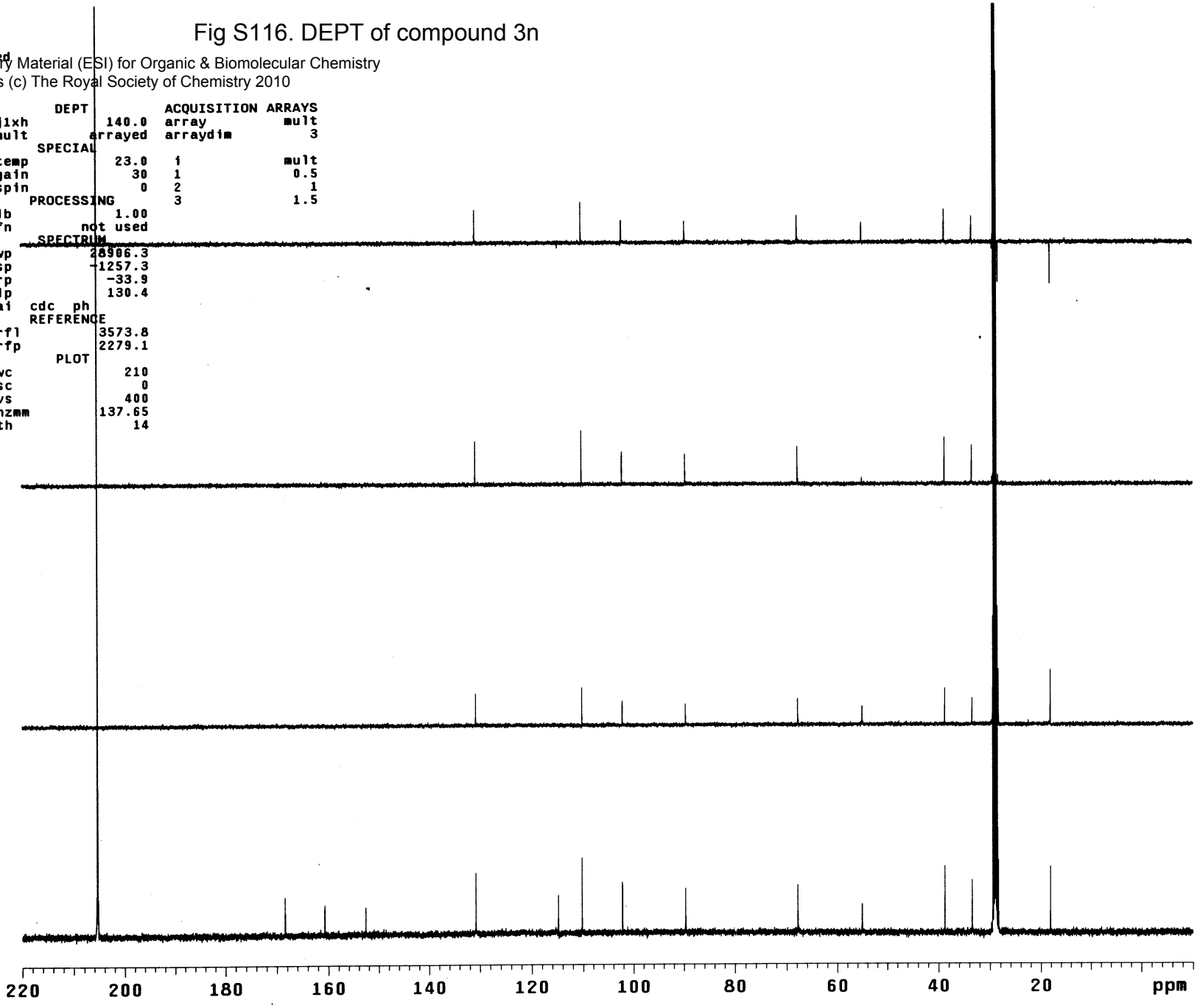


Fig S117. HSQC of compound 3n

PMK-02-404-f2-oxidized
exp38 #gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Aug 21 2010	hs	n	array	phase
solvent	Acetone	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4490.3	SPECIAL		1	1
at	0.228	temp	23.0	2	2
np	2048	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	4	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.105		
tn	H1	gfs	not used		
sfrq	499.832	fn	2048		
tof	-250.0	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	491.9		
dmm	ccp	wp	3723.0		
dmf	32258	sp1	1244.7		
dpwr	36	wp1	17611.5		
pwxlvl	52	rfl	2268.9		
pwx	14.300	rfp	2265.2		
HSQC		rfl1	9805.0		
j1xh	140.0	rfp1	8524.8		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	200		
		th	5		
		ai cdc ph			

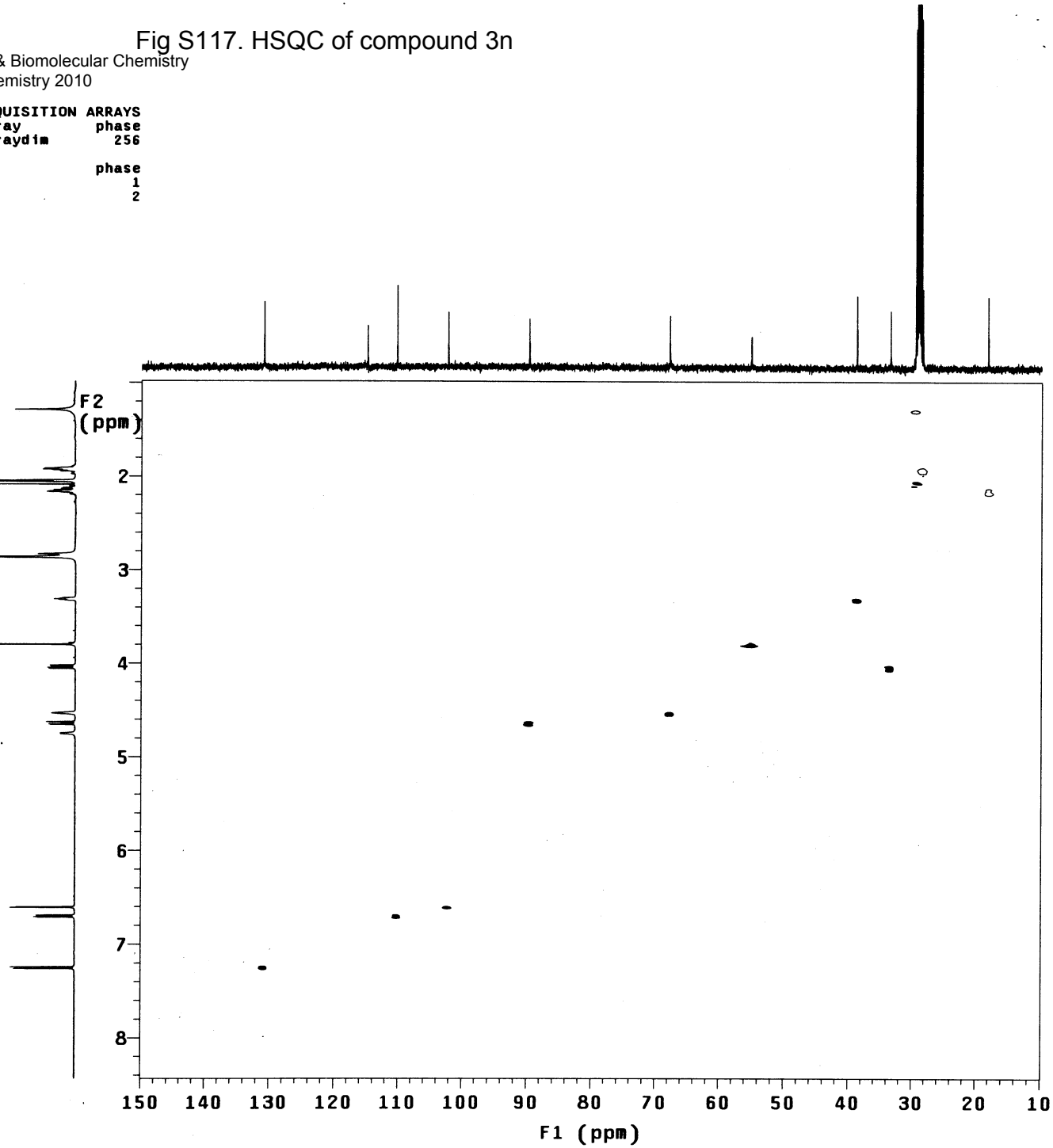


Fig S118. COSY of compound 3n

PMK-02-404-T2-oxidized
exp36 gCOSY

SAMPLE		FLAGS	
date	Aug 21 2010	hs	nn
solvent	Acetone	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4490.3	temp	23.0
at	0.228	gain	30
np	2048	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.114
d1	1.000	sbs	not used
nt	4	fn	2048
2D ACQUISITION		F1 PROCESSING	
sw1	4490.3	sb1	-0.029
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	2048
sfrq	499.832	DISPLAY	
tof	-250.0	sp	844.4
tpwr	58	wp	3249.4
pw	11.100	sp1	848.5
GRADIENTS		wp1	3249.4
gzlvl1	1003	rfl	2380.5
gt1	0.001000	rfp	2374.2
gstab	0.000500	rfl1	2380.7
DECOUPLER		rfp1	2374.2
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	200
		th	7
		ai cdc av	

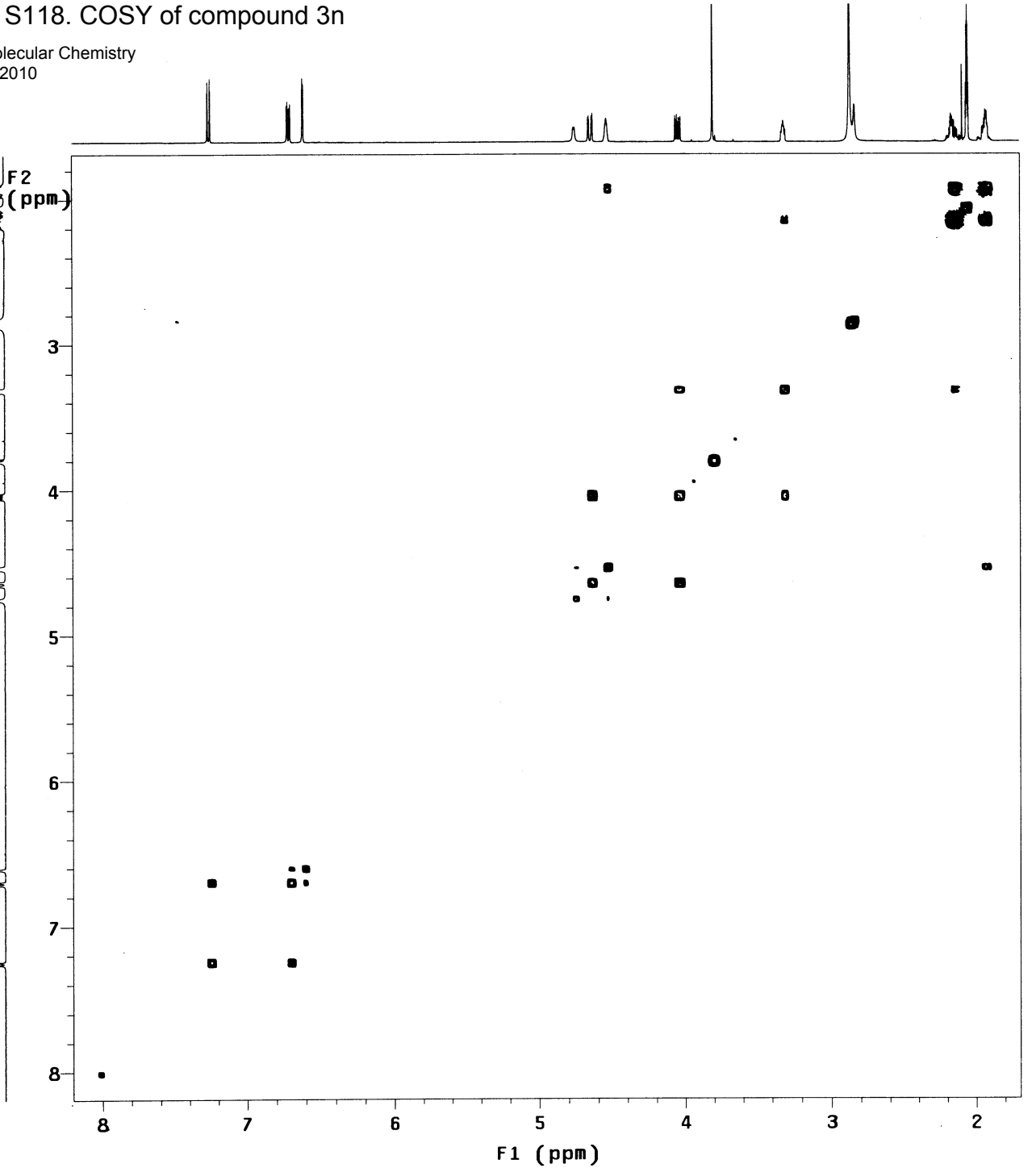
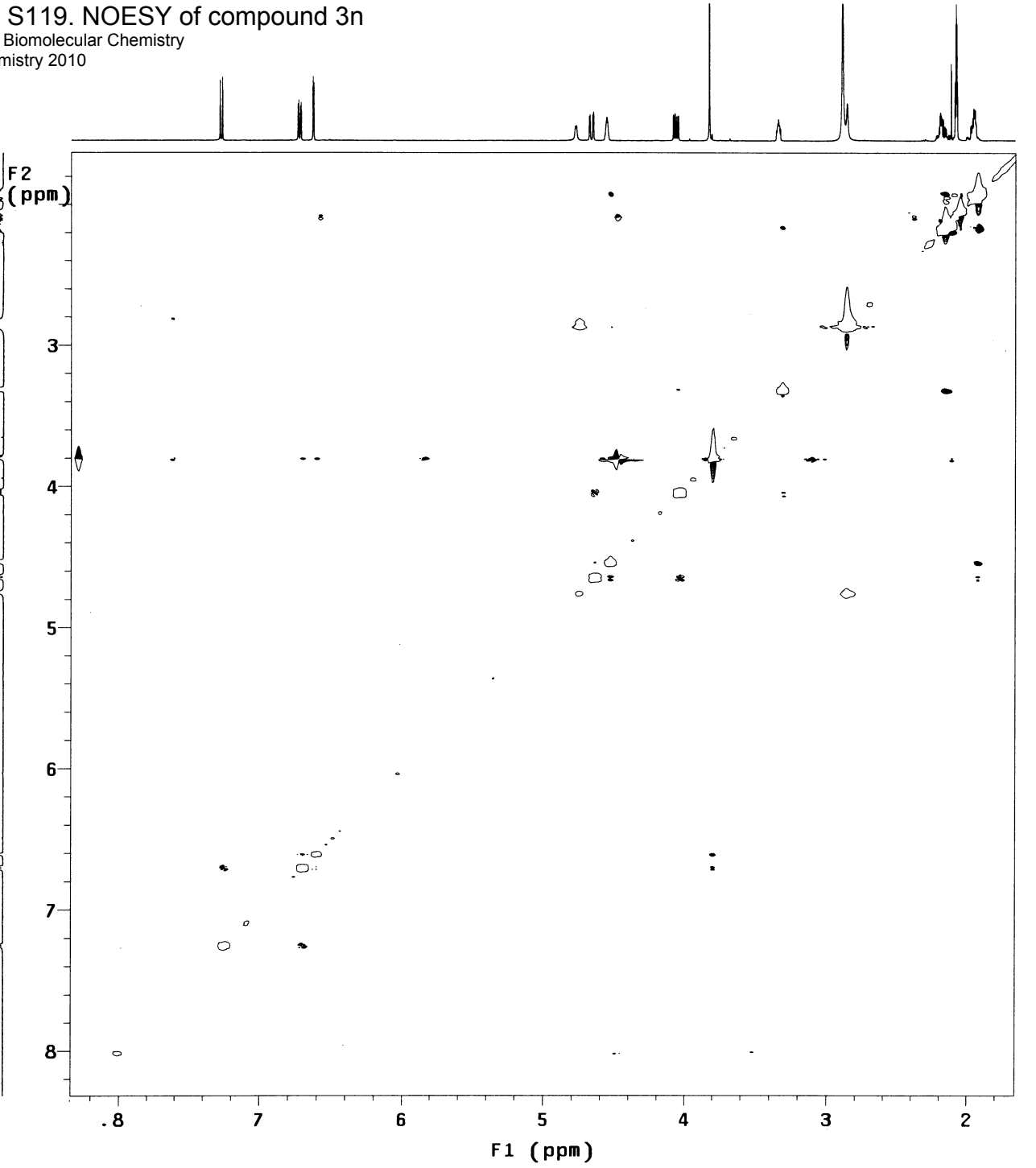


Fig S119. NOESY of compound 3n

PMK-02-404-f2.oxaligned

exp37 NOESY

SAMPLE		FLAGS	
date	Aug 21 2010	hs	n
solvent	Acetone	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1003
sw	4490.3	SPECIAL	
at	0.228	temp	23.0
np	2048	gain	30
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.105
nt	8	gfs	not used
2D ACQUISITION		fn	2048
sw1	4490.3	F1 PROCESSING	
ni	200	gf1	0.041
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.832	fn1	2048
tof	-250.0	DISPLAY	
tpwr	58	sp	815.1
pw	11.100	wp	3341.4
NOESY		sp1	831.2
mix	0.600	wp1	3337.1
PRESATURATION		rfl	2379.1
satmode	nnnn	rfp	2374.2
satpwr	0	rfl1	2380.5
satdly	0	rfp1	2374.2
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	200
		th	3
		ai ph	



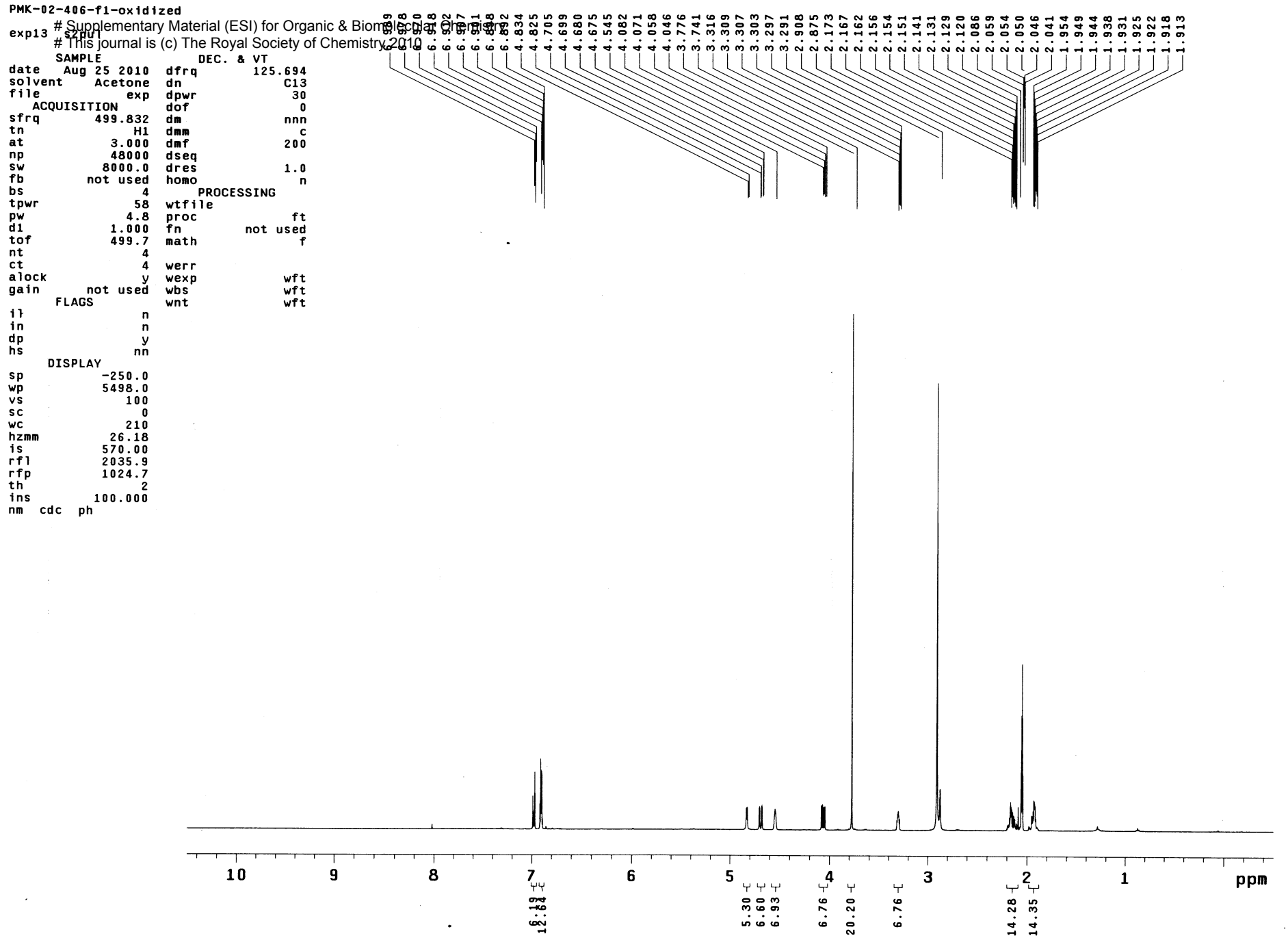
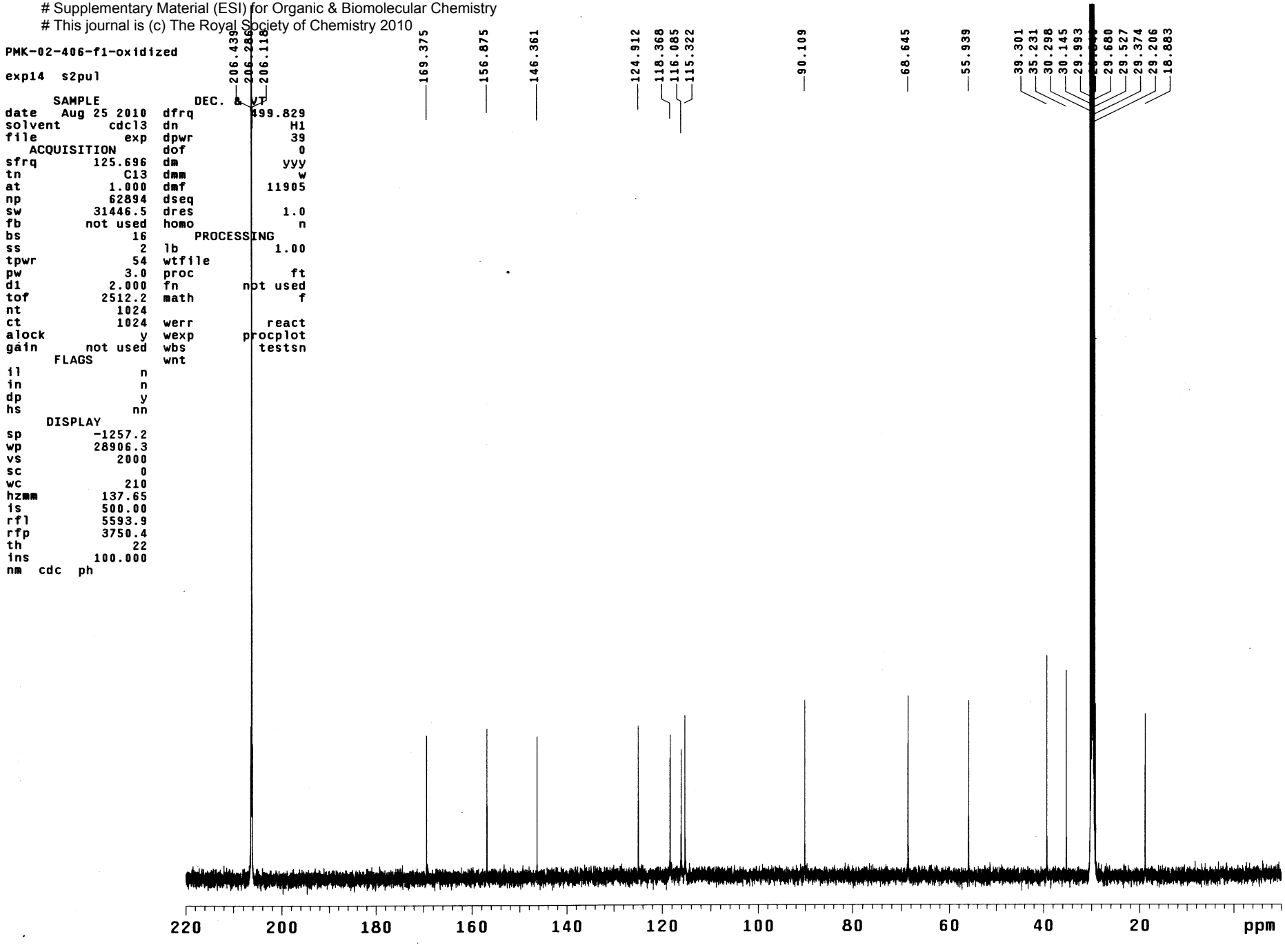


Fig S120. 1H NMR (acetone-d6, 500 MHz) of compound 3o

Fig S121. 13C NMR (acetone-d6, 125 MHz) of compound 3o



PMK-92-149

exp14 s2pul

```
      SAMPLE                 DEC. & VT
date   Aug 25 2010   dfrq        499.829
solvent      cdcl3   dn               H1
file           exp   dpwr             39
   ACQUISITION       dof               0
sfrq       125.696   dm              yyy
tn             C13   dmm               w
at           1.000   dmf           11905
np           62894   dseq
sw         31446.5   dres            1.0
fb        not used   homo              n
bs              16       PROCESSING
ss               2   lb             1.00
tpwr            54   wtfile
pw             3.0   proc             ft
d1           2.000   fn         not used
tof         2512.2   math              f
nt            1024
ct            1024   werr          react
alock            y   wexp       procplot
gain      not used   wbs          testsn
      FLAGS          wnt
il               n
in               n
dp               y
hs              nn
      DISPLAY
sp          3141.9
wp          1256.2
vs             750
sc               0
wc             210
hzmm          5.98
is          500.00
rfl         5593.9
rfp         3750.4
th              10
ins        100.000
nm  cdc  ph
```

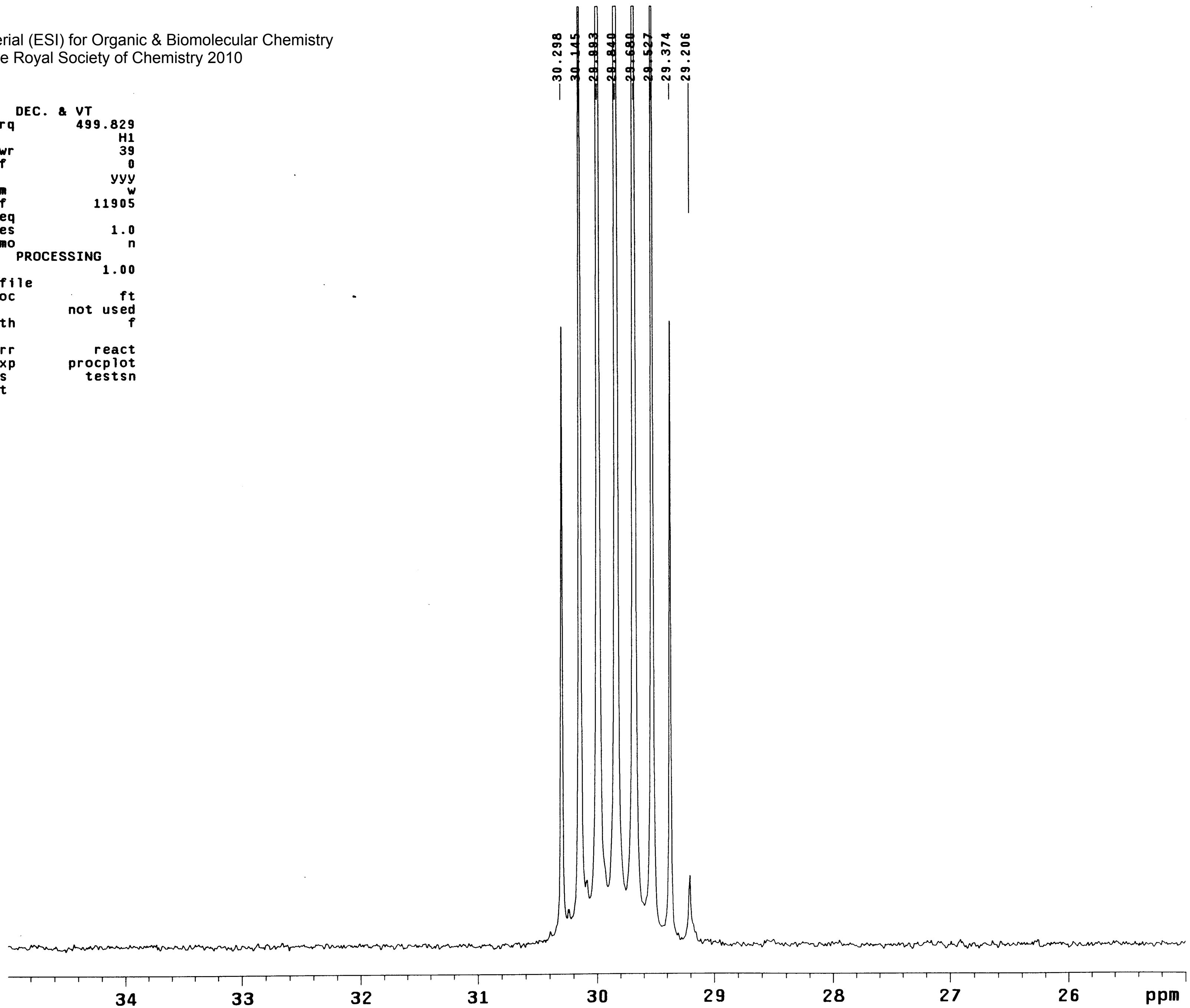


Fig S122. 13C NMR (acetone-d6, 125 MHz) of compound 3o (Expand).

Fig S123. DEPT of compound 3o

PMK-02-A06-f1-gx1d1zed

exp20 DEPT

SAMPLE		DEPT	
date	Aug 25 2010	j1xh	140.0
solvent	Acetone	mult	1.5
sample	undefined	SPECIAL	
ACQUISITION		temp	not used
sw	31446.5	gain	54
at	1.000	spin	0
np	62894	PROCESSING	
bs	16	lb	1.00
ss	-4	fn	not used
d1	1.000	SPECTRUM	
nt	1024	wp	28906.3
ct	1024	sp	-1191.0
TRANSMITTER		rp	-1920.4
tn	C13	lp	195.7
tof	2512.2	ai	cdc ph
tpwr	54	REFERENCE	
pw	11.500	rfl	1192.0
DECOUPLER		rfp	0
dn	H1	PLOT	
dof	0	wc	210
dpwr	39	sc	0
dm	nny	vs	500
dmm	ccw	hzmm	137.65
dmf	11905	th	68
pplvl	51		
pp	31.000		

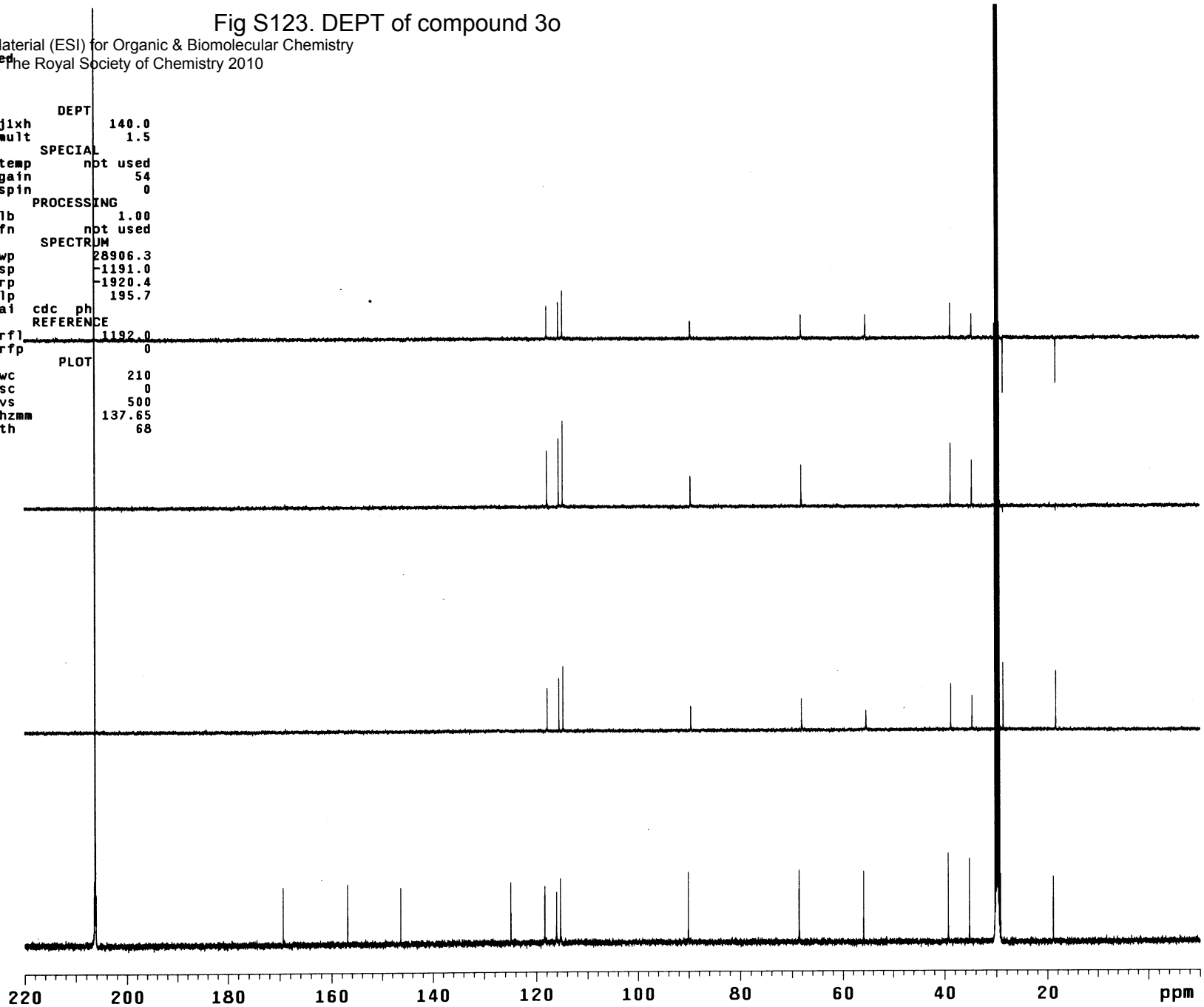


Fig S124. HSQC of compound 3o

PMK-02-406-TFA-Oxidized
exp18 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Aug 25 2010	hs	n	array	phase
solvent	Acetone	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1003	i	phase
sw	4001.6	SPECIAL		1	1
at	0.128	temp	not used	2	2
np	1024	gain	20		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1003		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.831	fn	1024		
tof	-499.9	F1 PROCESSING			
tpwr	58	gf1	0.006		
pw	11.100	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	789.6		
dmm	ccp	wp	2884.0		
dmf	32258	sp1	1155.3		
dpwr	36	wp1	15420.5		
pwxlvl	52	rfl	1894.9		
pwx	14.300	rfp	1887.4		
HSQC		rfl1	8212.3		
j1xh	140.0	rfp1	7030.5		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	100		
		th	5		
		ai cdc ph			

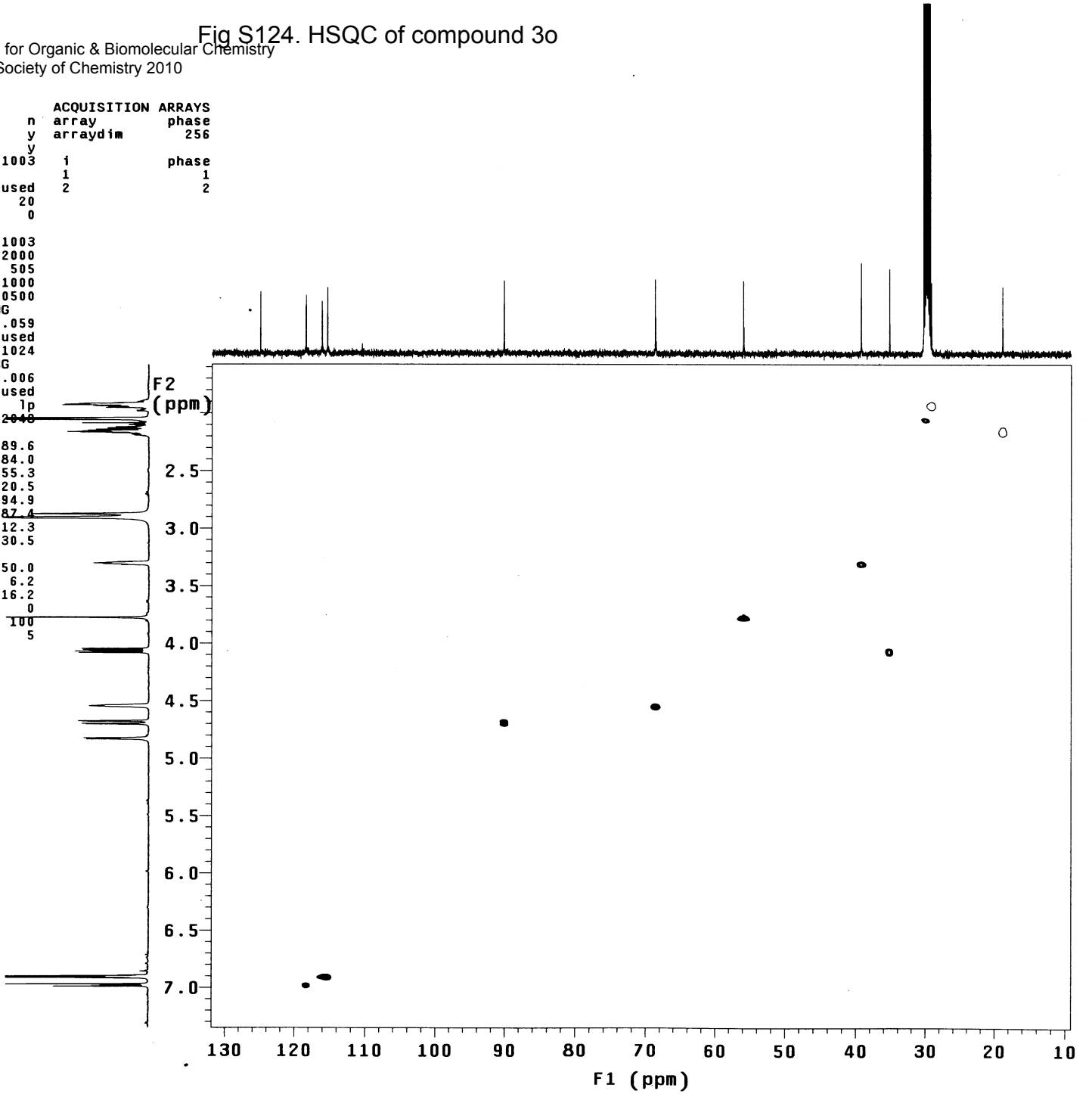


Fig S125. COSY of compound 3o

PMK-02-406-f1-oxidized

exp16 gCOSY

SAMPLE		FLAGS	
date	Aug 25 2010	hs	nn
solvent	Acetone	sspul	n
sample	undefined	hsglvl	1003
ACQUISITION		SPECIAL	
sw	4001.6	temp	not used
at	0.128	gain	28
np	1024	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.064
d1	1.000	sbs	not used
nt	8	fn	1024
2D ACQUISITION		F1 PROCESSING	
sw1	4001.6	sb1	-0.032
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	1024
sfrq	499.831	DISPLAY	
tof	-499.9	sp	816.9
tpwr	58	wp	2774.5
pw	11.100	sp1	819.0
GRADIENTS		wp1	2774.5
gzlvl1	1003	rfl	1898.9
gt1	0.001000	rfp	1887.4
gstab	0.000500	rfl1	1896.8
DECOUPLER		rfp1	1887.3
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	100
		th	8
		ai cdc av	

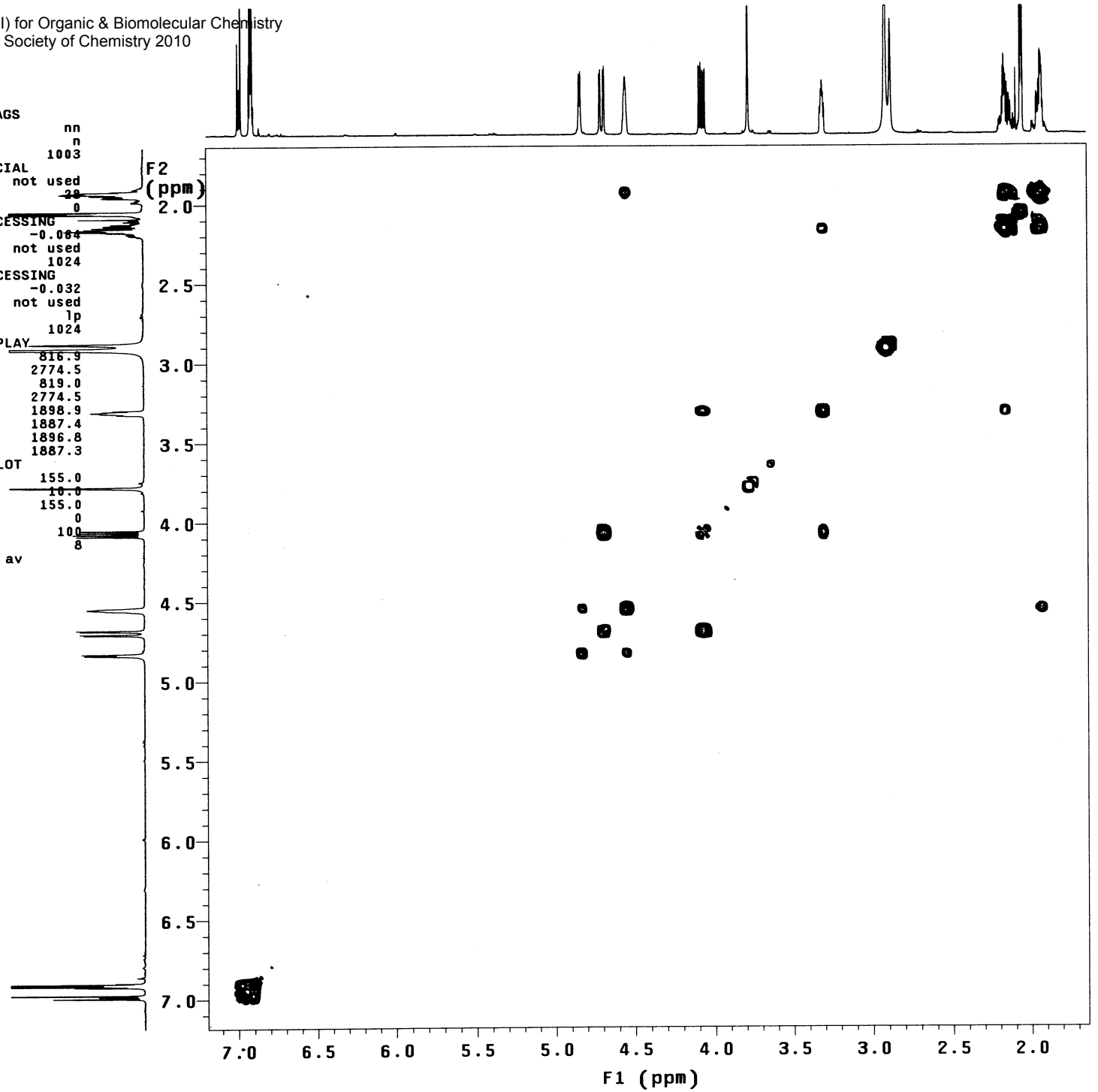


Fig S126. NOESY of compound 3o

PMK-02-106-f1-oxidized
exp17 NOESY

SAMPLE	
date	Aug 25 2010
solvent	Acetone
sample	undefined
ACQUISITION	
sw	4001.6
at	0.128
np	1024
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	4001.6
ni	200
TRANSMITTER	
tn	H1
sfrq	499.831
tof	-499.9
tpwr	58
pw	11.100
NOESY	
mix	0.600
PRESATURATION	
satmode	nnnn
satpwr	0
satdly	0
satfrq	0
DECOUPLER	
dn	C13
dm	nnn

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1003
SPECIAL	
temp	not used
gain	28
spin	0
F2 PROCESSING	
gf	0.059
gfs	not used
fn	1024
F1 PROCESSING	
gf1	0.046
gfs1	not used
proc1	lp
fn1	1024
DISPLAY	
sp	816.9
wp	2805.8
sp1	809.5
wp1	2805.8
rfl	1898.9
rfp	1887.4
rfl1	1898.5
rfp1	1887.3
PLOT	
wc	155.0
sc	10.0
wc2	155.0
sc2	0
vs	100
th	1
ai	ph

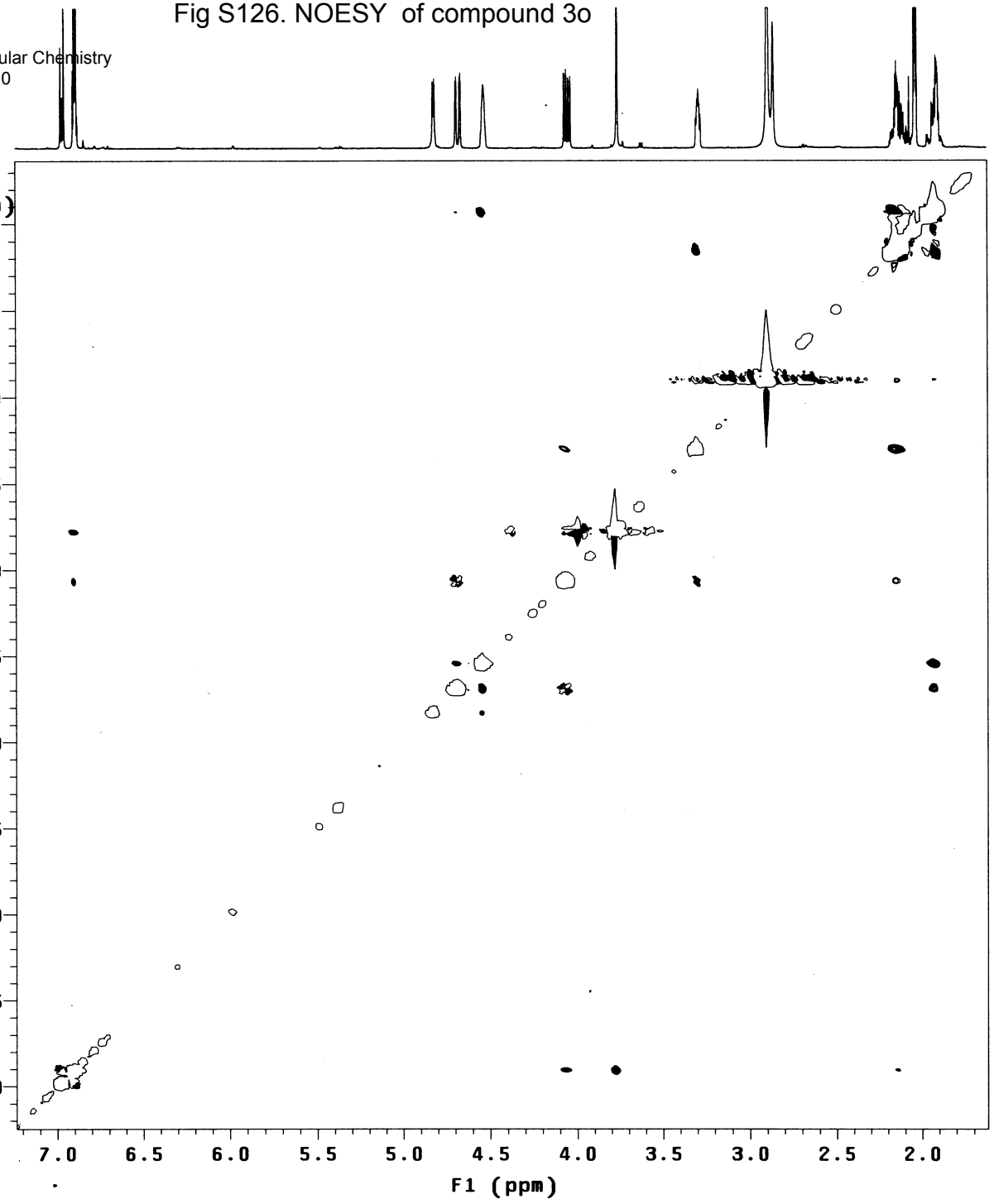
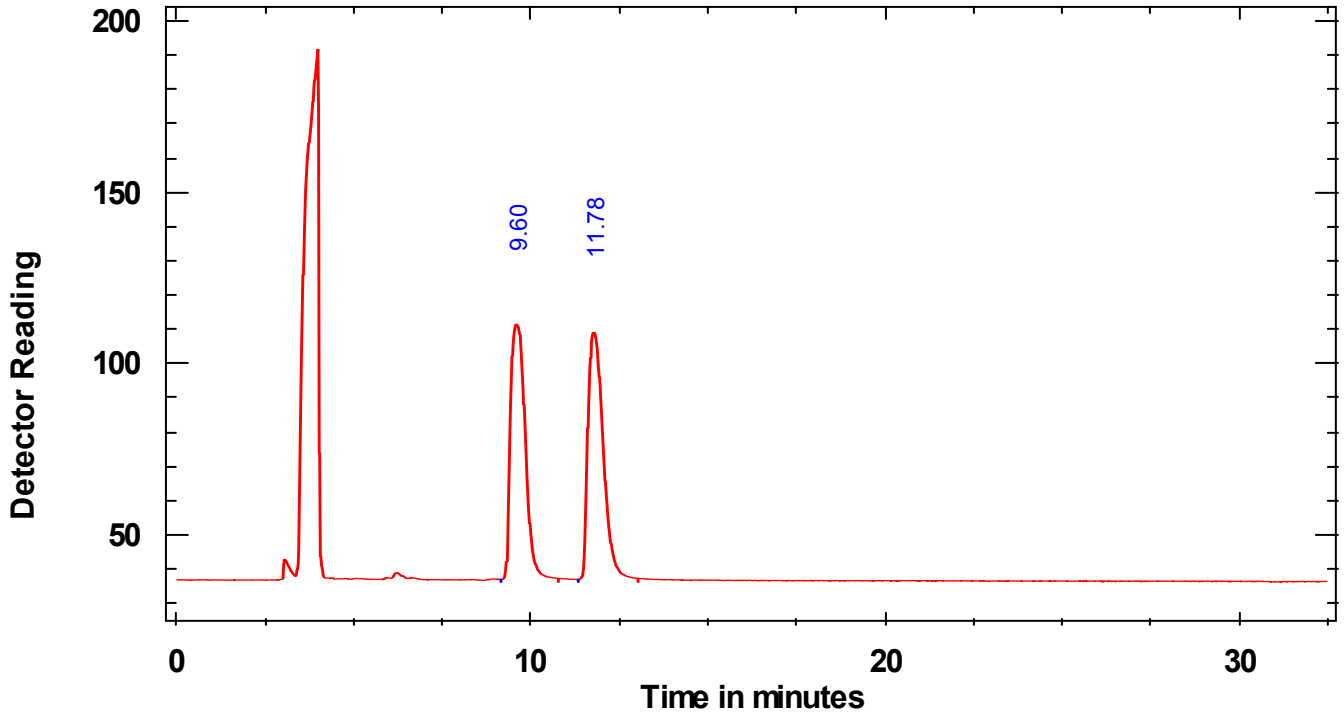


Fig S127. HPLC analysis of racemic compound 3a. (For comparison, Table 2, entry 1)

Chromatogram Report

PMK-02-340-racemate-colm-IA-8%ipa/hex

Report produced on 2010/3/19 at 上午 11:00:51 by Put your name here



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2010/3/19 ¤W¤È 11:00:01 Run stopped by operator

PEAK REPORT

#	begin	end	area	percent	maximum	time	begins as	name
1	9.15	10.78	2146	49.9	111.14	9.60	Baseline	
2	11.34	13.03	2151	50.1	108.96	11.78	Baseline	

Data taken from file [A01482 PMK-02-340-racemate-colm-IA-8%ipa_hex on 三月 19 2010 11 00 上午 11_00 .chr]

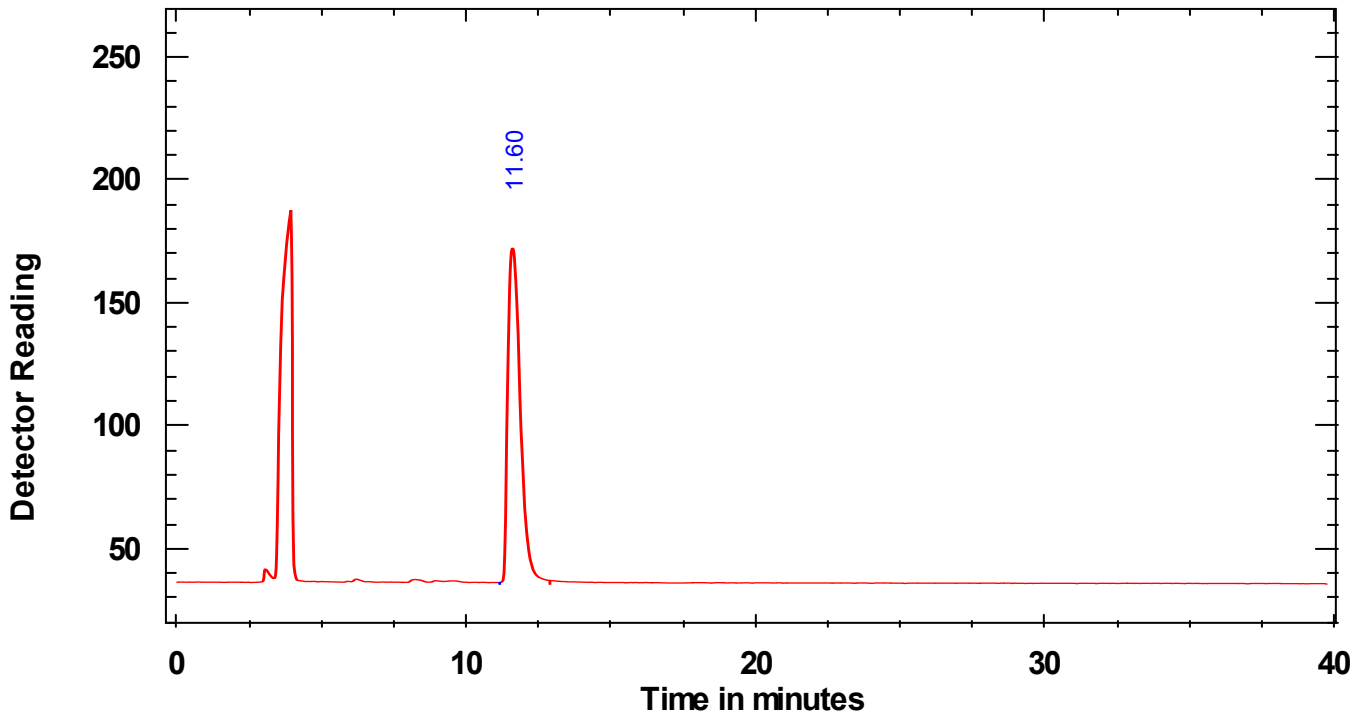
D-Star Instruments - 8424 Quarry Rd., Manassas VA 20110 - 800-DSTAR12

Fig S128. HPLC analysis of compound 3a obtained. (Table 2, entry 1)

Chromatogram Report

PMK-02-336-chiral-colm-IA-8%ipa/hex

Report produced on 2010/3/19 at 上午 11:44:07 by Put your name here



2010/3/19 ¤W¤È 11:03:54 Flow set to 1.00 at 0.00 minutes

2010/3/19 ¤W¤È 11:43:43 Run stopped by operator

PEAK REPORT

#	begin	end	area	percent	maximum	time	begins as	name
1	11.16	12.90	4041	100.0	172.09	11.60	Baseline	

Data taken from file [A01483 PMK-02-336-chiral-colm-IA-8%ipa_hex on 三月 19 2010 11 43 上午 11_43 .chr]

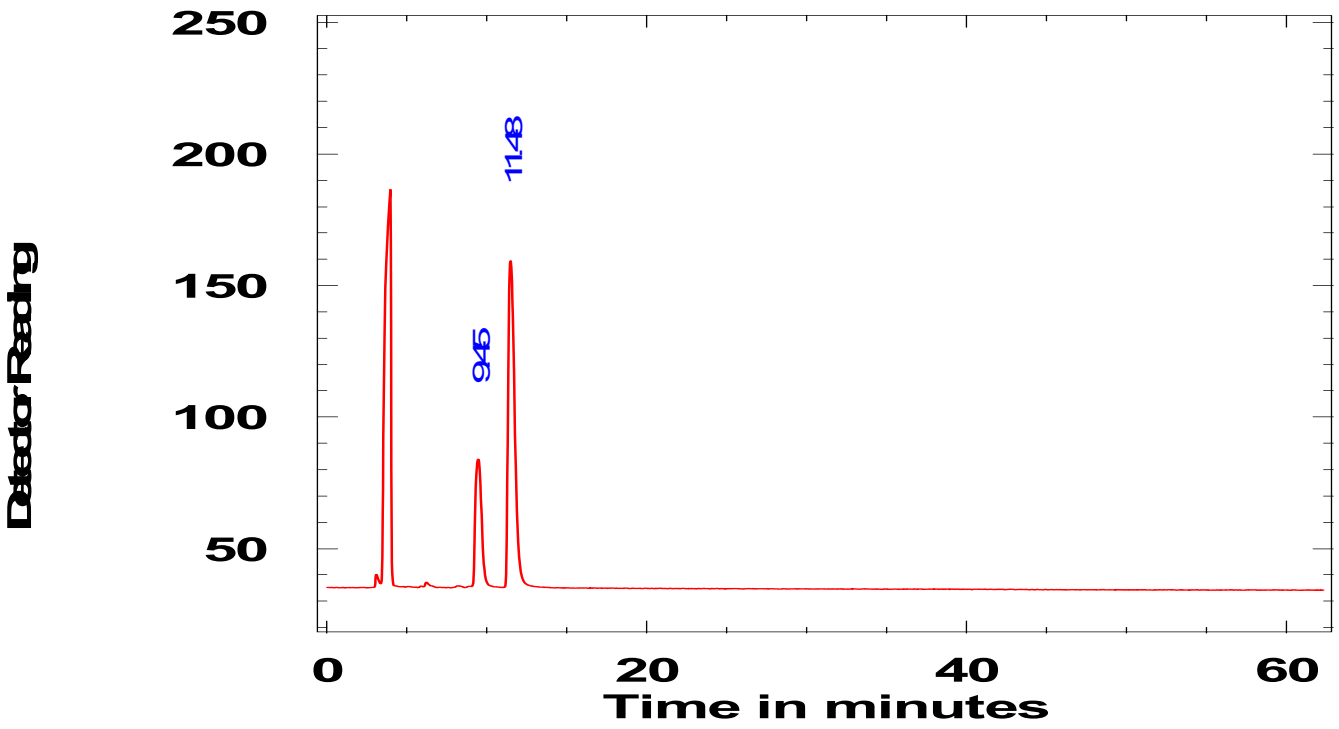
D-Star Instruments - 8424 Quarry Rd., Manassas VA 20110 - 800-DSTAR12

Fig S129. HPLC analysis of the mixture of racemic and chiral compound 3a obtained. (For comparison, Table 2, entry 1)

Peak Report

PMK-02-336-chiral+racemate-colm-IA-8%ipa/hex

Report produced on 2010/3/19 at 下午 01:26:19 by Put your name here



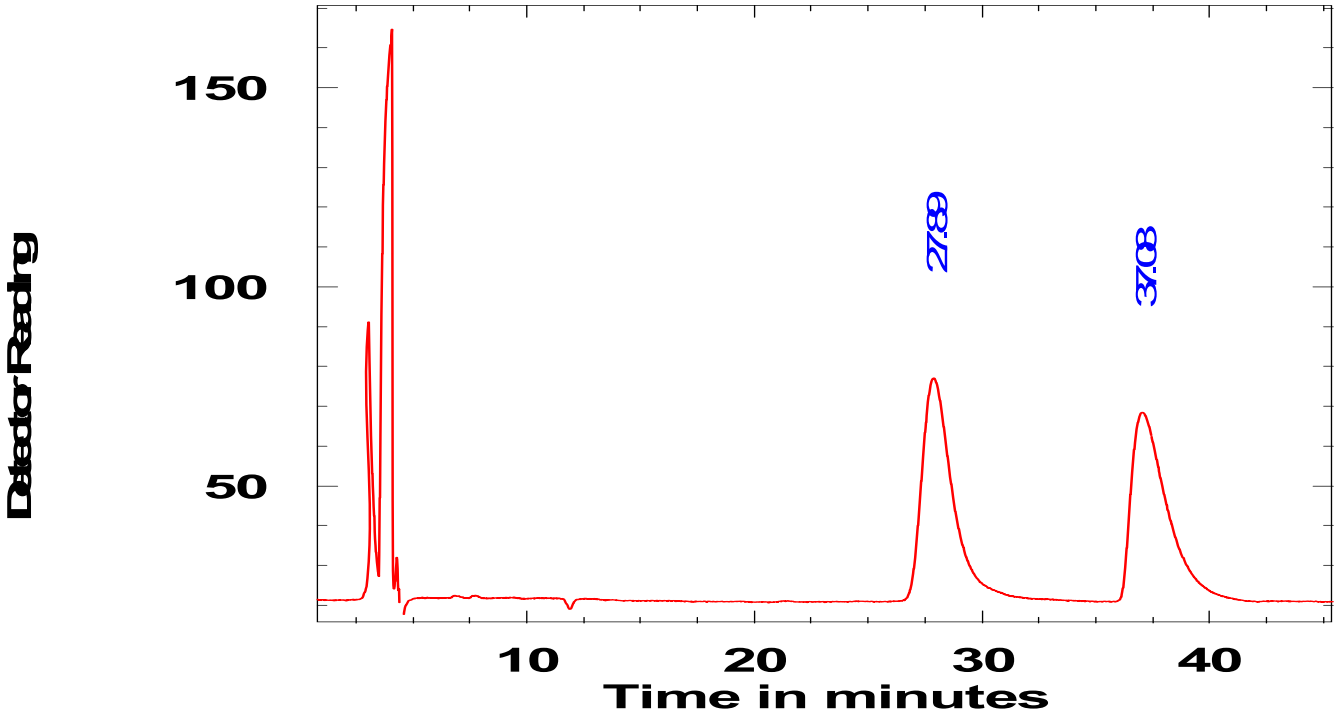
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	8.68	10.42	1322	83.91	9.45	27.3	Baseline
2	11.02	12.73	3519	159.29	11.48	72.7	Baseline

Fig S130. HPLC analysis of racemic compound 3b.
(For comparison, Table 2, entry 2)

Peak Report

PMK-02-364-racemate-colm-OD-6%ipa/Hex

Report produced on 2010/7/16 at 下午 05:44:16 by Put your name here



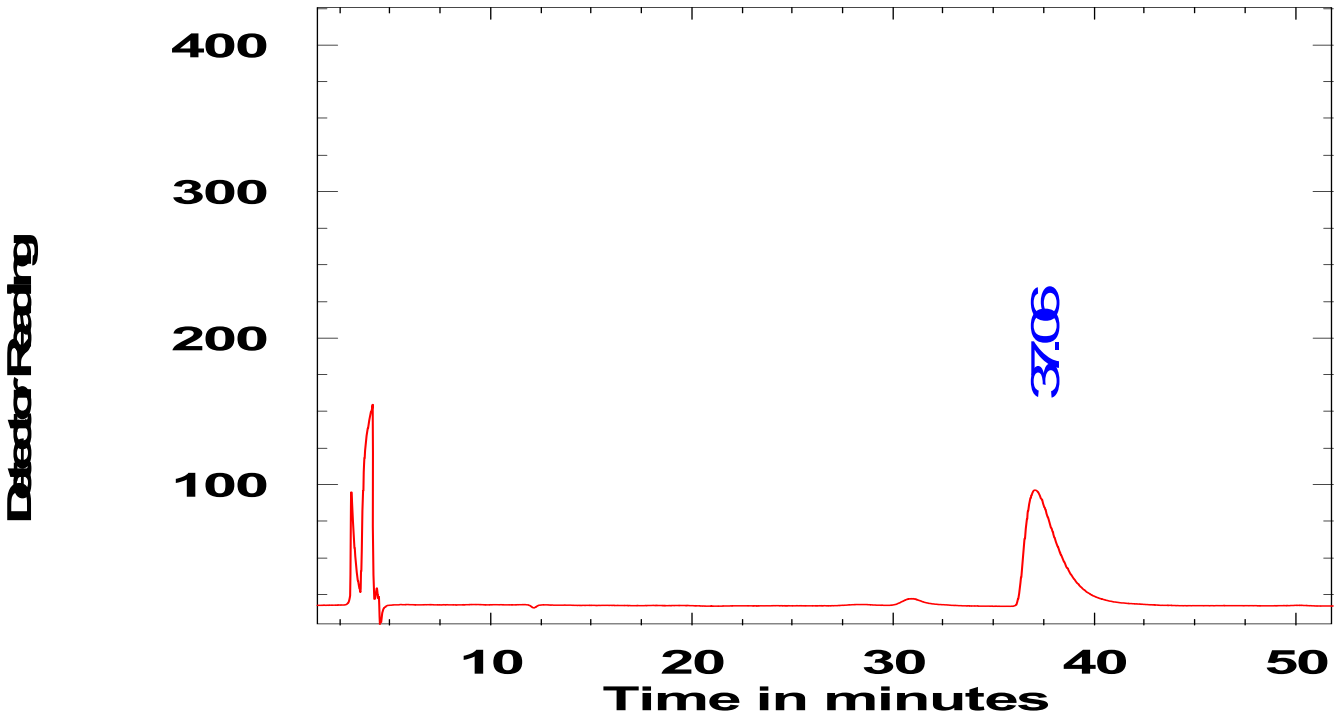
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	26.55	31.12	5008	77.07	27.89	50.7	Baseline
2	36.14	41.26	4874	68.42	37.08	49.3	Baseline

Fig S131. HPLC analysis of compound 3b obtained. (Table 2, entry 2)

Peak Report

PMK-02-364-Chiral-colm-OD-6%ipa/Hex

Report produced on 2010/7/16 at 下午 05:49:10 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	35.88	41.28	8854	96.16	37.06	100.0	Baseline

Fig S132. HPLC analysis of the mixture of racemic and chiral compound 3b obtained. (For comparison, Table 2, entry 2)

Peak Report

PMK-02-364-Chiral+racemate-colm-OD-6%ipa/Hex

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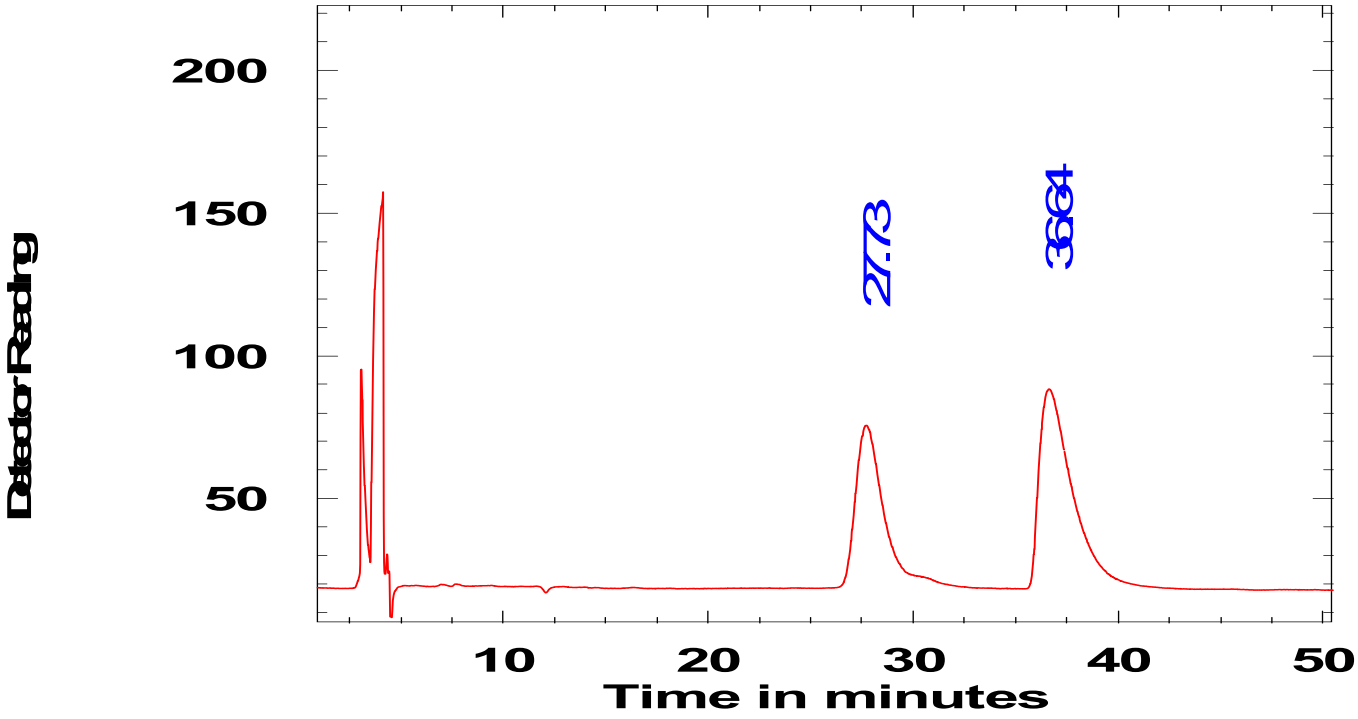


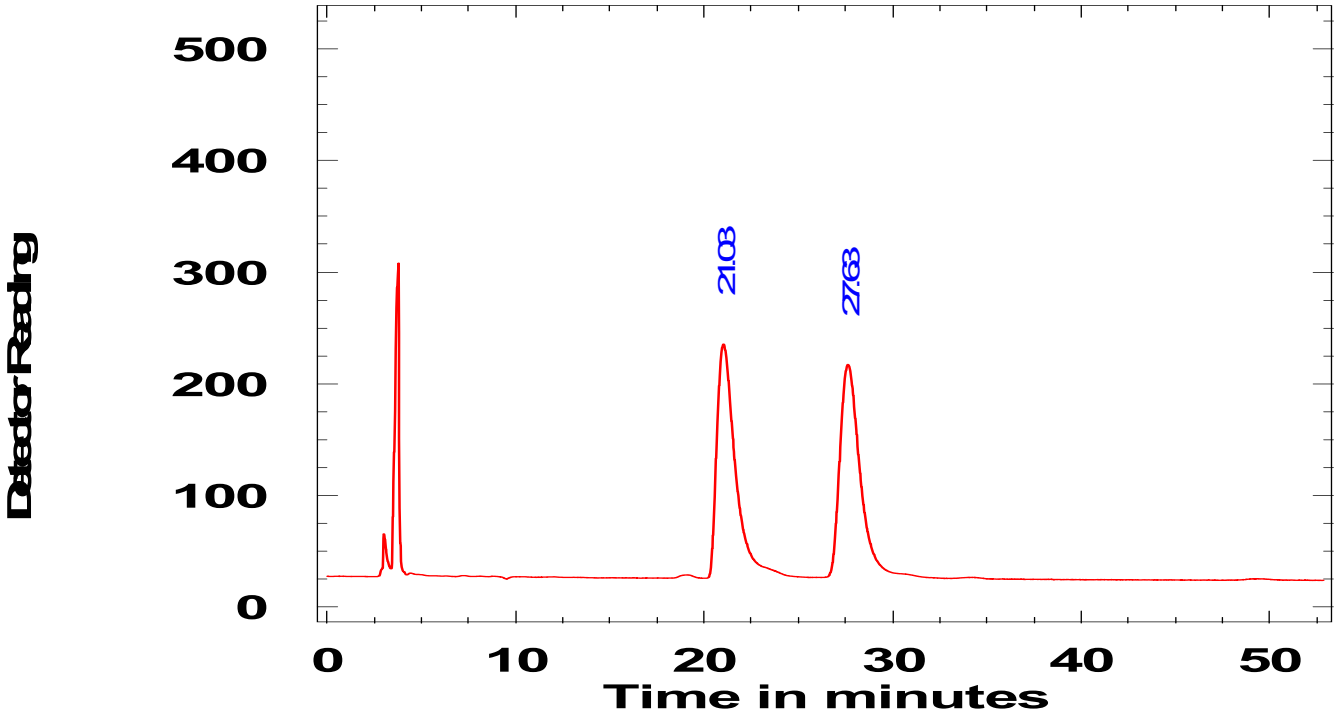
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	26.68	29.40	4120	75.59	27.73	34.2	Baseline
2	35.14	42.79	7942	88.31	36.64	65.8	Baseline

Fig S133. HPLC analysis of racemic compound 3c.
(For comparison, Table 2, entry 3)

Peak Report

PMK-02-365-racemate-colm-OD-10%ipa/Hex

Report produced on 2010/7/17 at 下午 04:27:56 by Put your name here



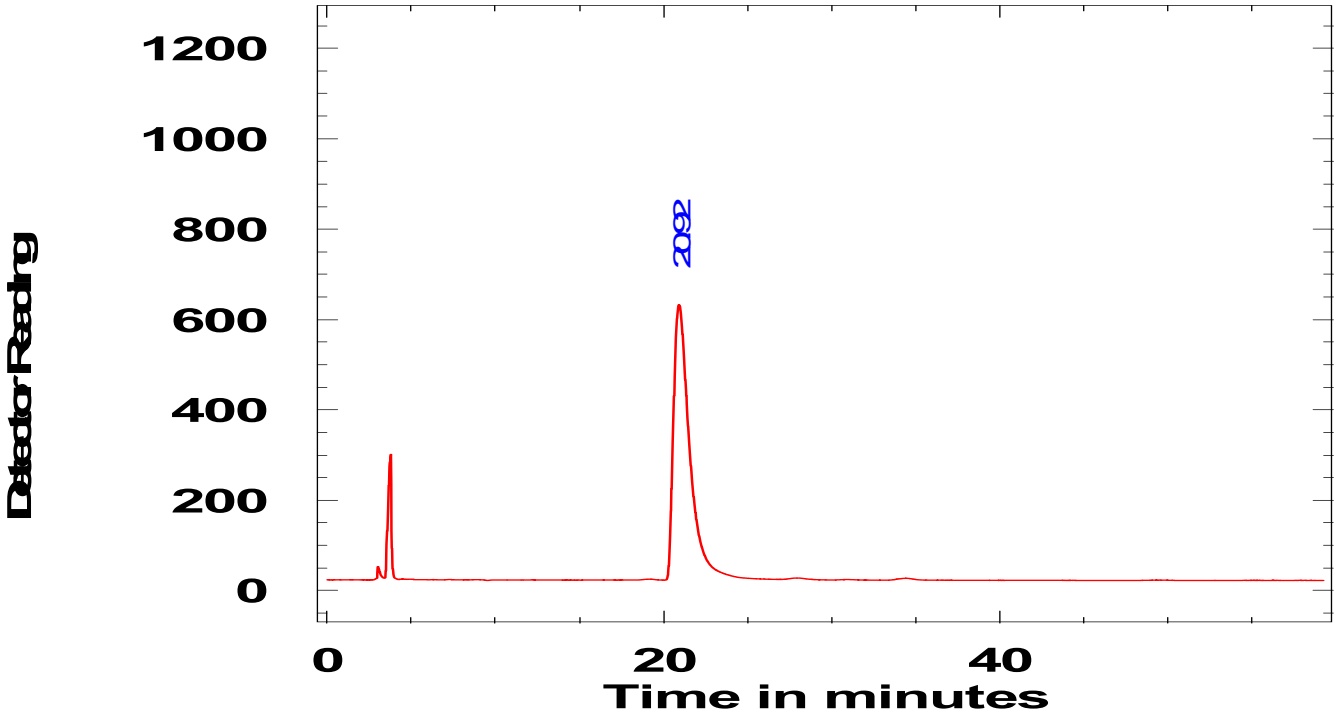
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	20.18	24.02	13992	235.50	21.03	50.6	Baseline
2	26.63	30.86	13674	217.21	27.63	49.4	Baseline

Fig S134. HPLC analysis of compound 3c obtained. (Table 2, entry 3)

Peak Report

PMK-02-365-chiral-colm-OD-10%ipa/Hex

Report produced on 2010/7/17 at 下午 04:26:18 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	20.28	24.92	37595	632.83	20.92	100.0	Baseline

Fig S135. HPLC analysis of the mixture of racemic and chiral compound 3c obtained. (For comparison, Table 2, entry 3)

Chromatogram Report

PMK-02-365-chiral+racemate-colm-OD-10%ipa/Hex

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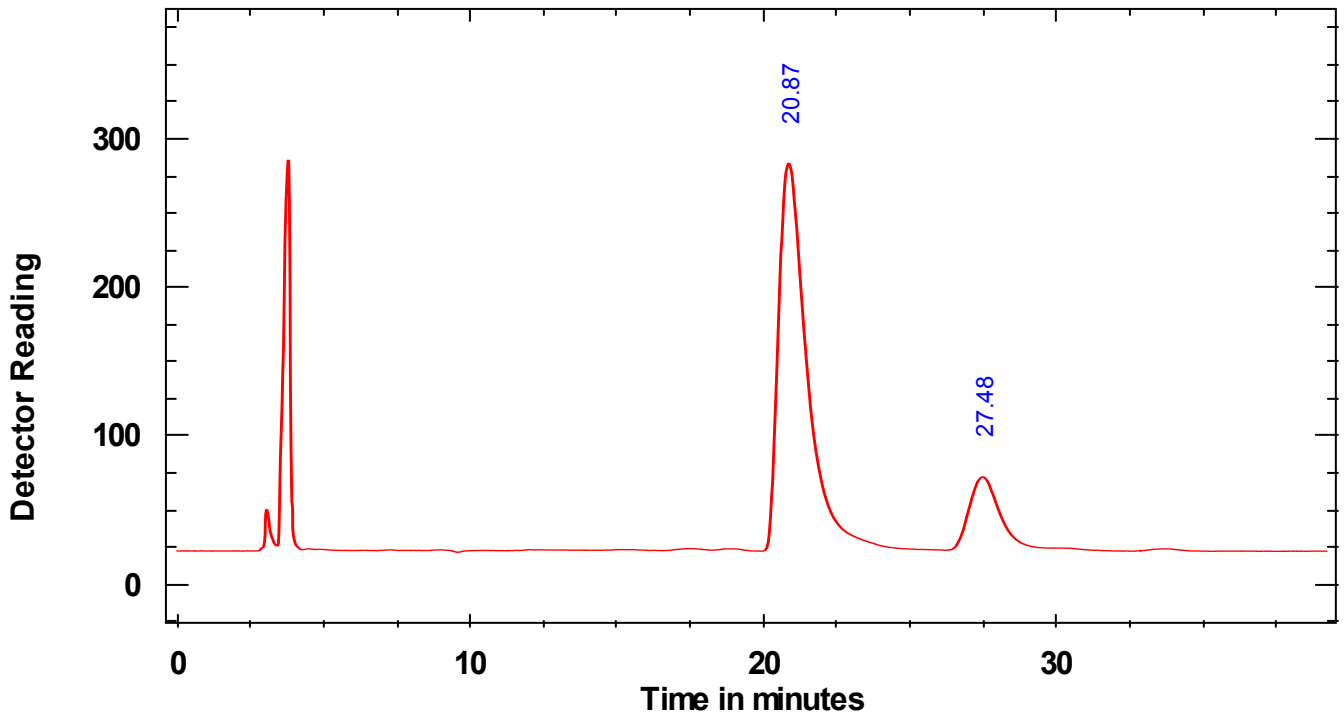


2010/7/17 ¤U¤È 03:42:50 Flow set to 1.00 at 0.00 minutes

2010/7/17 ¤U¤È 04:22:05 Run stopped by operator

PEAK REPORT

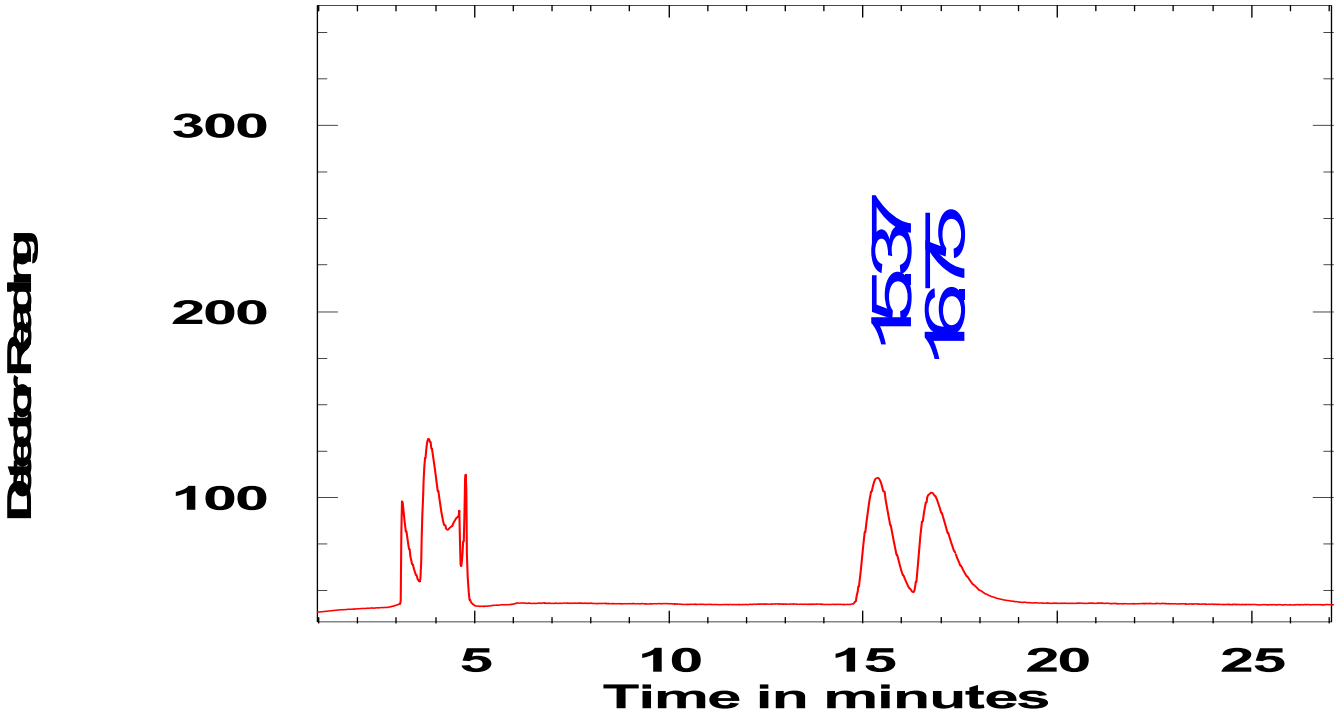
#	begin	end	area	percent	maximum	time	begins as	name
1	20.00	24.72	17725	83.5	283.50	20.87	Baseline	
2	26.22	29.42	3404	16.0	72.23	27.48	Baseline	

Fig S136. HPLC analysis of racemic compound 3d.
(For comparison, Table 2, entry 4)

Peak Report

PMK-02-368-racemate-colm-OD-4%ipa/Hex

Report produced on 2010/7/19 at 下午 08:10:52 by Put your name here



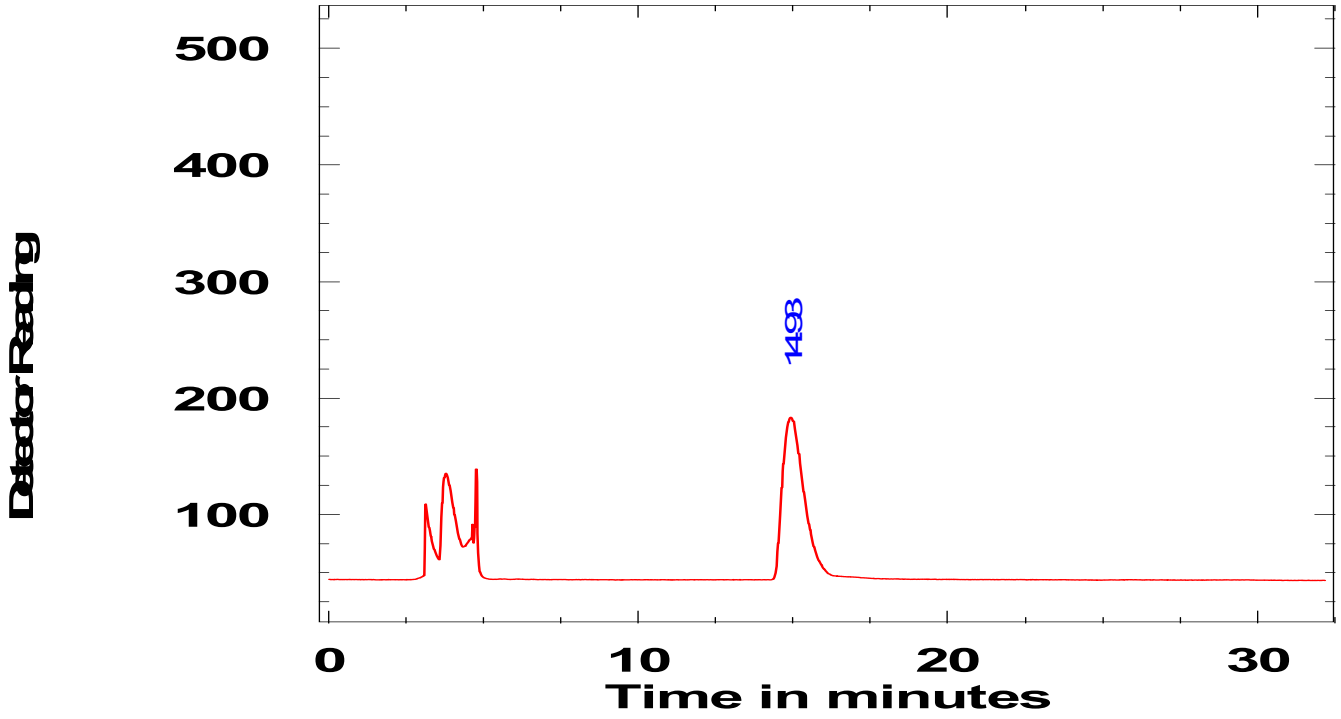
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	14.68	16.24	2877	110.55	15.37	51.7	Baseline
2	16.34	18.56	2686	102.65	16.75	48.3	Baseline

Fig S137. HPLC analysis of compound 3d obtained. (Table 2, entry 4)

Peak Report

PMK-02-368-chiral-colm-OD-4%ipa/Hex

Report produced on 2010/7/19 at 下午 06:18:06 by Put your name here



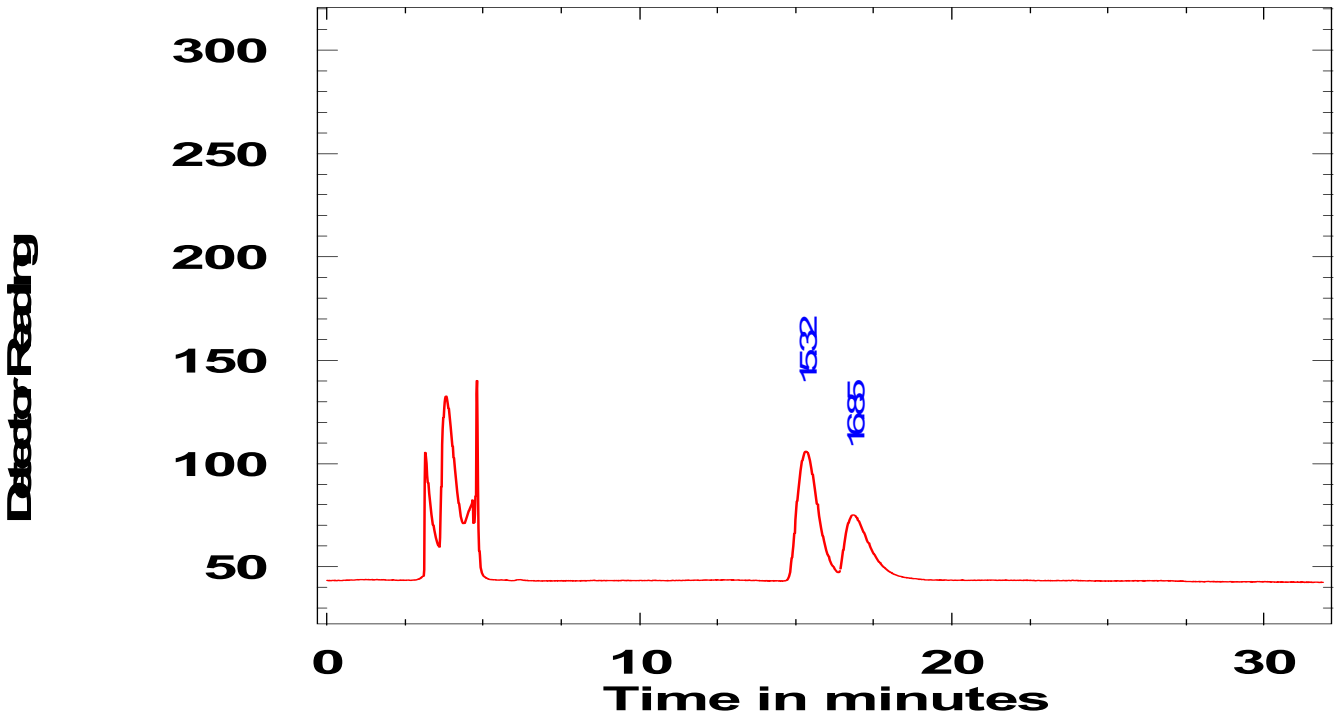
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	14.45	16.32	6140	183.38	14.93	100.0	Baseline

Fig S138. HPLC analysis of the mixture of racemic and chiral compound 3d obtained. (For comparison, Table 2, entry 4)

Peak Report

PMK-02-368-chiral+racemate-colm-OD-4%ipa/Hex

Report produced on 2010/7/19 at 下午 06:21:34 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	14.63	16.39	2759	105.92	15.32	77.3	Baseline
2	16.54	18.29	812	75.10	16.85	22.7	Baseline

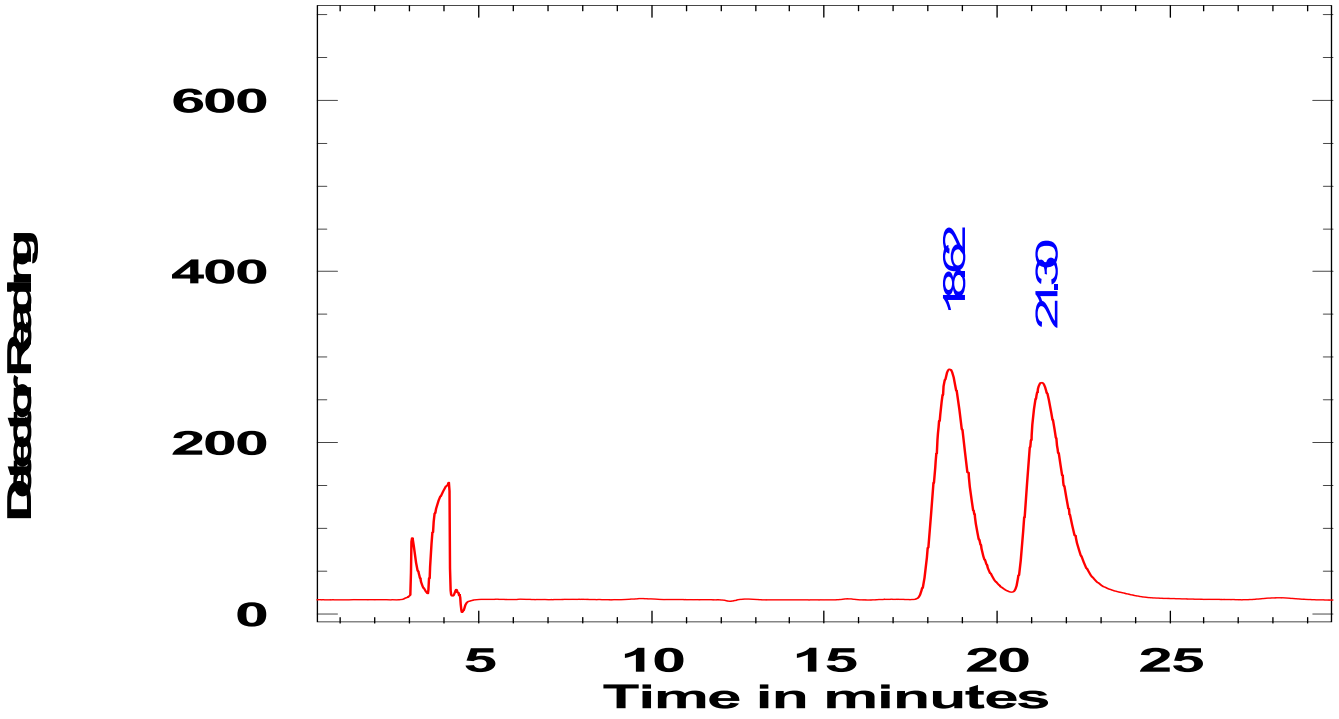
Data taken from file [PMK-02-368-chiral+racemate-colm-OD-4%ipa/Hex]
D-Star Instruments - 8424 Quarry Rd., Manassas VA 20110 - 800-DSTAR12

Fig S139. HPLC analysis of racemic compound cis-3e.
(For comparison, Table 2, entry 5)

Peak Report

PMK-02-371F1-racemate-colm-OD-6%ipa/Hex

Report produced on 2010/7/16 at 下午 05:42:02 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	17.55	20.51	17202	285.81	18.62	50.6	Baseline
2	20.56	24.02	16784	270.22	21.30	49.4	Baseline

Fig S140. HPLC analysis of compound cis-3e obtained. (Table 2, entry 5)

Peak Report

PMK-02-371F1-chiral-colm-OD-6%ipa/Hex

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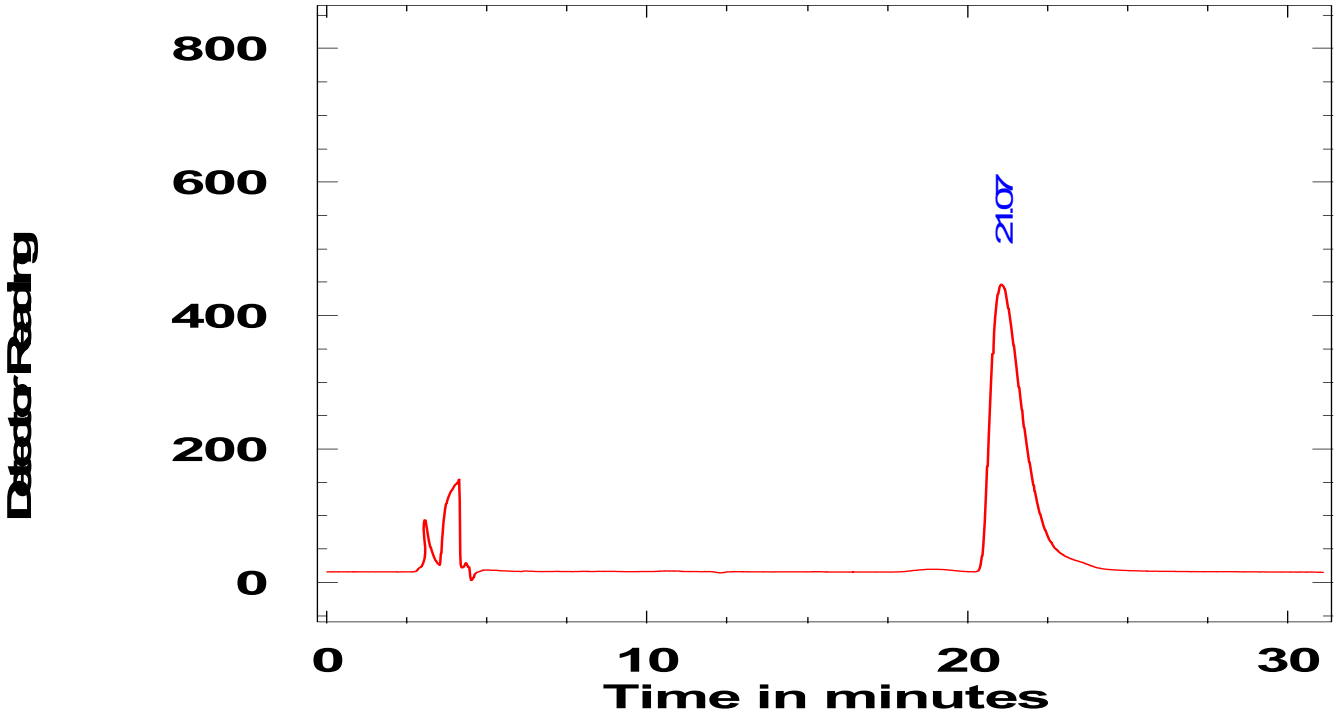


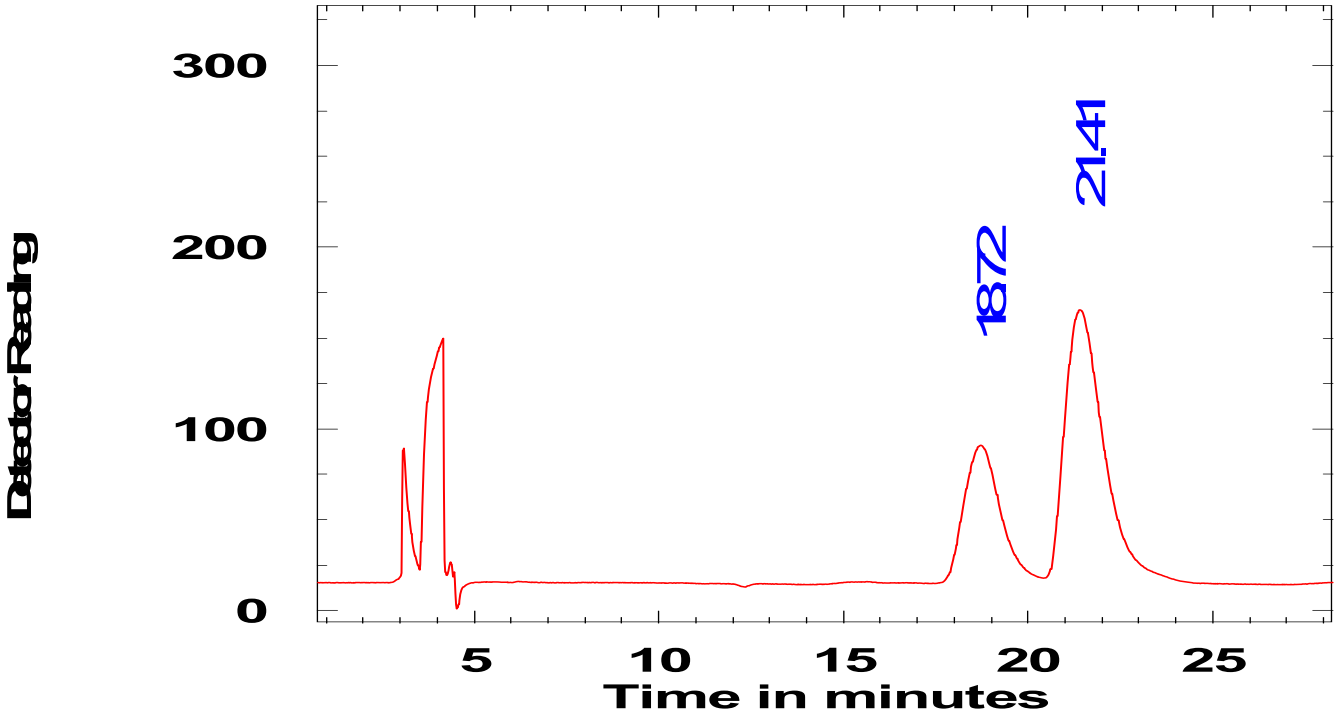
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	20.34	23.95	30647	446.82	21.07	100.0	Baseline

Fig S141. HPLC analysis of the mixture of racemic and chiral compound cis-3e obtained. (For comparison, Table 2, entry 5)

Peak Report

PMK-02-371F1-chiral+racemate-colm-OD-6%ipa/Hex

Report produced on 2010/7/16 at 下午 05:36:58 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	17.70	19.82	4353	90.94	18.72	29.4	Baseline
2	20.56	24.04	10442	165.37	21.41	70.6	Baseline

Data taken from file [PMK-02-371F1-chiral+racemate-colm-OD-6%ipa/Hex]
D-Star Instruments - 8424 Quarry Rd., Manassas VA 20110 - 800-DSTAR12

Fig S142. HPLC analysis of racemic compound trans-3e.
(For comparison, Table 2, entry 5)

Peak Report

PMK-02-371F2-racemate-colm-IA-5%ipa/hex

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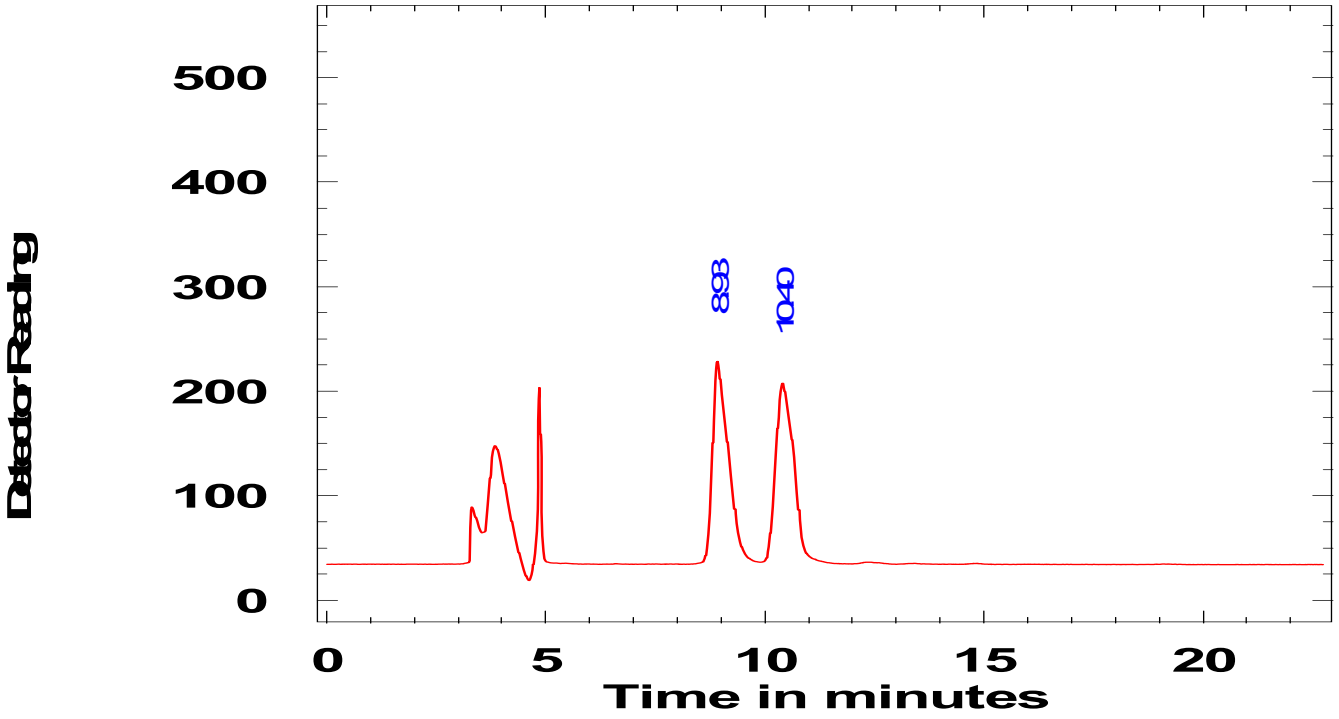

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	8.56	9.71	4730	228.35	8.93	49.9	Baseline
2	10.01	11.24	4747	207.41	10.40	50.1	Baseline

Fig S143. HPLC analysis of compound trans-3e obtained. (Table 2, entry 5)

Peak Report

PMK-02-371F2-chiral-colm-IA-5%ipa/hex

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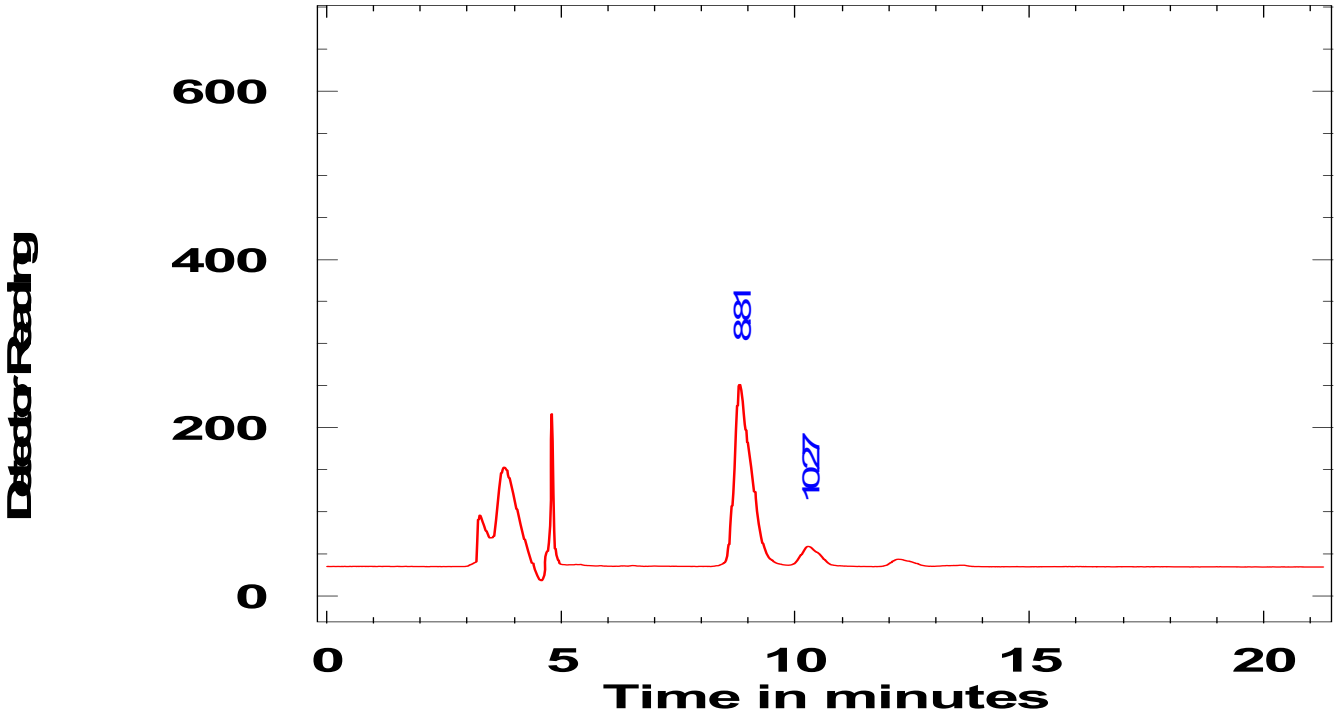

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	8.38	9.71	5329	251.24	8.81	93.1	Baseline
2	9.93	10.63	398	58.78	10.27	6.9	Baseline

Fig S144. HPLC analysis of the mixture of racemic and chiral compound trans-3e obtained. (For comparison, Table 2, entry 5)

Peak Report

PMK-02-371F2-chiral+racemate-colm-IA-5%ipa/hex

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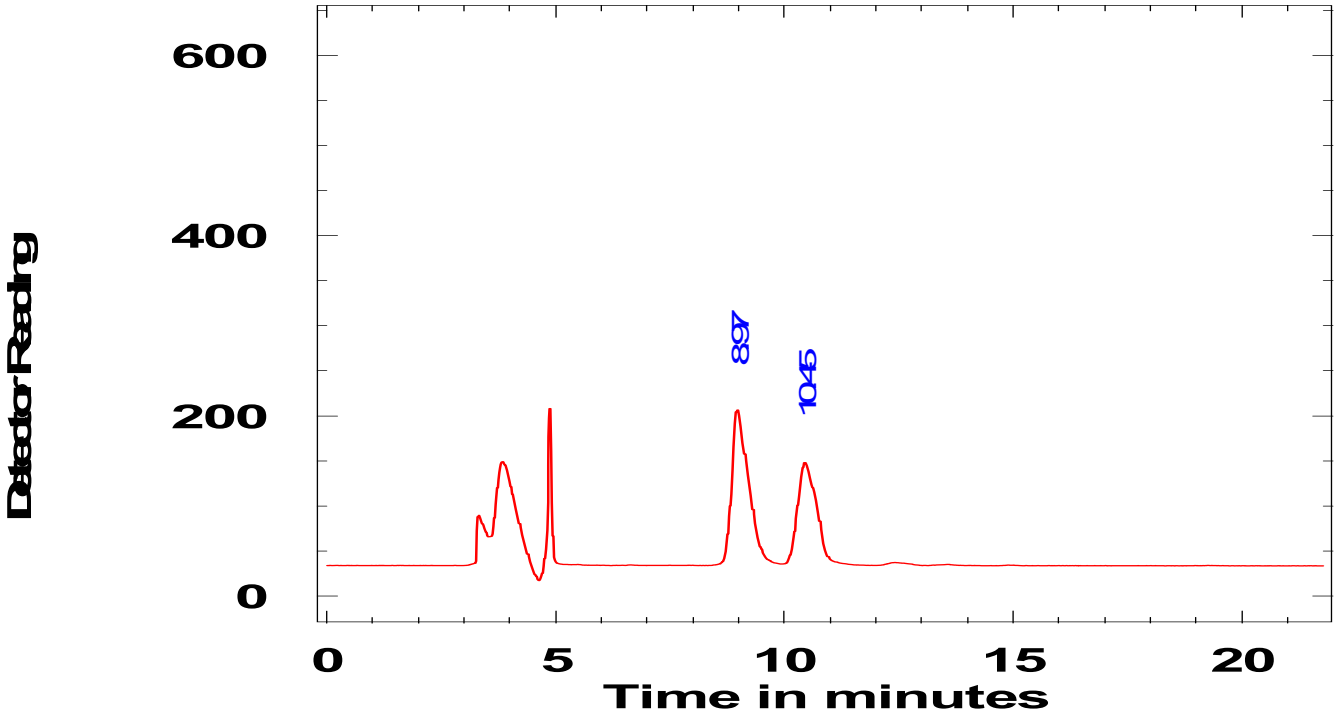

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	8.38	10.02	4388	206.33	8.97	59.9	Baseline
2	10.09	11.07	2943	147.92	10.45	40.1	Baseline

Fig S145. HPLC analysis of racemic compound 3f.(For comparison, Table 2, entry 6)

Peak Report

PMK-02-375-racemate-colm-IA-6%ipa/Hex

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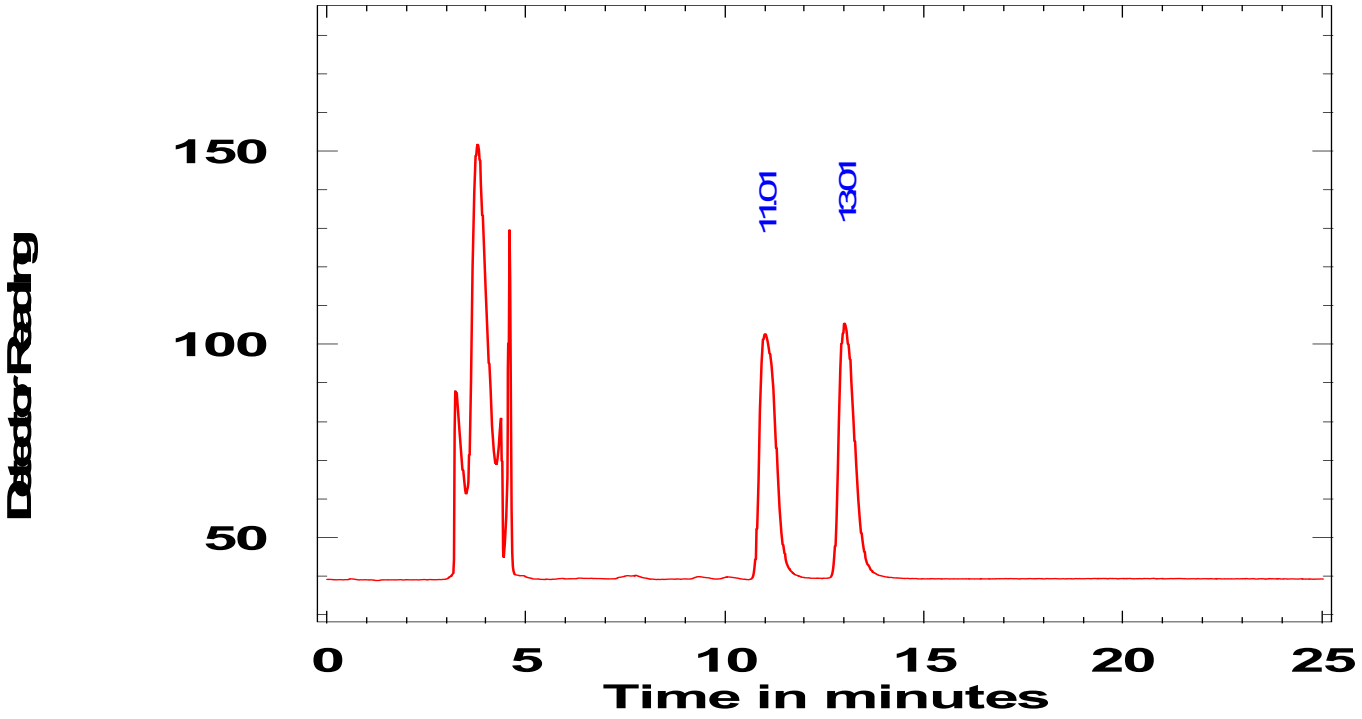

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	10.61	11.78	1741	102.71	11.01	50.0	Baseline
2	12.70	14.04	1740	105.43	13.01	50.0	Baseline

Fig S146. HPLC analysis of compound 3f obtained. (Table 2, entry 6)

Peak Report

PMK-02-375-chiral-colm-IA-6%ipa/Hex

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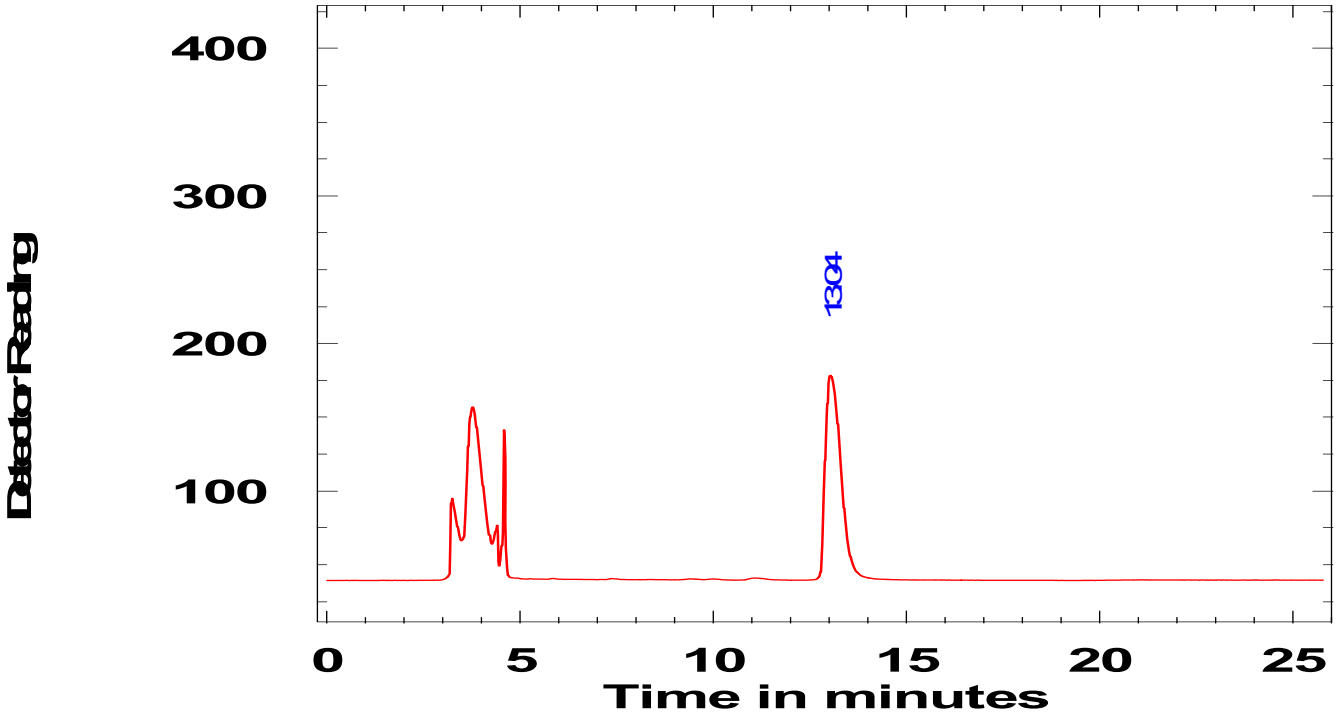

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	12.63	14.06	3850	178.32	13.04	100.0	Baseline

Fig S147. HPLC analysis of the mixture of racemic and chiral compound 3f obtained. (For comparison, Table 2, entry 6)

Peak Report

PMK-02-375-chiral+racemate-colm-IA-6%ipa/Hex

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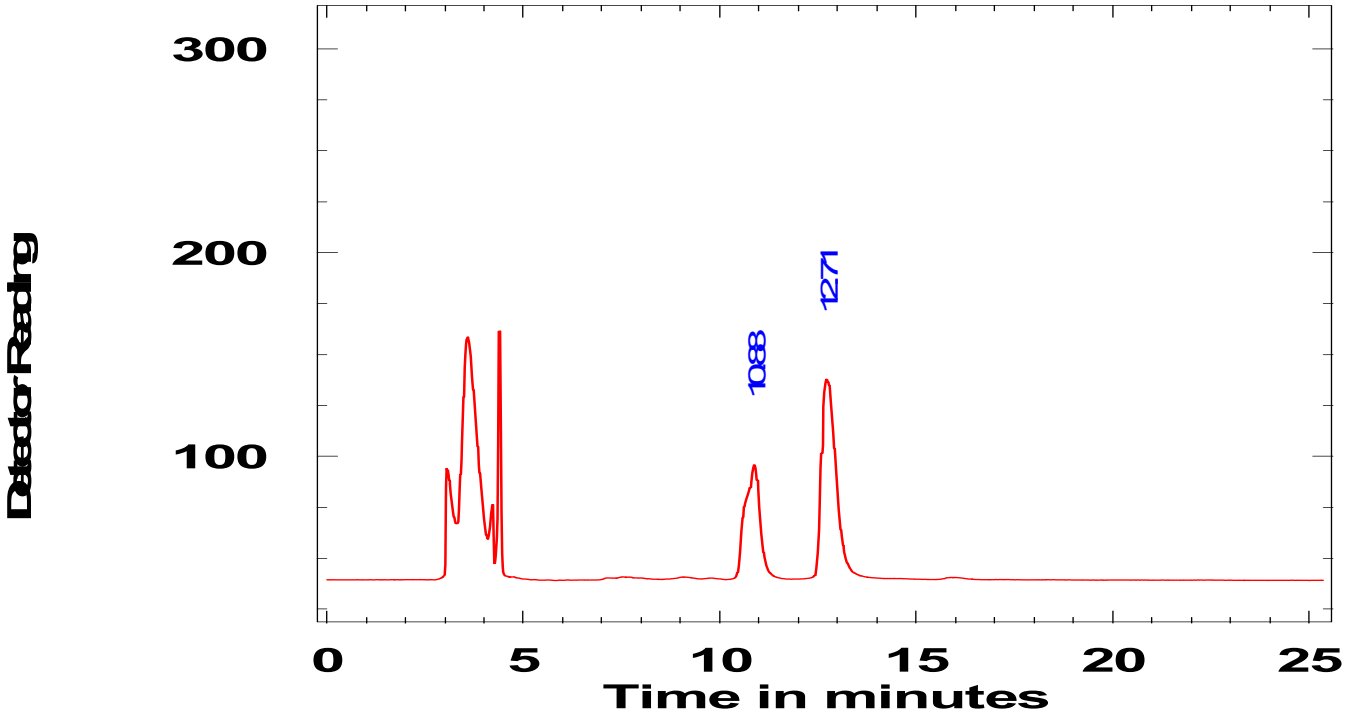

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	10.39	11.35	1413	95.81	10.88	37.8	Baseline
2	12.45	13.40	2320	137.83	12.71	62.2	Baseline

Fig S148. HPLC analysis of racemic compound 3g.

(For comparison, Table 2, entry 7)

Peak Report

PMK-02-379-racemate-colm-IA-6%ipa/Hex

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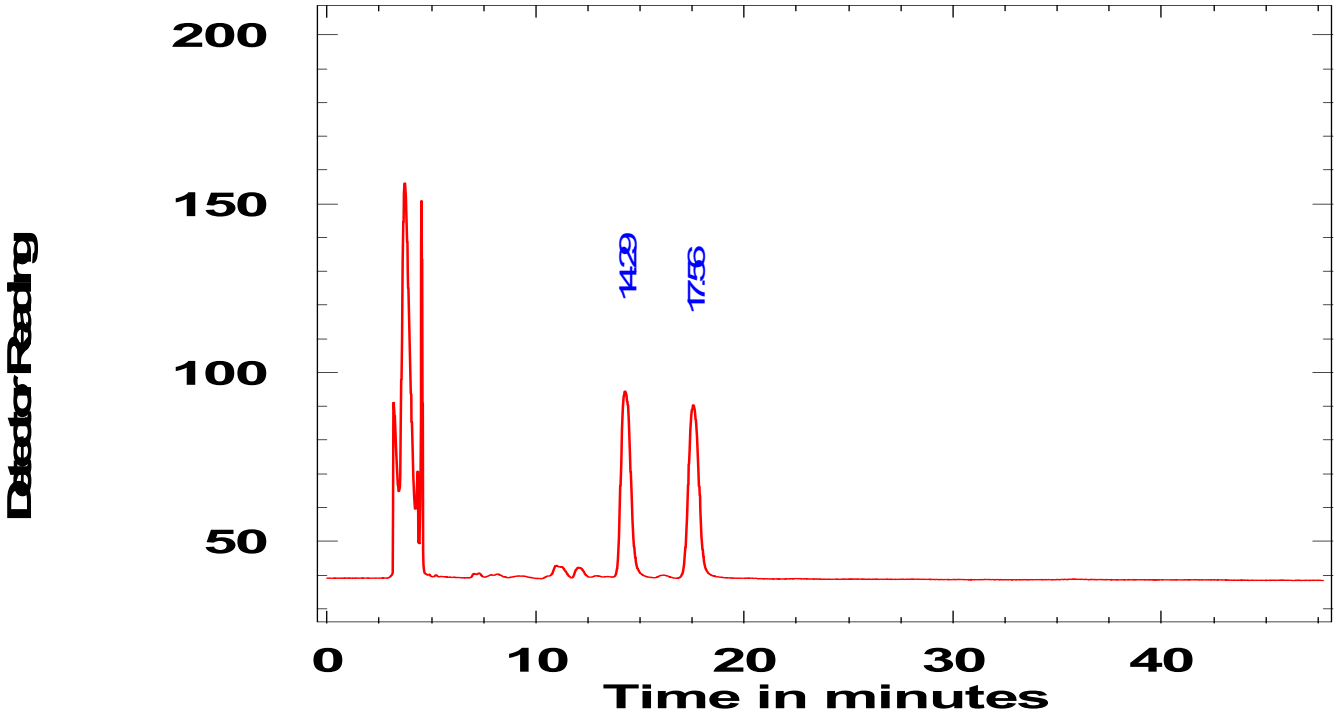

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	13.65	15.31	1759	94.46	14.29	49.9	Baseline
2	16.90	18.79	1768	90.40	17.56	50.1	Baseline

Fig S149. HPLC analysis of compound 3g obtained. (Table 2, entry 7)

Peak Report

PMK-02-379-chiral-colm-IA-6%ipa/Hex

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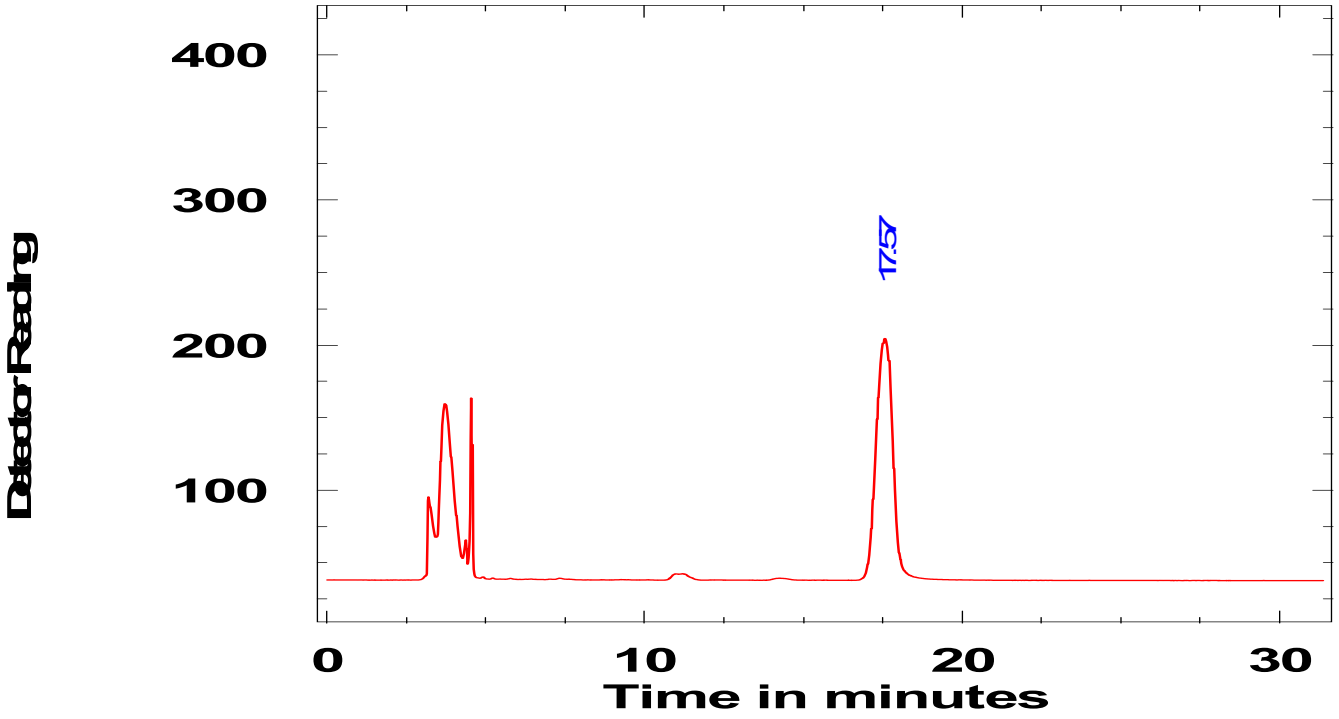

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	16.77	18.51	5938	204.58	17.57	100.0	Baseline

Fig S150. HPLC analysis of the mixture of racemic and chiral compound 3g obtained. (For comparison, Table 2, entry 7)

Peak Report

PMK-02-379-chiral+racemate-colm-IA-6%ipa/Hex

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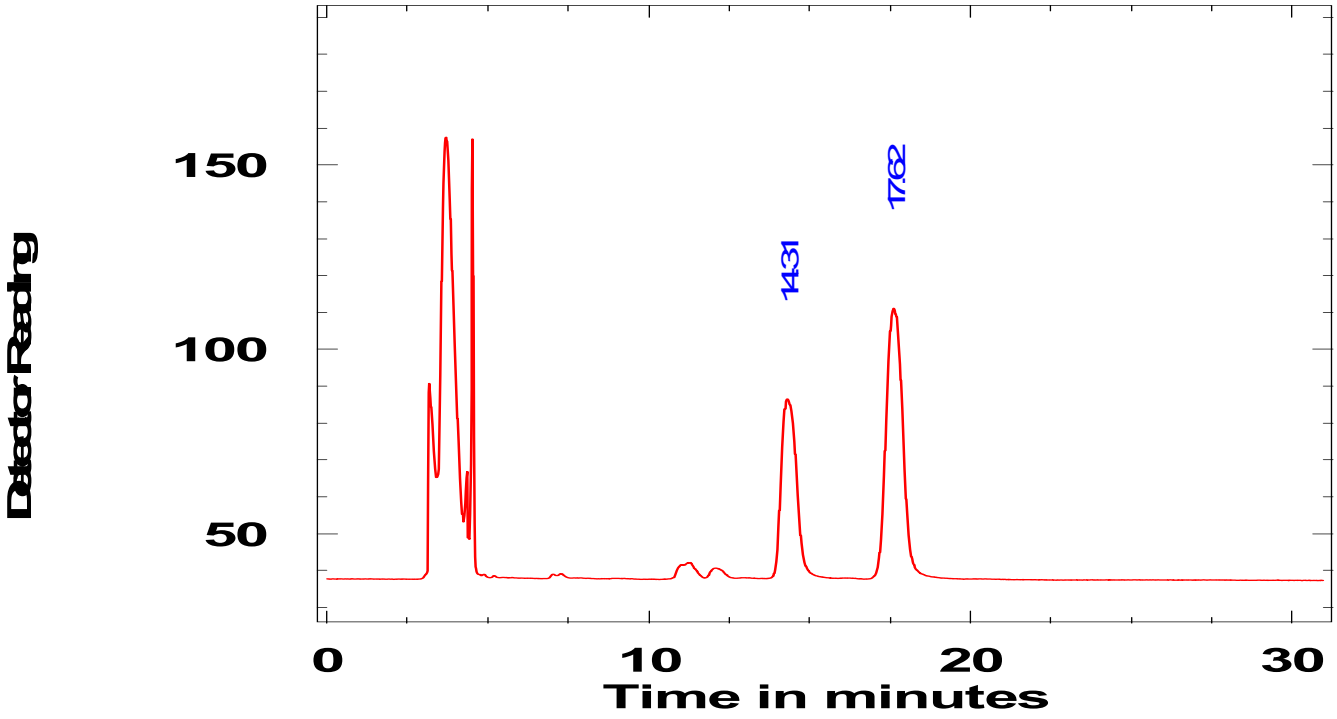

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	13.85	15.06	1529	86.55	14.31	36.8	Baseline
2	16.82	18.93	2623	111.13	17.62	63.2	Baseline

Fig S151. HPLC analysis of racemic compound 3h.
(For comparison, Table 2, entry 8)

Peak Report

PMK-02-390-racemate-colm-OD-10%ipa/hex

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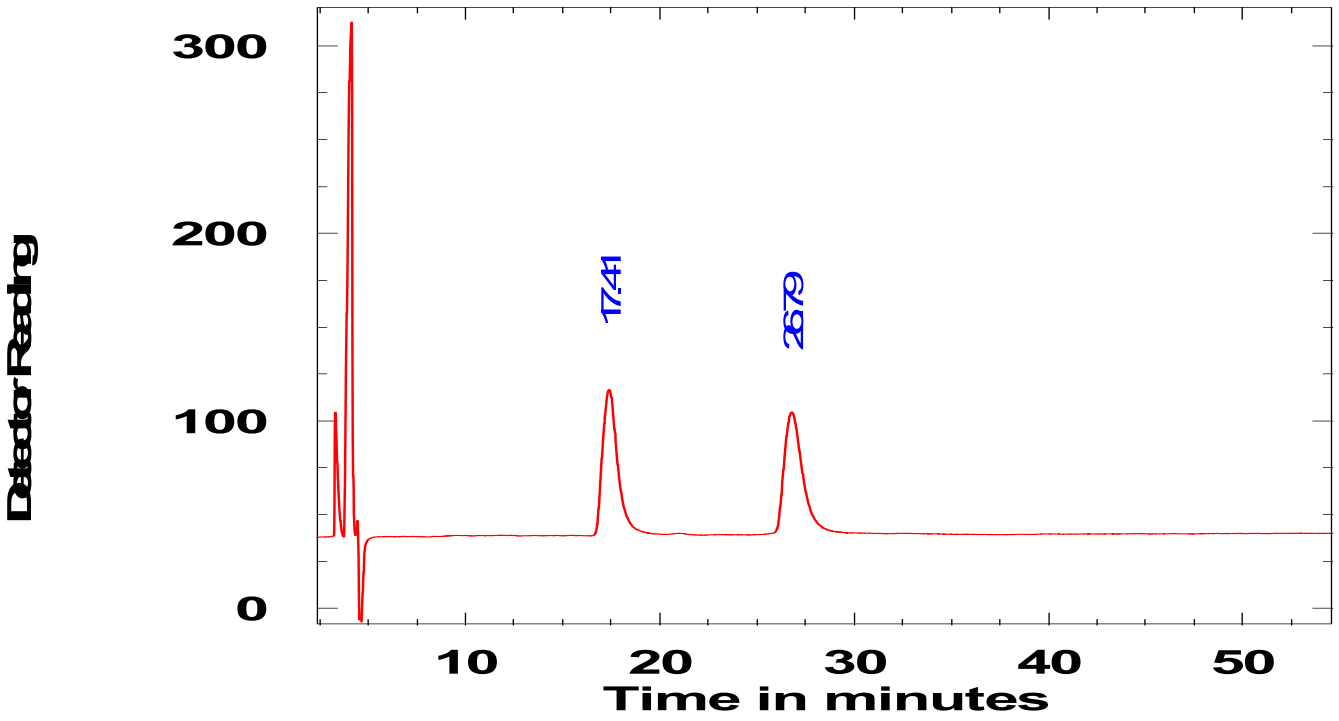

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	16.60	19.11	4085	116.60	17.41	49.2	Baseline
2	25.64	29.47	4212	104.63	26.79	50.8	Baseline

Fig S152. HPLC analysis of compound 3h obtained. (Table 2, entry 8)

Peak Report

PMK-02-390-chiral-colm-OD-10%ipa/hex

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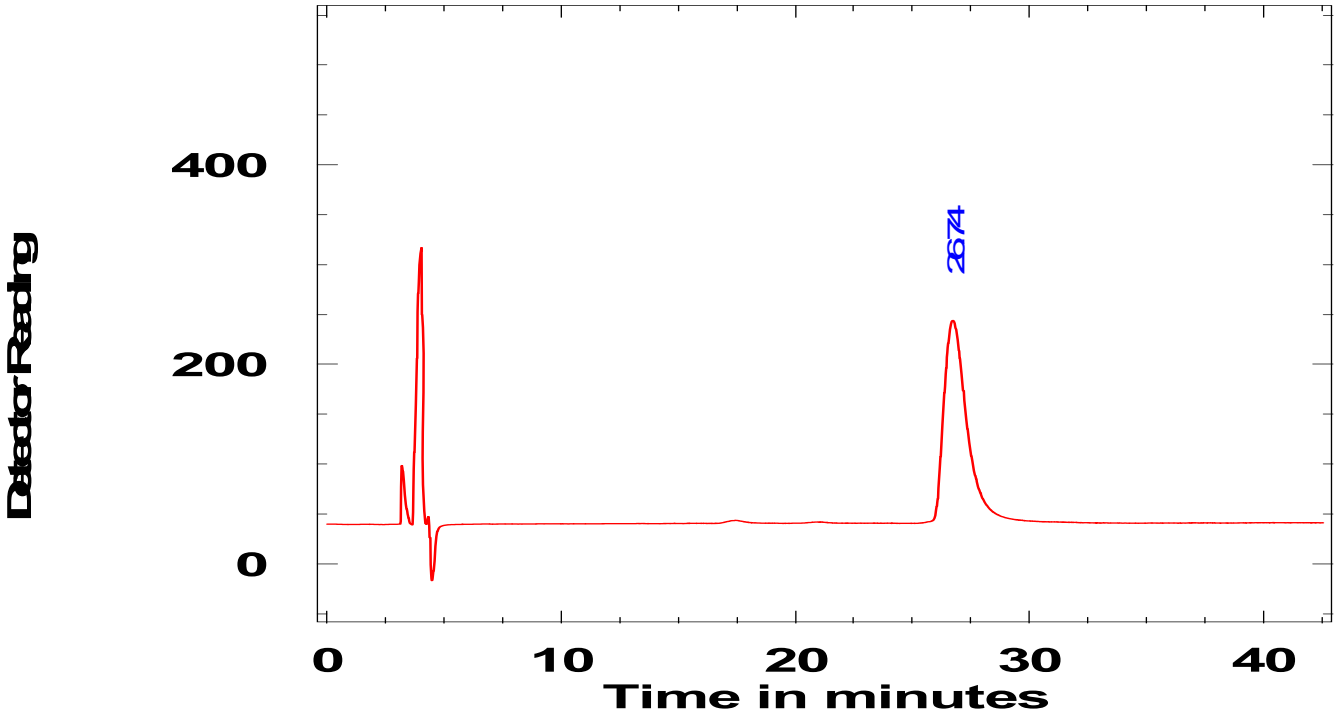

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	25.71	30.60	13799	244.11	26.74	100.0	Baseline

Fig S153. HPLC analysis of the mixture of racemic and chiral compound 3h obtained. (For comparison, Table 2, entry 8).

Peak Report

PMK-02-390-chiral+racemate-colm-OD-10%ipa/hex

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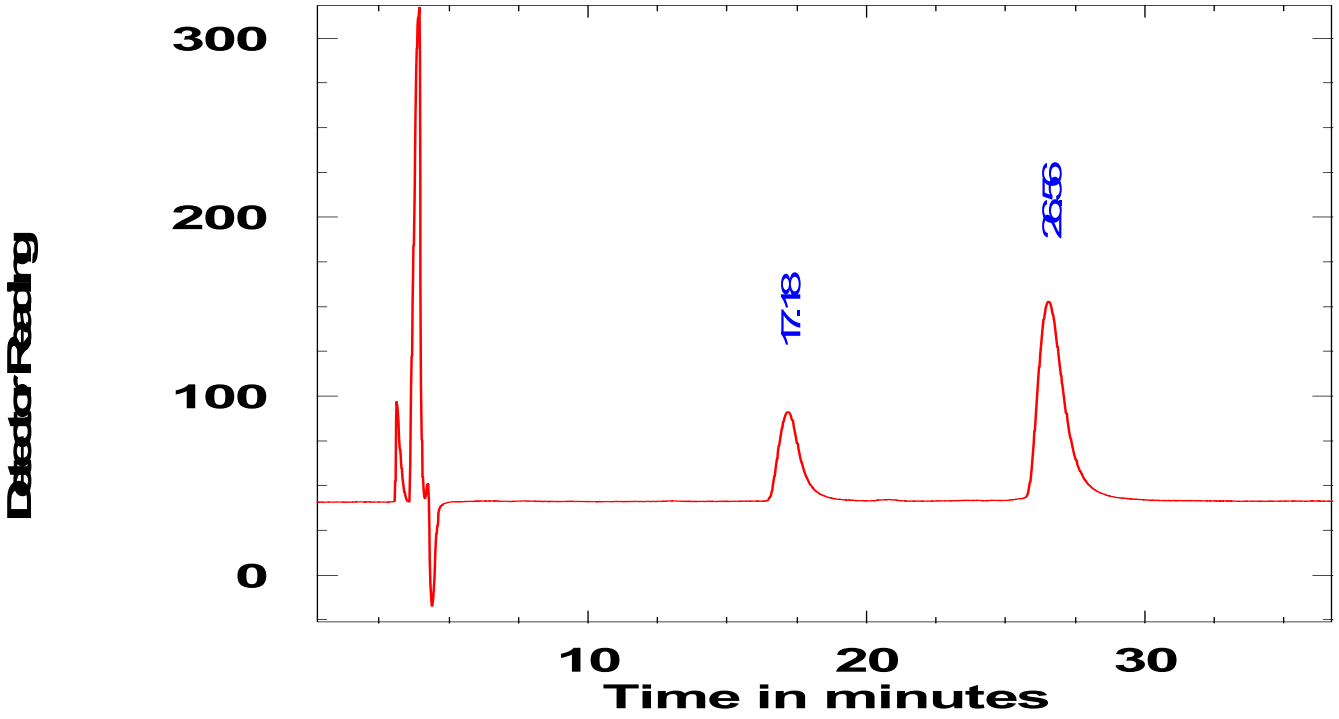

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	16.57	18.87	2367	91.32	17.18	24.8	Baseline
2	25.51	29.25	7195	152.92	26.56	75.2	Baseline

Fig S154. HPLC analysis of racemic compound cis-3i.
(For comparison, Table 2, entry 9)

Peak Report

PMK-02-377-racemate-2%ipa/Hex

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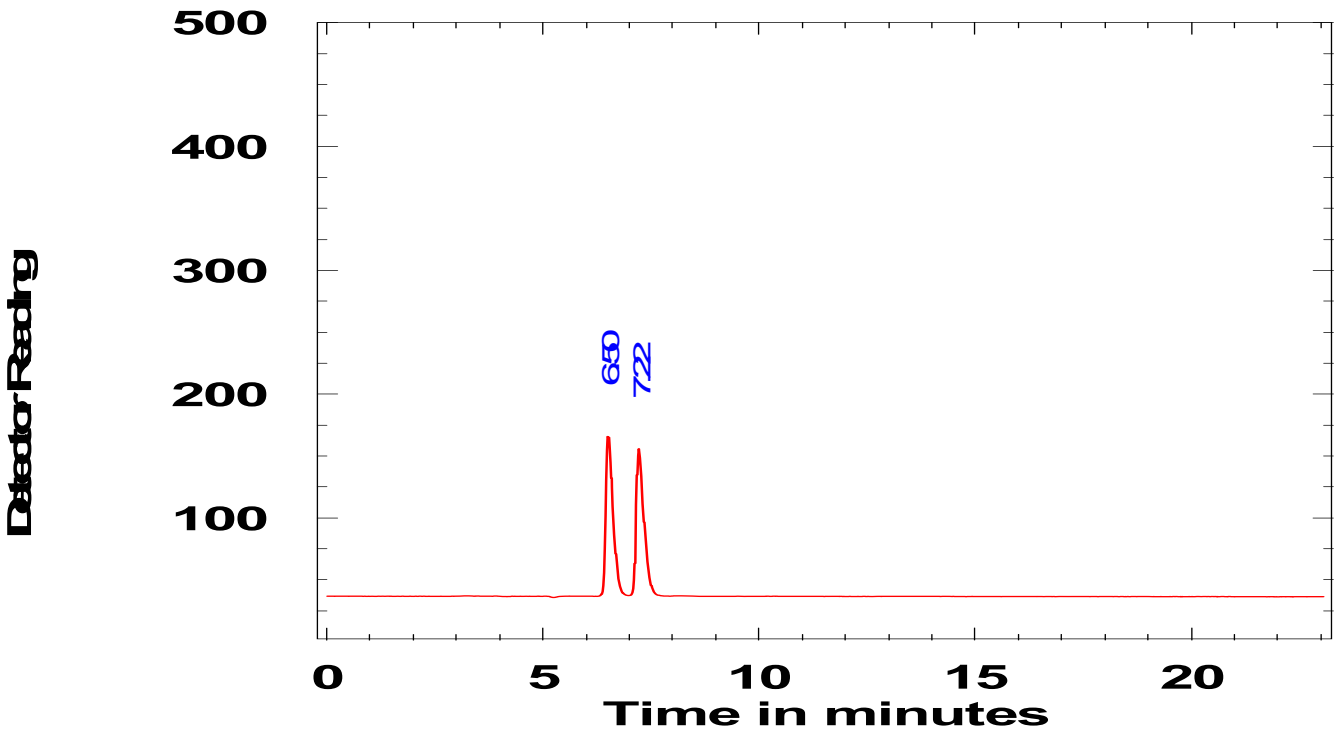

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	6.33	6.86	1476	165.73	6.50	49.8	Baseline
2	7.04	7.81	1486	155.76	7.22	50.2	Baseline

Fig S155. Fig S99. HPLC analysis of compound cis-3i obtained. (Table 2, entry 9)

Peak Report

PMK-02-377-chiral-2%ipa/Hex

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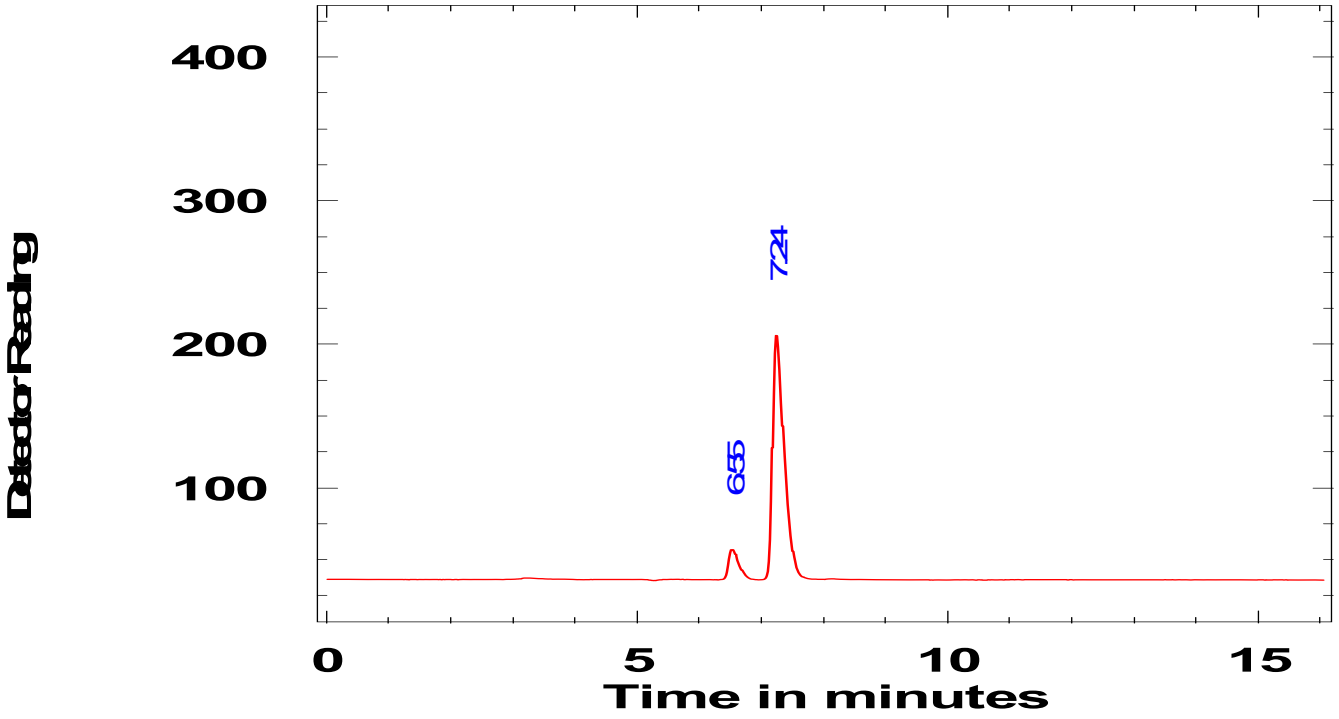

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	6.40	6.69	156	57.05	6.55	6.6	Baseline
2	7.05	7.94	2196	206.15	7.24	93.4	Baseline

Fig S156. HPLC analysis of the mixture of racemic and chiral compound cis-3i obtained. (For comparison, Table 2, entry 9)

Peak Report

PMK-02-377-chiral+racemate-2%ipa/Hex

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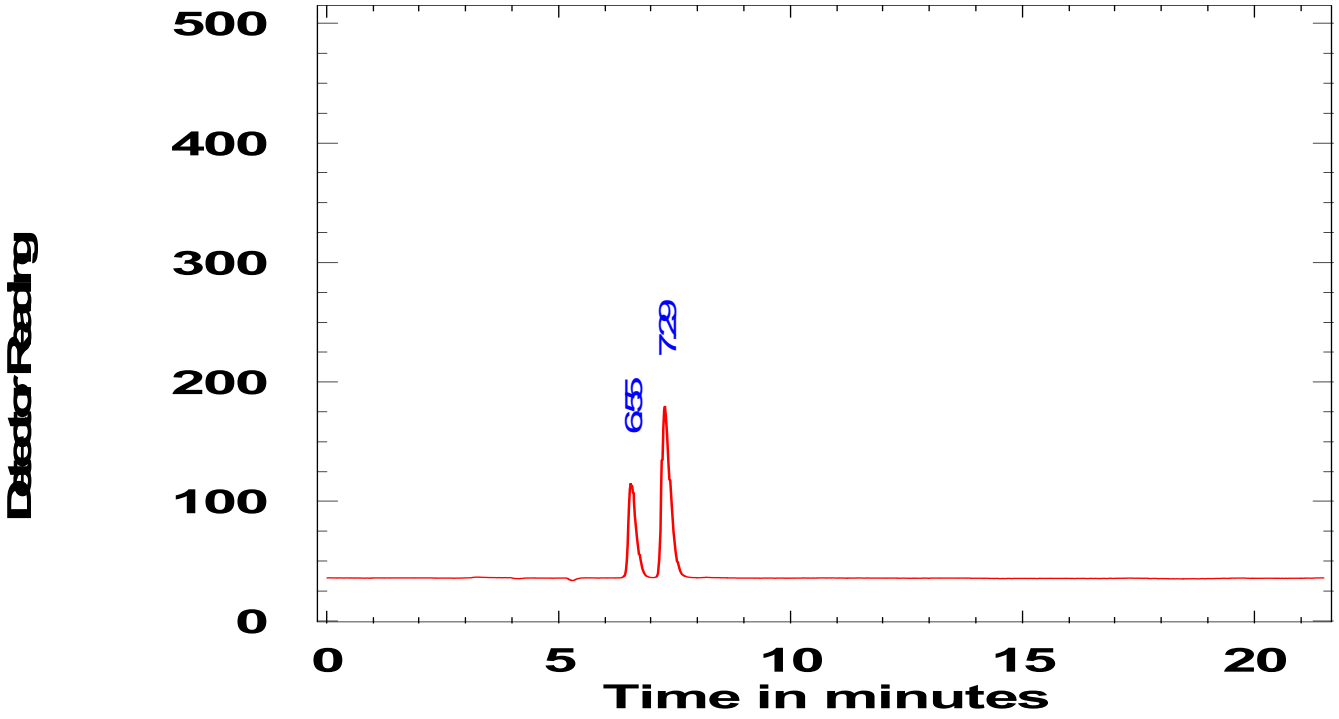

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	6.34	6.81	818	114.93	6.55	30.7	Baseline
2	7.06	7.83	1849	179.51	7.29	69.3	Baseline

Fig S157. HPLC analysis of the racemic compound trans-3i.
(For comparison, Table 2, entry 9)

Peak Report

PMK-02-377-F2-racemate-colm-IB-3%ipa-hex

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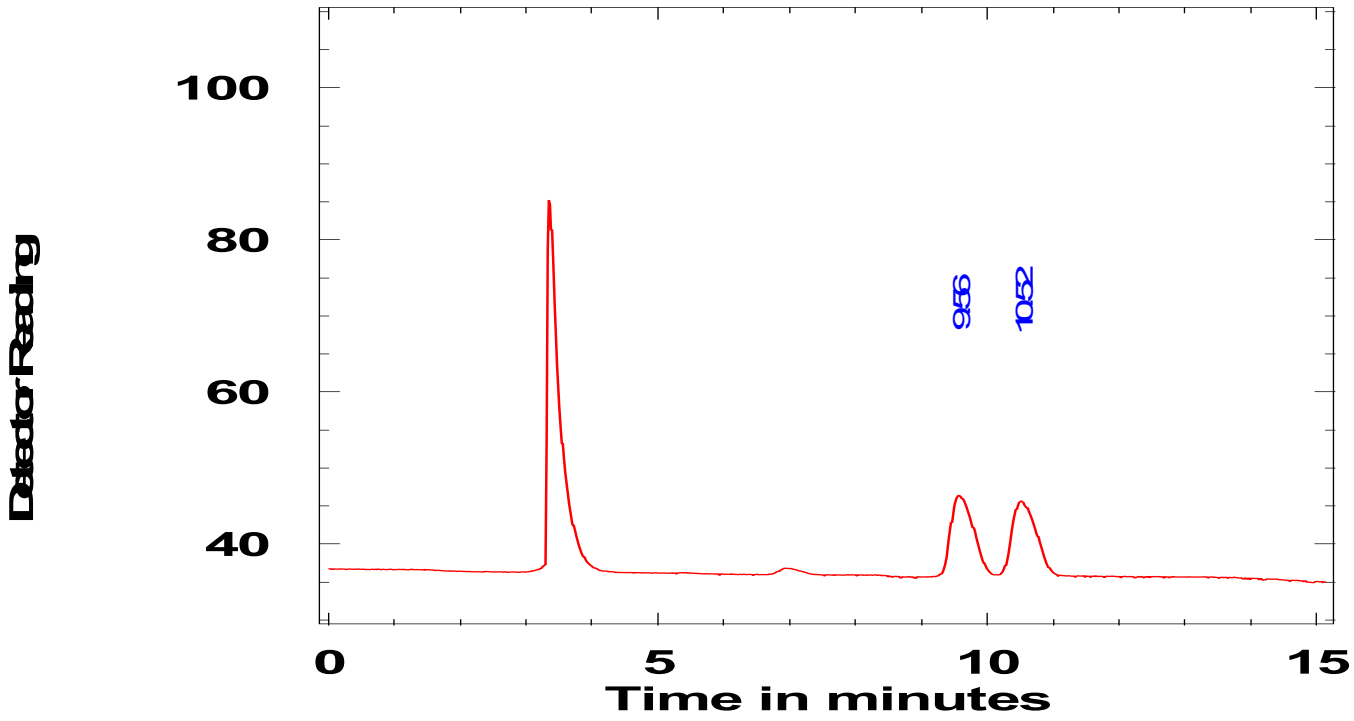

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	9.20	10.09	249	46.40	9.56	51.0	Baseline
2	10.19	11.04	240	45.66	10.52	49.0	Baseline

Fig S158. HPLC analysis of compound trans-3i obtained. (Table 2, entry 9)

Peak Report

PMK-02-377-F2-chiral-colm-IB-3%ipa-hex

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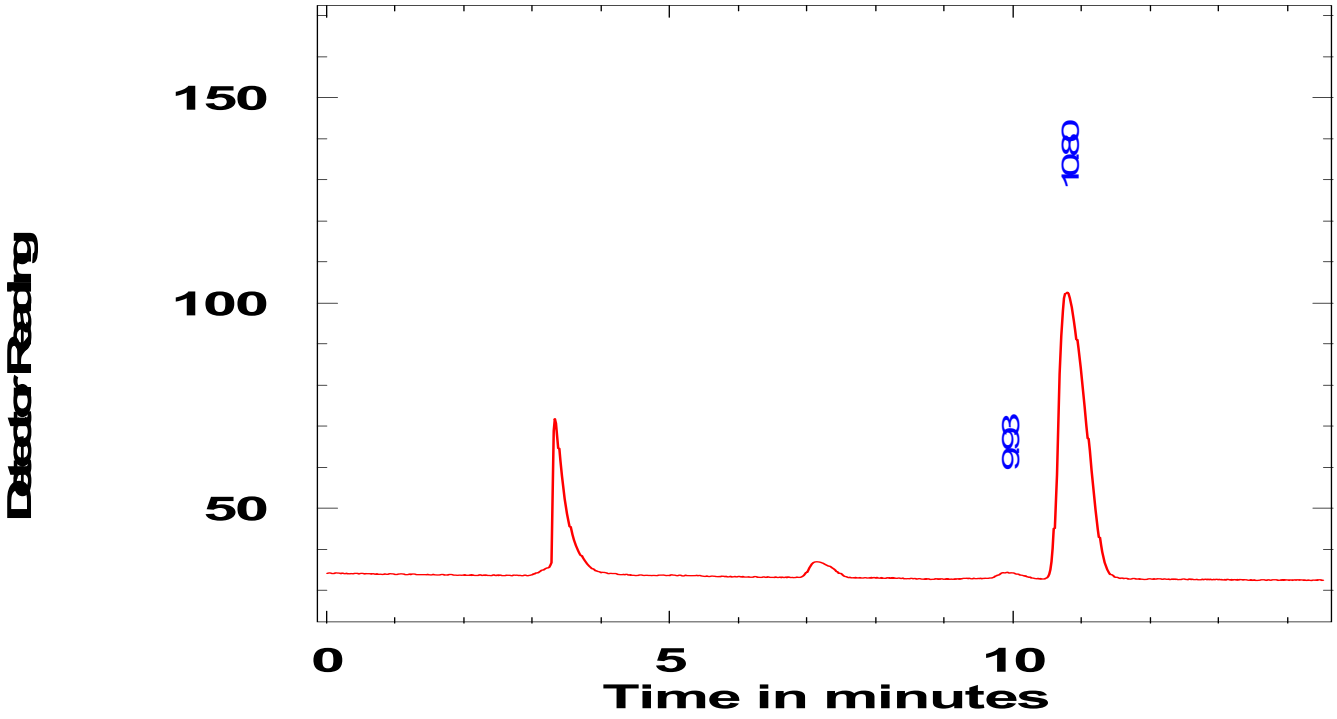

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	9.71	10.35	29	34.46	9.93	1.6	Baseline
2	10.50	11.42	1811	102.62	10.80	98.4	Baseline

Fig S159. HPLC analysis of the mixture of racemic and chiral compound trans-3i obtained. (For comparison, Table 2, entry 9)

Peak Report

PMK-02-377-F2-chiral+racemate-colm-IB-3%ipa-hex

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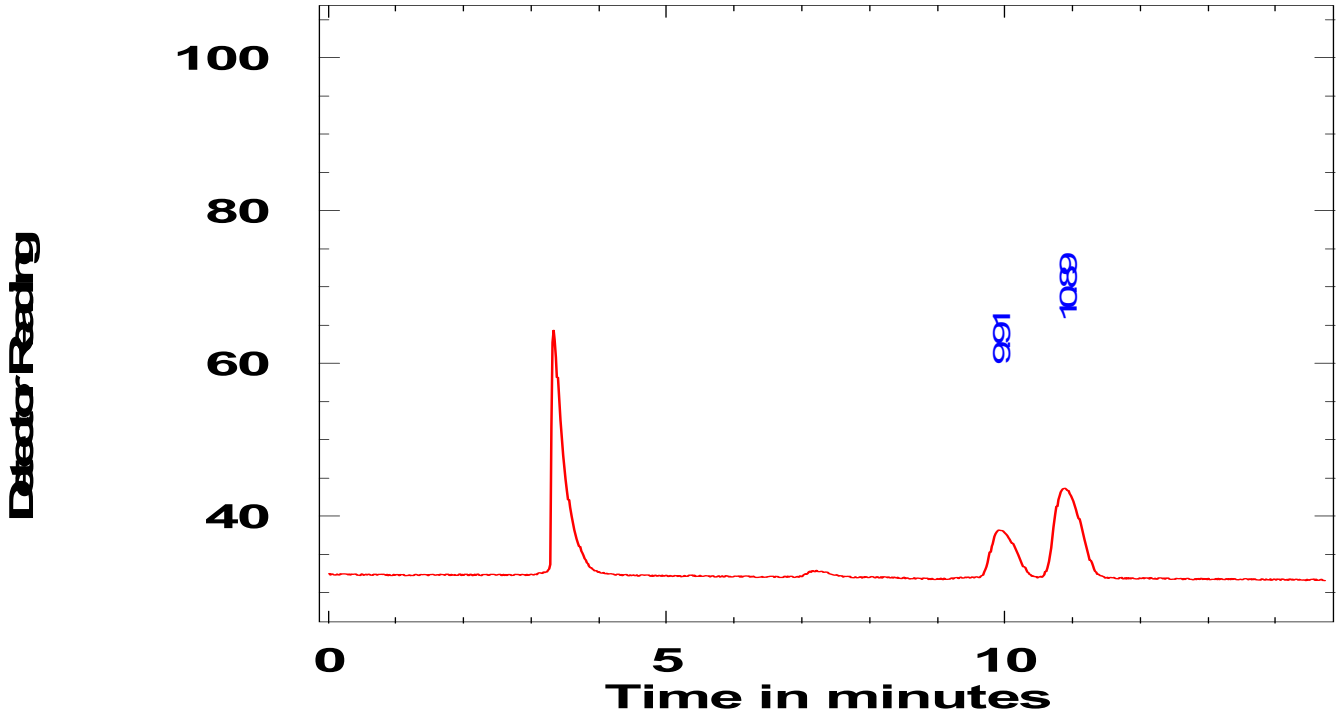

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	9.68	10.45	143	38.19	9.91	32.6	Baseline
2	10.55	11.37	297	43.67	10.89	67.4	Baseline

Fig S160. HPLC analysis of racemic compound 3j.
(For comparison, Table 2, entry 10)

Peak Report

PMK-02-360-racemate-colm- OD-8%ipa/hex

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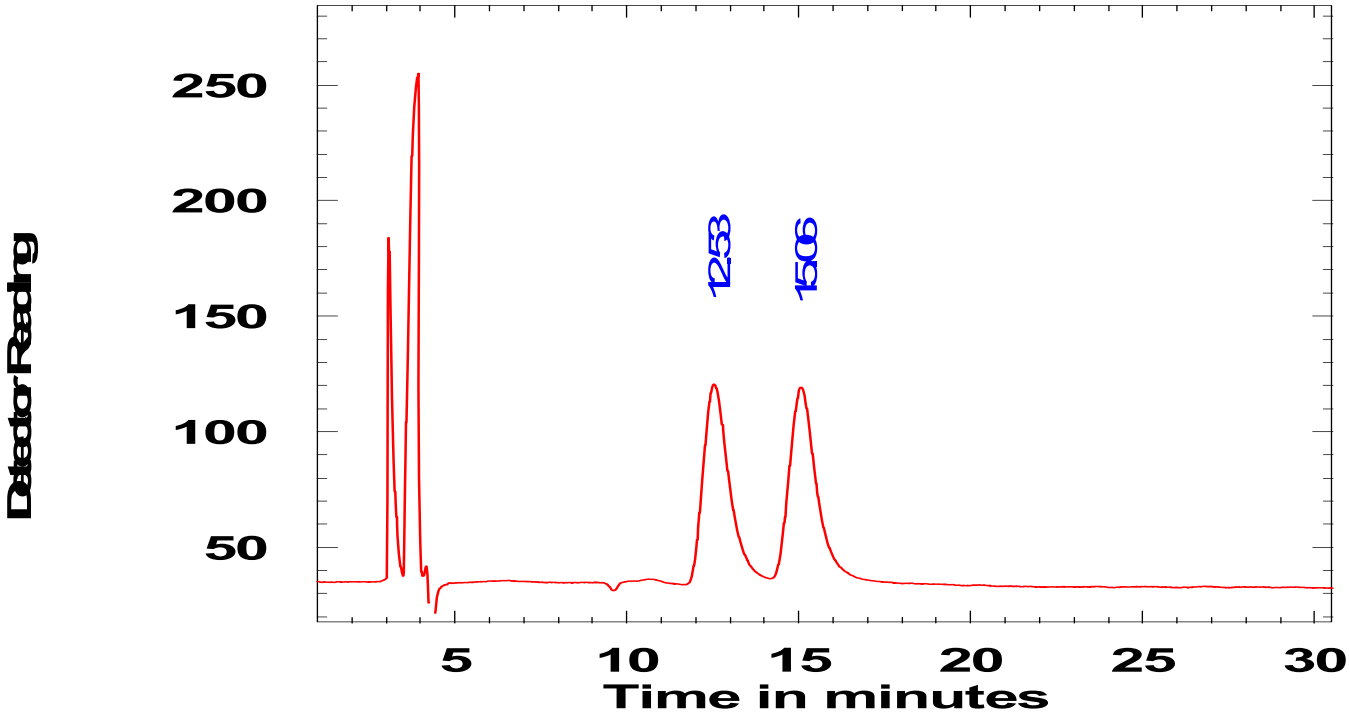

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	11.86	14.22	4302	120.59	12.53	50.4	Baseline
2	14.36	17.00	4231	119.17	15.06	49.6	Baseline

Fig S161. HPLC analysis of compound 3j obtained. (Table 2, entry 10)

Peak Report

PMK-02-360-Chiral-colm- OD-8%ipa/hex

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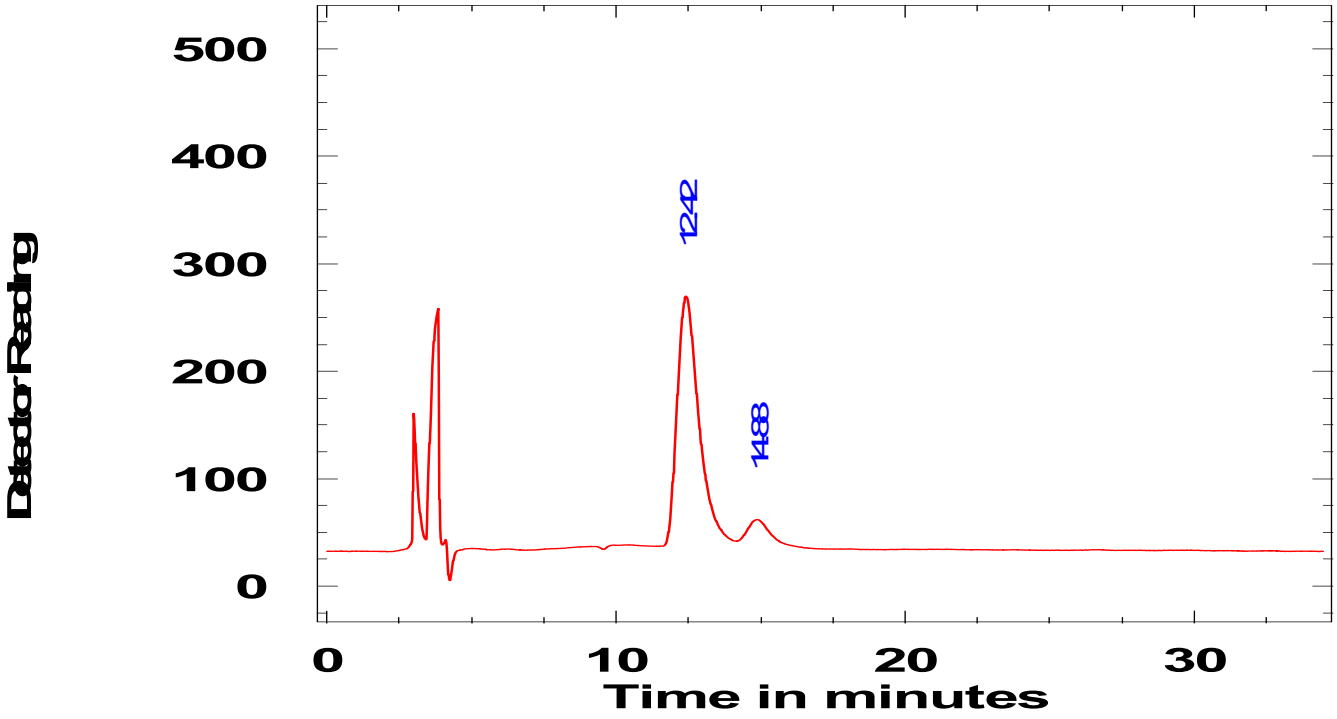

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	11.65	14.40	11600	269.84	12.42	95.9	Baseline
2	14.52	15.57	492	61.90	14.88	4.1	Baseline

Fig S162. HPLC analysis of the mixture of racemic and chiral compound 3j obtained.
(For comparison, Table 2, entry 10)

Peak Report

PMK-02-360-Chiral+racemate-colm- OD-8%ipa/hex

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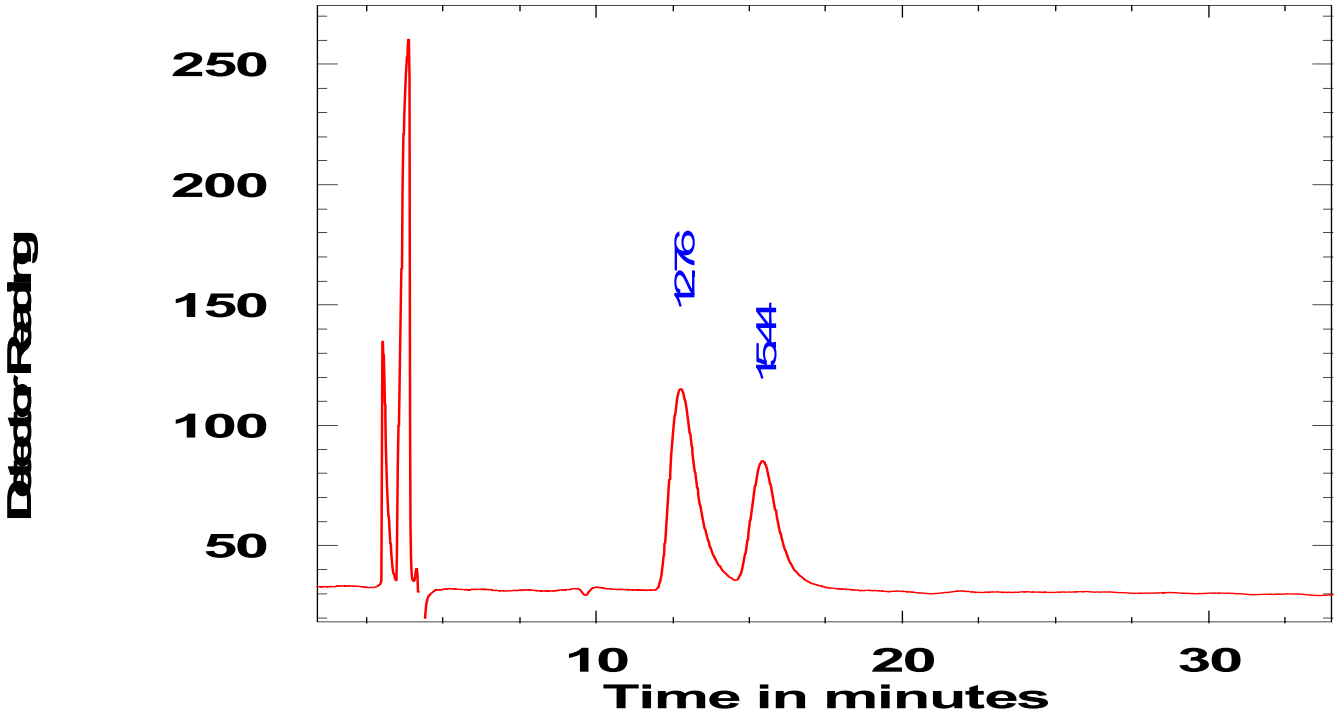

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	11.98	14.54	4933	115.19	12.76	63.2	Baseline
2	14.63	17.00	2876	85.31	15.44	36.8	Baseline

Fig S163. HPLC analysis of racemic compound 3k.

(For comparison, Table 2, entry 11)

Peak Report

PMK-02-394-racemate-colm-IA-5%ipa/Hex

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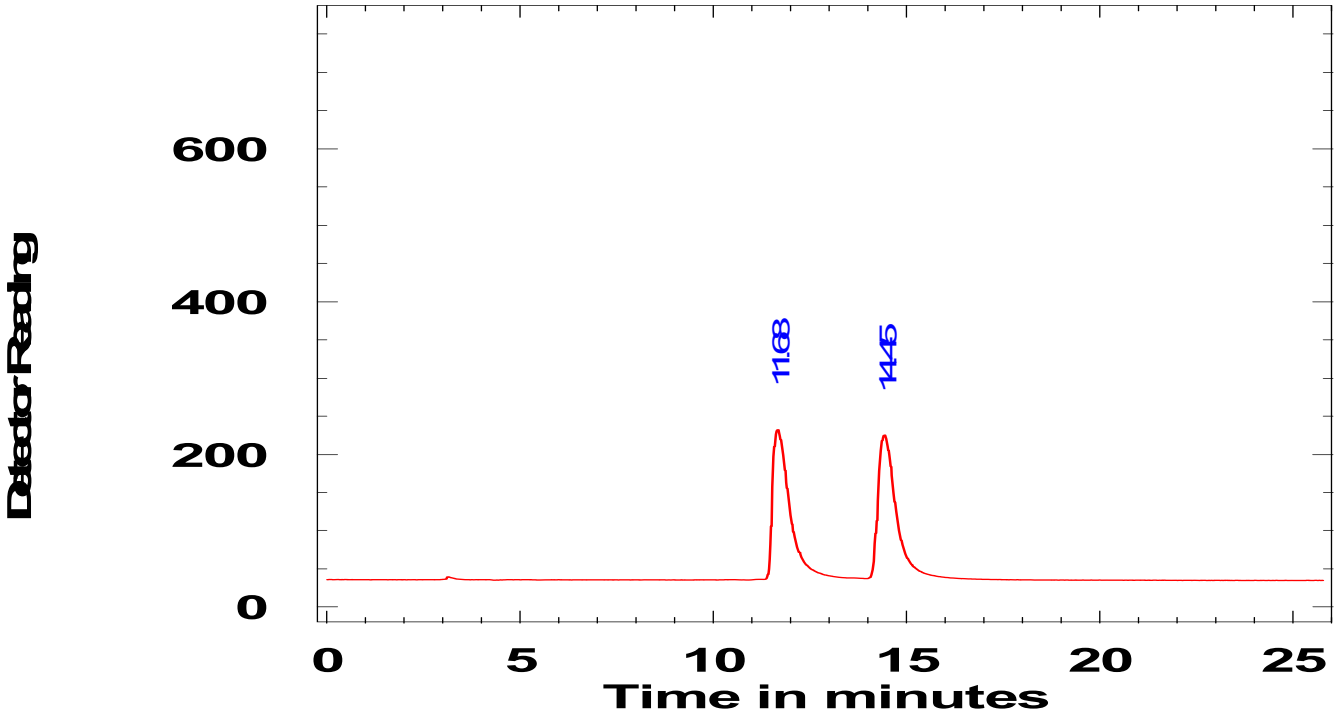

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	11.32	13.16	5997	232.01	11.68	49.8	Baseline
2	14.06	16.54	6051	225.15	14.45	50.2	Baseline

Fig S164. HPLC analysis of compound 3k obtained. (Table 2, entry 11)

Peak Report

PMK-02-394-chiral-colm-IA-5%ipa/Hex

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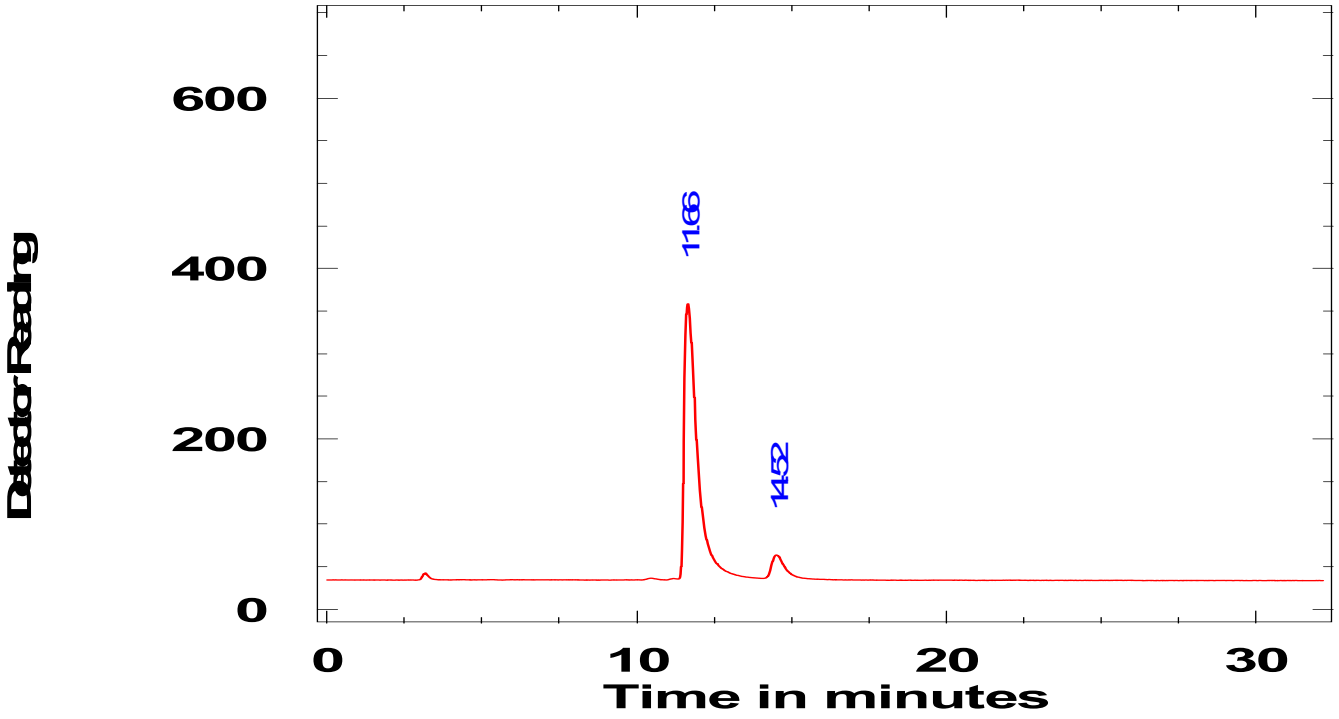

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	11.30	13.73	9585	358.51	11.66	95.0	Baseline
2	14.19	14.90	507	64.00	14.52	5.0	Baseline

Fig S165. HPLC analysis of the mixture of racemic and chiral compound 3k obtained.
(For comparison, Table 2, entry 11)

Peak Report

PMK-02-394-chiral+racemate-colm-IA-5%ipa/Hex

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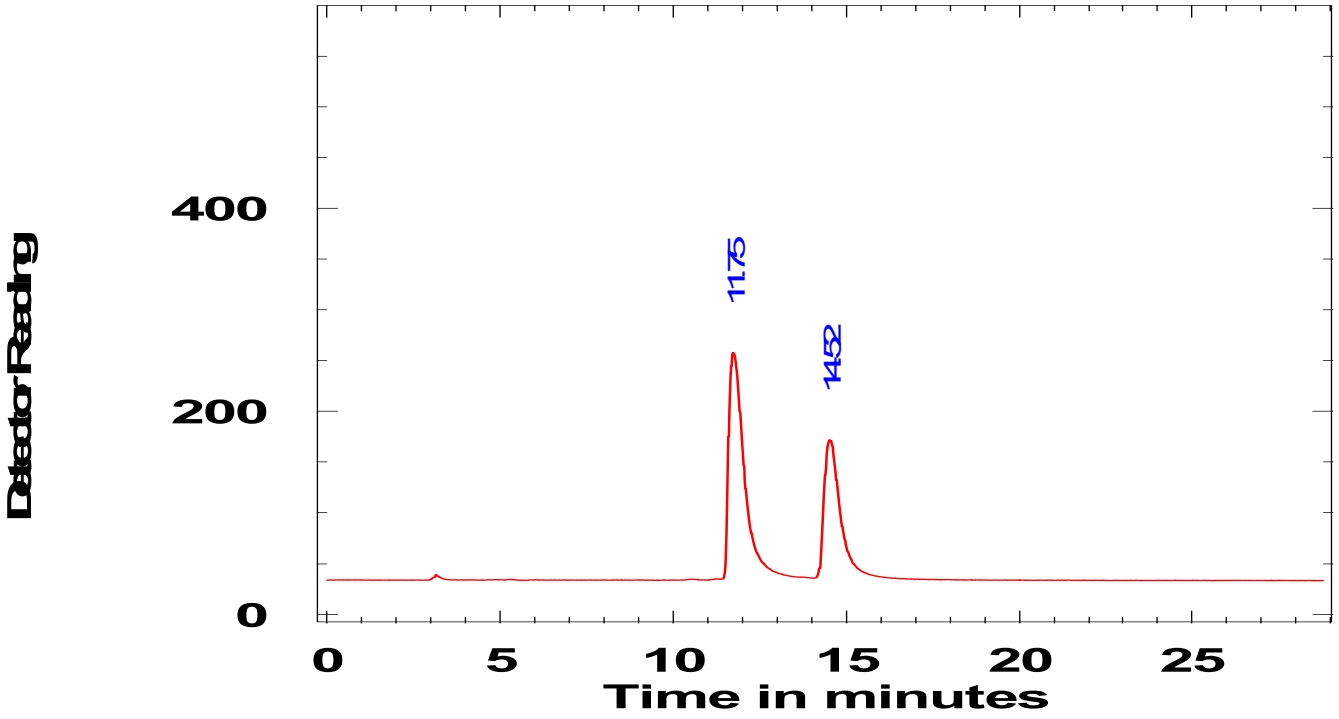

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	11.34	13.42	6963	257.86	11.75	60.8	Baseline
2	14.09	16.47	4490	172.03	14.52	39.2	Baseline

Fig S166. HPLC analysis of racemic compound 3l. (For comparison, Table 2, entry 12)

Peak Report

PMK-02-395-racemate-colm-IA-5%ipa/Hex

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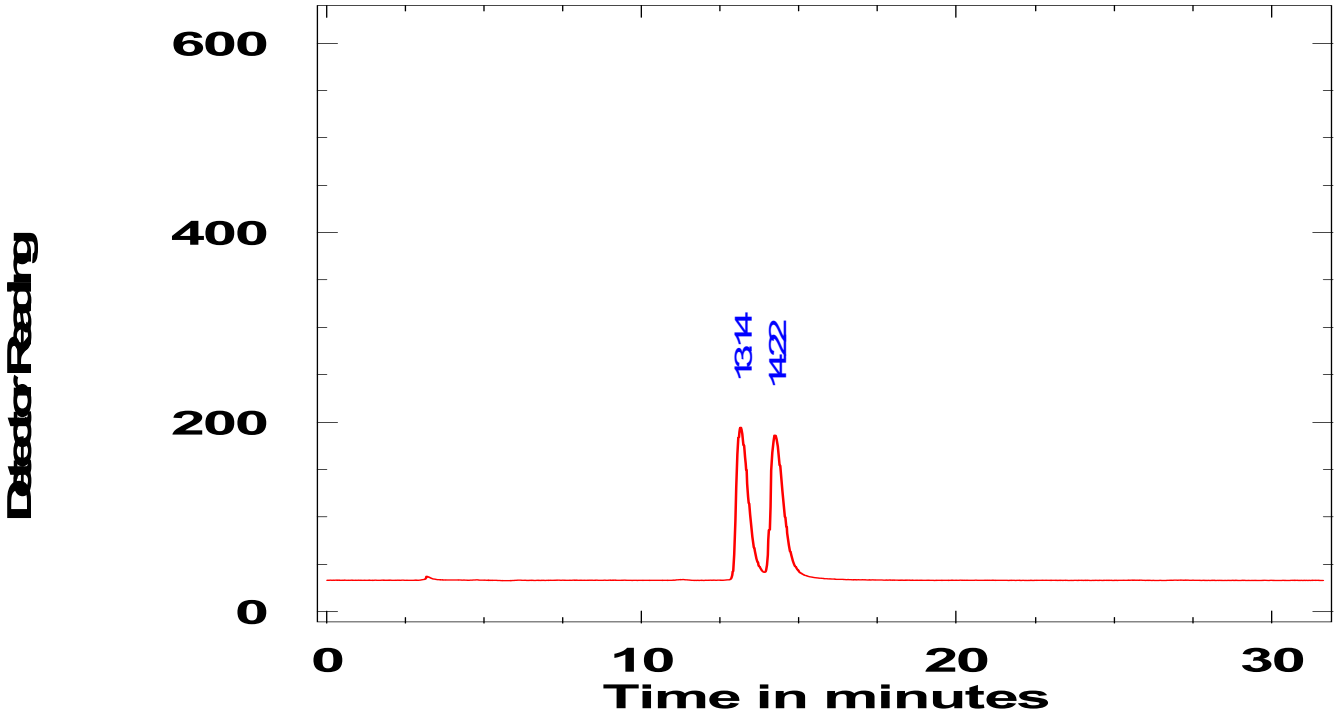

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	12.83	13.88	4079	194.69	13.14	49.9	Baseline
2	13.93	15.68	4102	186.28	14.22	50.1	Baseline

Fig S167. HPLC analysis of compound 3l obtained. (Table 2, entry 12)

Peak Report

PMK-02-395-chiral-colm-IA-5%ipa/Hex

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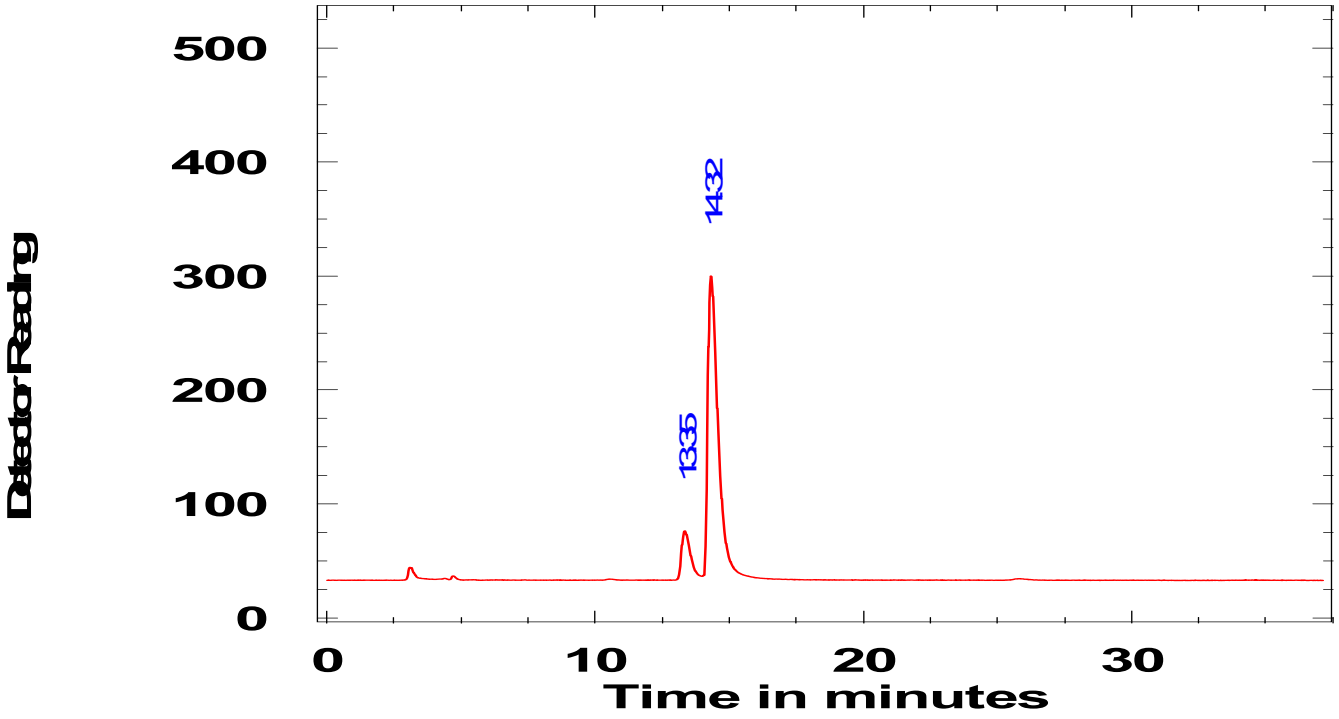

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	13.11	13.75	763	76.32	13.35	9.8	Baseline
2	13.91	16.57	7046	300.01	14.32	90.2	Baseline

Fig S168. HPLC analysis of the mixture of racemic and chiral compound 3l obtained.
(For comparison, Table 2, entry 12)

Peak Report

PMK-02-395-chiral+racemate-colm-IA-5%ipa/Hex

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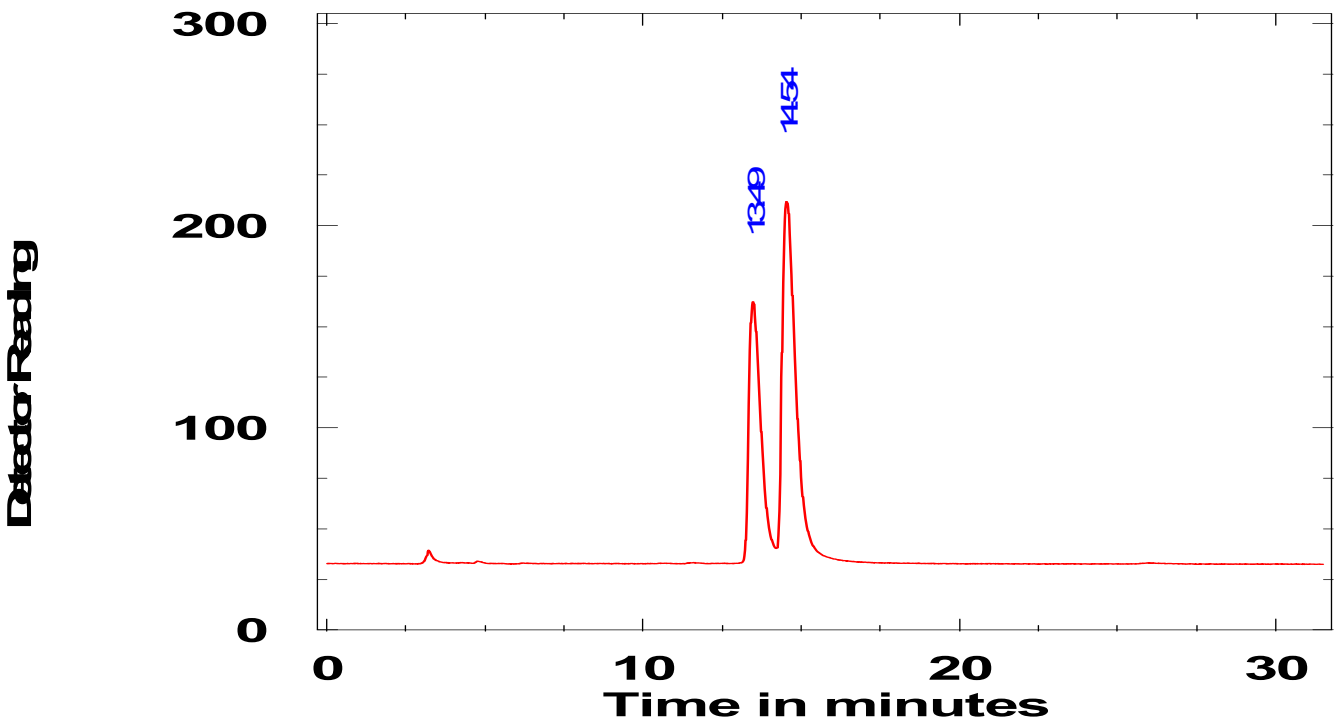


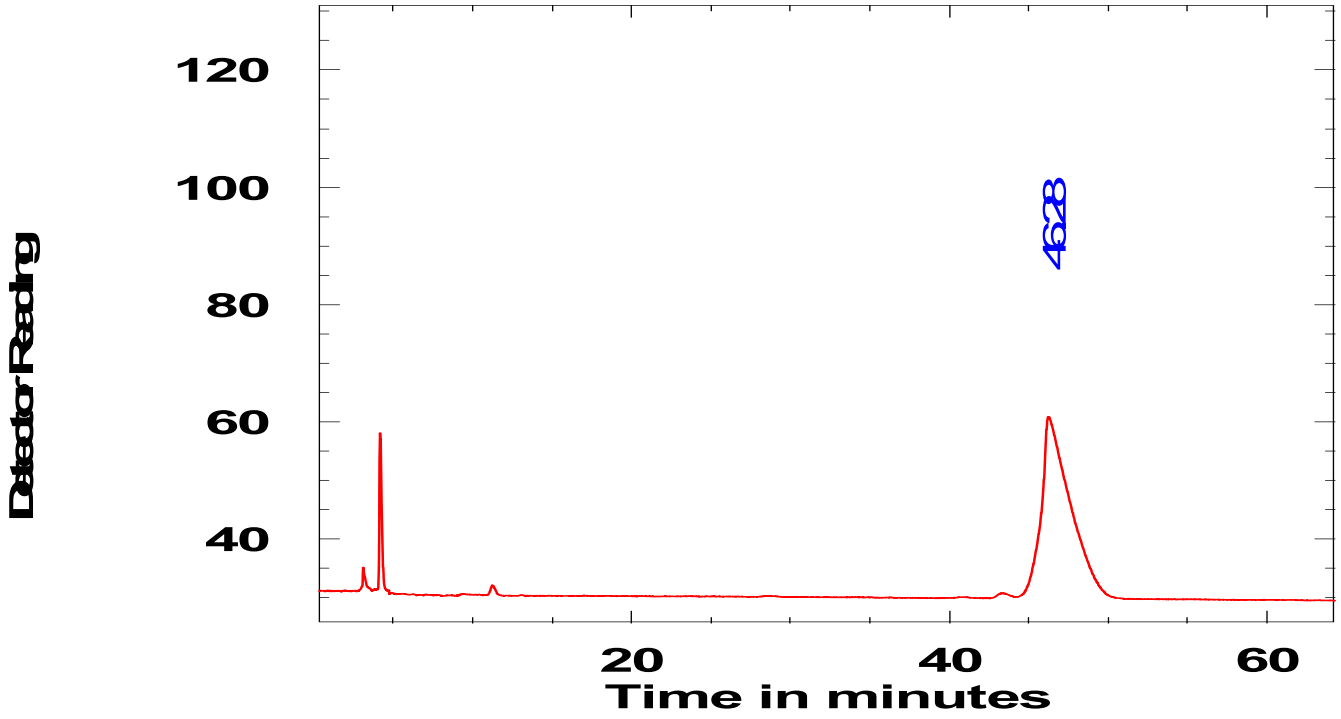
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	13.13	14.09	3097	162.45	13.49	38.6	Baseline
2	14.22	16.85	4916	211.95	14.54	61.4	Baseline

Fig S169. HPLC analysis of compound 3m obtained from cat (S)-I. (Table 2, entry 13)

Peak Report

PMK-02-393-chiral-(S-Enatiomer)-colm-IA-8%ipa/Hex

Report produced on 2010/9/7 at 下午 03:02:29 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	44.35	50.20	3848	60.84	46.28	100.0	Baseline

Fig S170. HPLC analysis of compound 3m obtained from cat (R)-I.
(For comparison, Table 2, entry 13)

Peak Report

PMK-02-405-chiral-(R-Enatiomer)-colm-IA-8%ipa/Hex

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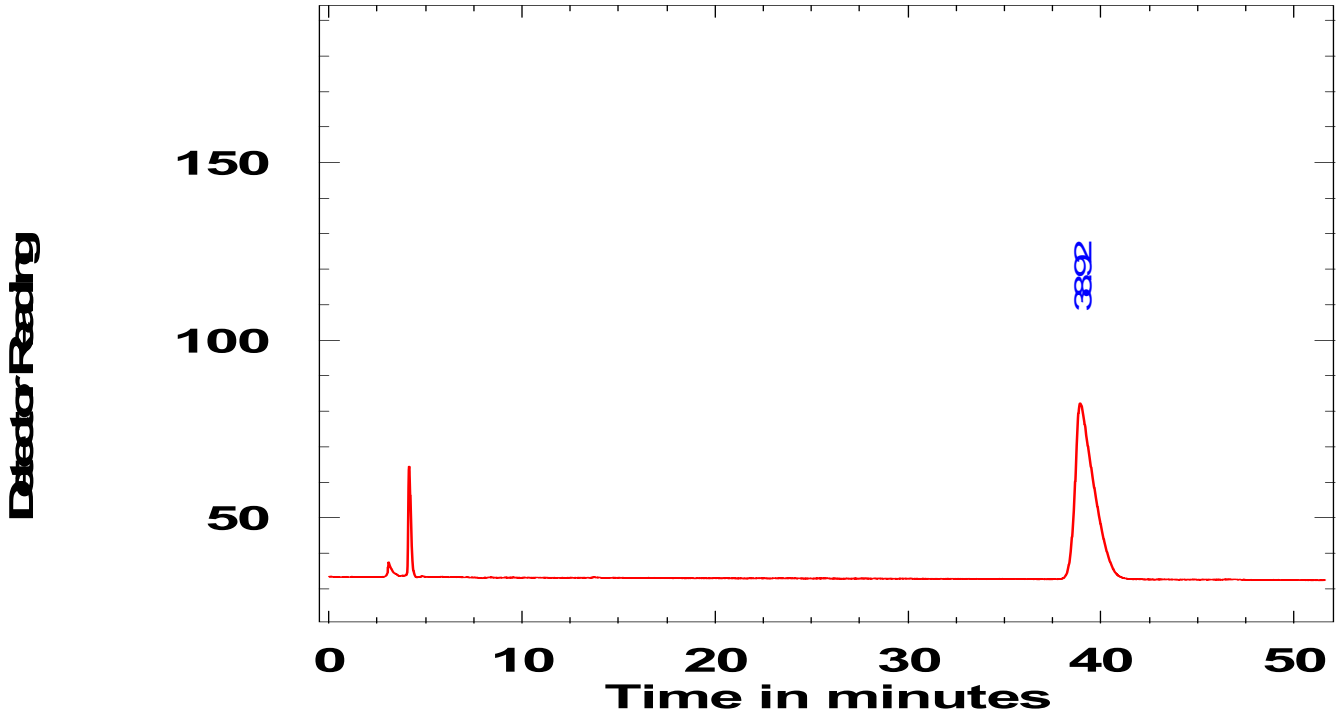


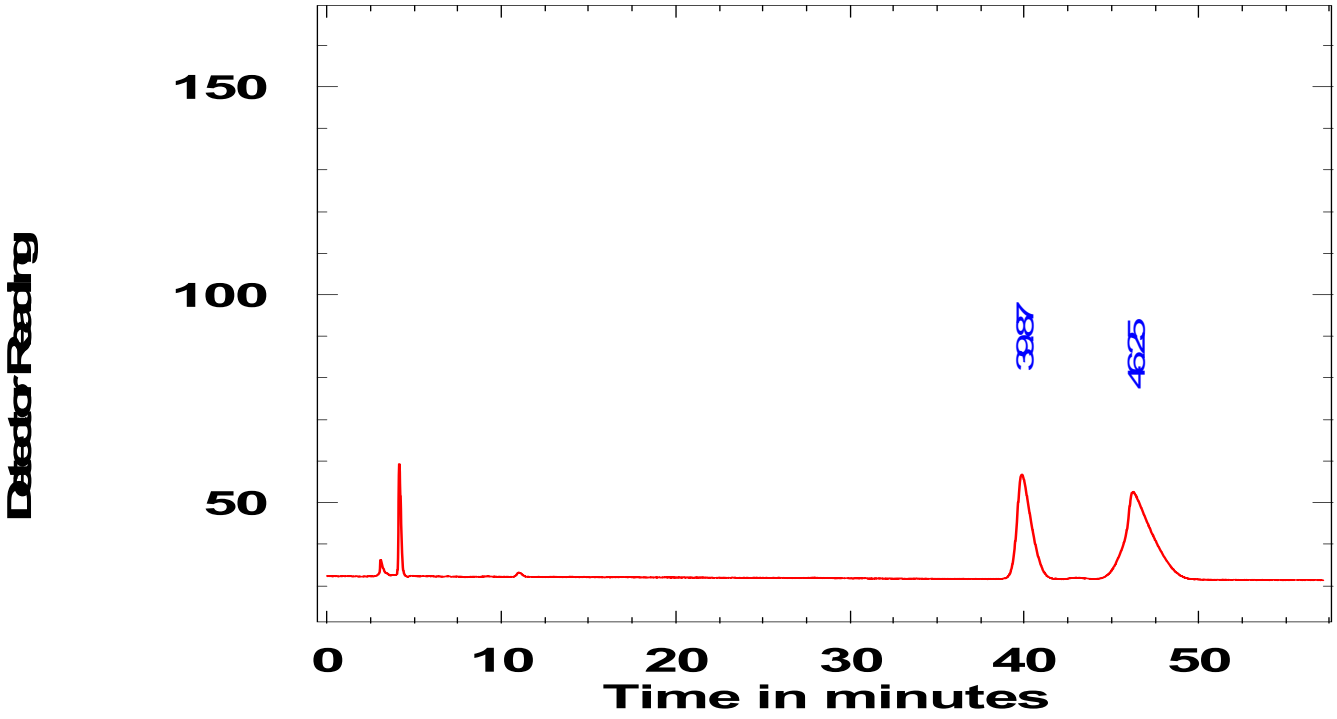
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	38.01	41.39	3354	82.25	38.92	100.0	Baseline

Fig S171. HPLC analysis of the mixture of (+)- and (-)-3m.
(For comparison, Table 2, entry 13)

Peak Report

PMK-02-393+405-chiral-(S+R-Enatiomer)-colm-IA-8%ipa/Hex

Report produced on 2010/9/7 at 下午 01:34:43 by Put your name here



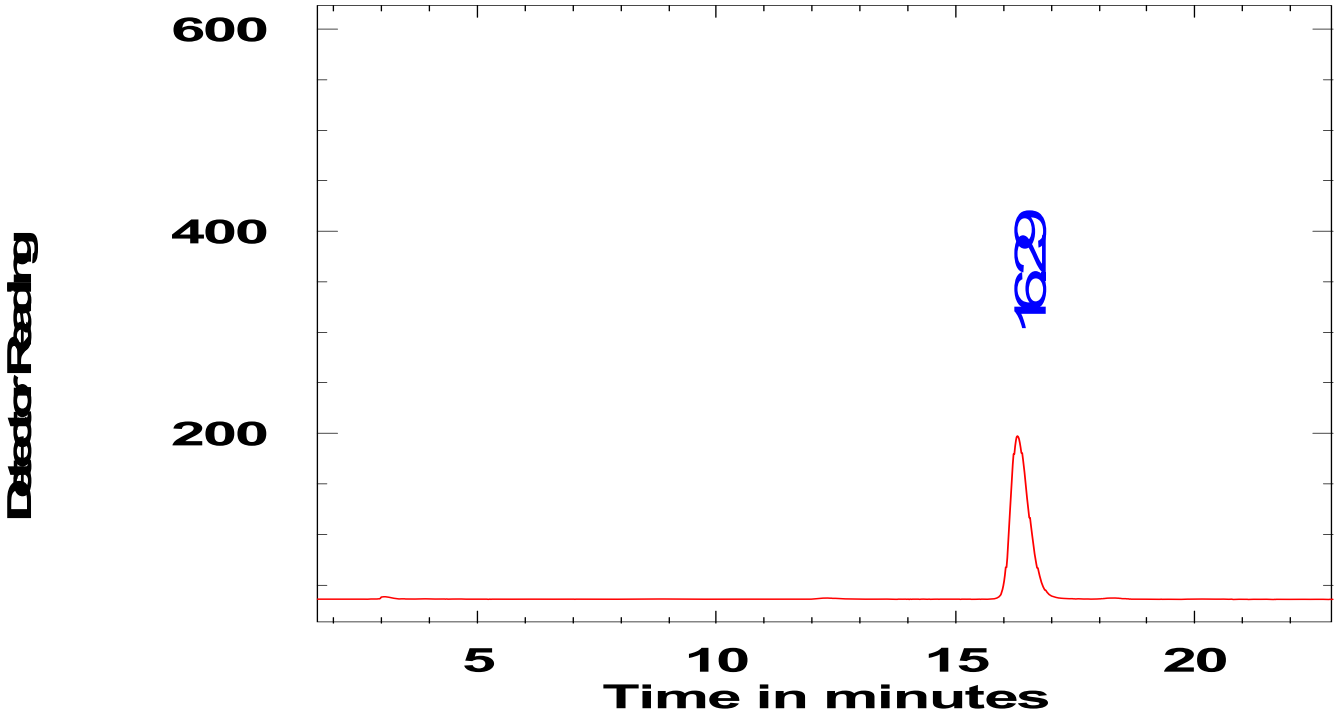
Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	38.67	41.33	1398	56.79	39.87	37.9	Baseline
2	44.25	49.48	2288	52.65	46.25	62.1	Baseline

Fig S172. HPLC analysis of compound 3n obtained from cat (S)-I. (Table 2, entry 14)

Peak Report

PMK-02-404-(S-enatiomer)chiral-colm-IA-15%ipa/Hex

Report produced on 2010/9/8 at 下午 02:58:01 by Put your name here



Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	15.82	17.41	4343	197.54	16.29	100.0	Baseline

Fig S173. HPLC analysis of compound 3n obtained from cat (R)-I. (Table 2, entry 14)
(For comparison, Table 2, entry 14)

Peak Report

PMK-02-407-(R-enatiomer)chiral-colm-IA-15%ipa/Hex

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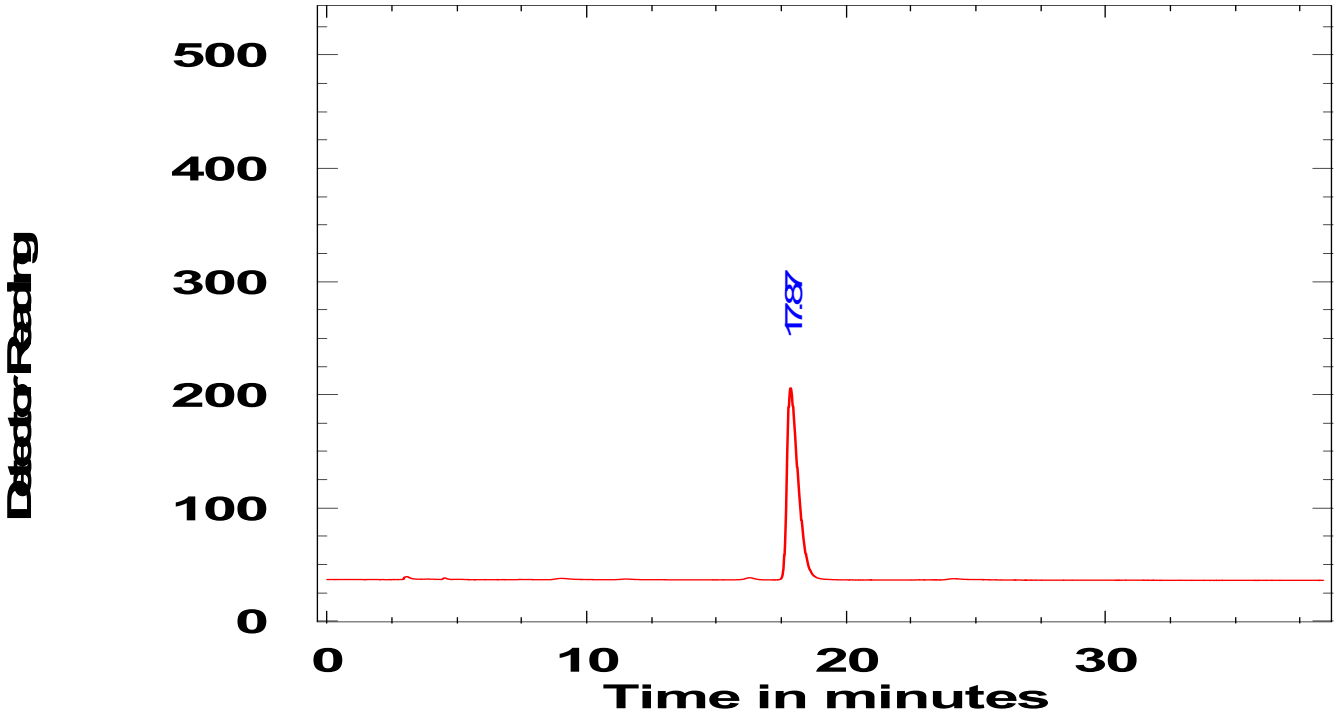

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	17.51	19.01	4982	206.04	17.87	100.0	Baseline

Fig S174. HPLC analysis of the mixture of (+)- and (-)-3n.
(For comparison, Table 2, entry 14)

Peak Report

PMK-02-404+407-(S+R-enatiomer)chiral-colm-IA-15%ipa/Hex

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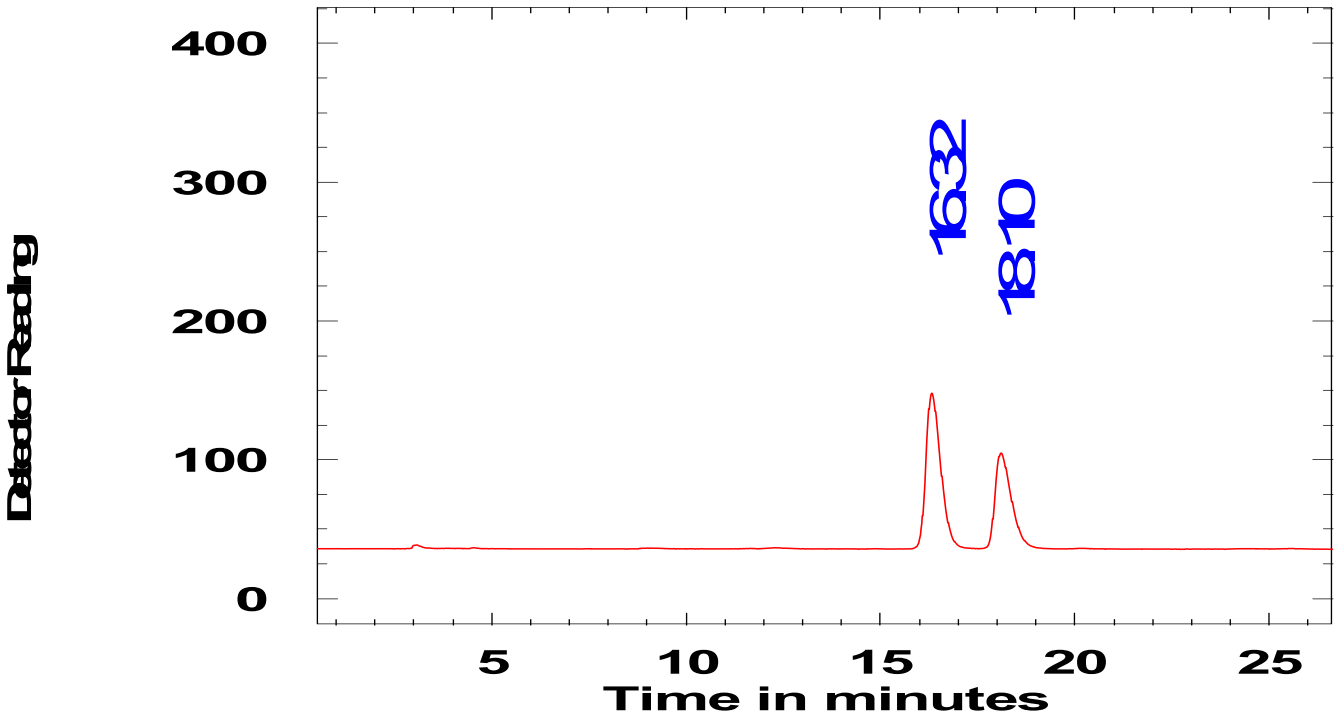

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	15.80	16.98	2859	147.92	16.32	59.7	Baseline
2	17.70	18.92	1929	104.66	18.10	40.3	Baseline

Fig S175. HPLC analysis of compound 3o obtained from cat (S)-I. (Table 2, entry 15)

Peak Report

PMK-02-406-(S-enatiomer)chiral-colm-IA-15%ipa/Hex

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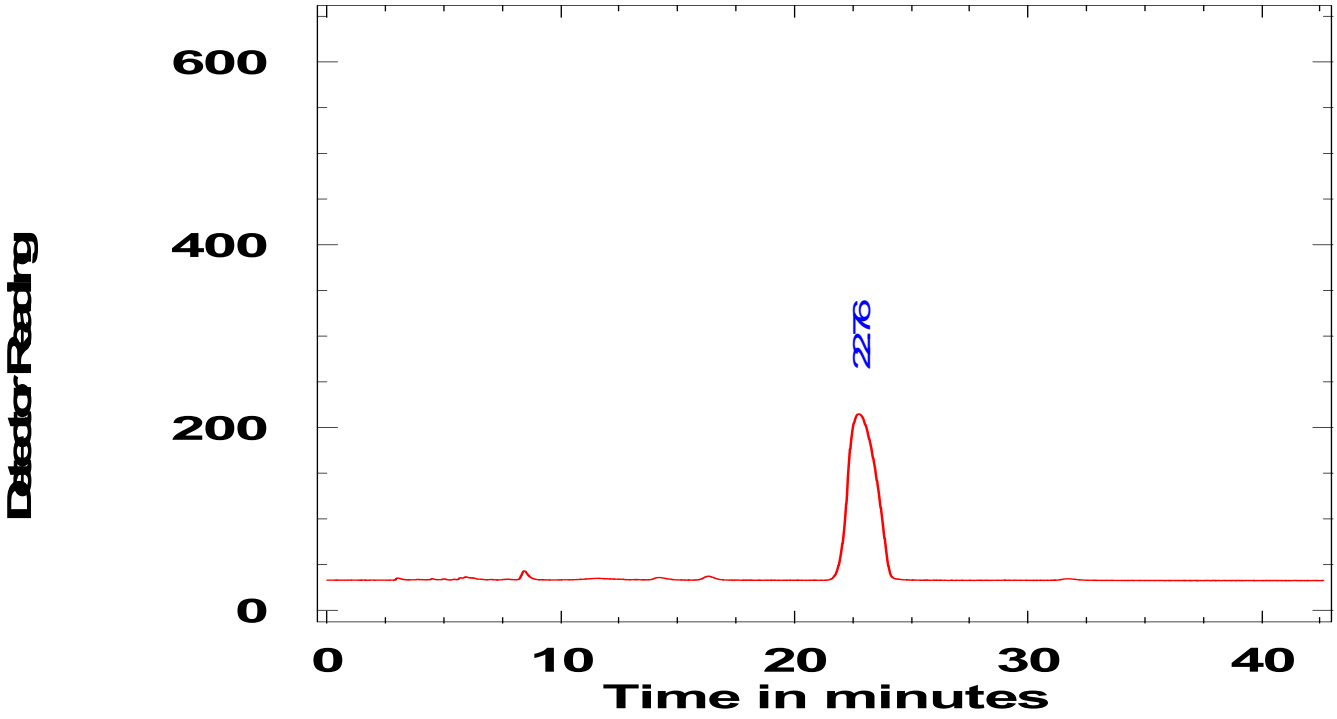

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	21.54	24.26	14841	215.11	22.76	100.0	Baseline

Fig S176. HPLC analysis of compound 3o obtained from cat (R)-I.
(For comparison, Table 2, entry 15)

Peak Report

PMK-02-408-(R-enatiomer)chiral-colm-IA-15%ipa/Hex

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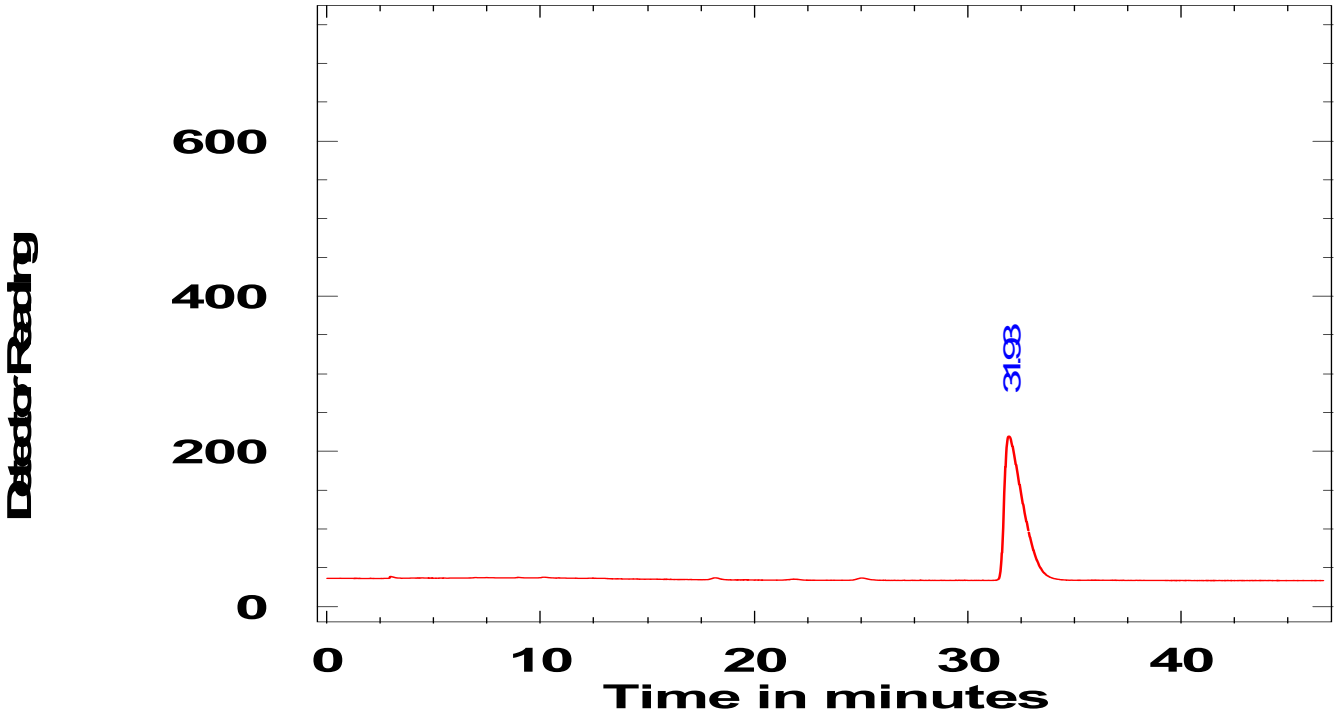

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	31.45	34.16	11019	219.65	31.93	100.0	Baseline

Fig S177. HPLC analysis of the mixture of (+)- and (-)-3o.
(For comparison, Table 2, entry 15)

Peak Report

PMK-02-406+408-(S+R-enatiomer)chiral-colm-IA-15%ipa/Hex

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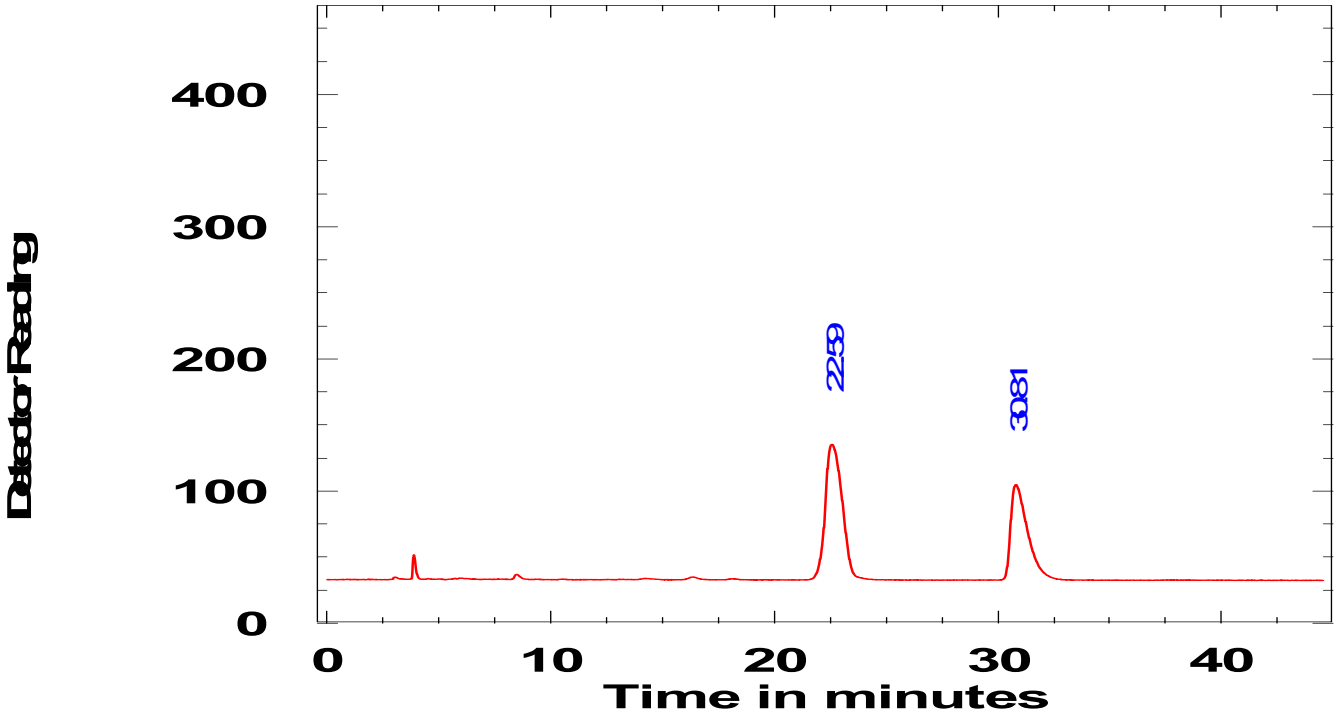

Peak #	Begin	End	Peak Area	Maximum	Time	Area %	Begins as
1	21.61	23.87	5268	135.17	22.59	58.9	Baseline
2	30.22	32.55	3677	104.75	30.81	41.1	Baseline