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SUPPORTING INFORMATION

Title: Sulfonamide Ligands Attained Through Opening of Saccharin Derivatives
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General procedure for the preparation of oxazoline sulfonamides (3):

To a stirred solution of 5% ethanolic KOH (20 mL) was added **13** (4.8 mmol). The reaction mixture was stirred at room temperature for the stated amount of time, followed by the addition of CH_2Cl_2 (50 mL). The reaction mixture was then acidified by the addition of an aqueous 2.0 M HCl solution, the organic was extracted, washed with H_2O (40 mL), brine (30 mL), dried (Na_2SO_4) and concentrated *in vacuo* to give a pale yellow oil. Purification by flash chromatography (CH_2Cl_2) gave **3** as a colourless oil.

***N*-benzyl-2-(4,5-dihydro-1,3-oxazol-2-yl)benzenesulfonamide (3ab):**

Prepared from **13ab** (1.70 g, 4.8 mmol), stirring for 3h. Purification by flash chromatography (CH_2Cl_2) gave **3ab** (1.23 g, 81 %) as a colourless oil. R_f 0.21 (CH_2Cl_2). ^{1}H (400 MHz, $CDCl_3$): δ = 8.41 (t, *J* = 6.2 Hz, 1H, SO_2NH), 8.06 (dd, *J* = 7.8, 1.0 Hz, 1H, Ar), 7.84 (dd, *J* = 7.8, 1.0 Hz, 1H, Ar), 7.59-7.51 (m, 2H, Ar), 7.23-7.17 (m, 5H, Ar), 4.46 (t, *J* = 9.5 Hz, 2H, CH_2Ph), 4.15-4.09 (m, 4H, CH_2CH_2) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 163.6, 139.7, 136.8, 132.0, 131.1, 131.0, 129.9, 128.5, 128.0, 127.5, 126.0, 67.7, 55.3, 47.6 ppm. IR (solid state): ν = 1659, 1497, 1452, 1431, 1365, 1330, 1256, 1168, 1132, 1102, 1051, 1030, 940, 791, 757, 722 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 317.0964, $C_{16}H_{17}N_2O_3S$ requires 317.0960.

***N*-benzhydryl-2-(4,5-dihydro-1,3-oxazol-2-yl)benzenesulfonamide (3ad):**

Prepared from **13ad** (0.35 g, 0.8 mmol), stirring for 16 h. Purification by flash chromatography (CH_2Cl_2) gave **3ad** (0.29 g, 86 %) as a white solid. R_f 0.15 (CH_2Cl_2). Mp. 115 – 118 ºC. ^{1}H (400 MHz, $CDCl_3$): δ = 9.00 (d, *J* = 9.0 Hz, 1H, SO_2NH), 7.75 (dd, *J* = 7.3, 1.2 Hz, 1H, Ar), 7.69 (dd, *J* = 7.3, 1.2 Hz, 1H, Ar), 7.43 (dt, *J* = 7.5, 1.2 Hz, 1H, Ar), 7.32 (dt, *J* = 7.5, 1.2 Hz, 1H, Ar), 7.16-7.12 (m, 10H, Ar), 5.67 (d, *J* = 8.8 Hz, 1H, $NHCH(Ph)_2$), 4.44 (t, *J* = 9.3 Hz, 2H, NCH_2), 4.11 (t, *J* = 9.3 Hz, 2H, OCH_2) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 163.5, 140.7, 140.6, 131.4, 130.7, 130.6, 129.2, 128.2, 127.6, 127.3, 125.3, 67.5, 62.4, 55.2 ppm. IR (solid state): ν = 1655, 1599, 1494,

1456, 1362, 1328, 1265, 1252, 1165, 1097, 1047, 1029, 937, 832, 749, 737, 700, 692 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 393.1270, $C_{22}H_{21}N_2O_3S$ requires 393.1273.

_N_-benzyl-2-[(4*S*)-4-methyl-4,5-dihydro-1,3-oxazol-2-yl]benzenesulfonamide (3bb):

Prepared from **13bb** (2.50 g, 6.8 mmol), stirring for 3 h. Purification by flash chromatography (CH_2Cl_2) gave **3bb** (2.06 g, 92 %) as a white solid. R_f 0.37 (CH_2Cl_2). Mp. 134 – 136 ºC. $[\alpha]_D$ = -20.0 [c 0.5, $CHCl_3$]. ^1H (500 MHz, $CDCl_3$): δ = 8.46 (br s, 1H, SO_2NH), 8.06 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.82 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.57-7.54 (m, 2H, Ar), 7.22-7.20 (m, 5H, Ar), 4.54-4.44 (m, 2H, $OCH_{2\alpha}$ and CHN), 4.14 (br s, 2H, $NHCH_2Ph$), 4.00 (dd, *J* = 7.8, 3.5 Hz, 1H, $OCH_{2\beta}$), 1.33 (d, *J* = 7.0 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 162.3, 139.5, 136.7, 132.0, 131.1, 130.9, 130.2, 128.4, 128.0, 127.5, 126.8, 74.1, 62.5, 47.6, 20.8 ppm. IR (solid state): ν = 1648, 1455, 1438, 1333, 1252, 1164, 1134, 1114, 1088, 1043, 959, 825, 779, 752, 697 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 330.1045, $C_{17}H_{18}N_2O_3S$ requires 330.1038.

_N_-benzyl-2-[(4*S*)-4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl]benzenesulfonamide (3cb):

Prepared from **13cb** (0.60 g, 1.50 mmol), stirring for 12 h. Purification by flash chromatography (CH_2Cl_2) gave **3cb** (0.40 g, 74 %) as a white solid. R_f 0.30 (CH_2Cl_2). Mp. 76 – 77 ºC. $[\alpha]_D$ = -23.0 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 8.71 (t, *J* = 6.1 Hz, 1H, SO_2NH), 8.01 (dd, *J* = 7.1, 1.5 Hz, 1H, Ar), 7.86 (dd, *J* = 7.1, 1.5 Hz, 1H, Ar), 7.59-7.56 (m, 2H, Ar), 7.27-7.21 (m, 5H, Ar), 4.49-4.43 (m, 1H, CHN), 4.16-4.08 (m, 4H, OCH_2 and CH_2Ph), 1.79 (octet, *J* = 6.7 Hz, 1H, $CHMe_2$), 0.91 (d, *J* = 6.7 Hz, 3H, *Me*), 0.88 (d, *J* = 6.7 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 162.4, 139.6, 136.7, 132.0, 131.2, 130.9, 130.0, 128.4, 128.1, 127.6, 126.1, 73.5, 70.7, 47.7, 32.8, 18.8, 18.7 ppm. IR (solid state): ν = 1654, 1457, 1359, 1334, 1318, 1254, 1168, 1103, 1053, 1032, 956, 794, 779, 736, 694. MS (EI+): *m/z* Found $[M]^+$ 358.1345, $C_{19}H_{22}N_2O_3S$ requires 358.1351.

_N_-isopropyl-2-[(4S)-4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl]benzenesulfonamide (3cc):

Prepared from **13cc** (0.45 g, 1.3 mmol), stirring for 3 h. Purification by flash chromatography (CH_2Cl_2) gave **3cc** (0.39 g, 98 %) as a viscous colourless oil. R_f 0.32 (CH_2Cl_2). $[\alpha]_D$ = -32.4 [c 0.45, $CHCl_3$]. ^1H (500 MHz, $CDCl_3$): δ = 8.27 (d, *J* = 6.4 Hz, 1H, SO_2NH), 8.16 (*apparent* dd, *J* = 5.6, 3.5 Hz, 1H, Ar), 7.88 (*apparent* dd, *J* = 5.8, 3.3 Hz, 1H, Ar), 7.60 (*apparent* dd, *J* = 5.8, 3.3 Hz, 2H, Ar), 4.50 (dd, *J* = 9.1, 8.0 Hz, 1H, $OCH_{2\alpha}$), 4.22-4.13 (m, 2H, CHN and $OCH_{2\beta}$), 3.48 (d septet, *J* = 6.6, 6.4 Hz, 1H, $NHCHMe_2$), 1.86 (d septet, *J* = 6.7, 6.5 Hz, 1H, $CHCHMe_2$), 1.11 (d, *J* = 6.6 Hz, 3H, *Me*), 1.07 (d, *J* = 6.7 Hz, 3H, *Me*), 1.04 (d, *J* = 6.6 Hz, 3H, *Me*), 0.98 (d, *J* = 6.7 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 162.5, 141.0, 131.8, 131.3, 130.8, 129.4, 126.1, 73.6, 70.7, 46.6,

33.0, 23.8, 23.4, 19.0, 18.9 ppm. IR (solid state): ν = 2968, 1651, 1468, 1438, 1387, 1335, 1250, 1176, 1144, 1133, 1096, 1050, 990, 961, 895, 855, 774, 749, 696 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 311.1432, $C_{15}H_{23}N_2O_3S$ requires 311.1429.

N-Benzhydryl-2-((4S)-4-isopropyl-4,5-dihydro-oxazol-2-yl)-benzene sulfonamide (3cd):

Prepared from **13cd** (0.30 g, 0.64 mmol), stirring for 3h. Purification by flash chromatography (CH_2Cl_2) gave **3cd** (0.12 g, 44 %) as a colourless oil as an inseparable mixture from the starting material (5.7:1 ratio). Data was collected on the inseparable mixture. R_f 0.27 (CH_2Cl_2). $[\alpha]_D$ = -17.4 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 9.28 (d, *J* = 8.4 Hz, 1H, SO_2NH), 7.77 (dd, *J* = 7.6, 0.8 Hz, 1H, Ar), 7.69 (dd, *J* = 7.6, 1.2 Hz, 1H, Ar), 7.43 (dt, *J* = 8.0, 1.6 Hz, 1H, Ar), 7.32 (dt, *J* = 7.6, 1.2 Hz, 1H, Ar), 7.19-7.08 (m, 10H, Ar), 5.66 (d, *J* = 8.4 Hz, 1H, $NHCH(Ph)_2$), 4.48-4.40 (m, 1H, NC*H*), 4.15-4.07 (m, 2H, OCH_2), 1.83-1.74 (m, 1H, $CHMe_2$), 0.94 (d, *J* = 6.8 Hz, 3H, Me), 0.89 (d, *J* = 6.8 Hz, 3H, Me) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 162.3, 140.0, 131.3, 130.5, 129.9, 129.0, 128.1, 127.7, 127.2, 125.3, 73.3, 70.5, 62.3, 32.7, 18.8 ppm. IR (solid state): υ = 2960, 1954, 1650, 1591, 1572, 1454, 1334, 1250, 1165, 1097, 1051, 960, 918, 830, 773, 697 cm^{-1}. MS (EI+): *m/z* Found $[M+H]^+$ 435.1748, $C_{25}H_{27}N_2O_3S$ requires 435.1743.

2-[(4S)-4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl]-*N*-[(1R)-1-phenylethyl]benzenesulfonamide (3ce):

Prepared from **13ce** (0.93 g, 2.2 mmol), stirring for 3 h. Purification by flash chromatography (CH_2Cl_2) gave **3ce** (0.63 g, 76 %) as a white solid. R_f 0.34 (CH_2Cl_2). Mp. 104-106 °C. $[\alpha]_D$ = -36.3 [c 0.4, $CHCl_3$]. ^1H (400 MHz, $CDCl_3$): δ = 8.71 (d, *J* = 7.9 Hz, 1H, SO_2NH), 7.70-7.64 (m, 2H, Ar), 7.42 (dt, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.30 (dt, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.14-7.02 (m, 5H, Ar), 4.53 (*apparent* q, *J* = 7.0 Hz, 1H, C*H*Me), *overlapped by* 4.49 (dd, *J* = 9.6, 8.2 Hz, 1H, $OCH_{2\alpha}CH$), 4.23 (ddd, *J* = 9.6, 8.9, 6.8 Hz, 1H, C*H*N), 4.12 (*apparent* t, *J* = 8.2 Hz, 1H, $OCH_{2\beta}CH$), 1.83 (octet, *J* = 6.8 Hz, 1H, $CHMe_2$), 1.46 (d, *J* = 7.0 Hz, 3H, ArCH*Me*), 1.03 (d, *J* = 6.8 Hz, 3H, *Me*), 0.95 (d, *J* = 6.8 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 162.7, 142.1, 140.7, 133.2, 131.3, 130.6, 130.5, 129.2, 128.1, 126.4, 125.6, 73.5, 70.7, 54.8, 32.9, 23.7, 18.9, 18.8 ppm. IR (solid state): ν = 1654, 1438, 1326, 1185, 1159, 1121, 1098, 1051, 954, 754, 718, 721, 697 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 373.1583, $C_{20}H_{25}N_2O_3S$ requires 373.1586.

2-[(4S)-4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl]-*N*-propylbenzenesulfonamide (3cg):

Prepared from **13cg** (0.71 g, 2.0 mmol), stirring for 3 h. Purification by flash chromatography (CH_2Cl_2) gave **3cg** (0.58 g, 92 %) as a viscous colourless oil. R_f 0.30 (CH_2Cl_2). $[\alpha]_D$ = -26.9 [c 1.0,

EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 8.19-8.13 (m, 2H, Ar), 7.89-7.85 (m, 1H, SO_2NH), 7.65-7.58 (m, 2H, Ar), 4.49 (ddd, *J* = 7.0, 3.5, 3.1 Hz, 1H, C*H*N), 4.21-4.14 (m, 2H, $OCH_{2\alpha}$ and $OCH_{2\beta}$), 2.59 (*apparent* q, *J* = 6.8 Hz, 2H, CH_2Et), 1.84 (octet, *J* = 6.9 Hz, 1H, C*H*Me$_2$), 1.50 (dt, *J* = 6.8 Hz, 2H, CH_2CH_3), 1.07 (d, *J* = 6.9 Hz, 3H, *Me*), 1.01 (d, *J* = 6.9 Hz, 3H, *Me*), 0.89 (t, *J* = 6.8 Hz, 3H, $CH_2CH_2CH_3$) ppm. ^{13}C (67.8 MHz, $CDCl_3$): δ = 163.2, 139.4, 131.9, 131.2, 130.8, 129.9, 125.7, 73.5, 70.7, 45.3, 32.9, 22.9, 19.0, 18.8, 11.4 ppm. IR (solid state): ν = 2962, 1651, 1459, 1334, 1250, 1168, 1136, 1097, 1050, 997, 960, 798, 779, 752, 701 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 311.1433, $C_{15}H_{23}N_2O_3S$ requires 311.1429.

***N*-(*tert*-butyl)-2-[(4S)-4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl]benzenesulfonamide (3ch):**
Prepared from **13ch** (0.13 g, 0.35 mmol), stirring for 3h. Purification by flash chromatography (CH_2Cl_2) gave **3ch** (0.08 g, 66 %) as a viscous oil. R_f 0.37 (CH_2Cl_2). $[\alpha]_D$ = -16.6 [c 1.0, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 8.39 (s, 1H, SO_2NH), 8.18-8.13 (m, 1H, Ar), 7.85-7.82 (m, 1H, Ar), 7.58-7.51 (m, 2H, Ar), 4.49-4.45 (m, 1H, $OCH_{2\alpha}$), 4.21-4.15 (m, 1H, C*H*N), *overlapped by* 4.16-4.10 (m, 1H, $OCH_{2\beta}$), 1.91 (m, 1H, C*H*Me$_2$), 1.22 (s, 9H, *t*Bu), 1.06 (d, *J* = 6.6 Hz, 3H, *Me*), 0.97 (d, *J* = 6.6 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 162.5, 143.4, 131.3, 130.6, 128.7, 125.5, 73.5, 70.4, 54.5, 32.7, 30.1, 18.9 ppm. IR (CH_2Cl_2 solution): υ = 3171, 2873, 1650, 1481, 1353, 1336, 1096, 1050, 965 cm^{-1}. MS (EI+): *m/z* Found $[M+H]^+$ 325.1557, $C_{16}H_{25}N_2O_3S$ requires 325.1586.

***N*-benzyl-2-[(4*S*)-4-benzyl-4,5-dihydro-1,3-oxazol-2-yl]benzenesulfonamide (3db):**
Prepared from **13db** (1.30 g, 2.93 mmol), stirring for 3 h. Purification by flash chromatography (CH_2Cl_2) gave **3db** (1.14 g, 95 %) as a white solid. R_f 0.35 (CH_2Cl_2). Mp. 90 – 93 ^{o}C (dec. CH_2Cl_2). $[\alpha]_D$ = -25.8 [c 1.2, $CHCl_3$]. ^{1}H (400 MHz, $CDCl_3$): δ = 8.22 (br s, 1H, SO_2NH *exchanges with* H_2O), 8.04 (dd, *J* = 7.2, 1.7 Hz, 1H, Ar), 7.77 (dd, *J* = 7.2, 1.7 Hz, 1H, Ar), 7.54 (m, 2H, Ar), 7.19-7.10 (m, 10H, Ar), 4.68-4.63 (m, 1H, C*H*N), 4.47 (dd, *J* = 9.5, 8.5 Hz, 1H, $OCH_{2\alpha}$), 4.17 (dd, *J* = 9.5, 8.5 Hz, 1H, $OCH_{2\beta}$), 4.11 (d, *J* = 14.0 Hz, 1H, $NHCH_{2\alpha}Ph$), 4.04 (d, *J* = 14.0 Hz, 1H, $NHCH_{2\beta}Ph$), 3.06 (dd, *J* = 14.0, 7.2 Hz, 1H, $CHCH_{2\alpha}Ph$), 2.84 (dd, *J* = 14.0, 7.2 Hz, 1H, $CHCH_{2\beta}Ph$) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 163.0, 139.7, 137.7, 136.8, 131.9, 131.1, 131.0, 129.8, 129.2, 128.7, 128.4, 127.9, 127.5, 126.7, 126.0, 72.2, 68.6, 47.5, 41.4 ppm. IR (solid state): ν = 1644, 1452, 1438, 1370, 1327, 1310, 1252, 1165, 1137, 1103, 1059, 1026, 963, 781, 770, 732, 699 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 406.1353, $C_{23}H_{22}N_2O_3S$ requires 406.1351.

General procedure for the reaction of saccharin 6 with 1,2-aminoalcohols, preparation of (4):
To a stirred solution of appropriate *N*-alkylsaccharin **6** (40.0 mmol) in dioxane (60 mL) was added

the required 1,2-aminoalcohol (40.0 mmol, see Scheme 3 for range employed). The reaction mixture was refluxed for 8-96 h, until no further saccharin was consumed by TLC analysis. [For the preparation of **4ab** and **4db** the reactions were carried out in THF (b.p. 67 ^{o}C). For **4ad**, **4cd**, **4ce** and **4ch** reaction was performed in dioxane or toluene in a sealed tube at 130 ^{o}C (6-20 h). The solvent was concentrated *in vacuo* to yield a colourless viscous oil. Purification by gradient chromatography (CH_2Cl_2 followed by CH_2Cl_2/MeOH 5 %) gave **4**, which was isolated as a viscous oil. After several days drying and standing under high vacuum (0.1 mm Hg, 20-40 ^{o}C) in most cases the oils solidified to white solids (yields, 52-95%).

2-[(Benzylamino)sulfonyl]-*N*-(2-hydroxyethyl)benzamide (4ab):

Prepared from ethanolamine (1.55 mL, 25.6 mmol) and **6b** (7.00 g, 25.6 mmol) in THF (20 mL), refluxing for 16 h. Purification by flash chromatography gave **4ab** (7.72 g, 90 %) as a viscous oil. R_f 0.25 (5 % MeOH/CH_2Cl_2). ^{1}H (400 MHz, $CDCl_3$): δ = 7.80 (d, *J* = 7.0 Hz, 1H, Ar), 7.61-7.48 (m, 3H, Ar), 7.25-7.19 (m, 5H, Ar), 6.67 (br t, 1H, SO_2NH), *overlapped by* 6.62 (t, *J* = 6.5 Hz, 1H, CON*H*), 4.13 (d, *J* = 6.5 Hz, CH_2Ph), 3.82 (br s, 2H, NCH_2), 3.56 (*apparent* q, *J* = 5.3 Hz, 2H, CH_2OH), 2.62 (br s, 1H, O*H*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 170.1, 137.7, 136.2, 135.2, 132.7, 130.2, 129.2, 128.6, 128.4, 128.1, 127.7, 60.9, 47.7, 42.9 ppm. IR (solid state): ν = 1644, 1541, 1456, 1425, 1327, 1162, 1130, 1061, 1029, 822, 785, 739, 698 cm^{-1}. MS (FAB+): *m/z* Found $[M]^+$ 335.1080, $C_{16}H_{19}N_2O_4S$ requires 335.1066. Anal. Calc. for. $C_{16}H_{18}N_2O_4S$: C, 57.5; H, 5.4; N, 8.4 %. Found: C, 57.4; H, 5.4; N, 8.0 %.

2-[(Benzhydrylamino)sulfonyl]-*N*-(2-hydroxyethyl)benzamide (4ad):

Prepared from ethanolamine (0.17 mL, 2.8 mmol) and **6d** (1.00 g, 2.8 mmol) in dioxane (10 mL), stirring at 130 ^{o}C for 6 h. Purification by flash chromatography gave **4ad** (0.75 g, 64 %) as a white low melting foam. R_f 0.40 (5 % MeOH/CH_2Cl_2). Mp. 54 – 55 °C (CH_2Cl_2). ^{1}H (400 MHz, $CDCl_3$): δ = 7.46 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.41-7.36 (m, 2H, Ar), 7.16-7.12 (m, 12H, 11-*Ar* and SO_2NH), 6.52 (br d, *J* = 5.5 Hz, 1H, CON*H*), 5.67 (d, *J* = 9.1 Hz, 1H, C*H*Ph_2), 3.80 (t, *J* = 5.5 Hz, 2H, NCH_2), 3.52 (t, *J* = 5.8 Hz, 2H, CH_2OH), 2.63 (t, *J* = 6.0 Hz, 1H, CH_2OH) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 170.4, 140.0, 138.9, 134.4, 132.1, 129.9, 129.8, 128.5, 128.4, 128.2, 127.6, 127.5, 62.4, 60.9, 43.0 ppm. IR (solid state): ν = 1643, 1541, 1454, 1421, 1327, 1164, 1131, 1051, 1026, 767, 757, 742, 719, 699, 684 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 411.1380, $C_{22}H_{23}N_2O_4S$ requires 411.1379. Anal. Calc. for $C_{22}H_{22}N_2O_4S \cdot ½H_2O$: C, 63.0; H, 5.5; N, 6.7 %. Found: C, 63.4; H, 5.5; N, 6.6 %.

***N*-(2-Hydroxyethyl)-2-{[(1-phenylethyl)amino]sulfonyl}benzamide (±)-(4ae):**

Prepared from ethanolamine (0.02 mL, 0.4 mmol) and (±)-**6e** (0.10 g, 0.4 mmol) in dioxane (5 mL), refluxing for 16 h. Purification by flash chromatography gave (±)-**4ae** (0.12 mg, 95 %) as a colourless viscous oil. R_f 0.31 (5 % MeOH/CH_2Cl_2). ^{1}H (400 MHz, CD_3OD): δ = 7.51 (d, *J* = 7.5 Hz, 1H, Ar), 7.44-7.41 (m, 2H, Ar), 7.26-7.22 (m, 1H, Ar), 7.13-7.11 (m, 1H, Ar), 7.07-7.05 (m, 4H, Ar), 6.69 (d, *J* = 7.3 Hz, 1H, SO_2NH), *overlapped by* 6.65 (br t, 1H, CON*H*), 4.51 (dt, *J* = 7.3, 6.9 Hz, 1H, C*H*(Me)Ph), 3.88-3.84 (m, 1H, $CH_{2\alpha}OH$), 3.82-3.78 (m, 1H, $CH_{2\beta}OH$), 3.62-3.58 (m, 1H, $NHCH_{2\alpha}$), 3.50-3.46 (m, 1H, $NHCH_{2\beta}$), 2.74 (br s, 1H, O*H*), 1.42 (d, *J* = 6.9 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 170.4, 141.8, 138.8, 134.6, 132.1, 129.9, 128.6, 128.3, 127.9, 127.3, 126.3, 60.9, 54.8, 43.0, 23.7 ppm. IR (solid state): ν = 1645, 1593, 1544, 1458, 1423, 1325, 1267, 1209, 1164, 1118, 1072, 952, 786, 758, 735, 700, 682 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 349.1221, $C_{17}H_{21}N_2O_4S$ requires 349.1222.

2-[(Benzylamino)sulfonyl]-*N*-[(1*S*)-2-hydroxy-1-methylethyl]benzamide (4bb):

Prepared from *L*-alaninol (2.00 g, 26.7 mmol) and **6b** (7.28 g, 26.7 mmol) in dioxane (25 mL), refluxing for 24 h. Purification by flash chromatography gave **4bb** (5.90 g, 64 %) as a viscous oil which solidified upon prolonged drying to a white solid. R_f 0.35 (5 % MeOH/CH_2Cl_2). Mp. 115 – 116 ^{o}C (CH_2Cl_2). $[\alpha]_D$ = -11.6 [c 1.0, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.55-7.46 (m, 3H, Ar), 7.22-7.20 (m, 5H, Ar), 6.49 (d, *J* = 8.0 Hz, 1H, CON*H*), 4.17-4.13 (m, 1H, NC*H*), *overlapped by* 4.15 (d, *J* = 13.9 Hz, 1H, $CH_{2\alpha}Ph$), 4.02 (d, *J* = 13.9 Hz, 1H, $CH_{2\beta}Ph$), 3.80 (dd, *J* = 11.4, 3.5 Hz, 1H, $CH_{2\alpha}OH$), 3.50 (dd, *J* = 11.4, 6.0 Hz, 1H, $CH_{2\beta}OH$), 1.20 (d, *J* = 6.9 Hz, 3H, *Me*) ppm. Missing SO_2NH and O*H* due to exchange with MeOH. ^{13}C (100 MHz, $CDCl_3$): δ = 169.4, 137.7, 136.2, 135.4, 132.6, 130.1, 129.2, 128.5, 128.4, 128.0, 127.7, 65.5, 48.5, 47.6, 16.6 ppm. IR (solid state): ν = 3512, 3236, 1641, 1565, 1458, 1442, 1331, 1216, 1164, 1128, 1065, 1039, 849, 747, 703 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 349.1214, $C_{17}H_{21}N_2O_4S$ requires 349.1222. Anal. Calc. for $C_{17}H_{20}N_2O_4S$: C, 58.6; H, 5.8; N, 8.0 %. Found: C, 58.4; H, 5.8; N, 8.1 %.

2-[(Benzylamino)sulfonyl]-*N*-[(1*S*)-1-hydroxymethyl)-2-methylpropyl]benzamide (4cb):

Prepared from *L*-valinol (3.96 g, 38.4 mmol) and **6b** (10.5 g, 38.4 mmol) in dioxane (60 mL), refluxing for 72 h. Purification by flash chromatography gave **4cb** as a viscous oil. After several days drying and standing under high vacuum the oil solidified to a white solid (10.0 g, 69 %). R_f 0.36 (5 % MeOH/CH_2Cl_2). Mp 136 – 137 ^{o}C (CH_2Cl_2). $[\alpha]_D$ = -28.9 [c 1.0, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 7.90 (dd, *J* = 7.9, 1.0 Hz, 1H, Ar), 7.60-7.46 (m, 3H, Ar), 7.22-7.18 (m, 5H, Ar), 6.72

(t, J = 7.0 Hz, 1H, SO_2NH), 6.39 (d, J = 8.9 Hz, 1H, CONH), 4.18 (dd, J = 13.8, 7.0 Hz, 1H, $PhCH_{2\alpha}$), 4.00 (dd, J = 13.8, 5.2 Hz, 1H, $PhCH_{2\beta}$), 3.88-3.82 (m, 1H, NCH), 3.78 (dd, J = 12.2, 6.3 Hz, 1H, $CH_{2\alpha}OH$), 3.65 (dd, J = 12.2, 3.0 Hz, 1H, $CH_{2\beta}OH$), 2.95 (br s, 1H, OH), 1.80 (octet, J = 6.9 Hz, 1H, $CHMe_2$), 1.00 (d, J = 6.9 Hz, 3H, *Me*), 0.99 (d, J = 6.9 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.8, 137.7, 136.2, 135.6, 132.7, 130.1, 129.3, 128.5, 128.4, 128.1, 127.7, 63.0, 58.1, 47.6, 29.1, 19.7, 19.1 ppm. IR (solid state): ν = 3512, 3232, 1640, 1565, 1431, 1330, 1215, 1166, 1015, 749, 736, 701 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 377.1352, $C_{19}H_{24}N_2O_4S$ requires 377.1535. Anal. Calc. for $C_{19}H_{24}N_2O_4S$: C, 60.6; H, 6.4; N, 7.4 %. Found: C, 60.7; H, 6.4; N, 7.4 %

***N*-[(1*S*)-1-Hydroxymethyl)-2-methylpropyl]-2-[(isopropylamino)sulfonyl]benzamide (4cc):** Prepared from *L*-valinol (1.50 g, 14.5 mmol) and **6c** (3.26 g, 14.5 mmol) in dioxane (25 mL), refluxing for 48 h. Purification by flash chromatography gave **4cc** (1.50 g, 82 %) as a white solid. R_f 0.46 (5 % MeOH/CH_2Cl_2). Mp. 171 – 175 °C (dec. MeOH). $[\alpha]_D$ = -46.7 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 8.05 (dd, J = 8.0, 1.0 Hz, 1H, Ar), 7.64-7.56 (m, 3H, Ar), 6.35 (d, J = 9.2 Hz, 1H, CONH), 6.15 (d, J = 6.5 Hz, 1H, SO_2NH), 4.00-3.94 (m, 2H, CH_2OH), 3.76-3.70 (m, 1H, NHCH), 3.43 (septet, J = 6.5 Hz, 1H, $NHCH(Me)_2$), 2.50 (t, J = 6.7 Hz, 1H, OH), 1.92 (septet, J = 6.8 Hz, 1H, $CHCHMe_2$), 1.80 (d, J = 6.5 Hz, 3H, *Me*), 1.06 (d, J = 6.8 Hz, 3H, *Me*), *overlapped by* 1.05 (d, J = 6.8 Hz, 3H, *Me*), 0.98 (d, J = 6.5 Hz, 3H, *Me*) ppm. ^{13}C (67.8 MHz, $CDCl_3$): δ = 169.5, 138.7, 135.4, 132.5, 130.1, 128.8, 128.6, 63.1, 58.0, 46.6, 29.1, 24.1, 22.8, 19.6, 19.0 ppm. IR (solid state): ν = 3321, 3085, 2963, 1633, 1550, 1462, 1438, 1335, 1317, 1254, 1155, 1142, 1117, 1068, 1016, 982, 906, 887, 793, 761, 736, 676 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 329.1532, $C_{15}H_{25}N_2O_4S$ requires 329.1535. Anal. Calc. for $C_{15}H_{24}N_2O_4S$: C, 54.9; H, 7.4; N, 8.5 %. Found: C, 54.9; H, 7.4; N, 8.5 %.

***N*-[(1*S*)-1-(Hydroxymethyl)-2-methylpropyl]-2-[(benzhydrylamino)sulfonyl]benzamide (4cd):** Prepared from *L*-valinol (1.00 g, 9.7 mmol) and **6d** (3.38 g, 9.7 mmol) in dioxane (20 mL), stirring at 130 °C for 20 h. Purification by flash chromatography gave **4cd** (3.19 g, 73 %) as a white foam. R_f 0.27 (5 % MeOH/CH_2Cl_2). Mp. 88 – 89 °C (CH_2Cl_2). $[\alpha]_D$ = -24.0 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.51 (dd, J = 8.0, 1.0 Hz, 1H, Ar), 7.45-7.38 (m, 3H, Ar), 7.24-7.19 (m, 6H, 5-Ar and SO_2NH), 7.13-7.05 (m, 5H, Ar), 6.08 (d, J = 8.7 Hz, 1H, CONH), 5.70 (d, J = 9.1 Hz, 1H, $CHPh_2$), 3.94-3.88 (m, 2H, CHN and $CH_{2\alpha}OH$), 3.73-3.70 (m, 1H, $CH_{2\beta}OH$), 2.43 (br t, J = 5.6 Hz, 1H, CH_2OH), 1.93 (septet, J = 6.9 Hz, 1H, $CHMe_2$), 1.02 (d, J = 6.9 Hz, 3H, *Me*), 1.01 (d, J = 6.9 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 170.2, 140.1, 139.6, 134.9, 132.0, 129.8, 128.5, 128.3, 127.8, 127.5, 127.4, 62.9, 62.4, 58.2, 29.1, 19.7, 19.3 ppm. IR (solid state): ν = 1647, 1556, 1494,

1458, 1433, 1325, 1165, 1133, 1080, 1024, 1010, 869, 790, 748, 737, 701 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 453.1862, $C_{25}H_{29}N_2O_4S$ requires 453.1848. Anal. Calc. for $C_{25}H_{28}N_2O_4S$: C, 66.4; H, 6.2; N, 6.2 %. Found: C, 66.1; H, 6.2; N, 6.0 %.

***N*-[(1S)-1-(hydroxymethyl)-2-methylpropyl]-2-({[(1R)-1-phenylethyl]amino}sulfonyl)benzamide (4ce):**

Prepared from L-valinol (0.20 g, 1.94 mmol) and **11e** (0.45 g, 1.41 mmol) in toluene (2 mL), stirring at reflux in a sealed tube for 24h. Purification by flash chromatography gave **4ch** (0.40 g, 73 %) as a viscous oil, which crystallised on standing. R_f 0.32 (5 % MeOH/CH_2Cl_2). Mp. 98 – 99 ^oC (CH_2Cl_2). $[\alpha]_D$ = +5.0 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.36-7.35 (m, 3H, Ar), 7.12-7.04 (m, 3H, Ar), 6.99-6.97 (m, 3H, Ar), 6.71 (d, *J* = 8.4 Hz, 1H, SO_2NH), 6.34 (br s, 1H, CON*H*), 4.53 (dq, *J* = 8.4, 7.0 Hz, 1H, C*H*(Me)Ph), 3.93-3.83 (m, 2H, NC*H* and $CH_{2\alpha}OH$), 3.65 (dd, *J* = 10.7, 6.4 Hz, 1H, $CH_{2\beta}OH$), 2.87 (br s, 1H, CH_2OH), 1.90 (septet, *J* = 6.9 Hz, 1H, C*H*Me_2), 1.46 (d, *J* = 7.0 Hz, 3H, PhCH*Me*), 1.01 (d, *J* = 6.9 Hz, 6H, Me) ppm. ^{13}C (67.8 MHz, $CDCl_3$): δ = 170.1, 141.1, 138.6, 134.7, 131.8, 129.5, 128.2, 128.0, 127.4, 127.1, 126.3, 62.8, 58.0, 55.0, 28.9, 23.9, 19.7, 19.1 ppm. IR (CH_2Cl_2 solution): υ = 3543, 3412, 3253, 2930, 2876, 1658, 1650, 1519, 1337, 1172, 1070, 1030 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 391.1674, $C_{20}H_{27}N_2O_4S$ requires 391.1692.

N-[(1S)-1-Hydroxymethyl-2-methyl-propyl]-2-propylsulfamoyl-benzamide (4cg):

Prepared from L-valinol (0.60 g, 5.8 mmol) and **6g** (1.30 g, 5.8 mmol) in dioxane (5 mL), refluxing for 24 h. Purification by flash chromatography gave **4cg** (0.97 g, 51 %) as a viscous oil. R_f 0.50 (5 % MeOH/CH_2Cl_2). $[\alpha]_D$ = -28.7 [c 1.0 EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.91 (dd, *J* = 7.6, 1.0 Hz, 1H, Ar), 7.59-7.49 (m, 3H, Ar), 6.63 (d, *J* = 8.8 Hz, 1H, SO_2NH), 6.30-6.27 (m, 1H, CON*H*), 3.89-3.79 (m, 2H, C*H*N and $CH_{2\alpha}OH$), 3.68-3.64 (m, 1H, $CH_{2\beta}OH$), 3.65 (br. s, 1H, CH_2OH), 2.90-2.85 (m, 1H, $NCH_{2\alpha}CH_2CH_3$), 2.77-2.73 (m, 1H, $NCH_{2\beta}CH_2CH_3$), 1.91-1.86 (m, 1H, C*H*Me_2), 1.45-1.42 ($NCH_2CH_{2\alpha}CH_3$ and $NCH_2CH_{2\beta}CH_3$), 0.98 (dd, *J* = 6.8, 4.0 Hz, 6H, CH*Me*$_2$), 0.83 (t, *J* = 7.4 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.6, 137.4, 135.6, 132.6, 129.9, 129.1, 128.5, 62.8, 58.0, 45.2, 29.0, 22.7, 19.6, 19.0, 11.1 ppm. IR (CH_2Cl_2 solution): ν = 3412, 3249, 2932, 2876, 1657, 1520, 1335, 1169, 1126, 1071 cm^{-1}. MS (EI+): *m/z* Found $[M+H]^+$ 329.1536, $C_{15}H_{25}N_2O_4S$ requires 329.1541.

***N*-[(1S)-1-(hydroxymethyl)-2-methylpropyl]-2-[(*tert*-butylamino)sulfonyl]benzamide** (**4ch**):

Prepared from L-valinol (0.24 g, 2.29 mmol) and **11h** (0.39 g, 1.53 mmol) in toluene (15 mL), stirring under Dean-Stark conditions for 120h. Purification by flash chromatography gave **4ch** (0.23 g, 45 %) as a viscous oil, which crystallised on standing. R_f 0.37 (5 % MeOH / CH_2Cl_2). Mp. 78 –

79 °C (CH_2Cl_2). $[\alpha]_D$ = +2.0 [c 1.0, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 8.01 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.58-7.53 (m, 3H, Ar), 6.55 (d, *J* = 9.1 Hz, 1H, CON*H*), 6.50 (s, 1H, SO_2N*H*), 3.96 (ddd, *J* = 12.5, 5.1, 3.3 Hz, 1H, C$H_{2\alpha}$OH), *overlapped by* 3.91-3.88 (m, 1H, C*H*N), 3.70 (ddd, *J* = 12.5, 7.2, 5.3 Hz, 1H, C$H_{2\beta}$OH), 2.84 (t, *J* = 5.9 Hz, 1H, CH_2O*H*), 1.88 (septet, *J* = 6.9 Hz, 1H, C*H*Me_2), 1.20 (s, 9H, ^{t}Bu), 1.00 (d, *J* = 6.9 Hz, 3H, Me), *overlapped by* 0.99 (d, *J* = 6.9 Hz, 3H, Me) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.7, 141.3, 135.4, 132.2, 130.2, 128.9, 128.2, 63.2, 58.0, 55.0, 30.1, 29.3, 19.7, 19.1 ppm. IR (solid state): ν = 3310, 1715, 1466, 1439, 1427, 1393, 1371, 1327, 1287, 1265, 1230, 1194, 1153, 1170, 1138, 1118, 1059, 986, 956, 859, 793, 761, 729, 713 cm^{-1}. MS (EI+): *m/z* Found $[M+Na]^+$ 365.1511, $C_{16}H_{26}NaN_2O_4S$ requires 365.1505.

2-[(Benzylamino)sulfonyl]-*N*-[(1*S*)-1-benzyl-2-hydroxyethyl]benzamide (4db):
Prepared from *L*-phenylalaninol (2.70 g, 18.3 mmol) and **6b** (5.00 g, 18.3 mmol) in THF (30 mL), refluxing for 96h. Purification by flash chromatography gave **4db** (3.70 g, 48 %) as a white foam. R_f 0.43 (5 % MeOH/CH_2Cl_2). Mp. 125 – 127 °C (CH_2Cl_2). $[\alpha]_D$ = -35.5 [c 1.0, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 7.85 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.49-7.44 (m, 2H, Ar), 7.32-7.30 (m, 2H, Ar), 7.23-7.19 (m, 9H, Ar), 6.55 (t, *J* = 6.3 Hz, 1H, SO_2N*H*), 6.32 (d, *J* = 8.3 Hz, 1H, CON*H*), 4.33-4.32 (m, 1H, NC*H*), 4.18 (dd, *J* = 13.9, 7.1 Hz, 1H, NC$H_{2\alpha}$), 4.00 (dd, *J* = 13.9, 5.5 Hz, 1H, NC$H_{2\beta}$), 3.85 (dd, *J* = 11.5, 3.4 Hz, 1H, C$H_{2\alpha}$OH), 3.66 (dd, *J* = 11.5, 5.6 Hz, 1H, C$H_{2\beta}$OH), 2.93 (d, *J* = 7.6 Hz, 2H, CH_2Ph), 2.73 (br s, 1H, O*H*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.5, 137.7, 137.6, 136.2, 135.4, 132.7, 130.1, 129.3, 129.2, 128.8, 128.6, 128.2, 128.1, 127.7, 126.9, 63.2, 53.8, 47.7, 36.7 ppm. IR (solid state): ν = 1655, 1638, 1558, 1524, 1496, 1455, 1440, 1330, 1164, 1130, 1041, 746, 733, 701 cm^{-1}. MS (ES+): *m/z* Found $[M+H]^+$ 425.1569, $C_{23}H_{25}N_2O_4S$ requires 425.1535. Anal. Calc. for $C_{23}H_{24}N_2O_4S.½H_2O$: C, 63.7; H, 5.8; N, 6.5 %. Found: C, 64.0; H, 5.5; N, 6.4 %.

***N*-[(1*R*)-1-(Hydroxymethyl)-3-(methylthio)propyl]-2-[(benzylamino)sulfonyl]benzamide (4eb):**
Prepared from *L*-methionol (0.50 g, 3.7 mmol) and **6b** (1.00 g, 3.7 mmol) in dioxane (10 mL), refluxing for 60 h. Purification by flash chromatography gave **4eb** (0.70 g, 52 %) as a viscous oil. R_f 0.31 (5 % MeOH/CH_2Cl_2). $[\alpha]_D$ = -17.0 [c 0.37, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 7.91 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.59-7.55 (m, 3H, Ar), 7.24-7.22 (m, 5H, Ar), 6.60 (t, *J* = 6.5 Hz, 1H, SO_2N*H*), 6.47 (d, *J* = 8.5 Hz, 1H, CON*H*), 4.22-4.19 (m, 1H, NC*H*), *overlapped by* 4.18 (dd, *J* = 14.0, 7.0 Hz, 1H, C$H_{2\alpha}$Ph), 4.05 (dd, *J* = 14.0, 6.0 Hz, 1H, C$H_{2\beta}$Ph), 3.90 (dd, *J* = 11.5, 3.5 Hz, 1H, C$H_{2\alpha}$OH), 3.66 (dd, *J* = 11.5, 5.5 Hz, 1H, C$H_{2\beta}$OH), 2.64 (m, 2H, CH_2CH_2S), 2.13 (s, 3H, S*Me*), 1.91 (q, *J* = 7.2; 7.1 Hz, 2H, C$H_2$$CH_2$S) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.8, 137.8, 136.2, 135.4, 132.7, 130.2, 129.3, 128.6, 128.2, 128.1, 127.7, 63.9, 52.2, 47.7, 30.9, 30.1, 28.3 ppm. IR

(solid state): ν = 2917, 1644, 1537, 1455, 1437, 1329, 1163, 1129, 1062, 786, 738 cm^{-1}. MS (FAB+): *m/z* Found $[M]^+$ 409.1257, $C_{19}H_{24}N_2O_4S$ requires 409.1256

General procedure for the preparation of *N*-alkylated saccharin derivatives (6):

To a stirred solution of sodium saccharin dihydrate (10.0 mmol) in DMF (20 mL) was added the appropriate alkyl halide (10.0 mmol), the reaction mixture was refluxed for 24-48 h at 90 °C, after which time a white precipitate had formed. The reaction mixture was cooled and CH_2Cl_2 (10 mL) and water (10 mL) were added. The reaction mixture was transferred to a separating funnel and 2 M HCl (10 mL) was added. The layers were separated and the aqueous layer washed with more CH_2Cl_2 (2 x 10 mL). The combined organic layers were washed with brine (2 x 10 mL), dried over $MgSO_4$ and concentrated *in vacuo* to yield a colourless viscous oil. Purification was carried out by either flash silica column chromatography or by recrystallisation as stated respectively for each compound.

2-benzyl-1,2-benzisothiazol-3(2H)-one 1,1-dioxide or *N*-benzyl saccharin (6b):

Prepared from sodium saccharin dihydrate (20.00 g, 90.0 mmol) and benzyl bromide (10.80 mL, 90.0 mmol) in DMF (40 mL). Purification by recrystallisation from cold EtOH gave **6b** (23.60 g, 96 %), as large colourless crystals. R_f 0.35 (25 % EtOAc/Hexane) Mp. 108-110 °C (EtOH; *lit.*[1] 109 °C, EtOH). ^1H (500 MHz, $CDCl_3$): δ = 8.02 (d, *J* = 7.0 Hz, 1H, Ar), 7.91-7.89 (m, 1H, Ar), 7.83-7.75 (m, 2H, Ar), 7.53-7.51 (m, 2H, Ar), 7.38-7.30 (m, 3H, Ar), 4.91 (s, 2H, C*H*$_2$Ph) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 158.9, 137.7, 134.8, 134.5, 134.4, 128.7, 128.7, 128.3, 127.2, 125.2, 121.0, 42.7 ppm. IR (solid state): ν = 2923, 2853, 1737, 1593, 1458, 1438, 1323, 1296, 1266, 1182, 1050, 959, 860, 756 cm^{-1}. MS (EI+): m/z Found $[M+H]^+$ 274.0539, $C_{14}H_{12}NO_3S$ requires 274.0543. Anal. Calc. for $C_{14}H_{11}NO_3S$: C, 61.5; H, 4.1; N, 5.1 %. Found: C, 61.4; H, 4.0; N, 5.0 %.

2-isopropyl-1,2-benzisothiazol-3(2H)-one 1,1-dioxide or isopropyl saccharin (6c):

Prepared from sodium saccharin dihydrate (12.00 g, 58.5 mmol) and 2-bromopropane (5.50 mL, 58.5 mmol) in DMF (30 mL). Purification by recrystallisation from cold EtOH gave **6c** as colourless crystals (10.00 g, 76 %). R_f 0.13 (50 % CH_2Cl_2/light petroleum). Mp. 63 – 64 ^oC (EtOH; *lit.*[2] 62 – 64 ^oC, MeOH). ^1H (400 MHz, $CDCl_3$): δ = 8.07 (dd, *J* = 7.3, 1.0 Hz, 1H, Ar), 8.02 (dd, *J* = 7.3, 1.0 Hz, 1H, Ar), 7.97 (dt, *J* = 7.3, 1.0 Hz, 1H, Ar), 7.95 (dt, *J* = 7.3, 1.0 Hz, 1H, Ar), 4.52 (septet, *J* = 7.0 Hz, 1H, C*H*Me$_2$), 1.61 (d, *J* = 7.0 Hz, 6H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 160.3, 139.4, 136.4, 135.8, 128.5, 125.9, 121.9, 48.2, 20.6 ppm. IR (solid state): ν = 1732, 1455, 1321, 1289, 1250, 1211, 1199, 1178, 1159, 1060, 984, 747 cm^{-1}. MS (ES+): *m/z* Found $[M]^+$

225.0461, $C_{10}H_{11}NO_3S$ requires 225.0460. Anal. Calc. for $C_{10}H_{11}NO_3S$: C, 53.3; H, 4.9; N, 6.2 %. Found: C, 53.2; H, 4.9; N, 6.2 %.

2-benzhydryl-1,2-benzisothiazol-3(2H)-one 1,1-dioxide (6d):

Prepared from sodium saccharin dihydrate (10.00 g, 48.7 mmol) and bromodiphenylmethane (12.00 g, 48.7 mmol) and DMF (40 mL). Purification by recrystallisation from cold EtOH gave **6d** (13.93 g, 82 %) as colourless crystals. R_f 0.38 (33 % CH_2Cl_2/light petroleum). Mp. 156 – 158 ºC. ^{1}H (400 MHz, CD_3OD): δ = 7.98 (dd, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.81 (dd, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.84 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.79 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.45-7.44 (m, 4H, Ar), 7.36-7.34 (m, 6H, Ar), 6.51 (s, 1H, C*H*Ph$_2$) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 158.5, 137.8, 136.6, 134.9, 134.4, 129.1, 128.5, 128.3, 127.2, 125.4, 121.0, 60.5 ppm. IR (solid state): ν = 1721, 1495, 1456, 1338, 1293, 1250, 1181, 1061, 1010, 820, 780, 752, 702 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 349.0758, $C_{20}H_{16}NO_3S$ requires 349.0773.

(±)-2-(1-phenylethyl)-1,2-benzisothiazol-3(2H)-one 1,1-dioxide or α-methyl-benzyl saccharin (6e):

Prepared from sodium saccharin dihydrate (10.00 g, 48.7 mmol) and (1-bromoethyl)benzene (6.70 mL, 48.7 mmol) in DMF (30 mL). Purification by recrystallisation from cold EtOH gave **6e** as a white solid (12.80 g, 92 %). R_f 0.21 (50 % CH_2Cl_2/light petroleum). Mp. 96 – 97 ^{o}C (EtOH; *lit.*[3] 83 ^{o}C, EtOH). ^{1}H (400 MHz, $CDCl_3$): δ = 7.96 (dd, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.88 (dd, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.83 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.80 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.60 (d, *J* = 7.5 Hz, 2H, Ar), 7.36-7.34 (m, 2H, Ar), 7.32-7.28 (m, 1H, Ar), 5.44 (q, *J* = 6.9 Hz, 1H, C*H*(Me)Ph), 2.02 (d, *J* = 6.9 Hz, 3H, Me) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 158.6, 138.8, 137.9, 134.7, 134.3, 128.6, 128.3, 127.8, 127.4, 125.1, 120.8, 53.2, 17.7 ppm. IR (solid state): ν = 1723, 1457, 1329, 1294, 1256, 1184, 968, 751, 704 cm^{-1}. MS (EI+): *m/z* Found $[M+Na]^+$ 310.0514, $C_{15}H_{13}NaNO_3S$ requires 310.0508. Anal. Calc. for $C_{15}H_{13}NO_3S$: C, 62.7; H 4.6; N 4.9 %. Found: C, 62.6; H 4.4; N 4.8 %.

2-(2-bromoethyl)-1,2-benzisothiazol-3(2H)-one 1,1-dioxide (6f):

Prepared from sodium saccharin dihydrate (5.00 g, 20.7 mmol) and 1,2-dibromoethane (1.80 mL, 20.9 mmol) in DMF (20 mL). Purification by flash silica chromatography followed by recrystallisation from *i*PrOH gave **6f** (2.67 g, 44 %) as white crystals. R_f 0.58 (50 % EtOAc/light petroleum). Mp. 96-98 ºC (*i*PrOH; *lit.*[4] 99 ºC *aq*. EtOH). ^{1}H (400 MHz, $CDCl_3$): δ = 8.10-8.08 (m, 1H, Ar), 7.96-7.84 (m, 3H, Ar), 4.17 (t, *J* = 7.6 Hz, 2H, NC*H*$_2$), 3.67 (t, *J* = 7.6 Hz, 2H, C*H*$_2$Br) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 158.5, 137.4, 135.0, 134.4, 126.9, 125.3, 121.0, 39.8, 26.8 ppm.

IR (solid state): ν = 2360, 2340, 1732, 1462, 1446, 1335, 1296, 1257, 1229, 1176, 1162, 1033, 960, 752 cm^{-1}.MS (EI+): *m/z* Found $[M]^+$ 290.9383, $C_9H_8BrNO_3S$ requires 290.9388 Anal. Calc. for $C_9H_8BrNO_3S$: C, 37.3; H, 2.8; N 4.8 %. Found: C, 37.4; H, 2.8; N 4.8 %.

2-propyl-1,2-benzisothiazol-3(2H)-one 1,1-dioxide or n-propyl saccharin (6g):

Prepared from sodium saccharin dihydrate (10.00 g, 48.8 mmol) and 1-bromopropane (4.30 mL, 48.0 mmol) in DMF (30 ml). Purification by recrystallisation from cold EtOH gave **6g** as colourless crystals (8.30 g, 71 %). R_f 0.35 (75 % CH_2Cl_2/light petroleum). Mp. 76 °C (EtOH; *lit.*[5] 75 °C, EtOH). ^1H (400 MHz, $CDCl_3$): δ = 8.06 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.92 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.88-7.81 (m, 2H, Ar), 3.74 (*apparent* dt, *J* = 8.9, 7.4 Hz, 2H, NCH_2), 1.89 (dq, *J* = 7.4, 6.7 Hz, 2H, CH_2CH_3), 1.03 (t, 3H, *J* = 6.7 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 159.1, 137.8, 134.7, 134.3, 127.5, 125.2, 121.0, 41.1, 21.9, 11.4 ppm. IR (solid state): ν = 1732, 1468, 1460, 1323, 1310, 1300, 1267, 1181, 1166, 1127, 1062, 994, 793, 757, 750, 693, 677 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 225.0465, $C_{10}H_{11}NO_3S$ requires 225.0460. Anal. Calc. for $C_{10}H_{11}NO_3S$: C, 53.3; H, 4.9; N, 6.2 %. Found: C, 53.4; H, 4.8; N, 6.0 %.

2-tert-butyl-1,2-benzisothiazol-3(2H)-one 1,1-dioxide (6h):

Compound **6h** was isolated as a side product in the preparation of **4ch** from **11h** (0.32 g, 1.3 mmol). Purification by flash chromatography (CH_2Cl_2) gave **6h** (0.10 mg, 32 %) as a white solid. R_f 0.67 (CH_2Cl_2). Mp. 99 – 100 ºC (CH_2Cl_2; *lit.*[6] 100 – 101 ºC, CH_2Cl_2). ^1H (400 MHz, $CDCl_3$): δ = 8.00 (dd, *J*= 7.0, 1.0 Hz, 1H, Ar), 7.85-7.76 (m, 3H, Ar), 1.78 (s, 9H, ^{t}Bu) ppm. ^{13}C (67.8 MHz, $CDCl_3$): δ = 160.0, 137.9, 134.4, 134.0, 127.4, 124.6, 120.1, 61.1, 27.8 ppm. IR (solid state): ν = 1719, 1458, 1371, 1325, 1288, 1251, 1196, 1178, 1162, 1125, 1060, 967, 787, 752, 732 cm^{-1}. MS (CI+): *m/z* $[M+H]^+$ 240.0690, $C_{11}H_{14}NO_3S$ requires 240.0694. Anal. Calc. for $C_{11}H_{13}NO_3S$: C, 55.2; H, 5.5; N, 5.9 %. Found: C, 55.1; H, 5.4; N, 5.7 %.

2-phenyl-1,2-benzisothiazol-3(2H)-one 1,1-dioxide (6i):

To a stirred solution of **11i** (0.52 g, 1.79 mmol) in toluene (10 mL) was added DMAP (0.02 g, 0.18 mmol). The reaction was refluxed for 24 h, with no sign of product formation. Pyridine (0.13 mL, 1.79 mmol) was added and the reaction was refluxed for 18 h. The reaction mixture was concentrated *in vacuo* to give a viscous oil. Purification by recrystallisation in EtOH gave **6i** (0.27 mg, 66 %). R_f 0.20 (50 % CH_2Cl_2/light petroleum). Mp. 190 – 191 °C (EtOH; *lit.*[7] 189 – 191 °C, EtOH). ^1H (400 MHz, CD_3OD): δ = 8.17 (dd, *J* = 7.2, 1.0 Hz, 1H, Ar), 7.97-7.82 (m, 3H, Ar), 7.56-7.52 (m, 5H, Ar) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 158.5, 137.8, 135.2, 134.6, 130.5, 130.0, 129.1,

128.9, 127.6, 126.0, 121.3 ppm. IR (solid state): ν = 1746, 1726, 1590, 1491, 1462, 1339, 1328, 1309, 1271, 1214, 1184, 1160, 1127, 1100, 998, 987, 787, 754, 742, 694 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 259.0312, $C_{13}H_9NO_3S$ requires 259.0303. Anal. Calc. for $C_{13}H_9NO_3S$: C, 60.2; H, 3.5; N, 5.4 %. Found: C, 60.2; H 3.5; N, 5.5 %.

General procedure for the preparation of *O*-alkylated saccharin derivatives (7):

To a stirred solution of saccharin **6a** (5.5 mmol) and triphenylphosphine (6.8 mmol) in dry THF (20 mL) was added anhydrous benzyl alcohol (5.5 mmol) under inert atmosphere. The reaction mixture was cooled to 0 °C and diethyl azodicarboxylate (6.8 mmol) was added dropwise. The reaction mixture was allowed to warm to room temperature and was stirred for 16 h, after which time the solvent was concentrated *in vacuo*. Purification by flash silica chromatography and subsequent recrystallisation from cold EtOH gave the desired O-alkylated product.

3-Benzyloxy-1,2-benzisothiazole 1,1-dioxide (7b):

Prepared from saccharin **6a** (1.00 g, 5.5 mmol), triphenylphosphine (1.79 g, 6.8 mmol), anhydrous benzyl alcohol (0.59 mL, 5.5 mmol), diethyl azodicarboxylate (1.07 mL, 6.8 mmol) and dry THF (20 mL). Purification by flash silica chromatography and subsequent recrystallisation from cold EtOH gave **7b** (0.88 g, 59 %) as colourless crystals. Also isolated from the reaction mixture was compound **6b** (0.48 g). R_f 0.60 (1:1 Hexane/EtOAc) Mp. 118-120 °C (EtOH, *lit.*[8] 128-129 °C). ^1H (400 MHz, $CDCl_3$): δ = 7.88 (dt, *J* = 7.6, 0.8 Hz, 1H, Ar), 7.78-7.66 (m, 3H, Ar), 7.51-7.42 (m, 5H, Ar), 5.59 (s, 2H, OCH_2Ph) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.0, 143.5, 134.1, 133.4, 133.4, 129.4, 129.1, 128.9, 128.6, 126.9, 123.4, 121.9, 73.5 ppm. IR (solid state): ν = 2985, 2919, 2361, 2338, 1712, 1618, 1561, 1470, 1413, 1354, 1319, 1174, 1157, 1055, 973, 771 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 290.9405, $C_9H_8BrNO_3S$ requires 290.9418. Anal. Calc. for $C_9H_8BrNO_3S$: C, 37.3; H, 2.8; N, 4.8%. Found: C, 37.1; H, 2.8; N, 4.8%.

3-(2-Bromo-ethoxy)-benzo[d]isothiazole 1,1-dioxide (7f):

Prepared from saccharin **6a** (2.00 g, 10.9 mmol), triphenylphosphine (3.59 g, 13.7 mmol), anhydrous 2-bromoethanol (0.78 mL, 10.9 mmol), diethyl azodicarboxylate (2.15 mL, 13.7 mmol) and dry THF (20 mL). Purification by flash silica chromatography (25-100 % CH_2Cl_2/light petroleum) and subsequent recrystallisation from cold EtOH gave **7f** (1.23 g, 37 %) as colourless crystals. Also isolated from the reaction mixture was compound **6f** (0.74 g). R_f 0.40 Mp. 172-174 °C. ^1H (400 MHz, $CDCl_3$): δ = 7.91-7.89 (m, 1H, Ar), 7.82-7.71 (m, 3H, Ar), 4.89 (t, *J* = 6.0 Hz, 2H, OC$H_2$$CH_2$Br), 3.74 (t, *J* = 6.0 Hz, 2H, OCH_2CH_2Br) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 168.9, 143.5, 134.4, 133.6, 126.4, 123.5, 122.0, 70.5, 27.1 ppm. IR (solid state): ν = 2361, 1615, 1556,

1457, 1414, 1357, 1327, 1273, 1175, 1273, 1175, 1166, 957, 819, 788, 771, 752 cm^{-1}. MS (EI+): *m/z* Found $[M]^+$ 290.9405, $C_9H_8BrNO_3S$ requires 290.9418. Anal. Calc. for $C_9H_8BrNO_3S$: C, 37.3; H, 2.8; N, 4.8%. Found: C, 37.1; H, 2.7; N, 4.8%.

2-{2-[(1,1-dioxido-1,2-benzisothiazol-3-yl)oxy]ethyl}-1,2-benzisothiazol-3(2H)-one 1,1-dioxide (8a):

Prepared following the general procedure for the synthesis of **6**. Prepared from sodium saccharin dihydrate (1.44 g, 7.02 mmol) and 1,2-dibromoethane (0.3 mL, 3.51 mmol) in DMF (25 mL). Purification by recrystallisation from CH_2Cl_2 gave **8a** (0.77 g, 56 %). R_f 0.75 (CH_2Cl_2). Mp. > 260 ^oC (dec., CH_2Cl_2). ^1H (400 MHz, $CDCl_3$): δ = 8.10 (dd, *J* = 6.5, 1.5 Hz, 1H, Ar), 7.94-7.85 (m, 5H, Ar), 7.76 (dt, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.71 (dt, *J* = 7.5, 1.0 Hz, 1H, Ar), 4.91 (t, *J* = 6.2 Hz, 2H, C*H*$_2$O), 4.27 (t, *J* = 6.2 Hz, 2H; NC*H*$_2$) ppm. ^{13}C (67.8 MHz, $CDCl_3$): δ = 169.2, 158.9, 143.5, 137.7, 135.2, 134.6, 134.2, 133.6, 127.5, 127.4, 125.5, 124.0, 121.8, 121.2, 67.9, 38.0 ppm. IR (solid state): ν = 1739, 1616, 1556, 1462, 1403, 1322, 1258, 1179, 1159, 1122, 1058, 1040, 953, 930, 788, 755 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 393.0206, $C_{16}H_{13}N_2O_6S_2$ requires 393.0215. Anal. Calc. for $C_{16}H_{12}N_2O_6S_2$: C, 49.0; H, 3.1; N, 7.2 %. Found: C, 48.8; H, 3.0; N, 7.1 %.

1,3-propyl-bis-(1,2-benzisothiazol-3(2H)-one 1,1-dioxide) (8b):

Prepared following the general procedure for the synthesis of **6**. Prepared from sodium saccharin dihydrate (5.00 g, 24.4 mmol) and 1,3-dibromopropane (1.20 mL, 12.0 mmol) in DMF (10 ml). Purification by trituration of the crude in hot *i*PrOH gave **8b** (2.37 g, 50 %) as a white insoluble solid. R_f 0.35 Mp. > 185 ^oC (dec. EtOH; *lit.*[9] 196 ^oC, $CHCl_3$). ^1H (400 MHz, $CDCl_3$): δ = 8.06 (dd, *J* = 7.0, 1.0 Hz, 2H, Ar), 7.94-7.84 (m, 6H, Ar), 3.95 (t, *J* = 7.0 Hz, 4H, NC*H*$_2$), 2.43 (quintet, *J* = 7.0 Hz, 2H, CH_2C*H*$_2CH_2$) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 159.0, 137.7, 134.9, 134.5, 127,4, 125.4, 121.1, 36.8, 27.3 ppm. IR (solid state): ν = 1728, 1710, 1461, 1329, 1296, 1255, 1182, 1173, 1156, 1060, 955, 787, 750 cm^{-1}. MS (EI+): *m/z* Found $[M+H]^+$ 407.0372, $C_{17}H_{15}N_2O_6S_2$ requires 407.0377. Anal. Calc. for $C_{17}H_{14}N_2O_6S_2$: C, 50.2; H, 3.5; N 6.9%. Found: C, 50.2; H, 3.4; N, 7.0%.

Methyl 2-({[(1R)-1-phenylethyl]amino}sulfonyl)benzoate (11e):

To a stirred solution of methyl 2-(chlorosulfonyl)benzoate (1.00 g, 4.3 mmol) in dry CH_2Cl_2 (20 mL) under an inert atmosphere was added triethylamine (0.71 mL, 5.1 mmol). The reaction mixture was stirred at room temperature and (R)-α-methylbenzylamine (0.65 mL, 5.1 mmol) was added in one portion. The reaction mixture was stirred for 4 h at room temperature. The reaction was quenched by the addition of 2.0 M HCl (5 mL), the organic was extracted and washed with 2.0 M HCl (2 x 50 mL), brine (50 mL), and then dried (Na_2SO_4) and concentrated *in vacuo*. Purification

by flash chromatography (CH_2Cl_2) gave **11e** (1.05 g, 77%) as a colourless oil. R_f 0.42 (CH_2Cl_2). $[\alpha]_D$ = +33.1 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.74-7.65 (m, 2H, Ar), 7.47 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.37 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.12-7.05 (m, 5H, Ar), 6.45 (d, *J* = 6.5 Hz, 1H, SO_2NH), 4.58 (quintet, *J* = 6.5 Hz, 1H, C*H*Me), 4.00 (s, 3H, O*Me*), 1.44 (d, *J* = 6.5 Hz, 3H, CH*Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 168.7, 142.2, 140.7, 132.0, 131.7, 130.6, 130.2, 129.7, 128.6, 127.7, 126.5, 55.0, 53.7, 24.1 ppm. IR (solid state): υ = 1718, 1435, 1334, 1295, 1167, 1167, 1138, 1116, 1084, 1060, 952, 786, 757, 733, 700 cm^{-1}. MS (EI+): m/z Found $[M\text{-}Me]^+$ 304.0643, $C_{15}H_{14}NO_4S$ requires 304.0644.

Methyl 2-[(*tert*-butylamino)sulfonyl]benzoate (11h):

To a stirred solution of methyl 2-(chlorosulfonyl)benzoate (0.50 g, 2.1 mmol) in dry CH_2Cl_2 (10 mL) under an inert atmosphere was added triethylamine (0.31 mL, 2.2 mmol). The reaction mixture was cooled to 0 °C and *tert*-butylamine (0.22 mL, 2.1 mmol) was added in one portion. The reaction mixture was stirred at 0 °C for 1 h then was allowed to warm to room temperature for 3 h. The reaction was quenched by the addition of 2.0 M HCl (5 mL), the organic was extracted and washed with 2.0 M HCl (2 x 50 mL), brine (50 mL), and then dried (Na_2SO_4) and concentrated *in vacuo*. Purification by flash chromatography (CH_2Cl_2) gave **11h** (0.73 g, 79 %), as a white solid. R_f 0.5 (CH_2Cl_2). Mp. 80 – 81 ^oC (CH_2Cl_2). ^1H (400 MHz, $CDCl_3$): δ = 8.13 (dd, *J* = 7.8, 1.7 Hz, 1H, Ar), 7.82 (dd, *J* = 7.8, 1.7 Hz, 1H, Ar), 7.62 (dt, *J* = 7.8, 1.7 Hz, 1H, Ar), *overlapped by* 7.60 (dt, *J* = 7.8, 1.7 Hz, 1H, Ar), 6.08 (s, 1H, SO_2NH), 3.97 (s, 3H, O*Me*), 1.26 (s, 9H, ^{t}*Bu*) ppm. ^{13}C (67.8 MHz, $CDCl_3$): δ = 168.2, 143.0, 131.7, 130.6, 129.9, 128.9, 54.8, 53.3, 30.2 ppm. IR (solid state): ν = 1710, 1442, 1429, 1390, 1332, 1288, 1265, 1158, 1120, 1062, 980, 955, 750, 733, 714 cm^{-1}. MS (EI+): *m/z* Found $[M\text{-}Me]^+$ 256.0650, $C_{11}H_{14}NO_4S$ requires 256.0644. Anal. Calc. for $C_{12}H_{17}NO_4S$: C, 53.1; H, 6.3; N, 5.2 %. Found: C, 53.0; H, 6.3; N, 5.1 %.

Methyl 2-(anilinosulfonyl)benzoate (11i):

To a stirred solution of methyl 2-(chlorosulfonyl)benzoate (0.30 g, 1.3 mmol) in dry CH_2Cl_2 (10 mL) under an inert atmosphere was added triethylamine (0.21 mL, 1.5 mmol). The reaction mixture was cooled to 0 °C and aniline (0.14 mL, 1.5 mmol) was added in one portion. The reaction mixture was stirred at 0 °C for 1 h then was allowed to warm to room temperature for 3 h. The reaction was quenched by the addition of 2.0 M HCl (5 mL), the organic was extracted and washed with 2.0 M HCl (2 x 15 mL), brine (15 mL), and then dried (Na_2SO_4) and concentrated *in vacuo* to give an orange oil. Purification by flash chromatography (CH_2Cl_2) gave **11i** (0.23 g, 62 %) as a colourless oil. R_f 0.38 (CH_2Cl_2). ^1H (400 MHz, $CDCl_3$): δ = 8.01 (s, 1H, SO_2NH), 7.83 (d, *J* = 7.0 Hz, 1H, Ar), *overlapped by* 7.83 (d, *J* = 7.0 Hz, 1H, Ar), 7.57 (dt, *J* = 7.0, 1.0 Hz, 1H, Ar), 7.48 (dt, *J* = 7.0, 1.0

Hz, 1H, Ar), 7.27-7.06 (m, 5H, Ar), 4.03 (s, 3H, OMe) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 168.7, 138.4, 137.0, 133.0, 131.9, 131.0, 130.9, 130.8, 129.6, 126.2, 123.3, 53.9 ppm. IR (CH_2Cl_2 solution): ν = 3286, 1728, 1714, 1598, 1574, 1496, 1352, 1114, 1060, 1030, 956 cm^{-1}. MS (EI+): *m/z* Found $[M+Na]^+$ 314.0463, $C_{14}H_{13}NaNO_4S$ requires 314.0457.

General Procedure for the preparation of chloro sulfonamides (13):

To a stirred solution of **4** (1.30 mmol) in the stated solvent (5 mL), was added triphenylphosphine (1.30 mmol) and carbon tetrachloride (5 mL, 51.7 mmol). The reaction mixture was stirred at room temperature for the stated time, until complete conversion by TLC was noted. The reaction was concentrated *in vacuo*, to give a crude colourless oil. Purification by flash chromatography gave **13** as either a viscous colourless oil or a foamy white solid.

2-[(benzylamino)sulfonyl]-*N*-(2-chloroethyl)benzamide (13ab):

To a stirred solution of **4ab** (2.00 g, 6.0 mmol) in anhydrous CH_2Cl_2 (10 mL) under an inert atmosphere at 0 °C was added dropwise thionyl chloride (3.50 mL, 30.0 mmol), making sure the internal temperature did not rise above 5 °C. The reaction mixture was then allowed to warm to room temperature, and then was refluxed for 2 – 3 h. The reaction mixture was concentrated *in vacuo*, to give a crude viscous oil. Purification by flash chromatography gave **13ab** (1.74 g, 82 %) as a colourless viscous oil. R_f 0.17 (CH_2Cl_2). ^{1}H (400 MHz, CD_3OD): δ = 7.90 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.60-7.56 (m, 1H, Ar), 7.53-7.45 (m, 2H, Ar), 7.24-7.16 (m, 5H, Ar), 6.47 (br, 1H, CON*H*), 6.41 (t, *J* = 6.4 Hz, 1H, SO_2NH), 4.14 (d, *J* = 6.4 Hz, 2H, CH_2Ph), 3.81-3.73 (m, 4H, CH_2CH_2Cl) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.9, 138.3, 136.3, 134.9, 132.6, 130.5, 129.5, 128.6, 128.1, 128.0, 127.7, 47.8, 43.4, 42.0 ppm. MS (EI+): *m/z* Found $[M-HCl]^+$ 316.0885, $C_{16}H_{16}N_2O_3S$ requires 316.0882.

2-[(benzhydrylamino)sulfonyl]-*N*-(2-chloroethyl)benzamide (13ad):

Prepared from **4ad** (0.70 g, 1.7 mmol), triphenylphosphine (0.50 g, 1.8 mmol) and carbon tetrachloride (5 mL, 51.7 mmol) in 50% CH_2Cl_2/MeCN (5 mL), stirring for 78 h. Purification by flash chromatography gave **13ad** (0.40 g, 54 %), as a white foamy solid after prolonged drying under high vacuum. R_f 0.20 (CH_2Cl_2). Mp. 68 – 70 °C. ^{1}H (500 MHz, $CDCl_3$): δ = 7.49 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.42-7.37 (m, 2H, Ar), 7.21-7.10 (m, 11H, Ar), 7.04 (d, *J* = 9.5 Hz, 1H, SO_2NH), 6.37 (br, 1H, CON*H*), 5.70 (d, *J* = 9.5 Hz, 1H, C*H*Ph$_2$), 3.79-3.74 (m, 4H, CH_2CH_2Cl) ppm. ^{13}C (125.8 MHz, $CDCl_3$): δ = 170.0, 140.1, 139.4, 134.0, 132.0, 130.2, 128.8, 128.4, 127.7, 127.5, 127.4, 62.4, 43.5, 42.0. IR (CH_2Cl_2 solution): ν = 3427, 3236, 1666, 1592, 1573, 1530, 1349, 1174, 1131, 1045, 1027 cm^{-1}. MS (EI+): m/z Found $[M+Na]^+$ 451.0859, $C_{22}H_{21}ClNaN_2O_3S$ requires

451.0854. Anal. Calc. for $C_{22}H_{21}ClN_2O_3S$: C, 61.6; H, 4.9; N, 6.5 %. Found: C, 61.4; H, 5.0; N, 6.5 %.

***N*-[(1S)-2-chloro-1-methylethyl]-2-[(benzylamino)sulfonyl]benzamide (13bb):**

Prepared from **4bb** (5.80 g, 16.7 mmol), triphenylphosphine (4.40 g, 16.9 mmol) and carbon tetrachloride (8 mL, 83.3 mmol) in MeCN (50 mL), stirring for 2 h. Purification by flash chromatography gave **13bb** (4.60 g, 75 %) as a white solid after prolonged drying under high vacuum. R_f 0.30 (CH_2Cl_2). Mp. 65 – 66 ^oC (EtOH). $[\alpha]_D$ = +1.4 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.85 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.56-7.47 (m, 3H, Ar), 7.22-7.20 (m, 5H, Ar), 6.44 (t, *J* = 6.4 Hz, 1H, SO_2NH), 6.27 (d, *J* = 8.1 Hz, 1H, CONH), 4.48 (dddd, *J* = 8.1, 6.7, 4.5, 3.8 Hz, 1H, NCH), 4.11 (dd, *J* = 14.0, 6.6 Hz, 1H, $CH_{2\alpha}$Ph), 4.09 (dd, *J* = 14.0, 6.3 Hz, 1H, $CH_{2\beta}$Ph), 3.80 (dd, *J* = 11.2, 4.5 Hz, 1H, $CH_{2\alpha}$Cl), 3.65 (dd, *J* = 11.2, 3.8 Hz, 1H, $CH_{2\beta}$Cl), 1.36 (d, *J* = 6.7 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.1, 138.2, 136.3, 135.0, 132.6, 130.3, 129.4, 128.5, 128.2, 128.0, 127.7, 48.7, 47.8, 46.5, 17.6 ppm. IR (solid state): ν = 1647, 1534, 1455, 1437, 1329, 1294, 1264, 1164, 1128, 1082, 1063, 1028, 785, 738, 697 cm^{-1}. Anal. Calc. for $C_{17}H_{19}ClN_2O_3S$: C, 55.7; H, 5.2; N, 7.6 %. Found: C: 55.4; H, 5.1; N, 7.7 %.

2-[(benzylamino)sulfonyl]-*N*-[(1S)-1-(chloromethyl)-2-methylpropyl]benzamide (13cb):

Prepared from **4cb** (0.50 g, 1.30 mmol), triphenylphosphine (0.35 g, 1.30 mmol) and carbon tetrachloride (5 mL, 51.7 mmol) in CH_2Cl_2 (5 mL). Purification by flash chromatography gave **13cb** (0.42 g, 81 %) as a white solid. R_f 0.30 (CH_2Cl_2). Mp. 128 – 129 ^oC (EtOH). $[\alpha]_D$ = -11.2 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.89 (d, *J* = 7.2 Hz, 1H, Ar), 7.59-7.47 (m, 3H, Ar), 7.22-7.16 (m, 5H, Ar), 6.46 (t, *J* = 6.4 Hz, 1H, SO_2NH), 6.22 (d, *J* = 8.8 Hz, 1H, CONH), 4.12 (d, *J* = 6.4 Hz, 2H, CH_2Ph), *overlapped by* 4.13-4.07 (m, 1H, NCH), 3.83 (dd, *J* = 11.5, 3.7 Hz, 1H, $CH_{2\alpha}$Cl), *overlapped by* 3.80 (dd, *J* = 11.5, 4.0 Hz, 1H, $CH_{2\beta}$Cl), 2.08-1.99 (m, 1H, C*H*Me_2), 1.06 (d, *J* = 9.6 Hz, 3H, *Me*), 1.04 (d, *J* = 6.7 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.5, 138.4, 136.4, 135.3, 132.6, 130.4, 129.6, 128.5, 128.1, 127.9, 127.7, 56.0, 47.8, 46.4, 29.4, 19.4, 19.0 ppm. IR (solid state): ν = 1620, 1591, 1554, 1456, 1437, 1437, 1338, 1168, 1069, 754, 734, 700 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 395.1210, $C_{19}H_{24}ClN_2O_3S$ requires 395.1196. Anal. Calc. for $C_{19}H_{23}ClN_2O_3S \cdot \frac{1}{2}H_2O$: C, 56.5; H, 6.0; N, 6.9 %. Found: C, 56.8; H, 6.1; N, 6.8 %.

***N*-[(1S)-1-(chloromethyl)-2-methylpropyl]-2-[(isopropylamino)sulfonyl]benzamide (13cc):**

Prepared from **4cc** (1.29 g, 3.9 mmol), triphenylphosphine (1.05 g, 4.0 mmol) and carbon tetrachloride (6 mL, 62.0 mmol) in MeCN (6 mL), stirring for 48 h. Purification by flash

chromatography gave **13cc** (0.45 g, 60 %) as a white solid. R_f 0.16 (CH_2Cl_2). Mp. > 198 °C (dec. MeOH). $[\alpha]_D$ = -55.2 [c 0.5, $CHCl_3$]. ^1H (400 MHz, $CDCl_3$): δ = 8.05 (*apparent* dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.62-7.56 (m, 3H, Ar), 6.16 (d, *J* = 9.0 Hz, 1H, CON*H*), 5.88 (d, *J* = 6.7 Hz, 1H, SO_2N*H*), 4.17-4.11 (m, 1H, NC*H*), 3.86 (dd, *J* = 11.5, 3.7 Hz, 1H, $CH_{2\alpha}Cl$), 3.82 (dd, *J* = 11.5, 4.0 Hz, 1H, $CH_{2\beta}Cl$), 3.48 (octet, *J* = 6.7 Hz, 1H, NHC*H*Me_2), 2.06 (octet, *J* = 6.7 Hz, 1H, C*H*Me_2), 1.09-1.05 (m, 12H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.5, 139.5, 135.2, 132.4, 130.5, 129.1, 128.3, 56.0, 46.8, 46.4, 29.3, 23.6, 19.4, 19.0 ppm. IR (solid state): ν = 2966, 1650, 1592, 1533, 1466, 1426, 1388, 1370, 1332, 1264, 1172, 1122, 1070, 990, 889, 785, 761, 736, 680 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 349.1178, $C_{15}H_{24}{}^{37}ClN_2O_3S$ requires 349.1167. Anal. Calc. for $C_{15}H_{23}ClN_2O_3S$: C, 51.9; H, 6.7; N, 8.1 %. Found: C, 52.1; H, 6.7; N, 7.9 %.

***N*-[(1S)-1-(chloromethyl)-2-methylpropyl]-2[(benzhydrylamino)sulfonyl]benzamide (13cd):**

Prepared from **4cd** (1.98 g, 4.38 mmol), triphenylphosphine (1.38 g, 5.25 mmol) and carbon tetrachloride (5 mL, 51.7 mmol) in 50 % MeCN/CH_2Cl_2 (5 mL), stirring for 78 h. Purification by flash chromatography gave **13cd** (1.47 g, 65 %) as a viscous oil. R_f 0.32 (CH_2Cl_2). $[\alpha]_D$ = -10.4 [c 1.0, EtOH]. ^1H (400 MHz, $CDCl_3$): δ = 7.47 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar), 7.42-7.37 (m, 3H, Ar), 7.25-7.09 (m, 10H, Ar), 7.06 (d, *J* = 9.3 Hz, 1H, SO_2N*H*), 6.05 (d, *J* = 9.1 Hz, 1H, CON*H*), 5.70 (d, *J* = 9.3 Hz, 1H, C*H*Ph_2), 4.14-4.09 (m, 1H, NC*H*), 3.85 (dd, *J* = 12.0, 3.7 Hz, 1H, $CH_{2\alpha}Cl$), 3.79 (dd, *J* = 12.0, 3.8 Hz, 1H, $CH_{2\beta}Cl$), 2.06-2.00 (m, 1H, C*H*Me_2), 1.06 (d, *J* = 6.8 Hz, 3H, *Me*), 1.04 (d, *J* = 6.7 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.7, 140.1, 139.5, 134.3, 131.9, 130.1, 128.7, 128.3, 127.7, 127.4, 126.6, 62.4, 55.8, 46.4, 29.2, 19.3, 19.1 ppm. IR (solid state): ν = 2968, 1652, 1522, 1496, 1456, 1424, 1329, 1165, 1129, 1047, 1026, 834, 744, 698 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 471.1508, $C_{25}H_{28}ClN_2O_3S$ requires 471.1509. Anal. Calc. for $C_{25}H_{27}ClN_2O_3S$: C, 63.8; H, 5.8; N, 6.0 %. Found: C, 64.2; H, 5.9; N, 5.9 %.

***N*-[(1S)-1-(chloromethyl)-2-methylpropyl]-2-({[(1R)-1-phenylethyl]amino}sulfonyl)benzamide (13ce):**

Prepared from **4ce** (0.35 g, 0.91 mmol), triphenylphosphine (0.24 g, 0.92 mmol) and carbon tetrachloride (6 mL, 62.0 mmol) in MeCN (6 mL), stirring for 48 h. Purification by flash chromatography gave **13ce** (0.32 g, 71 %) as a viscous oil. R_f 0.35 (CH_2Cl_2). $[\alpha]_D$ = -11.5 [c 0.7, $CHCl_3$]. ^1H (400 MHz, CD_3OD): δ = 7.54 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.46-7.43 (m, 2H, Ar), 7.28 (dt, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.16- 7.14 (m, 2H, Ar), 7.09-7.06 (m, 3H, Ar), 6.54 (d, *J* = 8.3 Hz, 1H, SO_2N*H*), 6.13 (d, *J* = 9.0 Hz, 1H, CON*H*), 4.57 (dq, *J* = 9.0, 7.1 Hz, 1H, C*H*(Me)Ph), 4.18-4.12 (m, 1H, NC*H*), 3.90 (dd, *J* = 11.5, 3.7 Hz, 1H, $CH_{2\alpha}Cl$), 3.83 (dd, *J* = 11.5, 3.8 Hz, 1H, $CH_{2\beta}Cl$), 2.10

(octet, J = 6.9 Hz, 1H, C*H*Me$_2$), 1.44 (d, J = 7.1 Hz, 3H, CH*Me*), 1.10 (d, J = 6.9 Hz, 3H, *Me*), 1.08 (d, J = 6.9 Hz, 3H, *Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.8, 141.9, 139.4, 134.5, 131.9, 130.1, 128.8, 128.2, 127.4, 127.2, 126.3, 55.9, 54.8, 46.2, 29.1, 23.9, 19.4, 19.0 ppm. IR (solid state): ν = 1643, 1551, 1437, 1333, 1266, 1171, 1118, 1070, 721, 695 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 411.1307, $C_{20}H_{26}{}^{37}ClN_2O_3S$ requires 411.1323.

_N_-[(1S)-1-(chloromethyl)-2-methylpropyl]-2-[(propylamino)sulfonyl]benzamide(13cg):

Prepared from **4cg** (0.97 g, 2.96 mmol), triphenylphosphine (0.85 g, 3.25 mmol) and carbon tetrachloride (3 mL, 31.0 mmol) in MeCN (5 mL), stirring for 24 h. Purification by flash chromatography gave **13cg** (0.67 g, 67 %), as a viscous oil which solidified upon prolonged drying to a white solid. R_f 0.15 (CH_2Cl_2). Mp. 122 – 124 ^{o}C. $[\alpha]_D$ = -11.3 [c 1.2, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 8.01 (dd, J = 8.0, 1.0 Hz, 1H, Ar), 7.62-7.50 (m, 3H, Ar), 6.14 (d, J = 8.9 Hz, 1H, CON*H*), 5.95 (t, J = 6.9 Hz, 1H, SO_2N*H*), 4.15-4.11 (m, 1H, NC*H*), 3.90 (dd, J = 11.5, 3.8 Hz, 1H, C$H_{2\alpha}$Cl), 3.86 (dd, J = 11.5, 4.1 Hz, 1H, C$H_{2\beta}$Cl), 2.95 (*apparent* q, J = 6.9 Hz, 2H, NHCH_2Et), 2.06 (octet, J = 6.9 Hz, 1H, C*H*Me$_2$), 1.57-1.42 (m, 2H, CH$_2$CH_2Me), 1.11 (d, J = 6.9 Hz, 3H, CH*Me*), 1.09 (d, J = 6.9 Hz, 3H, CH*Me*), 0.87 (t, J = 6.7 Hz, 3H, CH$_2$*Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.5, 138.4, 135.5, 132.6, 130.4, 129.6, 128.2, 56.0, 46.4, 45.6, 29.4, 22.9, 19.4, 19.0, 11.3 ppm. IR (CH_2Cl_2 solution): ν = 3414, 3256, 2876, 1660, 1595, 1573, 1524, 1342, 1175, 1126, 1069, MS (EI+): *m/z* Found $[M+H]^+$ 347.1196, $C_{15}H_{24}{}^{37}ClN_2O_3S$ requires 347.1191.

_N_-[(1S)-1-(chloromethyl)-2-methylpropyl]-2-[(*tert*-butylamino)sulfonyl]benzamide(13ch):

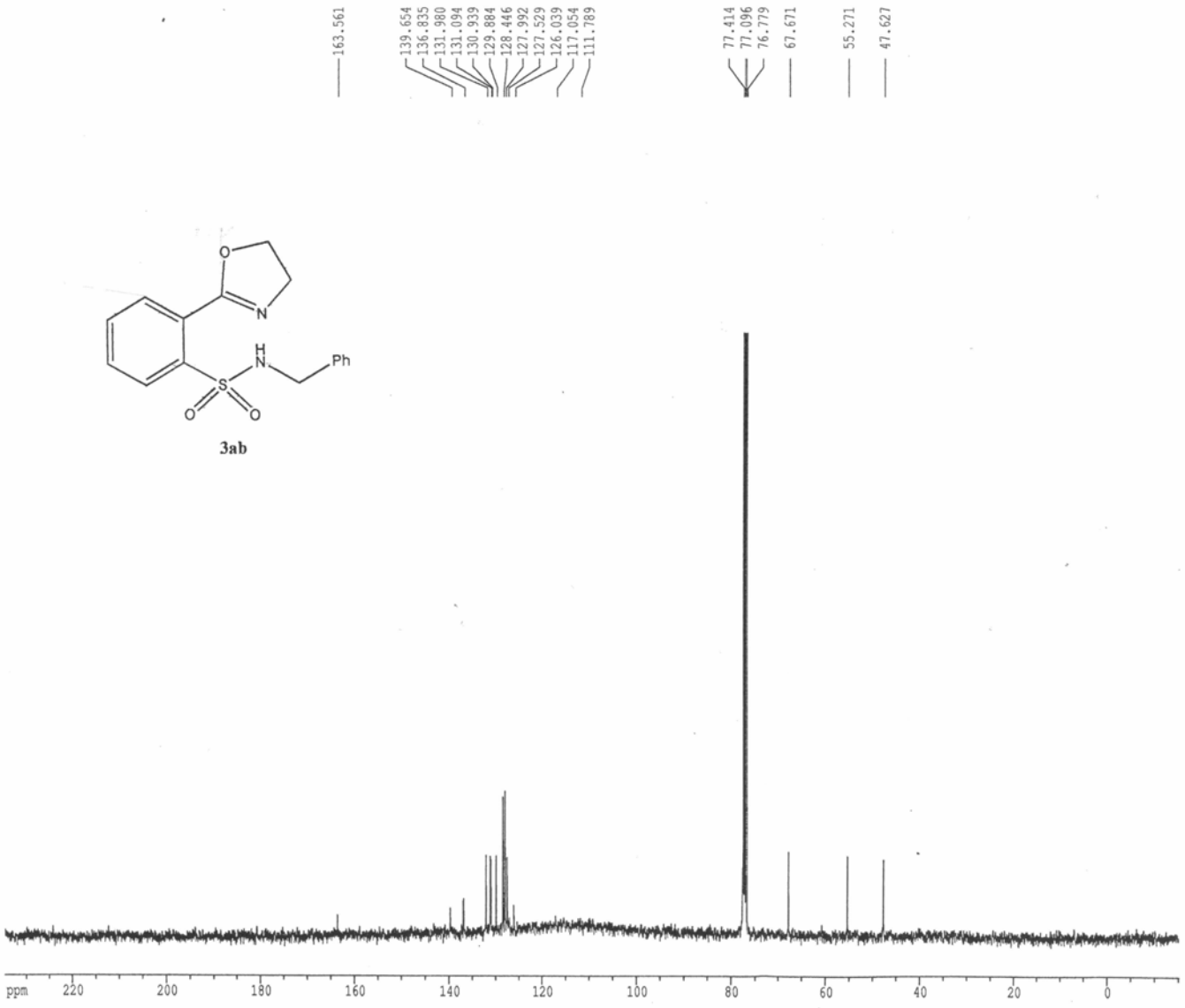
Prepared from **4ch** (0.14 g, 0.4 mmol), triphenylphosphine (0.11 g, 0.4 mmol) and carbon tetrachloride (3 mL, 31.0 mmol) in MeCN (5 mL), stirring for 24 h. Purification by flash chromatography gave **13ch** (0.09 g, 61 %) as a white solid. R_f 0.22 (CH_2Cl_2). $[\alpha]_D$ = -3.8 [c 1.2, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 8.05 (dd, J = 8.0, 1.0 Hz, 1H, Ar), 7.59-7.54 (m, 3H, Ar), 6.21 (d, J = 9.1 Hz, 1H, CON*H*), 6.10 (s, 1H, SO_2N*H*), 4.17-4.09 (*m*, 1H, NC*H*), 3.86 (dd, J = 11.5, 3.7 Hz, 1H, C$H_{2\alpha}$Cl), 3.80 (dd, J = 11.5, 4.1 Hz, 1H, C$H_{2\beta}$Cl), 2.04 (octet, J = 6.7, 1H, C*H*Me$_2$), 1.25 (s, 9H, ^{t}*Bu*), 1.07 (d, J = 6.7 Hz, 3H, CH*Me*), 1.05 (d, J = 6.7, 3H, CH*Me*) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.7, 142.0, 135.0, 132.1, 130.5, 128.6, 128.2, 56.0, 55.0, 46.4, 30.2, 29.3, 19.3, 19.0 ppm. IR (solid state): ν = 3365, 1645, 1336, 1472, 1443, 1392, 1364, 1313, 1198, 1154, 1128, 1071, 985, 790, 764, 731, 681 cm^{-1}. MS (CI+): *m/z* Found $[M+H]^+$ 361.1300, $C_{16}H_{26}ClN_2O_3S$ requires 361.1292.

***N*-[(1S)-1-benzyl-2-chloroethyl]- 2-[(benzylamino)sulfonyl]benzamide (13db):**

Prepared from **4db** (2.47 g, 5.8 mmol), triphenylphosphine (1.57 g, 6.0 mmol) and carbon tetrachloride (3 mL, 31.0 mmol) in MeCN (20 mL), stirring for 12 h. Purification by flash chromatography gave **13db** (1.83 g, 71 %) as a foamy white solid. R_f 0.23 (CH_2Cl_2). Mp. 48-50 ºC. $[\alpha]_D$ = -14.9 [c 1.0, EtOH]. ^{1}H (400 MHz, $CDCl_3$): δ = 7.90 (dd, *J* = 8.0, 1.0, 1H, Ar), 7.57-7.47 (m, 2H, Ar), 7.38 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar), 7.33-7.20 (m, 11H, Ar), 6.38 (t, *J* = 6.4 Hz, 1H, SO_2NH), 6.24 (d, *J* = 8.6 Hz, 1H, CO*NH*), 4.62 (m, 1H, NC*H*), 4.14 (dd, *J* = 14.0, 6.4 Hz, 1H, $NCH_{2\alpha}Ph$), *overlapped by* 4.13 (dd, *J* = 14.0, 6.4 Hz, 1H, $NCH_{2\beta}Ph$), 3.76 (dd, *J* = 11.3, 4.4 Hz, 1H, $CH_{2\alpha}Cl$), 3.60 (dd, *J* = 11.3, 3.5 Hz, 1H, $CH_{2\beta}Cl$), 3.08 (dd, *J* = 13.7, 6.3, 1H, $CH_{2\alpha}Ph$), 3.03 (dd, *J* = 13.7, 8.3 Hz, 1H, $CH_{2\beta}Ph$) ppm. ^{13}C (100 MHz, $CDCl_3$): δ = 169.2, 138.3, 136.6, 136.3, 135.0, 132.6, 130.4, 129.5, 129.4, 128.9, 128.6, 128.0, 128.0, 127.7, 127.2, 51.5, 47.8, 46.1, 37.1 ppm. IR (solid state): ν = 1649, 1526, 1497, 1455, 1438, 1331, 1164, 1129, 1064, 1029, 741, 698 cm^{-1}. MS (FAB+): *m/z* Found $[M+H]^+$ 443.1160, $C_{23}H_{24}ClN_2O_3S$ requires 443.1196. Anal. Calc. for $C_{23}H_{23}ClN_2O_3S$: C, 62.4; H, 5.2; N, 6.3 %. Found: C, 62.1; H, 5.2; N, 6.2 %.

CCDC - 604476-604480 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

RIR 309



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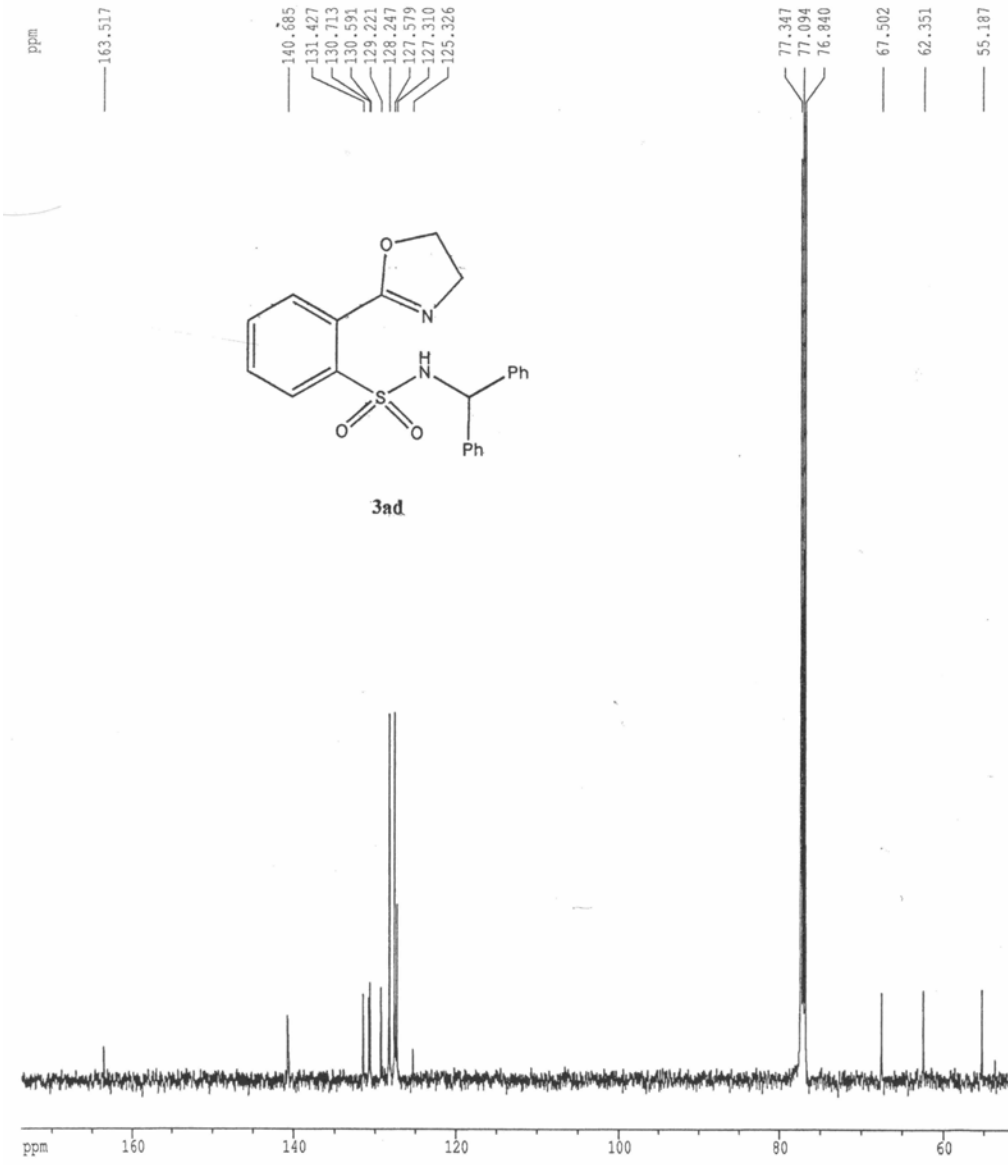
RIR 378



3ad

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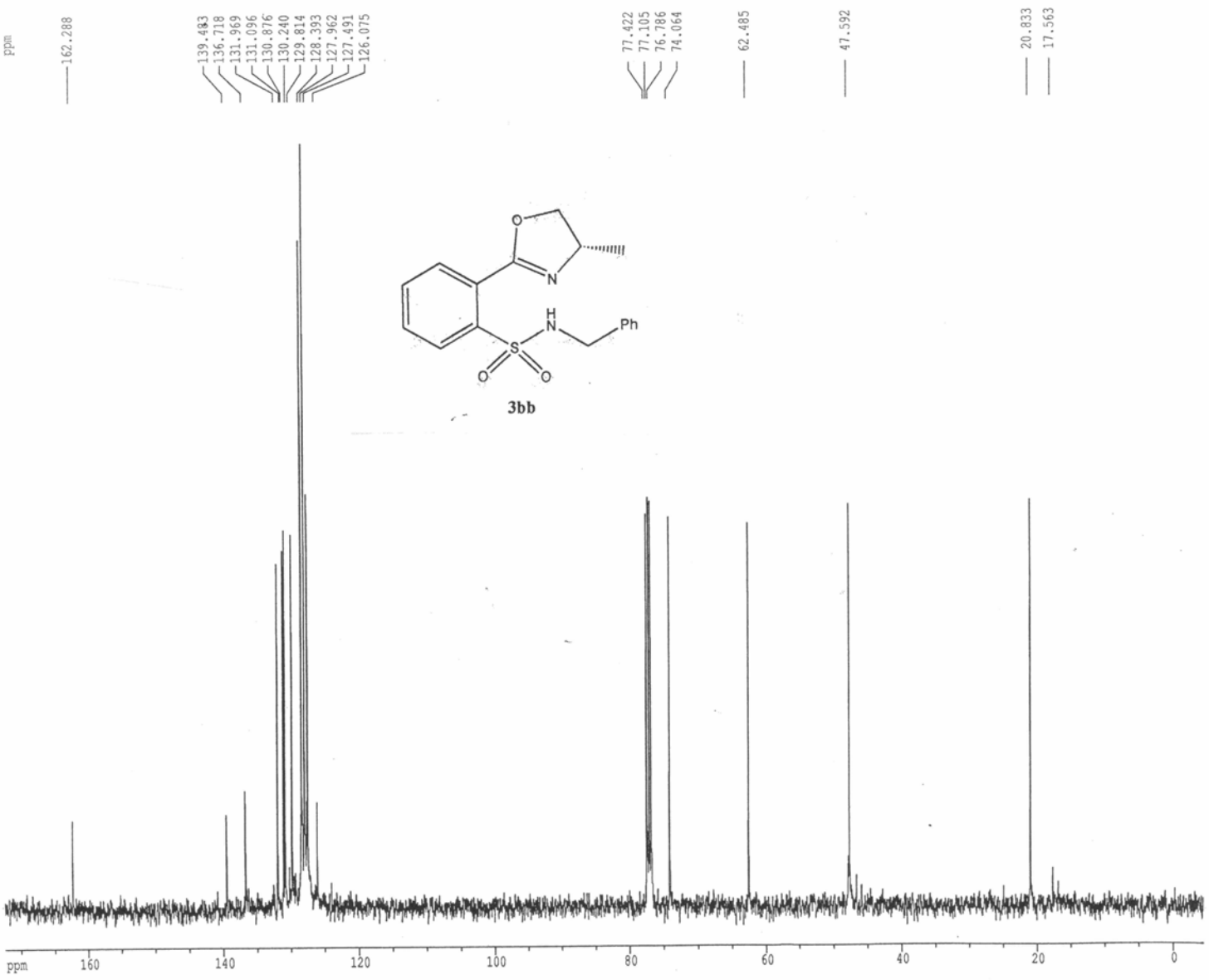
RIR 384



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NUC2	1H
PCPD2	90.00 usec
PL2	4.00 dB
PL12	25.58 dB
PL13	29.00 dB
SFO2	400.1316005 MHz

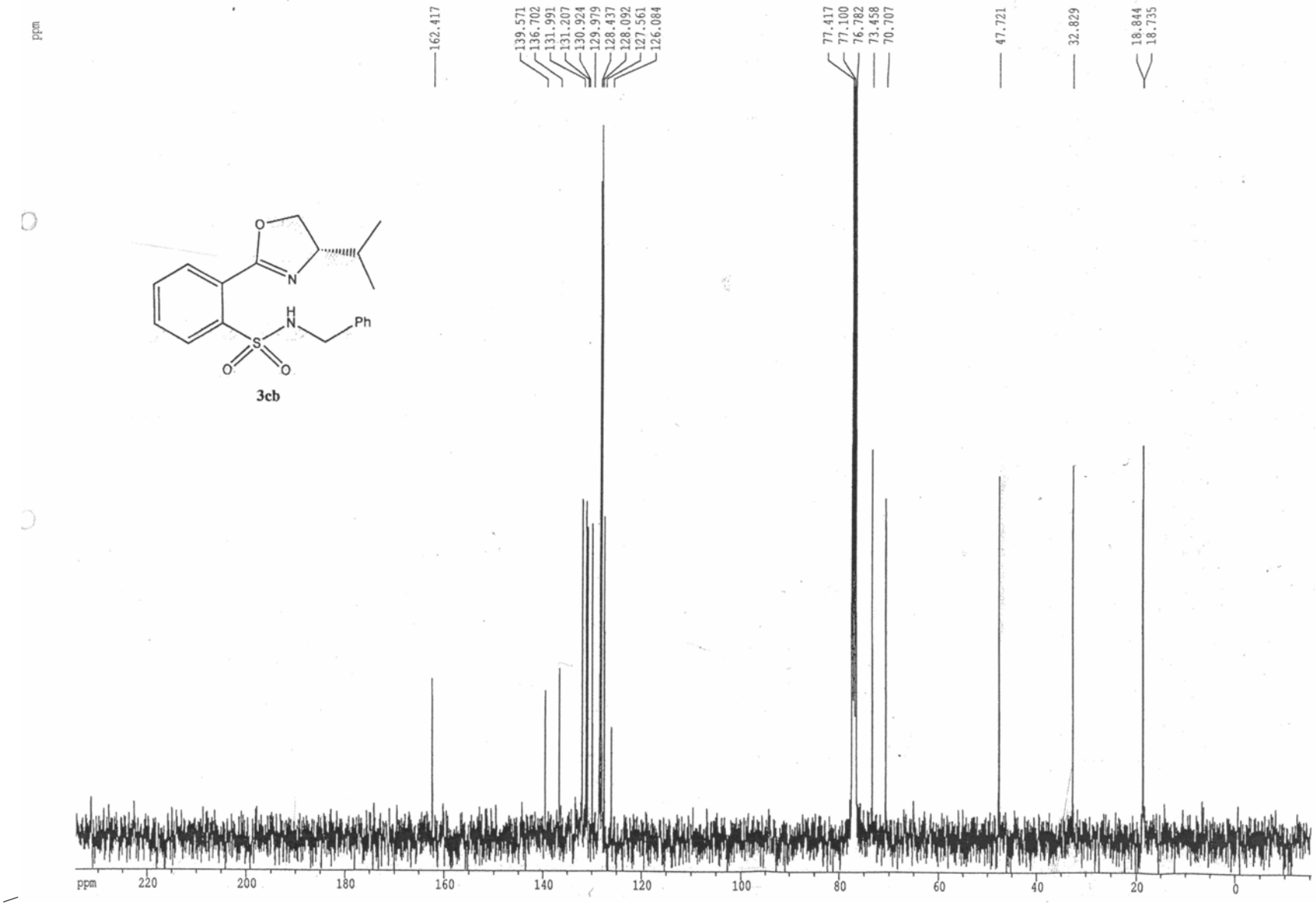
F2 - Processing parameters

SI	32768
SF	100.6127684 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

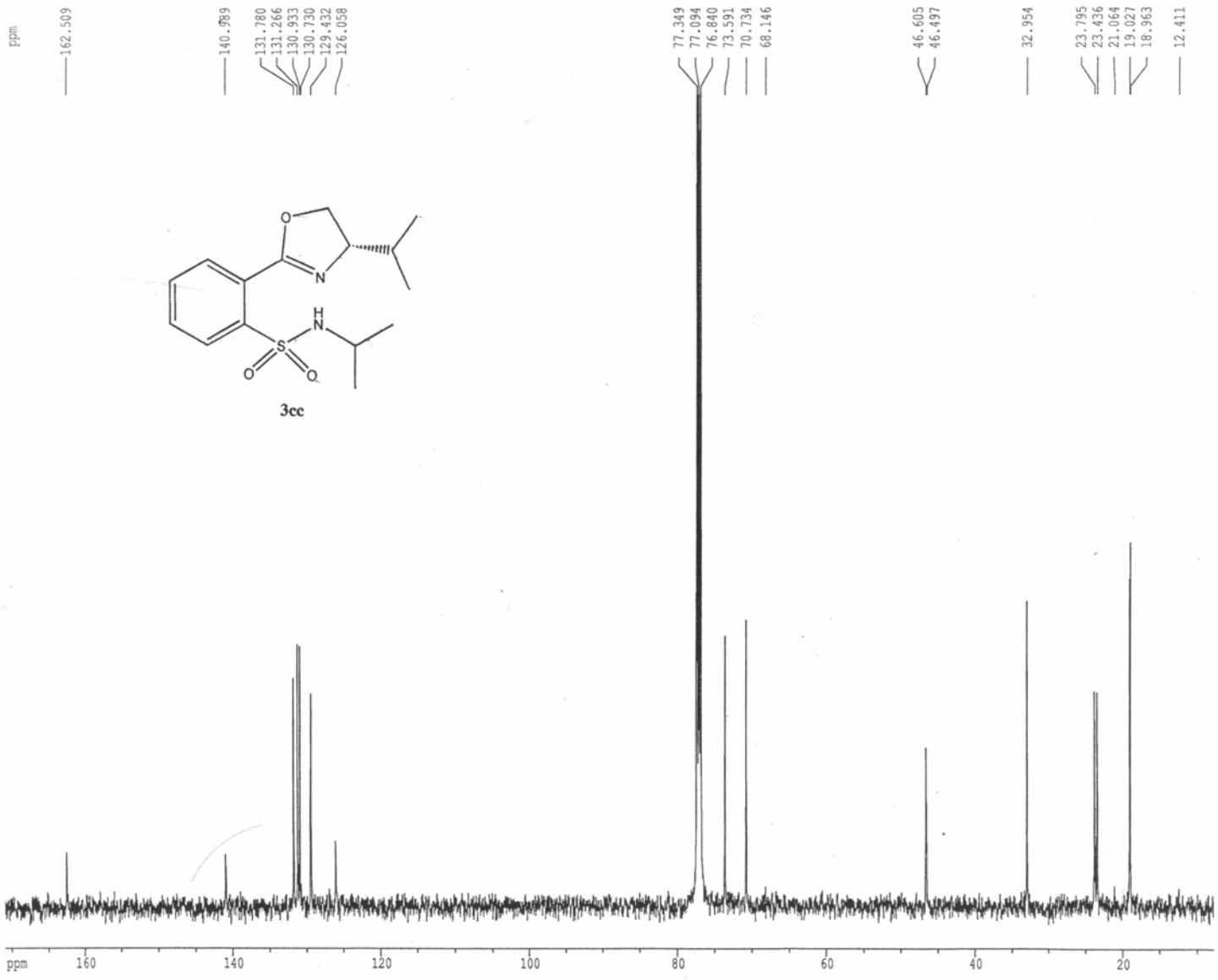
CX	30.00 cm
CY	19.00 cm
F1P	172.429 ppm
F1	17348.54 Hz
F2P	-4.552 ppm
F2	-458.02 Hz
PPMCM	5.89937 ppm/cm
HZCM	593.55200 Hz/cm

RIR 324


ppm
162.417
139.571
136.702
131.991
131.207
130.924
129.979
128.437
128.092
127.561
126.084
77.417
77.100
76.782
73.458
70.707
47.721
32.829
18.844
18.735
O
N
H
N
Ph
S
O
O
ppm
220
200
180
160
140
120
100
80
60
40
20
0

3cb

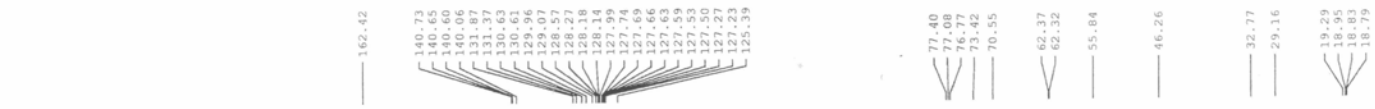
RIR 596

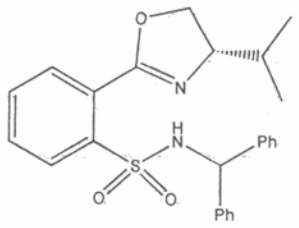


3cc

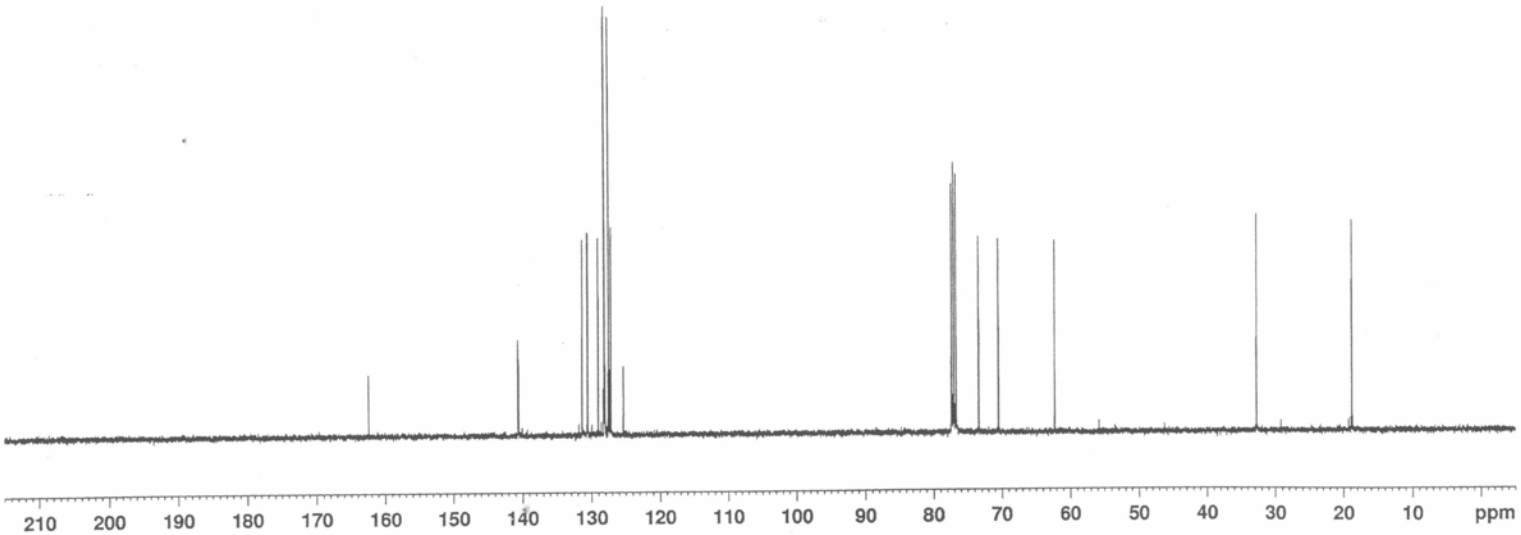
```
Current Data Parameters
NAME                 RIR596
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20021116
Time                 14.48
INSTRUM              DRX500
PROBHD    5 mm Multinu
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                     1728
DS                        2
SWH              35211.270 Hz
FIDRES            1.074563 Hz
AQ                0.4653556 sec
RG                   7298.2
DW                  14.200 usec
DE                   5.50 usec
TE                  300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

========== CHANNEL f1 ==========
NUC1                    13C
P1                    9.50 usec
PL1                   5.00 dB
SFO1           125.7715724 MHz

========== CHANNEL f2 ==========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               115.00 usec
PL2                  -3.00 dB
PL12                 16.00 dB
PL13                 16.00 dB
SFO2           500.1327507 MHz

F2 - Processing parameters
SI                     16384
SF             125.7577845 MHz
WDW                       EM
SSB                        0
LB                    3.00 Hz
GB                         0
PC                     1.00

1D NMR plot parameters
CX                    30.00 cm
F1P                  170.909 ppm
F1                 21493.12 Hz
F2P                    7.758 ppm
F2                   975.62 Hz
PPMCM                5.43837 ppm/cm
HZCM               683.91693 Hz/cm
```

UserID r_fry SampleID rf214 SupervisorID swood Lab Phone No. 13538 Slot Number 51



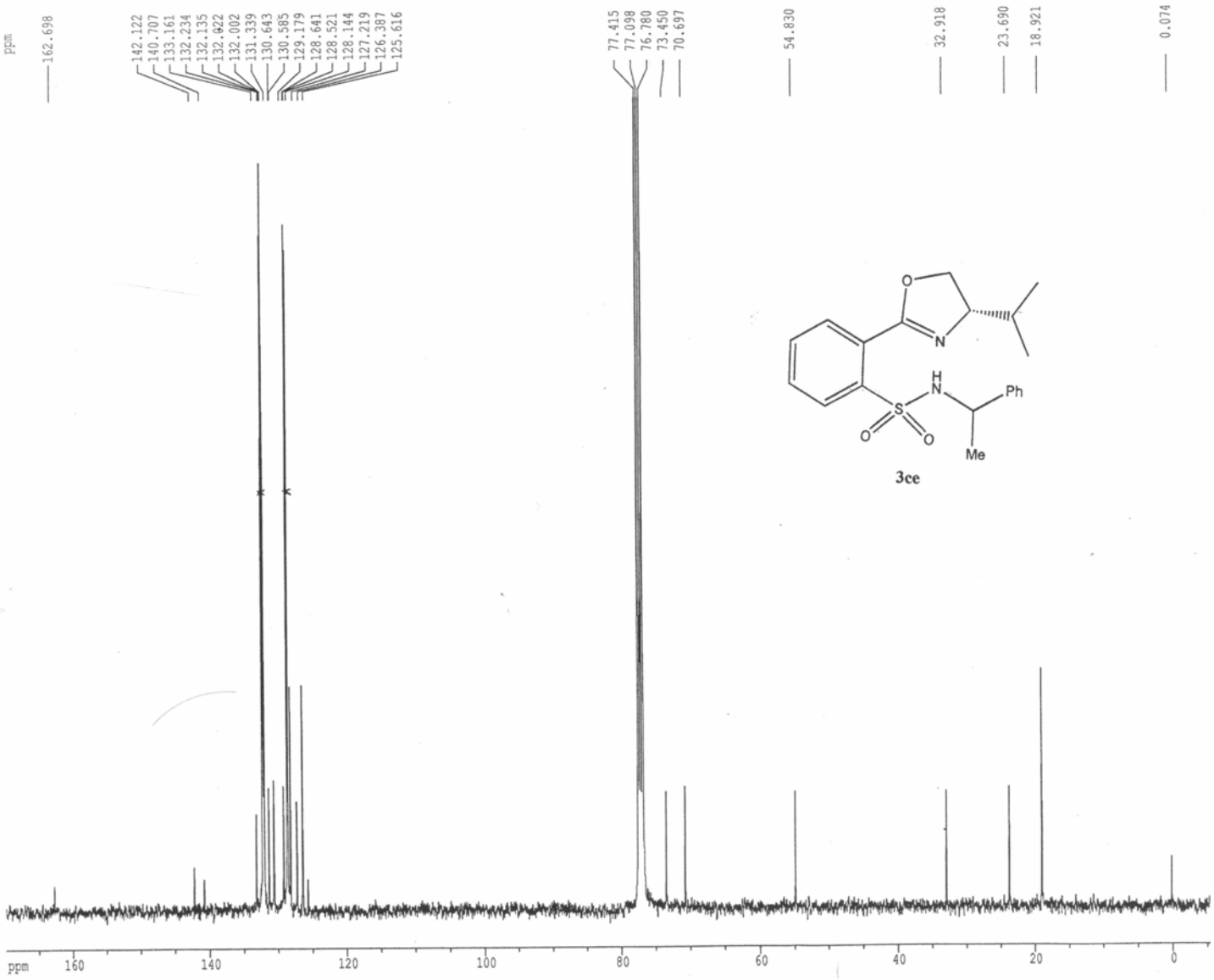


3cd




```
Current Data Parameters
NAME                  r_fry.rf214
EXPNO                           7
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20060316
Time                        14.41
INSTRUM                      av400
PROBHD    5 mm BBO BB-1H
PULPROG                   zgpg30
TD                           32768
SOLVENT                      CDCl3
NS                             512
DS                               2
SWH                    25125.629 Hz
FIDRES                   0.766773 Hz
AQ                     0.6521332 sec
RG                        5792.6
DW                         19.900 use
DE                          6.00 use
TE                         298.2 K
D1                    1.00000000 sec
d11                   0.03000000 sec
DELTA                 0.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 =======
NUC1                         13C
P1                          7.55 use
PL1                         6.00 dB
SFO1                100.6238364 MHz

======== CHANNEL f2 =======
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                      87.00 use
PL2                         0.00 dB
PL12                       21.00 dB
PL13                       26.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                           32768
SF                  100.6127690 MHz
WDW                             EM
SSB                              0
LB                          1.00 Hz
GB                               0
PC                          1.40
```

RIR 597



3ce

Current Data Parameters

NAME	rir597
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20021116
Time	1.44
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	10000
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	16384
DW	19.900 usec
DE	20.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	13.00 usec
PL1	-0.10 dB
SFO1	100.6237959 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.00 dB
PL12	25.58 dB
PL13	29.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

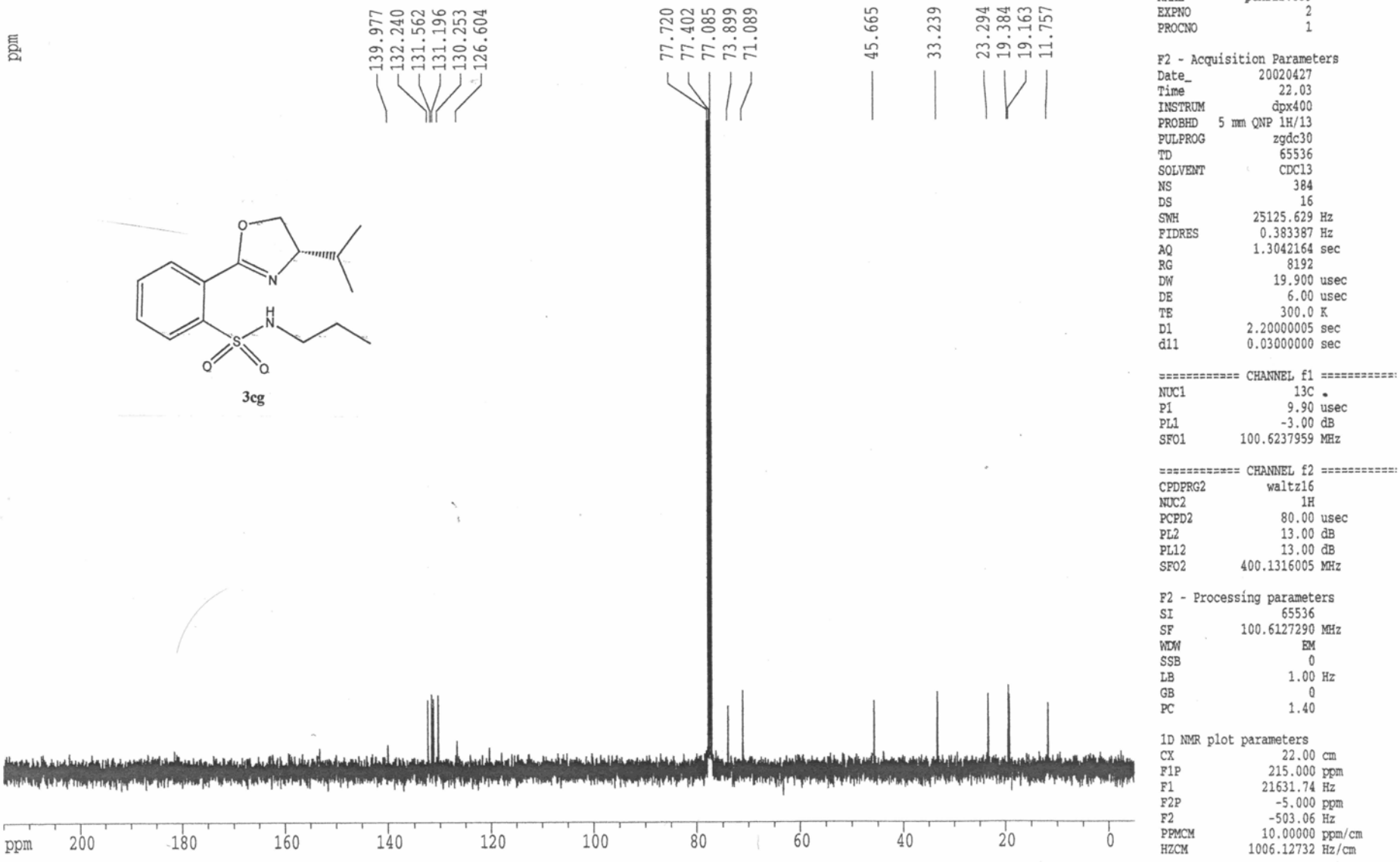
SI	32768
SF	100.6127615 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	19.00 cm
F1P	169.803 ppm
F1	17084.34 Hz
F2P	-5.374 ppm
F2	-540.64 Hz
PPMCM	5.83921 ppm/cm
HZCM	587.49933 Hz/cm

User/Group Robinson/Pfaltz

RIR 446



3cg

Current Data Parameters

NAME	pcxrir.039
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020427
Time	22.03
INSTRUM	dpx400
PROBHD	5 mm QNP 1H/13
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	384
DS	16
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	8192
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.20000005 sec
d11	0.03000000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.90 usec
PL1	-3.00 dB
SFO1	100.6237959 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	13.00 dB
PL12	13.00 dB
SFO2	400.1316005 MHz

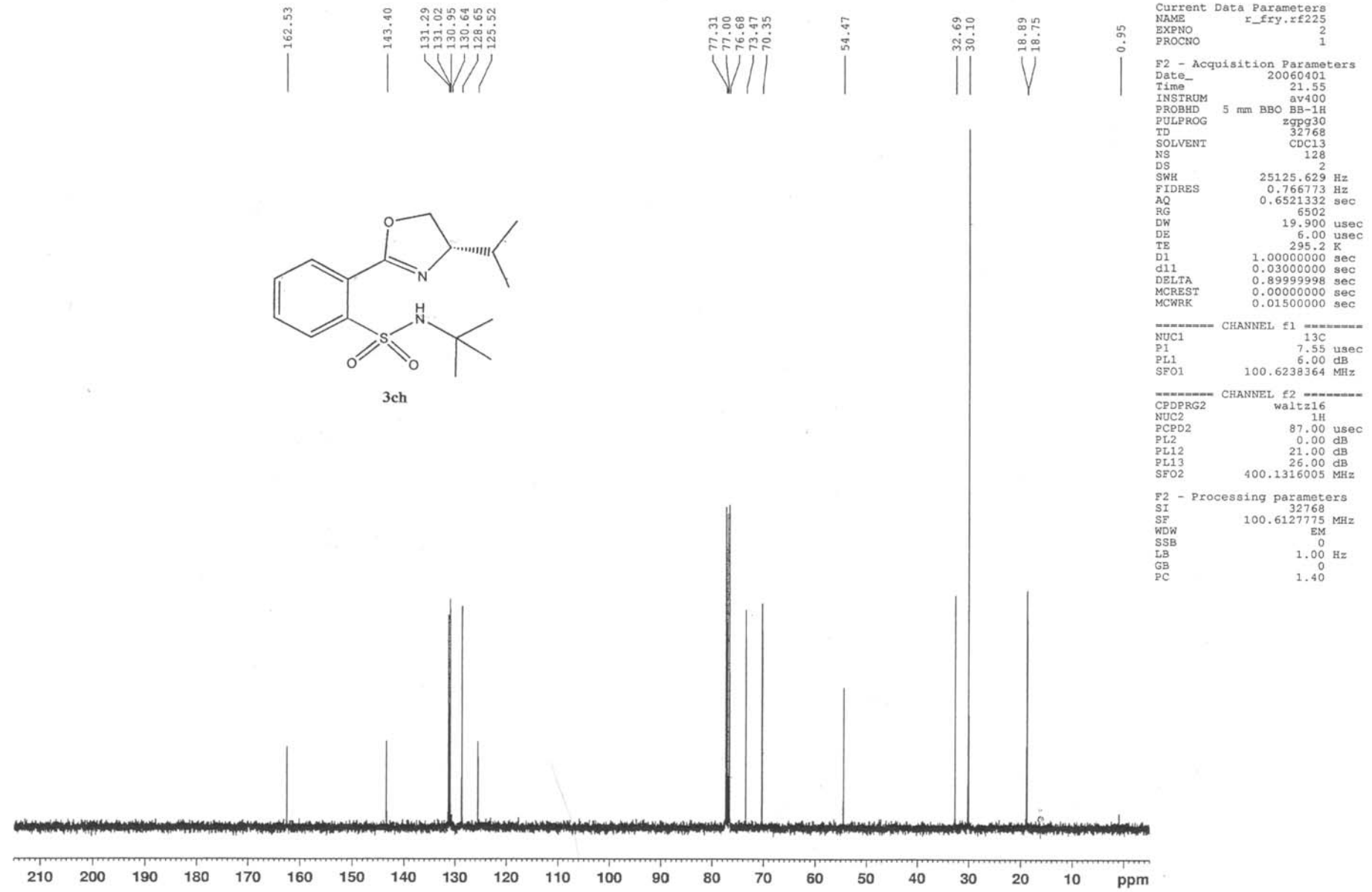
F2 - Processing parameters

SI	65536
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	22.00 cm
F1P	215.000 ppm
F1	21631.74 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.00000 ppm/cm
HZCM	1006.12732 Hz/cm

UserID r_fry SampleID rf225 SupervisorID swood Lab Phone No. 13538 Slot Number 1



Current Data Parameters

NAME	r_fry.rf225
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20060401
Time	21.55
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	128
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	6502
DW	19.900 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
d11	0.03000000 sec
DELTA	0.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	7.55 usec
PL1	6.00 dB
SFO1	100.6238364 MHz

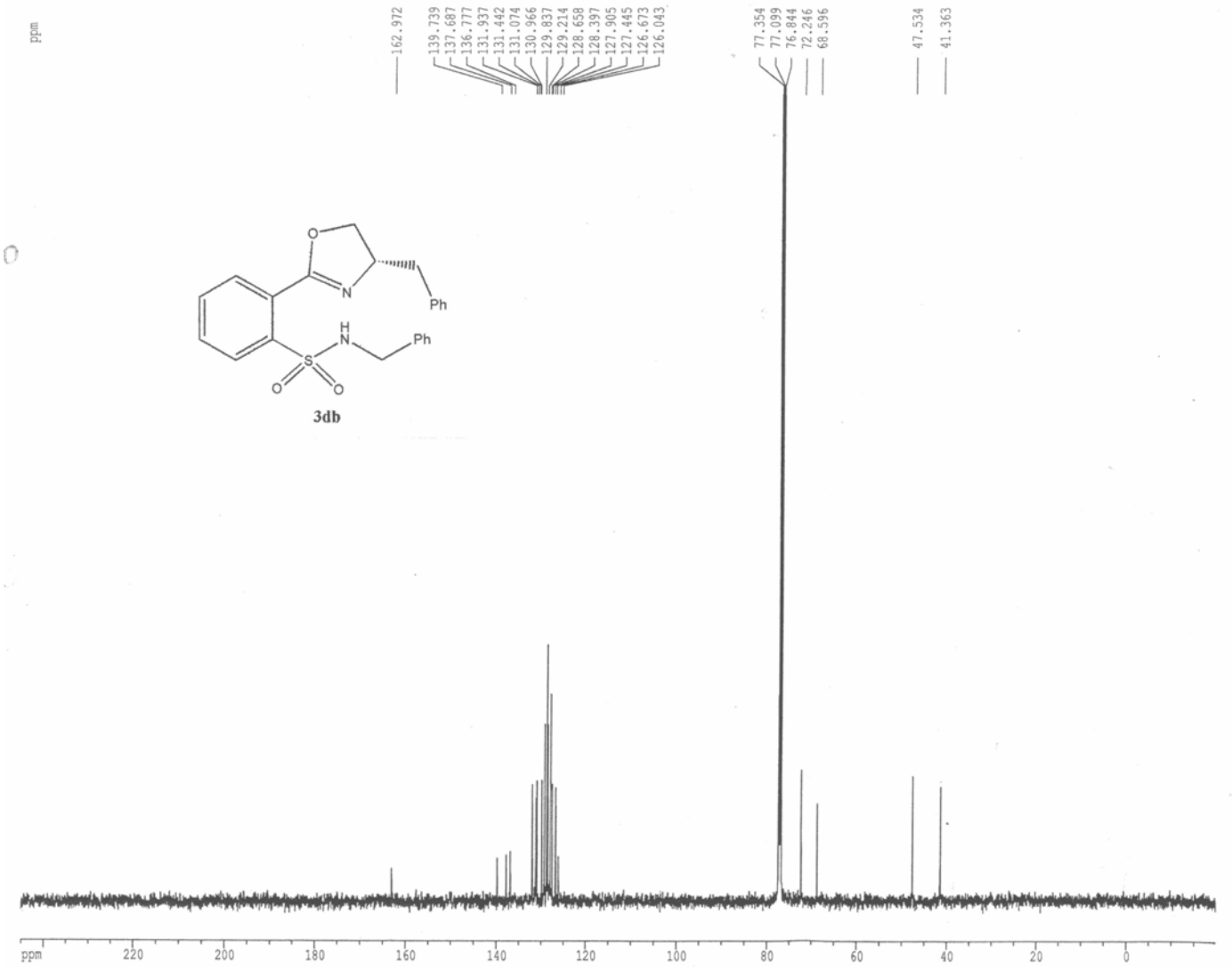
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	87.00 usec
PL2	0.00 dB
PL12	21.00 dB
PL13	26.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127775 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Carbon:RIR 360



3db

Current Data Parameters

NAME	rir360
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020116
Time	14.28
INSTRUM	DRX500
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	889
DS	2
SWH	35211.270 Hz
FIDRES	1.074563 Hz
AQ	0.4653556 sec
RG	7298.2
DW	14.200 usec
DE	5.50 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.50 usec
PL1	5.00 dB
SFO1	125.7715724 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	-3.00 dB
PL12	16.00 dB
PL13	16.00 dB
SFO2	500.1327507 MHz

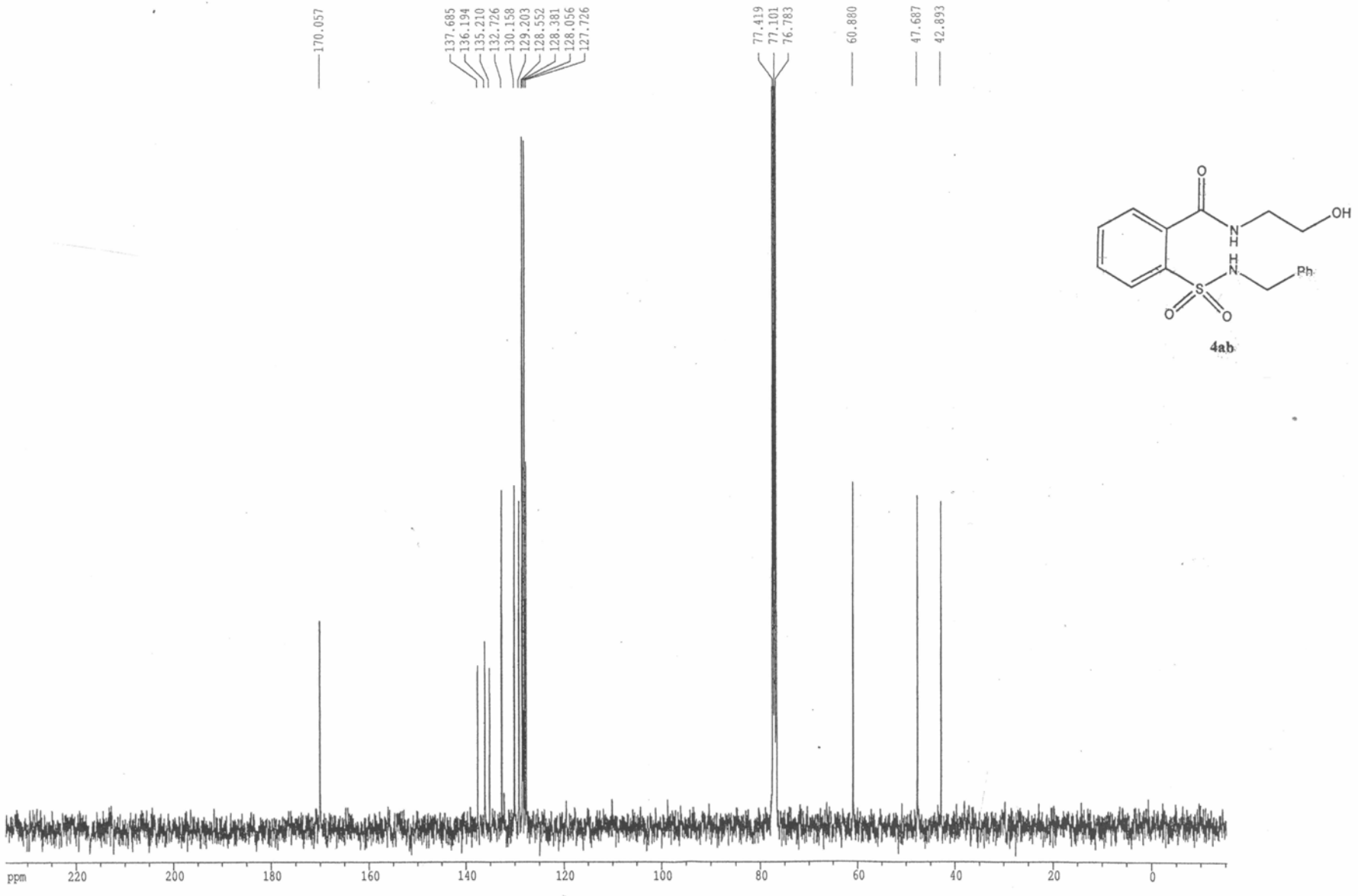
F2 - Processing parameters

SI	16384
SF	125.7577845 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
F1P	245.000 ppm
F1	30810.66 Hz
F2P	-20.000 ppm
F2	-2515.16 Hz
PPMCM	8.83333 ppm/cm
HZCM	1110.86035 Hz/cm

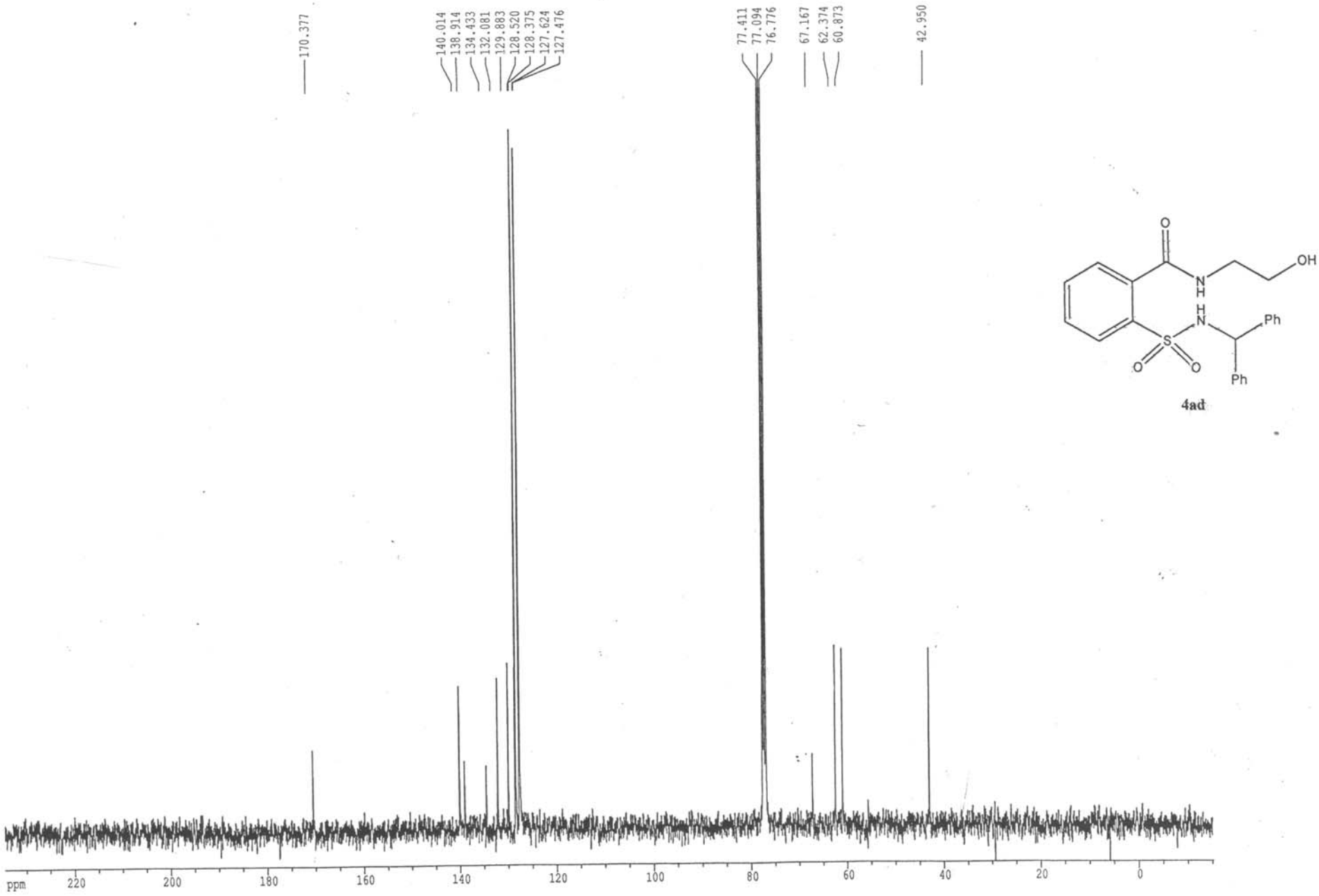
RIR 312


170.057
137.685
136.194
135.210
132.726
130.158
129.203
128.552
128.381
128.056
127.726
77.419
77.101
76.783
60.880
47.687
42.893
ppm
220
200
180
160
140
120
100
80
60
40
20
0

O
OH
N
H
H
N
Ph
S
O
O

4ab

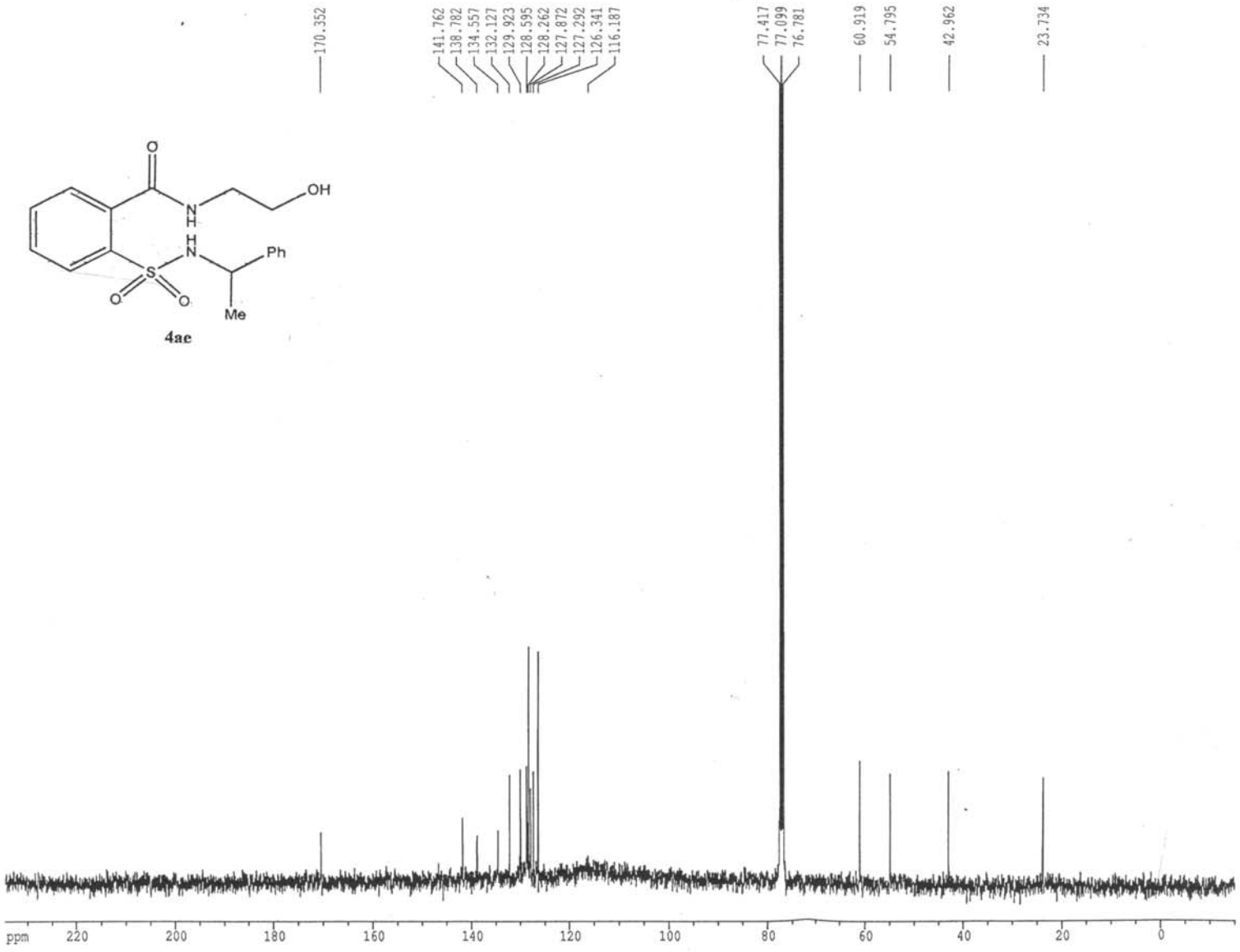
RIR 358


170.377
140.014
138.914
134.433
132.081
129.883
128.520
128.375
127.624
127.476
77.411
77.094
76.776
67.167
62.374
60.873
42.950
ppm
220
200
180
160
140
120
100
80
60
40
20
0

O
OH
N
H
H
N
Ph
S
O
O
Ph

4ad

RIR 454



Current Data Parameters

NAME	rir454
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020912
Time	16.22
INSTRUM	av400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1036
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	16384
DW	19.900 usec
DE	20.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	14.00 usec
PL1	-3.00 dB
SFO1	100.6237959 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	10.00 dB
PL12	30.00 dB
PL13	29.00 dB
SFO2	400.1316005 MHz

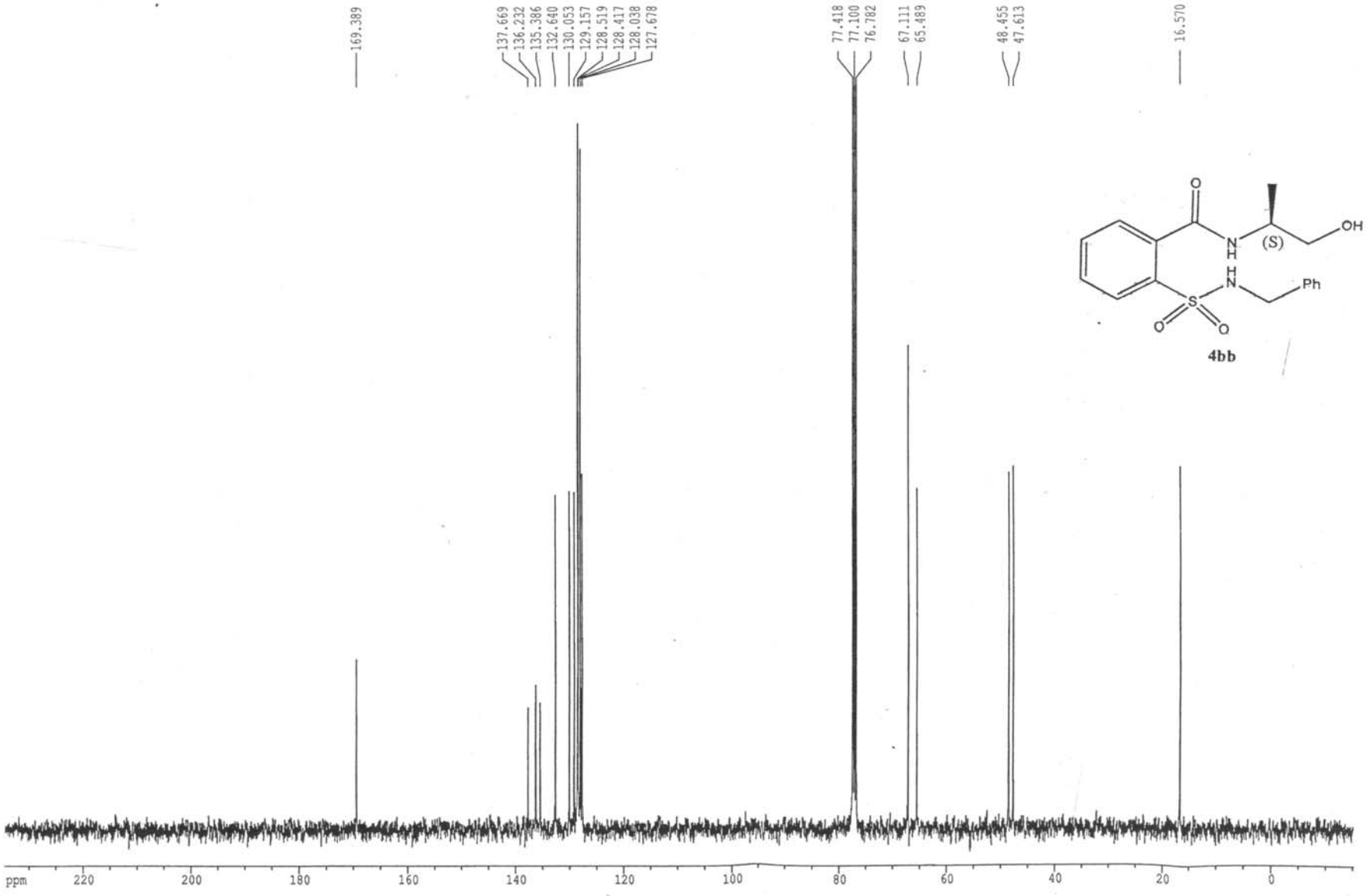
F2 - Processing parameters

SI	32768
SF	100.6127615 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

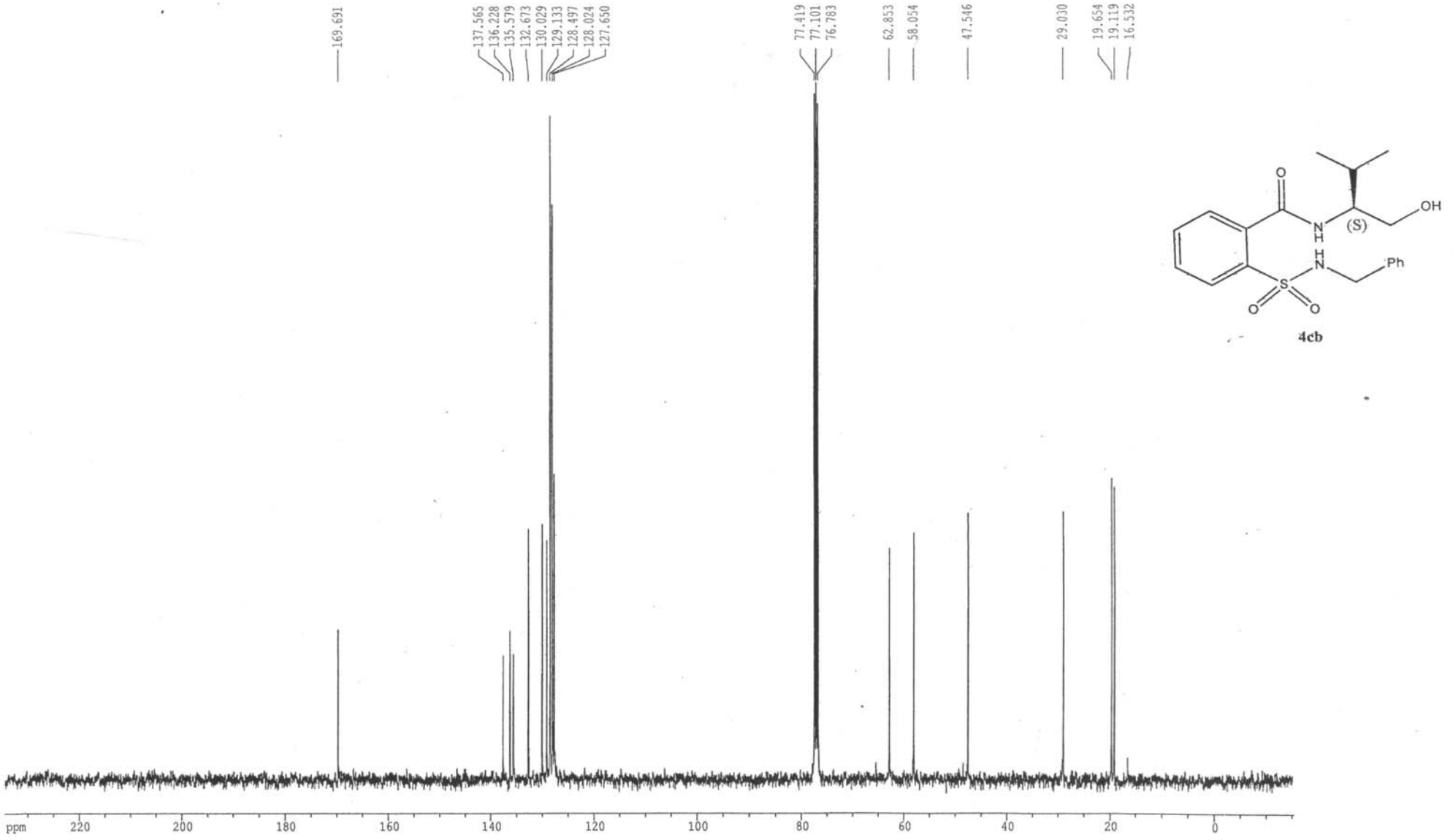
CX	30.00 cm
CY	28.62 cm
F1P	245.000 ppm
F1	24650.13 Hz
F2P	-20.000 ppm
F2	-2012.26 Hz
PPMCM	8.83333 ppm/cm
HZCM	888.74609 Hz/cm

RIR 351

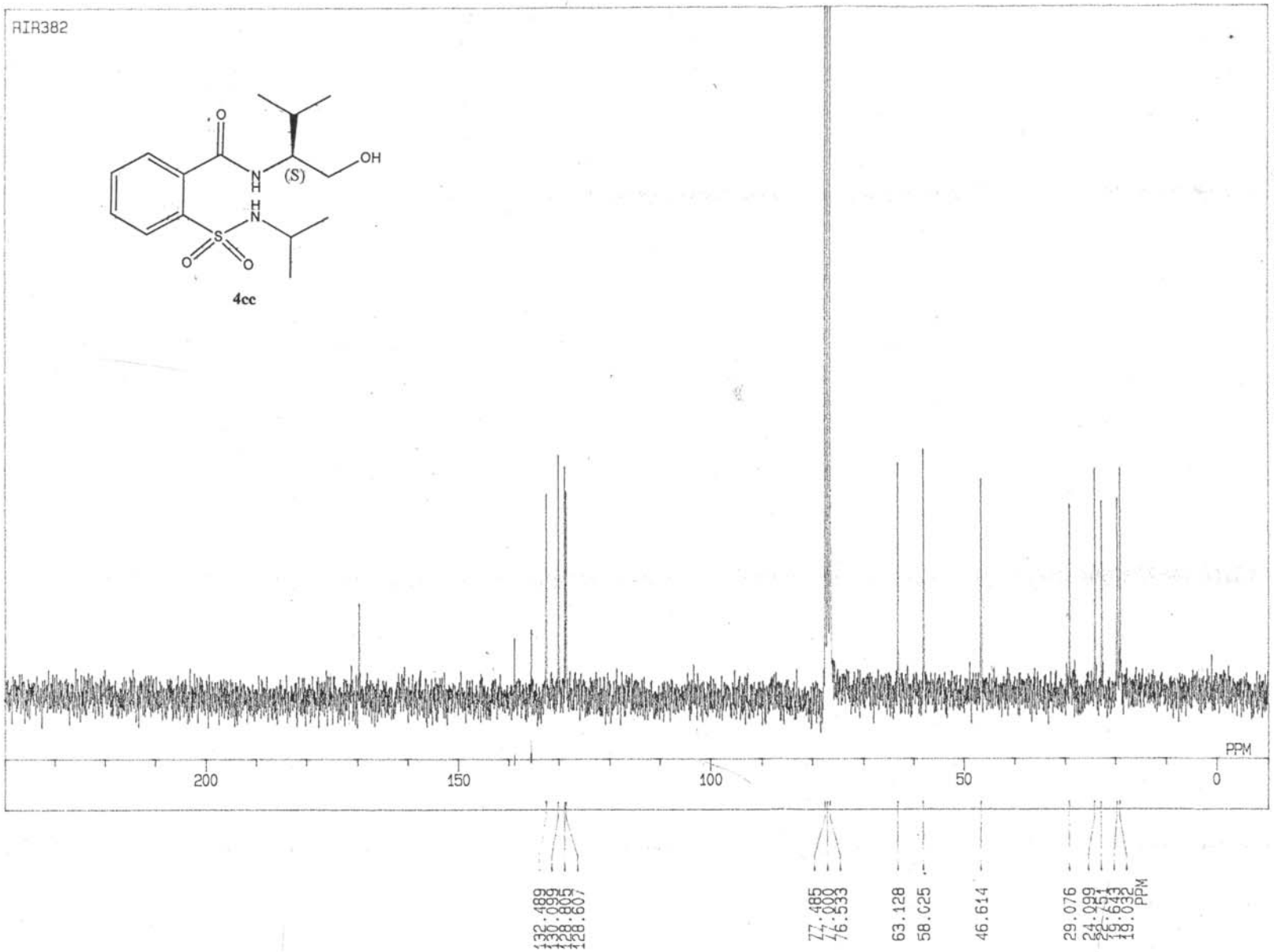

169.389
137.669
136.232
135.386
132.640
130.053
129.157
128.519
128.417
128.038
127.678
77.418
77.100
76.782
67.111
65.489
48.455
47.613
16.570
O
N
H
(S)
OH
N
H
Ph
S
O
O
ppm
220
200
180
160
140
120
100
80
60
40
20
0

4bb

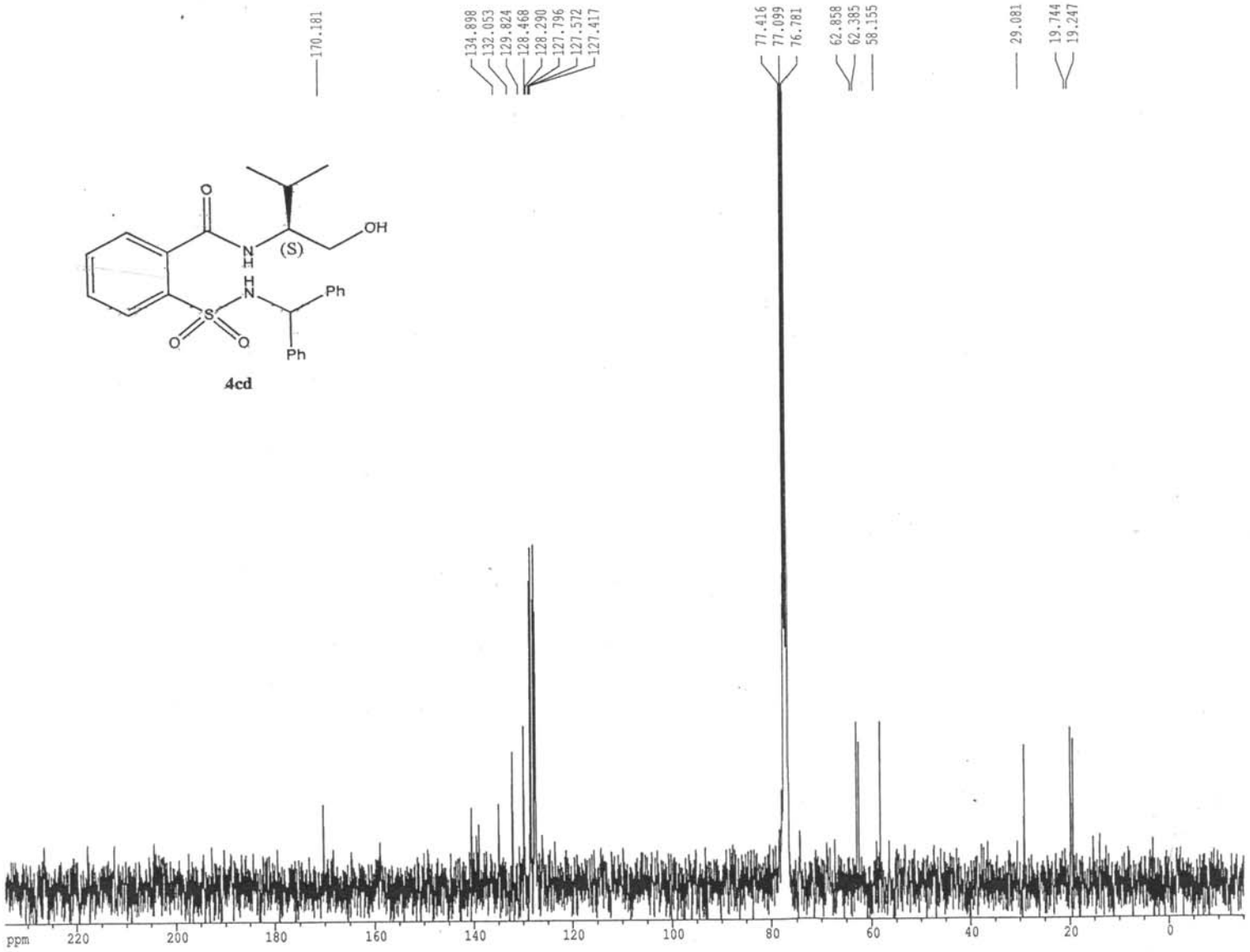
RIR 317 fr 24-34



4cb


RIR382
O
N
H
(S)
OH
N
H
S
O
O
4cc
UofN
13-OCT-02 20:39:15
DFILE Q13C
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 1197
ACQTM 0.819 sec
PD 1.181 sec
PW1 3.5 us
IRNUC 1H
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.50 Hz
RGAIN 28
OPERATOR :
PPM
200
150
100
50
0
132.489
130.099
128.805
128.807
77.485
77.000
76.533
63.128
58.625
46.614
29.076
24.099
22.471
19.643
19.032
PPM

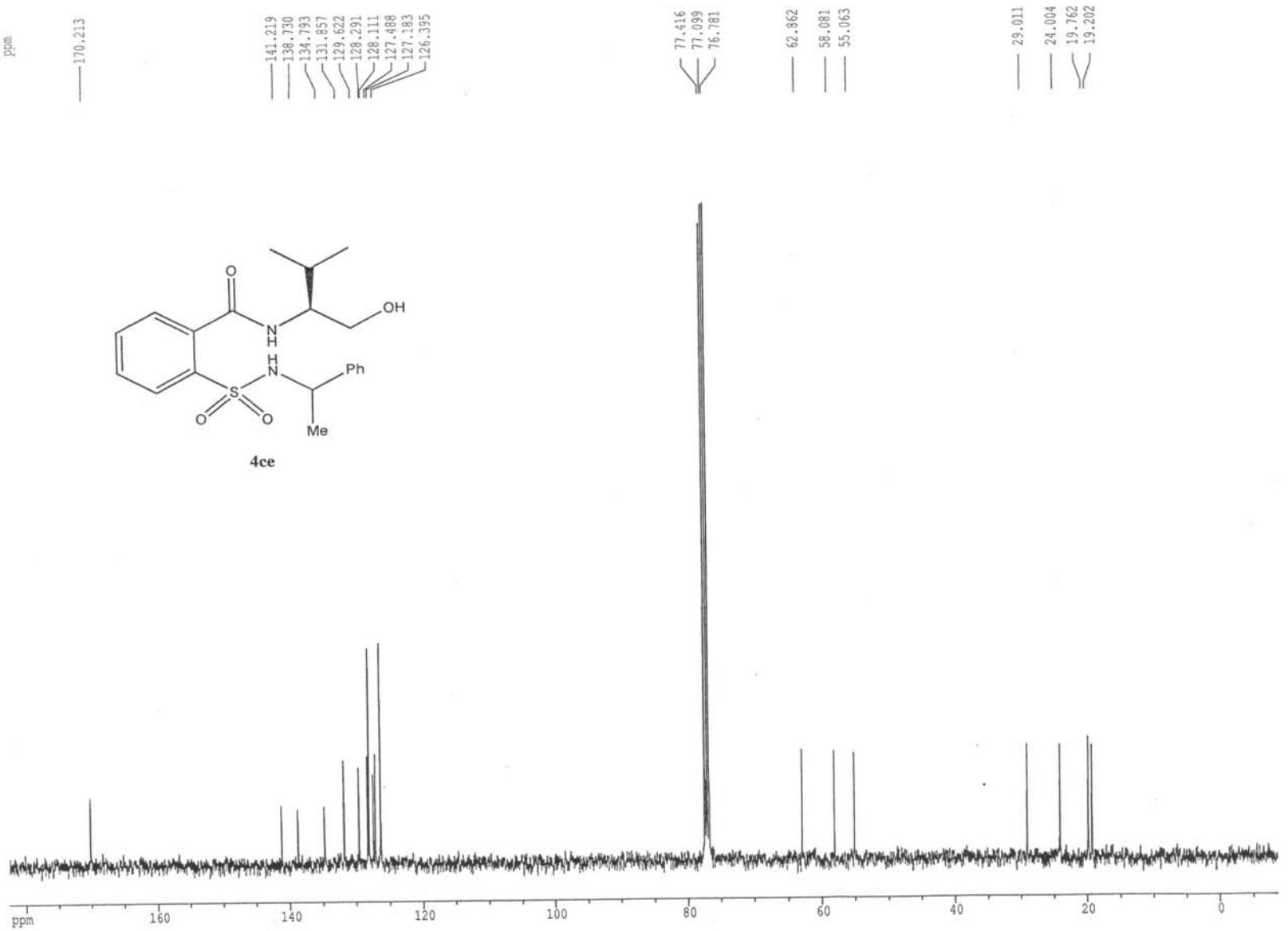
RIR 381



4cd

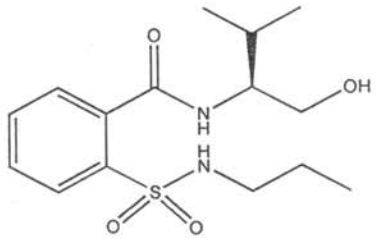
```
Current Data Parameters
NAME                  rir381
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20021012
Time                  15.48
INSTRUM               av400
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                     2000
DS                        2
SWH               25125.629 Hz
FIDRES             0.766773 Hz
AQ                 0.6521332 sec
RG                    16384
DW                   19.900 usec
DE                   20.00 usec
TE                   300.0 K
D1              1.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.50 usec
PL1                   -0.10 dB
SFO1          100.6237959 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   2.90 dB
PL12                 25.50 dB
PL13                 29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6127607 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    30.00 cm
CY                   127.80 cm
F1P                 245.000 ppm
F1                24650.13 Hz
F2P                -20.000 ppm
F2                -2012.26 Hz
PPMCM              8.83333 ppm/cm
HZCM             888.74609 Hz/cm
```

RIR 591 A




```
Current Data Parameters
NAME                  rir591A
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20021006
Time                  22.55
INSTRUM               av400
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     268
DS                       2
SWH              25125.629 Hz
FIDRES            0.766773 Hz
AQ                0.6521332 sec
RG                   16384
DW                   19.900 usec
DE                   20.00 usec
TE                   300.0 K
D1               1.00000000 sec
d11              0.03000000 sec
d12              0.00002000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.50 usec
PL1                  -0.10 dB
SFO1          100.6237959 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                   2.90 dB
PL12                 25.50 dB
PL13                 29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127638 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                   30.00 cm
CY                   17.19 cm
F1P                182.525 ppm
F1                18364.33 Hz
F2P                -8.715 ppm
F2                -876.86 Hz
PPMCM              6.37467 ppm/cm
HZCM             641.37292 Hz/cm
```

UserID r_fry SampleID rf216 SupervisorID swood Lab Phone No. 13538 Slot Number 51



4cg

Current Data Parameters

NAME	r_fry.rf216
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20060316
Time	10.12
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	512
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	9195.2
DW	19.900 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
d11	0.03000000 sec
DELTA	0.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	7.55 usec
PL1	6.00 dB
SFO1	100.6238364 MHz

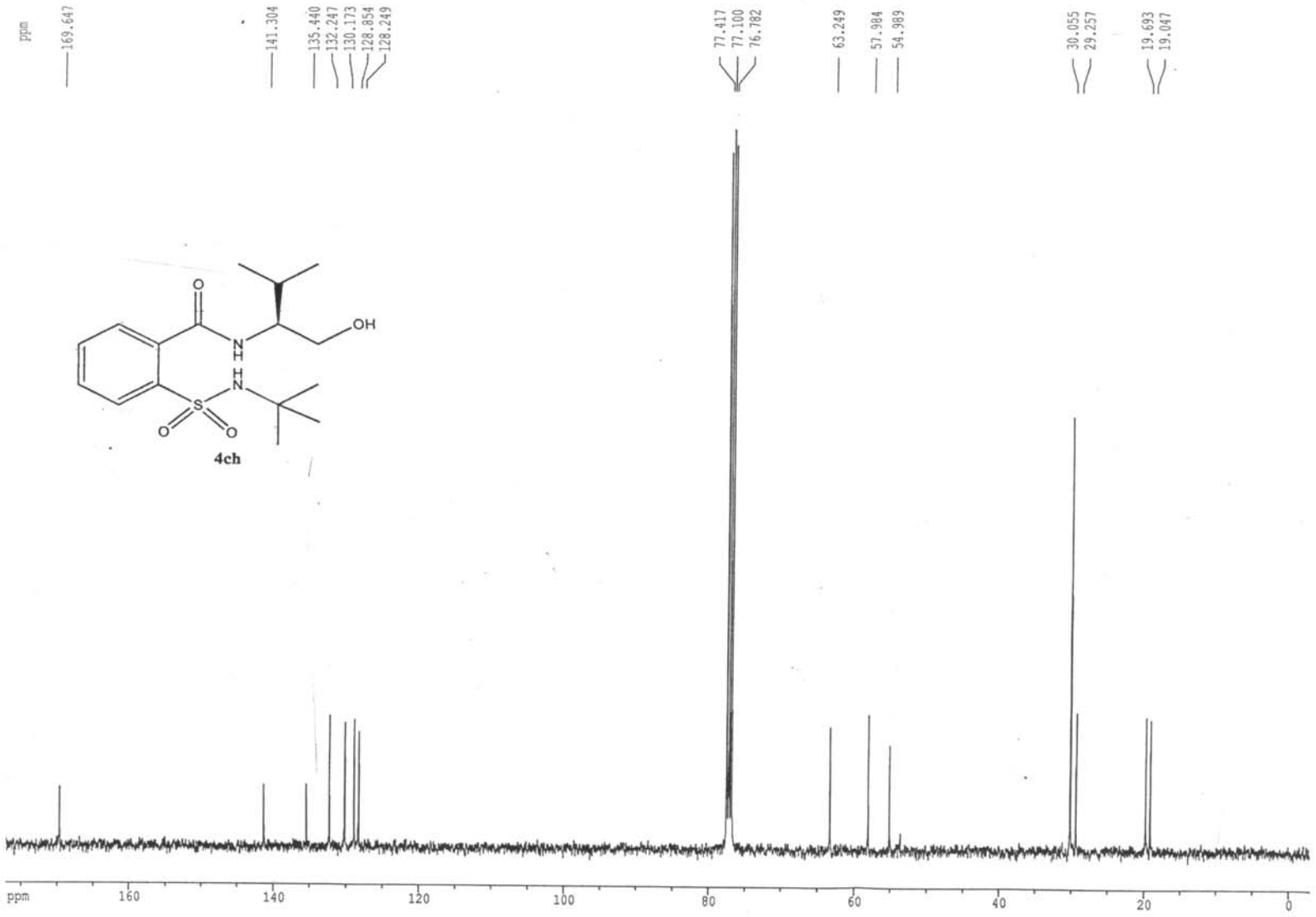
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	87.00 usec
PL2	0.00 dB
PL12	21.00 dB
PL13	26.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127752 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

RIR 590



Current Data Parameters

NAME	rir590
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020922
Time	18.35
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	759
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	16384
DW	19.900 usec
DE	20.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.50 usec
PL1	-0.10 dB
SFO1	100.6237959 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	2.90 dB
PL12	25.50 dB
PL13	29.00 dB
SFO2	400.1316005 MHz

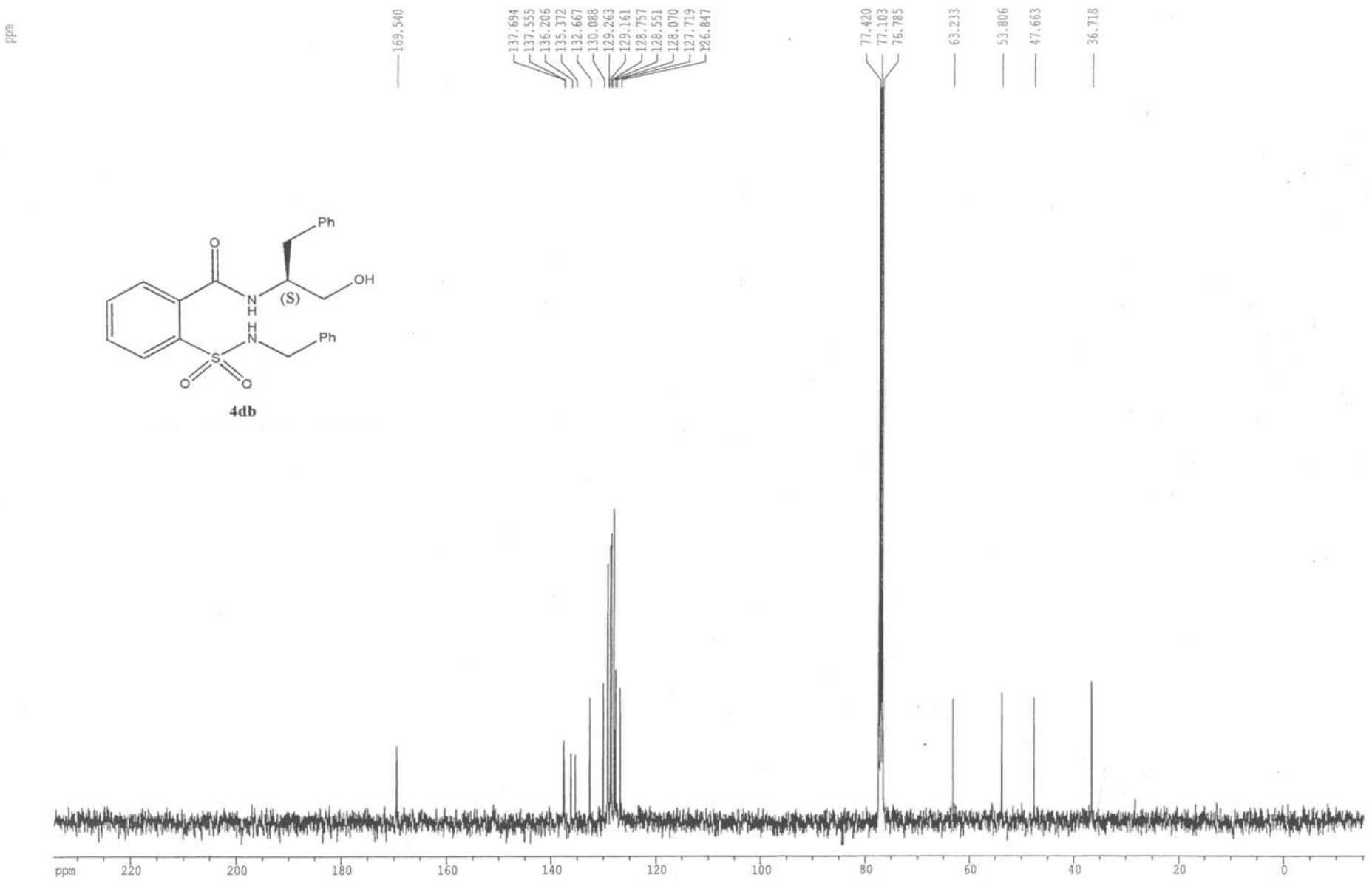
F2 - Processing parameters

SI	32768
SF	100.6127622 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

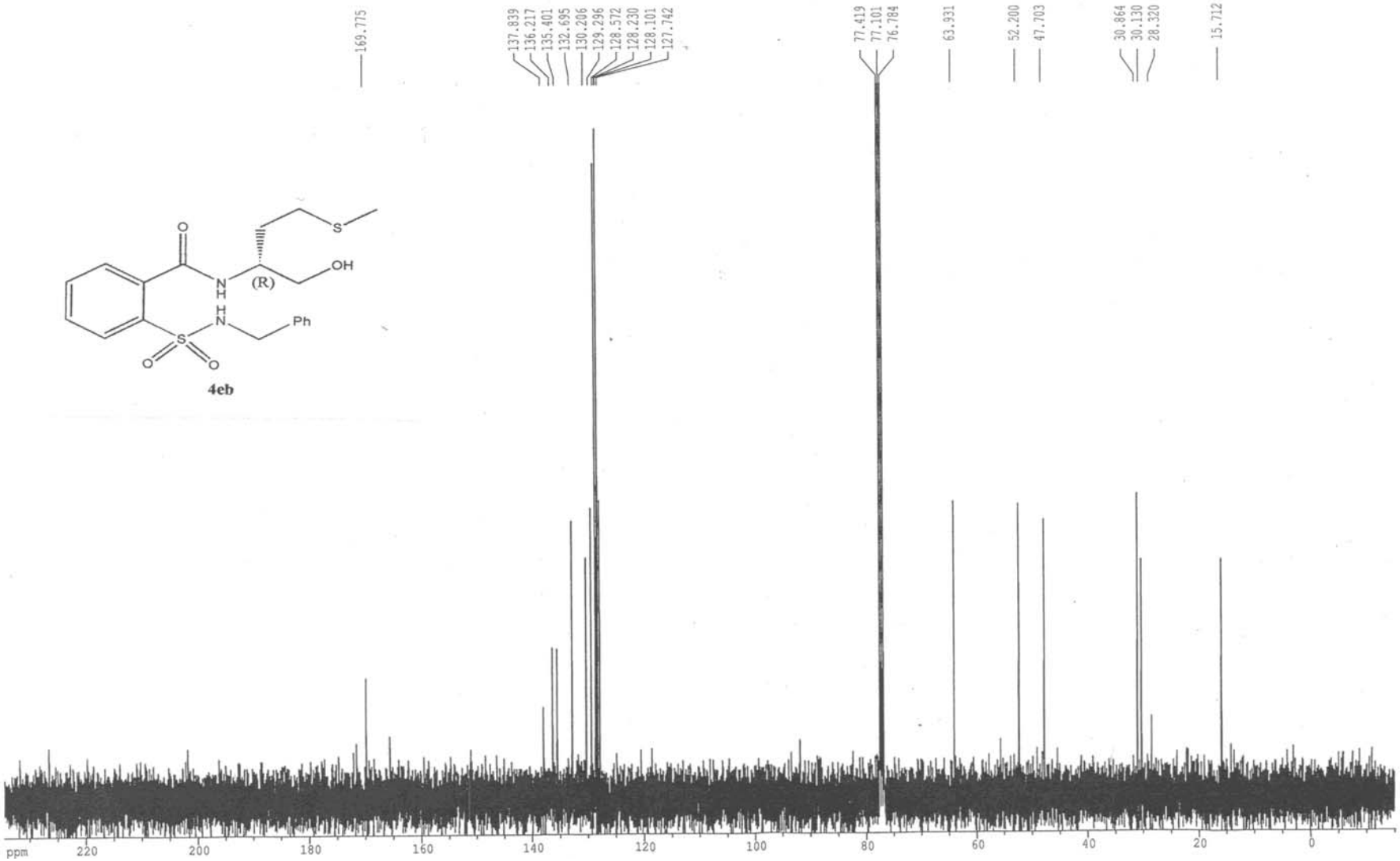
1D NMR plot parameters

CX	30.00 cm
CY	19.00 cm
F1P	177.091 ppm
F1	17817.58 Hz
F2P	-3.024 ppm
F2	-304.27 Hz
PPMCM	6.00383 ppm/cm
HZCM	604.06195 Hz/cm

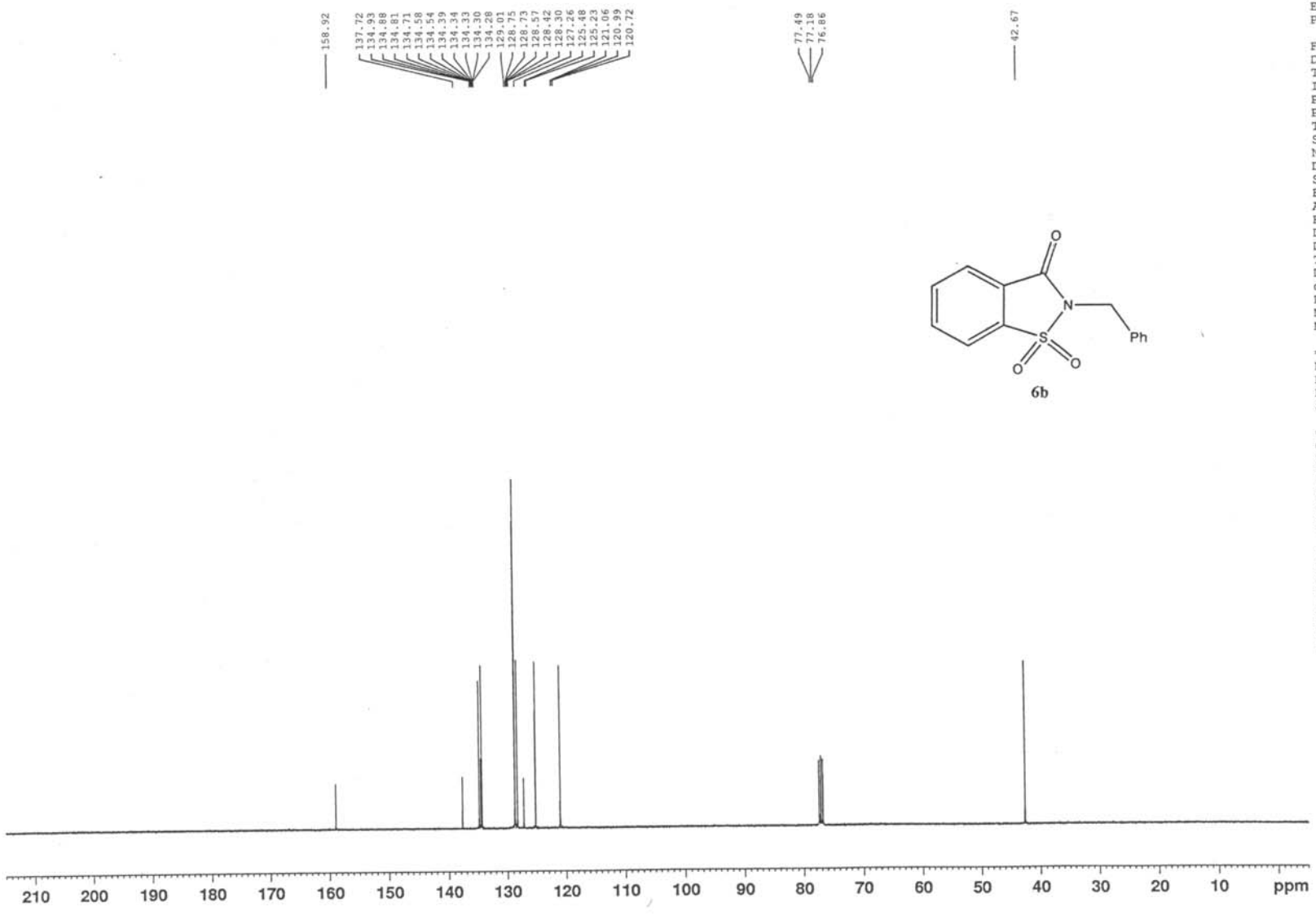
RIR 318


ppm
169.540
137.694
137.555
136.206
135.372
132.667
130.088
129.263
129.161
128.757
128.551
128.070
127.719
126.847
77.420
77.103
76.785
63.233
53.806
47.663
36.718
Ph
O
N
H
(S)
OH
N
H
Ph
S
O
O
4db
ppm
220
200
180
160
140
120
100
80
60
40
20
0

RIR 452


ppm
169.775
137.839
136.217
135.401
132.695
130.206
129.296
128.572
128.230
128.101
127.742
77.419
77.101
76.784
63.931
52.200
47.703
30.864
30.130
28.320
15.712
O
S
OH
N
H
(R)
N
H
Ph
S
O
O
4eb
ppm
220
200
180
160
140
120
100
80
60
40
20
0

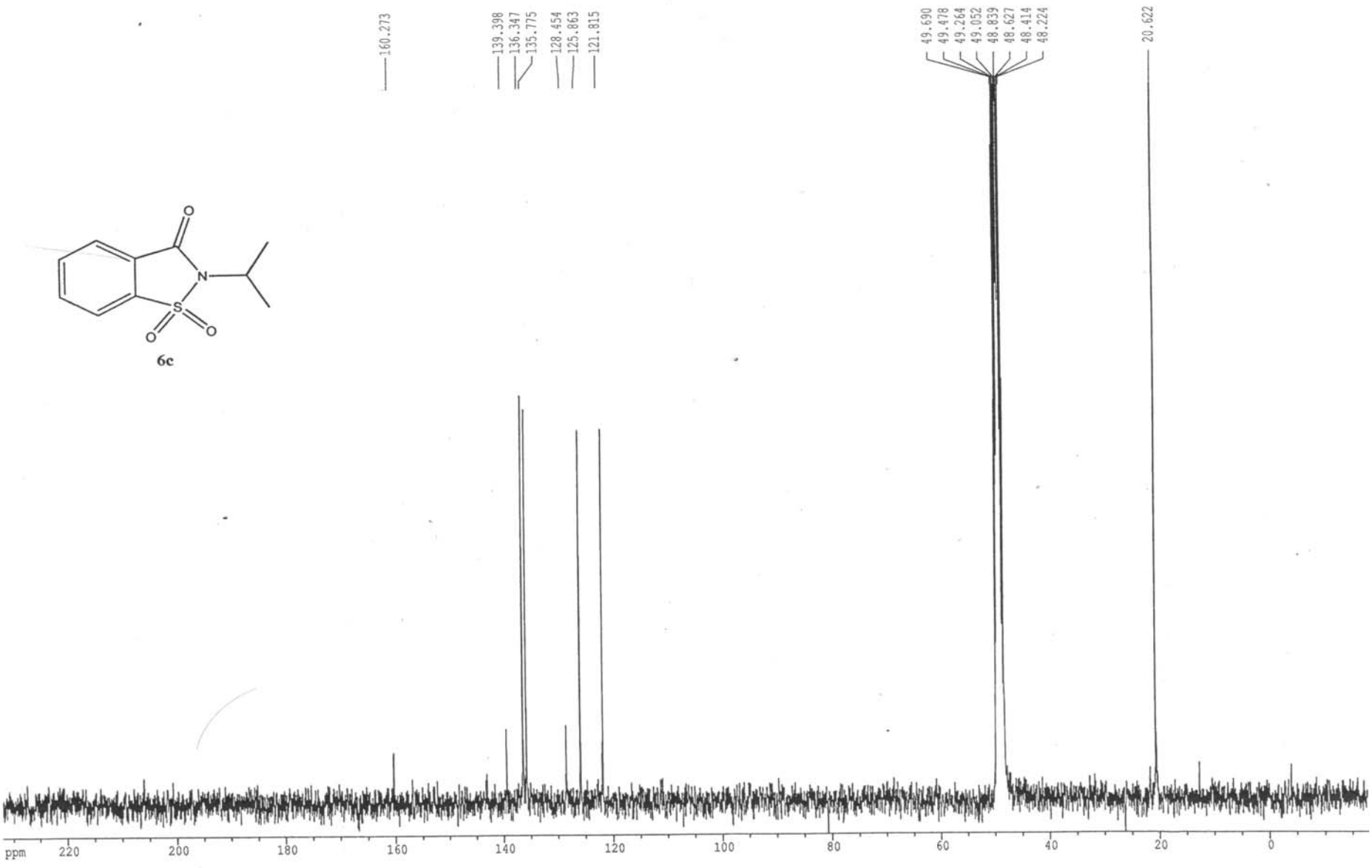
UserID r_fry SampleID rf209 SupervisorID swood Lab Phone No. 13538 Slot Number 1



6b

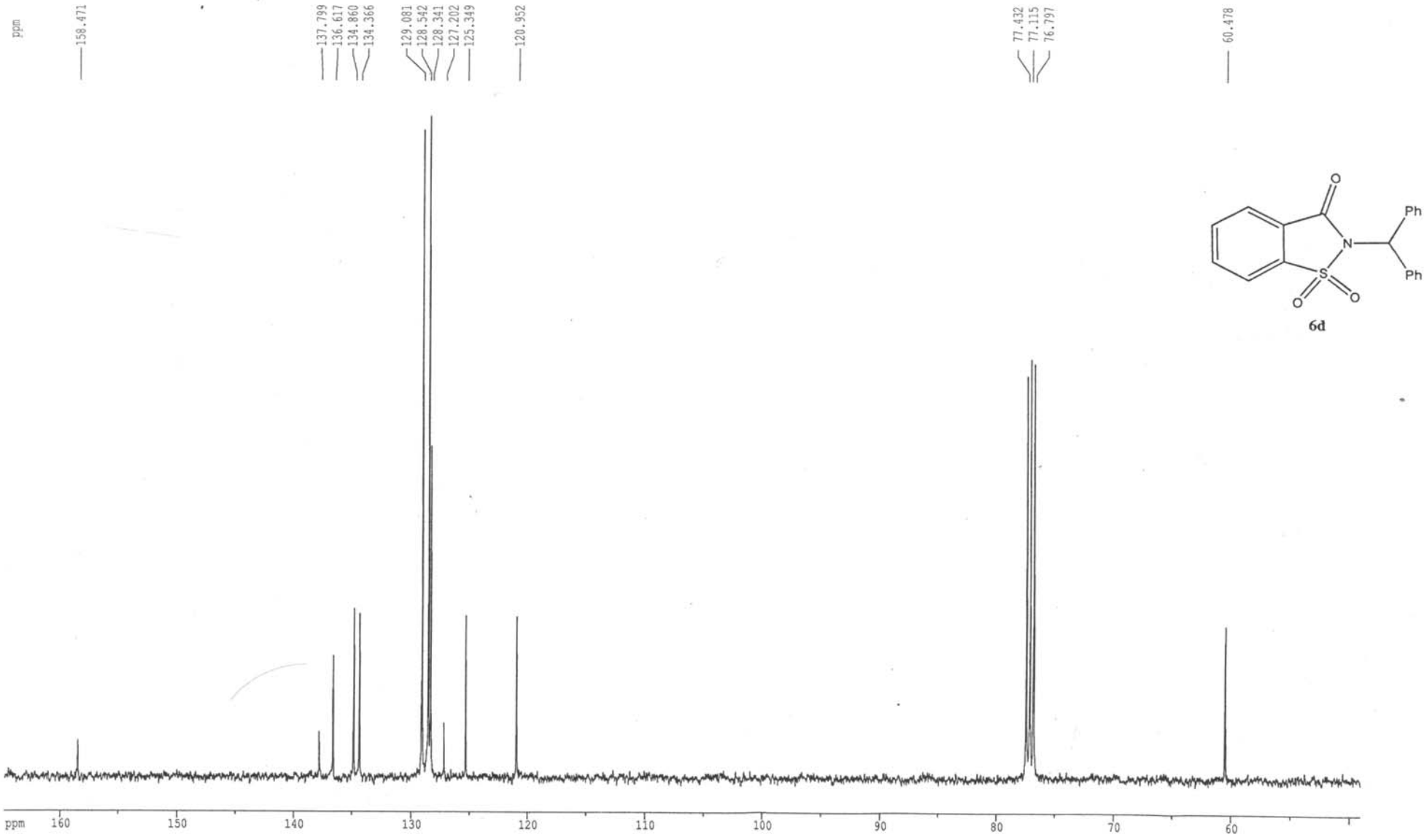
```
Current Data Parameters
NAME                  r_fry.rf209
EXPNO                           6
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20060320
Time                        11.27
INSTRUM                     av400
PROBHD    5 mm BBO BB-1H
PULPROG                   zgpg30
TD                         32768
SOLVENT                    CDCl3
NS                           512
DS                             2
SWH                  25125.629 Hz
FIDRES                0.766773 Hz
AQ                    0.6521332 sec
RG                       7298.2
DW                       19.900 use
DE                        6.00 use
TE                       298.2 K
D1                    1.00000000 sec
d11                   0.03000000 sec
DELTA                 0.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                        13C
P1                         7.55 use
PL1                        6.00 dB
SFO1                100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     87.00 use
PL2                        0.00 dB
PL12                      21.00 dB
PL13                      26.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127690 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                         1.40
```

RIR 361


160.273
139.398
136.347
135.775
128.454
125.863
121.815
49.690
49.478
49.264
49.052
48.839
48.627
48.414
48.224
20.622
ppm
220
200
180
160
140
120
100
80
60
40
20
0

6c

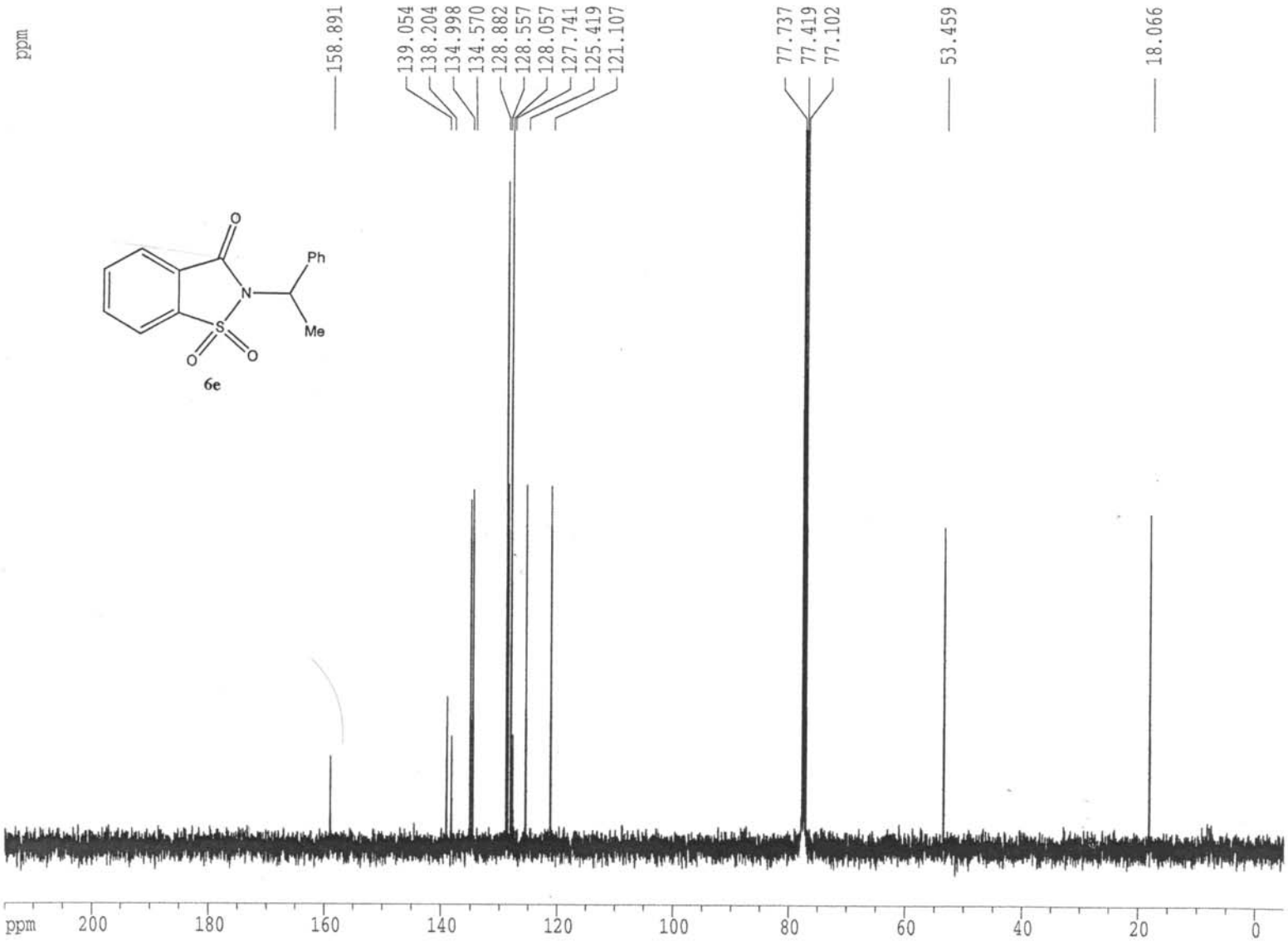
RIR 353


ppm
158.471
137.799
136.617
134.860
134.366
129.081
128.542
128.341
127.202
125.349
120.952
77.432
77.115
76.797
60.478
ppm 160 150 140 130 120 110 100 90 80 70 60

O
Ph
N
Ph
S
O
O

6d

User/Group Robinson/Pfaltz



Current Data Parameters
NAME pcxrir.002
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020410
Time 18.17
INSTRUM dpx400
PROBHD 5 mm QNP 1H/13
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 384
DS 16
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000005 sec
D11 0.03000000 sec

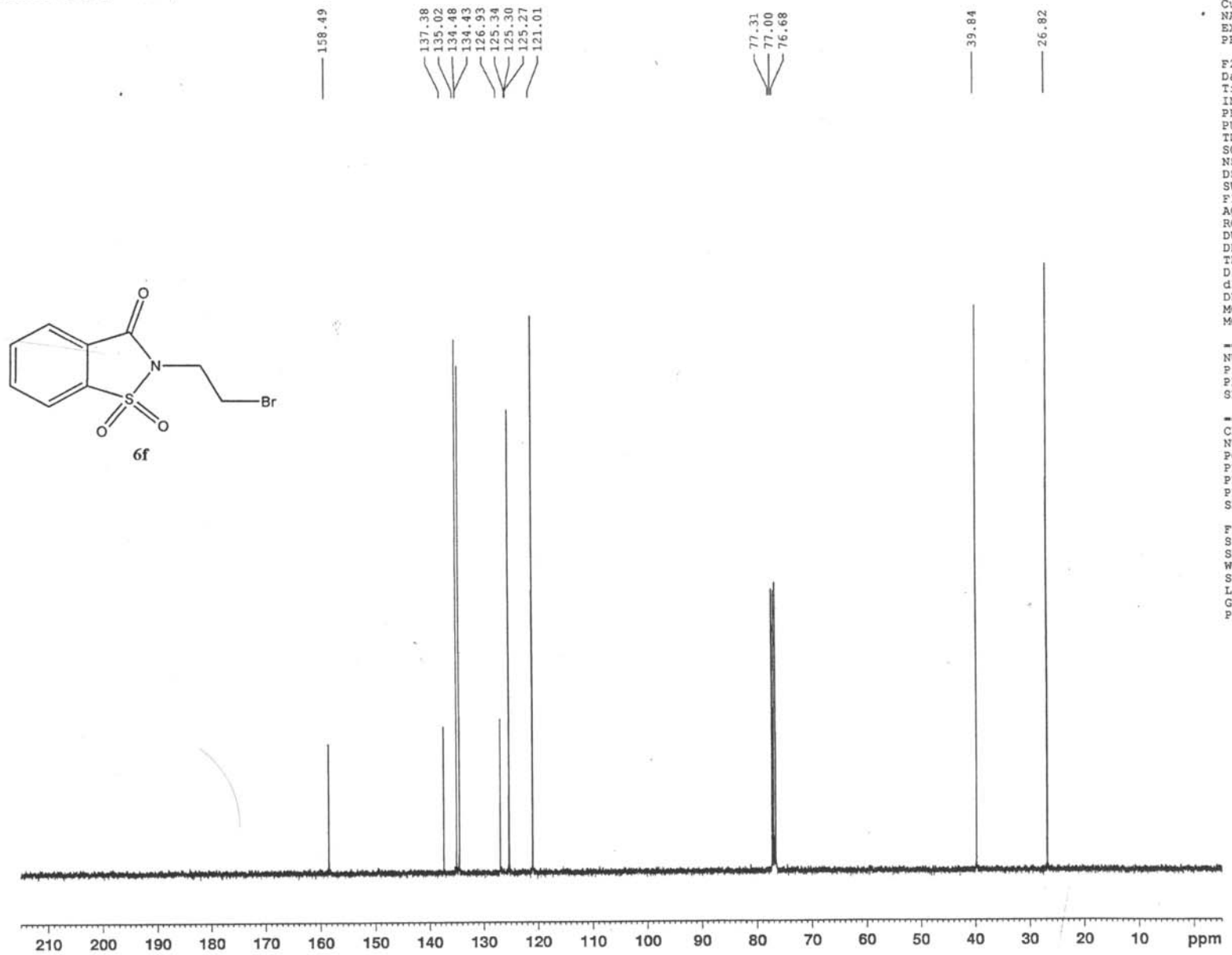
========== CHANNEL f1 ==== ======
NUC1 13C
P1 9.90 usec
PL1 -3.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==== ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 13.00 dB
PL12 13.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
F1P 225.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.05 Hz
PPMCM 10.00000 ppm/ m
HZCM 1006.12732 Hz/cm

UserID r_fry SampleID rf017 SupervisorID swood Lab Phone No. 13538 Slot Number 54



Current Data Parameters

NAME	r_fry.rf017
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20060316
Time	10.42
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	512
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	10321.3
DW	19.900 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
d11	0.03000000 sec
DELTA	0.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

-------- CHANNEL f1 --------

NUC1	13C
P1	7.55 usec
PL1	6.00 dB
SFO1	100.6238364 MHz

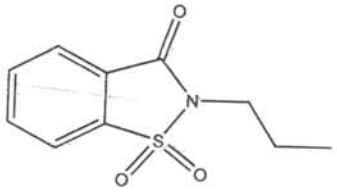
-------- CHANNEL f2 --------

CPDPRG2	waltz16
NUC2	1H
PCPD2	87.00 usec
PL2	0.00 dB
PL12	21.00 dB
PL13	26.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127798 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

RIR 394



6g

Current Data Parameters
NAME rir394
EXPNO 2
PROCNO 1

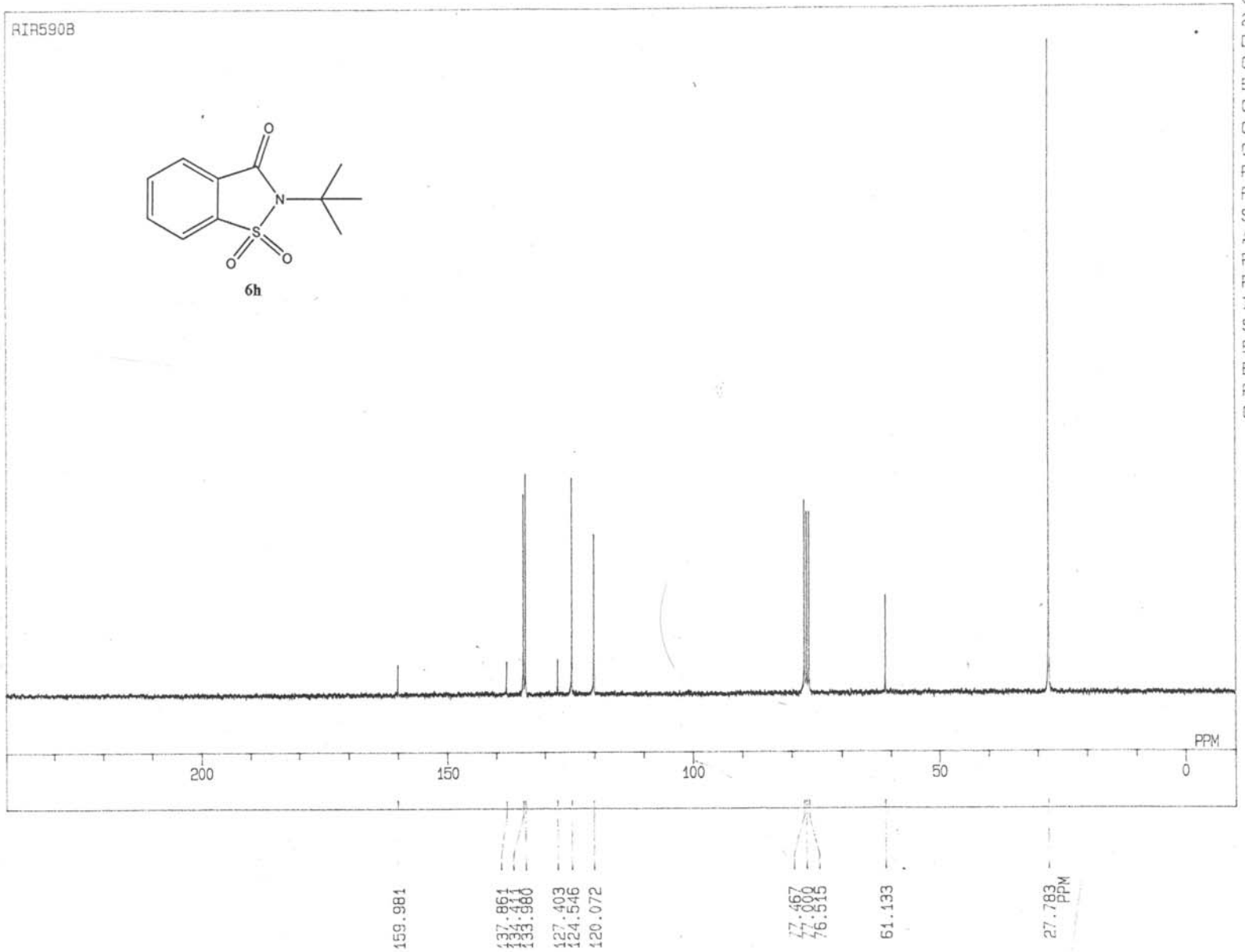
F2 - Acquisition Parameters
Date_ 20020308
Time 16.39
INSTRUM av400
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 245
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 16384
DW 19.900 usec
DE 20.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

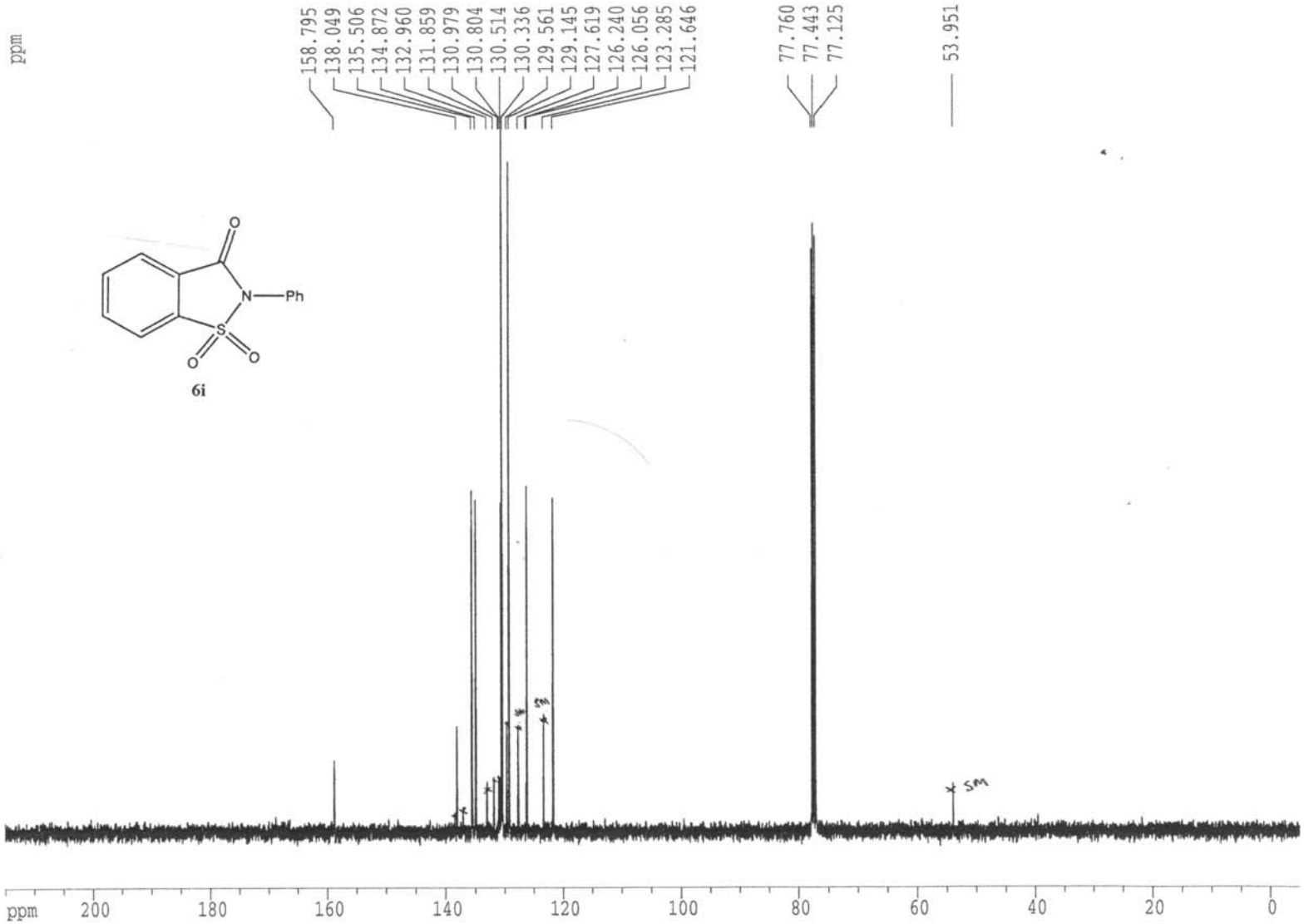
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127630 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 35.00 cm
CY 19.00 cm
F1P 246.000 ppm
F1 [illegible] Hz
F2P [illegible].000 ppm
F2 -2012.26 Hz
PPMCM 7.57143 ppm/cm
HZCM 761.78235 Hz/cm


RIR590B
O
N
S
O
O
6h
UofH
26-SEP-02 18:23:50
DFILE G13C
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 1807
ACQTM 0.819 sec
PD 1.181 sec
PW1 3.5 us
IRNUC 1H
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.50 Hz
RGAIN 28
OPERATOR :
PPM
200
150
100
50
0
159.981
137.861
134.411
133.980
127.403
124.546
120.072
77.467
77.000
76.515
61.133
27.783
PPM

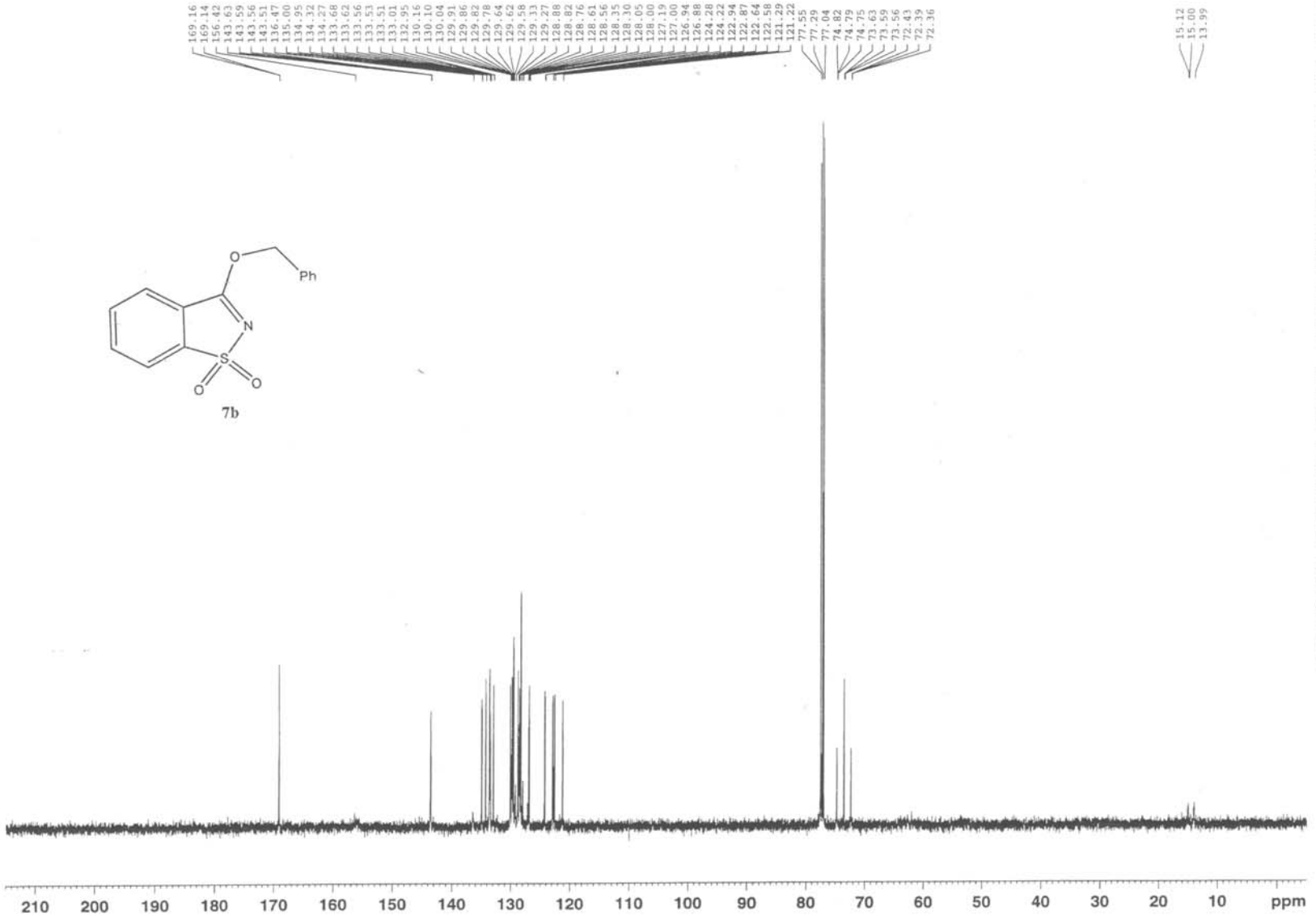
User/Group Robinson/Pfaltz
RIR 443 EtOH recryst.



6i

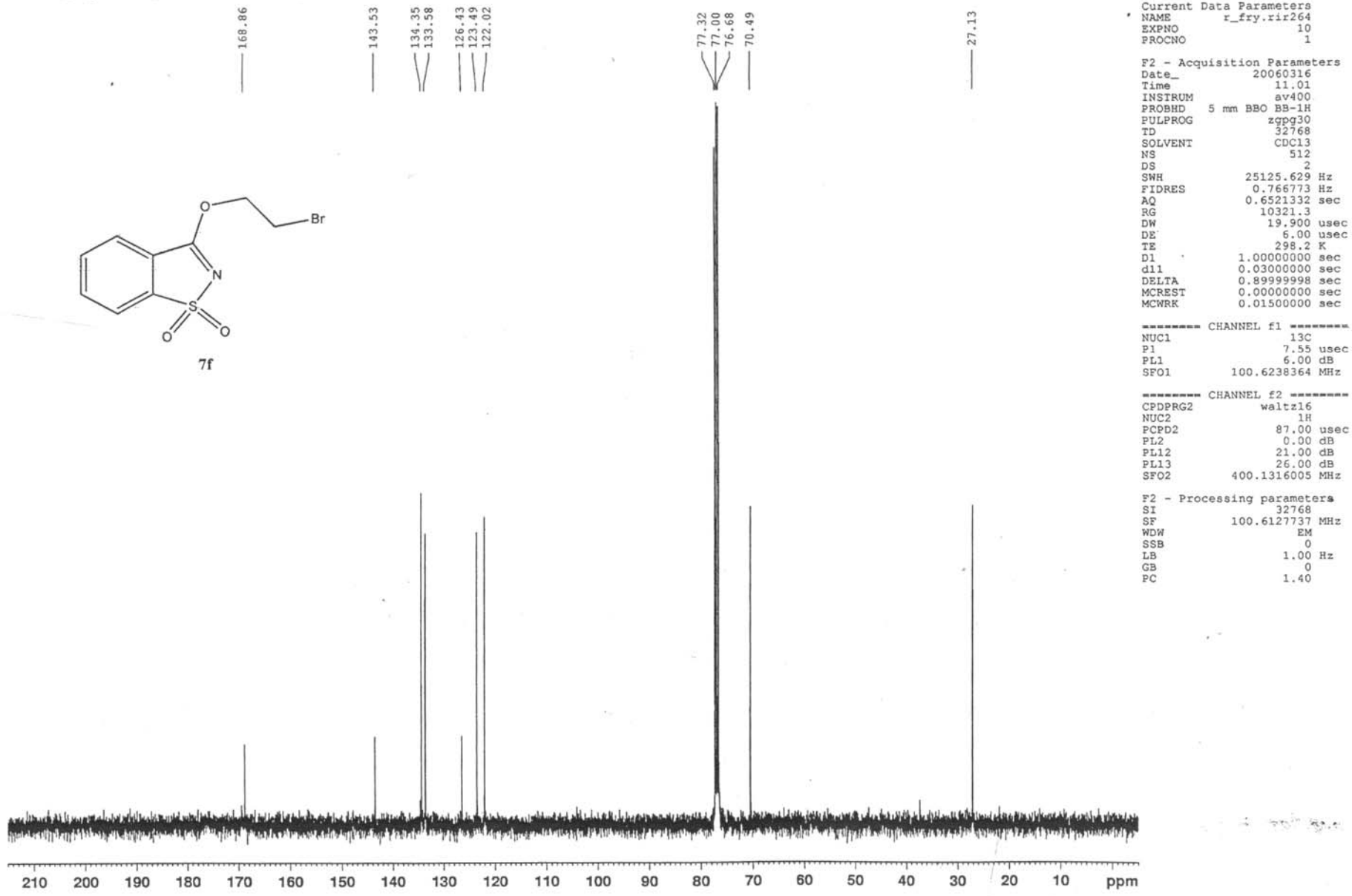
```
Current Data Parameters
NAME          pcxrir.040
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20020428
Time               16.25
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/13
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                   16
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG               11585.2
DW                19.900 usec
DE                 6.00 usec
TE                300.0 K
D1            2.20000005 sec
d11           0.03000000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                 9.90 usec
PL1               -3.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               13.00 dB
PL12              13.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127290 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
F1P               215.000 ppm
F1              21631.74 Hz
F2P                -5.000 ppm
F2               -503.06 Hz
PPMCM           10.00000 ppm/cm
HZCM          1006.12732 Hz/cm
```

userID: r_fry sampleID: rf222
13C[CPD] Spectrum




```
Current Data Parameters
NAME                  r_fry.rf222
EXPNO                           6
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20060325
Time                        14.59
INSTRUM                    DRX500
PROBHD    5 mm Multinucl
PULPROG                   zgpg30
TD                         32768
SOLVENT                    CDCl3
NS                           512
DS                             2
SWH                    31446.541 Hz
FIDRES                 0.959672 Hz
AQ                     0.5210612 sec
RG                          3251
DW                        15.900 use
DE                         6.00 use
TE                        300.0 K
D1                  1.00000000 sec
d11                 0.03000000 sec
DELTA               0.89999998 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 =======
NUC1                         13C
P1                         9.70 use
PL1                        4.00 dB
SFO1                125.7716219 MHz

======== CHANNEL f2 =======
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    100.00 use
PL2                       -3.00 dB
PL12                      16.00 dB
PL13                      18.00 dB
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577890 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

UserID r_fry SampleID rf264 SupervisorID swood Lab Phone No. 13538 Slot Number 55



7f

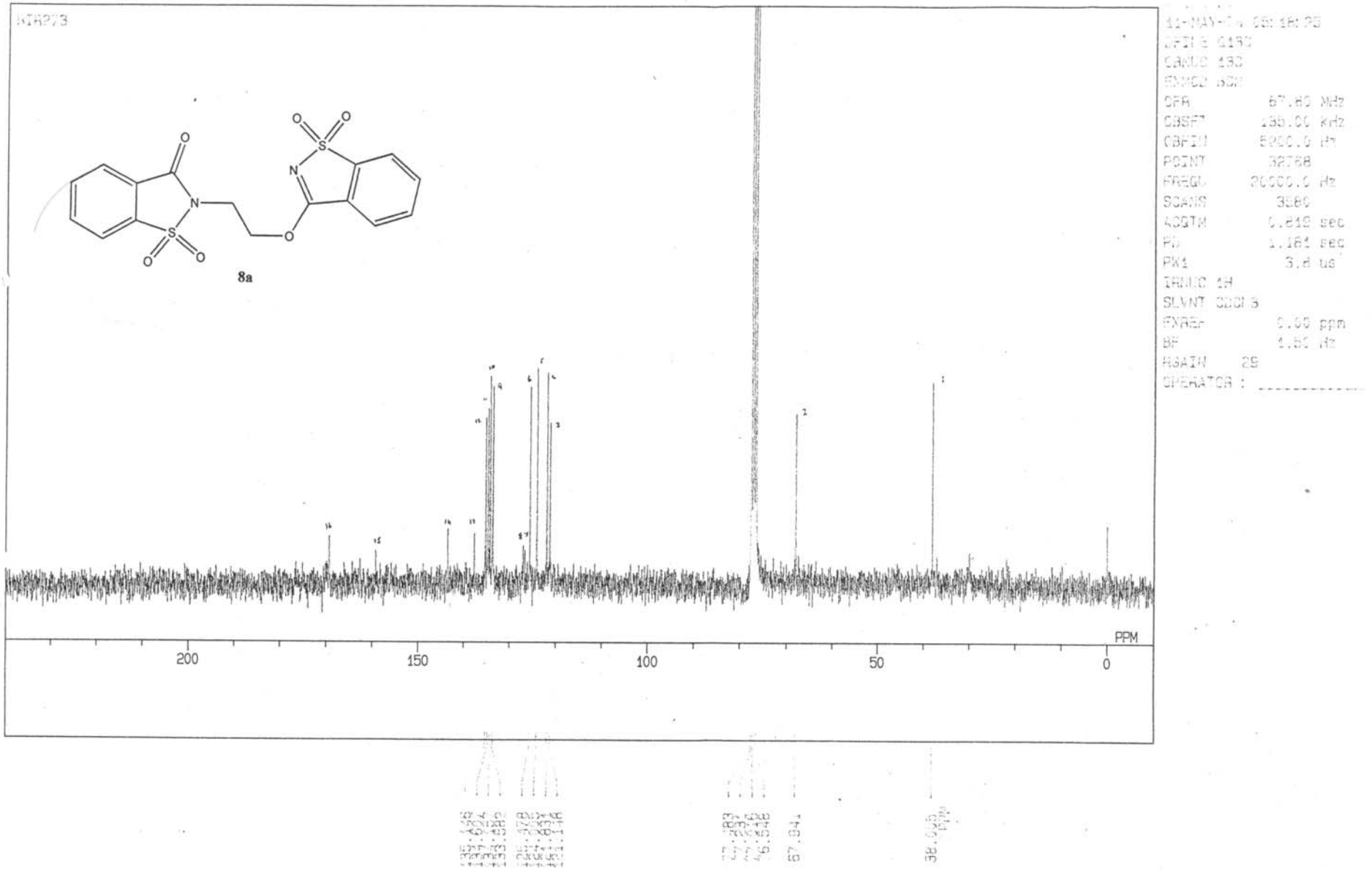
Current Data Parameters
NAME r_fry.rir264
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060316
Time 11.01
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 10321.3
DW 19.900 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

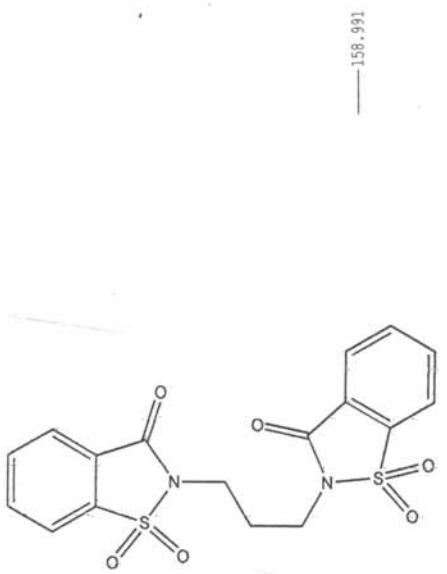
======== CHANNEL f1 ========
NUC1 13C
P1 7.55 usec
PL1 6.00 dB
SFO1 100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 0.00 dB
PL12 21.00 dB
PL13 26.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


NTH273
O
N
S
O
O
O
S
N
O
O
O
8a
200
150
100
50
0
PPM

RIR 372 (IPA insol.)



8b

Current Data Parameters

NAME	rir372
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020206
Time	12.36
INSTRUM	DRX500
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	879
DS	2
SWH	35211.270 Hz
FIDRES	1.074563 Hz
AQ	0.4653556 sec
RG	7298.2
DW	14.200 usec
DE	5.50 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.50 usec
PL1	5.00 dB
SFO1	125.7715724 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	-3.00 dB
PL12	16.00 dB
PL13	16.00 dB
SFO2	500.1327507 MHz

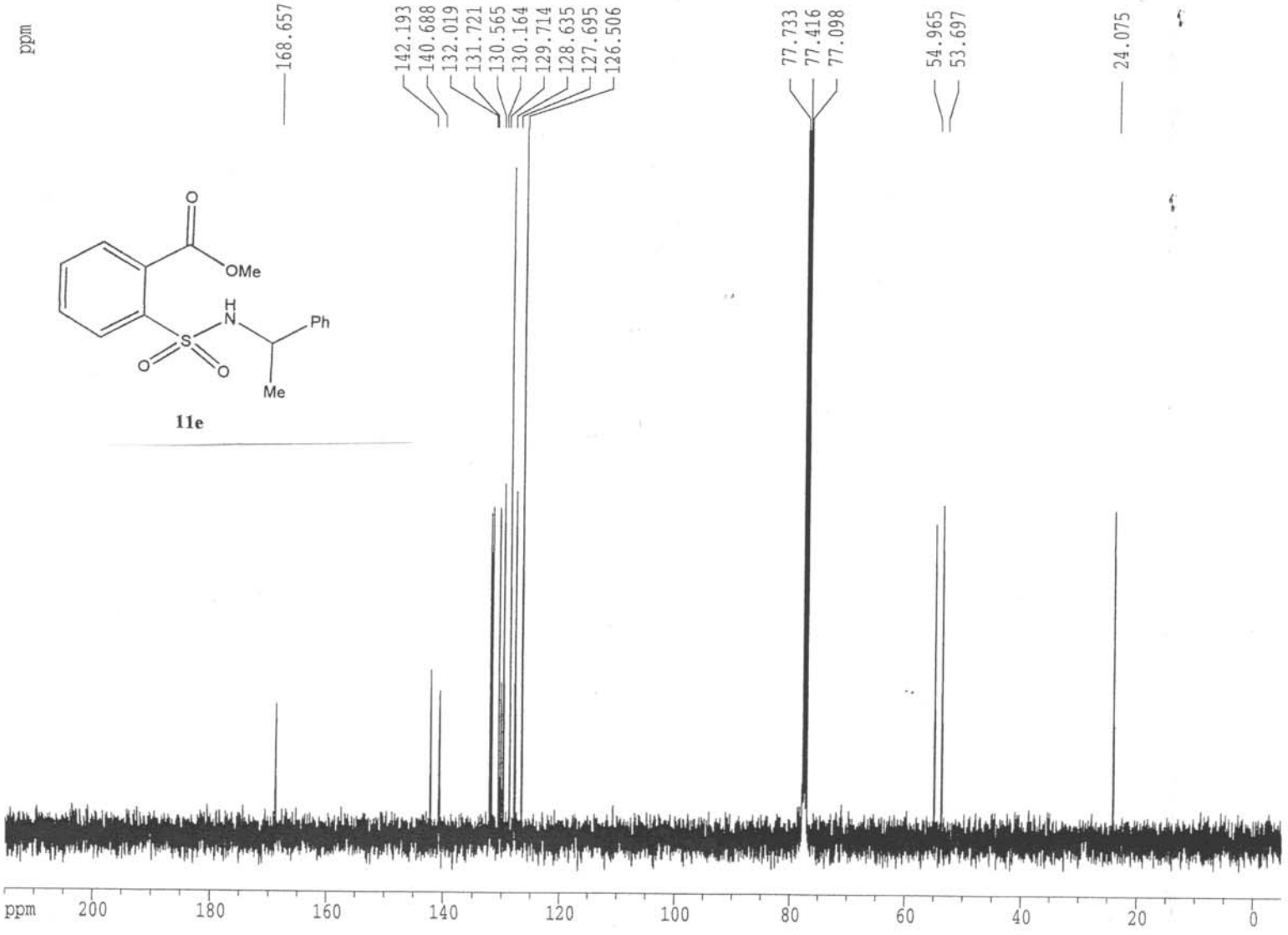
F2 - Processing parameters

SI	16384
SF	125.7577845 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

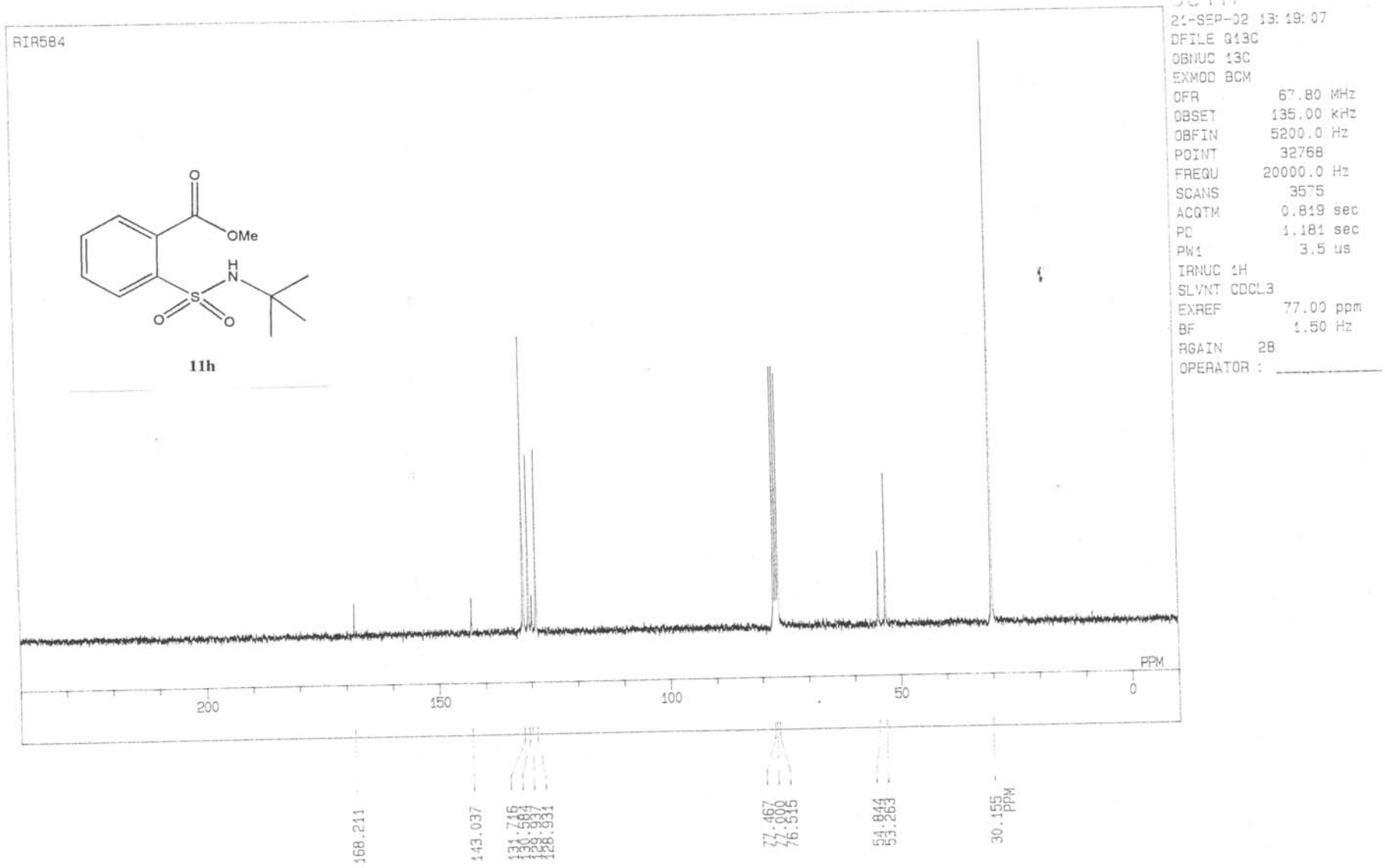
CX	30.00 cm
F1P	245.000 ppm
F1	30810.66 Hz
F2P	-20.000 ppm
F2	-2515.16 Hz
PPMCM	8.83333 ppm/cm
HZCM	1110.86035 Hz/cm

User/Group Robinson/Pfaltz
RIR 440c



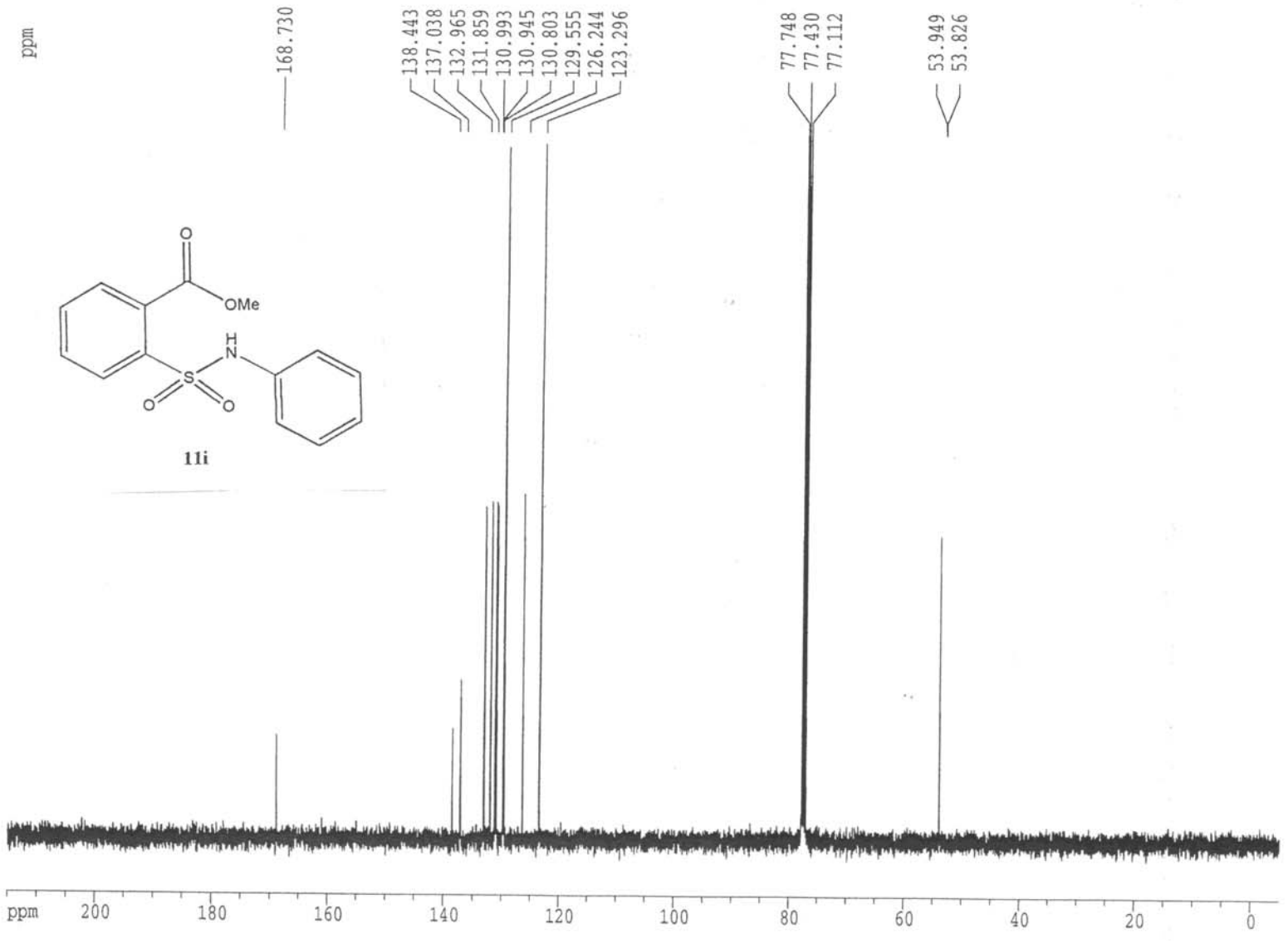
11e

```
Current Data Parameters
NAME                 pcxrir.031
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20020420
Time                       2.49
INSTRUM                  dpx400
PROBHD    5 mm QNP 1H/13
PULPROG                 zgdc30
TD                       65536
SOLVENT                  CDC13
NS                         384
DS                          16
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                        8192
DW                      19.900 usec
DE                       6.00 usec
TE                      300.0 K
D1                  2.20000005 sec
d11                 0.03000000 sec

============ CHANNEL f1 ============
NUC1                       13C
P1                        9.90 usec
PL1                      -3.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 ============
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      13.00 dB
PL12                     13.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                       65536
SF                  100.6127290 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       22.00 cm
F1P                     215.000 ppm
F1                    21631.74 Hz
F2P                    -5.000 ppm
F2                    -503.06 Hz
PPMCM                 10.00000 ppm/cm
HZCM                1006.12732 Hz/cm
```


RIR584
OMe
H
N
S
O
O
11h
200
150
100
50
0
PPM
168.211
143.037
131.716
130.584
130.037
129.837
128.931
77.467
77.000
76.515
54.844
53.263
30.155
PPM
13CFH
21-SEP-02 13:19:07
DFILE Q13C
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 3575
ACQTM 0.819 sec
PD 1.181 sec
PW1 3.5 us
IRNUC 1H
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.50 Hz
RGAIN 28
OPERATOR :

User/Group Robinson/Pfaltz

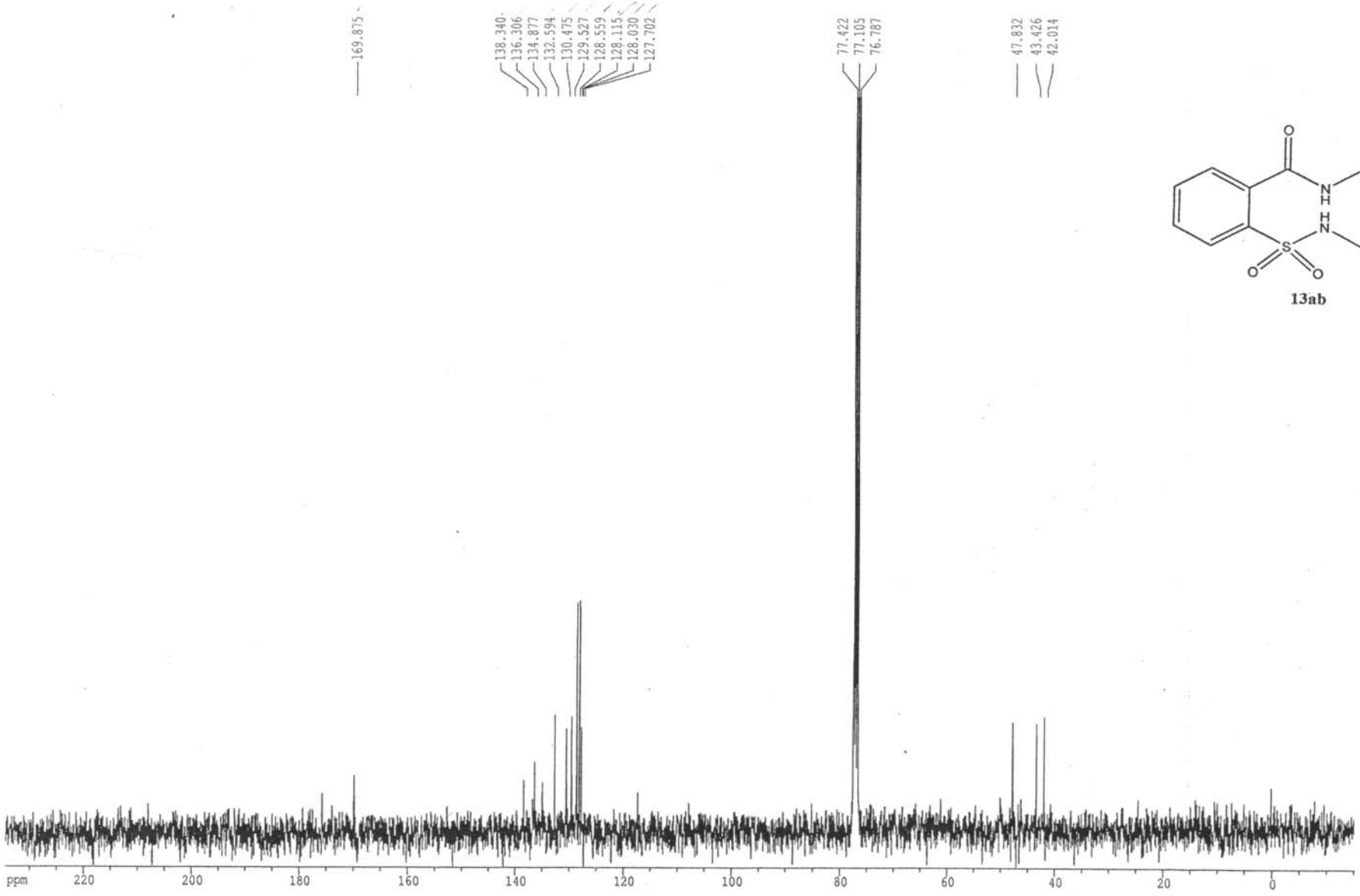
RIR 439



11i

```
Current Data Parameters
NAME          pcxrir.028
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20020419
Time               22.40
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/13
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                   16
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                 8192
DW               19.900 usec
DE                6.00 usec
TE               300.0 K
D1           2.20000005 sec
d11          0.03000000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                 9.90 usec
PL1               -3.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              13.00 dB
PL12             13.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127290 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                22.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12732 Hz/cm
```

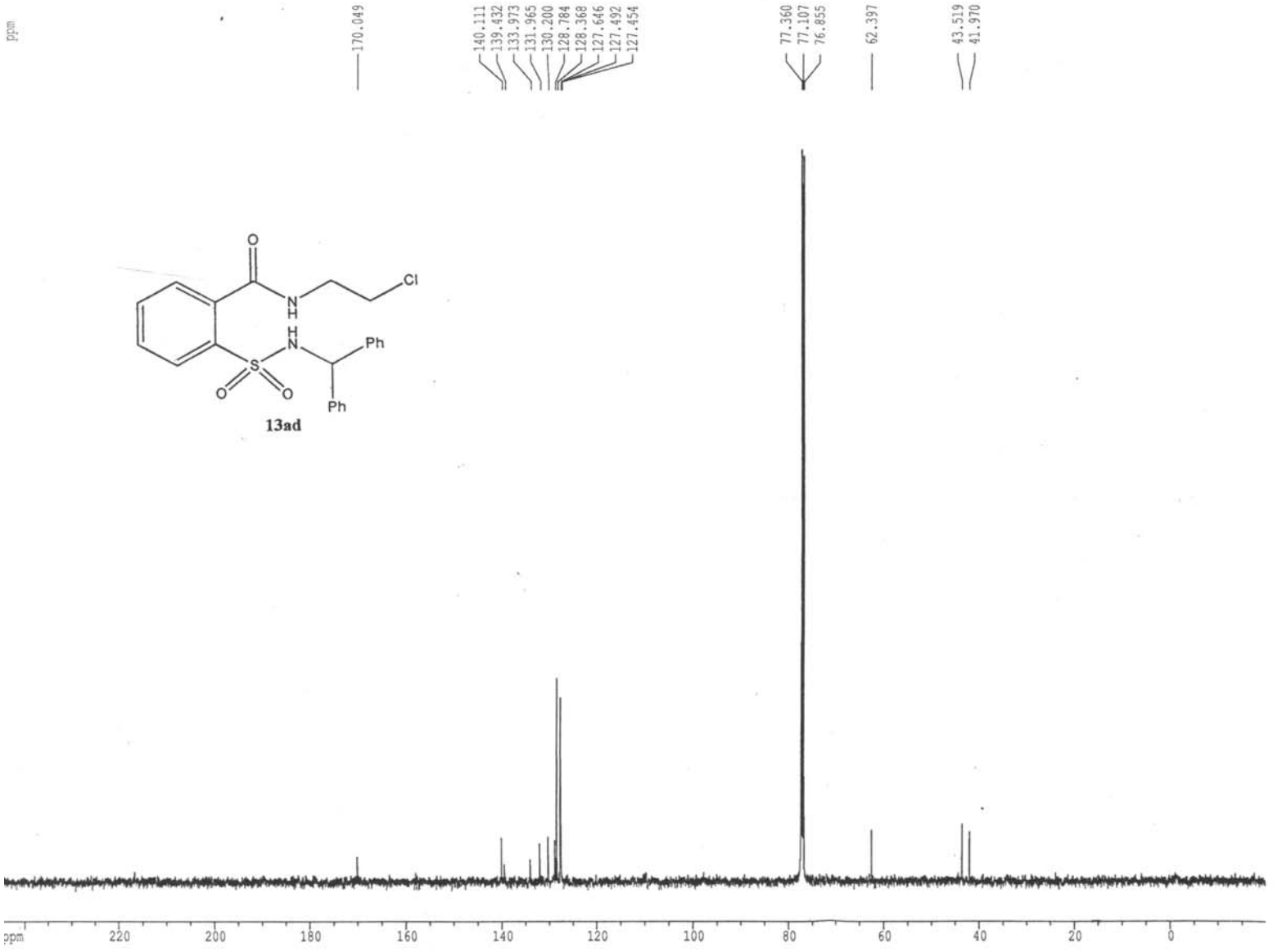
RIR 306


169.875
138.340
136.306
134.877
132.594
130.475
129.527
128.559
128.115
128.030
127.702
77.422
77.105
76.787
47.832
43.426
42.014
ppm
220
200
180
160
140
120
100
80
60
40
20
0

O
N
H
Cl
H
N
Ph
S
O
O

13ab

RIR 377



Current Data Parameters

NAME	rir377
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020212
Time	12.48
INSTRUM	DRX500
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1083
DS	2
SWH	35211.270 Hz
FIDRES	1.074563 Hz
AQ	0.4653556 sec
RG	7298.2
DW	14.200 usec
DE	5.50 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.50 usec
PL1	5.00 dB
SFO1	125.7715724 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	-3.00 dB
PL12	16.00 dB
PL13	16.00 dB
SFO2	500.1327507 MHz

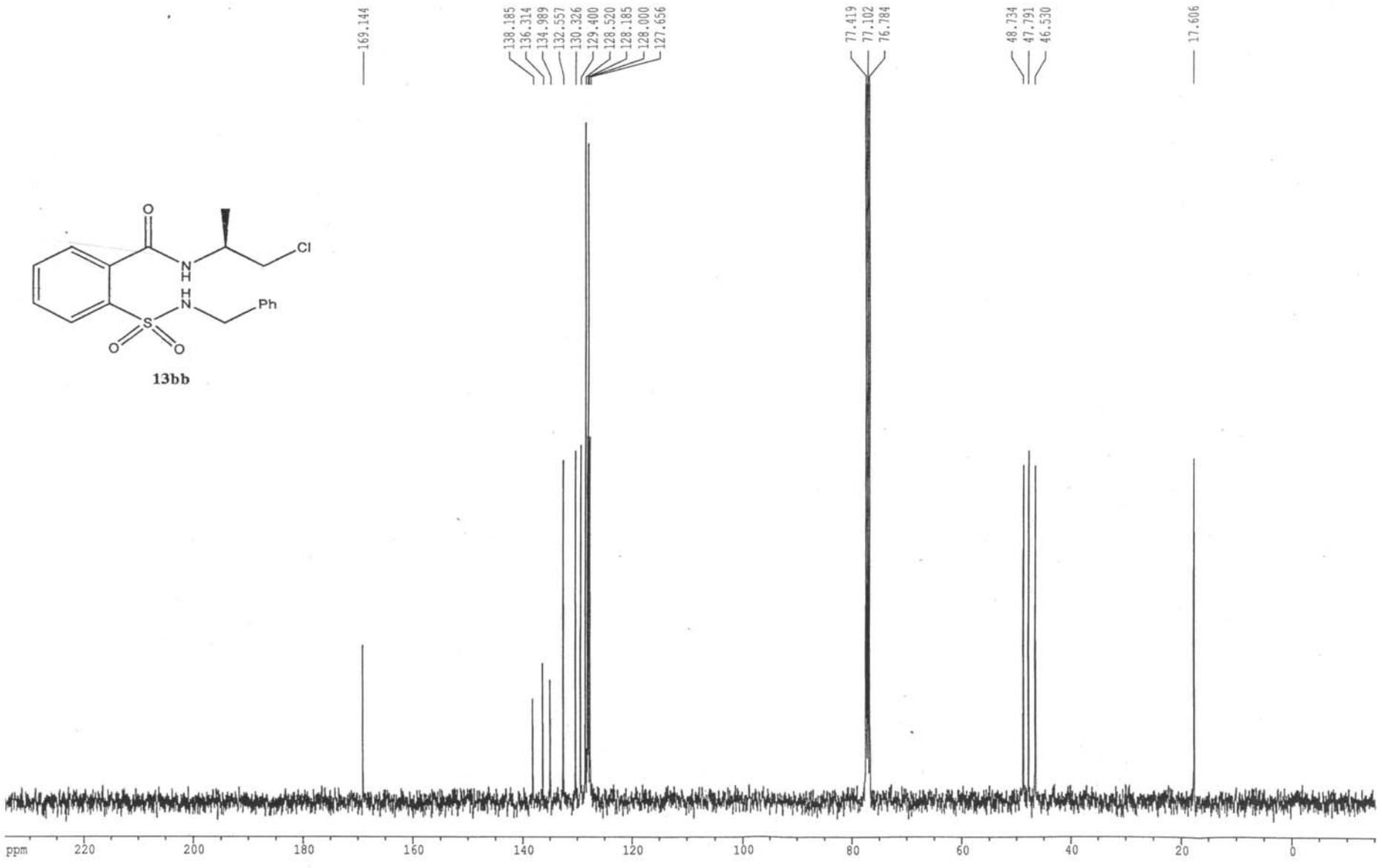
F2 - Processing parameters

SI	16384
SF	125.7577823 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

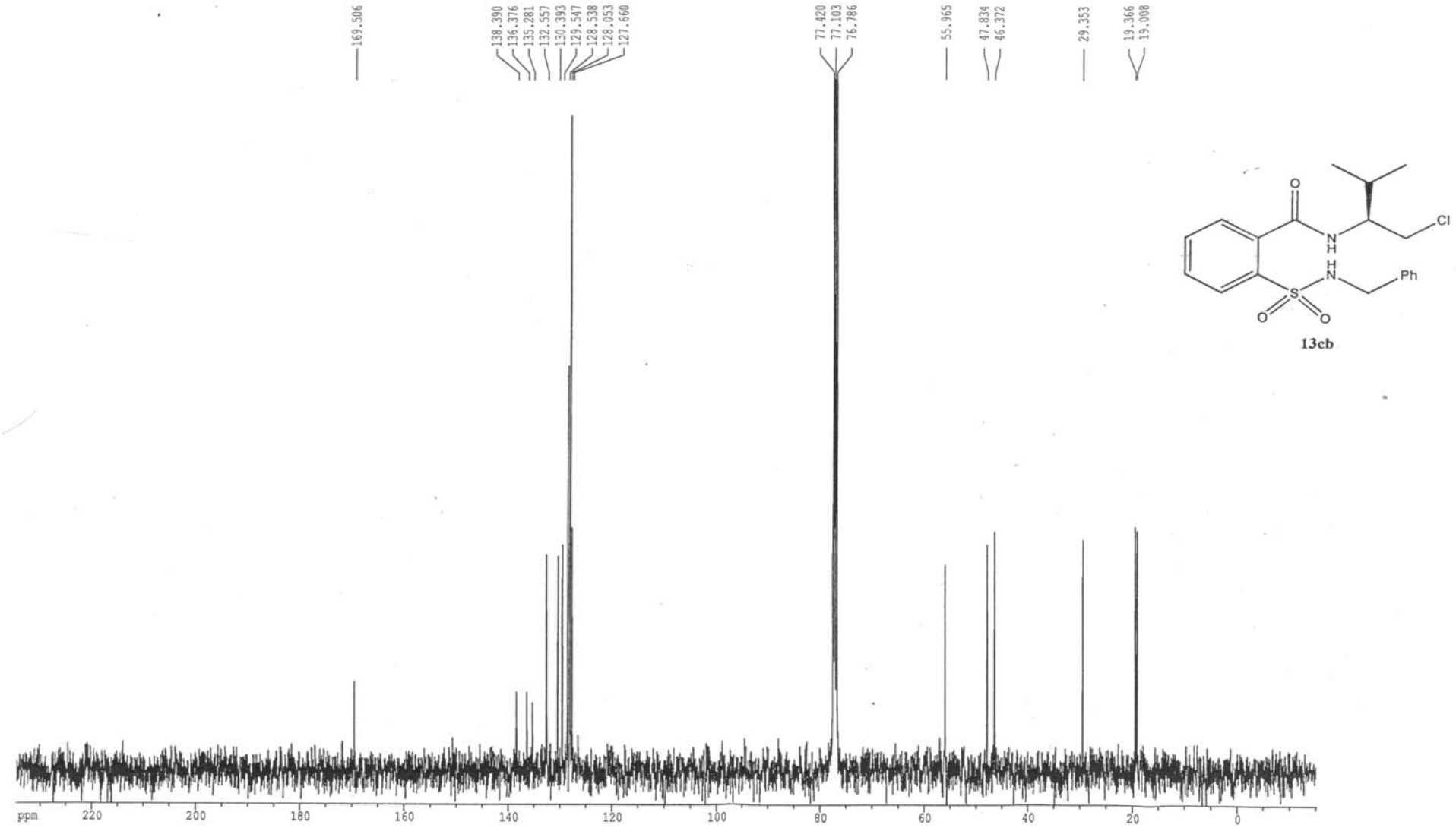
CX	30.00 cm
F1P	245.000 ppm
F1	30810.66 Hz
F2P	-20.000 ppm
F2	-2515.16 Hz
PPMCM	8.83333 ppm/cm
HZCM	1110.86035 Hz/cm

RIR 356


169.144
138.185
136.314
134.989
132.557
130.326
129.400
128.520
128.185
128.000
127.656
77.419
77.102
76.784
48.734
47.791
46.530
17.606
O
N
H
Cl
HN
Ph
S
O
O
ppm
220
200
180
160
140
120
100
80
60
40
20
0

13bb

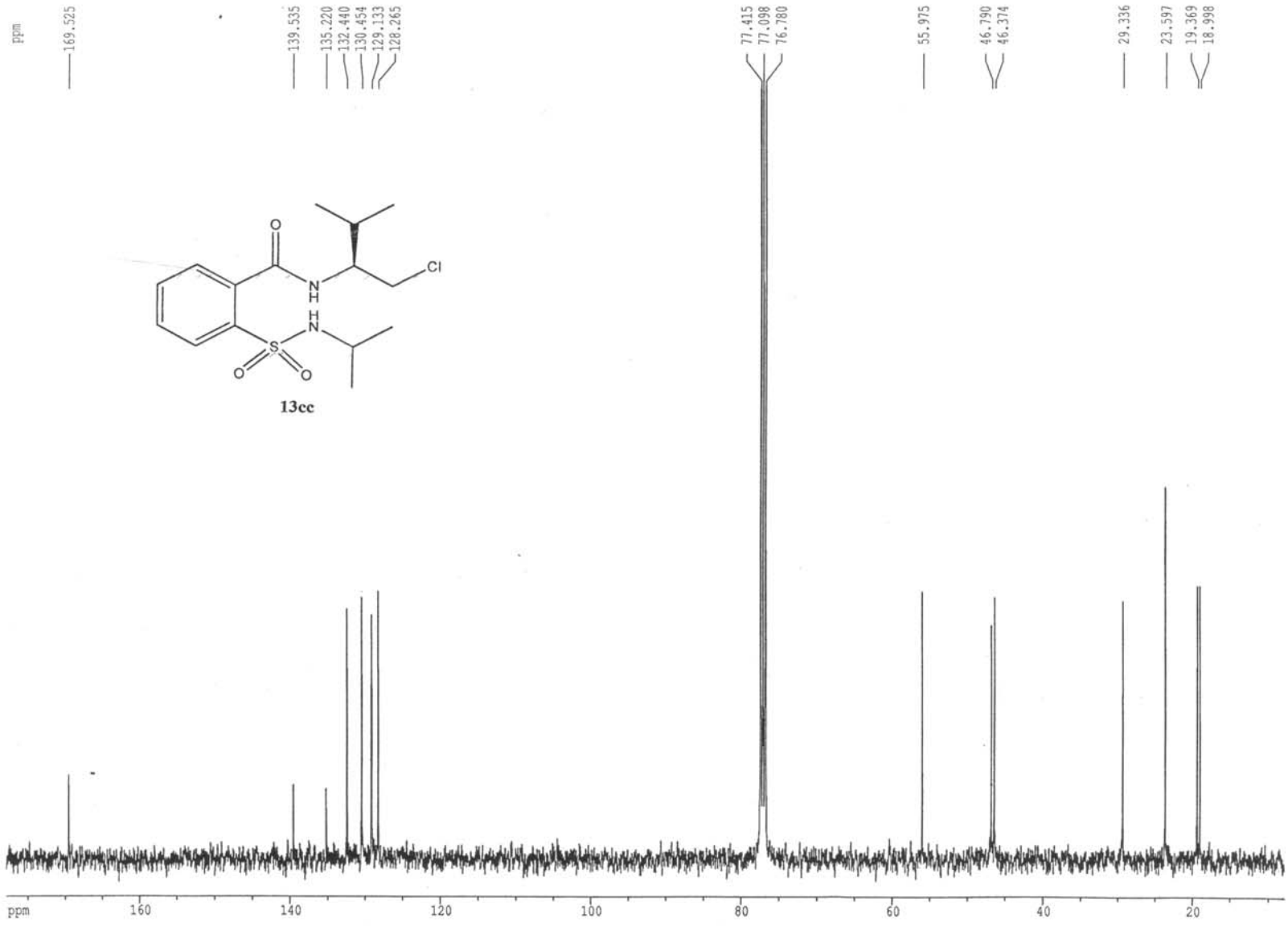
RIR 341


169.506
138.390
136.376
135.281
132.557
130.393
129.547
128.538
128.053
127.660
77.420
77.103
76.786
55.965
47.834
46.372
29.353
19.366
19.008
ppm
220
200
180
160
140
120
100
80
60
40
20
0

O
N
H
N
H
Cl
Ph
S
O
O

13cb

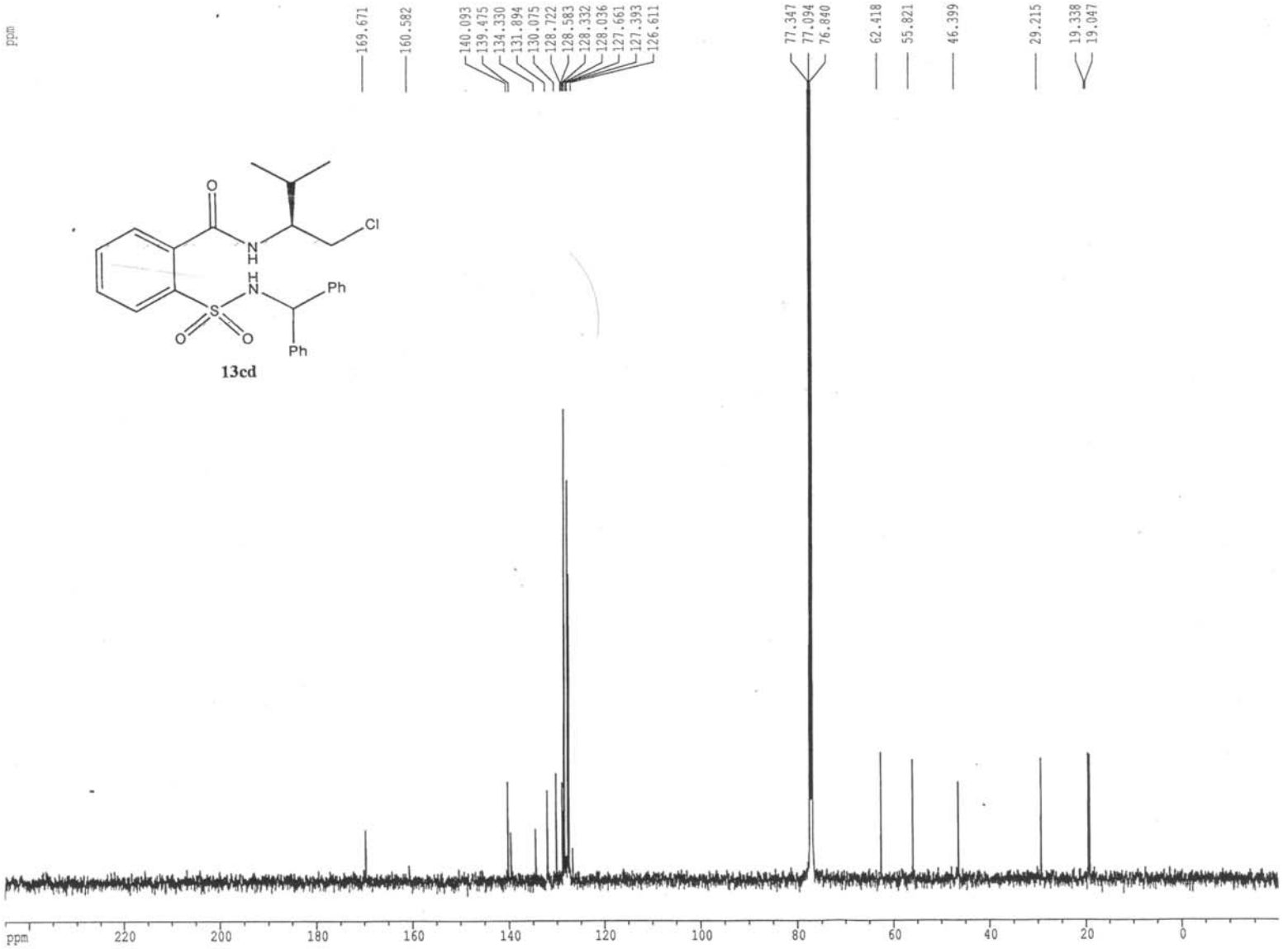
RIR 595



13cc

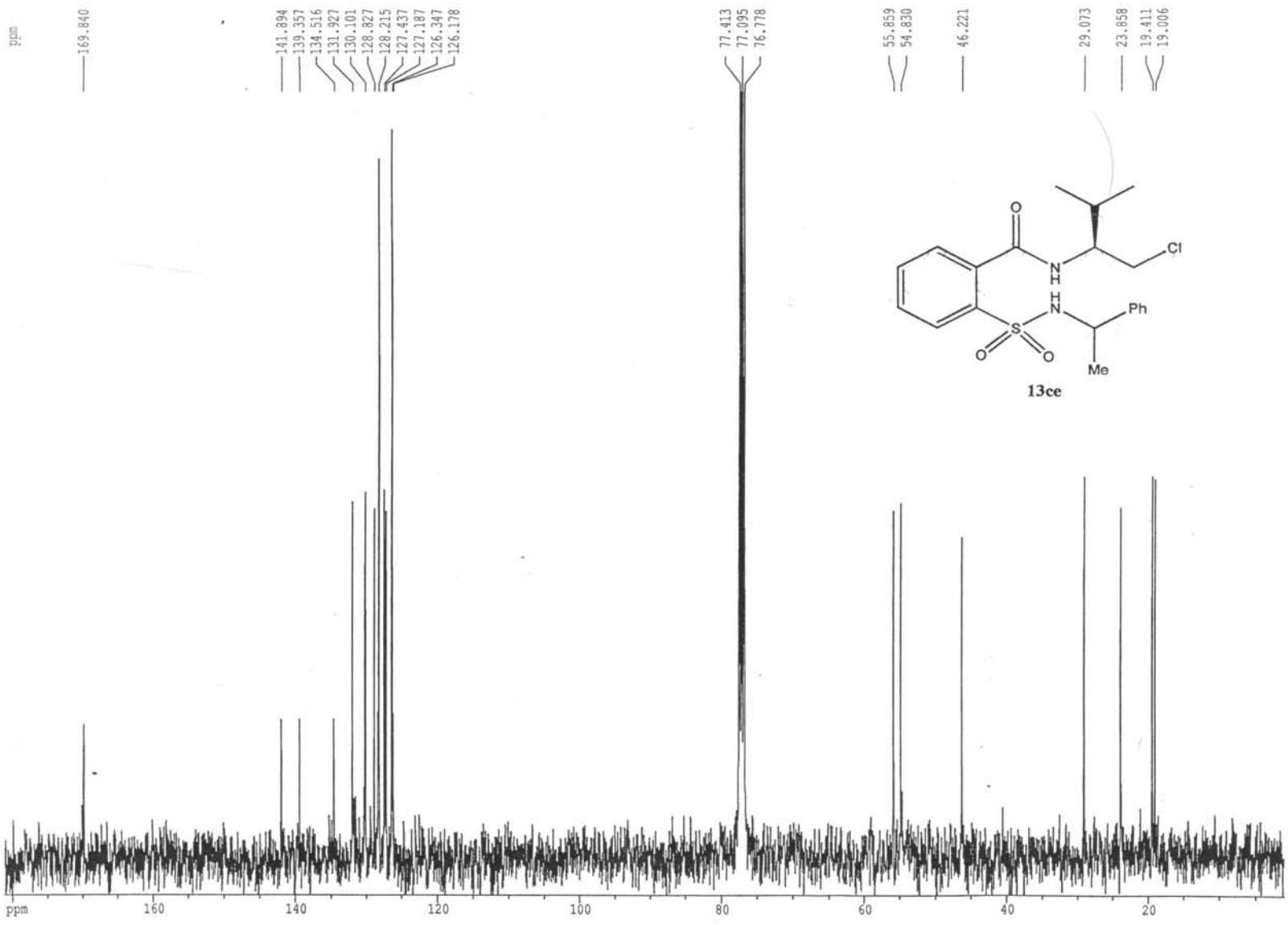
```
Current Data Parameters
NAME                 rir595
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_             20021110
Time                 16.31
INSTRUM              av400
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      645
DS                        2
SWH              25125.629 Hz
FIDRES           0.766773 Hz
AQ               0.6521332 sec
RG                    16384
DW                 19.900 usec
DE                 20.00 usec
TE                 300.0 K
D1              1.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 13.00 usec
PL1                -0.10 dB
SFO1          100.6237959 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 4.00 dB
PL12               25.58 dB
PL13               29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127615 MHz
WDW                     EM
SSB                      0
LB                  3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   30.00 cm
CY                   26.34 cm
F1P                177.851 ppm
F1               17894.08 Hz
F2P                  7.798 ppm
F2                 784.58 Hz
PPMCM             5.66843 ppm/cm
HZCM            570.31689 Hz/cm
```

RIR 390




```
Current Data Parameters
NAME                  rir390
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20020228
Time                  18.23
INSTRUM               DRX500
PROBHD      5 mm Multinu
PULPROG               zgpg30
TD                     32768
SOLVENT                CDCl3
NS                      1370
DS                         2
SWH               35211.270 Hz
FIDRES             1.074563 Hz
AQ                 0.4653556 sec
RG                      6502
DW                    14.200 usec
DE                     5.50 usec
TE                    300.0 K
D1                 1.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

========== CHANNEL f1 ==========
NUC1                      13C
P1                       9.50 usec
PL1                      5.00 dB
SFO1              125.7715724 MHz

========== CHANNEL f2 ==========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  115.00 usec
PL2                     -3.00 dB
PL12                    16.00 dB
PL13                    16.00 dB
SFO2              500.1327507 MHz

F2 - Processing parameters
SI                        16384
SF                125.7577845 MHz
WDW                        EM
SSB                         0
LB                       3.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      30.00 cm
F1P                    245.000 ppm
F1                   30810.66 Hz
F2P                   -20.000 ppm
F2                   -2515.16 Hz
PPMCM                 8.83333 ppm/cm
HZCM                1110.86035 Hz/cm
```

RIR 592

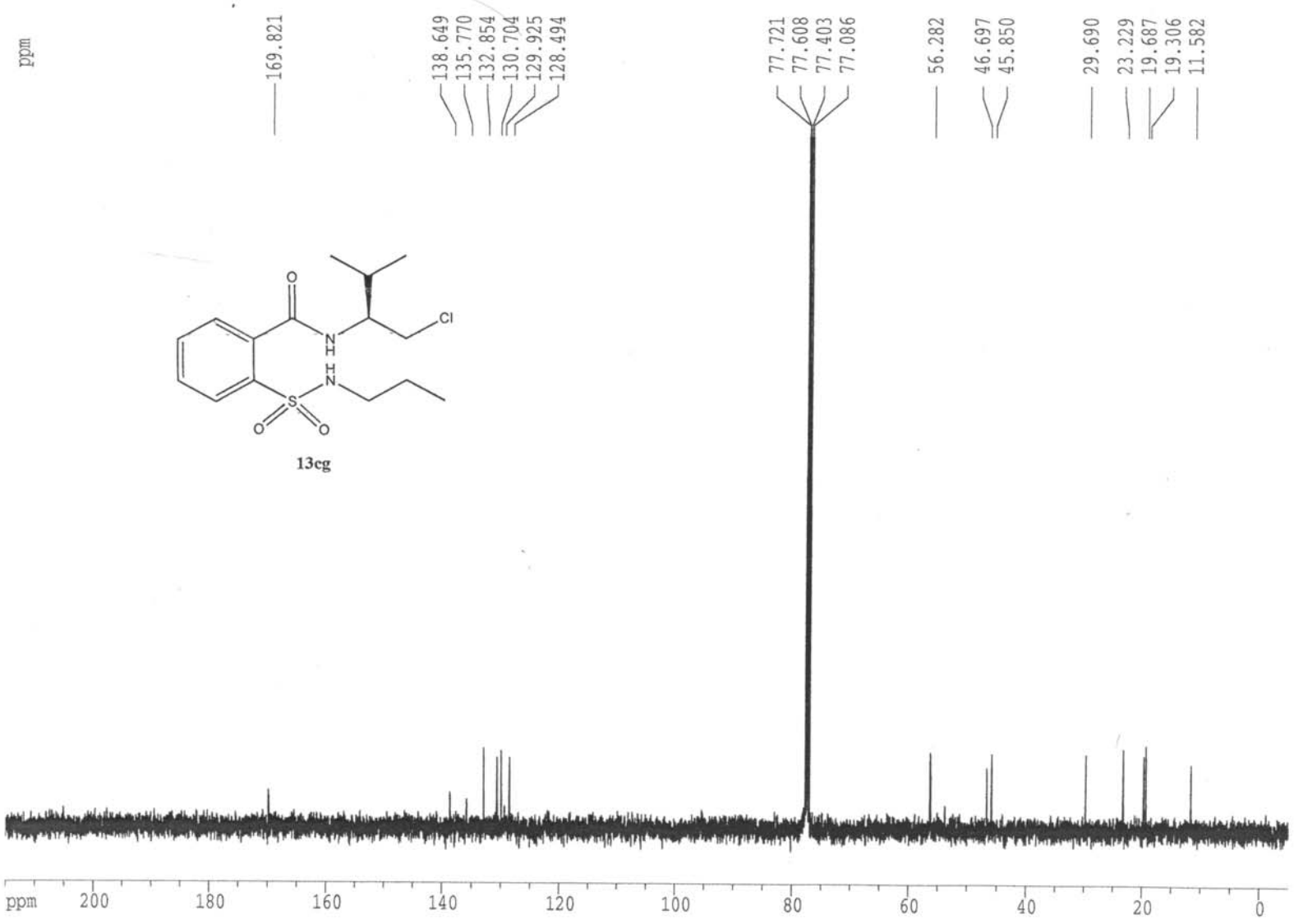


13ce

```
Current Data Parameters
NAME                 rir592
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20021027
Time                 17.41
INSTRUM              av400
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                    32768
SOLVENT               CDC13
NS                      672
DS                        2
SWH              25125.629 Hz
FIDRES           0.766773 Hz
AQ               0.6521332 sec
RG                    16384
DW                19.900 usec
DE                20.00 usec
TE                300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 13.00 usec
PL1                -0.10 dB
SFO1         100.6237959 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 2.90 dB
PL12               25.50 dB
PL13               29.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127622 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   30.00 cm
CY                   19.00 cm
F1P                181.087 ppm
F1               18219.66 Hz
F2P                  0.935 ppm
F2                  94.04 Hz
PPMCM              6.00508 ppm/cm
HZCM             604.18732 Hz/cm
```

User/Group Robinson/Pfaltz

RIR 426



13cg

Current Data Parameters

NAME	pcxrir.023
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020417
Time	0.08
INSTRUM	dpx400
PROBHD	5 mm QNP 1H/13
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	384
DS	16
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	8192
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.20000005 sec
d11	0.03000000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.90 usec
PL1	-3.00 dB
SFO1	100.6237959 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	13.00 dB
PL12	13.00 dB
SFO2	400.1316005 MHz

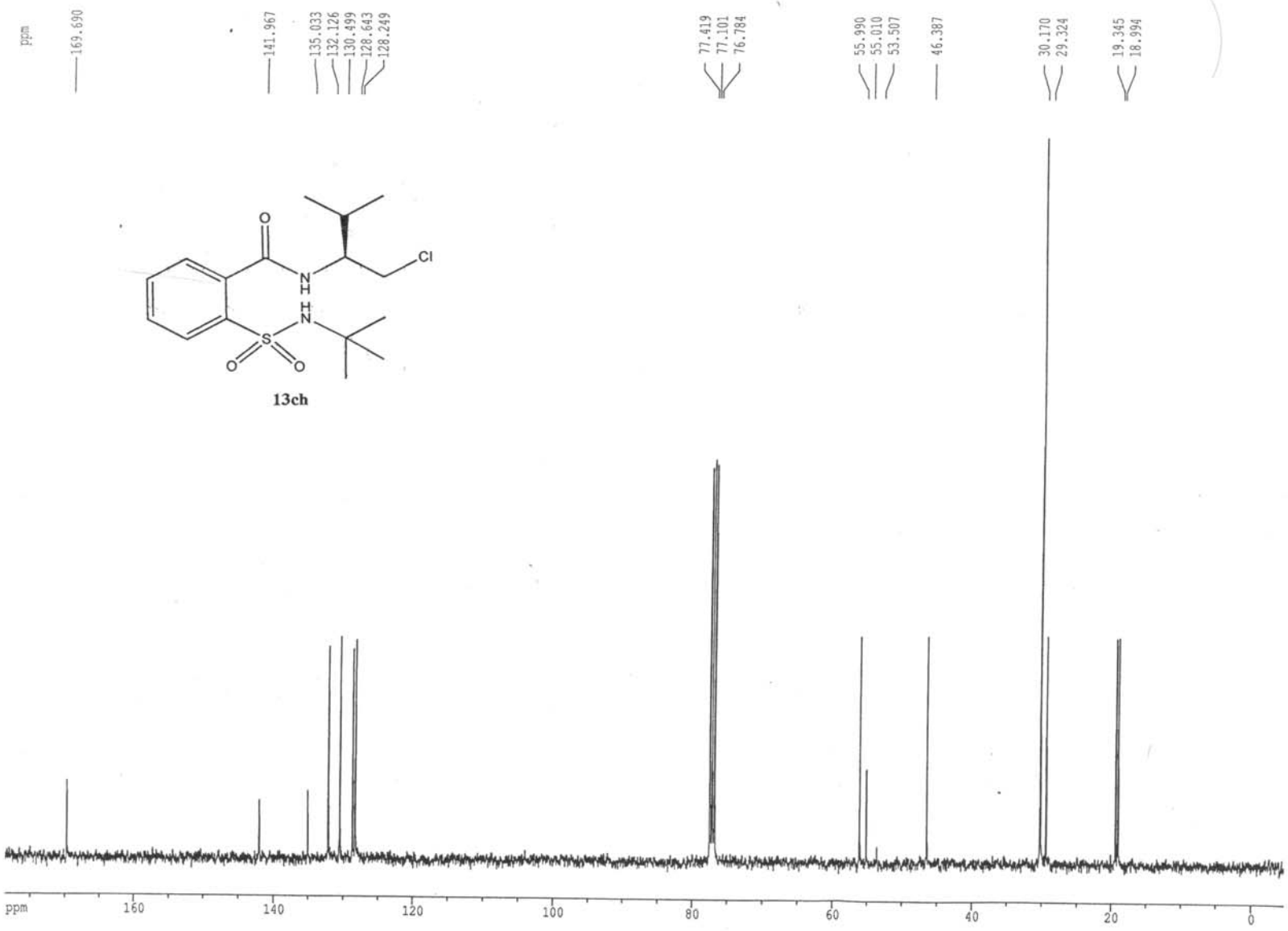
F2 - Processing parameters

SI	65536
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	22.00 cm
F1P	215.000 ppm
F1	21631.74 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.00000 ppm/cm
HZCM	1006.12732 Hz/cm

RIR 593



Current Data Parameters

NAME	rir593
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20021103
Time	12.06
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	499
DS	2
SWH	25125.629 Hz
FIDRES	0.766773 Hz
AQ	0.6521332 sec
RG	16384
DW	19.900 usec
DE	20.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	13.00 usec
PL1	-0.10 dB
SFO1	100.6237959 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.00 dB
PL12	25.58 dB
PL13	29.00 dB
SFO2	400.1316005 MHz

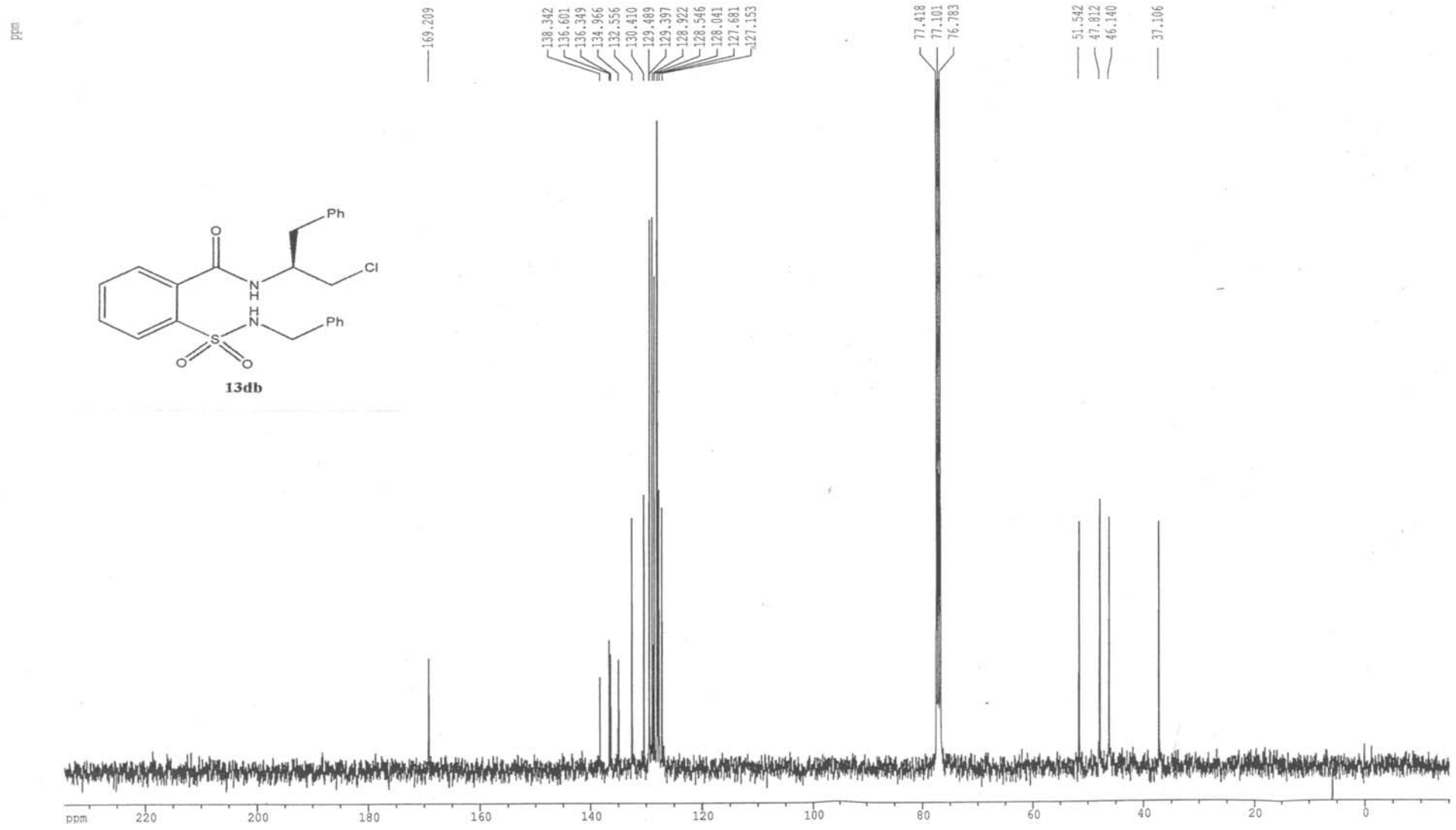
F2 - Processing parameters

SI	32768
SF	100.6127622 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	19.00 cm
F1P	178.574 ppm
F1	17966.79 Hz
F2P	-4.724 ppm
F2	-475.31 Hz
PPMCM	6.10993 ppm/cm
HZCM	614.73669 Hz/cm

RIR 357


ppm
169.209
138.342
136.601
136.349
134.966
132.556
130.410
129.489
129.397
128.922
128.546
128.041
127.681
127.153
77.418
77.101
76.783
51.542
47.812
46.140
37.106
Ph
Cl
Ph
O
O
O
N
H
N
H
S
ppm
220
200
180
160
140
120
100
80
60
40
20
0

13db

References

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