Electronic Supporting Information for

Facile oxidation of electron-poor benzo[*b*]thiophenes to the corresponding sulfones with an aqueous solution of H_2O_2 and P_2O_5

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General Synthetic Methods

Melting points were measured using a digital melting point apparatus (Electrothermal). IR spectra were recorded on a Perkin-Elmer Spectrum 1000 FT IR Spectrometer. ^{1}H- and ^{13}C-NMR spectra were acquired at 300 K using a Bruker Avance NMR spectrometer at 400 and 100 MHz, respectively. Chemical shifts are reported relative to TMS (δ = 0.0 ppm), and signals are designated as s (singlet), d (doublet), t (triplet), dt (double triplet), dd (doublet of doublets), ddd (double doublet of doublets) or m (multiplet), with coupling constants given in Hertz (Hz). Mass spectral data were collected using a Waters Micromass ZQ instrument coupled to a Waters 2695 HPLC with a Waters 2996 PDA. Waters Micromass ZQ parameters used were: Capillary (kV): 3.38, Cone (V): 35, Extractor (V): 3.0, Source temperature (°C): 100, Desolvation Temperature (°C): 200, Cone flow rate (L/h): 50, De-solvation flow rate (L/h): 250. High resolution mass spectroscopy data were recorded on a Waters Micromass QTOF Global in positive W-mode using metal-coated borosilicate glass tips to introduce the samples into the instrument. Thin Layer Chromatography (TLC) was performed on silica gel aluminium plates (Merck 60, F_{254}), and flash chromatography utilised silica gel (Merck 60, 230-400 mesh ASTM). 60% hydrogen peroxide aqueous solution was purchased from Fisher Scientific Inc. and used within one week. All other reagents were purchased from Sigma-Aldrich Co.

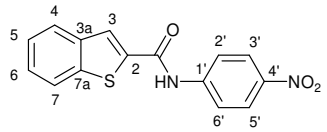
Synthesis of the benzo[*b*]thiophenecarboxamides

Benzo[*b*]thiophenecarboxamides used as substrates in the oxidation reactions were synthesised *via* three different routes.

The nitro-substituted starting materials employed in the oxidation reactions for entries **3**, **9**, and **12** of **Table 1** were synthesised according to representative procedure as follows:

Oxalyl chloride (293 µl, 3.36 mmol, 2.2 eq) was added to a magnetically stirred solution of benzo[*b*]thiophene-5-carboxylic acid (300 mg, 1.68 mmol, 1.1 eq) in anhydrous DCM (20 ml) at room temperature. Triethylamine (1.28 ml, 9.24 mmol, 5.5 eq) was then added to the mixture and the reaction was stirred at room temperature for 15 min under nitrogen atmosphere. A solution of 4-nitroaniline (210 mg, 1.52 mmol, 1.0 eq) in DCM (15 ml) was then cannulated into the flask and the reaction mixture was allowed to stir at room temperature for 18 h. The solution was diluted with DCM (40 ml) and washed with water (2 x 50 ml). The organic phase was dried over $MgSO_4$ and concentrated under reduced pressure to afford *N*-(4-nitrophenyl)benzo[*b*]thiophene-5-carboxamide as brown solid (360 mg, 80%).

N-(4-nitrophenyl)benzo[*b*]thiophene-2-carboxamide

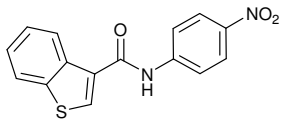


Molecular Weight: 298.32

Yellow crystals, 90% yield. m.p. 279-281 ºC. IR (ATR, ν_{max}/cm^{-1}): 3386 (sharp NH band), 3049, 2956, 1672, 1607, 1593, 1540, 1496, 1482, 1403, 1326, 1300, 1246, 1176, 1110, 1037, 879, 844, 748, 689, 639, 611. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28-

7.32 (m, 2 H, H5 and H6), 7.72-7.77 (m, 2 H, H4 and H7), 7.91 (d, 2 H, J = 7.3 Hz, H2' and H6'), 8.07 (d, 2 H, J = 7.4 Hz, H3' and H5'), 8.09 (s, 1 H, H3), 10.28 (s, 1 H, NH). ^{13}C NMR (100 MHz, $CDCl_3$) : δ 119.8 (C2' and C6'), 122.7 (C7), 124.6 (C3' and C5'), 124.9 (C4), 125.1 (C5), 126.5 (C3), 126.6 (C7), 139.0 (C4a), 141.3 (C4'), 141.5 (C7a), 143.2 (C1'), 144.3 (C2), 162.0 (C=O) Note: d_6-DMSO was added to the NMR tube for dissolving the analyte. MS (ESI^+) *m/z* (relative intensity): 298.68 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 299.0490; Measured mass $[M + H]^{\cdot+}$, 299.0505 (δ 5 ppm). Elem. Anal. calculated for $C_{15}H_{10}N_2O_3S$: C, 60.39; H, 3.38; N, 9.39%. Found: C, 60.11; H, 3.50; N, 9.28%.

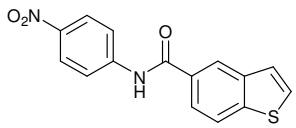
N-(4-nitrophenyl)benzo[*b*]thiophene-3-carboxamide



Molecular Weight: 298.32

Brown solid, 40% yield. IR (ATR: ν_{max}/cm^{-1}); 3386, 3110, 1560, 1538, 1488, 1300, 1250, 1215, 1175, 1151, 1109, 1055, 956, 842, 763, 744, 718, 686. ^{1}H NMR (d_6-DMSO, 400 MHz): rotamers δ 7.43-7.54 (m, 2 H, H5 and H6), 8.07 (d, 2 H, J = 9.2 Hz, H2' and H6'), 8.11 (dd, 1 H, J = 1.0, 7.0 Hz, H7), 8.32 (d, 2 H, J = 9.2 Hz, H3' and H5'), 8.43 (d, 1 H, J = 7.6 Hz, H4), 8.71 (s, 1 H, H2), 10.93 (s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 100 MHz): rotamers δ 119.6 (C2' and 6'), 122.9 (C7), 124.1 (C4), 124.9 (C3' and C5'), 125.2 and 125.3 (C5 and C6), 130.2 (C3), 133.5 (C2), 136.9 (C3a), 139.4 (C7a), 142.3 (C4'), 145.4 (C1'), 162.2 (C=O). MS (ESI^+) *m/z* (relative intensity): 299.03 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 299.0490; Measured mass $[M + H]^{\cdot+}$, 299.0476 (δ 4.7 ppm).

N-(4-nitrophenyl)benzo[*b*]thiophene-5-carboxamide



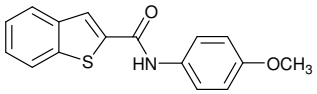
Molecular Weight: 298.32

Brown solid, 80% yield. IR (ATR: ν_{max}/cm^{-1}); 3282, 3080, 1775, 1654, 1594, 1498, 1405, 1325, 1248, 1150, 1112, 1049, 846, 745, 689. ^{1}H NMR (d_6-DMSO, 400 MHz): rotamers δ 7.63 (d, 1 H, J = 5.4 Hz, H3), 7.92 (d, 1 H, J = 5.4 Hz, H2), 7.95 (dd, 1 H, J = 8.5, 1.6 Hz, H6), 8.10 (d, 2 H, J = 9.1 Hz, H2' and H6'), 8.20 (d, 1 H, J = 8.5 Hz, H7), 8.29 (d, 2 H, J = 9.1 Hz, H3' and H5'), 8.55 (d, 1 H, J = 1.3 Hz, H4), 10.92 (s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 100 MHz): rotamers δ 119.8 (C2' and 6'), 122.7 (C7), 123.4 (C6), 123.6 (C4), 124.5 (C3), 124.8 (C3' and C5'), 129.3 (C2), 130.5 (C5), 139.1 (C3a), 142.4 (C7a), 142.6 (C4'), 145.6 (C1'), 166.4 (C=O). MS (ESI^+) *m/z* (relative intensity): 298.50 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 299.0478; Measured mass $[M + H]^{\cdot+}$, 299.0490 (δ 4 ppm).

Benzo[*b*]thiophene-2-carboxamides used as starting material in oxidation reactions for entries **2** and **4** of **Table 1** were synthesised from benzothiophene-2-carbonylchloride according to representative procedure as follows:

Benzo[*b*]thiophene-2-carbonyl chloride (500 mg, 2.54 mmol, 1.0 eq) was added in portions to a solution of 4-nitroaniline (315 mg, 2.3 mmol, 0.9 eq) and TEA (0.68 ml, 5.0 mmol, 2.0 eq) in anhydrous THF (10 ml) at room temperature under magnetic stirring. The reaction mixture was stirred for 4 h at room temperature. The product precipitated out of solution and was collected by filtration, and washed with small amounts of THF to give *N*-(4-methoxyphenyl)benzo[*b*]thiophene-2-carboxamide as gray solid (548 mg, 76%).

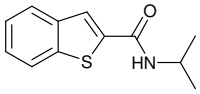
N-(4-methoxyphenyl)benzo[*b*]thiophene-2-carboxamide



Molecular Weight: 283.34

Gray solid, 76% yield. m.p. 226-229 ºC. IR (ATR, ν_{max}/cm^{-1}): 3344, 2977, 2946, 1634, 1597, 1514, 1476, 1397, 1243, 1173, 1031, 820, 808, 754, 734, 634. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 3 H, H-(OCH_3)), 6.86 (d, 2 H, *J* = 8.8 Hz, H3' and H5'), 7.38 (qt, 2 H, *J* = 6.8 Hz, H5 and H6), 7.68 (d, 2 H, *J* = 8.8 Hz, H2' and H6'), 7.84 (d, 2 H, *J* = 7.3 Hz, H4 and H7), 8.18 (s, 1 H, H3), 8.77 (s, 1 H, NH). ^{13}C NMR (100 MHz, $CDCl_3$) : δ 55.5 (C-(OCH_3)), 114.1 (C3' and C5'), 122.4 (C2' and C6'), 122.6 (C7), 124.8 (C4), 125.2 (C5), 125.9 (C3), 126.3 (C6), 131.2 (C1'), 139.4 (C2), 139.5 (C4a), 141.1 (C7a), 156.5 (C4'), 160.5 (C=O). MS (ESI^+) *m/z* (relative intensity): 283.70 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 284.0745; Measured mass $[M + H]^{\cdot+}$, 284.0734 (δ 4 ppm).

N-isopropylbenzo[*b*]thiophene-2-carboxamide

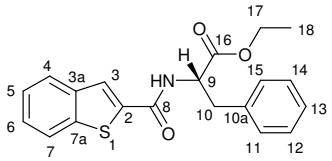


Molecular Weight: 219.3

White solid, 80% yield. m.p. 148–149 ^{o}C. IR (ATR, ν_{max}/cm^{-1}): 3264, 2972, 1625, 1549, 1455, 1287, 1198, 841, 742. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.19 (m, 6H, H-$(CH_3)_2$), 4.22 (s, 1H, H-(CH)), 5.88 (s, 1H, NH), 7.30 (m, 2H, H5 and H6), 7.74 (m, 2H, H4 and H7), 7.67 (s, 1H, H3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.8 (C-$(CH_3)_2$), 42.3 (C-(CH)), 122.7 (C3), 124.8 (C4), 124.9 (C7), 126.2 (C5 and C6), 138.9 (C3a), 139.1 (C7a), 140.7 (C2), 161.5 (C=O).

(*S*)-Ethyl 2-(benzo[*b*]thiophene-2-carboxamido)-3-phenylpropanoate (stating material forentry **6**) was synthesised according to Yoo and Li[1] as follows:

(*S*)-Ethyl 2-(benzo[*b*]thiophene-2-carboxamido)-3-phenylpropanoate



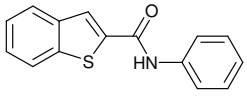
Molecular Weight: 353.43

T-Hydro® (2.1 ml, 6.5 mmol, 1.1 eq) and thianaphtene-2-carboxaldehyde (1.001 g, 6.17 mmol, 1 eq) were added to a mixture of CuI (11.21 mg, 0.06 mmol, 1 % mol), $AgIO_3$ (17.8 mg, 0.06 mmol, 1 % mol), *L*-phenylalanine ethyl ester hydrochloric acid (2.770 g, 9.49 mmol, 1.5 eq) and $CaCO_3$ (0.710 g, 7.10 mmol, 1.1 eq) in MeCN (2 ml). The mixture was allowed to stir at room temperature for 32 h. The mixture was then purified by flash chromatography EtOAc /hexane gradient to provide (*S*)-Ethyl 2-(benzo[*b*]thiophene-2-carboxamido)-3-phenylpropanoate as a colorless oil (1.7 g, 69%). IR (ATR, ν_{max}/cm^{-1}): 695, 716, 746, 766, 806, 863, 1042, 1081, 1112, 1180, 1219, 1277, 1366, 1427, 1450, 1538, 1643, 1733, 3266. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.31 (t, 3 H, *J* = 7.1 Hz, H18), 3.25 – 3.35, (dd, 2 H, *J* = 5.9 and 8.6 Hz, H10a and H10b), 4.23 – 4.28 (q, 2 H, *J* = 7.2 Hz, H17a and H17b), 5.07 – 5.12 (q, 1 H, *J* = 7.3 and 5.9 Hz, H9), 6.62 (d, 1 H, *J* = 7.3 Hz, NH), 7.18 (d, 2 H, *J* = 7.8, H11 and H15), 7.29 – 7.31 (m, 3 H, H13, H12 and H14), 7.40 – 7.44 (m, 2 H, *J* = 1.4, 1.7 and 7.1 Hz, H5 and H6), 7.75, (s, 1 H, H3), 7.84 (t, 2 H, *J* = 9.0, H4 and H7). ^{13}C NMR (100 MHz, $CDCl_3$) **:** δ 14.2 (CH_3), 38.0 (CH_2), 53.6 (C9), 61.8 (OCH_2), 122.7 (C7), 125.0 (C4), 125.1 (C3), 125.5 (C5), 126.5 (C6), 127.2 (C12 and C14), 128.6 (C13), 129.5 (C11 and C15), 135.7 (C10a), 137.9 (C3a), 139.0 (C7a), 141.0 (C2), 161.6 (NC=O), 171.3 (OC=O). MS (ESI $^+$) *m/z* (relative intensity): 160.97 ([*M* – Phe + H]$^+$ 100 %), 280.08 ([*M* – $COOCH_2CH_3$] 10 %), 376.09 ([*M* + Na]$^+$ 20%).

All other benzo[*b*]thiophenecarboxamides used as starting materials in oxidation reactions for entries **1**, **5**, **7**, **8**, **10**, and **11** of **Table 1** were synthesised from the commercially available benzo[*b*]thiophenecarboxilic acids using standard amide coupling conditions according to representative procedure as follows:

Aniline (105 μg, 1.15 mmol, 1.03 eq) was added to a solution of benzo[b]thiophene-3-carboxilic acid (200 mg, 1.12 mmol, 1.0 eq), *N*-(3-dimethylaminopropyl)-*N*-ethyl carbodiimide hydrochloride (257 mg, 1.34 mmol, 1.2 eq), and dimethylaminopyridine (27 mg, 0.22 mmol, 0.2 eq) in dry DCM (5 ml) at room temperature. The reaction mixture was magnetically stirred for 16 h at room temperature when a precipitate crashed out of solution. The solids were collect by filtration and washed with minimum DCM to give *N*-(4-methoxyphenyl)benzo[*b*]thiophene-3-carboxamide as a white solid (281 mg, 98 %).

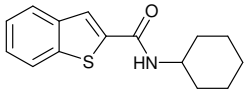
N-phenylbenzo[*b*]thiophene-2-carboxamide



Molecular Weight: 253.32

White solid, 72% yield. m.p. 205-207 ºC. IR (ATR, ν_{max}/cm^{-1}): 3375 (sharp NH band), 3049, 1638, 1595, 1522, 1438, 1315, 1244, 1187, 1080, 941, 869, 840, 752, 735, 685. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.14 (t, 1 H, *J* = 7.4 Hz, H4'), 7.41 (t, 2 H, *J* = 7.5 Hz, H3' and H5'), 7.50 (m, 2 H, H5 and H6), 7.79 (d, 2 H, *J* = 7.6 Hz, H2' and H6'), 8.02 (d, 1 H, *J* = 8.8 Hz, H6), 8.06 (d, 1 H, *J* = 9.0 Hz, H5), 8.38 (s, 1 H, H3), 10.50 (s, 1 H, NH). ^{13}C NMR (100 MHz, $CDCl_3$) : δ 120.3 (C2' and C6'), 122.8 (C7), 123.9 (C4'), 125.0 (C4), 125.8 (C5), 126.5 (C6), 128.7 (C3' and C5'), 138.6 (C1'), 139.1 (C4a), 140.0 (C7a), 140.5 (C2), 160.3 (C=O). MS (ESI^+) *m/z* (relative intensity): 253.74 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 254.0640; Measured mass $[M + H]^{\cdot+}$, 254.0633 (δ 3 ppm). Elem. Anal. calculated for $C_{15}H_{11}NOS$: C, 71.12; H, 4.38; N, 5.53%. Found: C, 71.08; H, 4.31; N, 5.49%.

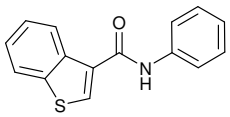
N-cyclohexylbenzo[*b*]thiophene-2-carboxamide



Molecular Weight: 259.37

Off-white solid, 74% yield. m.p. 165.2 – 166.8 ^{o}C. IR (ATR, ν_{max}/cm^{-1}): 3287, 2930, 1609, 1540, 1448, 1338, 1205, 1156, 1071, 874, 719, 662. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.19 (m, 2H, H2`and H6`), 1.14 (m, 2H, H4`), 1.64 (m, 4H, H3` and H5`), 1.97 (s, 2H, H2` and H6`), 3.91 (m, 1H, H1`), 5.92 (s, 1H, NH), 7.32 (t, 2H, *J* = 6.0 Hz, H5 and H6), 7.75 (m, 2H, H4 and H7), 7.67 (s, 1H, H3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.9 (C3` and C5`), 25.3 (C4`), 33.2 (C2` and C6`), 49.0 (C1`), 122.7 (C3), 124.86 (C7), 124.9 (C5 or C6), 124.93 (C4), 126.2 (C5 or C6), 138.97 (C3a), 139.15 (C7a), 140.7 (C2), 161.3 (C=O). MS (ESI^+) *m/z* (relative intensity): 281.06 ($[M + Na]^+$, 100%.).

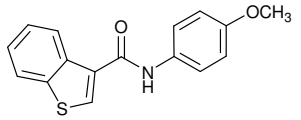
N-phenylbenzo[*b*]thiophene-3-carboxamide



Molecular Weight: 253.32

White solid, 98 % yield. m.p. 179 - 182 ºC. IR (ATR, ν_{max}/cm^{-1}): 3281 (sharp NH band), 3090, 3059, 1642, 1596, 1520, 1496, 1440, 1314, 1251, 1217, 1066, 1021, 884, 854, 765, 753, 732, 708, 689. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 (t, 1 H, *J* = 7.4 Hz, H4'), 7.38 (t, 2 H, *J* = 8.1 Hz, H3' and H5'), 7.42 – 7.50 (m, 2 H, H5 and H6), 7.64 (d, 2 H, *J* = 7.7 Hz, H2' and H6'), 7.81 (br s, 1 H, NH), 7.89 (d, 1 H, *J* = 8.1 Hz, H7), 7.98 (s, 1 H, H2), 8.41 (d, 1 H, *J* = 7.8 Hz, H4). ^{13}C NMR (100 MHz, $CDCl_3$) : δ 120.3 (C2' and C6'), 122.6 (C7), 124.3 (C4), 124.7 (C4'), 125.3 (C5 and C6), 129.2 (C3' and C5'), 129.6 (C2), 132.4 (C3), 136.7 (C4a), 137.8 (C1'), 140.3 (C7a), 162.0 (C=O). MS (ESI^+) *m/z* (relative intensity): 253.89 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 254.0640; Measured mass $[M + H]^{\cdot+}$, 254.0625 (δ 5 ppm).

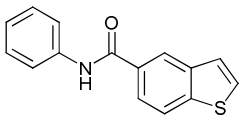
N-(4-methoxyphenyl)benzo[*b*]thiophene-3-carboxamide



Molecular Weight: 283.34

Red solid, 64% yield. m.p. 225 - 226 ºC. IR (ATR, ν_{max}/cm^{-1}): 3287, 3085, 2956, 1637, 1509, 1458, 1411, 1300, 1231, 1171, 1034, 822, 813, 766, 703, 685. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.77 (s, 3 H, H-(OCH_3)), 6.95 (d, 2 H, *J* = 9.0 Hz, H3' and H5'), 7.46 (q, 2 H, *J* = 7.1 Hz, H5 and H6), 7.68 (d, 2 H, *J* = 9.0 Hz, H2' and H6'), 8.05 (d, 1 H, *J* = 7.0 Hz, H7), 8.42 (d, 1 H, *J* = 7.1 Hz, H4), 8.52 (s, 1 H, H3), 10.20 (s, 1 H, NH). ^{13}C NMR (100 MHz, $CDCl_3$) : δ 55.2 (C-(OCH_3)), 113.8 (C3' and C5'), 121.8 (C2' and C6'), 122.8 (C7), 124.3 (C4), 124.8 (C5), 124.9 (C6), 131.2 (C2), 131.3 (C3), 132.1 (C1'), 137.2 (C4a), 139.4 (C7a), 155.5 (C4'), 161.5 (C=O). MS (ESI^+) *m/z* (relative intensity): 283.83 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 284.0667; Measured mass $[M + H]^{\cdot+}$, 284.0736. Elem. Anal. calculated for $C_{16}H_{13}NO_2S$: C, 67.82; H, 4.62; N, 4.94%. Found: C, 67.62; H, 4.61; N, 4.94%.

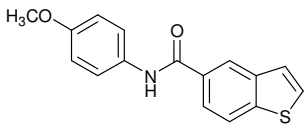
N-phenylbenzo[*b*]thiophene-5-carboxamide



Molecular Weight: 253.32

Red solid, 77 % yield. m.p. 187-188 ºC. IR (ATR, ν_{max}/cm^{-1}): 3359 (sharp NH band), 3080, 1770, 1654, 1594, 1522, 1440, 1321, 1238, 1158, 1120, 993, 901, 822, 750, 730, 686, 624. ^{1}H NMR (400 MHz, d_6-DMSO): δ 7.12 (t, 1 H, *J* = 7.3 Hz, H4'), 7.37 (t, 2 H, *J* = 7.7 Hz, H3' and H5'), 7.62 (d, 1 H, *J* = 5.4 Hz, H3), 7.82 (d, 2 H, *J* = 7.8 Hz, H2' and H6'), 7.90 (d, 1 H, *J* = 5.4 Hz, H2), 7.94 (dd, 1 H, *J* = 1.3, 8.4 Hz, H6), 8.16 (d, 1 H, *J* = 8.4 Hz, H7), 8.53, (s, 1 H, H4), 10.34 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO) : δ 120.3 (C2' and C6'), 122.5 (C7), 123.2 (C4), 123.3 (C6), 123.6 (C4'), 124.4 (C3), 128.6 (C3' and C5'), 128.9 (C2), 131.3 (C5), 139.1 (C4a), 139.3 (C1'), 142.0 (C7a), 165.6 (C=O). MS (ESI^+) *m/z* (relative intensity): 253.74 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 254.0640; Measured mass $[M + H]^{\cdot+}$, 254.0634 (δ 2 ppm). Elem. Anal. calculated for $C_{15}H_{11}NOS$: C, 71.12; H, 4.38; N, 5.53%. Found: C, 71.18; H, 4.31; N, 5.48%.

N-(4-methoxyphenyl)benzo[*b*]thiophene-5-carboxamide



Molecular Weight: 283.34

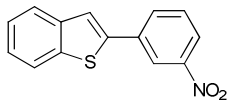
Colourless solid, 96 % yield. m.p. 211-213 ºC. IR (ATR, ν_{max}/cm^{-1}): 3349 (sharp NH band), 3075, 1774, 1648, 1599, 1508, 1407, 1321, 1234, 1158, 1120, 1052, 993, 900, 821, 804, 756, 730, 695, 646, 622. ^{1}H NMR (400 MHz, d_6-DMSO): δ 3.76 (s, 3 H, OCH_3), 6.95 (d, 2 H, *J* = 8.9 Hz, H3' and H5'), 7.61 (d, 1 H, *J* = 5.3 Hz, H3), 7.71 (d,

2 H, J = 8.8 Hz, H2' and H6'), 7.89 (d, 1 H, J = 5.4 Hz, H2), 7.93 (d, 1 H, J = 8.4 Hz, H6), 8.15 (d, 1 H, J = 8.4 Hz, H7), 8.51 (s, 1 H, H4), 10.22 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO) : δ 55.2 (OCH_3), 113.7 (C3' and C5'), 121.9 (C3' and C5'), 122.5 (C7), 123.1 (C4), 123.2 (C6), 124.4 (C3), 128.8 (C2), 131.4 (C5), 132.3 (C1'), 139.1 (C4a), 141.9 (C7a), 155.5 (C4'), 165.2 (C=O). MS (ESI^+) *m/z* (relative intensity): 283.84 ($[M + H]^{+}$, 100%). HRMS: Theoretical mass $[M + H]^{+}$, 284.0745; Measured mass $[M + H]^{+}$, 284.0750 (δ 2 ppm). Elem. Anal. calculated for $C_{16}H_{13}NO_2S$: C, 67.82; H, 4.62; N, 4.94%. Found: C, 67.67; H, 4.71; N, 4.91%.

Synthesis of sulfur-containing substrates

Sulfur-containing starting materials in oxidation reactions shown in **Table 3** that had not been previously reported in literature[2] or were not commercially available were synthesized as described below.

2-(3-nitrophenyl)benzo[*b*]thiophene



Molecular Weight: 255.29

$Pd(PPh_3)_4$ (129 mg, 0.11 mmol, 0.1 eq) was added to a solution of 3-nitrobromobenzene (272 mg, 1.34 mmol, 1.1 eq), thianaphthlene boronic acid (200 mg, 1.12 mmol, 1.0 eq) and CsF[2] (340 mg, 2.24 mmol, 2.0 eq) in acetonitrile/water (3:1 ratio; 5 ml) under nitrogen atmosphere and magnetic stirring in an Emrys™ Process vial. The vial was sealed with Reseal™ septa, and the solution was heated at 110°C under microwave radiation[1] for 20 minutes. The product 013-DA-118-01 crystallised out of the reaction mixture and was collected by filtration (gently washed with diethyl ether) as yellow solid (242 mg, 84%). m.p. 156-158 ºC. IR (ATR, ν_{max}/cm^{-1}): 3055, 3075, 1511, 1432, 1345, 1251, 1189, 1074, 985, 832, 805, 748, 723, 670. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.37-7.39 (m, 2 H, (H5 and H6), 7.60 (t, 1 H, H5'), 7.67 (s, 1 H, H3), 7.82 (dd, 1 H, J = 1.88, 8.34 Hz, H4), 7.85 (dd, 1 H, J = 1.72, 8.63 Hz, H7), 7.99 (dd, 1 H, J = 0.83, 7.80 Hz, H6'), 8.18 (dd, 1 H, J = 1.30, 7.76 Hz, H4'), 8.55 (t, 1 H, J = 1.81 Hz, H2'). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 149.2 (C3'), 141.2 (C2), 140.3 (C3a), 139.8 (C7a), 136.1 (C1'), 132.1 (C6'), 130.0 (C5'), 125.3 (C6), 125.0 (C5), 124.1 (C4), 122.6 (C4'), 122.4 (C7), 121.4 (C3), 121.1 (C2'). MS (ES^+) *m/z* (relative intensity) mass peak not observed by electron spray ionization. HRMS: Theoretical mass $[M + H]^{+}$, 256.0427; Measured mass $[M + H]^{+}$, 256.0436 (δ 4 ppm).

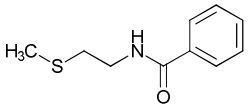
[1] Emrys™ Optimizer microwave station (Personal Chemistry).

[2] Some drops of TEA were also added.

N-(2-(methylthio)ethyl)benzamide and *N*-(4-(methylthio)phenyl)acetamide used as starting materials [*] for entries **5** and **6** of **Table 3** were synthesized as follows:

Acetyl (or benzoyl) chloride (1.01 eq) was added dropwise to a stirred solution of amine (or aniline) (1.0 eq) and TEA (2.0 eq) in THF (25 ml) at room temperature. After total consumption of the starting material (~1 h) the solution was diluted with EtOAc (100 ml) and washed with water (3 × 100 ml), dried with $MgSO_4$ and concentrated *in vacuo* to afford the products as colourless solids.

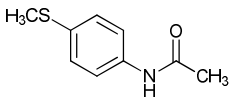
N-(2-(methylthio)ethyl)benzamide



Molecular Weight: 195.28

Colourless oil (96% yield), IR (ATR, ν_{max}/cm^{-1}): 3322, 2918, 1632, 1527, 1487, 1434, 1358, 1295, 1229, 1189, 1156, 1077, 1036, 1002, 944, 852, 802, 765, 721, 692, 660. ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 2.14 (s, 3 H, CH_3), 2.76 (t, 2 H, *J* = 6.4 Hz, SCH_2), 3.66 (q, 2 H, *J* = 6.3 Hz, $NHCH_2$), 6.64 (br s, 1 H, NH), 7.43 (t, 2 H, *J* = 7.0 Hz, H3' and H5'), 7.50 (t, 1 H, *J* = 7.4 Hz, H4'), 7.78 (d, 2 H, *J* = 7.1 Hz, H2' and H6'). ^{13}C NMR (100 MHz, $CDCl_3$): δ 15.0 (SCH_3), 33.9 (SCH_2), 38.0 ($NHCH_2$), 126.9 (C2' and C6'), 128.6 (C3' and C5'), 131.5 (C4'), 134.5 (C1'), 167.5 (C=O). MS (ESI^+) *m/z* (relative intensity): 196.44 ($[M + H]^+$ 95%).

N-(4-(methylthio)phenyl)acetamide



Molecular Weight: 181.25

Colourless oil, (90% yield) IR (ATR, ν_{max}/cm^{-1}): 3272, 3168, 3096, 2916, 1656, 1644, 1591, 1540, 1491, 1436, 1393, 1366, 1320, 1291, 1256, 1096, 1042, 1012, 952, 844, 820, 760, 608. ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 2.15 (s, 3 H, CH_3), 2.45 (s, 3 H, SCH_3), 7.22 (d, 2 H, *J* = 8.6 Hz, H3' and H5'), 7.37 (br s, 1 H, NH), 7.42 (d, 2 H, *J* = 8.6 Hz, H2' and H6'). ^{13}C NMR (100 MHz, $CDCl_3$): δ 16.7 (SCH_3), 24.5 (CH_3), 120.6 (C3 and C5), 128.0 (C2 and C6), 133. 6 (C1), 135.6 (C4), 168.3 (C=O). MS (ESI^+) *m/z* (relative intensity): 182.37 ($[M + H]^+$ 100%).

Syntheses of benzo[*b*]thiophene 1,1-dioxides

Method A, H_2O_2-P_2O_5 Reagent (Representative procedure).[*] The H_2O_2-P_2O_5 reagent (0.78 ml, 0.68 mmol, 2.0 eq) was added dropwise to a magnetically stirring suspension of *N*-phenylbenzo[*b*]thiophene-2-carboxamide (86 mg, 0.34 mmol, 1.0 eq) in CH_3CN (4 ml) in a test tube. The tube was sealed and the reaction mixture was stirred at room temperature until LC-MS showed complete consumption of starting material (9 h). Water (4 x volume of CH_3CN) was then added to the reaction mixture while stirring and the precipitate was collected by vacuum filtration and briefly

[*] The acetyl and benzoyl protecting groups were introduced to facilitate analyses (monitoring reaction time) of the oxidation reactions by LC-MS and TLC.

washed with water and small amounts of diethyl ether. The precipitate was dried over $CaCl_2$ (desiccator, overnight) to give benzo[*b*]thiophene 1,1-dioxide **4a** as yellow solids (95 mg, 98%).

* *Preparation of the H_2O_2-P_2O_5 reagent.*

Recently purchased 60% hydrogen peroxide aqueous solution (cold, ice-bath) was slowly pipetted onto 20.02 g of phosphorous pentoxide in a conical flask while moving the flask in an ice slurry. The formed lumps were smashed with a glass rod and the resulting solution (~ 40 ml) was transferred to a volumetric flask and additional 60% hydrogen peroxide was added to complete a final volume of 50 ml.[1] The solution was transferred to a glass bottle and stored at 4°C in a refrigerator.[2, 3]

Note 1: The final volume was completed with 60% hydrogen peroxide solution used to rinse (2 × ~3 ml) the conical flask.

Note 2: We observed that the concentration of the reactive phosphorous species (peroxymonophosphoric acid) in the P_2O_5-H_2O_2 reagent may vary depending on the quality of the 60% hydrogen peroxide aqueous solution. A titration can be easily carried out to determine the concentration of peroxymonophosphoric acid using small amounts (100 mg) of thianaphthene (**1**, MW: 134.20) at room temperature (app. 25 ºC). This requires 1.70 ml of the H_2O_2-P_2O_5 reagent for 100% sulfone conversion (app. 45 min), thus giving the concentration of the reactive species as 0.88 M. This titration was repeated four times (in the same day the reagent was prepared) to check its reproducibility.

Note 3: Titration (see Note 2) indicated a 50% reduction in the oxidizing properties of the reagent after three weeks which demanded larger volumes of the H_2O_2-P_2O_5 reagent for complete conversion to sulfone. It is recommended that the reagent should be used within two weeks of preparation.

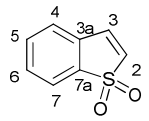
Method B, H_2O_2-TFAA (Representative procedure). 60% hydrogen peroxide aqueous solution (189 µl, 3.95 mmol, 10.0 eq) was added dropwise to a magnetically stirring solution of trifluoroacetic anhydride (0.55 ml, 3.95 mmol, 10.0 eq) in CH_3CN (1.0 ml) in a test tube in an ice-bath. The tube was sealed and the mixture was allowed to stir for one hour in the same temperature. *N*-phenylbenzo[*b*]thiophene-5-carboxamide (83 mg, 0.32 mmol, 1.0 eq) was then added in portion to the previous solution. The ice-bath was removed and the sealed vessel was allowed to reach room temperature while the reaction mixture was monitored by LC-MS. A precipitated was formed and the reaction was monitored for 8 h when LC-MS showed complete consumption of the starting material. The reaction was quenched with water (5 ml) and the precipitated collected by filtration, and washed with water (3 ml), and dried over $CaCl_2$ (desiccator, overnight) to give **4j** as a colourless solid. (88 mg, 98%).

Method C, DMDO (Representative procedure). 0.07-0.09M dimethyldioxirane acetonic solution* (6.85 ml, 0.48 mmol, 2.4 eq, as for 0.07 M) was added to a solution of *N*-phenylbenzo[*b*]thiophene-2-carboxamide (50 mg, 0.20 mmol, 1.0 eq) in DCM (1.0 ml) under magnetic stirring at room temperature. The excess solvent was removed after 15min to afford **4a** as a yellow solid. (53 mg, 98%)

* Freshly prepared according to *Organic Syntheses, Coll. Vol. 9, p.288 (1998); Vol. 74, p.91 (1997).*

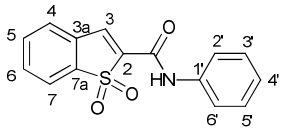
Analytical data for the oxidized products

benzo[*b*]thiophene 1,1-dioxide (**2**)[2]



Colourless solid. m.p. 92–98 °C. IR (ATR: ν_{max}/cm^{-1}); 3114, 3062 (Ar C-H), 1551, 1458, 1448, 1280, 1191, 1146, 1121, 1060, 940, 867, 794, 764, 766, 752, 696, 725. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.72 (d, 1 H, *J* = 6.93 Hz, H2); 7.22 (dd, 1 H, *J* = 6.91, 0.77 Hz, H3); 7.36 (dd, 1 H, *J* = 6.29, 1.52 Hz, H7); 7.55 (m, 2 H, H5 and H6); 7.73 (dd, 1 H, *J* = 6.03, 1.10 Hz, H4).

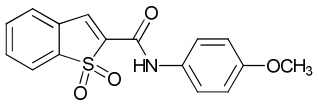
N-phenylbenzo[*b*]thiophene-2-carboxamide 1,1-dioxide (**4a**)



Molecular Weight: 285.32

Yellow solid. m.p. 257-258 ºC. IR (ATR, ν_{max}/cm^{-1}): 3270, (sharp NH band), 3111, 2992, 1681, 1606, 1544, 1501, 1440, 1280, 1251, 1070, 1049, 1018, 878, 811, 748, 700, 679. ^{1}H NMR (400 MHz, d_6-DMSO): δ 7.16 (t, 1 H, *J* = 7.4 Hz, H4'), 7.40 (t, 2 H, *J* = 7.7 Hz, H3' and H5'), 7.69 (d, 2 H, *J* = 7.8 Hz, H2' and H6'), 7.75-7.83 (dm, 3 H, H4, H5, and H6), 7.94 (d, 1 H, *J* = 6.8 Hz, H7), 8.39 (s, 1 H, H3), 10.47 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO) : δ 120.1 (C2' and C6'), 121.4 (C7), 124.4 (C4'), 127.7 (C4), 128.1 (C2), 128.9 (C3' and C5'), 132.7 (C6), 134.3 (C2 and C5), 136.3 (C4a), 137.3 (C7a), 138.0 (C1'), 156.6 (C=O). MS (ESI$^+$) *m/z* (relative intensity): 285.82 ([*M* + H]$^{\cdot+}$, 100%). HRMS: Theoretical mass [*M* + H]$^{\cdot+}$, 286.0538; Measured mass [*M* + H]$^{\cdot+}$, 286.0545 (δ 2 ppm). Elem. Anal. calculated for $C_{15}H_{11}NO_3S$: C, 63.14; H, 3.89; N, 4.91%. Found: C, 63.08; H, 3.97; N, 4.87%.

N-(4-methoxyphenyl)benzo[*b*]thiophene-2-carboxamide 1,1-dioxide (**4b**)

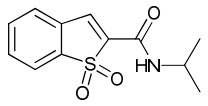


Molecular Weight: 315.34

Yellow solid. m.p. 234 - 236 ºC. IR (ATR, ν_{max}/cm^{-1}): 3390, 3058, 2951, 1654, 1616, 1573, 1535, 1507, 1454, 1439, 1411, 1291, 1243, 1148, 1128, 1033, 881, 825, 765, 744, 661. ^{1}H NMR (400 MHz, d_6-DMSO): δ 3.77 (t, 3 H, H-(OCH_3)), 6.97 (d, 2 H, *J* = 9.0 Hz, H3' and H5'), 7.60 (d, 2 H, *J* = 9.0 Hz, H2' and H6'), 7.75 – 7.82 (m, 3 H, H5, H6 and H7), 7.93 (d, 1 H, *J* = 7.6 Hz, H7), 8.33 (s, 1 H, H3), 10.37 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO) : δ 55.2 (C-(OCH_3)), 114.0 (C3' and C5'), 121.4 (C7), 121.7 (C2' and C6'), 127.6 (C4), 128.1 (C2), 131.0 (C1'), 132.6 (C6), 133.8 (C3), 134.3 (C5), 136.5 (C4a), 137.3 (C7a), 156.0 (C4'), 156.2 (C=O). MS (ESI$^+$) *m/z* (relative intensity): 315.67 ([*M* + H]$^{\cdot+}$, 100%). HRMS: Theoretical mass [*M* + H]$^{\cdot+}$,

316.0644; Measured mass $[M + H]^{+}$, 316.0653 (δ 3 ppm). Elem. Anal. calculated for $C_{16}H_{13}NO_4S$: C, 60.94; H, 4.16; N, 4.44%. Found: C, 60.95; H, 4.25; N, 4.32%.

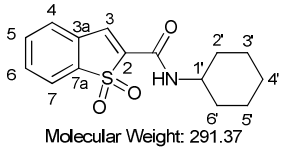
N-isopropylbenzo[*b*]thiophene-2-carboxamide 1,1-dioxide (**4d**)



Molecular Weight: 251.30

White solid. m.p. 181.1 – 182.8 °C. IR (ATR, ν_{max}/cm^{-1}): 3324, 2982, 1622, 1540, 1454, 1305, 1148, 918, 766, 663. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.21 (d, 6H, *J* = 6.8 Hz, H-$(CH_3)_2$), 4.18 (m, 1H, H-(CH)), 6.15 (s, 1H, NH), 7.43 (m, 1H, H4), 7.68 (m, 1H, H7), 7.56 (t, 2H, *J* = 4.0 Hz, H5 and H6), 7.74 (s, 1H, H3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.5 (C-$(CH_3)_2$), 42.4 (C-(CH)), 121.9 (C7), 126.9 (C4), 129.4 (C2), 132.2 (C5 or C6), 134.2 (C5 or C6), 136.2 (C3), 137.1 (C3a), 137.4 (C7a), 156.01 (C=O). MS (ESI^+) *m/z* (relative intensity): 251.19 ($[M + H]^+$, 100%.). HRMS: Theoretical mass $[M + H]^+$, 252.0694; Meseaured mass [M + H]+, 252.0688 (δ 3 ppm). Elem. Anal. calculated for $C_{12}H_{13}NO_3S$: C, 57.35; H, 5.21; N, 5.57%. Found: C, 57.35; H, 5.15; N, 5.52%.

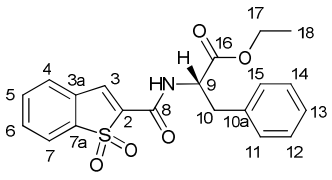
N-cyclohexylbenzo[*b*]thiophene-2-carboxamide 1,1-dioxide (**4e**)



Molecular Weight: 291.37

White solid. m.p. 196.4 – 197.8 °C. IR (ATR, ν_{max}/cm^{-1}): 3358, 2931, 1639, 1522, 1295, 1146, 918, 763, 708. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.30 (m, 4H, H3` and H5`), 1.54 (m, 2H, H2`and H6`), 1.69 (m, 2H, H4`), 1.94 (d, 2H, *J* = 12.0 Hz, H2` and H6`), 3.90 (m, 1H, H1`), 6.23 (s, 1H, NH), 7.42 (m, 1H, H4), 7.68 (m, 1H, H7), 7.54 (t, 2H, *J* = 3.6 Hz, H5 and H6), 7.74 (s, 1H, H3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.6 (C3` and C5`), 25.4 (C4`), 32.6 (C2` and C6`), 49.0 (C1`), 121.9 (C7), 126.9 (C4), 129.40 (C2), 132.1 (C5 or C6), 134.2 (C5 or C6), 136.2 (C3), 137.1 (C3a), 137.5 (C7a), 155.9 (C=O). MS (ESI^+) *m/z* (relative intensity): 291.88 ($[M + H]^+$, 100%.). HRMS: Theoretical mass $[M + H]^+$, 292.1007; Meseaured mass [M + H]+, 292.1002 (δ 2 ppm). Elem. Anal. calculated for $C_{15}H_{17}NO_3S$: C, 61.83; H, 5.88; N, 4.81%. Found: C, 61.85; H, 5.69; N, 4.81%.

(*S*)-Ethyl 2-(benzo[*b*]thiophene-2-carboxamido)-3-phenylpropanoate 1,1-dioxide (**4f**)

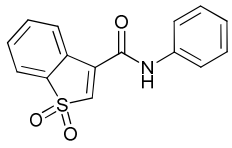


Molecular Weight: 385.43

Colourless oil. IR (ATR: ν_{max}/cm^{-1}); 3341, 3070, 1736, 1661, 1530, 1302, 1211, 1174, 1107, 1028, 849, 762, 730, 700. ^{1}H NMR (400MHz,$CDCl_3$): δ 1.31 (t, 3 H, *J* = 6.1

Hz, H18), 3.16 (dd, 1 H, *J* = 6.3, 14.0 Hz, H10), 3.31 (dd, 1 H, *J* = 6.3, 14.0 Hz, H10), 4.25 (q, 2 H, *J* = 7.1 Hz, H17), 5.01 (q, 1 H, *J* = 7.1 Hz, H9), 6.60 (d, *J* = 7.7 Hz, NH), 6.73 (s, 1 H, H3), 7.12 (d, *J* = 7.7 Hz, H11 and H15), 7.26-7.54 (m, 3 H, H12, H13 and H14), 7.54 (m, 2 H, H5 and H6), 7.68-7.71 (m, 2 H, H4 and H7). ^{13}C NMR (100 MHz, $CDCl_3$)[a] : δ 12.6 (C18), 36.1 (C10), 51.8 (C9), 60.6 (C17), 120.0 (C7), 124.0 (C4), 126.0 (C13), 127.3 (C12 and C14), 127.4 (C3), 127.7 (C11 and C15), 129.6 (C6), 132.3 (C5), 133.7 (C10a), 135.4 (C3a), 136.6 (C7a), 159.4 (NC=O), 169.2 (OC=O). MS (ESI^+) *m/z* (relative intensity): 386.5 ($[M + H]^{+}$, 100%).
[a] C2 carbon not observed using default relaxation time (1.00 sec.).

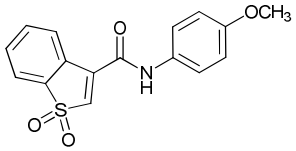
N-phenylbenzo[*b*]thiophene-3-carboxamide 1,1-dioxide (**4g**)



Molecular Weight: 285.32

Light-yellow solid. m.p. 188 - 190 ºC. IR (ATR, ν_{max}/cm^{-1}): 3356, 3075, 1672, 1597, 1539, 1488, 1440, 1288, 1243, 1205, 1175, 1137, 1109, 877, 834, 758, 694. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.00 (s, 1 H, H2), 7.21 (t, 1 H, *J* = 7.4 Hz, H4'), 7.41 (d, 2 H, *J* = 7.4 Hz, H3' and H5'), 7.54 – 7.66 (m, 4 H, H2', H6', H5, and H6), 7.72 (d, 1 H, *J* = 7.4 Hz, H4), 7.93 (d, 1 H, *J* = 7.4 Hz, H7), 8.26 (s, 1 H, H, NH). ^{13}C NMR (100 MHz, $CDCl_3$): δ 120.4 (C2' and C6'), 121.5 (C4), 125.7 (C4'), 126.0 (C7), 128.4 (C2), 129.2 (C1'), 129.3 (C3' and C5'), 131.2 (C5), 134.1 (C6), 136.7 (C3), 136.8 (C4a), 138.9 (C7a), 159.5 (C=O). MS (ESI^+) *m/z* (relative intensity): 285.80 ($[M + H]^{+}$, 100%). HRMS: Theoretical mass $[M + H]^{+}$, 286.0538; Measured mass $[M + H]^{+}$, 286.0526 (δ 4 ppm). Elem. Anal. calculated for $C_{15}H_{11}NO_3S$: C, 63.14; H, 3.89; N, 4.91%. Found: C, 63.10; H, 3.81; N, 5.01%.

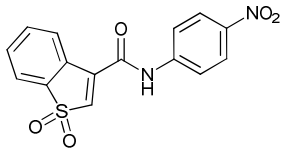
N-(4-methoxyphenyl)benzo[*b*]thiophene-3-carboxamide 1,1-dioxide (**4h**)



Molecular Weight: 315.34

Light-yellow solid. m.p. 135 - 138 ºC. IR (ATR, ν_{max}/cm^{-1}): 3245, 1649, 1610, 1566, 1513, 1297, 1242, 1172, 1105, 1030, 831, 760, 729, 682. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.83 (s, 1 H, H-(OCH_3)), 6.93 (d, 2 H, *J* = 9.0 Hz, H3' and H5'), 6.96 (s, 1 H, H2), 7.54 (d, 2 H, = 9.0 Hz, H2' and H6'), 7.57 – 7.63 (m, 2 H, H5 and H6), 7.73 (d, 1 H, *J* = 6.6 Hz, H4), 7.94 (d, 1 H, *J* = 6.8 Hz, H4), 7.97 (s, 1 H, NH). ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.5 (C-(OCH_3)), 114.5 (C3' or C5'), 121.5 (C4), 122.1 (C2' and C6'), 125.9 (C7), 128.2 (C2), 129.2 (C3), 129.6 (C1'), 131.2 (C6), 134.0 (C5), 137.0 (C4a), 139.0 (C7a), 157.4 (C4'), 159.3 (C=O). MS (ESI^+) *m/z* (relative intensity): 315.83 ($[M + H]^{+}$, 100%). HRMS: Theoretical mass $[M + H]^{+}$, 316.0644; Measured mass $[M + H]^{+}$, 316.0633 (δ 3 ppm). Elem. Anal. calculated for $C_{16}H_{13}NO_4S$: C, 60.94; H, 4.16; N, 4.44%. Found: C, 59.91; H, 4.15; N, 4.40%.

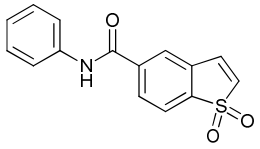
N-(4-nitrophenyl)benzo[*b*]thiophene-3-carboxamide 1,1-dioxide (**4i**)



Molecular Weight: 330.32

Yellow solid. m.p. 263-264°C. IR (ATR: ν_{max}/cm^{-1}); 3343, 3092, 1802, 1686, 1615, 1599, 1550, 1504, 1331, 1286, 1198, 1169, 1135, 1106, 978,882, 828, 764, 751, 686, 662. ^{1}H NMR (d_6-DMSO, 400 MHz): rotamers δ 7.70-7.79 (m, 2 H, H5 and H6), 7.87 (d, 1 H, *J* = 7.3 Hz, H4), 7.95 (s, 1 H, H2), 8.00-8.05 (m, 3 H, H2', H6', and H7), 8.32 (d, 2 H, *J* = 9.0 Hz, H3' and H5'), 11.4 (s, 1 H, NH). ^{13}C NMR (d_6-DMSO, 100 MHz): rotamers δ 120.0 (C2' and 6'), 121.7 (C7), 125.0 (C3' and C5'), 125.3 (C4), 128.5 (C3), 130.9 (C2), 131.4 (C6), 134.3 (C5), 136.5 (C3a), 136.9 (C7a), 143.1 (C4'), 144.1 (C1'), 160.7 (C=O). MS (ESI^-) *m/z* (relative intensity): 329.38 ($[M - H]^{\cdot-}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 331.0389; Measured mass $[M + H]^{\cdot+}$, 331.0375 (δ 4.2 ppm). HRMS (positive mode): Theoretical mass $[M + H]^{\cdot+}$, 331.0389; Measured mass $[M + H]^{\cdot+}$, 331.0375 (δ 4.2 ppm). HRMS (negative mode): Theoretical mass $[M - H]^{\cdot-}$, 329.0232; Measured mass $[M - H]^{\cdot-}$, 329.0227 (δ 2 ppm).

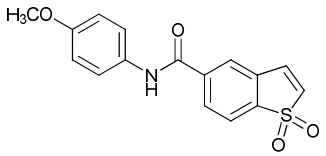
N-phenylbenzo[*b*]thiophene-5-carboxamide 1,1-dioxide (**4j**)



Molecular Weight: 285.32

Colourless solid. m.p. 247 - 249 ºC. IR (ATR, ν_{max}/cm^{-1}): 3338, 3090, 3049, 1655, 1597, 1530, 1498, 1440, 1337, 1289, 1194, 1151, 1134, 1101, 1069, 904, 800, 750, 714, 682, 626. ^{1}H NMR (400 MHz, d_6-DMSO): δ 7.14 (t, 1 H, *J* = 7.2 Hz, H4'), 7.39 (t, 2 H, *J* = 7.7 Hz, H3' and H5'), 7.49 (d, 1 H, *J* = 6.8 Hz, H2), 7.74 -7.78 (m, 3 H, H3, H2' and H6'), 8.03 (d, 1 H, *J* = 7.7 Hz, H7), 8.11 (s, 1 H, H4), 8.16 (d, 1 H, *J* = 7.7 Hz, H6), 10.5 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): δ 120.4 (C2' or C6'), 121.1 (C7), 124.1 (C4'), 125.3 (C4), 128.7 (C3' and C5'), 130.2 (C6), 131.6 (C4a), 131.9 (C2), 132.5 (C3), 138.4 (C5), 138.7 (C1'), 140.3 (C7a), 164.0 (C=O). MS (ESI^+) *m/z* (relative intensity): 285.8 ($[M + H]^{\cdot+}$, 100%).

N-(4-methoxyphenyl)benzo[*b*]thiophene-5-carboxamide 1,1-dioxide (**4k**)

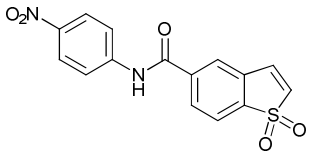


Molecular Weight: 315.34

Light-yellow solid. m.p. 251 - 253 ºC. IR (ATR, ν_{max}/cm^{-1}): 3307, 3096, 1652, 1528, 1517, 1290, 1235, 1196, 1152, 1135, 1032, 838, 820, 747, 682. ^{1}H NMR (400 MHz, d_6-DMSO): δ 3.76 (s, 1 H, OCH_3), 6.96 (d, 2 H, *J* = 6.9 Hz, H3' and H5'), 7.48 (d, 1

H, *J* = 6.9 Hz, H2), 7.67 (d, 2 H, *J* = 9.0 Hz, H2' and H6'), 7.73 (dd, 1 H, *J* = 0.6, 6.9 Hz, H3), 8.02 (d, 1 H, *J* = 7.8 Hz, H7), 8.10 (s, 1 H, H4), 8.14 (dd, 1 H, *J* = 1.2, 7.9 Hz, H6), 10.4 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): δ 55.2 (OCH_3), 113.8 (C3' or C5'), 121.1 (C7), 122.0 (C2' and C6'), 125.2 (C4), 130.1 (C6), 131.6 (C1'), 131.7 (C4a), 131.9 (C2), 132.5 (C3), 138.3 (C7a), 140.3 (C5), 155.8 (C4'), 163.5 (C=O). MS (ESI^+) *m/z* (relative intensity): 315.76 ($[M + H]^{\cdot+}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 316.0644; Measured mass $[M + H]^{\cdot+}$, 316.0644 (δ 0 ppm). Elem. Anal. calculated for $C_{16}H_{13}NO_4S$: C, 60.94; H, 4.16; N, 4.44%. Found: C, 60.94; H, 4.10; N, 4.31%.

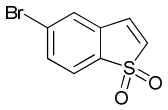
N-(4-nitrophenyl)benzo[*b*]thiophene-5-carboxamide 1,1-dioxide (**4l**)



Molecular Weight: 330.32

Light-yellow solid. m.p. 269-270°C decomposes and 291-293°C melts. IR (ATR: ν_{max}/cm^{-1}); 3328, 3070, 1796, 1677, 1590, 1543, 1497, 1330, 1294, 27, 1148, 1109, 1070, 855, 789, 750, 688. ^{1}H NMR (d_6-DMSO, 400 MHz): rotamers δ 7.52 (d, 1 H, *J* = 6.6 Hz, H2), 7.76 (d, 1 H, *J* = 6.9 Hz, H3), 8.04 (d, 2 H, *J* = 9.2 Hz, H2' and H6'), 8.07 (d, 1 H, *J* = 8.0 Hz, H7), 8.13 (s, 1 H, H4), 8.18 (d, 1 H, *J* = 7.8 Hz, H6), 8.28 (d, 2 H, *J* = 9.2 Hz, H3' and H5'), 11.05 (s, 1 H, NH). ^{13}C NMR (d_6-DMSO, 100 MHz): rotamers δ 120.0 (C2' and 6'), 121.3 (C7), 124.8 (C3' and C5'), 125.5 (C4), 130.6 (C6), 131.6 (C2), 132.0 (C3), 132.4 (C3a), 138.8 (C5), 139.5 (C7a), 142.8 (C4'), 144.9 (C1'), 164.7 (C=O). MS (ESI^-) *m/z* (relative intensity): 329.53 ($[M - H]^{\cdot-}$, 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 331.0389; Measured mass $[M + H]^{\cdot+}$, 331.0388 (δ >1 ppm).

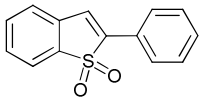
5-bromobenzo[b]thiophene 1,1-dioxide[3](**5**)



Molecular Weight: 245.09

White solid. HRMS: Theoretical mass $[M + Na]^{\cdot+}$, 266.9091; Measured mass $[M + Na]^{\cdot+}$, 266.9079 (δ 4 ppm). Elem. Anal. calculated for $C_8H_5BrO_2S$: C, 39.20; H, 2.06. Found: C, 39.21; H, 1.99%.

2-phenylbenzo[b]thiophene 1,1-dioxide[2](**6**)

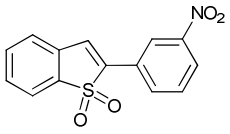


Molecular Weight: 242.29

Light-yellow solid. ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 7.55-7.65 (m, 3 H, H3', H4', and H5'), 7.68 (t, 1 H, *J* = 7.6 Hz, H6), 7.71 (d, 1 H, *J* = 7.2 Hz, H4), 7.80 (t, 1 H, *J* = 7.6

Hz, H5), 7.92 (d, 2 H, *J* = 8.0 Hz, H2' and H6'), 7.99 (d, 1 H, *J* = 7.2 Hz, H7), 8.05 (s, 1 H, H2). MS (ESI^+) *m/z* (relative intensity): 243.30 ($[M + H]^+$ 100%).

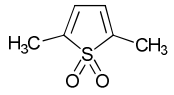
2-(3-nitrophenyl)benzo[b]thiophene 1,1-dioxide (**7**)



Molecular Weight: 287.29

Colourless solid. m.p. 240-242 ºC. IR (ATR, ν_{max}/cm^{-1}): 3092, 1569, 1530, 1453, 1337, 1292, 1247, 1200, 1152, 1125, 1094, 896, 869, 831, 762, 737, 666. ^1H NMR ($CDCl_3$, 400 MHz) δ7.49 (m, 2 H, (H4 and H3), 7.58 (dt, 1 H, *J* = 0.89, 7.51 Hz, H6), 7.64 (dt, 1 H, *J* = 1.05, 7.52 Hz, H5), 7.70 (t, 1 H, *J* = 8.04 Hz, H5'), 7.81 (d, 1 H, *J* = 7.45 Hz, H7), 8.23 (dd, 1 H, *J* = 1.07, 7.87 Hz, H6'), 8.31 (dd, 1 H, *J* = 1.32, 8.28 Hz, H4'), 8.61 (t, 1 H, *J* = 1.86 Hz, H2'). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.5 (C3'), 140.5 (C2), 137.1 (C7a), 134.1 (C5), 132.1 (C6'), 130.8 (C6), 130.5 (C5'), 129.5 (C3a), 129.0 (C1'), 126.4 (C3), 125.7 (C4), 124.6 (C4'), 121.8 (C7), 121.6 (C2'). MS (ES^+) *m/z* (relative intensity) mass peak not observed by electron spray ionization. HRMS: Theoretical mass $[M + H]^{\cdot+}$, 288.0325; Measured mass $[M + H]^{\cdot+}$, 288.0326 (δ 3 ppm).

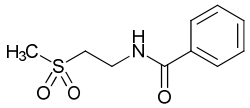
2,5-dimethylthiophene 1,1-dioxide[4] (**8**)



Molecular Weight: 144.19

MS (ESI^+) *m/z* (relative intensity): 145.26 ($[M + H]^+$ 100%)].

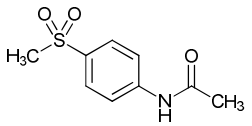
N-(2-(methylsulfonyl)ethyl)benzamide (**9**)



Molecular Weight: 227.28

Colourless oil. IR (ATR, ν_{max}/cm^{-1}): 3310, 3013, 2920, 1634, 1537, 1488, 1369, 1278, 1185, 1164, 1120, 1076, 1058, 961, 880, 804, 694, 668. ^1H-NMR ($CDCl_3$, 400 MHz) rotamers: δ 2.99 (s, 3 H, SCH_3), 3.38 (t, 2 H, *J* = 5.7 Hz, $NHCH_2$), 3.98 (q, 3 H, *J* = 5.97 Hz, SO_2CH_2), 7.26-7.30 (br s, 1 H, NH), 7.42 (t, 2 H, *J* = 7.3 Hz, H3' and H5'), 7.50 (t, 2 H, *J* = 7.3 Hz, H4'), 7.77 (d, 2 H, *J* = 7.3 Hz, H2' and H6'). ^{13}C NMR (100 MHz, $CDCl_3$): δ 33.8 ($NHCH_2$), 41.6 (SCH_3), 53.7 (SCH_2), 127.1 (C2' and C6'), 128.8 (C3' and C5'), 132.2 (C4'), 133.3 (C1'), 168.2 (C=O). MS (ESI^+) *m/z* (relative intensity): 228.43 ($[M + H]^+$ 100%). HRMS: Theoretical mass $[M + H]^{\cdot+}$, 228.0694; Measured mass $[M + H]^{\cdot+}$, 228.0699 (δ 2 ppm).

N-(4-(methylsulfonyl)phenyl)acetamide (**10**)



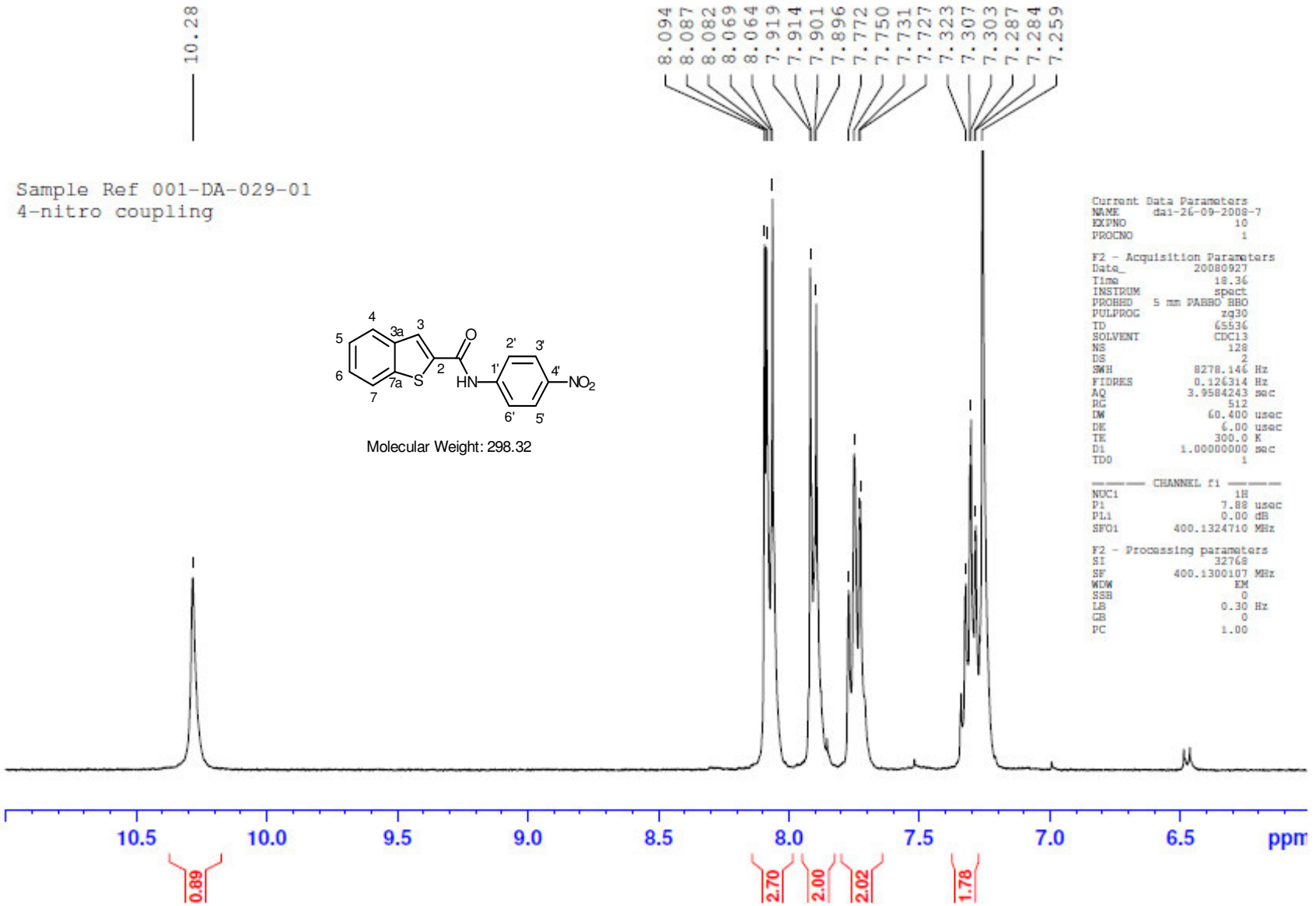
Molecular Weight: 213.25

White crystals. m.p. 202-204 ºC. IR (ATR, ν_{max}/cm^{-1}): 3351, 3027, 3003, 2925, 1670, 1588, 1532, 1503, 1438, 1388, 1276, 1132, 1086, 965, 836, 821, 777, 660. ^{1}H-NMR (400 MHz, d_6-DMSO): δ 2.15 (s, 3 H, CH_3), 3.20 (s, 3 H, SCH_3), 7.86 (d, 2 H, J = 9.0 Hz, H2' and H6'), 7.90 (d, 2 H, J = 8.9 Hz, H3' and H5'), 10.42 (s, 1 H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): δ 24.1 (CH_3), 43.8 (SCH_3), 118.6 (C3' and C5'), 128.1 (C2' and C6'), 134.4 (C1'), 143.6 (C4'), 169.1 (C=O). MS (ESI^+) *m/z* (relative intensity): 214.29 ($[M + H]^+$ 100%). HRMS: Theoretical mass $[M + H]^{+}$, 214.0538; Measured mass $[M + H]^{+}$, 214.0529 (δ 4 ppm). Elem. Anal. calculated for $C_9H_{11}NO_3S$: C, 71.63; H, 5.51; N, 6.96%. Found: C, 71.58; H, 5.48; N, 6.93%.

References

1. Yoo, W. J.; Li, C. J. *J. Am. Chem. Soc.* **2006,** *128*, 13064-13065.
2. Geneste, P.; Olive, J. L.; Ung, S. N.; El Faghi, M. E. A.; Easton, J. W.; Beierbeck, H.; Saunders, J. K. *J. Org. Chem.* **1979,** *44*, 2887-2892.
3. Chapman, N. B.; Ewing, D. F.; Scrowston, R. M.; Westwood, R. *J. Chem. Soc. C* **1968**, 764-769.
4. Rozen, S.; Bareket, Y. *J. Org. Chem.* **1997,** *62*, 1457-1462.

NMR spectra for all novel compounds



Sample Ref 001-DA-029-01
4-nitro coupling

126.61
126.50
125.13
124.94
124.64
122.69
119.77

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Current Data Parameters
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EXPNO                   12
PROCNO                   1

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TD                  65536
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NS                   1024
DS                      4
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FIDRES          0.365919 Hz
AQ              1.3664756 sec
RG                 1290.2
DW                20.850 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
d11            0.03000000 sec
DELTA          0.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                    7.87 usec
PL1                   0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                   0.00 dB
PL12                 20.00 dB
PL13                 20.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
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SF           100.6127690 MHz
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GB                       0
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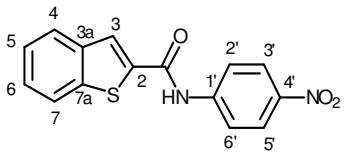
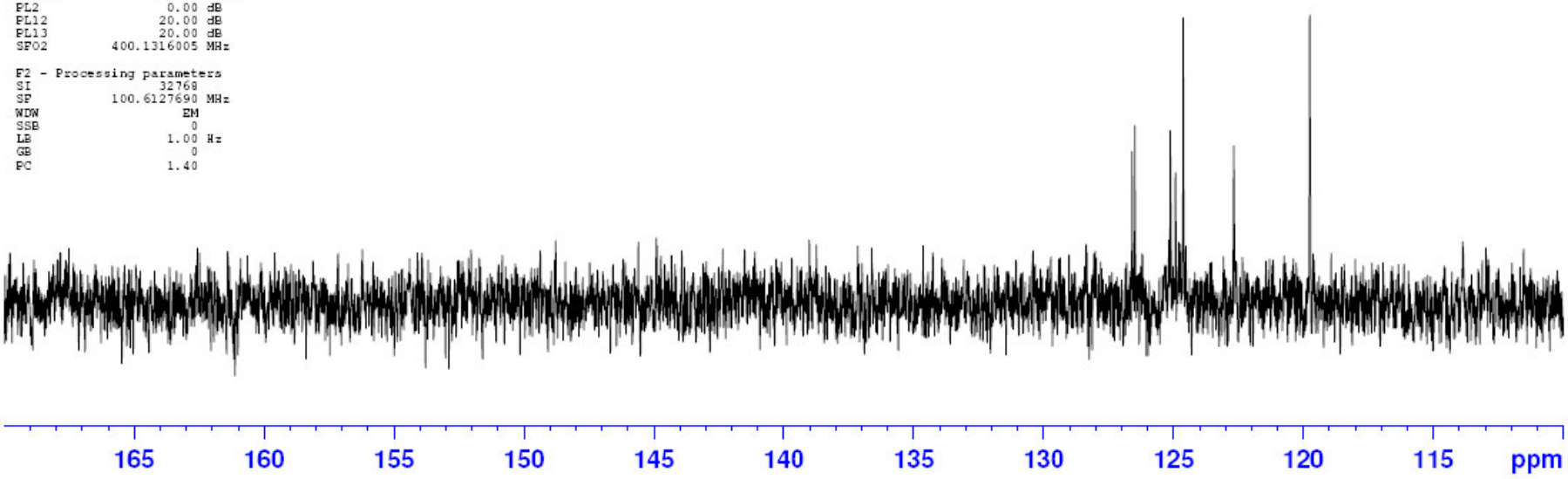
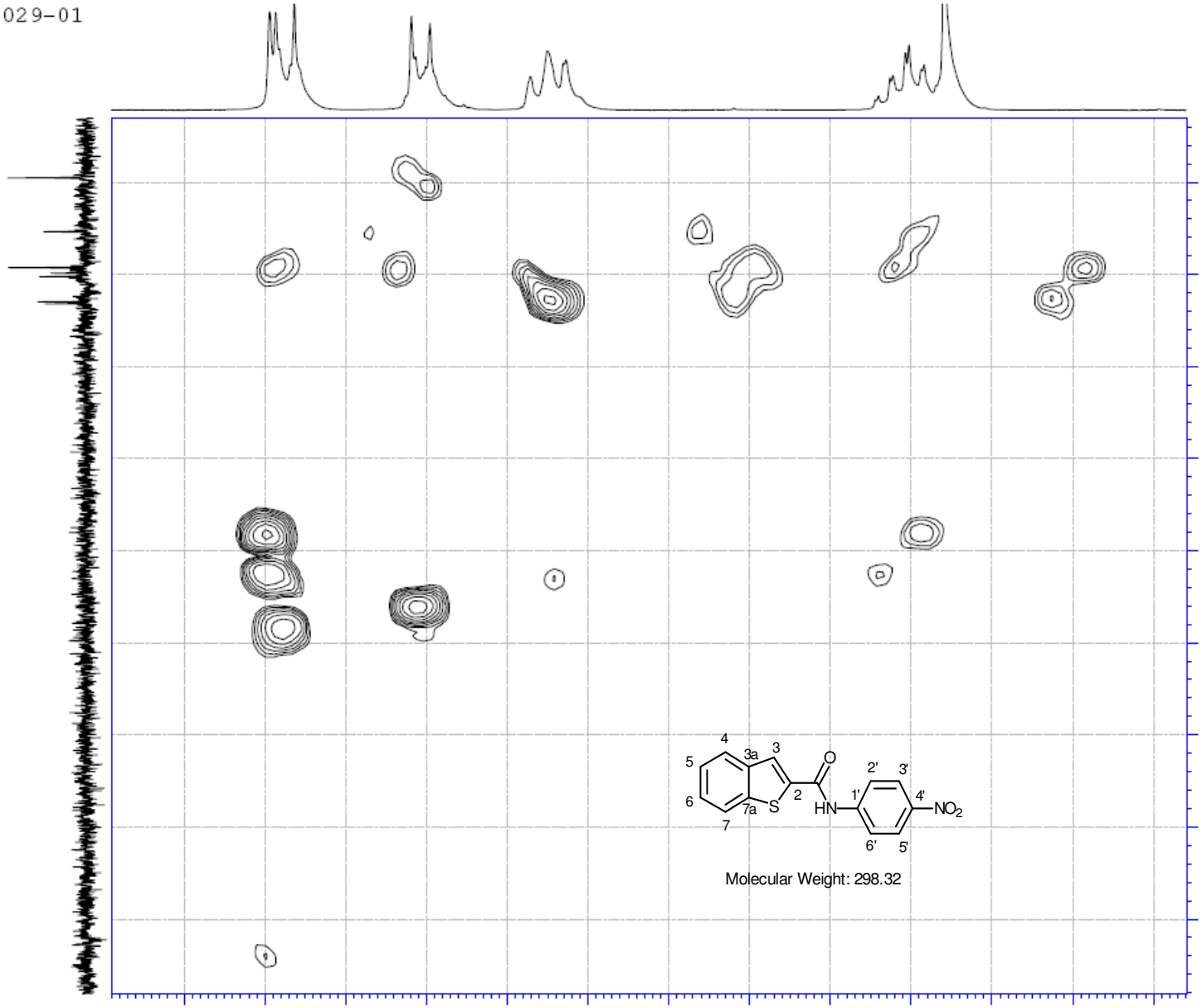


Molecular Weight: 298.32



Sample Ref 001-DA-029-01
4-nitro coupling



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Current Data Parameters
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EXPNO                   15
PROCNO                   1

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INSTRUM             spect
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PULPROG    inv4gplrndgf
TD                 1024
SOLVENT            CDCl3
NS                    8
DS                   16
SWH            4432.624 Hz
FIDRES         4.328734 Hz
AQ             0.1155572 sec
RG                 16384
DW              112.800 usec
DE                6.00 usec
TE                300.0 K
d0             0.00000300 sec
D1             1.48013401 sec
D6             0.07100000 sec
d13            0.00000400 sec
D16            0.00010000 sec
IN0            0.00002240 sec

--------- CHANNEL f1 --------
NUC1                   1H
P1                   7.88 usec
p2                  15.76 usec
PL1                  0.00 dB
SFO1         400.1322235 MHz

--------- CHANNEL f2 --------
NUC2                  13C
P3                   7.65 usec
PL2                  0.00 dB
SFO2         100.6227746 MHz

------ GRADIENT CHANNEL -----
GPNAM1          SINE.100
GPNAM2          SINE.100
GPNAM3          SINE.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
P16              1000.00 usec

F1 - Acquisition parameters
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TD                   128
SFO1         100.6228 MHz
FIDRES       174.386154 Hz
SW            221.833 ppm
FnMODE               QF

F2 - Processing parameters
SI                  1024
SF           400.1299787 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           100.6127690 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
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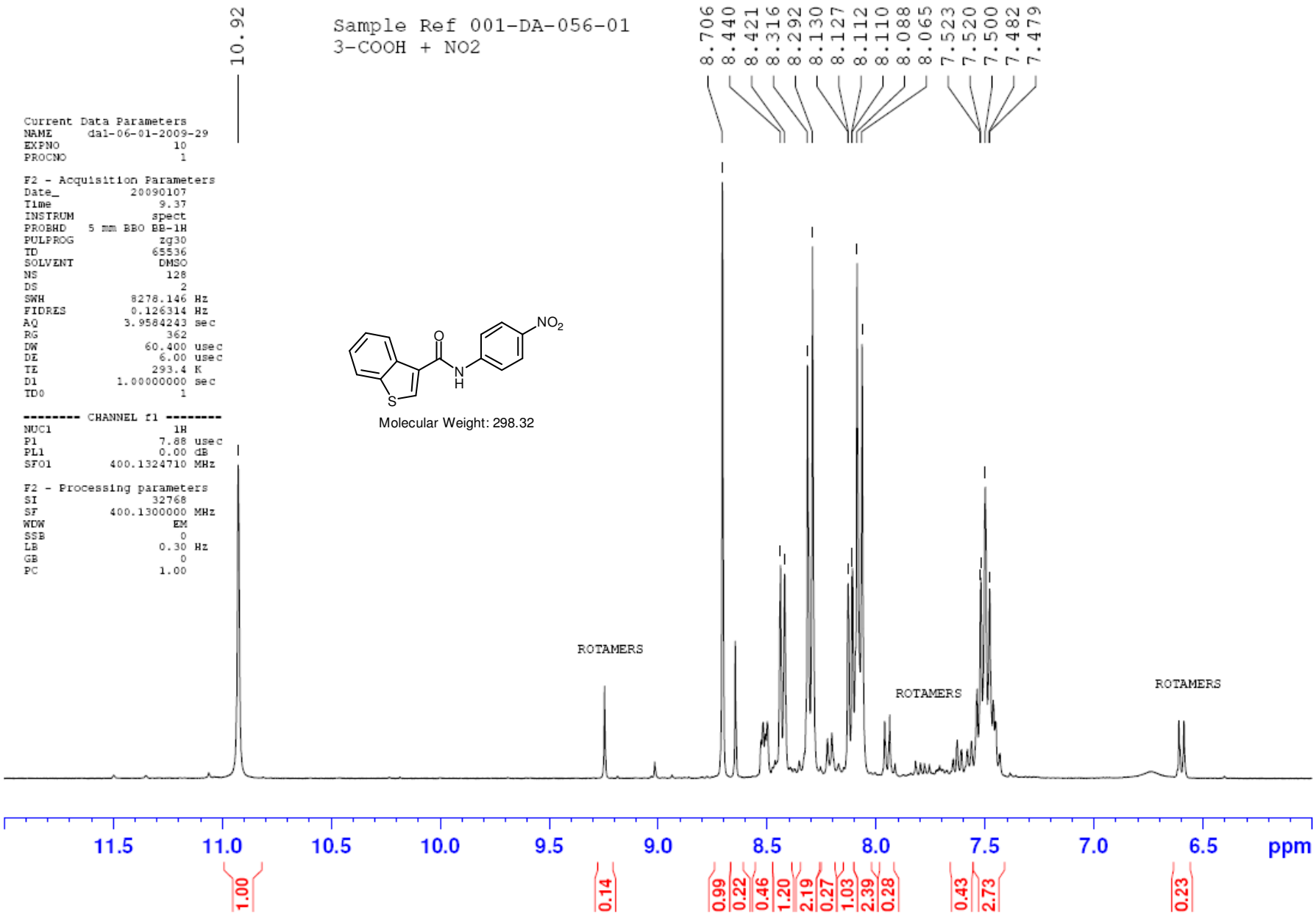
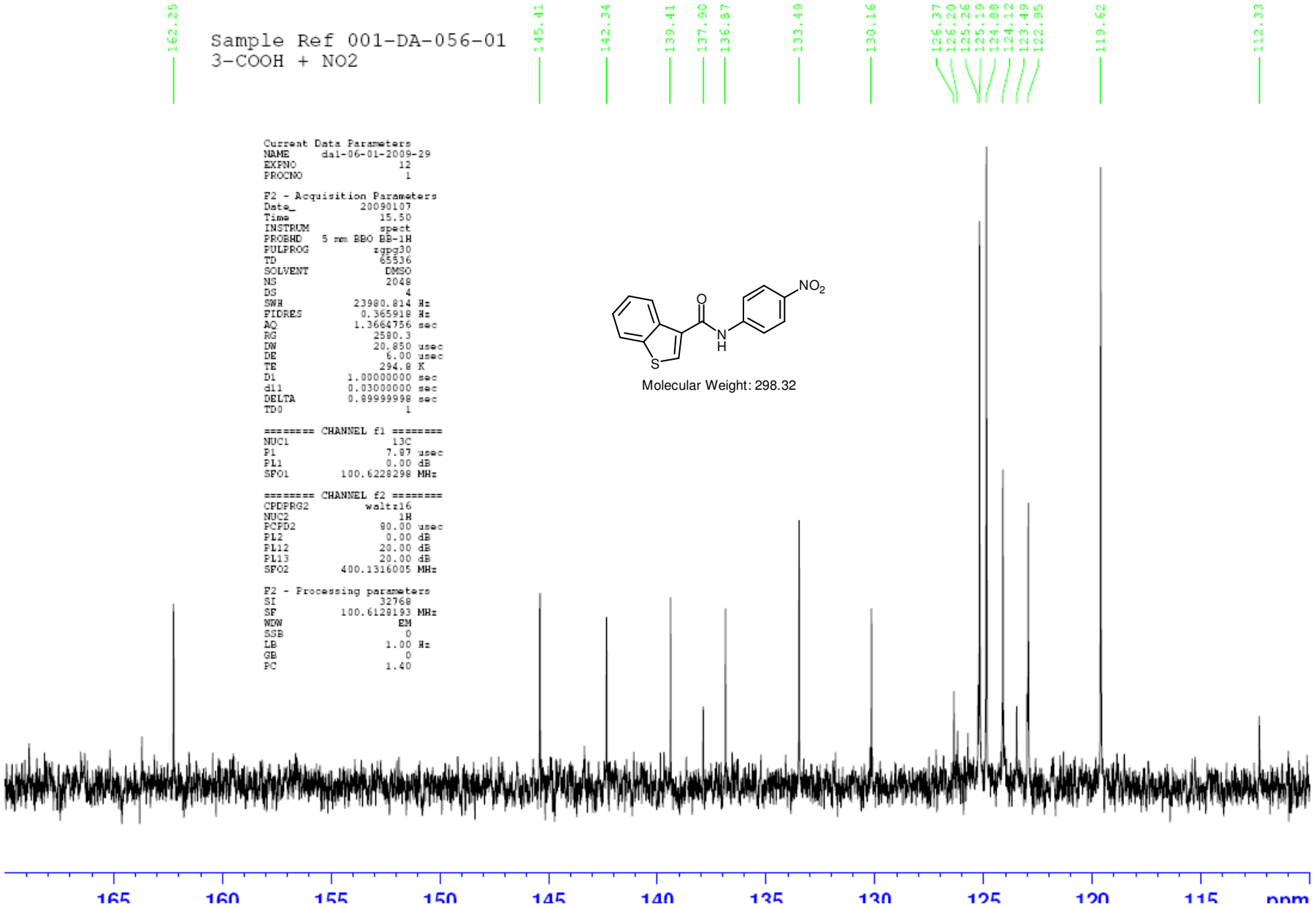
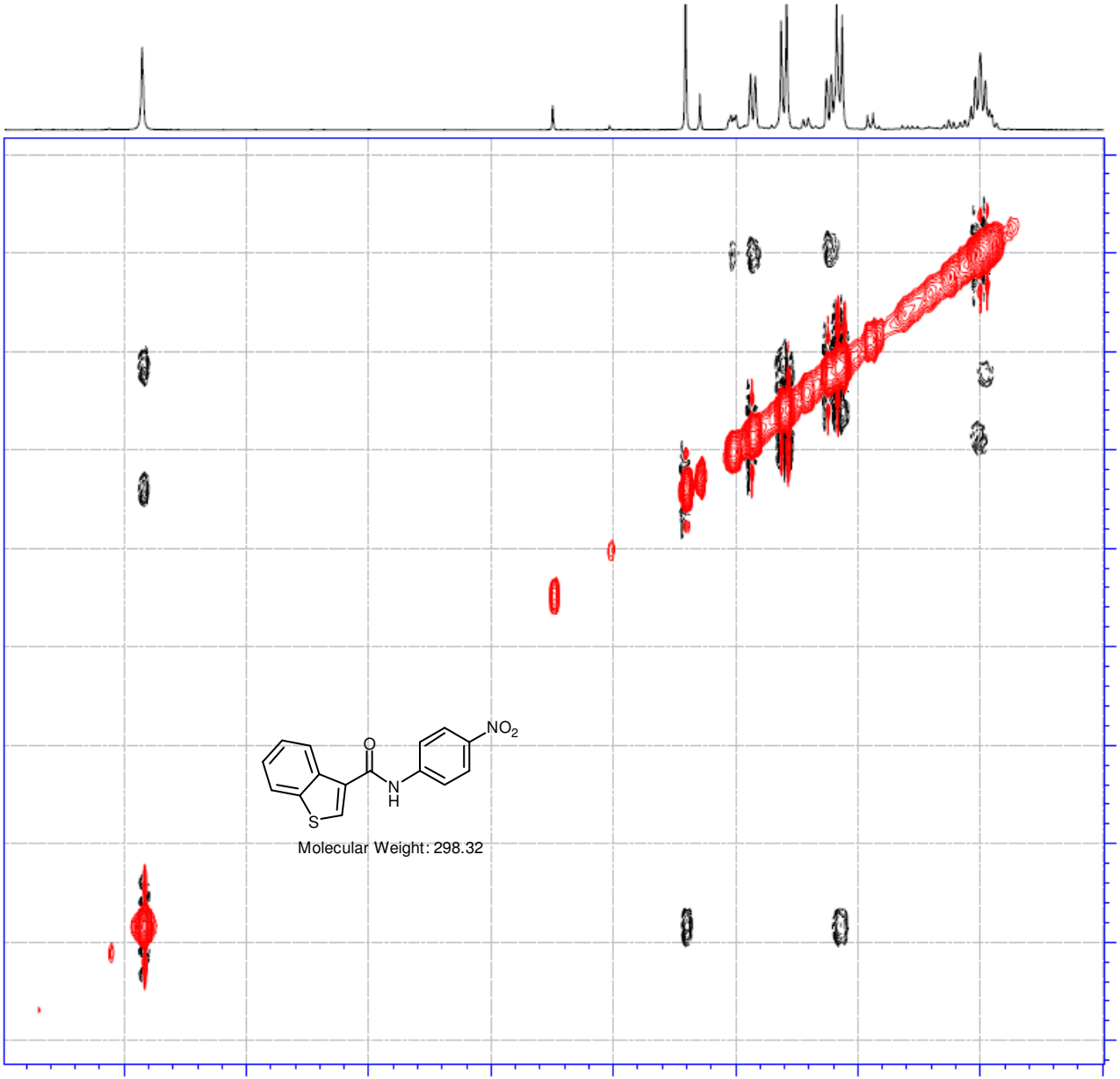
Molecular Weight: 298.32


Sample Ref 001-DA-056-01
3-COOH + NO2
10.92
8.706
8.440
8.421
8.316
8.292
8.130
8.127
8.112
8.110
8.088
8.065
7.523
7.520
7.500
7.482
7.479
Current Data Parameters
NAME dal-06-01-2009-29
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090107
Time 9.37
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
H
S
NO2
Molecular Weight: 298.32
ROTAMERS
ROTAMERS
ROTAMERS
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
ppm
1.00
0.14
0.99
0.22
0.46
1.20
2.19
0.27
1.03
2.39
0.28
0.43
2.73
0.23


Sample Ref 001-DA-056-01
3-COOH + NO2
162.25
145.41
142.34
139.41
137.90
136.87
133.49
130.16
126.37
126.20
125.26
125.19
124.88
124.12
123.49
122.95
119.62
112.33
Current Data Parameters
NAME da1-06-01-2009-29
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090107
Time 15.50
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
H
S
NO2
Molecular Weight: 298.32
165
160
155
150
145
140
135
130
125
120
115
ppm

Sample Ref 001-DA-056-01
3-COOH + NO2



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Current Data Parameters
NAME      da1-06-01-2009-29
EXPNO                   16
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090107
Time                 19.11
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG           noesyph
TD                    2048
SOLVENT               DMSO
NS                       4
DS                       4
SWH               5555.556 Hz
FIDRES            2.712674 Hz
AQ                0.1843700 sec
RG                     256
DW                  90.000 usec
DE                   6.00 usec
TE                  293.8 K
d0              0.00007997 sec
D1              1.33188999 sec
D8              0.80000001 sec
IN0             0.00018000 sec
ST1CNT                 128

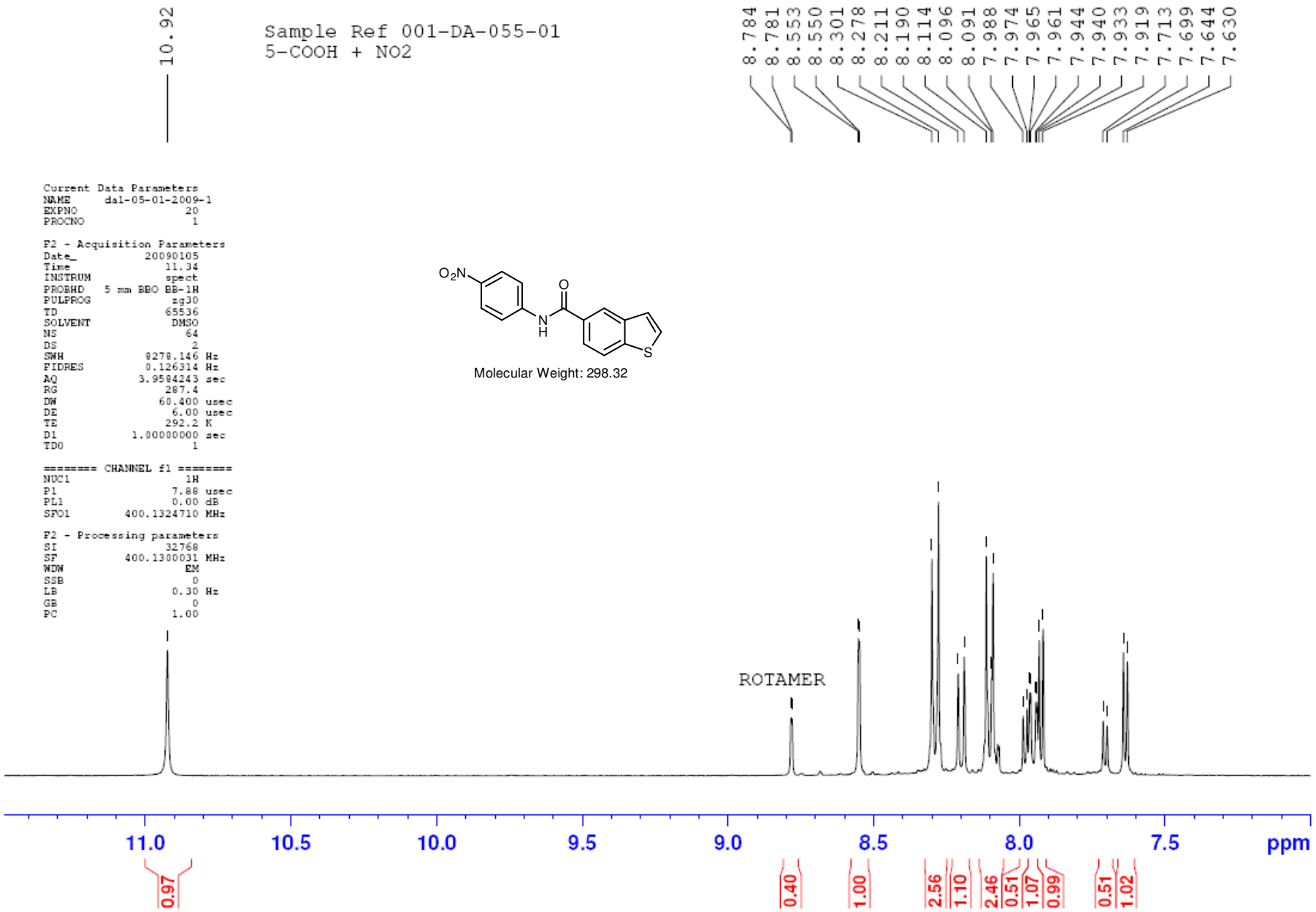
-------- CHANNEL f1 --------
NUC1                    1H
P1                   7.88 usec
PL1                  0.00 dB
SFO1          400.1327909 MHz

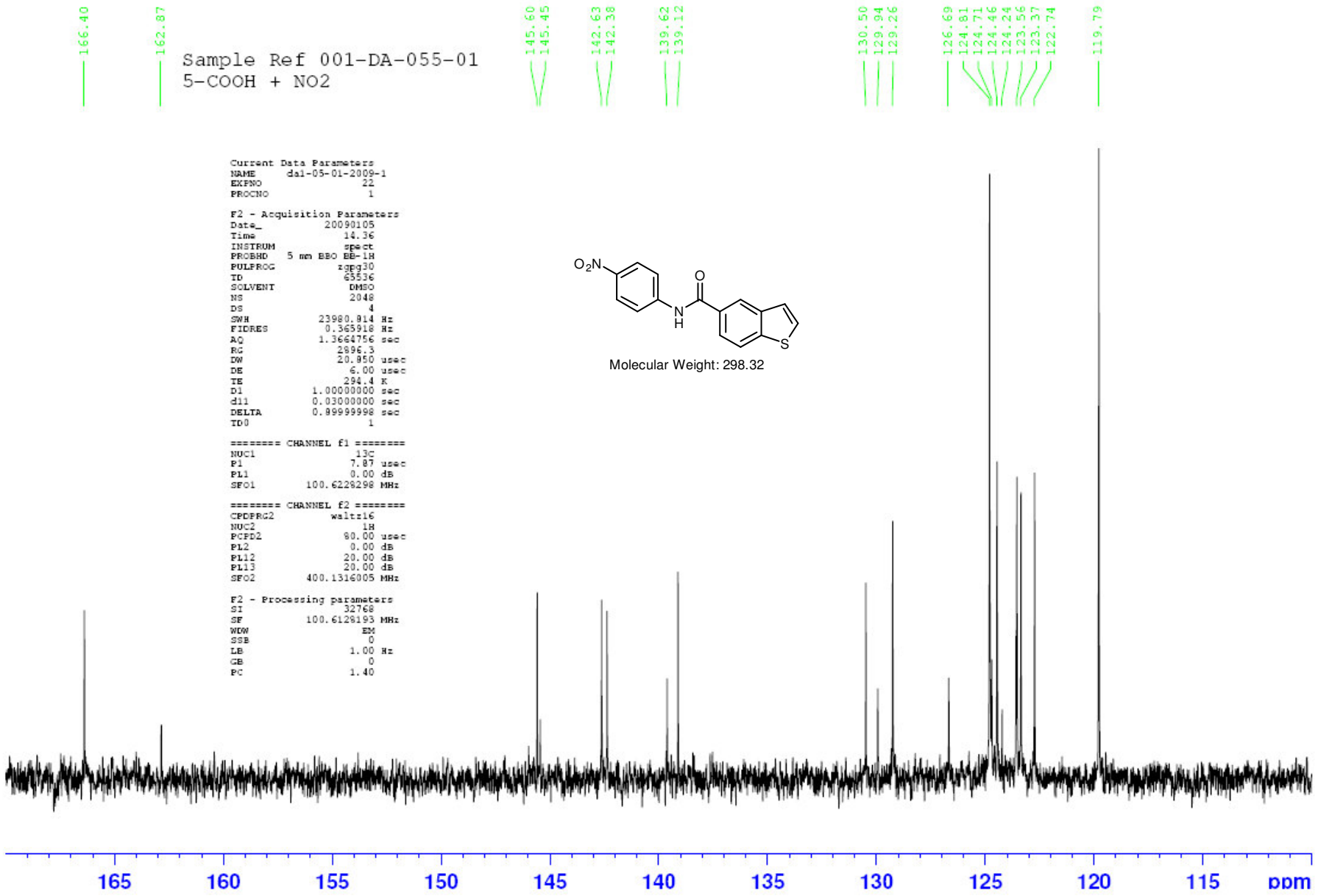
F1 - Acquisition parameters
ND0                      1
TD                     256
SFO1            400.1328 MHz
FIDRES          21.701389 Hz
SW                13.884 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                    1024
SF            400.1300000 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.00

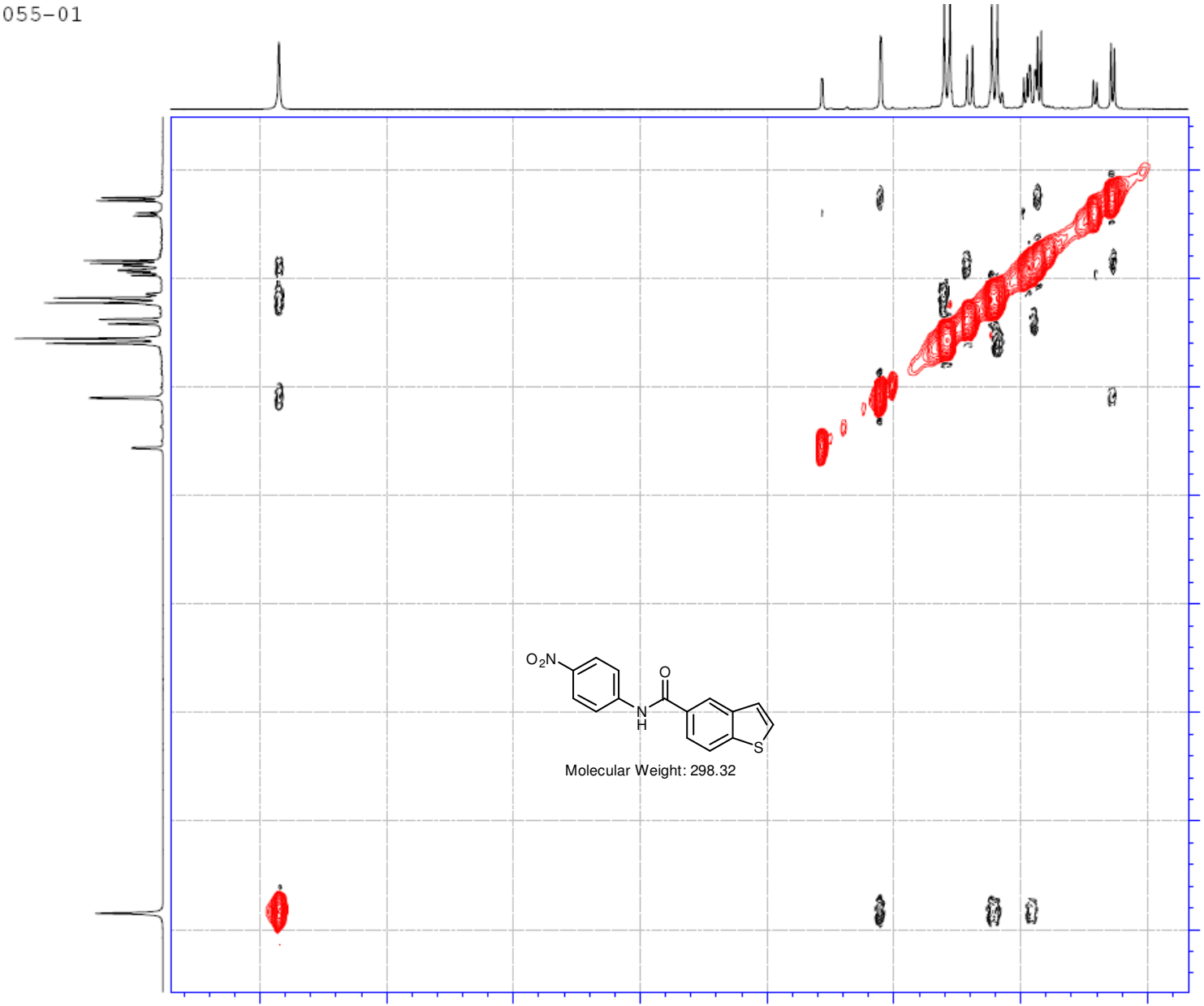
F1 - Processing parameters
SI                    1024
MC2          States-TPPI
SF            400.1300000 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
```


Molecular Weight: 298.32


10.92
Sample Ref 001-DA-055-01
5-COOH + NO2
8.784
8.781
8.553
8.550
8.301
8.278
8.211
8.190
8.114
8.096
8.091
7.988
7.974
7.965
7.961
7.944
7.940
7.933
7.919
7.713
7.699
7.644
7.630
Current Data Parameters
NAME da1-05-01-2009-1
EXPNO 20
PROCNO 1
P2 - Acquisition Parameters
Date_ 20090105
Time 11.34
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O2N
O
N
H
S
Molecular Weight: 298.32
ROTAMER
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
ppm
0.97
0.40
1.00
2.56
1.10
2.46
0.51
1.07
0.99
0.51
1.02


166.40
162.87
145.60
145.45
142.63
142.38
139.62
139.12
130.50
129.94
129.26
126.69
124.81
124.71
124.46
124.24
123.56
123.37
122.74
119.79
Sample Ref 001-DA-055-01
5-COOH + NO2
Current Data Parameters
NAME dal-05-01-2009-1
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090105
Time 14.36
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 usec
DE 6.00 usec
TE 294.4 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O2N
O
N
H
S
Molecular Weight: 298.32
165
160
155
150
145
140
135
130
125
120
115
ppm

Sample Ref 001-DA-055-01
5-COOH + NO2



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Current Data Parameters
NAME      da1-05-01-2009-1
EXPNO                   26
PROCNO                   1

F2 - Acquisition Parameters
Date_           20090105
Time               16.36
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          noesyph
TD                  2048
SOLVENT             DMSO
NS                     4
DS                     4
SWH             4708.098 Hz
FIDRES          2.298876 Hz
AQ              0.2175476 sec
RG                   181
DW                106.200 usec
DE                  6.00 usec
TE                 293.3 K
d0              0.00009617 sec
D1              1.29912198 sec
D8              0.80000001 sec
IN0             0.00021240 sec
ST1CNT               128

-------- CHANNEL f1 --------
NUC1                  1H
P1                 7.88 usec
PL1                0.00 dB
SFO1         400.1323434 MHz

F1 - Acquisition parameters
ND0                    1
TD                   256
SFO1           400.1323 MHz
FIDRES        18.391008 Hz
SW               11.766 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                  1024
SF           400.1300352 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.00

F1 - Processing parameters
SI                  1024
MC2          States-TPPI
SF           400.1300352 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
```
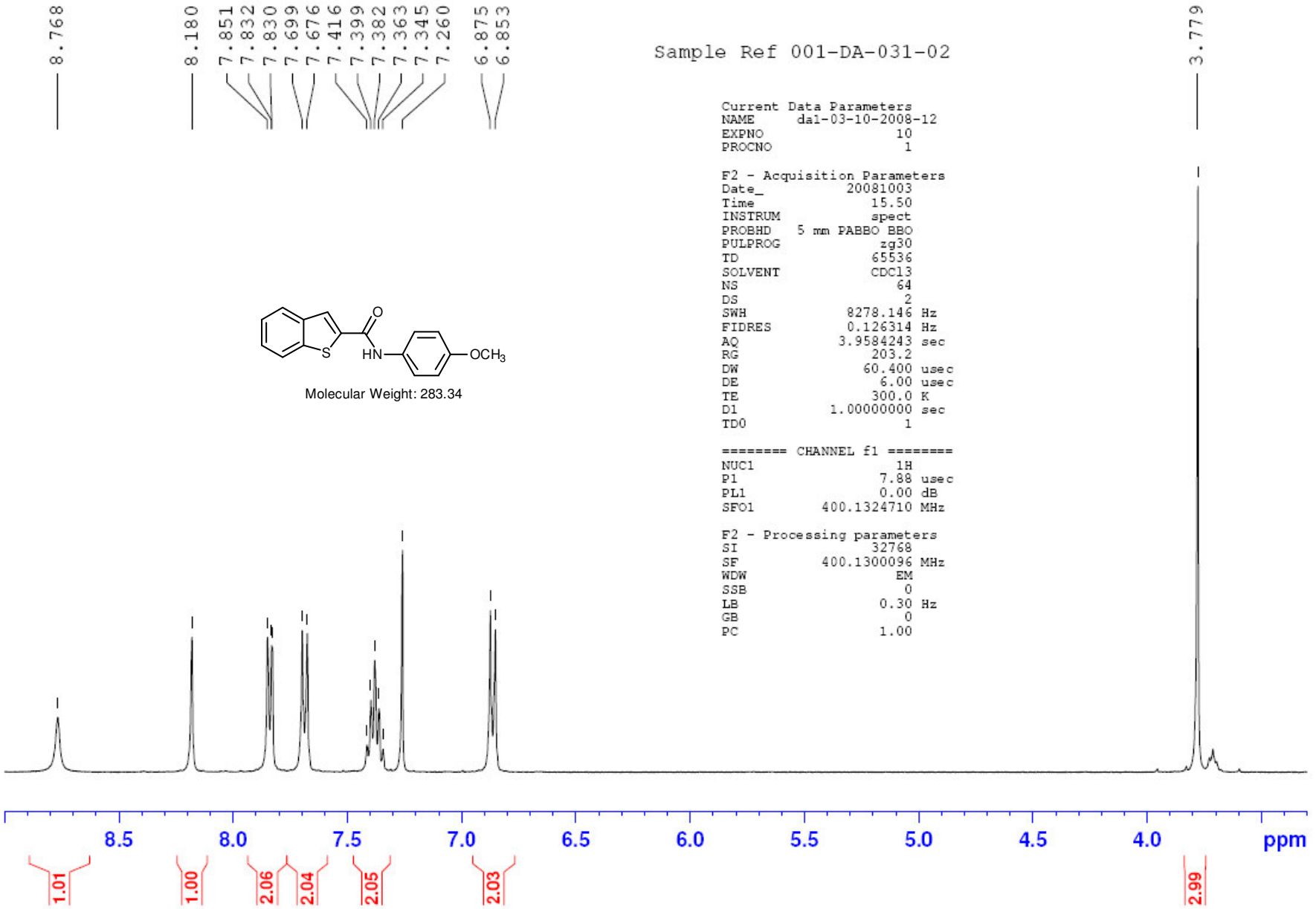
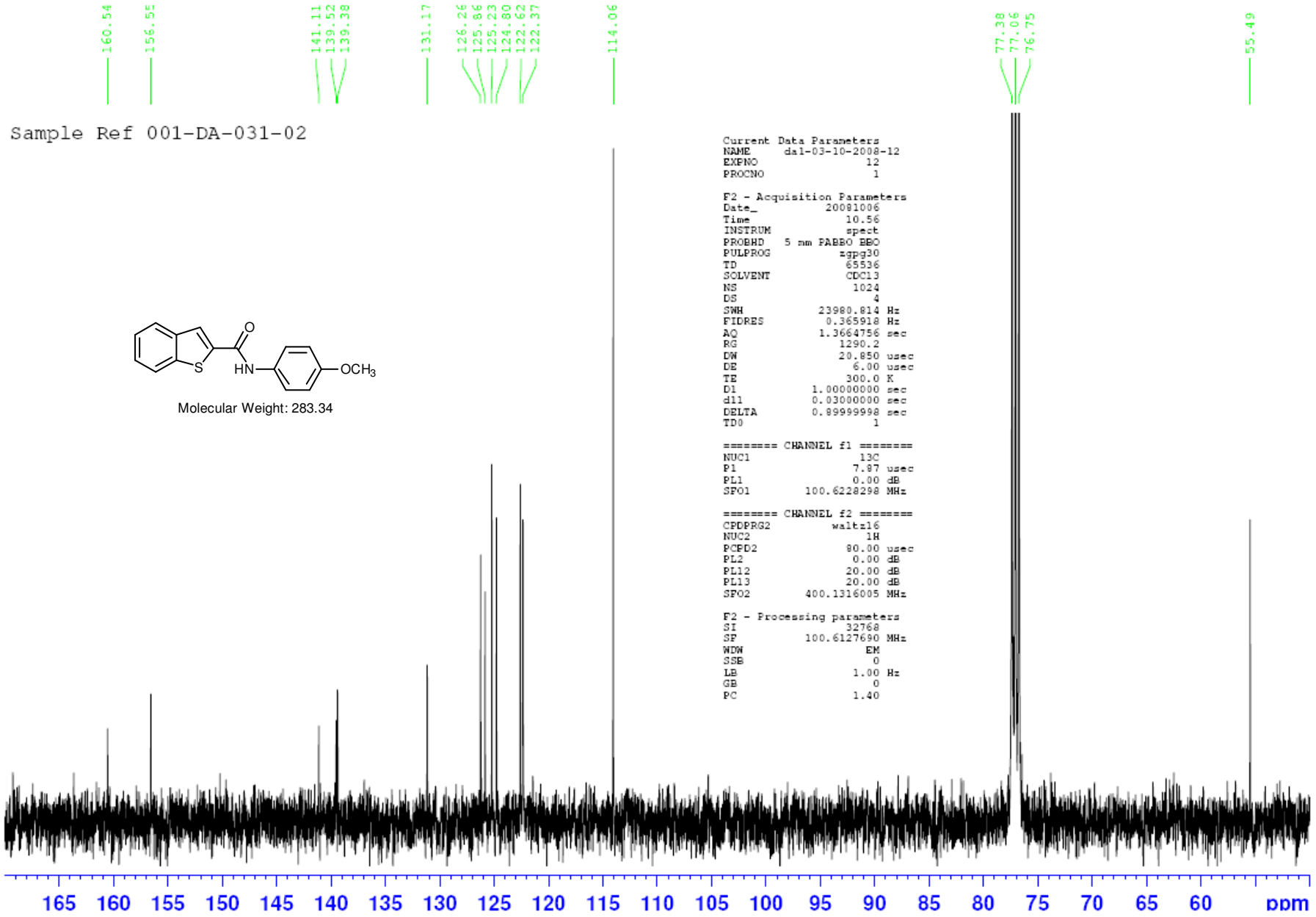
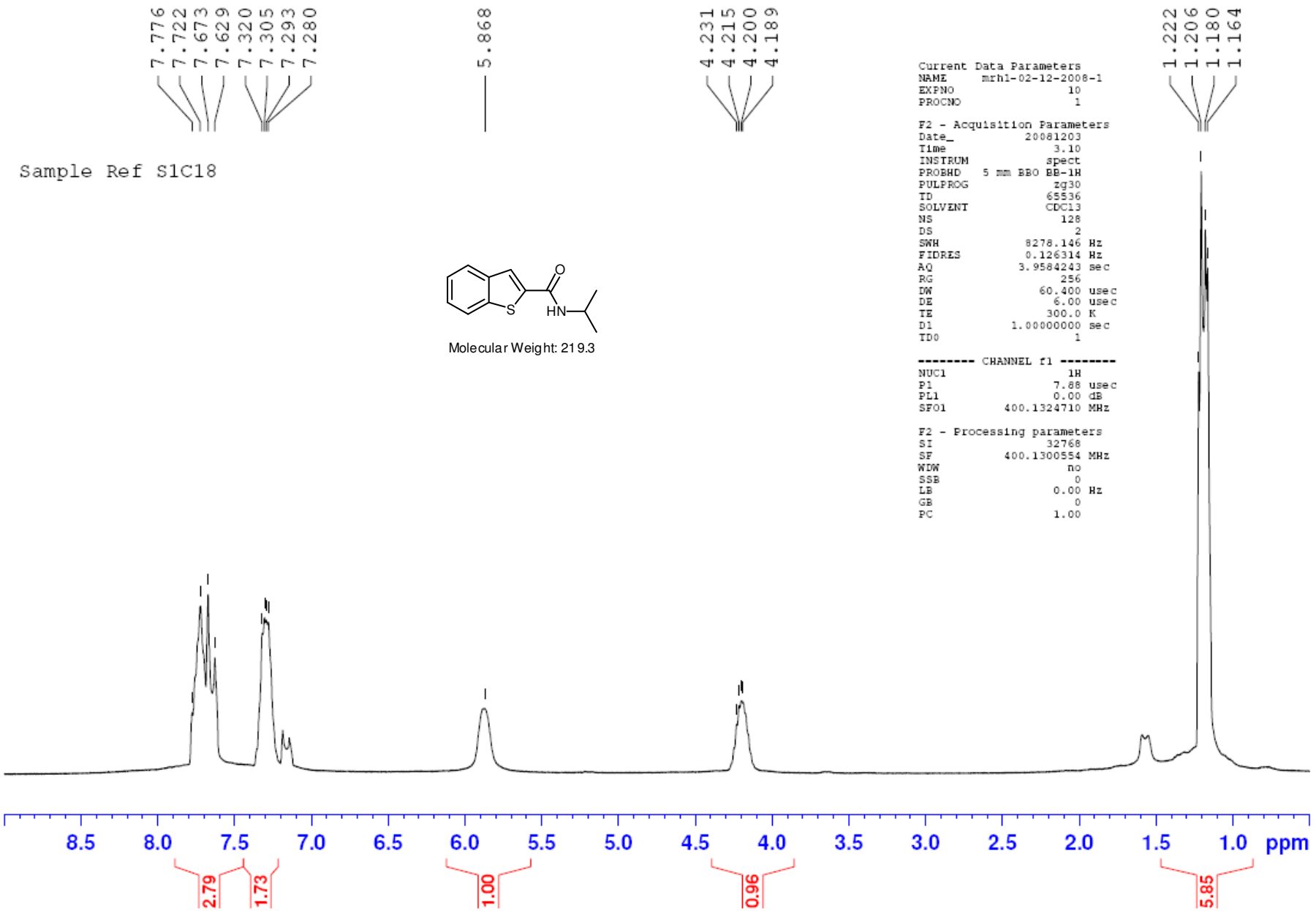
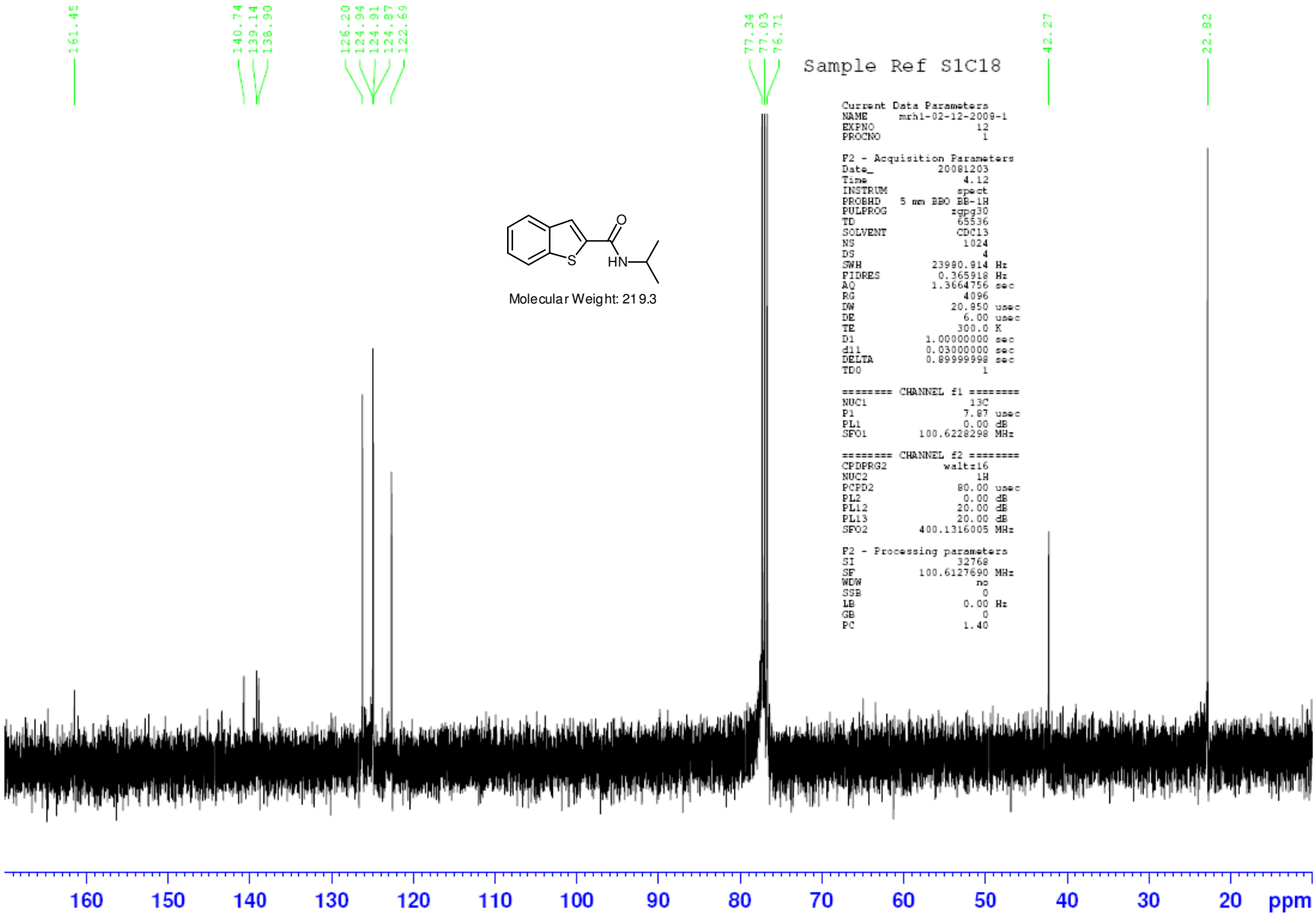


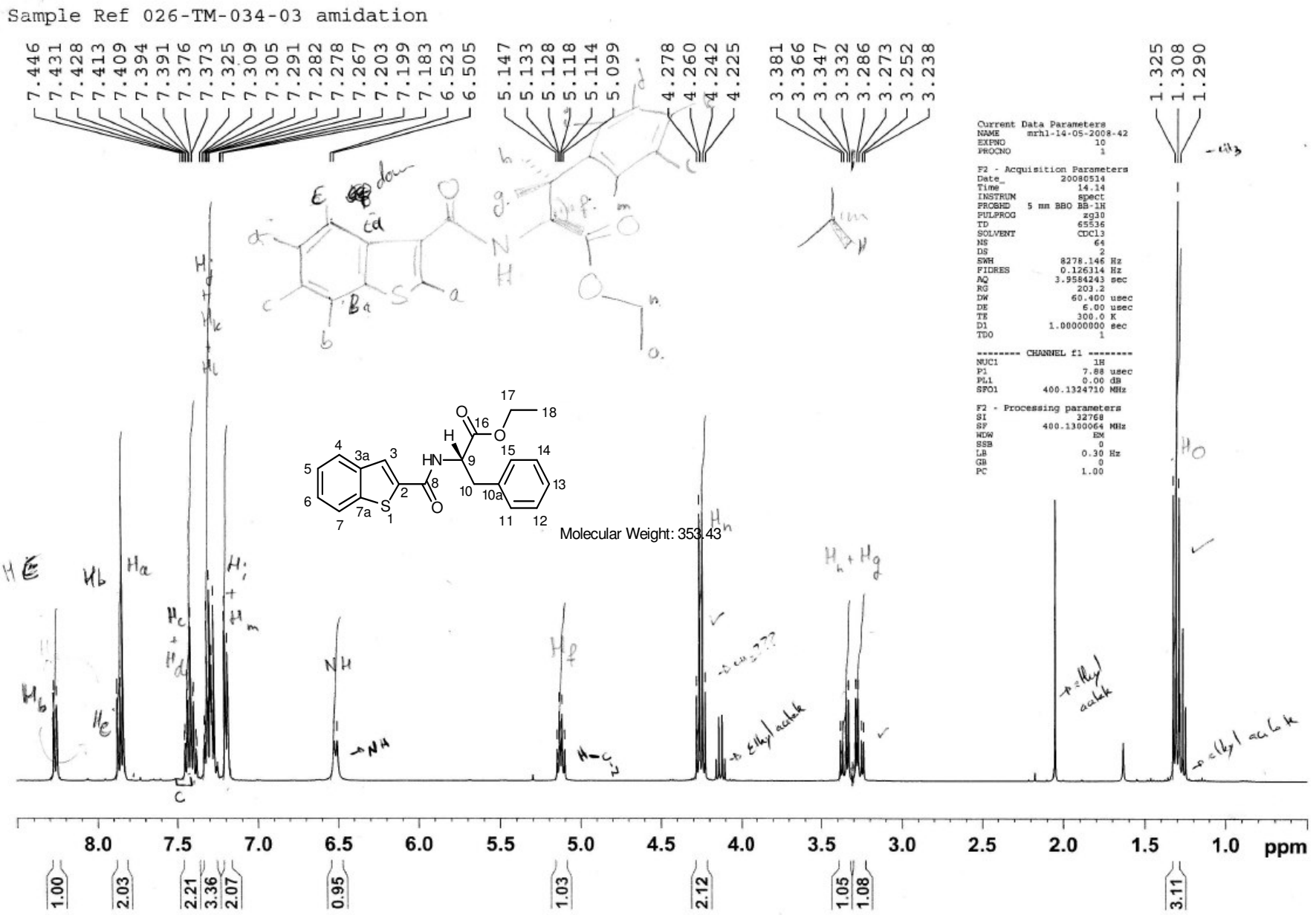
Molecular Weight: 298.32

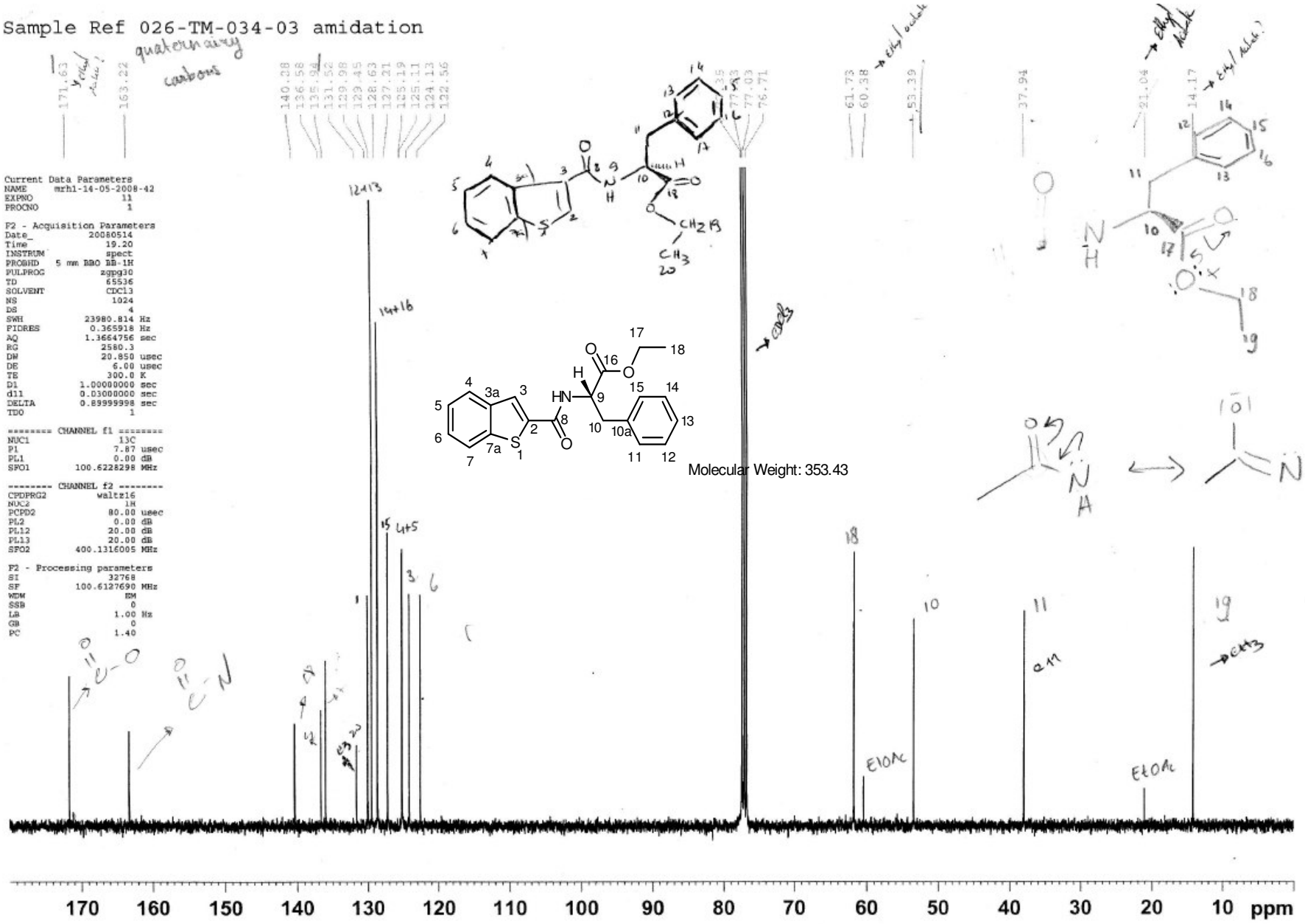

8.768
8.180
7.851
7.832
7.830
7.699
7.676
7.416
7.399
7.382
7.363
7.345
7.260
6.875
6.853
3.779
Sample Ref 001-DA-031-02
Current Data Parameters
NAME da1-03-10-2008-12
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081003
Time 15.50
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
HN
S
OCH3
Molecular Weight: 283.34
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
1.01
1.00
2.06
2.04
2.05
2.03
2.99

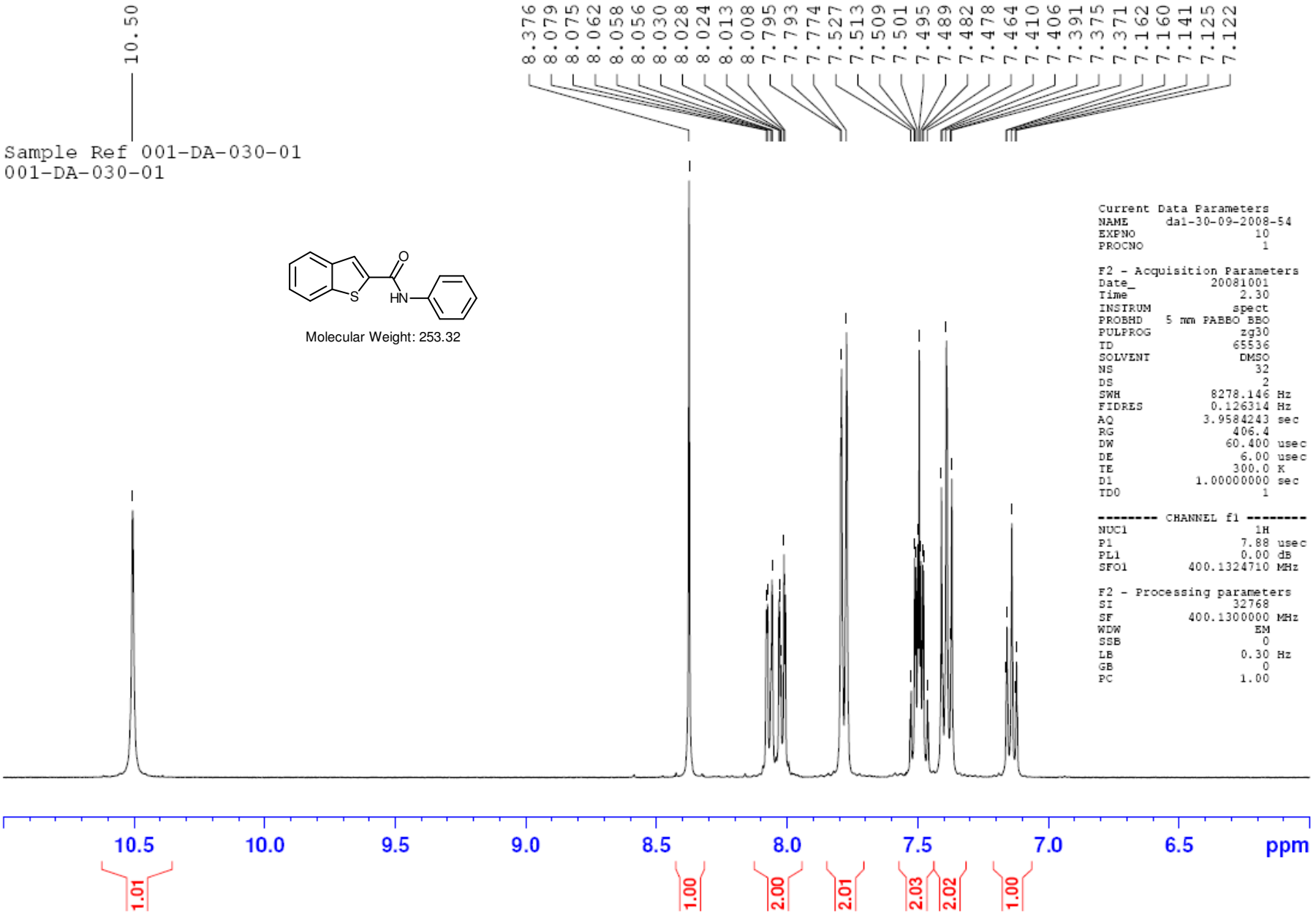

160.54
156.55
141.11
139.52
139.38
131.17
126.26
125.86
125.23
124.80
122.62
122.37
114.06
77.38
77.06
76.75
55.49
Sample Ref 001-DA-031-02
Molecular Weight: 283.34
Current Data Parameters
NAME da1-03-10-2008-12
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081006
Time 10.56
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 ppm

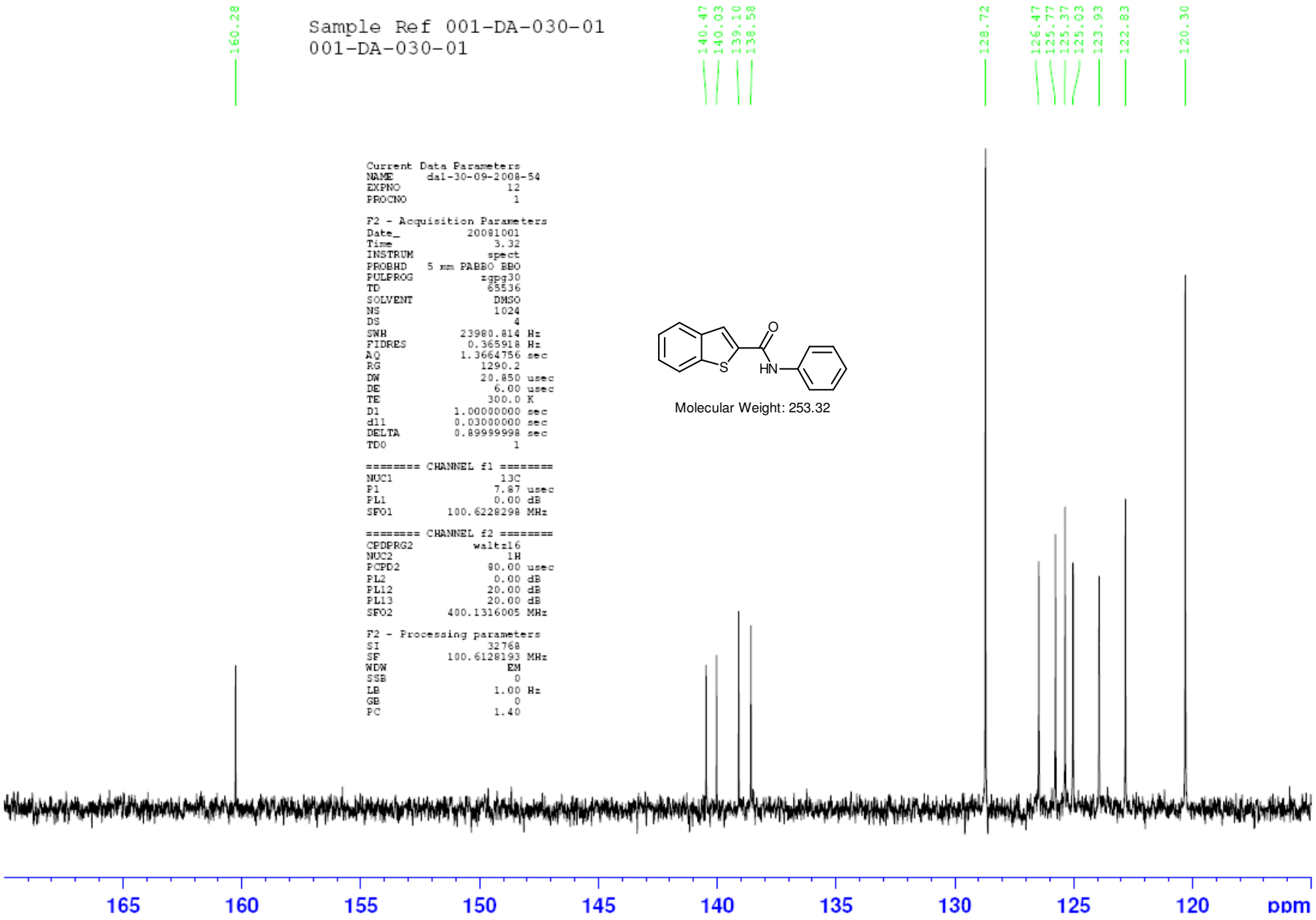

7.776
7.722
7.673
7.629
7.320
7.305
7.293
7.280
5.868
4.231
4.215
4.200
4.189
1.222
1.206
1.180
1.164
Sample Ref S1C18
Molecular Weight: 219.3
Current Data Parameters
NAME mrh1-02-12-2008-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081203
Time 3.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300554 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.79
1.73
1.00
0.96
5.85

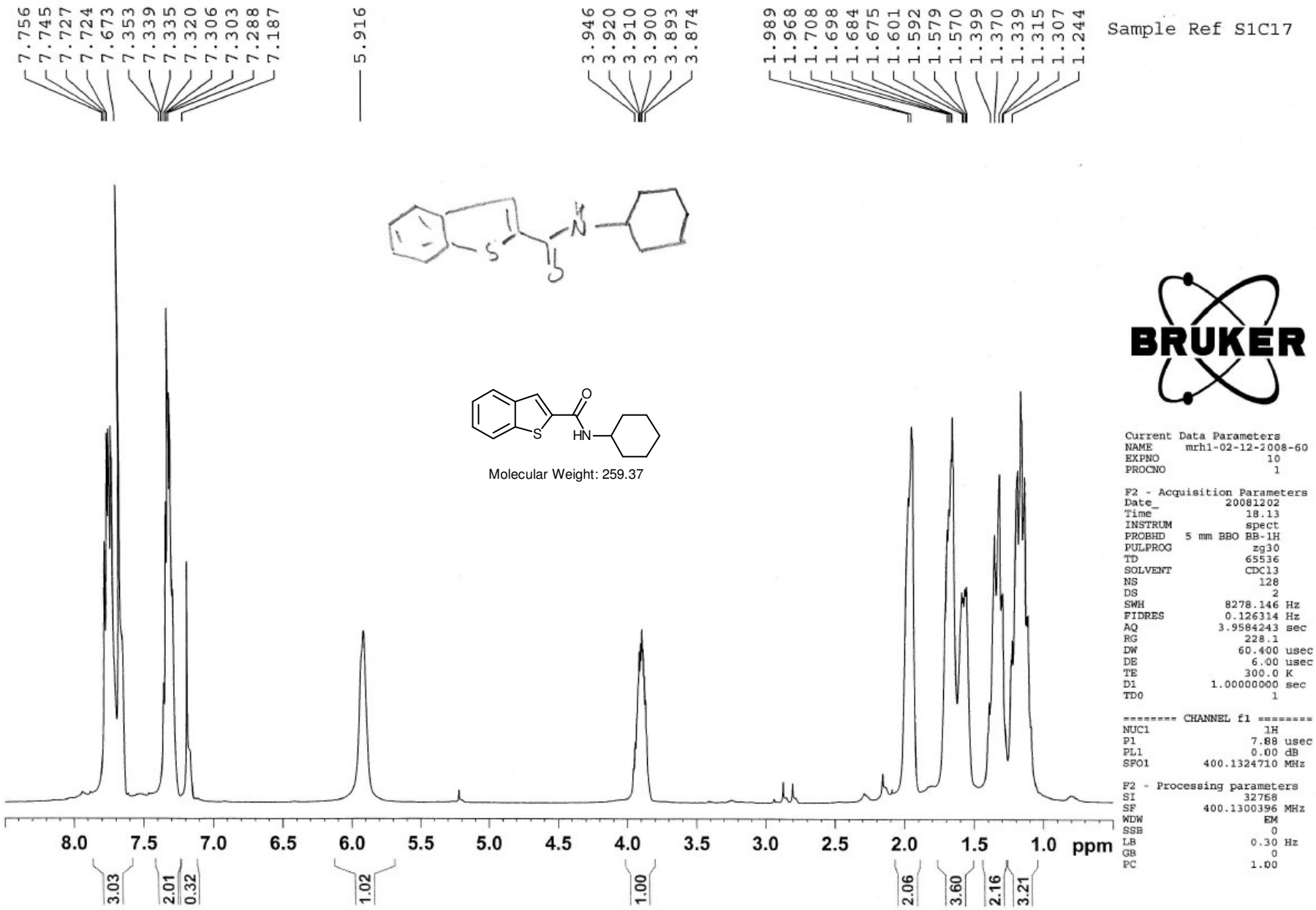

161.45
140.74
139.14
138.90
126.20
124.94
124.91
124.87
122.69
77.34
77.03
76.71
42.27
22.82
Sample Ref S1C18
O
S
HN
Molecular Weight: 219.3
Current Data Parameters
NAME mrh1-02-12-2008-1
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081203
Time 4.12
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.914 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

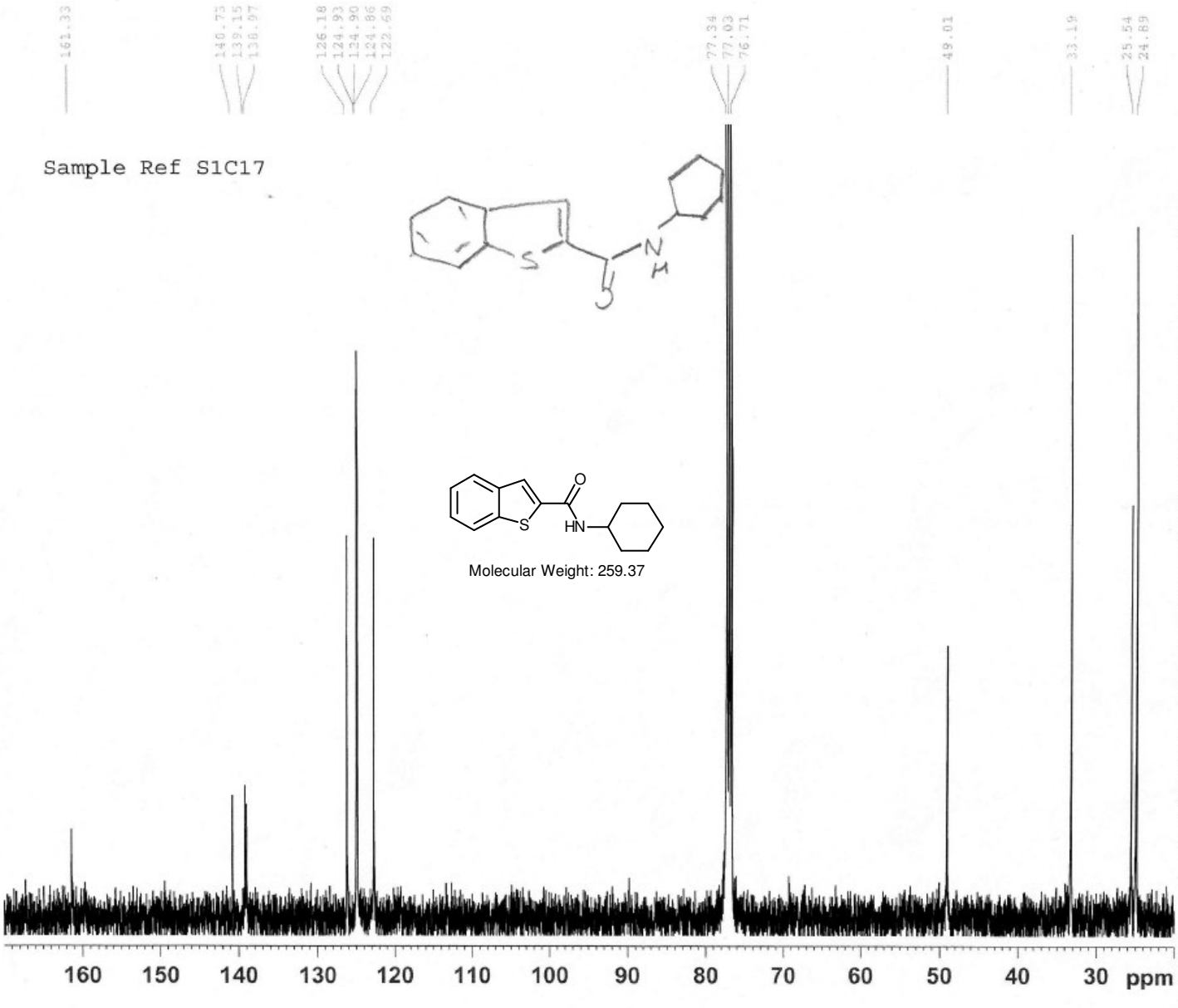

Sample Ref 026-TM-034-03 amidation
7.446 7.431 7.428 7.413 7.409 7.394 7.391 7.376 7.373 7.325 7.309 7.305 7.291 7.282 7.278 7.267 7.203 7.199 7.183 6.523 6.505
5.147 5.133 5.128 5.118 5.114 5.099
4.278 4.260 4.242 4.225
3.381 3.366 3.347 3.332 3.286 3.273 3.252 3.238
1.325 1.308 1.290
Current Data Parameters
NAME mrh1-14-05-2008-42
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080514
Time 14.14
INSTRUN spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Molecular Weight: 353.43
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 2.03 2.21 3.36 2.07 0.95 1.03 2.12 1.05 1.08 3.11


Sample Ref 026-TM-034-03 amidation
quaternary carbons
Current Data Parameters
NAME mrh1-14-05-2008-42
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080514
Time 19.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
Molecular Weight: 353.43
EtOAc
EtOAc
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


10.50
8.376
8.079
8.075
8.062
8.058
8.056
8.030
8.028
8.024
8.013
8.008
7.795
7.793
7.774
7.527
7.513
7.509
7.501
7.495
7.489
7.482
7.478
7.464
7.410
7.406
7.391
7.375
7.371
7.162
7.160
7.141
7.125
7.122
Sample Ref 001-DA-030-01
001-DA-030-01
Molecular Weight: 253.32
Current Data Parameters
NAME da1-30-09-2008-54
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081001
Time 2.30
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 406.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
ppm
1.01
1.00
2.00
2.01
2.03
2.02
1.00


160.28
140.47
140.03
139.10
138.58
128.72
126.47
125.77
125.37
125.03
123.93
122.83
120.30
Sample Ref 001-DA-030-01
001-DA-030-01
Current Data Parameters
NAME da1-30-09-2008-54
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081001
Time 3.32
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
S
HN
Molecular Weight: 253.32
165
160
155
150
145
140
135
130
125
120
ppm


7.756
7.745
7.727
7.724
7.673
7.353
7.339
7.335
7.320
7.306
7.303
7.288
7.187
5.916
3.946
3.920
3.910
3.900
3.893
3.874
1.989
1.968
1.708
1.698
1.684
1.675
1.601
1.592
1.579
1.570
1.399
1.370
1.339
1.315
1.307
1.244
Sample Ref S1C17
S
N
O
S
HN
O
Molecular Weight: 259.37
BRUKER
Current Data Parameters
NAME mrh1-02-12-2008-60
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081202
Time 18.13
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300396 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
3.03
2.01
0.32
1.02
1.00
2.06
3.60
2.16
3.21



BRUKER

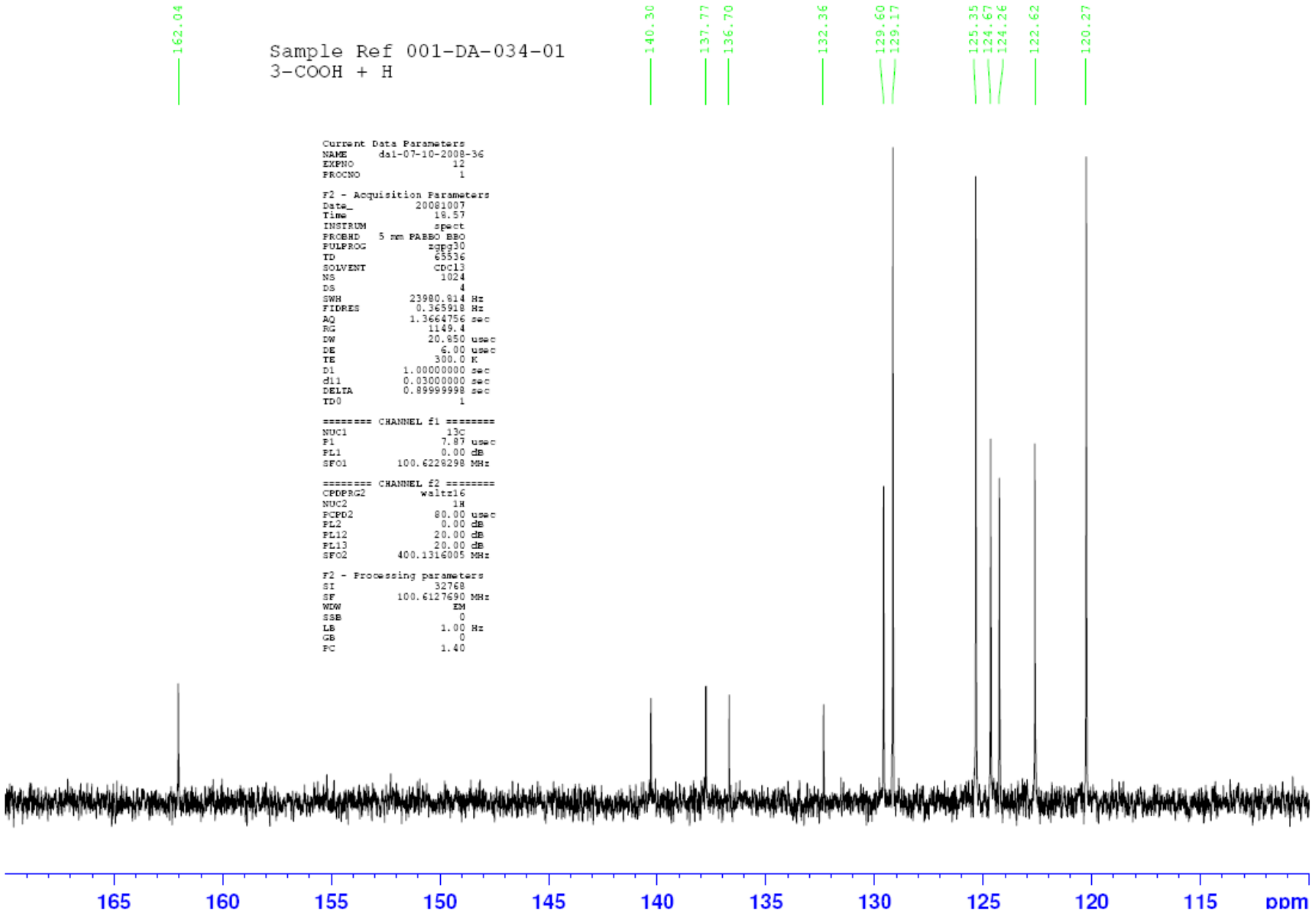
Current Data Parameters
NAME mrh1-02-12-2008-60
EXPNO 11
PROCNO 1

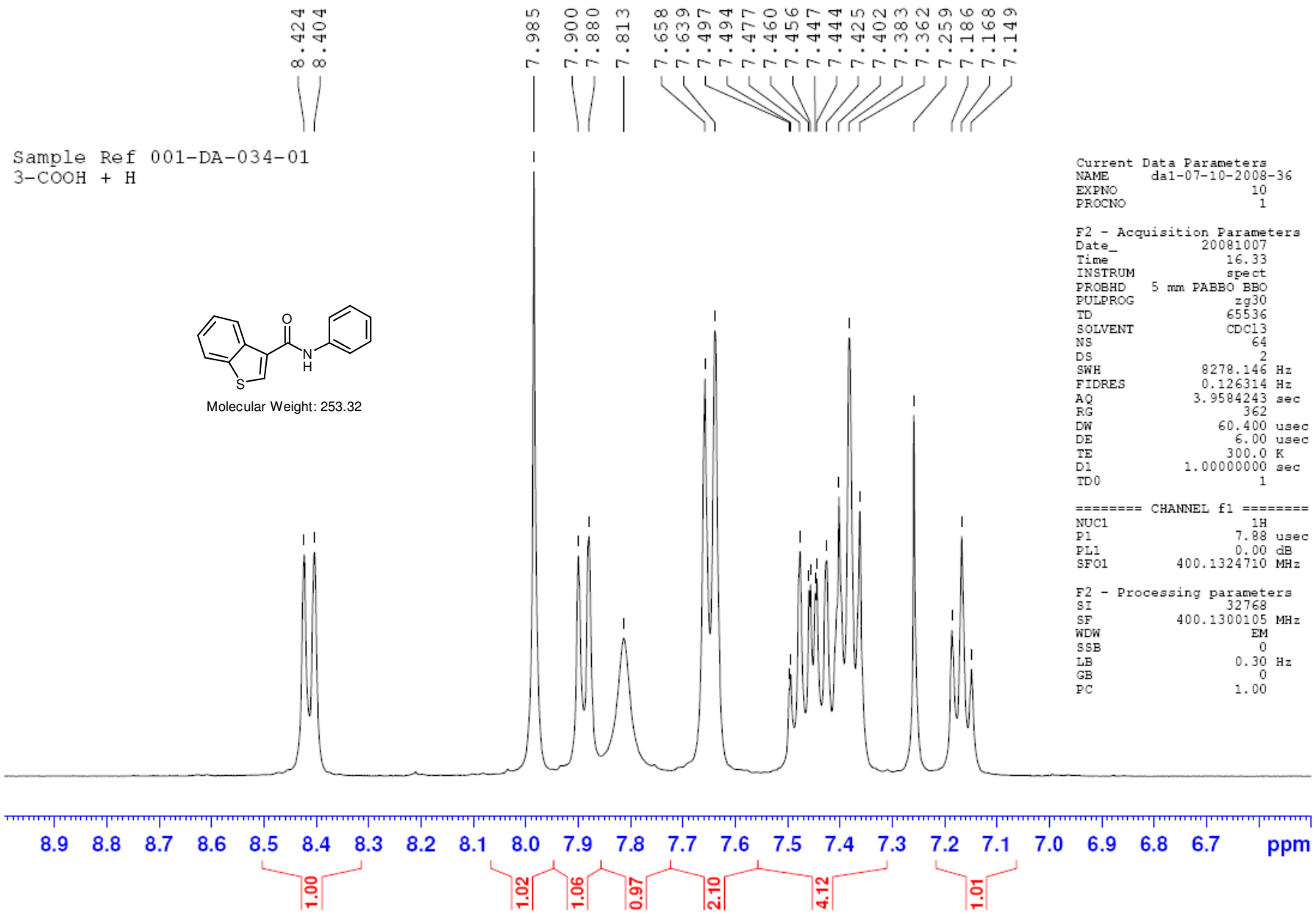
F2 - Acquisition Parameters
Date_ 20081202
Time 19.00
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5160.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

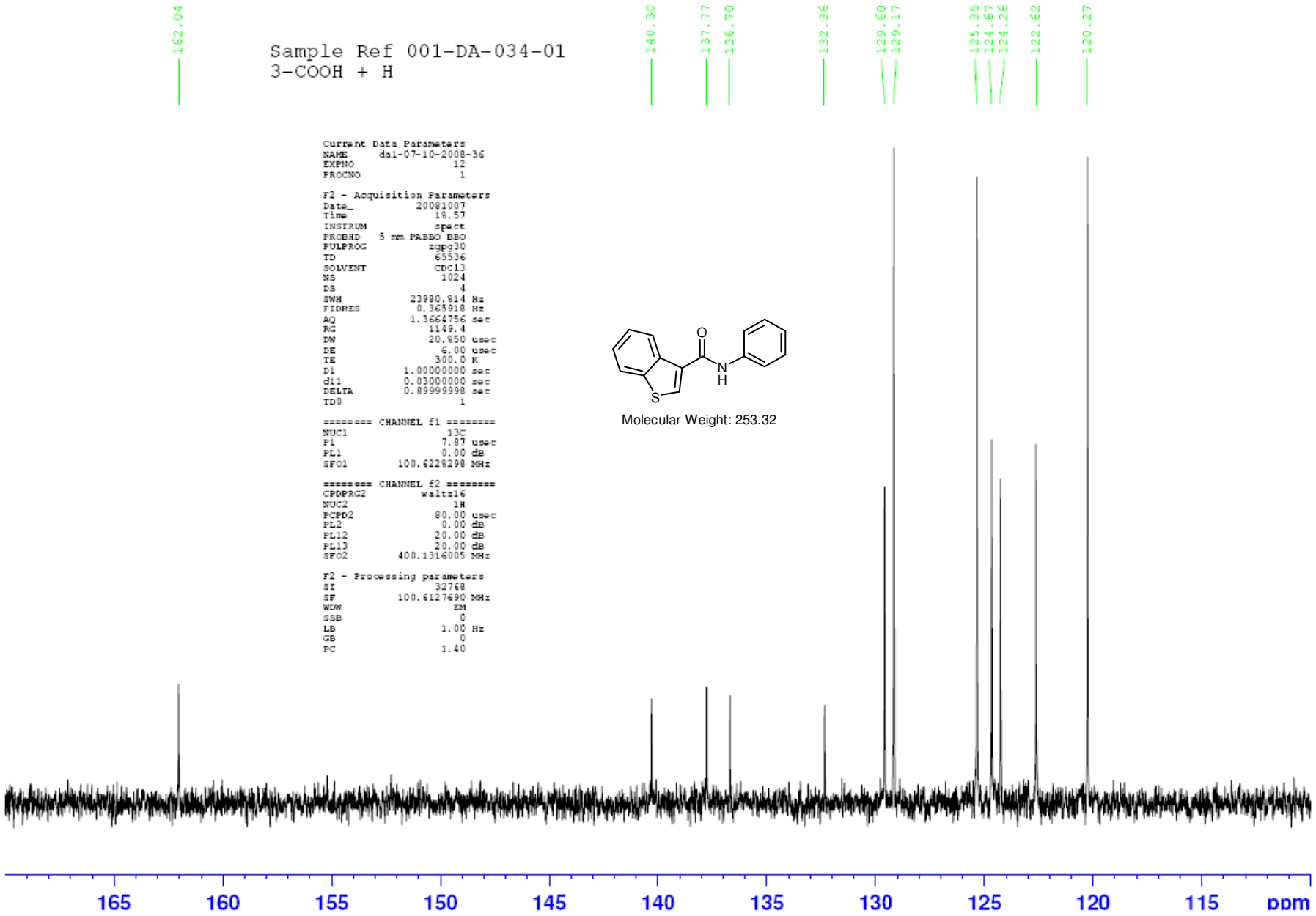
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

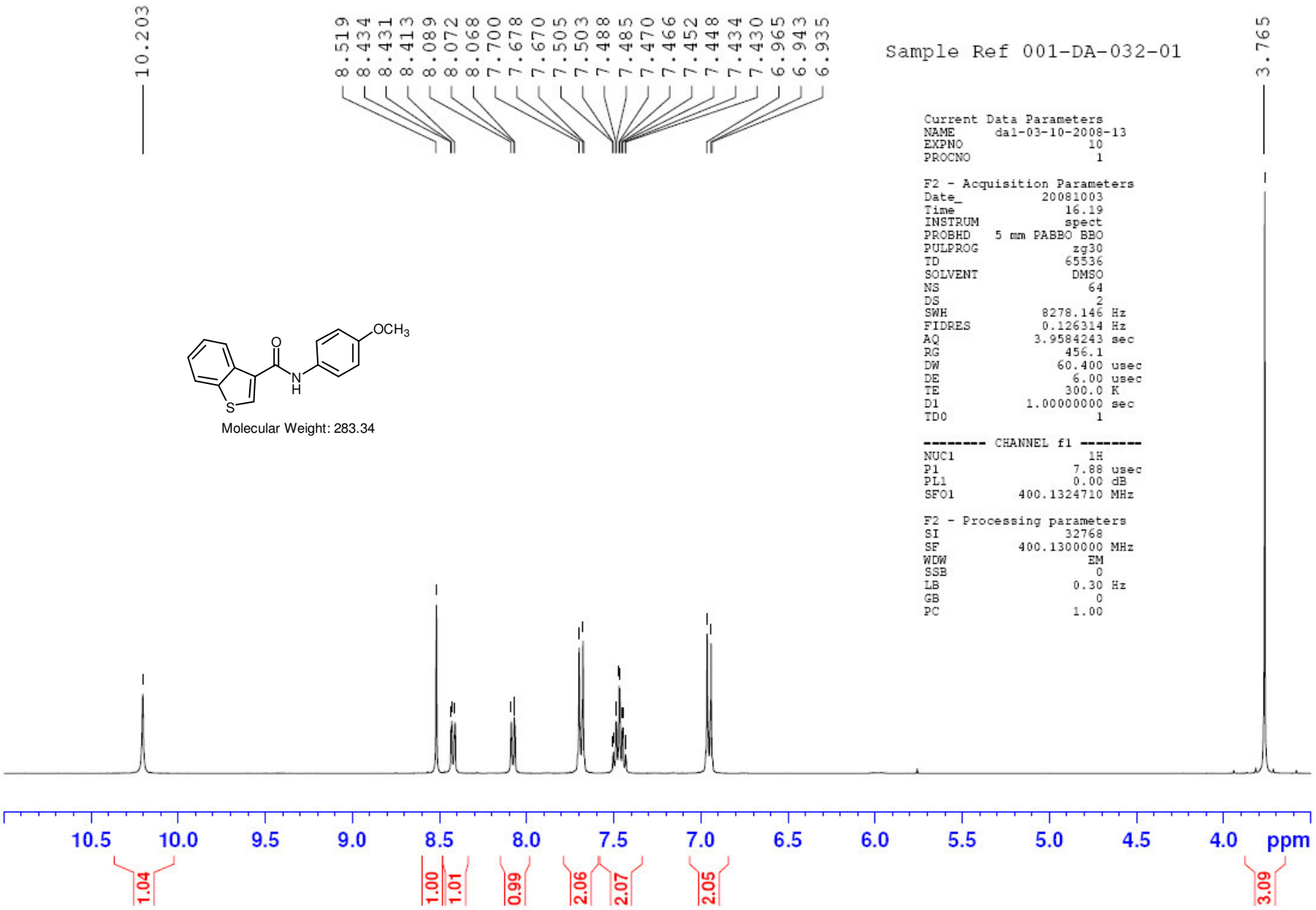
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

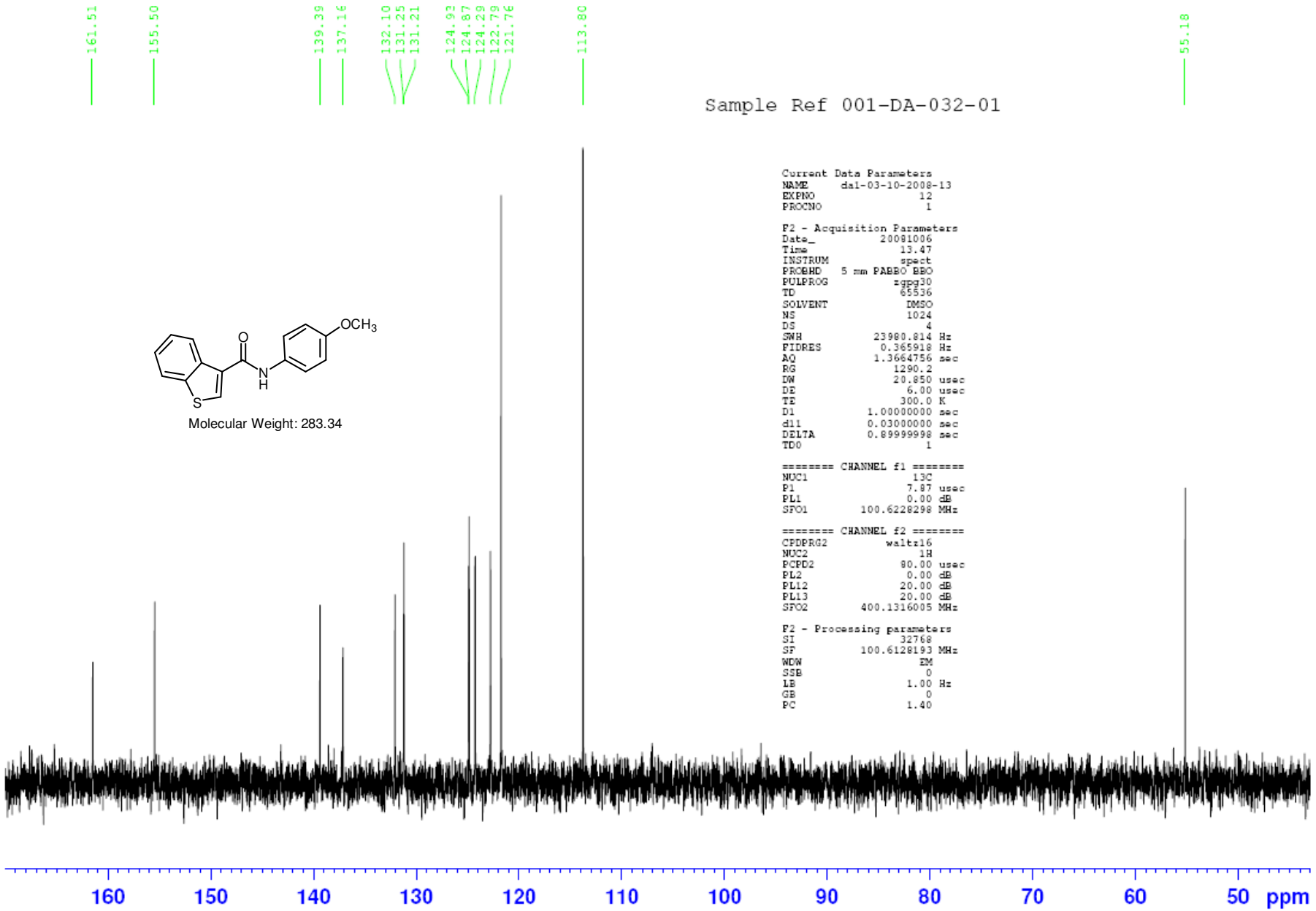
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


162.04
140.30
137.77
136.70
132.36
129.60
129.17
125.35
124.67
124.26
122.62
120.27
Sample Ref 001-DA-034-01
3-COOH + H
Current Data Parameters
NAME da1-07-10-2008-36
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081007
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13c
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
165
160
155
150
145
140
135
130
125
120
115
ppm

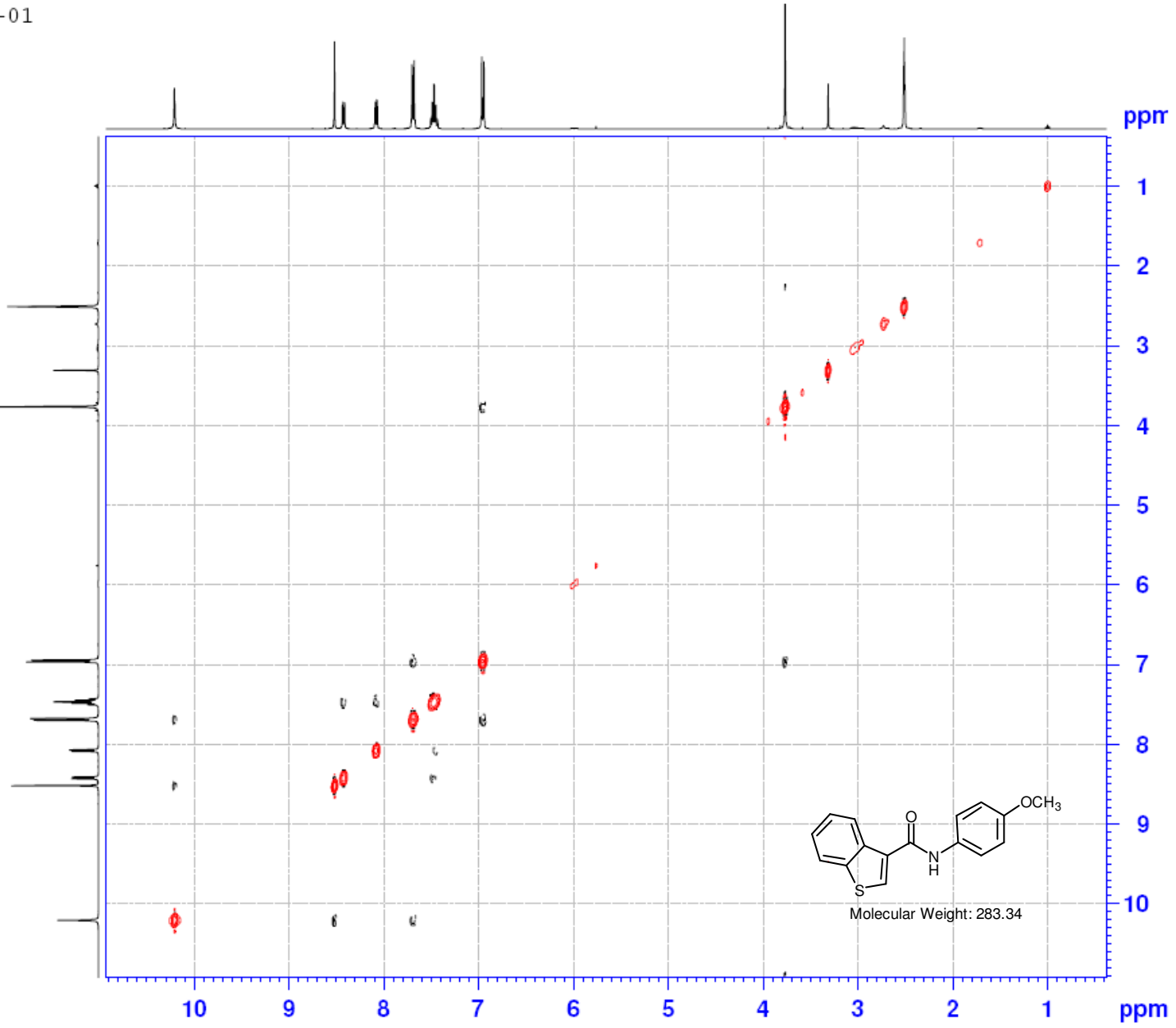

8.424
8.404
7.985
7.900
7.880
7.813
7.658
7.639
7.497
7.494
7.477
7.460
7.456
7.447
7.444
7.425
7.402
7.383
7.362
7.259
7.186
7.168
7.149
Sample Ref 001-DA-034-01
3-COOH + H
Molecular Weight: 253.32
Current Data Parameters
NAME da1-07-10-2008-36
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081007
Time 16.33
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 ppm
1.00
1.02
1.06
0.97
2.10
4.12
1.01


162.04
140.30
137.77
136.70
132.36
129.60
129.17
125.35
124.67
124.26
122.62
120.27
Sample Ref 001-DA-034-01
3-COOH + H
Current Data Parameters
NAME da1-07-10-2008-36
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081007
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
O
N
H
S
Molecular Weight: 253.32
165
160
155
150
145
140
135
130
125
120
115
ppm

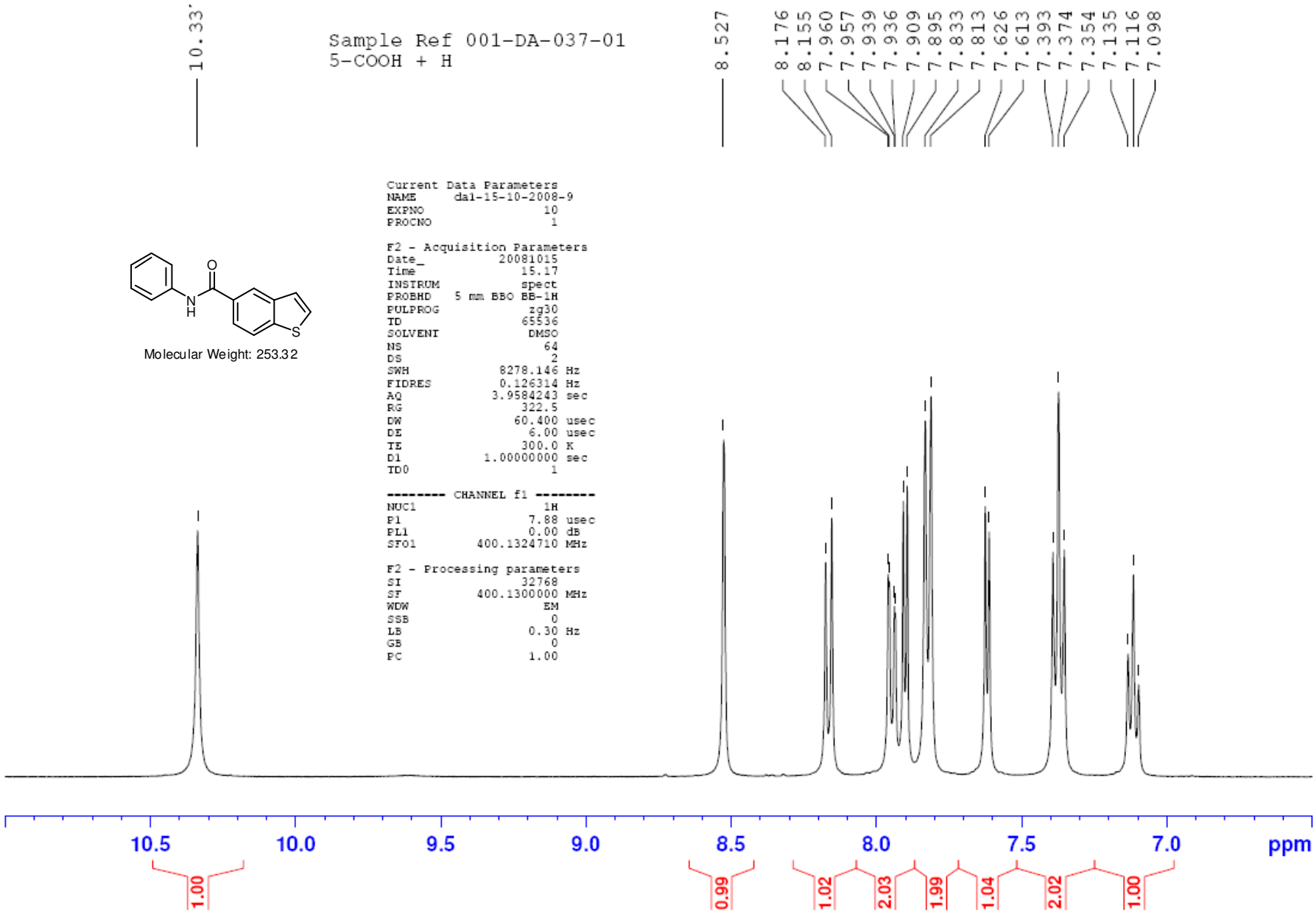

10.203
8.519
8.434
8.431
8.413
8.089
8.072
8.068
7.700
7.678
7.670
7.505
7.503
7.488
7.485
7.470
7.466
7.452
7.448
7.434
7.430
6.965
6.943
6.935
3.765
Sample Ref 001-DA-032-01
Current Data Parameters
NAME da1-03-10-2008-13
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081003
Time 16.19
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
H
S
OCH3
Molecular Weight: 283.34
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
1.04
1.00
1.01
0.99
2.06
2.07
2.05
3.09

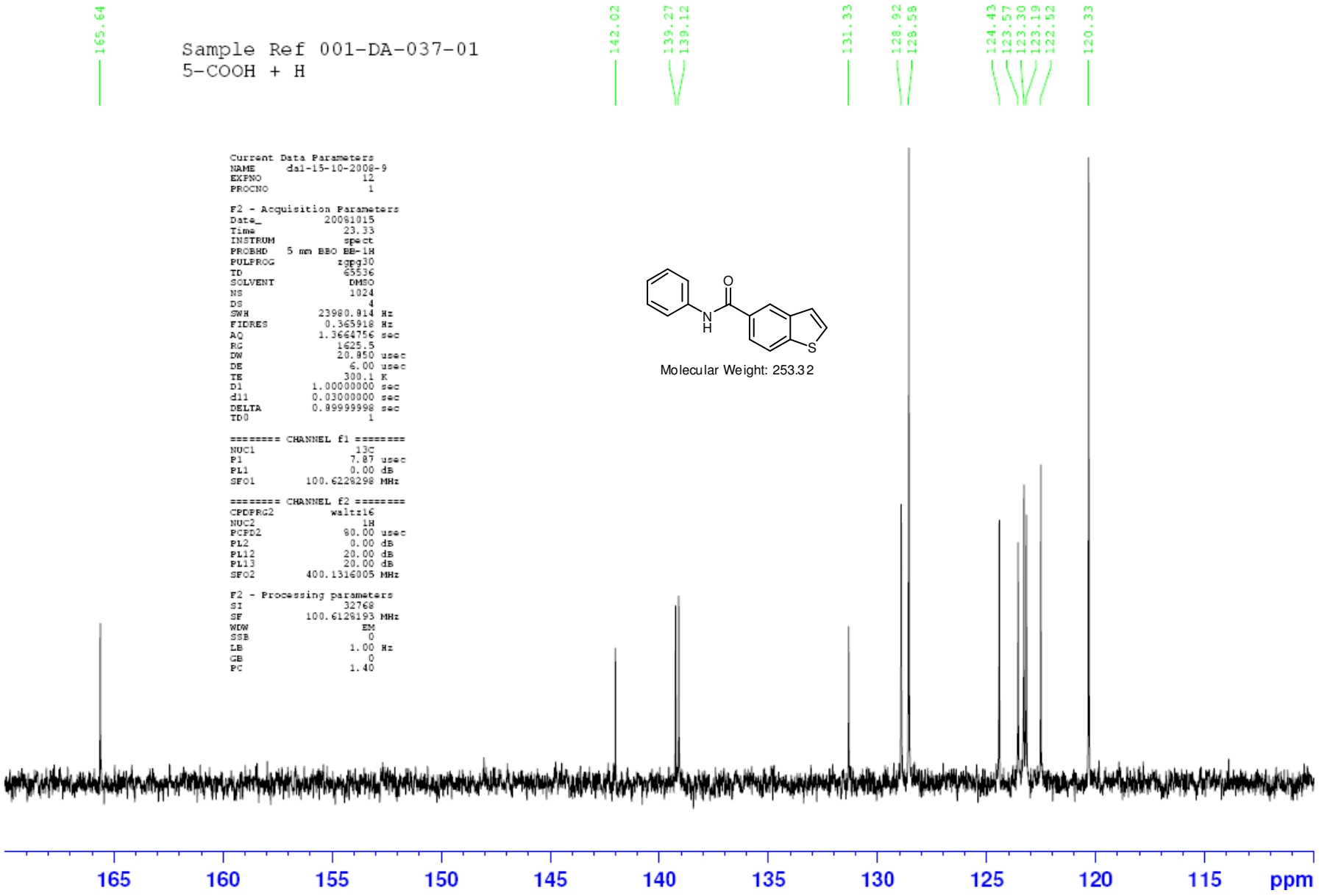

161.51
155.50
139.39
137.16
132.10
131.25
131.21
124.93
124.87
124.29
122.79
121.76
113.80
55.18
Sample Ref 001-DA-032-01
Current Data Parameters
NAME da1-03-10-2008-13
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081006
Time 13.47
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
H
S
OCH3
Molecular Weight: 283.34
160
150
140
130
120
110
100
90
80
70
60
50
ppm

Sample Ref 001-DA-032-01

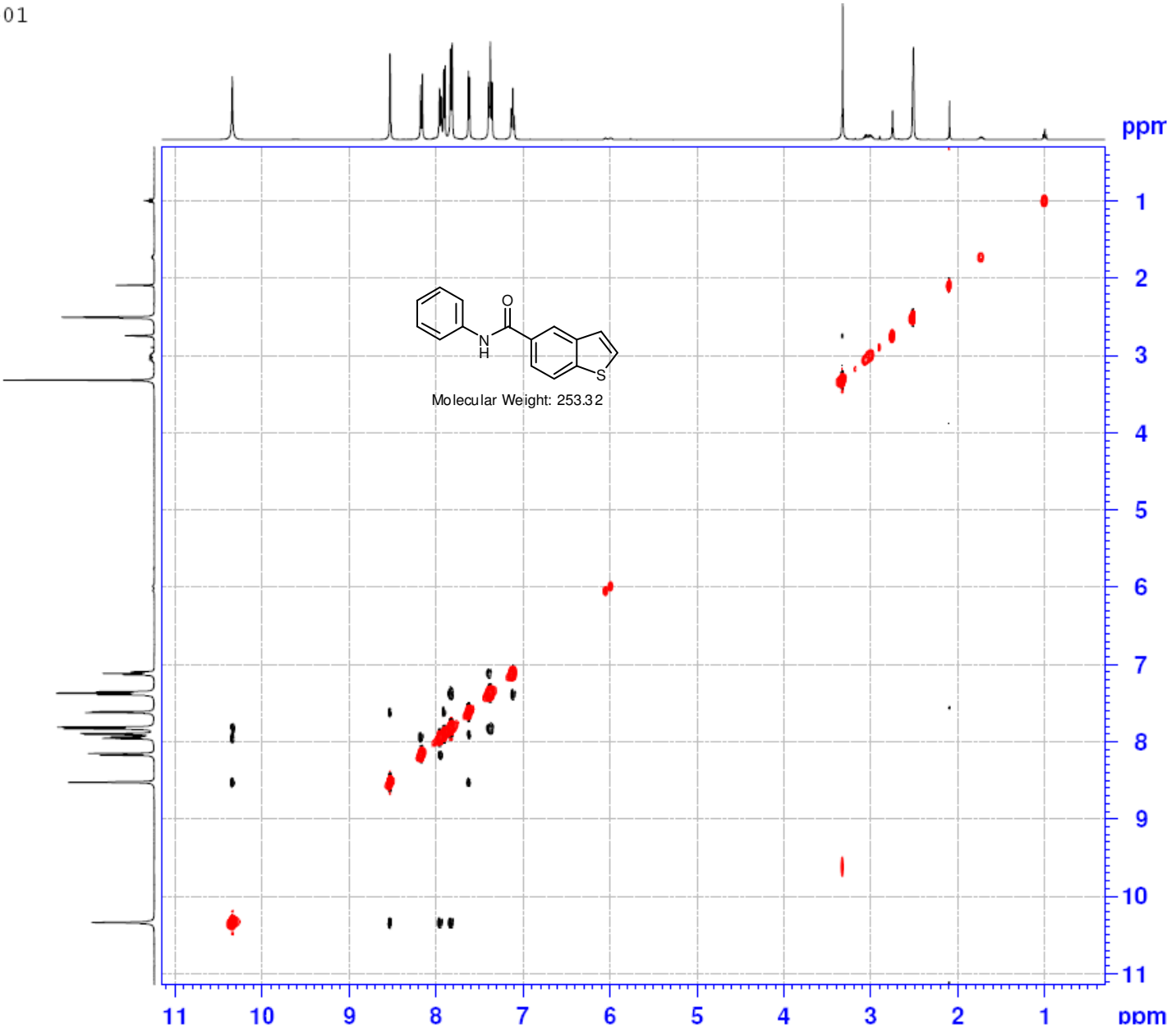



```
Current Data Parameters
NAME      da1-03-10-2008-13
EXPNO                   16
PROCNO                   1

F2 - Acquisition Parameters
Date_           20081006
Time               15.45
INSTRUM            spect
PROBHD    5 mm PABBO BBO
PULPROG          noesyph
TD                  2048
SOLVENT             DMSO
NS                     4
DS                     4
SWH             4222.973 Hz
FIDRES          2.061999 Hz
AQ              0.2425332 sec
RG                   256
DW                118.400 usec
DE                  6.00 usec
TE                 300.0 K
d0            0.00010837 sec
D1            1.27454603 sec
D8            0.80000001 sec
INO           0.00023680 sec
ST1CNT               128

-------- CHANNEL f1 --------
NUC1                 1H
P1                7.88 usec
PL1                0.00 dB
SFO1        400.1322609 MHz

F1 - Acquisition parameters
NDO                  1
TD                 256
SFO1          400.1323 MHz
FIDRES         16.495989 Hz
SW               10.554 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                1024
SF          400.1300000 MHz
WDW               QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
PC                 1.00

F1 - Processing parameters
SI                1024
MC2          States-TPPI
SF          400.1300000 MHz
WDW               QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
```

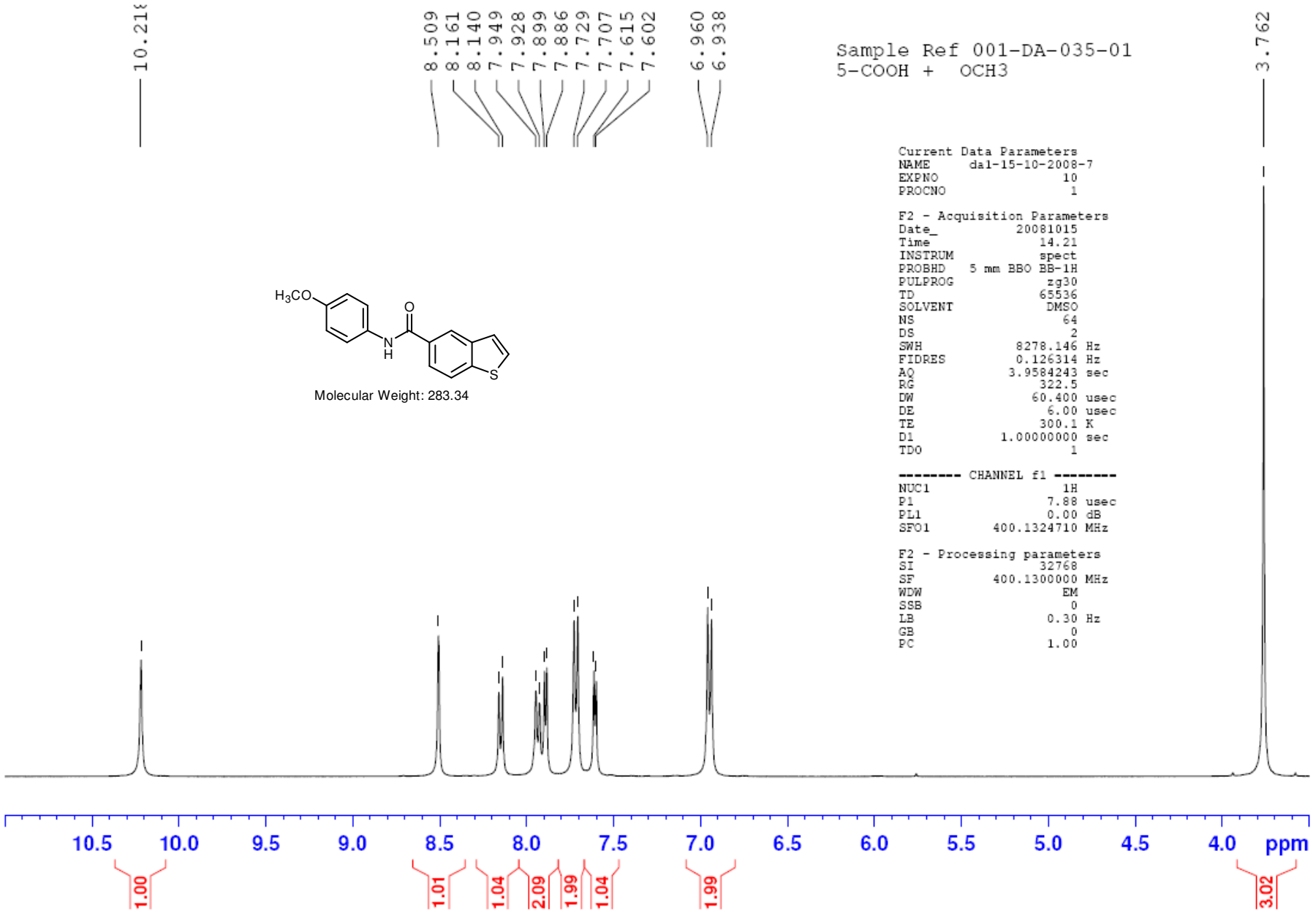

10.33
Sample Ref 001-DA-037-01
5-COOH + H
8.527
8.176
8.155
7.960
7.957
7.939
7.936
7.909
7.895
7.833
7.813
7.626
7.613
7.393
7.374
7.354
7.135
7.116
7.098
Current Data Parameters
NAME da1-15-10-2008-9
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081015
Time 15.17
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
H
S
Molecular Weight: 253.32
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
ppm
1.00
0.99
1.02
2.03
1.99
1.04
2.02
1.00

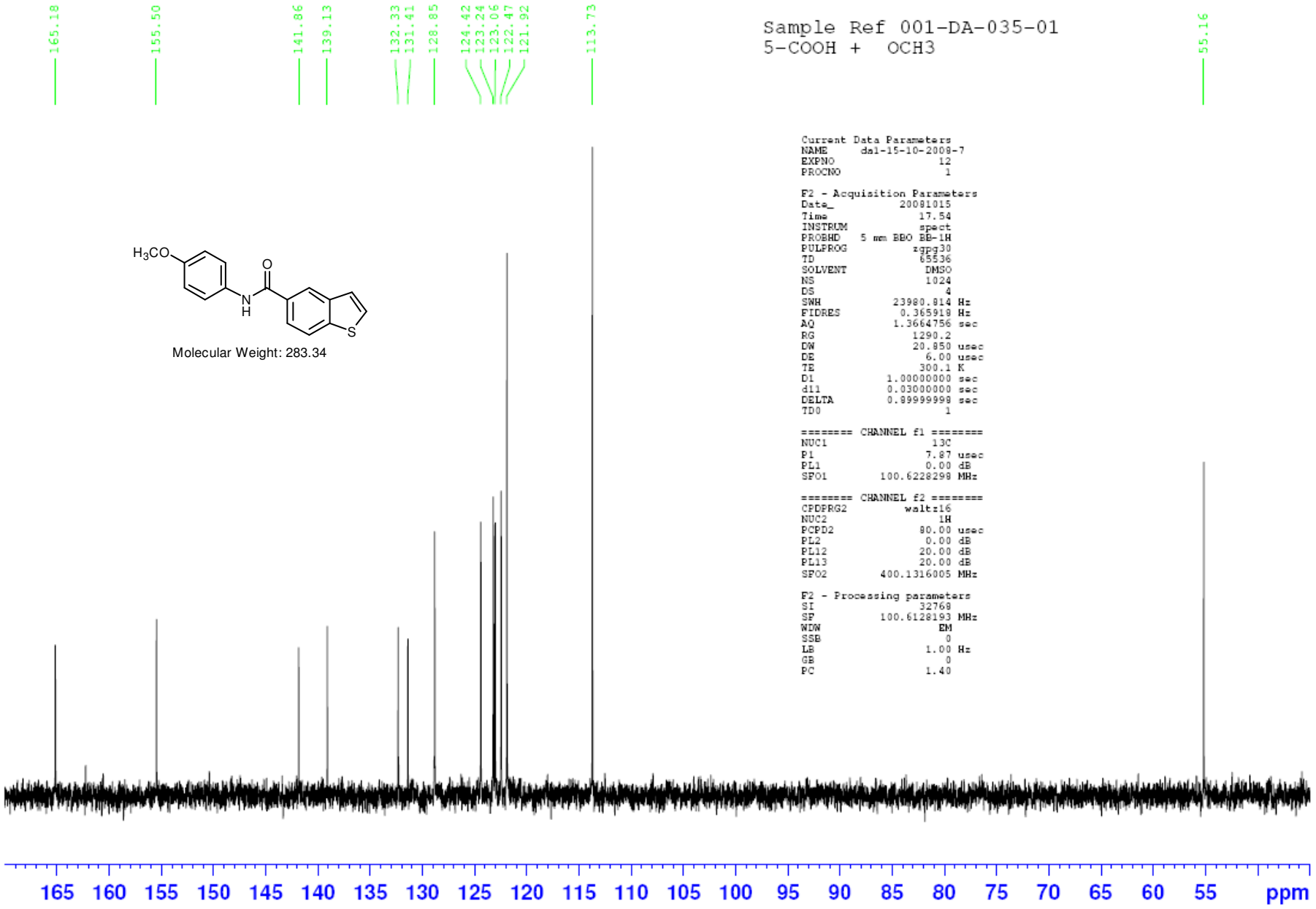

165.64
142.02
139.27
139.12
131.33
128.92
128.58
124.43
123.57
123.30
123.19
122.52
120.33
Sample Ref 001-DA-037-01
5-COOH + H
Current Data Parameters
NAME dal-15-10-2008-9
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081015
Time 23.33
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
H
S
Molecular Weight: 253.32
165
160
155
150
145
140
135
130
125
120
115
ppm

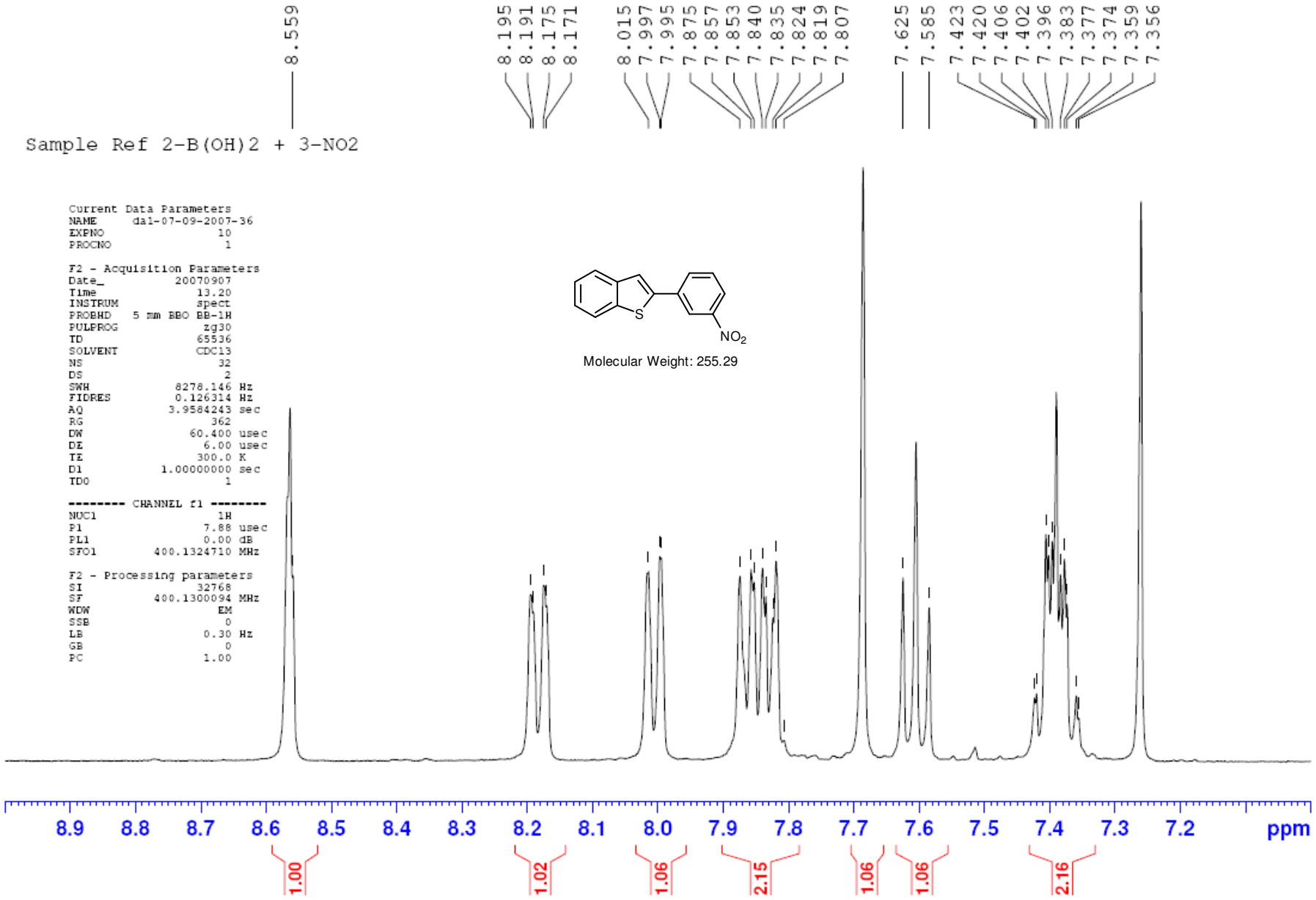
Sample Ref 001-DA-037-01
5-COOH + H



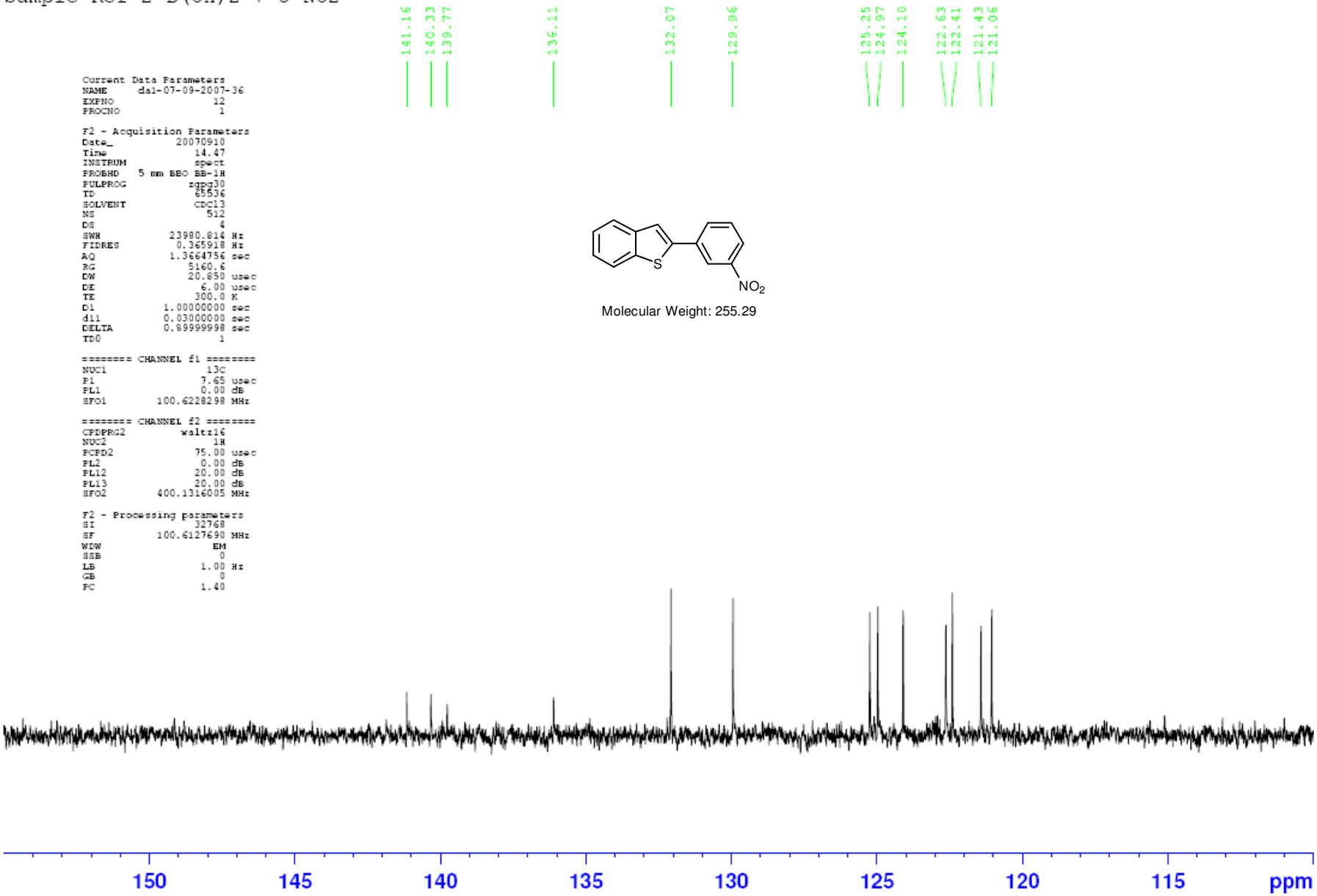

```
Current Data Parameters
NAME      da1-15-10-2008-9
EXPNO                   16
PROCNO                   1

F2 - Acquisition Parameters
Date_           20081016
Time                0.54
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          noesyph
TD                  2048
SOLVENT             DMSO
NS                     4
DS                     4
SWH              4340.278 Hz
FIDRES           2.119276 Hz
AQ               0.2359796 sec
RG                 143.7
DW                 115.200 usec
DE                   6.00 usec
TE                 300.0 K
d0             0.00010517 sec
D1             1.28068995 sec
D8             0.80000001 sec
IN0            0.00023040 sec
ST1CNT                128

-------- CHANNEL f1 --------
NUC1                   1H
P1                   7.88 usec
PL1                  0.00 dB
SFO1         400.1322912 MHz

F1 - Acquisition parameters
ND0                    1
TD                   256
SFO1             400.1323 MHz
FIDRES          16.954210 Hz
SW                10.847 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                  1024
SF           400.1300000 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.00

F1 - Processing parameters
SI                  1024
MC2          States-TPPI
SF           400.1300000 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
```


10.218
8.509
8.161
8.140
7.949
7.928
7.899
7.886
7.729
7.707
7.615
7.602
6.960
6.938
3.762
Sample Ref 001-DA-035-01
5-COOH + OCH3
Current Data Parameters
NAME da1-15-10-2008-7
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081015
Time 14.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3CO
O
N
H
S
Molecular Weight: 283.34
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
1.00
1.01
1.04
2.09
1.99
1.04
1.99
3.02


165.18
155.50
141.86
139.13
132.33
131.41
128.85
124.42
123.24
123.06
122.47
121.92
113.73
55.16
Sample Ref 001-DA-035-01
5-COOH + OCH3
H3CO
O
N
H
S
Molecular Weight: 283.34
Current Data Parameters
NAME da1-15-10-2008-7
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081015
Time 17.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.99999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 ppm

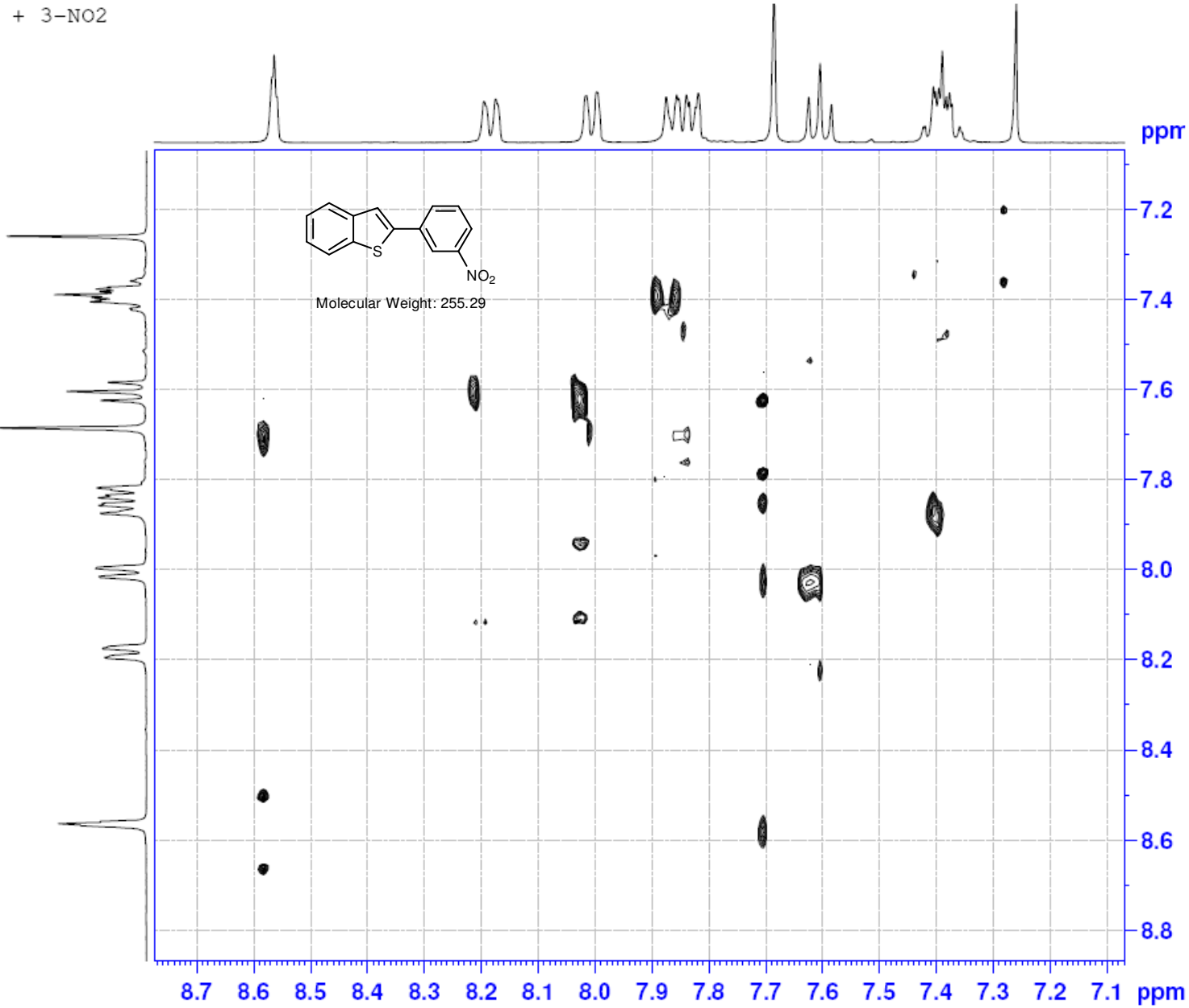

8.559
8.195
8.191
8.175
8.171
8.015
7.997
7.995
7.875
7.857
7.853
7.840
7.835
7.824
7.819
7.807
7.625
7.585
7.423
7.420
7.406
7.402
7.396
7.383
7.377
7.374
7.359
7.356
Sample Ref 2-B(OH)2 + 3-NO2
Current Data Parameters
NAME da1-07-09-2007-36
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070907
Time 13.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
S
NO2
Molecular Weight: 255.29
8.9
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
ppm
1.00
1.02
1.06
2.15
1.06
1.06
2.16

Sample Ref 2-B(OH)2 + 3-NO2

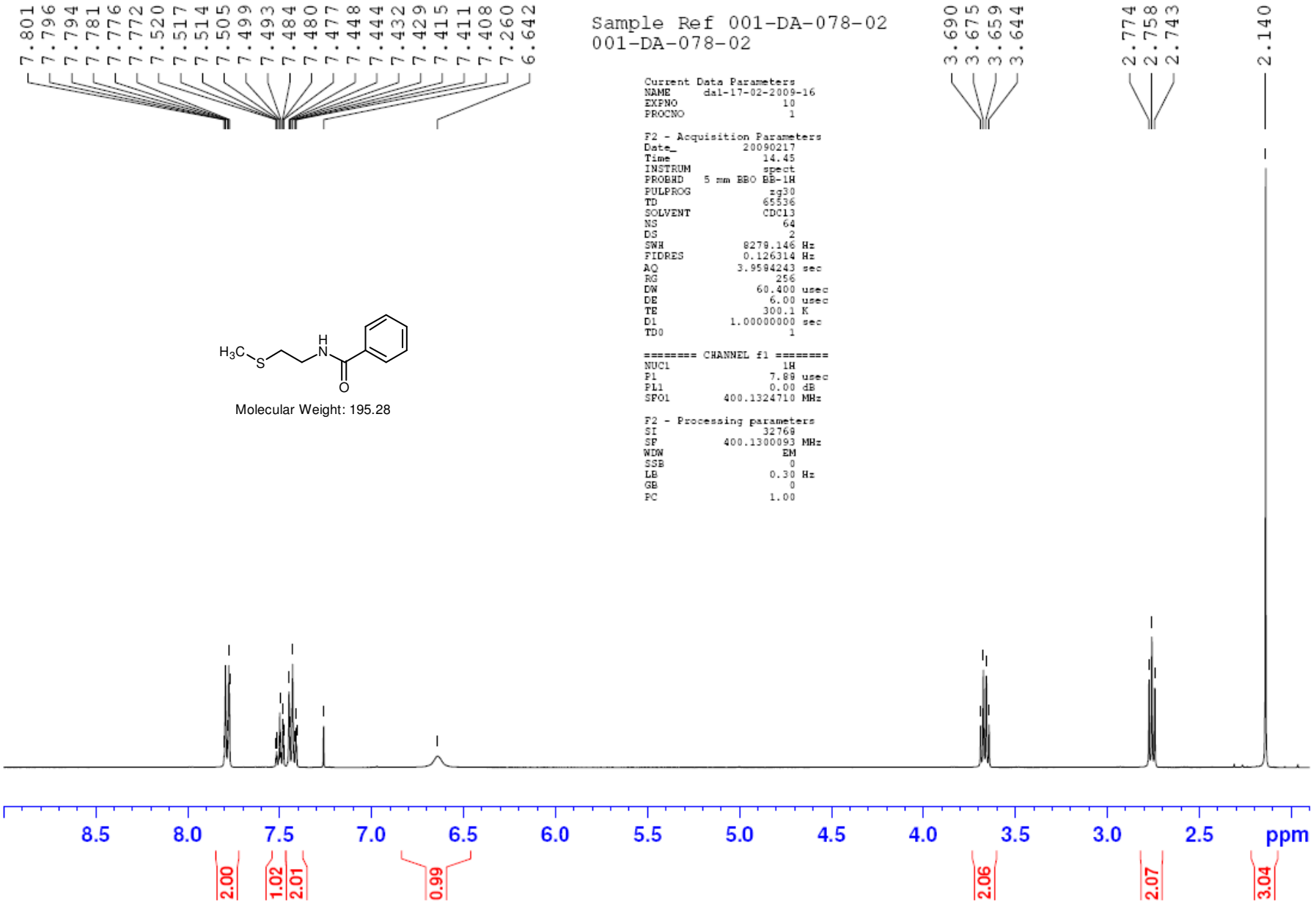


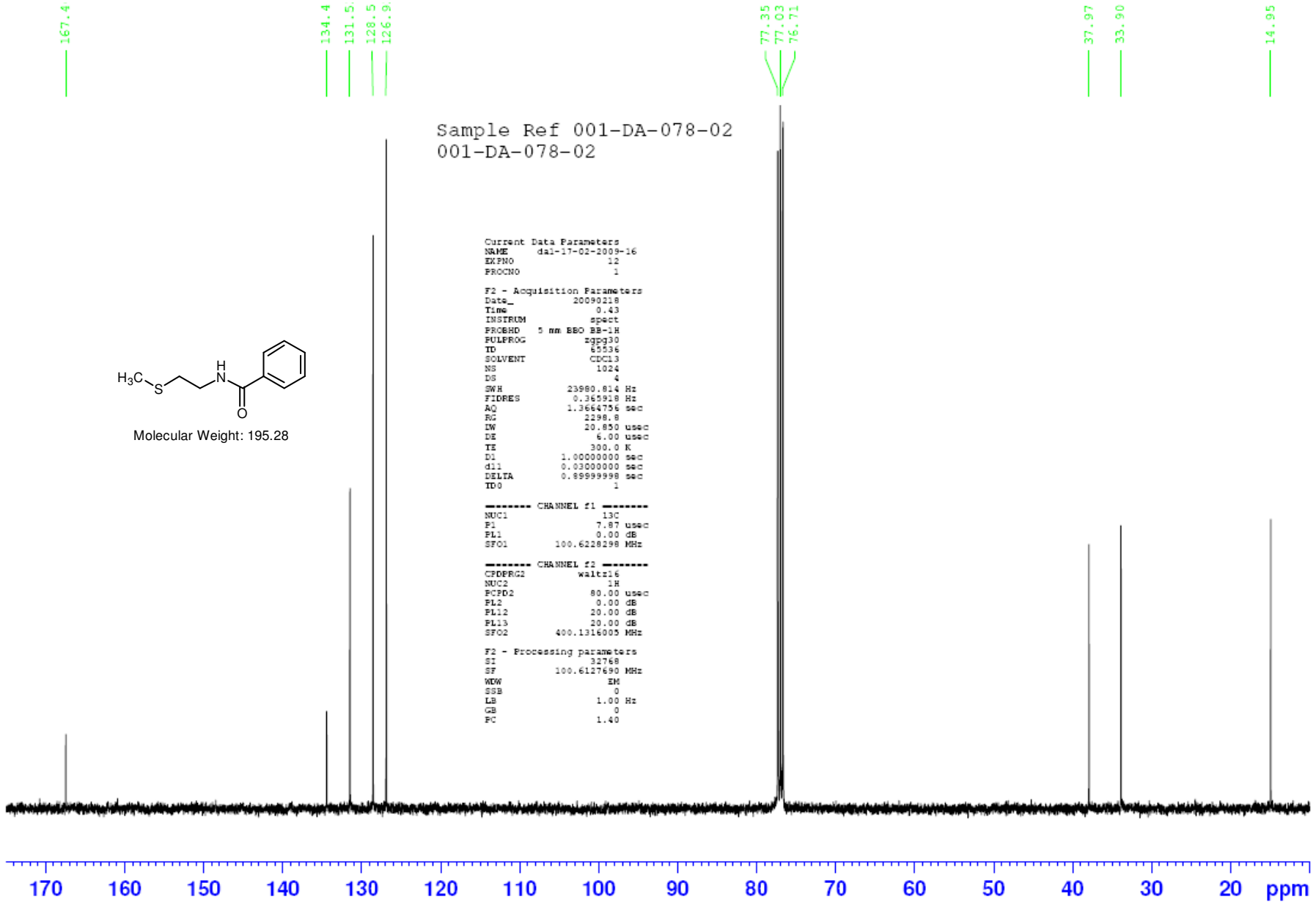

```
Current Data Parameters
NAME      da1-07-09-2007-36
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_              20070910
Time                  14.47
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH            23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                5160.6
DW                20.850 usec
DE                 6.00 usec
TE                300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
DELTA         0.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.65 usec
PL1                  0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2               75.00 usec
PL2                  0.00 dB
PL12                20.00 dB
PL13                20.00 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
```

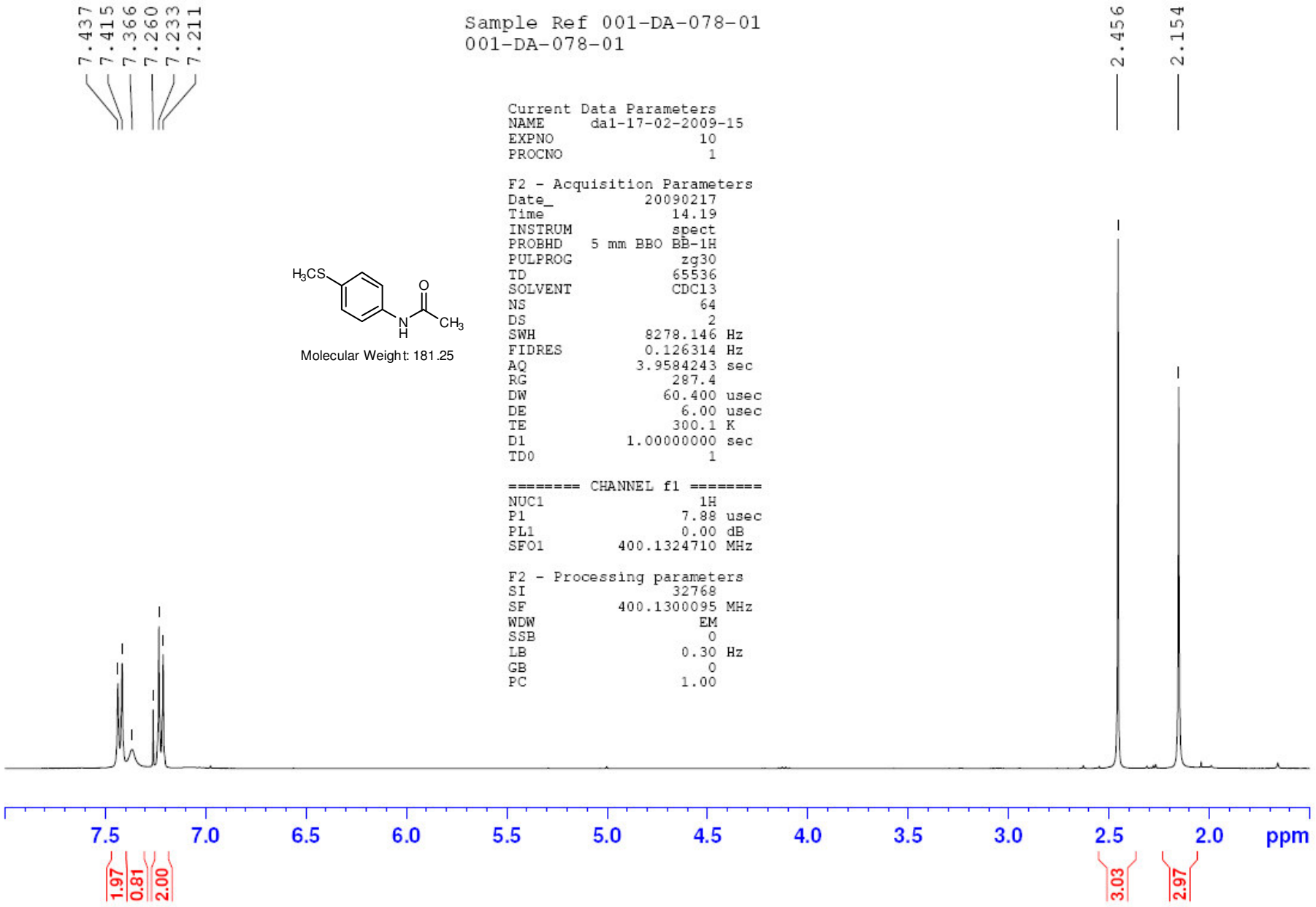
Sample Ref 2-B(OH)2 + 3-NO2

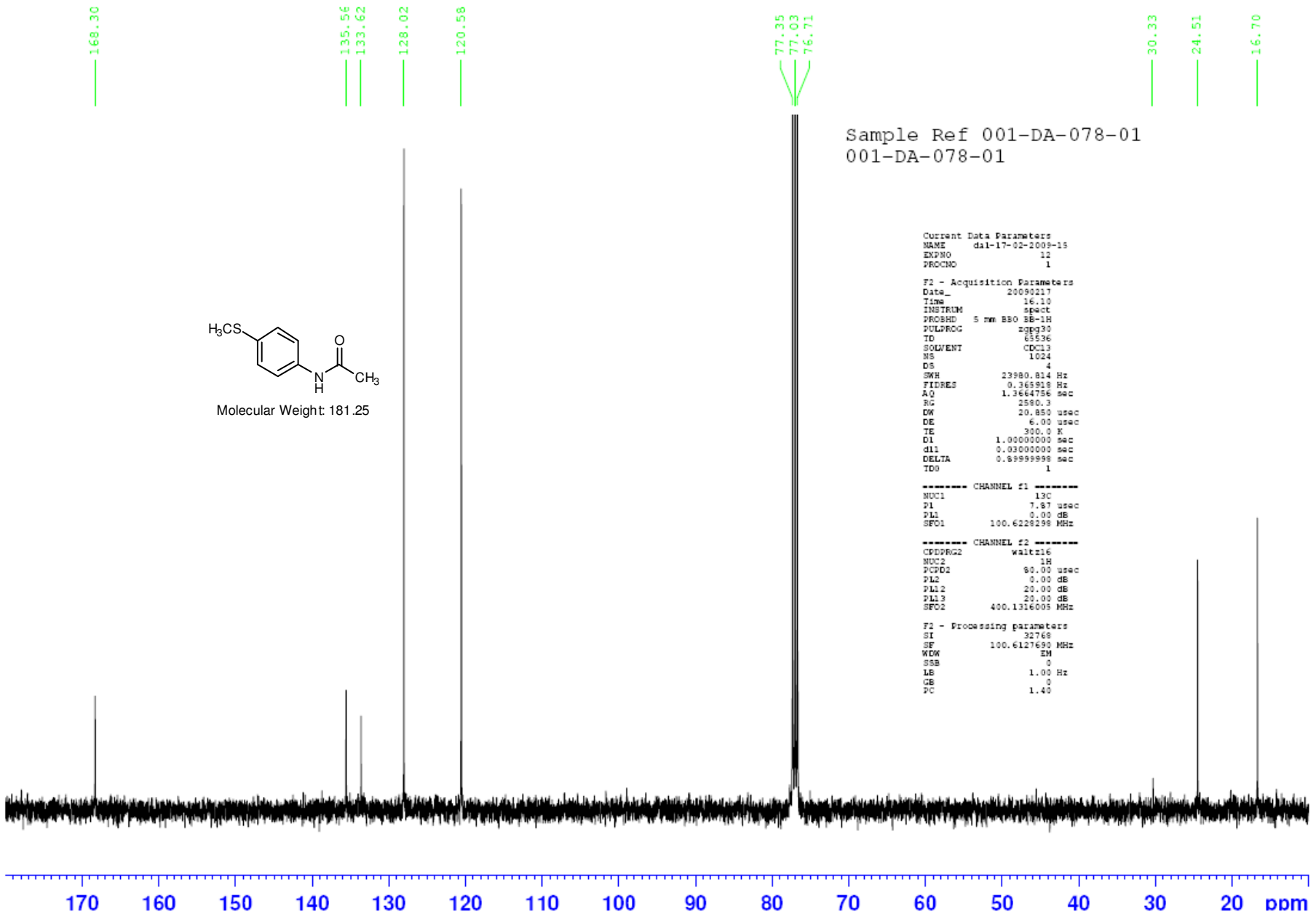


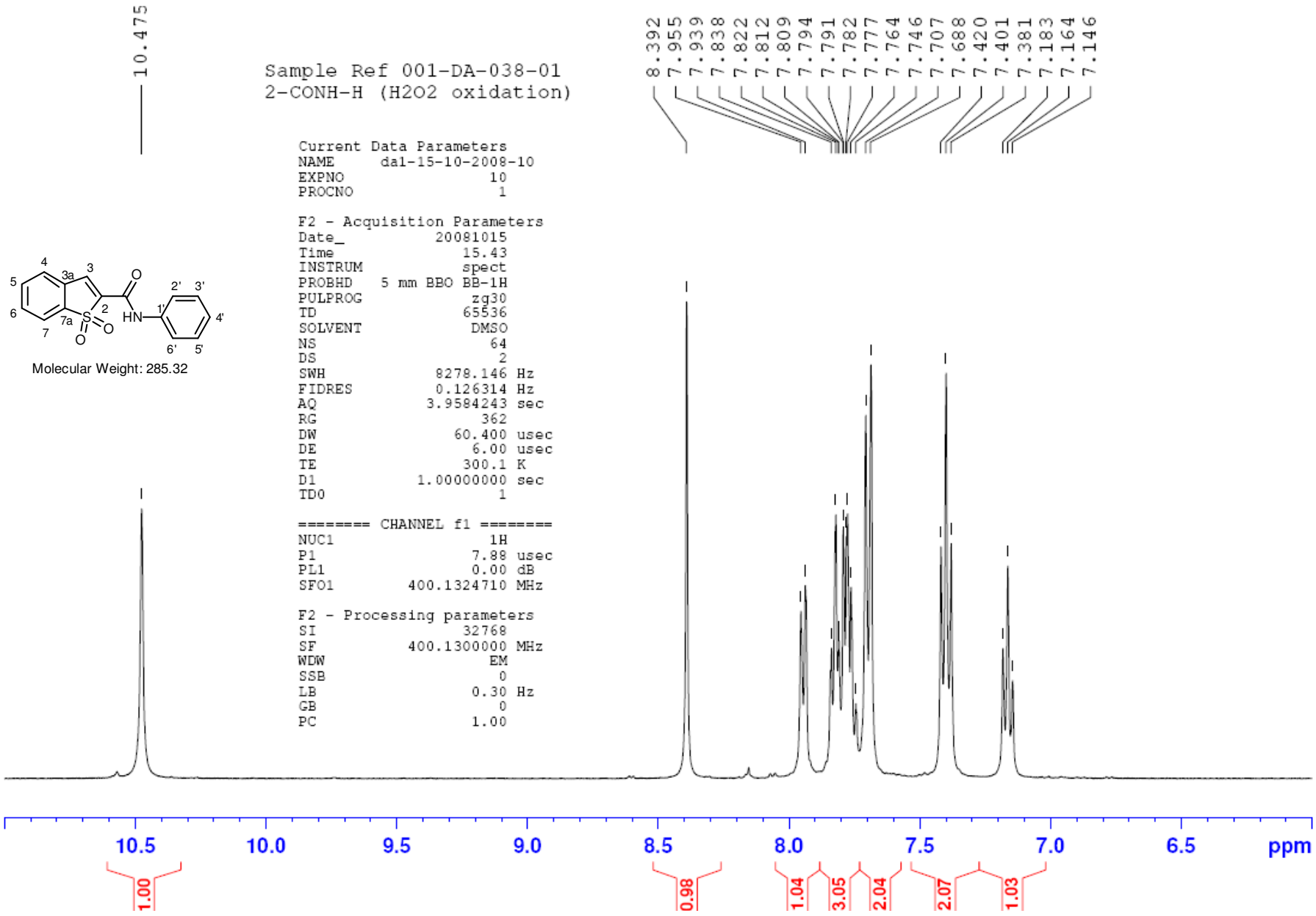

```
Current Data Parameters
NAME      da1-07-09-2007-36
EXPNO                   16
PROCNO                   1

F2 - Acquisition Parameters
Date_              20070910
Time                 20.15
INSTRUM              spect
PROBHD    5 mm BBO BB-1H
PULPROG            noesyph
TD                    2048
SOLVENT               CDCl3
NS                       4
DS                       4
SWH               3396.739 Hz
FIDRES            1.658564 Hz
AQ                0.3015156 sec
RG                   228.1
DW                 147.200 usec
DE                   6.00 usec
TE                  300.0 K
d0              0.00013717 sec
D1              1.08654106 sec
D8              0.80000001 sec
IN0             0.00029440 sec
ST1CNT                128

-------- CHANNEL f1 --------
NUC1                    1H
P1                   7.88 usec
PL1                  0.00 dB
SFO1          400.1319825 MHz

F1 - Acquisition parameters
ND0                      1
TD                     256
SFO1             400.132 MHz
FIDRES           13.268512 Hz
SW                   8.489 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                    1024
SF              400.1300000 MHz
WDW                  QSINE
SSB                      2
LB                   0.00 Hz
GB                       0
PC                   1.00

F1 - Processing parameters
SI                    1024
MC2            States-TPPI
SF              400.1300000 MHz
WDW                  QSINE
SSB                      2
LB                   0.00 Hz
GB                       0
```

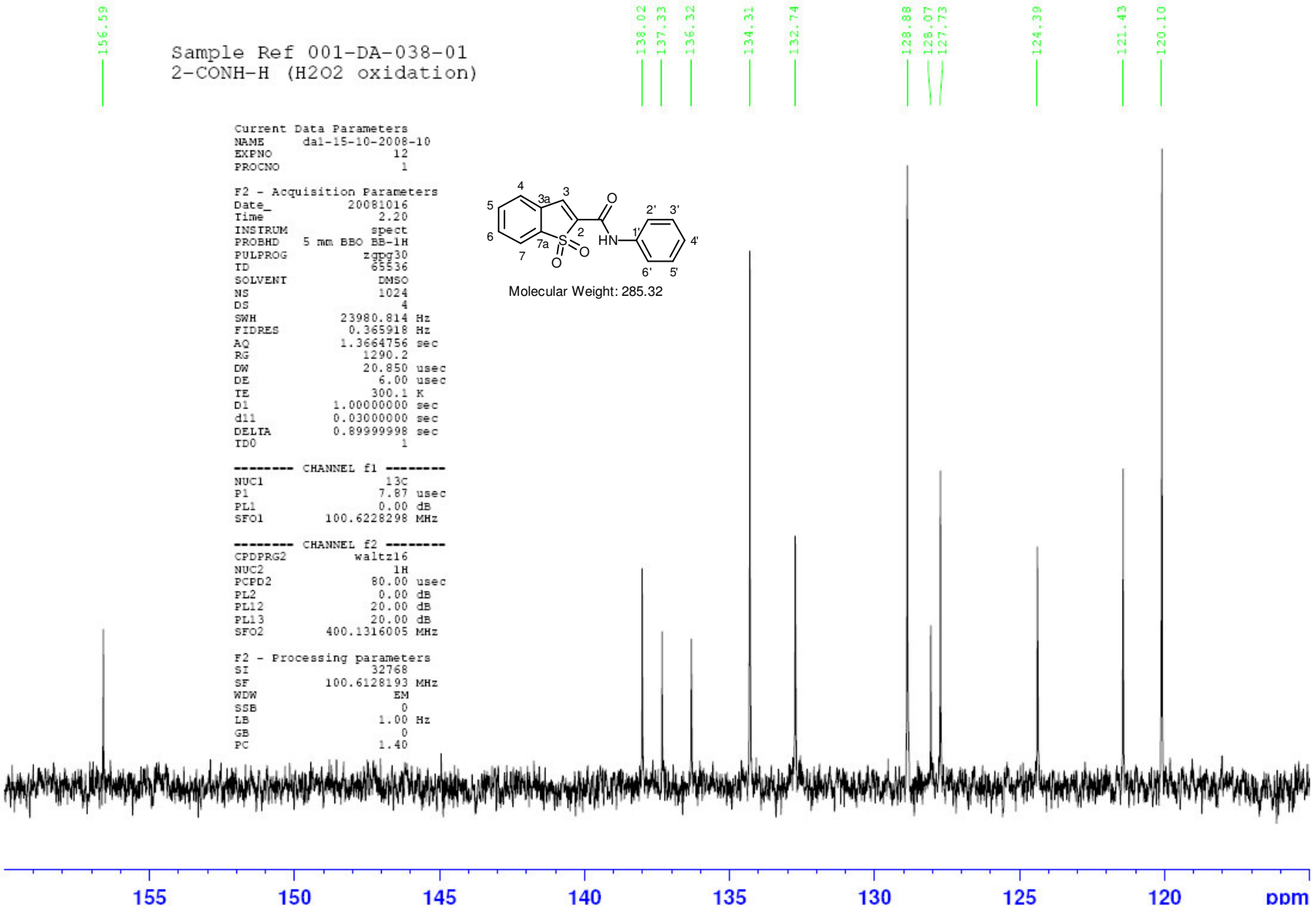

7.801
7.796
7.794
7.781
7.776
7.772
7.520
7.517
7.514
7.505
7.499
7.493
7.484
7.480
7.477
7.448
7.444
7.432
7.429
7.415
7.411
7.408
7.260
6.642
3.690
3.675
3.659
3.644
2.774
2.758
2.743
2.140
Sample Ref 001-DA-078-02
001-DA-078-02
Current Data Parameters
NAME da1-17-02-2009-16
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090217
Time 14.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3C
S
H
N
O
Molecular Weight: 195.28
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
2.00
1.02
2.01
0.99
2.06
2.07
3.04


167.4
134.4
131.5
128.5
126.9
77.35
77.03
76.71
37.97
33.90
14.95
Sample Ref 001-DA-078-02
001-DA-078-02
Current Data Parameters
NAME dal-17-02-2009-16
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090218
Time 0.43
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2298.8
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
H3C
S
H
N
O
Molecular Weight: 195.28
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

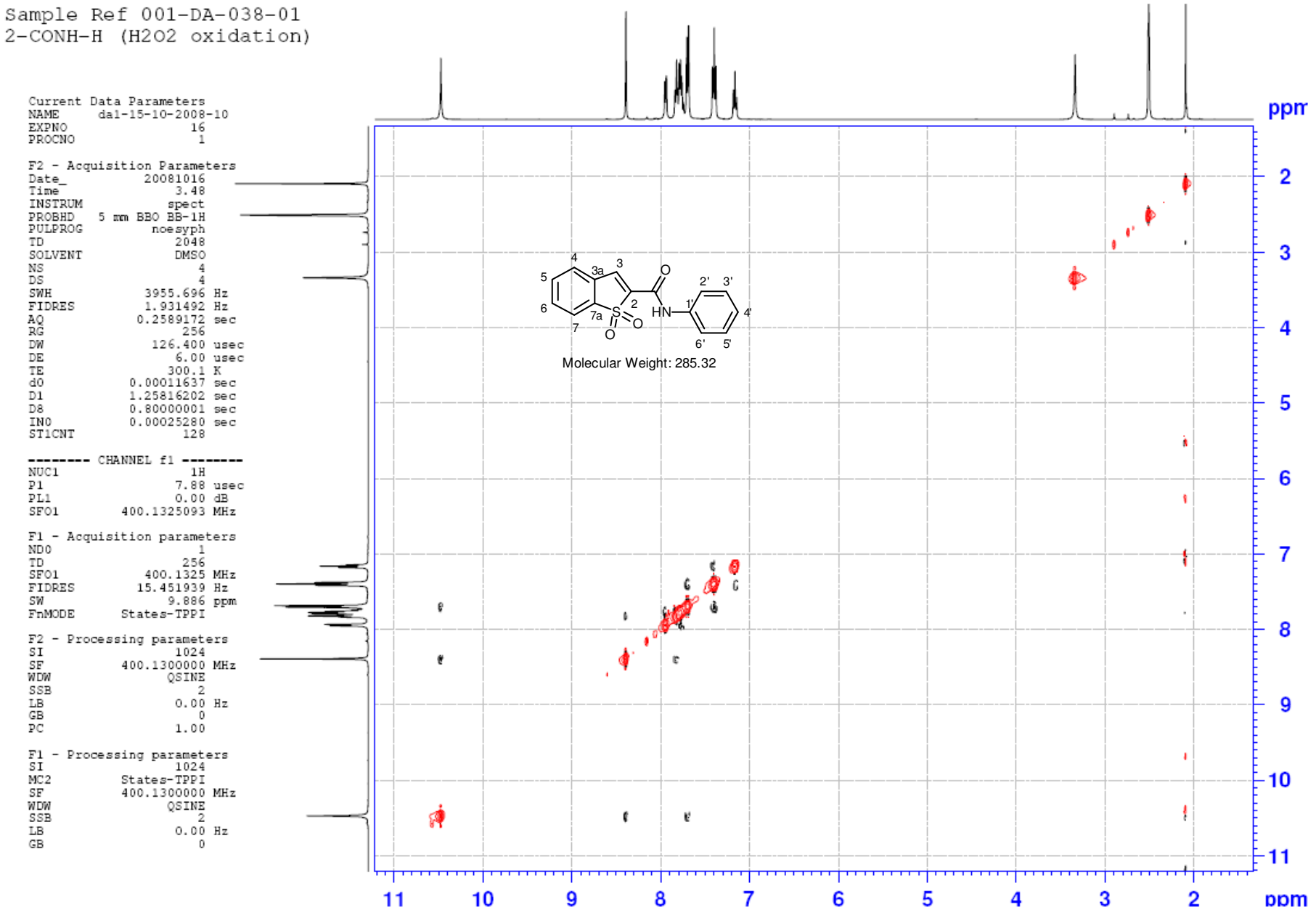

7.437
7.415
7.366
7.260
7.233
7.211
2.456
2.154
Sample Ref 001-DA-078-01
001-DA-078-01
Current Data Parameters
NAME dal-17-02-2009-15
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090217
Time 14.19
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3CS
O
N
H
CH3
Molecular Weight: 181.25
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
1.97
0.81
2.00
3.03
2.97

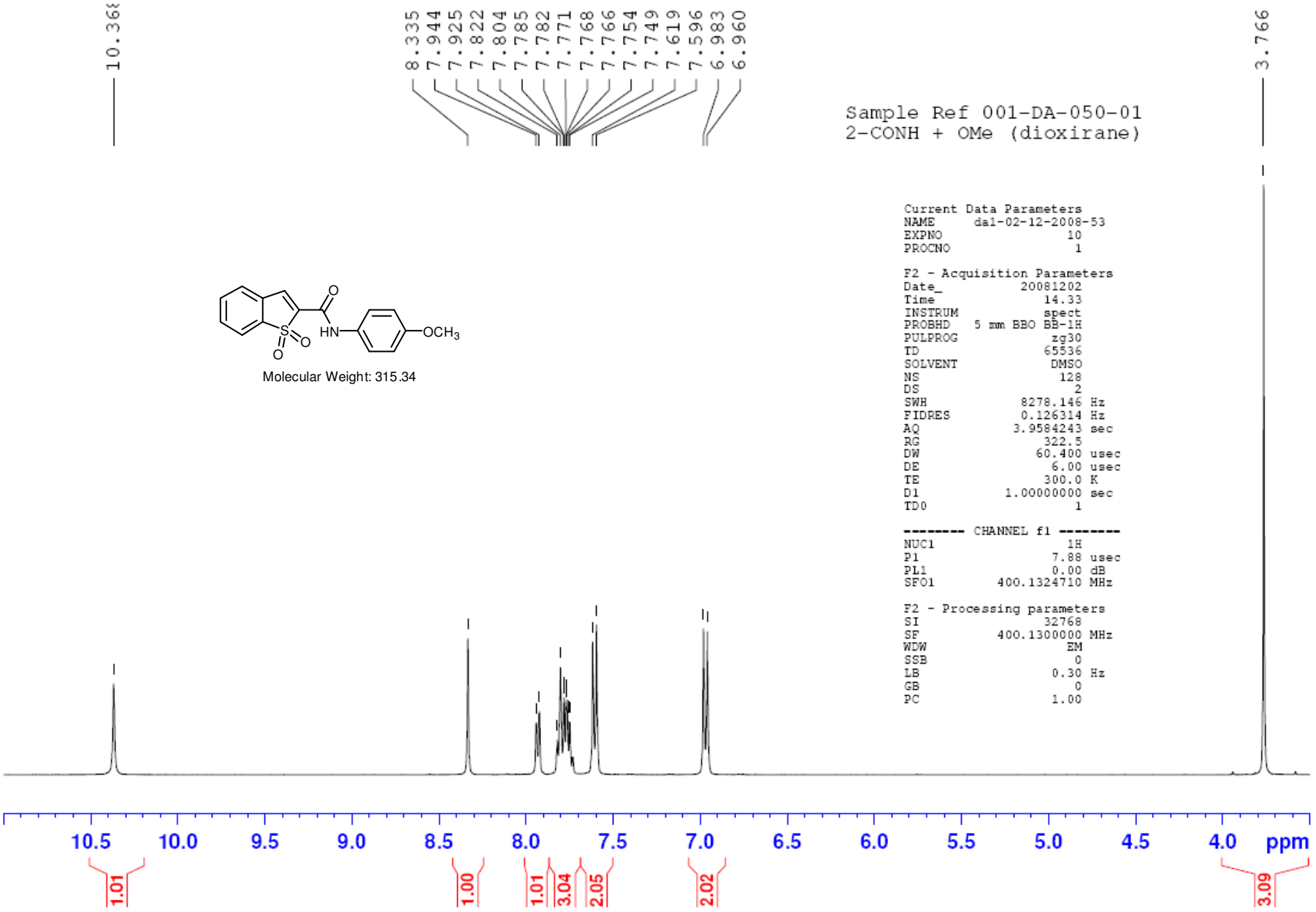

168.30
135.56
133.62
128.02
120.58
77.35
77.03
76.71
30.33
24.51
16.70
Sample Ref 001-DA-078-01
001-DA-078-01
H3CS
O
N
H
CH3
Molecular Weight: 181.25
Current Data Parameters
NAME da1-17-02-2009-15
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090217
Time 16.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

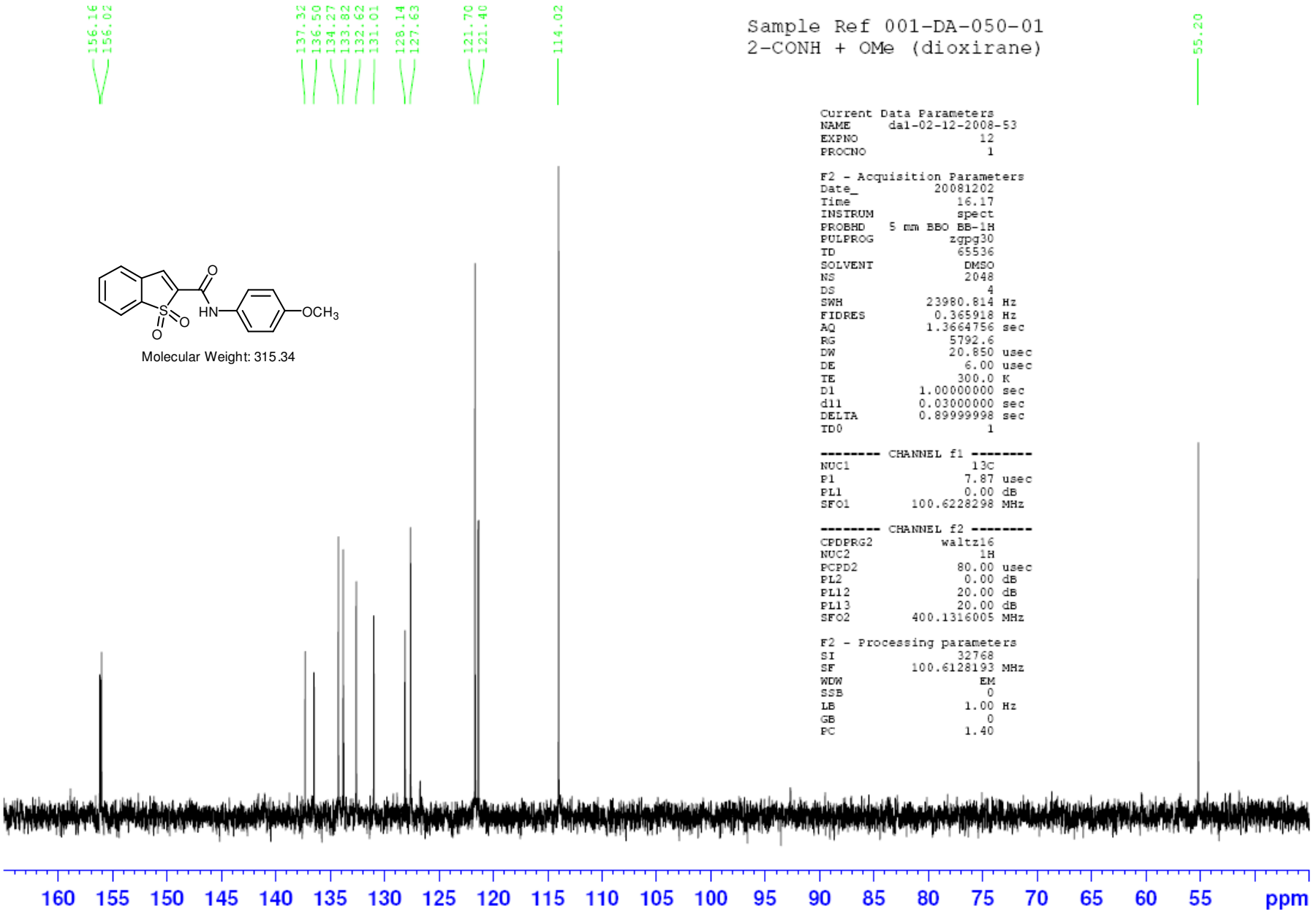

10.475
8.392
7.955
7.939
7.838
7.822
7.812
7.809
7.794
7.791
7.782
7.777
7.764
7.746
7.707
7.688
7.420
7.401
7.381
7.183
7.164
7.146
Sample Ref 001-DA-038-01
2-CONH-H (H2O2 oxidation)
Current Data Parameters
NAME da1-15-10-2008-10
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081015
Time 15.43
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Molecular Weight: 285.32
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
ppm
1.00
0.98
1.04
3.05
2.04
2.07
1.03

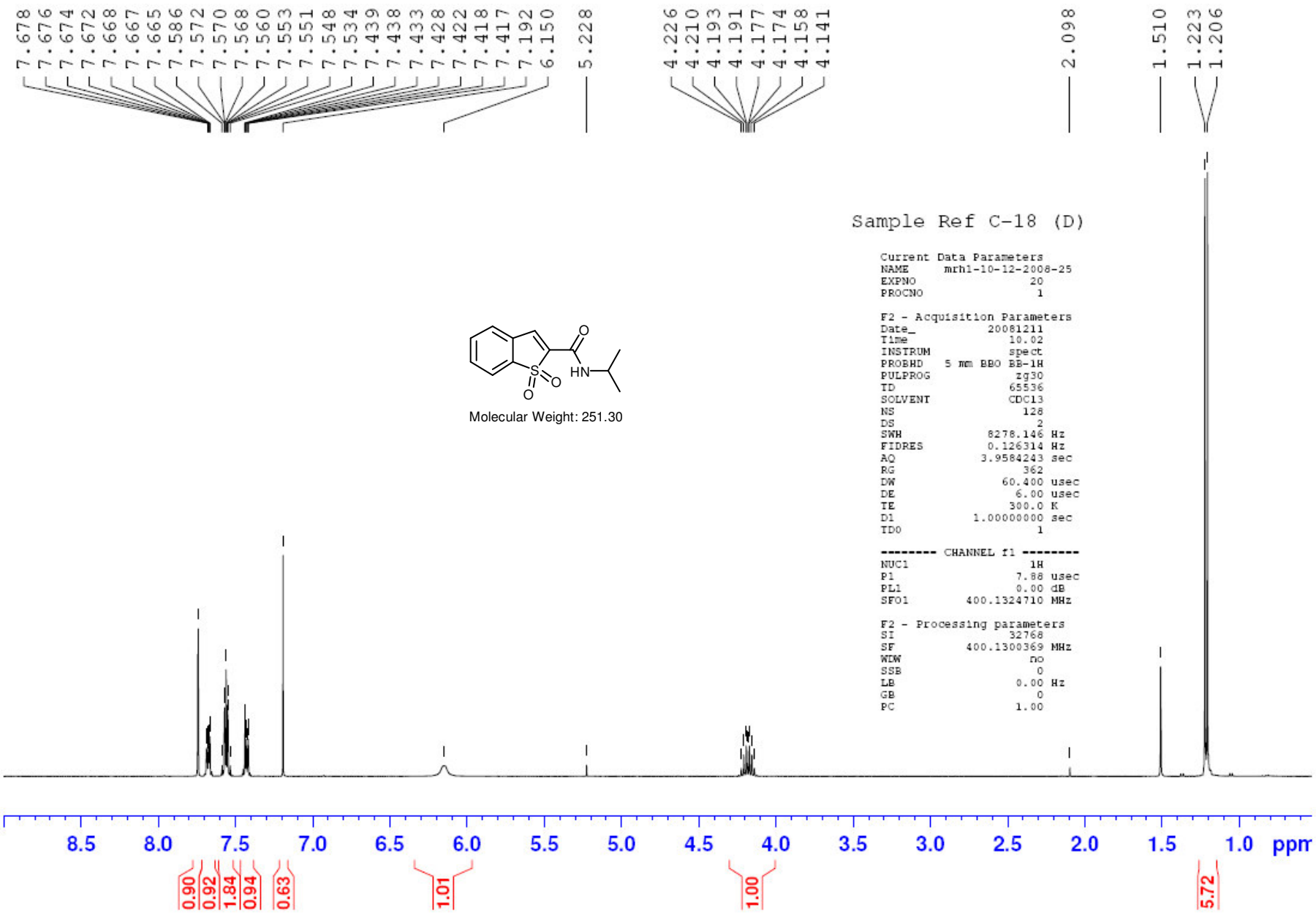

156.59
138.02
137.33
136.32
134.31
132.74
128.88
128.07
127.73
124.39
121.43
120.10
Sample Ref 001-DA-038-01
2-CONH-H (H2O2 oxidation)
Current Data Parameters
NAME da1-15-10-2008-10
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081016
Time 2.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Molecular Weight: 285.32
155
150
145
140
135
130
125
120
ppm

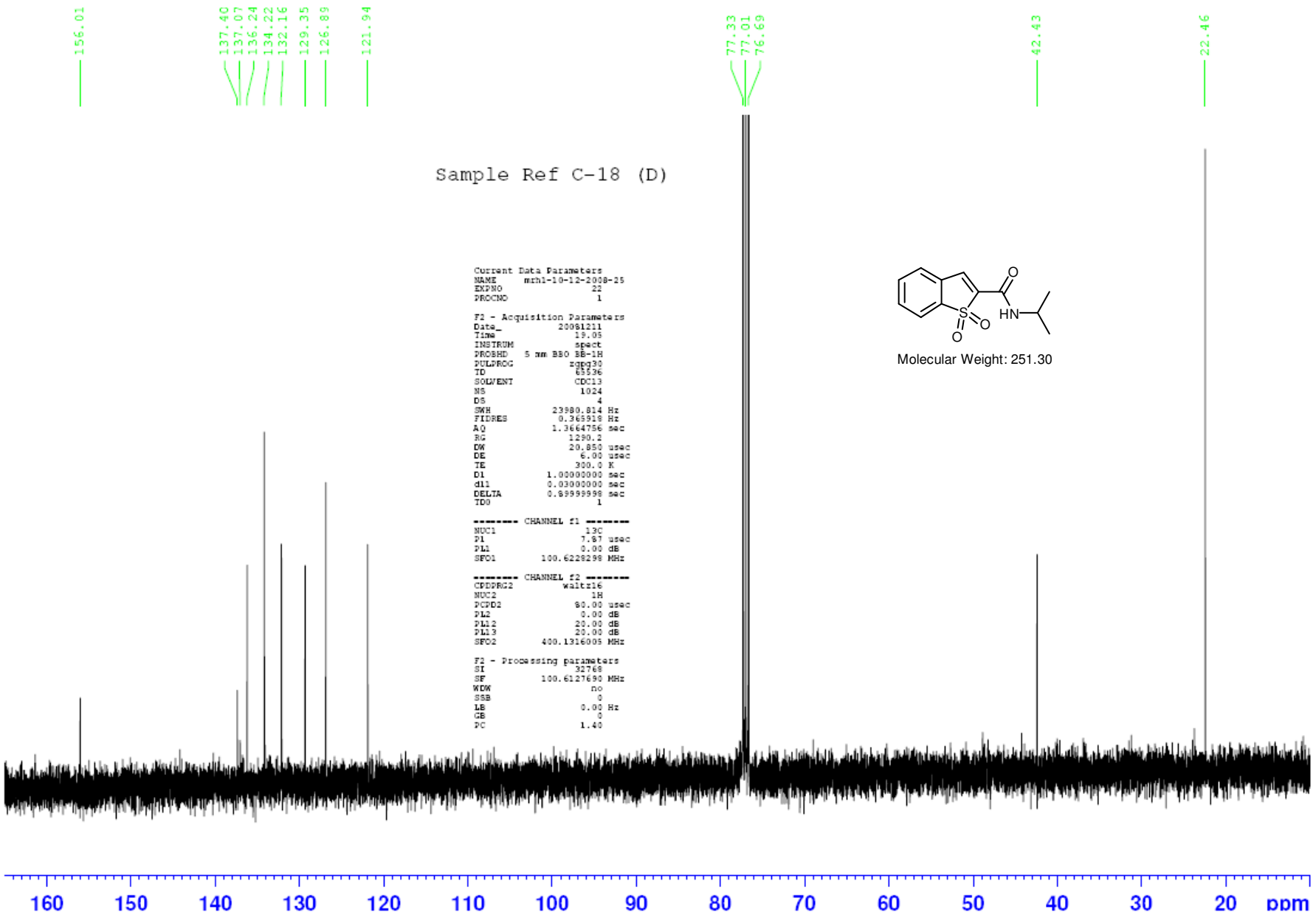
Sample Ref 001-DA-038-01
2-CONH-H (H2O2 oxidation)




10.366
8.335
7.944
7.925
7.822
7.804
7.785
7.782
7.771
7.768
7.766
7.754
7.749
7.619
7.596
6.983
6.960
3.766
Sample Ref 001-DA-050-01
2-CONH + OMe (dioxirane)
Molecular Weight: 315.34
Current Data Parameters
NAME da1-02-12-2008-53
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081202
Time 14.33
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
1.01
1.00
1.01
3.04
2.05
2.02
3.09


156.16
156.02
137.32
136.50
134.27
133.82
132.62
131.01
128.14
127.63
121.70
121.40
114.02
55.20
Sample Ref 001-DA-050-01
2-CONH + OMe (dioxirane)
Molecular Weight: 315.34
Current Data Parameters
NAME dal-02-12-2008-53
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081202
Time 16.17
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 ppm


7.678
7.676
7.674
7.672
7.668
7.667
7.665
7.586
7.572
7.570
7.568
7.560
7.553
7.551
7.548
7.534
7.439
7.438
7.433
7.428
7.422
7.418
7.417
7.192
6.150
5.228
4.226
4.210
4.193
4.191
4.177
4.174
4.158
4.141
2.098
1.510
1.223
1.206
Sample Ref C-18 (D)
Current Data Parameters
NAME mrh1-10-12-2008-25
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081211
Time 10.02
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300369 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
HN
S
O
O
Molecular Weight: 251.30
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.90
0.92
1.84
0.94
0.63
1.01
1.00
5.72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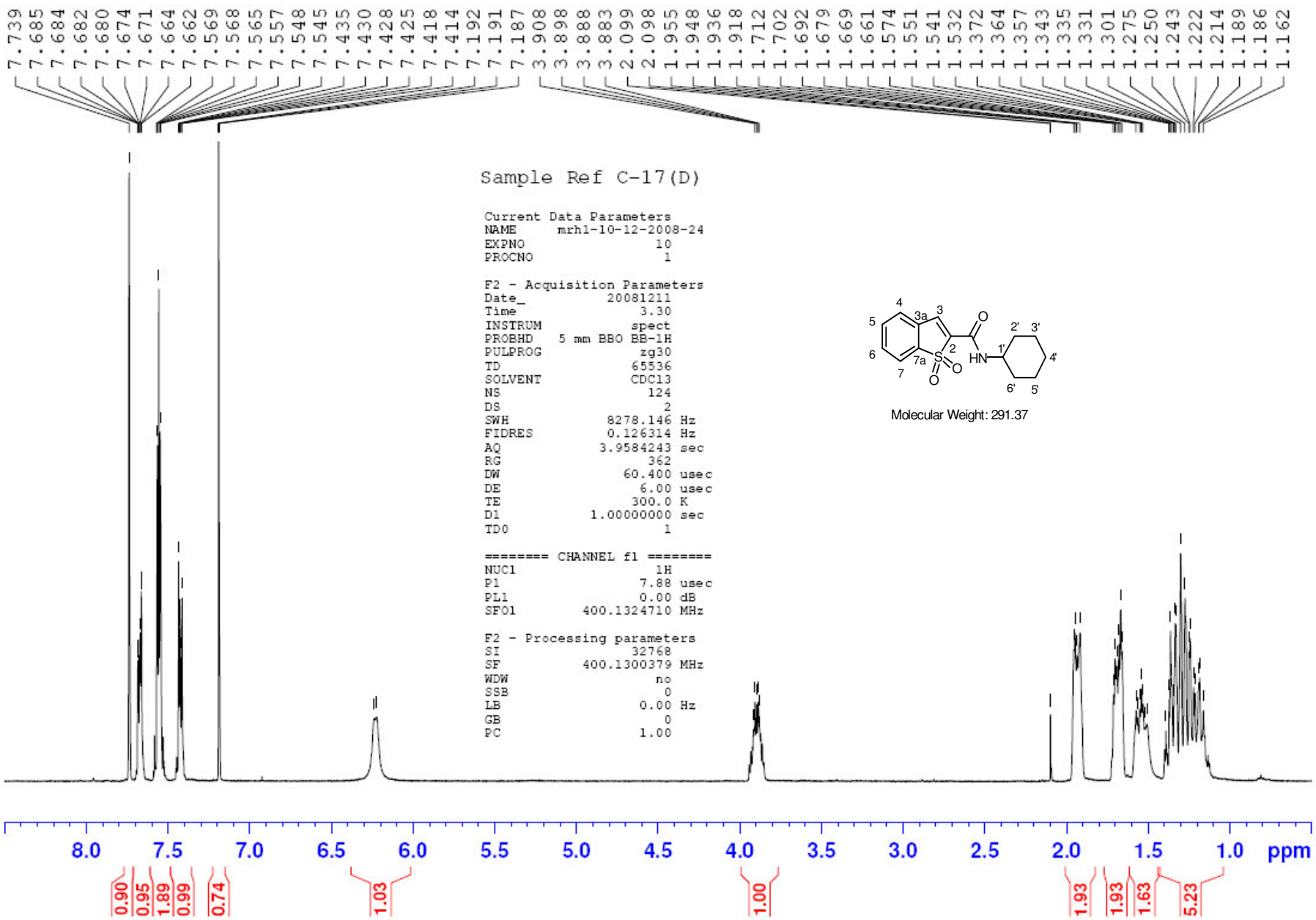
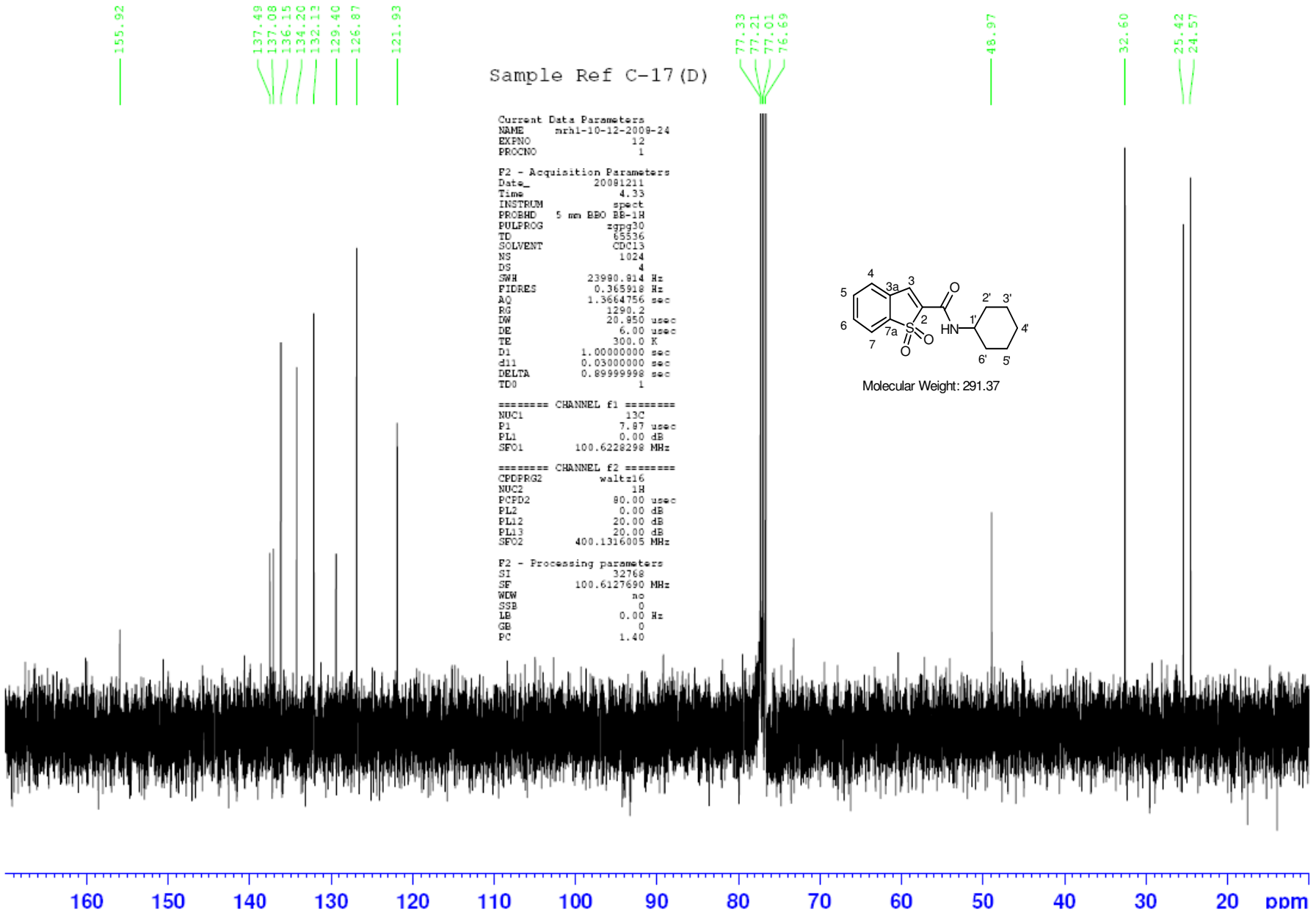
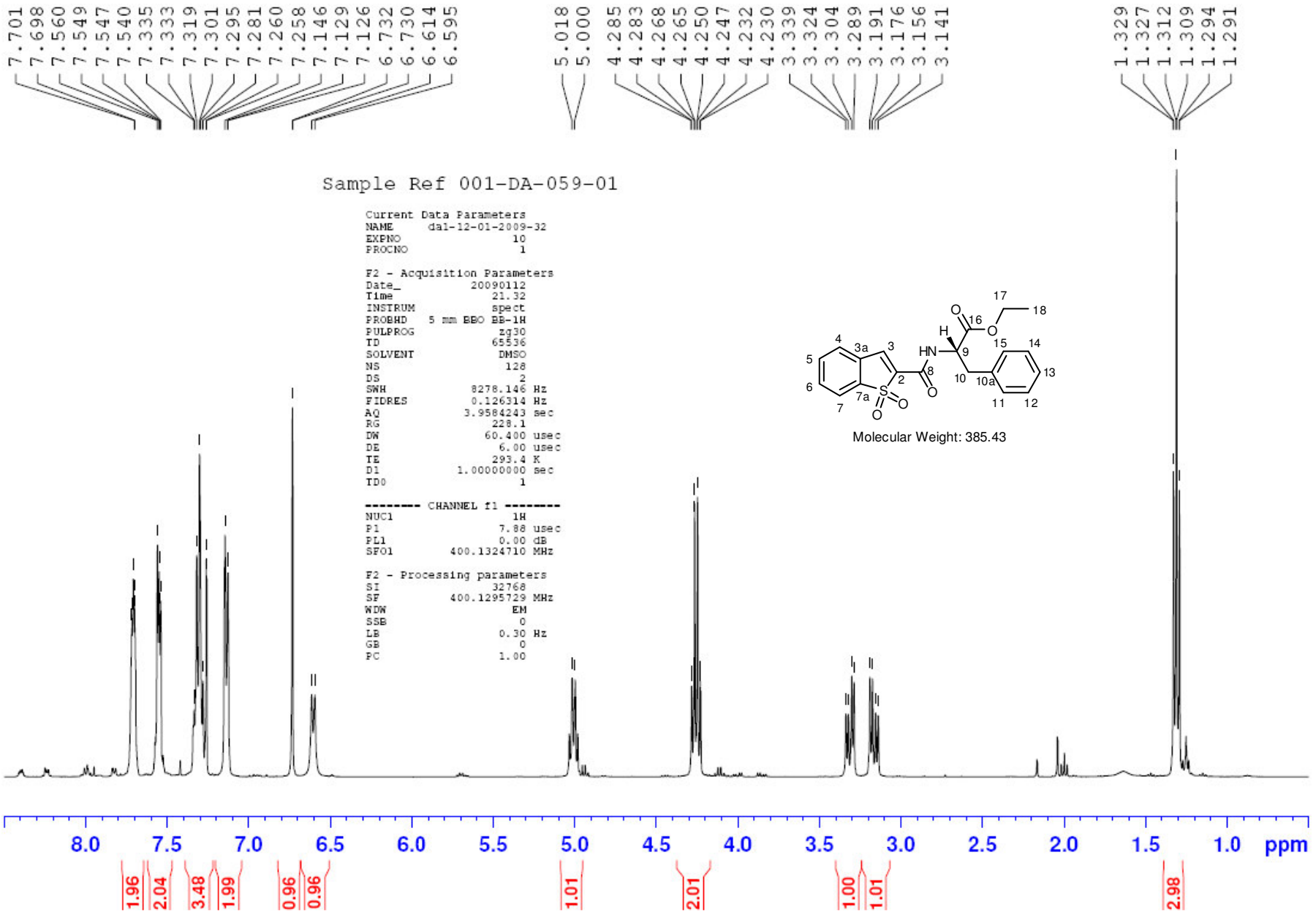
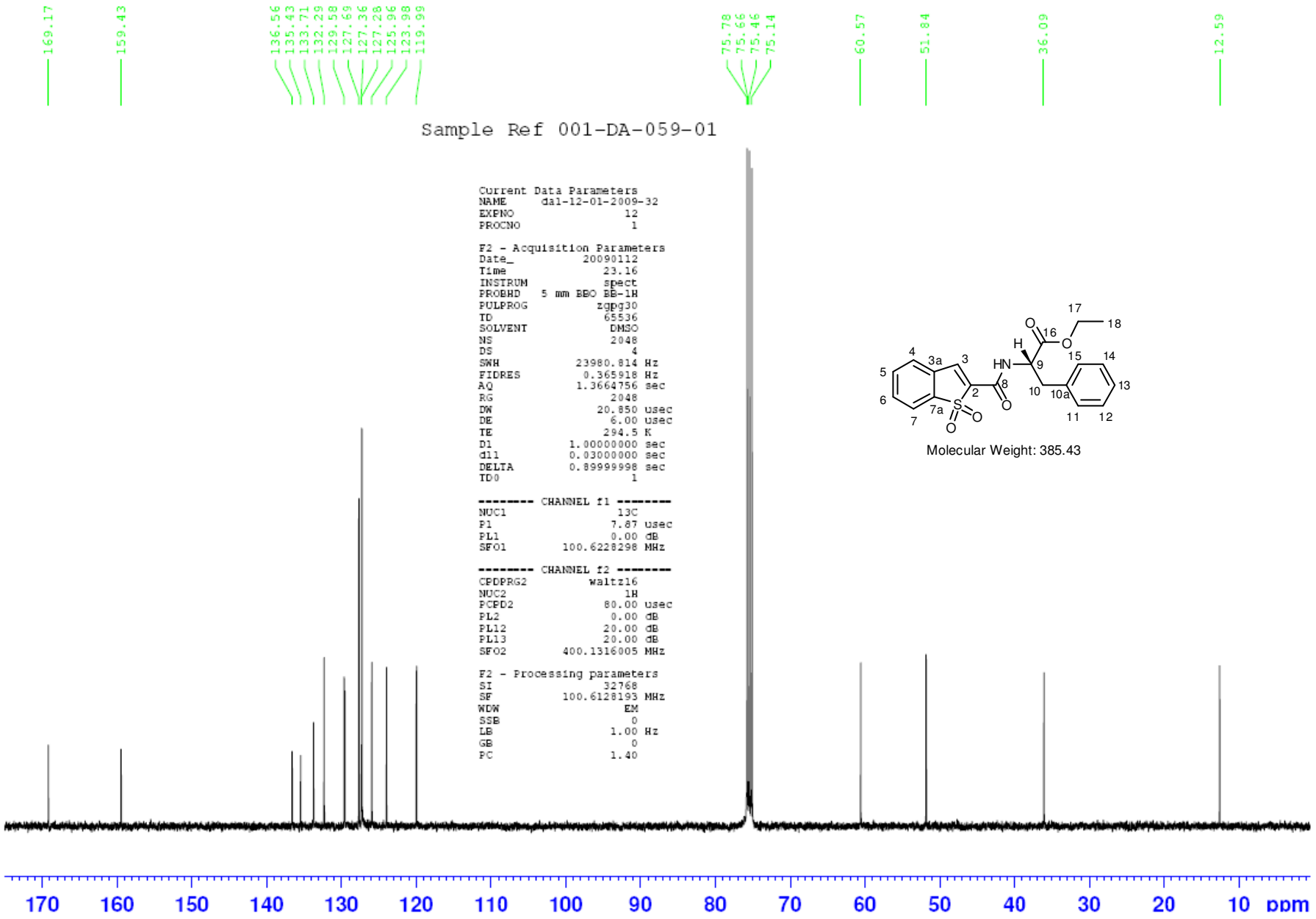
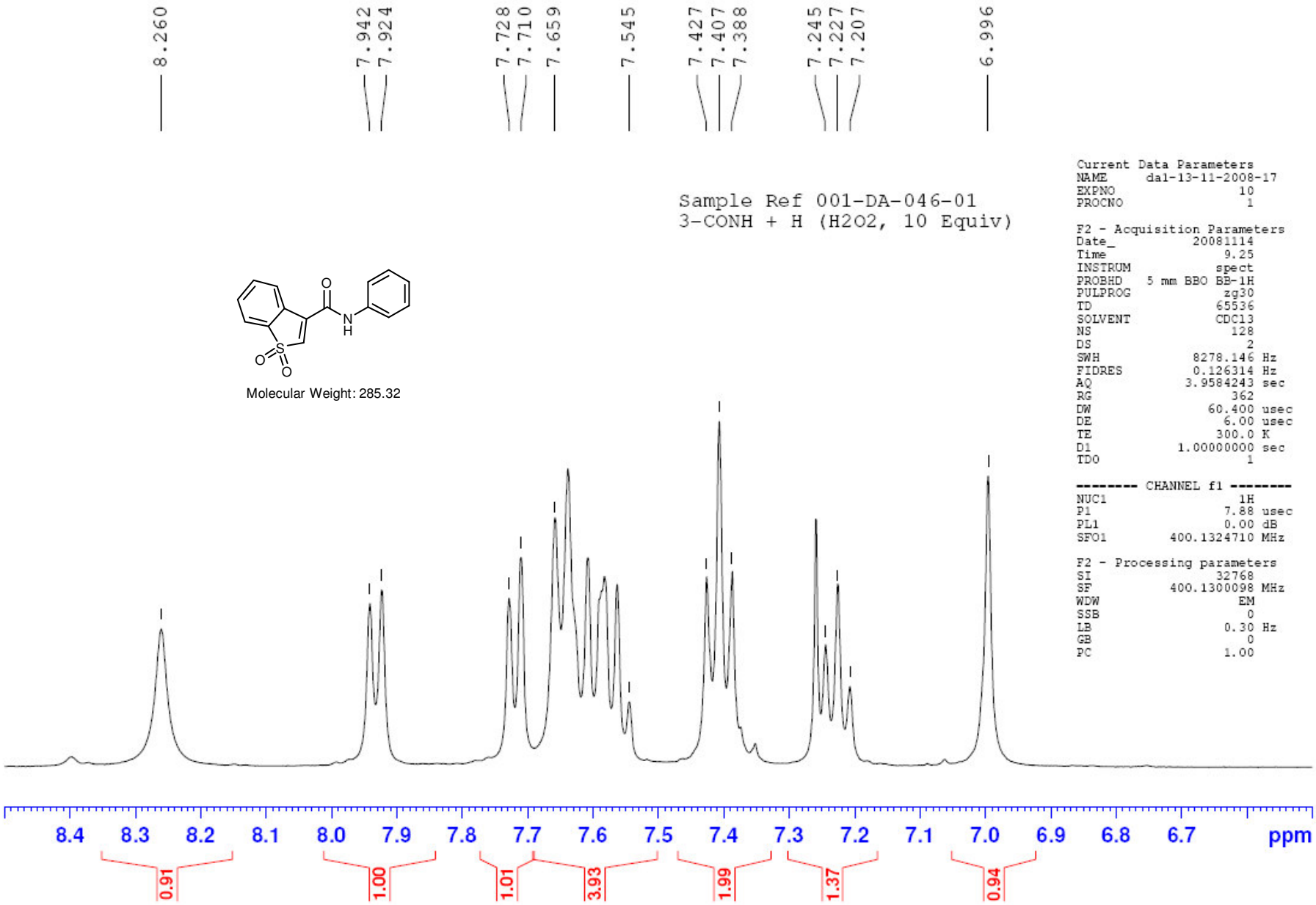
137.40
137.07
136.24
134.22
132.16
129.35
126.89
121.94
77.33
77.01
76.69
42.43
22.46
Sample Ref C-18 (D)
Current Data Parameters
NAME mrh1-10-12-2008-25
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081211
Time 19.05
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365919 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
O
HN
S
O
O
Molecular Weight: 251.30
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

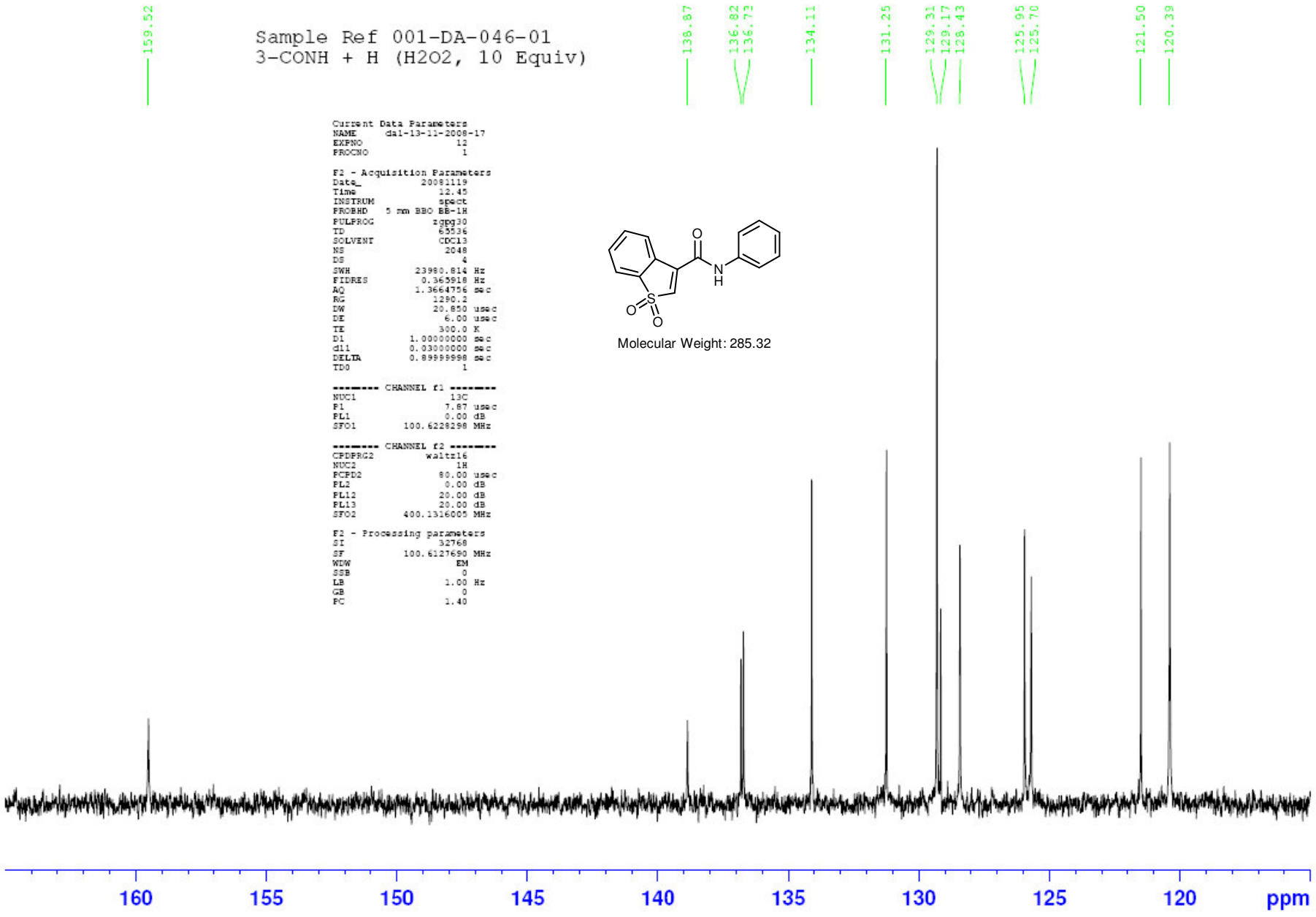

7.739
7.685
7.684
7.682
7.680
7.674
7.671
7.664
7.662
7.569
7.568
7.565
7.557
7.548
7.545
7.435
7.430
7.428
7.425
7.418
7.414
7.192
7.191
7.187
3.908
3.898
3.888
3.883
2.099
2.098
1.955
1.948
1.936
1.918
1.712
1.702
1.692
1.679
1.669
1.661
1.574
1.551
1.541
1.532
1.372
1.364
1.357
1.343
1.335
1.331
1.301
1.275
1.250
1.243
1.222
1.214
1.189
1.186
1.162
Sample Ref C-17(D)
Current Data Parameters
NAME mrh1-10-12-2008-24
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081211
Time 3.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 124
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300379 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Molecular Weight: 291.37
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.90
0.95
1.89
0.99
0.74
1.03
1.00
1.93
1.93
1.63
5.23

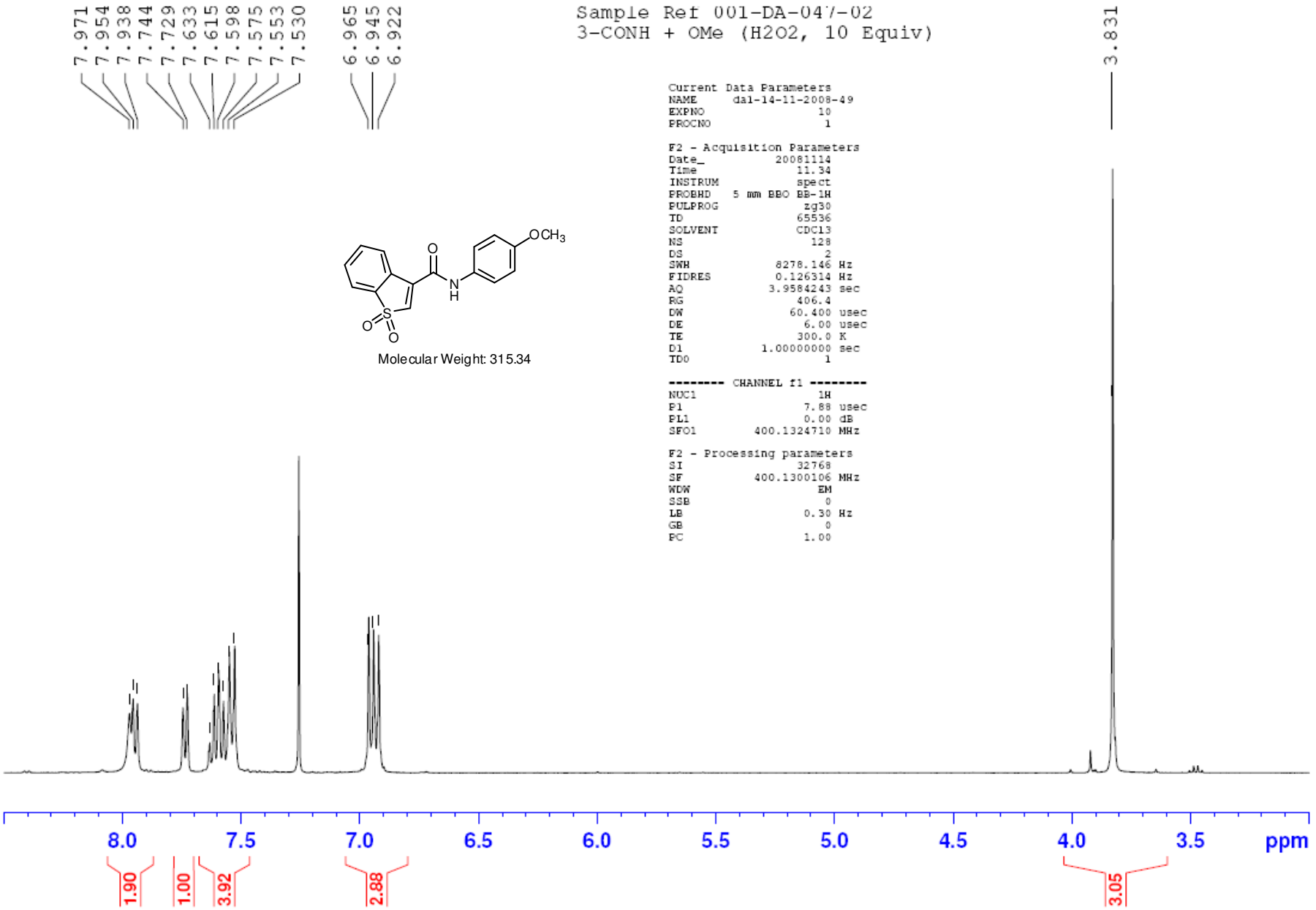

155.92
137.49
137.08
136.15
134.20
132.13
129.40
126.87
121.93
77.33
77.21
77.01
76.69
48.97
32.60
25.42
24.57
Sample Ref C-17(D)
Current Data Parameters
NAME mrh1-10-12-2008-24
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081211
Time 4.33
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
Molecular Weight: 291.37
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

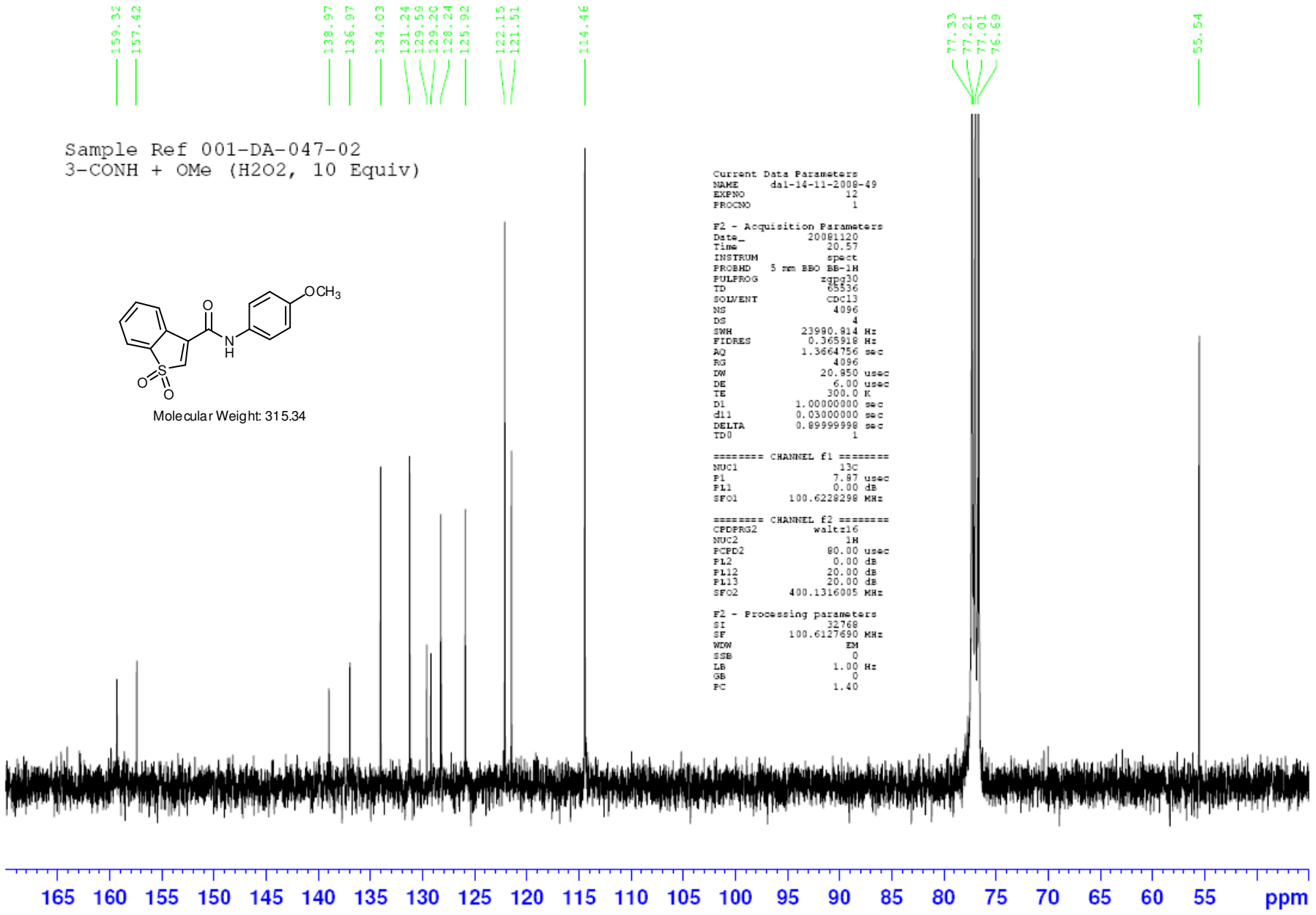

7.701
7.698
7.560
7.549
7.547
7.540
7.335
7.333
7.319
7.301
7.295
7.281
7.260
7.258
7.146
7.129
7.126
6.732
6.730
6.614
6.595
5.018
5.000
4.285
4.283
4.268
4.265
4.250
4.247
4.232
4.230
3.339
3.324
3.304
3.289
3.191
3.176
3.156
3.141
1.329
1.327
1.312
1.309
1.294
1.291
Sample Ref 001-DA-059-01
Current Data Parameters
NAME dal-12-01-2009-32
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090112
Time 21.32
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1295729 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Molecular Weight: 385.43
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.96
2.04
3.48
1.99
0.96
0.96
1.01
2.01
1.00
1.01
2.98


169.17
159.43
136.56
135.43
133.71
132.29
129.58
127.69
127.36
127.28
125.96
123.98
119.99
75.78
75.66
75.46
75.14
60.57
51.84
36.09
12.59
Sample Ref 001-DA-059-01
Current Data Parameters
NAME da1-12-01-2009-32
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090112
Time 23.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 294.5 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Molecular Weight: 385.43
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

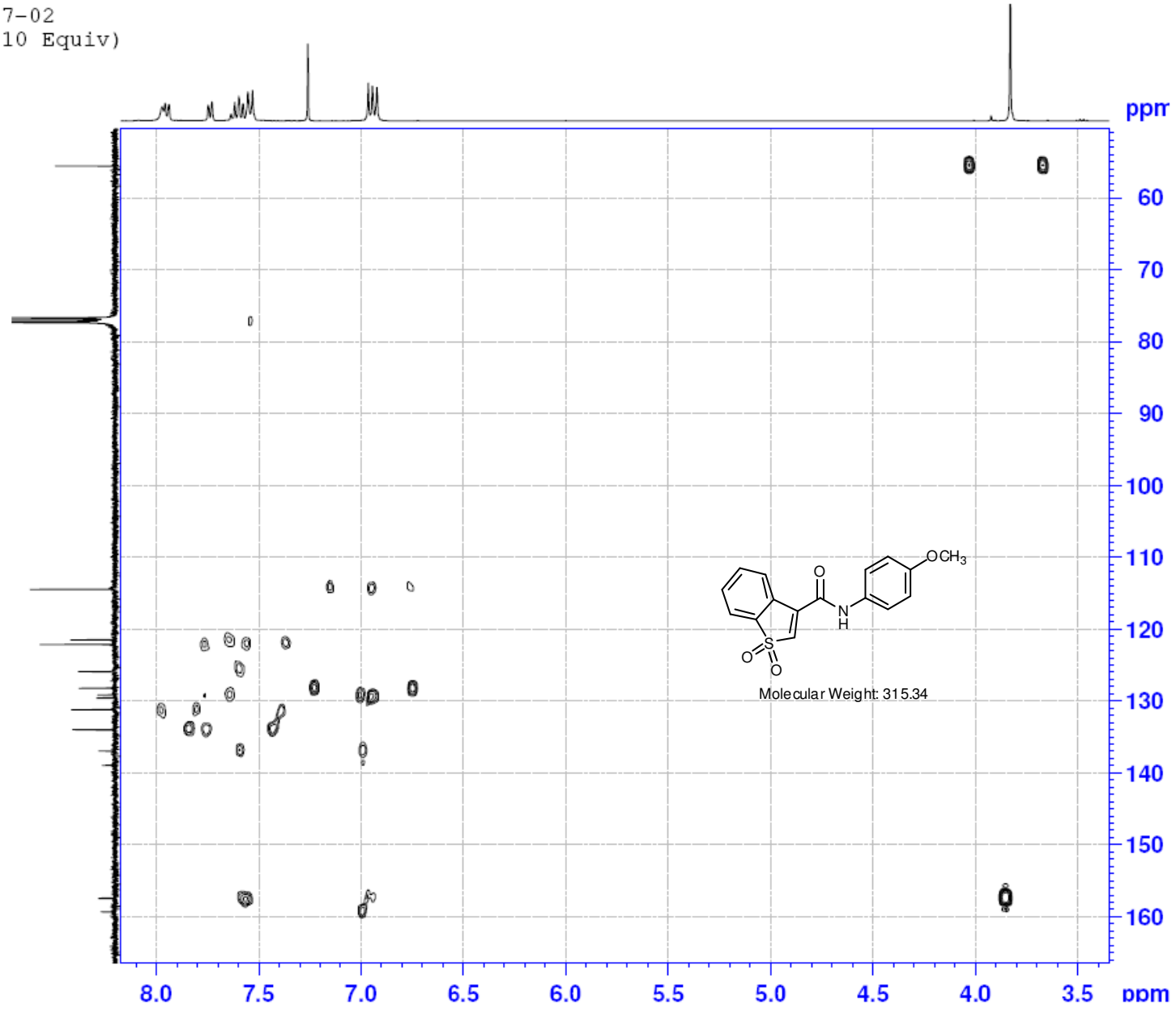

8.260
7.942
7.924
7.728
7.710
7.659
7.545
7.427
7.407
7.388
7.245
7.227
7.207
6.996
Sample Ref 001-DA-046-01
3-CONH + H (H2O2, 10 Equiv)
Molecular Weight: 285.32
Current Data Parameters
NAME dal-13-11-2008-17
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081114
Time 9.25
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 ppm
0.91
1.00
1.01
3.93
1.99
1.37
0.94


159.52
138.87
136.82
136.73
134.11
131.25
129.31
129.17
128.43
125.95
125.70
121.50
120.39
Sample Ref 001-DA-046-01
3-CONH + H (H2O2, 10 Equiv)
Current Data Parameters
NAME dal-13-11-2008-17
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081119
Time 12.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
O
N
H
O
S
O
Molecular Weight: 285.32
160
155
150
145
140
135
130
125
120
ppm

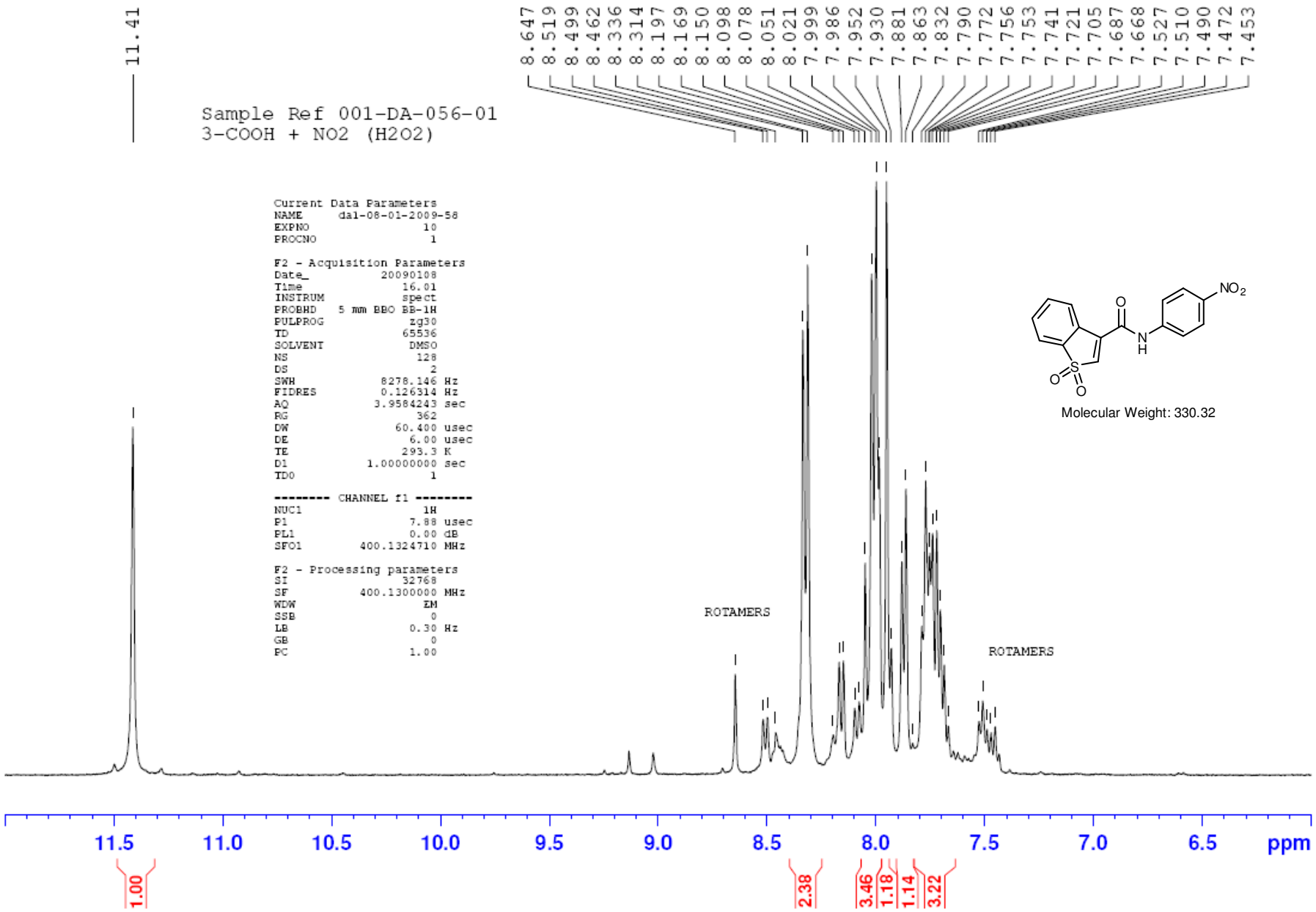

7.971
7.954
7.938
7.744
7.729
7.633
7.615
7.598
7.575
7.553
7.530
6.965
6.945
6.922
3.831
Sample Ref 001-DA-047-02
3-CONH + OMe (H2O2, 10 Equiv)
Current Data Parameters
NAME dal-14-11-2008-49
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081114
Time 11.34
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 406.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OCH3
O
N
H
O
S
O
Molecular Weight: 315.34
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
ppm
1.90
1.00
3.92
2.88
3.05

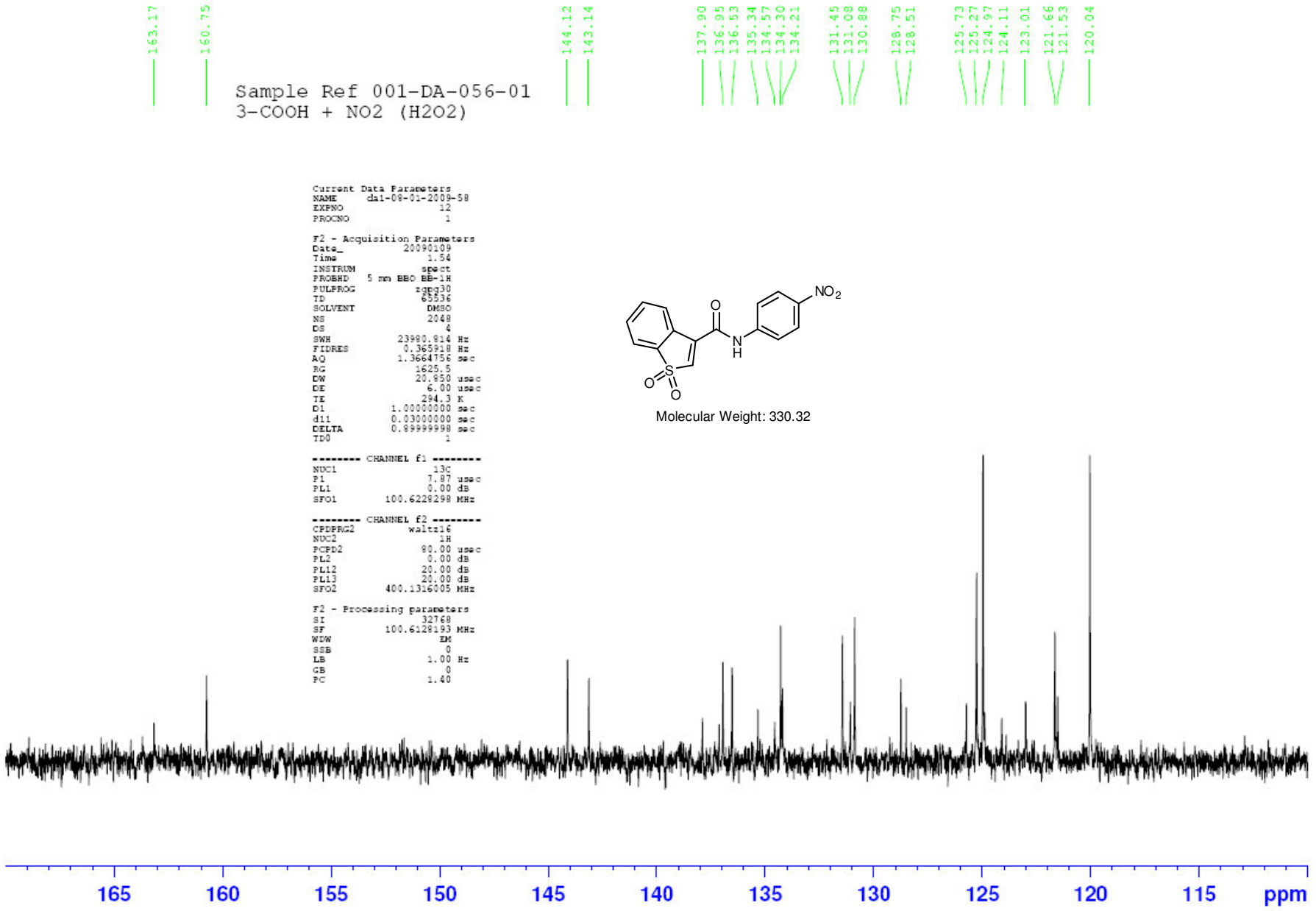

159.32
157.42
138.97
136.97
134.03
131.24
129.59
129.20
128.24
125.92
122.15
121.51
114.46
77.33
77.21
77.01
76.69
55.54
Sample Ref 001-DA-047-02
3-CONH + OMe (H2O2, 10 Equiv)
OCH3
O
N
H
S
O
O
Molecular Weight: 315.34
Current Data Parameters
NAME dai-14-11-2008-49
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081120
Time 20.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 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
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 ppm

Sample Ref 001-DA-047-02
3-CONH + OMe (H2O2, 10 Equiv)

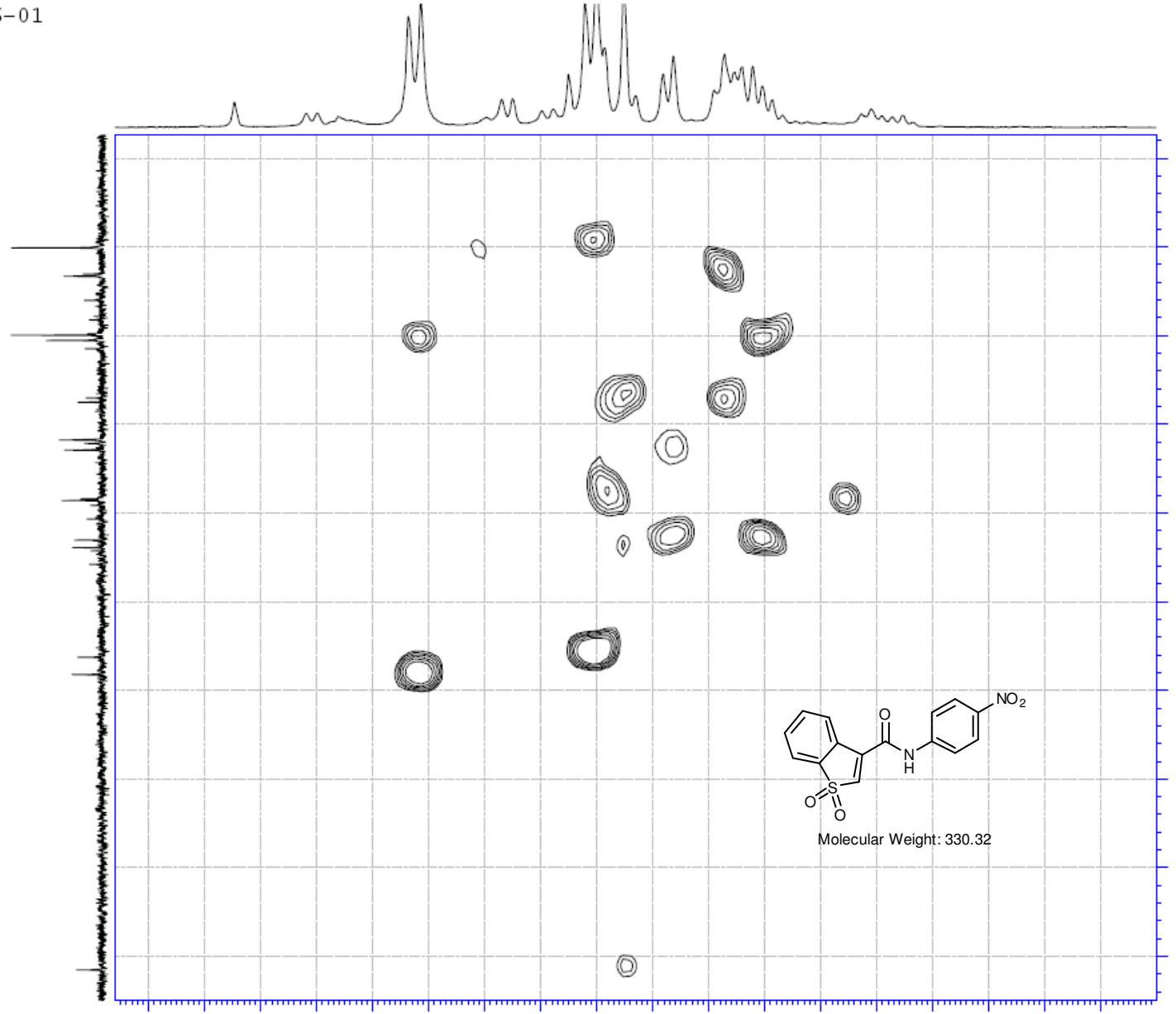



```
Current Data Parameters
NAME      da1-14-11-2008-49
EXPNO                   15
PROCNO                   1

F2 - Acquisition Parameters
Date_           20081120
Time              22.32
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG    inv4gplrndqf
TD                 1024
SOLVENT            CDCl3
NS                   16
DS                   16
SWH            3238.342 Hz
FIDRES         3.162443 Hz
AQ            0.1581556 sec
RG                16384
DW              154.400 usec
DE                6.00 usec
TE                300.0 K
d0            0.00000300 sec
D1            1.43815005 sec
D6            0.07100000 sec
d13           0.00000400 sec
D16           0.00010000 sec
IN0           0.00002240 sec

-------- CHANNEL f1 --------
NUC1                 1H
P1                 7.88 usec
p2                15.76 usec
PL1                0.00 dB
SFO1        400.1319948 MHz

-------- CHANNEL f2 --------
NUC2                13C
P3                 7.65 usec
PL2                0.00 dB
SFO2        100.6227746 MHz

------ GRADIENT CHANNEL -----
GPNAM1          SINE.100
GPNAM2          SINE.100
GPNAM3          SINE.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
P16              1000.00 usec

F1 - Acquisition parameters
ND0                  2
TD                 128
SFO1          100.6228 MHz
FIDRES        174.386154 Hz
SW              221.933 ppm
FnMODE               QF

F2 - Processing parameters
SI                1024
SF          400.1300000 MHz
WDW               SINE
SSB                  0
LB                0.00 Hz
GB                   0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                  QF
SF          100.6127690 MHz
WDW               SINE
SSB                  0
LB                0.00 Hz
GB                   0
```


11.41
8.647
8.519
8.499
8.462
8.336
8.314
8.197
8.169
8.150
8.098
8.078
8.051
8.021
7.999
7.986
7.952
7.930
7.881
7.863
7.832
7.790
7.772
7.756
7.753
7.741
7.721
7.705
7.687
7.668
7.527
7.510
7.490
7.472
7.453
Sample Ref 001-DA-056-01
3-COOH + NO2 (H2O2)
Current Data Parameters
NAME dal-08-01-2009-58
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090108
Time 16.01
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ROTAMERS
ROTAMERS
O
N
H
S
O
O
NO2
Molecular Weight: 330.32
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
ppm
1.00
2.38
3.46
1.18
1.14
3.22


163.17
160.75
144.12
143.14
137.90
136.95
136.53
135.34
134.57
134.30
134.21
131.45
131.08
130.88
128.75
128.51
125.73
125.27
124.97
124.11
123.01
121.66
121.53
120.04
Sample Ref 001-DA-056-01
3-COOH + NO2 (H2O2)
Current Data Parameters
NAME dai-09-01-2009-58
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090109
Time 1.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 294.3 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
H
S
O
O
NO2
Molecular Weight: 330.32
165
160
155
150
145
140
135
130
125
120
115
ppm

Sample Ref 001-DA-056-01
3-COOH + NO2 (H2O2)



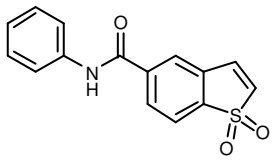
Molecular Weight: 330.32

```
Current Data Parameters
NAME      da1-08-01-2009-58
EXPNO                   15
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090109
Time                   3.06
INSTRUM                spect
PROBHD   5 mm BBO BB-1H
PULPROG     inv4gplrndgf
TD                     1024
SOLVENT                DMSO
NS                        8
DS                       16
SWH                5668.934 Hz
FIDRES             5.536068 Hz
AQ                 0.0903668 sec
RG                     16384
DW                    88.200 usec
DE                     6.00 usec
TE                    293.7 K
d0                 0.00000300 sec
D1                 1.50573397 sec
D6                 0.07100000 sec
d13                0.00000400 sec
D16                0.00010000 sec
IN0                0.00002240 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                      7.98 usec
p2                     15.76 usec
PL1                     0.00 dB
SFO1            400.1333063 MHz

======== CHANNEL f2 ========
NUC2                     13C
P3                      7.65 usec
PL2                     0.00 dB
SFO2            100.6227746 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPNAM3              SINE.100
GPZ1                   50.00 %
GPZ2                   30.00 %
GPZ3                   40.10 %
P16                  1000.00 usec

F1 - Acquisition parameters
ND0                       2
TD                      128
SFO1               100.6228 MHz
FIDRES          174.386154 Hz
SW                 221.933 ppm
FnMODE                   QF

F2 - Processing parameters
SI                      1024
SF              400.1300000 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF              100.6127690 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
```

Sample Ref 001-DA-043-01

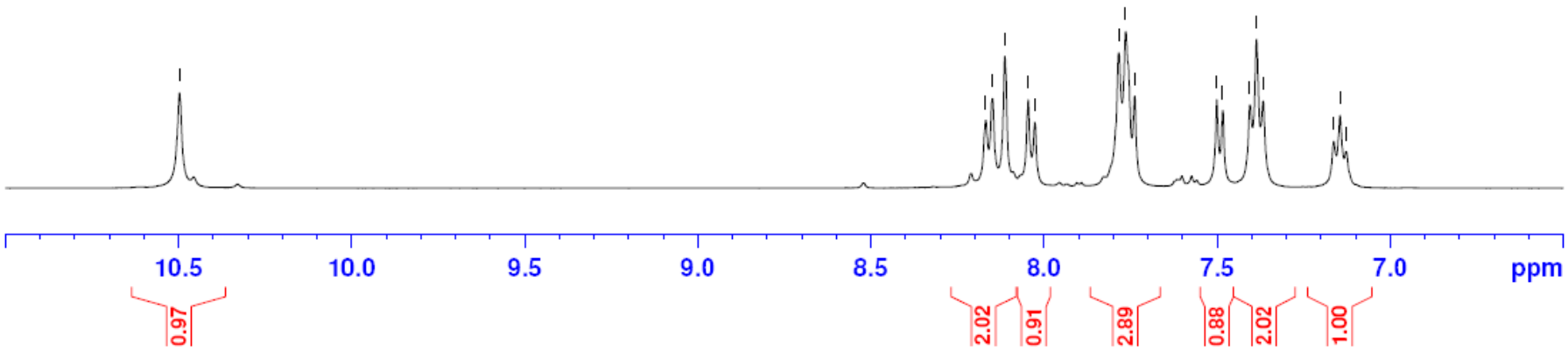
10.497

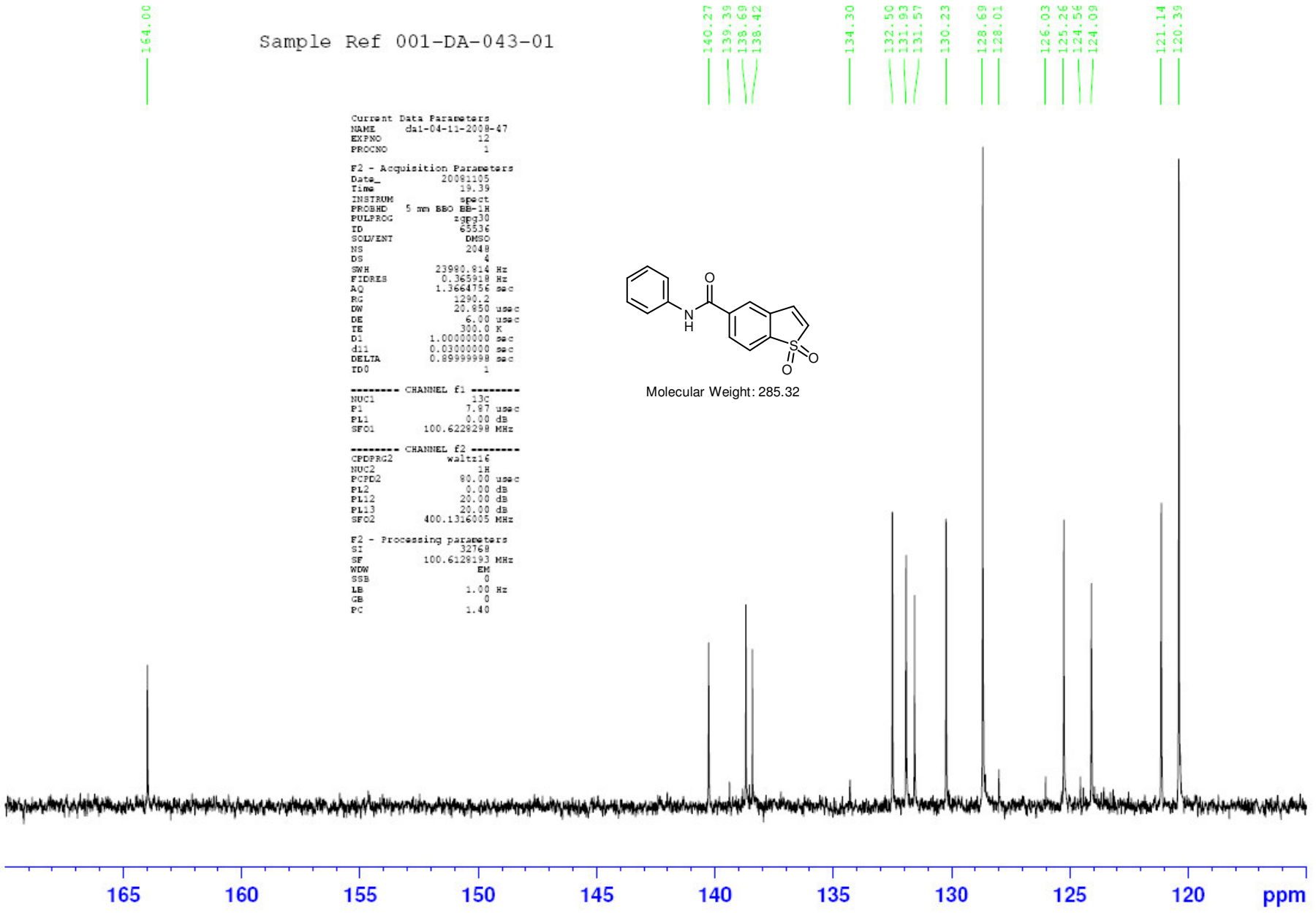
8.169 8.150 8.114 8.047 8.027 7.785 7.765 7.739 7.502 7.485 7.406 7.387 7.368 7.164 7.146 7.128



Molecular Weight: 285.32

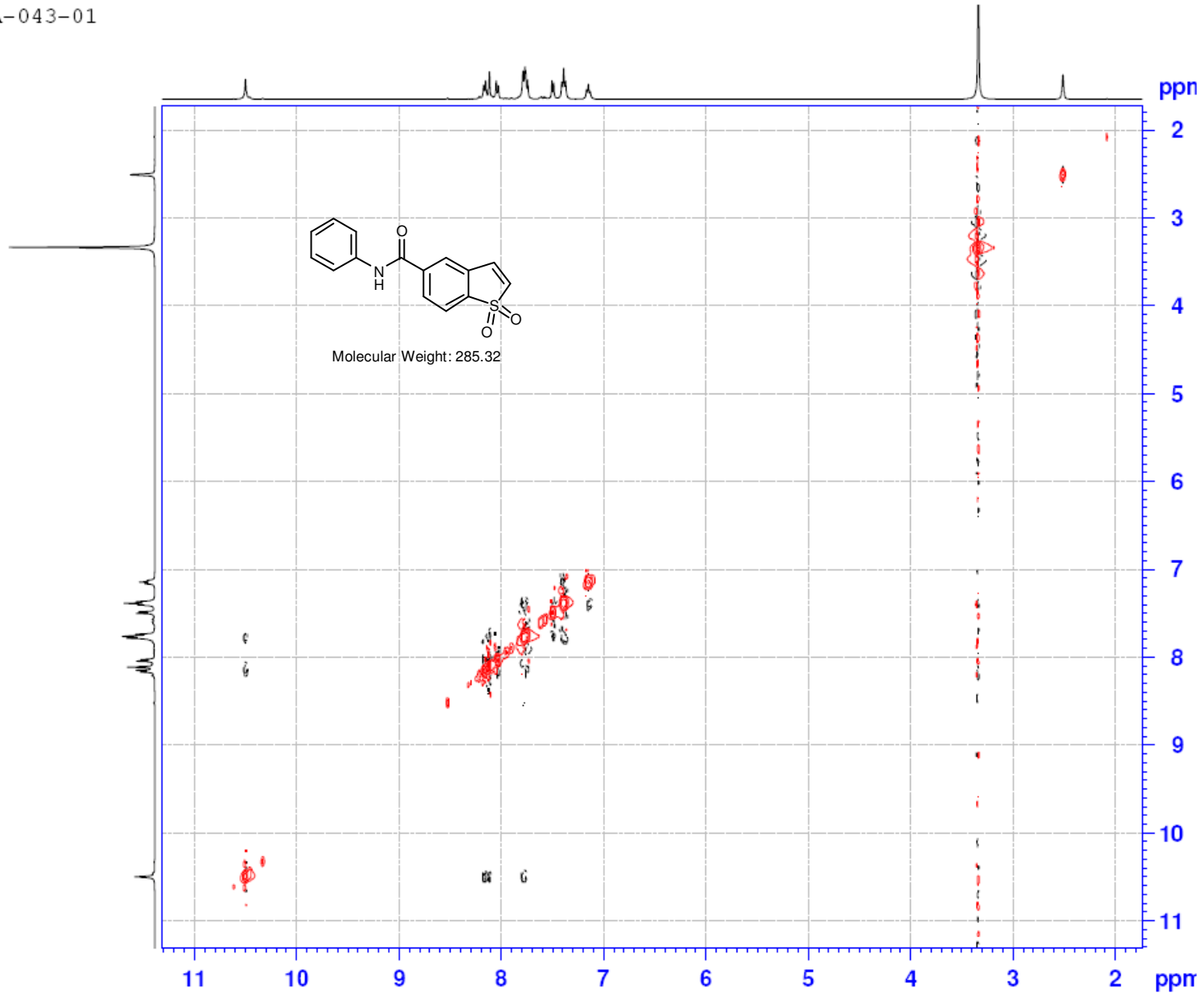
```
Current Data Parameters
NAME      da1-04-11-2008-47
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20081104
Time                 17.31
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                      64
DS                       2
SWH               8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                     256
DW                  60.400 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                      1

-------- CHANNEL f1 --------
NUC1                    1H
P1                   7.88 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                   32768
SF            400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

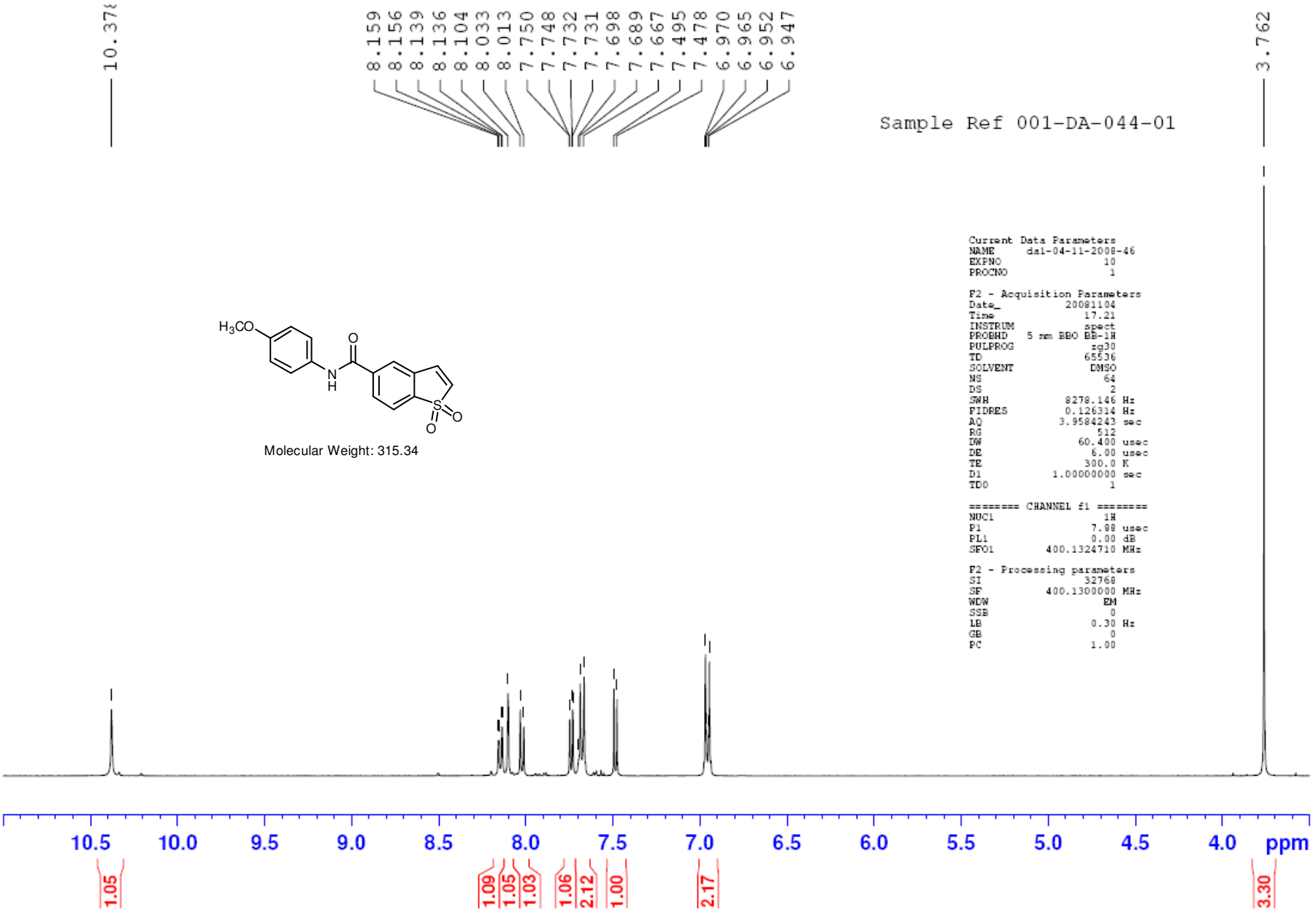


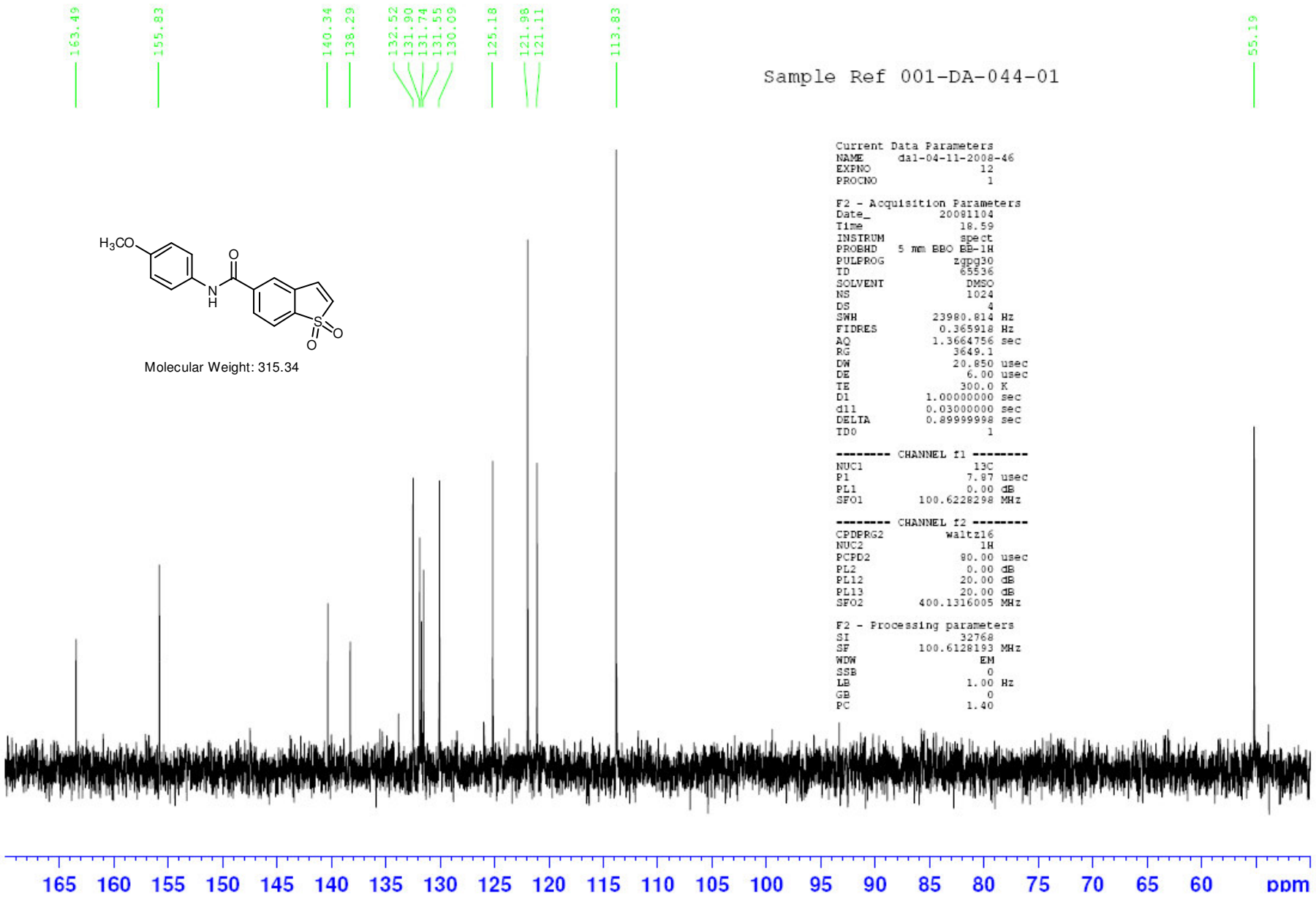

164.00
Sample Ref 001-DA-043-01
140.27
139.39
138.69
138.42
134.30
132.50
131.93
131.57
130.23
128.69
128.01
126.03
125.26
124.56
124.09
121.14
120.39
Current Data Parameters
NAME da1-04-11-2008-47
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081105
Time 19.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Molecular Weight: 285.32
165
160
155
150
145
140
135
130
125
120
ppm

Sample Ref 001-DA-043-01

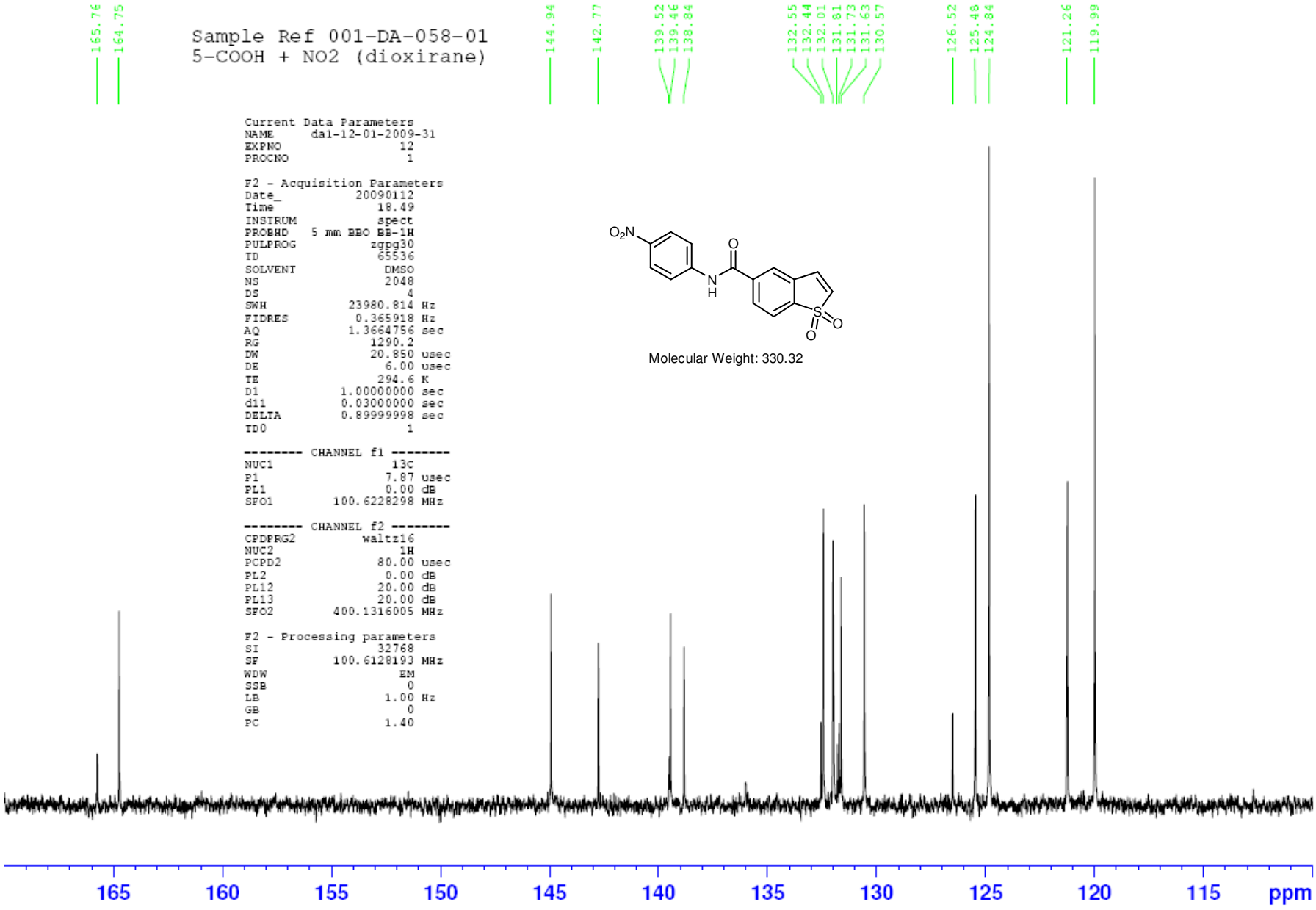
```
Current Data Parameters
NAME      da1-04-11-2008-47
EXPNO                   16
PROCNO                   1

F2 - Acquisition Parameters
Date_              20081105
Time                 21.24
INSTRUM              spect
PROBHD    5 mm BBO BB-1H
PULPROG           noesyph
TD                    2048
SOLVENT               DMSO
NS                       4
DS                       4
SWH                3834.356 Hz
FIDRES             1.872244 Hz
AQ                 0.2671092 sec
RG                     161.3
DW                   130.400 usec
DE                     6.00 usec
TE                    300.0 K
d0                 0.00012037 sec
D1                 1.24996996 sec
D8                 0.80000001 sec
IN0                0.00026080 sec
ST1CNT                  128

======== CHANNEL f1 ========
NUC1                     1H
P1                     7.00 usec
PL1                    0.00 dB
SFO1           400.1326100 MHz

F1 - Acquisition parameters
ND0                      1
TD                     256
SFO1             400.1326 MHz
FIDRES           14.977952 Hz
SW                   9.583 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                    1024
SF             400.1300000 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
PC                    1.00

F1 - Processing parameters
SI                    1024
MC2            States-TPPI
SF             400.1300000 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
```

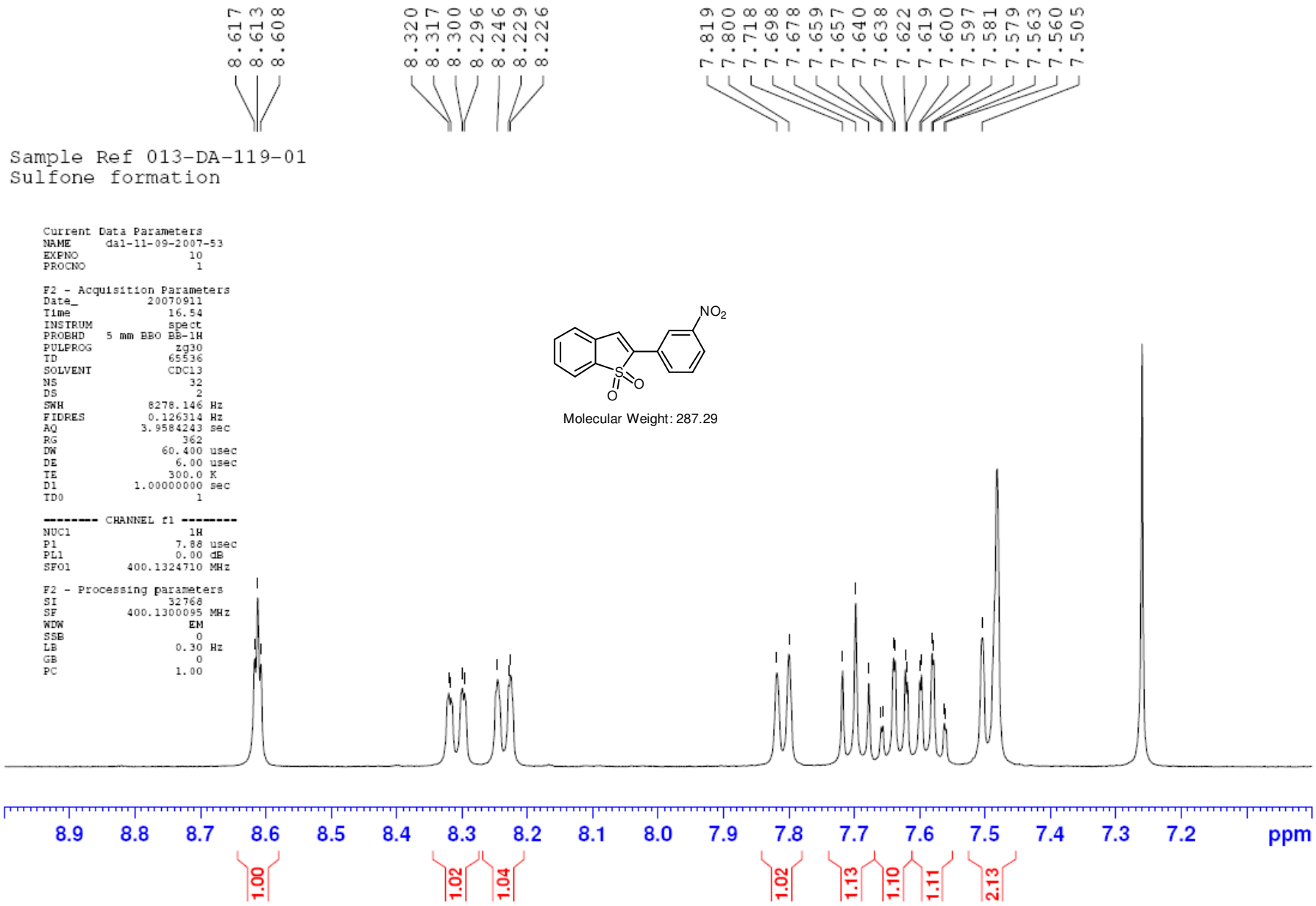



10.378
8.159
8.156
8.139
8.136
8.104
8.033
8.013
7.750
7.748
7.732
7.731
7.698
7.689
7.667
7.495
7.478
6.970
6.965
6.952
6.947
3.762
Sample Ref 001-DA-044-01
H3CO
O
N
H
S
O
O
Molecular Weight: 315.34
Current Data Parameters
NAME dai-04-11-2008-46
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081104
Time 17.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 512
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
1.05
1.09
1.05
1.03
1.06
2.12
1.00
2.17
3.30

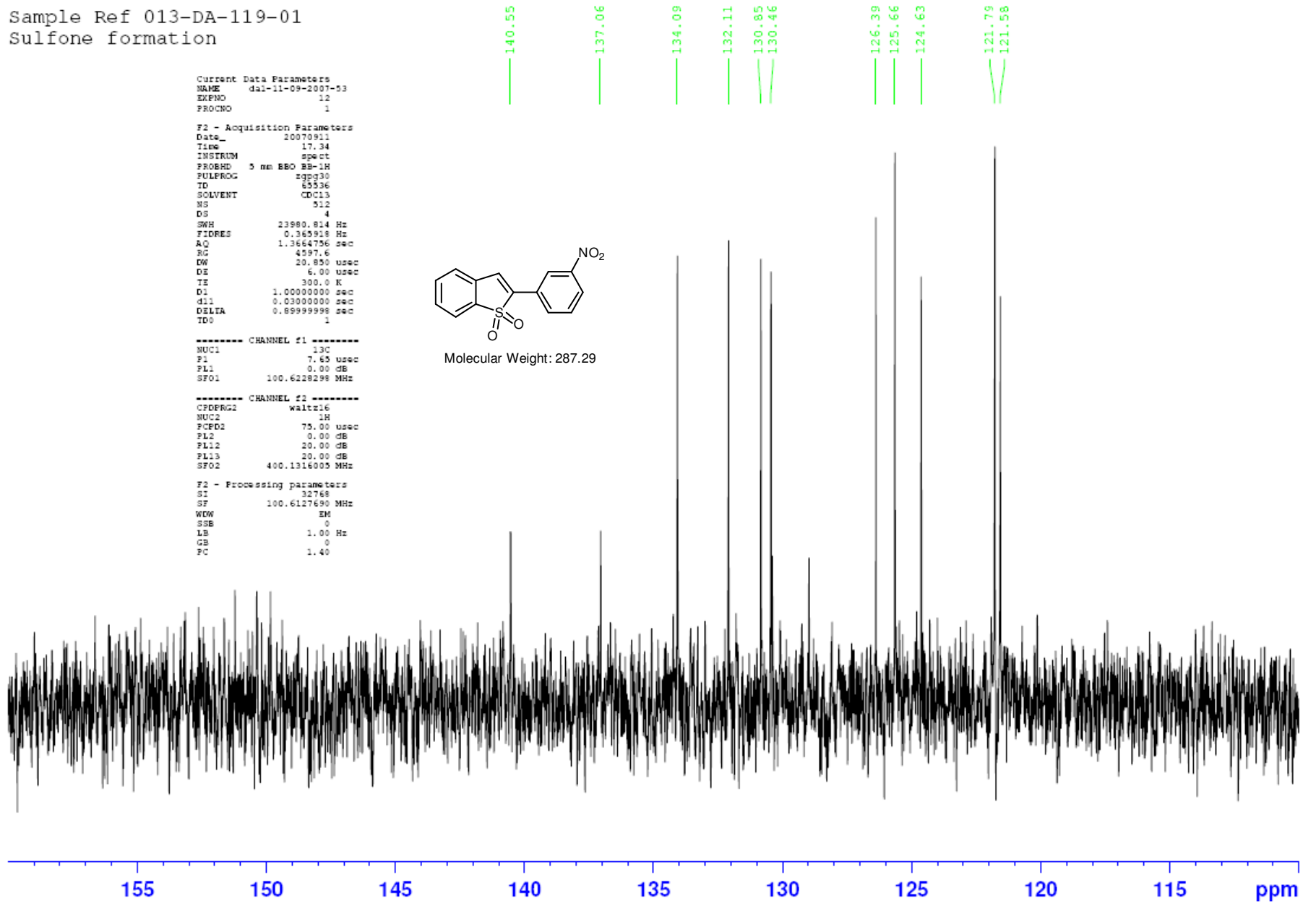

163.49
155.83
140.34
138.29
132.52
131.90
131.74
131.55
130.09
125.18
121.98
121.11
113.83
55.19
Sample Ref 001-DA-044-01
H3CO
N
H
O
S
O
O
Molecular Weight: 315.34
Current Data Parameters
NAME da1-04-11-2008-46
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081104
Time 18.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3649.1
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 ppm

11.04
8.306
8.195
8.176
8.143
8.130
8.089
8.069
8.061
8.038
7.979
7.960
7.770
7.753
7.737
7.720
7.530
7.513
7.487
7.470
Sample Ref 001-DA-058-01
5-COOH + NO2 (dioxirane)
Current Data Parameters
NAME dal-12-01-2009-31
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090112
Time 17.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O2N
O
N
H
S
O
O
Molecular Weight: 330.32
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
ppm
1.00
2.05
1.01
1.54
3.00
0.31
0.99
0.29
0.97
0.27

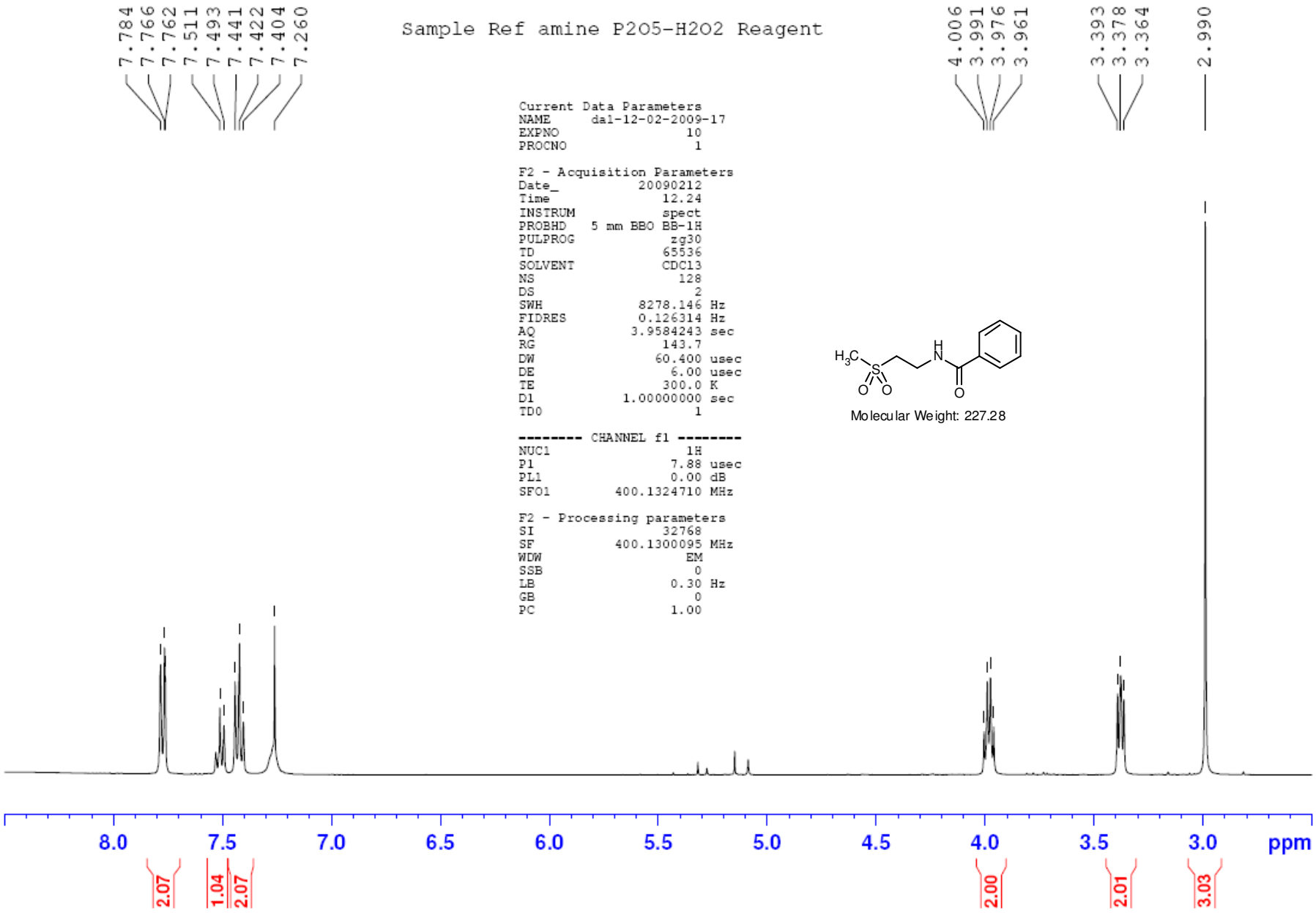

165.76
164.75
144.94
142.77
139.52
139.46
138.84
132.55
132.44
132.01
131.81
131.73
131.63
130.57
126.52
125.48
124.84
121.26
119.99
Sample Ref 001-DA-058-01
5-COOH + NO2 (dioxirane)
Current Data Parameters
NAME dai-12-01-2009-31
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090112
Time 18.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 294.6 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O2N
N
H
O
S
O
O
Molecular Weight: 330.32
165
160
155
150
145
140
135
130
125
120
115
ppm

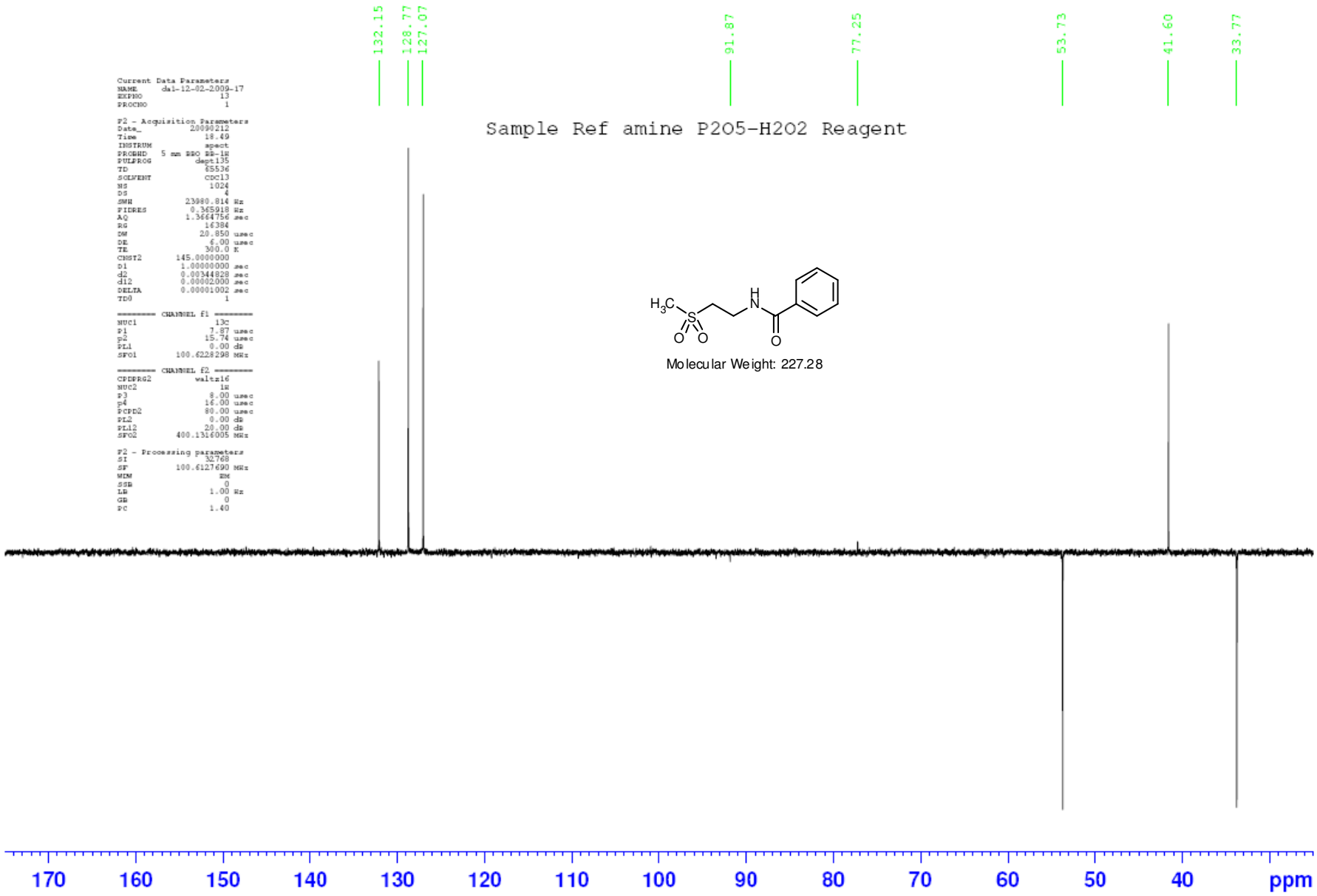

8.617
8.613
8.608
8.320
8.317
8.300
8.296
8.246
8.229
8.226
7.819
7.800
7.718
7.698
7.678
7.659
7.657
7.640
7.638
7.622
7.619
7.600
7.597
7.581
7.579
7.563
7.560
7.505
Sample Ref 013-DA-119-01
Sulfone formation
Current Data Parameters
NAME dal-11-09-2007-53
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070911
Time 16.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
S
O
O
Molecular Weight: 287.29
8.9
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
ppm
1.00
1.02
1.04
1.02
1.13
1.10
1.11
2.13

Sample Ref 013-DA-119-01
Sulfone formation

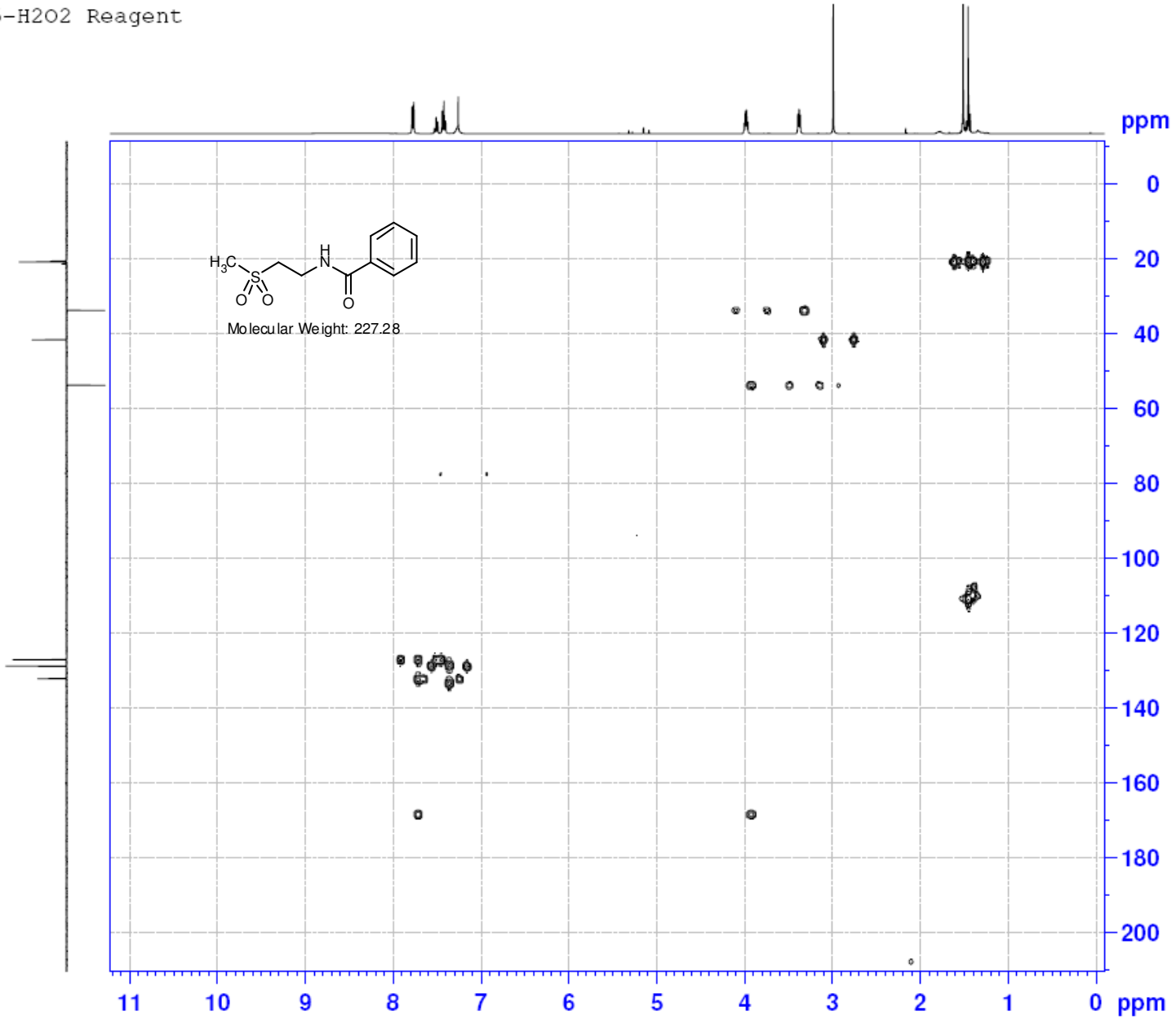



```
Current Data Parameters
NAME      da1-11-09-2007-53
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_              20070911
Time                  17.34
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                4597.6
DW                20.850 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
d11            0.03000000 sec
DELTA          0.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.65 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              75.00 usec
PL2                 0.00 dB
PL12               20.00 dB
PL13               20.00 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
```

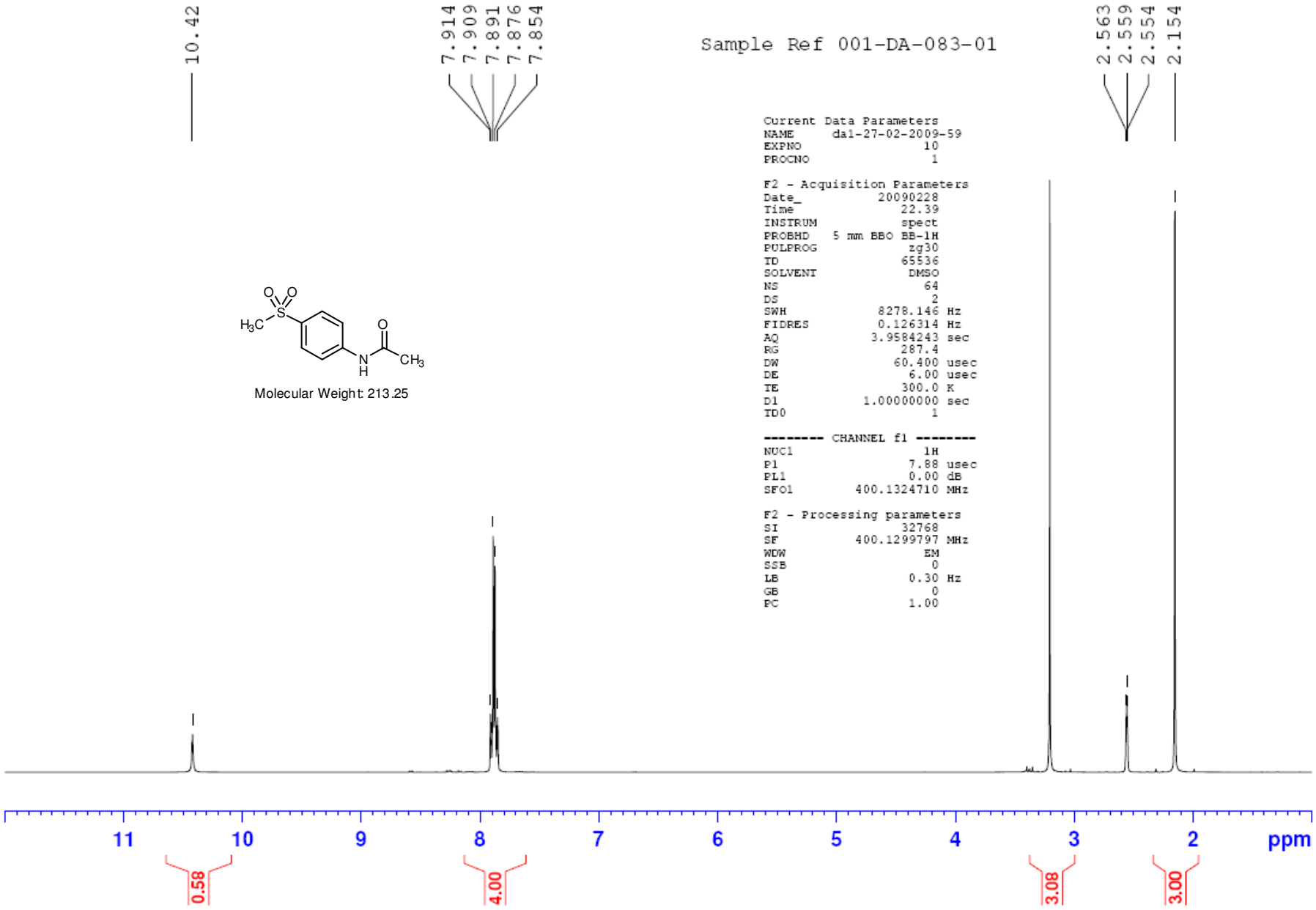

Sample Ref amine P2O5-H2O2 Reagent
7.784
7.766
7.762
7.511
7.493
7.441
7.422
7.404
7.260
4.006
3.991
3.976
3.961
3.393
3.378
3.364
2.990
Current Data Parameters
NAME dal-12-02-2009-17
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090212
Time 12.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Molecular Weight: 227.28
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
2.07
1.04
2.07
2.00
2.01
3.03

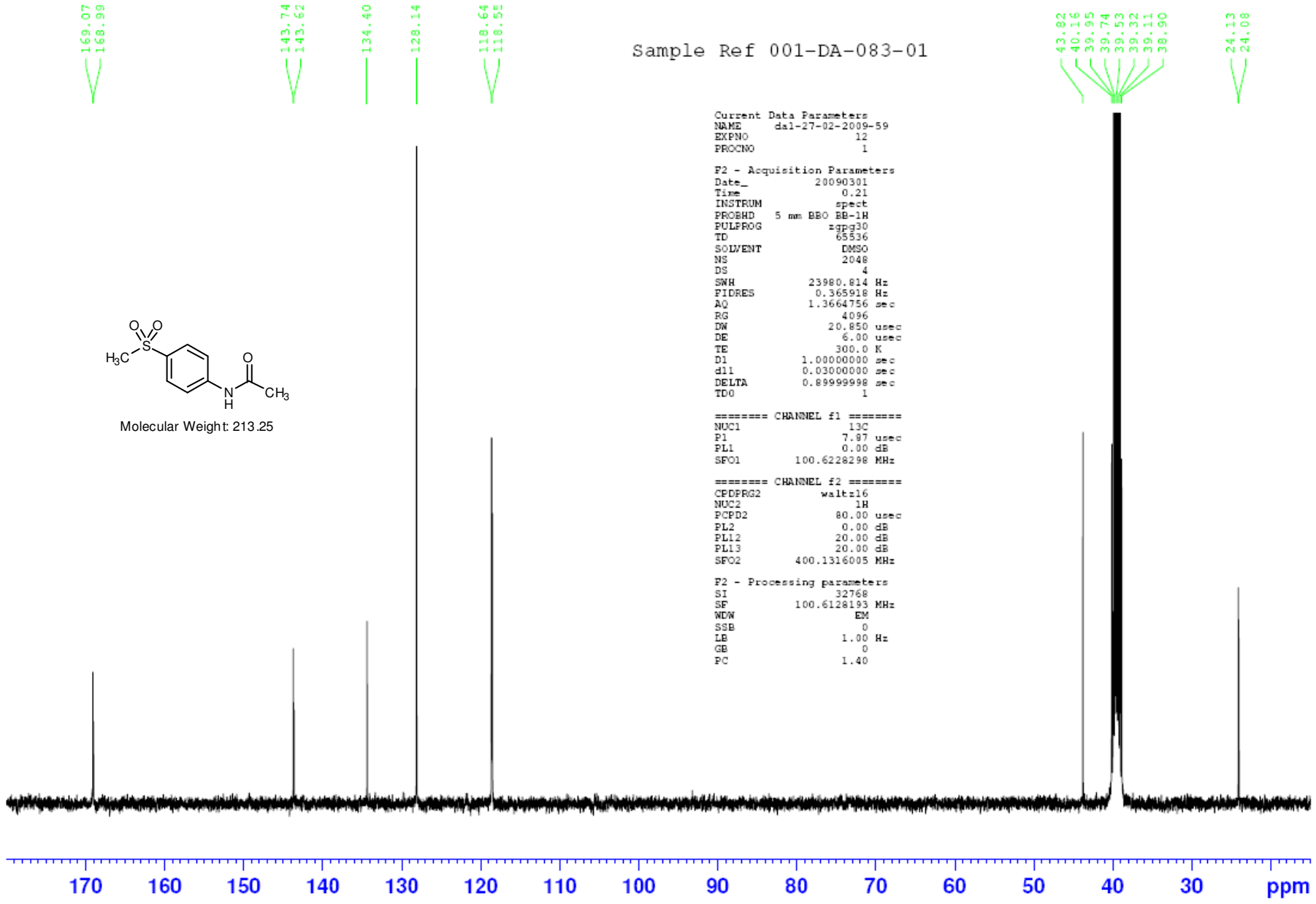

132.15
128.77
127.07
91.87
77.25
53.73
41.60
33.77
Sample Ref amine P205-H202 Reagent
Current Data Parameters
NAME dal-12-02-2009-17
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090212
Time 18.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 1.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001002 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
P2 15.74 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 8.00 usec
p4 16.00 usec
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 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
H3C
S
O
O
H
N
O
Molecular Weight: 227.28
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm

Sample Ref amine P2O5-H2O2 Reagent

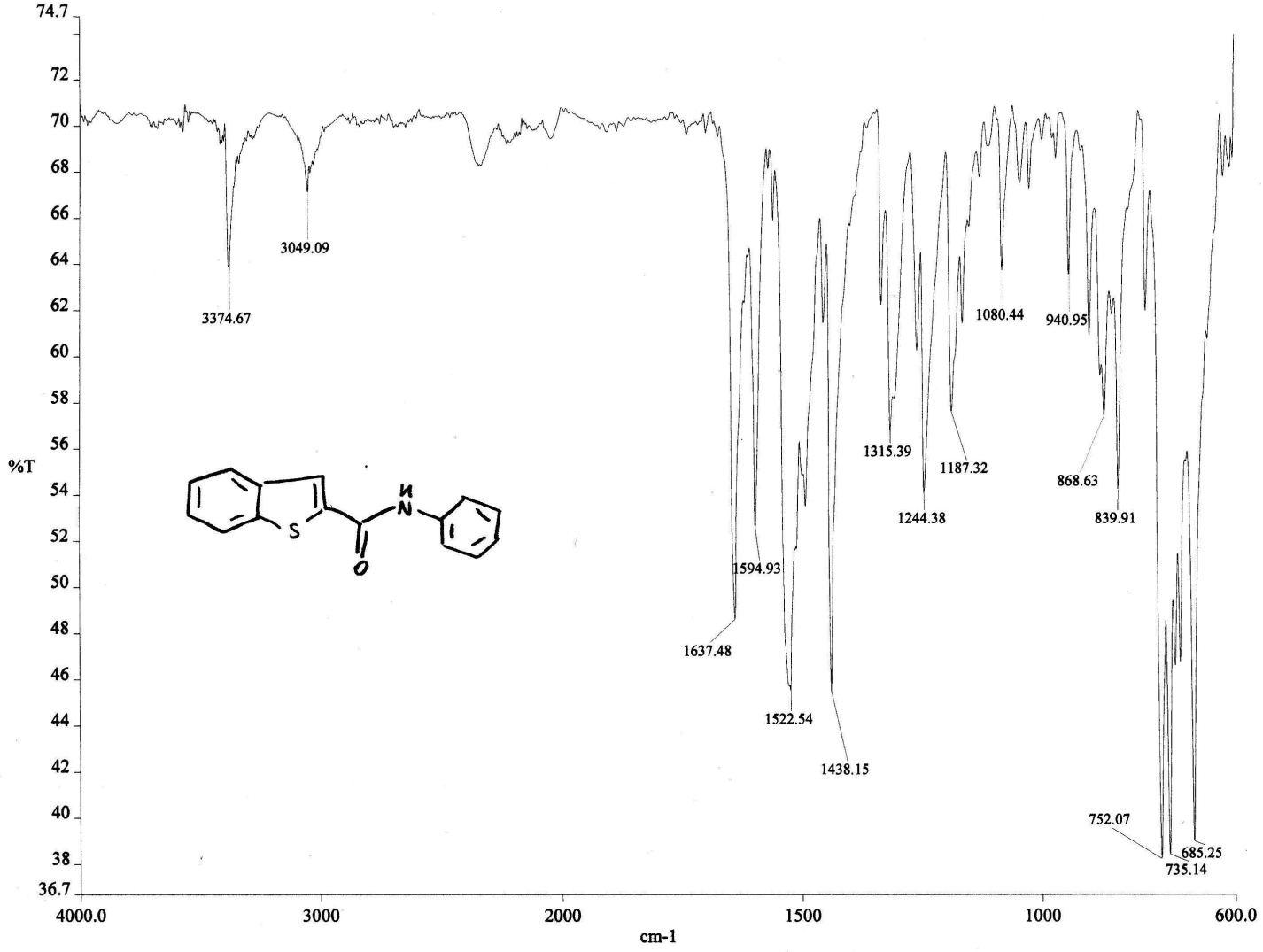



```
Current Data Parameters
NAME      da1-12-02-2009-17
EXPNO                   15
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090212
Time                 19.16
INSTRUM                spect
PROBHD   5 mm BBO BB-1H
PULPROG     inv4gplrndqf
TD                     1024
SOLVENT              CDCl3
NS                        8
DS                       16
SWH                4528.985 Hz
FIDRES             4.422837 Hz
AQ                 0.1130996 sec
RG                     16384
DW                   110.400 usec
DE                     6.00 usec
TE                    300.1 K
d0                 0.00000300 sec
D1                 1.48320603 sec
D6                 0.07100000 sec
d13                0.00000400 sec
D16                0.00010000 sec
IN0                0.00002240 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                      7.88 usec
P2                     15.76 usec
PL1                     0.00 dB
SFO1           400.1322595 MHz

======== CHANNEL f2 ========
NUC2                      13C
P3                      7.65 usec
PL2                     0.00 dB
SFO2           100.6227746 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPNAM3              SINE.100
GPZ1                   50.00 %
GPZ2                   30.00 %
GPZ3                   40.10 %
P16                  1000.00 usec

F1 - Acquisition parameters
ND0                       2
TD                      128
SFO1             100.6228 MHz
FIDRES          174.386154 Hz
SW                 221.833 ppm
FnMODE                  QF

F2 - Processing parameters
SI                      1024
SF             400.1300345 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                     QF
SF             100.6127690 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
```


10.42
7.914
7.909
7.891
7.876
7.854
2.563
2.559
2.554
2.154
Sample Ref 001-DA-083-01
Current Data Parameters
NAME da1-27-02-2009-59
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090228
Time 22.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 7.88 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1299797 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Molecular Weight: 213.25
11
10
9
8
7
6
5
4
3
2
ppm
0.58
4.00
3.08
3.00

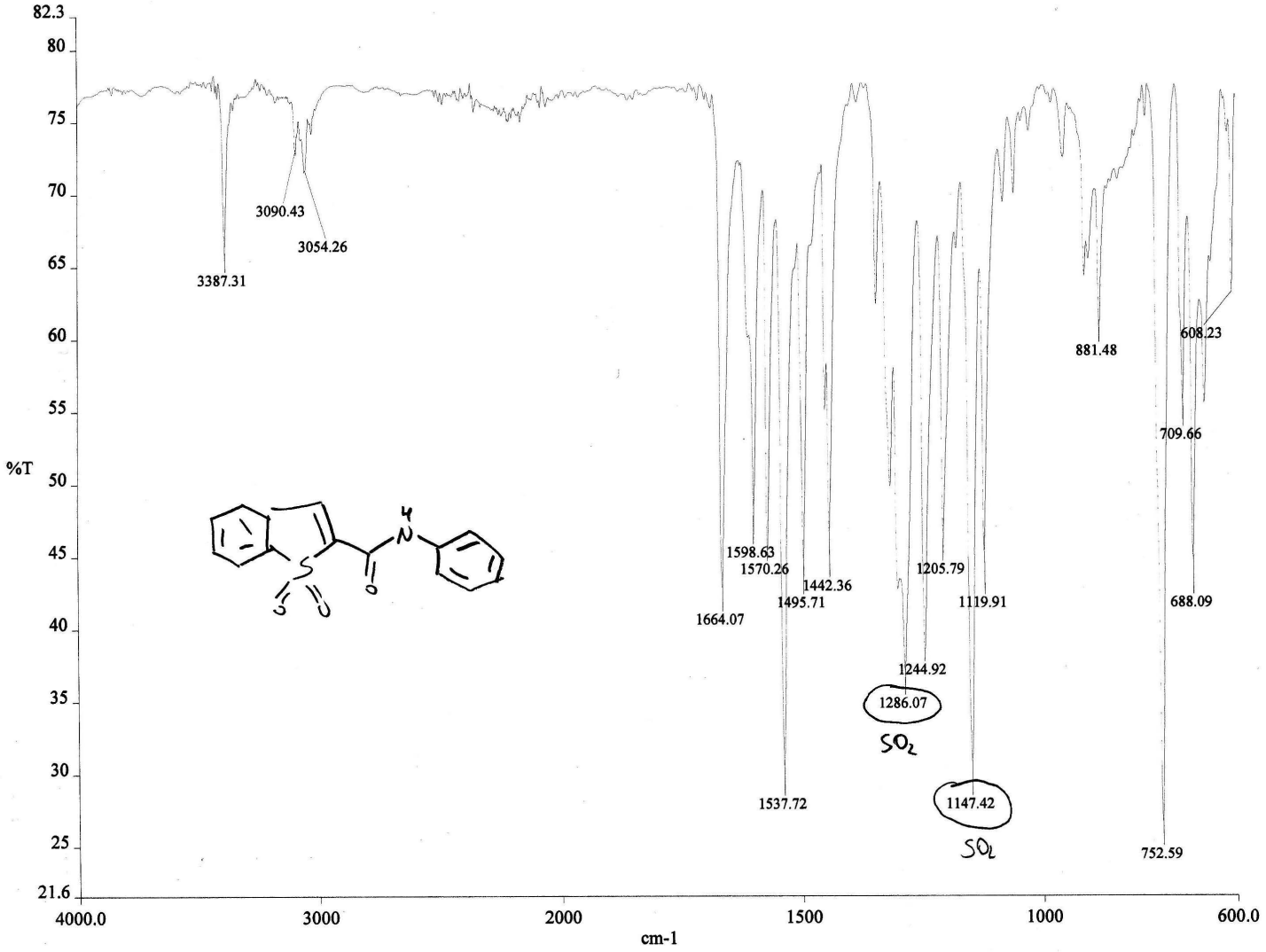

169.07
168.99
143.74
143.62
134.40
128.14
118.64
118.58
43.82
40.16
39.95
39.74
39.53
39.32
39.11
38.90
24.13
24.08
Sample Ref 001-DA-083-01
Molecular Weight: 213.25
Current Data Parameters
NAME dal-27-02-2009-59
EXPNO 12
PROCNO 1
P2 - Acquisition Parameters
Date_ 20090301
Time 0.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.87 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz
P2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

IR spectra for all novel compounds



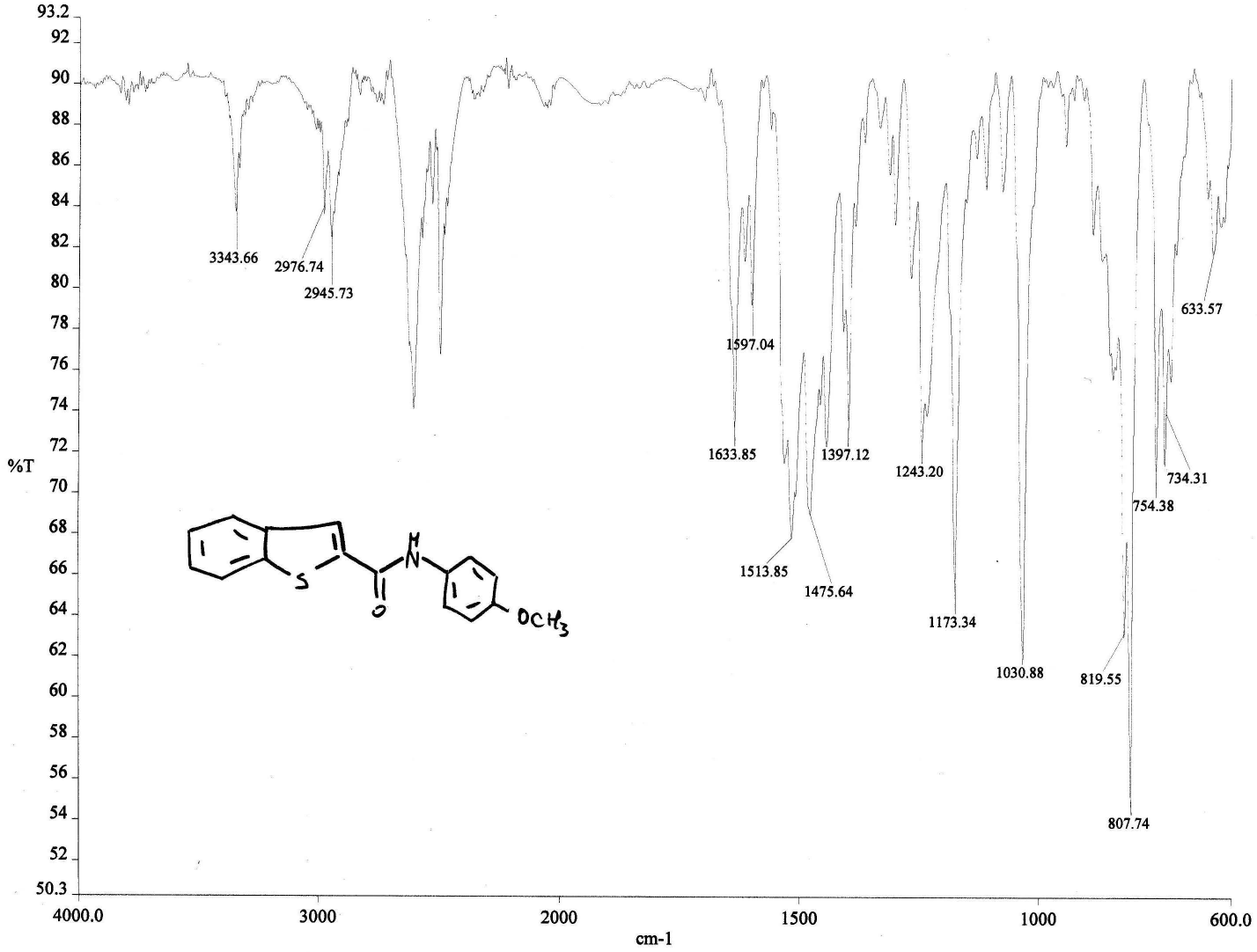
c:\pel_data\spectra\dyeison\001-da-030-01.sp - 001-DA-030-01

m.p. 205 - 207 °C

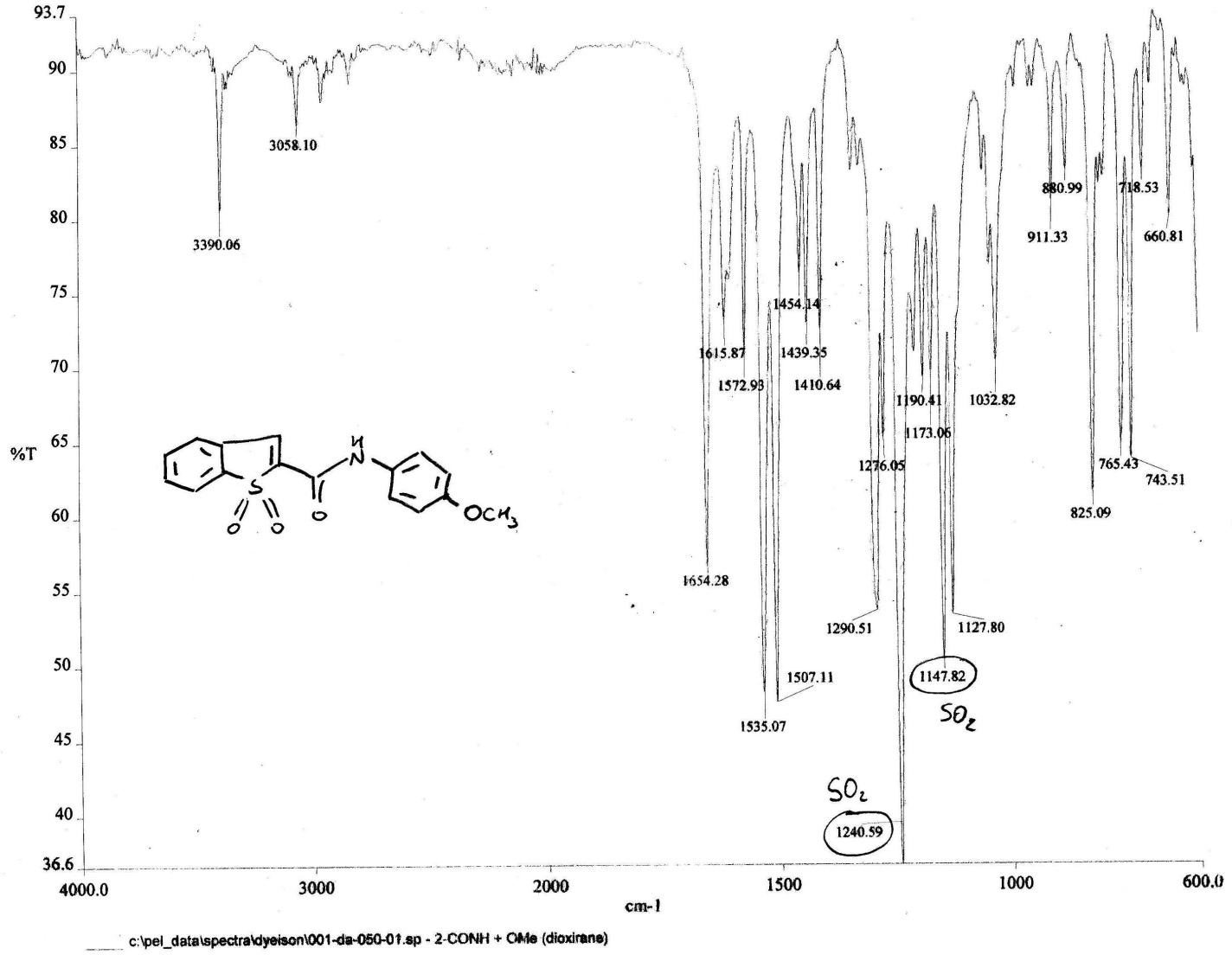


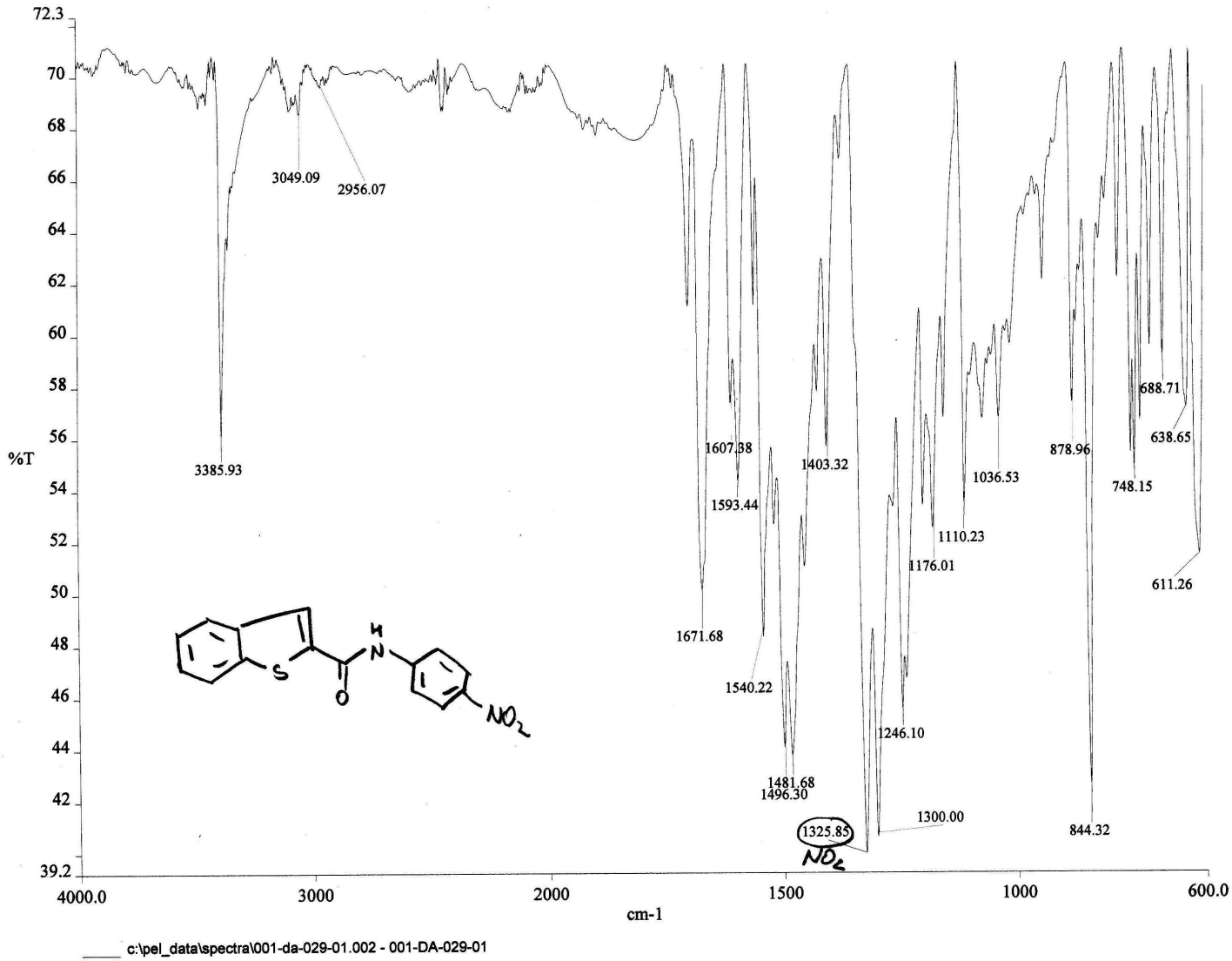
c:\pel_data\spectra\dyeison\001-da-038-01.sp - 001-DA-038-01

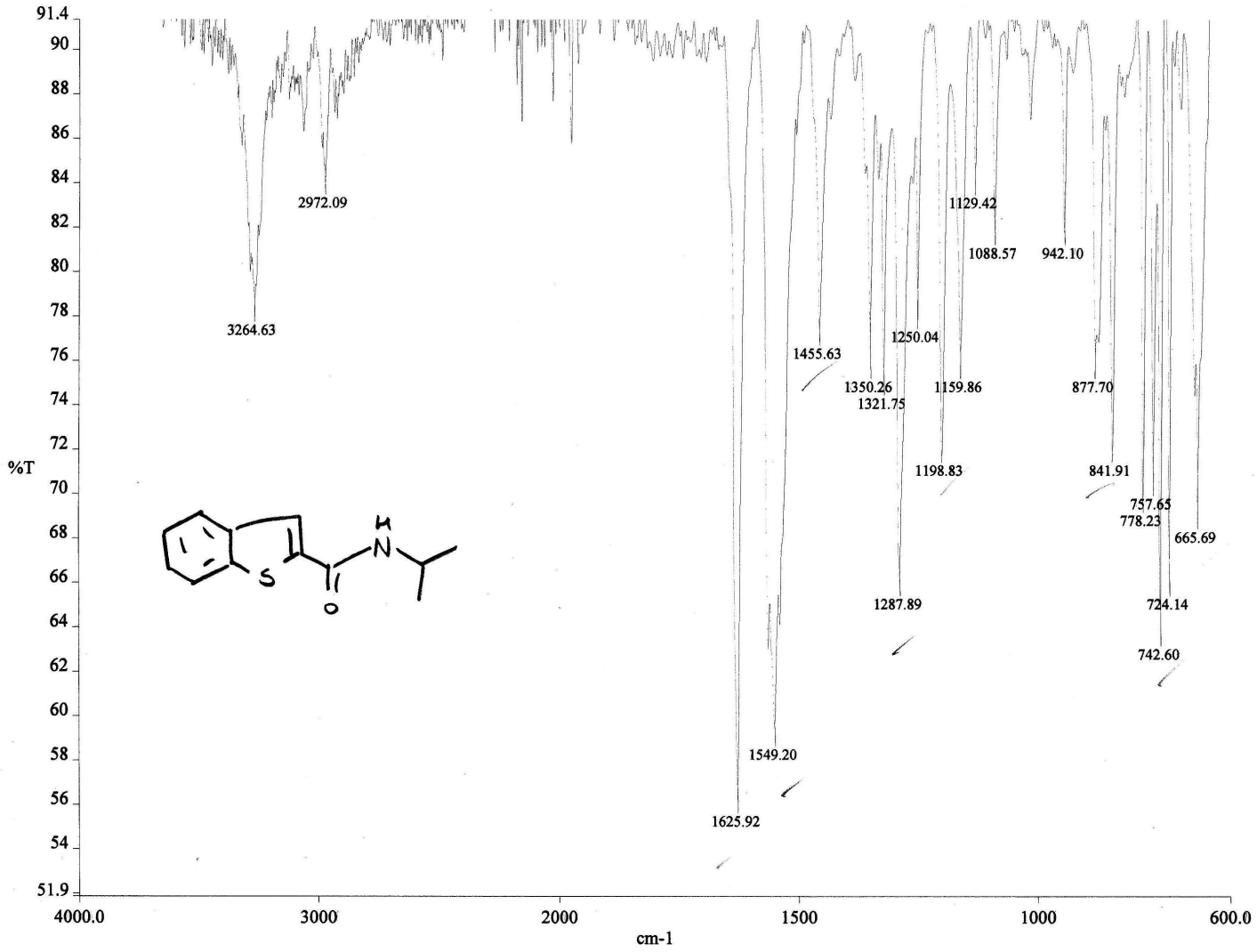
m.p. 245-246


93.2
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50.3
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3343.66
2976.74
2945.73
1597.04
1633.85
1397.12
1243.20
1513.85
1475.64
1173.34
1030.88
819.55
807.74
754.38
734.31
633.57
S
H
N
O
OCH3
c:\pel_data\spectra\dyeison\001-da-031-02.sp - 001-DA-031-02

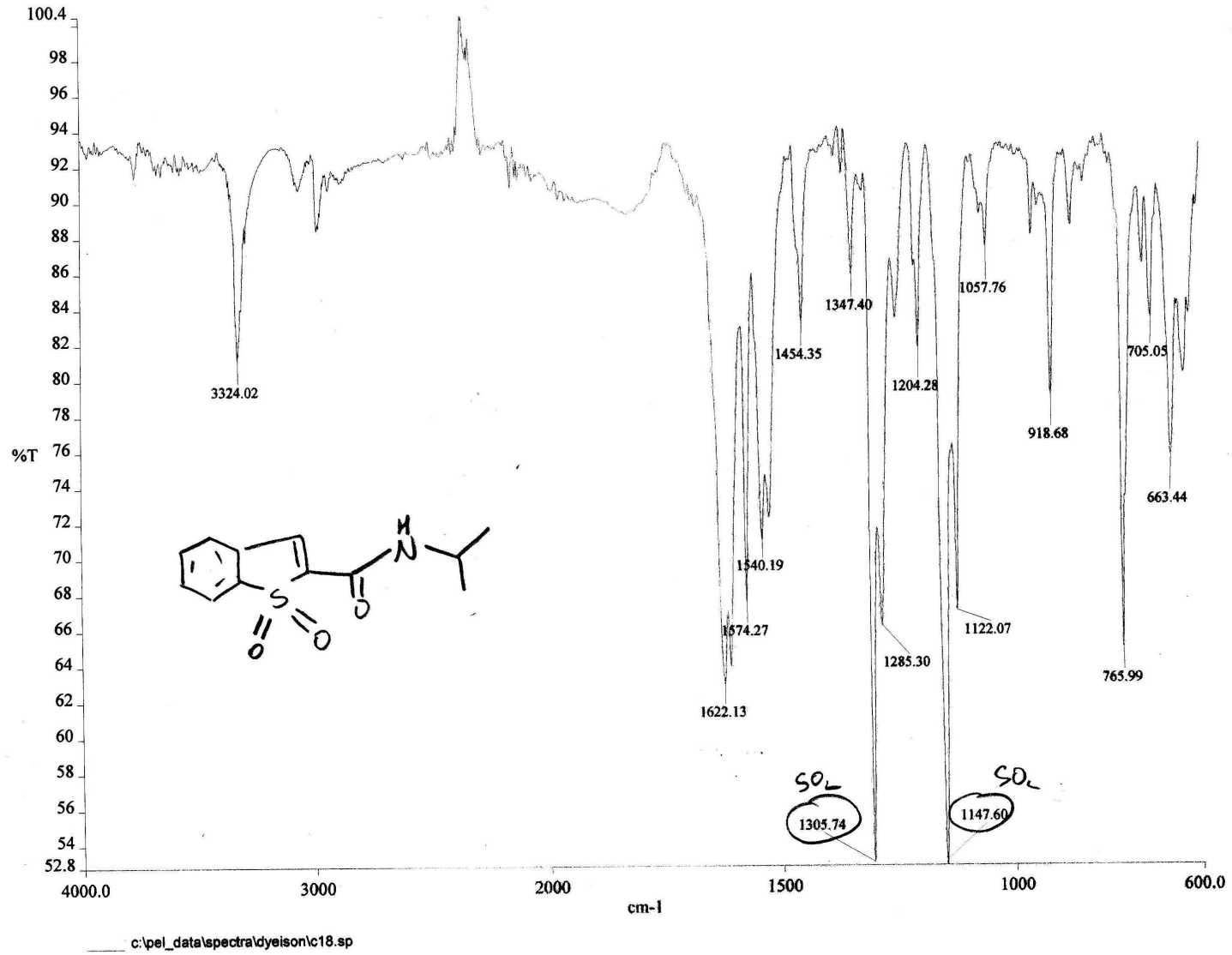
226 - 229

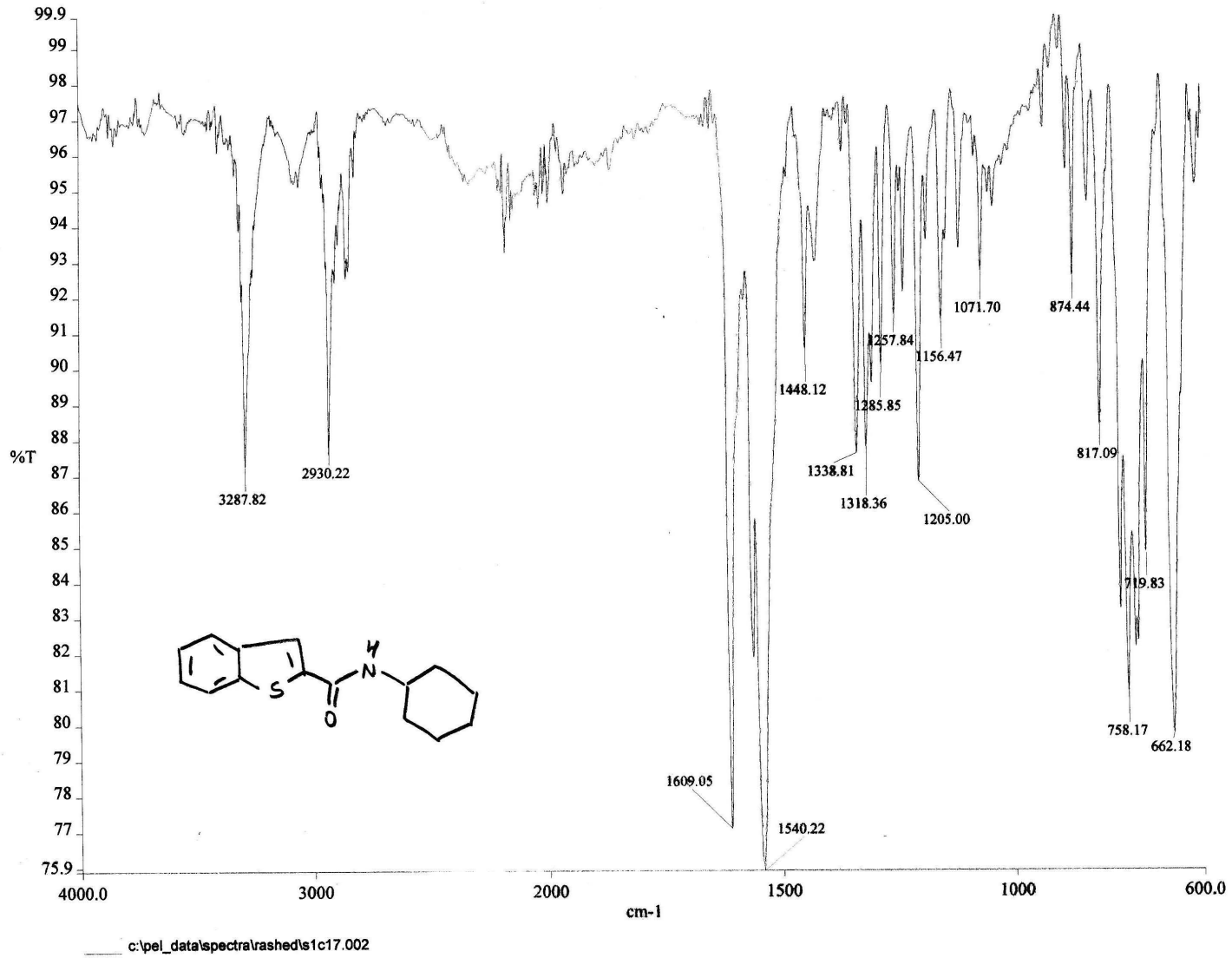

93.7
90
85
80
75
70
65
60
55
50
45
40
36.6
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3390.06
3058.10
1654.28
1615.87
1572.93
1535.07
1507.11
1454.14
1439.35
1410.64
1290.51
1276.05
1240.59
1190.41
1173.06
1147.82
1127.80
1032.82
911.33
880.99
825.09
765.43
743.51
718.53
660.81
SO2
SO2
OCH3
c:\pel_data\spectra\dyeison\001-da-050-01.sp - 2-CONH + OMe (dioxirane)

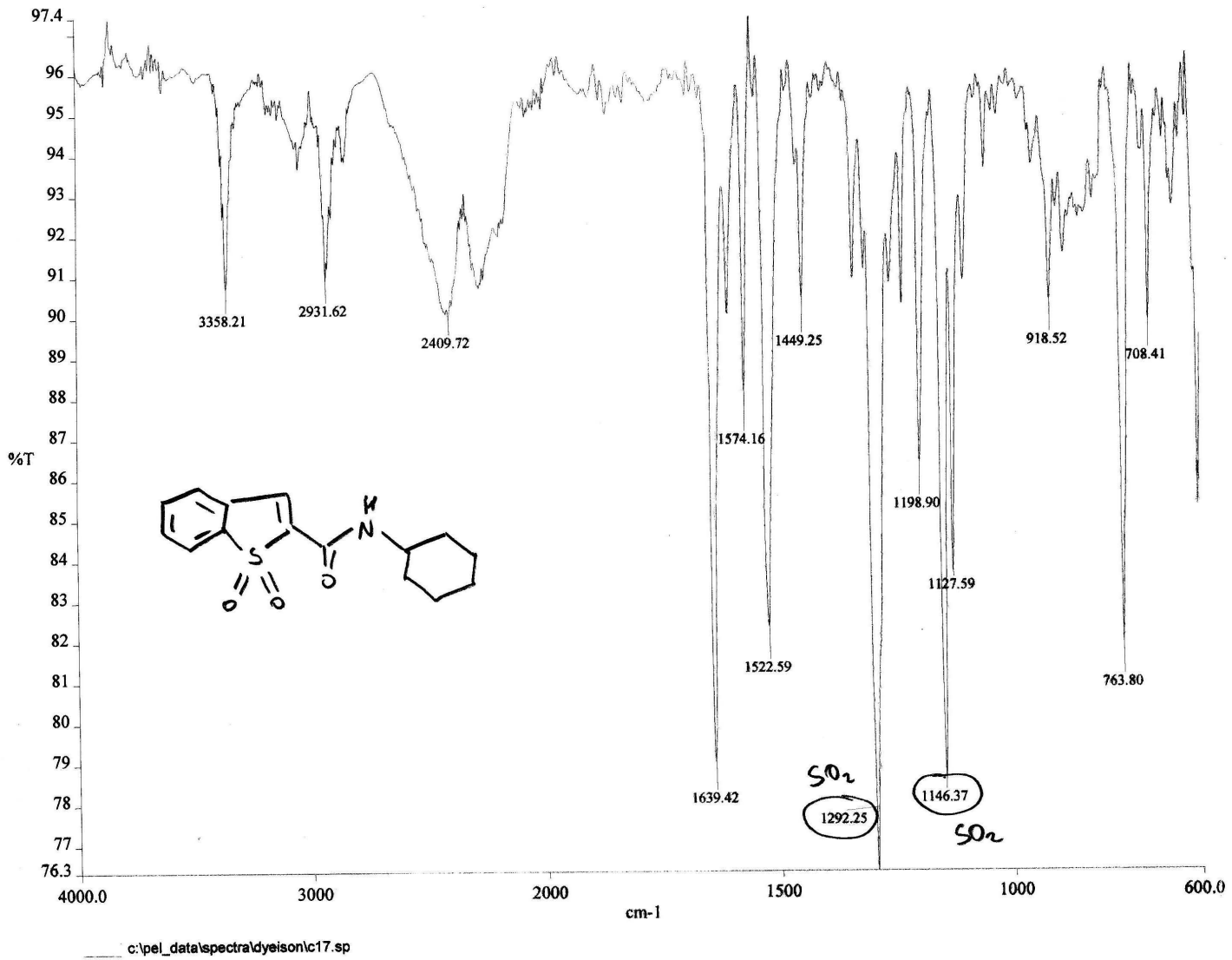

72.3
70
68
66
64
62
60
58
56
54
52
50
48
46
44
42
39.2
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3385.93
3049.09
2956.07
1671.68
1607.38
1593.44
1540.22
1481.68
1496.30
1403.32
1325.85
1300.00
1246.10
1176.01
1110.23
1036.53
878.96
844.32
748.15
688.71
638.65
611.26
NO2
c:\pel_data\spectra\001-da-029-01.002 - 001-DA-029-01

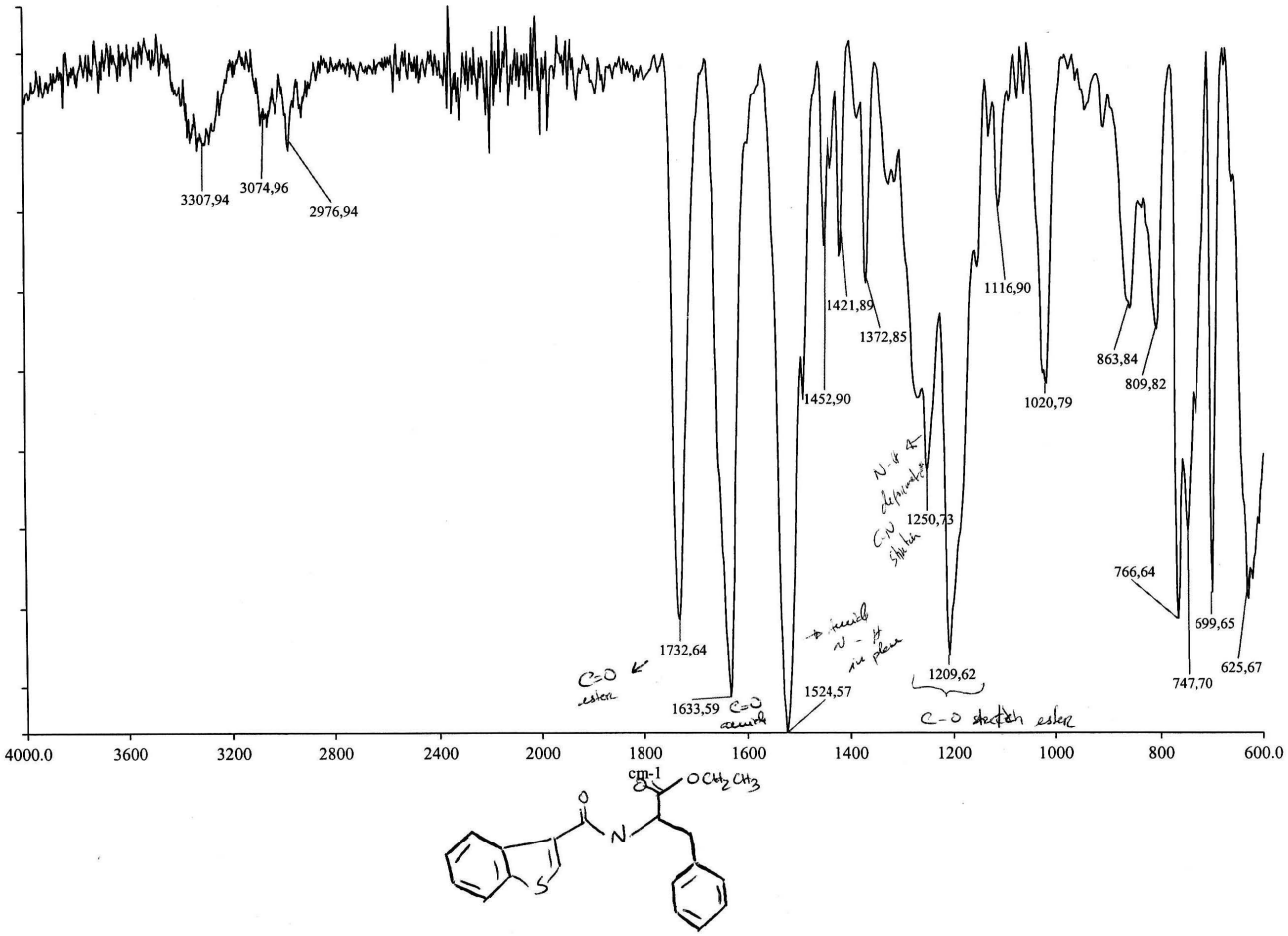


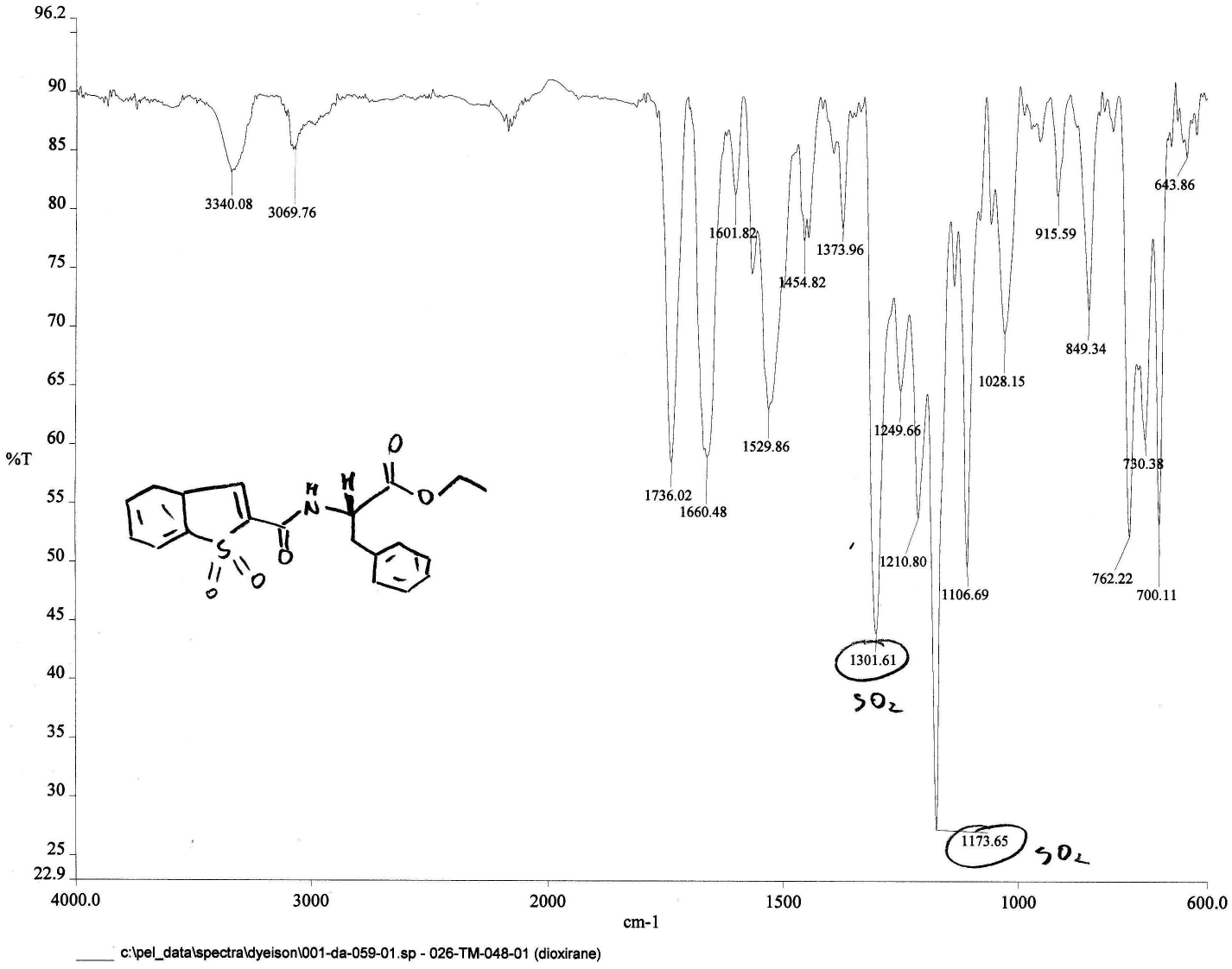
117.7 - 148.5°C Rf = 0.50 (EA)

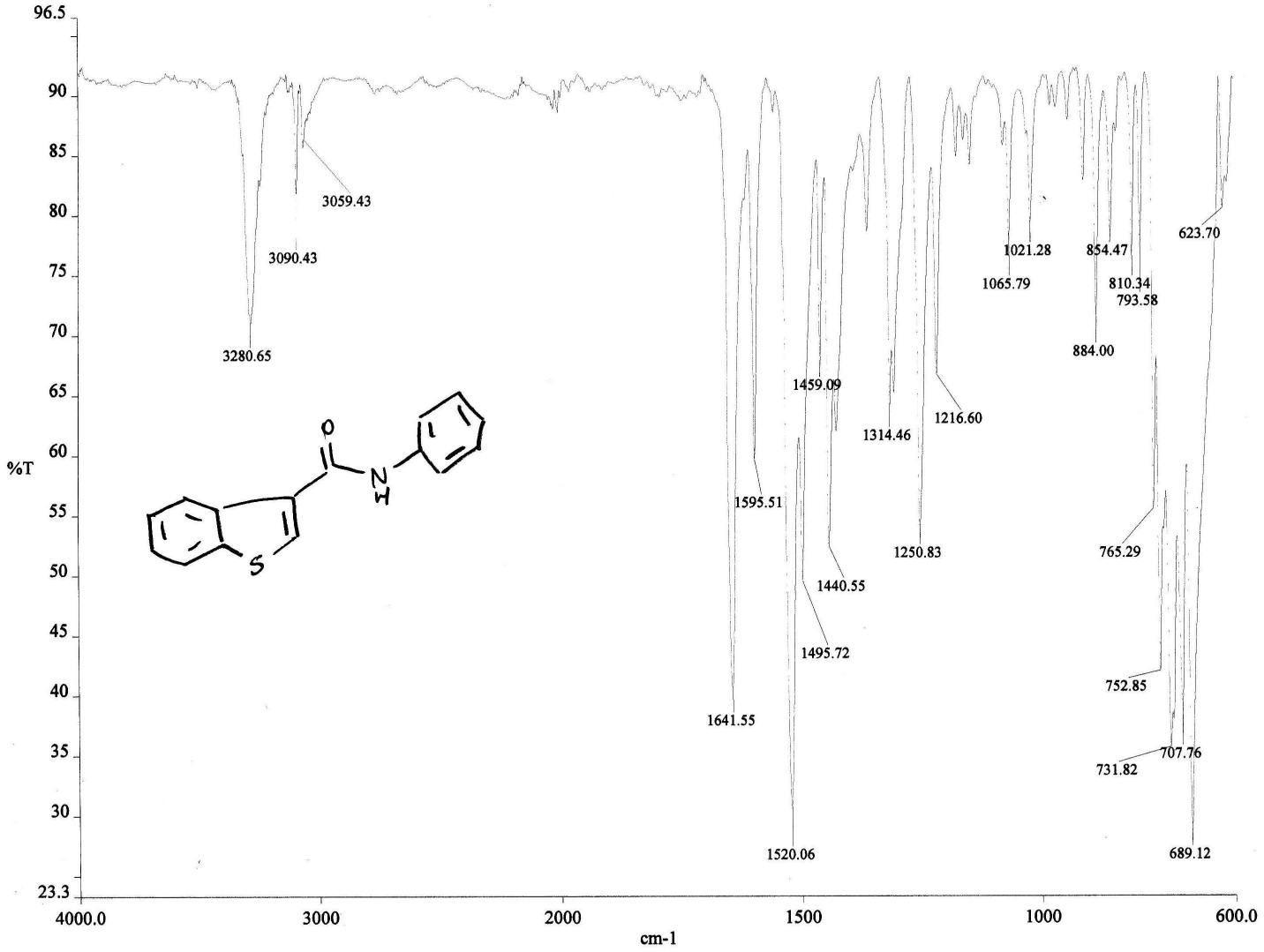

100.4
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52.8
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3324.02
1622.13
1574.27
1540.19
1454.35
1347.40
1285.30
1204.28
1122.07
1057.76
918.68
765.99
705.05
663.44
1305.74
1147.60
SO2
SO2
c:\pel_data\spectra\dyeison\c18.sp


99.9
99
98
97
96
95
94
93
92
91
90
89
88
87
86
85
84
83
82
81
80
79
78
77
75.9
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3287.82
2930.22
1609.05
1540.22
1448.12
1338.81
1318.36
1285.85
1257.84
1205.00
1156.47
1071.70
874.44
817.09
758.17
719.83
662.18
S
O
N
H
c:\pel_data\spectra\rashed\s1c17.002


97.4
96
95
94
93
92
91
90
89
88
87
86
85
84
83
82
81
80
79
78
77
76.3
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3358.21
2931.62
2409.72
1639.42
1574.16
1522.59
1449.25
1292.25
1198.90
1146.37
1127.59
918.52
763.80
708.41
SO2
SO2
H
N
O
S
O
O
c:\pel_data\spectra\dyeison\c17.sp

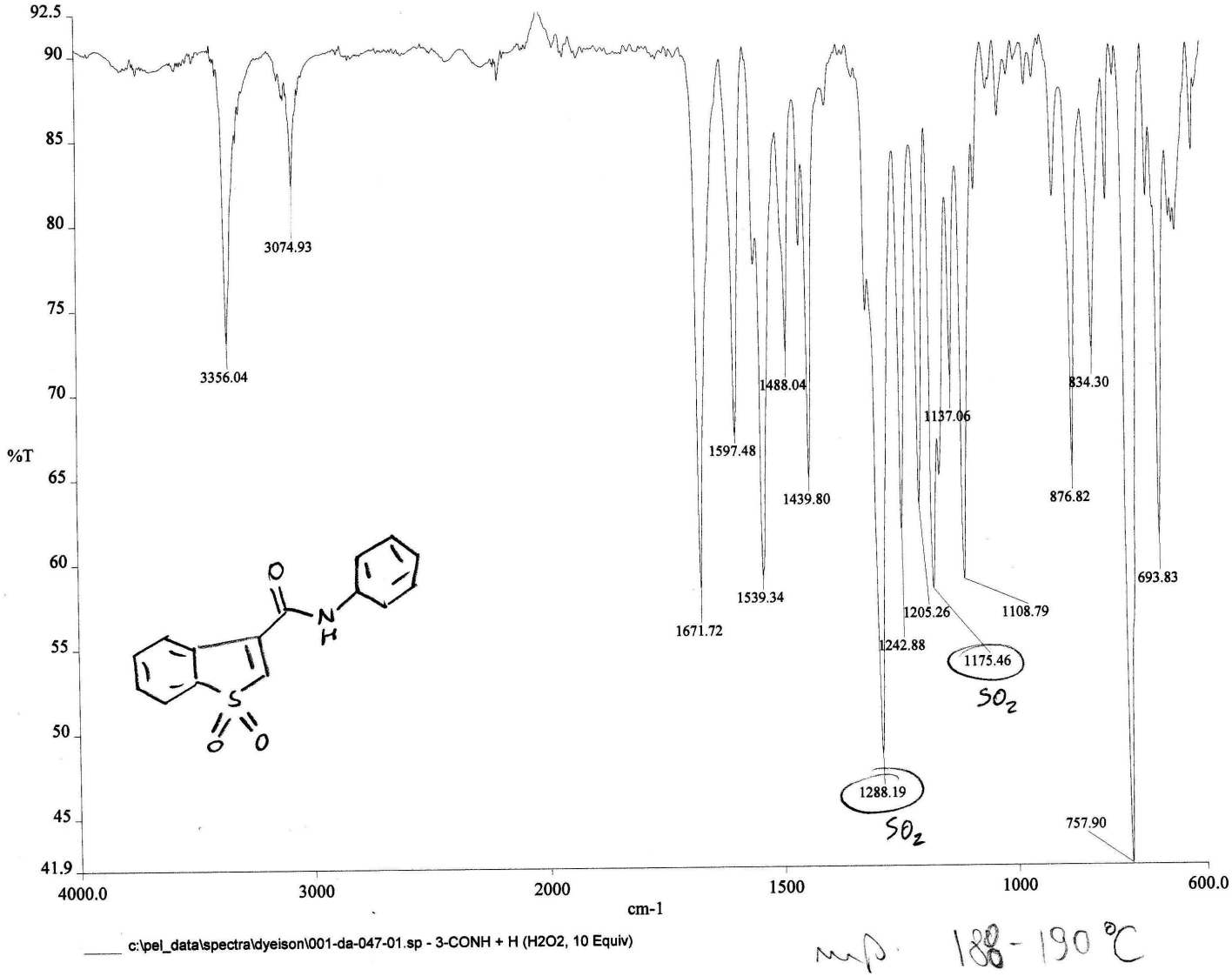

3307,94
3074,96
2976,94
1732,64
1633,59
1524,57
1452,90
1421,89
1372,85
1250,73
1209,62
1116,90
1020,79
863,84
809,82
766,64
747,70
699,65
625,67
4000.0
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600.0
cm-1
O
N
S
OCH2CH3


96.2
90
85
80
75
70
65
60
55
50
45
40
35
30
25
22.9
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3340.08
3069.76
1736.02
1660.48
1601.82
1529.86
1454.82
1373.96
1301.61
1249.66
1210.80
1173.65
1106.69
1028.15
915.59
849.34
762.22
730.38
700.11
643.86
SO2
SO2
c:\pel_data\spectra\dyeison\001-da-059-01.sp - 026-TM-048-01 (dioxirane)

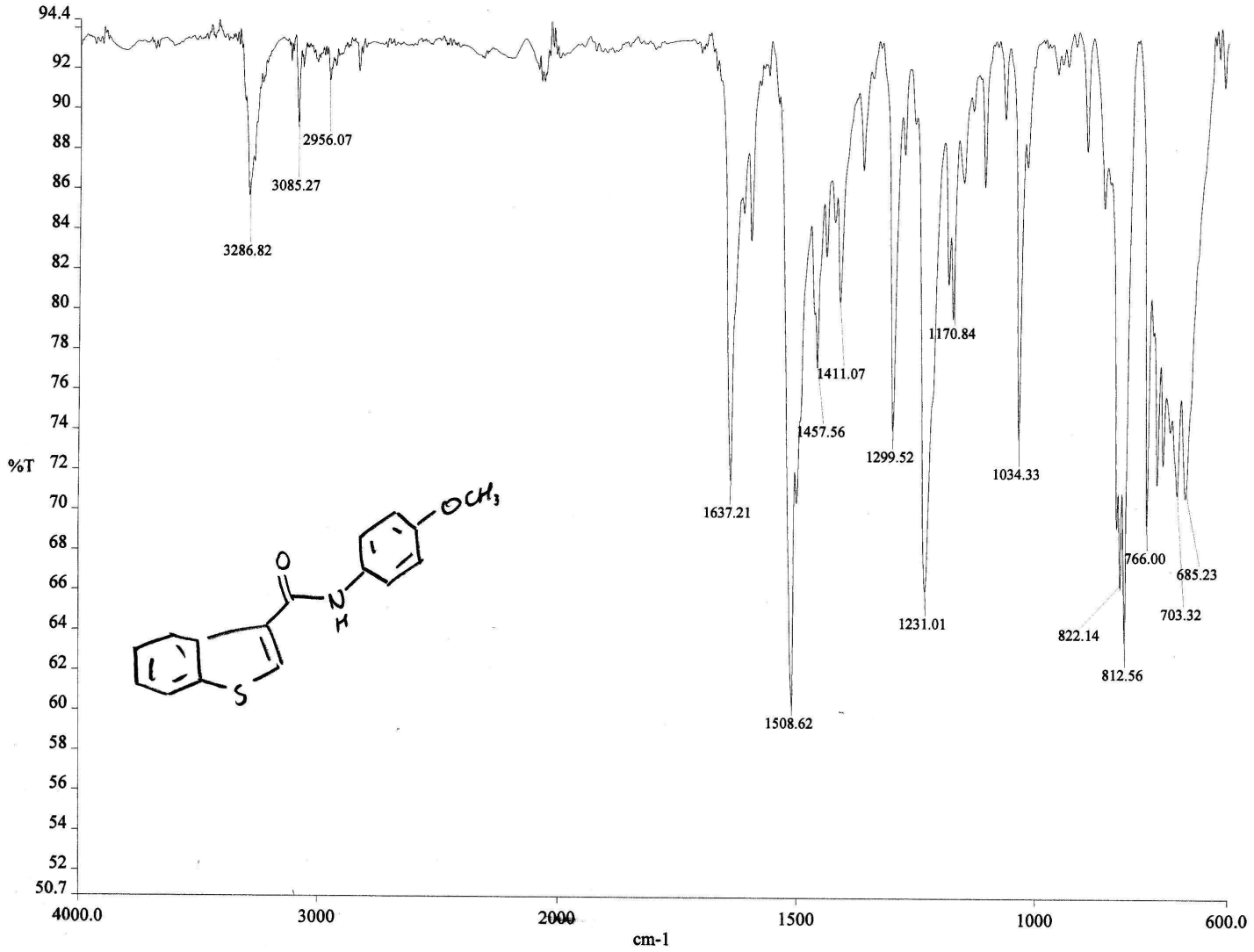


c:\pel_data\spectra\dyeison\001-da-034-01.sp - 001-DA-034-01

m.p. 179-182

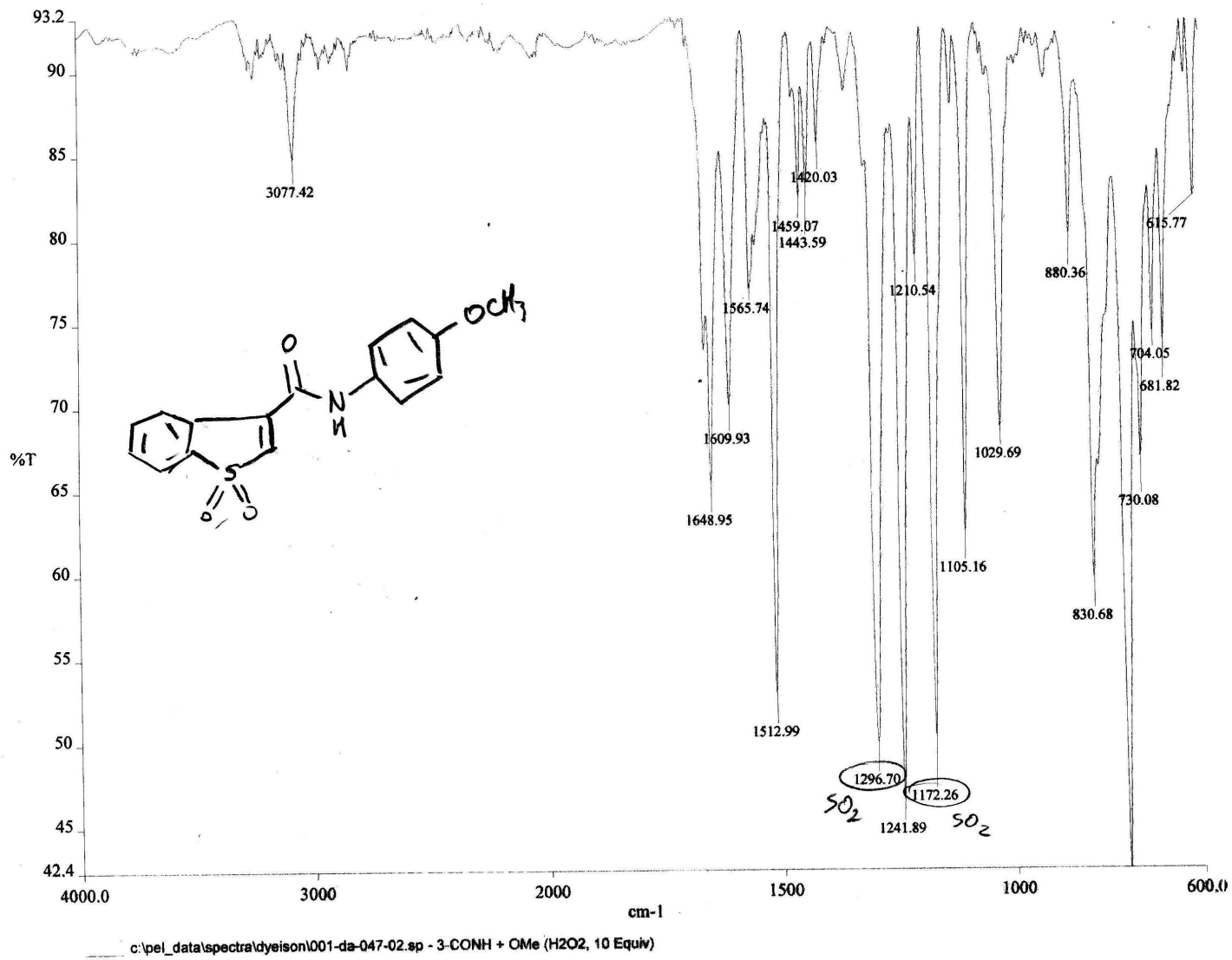

92.5
90
85
80
75
70
65
60
55
50
45
41.9
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3356.04
3074.93
1671.72
1597.48
1539.34
1488.04
1439.80
1288.19
SO2
1242.88
1205.26
1175.46
SO2
1137.06
1108.79
876.82
834.30
757.90
693.83
O
N
H
S
O
O
c:\pel_data\spectra\dyeison\001-da-047-01.sp - 3-CONH + H (H2O2, 10 Equiv)

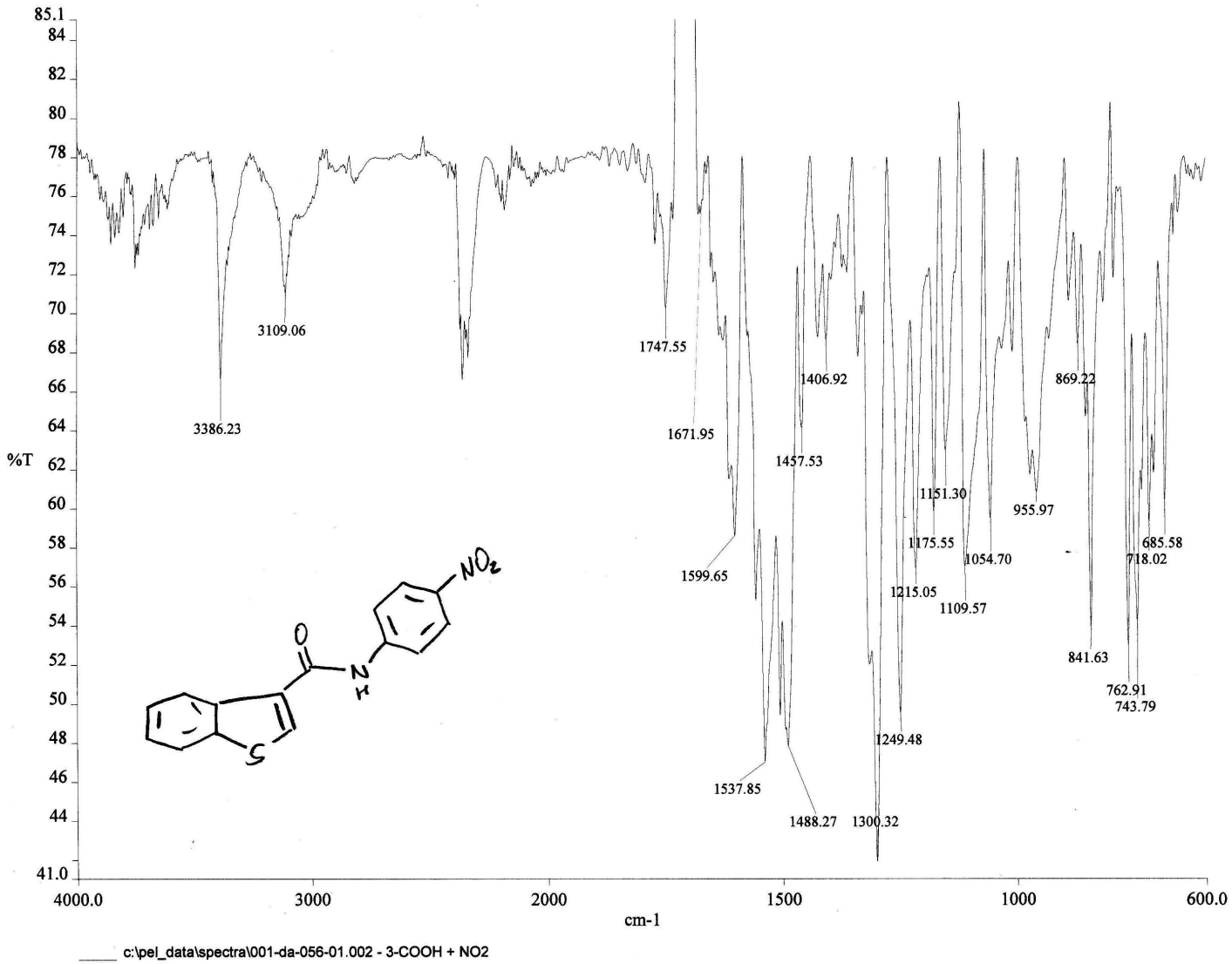
mp 188 - 190 °C

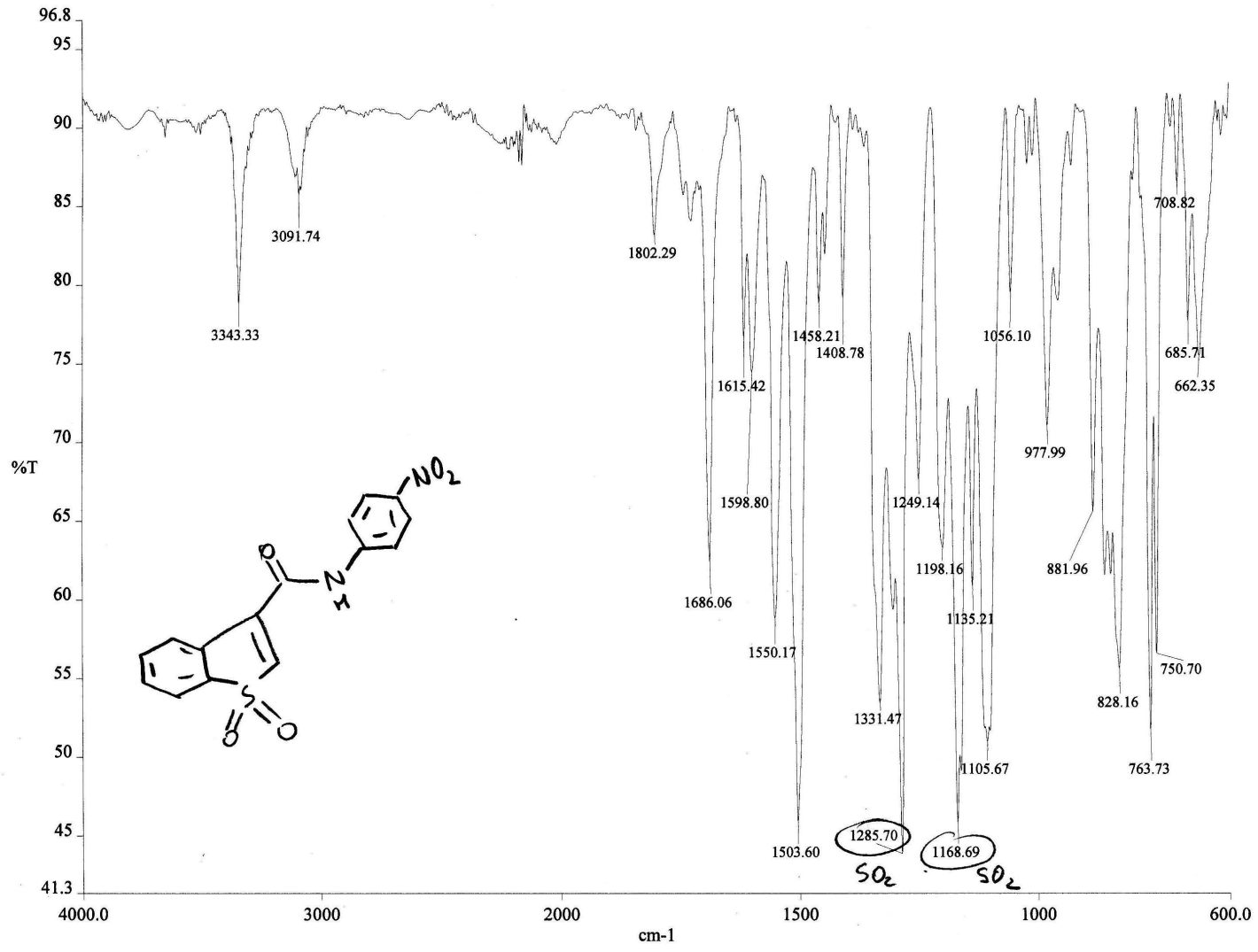


c:\pel_data\spectra\dyeison\001-da-032-01.sp - 001-DA-032-01

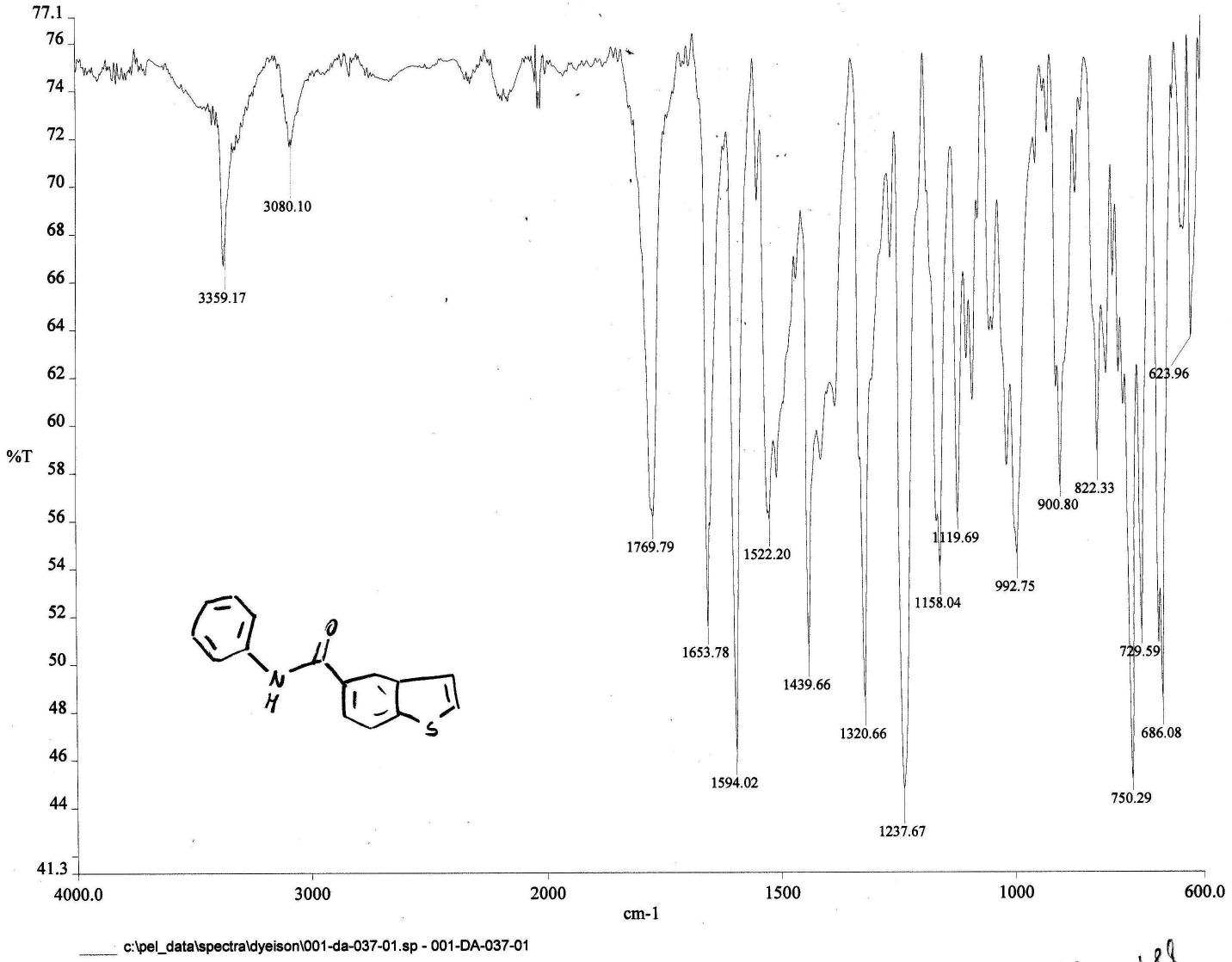
m.p. 225 - 226


93.2
90
85
80
75
70
65
60
55
50
45
42.4
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3077.42
1648.95
1609.93
1565.74
1512.99
1459.07
1443.59
1420.03
1296.70
1241.89
1210.54
1172.26
1105.16
1029.69
880.36
830.68
730.08
704.05
681.82
615.77
OCH3
O
N
H
S
O
O
SO2
SO2
c:\pel_data\spectra\dyeison\001-da-047-02.sp - 3-CONH + OMe (H2O2, 10 Equiv)

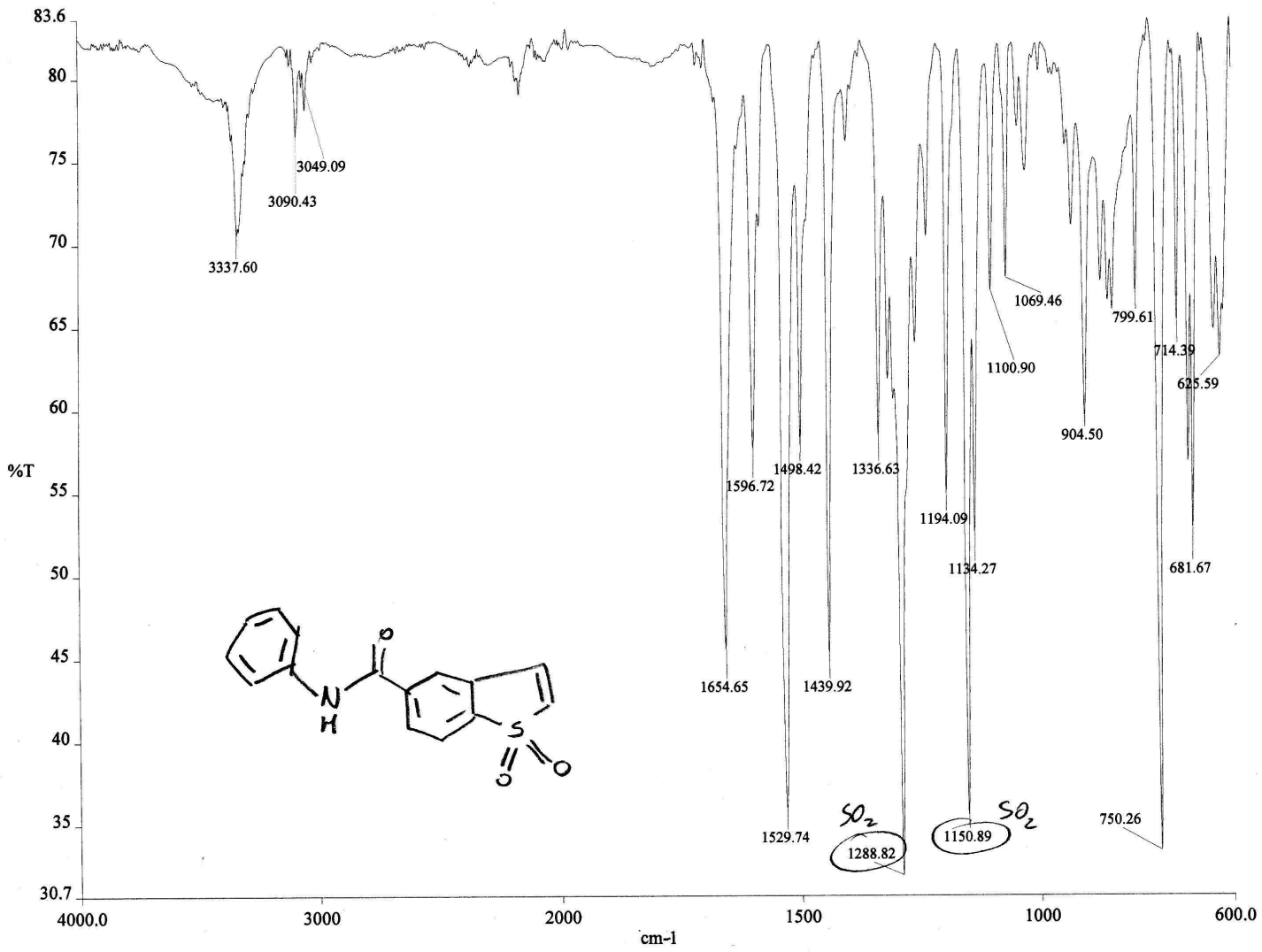

85.1
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
44
41.0
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3386.23
3109.06
1747.55
1671.95
1599.65
1537.85
1488.27
1457.53
1406.92
1300.32
1249.48
1215.05
1175.55
1151.30
1109.57
1054.70
955.97
869.22
841.63
762.91
743.79
718.02
685.58
O
N
H
NO2
S
c:\pel_data\spectra\001-da-056-01.002 - 3-COOH + NO2


96.8
95
90
85
80
75
70
65
60
55
50
45
41.3
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3343.33
3091.74
1802.29
1615.42
1598.80
1686.06
1550.17
1503.60
1458.21
1408.78
1331.47
1285.70
1249.14
1198.16
1168.69
1135.21
1105.67
1056.10
977.99
881.96
828.16
763.73
750.70
708.82
685.71
662.35
NO2
O
N
H
S
O
O
SO2
SO2
c:\pel_data\spectra\dyeison\001-da-057-01.sp - 3-COOH + NO2 (H2O2)

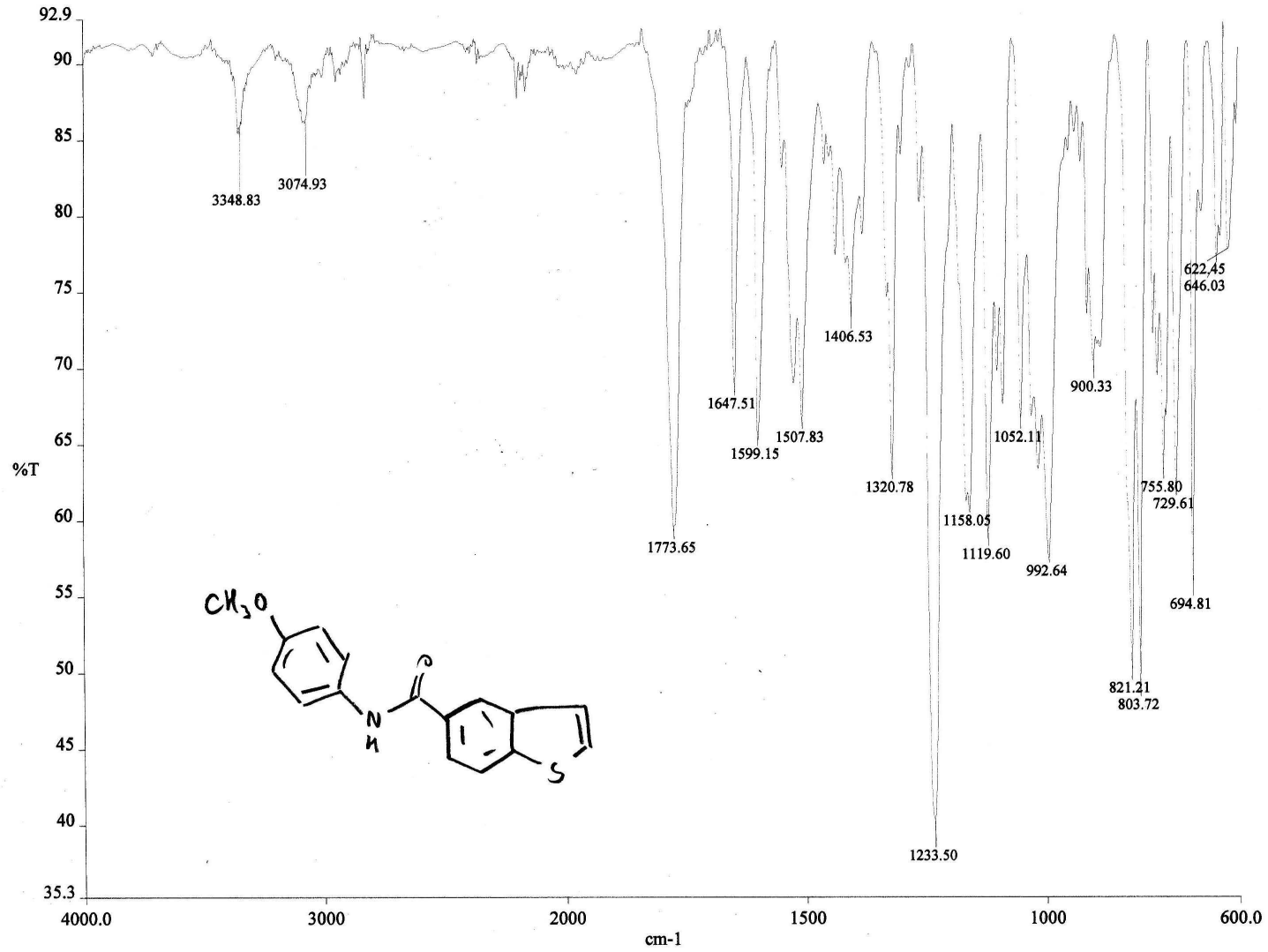
~ p. 263 - 264


77.1
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
44
41.3
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3359.17
3080.10
1769.79
1653.78
1594.02
1522.20
1439.66
1320.66
1237.67
1158.04
1119.69
992.75
900.80
822.33
729.59
686.08
623.96
750.29
O
N
H
S
c:\pel_data\spectra\dyeison\001-da-037-01.sp - 001-DA-037-01

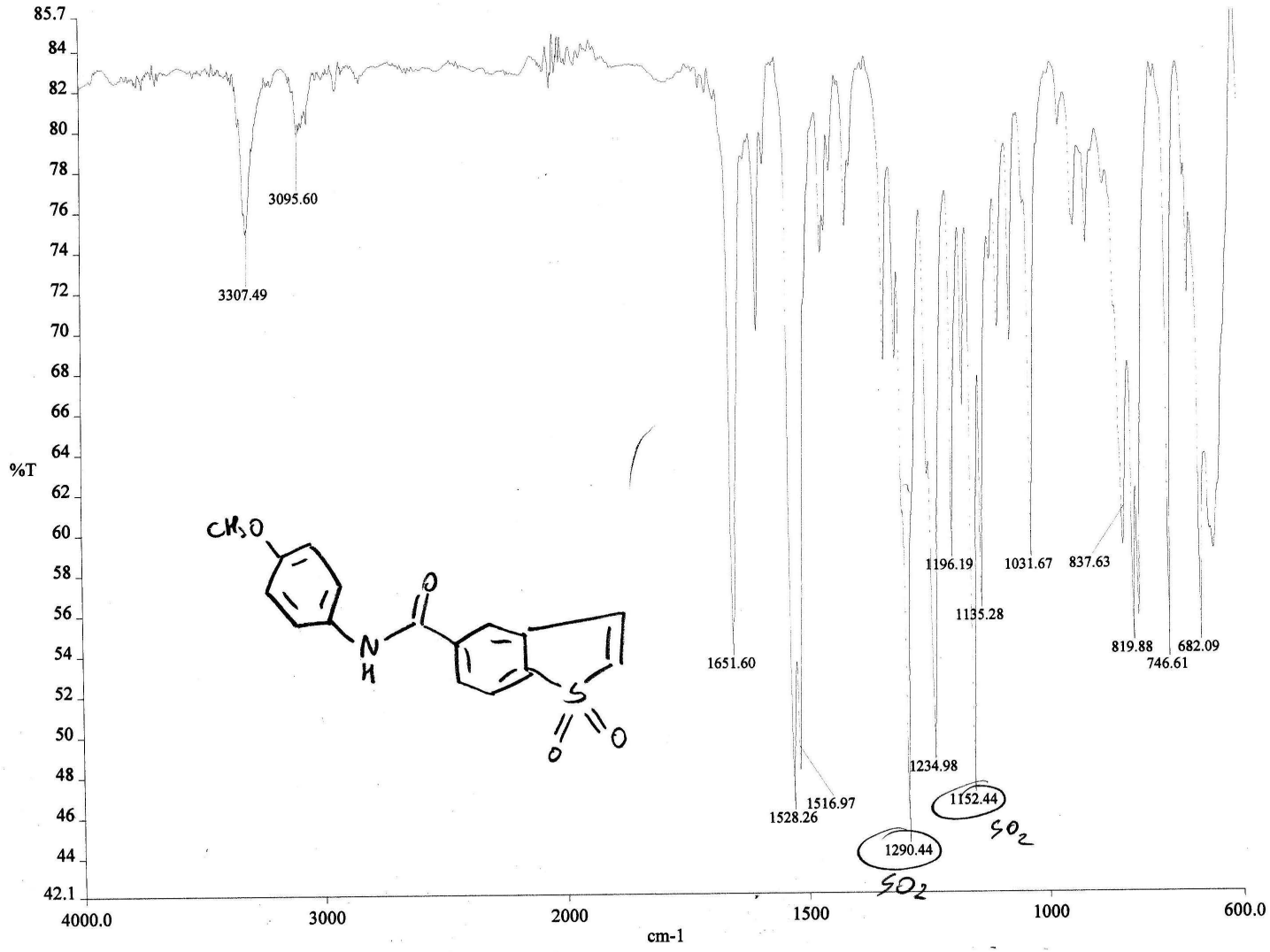
~ - N 187 - 189


83.6
80
75
70
65
60
55
50
45
40
35
30.7
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3337.60
3090.43
3049.09
1654.65
1596.72
1529.74
1498.42
1439.92
1336.63
1288.82
1194.09
1150.89
1134.27
1100.90
1069.46
904.50
799.61
750.26
714.39
681.67
625.59
SO2
SO2
O
N
H
S
O
O
c:\pel_data\spectra\dyeison\001-da-043-01.sp - 001-DA-043-01 (5-CONH + H, H2O2-oxidation, 10 Equiv)

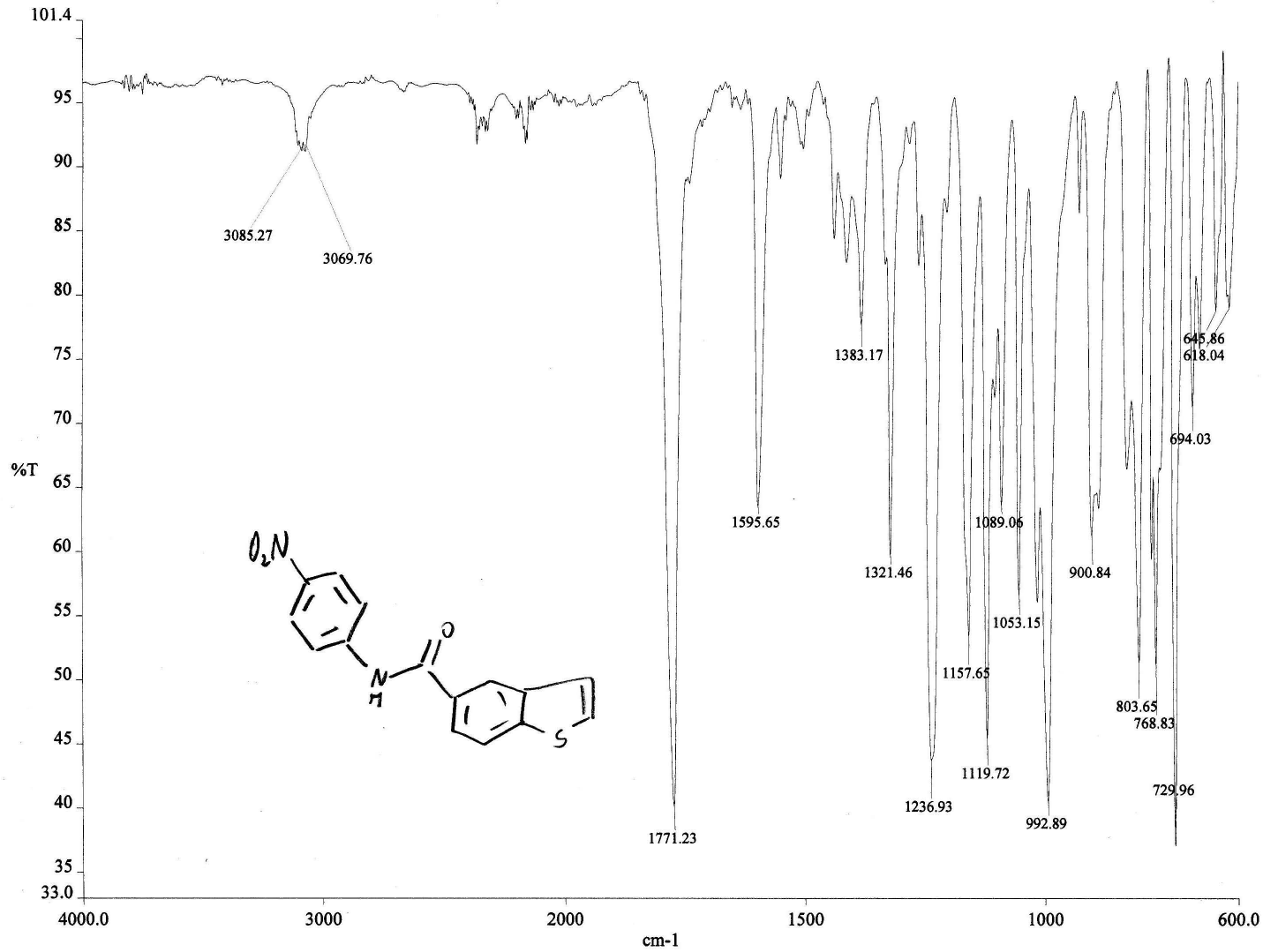
m.p. 247 - 249°C


92.9
90
85
80
75
70
65
60
55
50
45
40
35.3
%T
3348.83
3074.93
1773.65
1647.51
1599.15
1507.83
1406.53
1320.78
1233.50
1158.05
1119.60
1052.11
992.64
900.33
821.21
803.72
755.80
729.61
694.81
622,45
646.03
4000.0
3000
2000
1500
1000
600.0
cm-1
CH3O
N
H
S
c:\pel_data\spectra\dyeison\001-da-035-01.sp - 001-DA-035-01

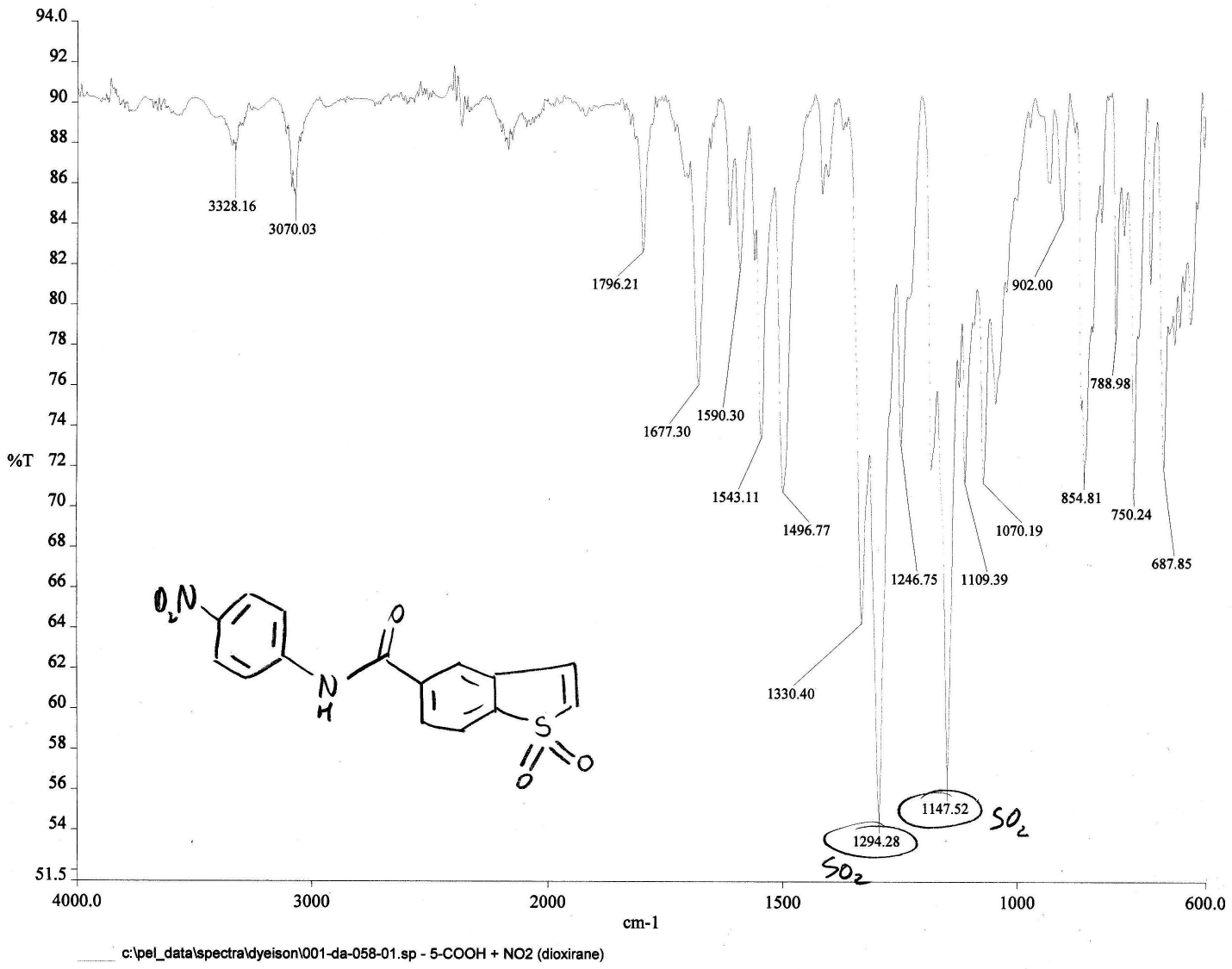
m.p. 211 - 213

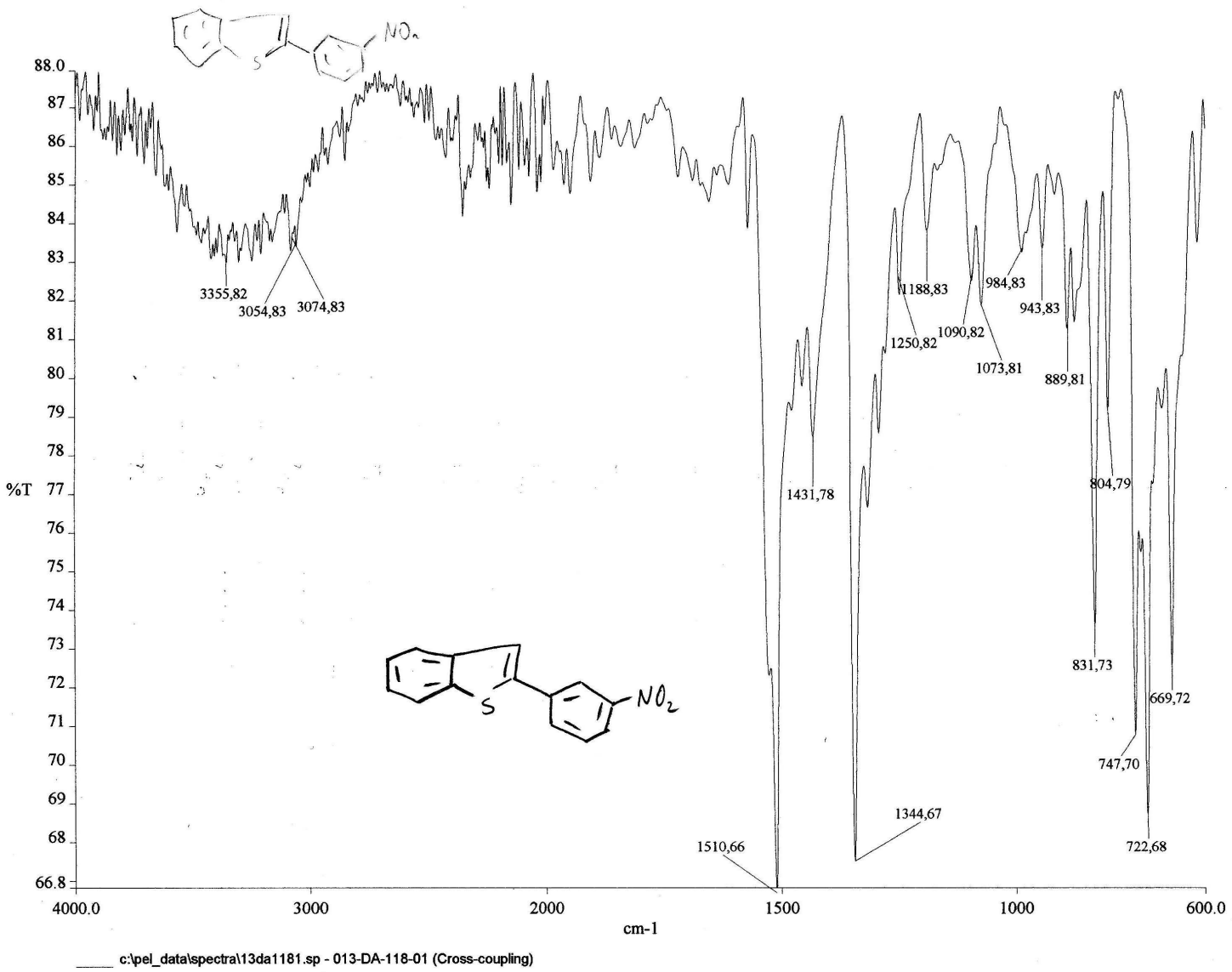

85.7
84
82
80
78
76
74
72
70
68
66
64
%T
62
60
58
56
54
52
50
48
46
44
42.1
4000.0
3000
2000
1500
1000
600.0
cm-1
3095.60
3307.49
1651.60
1528.26
1516.97
1290.44
SO2
1234.98
1196.19
1152.44
SO2
1135.28
1031.67
837.63
819.88
746.61
682.09
CH3O
N
H
O
S
O
O
c:\pel_data\spectra\dyeison\001-da-044-01.sp - 001-DA-044-01 (5-CONH + OMe, H2O2-oxidation, 10 Equiv)

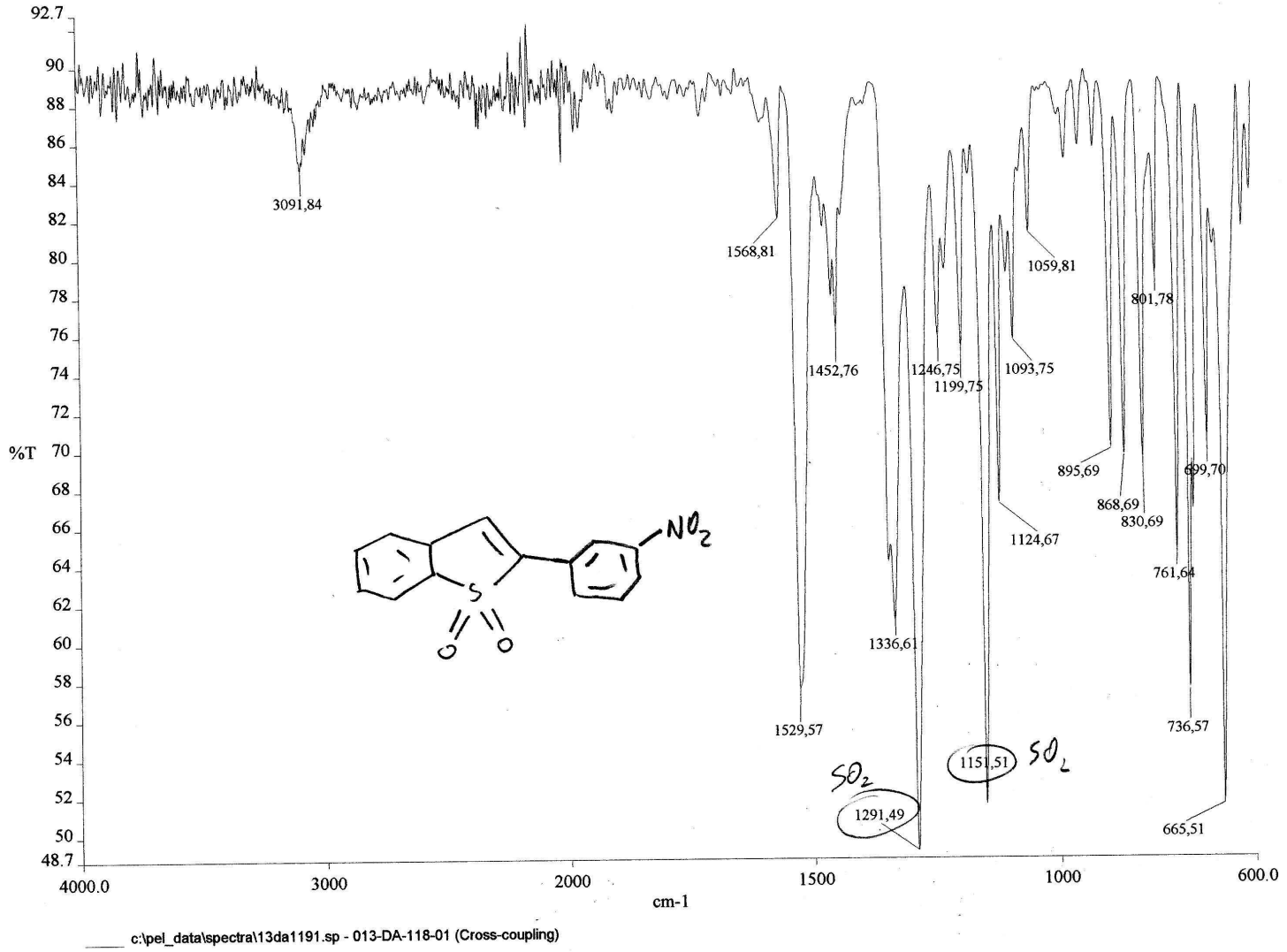
m.p. 251 - 253

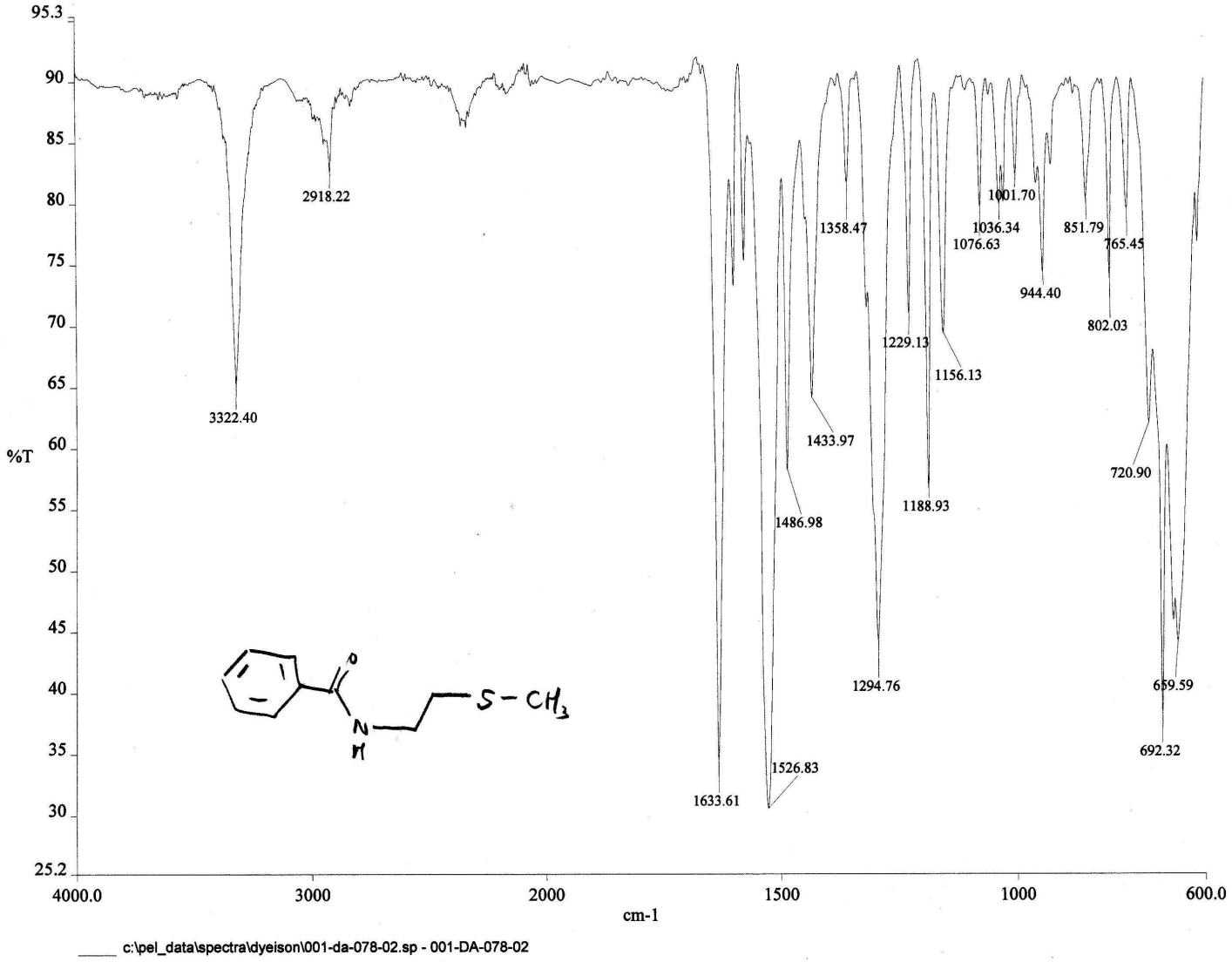

101.4
95
90
85
80
75
70
65
60
55
50
45
40
35
33.0
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3085.27
3069.76
1771.23
1595.65
1383.17
1321.46
1236.93
1157.65
1119.72
1089.06
1053.15
992.89
900.84
803.65
768.83
729.96
694.03
645.86
618.04
O2N
N
H
O
S
c:\pel_data\spectra\dyeison\001-da-036-01.sp - 001-DA-036-01

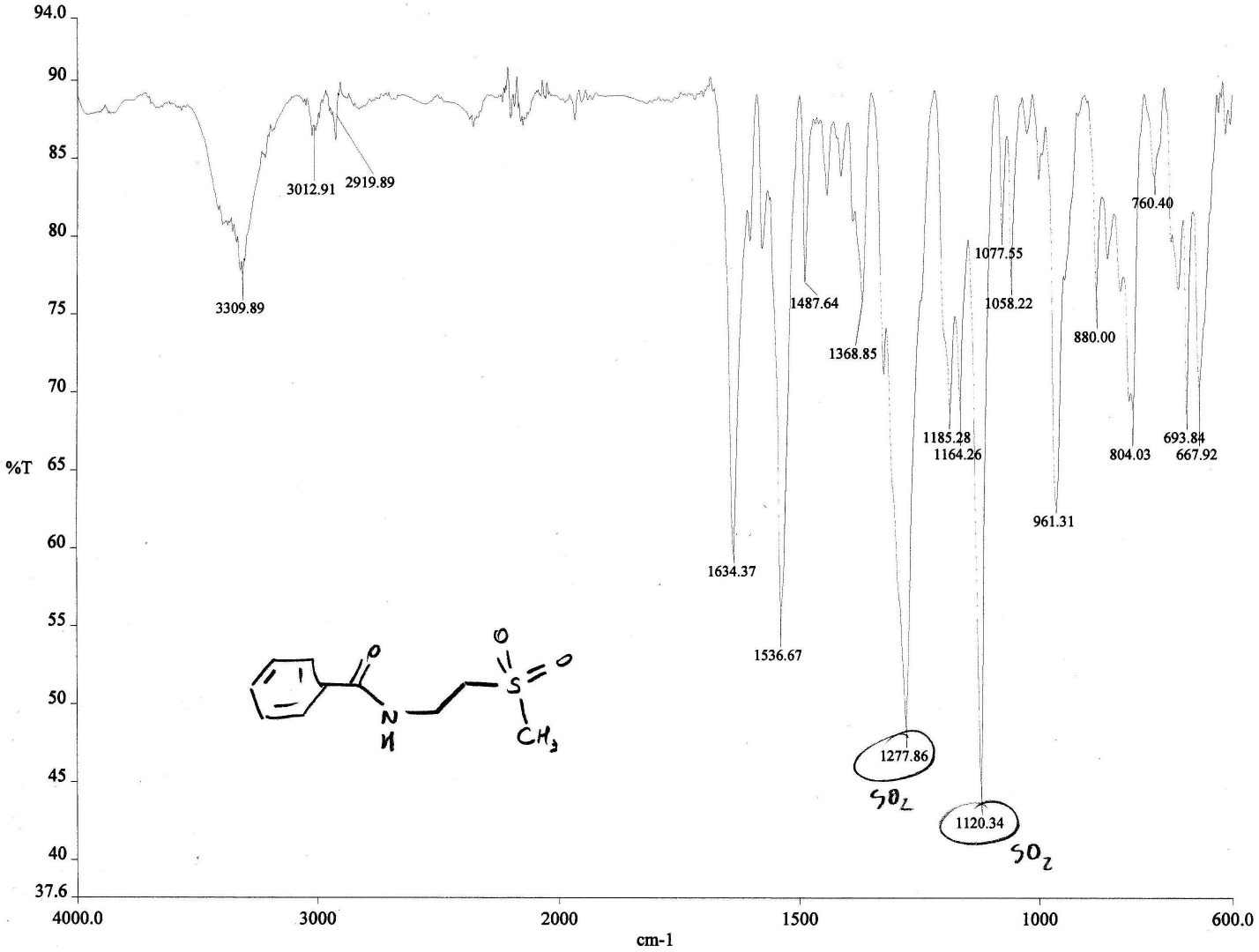
m.p 168 – 170

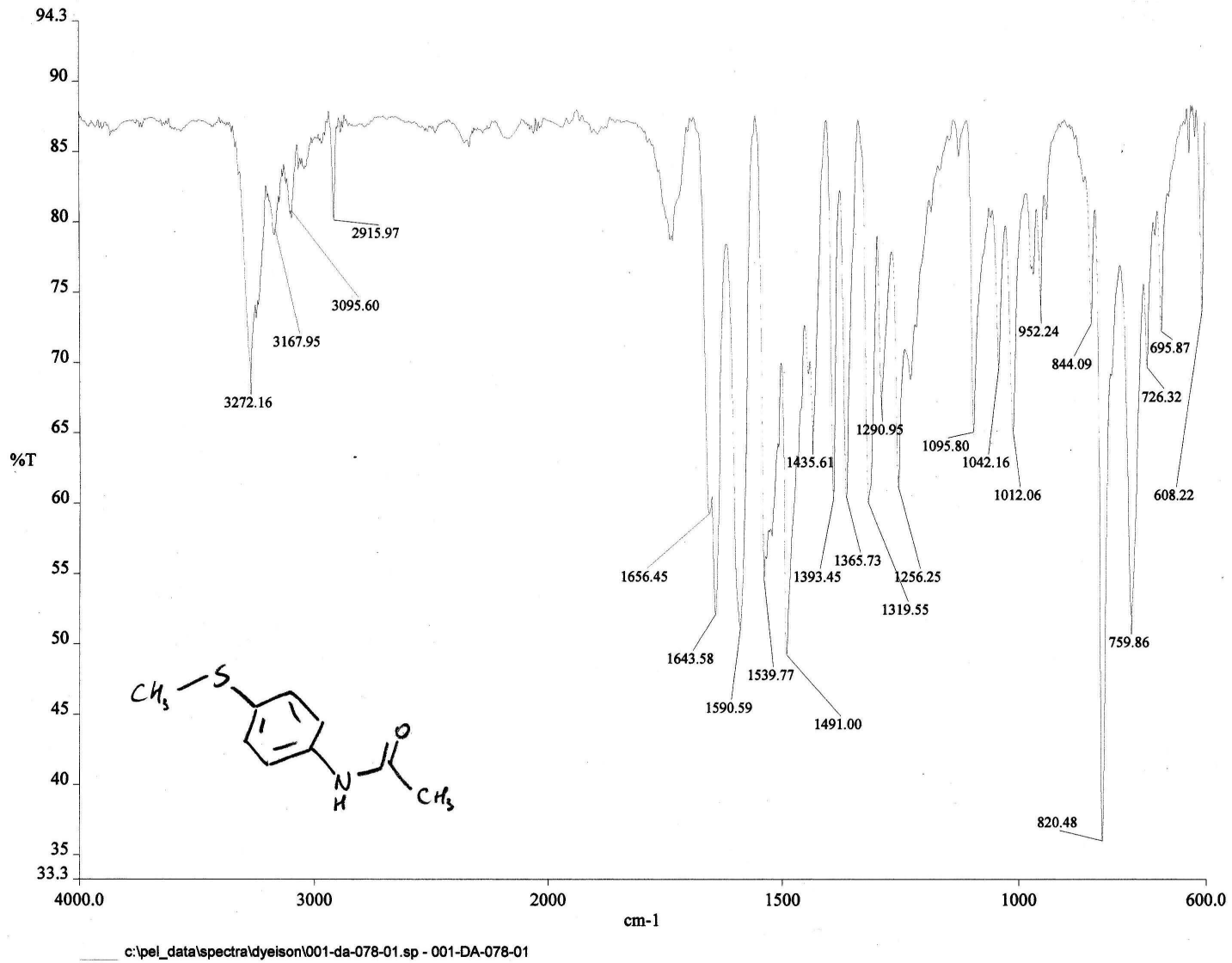

94.0
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
51.5
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3328.16
3070.03
1796.21
1677.30
1590.30
1543.11
1496.77
1330.40
1294.28
1246.75
1147.52
1109.39
1070.19
902.00
854.81
788.98
750.24
687.85
SO2
SO2
c:\pel_data\spectra\dyeison\001-da-058-01.sp - 5-COOH + NO2 (dioxirane)

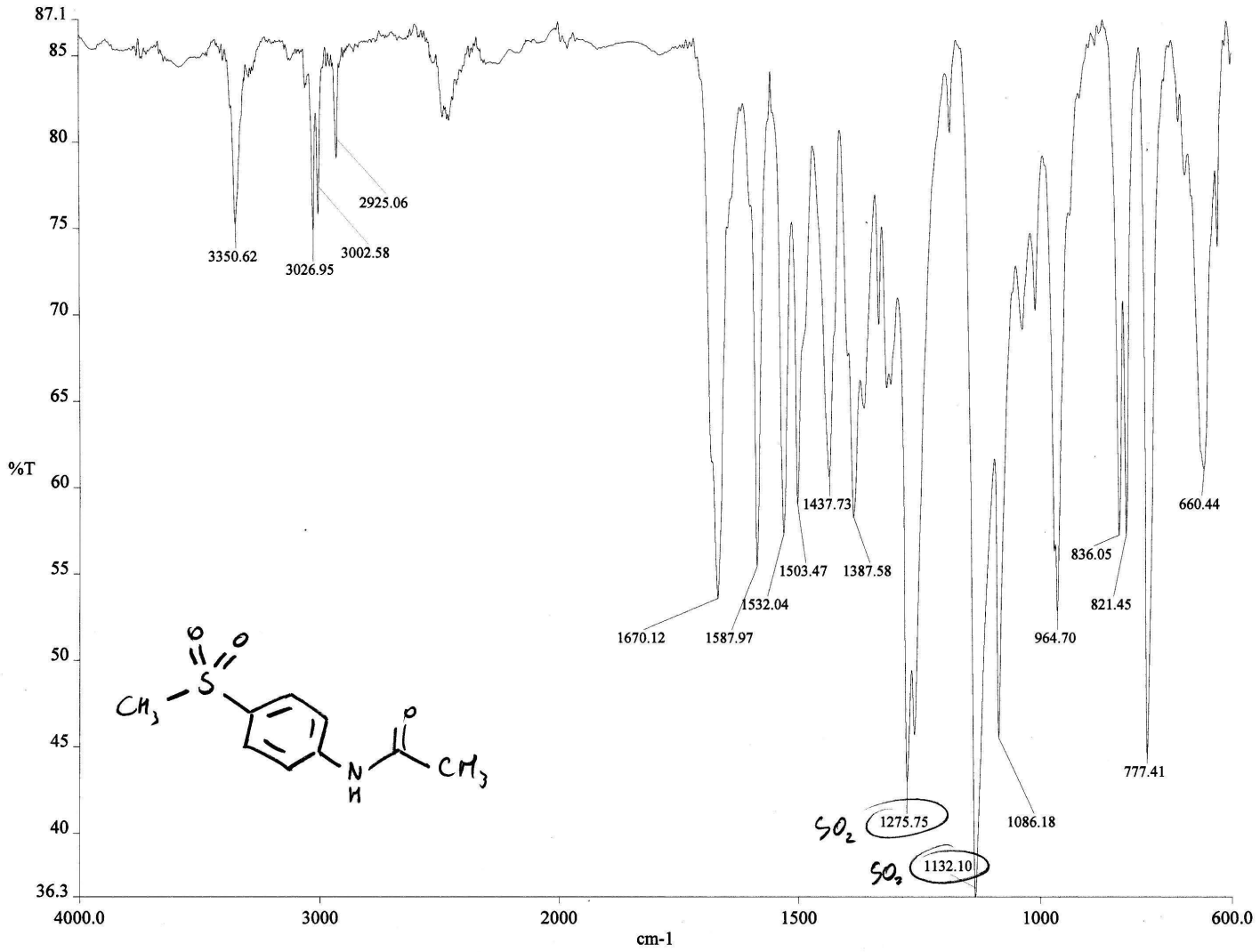

88.0
87
86
85
84
83
82
81
80
79
78
%T 77
76
75
74
73
72
71
70
69
68
66.8
4000.0
3000
2000
1500
1000
600.0
cm-1
3355,82
3054,83
3074,83
1510,66
1431,78
1344,67
1250,82
1188,83
1090,82
1073,81
984,83
943,83
889,81
831,73
804,79
747,70
722,68
669,72
NO2
S
c:\pel_data\spectra\13da1181.sp - 013-DA-118-01 (Cross-coupling)


92.7
90
88
86
84
82
80
78
76
74
72
%T 70
68
66
64
62
60
58
56
54
52
50
48.7
4000.0
3000
2000
1500
1000
600.0
cm-1
3091,84
1568,81
1452,76
1246,75
1199,75
1093,75
1059,81
801,78
895,69
868,69
830,69
699,70
1124,67
761,64
1336,61
1529,57
736,57
1151,51
1291,49
665,51
NO2
S
O
O
SO2
SO2
c:\pel_data\spectra\13da1191.sp - 013-DA-118-01 (Cross-coupling)


95.3
90
85
80
75
70
65
60
55
50
45
40
35
30
25.2
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3322.40
2918.22
1633.61
1526.83
1486.98
1433.97
1358.47
1294.76
1229.13
1188.93
1156.13
1076.63
1036.34
1001.70
944.40
851.79
802.03
765.45
720.90
692.32
659.59
O
N
H
S - CH3
c:\pel_data\spectra\dyeison\001-da-078-02.sp - 001-DA-078-02


94.0
90
85
80
75
70
65
60
55
50
45
40
37.6
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
3309.89
3012.91
2919.89
1634.37
1536.67
1487.64
1368.85
1277.86
1185.28
1164.26
1120.34
1077.55
1058.22
961.31
880.00
804.03
760.40
693.84
667.92
SO2
SO2
c:\pel_data\spectra\001-da-079-01.002 - 001-DA-079-01


94.3
90
85
80
75
70
65
60
55
50
45
40
35
33.3
%T
4000.0
3000
2000
1500
1000
600.0
cm-1
2915.97
3095.60
3167.95
3272.16
1656.45
1643.58
1590.59
1539.77
1491.00
1435.61
1393.45
1365.73
1290.95
1319.55
1256.25
1095.80
1042.16
1012.06
952.24
844.09
820.48
759.86
726.32
695.87
608.22
CH3
S
N
H
O
CH3
c:\pel_data\spectra\dyeison\001-da-078-01.sp - 001-DA-078-01


87.1
85
80
75
70
65
%T
60
55
50
45
40
36.3
4000.0
3000
2000
1500
1000
600.0
cm-1
3350.62
3026.95
3002.58
2925.06
1670.12
1587.97
1532.04
1503.47
1437.73
1387.58
1275.75
1132.10
1086.18
964.70
836.05
821.45
777.41
660.44
SO2
SO2
O
O
S
CH3
N
H
O
CH3
c:\pel_data\spectra\001-da-083-01.002

m.p. 202-204