

Supporting Information

Copper-Catalyzed One-Pot Synthesis of *N*-Substituted Benzo[*d*]isothiazol-3(2*H*)-ones via *C-S/N-S* Bond Formation

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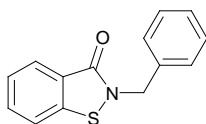
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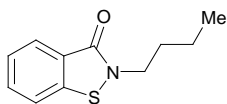
General Information. CuCl (98%) and sulfur powder (97%) were purchased from Rankem and used without further purification. Cu(OAc)₂·1H₂O (98%) was purchased from Merck and used without further purification. 2-Iodobenzoic acid (98%), 2-bromobenzoic acid (97%) and anilines were purchased from Aldrich. Column chromatography was performed with Rankem silica gel (60-120 mesh). The substituted 2-halobenzamides were prepared according to the reported procedures.¹¹ NMR (¹H and ¹³C) spectra were recorded with a Varian 400 spectrometer. Melting points were determined with a Büchi B-545 apparatus and are uncorrected. Elemental analyses were recorded using PerkinElmer CHNS analyzer. X-Ray data were collected on a Bruker SMART APEX equipped with a CCD area detector using Mo K α radiation. The structure was solved by direct method using *SHELLX-97* (Göttingen, Germany).

General Procedure for Preparation of *N*-Substituted Benzo[*d*]isothiazol-3(2*H*)-ones. An oven dried round bottom flask (10 mL) was charged with *N*-substituted 2-halobenzamide (0.5 mmol), CuCl (10 mol %), sulfur powder (1.5 mmol) and K₂CO₃ or Cs₂CO₃ (1.5 mmol) in DMF (1 mL) under nitrogen atmosphere. The resultant mixture was stirred at 75-135 °C under nitrogen balloon for the appropriate time. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluent. The reaction mixture was then cooled to room temperature and diluted with ethyl acetate (10 mL). The organic layer was separated and washed with water (3 x 5 mL) and brine (1 x 5 mL). Drying (Na₂SO₄) and evaporation of the solvent gave a residue that was purified on silica gel column chromatography using hexane and ethyl acetate as eluent.

Characterization Data of 2-Substituted Benzo[*d*]isothiazol-3(2*H*)-ones

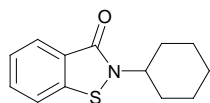


***N*-Benzylbenzo[*d*]isothiazol-3(2*H*)-one² (table 1, entry 1).** Colorless solid; 88% yield; mp = 88-89 °C (lit. mp³ 89 °C); ¹H NMR (CDCl₃, 400 MHz) δ 7.99 (d, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 1H), 7.33-7.23 (m, 6H), 4.96 (s, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 165.5, 140.5, 136.3, 131.9, 128.9, 128.5, 128.4, 126.9, 125.6, 124.5, 120.5, 47.6; FT-IR (KBr): 3078, 3022, 2962, 2923, 1667, 1592, 1445, 1336, 1261, 1243, 1184, 1064, 1029 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₁NOS: C, 69.68; H, 4.59; N, 5.80; S, 13.29, found: C, 69.64; H, 4.56; N, 5.83; S, 13.31.

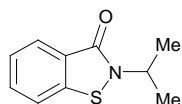


***N*-Butylbenzo[*d*]isothiazol-3(2*H*)-one⁴ (table 2, entry 1).** Yellow oil; 93% yield; ¹H NMR (CDCl₃, 400 MHz) δ 8.01 (d, *J* = 8.0 Hz, 1H), 7.58-7.50 (m, 2H), 7.36 (t, *J* = 7.2 Hz, 1H), 3.88 (t, *J* = 7.2 Hz, 2H), 1.75 (t, *J* = 7.2 Hz, 2H), 1.71-1.33 (m, 2H), 0.95 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 165.5, 140.3, 131.7, 126.8, 125.6, 125.2, 120.4, 43.8, 31.7, 19.9, 13.8. FT-IR (neat): 2962, 2857, 1682, 1490, 1449, 1261, 1091 cm⁻¹. Elemental analysis calcd

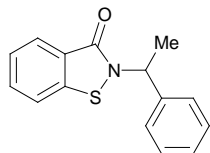
(%) for C₁₁H₁₃NOS: C, 63.74; H, 6.32; N, 6.76; S, 15.47, found: C, 63.71; H, 6.35; N, 6.73; S, 15.50.



N-Cyclohexylbenzo[d]isothiazol-3(2H)-one³ (table 2, entry 2). Colorless solid; 95% yield; mp = 86-87 °C (lit.³ mp 87-88 °C); ¹H NMR (CDCl₃, 400 MHz) δ 8.01 (d, *J* = 8.0 Hz, 1H), 7.54-7.50 (m, 1H), 7.36-7.31 (m, 2H), 4.59-4.53 (m, 1H), 2.01 (d, *J* = 10.4 Hz, 1H), 1.85 (d, *J* = 12.8 Hz, 2H), 1.71-1.67 (m, 1H), 1.55-1.38 (m, 5H), 1.21-1.12 (m, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.9, 140.4, 131.5, 126.6, 125.6, 125.4, 120.5, 53.3, 33.06, 25.7, 25.4; FT-IR (KBr): 2929, 2854, 1651, 1448, 1332, 1305, 1262, 1240, 1210, 1191, 1149, 1062 cm⁻¹. Elemental analysis calcd (%) C₁₃H₁₅NOS: C, 66.92; H, 6.48; N, 6.00; S, 13.74, found: C, 66.95; H, 6.46; N, 6.03; S, 13.76.

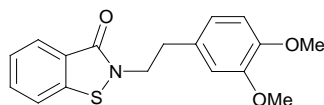


N-Isopropylbenzo[d]isothiazol-3(2H)-one⁶ (table 2, entry 3). Yellow oil; 93% yield; ¹H NMR (CDCl₃, 400 MHz) δ 8.01 (d, *J* = 7.6 Hz, 1H), 7.57-7.54 (m, 1H), 7.36 (t, *J* = 6.4 Hz, 2H), 5.01-4.95 (m, 1H), 1.40 (d, *J* = 6.4 Hz, 6H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.9, 140.1, 131.5, 126.4, 125.5, 125.4, 120.5, 46.1, 22.2. FT-IR (neat): 2935, 2851, 1641, 1447, 1332, 1239, 1192, 1034 cm⁻¹. Elemental analysis calcd (%) for C₁₀H₁₁NOS: C, 62.15; H, 5.74; N, 7.25; S, 16.59, found: C, 62.18; H, 5.76; N, 7.24; S, 16.57.

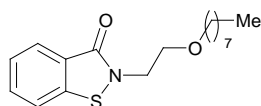


N-(1-Phenylethyl)benzo[d]isothiazol-3(2H)-one (table 2, entry 4). Yellow oil; 87%, yield; ¹H NMR (CDCl₃, 400 MHz) δ 7.83-7.81 (m, 1H), 7.40-7.25 (m, 7H), 7.14-7.05 (m, 1H), 5.33-5.26 (m, 1H), 1.63 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 165.1, 140.6, 131.7, 128.8, 128.6, 128.3, 127.4, 126.7, 125.5, 120.5, 52.3, 19.3. FT-IR (neat): 2935, 2851, 1640, 1440, 1331,

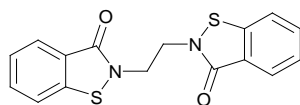
1229, 1192, 1092, 1034 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{15}\text{H}_{13}\text{NOS}$: C, 70.56; H, 5.13; N, 5.49; S, 12.56, found: C, 70.53; H, 5.15; N, 5.53; S, 12.52.



***N*-(3,4-Dimethoxyphenethyl)benzo[*d*]isothiazol-3(2*H*)-one (table 2, entry 5).** Yellow solid; 83% yield; mp 105-107 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 7.95 (d, $J = 7.6$ Hz, 1H), 7.51-7.47 (m, 1H), 7.43 (d, $J = 8.0$ Hz, 1H), 7.31 (t, $J = 7.2$ Hz, 1H), 6.71-6.65 (m, 3H), 4.04 (t, $J = 6.8$ Hz, 2H), 3.75 (s, 3H), 3.70 (s, 3H), 2.94 (t, $J = 6.8$ Hz, 2H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 165.4, 149.1, 147.9, 140.3, 131.8, 130.3, 126.7, 125.5, 124.6, 120.9, 120.2, 112.1, 111.4, 55.9, 55.8, 45.2, 35.2. FT-IR (KBr): 2935, 2067, 1639, 1518, 1446, 1337, 1263, 1230, 1185, 1143, 1026 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{17}\text{H}_{17}\text{NO}_3\text{S}$: C, 67.74; H, 5.43; N, 4.44; S, 10.17, found: C, 67.71; H, 5.45; N, 4.46; S, 10.15.

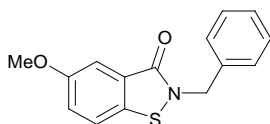


***N*-(2-(Octyloxy)ethyl)benzo[*d*]isothiazol-3(2*H*)-one (table 2, entry 6).** Yellow oil; 90% yield ^1H NMR (CDCl_3 , 400 MHz) δ 7.97 (d, $J = 8.0$ Hz, 1H), 7.52 (t, $J = 8.0$ Hz, 1H), 7.34-7.30 (m, 1H), 7.03 (t, $J = 7.6$ Hz, 1H), 4.03 (t, $J = 5.2$ Hz, 2H), 3.65 (t, $J = 4.8$ Hz, 2H), 3.43-3.39 (m, 2H), 1.57-1.49 (m, 2H), 1.28-1.19 (m, 10H), 0.83 (t, $J = 6.0$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 165.7, 140.0, 131.9, 126.7, 125.4, 124.3, 120.3, 71.6, 69.4, 44.2, 31.9, 29.8, 29.6, 26.4, 26.3, 22.8, 14.3. FT-IR (neat): 2935, 2851, 1641, 1467, 1331, 1192, 1034 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{17}\text{H}_{25}\text{NO}_2\text{S}$: C, 66.13; H, 7.79; N, 4.33; S, 9.91, found: C, 66.17; H, 7.76; N, 4.34; S, 9.87.

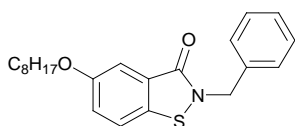


***N,N'*-(Ethane-1,2-diyl)dibenzo[*d*]isothiazol-3(2*H*)-one (table 2, entry 7).** Colorless solid; 81% yield; mp 165-167 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.01 (d, $J = 8.0$ Hz, 2H), 7.58 (t, $J = 7.2$ Hz, 2H), 7.46 (d, $J = 8.0$ Hz, 2H), 7.38 (t, $J = 7.2$ Hz, 2H), 4.21 (s, 4H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 165.8, 140.9, 132.2, 126.9, 126.8, 125.8, 120.6, 43.1. FT-IR (KBr): 2964, 2924, 2853,

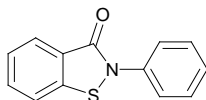
1644, 1505, 1447, 1330, 1304, 1261, 1097, 1065, 1019 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$: C, 58.52; H, 3.68; N, 8.53; S, 19.53, found: C, 58.55; H, 3.64; N, 8.50; S, 19.54.



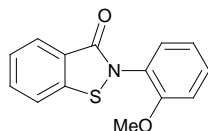
***N*-Benzyl-5-methoxybenzo[*d*]isothiazol-3(2*H*)-one (table 2, entry 8).** Colorless solid, 67% yield; m.p. 134-135 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.38 (s, 1H), 7.83-7.81(m, 1H), 7.33-7.31 (m, 5H), 7.24-7.21 (m, 1H), 5.01 (s, 2H), 3.8 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 163.9, 140.5, 139.9, 136.0, 135.8, 129.1, 128.6, 126.6, 122.2, 55.7, 47.9. FT-IR (KBr): 2917, 2840, 1651, 1591, 1576, 1408, 1266 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{15}\text{H}_{13}\text{NO}_2\text{S}$: C, 66.40; H, 4.83; N, 5.16; 11.82, found C, 66.44; H, 4.78; N, 5.19, S, 11.83.



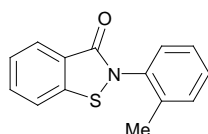
***N*-Benzyl-5-(octyloxy)benzo[*d*]isothiazol-3(2*H*)-one (table 2, entry 9).** Yellow liquid; 72% yield; ^1H NMR (400 MHz, CDCl_3) δ 8.38 (s, 1H), 7.83-7.81(m, 1H), 7.33-7.31 (m, 5H), 7.24-7.21 (m, 1H), 5.01 (s, 2H), 4.03 (t, $J = 5.2$ Hz, 2H), 1.57-1.49 (m, 3H), 1.28-1.19 (m, 10H), 0.83(t, $J = 6.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 163.9, 140.5, 139.9, 136.0, 135.8, 129.1, 128.6, 126.6, 122.2, 69.4, 55.7, 47.9, 31.9, 29.8, 29.6, 26.4, 26.3, 22.8, 14.3. FT-IR (KBr): 2917, 2840, 1651, 1591, 1576, 1408, 1376, 1299, 1266, 1021 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{22}\text{H}_{27}\text{NO}_2\text{S}$: C, 71.51; H, 7.36; N, 3.79; S, 8.68, found C, 71.54; H, 7.33; N, 3.81; S, 8.71.



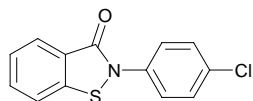
***N*-Phenylbenzo[*d*]isothiazol-3(2*H*)-one⁶ (table 3, entry 1).** Colorless solid; 90% yield; mp 140-142 °C (lit.⁷ mp 141.5-142.5 °C); ^1H NMR (400 MHz, CDCl_3) δ 8.12 (d, $J = 8.0$ Hz, 1H), 7.72-7.65 (m, 2H), 7.59 (d, $J = 8.0$ Hz, 2H), 7.49-7.42 (m, 2H), 7.34 (t, $J = 7.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 164.3, 139.8, 136.0, 132.8, 132.7, 129.7, 127.5, 126.2, 125.8, 120.4. FT-IR (KBr): 2963, 2923, 2840, 1633, 1412, 1261, 1095, 1022 cm^{-1} . Elemental analysis calcd (%) for $\text{C}_{13}\text{H}_9\text{NOS}$: C, 68.70; H, 3.99; N, 6.16; S, 14.11, found C, 68.72; H, 3.97; N, 6.18; S, 14.14.



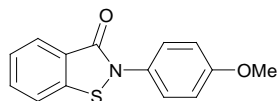
***N*-(2-Methoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one (table 3, entry 2).** Colorless solid; 84% yield; mp 105-107 °C; ¹H NMR (CDCl₃, 400 MHz) δ 8.09 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 6.8 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.42-7.37 (m, 3H), 7.03 (t, *J* = 8.0 Hz, 2H), 3.81 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 165.1, 156.9, 141.6, 132.1, 130.7, 130.3, 127.2, 125.4, 124.6, 123.9, 120.9, 112.5, 56.0; FT-IR (KBr): 2923, 2923, 2851, 1663, 1593, 1497, 1445, 1333, 1261, 1094, 1021 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₁NO₂S: C, 65.35; H, 4.31; N, 5.44; S, 12.46, found: C, 65.38; H, 4.29; N, 5.47; S, 12.44.



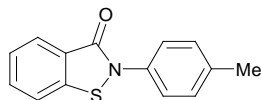
***N*-(2-Methylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (table 3, entry 3).** Colorless solid; mp 122-124 °C (lit. mp² 122-123 °C); ¹H NMR (CDCl₃, 400 MHz) δ 8.07 (d, *J* = 8.0 Hz, 1H), 7.63 (t, *J* = 8.0 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.49 (s, 1H), 7.46-7.38 (m, 2H), 7.34 (t, *J* = 8.0 Hz, 1H), 7.11 (d, *J* = 7.6 Hz, 1H), 2.38 (s, 3H); ¹³C NMR (CDCl₃, 100MHz) δ 164.4, 140.2, 139.6, 137.3, 132.5, 129.4, 128.2, 127.4, 125.9, 125.6, 125.1, 121.9, 120.3, 21.6. FT-IR (KBr): 2963, 2917, 1644, 1504, 1331, 1016 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₁NOS: C, 69.68; H, 4.59; N, 5.80; S, 13.29, found: C, 69.64; H, 4.61; N, 5.78; S, 13.27.



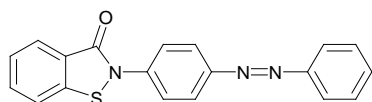
***N*-(4-Chlorophenyl)benzo[*d*]isothiazol-3(2*H*)-one⁷ (table 3, entry 5).** Colorless solid; 85% yield; m.p. 127-128 °C (lit.⁷ mp 128-129 °C); ¹H NMR (CDCl₃, 400MHz) δ: 8.08 (d, *J* = 8.0 Hz, 1H), 7.65-7.63 (m, 2H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.43-7.39 (m, 3H); ¹³C NMR (CDCl₃, 400MHz) δ 163.1, 138.6, 134.8, 131.6, 128.5, 128.1, 126.2, 124.9, 124.6, 123.6, 119.1; FT-IR (KBr): 2962, 2926, 2851, 1661, 1591, 1490, 1444, 1325, 1303, 1261, 1122, 1028 cm⁻¹. Elemental analysis calcd (%) for C₁₃H₈ClNOS: C, 59.66; H, 3.08; N, 5.35; S, 12.25, found C, 59.64; H, 3.10; N, 5.33; S, 12.27.



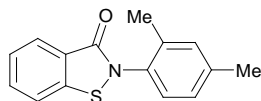
***N*-(4-Methoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one⁷ (table 3, entry 6):** Colorless solid; 89% yield; m.p. 146-147 °C (lit.⁷ mp 147-149 °C); ¹H NMR (CDCl₃, 400 MHz) δ 8.08 (d, *J* = 8.0 Hz, 1H), 7.62 (t, *J* = 6.8 Hz, 1H), 7.55-7.51 (m, 3H), 7.41 (t, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 6.8 Hz, 2H), 3.8 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.5, 158.9, 140.2, 132.3, 129.8, 127.3, 127.0, 125.9, 124.8, 120.2, 114.7, 55.7. FT-IR (KBr): 2923, 2854, 1663, 1591, 1490, 1445, 1331, 1267, 1095 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₁NO₂S: C, 65.35; H, 4.31; N, 5.44; S, 12.40, found C, 65.36; H, 4.33; N, 5.46; S, 12.42.



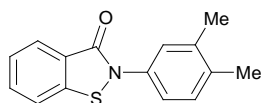
***N*-p-Tolylbenzo[*d*]isothiazol-3(2*H*)-one⁷ (table 3, entry 7).** Colorless solid; 93% yield; mp 135-136 °C (lit.⁷ mp 136-137 °C); ¹H NMR (CDCl₃, 400 MHz) δ 8.02 (d, *J* = 8.0 Hz, 1H), 7.56-7.54 (m, 1H), 7.47 (d, *J* = 6.4 Hz, 4H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 2.29 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.4, 140.1, 137.4, 134.7, 132.4, 130.1, 127.3, 125.9, 124.9, 120.2, 21.3. FT-IR (KBr): 2923, 2917, 2851, 1644, 1504, 1446, 1331, 1261, 1096, 1019 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₁NOS: C, 69.68; H, 4.59; N, 5.80; S, 13.29, found 69.65; H, 4.56; N, 5.77; S, 13.25.



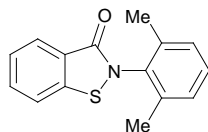
***N*-(4-(Phenyldiazenyl)phenyl)benzo[*d*]isothiazol-3(2*H*)-one (table 3, entry 9).** Orange solid; 63% yield; mp 147-149 °C; ¹H NMR (CDCl₃, 400 MHz) δ 8.10 (d, *J* = 8.0 Hz, 1H), 7.65-7.61 (m, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.45-7.36 (m, 8H), 7.22 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.4, 152.8, 150.6, 139.8, 132.8, 131.4, 129.3, 127.5, 127.3, 126.2, 124.3, 124.2, 124.1, 123.1, 120.4. FT-IR (KBr): 3066, 2961, 2924, 2854, 2852, 1653, 1594, 1526, 1497, 1446, 1327, 1261, 1101, 1018 cm⁻¹. Elemental analysis calcd (%) for C₁₉H₁₃N₃OS: C, 68.86; H, 3.95; N, 12.68; S, 9.68, found .C, 68.84; H, 3.96; N, 12.64 S, 9.71.



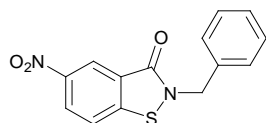
***N*-(2,4-Dimethylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (table 3, entry 10).** Colorless solid; 92% yield; mp 114-115 °C; ¹H NMR (CDCl₃, 400 MHz) δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.47 (d, *J* = 8.4 Hz, 1H), 7.30 (t, *J* = 8.4 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 1H), 7.04 (s, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 2.26 (s, 3H), 2.15 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.7, 141.3, 139.7, 137.4, 132.2, 132.0, 131.3, 128.8, 127.7, 127.2, 125.7, 124.1, 120.4, 21.3, 17.9. FT-IR (KBr): 2923, 2854, 1659, 1593, 1500, 1446, 1329, 1309, 1262, 1104, 1017 cm⁻¹. Elemental analysis calcd (%) for C₁₅H₁₃NOS: C, 73.56; H, 5.13; N, 5.49; S, 12.56, found C, 73.54; H, 5.16; N, 5.47; S, 12.54.



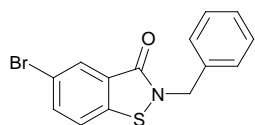
***N*-(3,4-Dimethylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (table 3, entry 11).** Colorless solid; 91% yield; mp 114-116 °C; ¹H NMR (CDCl₃, 400 MHz) δ 8.07 (d, *J* = 8.0 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.43-7.34 (m, 3H), 7.19 (d, *J* = 8.0 Hz, 1H), 2.28 (s, 3H), 2.26 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 164.3, 140.2, 137.9, 136.2, 134.8, 132.3, 130.5, 127.2, 126.2, 125.8, 124.9, 122.5, 120.2, 20.0, 19.5. FT-IR (KBr): 2920, 2849, 2920, 1637, 1467, 1350, 1307, 1261, 1101, 1021 cm⁻¹. Elemental analysis calcd (%) for C₁₅H₁₃NOS: C, 70.56; H, 5.13; N, 5.49; S, 12.56, found C, 70.59; H, 5.11; N, 5.51; S, 12.54.



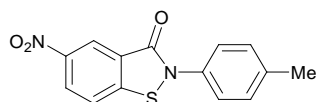
***N*-(2,6-Dimethylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (table 3, entry 12).** Colorless solid; 86% yield; mp 116-117 °C; ¹H NMR (CDCl₃, 400 MHz) 8.04 (d, *J* = 8.4 Hz, 1H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.36-7.34 (m, 1H), 7.12-7.07 (m, 3H), 2.26 (s, 3H), 2.12 (s, 3H); ¹³C NMR (CDCl₃, 400 MHz) δ 164.7, 141.4, 136.9, 134.6, 134.5, 132.3, 131.2, 130.5, 129.6, 127.3, 125.8, 124.1, 120.4, 20.9, 17.6. FT-IR (KBr): 2920, 2849, 2920, 1637, 1470, 1307, 1265, 1111, 1021 cm⁻¹. Elemental analysis calcd (%) for C₁₅H₁₃NOS: C, 70.56; H, 5.13; N, 5.49; S, 12.56, found C, 70.58; H, 5.15; N, 5.52; S, 12.54.



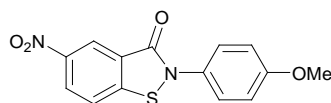
***N*-Benzyl-5-nitrobenzo[*d*]isothiazol-3(2*H*)-one (table 4, entry 2).** Yellow solid; 35% yield; mp 178-180 °C; ¹H NMR 8.33-8.31 (m, 1H), 7.36-7.35 (m, 1H), 6.93-6.90 (m, 1H), 6.46-6.34 (m, 5H), 5.02 (s, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) 165.1, 146.7, 139.3, 138.3, 137.8, 132.0, 129.1, 128.0, 127.7, 126.1, 124.3, 55.6. FT-IR (KBr): 3399, 2962, 2977, 2857, 1651, 1591, 1513, 1408, 1340, 1299, 1261, 1094, 1021 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₀N₂O₃S: C, 58.73; H, 3.52; N, 9.78; S, 11.20, found C, 58.76; H, 3.50; N, 9.75; S, 11.23.



***N*-Benzyl-5-bromobenzo[*d*]isothiazol-3(2*H*)-one (table 4, entry 3).** Colorless solid; 90% yield; m.p. 139-140 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.177-8.172 (m, 1H), 7.67 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.36-7.30 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 165.0, 138.8, 133.5, 131.7, 131.4, 131.3, 130.6, 129.9, 128.3, 126.9, 125.9, 42.5. FT-IR (KBr): 2930, 1627, 1331, 1356, 1252, 1075, 1041 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₀BrNOS: C, 52.51; H, 3.15; N, 4.37; S, 10.01, found C, 52.48; H, 3.16; N, 4.35; S, 10.08.



5-Nitro-*N*-p-tolylbenzo[*d*]isothiazol-3(2*H*)-one (table 4, entry 6). Yellow solid; 35% yield; m.p. 111-112 °C; ¹H NMR (CDCl₃, 400 MHz) δ 8.77-8.71 (m, 1H), 8.43-8.39 (m, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.18 (d, *J* = 8.4 Hz, 2H); 2.28 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 163.7, 148.4, 137.0, 134.7, 133.8, 130.8, 129.9, 129.8, 126.7, 123.0, 121.3, 21.2. FT-IR(KBr): 2930, 2923, 2256, 1651, 1048, 1025 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₀NO₃S: C, 58.73; H, 3.52; N, 9.78; S, 11.20, found C, 58.76; H, 3.55; N, 9.75; S, 11.18.

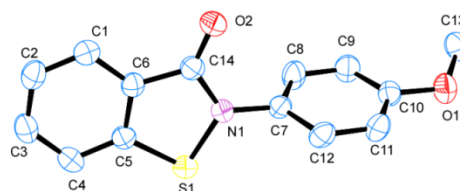
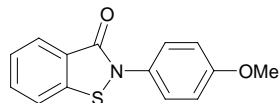


***N*-(4-Methoxyphenyl)-5-nitrobenzo[*d*]isothiazol-3(2*H*)-one** (table 5, entry 4). Yellow solid; 45% yield; 114-115 °C (DMSO); ¹H NMR (CDCl₃, 400 MHz) δ 8.68-8.67 (m, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 3H), 3.73 (s, 3H); ¹³C NMR (CDCl₃, 100MHz) δ 162.4, 156.0, 146.2, 137.9, 137.2, 131.6, 131.4, 125.6, 123.9, 121.5, 114.0, 55.3. FT-IR(KBr): 2255, 2128, 1651, 1510, 1246, 1048, 1025, 1002 cm⁻¹. Elemental analysis calcd (%) for C₁₄H₁₀NO₄S: C, 55.62; H, 3.33; N, 9.27, S, 10.61, found C, 55.66; H, 3.31; N, 9.30, S, 10.63.

References

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Crystal Structure of N-(4-methoxyphenyl)benzo[d]isothiazol-3(2H)-one



Crystal number: Summary of Data CCDC 876574 Thermal ellipsoids are drawn at a 40% probability level. Hydrogen atoms have been omitted for clarity.

Formula: C₁₄ H₁₁ N O₂ S

Unit cell parameters: a 10.5093(14) b 13.6166(17) c 9.1287(12) beta 114.028(6) space group P 21/c

Datablock:

Bond precision:	C-C = 0.0022 Å	Wavelength=0.71073	
Cell:	a=12.7577(7)	b=10.0993(6)	c=11.6512(7)
	alpha=90	beta=114.028(6)	gamma=90
Temperature:	296 K		

	Calculated	Reported
Volume	1193.1(3)	1193.1(3)
Space group	P 21/c	P2(1)/c
Hall group	-P 2ybc	?
Moiety formula	C ₁₄ H ₁₁ N O ₂ S	?
Sum formula	C ₁₄ H ₁₁ N O ₂ S	C ₁₄ H ₁₁ N O ₂ S
Mr	257.31	257.31
Dx, g cm ⁻³	1.433	1.432
Z	4	4
Mu (mm ⁻¹)	0.263	0.263
F000	536.0	536.0
F000'	536.72	
h,k,lmax	14,19,12	14,19,11
Nref	3452	3382
Tmin,Tmax	0.895,0.912	0.895,0.912
Tmin'	0.895	

Correction method MULTI-SCAN

Data completeness= 0.980
R(reflections)= 0.0388(3340)
S = 1.023

Theta(max)= 29.940
wR2(reflections)= 0.0947(3109)
Npar= 165

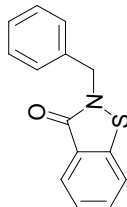
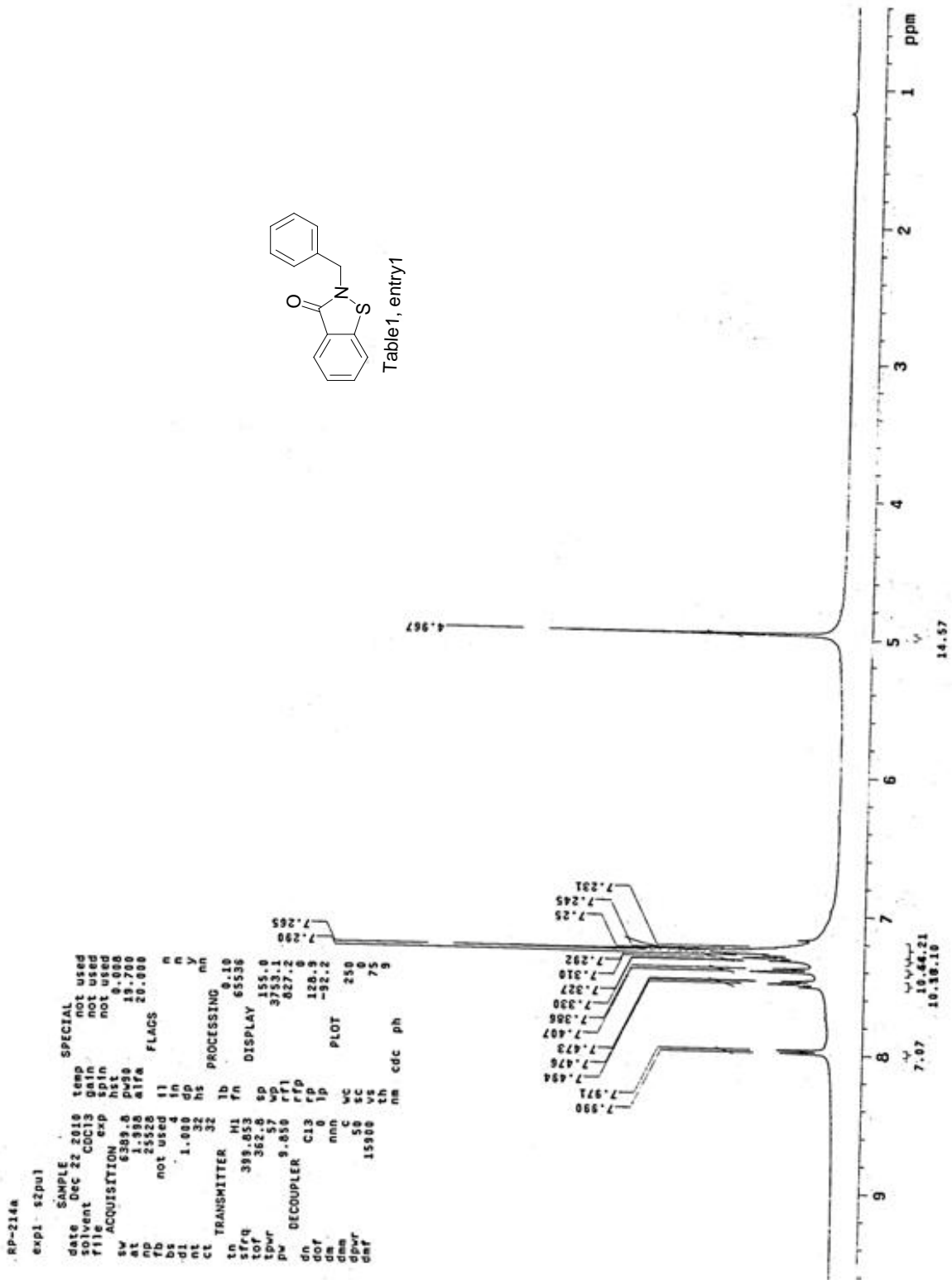


Table1, entry1

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exp1 s2p01
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date Dec 23 2010 temp not used
solvent C0C13 gain not used
file C0C13 exp mot used
ACQUISITION hst 0.005
sw 25125.6 Dv90 18.000
nt 6072 a1FA 20.000
fb 13508 11
bs 16 in n
dl 1.000 dp V
nt 3800 hs
cl 1804 PROCESSING mn
tn C13 fb 2.00
frrq 188.554 fn 65536
tor 1536.3 sp DISPLAY -722.0
tpr 9.811 wd 2178.0
pr 9.380 rfp 776.0
DECOUPLER H1 rfp -52.4
dn 0 lp -312.3
ds yyy wc PLOT 250
dm 42 sc 0
dpr 8908 th vs 02
cbr mb mo ph 5
    
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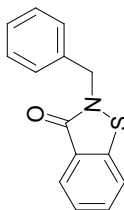
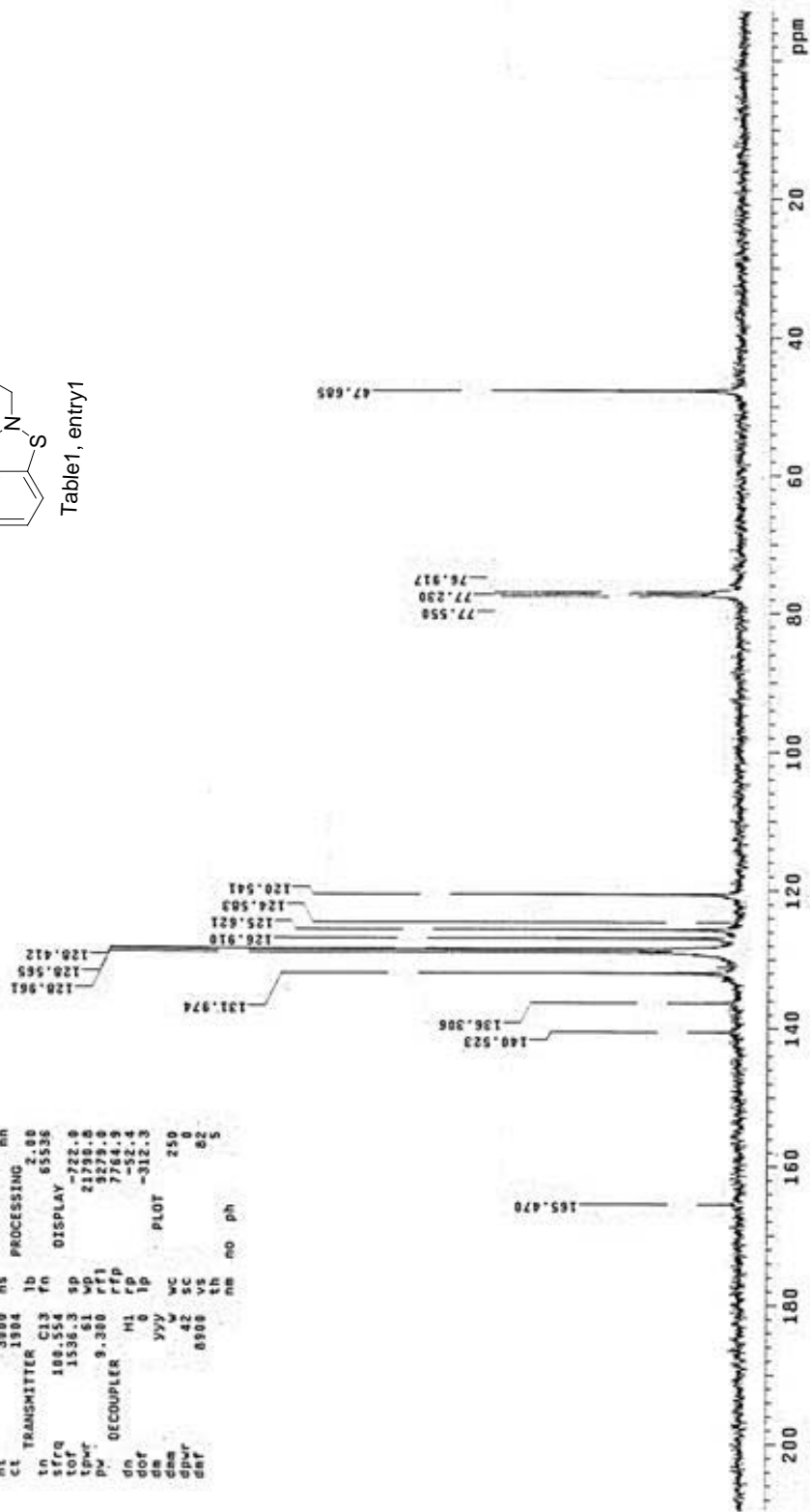


Table1, entry1



RP-227

exp1 s2pu1

date	Feb 26 2011	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	8.000
sw	6389.8	pw90	19.700
at	1.998	alfa	20.000
np	25528	FLAGS	
fb	not used	l1	n
bs	4	ln	n
dl	1.000	dp	y
nt	32	rs	nn
ct	TRANSMITTER 32	rs	PROCESSING
tn	399.453	fb	0.10
trfq	362.8	fn	65536
tor	57	sp	DISPLAY
tpwr	9.858	bp	136.7
pw	DECOUPLER C13	wp	4237.0
dn	8	rf1	3698.5
dof	nnn	rfp	2884.9
dsw	.c	lp	187.9
dwr	50	pl	-92.0
dwt	15900	mc	250
		sc	0
		vs	.70
		th	nm cdc ph
			3

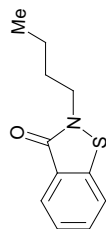
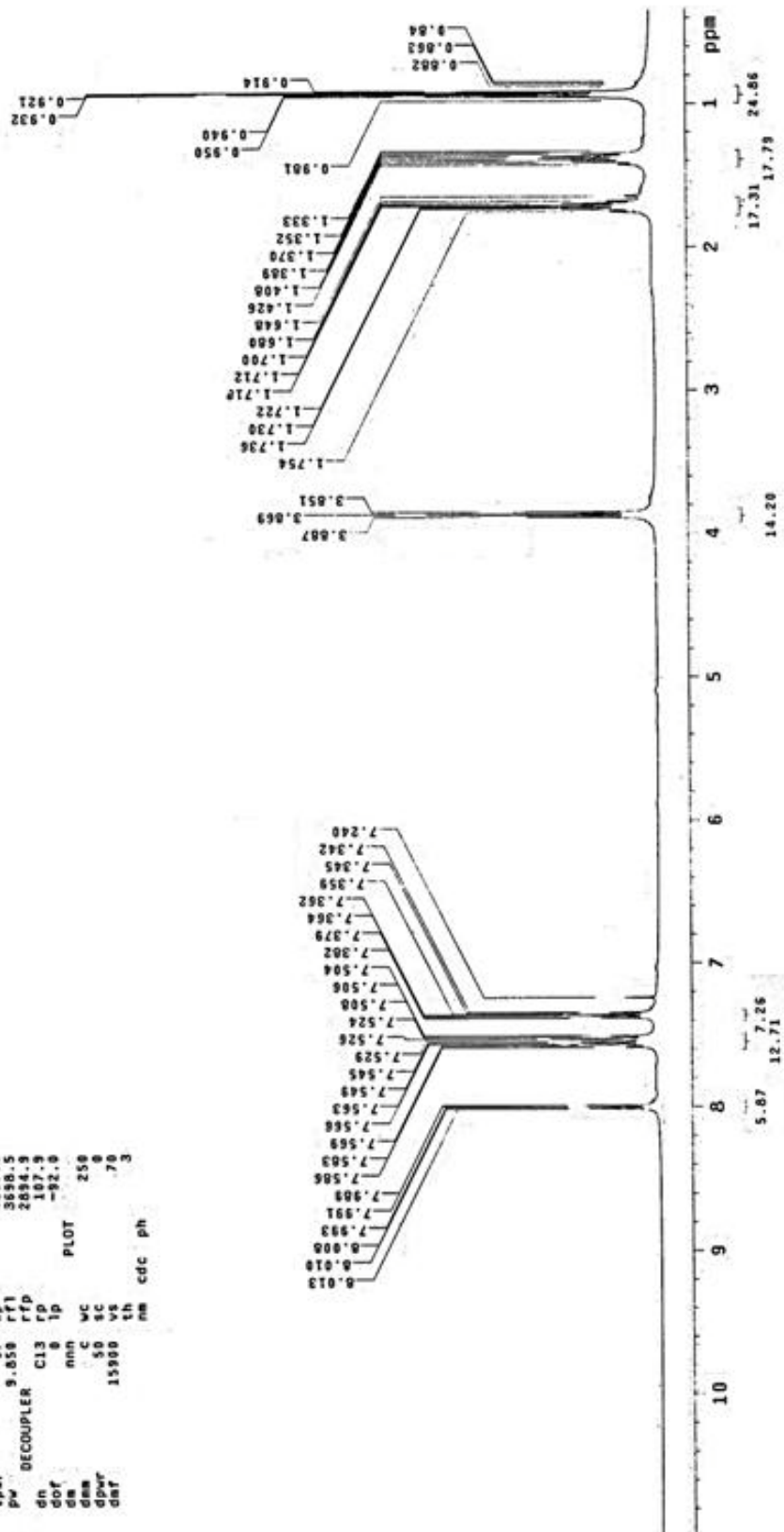


Table 2, entry 1



RP-227-13c

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exp1 s2pul
date Mar 25 2011 temp not used
solvent COC13 gain not used
file exp not used
sv ACQUISITION exp hst 0.000
st 25125.6 p490 16.600
sp 19270 a17a 28.000
sb 13532 11 FLAGS
d1 1.02 in n
d2 10000 ns y
d3 10000 ns y
ct TRANSMITTER C13 2.00
sfreq 100.554 fn 85536
tof 1538.3 sp DISPLAY -558.2
tpwr 61 wd 20692.3
pv DECOUPLER H1 rfp 1597.2
dn dof 0 lp -26.8
da dsv yyy wc PLOT -426.6
dsw 42 sc 258
dwt 8900 vs 8
dwt th no ph 29
dwt ms no ph 2
    
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SPECIAL

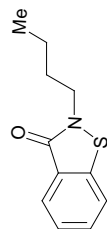
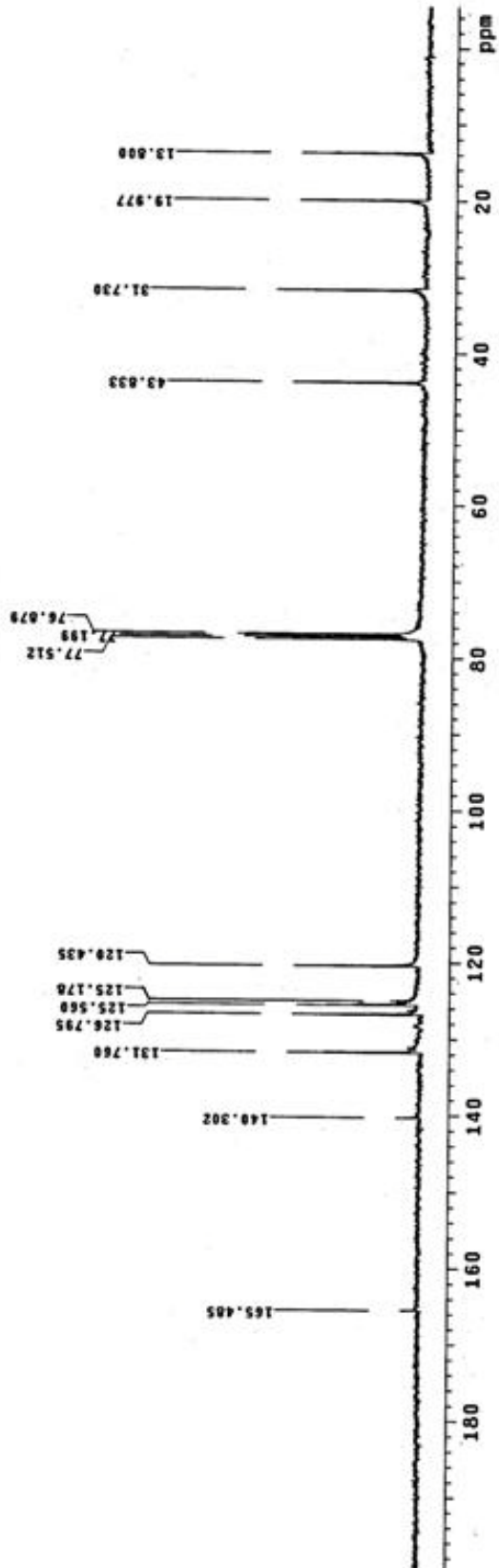


Table 2, entry 1



```

RP-224
expl szpul
SPECIAL
date Feb 1 2011 temp not used
solvent CDCl3 gain not used
file exp not used
sw ACQUISITION exp hst 0.008
at 6389.0 pw20 19.709
rb 1.998 a17a 20.008
np not used 11
bs not used 4
di 1.080 dp
nt 32 hs
ct TRANSMITTER M1 lb 6536
in 399.853 fn DISPLAY 43.3
top 362.0 sp 3595.6
tpr 57 vp 3599.5
pw 9.659 rfp 2894.9
deCOUPLER C13 rfp 116.4
dn dof 0 lp -84.0
dm nnn
dss c -250
dpr 50
dfr 34
dfr 15900
mh cdc ph
    
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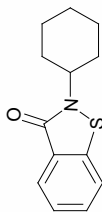
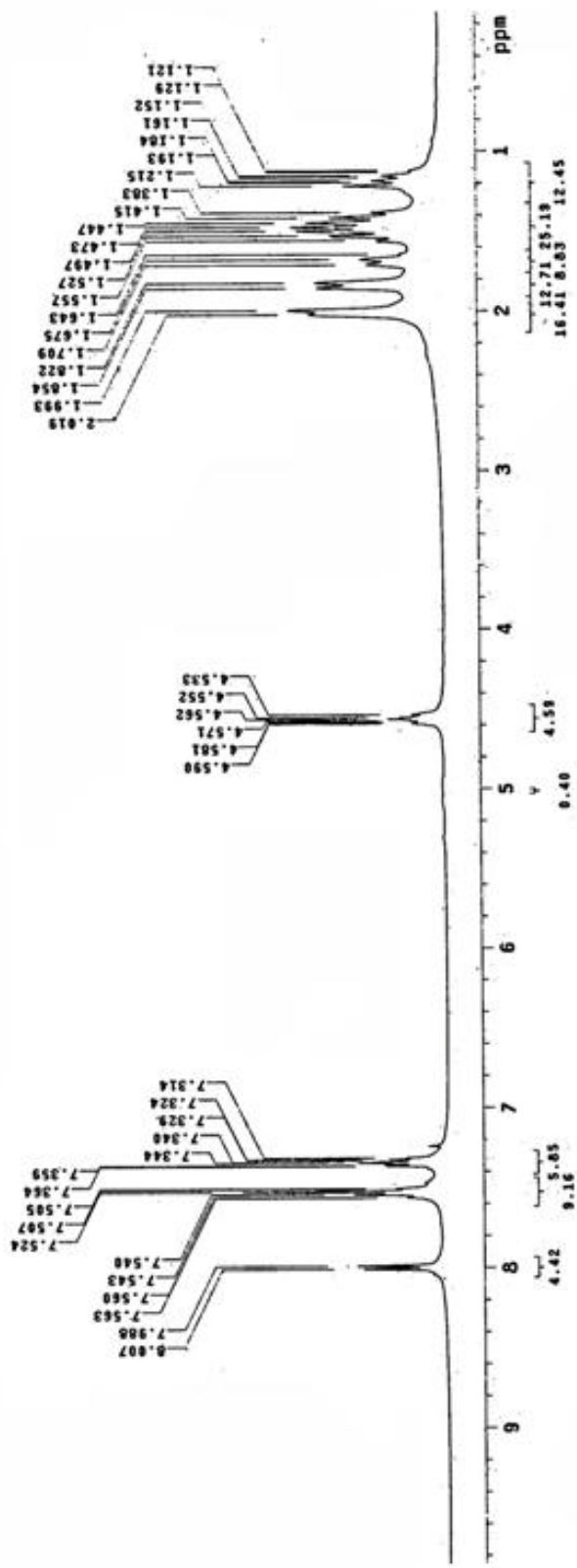


Table 2, entry 2




```

RP-224
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date SAMPLE Feb 22 2011 temp not used
solvent CDCl3 gain not used
file exp not used
sv ACQUISITION exp hst 9.000
at 25125.6 pps0 10.000
fd 65378 a1fa 20.000
fp 13600 f1
bd 16 n
bl 1.000 in
d1 7000 dp n
nt 7000 hs y
ct TRANSMITTER 7000 lb PROCESSING 2.00
tn C13 fn DISPLAY 6536
sfreq 100.554 sp -1509.5
tof 1538.3 sp 25125.6
tpwr 61 wp 9274.4
pw DECOUPLER 9.300 rfp 7764.9
dn HI rfp -47.3
dof 0 ip -351.5
dm yvy w VC 250
dwa 42 sc 0
dwr 8900 vs 60
dwt th no .ph
    
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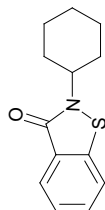
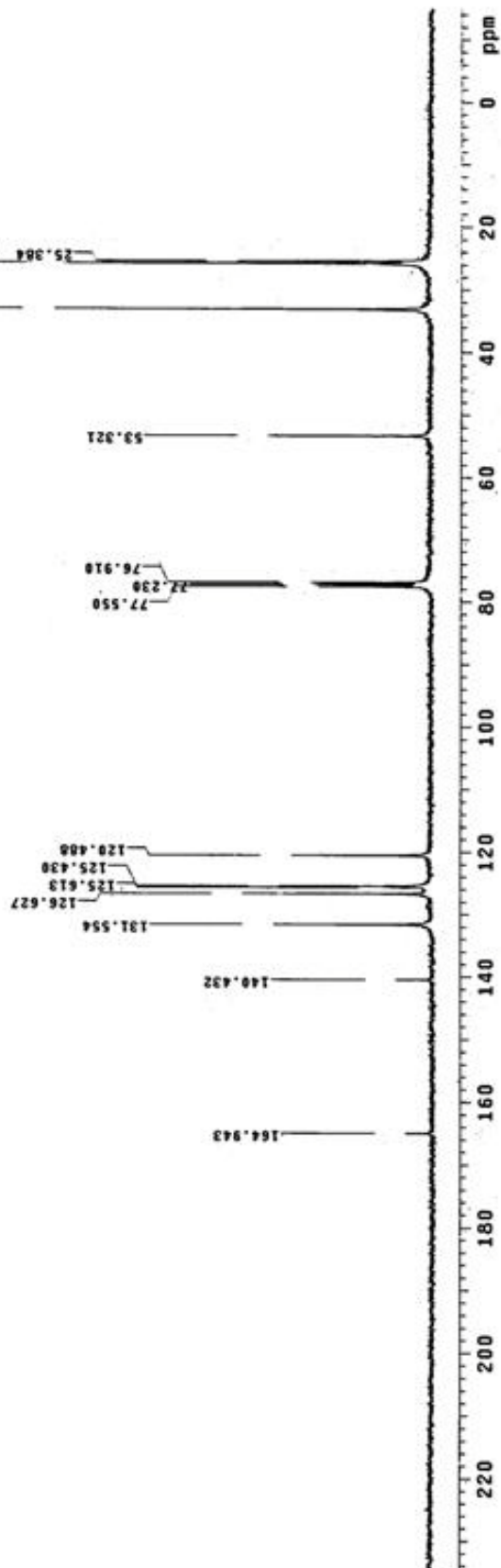
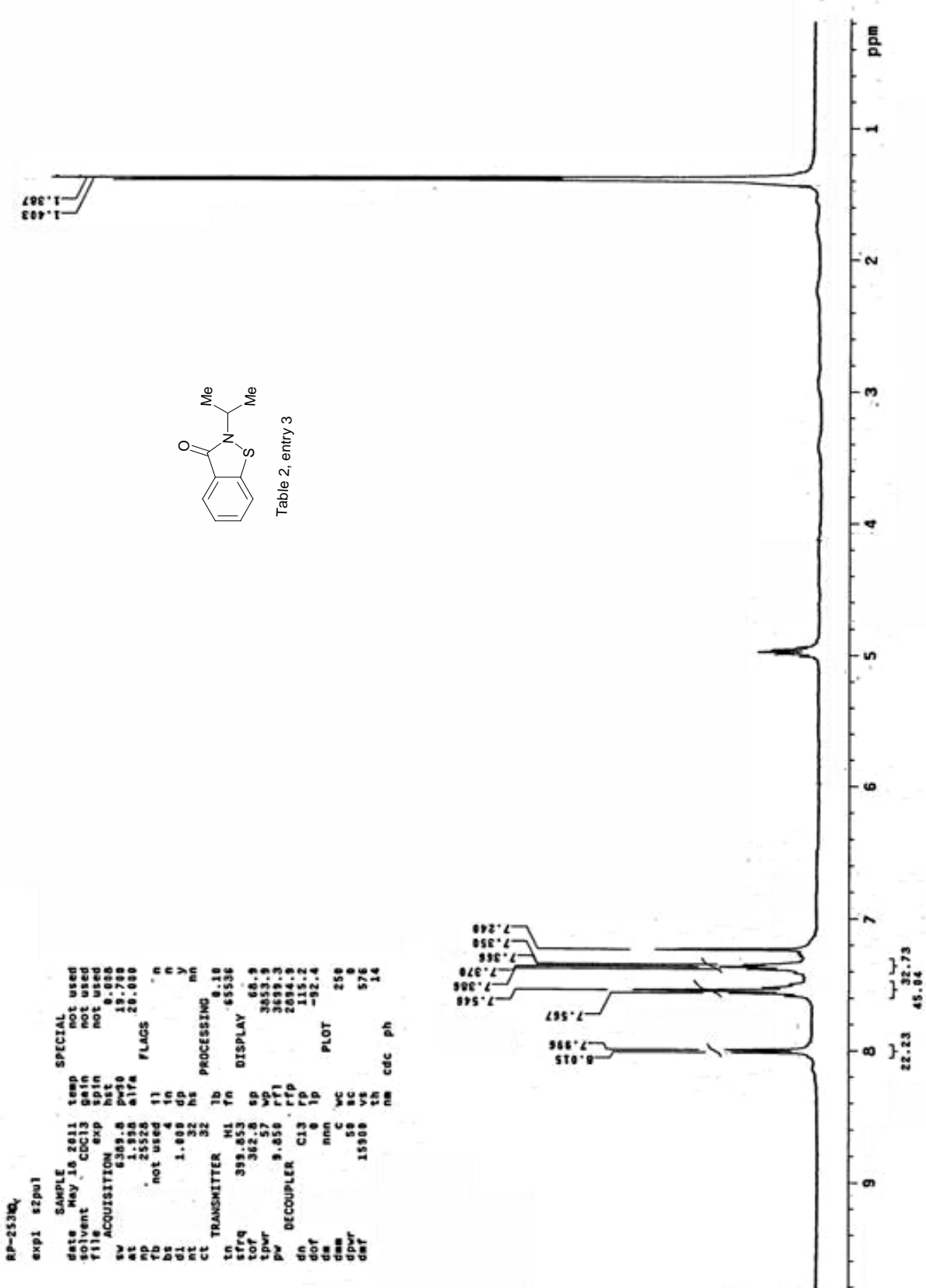


Table 2, entry 2





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date SAMPLE temp not used
solvent Aug 16 2011 temp not used
file CDC13 gain not used
file exp spin not used
ACQUISITION hst 0.808
sv 25125.6 pwr 16.600
nt 1.275 a17a 20.000
fp 8220 11 FLAGS
bs 15810 11 n
di 1.020 11 n
nt 7080 11 y
ct 860 11 y
TRANSMITTER CL3 lb Tn
tfrq .100.514 Tn DISPLAY 6536
tof 1536.3 SP -1517.2
pwr 61 wd 25125.6
pv 9.300 rfp 8202.1
DECOUPLER H1 rfp 7764.9
dn 0 1p -53.7
dm 0 1p -313.6
dmm yvy WC 250
dppr 42 SC 0
dar 8980 VS 14
nm no ph 3
    
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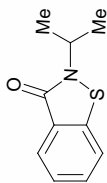
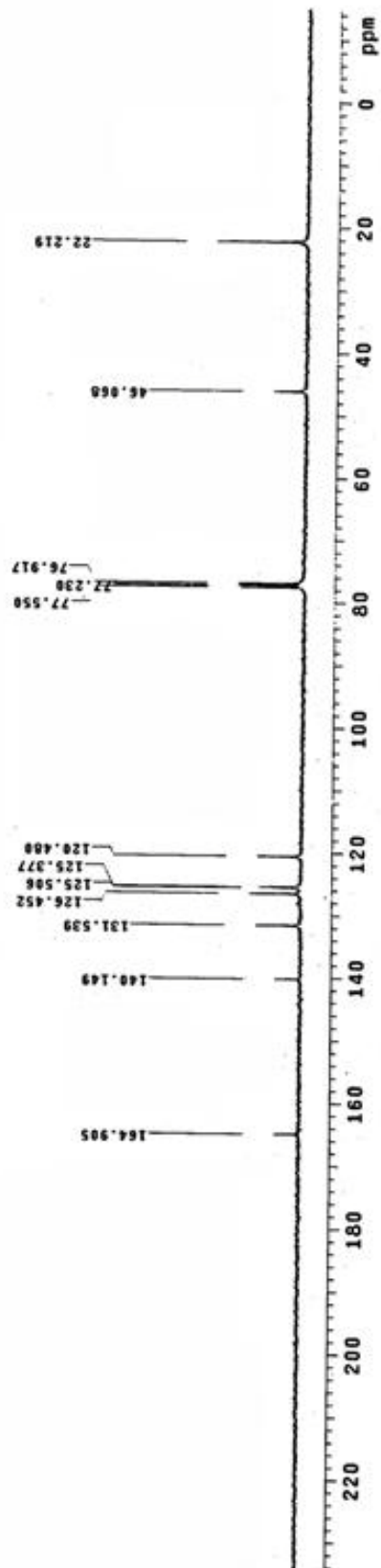


Table 2, entry 3



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RP-258
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SAMPLE
date   Feb 11 2012   temp not used
solvent C6Cl3      gain  not used
file    exp        not used
ACQUISITION exp    not used
sv      6389.8     Dv99  18.700
at      1.998     d1fa  26.000
np      not used  t1
bs      1.000     dp
dl      1.000     ds
nt      32       hs
ct      TRANSMITTER H1  lb  8.10
          399.853   fm  6558
          362.8    sp  328.2
          57       wd  3711.2
          9.650    rf1  795.8
          DECOUPLER C13 rp  191.2
          0       lp  -74.3
          nnn     wc  250
          50      sc  30
          15980  th  35
          nm cdc ph
    
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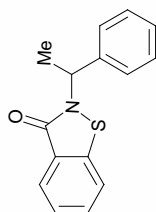
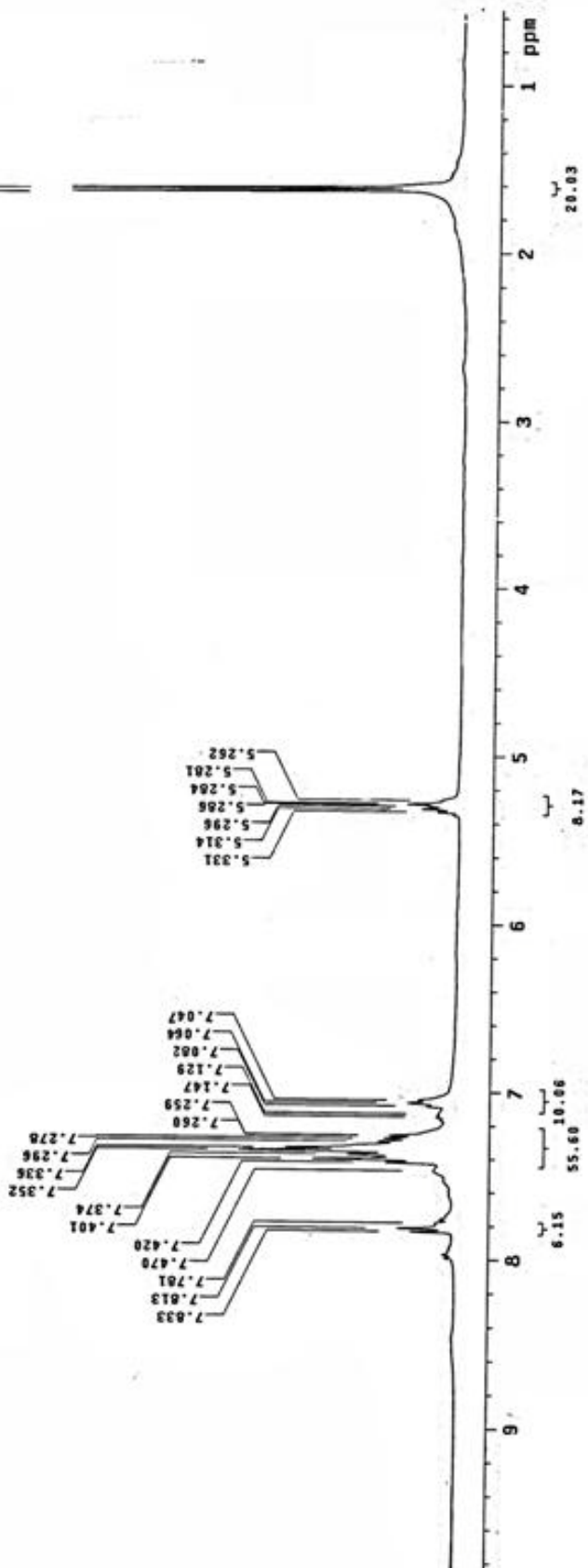


Table 2, entry 4



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solvent CDCl3 gain not used
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ACQUISITION .fid pvs0 10.000
          alfa 20.000
          flags
sv 25125.6
sc 1.375
td 13800
bs 18
dl 1.000
nt 7000
ct 7000
IN TRANSMITTER
ln C13 sp 168.2
sfrq 100.625
tof 1536.3
tpwr 61
pw 3.350
DECOUPLER M1
dn MC 250
dm yyw
dms vs
dpr 42
dmt 8500
nm no ph
    
```

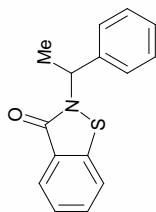
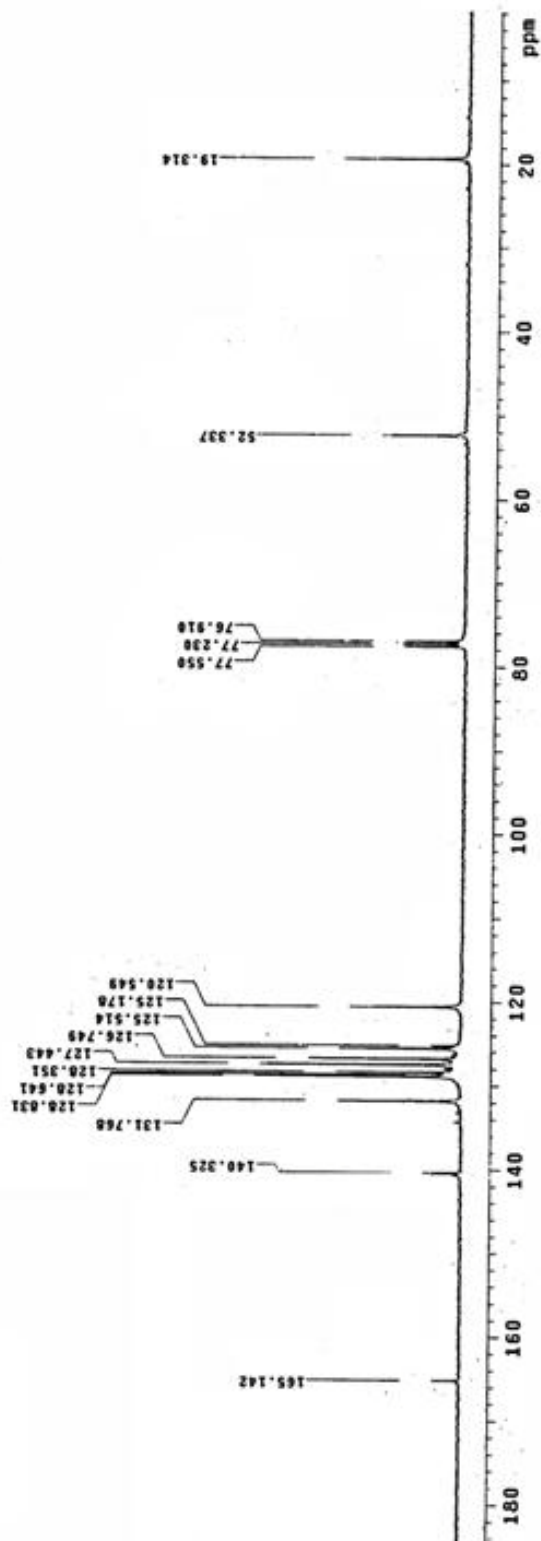
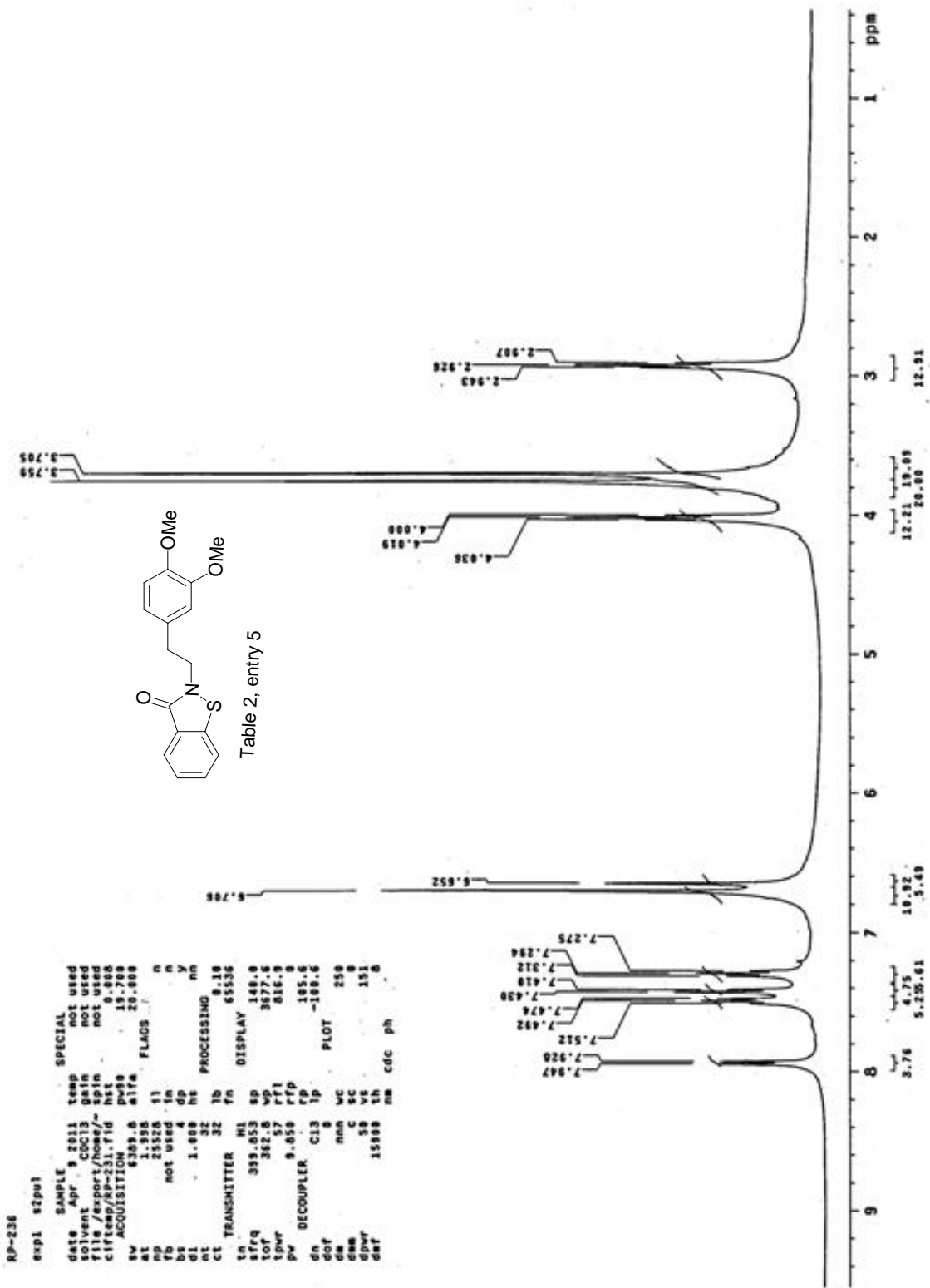


Table 2, entry 4





```

RP-231
exp1 s2pu1
SAMPLE SPECIAL
date Apr 1 2011 temp not used
solvent CDCl3 gain not used
file/export/home/~ spin not used
c1ftemp/RP-231-13C-ht 9.000
ACQUISITION .fid pw38 18.000
sv 25125.6 s1fa 29.000
FLAUS
at 1.139 f1 n
pb 49278 in n
fb 13692 dp y
bl 1.000 ht nn
bl 1.000 ht PROCESSING 2.00
nt 18000 fb DISPLAY 65536
CT 18000 FT
TRANSMITTER CL3 5D -383.0
sfrq 189.554 wd 20734.2
tof 1536.3 rfi 9279.0
tper 61 rfd 7764.9
pw 9.388 rfp -80.5
DECOUPLER H1 1P PLOT -271.4
dn 0 WC 250
da 0 SC 0
dm vvy vt 18
dpr w vs 3
dnt 42 th 3
8500 na no ph
    
```

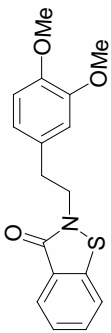
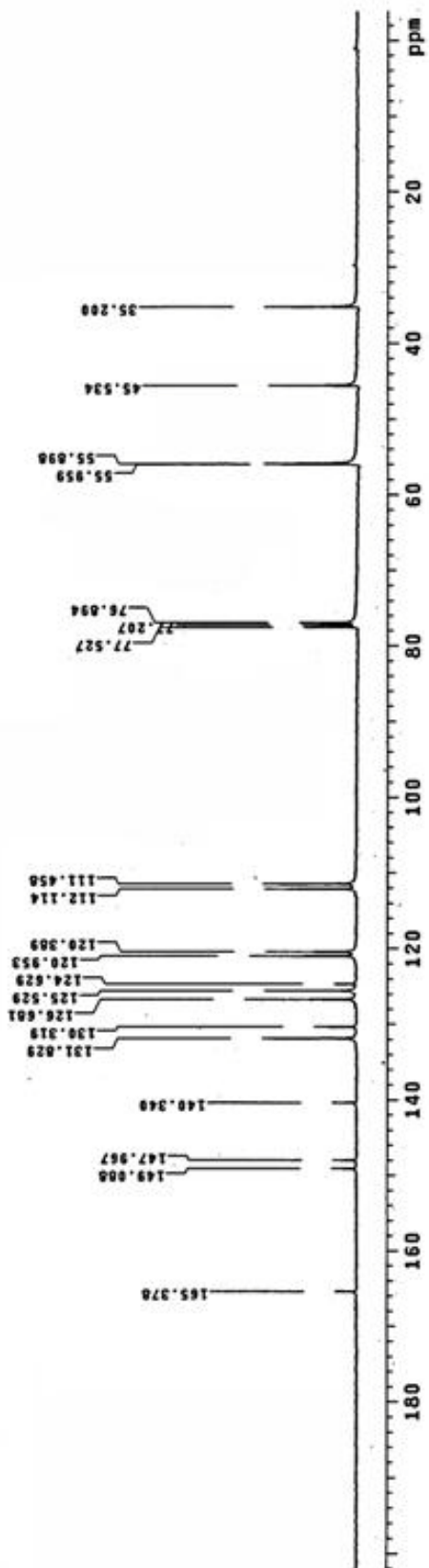


Table 2, entry 5



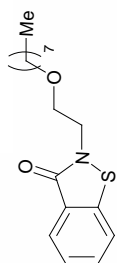
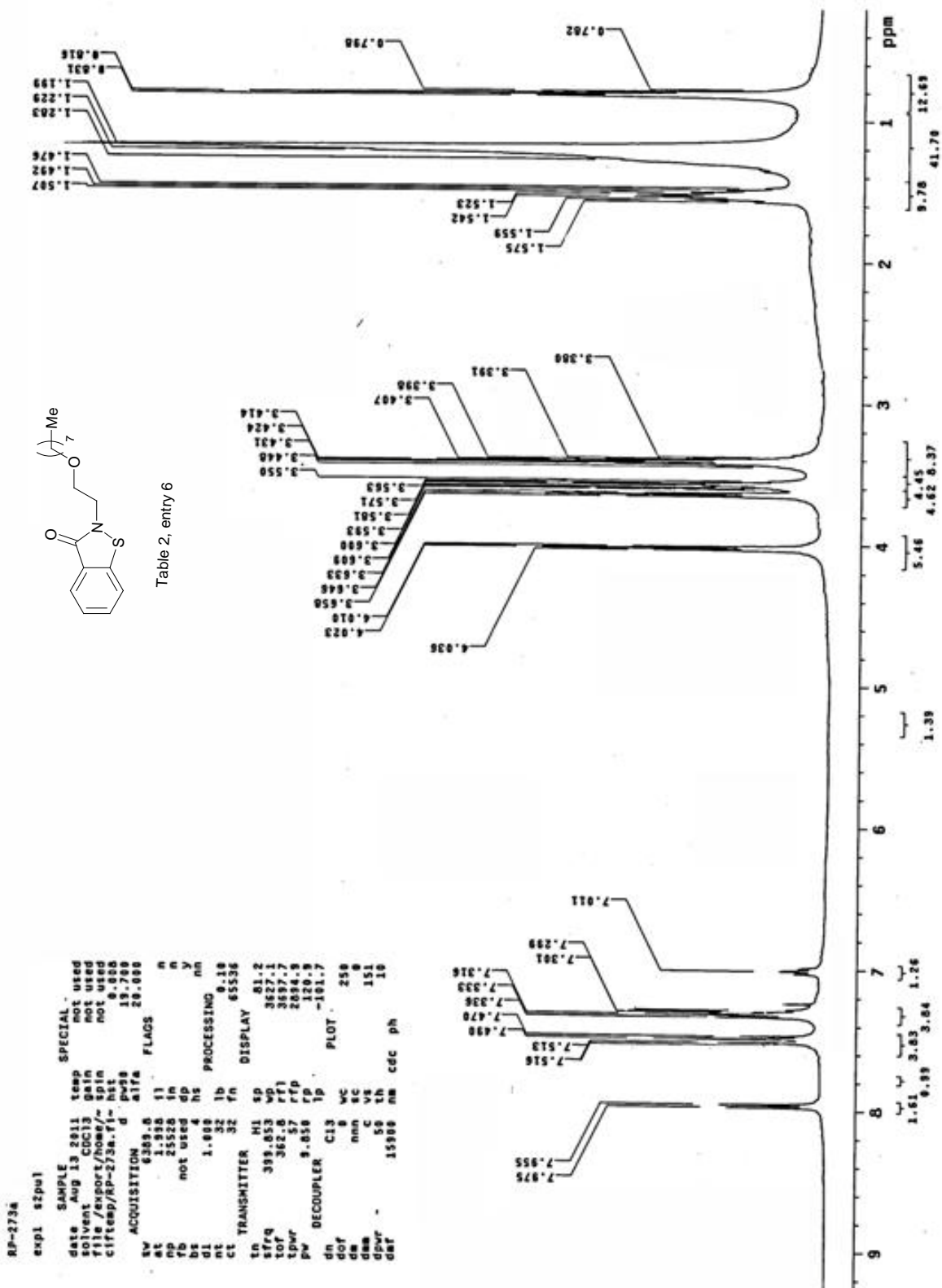


Table 2, entry 6




```

RP-273a
exp1 szpul
SAMPLE
date Aug 13 2011 temp not used
file /export/home/ not used
ciftemp/RP-273a-h1 spin not used
- ACQUISITION: h1 ps 8.888
10.000
20.000
SPECIAL
25125 5 11 FLAGS
41 1.148 11 n
58278 1n n
13800 dp y
bs 18 bp
nl 1.000 1b
nl 10000 1b
ct 1600 1a DISPLAY 65536
TRANSMITTER C13 SP -1502.1
sfrq 100.554 wd 25125.6
tof 1536.3 rfi 1583.1
tpwr 61 rfp -30.2
pw 9.350 rp -373.6
DECOUPLER H1 1p PLOT
dn 0 wc 250
dof 0 sc 0
dm vvy sc 32
dss w vs 42
dpr th no 2
dpr 6900 na ph
    
```

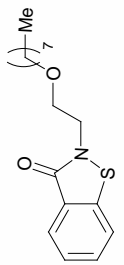
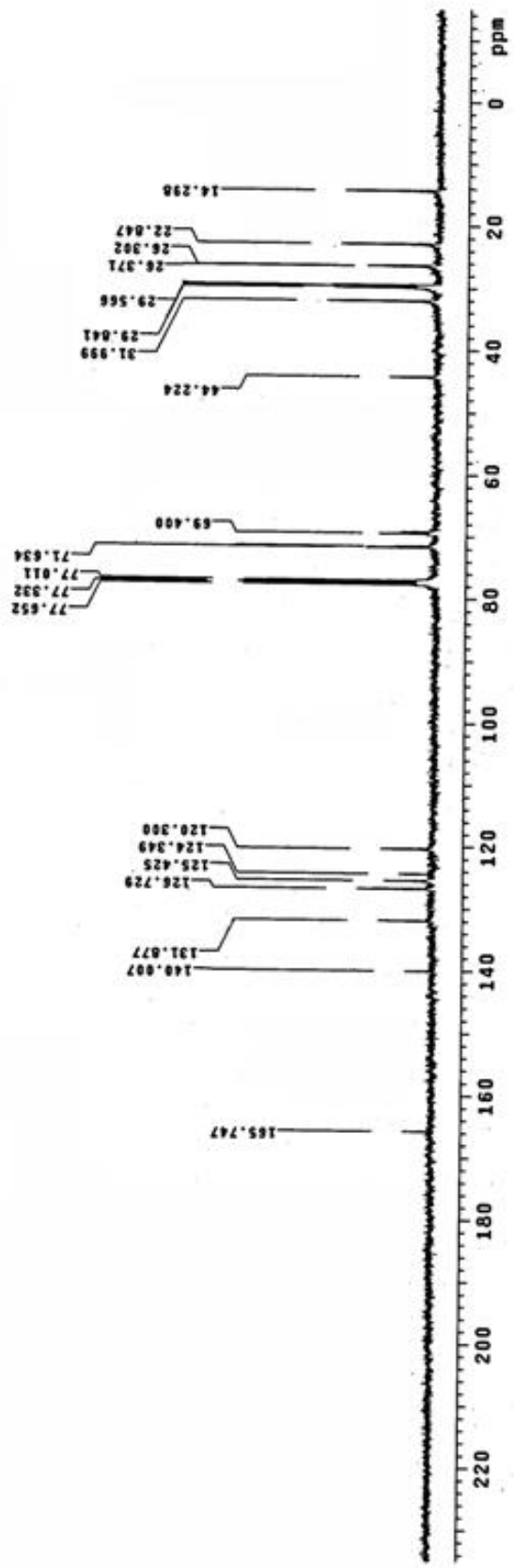
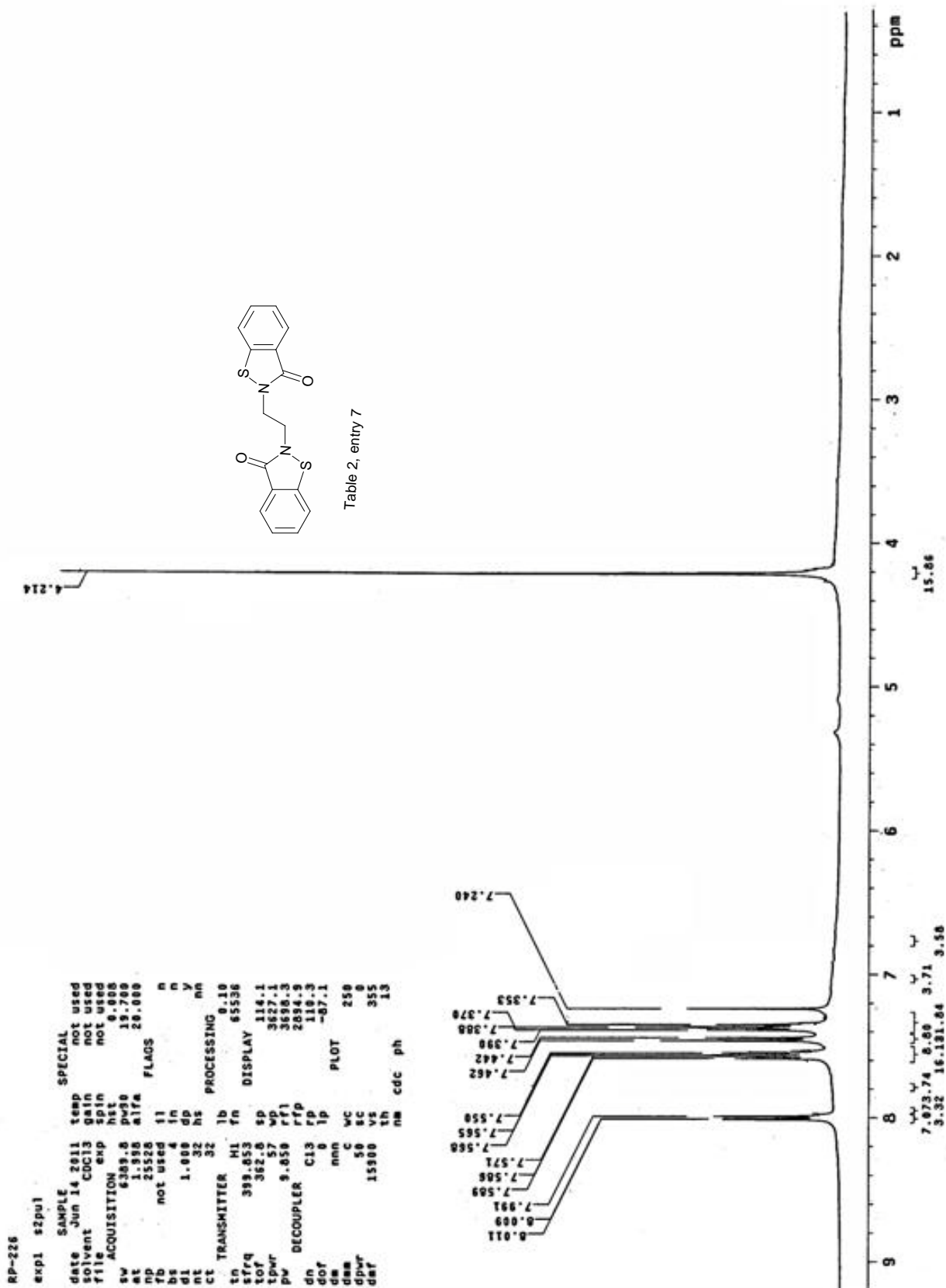


Table 2, entry 6





RP_217
exp1 42pul

SAMPLE		SPECIAL	
date	Jun 23 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		exp	not used
sw	251.2516	ns1	16.000
st	1.199	ns2	16.000
at	1.199	at1a	20.000
np	60276	FLAGS	
fb	13800	l1	n
bs	1.0	l2	n
dl	1.000	dp	y
nt	10000	hs	mn
ct	10000	PROCESSING	
tn	2.00	lb	2.00
tr	CDCl3	fm	6556
sfrq	100.554	dm	DISPLAY
tof	1536.3	sp	-760.3
tpw	61	wp	21056.0
pw	9.300	rf1	9271.3
de	9.300	rf2	7764.3
dm	NI	tp	-70.4
dmc	TP	pl	-237.0
dms	YVW	vc	258
dsv	42	sc	8
dpr	8100	vs	84
dwt		th	3
		ms	no
		ph	

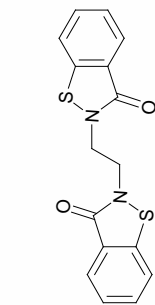
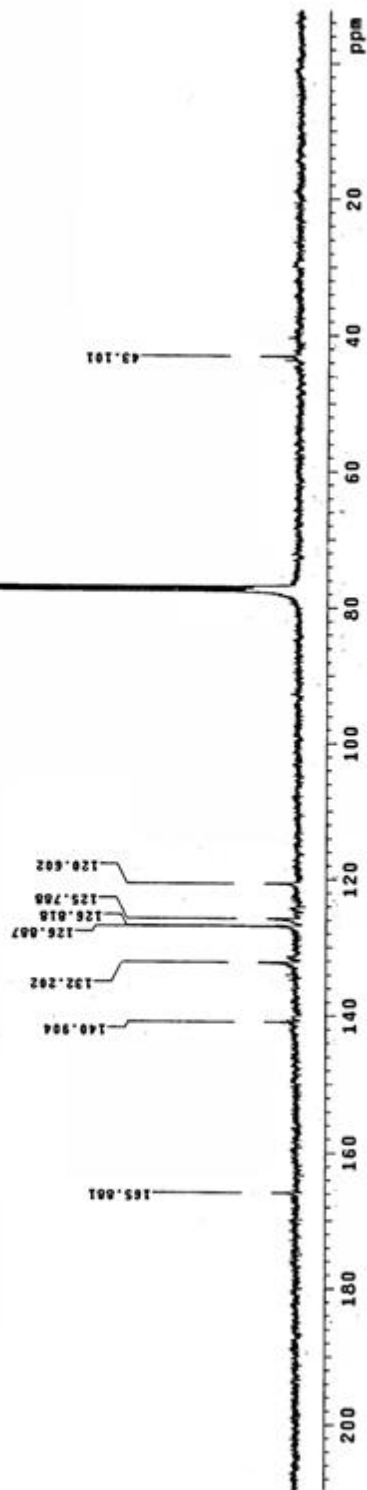
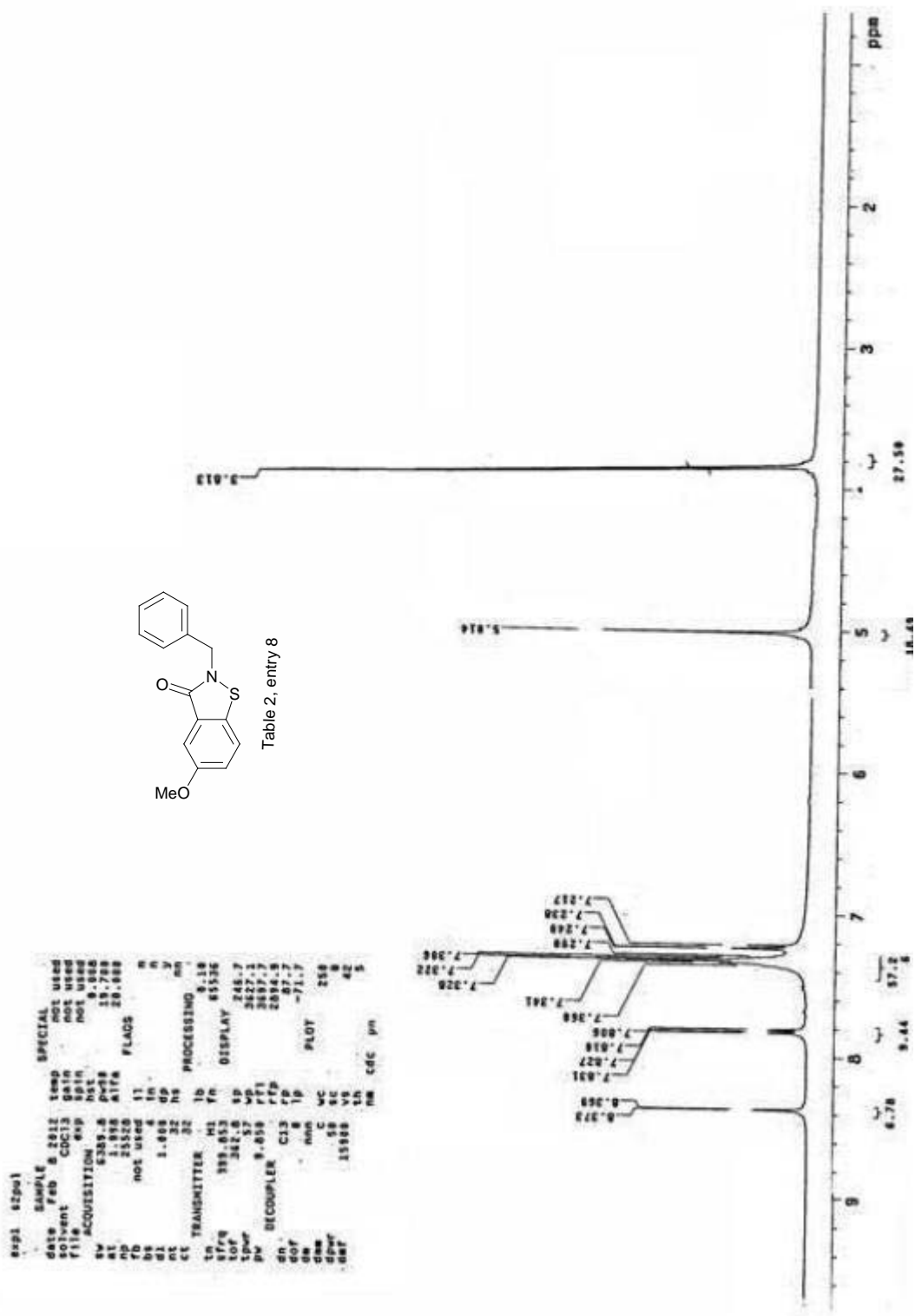


Table 2, entry 7





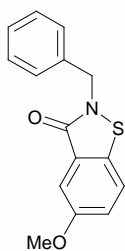
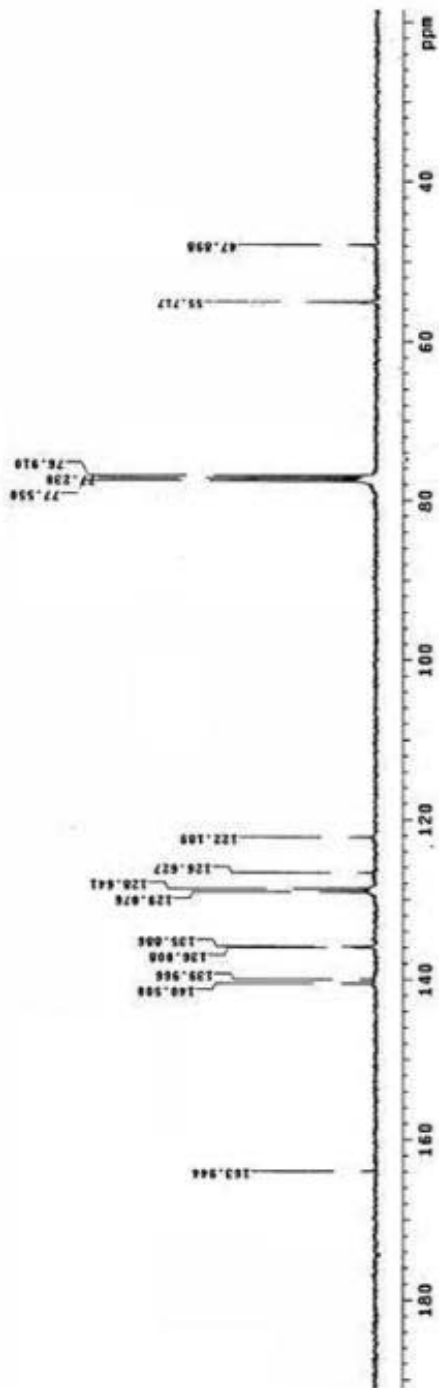


Table 2, entry 8

```

exp1 szpet
=====
NAME SAMPLE 8 3812 temp SPECIAL
SOLVENT PMS CDCl3 0min NOT USED
FILE /export/home/~spia NOT USED
CIFTEMP/PP_252b-13-051 8.000
C.FID P498 18.000
ACQUISITION SITE 28.000
=====
SI 28125.8 11 n
SI 1.199 11 n
SI 68278 11 n
SI 13000 40 y
SI 1.000 00 n
SI 1.000 10 n
SI 10000 10 n
SI 5926 70 n
SI 5538 5538 n
=====
CT TRANSMITTER C13 0P
SI 188 513 0P 1000.0
SI 107 553.3 0F1 1075.6
SI 61 776.8 61 776.8
SI 8.300 1P -34.0
SI 0.000 1P -34.0
=====
SI DECOUPLER NI 1F PLOT -391.0
SI 0 0 MC 258
SI 0 0 SC 8
SI 0 0 W 31
SI 42 10 n
SI 8908 00 n
SI 00 n
=====

```



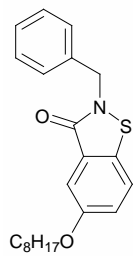
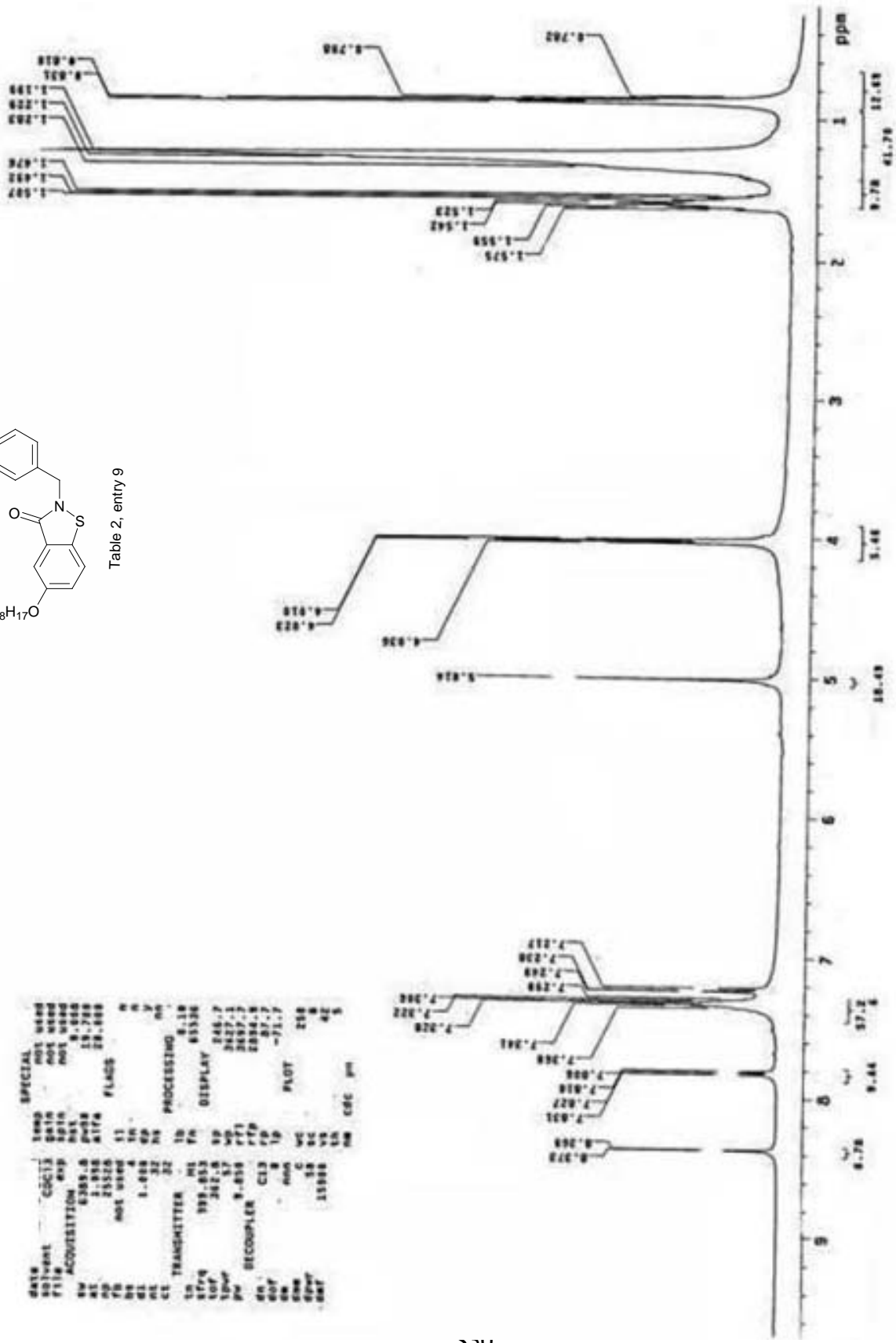


Table 2, entry 9




```
RP-25280~
expl stidh
SAMPLE
date Oct 25 2011 temp not used
solvent CDCl3 gain not used
fl 100000000 exp not used
sv ACQUISITION 6006.0 0450 19.780
at 1.915 01fa 28.000
np not used 11 flags n
rb not used in n
dl 1.000 in n
ct 32 ns PROCESSING mh n
tn M1 fn DISPLAY not used
stfz 399.853 sp 146.8
tolr 57.000 wf 3764.2
pwr 7.800 rfp 966.0
DECOUPLER CL3 10 105.4
dn dof nmn vc PLOT -78.9
dm dm c 250
dss c 8
dwt 50 22
SMT 15908 tn cdc ph
```

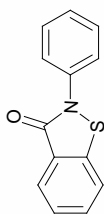
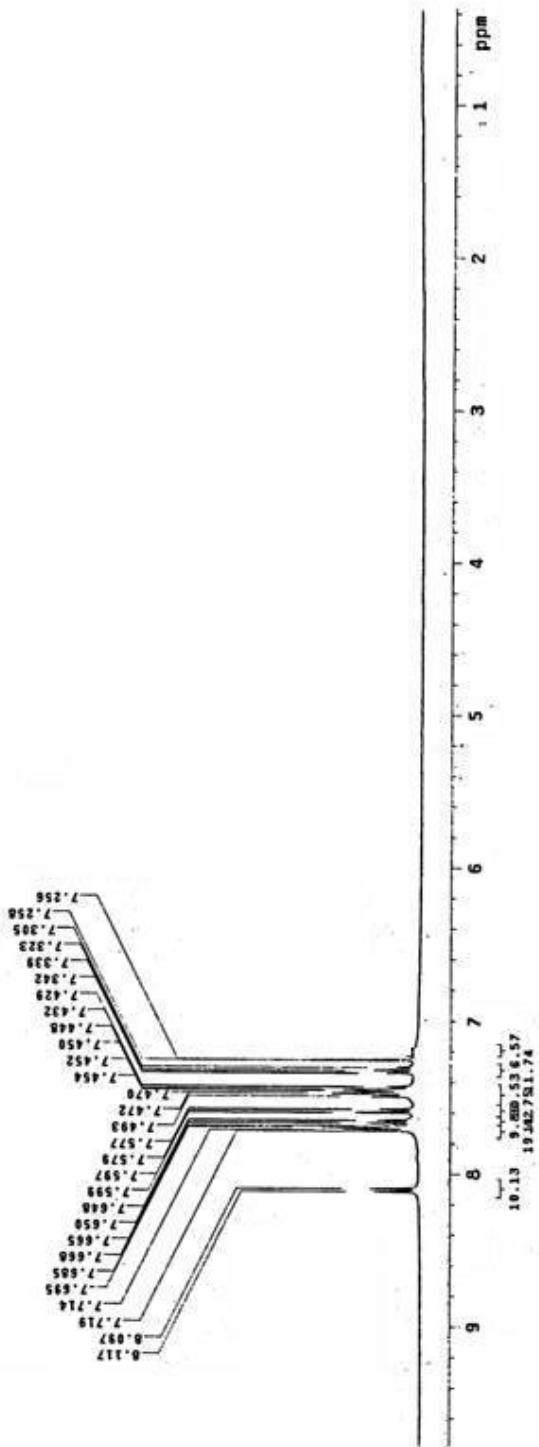


Table 3, entry 1




```

RP-252a
expl std13c
date SAMPLE Oct 25 2011 temp not used
solvent CDCl3 gain not used
file exp hst not used
ACQUISITION exp hst 0.008
sv 25000.0 pw38 10.000
at 1.199 m17a 20.000
np 59160
fb 13000
bs 16
di 0
nt 7000
ct 1072
TRANSMITTER lb 1.00
:fn not used
sfrq 100.552 :fn DISPLAY
tof 0 :sp -2976.6
tpr 61 :wp 25000.0
pw 0.667 :rfi 2976.6
DECOUPLER HI :rfp 0
dn 0 :lp -62.7
dof 0 :lp -270.0
dm yyy :w WC 250
dss w :s SC 0
dpr 42 :sc 23
dmf 8300 :vs VS 2
nm no ph 2
    
```

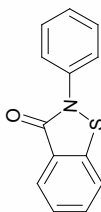
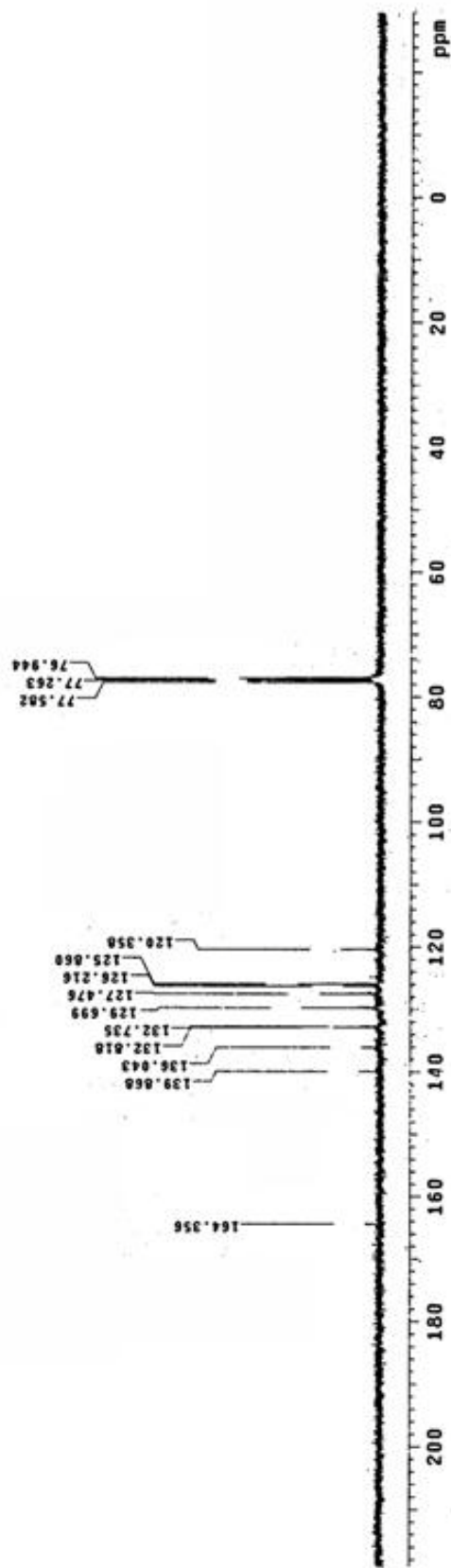


Table 3, entry 1



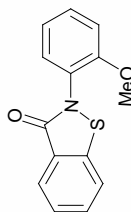
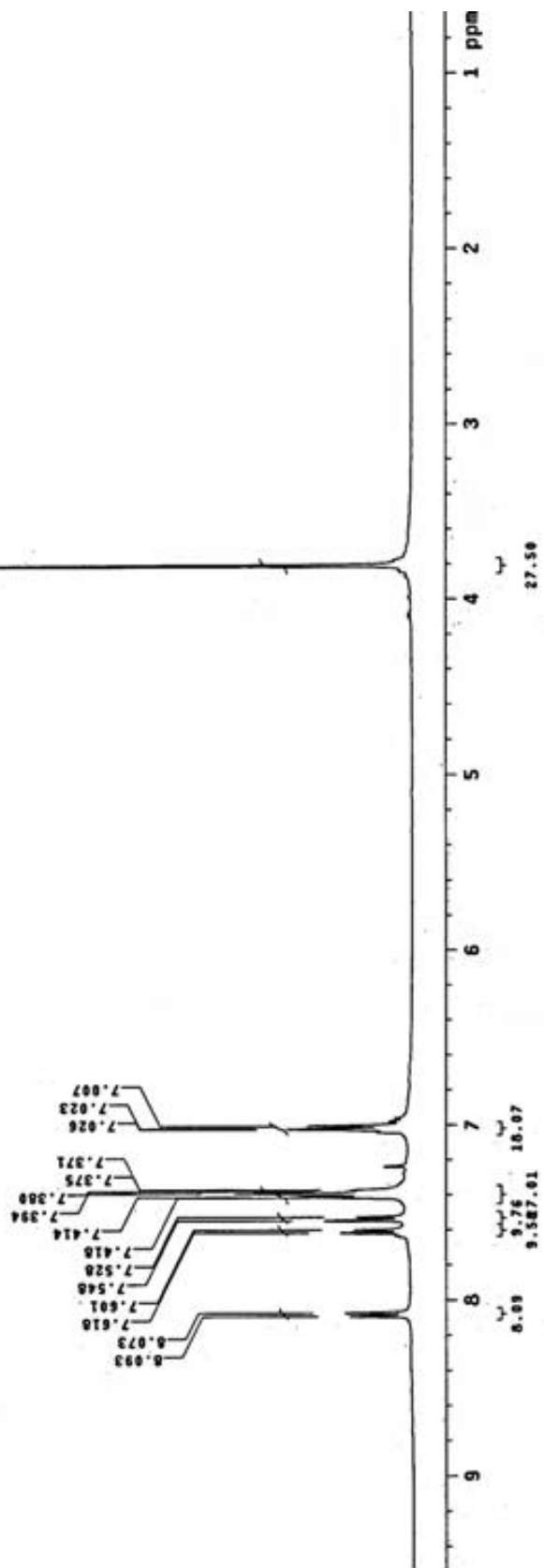


Table 3, entry 2

```

RP-287
expl s2pu1
SAMPLE
date Nov 17 2011
solvent CDCl3
ACQUISITION
sv 6388.8
at 3.250
pb 2520
bc not used
dl 1.000
nt 32
ct TRANSMITTER H1
tn 0.10
sfq 399.853
tof 382.9
tpr 3618.7
pv DECOUPLER C13
dn 8
dof 8
dm nnn
dms C
dpcr 58
cmf 15988
SPECIAL
temp not used
gain not used
hplm not used
hpln 8.888
hplp 18.788
d17a 28.608
n n
y y
mn mn
PROCESSING
lb 0.10
fn 5538
DISPLAY
sp 243.8
wp 3618.7
rft 3780.7
rfp 2684.4
rp 182.3
lp -85.7
wc 250
sc 91
ve 91
nm cdc
ph 3
    
```



```

RP-287
exp1 s2pu1
SAMPLE
date Nov 15 2011 temp not used
solvent CDCl3 gain not used
file exp not used
ACQUISITION
sw 25125.6 ppg 16.600
at 1.195 a1ya 20.000
pp 6270 11
pb 13000 11
dt 1.000 in
nl 7000 ns
ct 1000 ns
PROCESSING
tn TRANSMITTER 1b 2.00
fr 6556
sfrq 100.554 fr DISPLAY
tof 1536.3 sp 1682.7
tqvr 61 wd 17035.9
pw 9.300 rfp 9284.4
DECOUPLER HI rfp 7264.9
dn 6 lp -84.0
da yvy wc -271.4
dss y wc 250
davr 42 sc 11
dfr 6900 th no ph 2
    
```

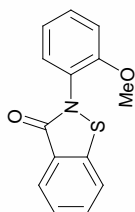
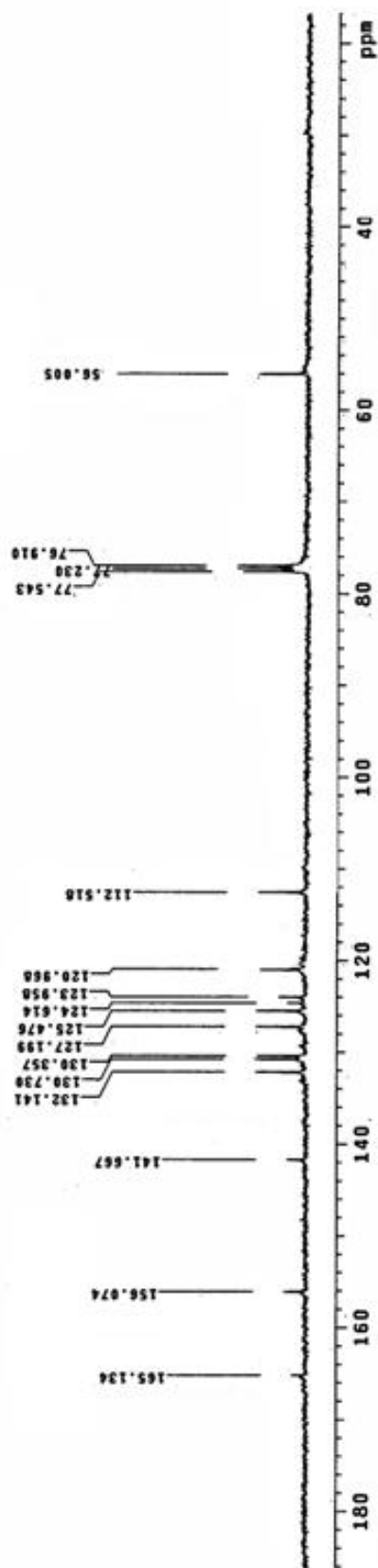


Table 3, entry 2



RP-289
 exp1 s2pu1

date	Sep 16 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION	exp	hist	9.000
sw	6309.8	pu99	19.700
et	1.898	al1a	20.000
np	25528	FLAOS	
fb	not used	l1	n
be	4	in	n
dl	1.000	y	y
nt	32	hs	mn
CT	TRANSMITTER 28	lb	0.10
tn	399.853	fn	65536
frq	399.853	sp	187.3
cor	362.9	wp	3045.5
lper	27	rf1	3880.3
pv	8.059	rfp	2894.9
DECOUPLER	Cl3	tp	113.6
dn	nmr	lp	-66.4
dof	nmr	VC	250
dwa	50	VC	151
dwr	15980	th	20
dat		nm	cdc. ph

SPECIAL
 temp not used
 gain not used
 spin not used
 hist 9.000
 pu99 19.700
 al1a 20.000
 FLAOS
 l1 n
 in n
 y y
 mn mn
 PROCESSING 0.10
 lb 65536
 fn DISPLAY 187.3
 sp 3045.5
 wp 3880.3
 rf1 2894.9
 rfp 113.6
 tp -66.4
 lp PLOT 250
 VC 151
 th 20
 nm cdc. ph

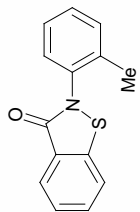
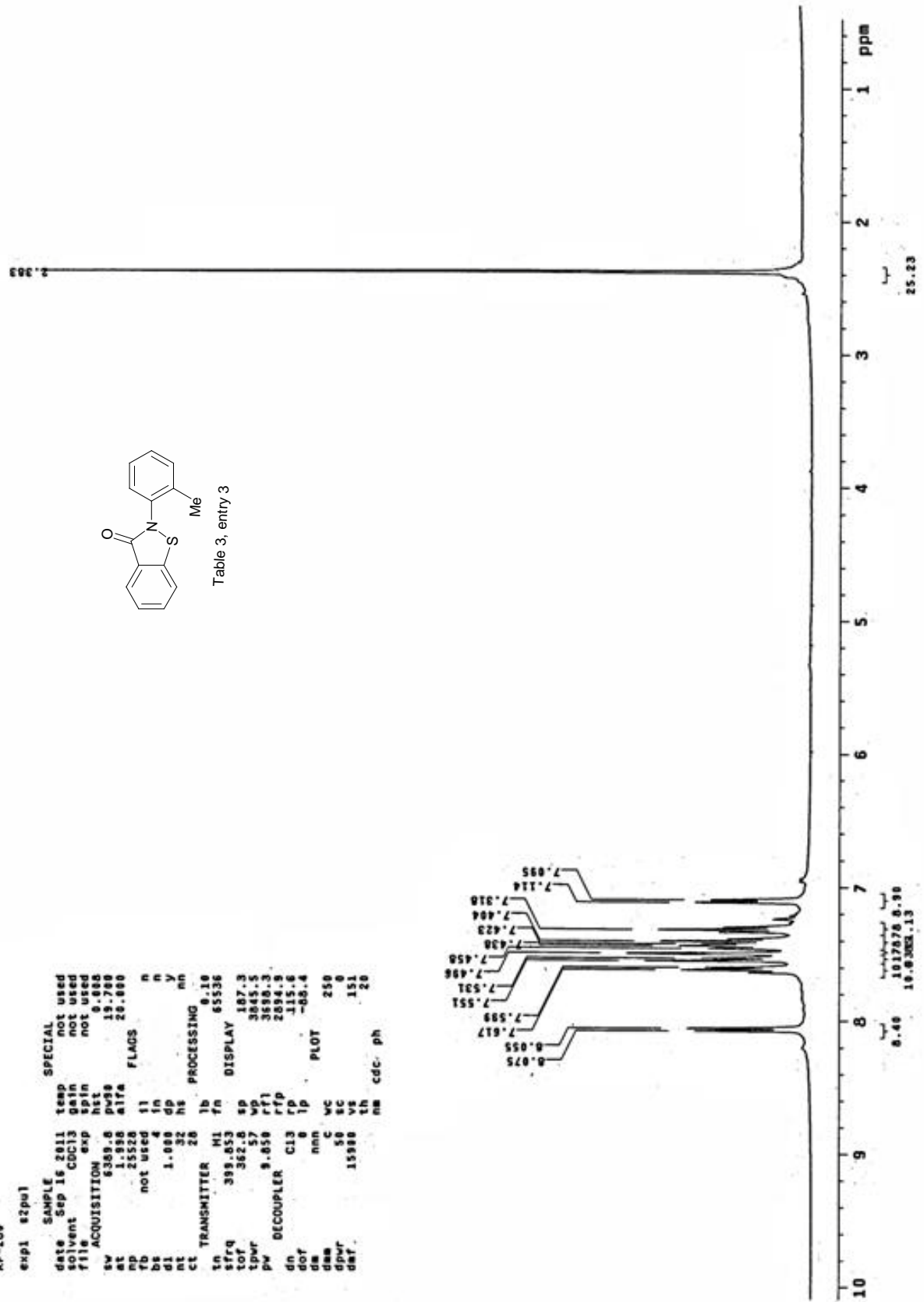


Table 3, entry 3



```

RP-280a
exp1 s2pu1
SAMPLE
date Sep 26 2011 temp not used
solvent CDC13 gain not used
file CDC13 exp file not used
ACQUISITION
sw 5125.6 pwr0 18.600
at 1.199 a1r4 20.000
np 68270
fb 13600 11
bs 16 1n n
d1 1.000 dp y
nt 7000 hs
ct TRANSMITTER 688 1b PROCESSING nm 2.80
tn TRANSMITTER C13 fn 65536
strq 100.554 sp DISPLAY
tof 1536.3 ep -1503.1
tpwr 61 wp 25125.6
pw 9.300 rfl 1503.1
DECOUPLER H1 rfp 0
do 0 rp -69.8
dof 0 lp -271.4
ds yyy w VC 250
dss w SC 0
dpr 42 tc 0
dar 8900 vs 17
nm no ph 2
    
```

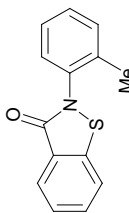
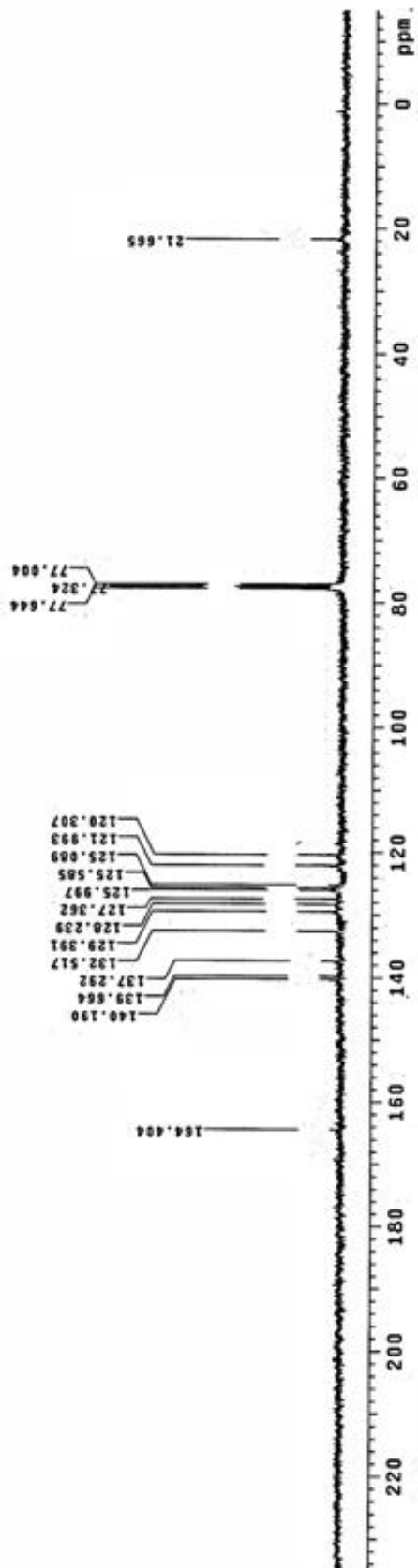


Table 3, entry 3



```

RP-251a
expl stdih
SAMPLE
date Oct 25 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ not used
clftemp/RP-252a-1C~ hst 0.908
1.1fid pw98 13.700
ACQUISITION alfa 20.000
sw 6006 0
sc 2395 11
fb 2394 1n
not used dp
bs 4 hs
d1 1.000 fn not used
ct 32 DISPLAY 162.3
TRANSMITTER H1 wp 3551.3
sfrq 399.853 rfl 3869.1
tof 0 rfp 2894.9
tpwr 57 rp 99.2
pw 7.000 lp -76.9
DECOUPLER C13 WC 250
dn 0 SC 0
dof 0 SC 48
dm nnn vs
dnn C th
dpwr 50 nm cdc ph
dnt 15900
    
```

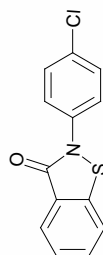
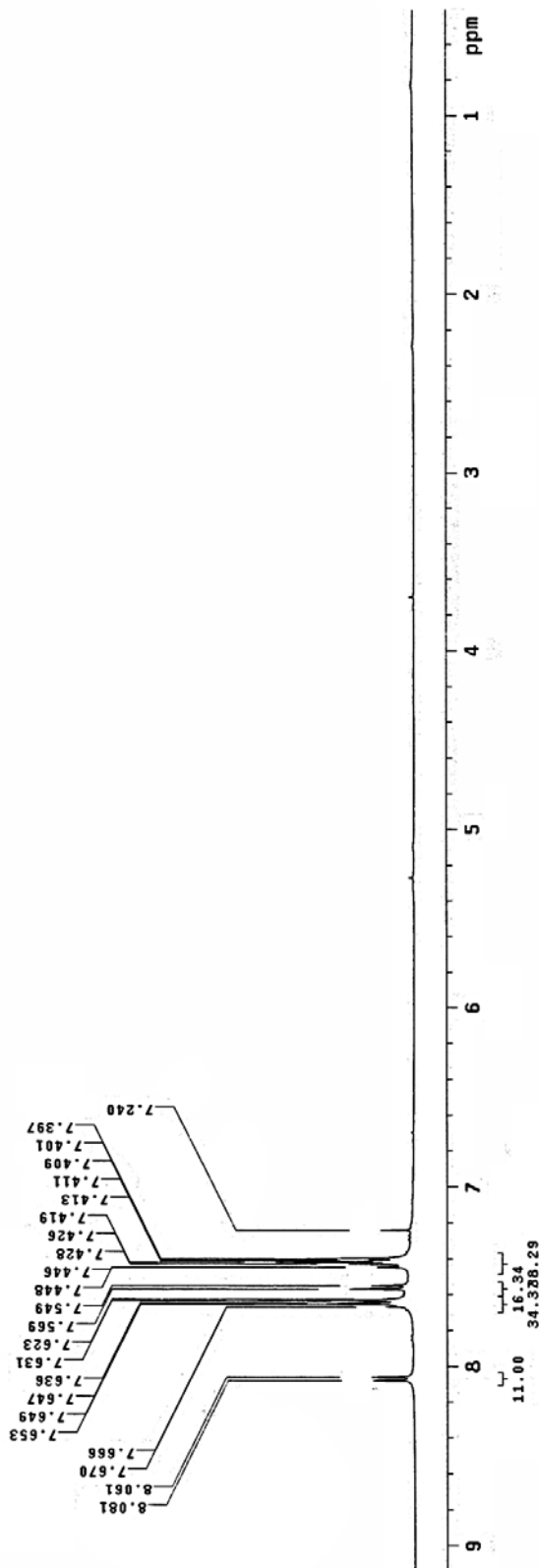


Table 3, entry 5



```

RP-251b;
expl s2pul
SAMPLE
date May 31 2011
solvent CDCl3
file not used
ACQUISITION
sv 25125.6
st 1.155
np 60270
pb 13000
bs 100
di 1.000
nt 15000
ct 15000
SPECIAL
temp not used
gain not used
spin not used
hst 8.000
pvs0 10.000
a17a 20.000
FLAGS
n n
n n
y y
nr nr
PROCESSING
lb 2.00
fn 6536
DISPLAY
sp -174.0
wp 22007.6
rf1 1627.1
rfp 0
MI 0
lp -33.5
-371.5
PLOT
wc 250
sc 0
vs 1172
th no
a1 ph
    
```

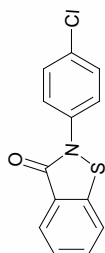
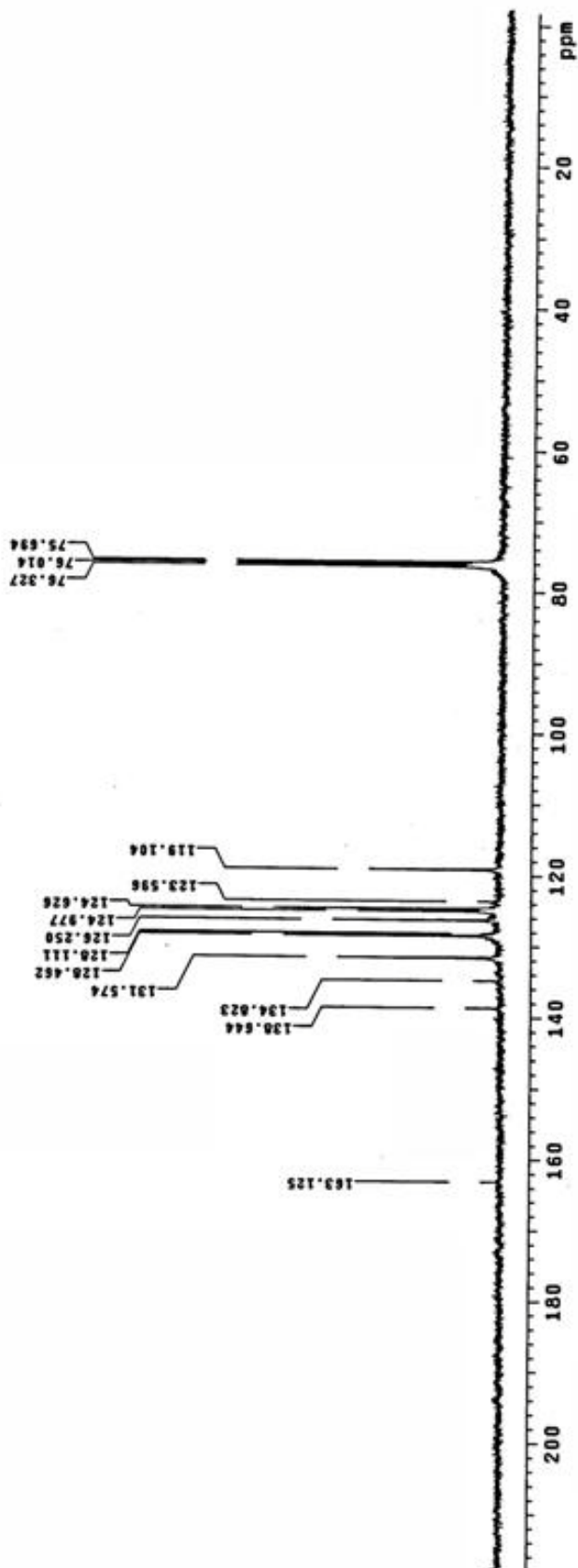


Table 3, entry 5



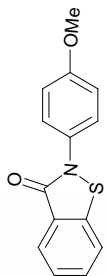
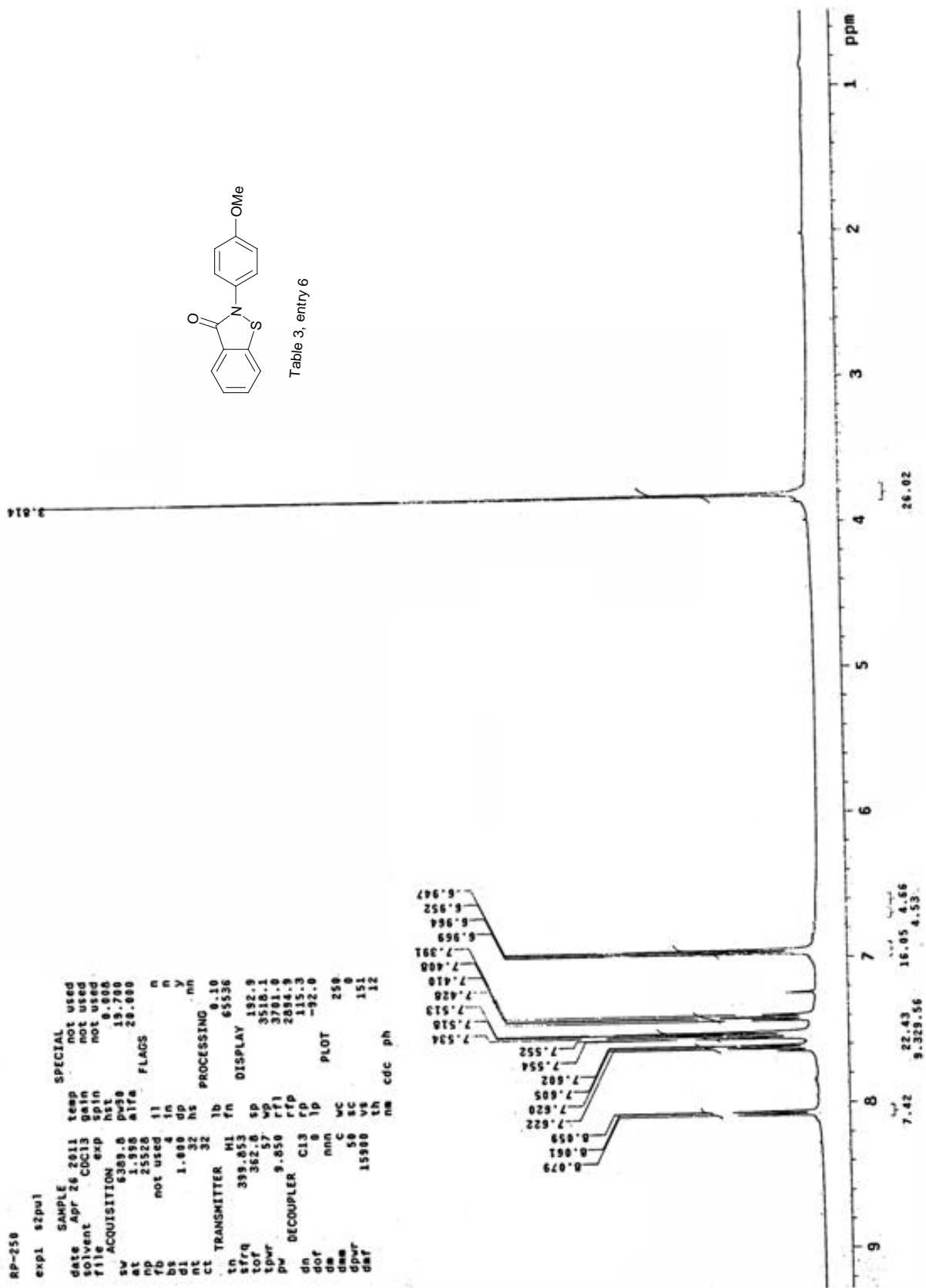


Table 3, entry 6

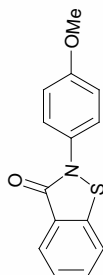
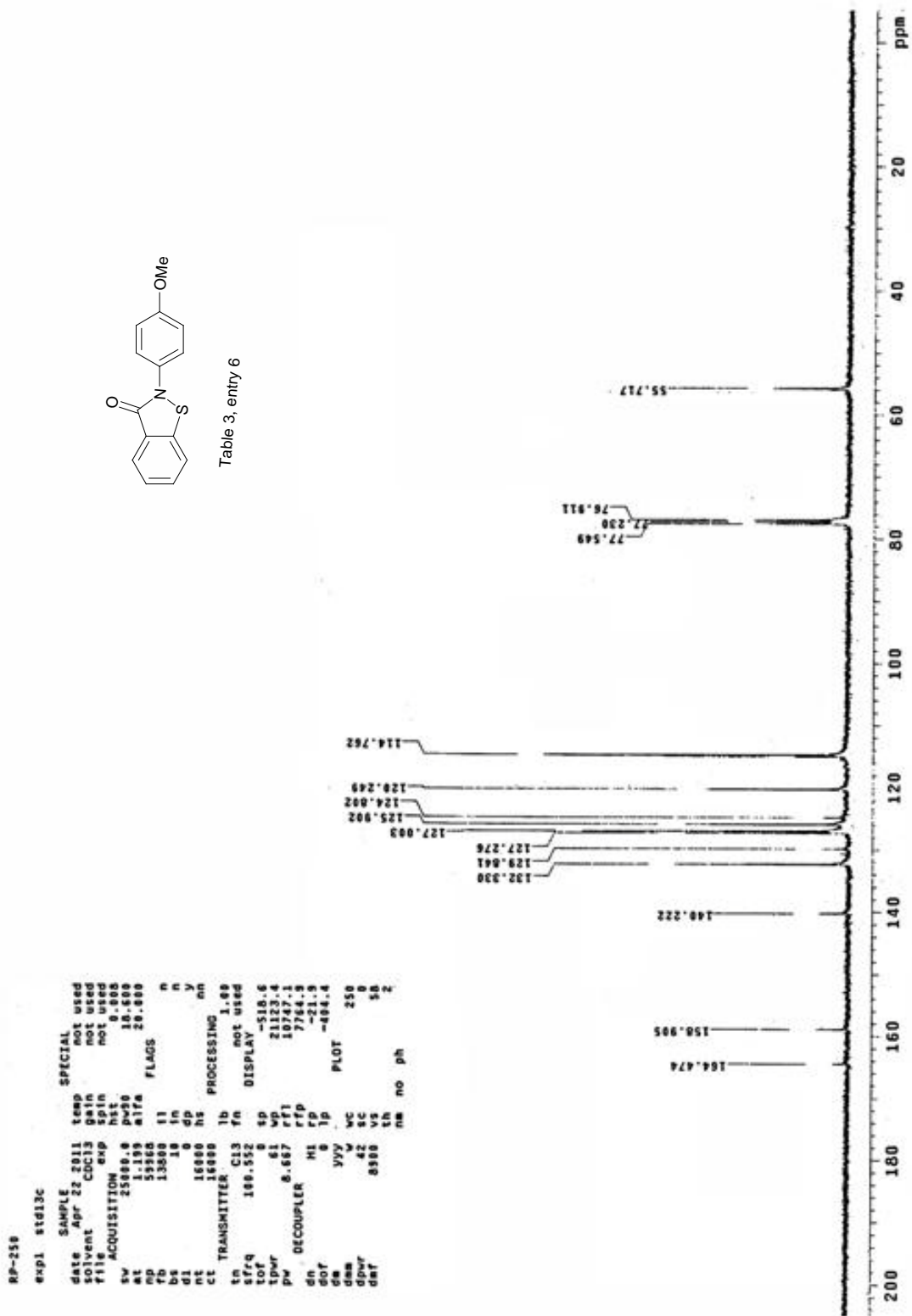


Table 3, entry 6



```

RP-251a
exp1 szpu1
SAMPLE SPECIAL
date Jun 16 2011 temp not used
solvent CDCl3 gas1 not used
file exp file not used
sw 6389.8 pw90 hst 0.008
at 1.998 a1fa 20.000
np not used f1 n
bs 4 in n
d1 1.000 dp y
nt 32 hs mn
ct TRANSMITTER 32 PROCESSING 0.10
tn HI fb 65536
sflq 399.853 fn DISPLAY 93.4
tof 362.8 sp 3434.1
tpwr 57 wf1 830.1
pw 9.850 rf1 rfp 116.4
dn DECOUPLER C13 rp -86.4
dm nnn tp PLOT 250
dms C 50
dpr 15900 sc 74
dat th nm cdc ph 8
    
```

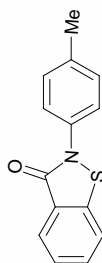
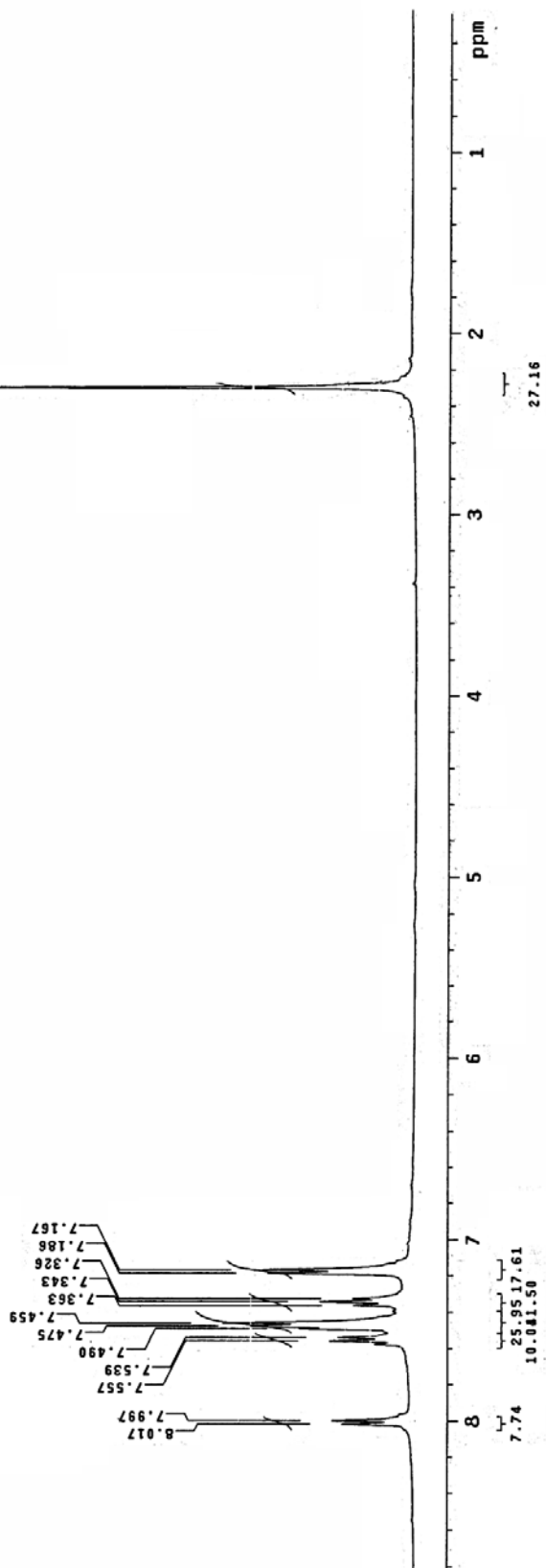


Table 3, entry 7



```

RP-25
exp1 s2pu1
date SAMPLE SPECIAL
solvent CCl3 gain not used
flig CCl3 exp not used
ACQUISITION exp not used
nt 10.000
st 25125.6
at 48270.8170
pd 48270.8170
pb 13882.11
bs 1.000 in n
dl 1.000 dd v
nl 7680 ht mn
ct TRANSMITTER 2000 lb PROCESSING 2.00
tn C13 fn 65536
sfrq 106.554 fn DISPLAY
tof 1536.3 sp -1585.7
tpr 61 wp 25125.6
pw 9.300 rf 9270.6
DECOUPLER H1 rfp 7764.9
dn 0 lp -2.7
dm VVY WC -460.5
dms W WC 250
dpr 42 SC 0
dfr 6900 th VS 20
mm no ph 2
    
```

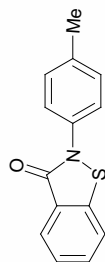
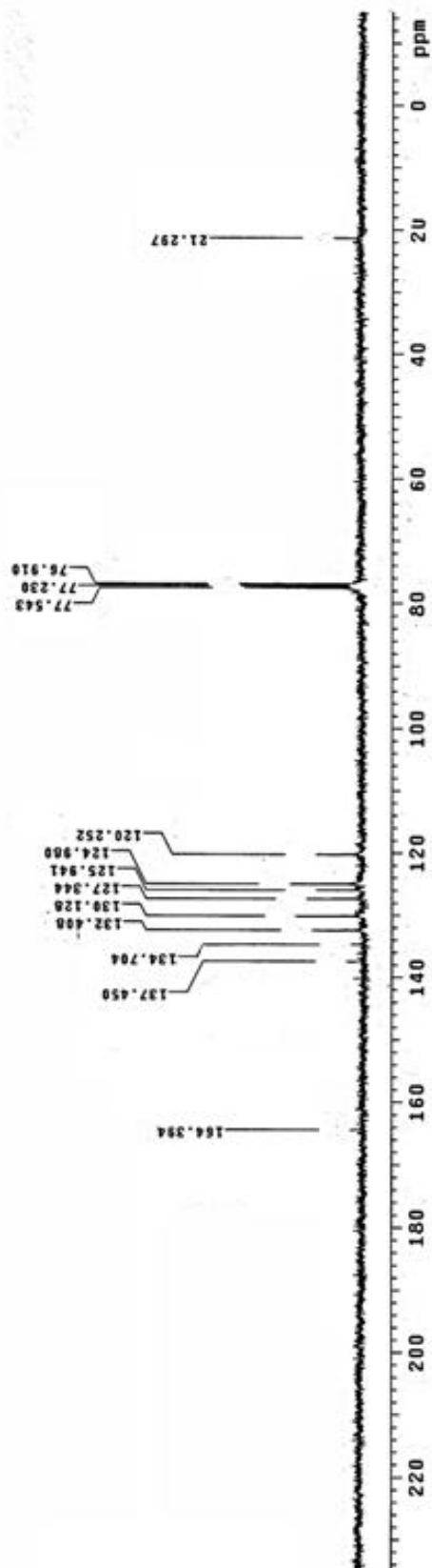


Table 3, entry 7



```

RP-262
exp1 s2pu1
date SAMPLE Jun 15 2011 temp not used
solvent CDC13 gain not used
file file not used
ACQUISITION exp hst 0.008
sw 6309.8 pps0 13.700
at 1.998 m174 26.000
np not used i1 n
bs 25528 4 n
d1 1.000 dp n
nt 32 hs PROCESSING nn
ct TRANSMITTER 32 lb 0.10
tn 399.853 fn 65536
sfrq 362.8 sp 102.6
tpwr 57 wp 3727.9
pv 9.850 rfl 795.8
DECOUPLER C13 rf0 0
dn 0 lp 111.2
dm nnn C PLOT 250
dsw 50 SC 0
dpr 13900 vs 86
dar ma cdc ph 7
    
```

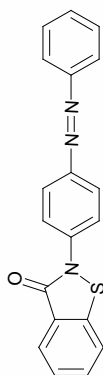
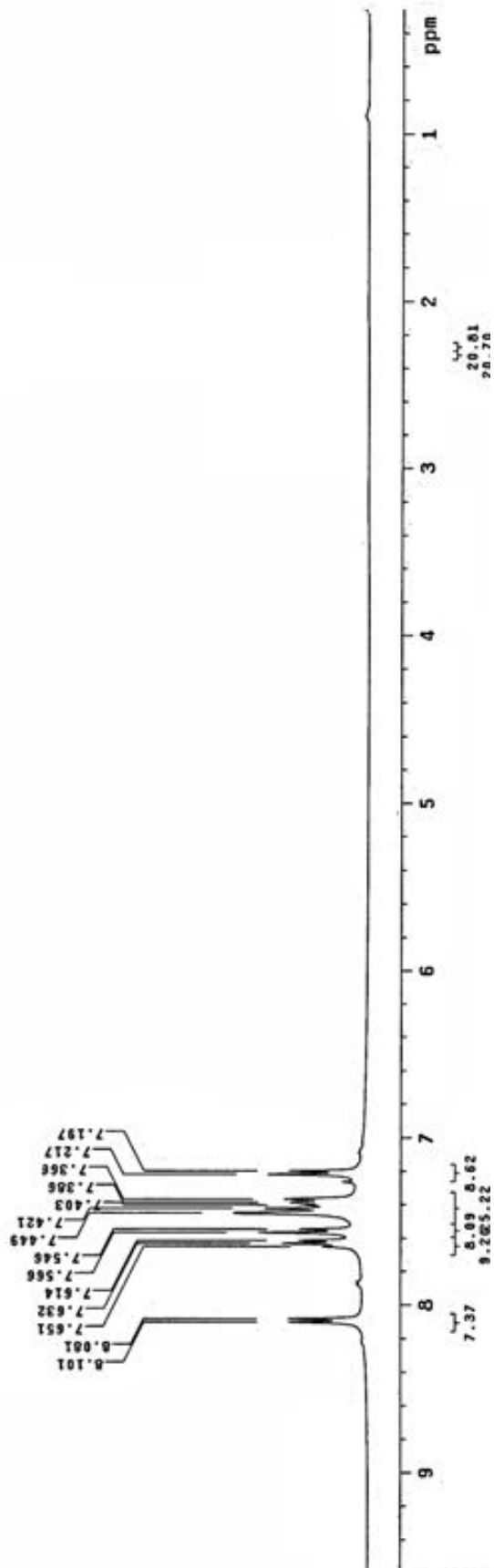


Table 3, entry 9



```

RP-262
exp1 szpul
date SAMPLE temp SPECIAL
  Jun 12 2011 not used
solvent COCl3 gain not used
file not used
ACQUISITION exp
  25125.6 P450 16.600
  1.195 d1fa 28.600
  6276 11 FLAGS
  15800 11 n
  1 480 11 n
  1 1200 11 y
  1 1200 11 n
  11300 11 n
  CT TRANSMITTER C13 2.00 PROCESSING nm
  TO 100.55 P1 65536
  FREQ 1536.3 EP -351.6 DISPLAY
  1536.3 EP 2025.6
  1536.3 EP 2025.6
  PV DECOUPLER 8.300 P1 7572.1
  8.300 P1 7572.1
  8.300 P1 7572.1
  dn HI 1p PLOT
  dof 0 1p -374.2
  dm yyy WC 250
  dm y WC 250
  dm 42 VC 25
  dm 8000 VA 25
  dm
  nm no ph
    
```

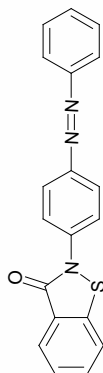
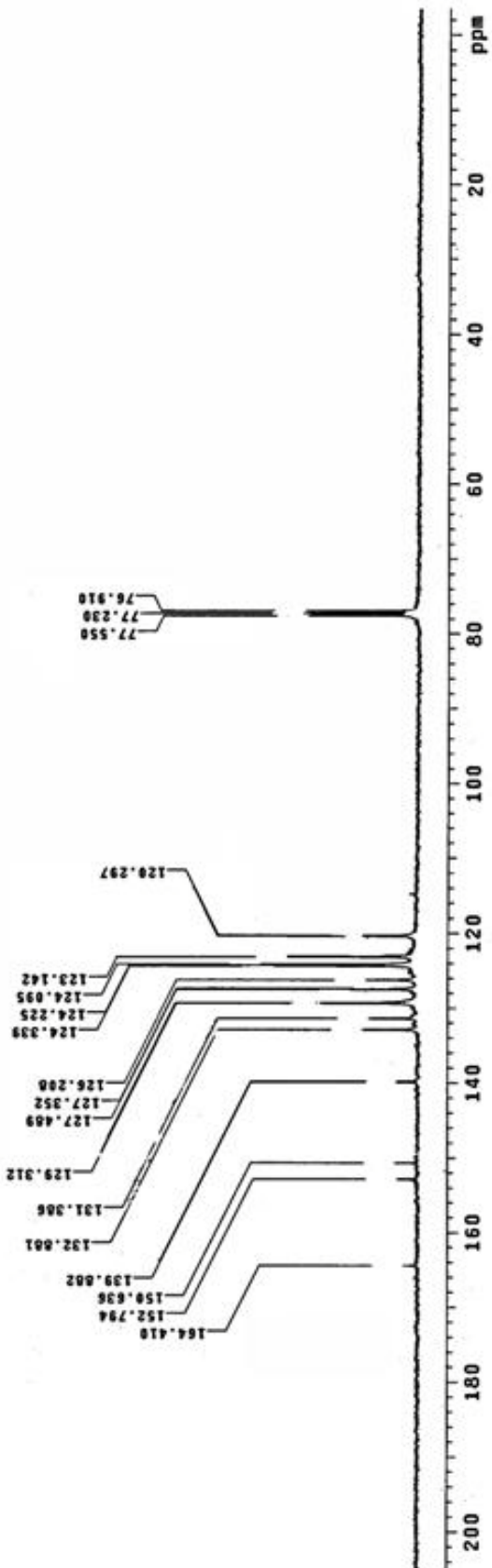


Table 3, entry 9



RP-255
 exp1 f2pul

date	Jun 11 2011	Temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION	exp	hst	8.808
sw	6389.8	pw30	19.708
at	1.998	altA	20.000
NP	25520	FLAGS	
fb	not used	l1	m
bs	4	l2	m
dl	1.000	dp	y
nt	32	hs	nn
ct	32	PROCESSING	
TRANSMITTER	lb	fb	0.10
tn	H1	fn	65536
sfrq	399.853	DISPLAY	
tof	362.0	sp	83.7
tpwr	57	wp	3803.6
pv	9.850	rf1	839.9
DECOUPLER	C13	rfp	8
dn	0	rp	116.0
ds	0	lp	-81.0
dm	minn	WC	250
dpr	C	SC	8
dat	15900	VS	83
		nm	cdc
		ph	5

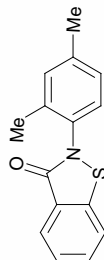
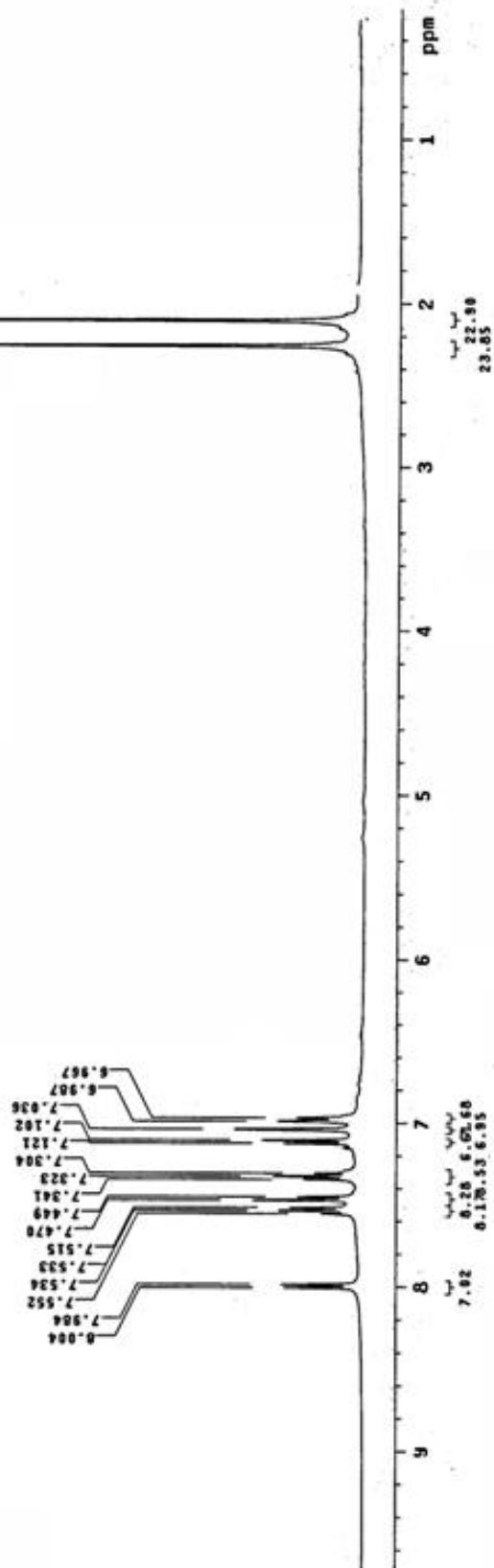


Table 3, entry 10



RP-254
 exp1 szpu1

SAMPLE		SPECIAL	
date	Jun 11 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
sw	25125.6	nu2	8.000
at	1.199	atfa	18.000
nd	6270	atfb	20.000
nb	15000	fl	
bs	64	fn	
dl	1.000	dp	
nt	8000	hs	
ct	5312	mn	
tn	TRANSMITTER	2.00	PROCESSING
sfreq	C13	fn	65536
tof	100.554	sp	297.1
tpwr	1536.3	wp	1852.5
pw	9.300	rf1	9283.6
deco	DECOUPLER	rfp	7764.9
dn	H1	rp	-57.7
da	0	lp	-332.5
dsm	VVY	vc	258
cpwr	42	sc	0
daf	0900	vs	30
		th	no
		ph	3

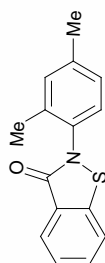
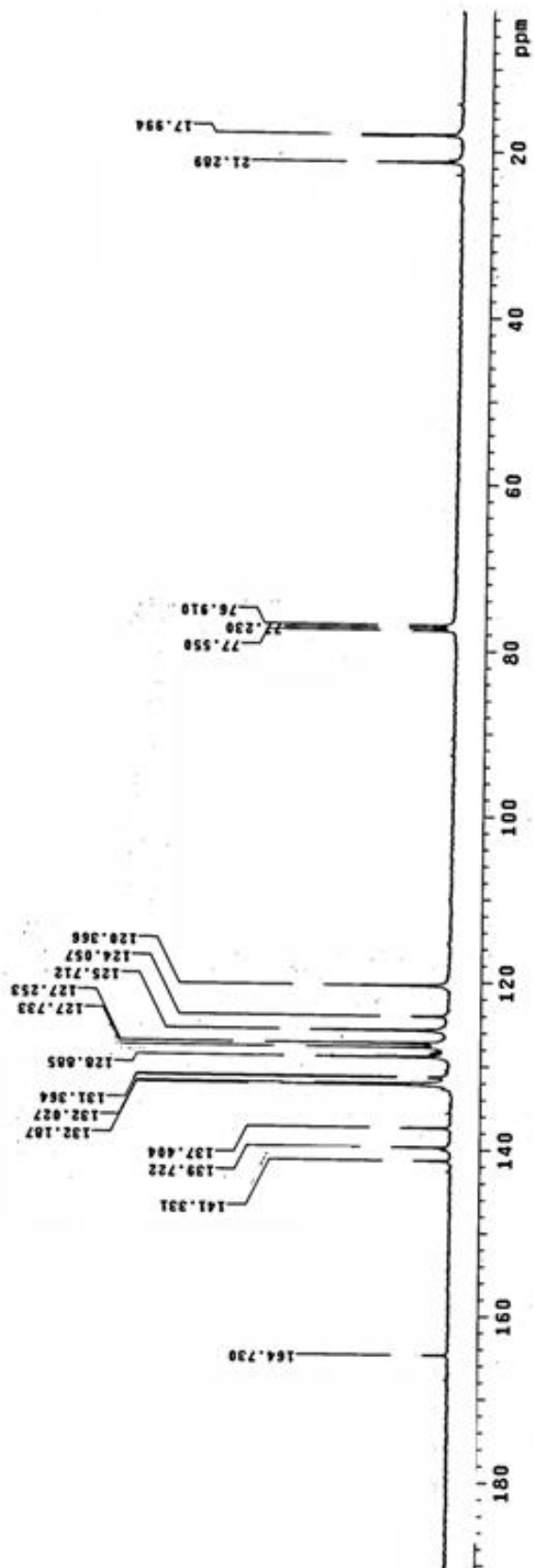


Table 3, entry 10



```

RP-255
exp1 s2pu1
SAMPLE
date Jun 21 2011 temp not used
solvent CDC13 gain not used
file /export/home/~ spin not used
ciftemp/RP-255.fid hst 0.008
ACQUISITION pw90 19.700
SW 6389.8 a1fa 20.000
at 1.998 n n
np 25528 l1 n n
7b not used in n y
bs 4 dp y
d1 1.000 hs
nt 32
ct TRANSMITTER 32 lb 0.10
fn 65536
tp H1 DISPLAY
sfrq 399.853 SP 196.8
tof 362.8 wd 4164.6
tpr 57 rfl 3697.1
pw 9.850 rfd 2894.9
DECOUPLER c13 lp 117.7
-93.7
dn dof
dm mnn wc 250
dm c 0
dpr 50 vs 151
dat 15900 th nm cdc ph
    
```

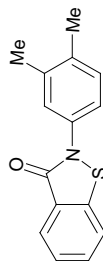
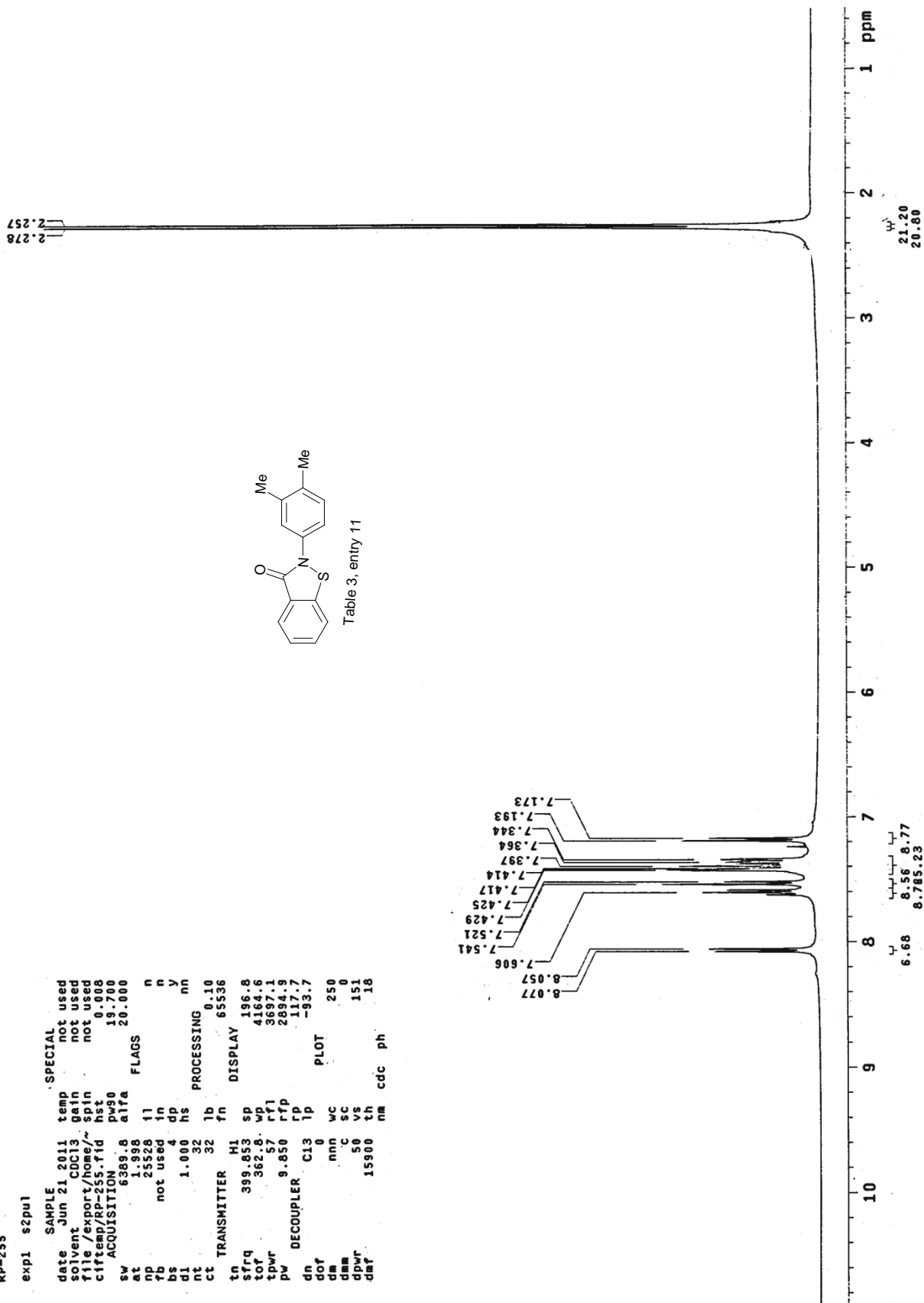


Table 3, entry 11




```

RP-255
exp1 s2pu1
SAMPLE
date Jun 21 2011 temp
f1 solvent CDCl3 gain not used
f2 f1 port/beamsc- spin not used
c1 ftemp/RP-255-410 hst 8.000
ACQUISITION f1d 8178 18.000
sv 25125.6 20.000
at 1.100 11
np 60270 in n
fb 13000 dp y
bs 100 hs
dl 1.000 2.00
nt 7000 lb
ct 300 fm 855.0
TRANSMITTER C13 SP
f1rq 180.554 wp -1513.3
tof 1538.3 rfi 25125.6
tper 41 rfp 9278.2
pv 9.300 tp 7684.9
DECOUPLER M1 PLOT -50.6
dn 8 MC -271.4
dof 8 VC 250
dm 8 SC 8
dmw 42 th 13
dsvr 42 th 13
dmf 8000 na no ph 2
    
```

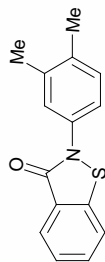
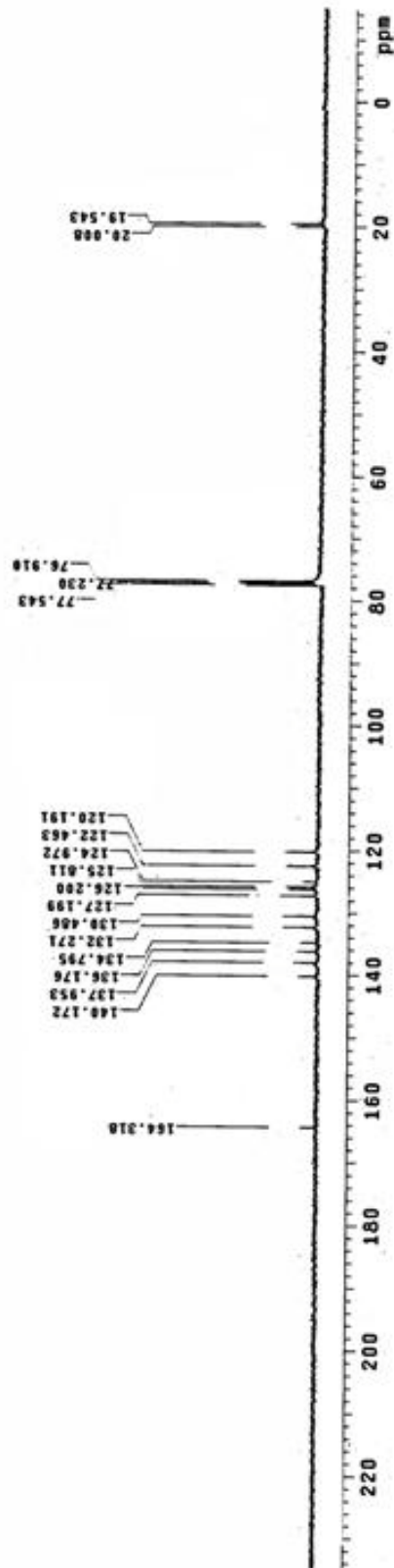
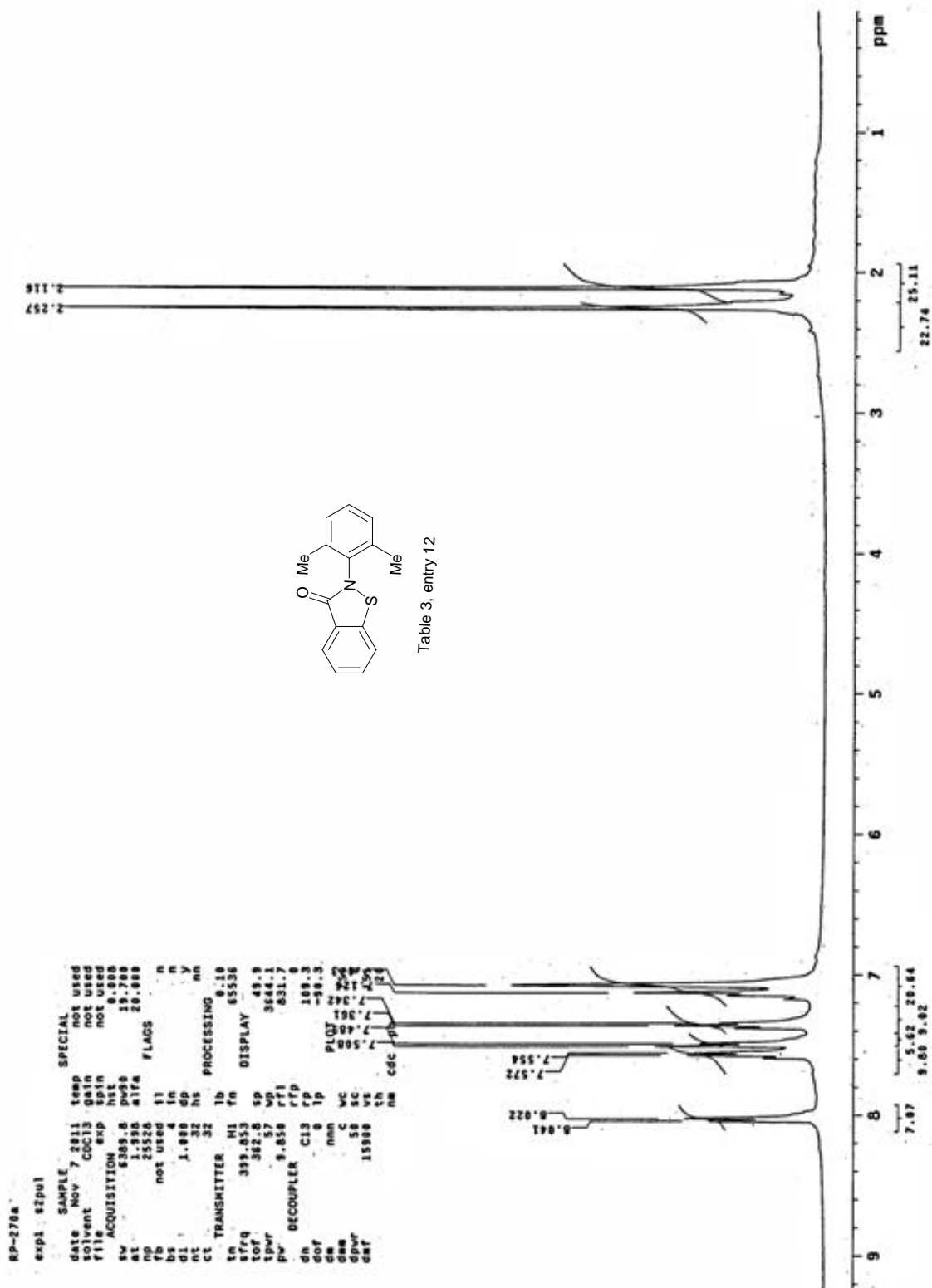


Table 3, entry 11





```

RP-270a
exp1 szpu1
SAMPLE 7 2011 temp not used
date Nov CDCl3 gasIn not used
solvent /export/home/~ spIn not used
file /export/RP-270a-13- hst 8.488
ciftemp/RP-270a-13- pvar8 18.488
C-7fd C-7fd 28.488
ACQUISITION alfa
SW 25125.6
ALFA 28.488
SPECIAL
at 1.199 11 n n
np 68278 1n n n
fb 13888 -dp y y
bs 16 hs
dl 1.000 1b
nt 7088 1b
ct TRANSMITTER C13 SP DISPLAY -653.8
IN 188.554 4p
17fQ 188.554 4p 18413.8
tof 1532.3 4f1 8176.7
tprv 61 4fp 7564.9
pw 8.358 1p -64.1
DECOUPLER H1 PLOT -271.4
dn 0 VC 258
dof 0 VC 258
da yyv sc 16
dam yyv vs 14
dprv 42 th
dat 8388 nm no ph
    
```

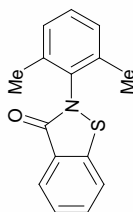
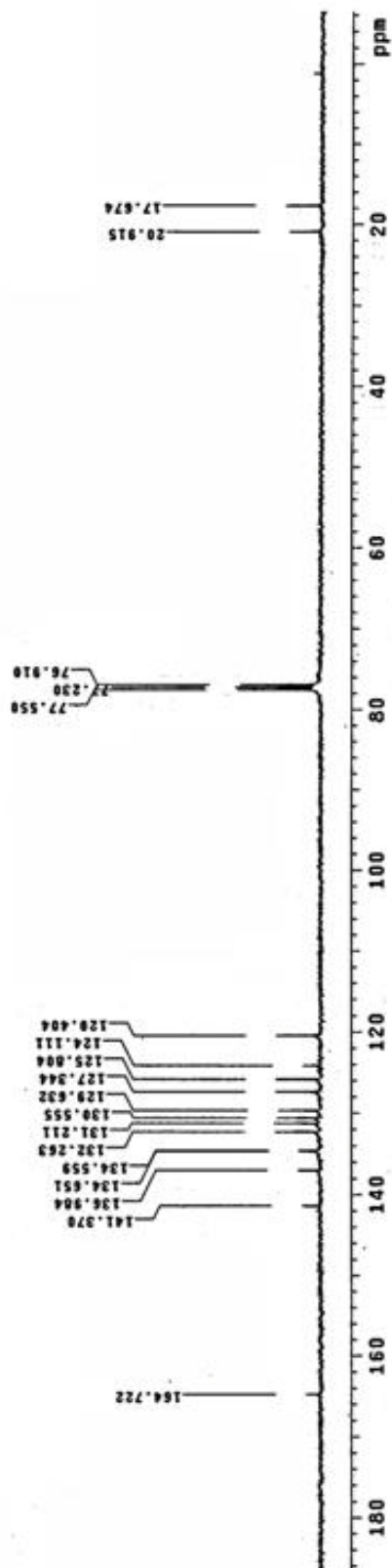


Table 3, entry 12



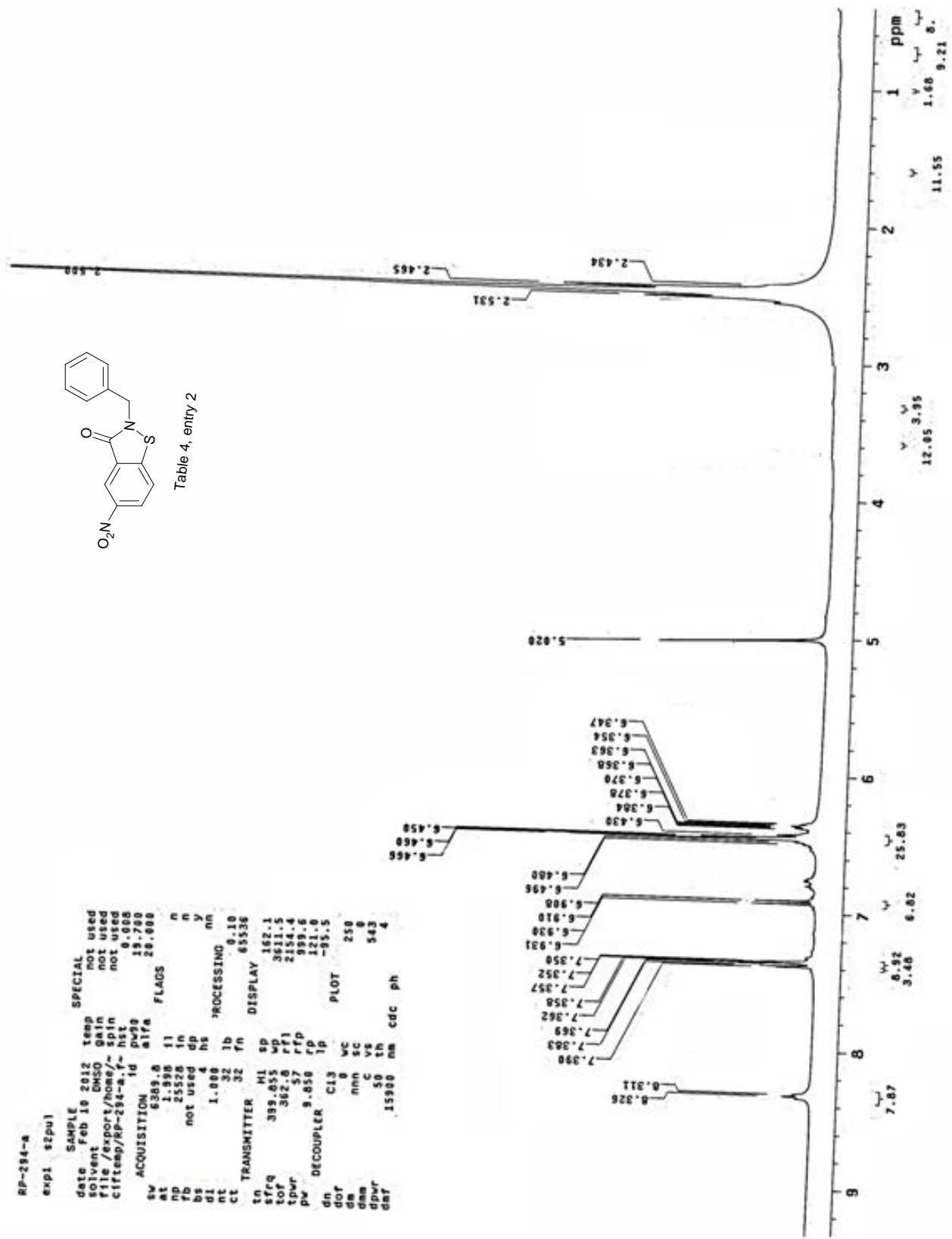


Table 4, entry 2

```

RP-234-a
expt s2pu1
SPECIAL
date Feb 10 2012 temp not used
solvent DMSO gain not used
file /export/home/~ spin not used
ciftemp/RP-234-a.f-hst 0.008
ACQUISITION id pw2g 15.780
          alfa 24.000
sw 6309.0
at 2526.0
nd 2526.0
fb not used
bs not used
d1 1.080
nt 32
ct 32
TRANSMITTER H1 SP DISPLAY 162.1
          hf 389.855
          tof 362.6
          tpr 57
          pw 9.850
DECOUPLER C13 PLOT -95.5
          dn 0
          dm nnn
          dan c
          dpr 50
          daf 15900
          ma cdc
          ph 4
    
```

```

RP-284a
expl szpul
SAMPLE date Feb 2 2012 temp not used
solvent DMSO gain not used
file /export/home/~ not used
ciftemp/RP-284a-13-hst 0.000
C.Fid pw30 18.600
ACQUISITION a1fa 20.000
sv 25125.6 f1
ns 5272 f2
fb 15000 dp
bs 100 hs
dl 1.000 nt
nt 10000 lb 2.00
ct 4500 f2 6556
TRANSMITTER C13 sp -980.0
sfreq 100.554 wp 1545.6
tor 1536.3 rfi 1503.1
tpr 0.61 rfp -85.9
pw 8.300 lp -389.4
DECOUPLER H1
dnt 6 wc PLOT 250
dof 6 sc 0
dm yyv vs 54
dwm 42 th nm 2
dpr 8900
dnt nm no ph
    
```

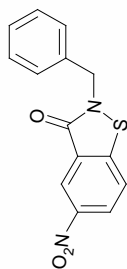
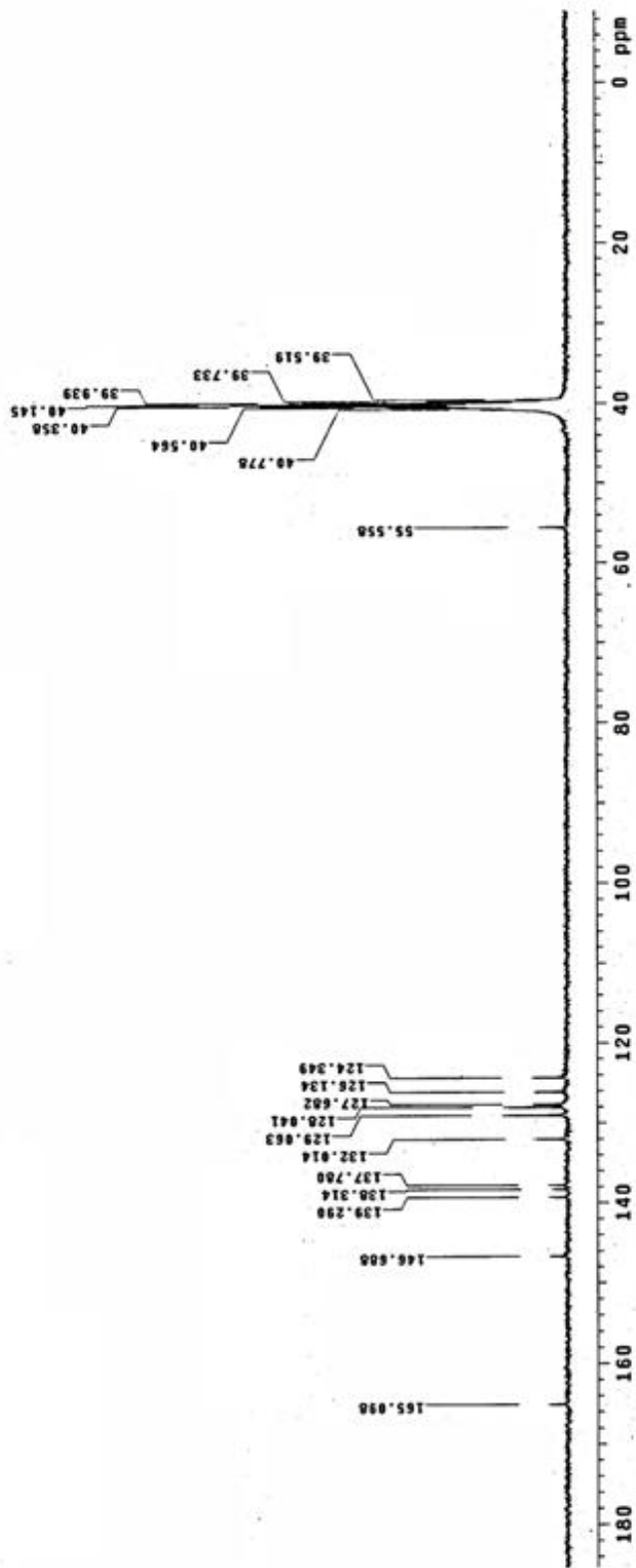


Table 4, entry 2



```

SM-26
exp1 s2pu1
SAMPLE
date Feb 9 2012 temp not used
solvent CDC13 gain not used
file CDC13 exp not used
ACQUISITION
sw 6309.6 pvs0 0.008
at 1.998 m176 19.700
np 25520 20.000
bs not used 11
di 1.000 dp 4
nt 32 hs
ct TRANSMITTER 32 PROCESSING mn
tn 0.10
trf9 399.053 HI fb 65536
tof 362.0 sp DISPLAY 137.7
tpr 57 wd 3711.2
pv 0.039 rfi 3837.5
DECOUPLER C13 rp 2084.9
dn 187.3
dof mn mc ip PLOT -87.4
dca nnc wc 258
dss No SC 34
dpr VS 34
dnt 15000 th cdc pn 3
    
```

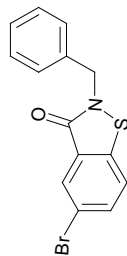
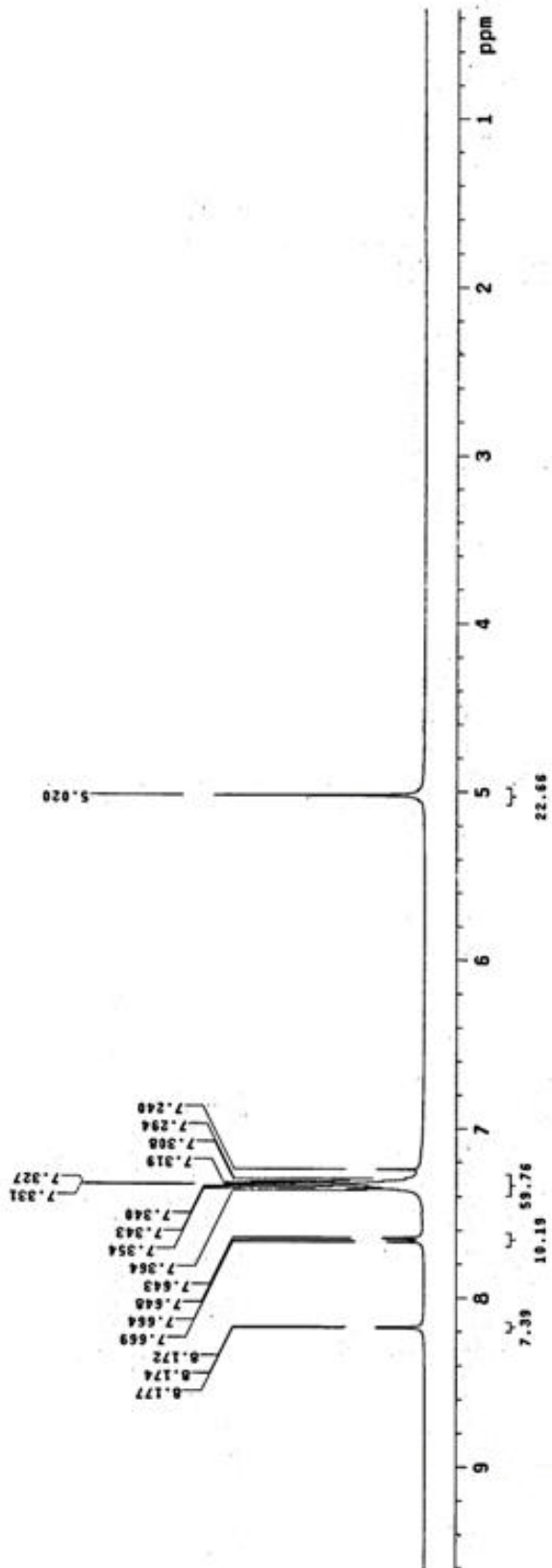
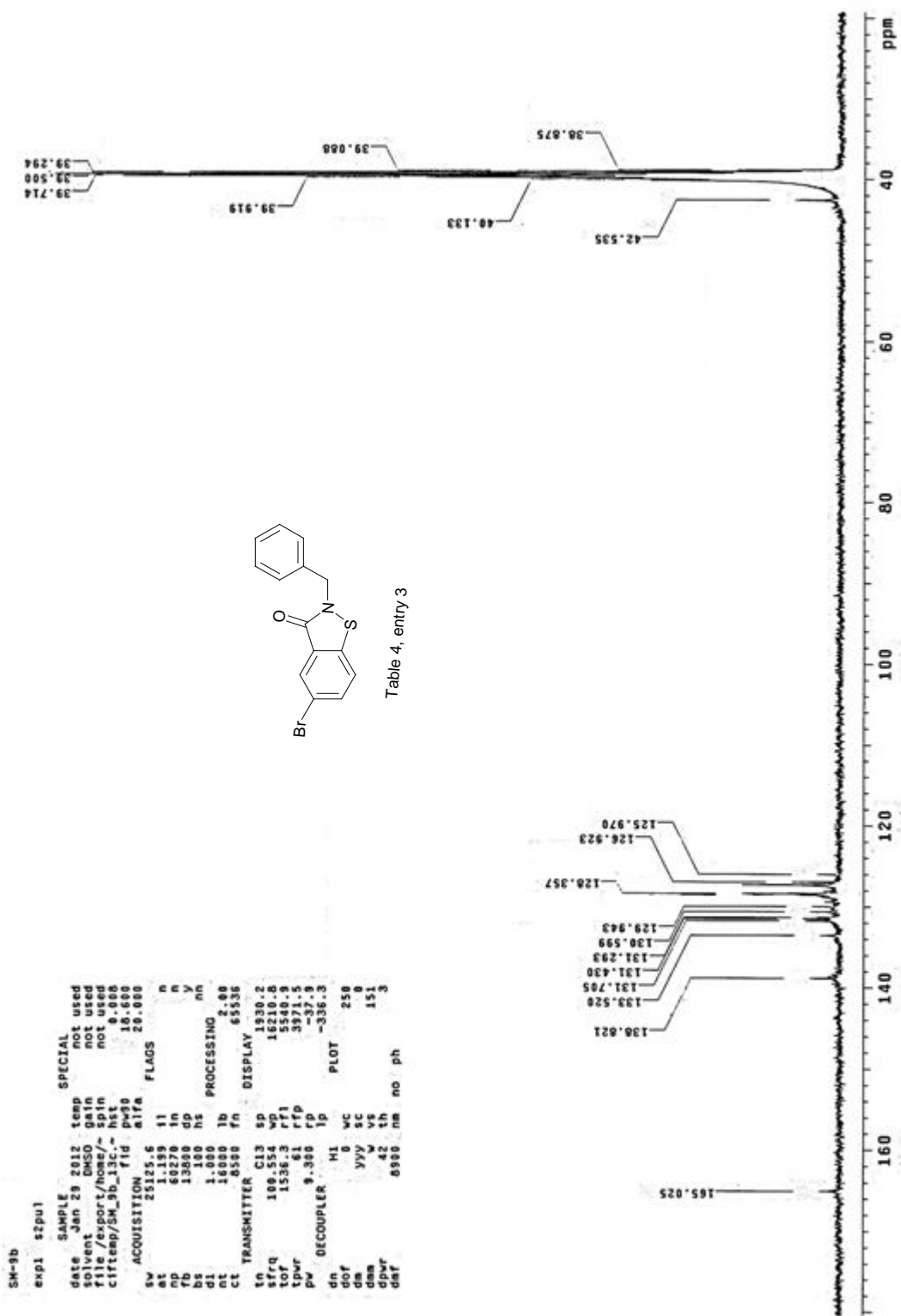


Table 4, entry 3





RP-234b

exp1 szpu1

```

SAMPLE SPECIAL
date Jan 4 2012 temp not used
solvent DMSO gain not used
file /export/home/~ spin not used
ciftemp/RP-234b.f1- hst 0.008
pv20 19.700
pv20 19.700
pv20 20.000
ACQUISITION d a1fa 20.000
sv 6289.0 FLAGS
at 1.998 11 n
np 2520 11 n
rb not used dp y
bs 4 hs PROCESSING nm
dl 1.000 32 fb 6536
rt 32 fr 6536
CT TRANSMITTER M1 SP DISPLAY 201.1
sfrq 399.855 wp 3026.0
tof 382.0 rfi 1789.2
tpwr 57 rfp 999.6
pv DECOUPLER C13 PLOT -87.3
dn dof wc 250
de nnn sc 0
das c vs 94
dpr 50 th 10
daf 15900 nm cdc ph
    
```

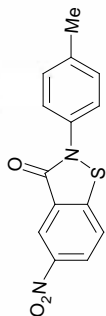
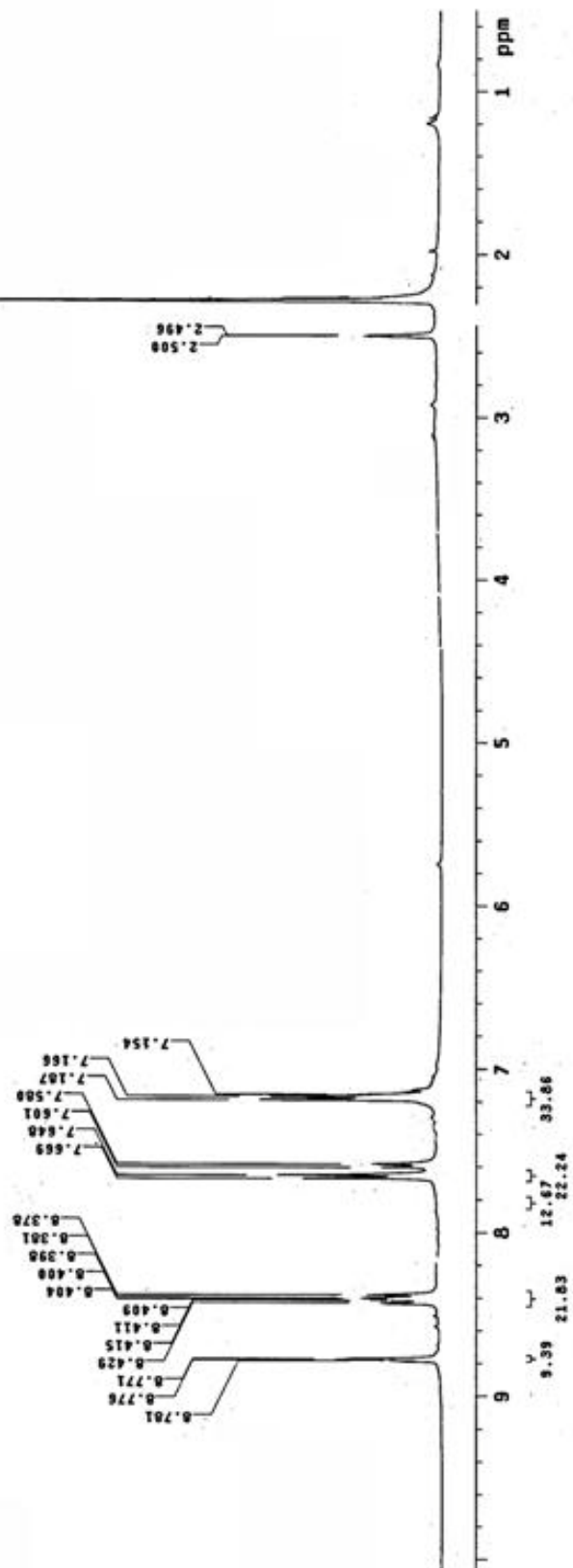


Table 4, entry 6




```

RP-294b
expl szpu1
SAMPLE
date Jan 16 2012 temp not used
solvent DMF0 gain not used
c1temp/RP-294b-13-ht not used
c1temp/RP-294b-13-ht 8.828
C.fid Dv88 10.600
ACQUISITION: alpha 20.000
SW 25125.6
AL 1.199 II
PD 1.199 n
TP 1.199 n
BS 1.199 n
DL 1.000 HS
DI 1.000 HS
NT 10000 lb
CT 13000 FN
TRANSMITTER CL3 SP
SFRQ 100.514 W
TOF 25125.6
TPR 1536.3 rfl
Pv 41 rfp
DECOUPLER 9.300 lp -56.4
MI VC PLOT
dof yyf WC 250
dms yvf VC 63
dpr 42 th no ph 1
dar 8500 nm no ph
    
```

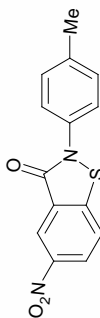
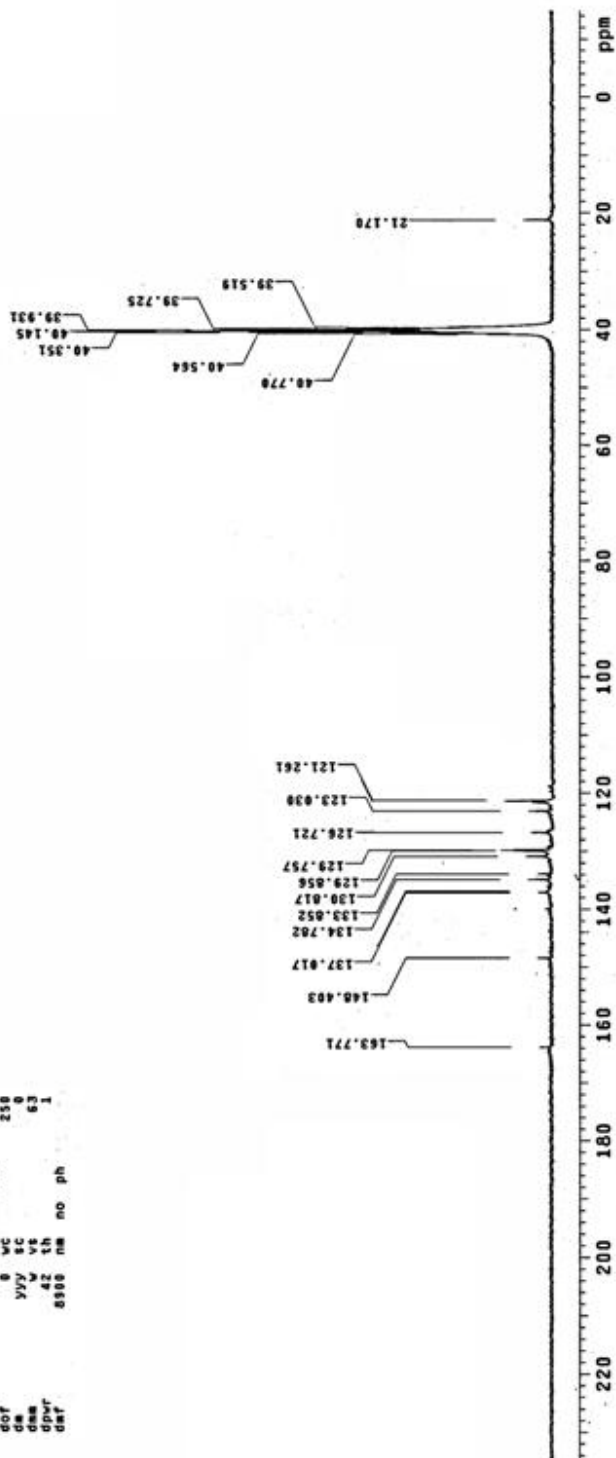


Table 4, entry 6



```

SM-9a
-exp1 tzpul
date Jan 17 2012 temp not used
solvent DMSO gain not used
p11n not used
sw ACQUISITION exp hst not used
at 8369.8 pw30 19.700
mp 1.998 m1fa 20.000
fb mot used 11 n
bs 4 in n
d1 1.000 dp y
nt 32 hs
ct TRANSMITTER H1 b PROCESSING 6.18
tn 6536
f1rq 399.815 fn DISPLAY 136.2
t0r 382.8 sp 4303.0
tpr 9.810 rfi 795.0
pw DECOUPLER C13 rf 98.4
dn 0 lp -85.0
dof 0 PLOT
dm mnn C 250
dms C 50 SC 0
dpr 15900 VS 384
dat nm cdc ph
    
```

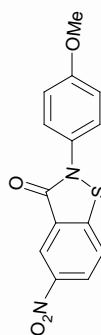
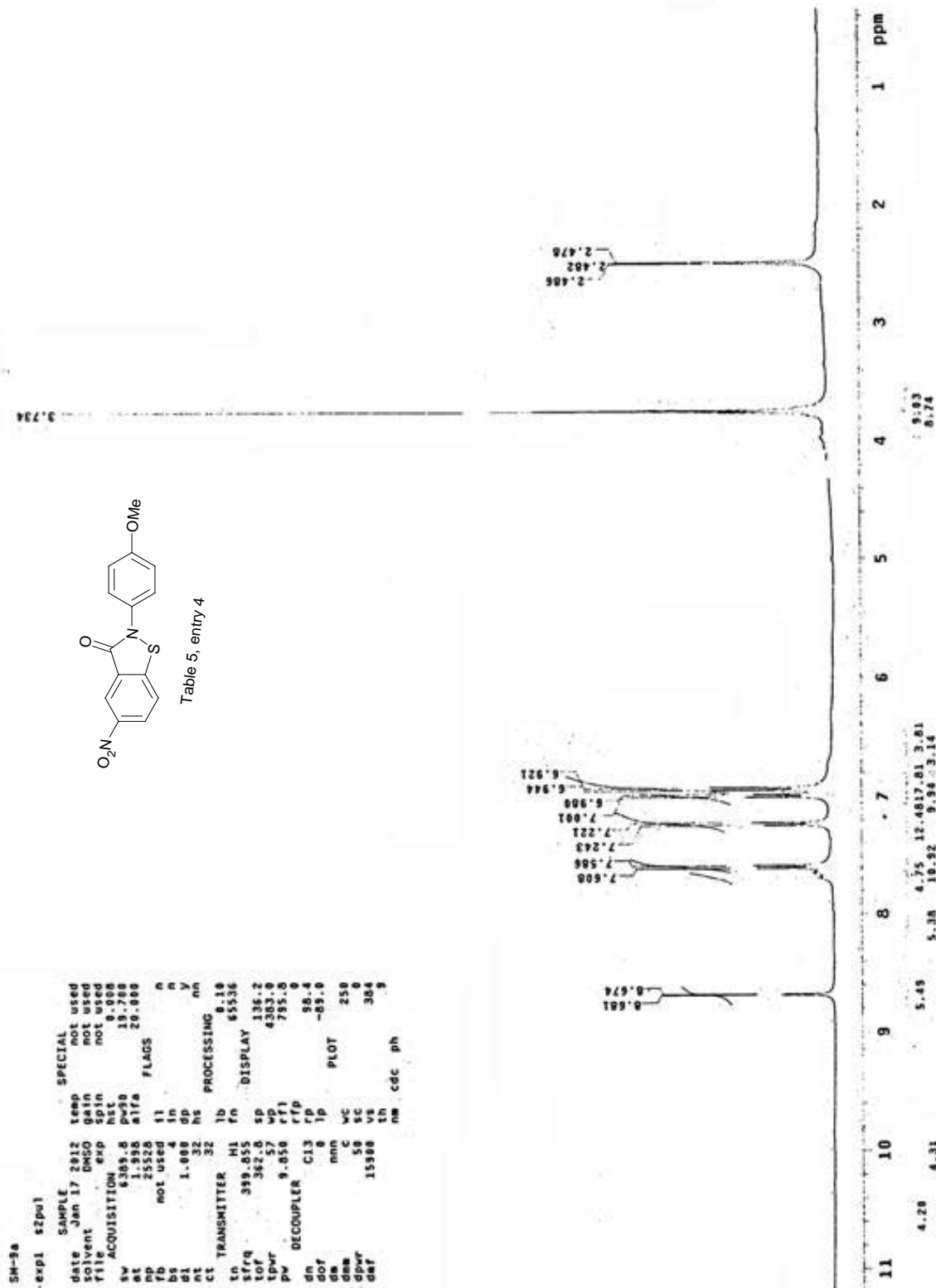


Table 5, entry 4



```

exp1 szpu1
SAMPLE
date Feb 18 2012 temp not used
solvent DMSO gain not used
file /export/home/~ not used
ciftemp/RP-SK-3a.1-hst 8.888
ACQUISITION 1d pvs 18.888
          a1fa 20.888
          flags
          n
          rd 64278 in
          tb 18888 dp
          dl 1.000 ns
          ct 7000 lb fn 6556
          tn TRANSMITTER C13 SP DISPLAY 217.2
          sfrq 100.554 vp 16489.0
          tof 1536.3 rf1 5537.0
          tpr 61 rfp 3971.5
          pw 8.300 rp -36.9
          dn DECOUPLER H1 PLOT -343.4
          dof 0 wc 250
          dca 0
          dpr 42 tk 93
          dbr 8908 nm no ph
    
```

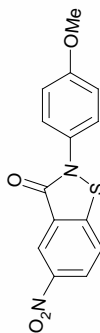


Table 5, entry 4

