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Structure Investigations

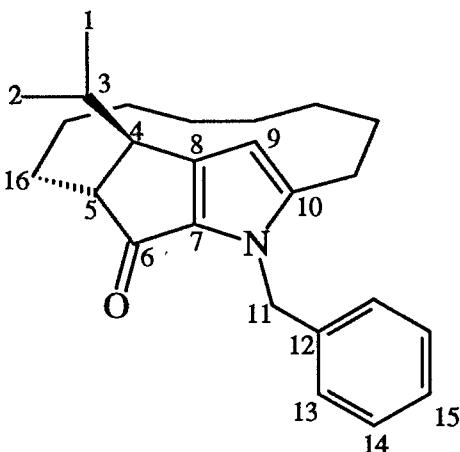
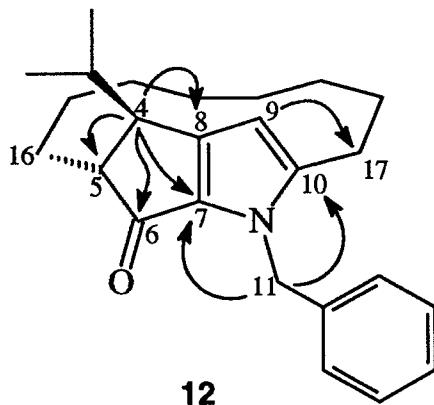
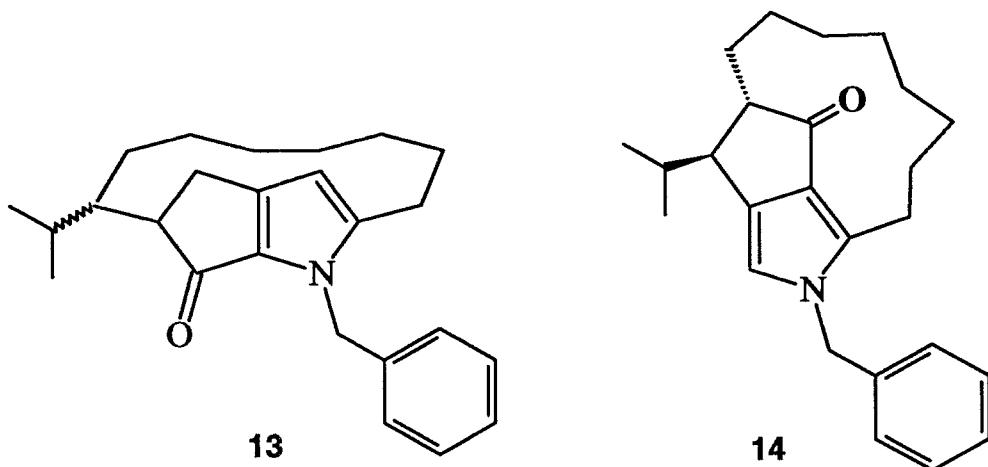


Table. Selected ^1H and ^{13}C NMR data of compound **12**. All assignments are unambiguous and were made by using COSY and ^{13}C , ^1H -chemical shift correlated NMR spectra. Arbitrary numbering as shown. The multiplicity of the ^{13}C signals refers to the geminal protons (DEPT).

Atom	^1H NMR (δ)	^{13}C NMR (δ)
1,2	1.00 (d), 0.88 (d)	21.20 (q), 19.68 (q)
3	1.78 (m)	33.28 (d)
4	2.63 (d)	48.39 (d)
5	2.70 (m)	59.44 (d)
6	-	193.41 (s)
7	-	134.87 (s)
8	-	153.87 (s)
9	6.00 (s)	109.13 (d)
10	-	146.72 (s)
11	5.67 (d), 4.98 (d)	47.67 (t)
12	-	138.40 (s)
13	7.20-7.18 (m)	126.90 (d)
14	7.32-7.29 (m)	128.74 (d)
15	7.27-7.24 (m)	127.55 (d)
16	1.98-1.93 (m), 1.80-1.75 (m)	31.82 (t)



Schematic representation of the longe range couplings observed in the $^nJ(\text{H,C})$ correlated spectra of compound **12** which define the site of acylation of the pyrrole ring and the location of the isopropyl side chain within the cyclopentanone ring (arbitrary numbering).



One might expect that strain and/or antiaromaticity of the product formed could prevent the desired elimination of the sulfone group of substrate **11** in the 5-membered ring; however, elimination in the ansa chain might occur which would lead to product **13** after the 1,4-addition of the zincate. The attached spectra, however, rigorously exclude such a pathway:

- (i) the observed coupling pattern (doublett) of the proton at the branching point does not match the one expected for **13** (ddd or dAB).
- (ii) the observed crosspeaks of H-4 (numbering as above) in the $^nJ(\text{C,H})$ correlated spectra - in particular those with two quarternary pyrrolic C-atoms - give an unambiguous proof of the site of attachment.

Similar arguments can be forwarded to establish the site of acylation of the pyrrole ring (**10** → **11** → **12**). In the product formed, crosspeaks are observed between the N-CH₂Ph (H-11) and *two quarternary* α-C-atoms of the trisubstituted pyrrole ring (C-7, C-10), while a crosspeak with its =CH atom (C-9) is missing. This excludes that acylation had occurred at C-4 of the pyrrole and hence definitely rules out the formation of the isomeric product **14**.

Experimental Procedures

Compound 3. To a solution of alcohol **2** (4.452 g, 41.8 mmol) in CH₂Cl₂ (80 ml) was added dropwise a solution of TBDMSCl (6.933 g, 46.0 mmol) and DBU (6.88 ml, 46.0 mmol) in CH₂Cl₂ (20 ml) over a period of 1 hr at room temperature. After stirring for additional 15 min, the reaction mixture was poured into NaHCO₃ (sat.), the aqueous layer was extracted with CH₂Cl₂, the organic phases were dried over Na₂SO₄ and evaporated. Purification of the residue by flash chromatography (SiO₂, pentane/ether = 20/1) afforded compound **3** (8.186 g, 90 %) as colorless liquid. ¹H NMR (200 MHz, CDCl₃): δ 5.22-5.18 (m, 2H), 4.22 (m, 2H), 4.07 (m, 2H), 0.90 (s, 9H), 0.07 (s, 6H). ¹³C NMR (75 MHz, CDCl₃): δ 144.6, 114.4, 63.4, 45.0, 25.9, 18.3, -5.4. MS: *m/z* (relative intensity) 165 (37), 164 (14), 163 (100), 127 (42), 125 (27), 123 (74), 95 (41), 93 (84), 75 (12), 73 (25), 57 (43), 41 (15). IR (neat): 3084, 2956, 2930, 2886, 2858, 1656, 1258, 1115, 1089, 915, 846, 777 cm⁻¹. C₁₀H₂₁ClOSi: *Calcd.*: C, 54.39; H, 9.59; *Found*: C, 54.55; H, 9.56.

Compound 4. A solution of allyl chloride **3** (1.369 g, 6.21 mmol) and NaI (0.932 g, 6.21 mmol) in acetone (10 ml) was stirred at 50°C for 16 h. After cooling to room temperature, the precipitated NaCl was filtered off under argon and washed with acetone (10 ml). Tetrahydrothiophene (1.10 ml, 12.42 mmol) was added to the yellow solution of the allyl iodide and the flask was wrapped with aluminum foil. After addition of AgBF₄ (1.209 g, 6.21 mmol), the resulting suspension was stirred for 1 h. The reaction mixture was diluted with CH₂Cl₂ (20 ml), the precipitated AgI was filtered off and the solvent evaporated. The crude solid was washed with hexane, the solvent removed by decantation and the resulting colorless crystals of the sulfonium salt **4** were dried *in vacuo* (1.639 g, 73 %). ¹H NMR (200 MHz, THF-d₈): δ 5.55 (d, *J* = 1.0 Hz, 1H), 5.51 (dd, *J* = 2.8, 1.5 Hz, 1H), 4.29 (s, 2H), 4.02 (s, 2H), 3.65-3.39 (m, 4H), 2.52-2.22 (m, 4H), 0.95 (s, 9H), 0.12 (s, 6H). ¹³C NMR (50 MHz, THF-d₈): δ 139.7, 120.8, 65.8, 45.7, 44.2, 29.4, 26.6, 19.3, -5.0. MS (ESI/pos): *m/z* (relative intensity) 633 ([2M⁺-BF₄], 20), 273 ([M⁺-BF₄], 100). IR (KBr): 2956, 2931, 2887, 2859, 1652, 1474, 1462, 1431, 1255, 1112, 1059, 843, 777 cm⁻¹. C₁₄H₂₉BF₄OSSi: *Calcd.*: C, 46.67; H, 8.11. *Found*: C, 46.53; H, 8.06.

Compound 5. To a solution of the sulfonium salt **4** (1.507 g, 4.19 mmol) in THF (50ml) was added *t*-BuLi (2.79 ml, 4.19 mmol, 1.5 M in hexane) via syringe at -78°C. After stirring for 15

min at -78°C, 9-bromononanal (0.842 g, 3.81 mmol) in THF (5 ml) was added via syringe, the mixture was stirred for further 15 min at -78°C and then slowly warmed to room temperature. The reaction mixture was extracted with H₂O/ethyl acetate, the organic layer was dried over Na₂SO₄ and evaporated. Purification of the crude product by flash chromatography (SiO₂, hexane/ethyl acetate = 50/1) afforded vinyl oxirane **5** (1.299 g, 84 %) as a colorless oil. ¹H NMR (200 MHz, CDCl₃): δ 5.21-5.15 (m, 1.5H), 4.99 (m, 0.5H), 4.16 (s, 1H), 4.09 (m, 1H), 3.44 (m, 0.5H), 3.38 (t, J = 6.8 Hz, 2H), 3.14 (d, J = 2.1 Hz, 0.5H), 3.03 (m, 0.5H), 2.85 (td, J = 5.3, 2.2 Hz, 0.5H), 1.83 (m, 2H), 1.53-1.18 (m, 12H), 0.90 (s, 4.5H), 0.89 (s, 4.5H), 0.06 (s, 3H), 0.05 (s, 3H). ¹³C NMR (50 MHz, CDCl₃): δ 144.9, 142.2, 111.9, 111.2, 64.5, 62.7, 60.1, 58.8, 58.4, 56.3, 33.9, 32.8, 32.2, 29.4, 29.3, 28.6, 28.1, 26.6, 26.2, 25.9, 18.4, 18.3, -5.4. MS: m/z (relative intensity) 350 (14), 349 (64), 348 (14), 347 (62), 157 (19), 144 (11), 143 (87), 127 (13), 113 (12), 109 (14), 107 (20), 105 (11), 95 (23), 93 (33), 81 (26), 79 (23), 75 (100), 73 (48), 69 (26), 67 (23), 59 (12), 57 (18), 55 (45), 43 (16), 41 (27), 29 (10). IR (neat): 2929, 2856, 1656, 1471, 1463, 1389, 1255, 1107, 1085, 911, 838, 777 cm⁻¹. C₁₉H₃₇BrO₂Si: Calcd.: C, 56.28; H, 9.20; Found: C, 56.32; H, 9.15.

Compound 6. Methyl (phenylsulfonyl)acetate (0.656 g, 3.06 mmol) was added to KH (0.123 g, 3.06 mmol) in DMF (15 ml) at room temperature. After hydrogen formation had ceased, substrate **5** (1.238 g, 3.06 mmol) in DMF (5 ml) was added and the solution stirred for 72 h at room temperature. The reaction mixture was extracted with H₂O/ethyl acetate, the organic layer was dried over Na₂SO₄ and evaporated. Purification of the residue by flash chromatography (SiO₂, hexane/ethyl acetate = 10/1) afforded compound **6** (1.112 g, 68 %) as a colorless oil. ¹H NMR (300 MHz, CDCl₃): δ 7.86-7.82 (m, 2H), 7.68-7.62 (m, 1H), 7.57-7.51 (m, 2H), 5.19-5.13 (m, 1.5H), 4.97 (m, 0.5H), 4.14-4.06 (m, 2H), 3.90 (dd, J = 10.7, 4.3 Hz, 1H), 3.63 (s, 3H), 3.41 (ddd, J = 4.3, 1.2, 0.6 Hz, 0.5H), 3.12 (d, J = 2.1 Hz, 0.5H), 3.02 (m, 0.5H), 2.83 (m, 0.5H), 1.99-1.91 (m, 2H), 1.59-1.48 (m, 1H), 1.37-1.23 (m, 13H), 0.88 (s, 4.5H), 0.87 (s, 4.5H), 0.04 (s, 3H), 0.03 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): δ 166.5, 144.8, 142.2, 137.1, 134.2, 129.3, 129.0, 111.9, 111.1, 70.9, 64.4, 62.7, 60.0, 58.7, 58.3, 56.2, 52.8, 32.2, 29.3, 29.2, 29.0, 28.9, 26.8, 26.7, 26.6, 26.2, 25.9, 25.8, 18.3, 18.2, -5.39, -5.42. MS: m/z (relative intensity) 483 (14), 482 (32), 481 (100), 199 (38), 143 (44), 135 (22), 125 (11), 95 (10), 81 (11), 77 (19), 75 (39), 73 (30), 67 (11), 55 (12). IR (neat): 3093, 2952, 2929, 2856, 1745, 1463, 1448, 1327, 1311, 1257, 1150, 1084, 838, 778, 723, 689 cm⁻¹. C₂₈H₄₆O₆SSi: Calcd.: C, 62.42; H, 8.61; Found: C, 62.32; H, 8.67.

Compound 7. To a refluxing solution of $\text{Pd}(\text{PPh}_3)_4$ (23 mg, 0.02 mmol) and dppe (16 mg, 0.04 mmol) in THF (100 ml) was added substrate **6** (108 mg, 0.20 mmol) in THF (40 ml) dropwise over a period of 6 h. The reaction mixture was refluxed for additional 10 h, cooled to room temperature, extracted with H_2O /ethyl acetate and the organic layer was dried over Na_2SO_4 . After evaporation of the solvent, the crude product was purified by flash chromatography (SiO_2 , hexane/ethyl acetate = 3/1), which afforded product **7** (92 mg, 85 %) as a pale yellow foam. ^1H NMR (400 MHz, CD_2Cl_2): δ 7.83-7.76 (m, 2H), 7.70-7.67 (m, 1H), 7.59-7.54 (m, 2H), 5.89 (d, J = 8.8 Hz), 5.71 (dd, J = 9.6, 1.5 Hz), 5.61 (d, J = 9.0 Hz), 5.25 (d, J = 7.7 Hz) [1H], 4.56 -4.07 (m), 4.01 (s), 3.85-3.72 (m) [3H], 3.60 (s), 3.57 (s), 3.56 (s), 3.50 (s) [3H], 3.13-2.78 (m, 2H), 2.20-1.21 (m, 17H), 0.95 (s), 0.92 (s), 0.84(s), 0.81 (s) [9H], 0.119 (s), 0.117 (s), 0.114 (s), 0.108 (s), 0.014 (s), -0.003 (s), -0.010 (s), -0.015 (s) [6H]. ^{13}C NMR (100 MHz, CD_2Cl_2): δ 169.21, 169.18, 168.92, 168.83, 140.06, 137.45, 137.26, 137.11, 136.73, 136.67, 136.66, 135.94, 135.74, 135.18, 134.45, 134.40, 134.33, 133.96, 132.55, 130.64, 130.57, 130.47, 130.37, 129.21, 129.08, 129.06, 129.01, 77.38, 76.71, 76.47, 76.21, 68.27, 67.94, 67.81, 66.77, 66.34, 66.28, 62.67, 60.52, 38.54, 37.29, 36.85, 36.30, 36.20, 33.95, 31.85, 31.10, 31.07, 30.80, 29.84, 29.30, 29.21, 29.04, 28.63, 27.94, 27.85, 27.43, 27.19, 26.88, 26.72, 26.45, 26.09, 25.99, 25.97, 25.47, 25.14, 24.97, 24.80, 24.67, 24.59, 24.29, 24.13, 24.08, 23.57, 23.41, 21.69, 21.06, 20.57, 18.65, 18.58, 18.44, 18.36, -5.17, -5.19, -5.25, -5.27, -5.30, -5.35, -5.30. MS: m/z (relative intensity) 481 (17), 431 (16), 339 (28), 322 (11), 321 (42), 307 (16), 265 (30), 233 (29), 200 (15), 199 (100), 187 (18) 135 (30), 91 (11), 81 (11), 79 (11), 75 (49), 73 (39), 67 (12), 55 (12), 41 (10). IR (neat): 3527, 3429, 3065, 2930, 2857, 1736, 1585, 1463, 1447, 1308, 1252, 1144, 1079, 1038, 1005, 838, 778, 721, 691, 610 cm^{-1} . $\text{C}_{28}\text{H}_{46}\text{O}_6\text{SSI}$: *Calcd.*: C, 62.42; H, 8.61; *Found*: C, 62.36; H, 8.54.

Compound 8. To a solution of silyl ether **7** (736 mg, 1.37 mmol) in THF (125 ml) was successively added NH_4F (253 mg, 6.84 mmol) and TBAF (6.85 ml, 6.85 mmol, 1 M in THF) at room temperature. After stirring for 30 min, the reaction mixture was extracted with saturated aq. NaCl /ethyl acetate, the organic layer was dried over Na_2SO_4 and evaporated. Purification of the residue by flash chromatography (SiO_2 , hexane/ethyl acetate = 2/1 → 1/1) afforded lactone **8** (338 mg, 63 %) as a colorless foam. ^1H NMR (400 MHz, CD_2Cl_2): δ 7.94-7.84 (m, 2H), 7.73-7.69 (m, 1H), 7.61-7.55 (m, 2H), 5.77-5.69 (m, 1H), 5.08 (dd, J = 13.3, 1.6 Hz), 4.98-4.84 (m), 4.73-4.69 (m), 4.58-4.45 (m), 4.36 (td, J = 9.5, 3.2 Hz) [3H], 3.92 (dd, J = 16.3, 2.6 Hz), 3.75-3.72 (m), 3.53-3.45 (m), 3.42-3.32 (m) 3.02 (dd, J = 16.1, 2.8

Hz), 2.81-2.72 (m) [2H], 2.07-0.89 (m, 17H). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 166.60, 166.45, 165.85, 136.28, 135.54, 135.28, 134.63, 134.53, 134.42, 134.35, 134.29, 133.72, 133.37, 133.32, 131.61, 131.45, 131.24, 131.20, 129.50, 129.27, 128.88, 128.76, 128.65, 128.62, 127.92, 127.87, 127.24, 75.73, 74.65, 72.81, 72.76, 72.36, 71.96, 71.88, 70.90, 70.54, 68.52, 67.43, 65.38, 38.32, 37.50, 36.53, 36.23, 35.86, 35.67, 35.38, 34.96, 33.46, 29.82, 29.03, 27.83, 27.76, 27.53, 27.24, 27.02, 26.92, 26.68, 26.03, 25.84, 25.83, 25.36, 25.05, 24.58, 24.39, 24.27, 24.18, 23.63, 23.30, 23.24, 22.71, 22.12. MS: m/z (relative intensity) 252 (16), 251 (100), 250 (60), 234 (13), 233 (76), 232 (50), 222 (11), 206 (15), 205 (31), 187 (24), 152 (22), 149 (10), 145 (11), 143 (26), 135 (12), 131 (12), 125 (16), 124 (11), 123 (10), 121 (15), 119 (12), 109 (16), 108 (14), 107 (19), 105 (15), 95 (34), 93 (24), 91 (23), 83 (11), 82 (21), 81 (43), 79 (39), 78 (12), 77 (43), 69 (21), 67 (41), 57 (18), 55 (55), 53 (13), 43 (33), 41 (57), 39 (12), 29 (20). IR (neat): 3515, 3066, 2932, 2860, 1730, 1584, 1448, 1307, 1143, 1080, 1000, 722, 689 cm^{-1} . $\text{C}_{21}\text{H}_{28}\text{O}_5\text{S}$: *Calcd.*: C, 64.26; H, 7.19; *Found*: C, 64.15; H, 7.26.

Compound 9. Dess-Martin-periodinane (825 mg, 1.94 mmol) was added to a solution of alcohol **8** (304 mg, 0.78 mmol) in CH_2Cl_2 (50 ml) at room temperature. After stirring for 3.5 h, the reaction mixture was filtered through a short pad of silica, the residues were washed with CH_2Cl_2 , the combined organic layers were extracted with sat. aq. NaHCO_3 and dried over Na_2SO_4 . The solvent was evaporated and the crude product purified by flash chromatography (SiO_2 , hexane/ethyl acetate = 6/1 → 4/1), which afforded ketone **9** (251 mg, 83 %) as a colorless foam. ^1H NMR (300 MHz, CDCl_3): δ 7.97-7.93 (m, 2H), 7.72-7.65 (m, 1H), 7.58-7.55 (m, 2H), 6.55 (m), 6.40 (d, J = 2.7 Hz) [1H], 5.58-5.52 (m), 5.13-5.02 (m), 4.50 (dd, J = 12.2, 2.7 Hz) [2H], 3.70-3.47 (m, 2H), 2.94 (dd, J = 13.0, 1.5 Hz), 2.85 (ddd, J = 16.0, 10.7, 3.1 Hz), 2.70 (m) [2H], 2.35-2.24 (m, 1H), 2.11-1.91 (m, 2H), 1.86-1.66 (m, 1H), 1.62-0.78 (m, 10H). ^{13}C NMR (75 MHz, CDCl_3): δ 202.2, 201.8, 166.2, 165.4, 143.8, 141.3, 134.9, 134.6, 134.5, 133.9, 131.5, 131.2, 129.2, 128.6, 128.5, 127.5, 73.4, 73.1, 70.9, 70.3, 41.8, 40.8, 38.4, 34.6, 34.1, 30.7, 26.4, 26.2, 26.0, 25.8, 25.3, 25.2, 24.8, 23.7, 22.9, 22.4, 22.3, 22.0. MS: m/z (relative intensity) 250 (16), 249 (100), 248 (92), 221 (16), 220 (11), 151 (14), 150 (15), 142 (11), 137 (21), 136 (11), 124 (21), 123 (18), 122 (11), 119 (10), 107 (11), 105 (13), 95 (14), 94 (14), 93 (15), 91 (22), 81 (18), 79 (28), 78 (18), 77 (54), 69 (11), 67 (30), 66 (12), 65 (16), 55 (42), 53 (13), 51 (17), 43 (25), 41 (51), 39 (19), 29 (16), 27

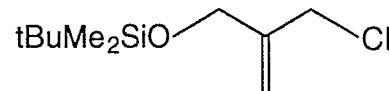
(12). IR (KBr): 3066, 2941, 2862, 1738, 1688, 1641, 1584, 1447, 1306, 1181, 1142, 1080, 754, 721 cm⁻¹. C₂₁H₂₆O₅S: *Calcd.*: C, 64.59; H, 6.71; *Found*: C, 64.38; H, 6.86.

Compound 10. A solution of Pd(PPh₃)₄ (175 mg, 0.15 mmol) and benzylamine (132 µl, 1.21 mmol) in THF (10 ml) was added to a solution of compound **9** (392 mg, 1.01 mmol) in THF (60 ml) at 35 °C. After stirring for 1.5 h, the reaction mixture was extracted with NH₄Cl/ethyl acetate, the organic layer was dried over Na₂SO₄ and evaporated. After purification of the residue by flash chromatography (SiO₂, hexane/ethyl acetate/acetic acid = 2/1/0 → 2/1/0.1), toluene and then CH₂Cl₂ were successively and repeatedly stripped off *in vacuo* in order to remove aceotropically any traces of HOAc. The resulting acid **10** was dried *in vacuo* and isolated as a yellow solid (340 mg, 70 %). ¹H NMR (400 MHz, THF-d₈): δ 7.84-7.81 (m, 2H), 7.62-7.57 (m, 1H), 7.51-7.47 (m, 2H), 7.21-7.10 (m, 3H), 6.95-9.93 (m, 2H), 6.46 (d, *J* = 2.0 Hz, 1H), 5.84 (d, *J* = 2.0 Hz, 1H), 4.97 (d, AB, *J* = 16.2 Hz, 1H), 4.91 (d, AB, *J* = 16.2 Hz, 1H), 3.42 (d, *J* = 13.8 Hz, 1H), 3.01 (d, *J* = 13.8 Hz, 1H), 2.52-2.46 (m, 1H), 2.42-2.35 (m, 1H), 2.22-2.14 (m, 1H), 1.65-1.60 (m, 1H), 1.32-0.85 (m, 11H), 0.65-0.62 (m, 1H); the -COOH is not detected. ¹³C NMR (100 MHz, THF-d₈): δ 169.9, 140.5, 138.6, 134.5, 132.4, 131.9, 129.5, 129.4, 128.1, 127.5, 122.3, 117.0, 113.3, 77.8, 51.2, 31.6, 30.3, 28.3, 27.8, 27.7, 27.6, 27.2, 26.7, 23.2. MS: *m/z* (relative intensity) 480 (16), 479 ([M⁺], 47), 339 (19), 338 (78), 337 (24), 91 (100), 44 (11). IR (KBr): 3400-2500, 3065, 2931, 2848, 1701, 1448, 1322, 1307, 1147, 1079, 811, 731, 688 cm⁻¹. HR-MS (C₂₈H₃₃NO₄S): *Calcd.*: 479.21303; *Found*: 479.21177.

Compound 11. N,N-(dimethyl)-1-amino-1-chloro-2-methylpropene-1 (122 mg, 0.92 mmol) in CH₂Cl₂ (2 ml) was added to a solution of acid **10** (400 mg, 0.84 mmol) in CH₂Cl₂ (50 ml) at room temperature. After stirring for 2 h, the solvent was removed *in vacuo* (10⁻² mbar) at room temperature and the resulting oil was dried for 2 h. The acid chloride thus obtained was dissolved in refluxing 1,2-dichloroethane (50 ml) and treated dropwise with SnCl₄ (110 µl, 0.94 mmol) via syringe. After 1 h reaction time, the solution was cooled to room temperature, extracted with NaHCO₃ (sat.)/CH₂Cl₂, the organic layer was dried over Na₂SO₄ and evaporated. Purification of the residue by flash chromatography (SiO₂, hexane/ethyl acetate = 6/1) afforded ketopyrrole **11** (275 mg, 71 %) as a colorless foam. ¹H NMR (300 MHz, CD₂Cl₂): δ 7.98-7.94 (m, 2H), 7.67-7.61 (m, 1H), 7.55-7.49 (m, 2H), 7.35-7.28 (m, 3H), 7.15-7.12 (m, 2H), 6.03 (s, 1H), 5.59 (d, *J* = 15.3 Hz, 1H), 4.96 (d, *J* = 15.3 Hz, 1H), 3.51

(d, $J = 16.4$ Hz, 1H), 2.90 (d, $J = 16.4$ Hz, 1H), 2.75-2.55 (m, 2H), 2.23-2.12 (m, 2H), 1.81-1.73 (m, 1H), 1.49-1.44 (m, 1H), 1.28-1.09 (m, 3H), 0.96-0.83 (m, 4H), 0.77-0.68 (m, 2H), 0.44-0.39 (m, 1H). ^{13}C NMR (75 MHz, CD_2Cl_2): δ 182.3, 150.3, 149.0, 137.4, 137.1, 133.7, 130.8, 128.8, 128.6, 127.9, 127.1, 108.1, 82.2, 47.9, 31.9, 29.8, 28.0, 27.5, 27.3, 26.4, 25.2, 24.7. MS: m/z (relative intensity) 461 ([M $^+$], 25), 321 (14), 320 (65), 319 (80), 91 (100). IR (KBr): 2928, 2856, 1680, 1623, 1484, 1457, 1446, 1391, 1301, 1261, 1143, 1084, 720, 689 cm^{-1} . HR-MS ($\text{C}_{28}\text{H}_{31}\text{NO}_3\text{S}$): *Calcd.*: 461.20247; *Found*: 461.19802.

Compound 12. To a solution of $\text{ZnCl}_2\text{-TMEDA}$ (197 mg, 0.78 mmol) in THF (30 ml) was added MeLi (1040 μl , 1.56 mmol, 1.5 M in THF) at 0°C and stirred for 15 min. *i*-PrMgCl (390 μl , 0.78 mmol, 2 M in THF) was then added and the resulting solution stirred for additional 5 min. After addition of *t*-BuOK (175 mg, 1.56 mmol) in THF (2 ml) and stirring for 5 min, sulfone **11** (60 mg, 0.13 mmol) in THF (3 ml) was introduced and the resulting solution was warmed to room temperature. The mixture was treated with additional *t*-BuOK (700 mg, 6.24 mmol) in THF (7 ml) and stirred for another 15 min. After extractive work-up with $\text{NH}_4\text{Cl}/$ ethyl acetate, the organic layer was dried over Na_2SO_4 , evaporated, and the residue purified by flash chromatography (SiO_2 , hexane/ethyl acetate = 20/1), which afforded product **12** (22 mg, 47 %) as a colorless oil. ^1H NMR (600 MHz, CD_2Cl_2): δ 7.32-7.29 (m, 2H), 7.27-7.24 (m, 1H), 7.20-7.18 (m, 2H), 6.00 (s, 1H), 5.67 (d, $J = 15.6$ Hz, 1H), 4.98 (d, $J = 15.6$ Hz, 1H), 2.70-2.65 (m, 2H), 2.63 (d, $J = 6.6$ Hz, 1H), 2.62-2.57 (m, 1H), 1.98-1.93 (m, 1H), 1.81-1.68 (m, 3H), 1.55-1.48 (m, 1H), 1.26-1.21 (m, 1H), 1.11-0.99 (m, 3H), 1.00 (d, $J = 6.7$ Hz, 3H), 0.94-0.89 (m, 3H), 0.88 (d, $J = 6.7$ Hz, 3H), 0.78-0.69 (m, 2H), 0.61-0.55 (m, 1H). ^{13}C NMR (150 MHz, CD_2Cl_2): δ 193.41 (s), 153.87 (s), 146.72 (s), 138.40 (s), 134.87 (s), 128.74 (d), 127.55 (d), 126.90 (d), 109.13 (d), 59.44 (d), 48.39 (d), 47.67 (t), 33.28 (d), 31.82 (t), 28.17 (t), 27.73 (t), 27.44 (t), 27.18 (t), 25.46 (t), 25.16 (t), 25.06 (t), 21.20 (q), 19.68 (q). MS: m/z (relative intensity) 364 (29), 363 ([M $^+$], 100), 348 (16), 321 (36), 320 (58), 91 (84). IR (KBr): 3064, 3031, 2929, 2856, 1673, 1496, 1473, 1456, 1391, 1260, 723, 697 cm^{-1} . HR-MS ($\text{C}_{25}\text{H}_{33}\text{NO}$): *Calcd.*: 363.25621; *Found*: 363.25401.



3



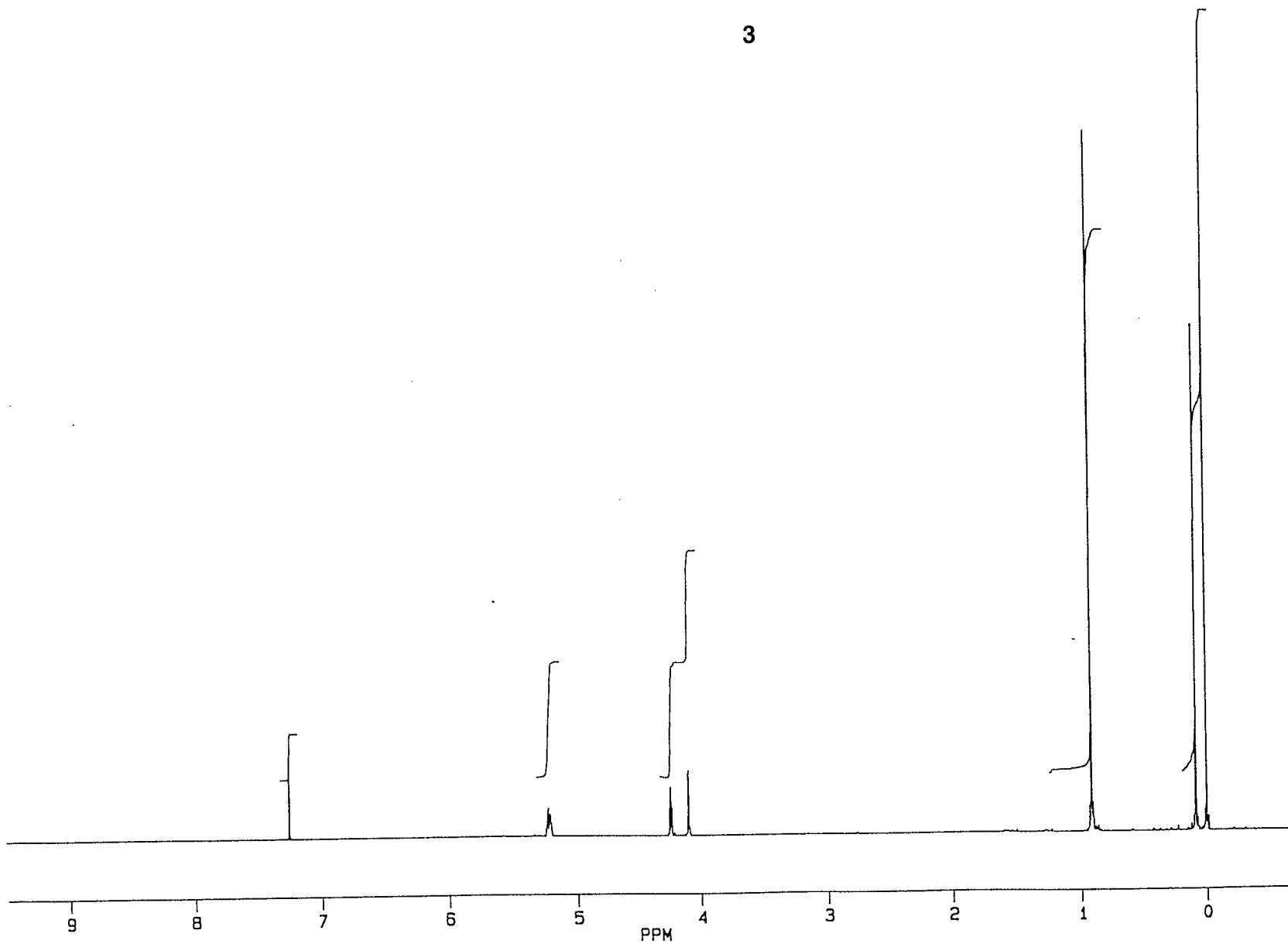
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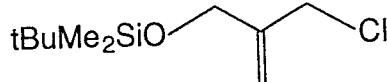
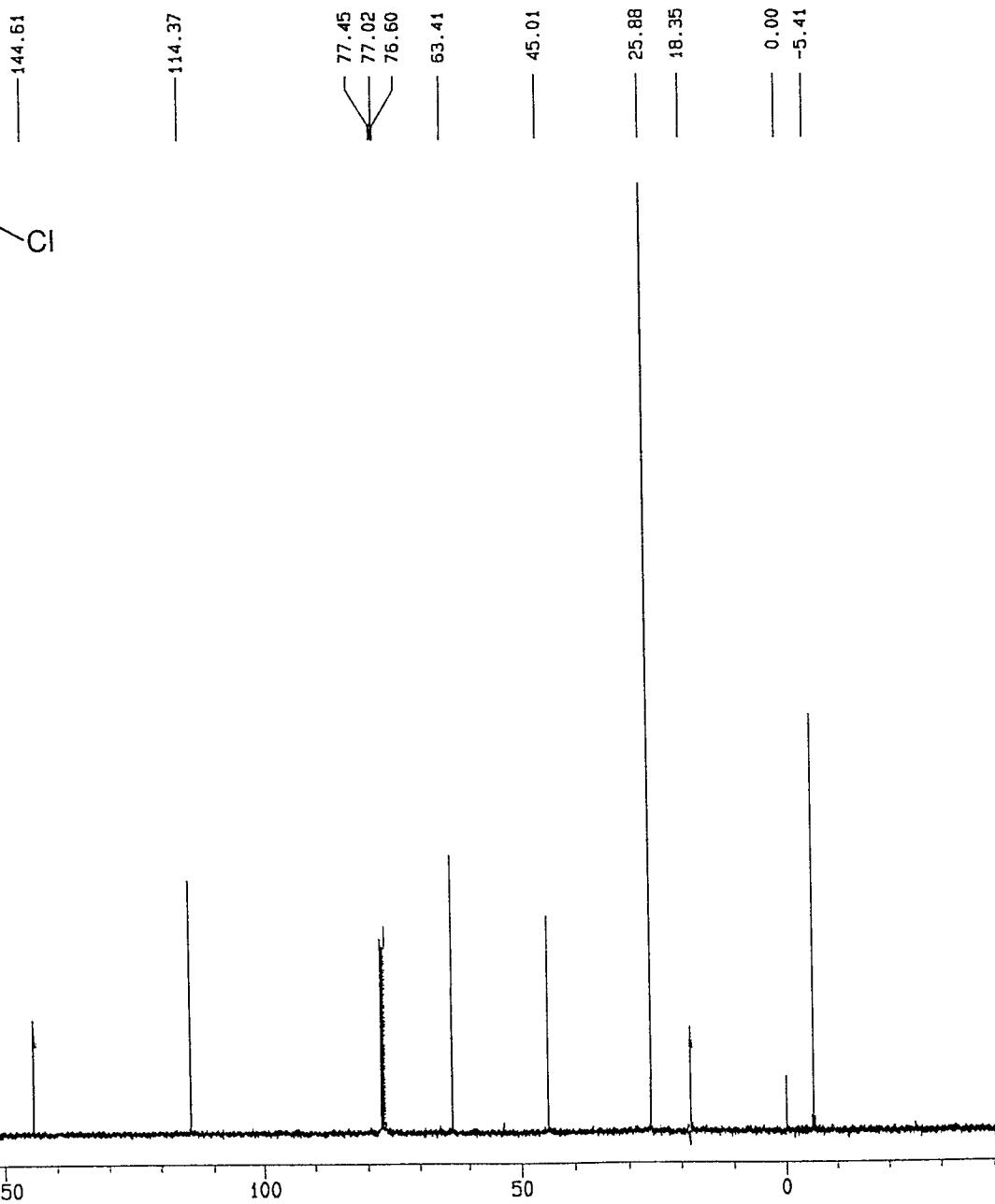
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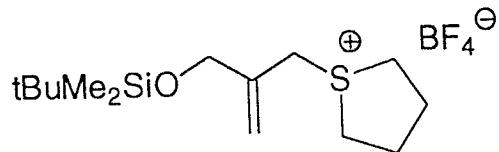
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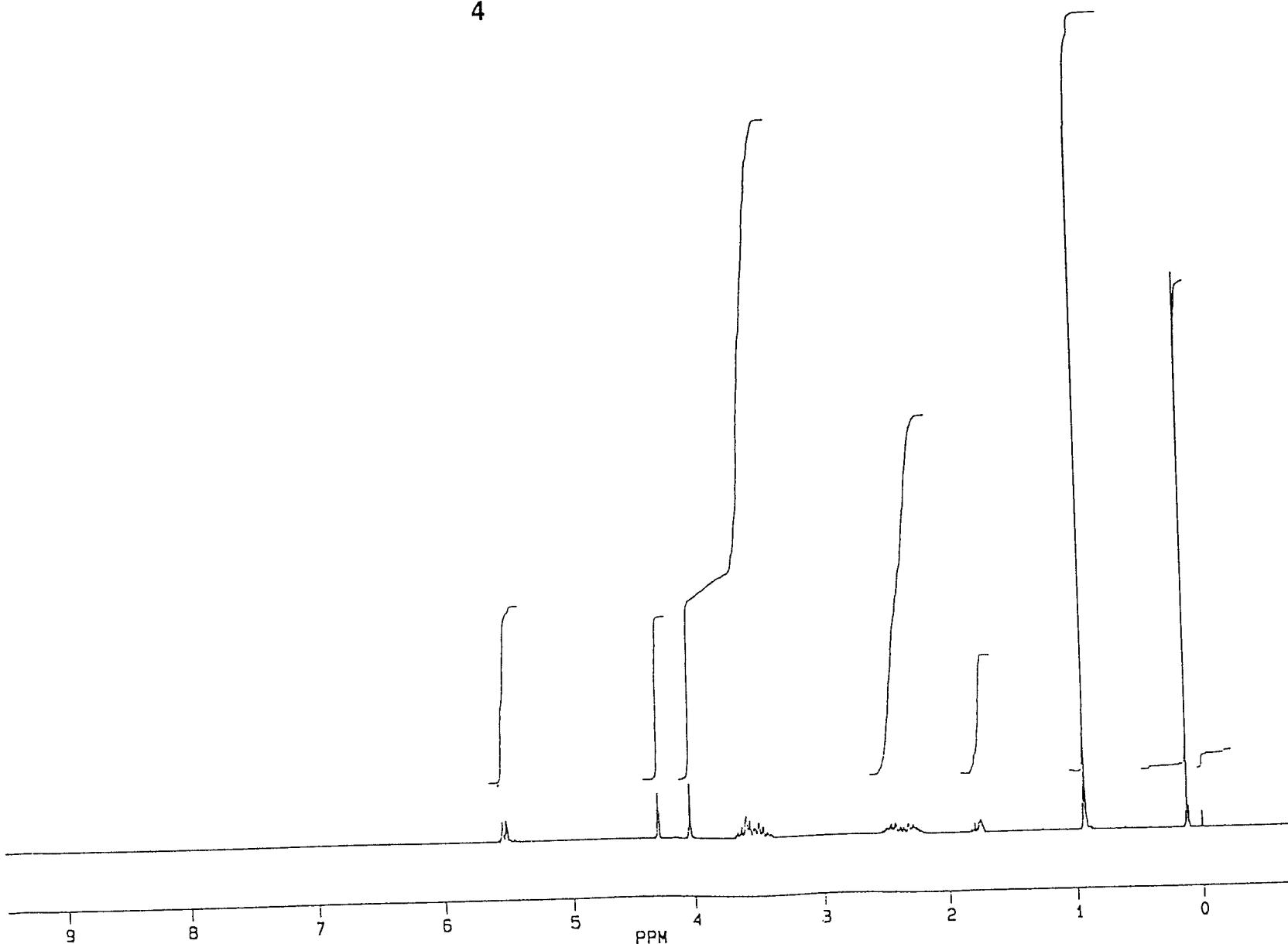
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4



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X51.AU

DATE 8-5-96

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SI 32768

TD 32768

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CY 12.00

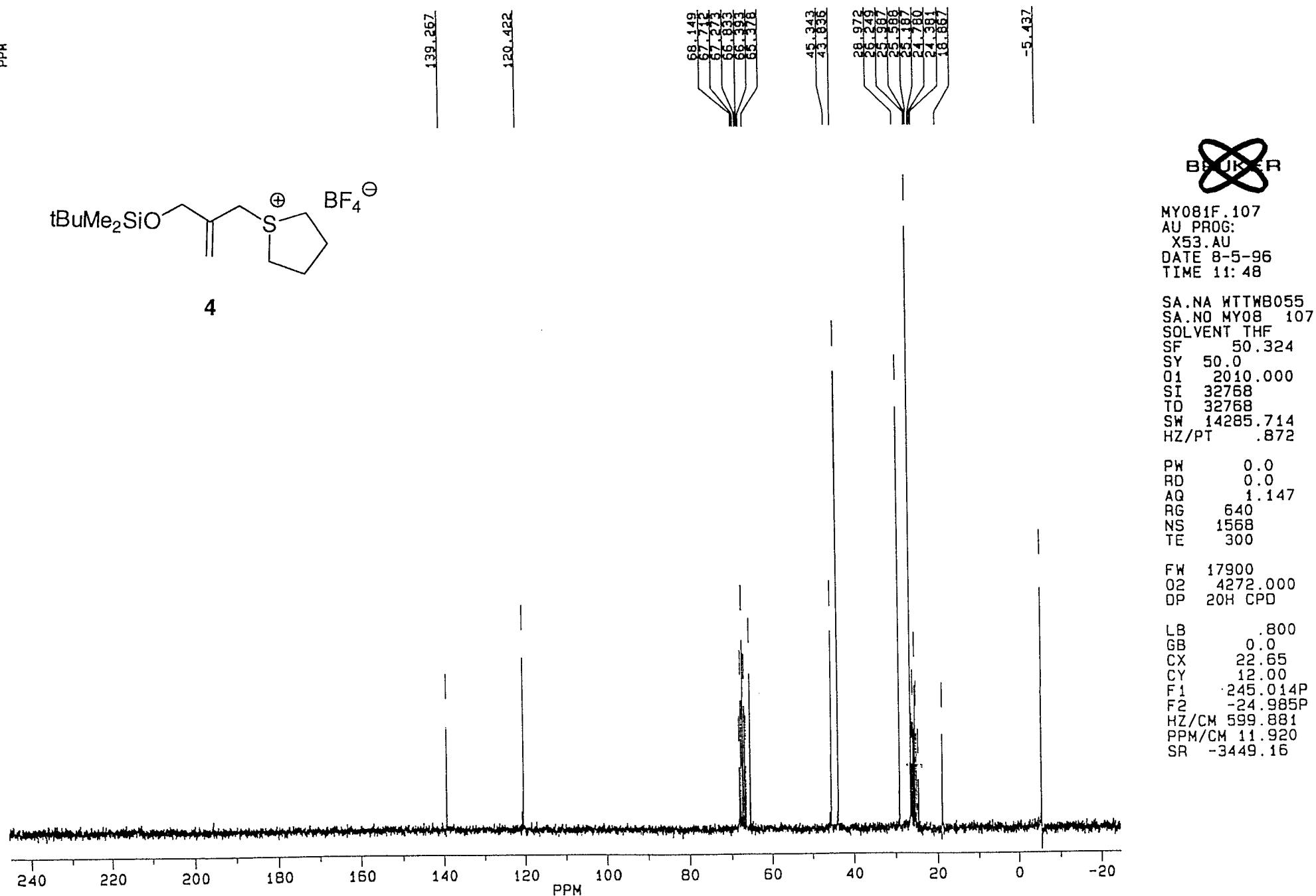
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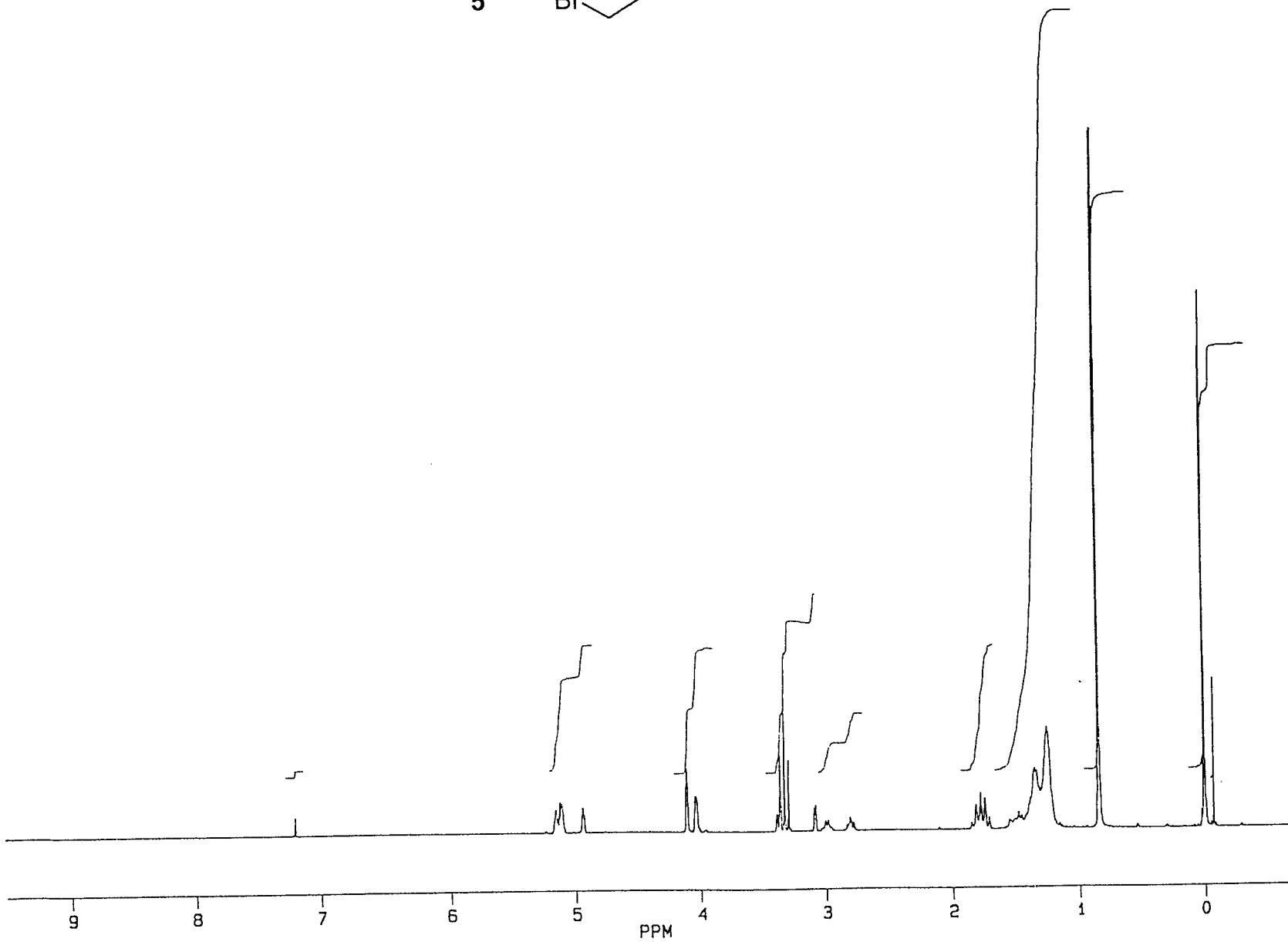
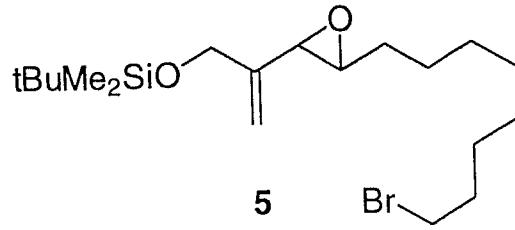
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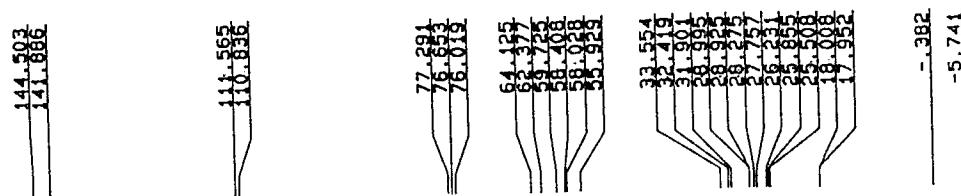
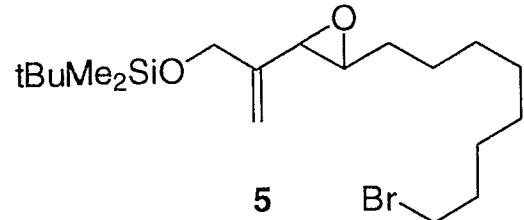
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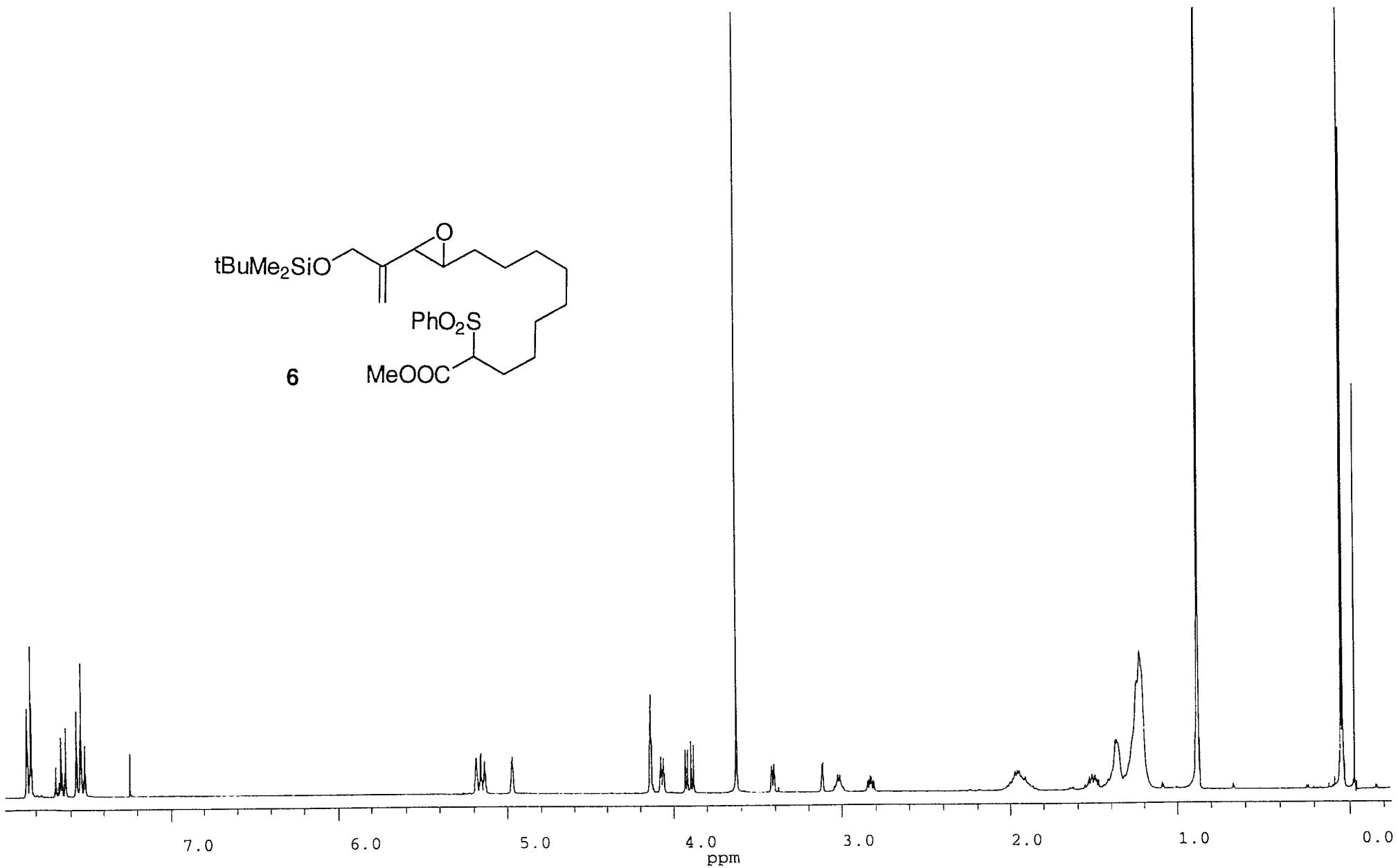
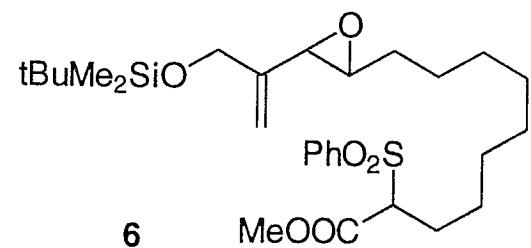
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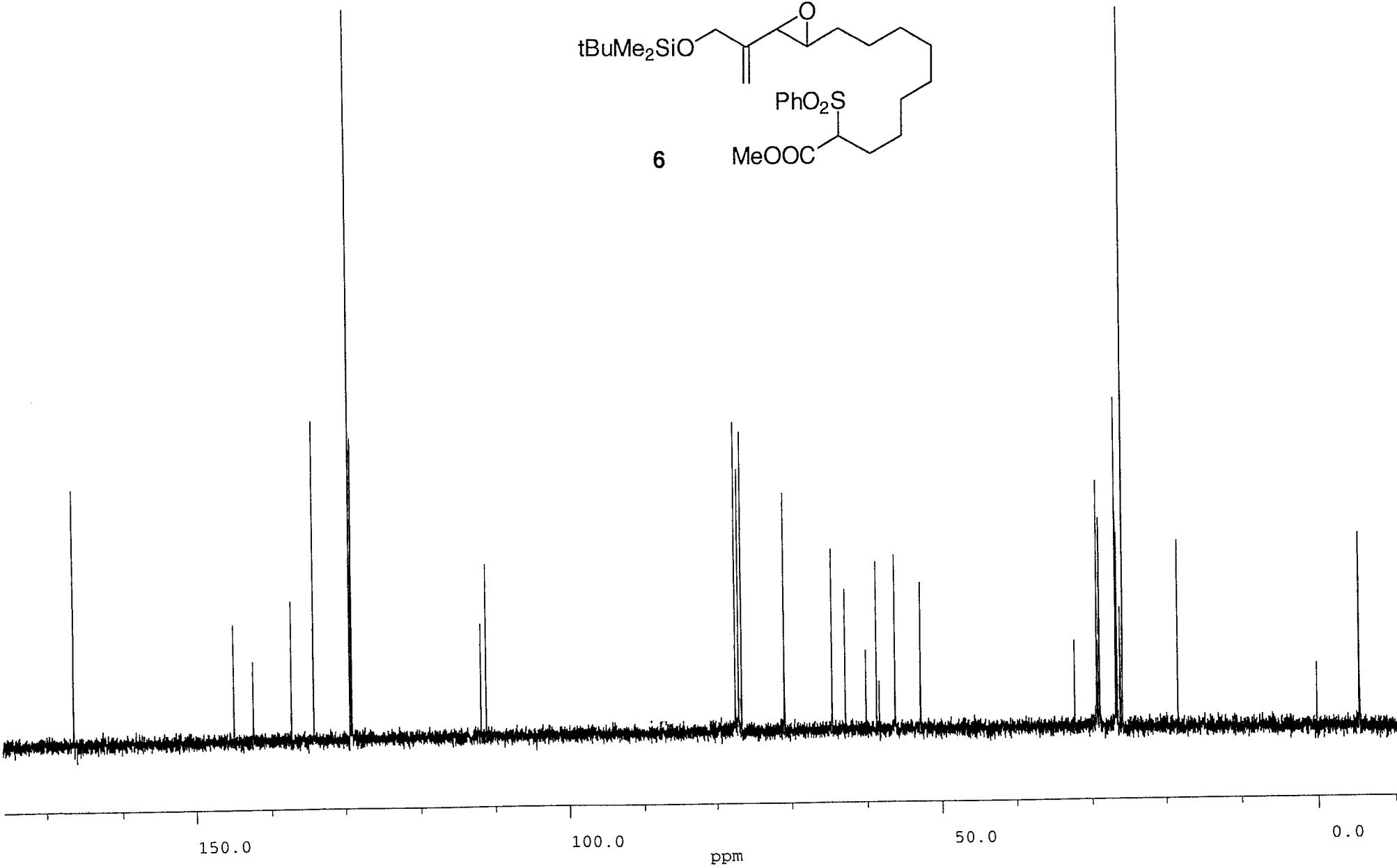
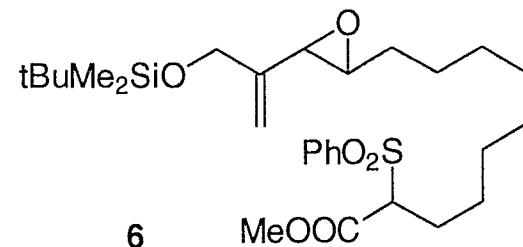
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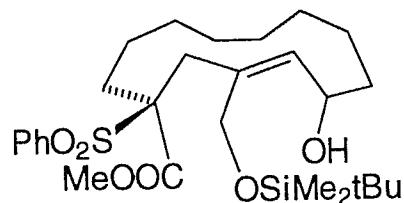


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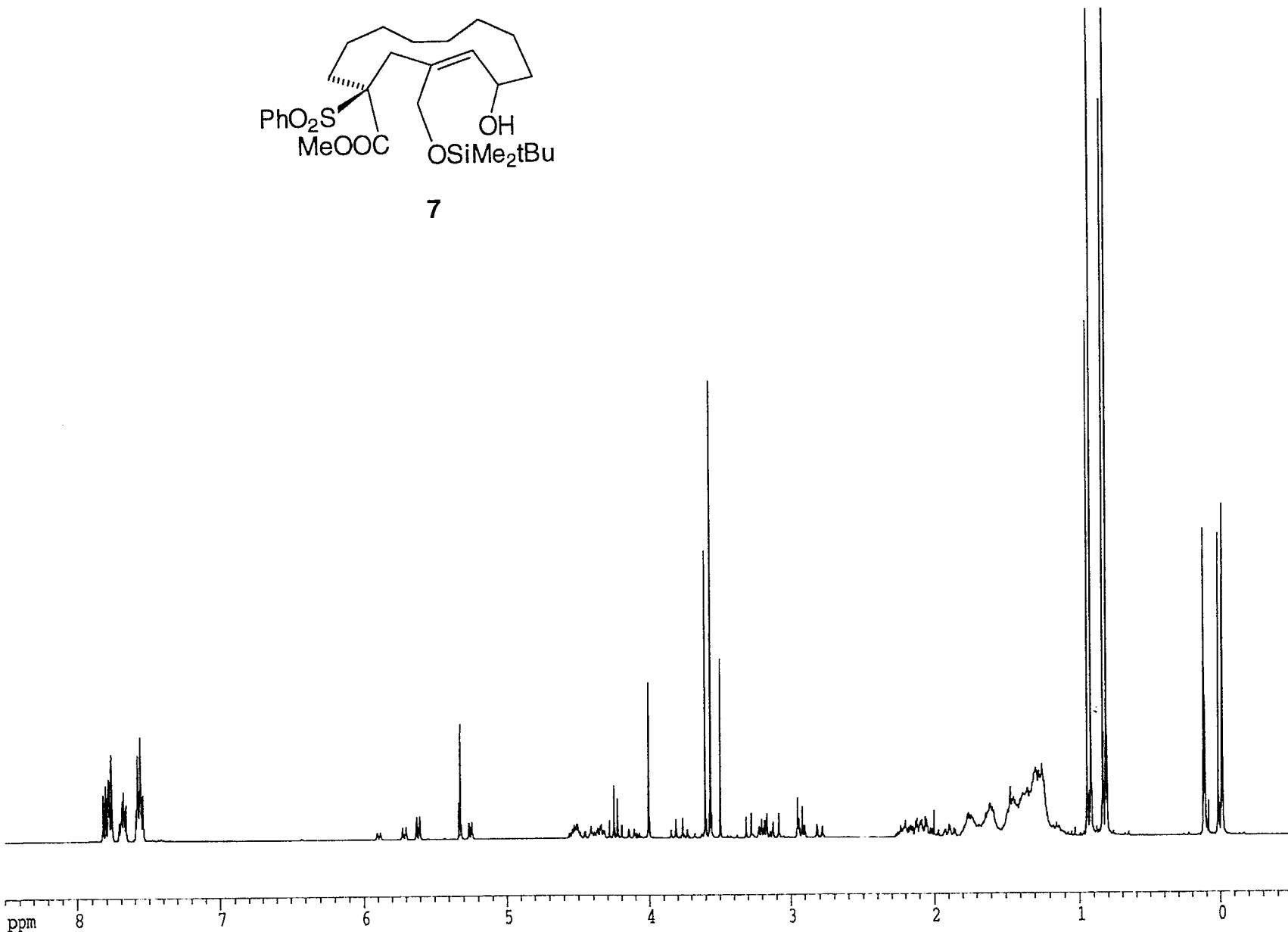
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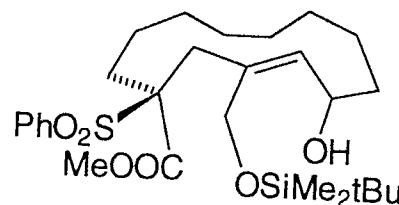


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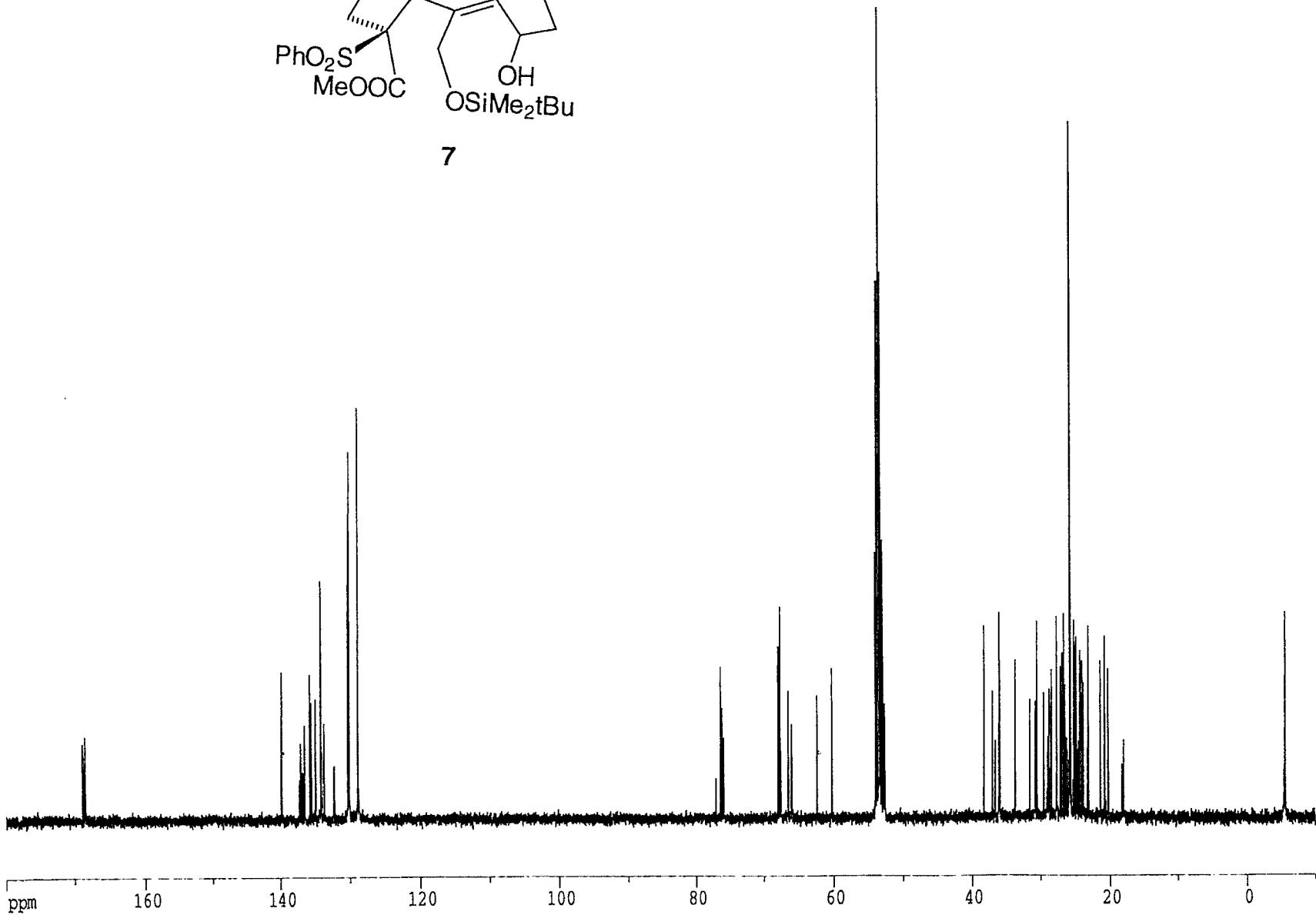
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7

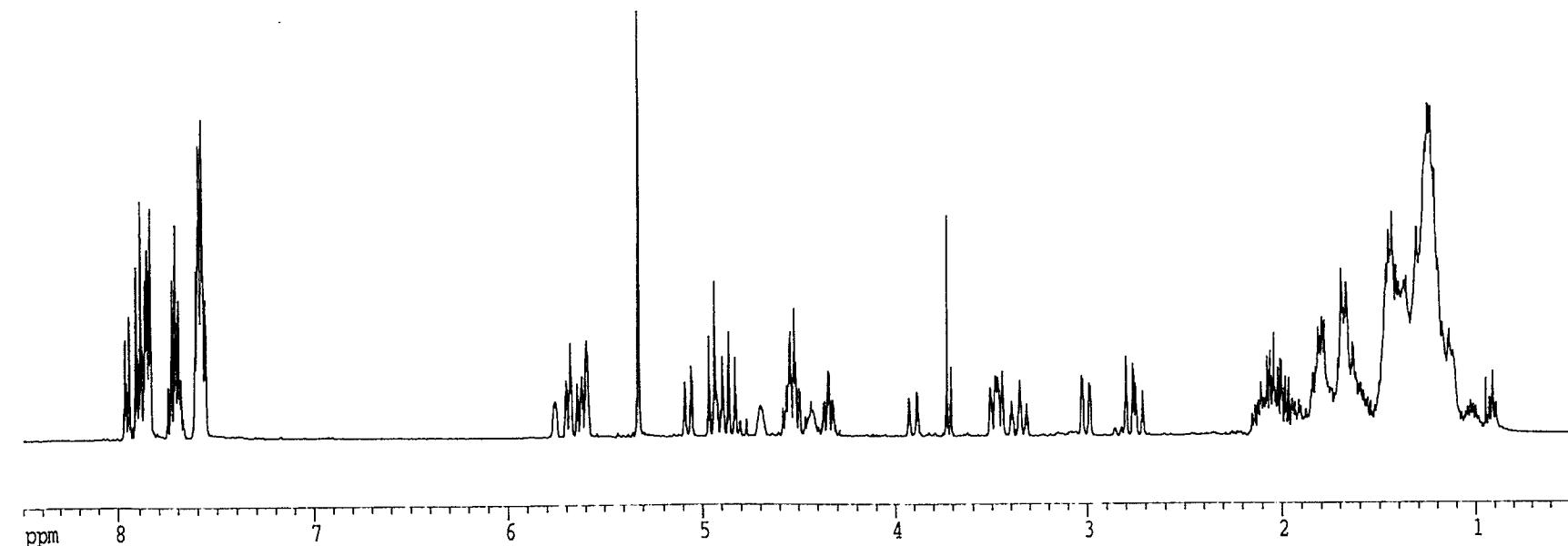
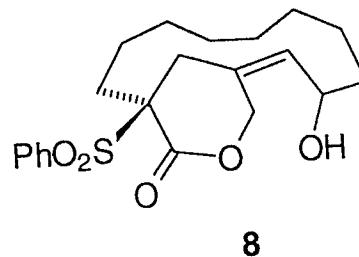


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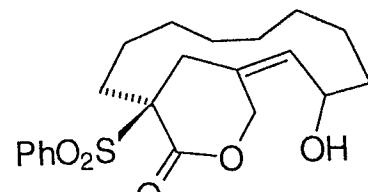


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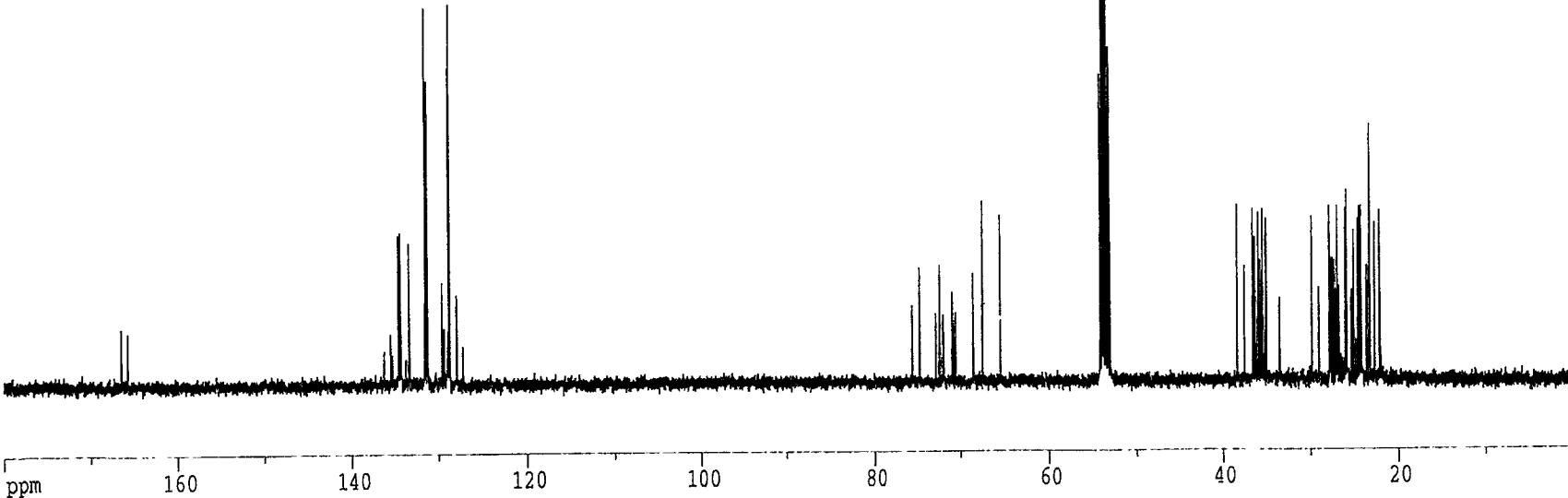
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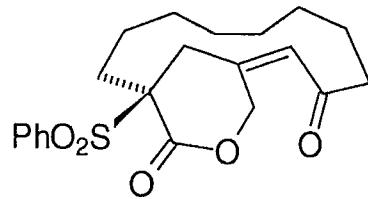
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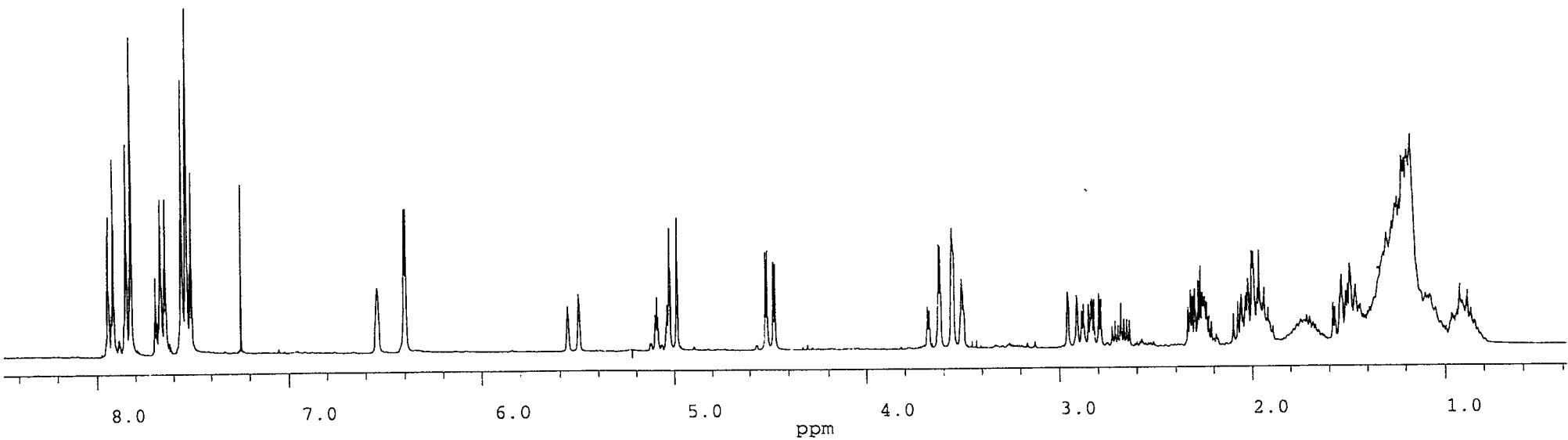


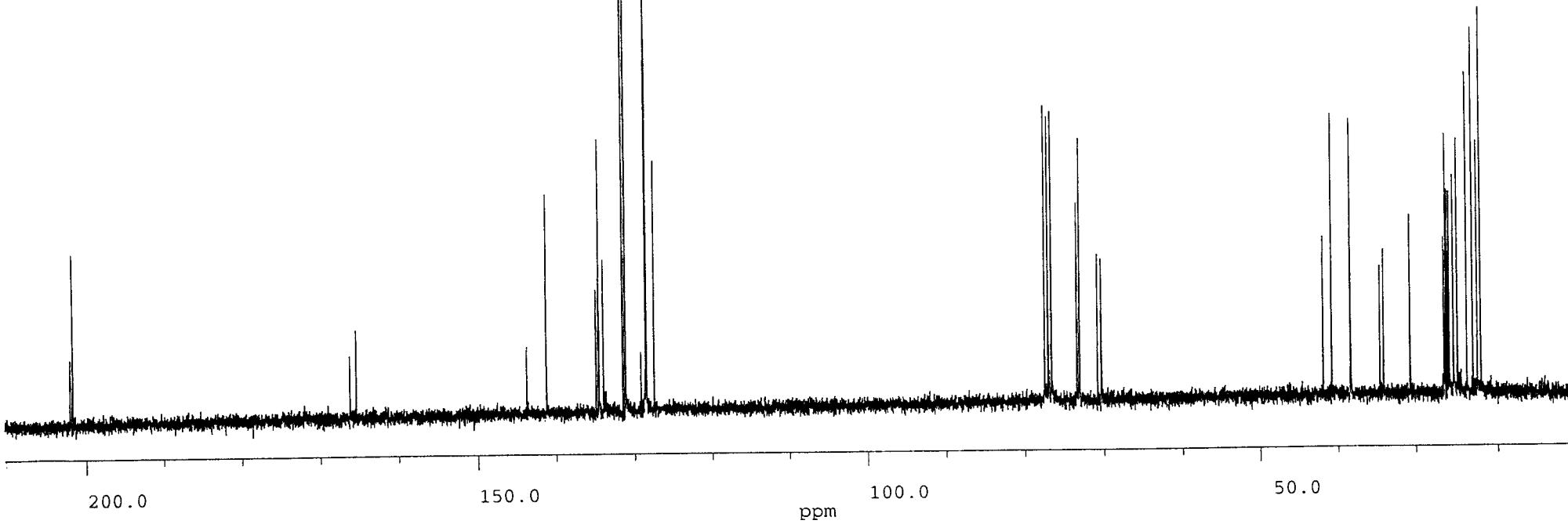
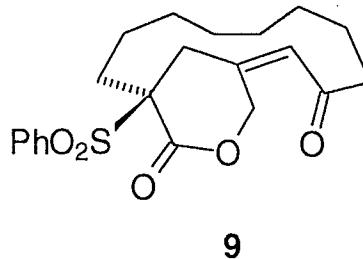
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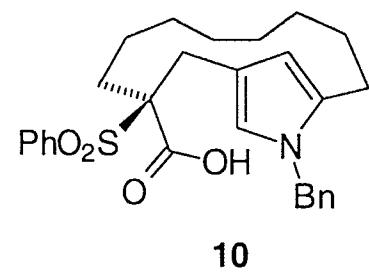




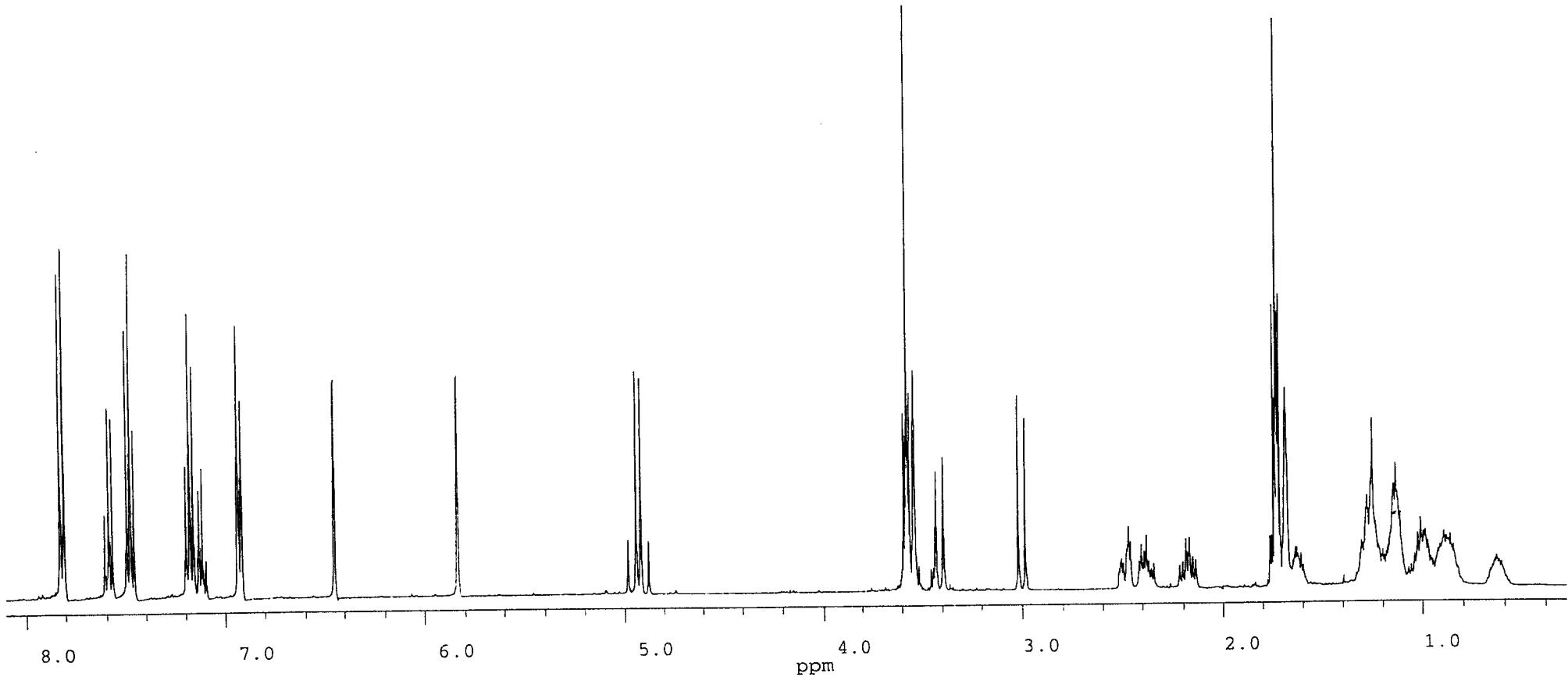
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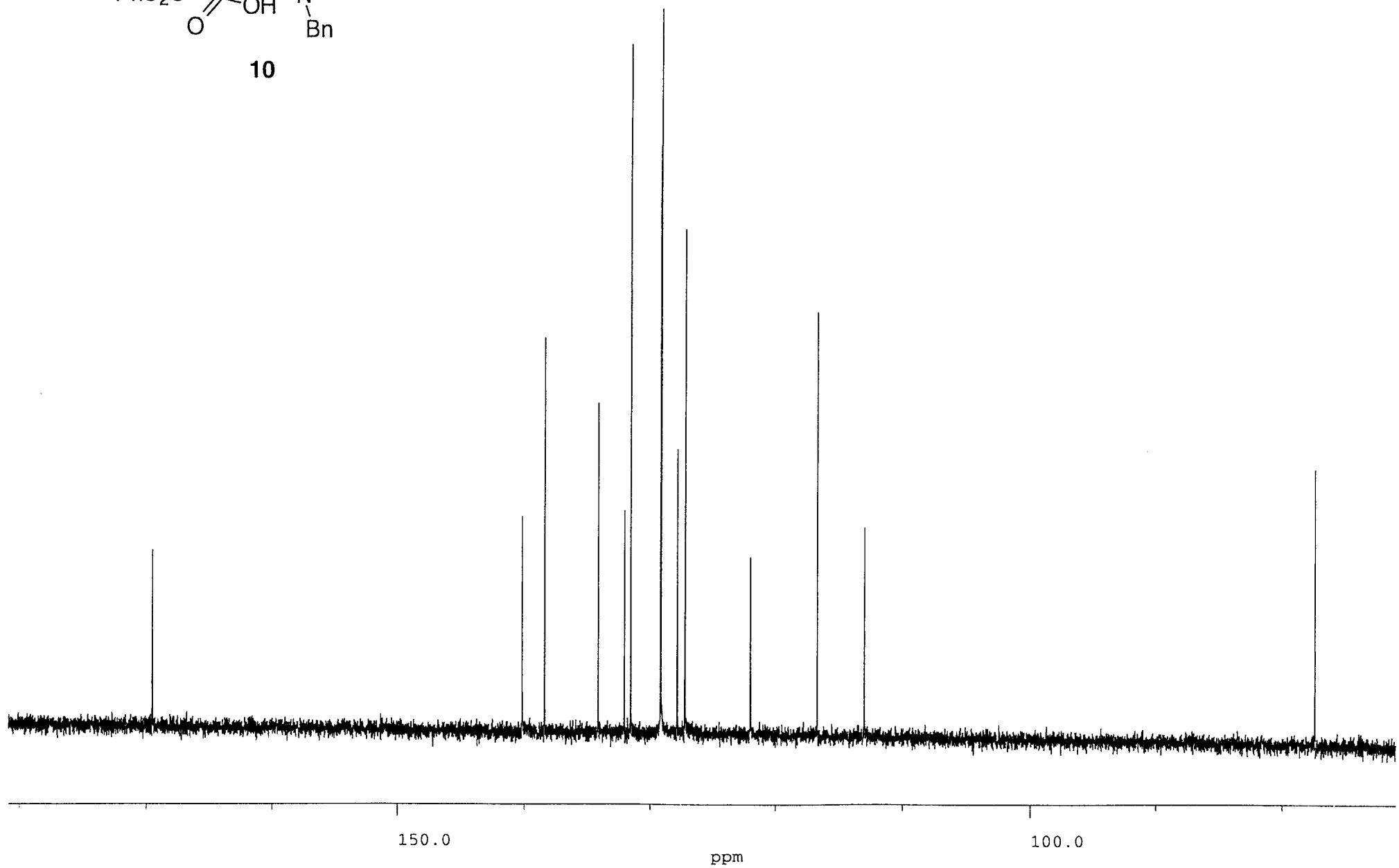
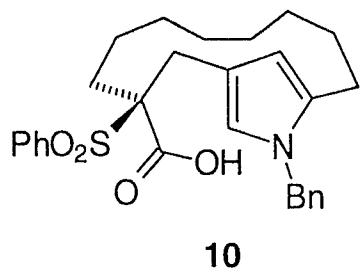


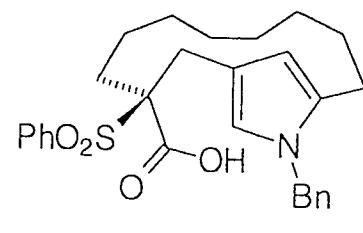




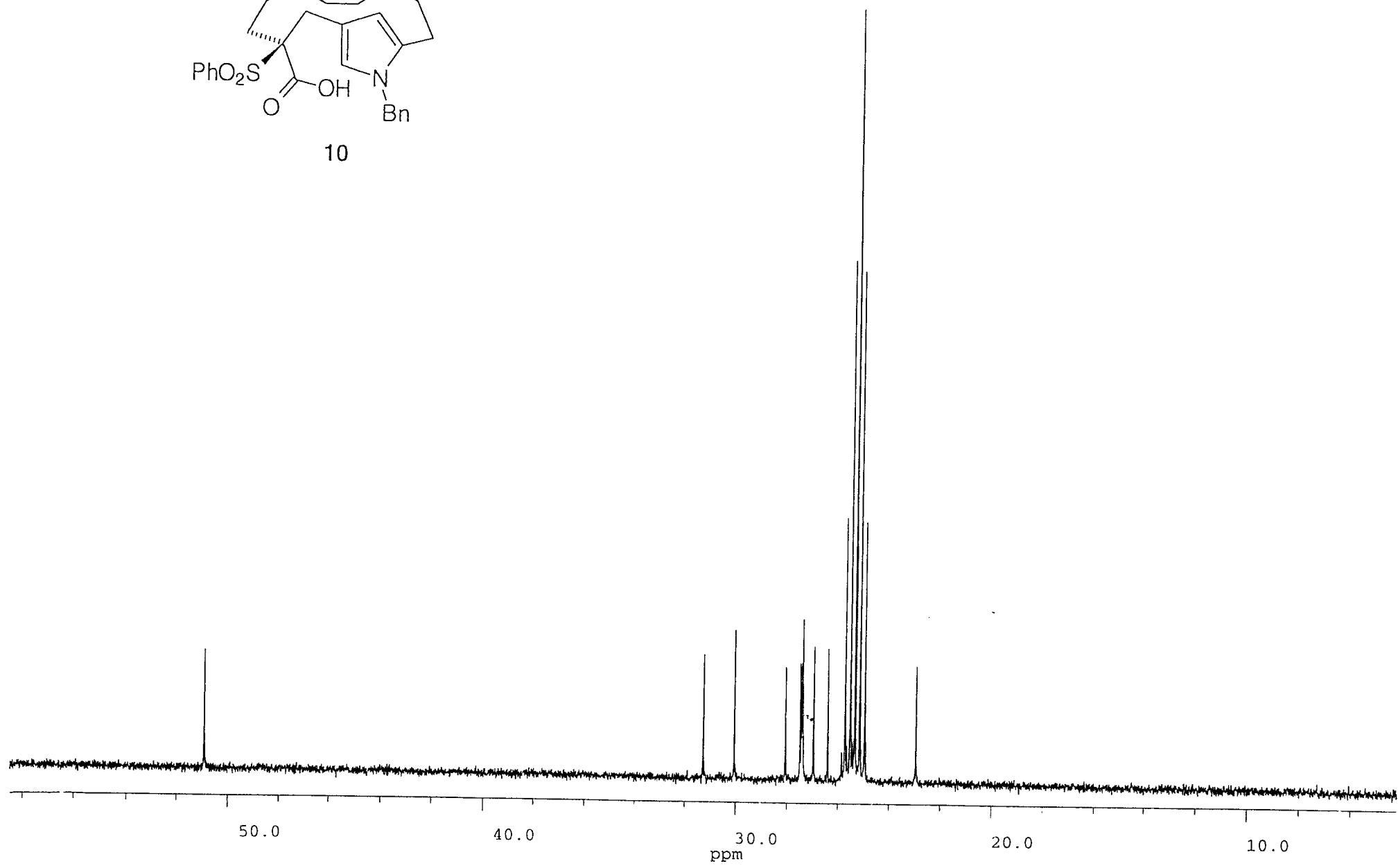
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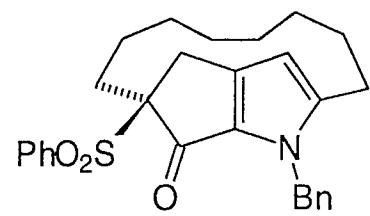




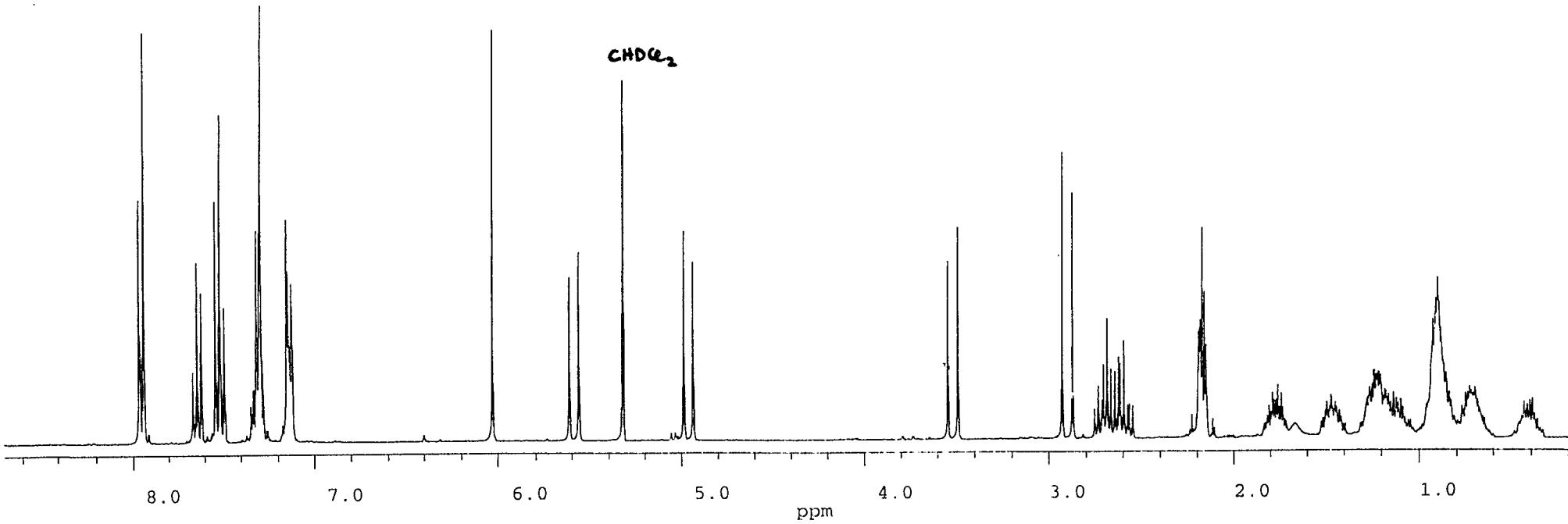


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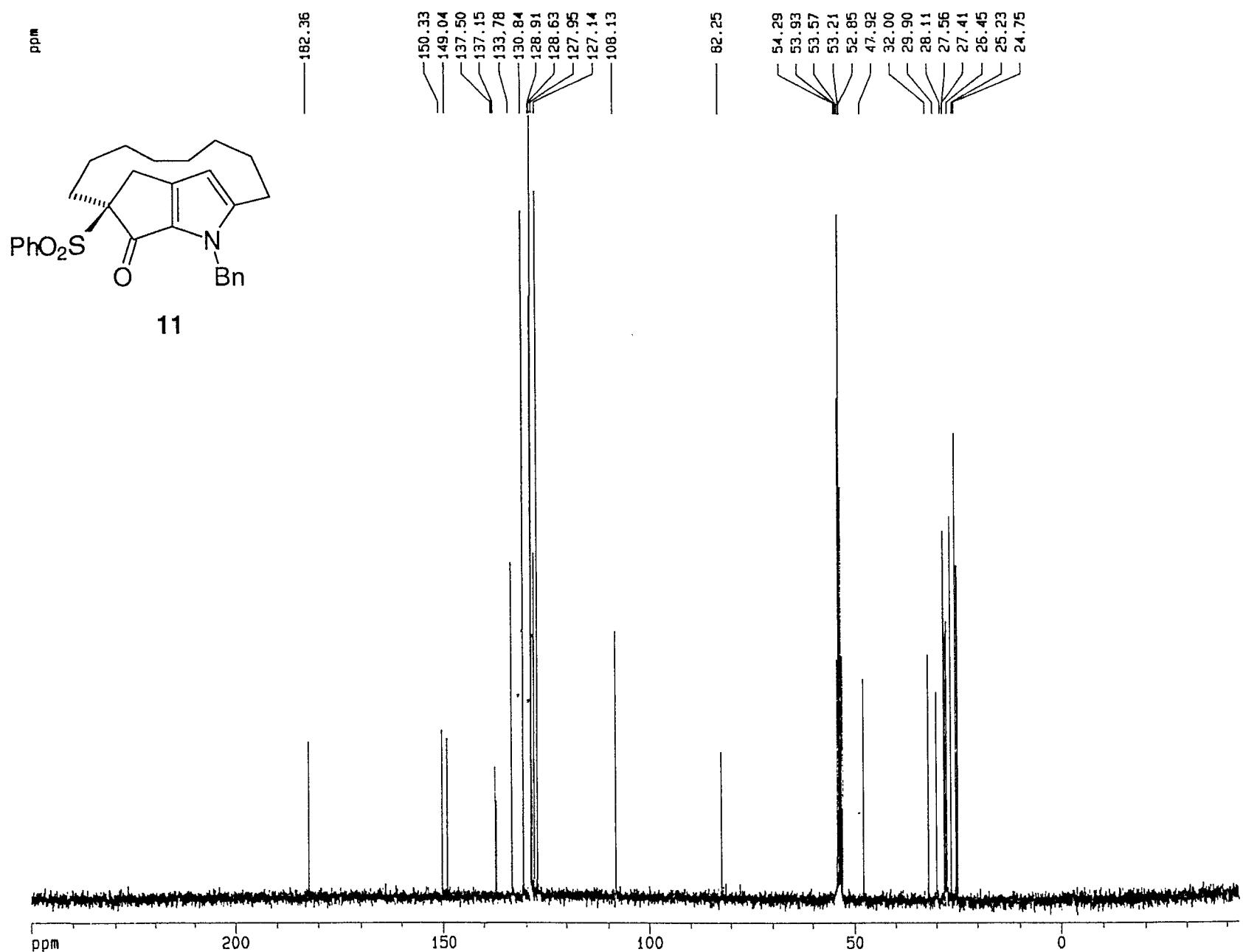




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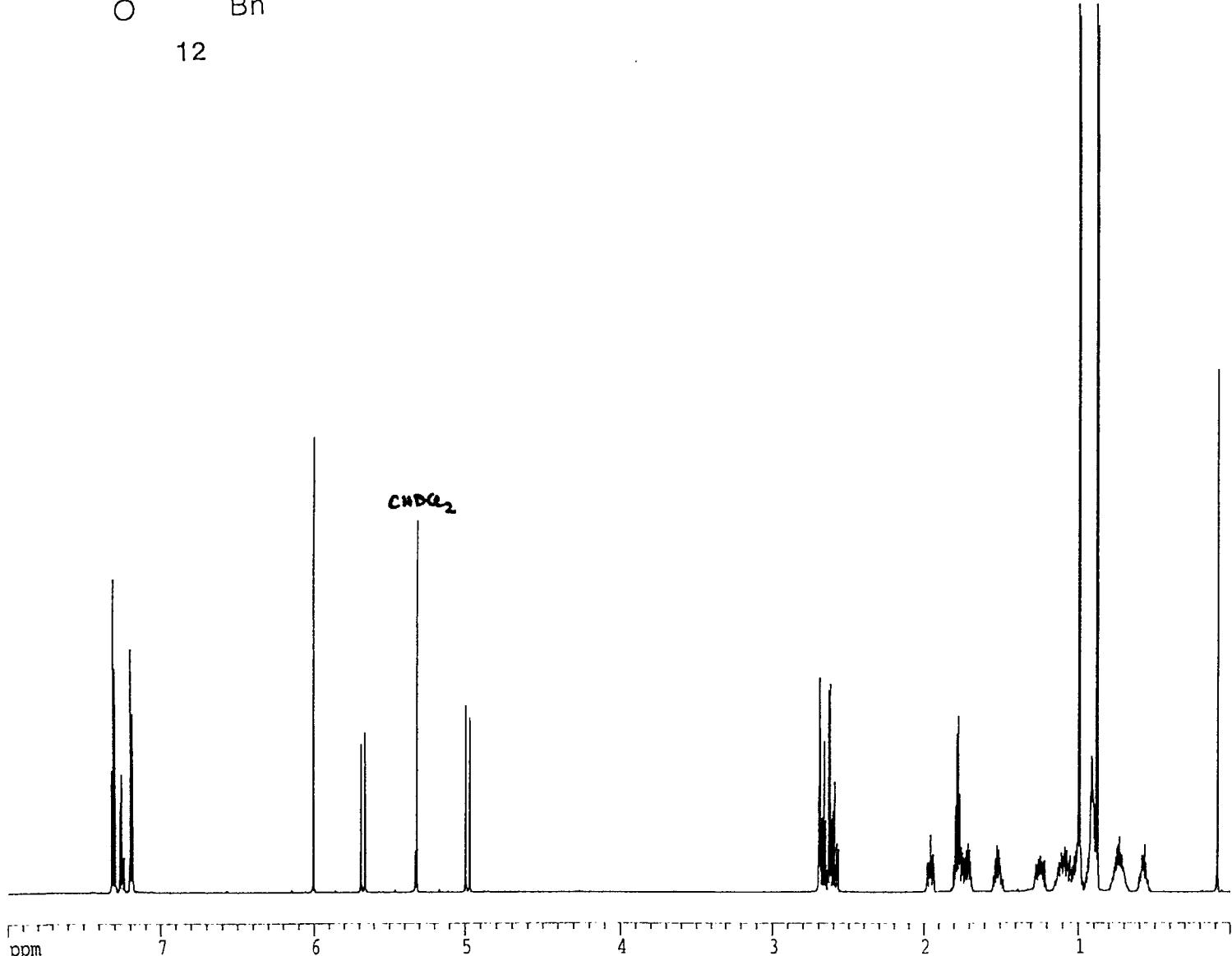
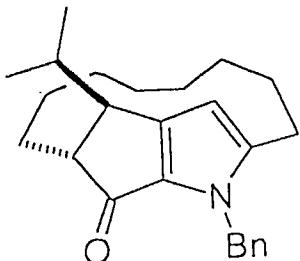


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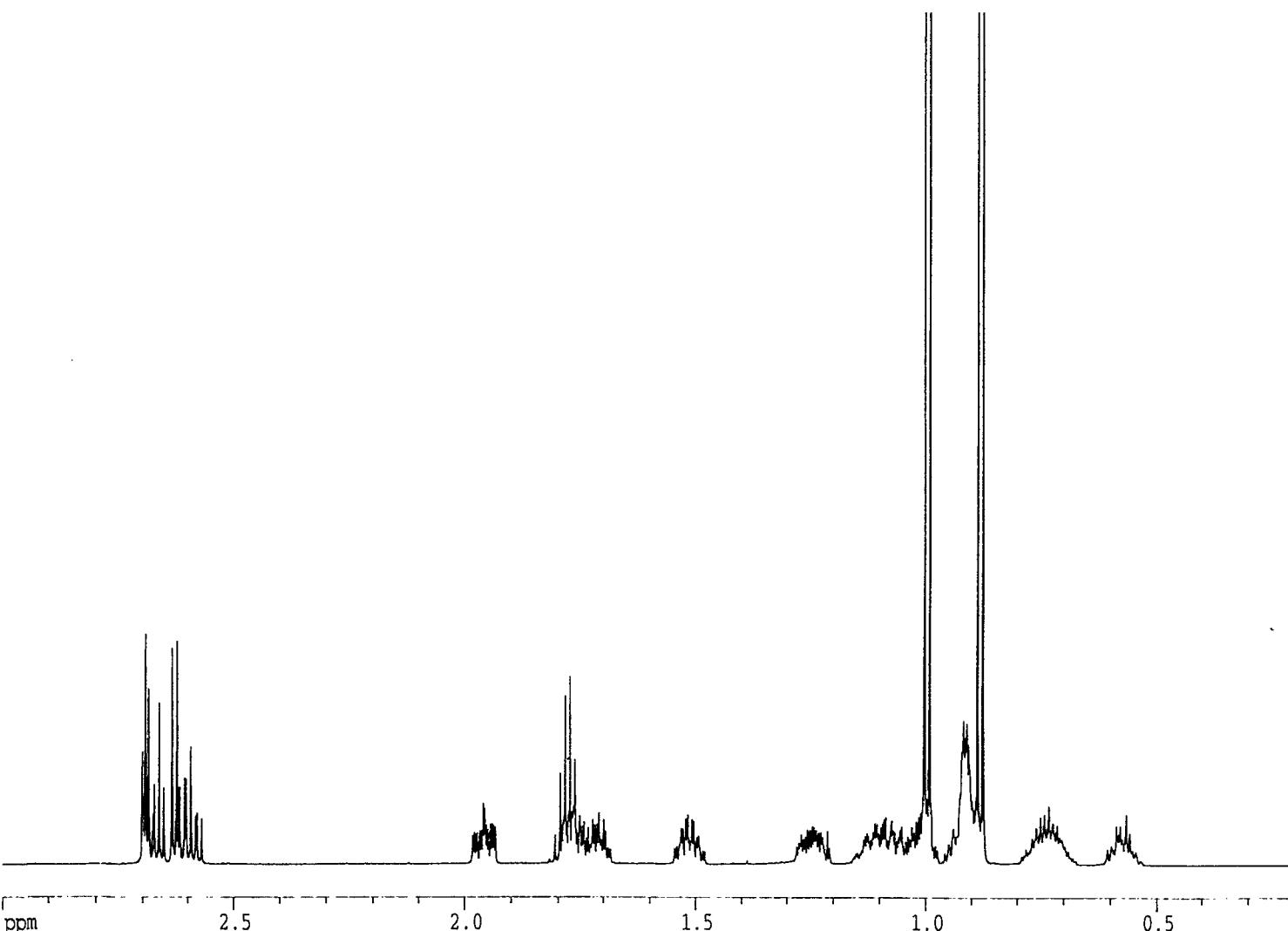
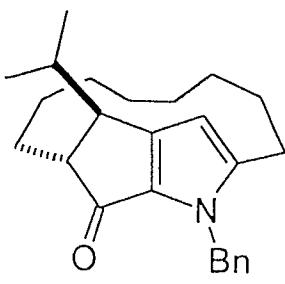


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 LB -0.30 Hz
 GB 0.18
 PC 0.50

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4801.76 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 240.08801 Hz/cm



Current Data Parameters

NAME wtt12601
EXPNO 10
PROCNO 1
DU mpi
USER wtt

F2 - Acquisition Parameters

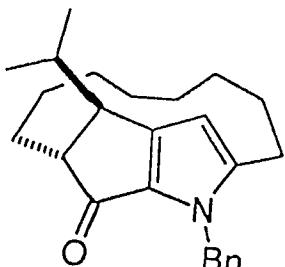
Date_ 961016
Time 14.59
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 32
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 128
DW 41.600 usec
DE 4.50 usec
TE 303.0 K
HL1 90 dB
D1 1.0000000 sec
P1 8.80 usec
DE 4.50 usec
SFO1 600.2242403 MHz
NUCLEUS 1H

F2 - Processing parameters

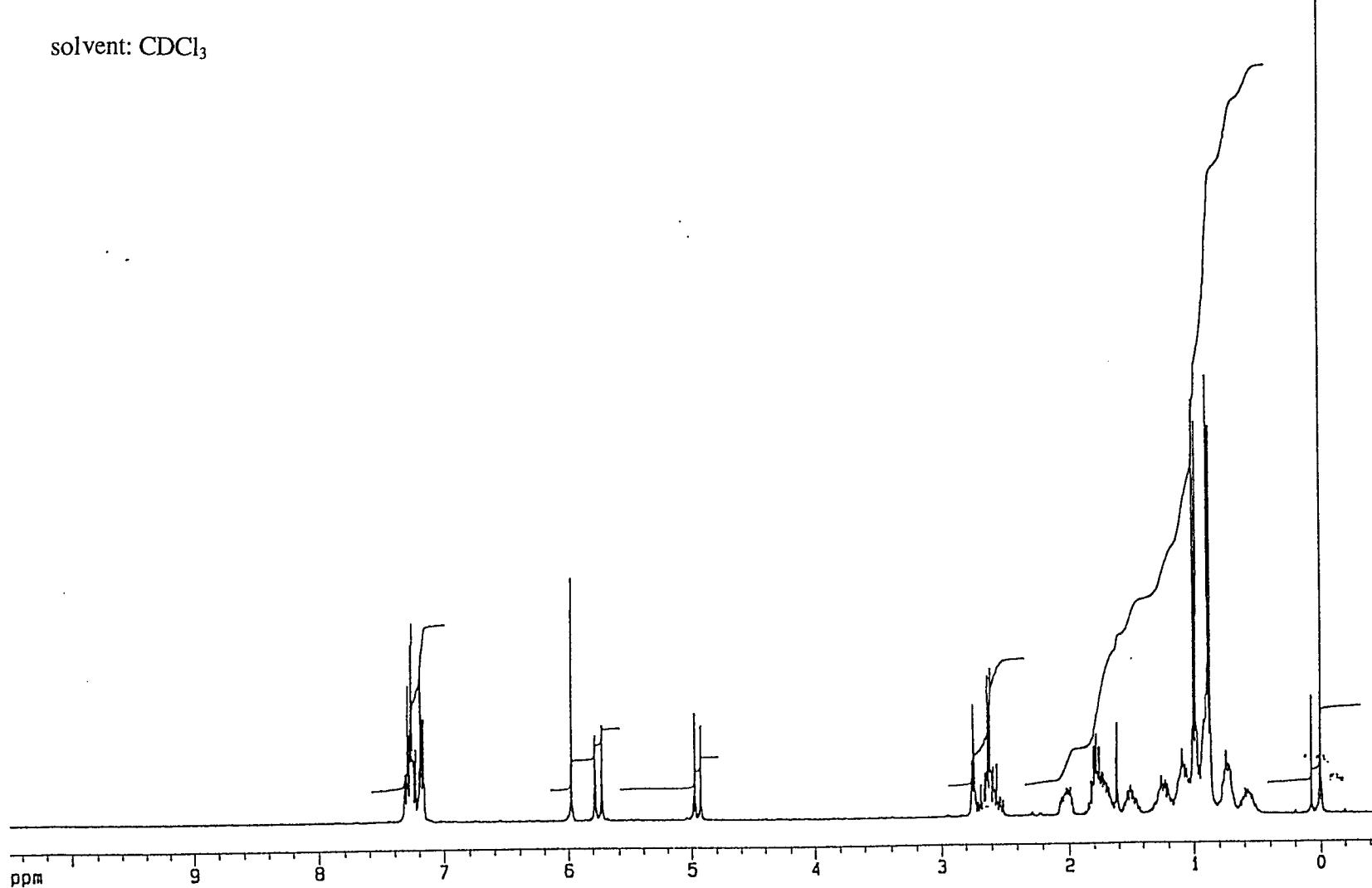
SI 65536
SF 600.2200217 MHz
WDW GM
SSB 0
LB -0.30 Hz
GB 0.18
PC 0.50

1D NMR plot parameters

CX 20.00 cm
F1P 3.000 ppm
F1 1800.66 Hz
F2P 0.200 ppm
F2 120.04 Hz
PPMCM 0.14000 ppm/cm
HZCM 84.03680 Hz/cm



solvent: CDCl₃

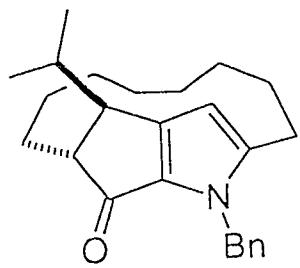


Current Data Parameters
NAME okt14102
EXPNO 10
PROCNO 1
DU u
USER et

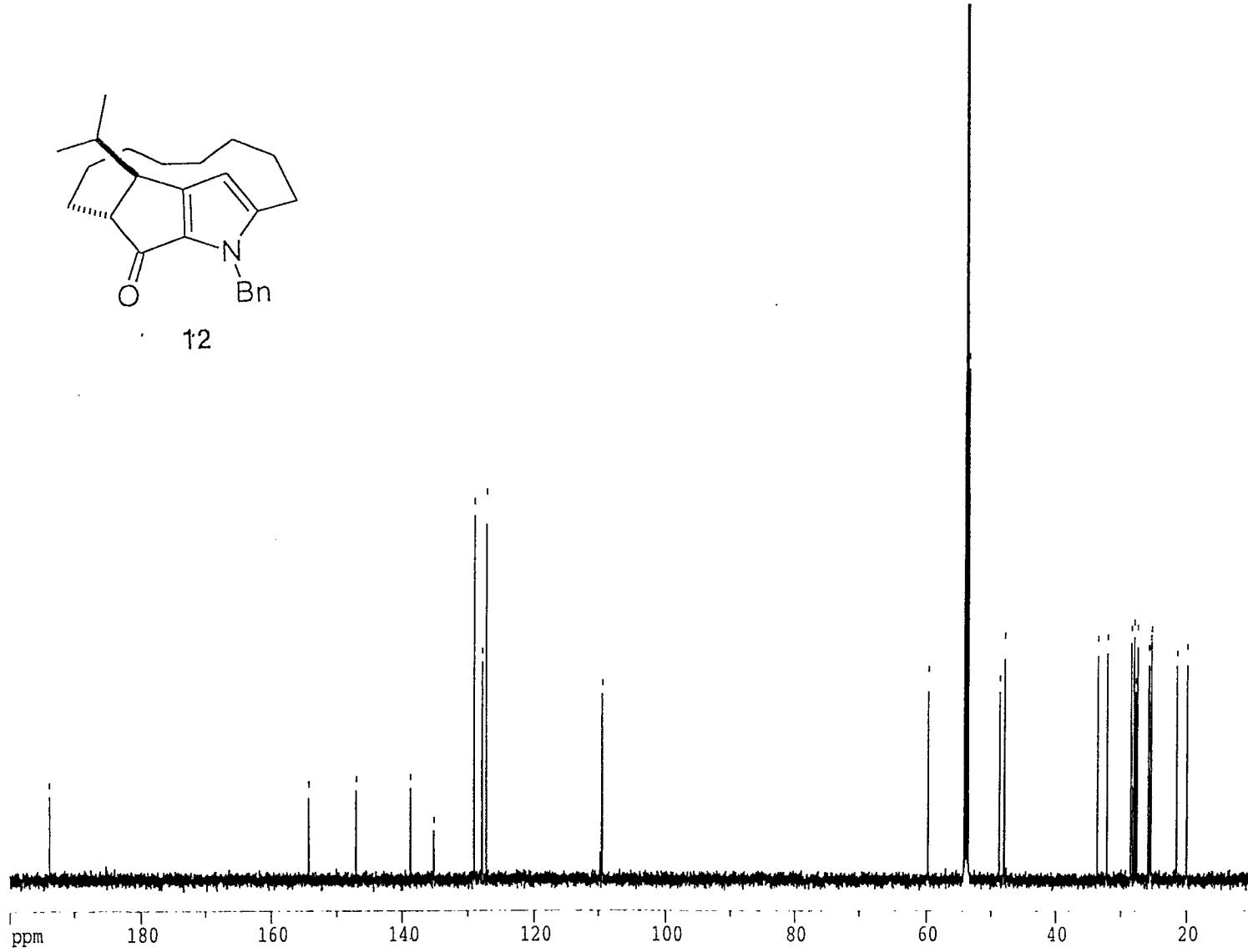
F2 - Acquisition Parameters
Date 961014
Time 16.57
PULPROG zg30
SOLVENT CDCl₃
AQ 2.5214736 sec
DW 80.0 usec
RG 512
NUCLEUS 1H
SF01 300.1349393 MHz
SF02 360.1319532 MHz
TE 302.0 K
HI 0 dB
D1 1.0000000 sec
P1 6.8 usec
DE 100.0 usec
SF01 300.1349393 MHz
SMH 6249.97 Hz
TD 32768
NS 32
DS 2

F2 - Processing parameters
SI 32768
SF 300.1333680 MHz
SR 3368.04 Hz
HzPPT 0.1907 Hz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
DC 8.00

1D NMR plot parameters
CX 22.10 cm
CY 14.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCH 0.49774 ppm/cm
HZCM 149.38765 Hz/cm



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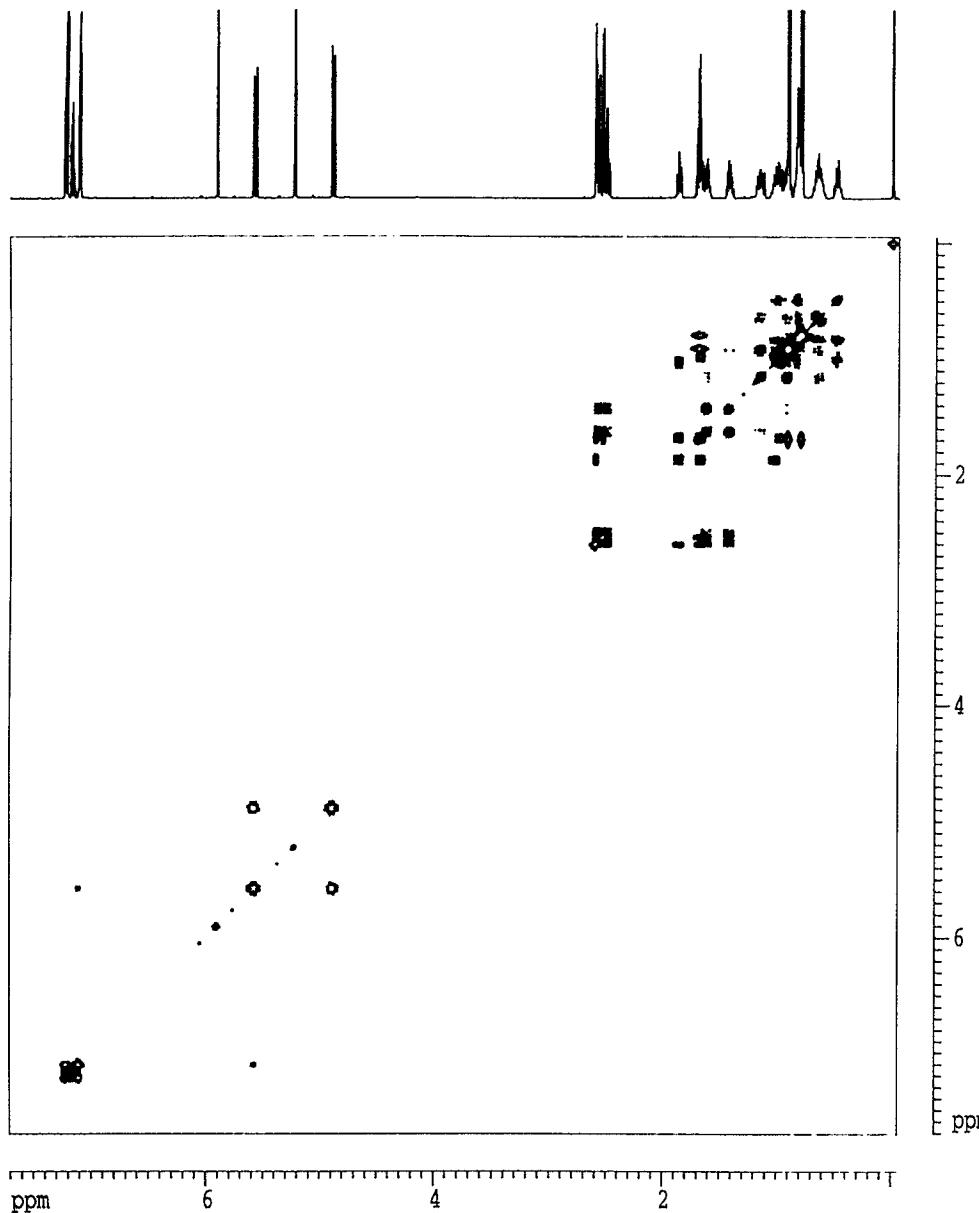
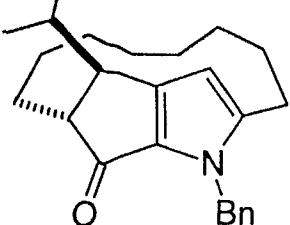


Current Data Parameters
 NAME wtt12601
 EXPNO 11
 PROCNO 1
 DU mpi
 USER wtt

F2 - Acquisition Parameters
 Date_ 961016
 Time 15.19
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgdc30
 TD 65536
 SOLVENT CD2Cl2
 NS 2269
 DS 8
 SWH 37593.984 Hz
 FIDRES 0.573639 Hz
 AQ 0.8716788 sec
 RG 16384
 DW 13.300 usec
 DE 4.50 usec
 TE 303.0 K
 D11 0.0300000 sec
 CPDPRG waltz16
 P31 75.00 usec
 S2 83 dB
 HLL 90 dB
 D1 0.0300000 sec
 P1 13.40 usec
 DE 4.50 usec
 SF01 150.9419346 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 65536
 SF 150.9253745 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 30185.07 Hz
 F2P 10.000 ppm
 F2 1509.25 Hz
 PPMCM 9.50000 ppm/cm
 HZCM 1433.79102 Hz/cm



Current Data Parameters
 NAME wtt12601
 EXPNO 14
 PROCHNO 1
 DU mpi
 USER wtt

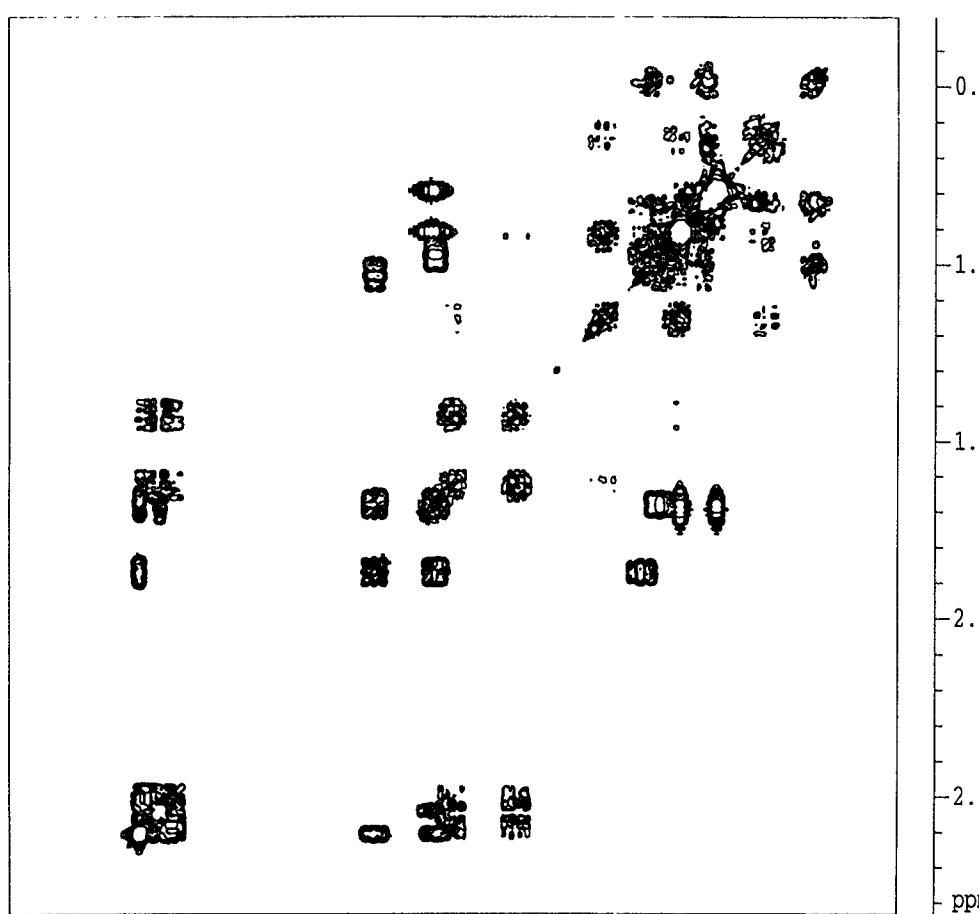
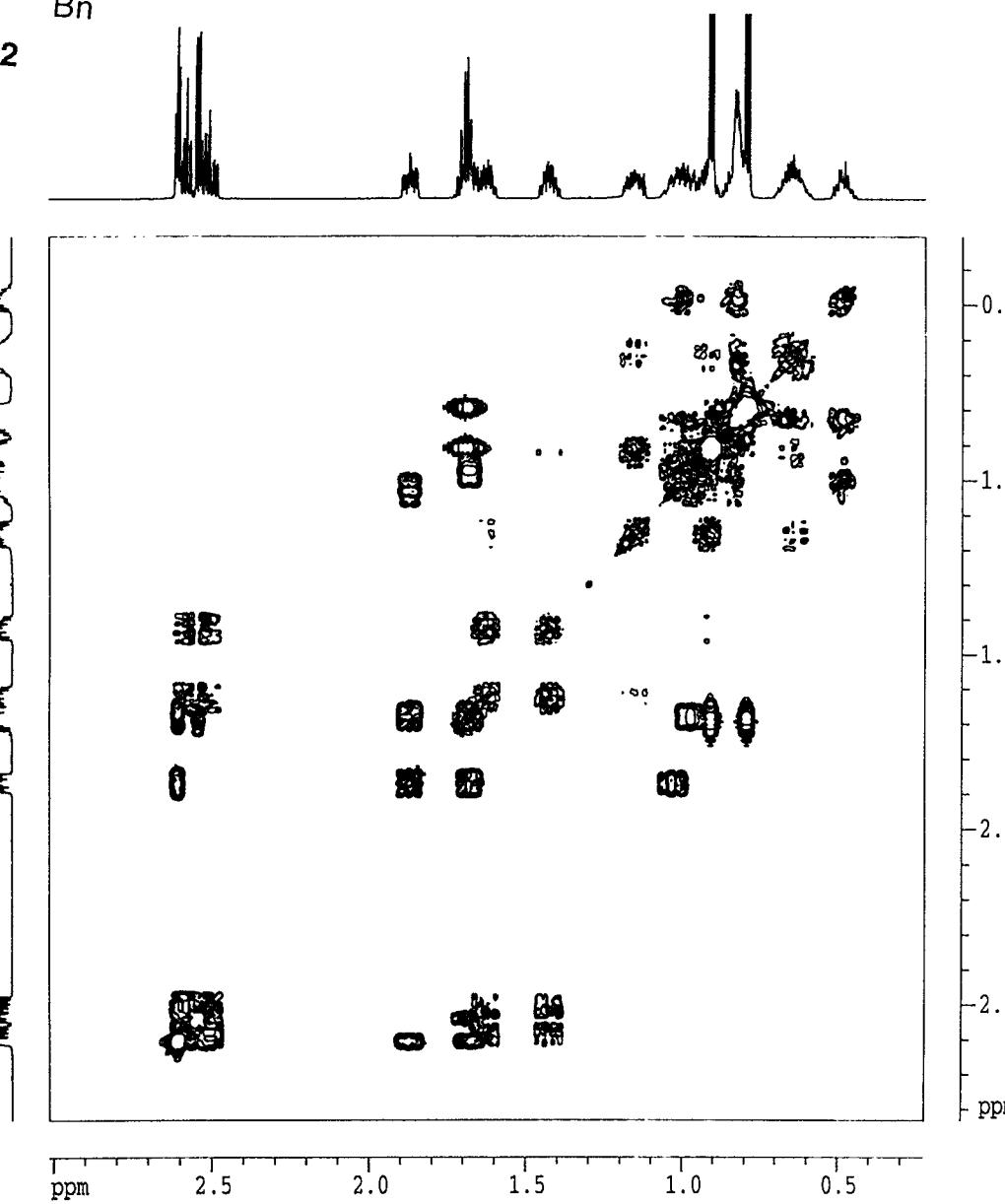
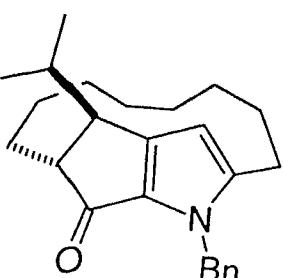
F2 - Acquisition Parameters
 Date_ 961016
 Time 18.01
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG cosys
 TD 1024
 SOLVENT CDCl₂
 NS 2
 DS 16
 SWH 4664.179 Hz
 AQ 0.1098228 sec
 RG 128
 DW 107.200 usec
 DE 4.50 usec
 TE 303.0 K
 D1 1.72118199 sec
 P1 8.80 usec
 D0 0.00000400 sec
 P16 1000.00 usec
 D16 0.00050000 sec
 P0 8.80 usec
 D13 0.00000400 sec
 DE 4.50 usec
 SFO1 600.2223708 MHz
 NUCL 1H
 PL1 0.00 dB
 INU 0.00021440 sec

F1 - Acquisition parameters
 NDO 1
 TD 512
 SFO1 600.2224 MHz
 FIDRES 9.109725 Hz
 SW 7.771 ppm

F2 - Processing parameters
 SI 1024
 SP 600.2200738 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 64.00

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 600.2200738 MHz
 MDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX2 12.00 cm
 CX1 12.00 cm
 F2FLO 7.712 ppm
 F2ILO 4629.04 Hz
 F2PHI -0.059 ppm
 F2HI -35.14 Hz
 F1FLO 7.712 ppm
 F1LO 4629.04 Hz
 F1PHI -0.059 ppm
 F1HI -35.14 Hz
 F2PPCM 0.64757 ppm/cm
 F2HZCM 388.68161 Hz/cm
 F1PPCM 0.64757 ppm/cm
 F1HZCM 388.68161 Hz/cm



Current Data Parameters
 NAME wtt12601
 EXPNO 14
 PROCNO 1
 DU mpi
 USER wtt

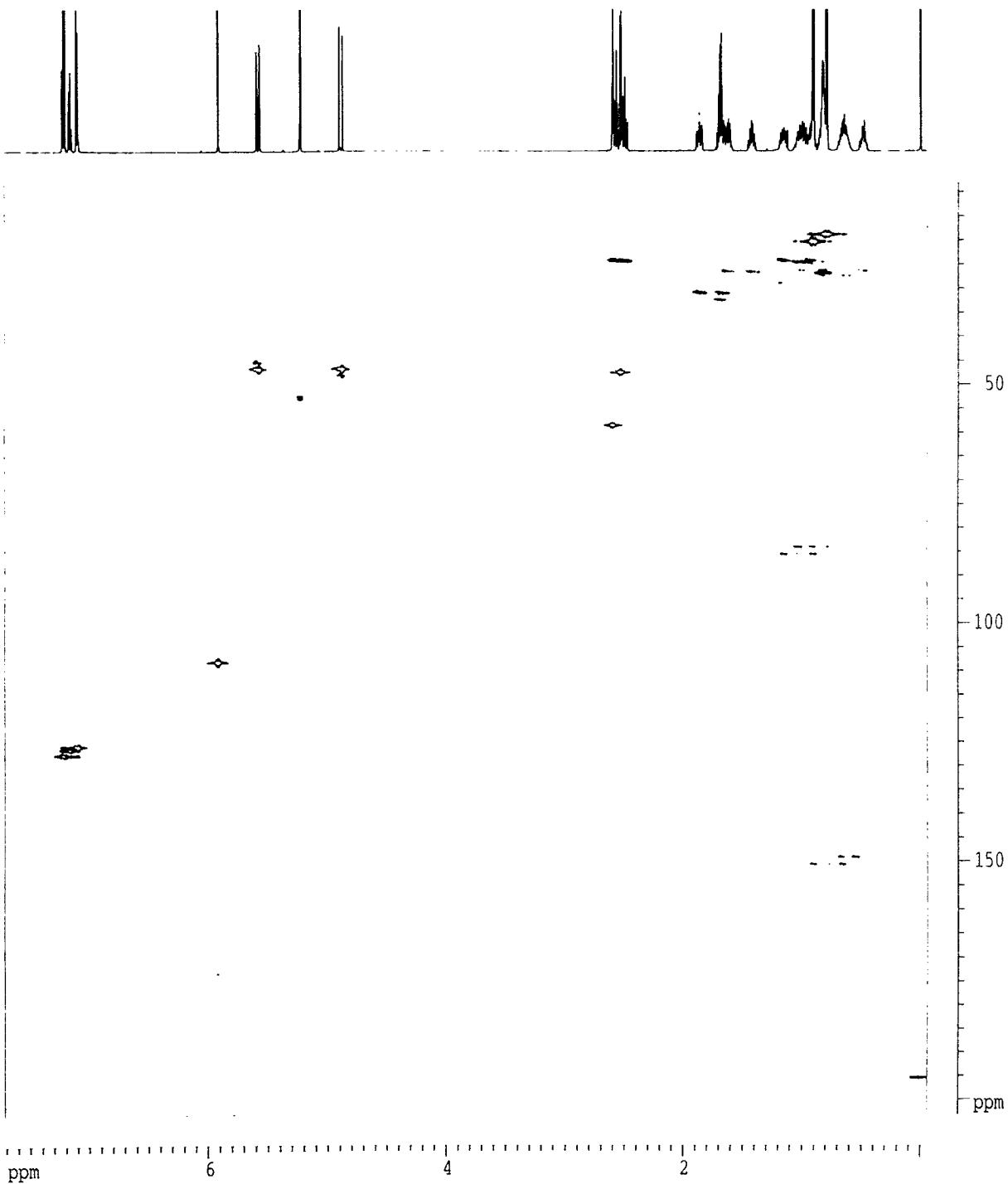
F2 - Acquisition Parameters
 Date_ 961016
 Time 18.01
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG cosy9s
 TD 1024
 SOLVENT CDCl3
 NS 2
 DS 16
 SWH 4664.179 Hz
 AQ 0.1098228 sec
 RG 128
 DW 107.200 usec
 DE 4.50 usec
 TE 303.0 K
 D1 1.72118199 sec
 P1 8.80 usec
 D0 0.00000400 sec
 P16 1000.00 usec
 D16 0.00050000 sec
 P0 8.80 usec
 D13 0.00000400 sec
 DE 4.50 usec
 SP01 600.2223708 MHz
 NMCL 1H
 PL1 0.00 dB
 IN0 0.00021440 sec

F1 - Acquisition parameters
 ND0 1
 TD 512
 SP01 600.2224 MHz
 FIDRES 9.109725 Hz
 SW 7.771 ppm

F2 - Processing parameters
 SI 1024
 SF 600.2200738 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 64.00

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 600.2200738 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX2 12.00 cm
 CX1 12.00 cm
 F2PLO 3.015 ppm
 F2LO 1809.58 Hz
 F2PHI 0.222 ppm
 F2HI 133.39 Hz
 F1PLO 2.833 ppm
 F1LO 1700.26 Hz
 F1PHI 0.306 ppm
 F1HI 183.49 Hz
 F2PPCM 0.23272 ppm/cm
 F2HZCM 139.68245 Hz/cm
 F1PPCM 0.21059 ppm/cm
 F1HZCM 126.39745 Hz/cm



Current Data Parameters
 NAME wtt12601
 EXPNO 15
 PROCNO 1
 DU mpu
 USER wtt

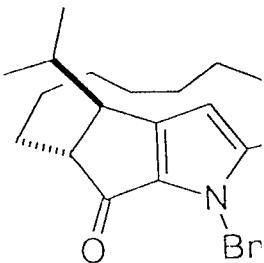
F2 - Acquisition Parameters:
 Date_ 961016
 Time 16:34
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG inv4gs
 TD 1024
 SOLVENT CD2Cl2
 NS 4
 DS 16
 SWH 4664.179 Hz
 AC 0.109228 sec
 RG 32768
 DW 107.000 usec
 DE 4.50 usec
 TE 301.0 ms
 D1 0.0001000 sec
 D2 0.0036000 sec
 P1 1000.00 usec
 D11 0.0000300 sec
 A2 0.0015770 sec
 D1 0.1927158 sec
 F1 8.35 usec
 PL 0.00 de
 P2 14.41 usec
 SPGS 150.9414971 MHz
 NMRI 1H
 D6 0.0000100 sec
 D16 0.0050000 sec
 P1 17.60 usec
 PL11 11.59 usec
 DE 4.50 usec
 SPG1 150.9414971 MHz
 NMRI 1H
 PLI 0.00 de
 CPDPG1 90deg
 PTDPL 15.50 usec
 INO 0.0011891 sec

F1 - Acquisition parameters:
 H60
 TD 1024
 CP01 150.9411 MHz
 FIDRES 24.807154 Hz
 SW 195.439 ppm

F2 - Processing parameters:
 SI 1024
 SF 600.1200778 MHz
 WMW SINE
 SWB 0
 LB 0.00 Hz
 GS 0
 PC 64.00

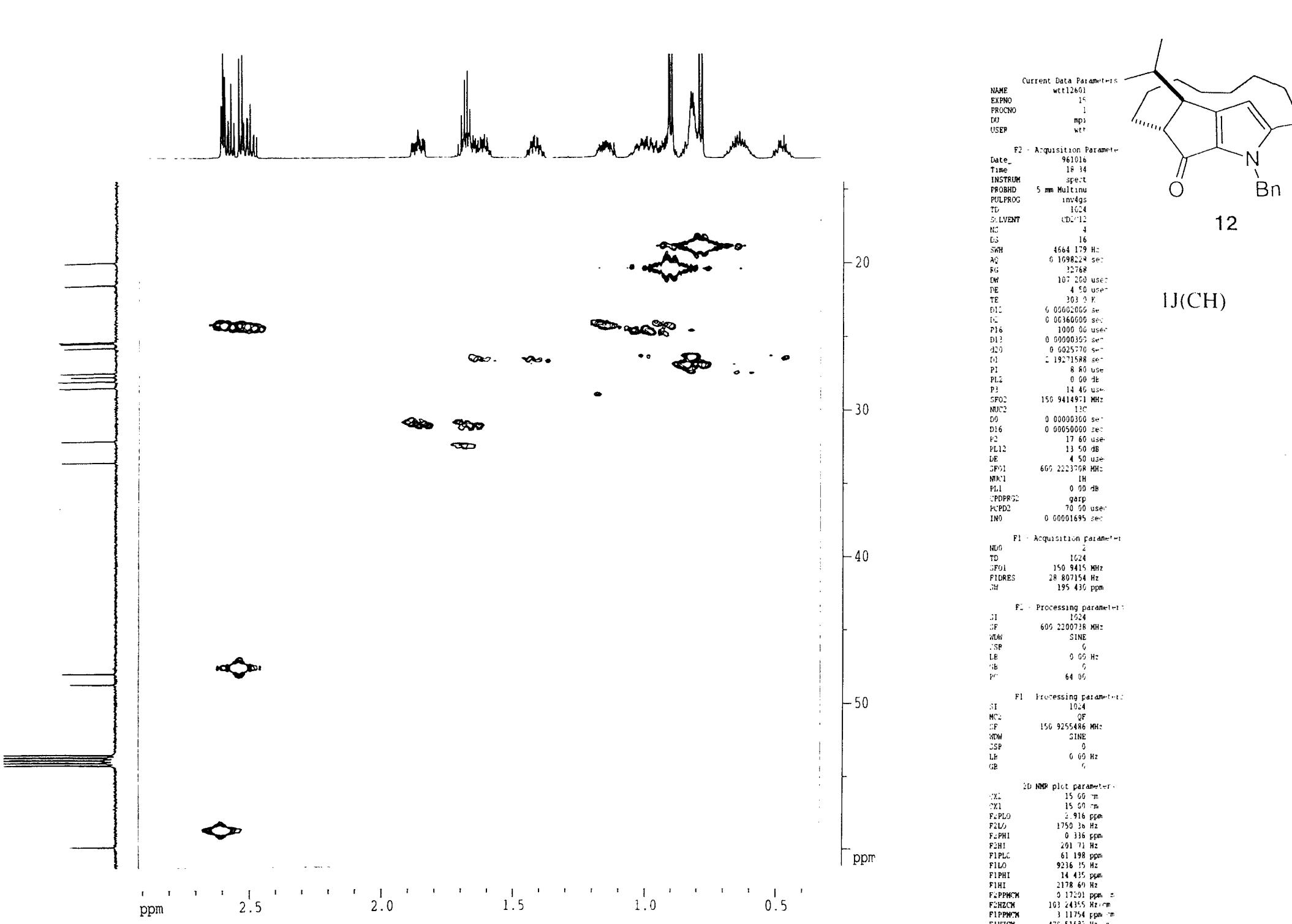
F1 - Processing parameters:
 SI 1024
 W0 16F
 SF 150.9255446 MHz
 WMW SINE
 SWB 0
 CSE 0
 LP 0.00 Hz
 GR 0

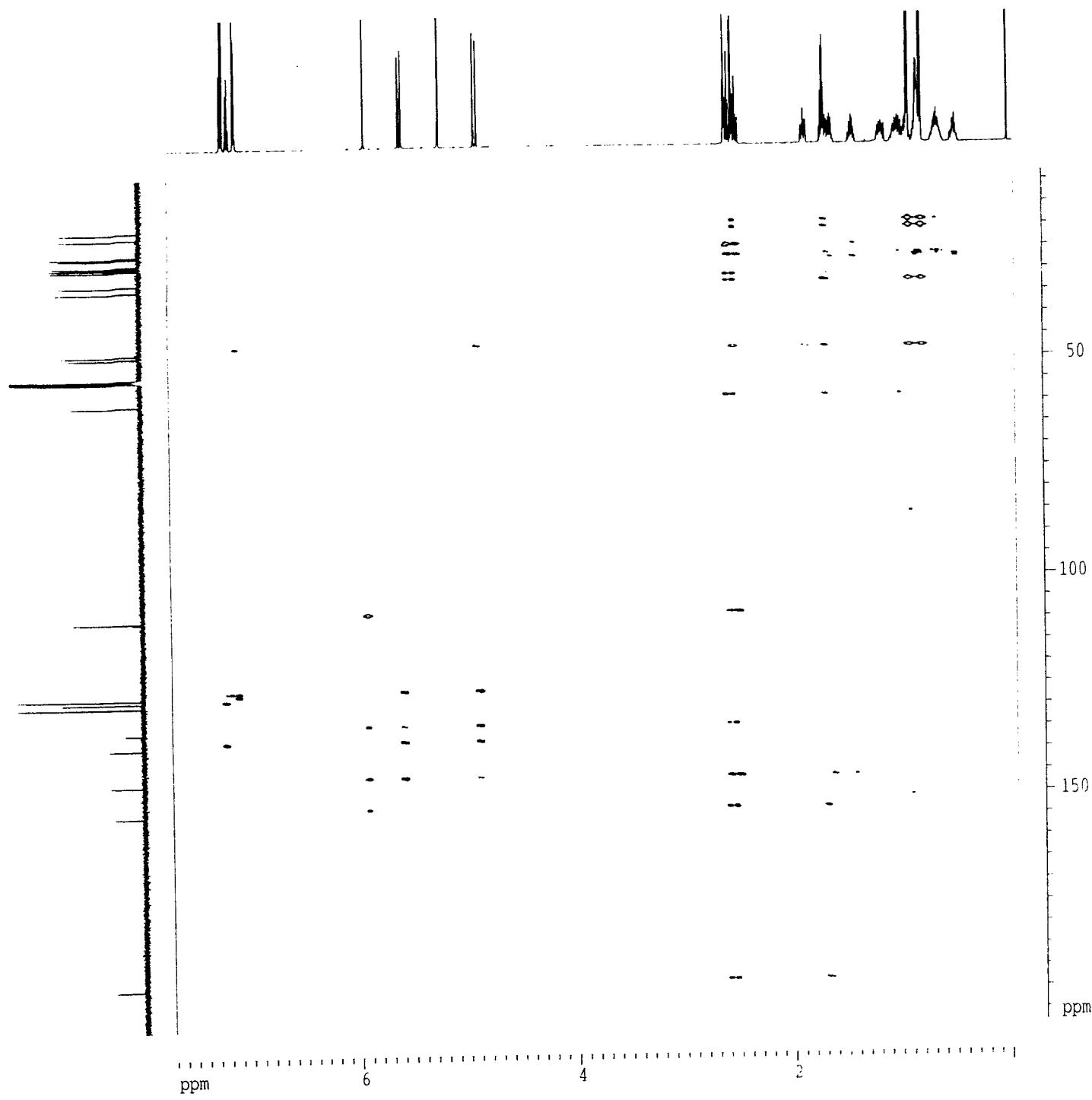
2D NMR plot parameters:
 CX1 15.00 cm
 CX2 15.00 cm
 F2PLQ 7.712 ppm
 F2LO 4629.04 Hz
 F2PH1 -9.059 ppm
 F2HI -15.14 Hz
 F1PLQ 203.396 ppm
 F1LO 39697.68 Hz
 F1PH1 7.945 ppm
 F1HI 1199.16 Hz
 F2PPMCM 0.51805 ppm/cm
 F1HZCM 310.94528 Hz·cm
 F1PPMCM 13.03066 ppm·cm
 F1HZCM 1966.56836 Hz·cm



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IJ(CH)





Current Data Parameters
NAME: att1601
EXPNO: 16
TD: 65536
TQ: 1
DT: 1024
SWH: 8000 Hz

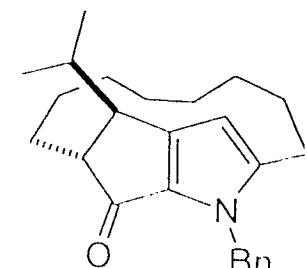
E1 Acquisition parameters:
Date: 9/16/06
Time: 11:14
INSTRUM: spect
PRSWP: 1 sec Multidim
DW128: 10.4 us
TE: 100 ms
D1: 65.6 ms
SF: 16 Hz
R1: 4000.179 Hz
A1: 10982.6 sec
F1: 31768 Hz
D11: 10.260 us
SF1: 4.50 us
TE1: 363.0 F
TD1: 65536000 sec
DW1: 6553600 sec
D12: 10.076 us
SF2: 2300300 sec
D13: 0.489375 sec
TE2: 19271588 sec
DW2: 8.80 us
D14: 0.001 sec
TE3: 14.40 us
DW3: 10.9414971 MHz
NUC1: 13C
PC1: 1.000000000 sec
TDn: 6 000500000 Le
P1: 11.50 us
TDd: 4.50 us
SF3: 6.251108 MHz
D15: 1H
TE4: 0.001 sec
DW4: 0.011 sec
P1: 0.001 sec
TD5: 0.001651 sec

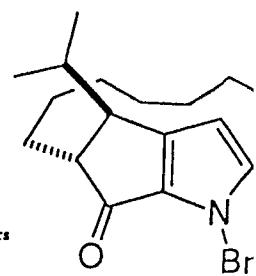
E1 Acquisition parameters:
DW: 16.4
TE: 100 ms
SF: 156.9415 MHz
P1: 2.8 807154 Hz
TD: 195410 rpm

E1 Processing parameters:
SF: 16.4
T1: 656.1250758 MHz
SW: 10000000 Hz
SF: 5
D1: 65.6 Hz
SF: 5
P1: 64.0

E1 FID scaling parameters:
SF: 16.4
M1: 16.4
SF: 140.9184466 MHz
SW: SINE
SF: 6
D1: 0.05 Hz
SF: 6

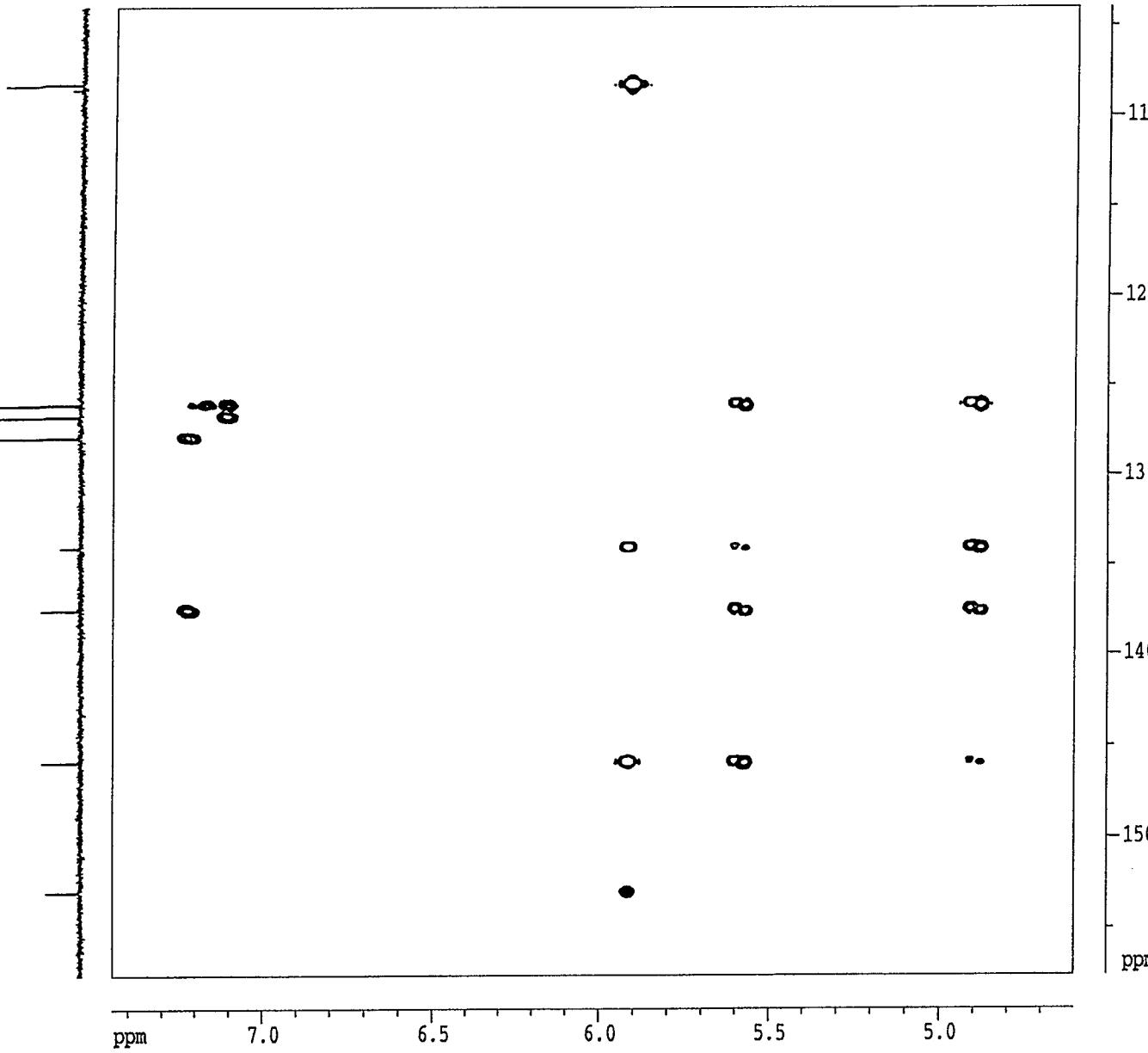
13C NMR plot parameters:
T1: 15.00 m
Z1: 15.00 m
F1,D1: 7.711 ppm
F1,D2: 4639.94 Hz
F1,D3: -0.059 ppm
F1,D4: 15.14 Hz
F1,D5: 201.396 ppm
F1,D6: 36697.65 Hz
F1,D7: 7.949 ppm
F1,D8: 1199.16 Hz
F1,D9: 0.41895 ppm, m
F1,D10: 110.94518 Hz, m
F1,D11: 13.01906 ppm, m
F1,D12: 1966.56816 Hz, m





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nJ(CH)



Current Data Parameters
NAME wtt12601
EXPNO 16
PROCNO 1
DU mpi
USER wtt

F2 - Acquisition Parameters
Date_ 961016
Time 21.14
INSTRUM spect
PROBOD 5 mm Multinucl
PULPROG invgs
TD 1024
SOLVENT CDCl3
NS 4
DS 16
SWH 4664.179 Hz
AQ 0.1098228 sec
RG 32768
DW 107.200 usec
DE 4.50 usec
TE 303.0 K
D12 0.00002000 sec
D2 0.05000000 sec
P16 1000.00 usec
D13 0.00000300 sec
d20 0.0485770 sec
D1 2.19271588 sec
P1 8.80 usec
PL2 0.00 dB
P3 14.40 usec
SF02 150.9414971 MHz
NUC2 13C
D0 0.00000300 sec
D16 0.00050000 sec
P2 17.60 usec
PL12 13.50 dB
DE 4.50 usec
SF01 600.2223708 MHz
NUC1 1H
PL1 0.00 dB
CPDPG2 garp
PCPD2 70.00 usec
IN0 0.00001695 sec

F1 - Acquisition parameters
ND0 2
TD 1024
SF01 150.9415 MHz
FIDRES 28.807154 Hz
SW 195.430 ppm

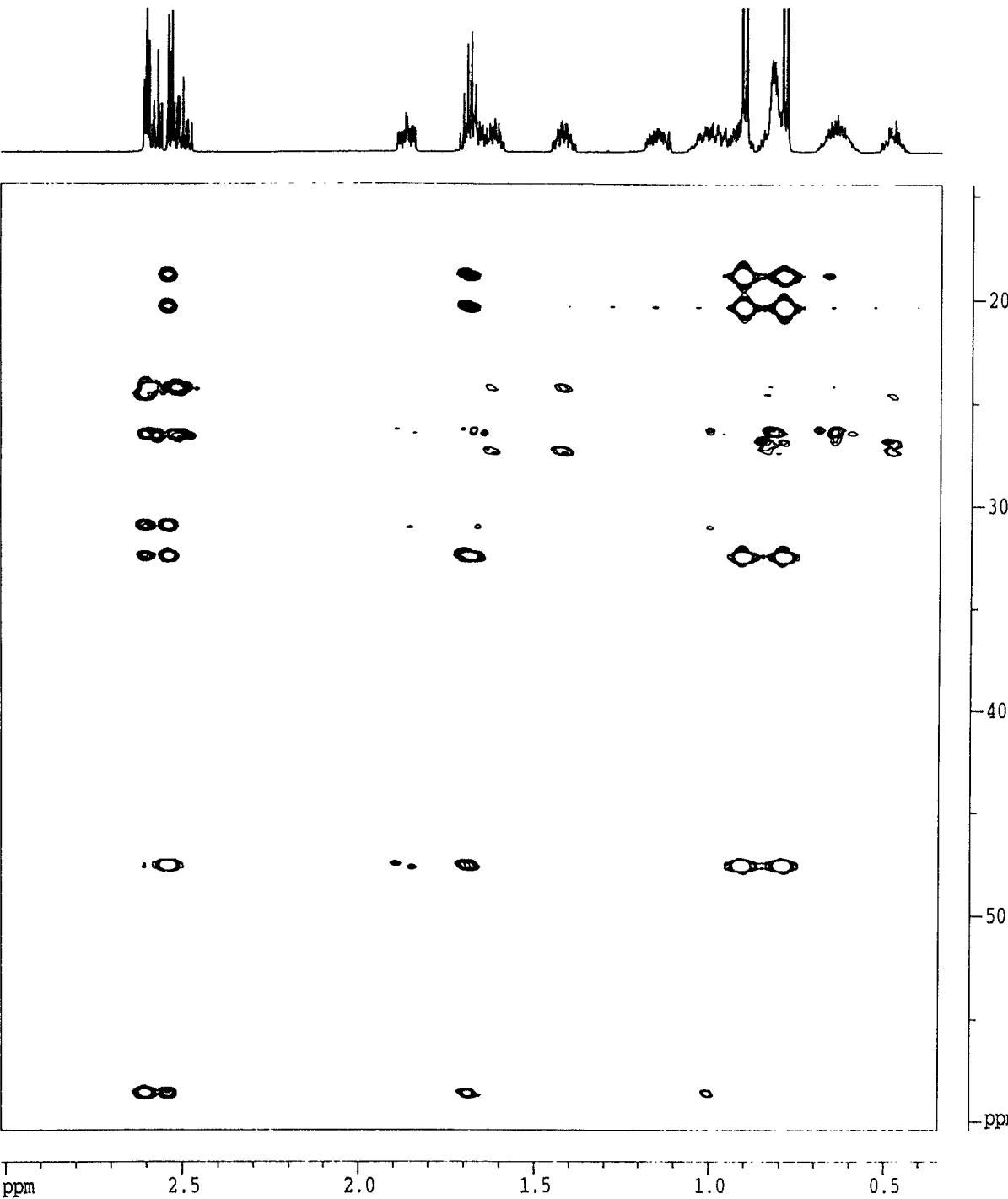
F2 - Processing parameters
SI 1024
SP 600.2200738 MHz
MDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 64.00

F1 - Processing parameters
SI 1024
MC2 QF
SP 150.9255486 MHz
W0W SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PL0 7.447 ppm
F1LO 4469.52 Hz
F2PHI 4.601 ppm
F2RHI 2761.54 Hz
F1M0 157.587 ppm
F1L0 23783.96 Hz
F1PHI 101.953 ppm
F1RHI 15689.16 Hz
F2PPHCH 0.18972 ppm/cm
F2R2CN 113.87156 Hz/cm
F1PPHCH 3.57563 ppm/cm
F1R2CH 539.65405 Hz/cm

110
120
130
140
150

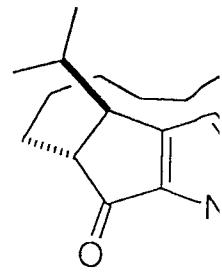
ppm



Current Data Parameters
 NAME wtt12601
 EXPNO 16
 PROCHD 1
 DU mpi
 USER wtt

F2 - Acquisition Parameters

Date_ 961016
 Time 21 14
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG invqgs
 TD 1024
 SOLVENT CD2Cl2
 NS 4
 DS 16
 SWH 4664.179 Hz
 AQ 0.1098228 sec
 RG 32768
 DM 107.200 usec
 D2 4.50 usec
 TZ 303.0 K
 D12 0.0000200 sec
 D2 0.0500000 sec
 P16 1000.00 usec
 D13 0.00000300 sec
 D20 0.048970 sec
 D1 2.19271588 sec
 P1 8.80 usec
 PL2 0.00 dB
 P3 14.40 usec
 SF02 150.9414971 MHz
 MUC2 13C
 D0 0.00000300 sec
 D16 0.0050000 sec
 P2 17.60 usec
 PL12 13.50 dB
 DE 4.50 usec
 SF01 600.2223708 MHz
 MUC1 1H
 PL1 0.00 dB
 CPDPG2 garp
 PCPD2 70.00 usec
 IND 0.00001695 sec



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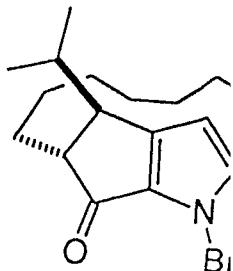
nJ(Ch)

F1 - Acquisition parameters
 NDD 2
 TD 1024
 SF01 150.9415 MHz
 FIDRES 28.807154 Hz
 SW 195.430 ppm

F2 - Processing parameters
 SI 1024
 SF 600.2200738 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 64.00

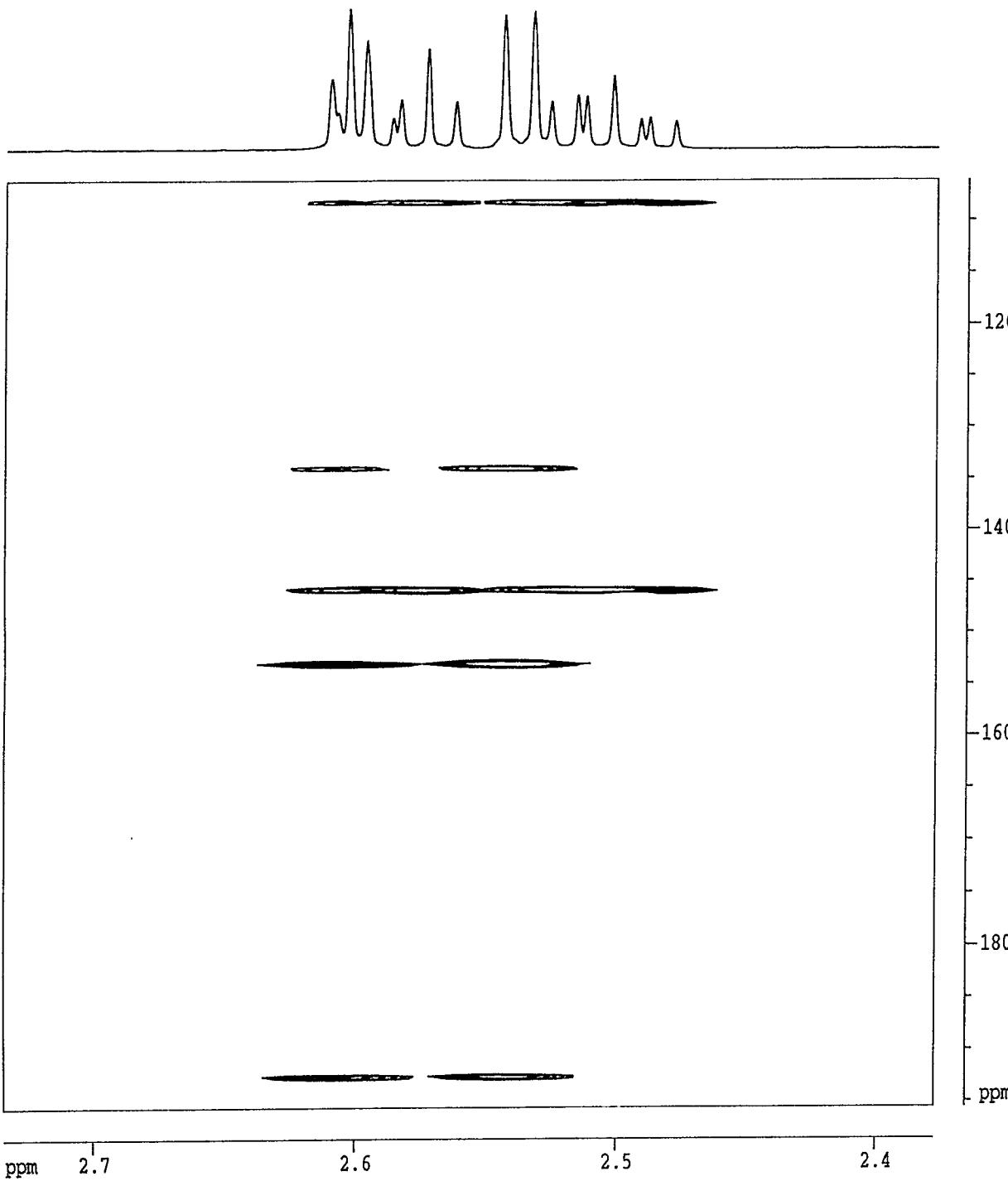
F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 150.9255486 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX2 15.00 cm
 CX1 15.00 cm
 F2PL0 3.015 ppm
 F2LO 1809.58 Hz
 F2PHI 0.344 ppm
 F2HI 206.27 Hz
 F1PL0 60.435 ppm
 F1LO 9121.12 Hz
 F1PHI 14.435 ppm
 F1HI 2178.60 Hz
 F2PPMCH 0.17808 ppm/cm
 F1HZCM 106.88744 Hz/cm
 F1PPMCH 3.06684 ppm/cm
 F1HZCM 462.83453 Hz/cm



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nJ(CH)



Current Data Parameters
NAME wti2601
EXPNO 16
PROCNO 1
DU mpi
USER wt

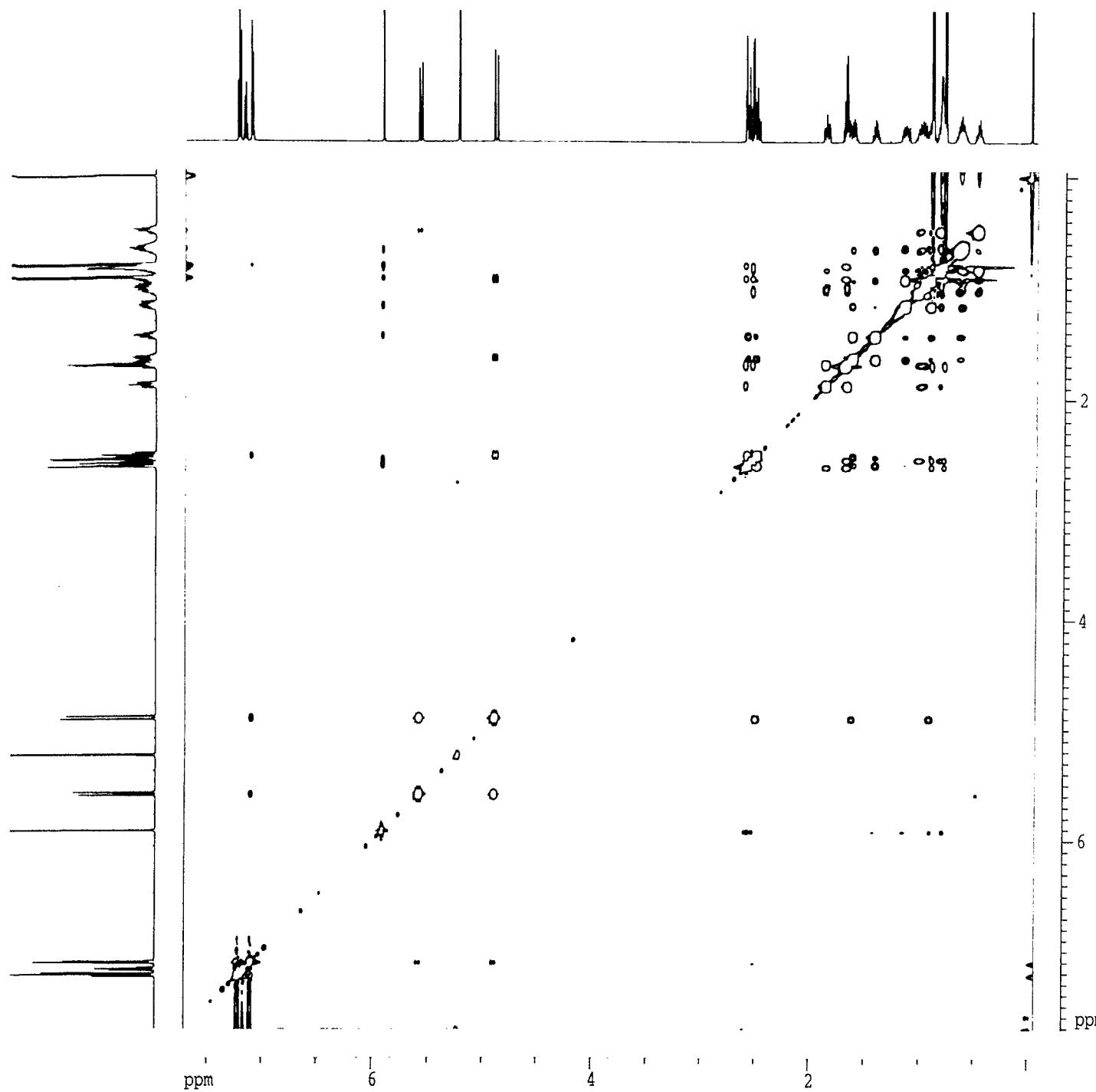
F2 - Acquisition Parameters
Date_ 961016
Time 21.14
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG inv1gs
TD 1024
SOLVENT CDCl₃
NS 4
DS 16
SWH 4664.179 Hz
AQ 0.1098228 sec
RG 32768
DM 107.200 usec
DE 4.50 usec
TE 303.0 K
D12 0.00002000 sec
D2 0.05000000 sec
P16 1000.00 usec
D13 0.00000300 sec
d20 0.0489770 sec
D1 2.19271581 sec
P1 8.00 usec
PL2 0.00 dB
P3 14.40 usec
SP02 150.9414971 MHz
NUC2 13C
D0 0.00000300 sec
D16 0.00050000 sec
P2 17.60 usec
PL12 13.50 dB
DR 4.50 usec
SP01 600.2223708 MHz
NUC1 1H
PL1 0.00 dB
CPDPBG2 garp
PCPD2 70.00 usec
INO 0.00001695 sec

F1 - Acquisition parameters
ND0 2
TD 1024
SP01 150.9415 MHz
FIDRES 28.807154 Hz
SW 195.430 ppm

F2 - Processing parameters
SI 1024
SF 600.2200738 MHz
WDW SINC
SSB 0
LB 0.00 Hz
GB 0
PC 64.00

F1 - Processing parameters
SI 1024
MC2 QP
SF 150.9255486 MHz
WDW SINC
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PL0 2.734 ppm
F2LO 1641.05 Hz
F2PHI 2.377 ppm
F2RI 1426.97 Hz
F1PL0 195.571 ppm
F1LO 29516.59 Hz
F1PHI 106.053 ppm
F1RI 16006.03 Hz
F2PMCH 0.02378 ppm/cm
F2NCH 14.27131 Hz/cm
F1PMCH 5.96787 ppm/cm
F1NCH 950.79361 Hz/cm



Current Data Parameters
 NAME: wt12601
 EXPNO: 21
 PROJNO: 1
 DU: mps
 USER: wxt

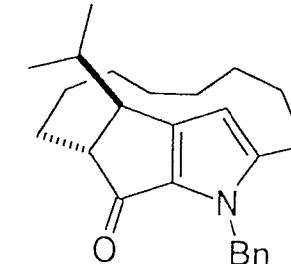
F1 - Acquisition Parameters
 Date: 961022
 Time: 21:01
 INSTRUM: spect
 PROBHD: 5 mm Multinucl
 PULPROG: noesypr1d
 TD: 1024
 SOLVENT: CDCl3
 NS: 5
 DS: 16
 SWH: 4664.179 Hz
 FIDRES: 4.554862 Hz
 AQ: 9.109228 sec
 RG: 128
 DW: 167.200 usec
 DE: 4.50 usec
 TE: 303.0 K
 H1I: 90 dB
 D1: 3.96293692 sec
 P1: 8.80 usec
 DO: 0.0000300 sec
 DR: 1.0000000 sec
 DE: 4.50 usec
 SF01: 600.2223708 MHz
 NUCLEUS: 1H
 INC: 0.00010720 sec

F1 - Acquisition parameters
 NDO: 2
 TD: 512
 SF01: 600.2224 MHz
 FIDRES: 9.109725 Hz
 SW: 7.771 ppm

F2 - Processing parameters
 SI: 1024
 SF: 600.2200738 MHz
 WDW: QSINE
 SSB: 2
 LB: 0.00 Hz
 GB: 0
 PC: 4.00

F1 - Processing parameters
 SI: 1024
 MC2: TPII
 SF: 600.2200738 MHz
 WDW: QSINE
 SSB: 2
 LB: 0.00 Hz
 GB: 0

2D NMR plot parameters
 CX2: 15.00 cm
 CX1: 15.00 cm
 F2PLO: 7.712 ppm
 F2LO: 4629.04 Hz
 F2PHI: -0.059 ppm
 F2HI: -35.14 Hz
 F1PLO: 7.712 ppm
 F1LO: 4629.04 Hz
 F1PHI: -0.059 ppm
 F1HI: -35.14 Hz
 F2PPCM: 0.51805 ppm/cm
 F2HZCM: 310.94528 Hz/cm
 F1PPCM: 0.51805 ppm/cm
 F1HZCM: 310.94528 Hz/cm



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NOESY

2
4
6
ppm