Supporting Information

Synthesis of Enaminones by Rhodium-Catalyzed Denitrogenative Rearrangement of 1-(*N*-Sulfonyl-1,2,3-triazol-4-yl)alkanols

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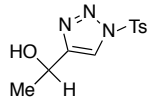
General Methods.

Rhodium(II)-catalyzed reactions were carried out with a Biotage Initiator 2.5 microwave synthesizer. IR measurements were performed on a FTIR SHIMADZU DR-8000 spectrometer fitted with a Pike Technologies MIRacle Single Reflection ATR adapter. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury-vx400 (^{1}H at 400.44 MHz and ^{13}C at 100.69 MHz) spectrometer. NMR data were obtained in $CDCl_3$. Proton chemical shifts were referenced to the residual proton signal of the solvent at 7.26 ppm ($CHCl_3$). Carbon chemical shifts were referenced to the carbon signal of the solvent at 77.0 ppm ($CDCl_3$). High-resolution mass spectra were recorded on a Thermo Scientific Exactive (ESI and APCI) spectrometer. Flash column chromatography was performed with silica gel 60N (Kanto) and diol-silica gel DIOL MB 100–40/75 (Fuji Silysia Chemical Ltd.). Preparative thin-layer chromatography (PTLC) was performed on silica gel plates with PF254 inidicator (Merck). Recycling preparative HPLC was carried out on COSMOSIL 5SL-II (Nacalai) with a Japan Analytical Industry LC-9110 NEXT. Gel permeation chromatography (GPC) was carried out with a Japan Analytical Industry LC-908.

Materials.

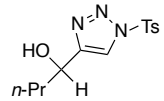
Chloroform (Wako, dehydrated, amylene as stabilizer) was distilled from phosphorus oxide (Wako). Toluene (Nacalai) was used as received from the commercial sources. $Rh_2(Oct)_4$ (Aldrich), $Cu(OAc)_2 \cdot H_2O$ (Wako), and *o*-aminophenol (nacalai) were used as received from the commercial sources. 3-Butyn-2-ol (**5a**, Aldrich), 1-ethynyl-1-cyclohexanol (**5c**, TCI), mestranol (**5k**, TCI) were used as received from the commercial sources. 1-(*N*-Sulfonyl-1,2,3-triazol-4-yl)alkanols **1a**–**h** and 1-(*N*-sulfonyl-1,2,3-triazol-4-yl)cycloalkanols **3a**–**j** were prepared from the corresponding propargylic alcohols according to the literature procedures.[1,2] The analytical data of compounds **1h**,[2] **2b**,[3] **2c**,[3] **2f**,[4] **3b**,[1] and **3c**[1] have already reported.

1a:



IR (ATR): 3315, 2978, 1595, 1394, 1192, 1178, 1113, 1009 cm^{-1}; ^{1}H NMR: δ = 1.57 (d, *J* = 6.8 Hz, 3H), 2.12–2.36 (br, 1H), 2.44 (s, 3H), 5.06 (q, *J* = 6.4 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.4 Hz, 2H), 8.05 (s, 1H); ^{13}C NMR: δ = 21.7, 22.8, 62.5, 120.3, 128.5, 130.4, 132.7, 147.3, 152.1; HRMS (ESI^+): Calcd for $C_{11}H_{14}N_3O_3S$, $M+H^+$ 268.0750. Found m/z 268.0743.

1b:



IR (ATR): 3342, 3267, 3153, 2955, 2870, 1595, 1387, 1171, 1018, 980 cm^{-1}; ^{1}H NMR: δ = 0.94 (t, *J* = 7.6 Hz, 3H), 1.31–1.55 (m, 2H), 1.74–1.91 (m, 2H), 2.36–2.68 (br, 1H), 2.45 (s, 3H), 4.89 (dd, *J* = 7.2, 5.6 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.4 Hz, 2H), 8.04 (s, 1H); ^{13}C NMR: δ = 13.6, 18.3, 21.7, 39.0, 66.3, 120.5, 128.5, 130.3, 132.8, 147.3, 151.4; HRMS (ESI^+): Calcd for $C_{13}H_{18}N_3O_3S$, $M+H^+$ 296.1063. Found m/z 296.1055.

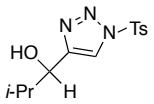
1 Raushel, J.; Fokin, V. V. *Org. Lett.* **2010***, 12,* 4952.

2 Liu, Y.; Wang, X.; Xu, J.; Zhang, Q.; Zhao, Y.; Hu, Y. *Tetrahedron* **2011**, *67*, 6294.

3 Liu, P.; Shan, G.; Chen, S.; Rao, Y. *Tetrahedron Lett.* **2012**, *53*, 936.

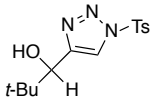
4 Xiao, F.; Wang, J. *J. Org. Chem.* **2006**, *71*, 5789

1c:



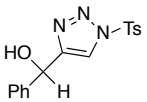
IR (ATR): 3298, 3101, 2968, 1593, 1393, 1379, 1194, 1177, 1024, 988 cm^{-1}; ^1H NMR: δ = 0.81–0.98 (m, 6H), 2.06–2.18 (m, 1H), 2.45 (s, 3H), 2.66–3.46 (br, 1H), 4.67 (d, *J* = 5.6 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.0 Hz, 2H), 8.04 (s, 1H); ^{13}C NMR: δ = 16.9, 18.2, 21.6, 33.7, 71.6, 121.1, 128.4, 130.3, 132.7, 147.2, 150.2; HRMS (ESI^+): Calcd for $C_{13}H_{18}N_3O_3S$, $M+H^+$ 296.1063. Found m/z 296.1055.

1d:



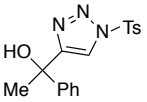
IR (ATR): 3263, 3103, 2968, 1389, 1196, 1177, 1057, 1024, 1016, 982 cm^{-1}; ^1H NMR: δ = 0.92 (s, 9H), 2.02–2.48 (br, 1H), 2.45 (s, 3H), 4.57 (s, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.4 Hz, 2H), 8.02 (s, 1H); ^{13}C NMR: δ = 21.7, 25.3, 35.3, 75.0, 121.3, 128.5 130.4, 132.9, 147.3, 149.0; HRMS (ESI^+): Calcd for $C_{14}H_{20}N_3O_3S$, $M+H^+$ 310.1220. Found m/z 310.1211.

1e:



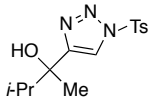
IR (ATR): 3336, 3155, 1593, 1456, 1387, 1217, 1194, 1177, 1043, 1011, 966 cm^{-1}; ^1H NMR: δ = 2.44 (s, 3H), 2.44–3.16 (br, 1H), 5.98 (s, 1H), 7.29–7.43 (m, 7H), 7.87 (s, 1H), 7.96 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR: δ = 21.7, 68.6, 121.2, 126.3, 128.2, 128.6, 130.3, 132.6, 140.8, 147.3, 150.9; HRMS (ESI^+): Calcd for $C_{16}H_{16}N_3O_3S$, $M+H^+$ 330.0907. Found m/z 330.0897.

1f:



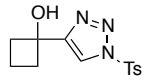
IR (ATR): 3422, 3162, 1391, 1196, 1177, 1138, 1113, 1005, 986 cm^{-1}; ^1H NMR: δ = 1.96 (s, 3H), 2.45 (s, 3H), 2.75–3.05 (br, 1H), 7.24–7.30 (m, 1H), 7.30–7.37 (m, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.43–7.48 (m, 2H), 7.92 (s, 1H), 7.98 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR: δ = 21.7, 30.3, 71.9, 120.4, 125.0, 127.4, 128.2, 128.6, 130.4, 132.7, 145.3, 147.3, 154.4; HRMS (ESI^+): Calcd for $C_{17}H_{18}N_3O_3S$, $M+H^+$ 344.1063. Found m/z 344.1053.

1g:



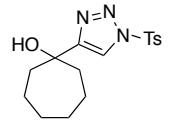
IR (ATR): 3422, 3123, 2966, 1593, 1385, 1192, 1178, 1092, 999 cm^{-1}; ^1H NMR: δ =0.81-0.89 (m, 6H), 1.53 (s, 3H), 2.08-2.34 (br, 1H), 2.10 (sept, *J* = 6.8 Hz, 1H), 2.45 (s, 3H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.991 (d, *J* = 7.6 Hz, 2H), 7.995 (s, 1H); ^{13}C NMR: δ = 16.8, 17.0, 21.7, 24.8, 37.8, 73.4, 120.3, 128.5, 130.3, 132.9, 147.2, 154.0; HRMS (ESI^+): Calcd for $C_{14}H_{20}N_3O_3S$, $M+H^+$ 310.1220. Found m/z 310.1211.

3a:



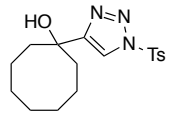
IR (ATR): 3287, 3113, 1593, 1396, 1196, 1177, 1015 cm^{-1}; ^{1}H NMR: δ = 1.74–1.91 (m, 1H), 1.87–2.02 (m, 1H), 2.30–2.44 (m, 2H), 2.44 (s, 3H), 2.47–2.59 (m, 2H), 2.80–2.98 (br, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 2H), 8.07 (s, 1H); ^{13}C NMR: δ = 12.5, 21.8, 37.1, 71.8, 119.5, 128.7, 130.4, 132.9, 147.3, 153.2; HRMS (ESI^+): Calcd for $C_{13}H_{16}N_3O_3S$, $M+H^+$ 294.0907. Found m/z 294.0902.

3d:



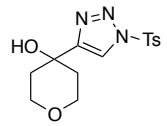
IR (ATR): 3385, 3148, 2920, 1385, 1192, 1177 cm^{-1}; ^{1}H NMR: δ = 1.48–1.77 (m, 8H), 1.96 (dd, *J* = 14.4, 8.4 Hz, 2H), 2.10 (dd, *J* = 14.8, 10.0 Hz, 2H), 2.28–2.44 (br, 1H), 2.44 (s, 3H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.4 Hz, 2H), 8.00 (s, 1H); ^{13}C NMR: δ = 21.77, 21.81, 29.2, 41.8, 73.4, 119.1, 128.7, 130.4, 133.0, 147.2, 156.3; HRMS (ESI^+): Calcd for $C_{16}H_{22}N_3O_3S$, $M+H^+$ 336.1376. Found m/z 336.1371.

3e:



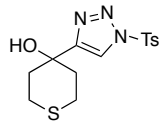
IR (ATR): 3487, 3130, 2895, 2843, 1593, 1389, 1194, 1178, 1013, 999 cm^{-1}; ^{1}H NMR: δ = 1.42–1.62 (m, 5H), 1.56–1.76 (m, 5H), 2.03–2.11 (m, 4H), 2.23–2.27 (br, 1H), 2.45 (s, 3H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.99 (d, *J* = 8.0 Hz, 2H), 8.00 (s. 1H); ^{13}C NMR: δ = 21.6, 21.8, 24.5, 28.0, 36.5, 73.1, 119.6, 128.7, 130.4, 133.0, 147.3, 155.0; HRMS (ESI^+): Calcd for $C_{17}H_{24}N_3O_3S$, $M+H^+$ 350.1533. Found m/z 350.1526.

3f:



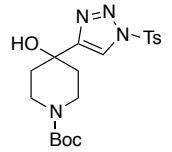
IR (ATR): 3402, 3125, 2860, 1595, 1389, 1196, 1180, 1020 cm^{-1}; ^{1}H NMR: δ = 1.83 (d, *J* = 13.2 Hz, 2H), 2.17 (td, *J* = 12.4, 4.8 Hz, 2H), 2.46 (s, 3H), 3.79 (d, *J* = 11.6 Hz, 2H), 3.89 (t, *J* = 10.8 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 8.00 (d, *J* = 8.4 Hz, 2H), 8.03 (s, 1H); ^{13}C NMR: δ = 21.8, 37.8, 63.3, 66.9, 119.5, 128.7, 130.5, 132.8, 147.5, 154.1; HRMS (ESI^+): Calcd for $C_{14}H_{18}N_3O_4S$, $M+H^+$ 324.1013. Found m/z 324.1007.

3g:



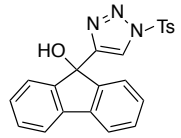
IR (ATR): 3400, 3153, 2980, 1591, 1391, 1194, 1184, 1020 cm^{-1}; ^{1}H NMR: δ = 2.13–2.25 (m, 4H), 2.34–2.48 (br, 1H), 2.46 (s, 3H), 2.44–2.53 (m, 2H), 3.05–3.15 (m, 2H), 7.40 (d, *J* = 8.8 Hz, 2H), 7.995 (d, *J* = 8.0 Hz, 2H), 8.003 (s, 1H); ^{13}C NMR: δ = 21.8, 23.7, 38.5, 68.1, 119.3, 128.8, 130.5, 132.8, 147.5, 154.6; HRMS (ESI^+): Calcd for $C_{14}H_{18}N_3O_3S_2$, $M+H^+$ 340.0784. Found m/z 340.0780.

3h:



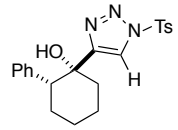
IR (ATR): 3427, 3161, 1666, 1591, 1427, 1387, 1173, 1146, 1076, 989 cm^{-1}; ^1H NMR: δ = 1.46 (s, 9H), 1.85 (d, *J* = 12.4 Hz, 2H), 2.01 (td, *J* = 12.0, 4.8 Hz, 2H), 2.46 (s, 3H), 3.29 (t, *J* = 10.8 Hz, 2H), 3.87 (br, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 8.00 (d, *J* = 8.0 Hz, 2H), 8.01 (s, 1H); ^{13}C NMR: (–60 °C) δ = 22.0, 28.2, 36.1, 36.3, 38.1, 39.1, 67.1, 79.8, 119.5, 128.6, 130.5, 131.6, 147.8, 154.2, 154.4; HRMS (ESI^+): Calcd for $C_{19}H_{27}N_4O_5S$, $M+H^+$ 423.1697. Found m/z 423.1688.

3i:



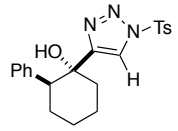
IR (ATR): 3256, 3169, 2980, 1591, 1452, 1389, 1192, 1178, 989 cm^{-1}; ^1H NMR: δ = 2.45 (s, 3H), 2.99–3.10 (br, 1H), 7.32 (td, *J* = 7.2, 0.8 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.41 (td, *J* = 7.6, 0.8 Hz, 2H), 7.61 (d, *J* = 7.6 Hz, 2H), 7.66 (d, *J* = 7.6 Hz, 2H), 7.84 (s, 1H), 7.96 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR: δ = 21.8, 78.3, 120.3, 120.5, 124.8, 128.5, 128.8, 129.8, 130.4, 132.7, 139.5, 146.8, 147.4, 149.9; HRMS (ESI^+): Calcd for $C_{22}H_{18}N_3O_3S$, $M+H^+$ 404.1063. Found m/z 404.1059.

cis-**3j:**



IR (ATR): 3362, 1389, 1192, 1177, 1005 cm^{-1}; ^1H NMR: δ = 1.48–1.62 (m, 1H), 1.68–1.97 (m, 5H), 2.07–2.32 (m, 3H), 2.47 (s, 3H), 3.15 (dd, *J* = 13.2, 3.6 Hz, 1H), 6.83 (d, *J* = 7.2 Hz, 2H), 6.96 (t, *J* = 7.6 Hz, 2H), 7.06 (tt, *J* = 7.6, 1.6 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.48 (s, 1H), 7.80 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR: δ = 21.0, 21.7, 25.8, 27.2, 38.4, 51.8, 72.5, 120.6, 126.4, 127.7, 128.3, 128.6, 130.2, 133.2, 140.9, 146.8, 155.0; HRMS (ESI^+): Calcd for $C_{21}H_{24}N_3O_3S$, $M+H^+$ 398.1533. Found m/z 398.1521.

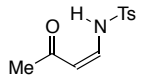
trans-**3j:**



IR (ATR): 3362, 1387, 1194, 1177, 1053, 1005 cm^{-1}; ^1H NMR: δ = 1.43–1.58 (m, 1H), 1.72–1.85 (m, 2H), 1.86 (dd, *J* = 13.2, 4.4 Hz, 1H), 1.94–2.04 (m, 1H), 2.11–2.54 (m, 4H), 2.48 (s, 3H), 2.89 (dd, *J* = 13.2, 3.6 Hz, 1H), 6.77 (d, *J* = 6.8 Hz, 2H), 7.02 (t, *J* = 7.6 Hz, 2H), 7.12 (t, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.57 (s. 1H), 7.89 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR: δ = 21.7, 22.7, 26.1, 28.3, 40.3, 55.2, 73.3, 122.1, 126.7, 127.7, 128.4, 128.9, 130.3, 133.2, 140.2, 147.0, 152.5; HRMS (ESI^+): Calcd for $C_{21}H_{24}N_3O_3S$, $M+H^+$ 398.1533. Found m/z 398.1521.

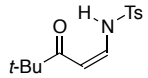
Typical Procedure for the Denitrogenative Rearrangement Reaction of 1-(*N*-Tosyl-1,2,3-triazol-4-yl)-alkanols (Table 1, entry 1). A 2-5 mL Biotage® microwave vial was charged with $Rh_2(Oct)_4$ (0.8 mg, 1 μmol), freshly prepared **1a** (53.5 mg, 0.20 mmol), and $CHCl_3$ (4 mL). The vial was capped with a Teflon pressure cap. The reaction mixture was heated at 140 °C for 15 min under microwave irradiation. After the reaction mixture was cooled, the solvent was removed under reduced pressure. The residue was purified by recycling preparative HPLC (CH_2Cl_2) to give the product **2a** (42.8 mg, 0.18 mmol, 89%).

2a:



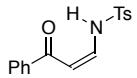
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3109, 1680, 1657, 1574, 1354, 1167, 1151, 1090, 966 cm^{-1}; ^1H NMR: δ = 2.13 (s, 3H), 2.42 (s, 3H), 5.45 (d, *J* = 8.4 Hz, 1H), 6.95 (t, *J* = 8.8 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 11.45 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR: δ = 21.5, 30.1, 103.2, 126.6, 130.0, 136.8, 139.7, 144.5, 200.5; HRMS (ESI^+): Calcd for $C_{11}H_{14}NO_3S$, $M+H^+$ 240.0689. Found m/z 240.0683.

2d:



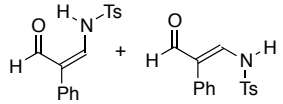
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3117, 2968, 1674, 1578, 1560, 1356, 1242, 1231, 1163, 1080, 924 cm^{-1}; ^1H NMR: δ = 1.11 (s, 9H), 2.42 (s, 3H), 5.65 (d, *J* = 8.4 Hz, 1H), 7.05 (dd, *J* = 10.4, 8.4 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.74 (d, *J* = 8.4 Hz, 2H), 11.57 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR: δ = 21.6, 26.6, 42.9, 98.7, 126.8, 130.0, 137.1, 140.7, 144.4, 208.5; HRMS (ESI^+): Calcd for $C_{14}H_{20}NO_3S$, $M+H^+$ 282.1158. Found m/z 282.1150.

2e:



Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate = 100:1); IR (ATR): 3115, 1638, 1558, 1456, 1354, 1232, 1159, 1015 cm^{-1}; ^1H NMR: δ = 2.42 (s, 3H), 6.19 (d, *J* = 8.8 Hz, 1H), 7.26 (t, *J* = 9.4 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.41–7.48 (m, 2H), 7.50–7.57 (m, 1H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.87 (d, *J* = 7.2 Hz, 2H), 11.94 (d, *J* = 10.0 Hz, 1H); ^{13}C NMR: δ = 21.5, 99.2, 126.7, 127.7, 128.6, 130.0, 132.8, 136.9, 137.5, 141.9, 144.5, 192.0; HRMS (ESI^+): Calcd for $C_{16}H_{16}NO_3S$, $M+H^+$ 302.0845. Found m/z 302.0837.

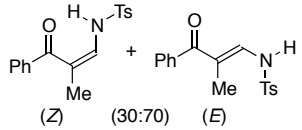
2e':



(*Z*) (70:30) (*E*)

Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate=100:1); IR (ATR): 3236, 1684, 1628, 1595, 1541, 1331, 1248, 1159, 1084 cm^{-1}; ^1H NMR: (*Z*) δ = 2.44 (s, 3H), 7.26-7.46 (m, 8H), 7.78 (d, *J* = 8.0 Hz, 2H), 9.68 (d, *J* = 3.6 Hz, 1H), 11.63 (d, *J* = 10.8 Hz, 1H); (*E*) δ = 2.47 (s, 3H), 7.11 (d, *J* = 8.0, 2H), 7.26-7.46 (m, 7H), 7.77 (d, *J* = 8.4 Hz, 2H), 9.42 (s, 1H); ^{13}C NMR: (*Z* and *E*) δ = 21.61, 21.65, 117.2, 124.5, 126.8, 126.9, 126.9, 128.7, 129.0, 129.1, 129.3, 129.7, 130.2, 130.3, 135.1, 136.0, 136.6, 139.0, 143.4, 144.9, 145.2, 189.3, 193.6; HRMS (ESI^+): Calcd for $C_{16}H_{16}NO_3S$, $M+H^+$ 302.0845. Found m/z 302.0837.

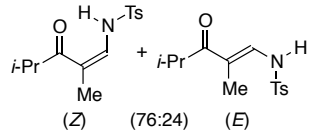
2f':



(*Z*) (30:70) (*E*)

It is difficult to get a large amount of **2f'** due to the minor products. Therefore, only ^{1}H NMR was shown here. Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate=100:1); ^{1}H NMR: (*Z*) δ = 1.93 (d, *J* = 1.2 Hz, 3H); 2.44 (s, 3H), 7.04 (dq, *J* = 10.8, 1.2 Hz, 1H), 7.30-7.55 (m, 7H), 7.79 (d, *J* = 8.8 Hz, 2H), 11.44 (d, *J* = 10.4 Hz, 1H); (*E*) δ = 1.83 (d, *J* = 1.2 Hz, 3H); 2.46 (s, 3H), 6.83 (d, *J* = 12.0 Hz, 1H), 7.10 (dq, *J* = 12.0, 1.2 Hz, 1H), 7.30-7.55 (m, 7H), 7.69 (d, *J* = 8.4 Hz, 2H).

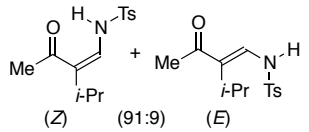
2g:



(*Z*) (76:24) (*E*)

Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate = 100:1); IR (ATR): 3354, 3260, 3192, 2970, 2932, 2872, 1715, 1607, 1597, 1342, 1157, 1088, 1047 cm^{-1}; ^{1}H NMR: (*Z*) δ = 1.02 (d, *J* = 6.8 Hz, 6H), 1.92 (d, *J* = 1.2 Hz, 3H), 2.40 (s, 3H), 2.86 (septet, *J* = 6.8 Hz, 1H), 6.82 (dq, *J* = 10.4, 1.2 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 11.56 (d, *J* = 10.4 Hz, 1H); (*E*) δ = 1.06 (d, *J* = 6.8 Hz, 6H), 1.64 (d, *J* = 1.2 Hz, 3H), 2.42 (s, 3H), 3.14 (septet, *J* = 6.8 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.41 (dd, *J* = 12.0, 1.2 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 2H), (N–H missing); ^{13}C NMR: (*Z*) δ = 16.9, 18.3, 21.5, 36.5, 109.2, 126.6, 129.9, 137.3, 137.5, 144.1, 208.4; (*E*) δ = 9.5, 19.7, 21.6, 33.7, 116.5, 130.1, 133.6, 136.8, 144.7, 203.2; HRMS (ESI$^+$): Calcd for $C_{14}H_{20}NO_3S$, M+H$^+$ 282.1158. Found m/z 282.1150.

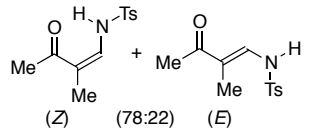
2g':



(*Z*) (91:9) (*E*)

Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate = 100:1); IR (ATR): 3204, 3051, 2963, 1651, 1574, 1433, 1360, 1263, 1155, 1090 cm^{-1}; ^{1}H NMR: (*Z*) δ = 1.10 (d, *J* = 6.4 Hz, 6H), 2.22 (s, 3H), 2.41 (s, 3H), 2.74 (quint, *J* = 6.8 Hz, 1H), 6.84 (d, *J* = 10.8 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 11.64 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR: (*Z*) δ = 21.5, 23.3, 27.9, 28.3, 121.7, 126.5, 129.9, 135.3, 137.4, 144.1, 202.4; HRMS (ESI$^+$): Calcd for $C_{14}H_{20}NO_3S$, M+H$^+$ 282.1158. Found m/z 282.1150.

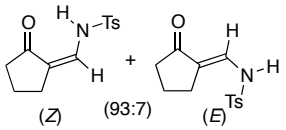
2h:



(*Z*) (78:22) (*E*)

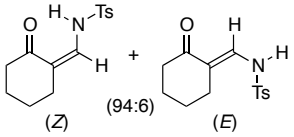
Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate = 100:1); IR (ATR): 3269, 2930, 1732, 1639, 1593, 1408, 1337, 1269, 1157, 1086 cm^{-1}; ^{1}H NMR: (*Z*) δ = 1.89 (d, *J* = 1.2 Hz, 3H), 2.16 (s, 3H), 2.42 (s, 3H), 6.78 (dq, *J* = 10.4, 1.2 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 11.41 (d, *J* = 10.4 Hz, 1H); (*E*) δ = 1.64 (d, *J* = 1.2 Hz, 3H), 2.26 (s, 3H), 2.44 (s, 3H), 6.96 (d, *J* = 12.0 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.35–7.40 (m, 1H), 7.77 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR: (*Z*) δ = 17.5, 21.5, 28.8, 110.3, 126.6, 129.9, 136.5, 137.4, 144.2, 202.4; (*E*) δ = 9.2, 21.6, 25.0, 118.3, 126.7, 130.2, 134.6, 136.7, 144.8, 196.5; HRMS (ESI$^+$): Calcd for $C_{12}H_{16}NO_3S$, M+H$^+$ 254.0845. Found m/z 254.0841.

4a:



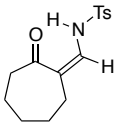
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3113, 1709, 1597, 1350, 1339, 1207, 1157, 1088, 1007 cm^{-1}; ^1H NMR: (*Z*) δ = 1.91 (quint, *J* = 7.6 Hz, 2H), 2.31 (t, *J* = 7.6 Hz, 2H), 2.41 (s, 3H), 2.55 (td, *J* = 7.2, 2.0 Hz, 2H), 6.79 (s, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 10.71 (s, 1H); ^{13}C NMR: (*Z*) δ = 21.2, 21.5, 27.2, 39.3, 114.5, 126.7, 130.0, 131.7, 137.2, 144.3, 209.5; HRMS (ESI^+): Calcd for $C_{13}H_{16}NO_3S$, $M+H^+$ 266.0845. Found m/z 266.0841.

4b:



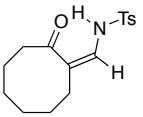
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3138, 2947, 1670, 1553, 1335, 1219, 1157, 1084 cm^{-1}; ^1H NMR: (*Z*) δ = 1.62–1.71 (m, 2H), 1.70–1.80 (m, 2H), 2.34 (t, *J* = 6.8 Hz, 2H), 2.38 (td, *J* = 6.8, 1.2 Hz, 2H), 2.41 (s, 3H), 6.81 (dt, *J* = 10.0, 1.2 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 11.58 (d, *J* = 10.0 Hz, 1H); ^{13}C NMR: (*Z*) δ = 21.5, 22.1, 23.2, 28.3, 38.6, 111.8, 126.6, 129.9, 137.2, 137.4, 144.2, 202.4; HRMS (ESI^+): Calcd for $C_{14}H_{18}NO_3S$, $M+H^+$ 280.1002. Found m/z 280.1002.

4c:



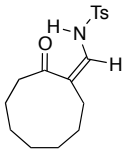
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3274, 3115, 2922, 1651, 1566, 1346, 1258, 1167, 1142, 1092 cm^{-1}; ^1H NMR: δ = 1.56–1.74 (m, 6H), 2.27–2.33 (m, 2H), 2.41 (s, 3H), 2.48–2.54 (m, 2H), 6.84 (d, *J* = 10.4 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 11.46 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR: δ = 21.5, 24.8, 30.4, 31.2, 32.4, 44.6, 116.6, 126.6, 129.9, 136.6, 137.3, 144.1, 206.6; HRMS (ESI^+): Calcd for $C_{15}H_{20}NO_3S$, $M+H^+$ 294.1158. Found m/z 294.1153.

4d:



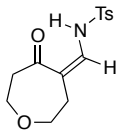
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3175, 3112, 2924, 1645, 1564, 1354, 1261, 1163, 1086 cm^{-1}; ^1H NMR: δ = 1.37–1.60 (m, 6H), 1.65–1.74 (m, 2H), 2.38 (t, *J* = 6.0 Hz, 2H), 2.41 (s, 3H), 2.52 (t, *J* = 6.4 Hz, 2H), 6.83 (d, *J* = 10.0 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 11.68 (d, *J* = 10.0 Hz, 1H); ^{13}C NMR: δ = 21.6, 25.6, 26.0, 28.9, 29.9, 32.7, 39.6, 115.8, 126.6, 129.9, 137.2, 137.3, 144.1, 207.4; HRMS (ESI^+): Calcd for $C_{16}H_{22}NO_3S$, $M+H^+$ 308.1315. Found m/z 308.1313.

4e:



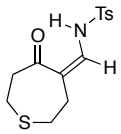
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3210, 2924, 1643, 1558, 1350, 1256, 1157, 1088 cm^{-1}; ^1H NMR: δ = 1.37–1.56 (m, 6H), 1.51–1.68 (m, 2H), 1.64–1.82 (m, 2H), 2.32–2.45 (m, 2H), 2.40 (s, 3H), 2.51 (t, *J* = 6.4 Hz, 2H), 6.84 (d, *J* = 10.4 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 11.72 (d, *J* = 10.0 Hz, 1H); ^{13}C NMR: δ = 21.5, 24.3, 24.6, 26.1, 27.6, 29.9, 31.0, 39.2, 117.2, 126.6, 129.9, 137.3, 138.2, 144.1, 207.7; HRMS (ESI^+): Calcd for $C_{17}H_{24}NO_3S$, $M+H^+$ 322.1471. Found m/z 322.1465.

4f:



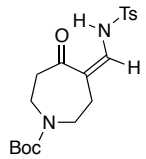
Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate =100:1); IR (ATR): 3308, 1682, 1651, 1595, 1566, 1346, 1263, 1159, 1146 cm^{-1}; ^1H NMR: δ = 2.41 (s, 3H), 2.46–2.51 (m, 2H), 2.71–2.75 (m, 2H), 3.68–3.76 (m, 4H), 6.89 (d, *J* = 10.4 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 11.42 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR: δ = 21.5, 35.0, 48.3, 66.0, 72.3, 114.5, 126.6, 130.0, 137.1, 137.8, 144.4, 204.4; HRMS (ESI^+): Calcd for $C_{14}H_{18}NO_4S$, $M+H^+$ 296.0951. Found m/z 296.0940.

4g:



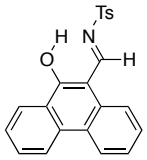
Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate=100:1); IR (ATR): 3179, 3028, 2897, 1647, 1560, 1354, 1263, 1155, 1146, 1082 cm^{-1}; ^1H NMR: δ = 2.42 (s, 3H), 2.64–2.80 (m, 6H), 2.92–2.95 (m, 2H), 6.92 (d, *J* = 10.4 Hz, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 11.48 (d, *J* = 10.8 Hz, 1H); ^{13}C NMR: δ = 21.5, 25.6, 32.4, 35.2, 47.5, 114.5, 126.6, 130.0, 137.0, 138.5, 144.4, 204.1; HRMS (ESI^+): Calcd for $C_{14}H_{18}NO_3S_2$, $M+H^+$ 312.0723. Found m/z 312.0717.

4h:



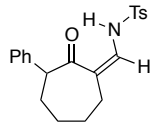
Purified by recycling preparative HPLC (CH_2Cl_2/ethyl acetate=100:1); IR (ATR): 3179, 2974, 2930, 1688, 1651, 1574, 1418, 1362, 1244, 1161, 1088 cm^{-1}; ^1H NMR: δ = 1.44 (s, 9H), 2.37–2.46 (m, 2H), 2.42 (s, 3H), 2.60–2.66 (m, 2H), 3.46–3.54 (m, 4H), 6.91 (d, *J* = 10.4 Hz, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 2H), 11.47 (d, *J* = 10.0 Hz, 1H); ^{13}C NMR: δ = 21.5, 28.3, 33.0–33.8 (br), 41.7–42.6 (br), 46.1, 47.6–48.9 (br), 80.2, 114.1, 126.6, 129.9, 137.0, 138.5, 144.4, 154.6, 204.2; HRMS (ESI^+): Calcd for $C_{19}H_{27}N_2O_5S$, $M+H^+$ 395.1635. Found m/z 395.1630.

4i:



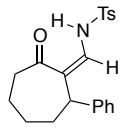
Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3065, 1589, 1541, 1487, 1321, 1294, 1153, 1088 cm^{-1}; ^1H NMR: δ = 2.44 (s, 3H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.55 (t, *J* = 6.8 Hz, 1H), 7.64 (t, *J* = 6.8 Hz, 2H), 7.81 (t, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 8.4 Hz, 2H), 8.19 (d, *J* = 8.4 Hz, 1H), 8.50 (d, *J* = 8.0 Hz, 1H), 8.55 (dd, *J* = 8.4, 5.6 Hz, 2H), 9.93 (s, 1H), (O–H missing); ^{13}C NMR: δ = 21.7, 105.2, 120.3, 122.8, 123.5, 124.8, 125.4, 125.7, 125.8, 127.3, 127.7, 128.4, 130.0, 132.0, 135.1, 135.9, 144.7, 165.6, 166.0; HRMS (ESI^+): Calcd for $C_{22}H_{18}NO_3S$, $M+H^+$ 376.1002. Found m/z 376.0995.

4j:



Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3192, 2924, 2853, 1651, 1574, 1352, 1250, 1167, 1072, 1055 cm^{-1}; ^1H NMR: δ = 1.32–1.46 (m, 1H), 1.66–1.80 (m, 1H), 1.90–2.13 (m, 4H), 2.35 (dd, *J* = 15.2, 6.0 Hz, 1H), 2.42 (s, 3H), 2.55–2.66 (m, 1H), 3.89 (d, *J* = 10.4 Hz, 1H), 6.95 (d, *J* = 10.4 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.24–7.37 (m, 5H), 7.70 (d, *J* = 8.4 Hz, 2H), 11.40 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR: δ = 21.5, 30.1, 30.6, 32.1, 33.0, 57.5, 115.7, 126.7, 126.8, 128.1, 128.4, 129.8, 137.2, 137.4, 140.8, 144.1, 204.7; HRMS (ESI^+): Calcd for $C_{21}H_{24}NO_3S$, $M+H^+$ 370.1471. Found m/z 370.1461.

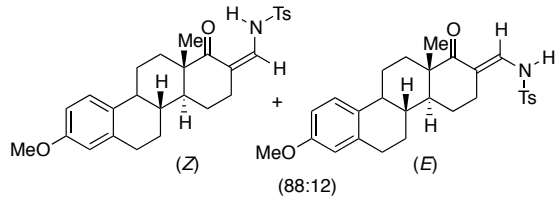
4j':



Purified by preparative thin-layer chromatography ($CHCl_3$/ethyl acetate = 25:1) and recycling preparative HPLC (Hexane/CH_2Cl_2/ethyl acetate=70:15:15); IR (ATR): 3179, 2926, 2856, 1645, 1568, 1360, 1259, 1167, 1150, 1086 cm^{-1}; ^1H NMR: δ = 1.51–1.65 (m, 1H), 1.63–1.82 (m, 2H), 1.83–1.96 (m, 1H), 2.06–2.23 (m, 2H), 2.36–2.48 (m, 1H), 2.40 (s, 3H), 2.58–2.69 (m, 1H), 3.72–3.80 (m, 1H), 6.39 (d, *J* = 10.8 Hz, 1H), 7.12 (d, *J* = 7.2 Hz, 2H), 7.23–7.30 (m, 3H), 7.35 (t, *J* = 7.2 Hz, 2H), 7.55 (d, *J* = 8.4 Hz, 2H), 11.57 (d, *J* = 10.8 Hz, 1H); ^{13}C NMR: δ = 21.6, 24.7, 28.5, 35.3, 44.1, 45.9, 119.9, 126.6, 126.7, 127.8, 128.8, 129.8, 137.2, 139.0, 142.9, 144.1, 206.3; HRMS (ESI^+): Calcd for $C_{21}H_{24}NO_3S$, $M+H^+$ 370.1471. Found m/z 370.1462.

Typical Procedure for the One-pot Synthesis of Enaminones from Propargylic Alcohols (equation 3).
A 2-5 mL Biotage® microwave vial was charged with 2-aminophenol (1.23 g, 11.3 μmol), $Cu(OAc)_2 \cdot H_2O$ (3.9 mg, 19.5 μmol), tosyl azide (38.4 mg, 0.19 mmol), but-3-yn-2-ol (**5a**, 14.4 mg. 0.21 mmol), and $CHCl_3$ (1 mL). The vial was capped with a Teflon pressure cap. The reaction mixture was stirred at room temperature for 24 h. To the resulting green solution were added $Rh_2(Oct)_4$ (1.57 mg, 2 μmol) and $CHCl_3$ (3 mL). Then, the reaction mixture was heated at 140 °C for 15 min under microwave irradiation. After being cooled to room temperature, the resulting mixture was passed through a pad of diol silica and eluted with ethyl acetate (50 mL). The filtrate was concentrated under reduced pressure. The residue was purified by recycling preparative HPLC (CH_2Cl_2) to give the product **2a** (33.8 mg, 0.14 mmol, 69%).

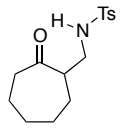
4k:



Purified by recycling preparative HPLC (CH_2Cl_2); IR (ATR): 3244, 2926, 1651, 1574, 1499, 1352, 1254, 1161, 1088 cm^{-1}; ^1H NMR: (*Z*) δ = 0.98 (s, 3H), 1.22–1.56 (m, 6H), 1.98–2.14 (m, 2H), 2.14–2.26 (m, 2H), 2.32–2.48 (m, 2H), 2.42 (s, 3H), 2.52–2.60 (m, 1H), 2.81–2.89 (m, 2H), 3.77 (s, 3H), 6.63 (d, *J* = 2.4 Hz, 1H), 6.72 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.82 (d, *J* = 10.4 Hz, 1H), 7.21 (d, *J* = 8.8 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.74 (d, *J* = 8.4 Hz, 2H), 11.38 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR: (*Z*) δ =17.5, 21.2, 21.5, 25.8, 26.3, 26.5, 30.0, 33.1, 39.3, 42.8, 44.8, 46.1, 55.1, 109.7, 111.6, 113.4, 126.2, 126.6, 129.9, 132.2, 136.9, 137.3, 137.6, 144.1, 157.5, 207.8; HRMS (ESI^+): Calcd for $C_{28}H_{34}NO_4S$, $M+H^+$ 480.2203. Found m/z 480.2192.

Procedure for the Hydrogenation Reaction of Enaminone 4c Catalyzed by Pd/C (Scheme 2). A side-arm tube equipped with a stirrer bar was charged with enaminone **4c** (57.7 mg, 0.20 mmol) and Pd/C (6.9 mg, 12 wt%), and ethyl acetate (3 mL). The tube was connected to a hydrogen balloon and immersed in a dry ice/acetone bath. After ten vacuum/H_2-filling cycles, the cooling bath was removed. The reaction mixture was stirred for 24 h at 40 °C, and then, cooled to room temperature. The resulting mixture was passed through a pad of Celite and eluted with ethyl acetate. The filtrate was concentrated under reduced pressure. The residue was purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 25:1) to give the product **6** (50.3 mg, 0.17 mmol, 86%).

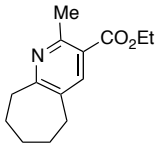
6:



IR (ATR): 3279, 2926, 1693, 1325, 1155, 1092 cm^{-1}; ^1H NMR: δ = 1.19–1.36 (m, 2H), 1.44–1.95 (m, 6H), 2.30–2.52 (m, 2H), 2.42 (s, 3H), 2.78–2.88 (m, 1H), 3.00 (t, *J* = 6.8 Hz, 2H), 5.06 (t, *J* = 6.8 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR: δ = 21.5, 23.3, 29.0, 29.1, 29.2, 43.5, 44.6, 51.3, 126.9, 129.7, 137.1, 143.3, 215.5; HRMS (ESI^+): Calcd for $C_{15}H_{22}NO_3S$, $M+H^+$ 296.1315. Found m/z 296.1308.

Procedure for the Reaction of Enaminone 4c with Ethyl Acetoacetate (Scheme 2). A side-arm tube equipped with a stirrer bar and reflux condenser was charged with enaminone **4c** (61.8 mg, 0.21 mmol) and ammonium acetate (23.0 mg, 0.30 mmol). The tube was evacuated and refilled with argon three times, and ethyl acetoacetate (34.0 mg, 0.26 mmol) and AcOH (2 mL) were added. After being heated at 140 °C for 12 h, the reaction mixture was cooled to room temperature and neutralized with 1 M NaOH aq. The aqueous layer was extracted with ethyl acetate (2 mL x 4). The combined organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by preparative thin-layer chromatography (hexane/ethyl acetate = 4:1) to give the product **7** (24.5 mg, 0.11 mmol, 50%).

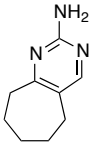
7:



IR (ATR): 2922, 1720, 1597, 1556, 1443 cm^{-1}; ^1H NMR: δ = 1.39 (t, *J* = 7.2 Hz, 3H), 1.54–1.76 (m, 4H), 1.84–1.92 (m, 2H), 2.74–2.82 (m, 2H), 2.77 (s, 3H), 3.01–3.08 (m, 2H), 4.36 (q, *J* = 7.2 Hz, 2H), 7.88 (s, 1H); ^{13}C NMR: δ = 14.3, 24.2, 26.3, 27.9, 32.4, 34.5, 39.4, 60.9, 122.8, 135.2, 138.6, 156.2, 166.0, 166.9; HRMS (ESI^+): Calcd for $C_{14}H_{20}NO_2$, $[M+H]^+$ 234.1489. Found m/z 234.1486.

Procedure for the Reaction of Enaminone 4c with Guanidine (Scheme 2). To a side-arm tube equipped with a stirrer bar and reflux condenser was charged with enaminone **4c** (76.2 mg, 0.26 mmol), guanidine hydrochloride (30.5 mg, 0.32 mmol) and NaOH (14.7 mg, 0.37 mmol). The tube was evacuated and refilled with argon three times, and *i*-PrOH (5 mL) was added. After being refluxed at 110 °C for 24 h, the reaction mixture was cooled to room temperature and concentrated under reduced pressure. The residue was purified by preparative thin-layer chromatography (hexane/ethyl acetate = 1:4) to give the product **8** (26.2 mg, 0.16 mmol, 62%).

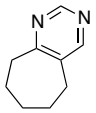
8:



IR (ATR): 3314, 3159, 2914, 1655, 1591, 1556, 1483, 1437 cm^{-1}; ^1H NMR: δ = 1.52–1.69 (m, 4H), 1.76–1.86 (m, 2H), 2.53–2.60 (m, 2H), 2.72–2.78 (m, 2H), 5.18 (br s, 2H), 7.92 (s, 1H); ^{13}C NMR: δ = 25.9, 28.3, 31.0, 32.3, 38.9, 124.9, 156.7, 161.5, 172.5; HRMS (ESI^+): Calcd for $C_9H_{14}N_3$, $[M+H]^+$ 164.1182. Found m/z 164.1183.

Procedure for the Reaction of Enaminone 4c with Formamidine (Scheme 2). To a side-arm tube equipped with a stirrer bar and reflux condenser was charged with enaminone **4c** (61.1 mg, 0.21 mmol) and formamidine hydrochloride (84.5 mg, 1.1 mmol). The tube was evacuated and refilled with argon three times, and pyridine (1 mL) was added. After being refluxed at 130 °C for 20 h, the reaction mixture was cooled to room temperature and concentrated under reduced pressure. The residue was purified by preparative thin-layer chromatography (1st.: hexane/ethyl acetate = 1:1, 2nd.: chloroform/ethyl acetate = 100:1) to give the product **9** (19.4 mg, 0.13 mmol, 63%).

9:[5]

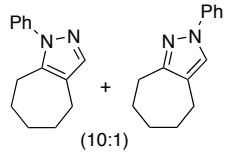


IR (ATR): 2922, 2853, 1572, 1551, 1456, 1447, 1396 cm^{-1}; ^1H NMR: δ = 1.62–1.75 (m, 4H), 1.84–1.95 (m, 2H), 2.71–2.79 (m, 2H), 2.94–3.02 (m, 2H), 8.37 (s, 1H), 8.89 (s, 1H); ^{13}C NMR: δ = 25.7, 27.4, 31.9, 32.3, 39.0, 135.5, 155.5, 156.3, 171.3; HRMS (ESI^+): Calcd for $C_9H_{13}N_2$, $[M+H]^+$ 149.1073 Found m/z 149.1074.

5 Boger, D. L.; Schumacher, J.; Mullican, M. D.; Patel, M.; Panek, J. S. *J. Org. Chem.* **1982**, *47*, 2673.

Procedure for the Reaction of Enaminone 4c with Phenylhydrazine (Scheme 2). To a side-arm tube equipped with a stirrer bar and reflux condenser was charged with enaminone **4c** (69.7 mg, 0.24 mmol). The tube was evacuated and refilled with argon three times, and phenyl hydrazine (28.9 mg, 0.27 mmol) and EtOH (4 mL) were added. After being refluxed at 100 °C for 12 h, the reaction mixture was cooled to room temperature and concentrated under reduced pressure. The residue was purified by preparative thin-layer chromatography (hexane/ethyl acetate = 5:1) to give the product **9** (42.9 mg, 0.20 mmol, 85% yield, 10:1 r.r.).

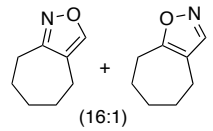
10:[6]



IR (ATR): 1501, 1398 cm^{-1}; ^1H NMR: δ = 1.60–1.76 (m, 4H), 1.81–1.90 (m, 2H), 2.60–2.67 (m, 2H), 2.74–2.82 (m, 2H), 7.33–7.42 (m, 4H), 7.42–7.49 (m, 2H); ^{13}C NMR: δ = 25.6, 27.09, 27.15, 28.5, 31.7, 121.9, 125.4, 127.4, 128.8, 139.6, 139.9, 142.1; HRMS (ESI^+): Calcd for $C_{14}H_{17}N_2$, $[M+H]^+$ 213.1386. Found m/z 213.1384.

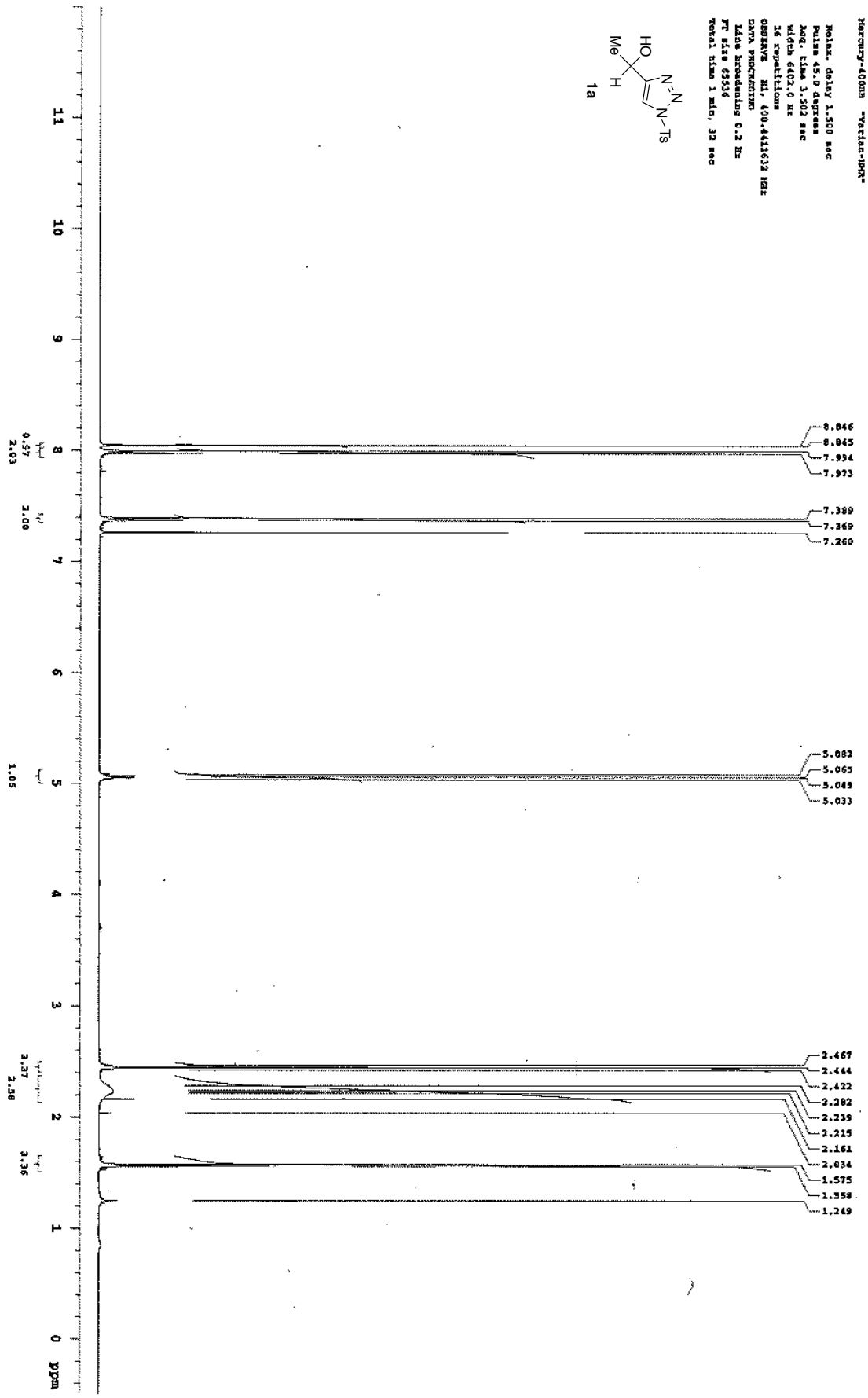
Procedure for the Reaction of Enaminone 4c with Hydroxylamine (Scheme 2). To a side-arm tube equipped with a stirrer bar was charged with enaminone **4c** (178.3 mg, 0.61 mmol) and hydroxylamine hydrochloride (218.9 mg, 3.2 mmol). The tube was evacuated and refilled with argon three times, and MeOH (3 mL) was added. After being heated at 70 °C for 4 h, the reaction mixture was cooled to room temperature and neutralized with $NaHCO_3$ aq. The aqueous layer was extracted with Et_2O (4 mL x 4). The combined organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by preparative thin-layer chromatography (hexane/ethyl acetate = 5:1) to give the product **10** (54.3 mg, 0.40 mmol, 66%, 16:1 r.r.).

11:[7]



IR (ATR): 2922, 2851, 1614, 1443, 1414 cm^{-1}; ^1H NMR: δ = 1.60–1.74 (m, 4H), 1.78–1.86 (m, 2H), 2.50–2.56 (m, 2H), 2.76–2.84 (m, 2H), 8.02 (s, 1H); ^{13}C NMR: δ = 23.2, 27.0, 27.2, 29.0, 31.9, 120.2, 153.6, 164.9; HRMS (APCI): Calcd for $C_8H_{12}NO$, $[M+H]^+$ 138.0913. Found m/z 138.0914.

6 Cho, C. S.; Patel, D. B. *Tetrahedron* **2006**, 62, 6388.
7 Ichino, T.; Arimoto, H.; Uemura, D. *Chem. Commun.* **2006**, 1742.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Pulse 45.0 degrees
Acq. time 3.502 sec
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16 repetitions
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DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
Me
H
N
N
Ts
1a
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8.045
7.994
7.973
7.389
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7.260
5.082
5.065
5.049
5.033
2.467
2.444
2.422
2.282
2.239
2.215
2.161
2.034
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1.558
1.249
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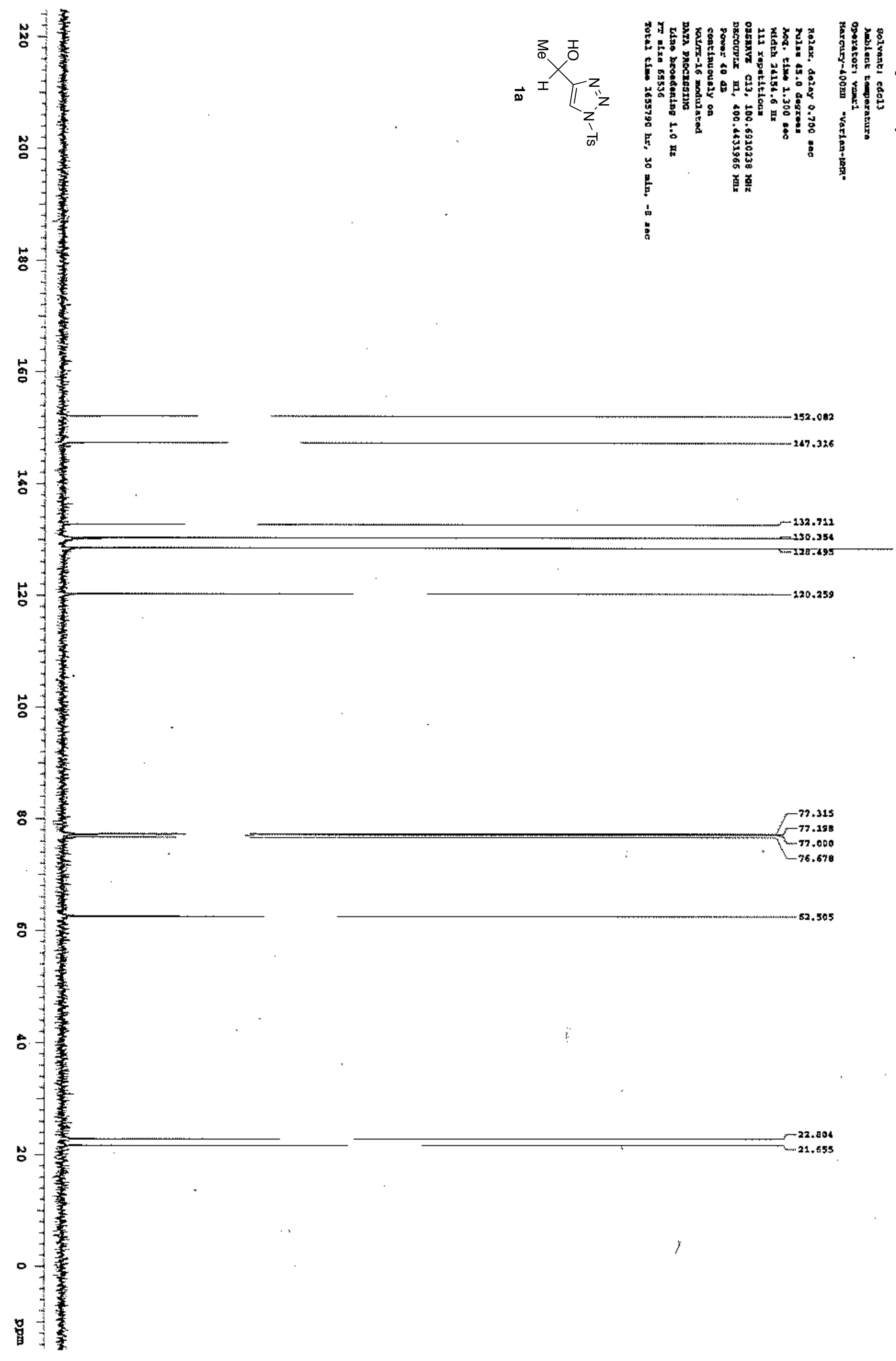
2.03
2.00
1.05
3.37
2.58
3.36
ppm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Ambient temperature
Operator: vnmr1
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Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
111 repetitions
OBSERVE C13, 100.6910238 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1655790 hr, 30 min, -8 sec
N
N
N
Ts
HO
Me
H
1a
152.082
147.316
132.711
130.354
128.495
120.259
77.315
77.198
77.000
76.678
62.505
22.804
21.655
220
200
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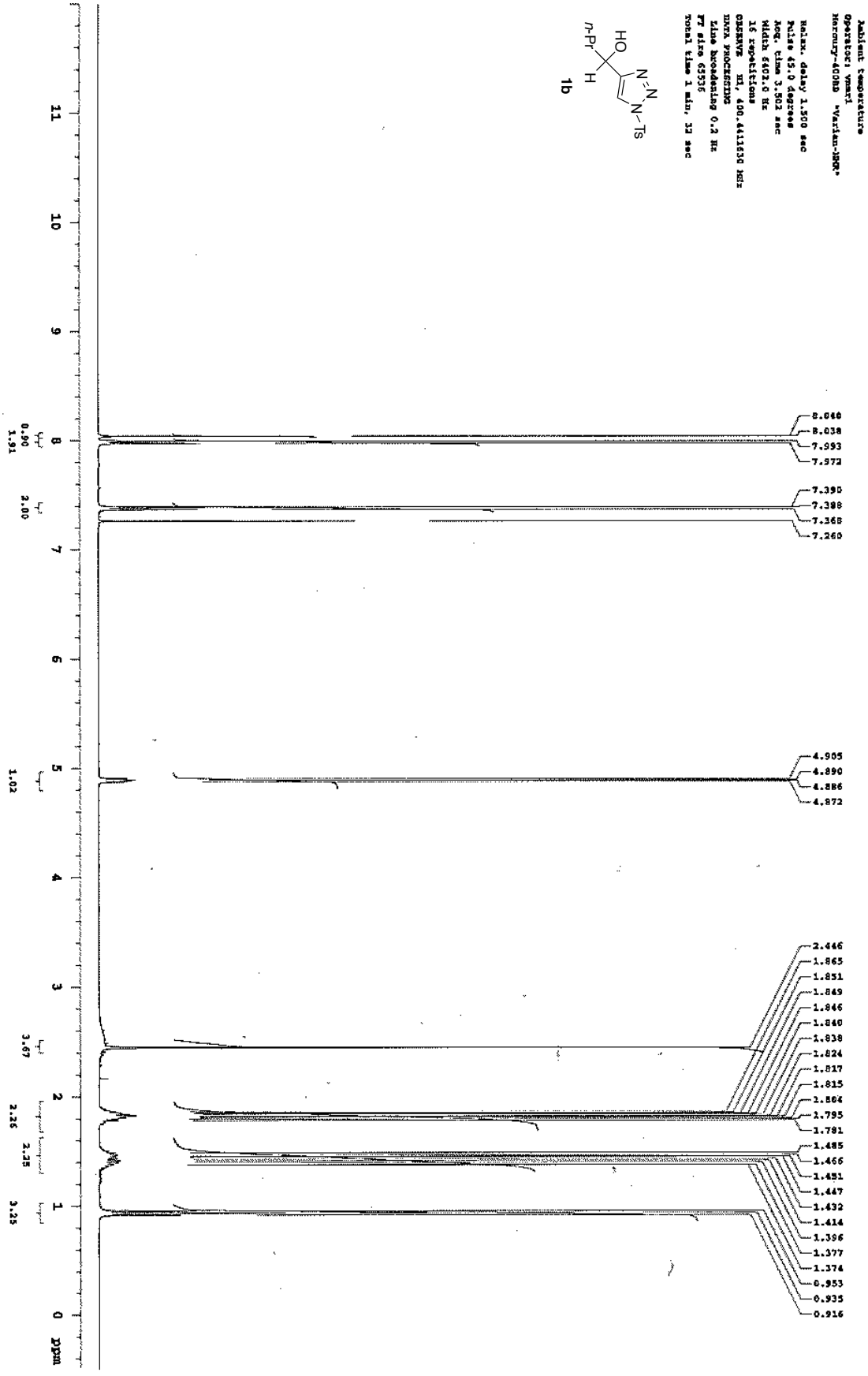
160
140
120
100
80
60
40
20
0
ppm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Ambient temperature
Operator: vmarl
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Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
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DATA PROCESSING
Line Broadening 0.2 Hz
FT size 65536
Total time 1 min, 33 sec
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n-Pr
H
N
N
N
Ts
1b
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8.038
7.993
7.972
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1.804
1.795
1.781
1.485
1.466
1.451
1.447
1.432
1.414
1.396
1.377
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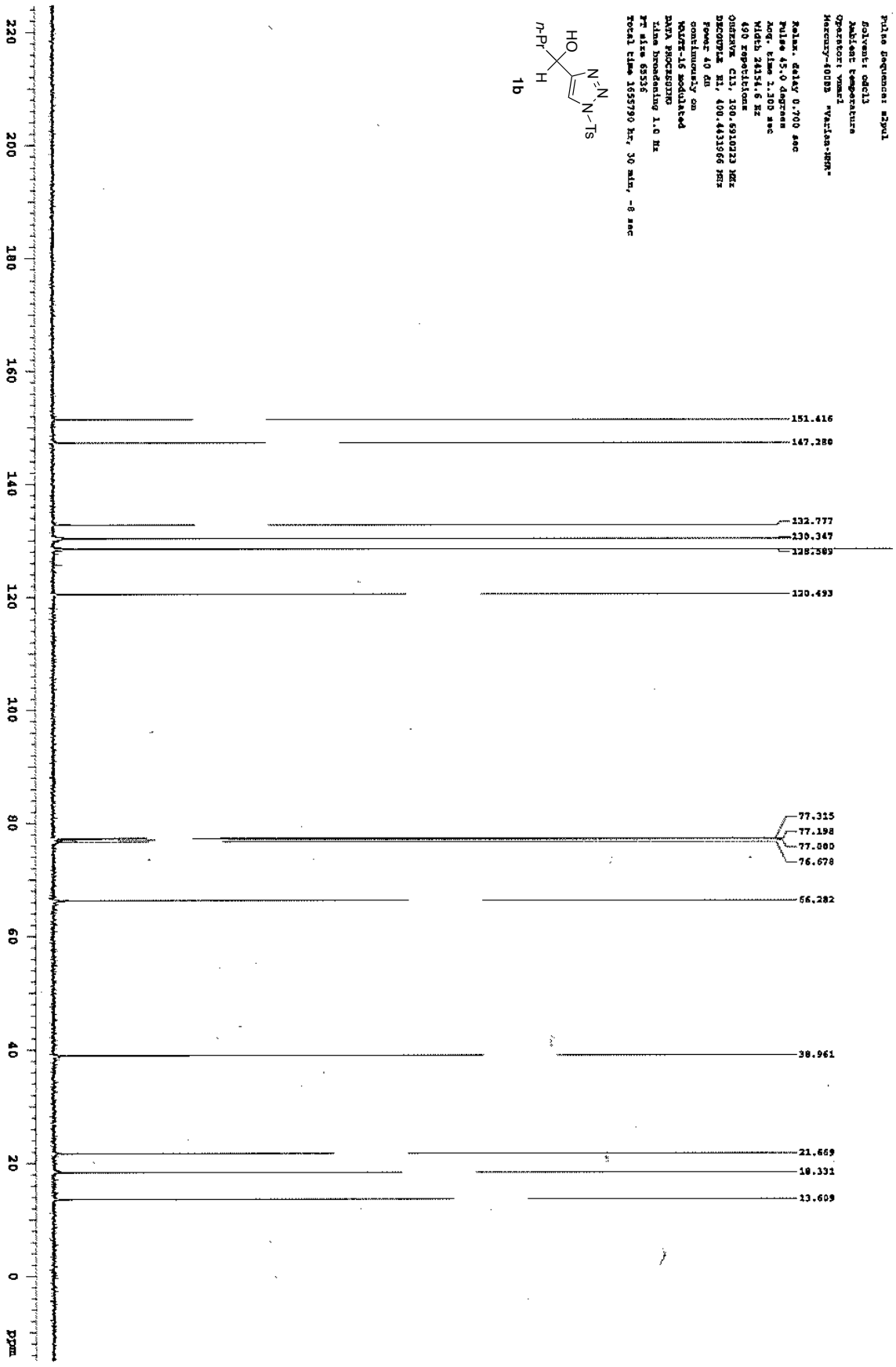
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Ambient temperature
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OBSERVE C13, 100.5910323 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 65536
Total time 165790 hr, 30 min, -8 sec
HO
n-Pr
H
N
N
N
Ts
1b
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147.280
132.777
130.347
128.569
120.493
77.315
77.198
77.080
76.678
56.282
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18.331
13.609
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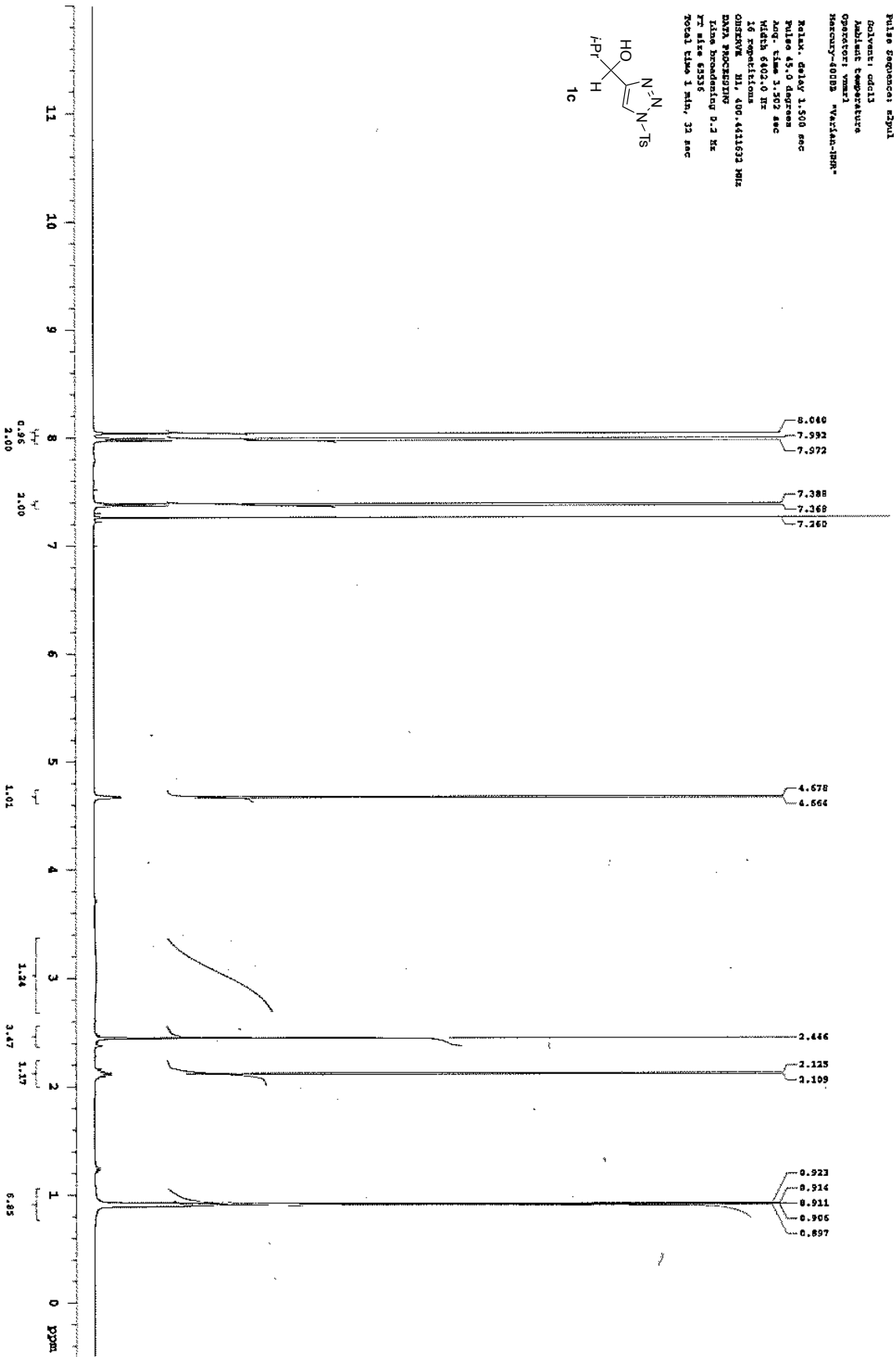
160
140
120
100
80
60
40
20
0
ppm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Ambient temperature
Operator: vnmrj
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Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min, 32 sec
HO
iPr
H
N
N
N
Ts
1C
8.040
7.992
7.972
7.388
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7.260
4.678
4.664
2.446
2.125
2.109
0.923
0.914
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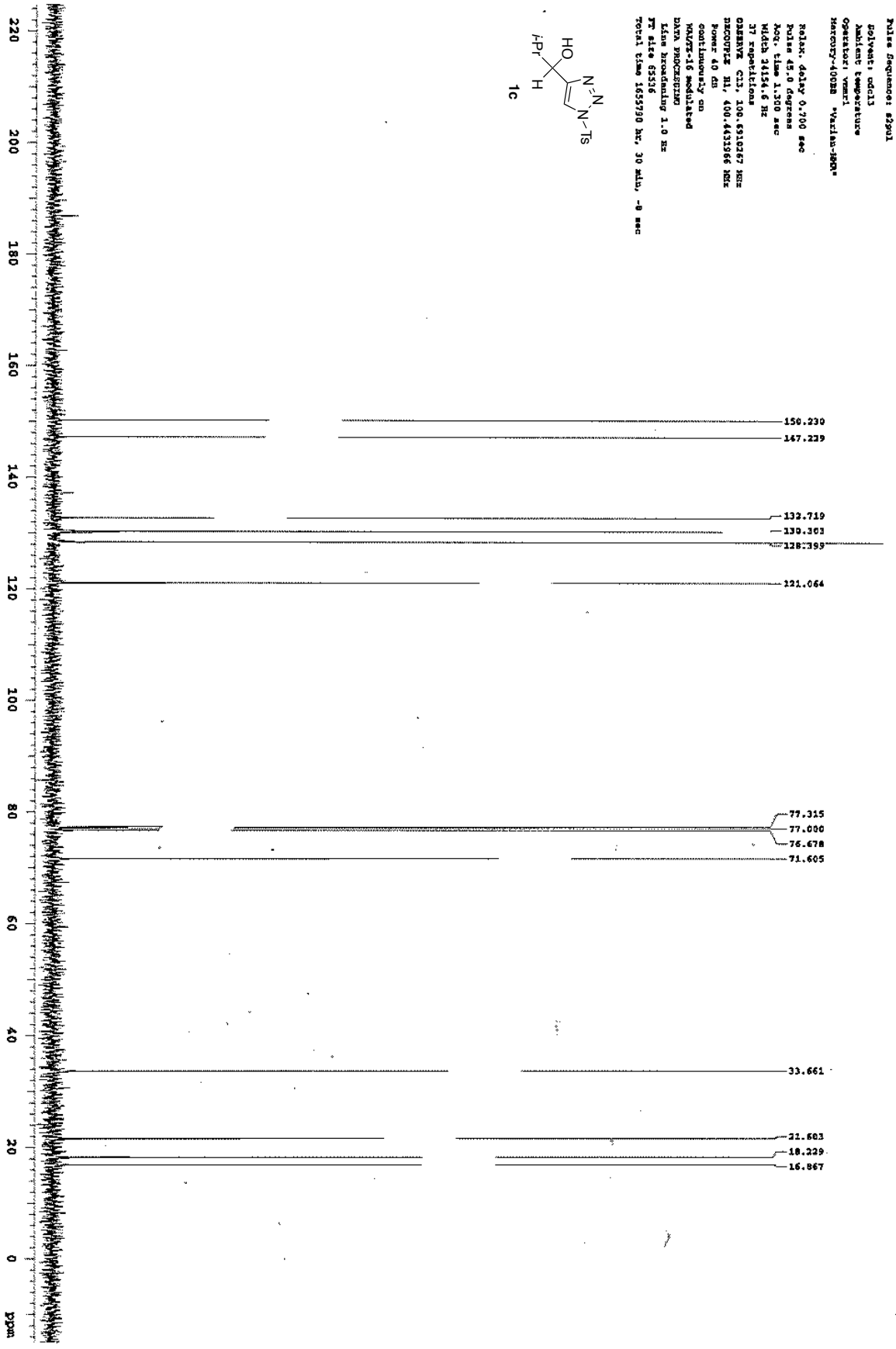
ppm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Solvent: cdcl3
Ambient temperature
Operator: vemri
Mercury-400BB "Varian-HOM"
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Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
37 repetitions
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DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1655790 sec, 30 min, -8 sec
HO
i-Pr
H
N
N
N
Ts
1c
150.230
147.239
132.719
130.303
128.399
121.064
77.315
77.000
76.678
71.605
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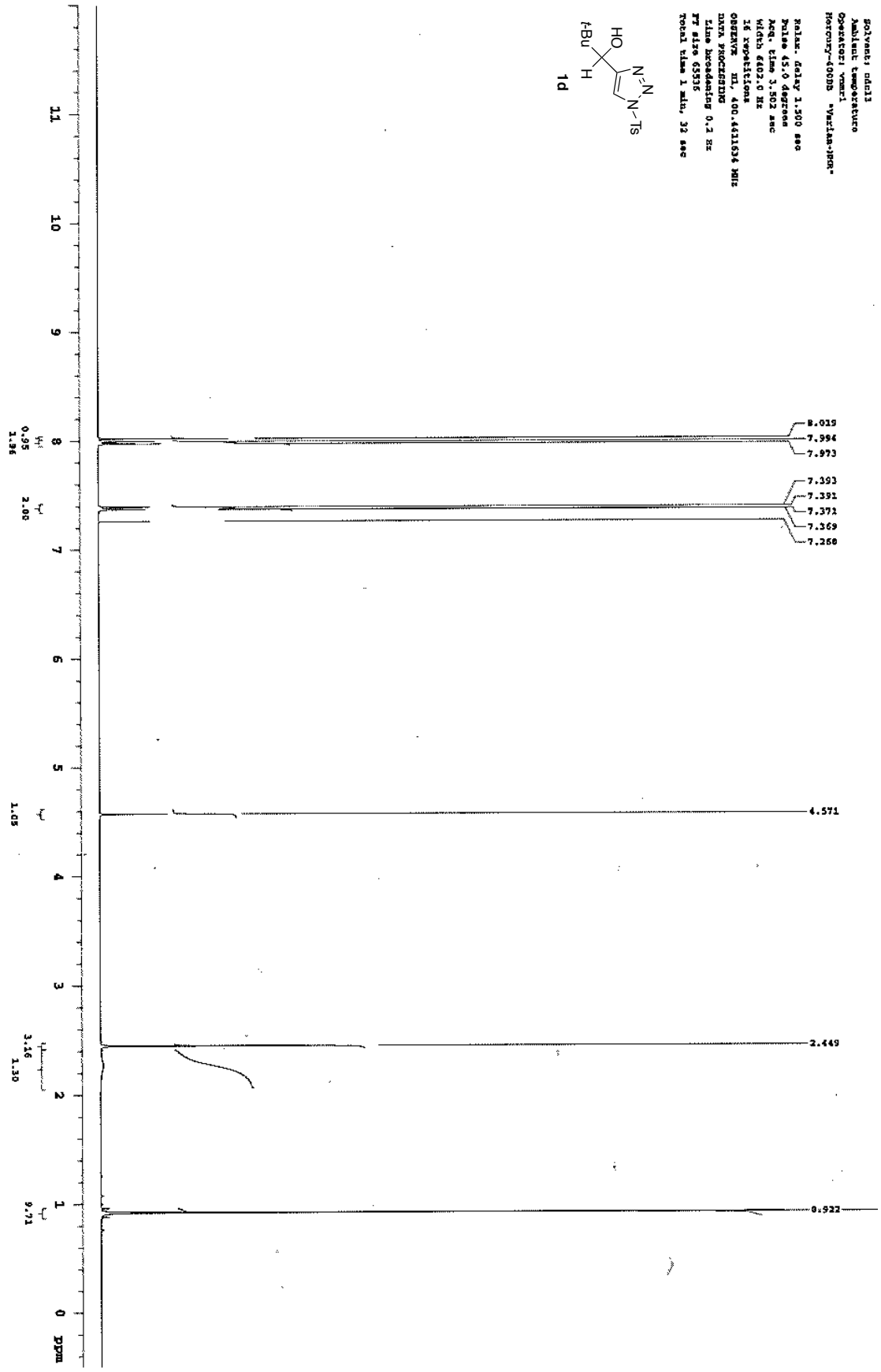
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Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-NMR"
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Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6602.0 Hz
16 repetitions
OBSERVE H1, 400.4411534 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
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tBu
H
N
N
N
Ts
1d
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7.994
7.973
7.393
7.391
7.371
7.369
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1.05
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1.30
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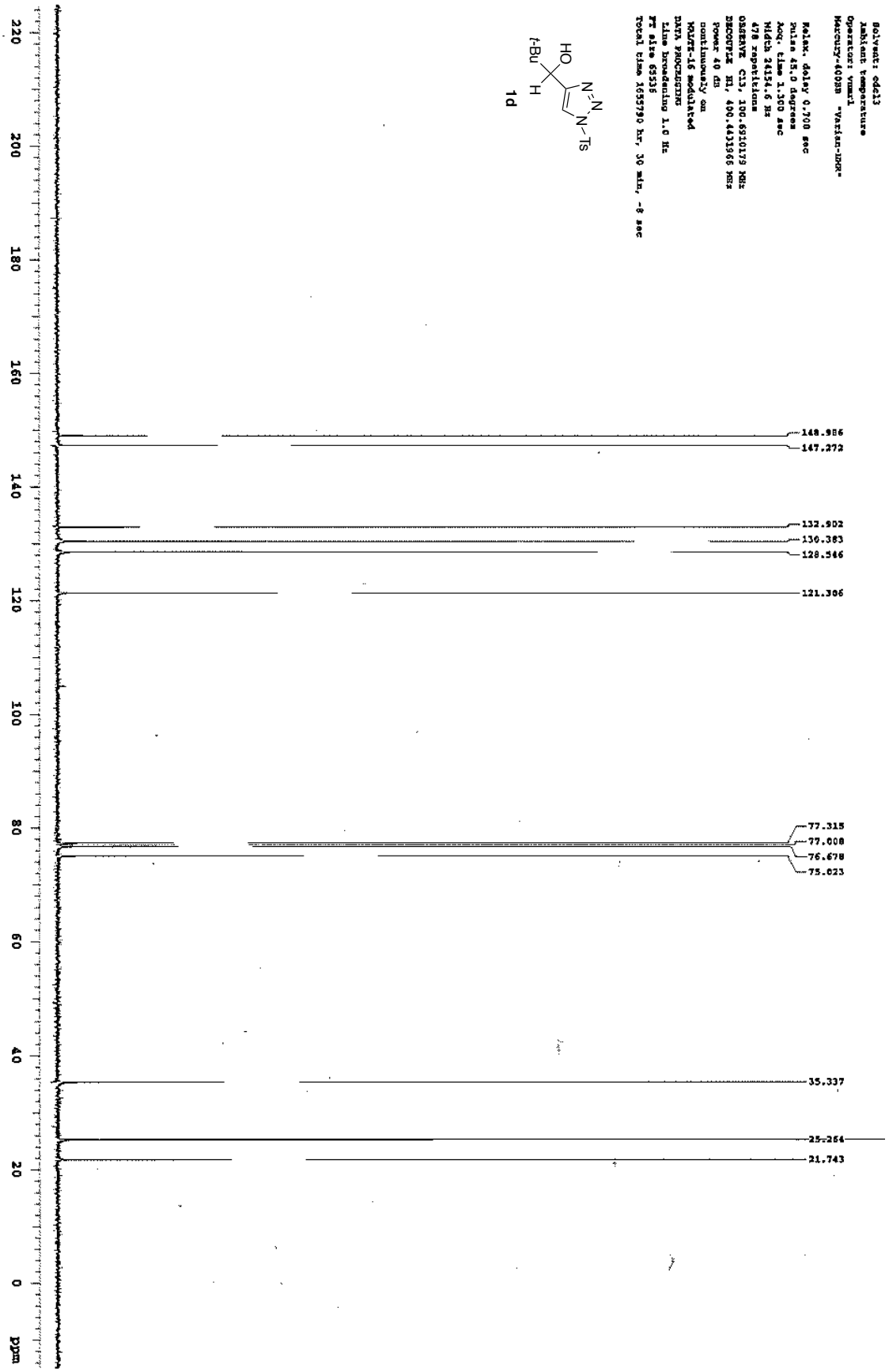
ppm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Ambient temperature
Operator: vnmrl
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478 repetitions
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DECOUPLE H1, 400.4413966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
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t-Bu
H
N
N
N
Ts
1d
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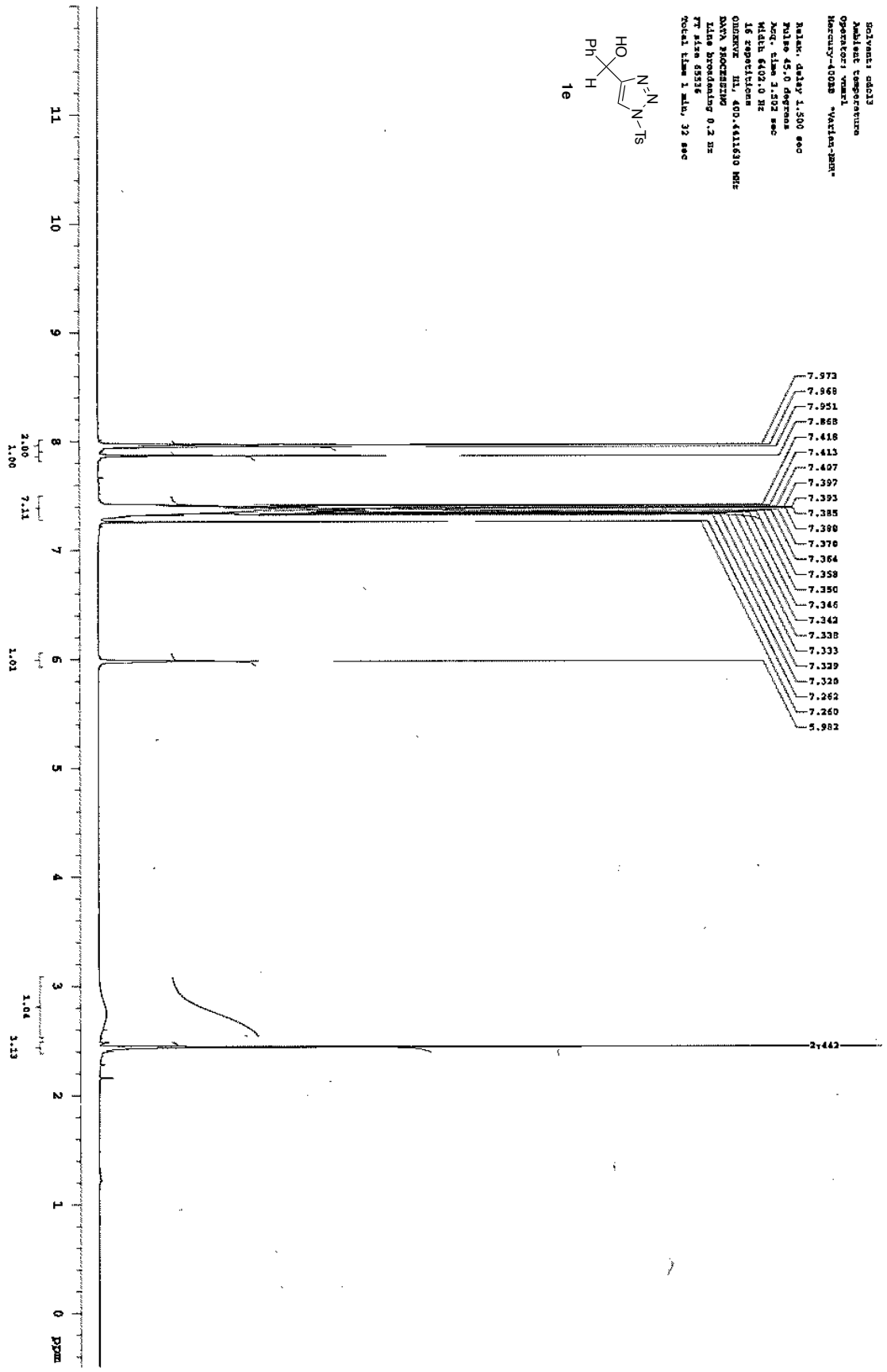
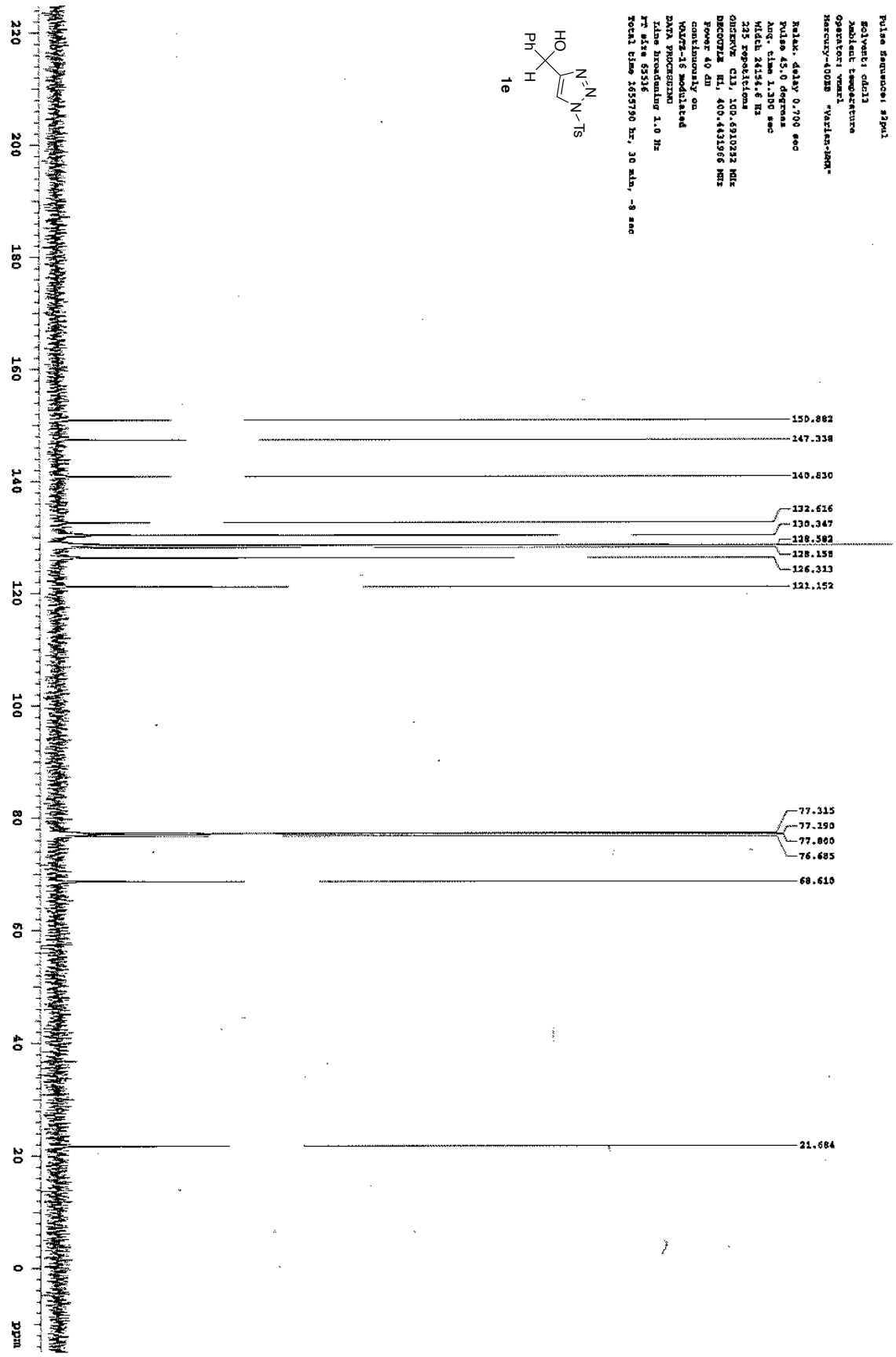
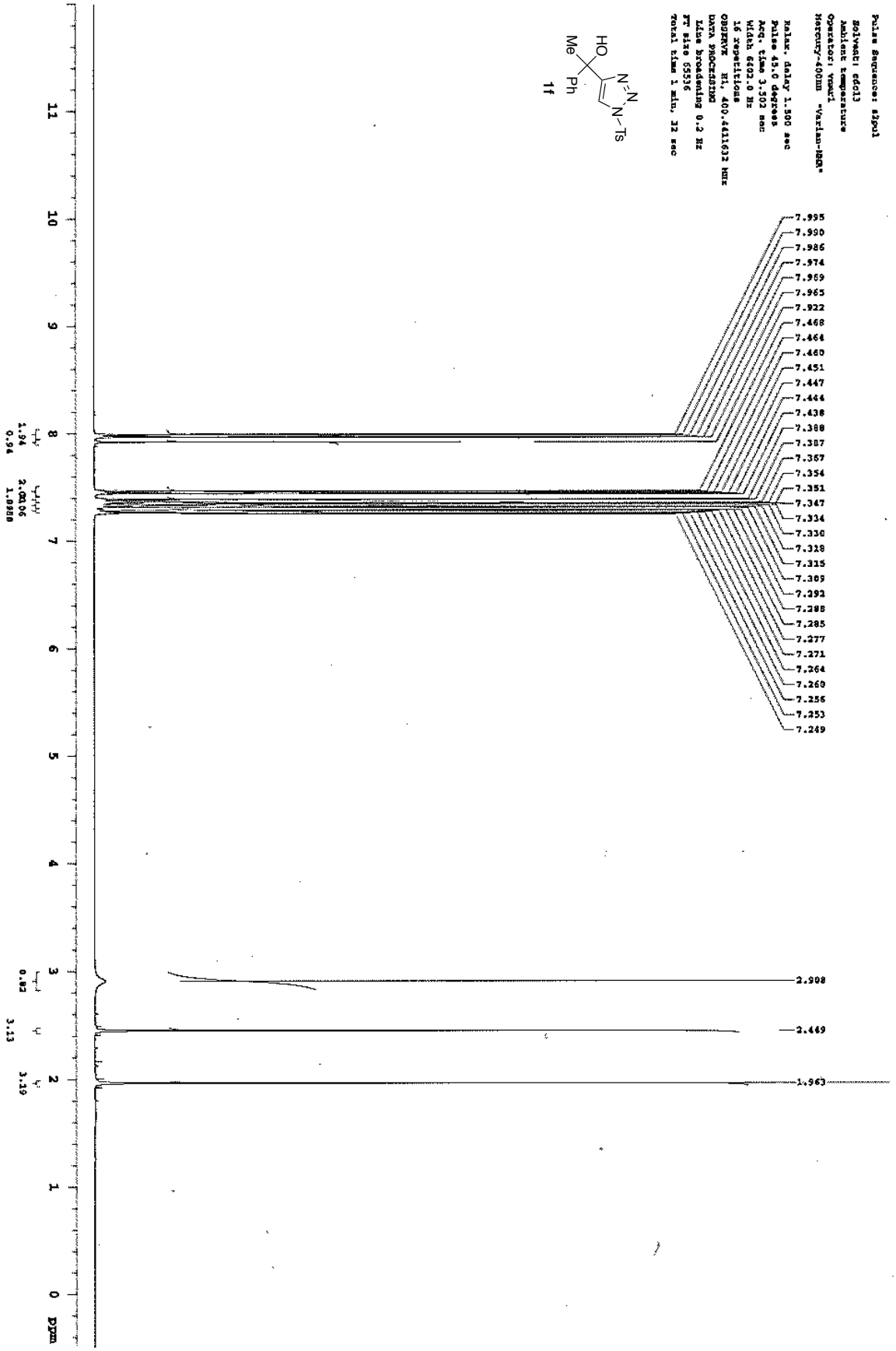
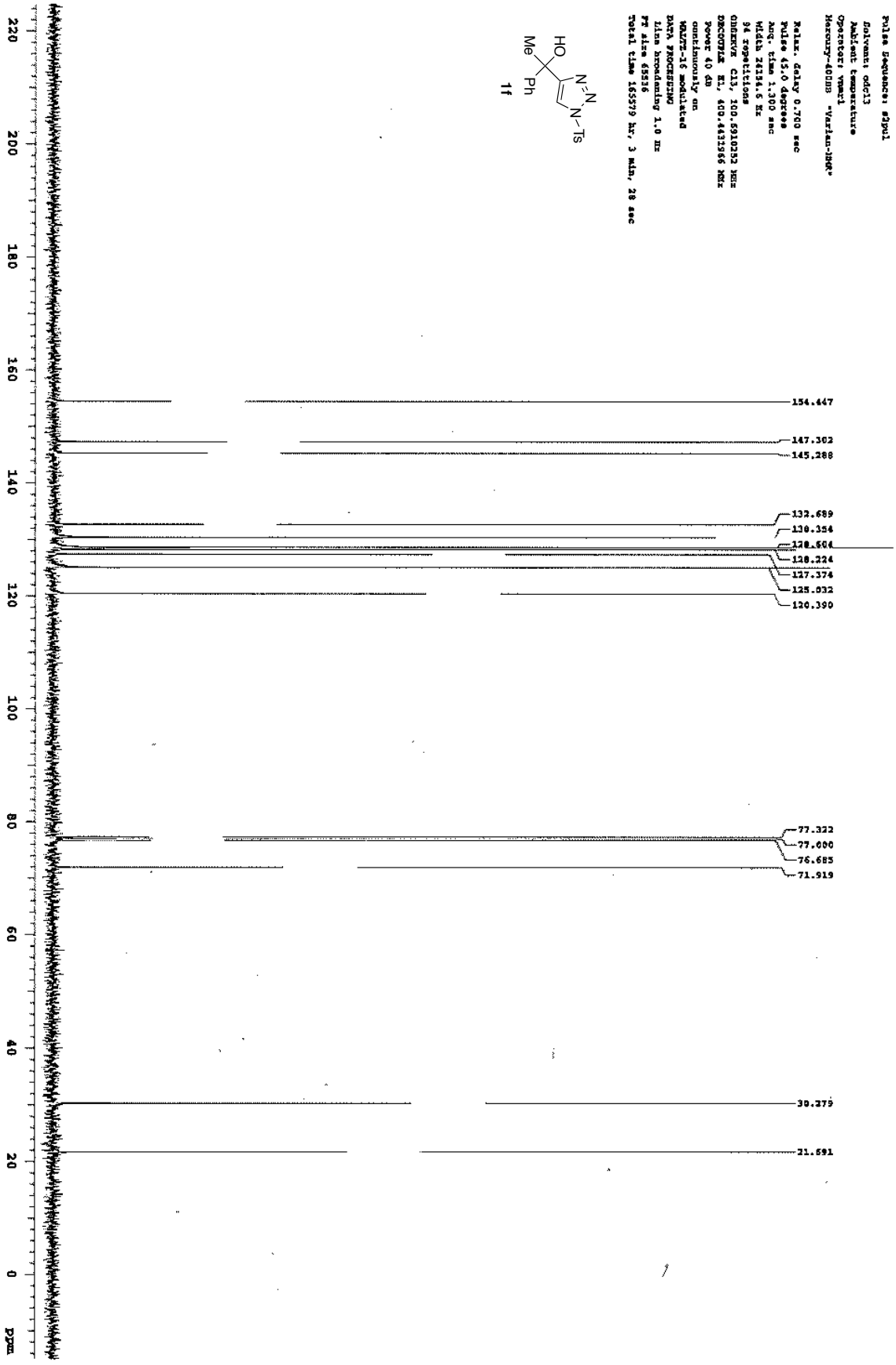
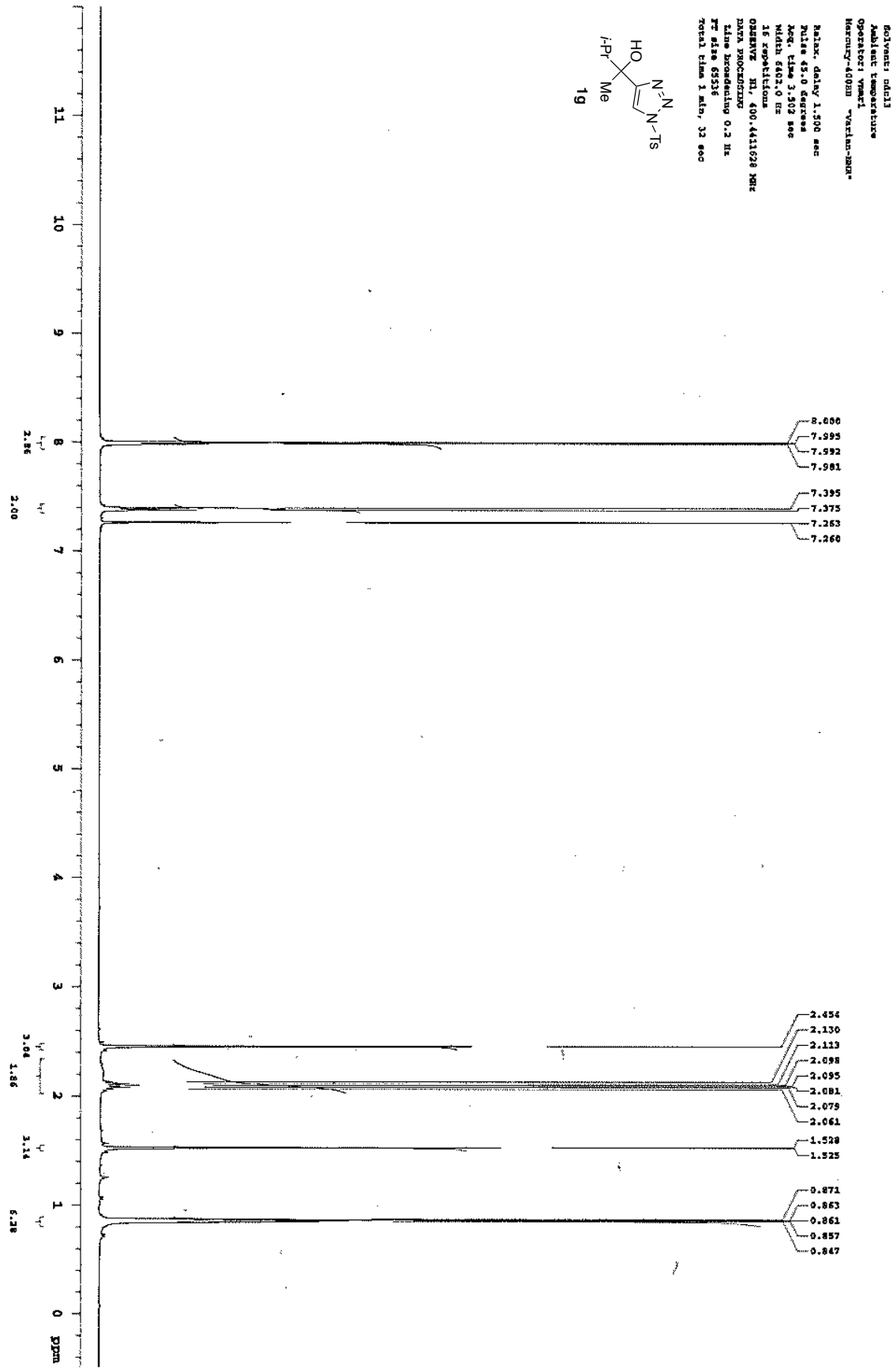
132.902
130.383
128.546
121.306
77.315
77.008
76.678
75.023
35.337
25.254
21.743
220
200
180
160
140
120
100
80
60
40
20
0
ppm

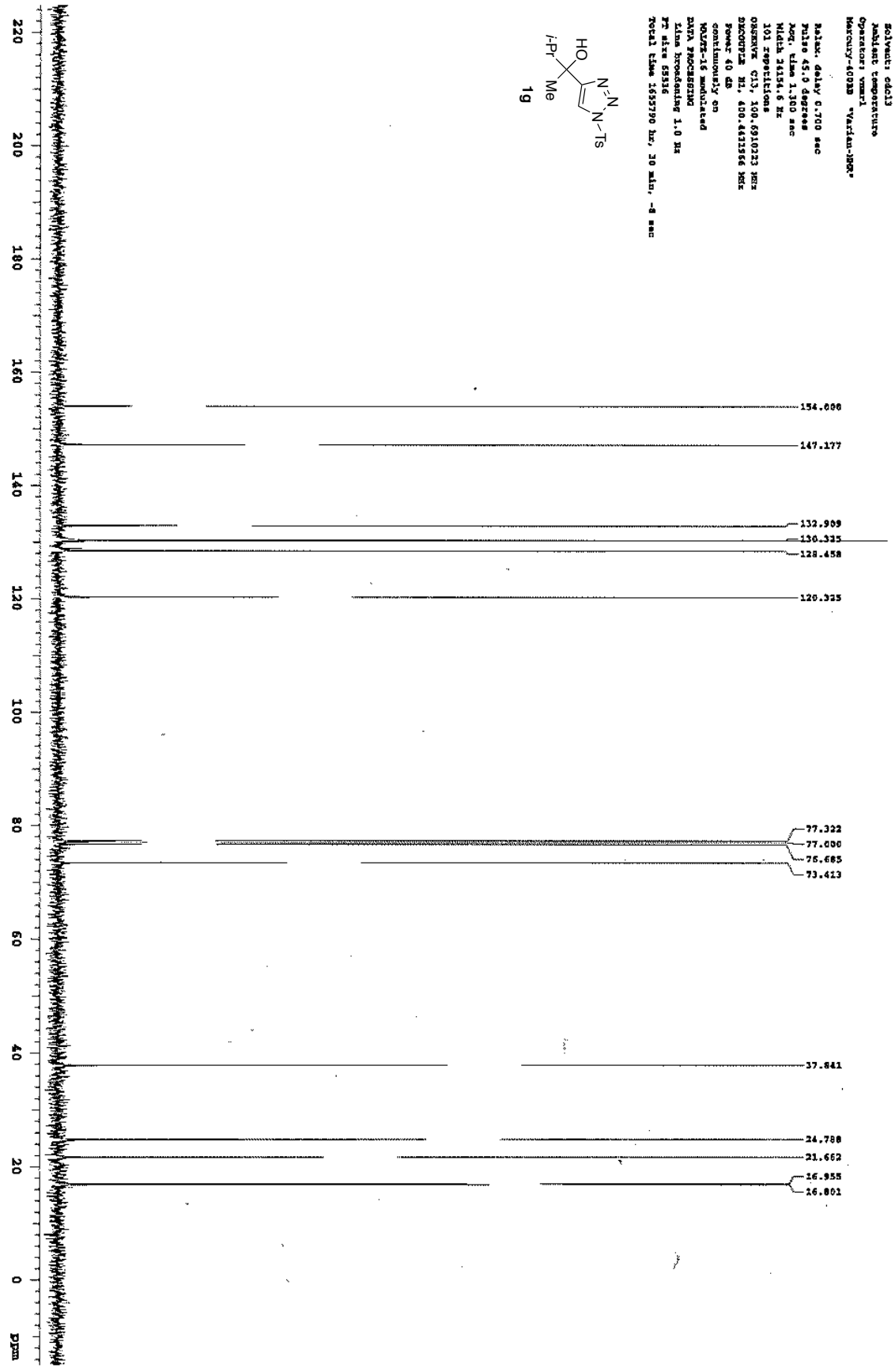

Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "varian-pmr"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4611630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
Ph
H
N
N
N
Ts
1e
7.973
7.968
7.951
7.868
7.418
7.413
7.407
7.397
7.393
7.385
7.380
7.376
7.364
7.358
7.350
7.346
7.342
7.338
7.333
7.329
7.326
7.262
7.260
5.982
2.442
2.00
7.11
1.00
1.01
1.04
3.13
11
10
9
8
7
6
5
4
3
2
1
0
ppm

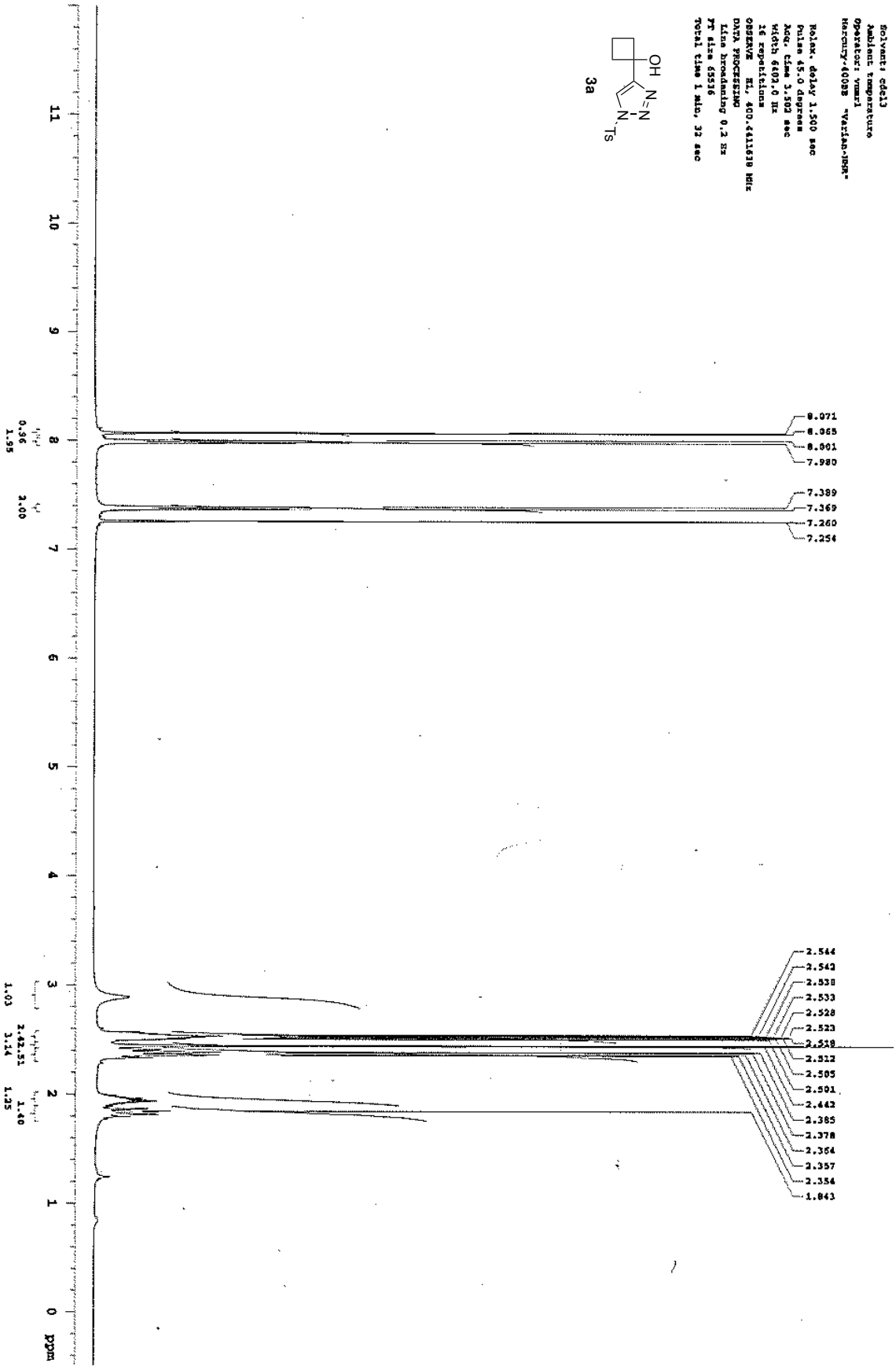

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmarl
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
235 repetitions
OBSERVE C13, 100.6210282 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1655790 hr, 30 min, -8 sec
HO
Ph
H
N
N
Ts
1e
150.882
147.338
140.830
132.616
130.347
128.582
128.158
126.313
121.152
77.315
77.190
77.800
76.685
68.610
21.684
220
200
180
160
140
120
100
80
60
40
20
0
ppm

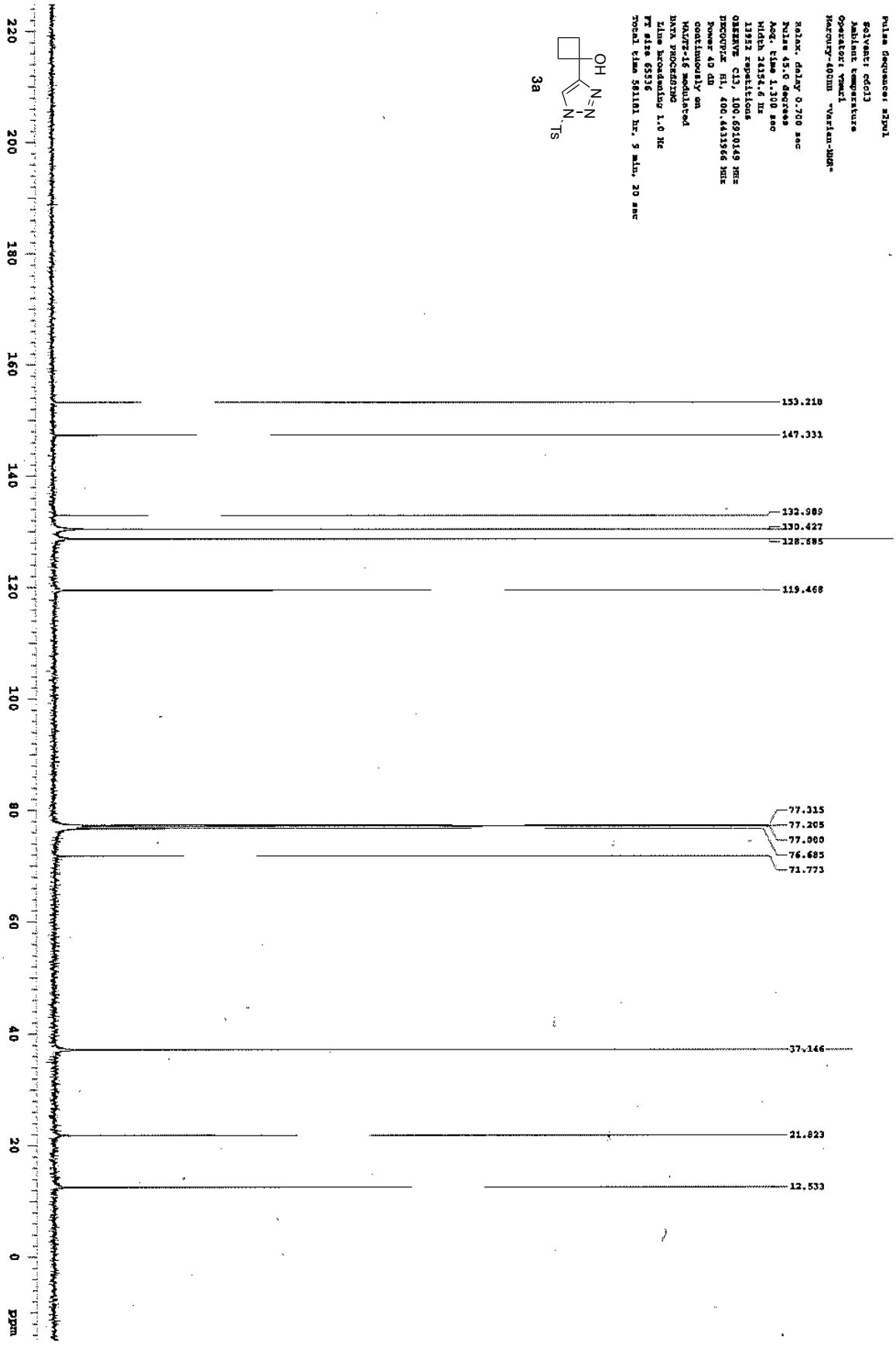

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-MRG"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
Me
Ph
N
N
N
Ts
1f
7.995
7.990
7.986
7.974
7.969
7.965
7.922
7.468
7.464
7.460
7.451
7.447
7.444
7.438
7.388
7.387
7.367
7.354
7.351
7.347
7.334
7.330
7.328
7.315
7.309
7.292
7.288
7.285
7.277
7.271
7.264
7.260
7.256
7.253
7.249
2.908
2.449
1.963
1.94
2.00
1.98
0.94
0.83
3.13
3.19
ppm

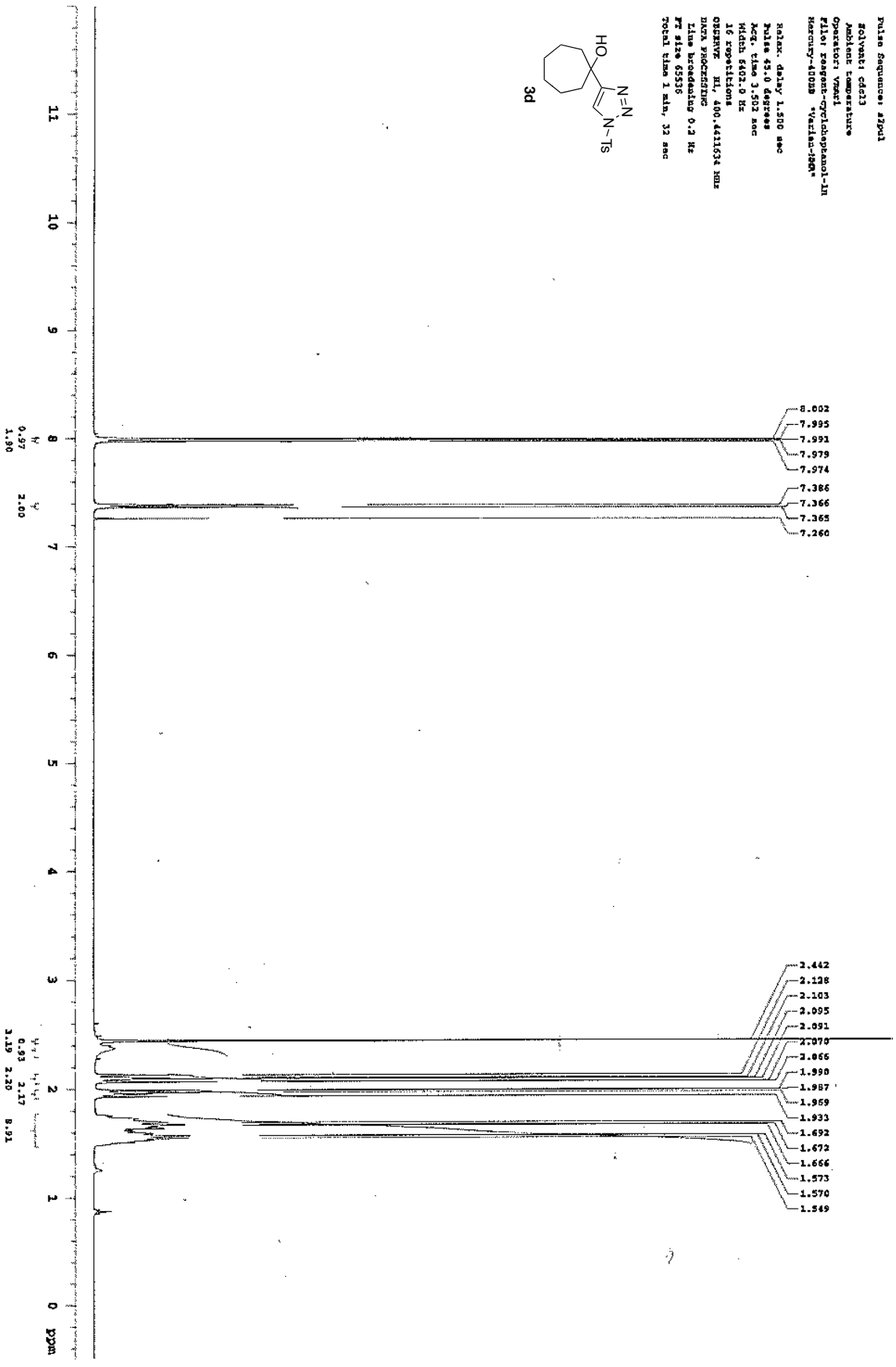

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-HRM"
Relax. delay 0.700 sec
Pulse 45.0 degree
Acq. time 1.300 sec
Width 26194.5 Hz
94 repetitions
OBSERVE C13, 100.5910252 MHz
DECOUPLE H1, 400.4433566 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 65536
Total time 165579 hr, 3 min, 28 sec
HO
Me
Ph
N
N
N
Ts
1f
154.447
147.302
145.288
132.689
130.354
128.504
128.224
127.374
125.032
120.390
77.322
77.000
76.685
71.919
39.279
21.591
220
200
180
160
140
120
100
80
60
40
20
0
ppm

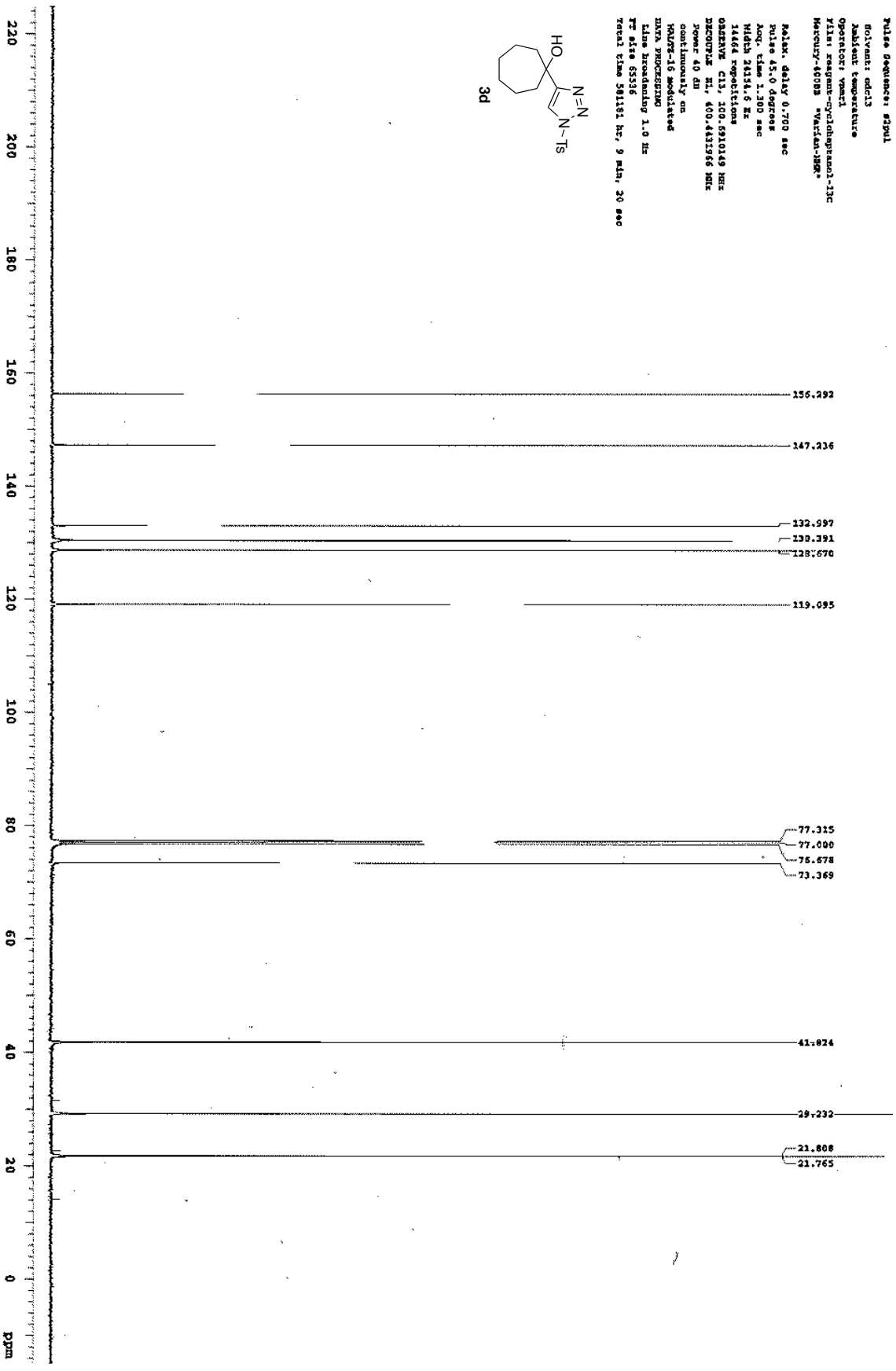

Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "varian-mrh"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411028 MHz
DATA PROCESSING
line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
i-Pr
Me
N
N
N
Ts
1g
8.000
7.995
7.992
7.981
7.395
7.375
7.263
7.260
2.454
2.130
2.113
2.098
2.095
2.081
2.079
2.061
1.528
1.525
0.871
0.863
0.861
0.857
0.847
2.86
2.00
3.04
1.86
3.24
5.28
11
10
9
8
7
6
5
4
3
2
1
0
ppm

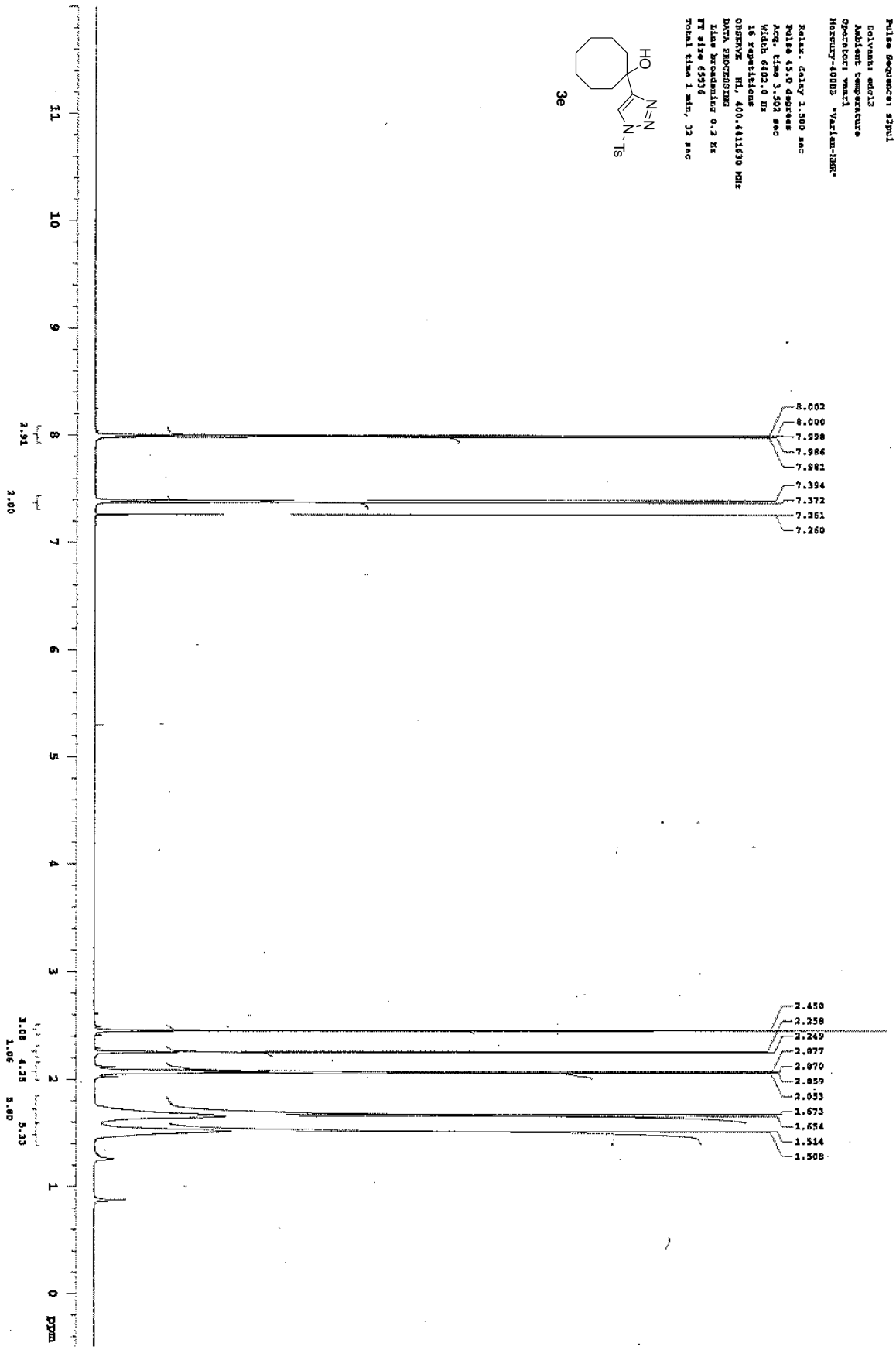

Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-MER"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
101 repetitions
OBSERVE C13, 100.6910223 MHz
DECOUPLE H1, 400.4433566 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1555790 hr, 30 min, -8 sec
HO
i-Pr
Me
N
N
N
Ts
1g
154.000
147.177
132.909
130.335
128.458
120.325
77.322
77.000
75.685
73.413
37.841
24.788
21.662
16.955
16.801
220
200
180
160
140
120
100
80
60
40
20
0
ppm

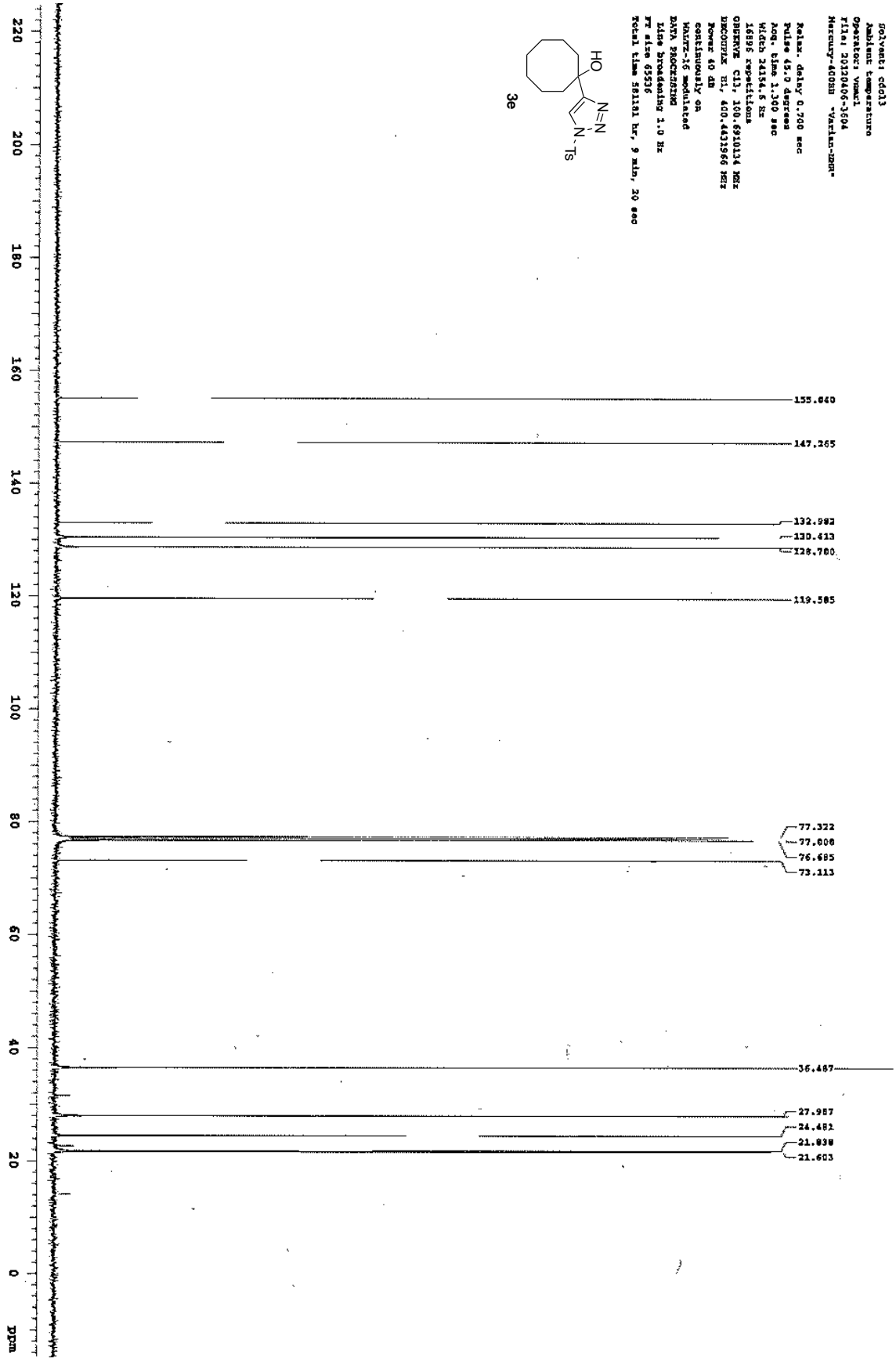

Solvent: cdcl3
Ambient temperature
Operator: vumri
Mercury-400BB "Varian-NMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6409.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
OH
N=N
N
Ts
3a
8.071
8.065
8.001
7.980
7.389
7.369
7.260
7.254
2.544
2.542
2.538
2.533
2.528
2.523
2.518
2.512
2.505
2.501
2.442
2.385
2.378
2.364
2.357
2.354
1.843
0.36
1.95
2.00
1.03
2.42.51
3.14
1.40
1.25
ppm

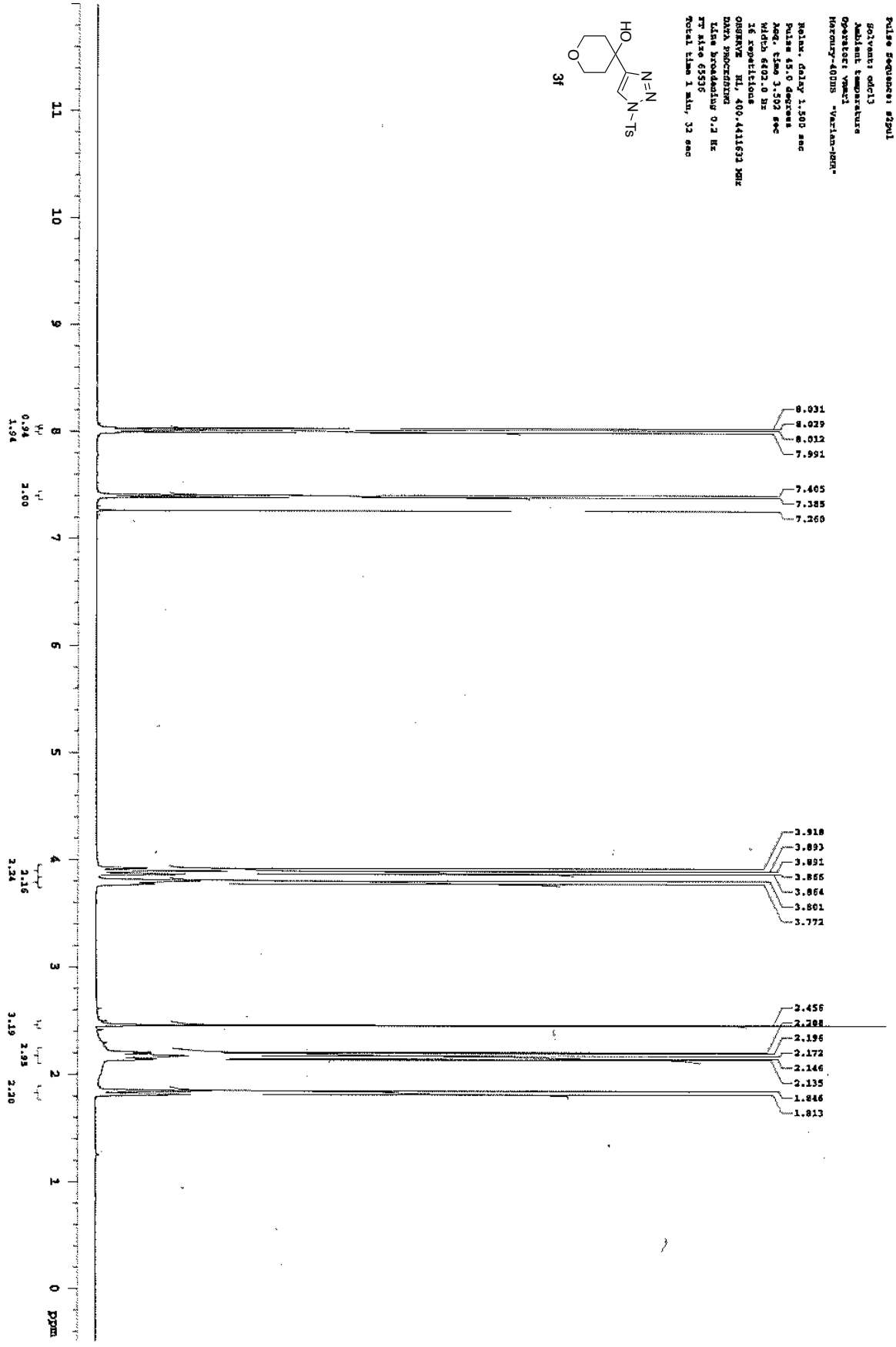

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmscl
Mercury-400BB "Varian-MBB"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24158.6 Hz
13952 repetitions
OBSERVE C13, 100.6910149 MHz
DECOUPLE H1, 400.4433566 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581101 hr, 9 min, 20 sec
OH
N
N
N
Ts
3a
153.218
147.331
132.989
130.427
128.585
119.468
77.315
77.205
77.000
76.685
71.773
37.146
21.823
12.533
220
200
180
160
140
120
100
80
60
40
20
0
ppm

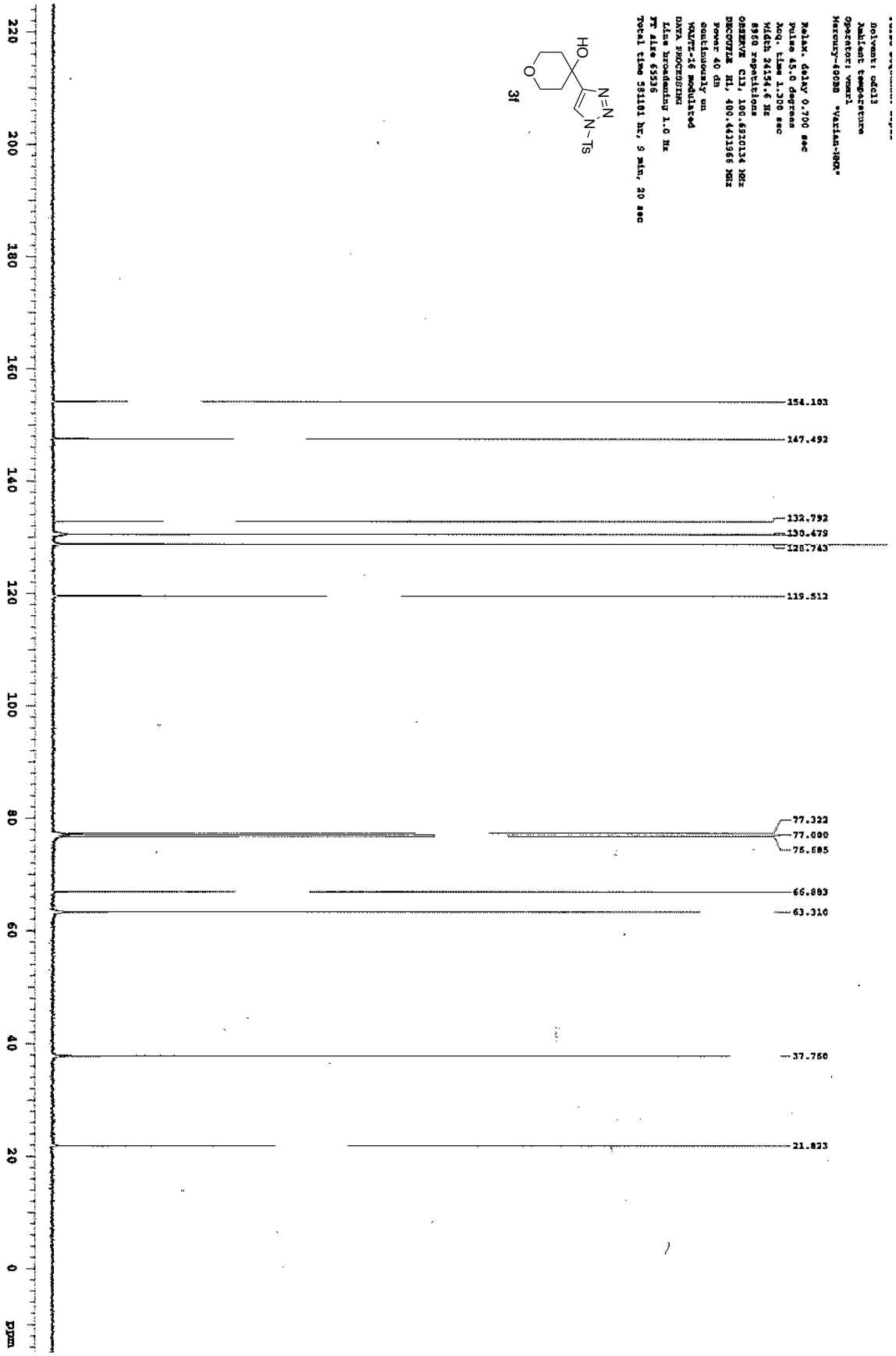

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
File: reagent-cyclohexanol-1H
Mercury-400BB "Varian-HRM"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5402.0 Hz
16 repetitions
OBSERVE H1, 400.4411634 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
N=N
N-Ts
3d
8.002
7.995
7.991
7.979
7.974
7.386
7.366
7.365
7.260
2.442
2.128
2.103
2.095
2.091
2.070
2.065
1.990
1.987
1.959
1.933
1.692
1.672
1.666
1.573
1.570
1.549
0.97
2.00
6.93
3.17
3.19
2.20
8.91
1.90
ppm

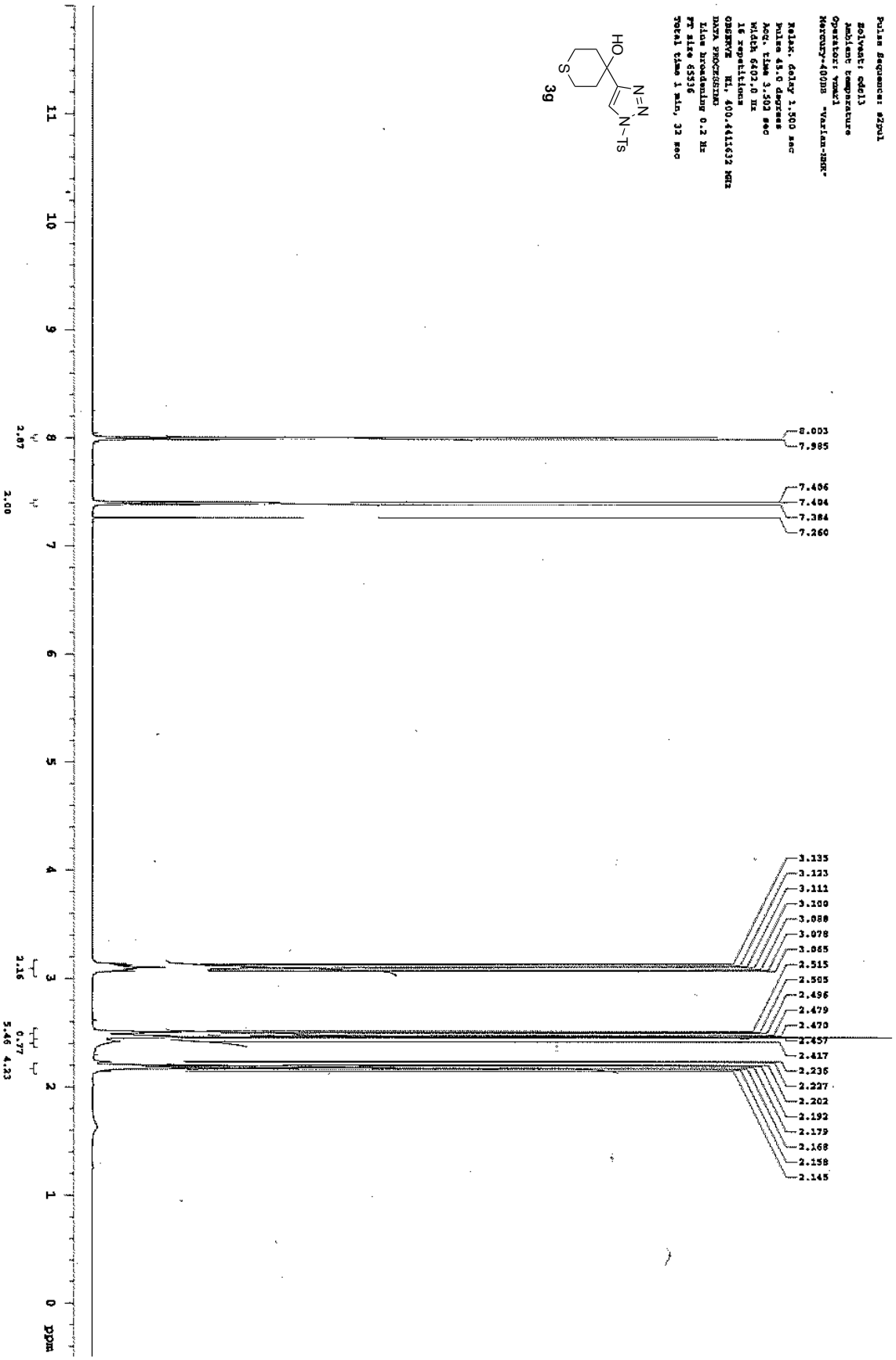

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmcl
File: reagent-cyclohexanol-13c
Mercury-400BB "varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degree
Acq. time 1.300 sec
Width 24154.6 Hz
14464 repetitions
OBSERVE C13, 100.5910149 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
EXPA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 501181 hr, 9 min, 20 sec
3d
156.292
147.236
132.997
130.391
128.670
119.095
77.315
77.000
75.678
73.369
41.824
29.232
21.808
21.765
220
200
180
160
140
120
100
80
60
40
20
0
ppm

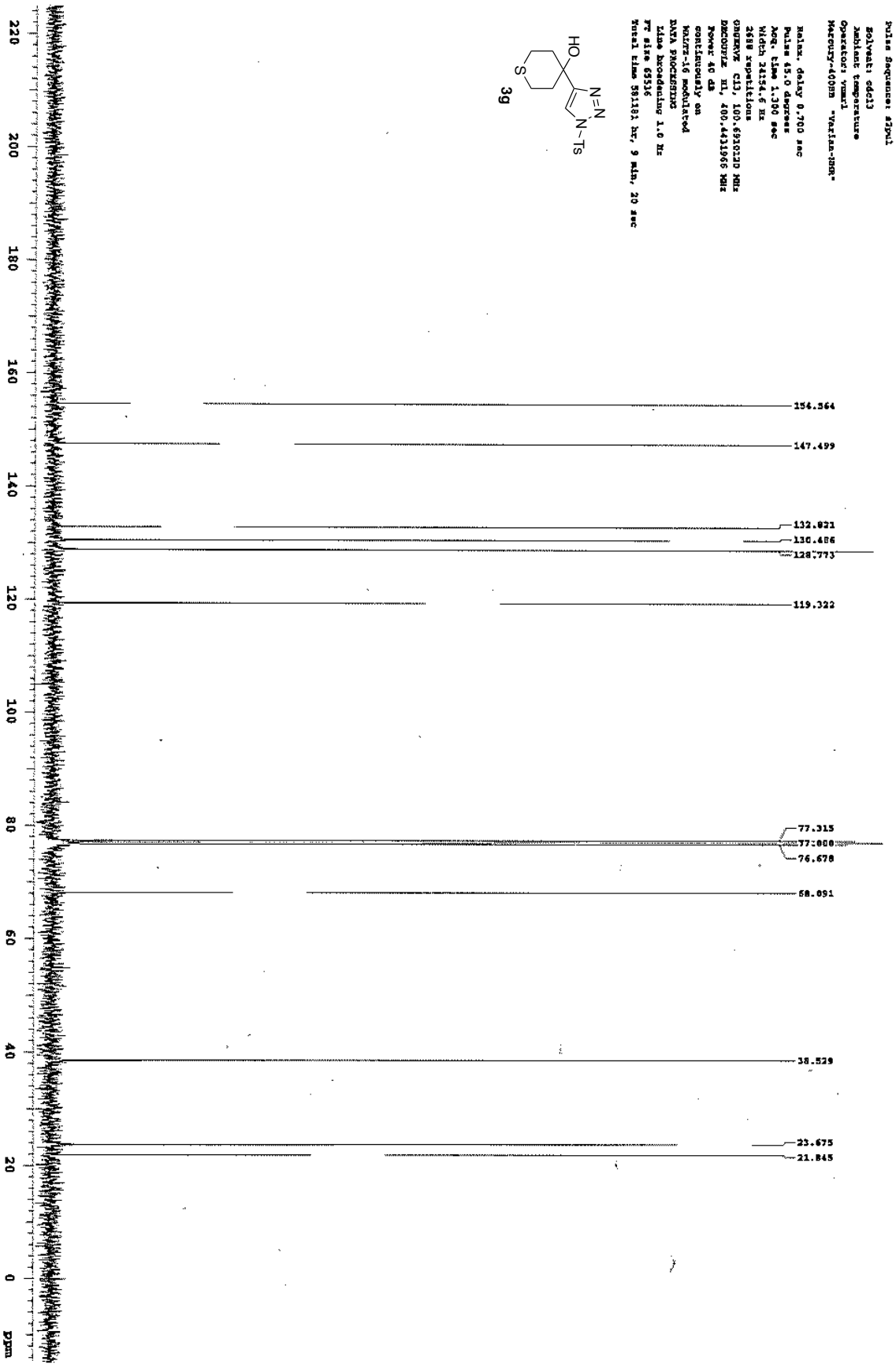

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-VNMR"
Relax. delay 2.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
N=N
N
Ts
3e
8.002
8.000
7.998
7.986
7.981
7.394
7.372
7.261
7.260
2.450
2.258
2.249
2.077
2.070
2.059
2.053
1.673
1.654
1.514
1.508
2.91
2.00
1.08
4.25
5.33
1.06
5.80
ppm

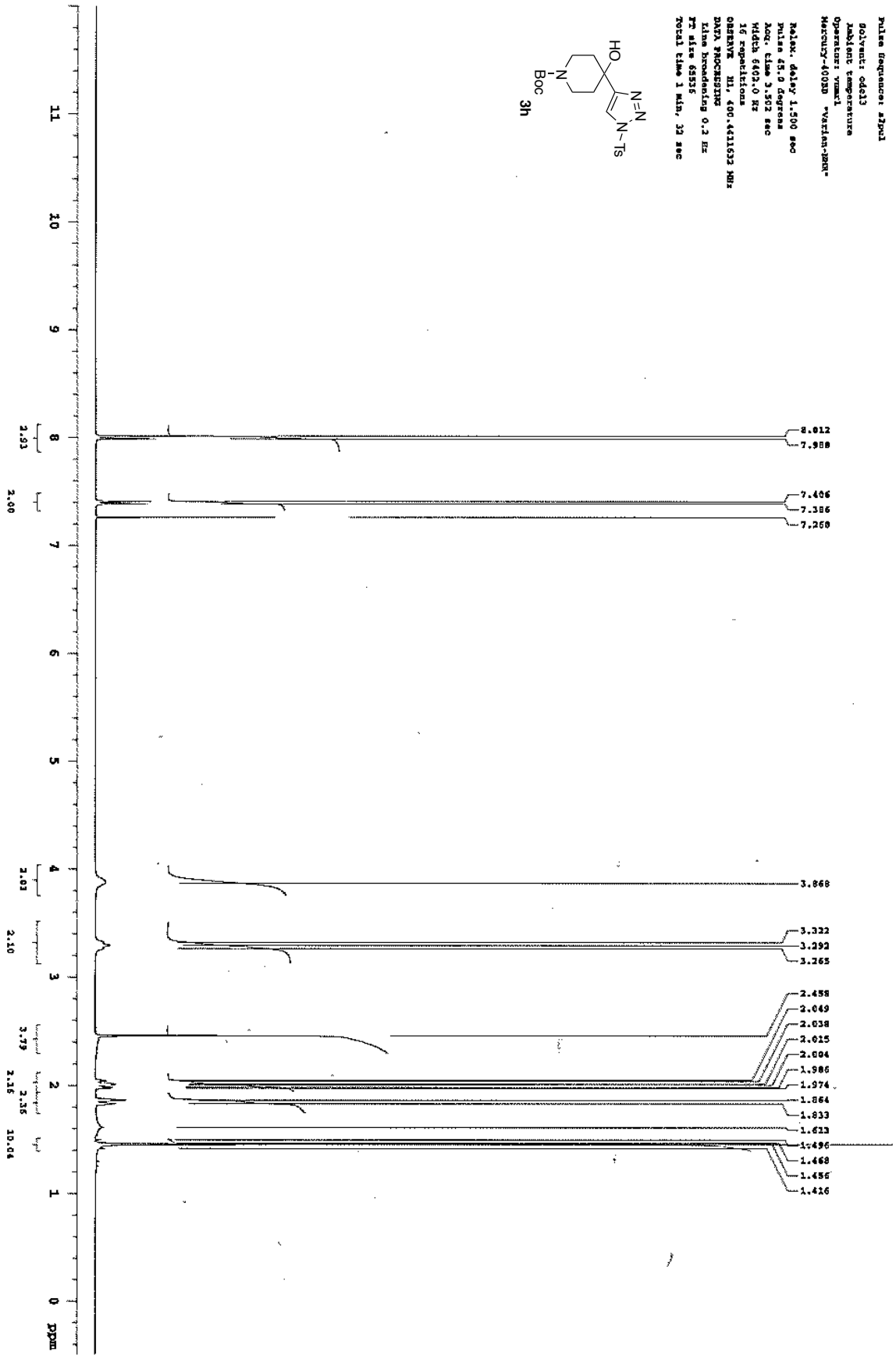

Solvent: cdcl3
Ambient temperature
Operator: vnmrl
File: 20120406-3604
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.5 Hz
16995 repetitions
OBSERVE C13, 100.6910134 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 501:01 hr, 9 min, 20 sec
HO
N=N
N
Ts
3e
155.640
147.265
132.982
130.413
128.760
119.585
77.322
77.000
76.685
73.113
36.487
27.987
24.481
21.938
21.603
220
200
180
160
140
120
100
80
60
40
20
0
ppm

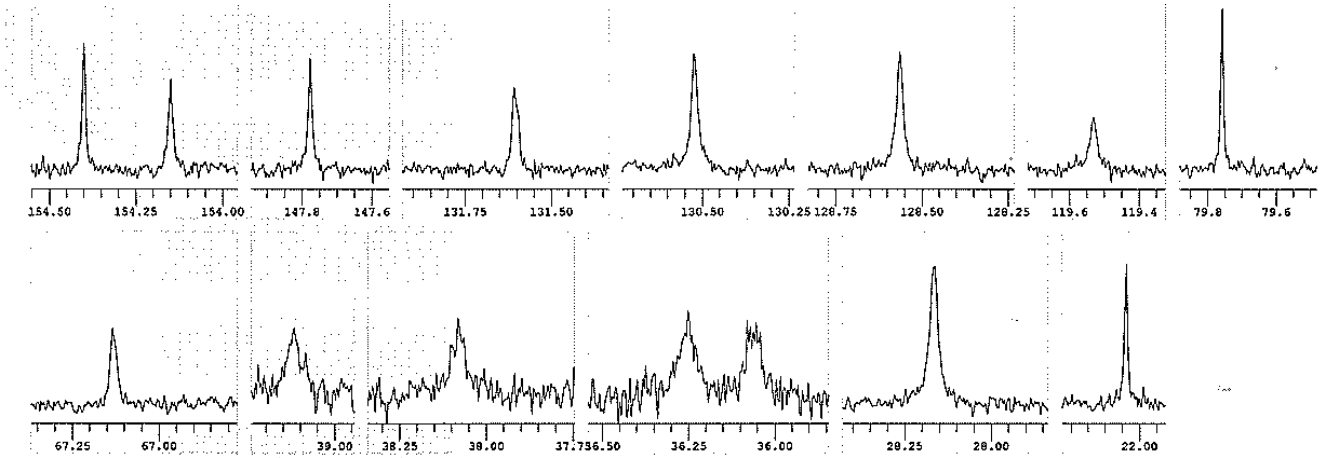

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400MS "Varian-MER"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.592 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411633 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
O
N=N
N-Ts
3f
8.031
8.029
8.012
7.991
7.405
7.385
7.260
3.918
3.893
3.891
3.865
3.864
3.801
3.772
2.456
2.208
2.196
2.172
2.146
2.135
1.846
1.813
0.94
2.00
2.16
2.24
3.19
2.95
2.20
1.94
ppm

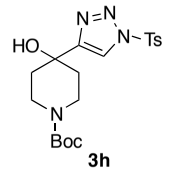

Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-HRX"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
8950 repetitions
OBSERVE C13, 100.6210134 MHz
DECOUPLE H1, 400.4413966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581101 hr, 9 min, 20 sec
HO
O
N=N
N
N
Ts
3f
154.103
147.492
132.792
130.479
128.743
119.512
77.322
77.000
76.585
66.803
63.310
37.760
21.823
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vamsi
Mercury-400BB "varian-mmr"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4416432 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
S
N=N
N
Ts
3g
8.003
7.985
7.496
7.494
7.384
7.260
3.135
3.123
3.111
3.100
3.088
3.078
3.065
2.515
2.505
2.496
2.479
2.470
2.457
2.417
2.236
2.227
2.202
2.192
2.179
2.168
2.158
2.145
2.87
2.00
2.16
0.77
5.46
4.23
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
2688 repetitions
OBSERVE C13, 100.6910230 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 58:181 hr, 9 min, 20 sec
HO
S
N=N
N
Ts
3g
154.364
147.499
132.921
130.486
128.773
119.322
77.315
77.000
76.678
58.091
38.529
23.675
21.845
220
200
180
160
140
120
100
80
60
40
20
0
ppm

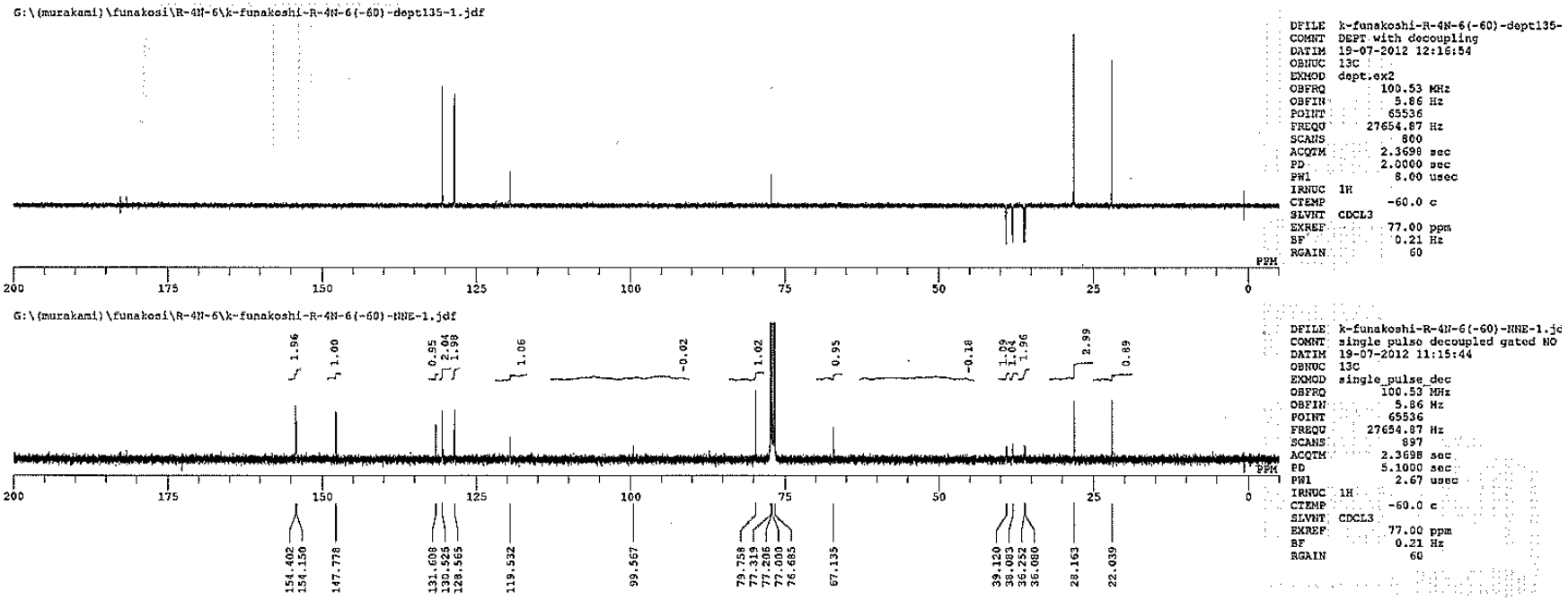

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "varian-2RCM"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
HO
N
Boc
3h
N=N
N
Ts
8.012
7.988
7.406
7.386
7.260
3.868
3.332
3.292
3.265
2.458
2.049
2.038
2.015
2.004
1.986
1.974
1.864
1.833
1.623
1.496
1.468
1.456
1.416
2.03
2.00
2.03
2.10
3.79
2.35
2.35
10.04
11
10
9
8
7
6
5
4
3
2
1
0
ppm

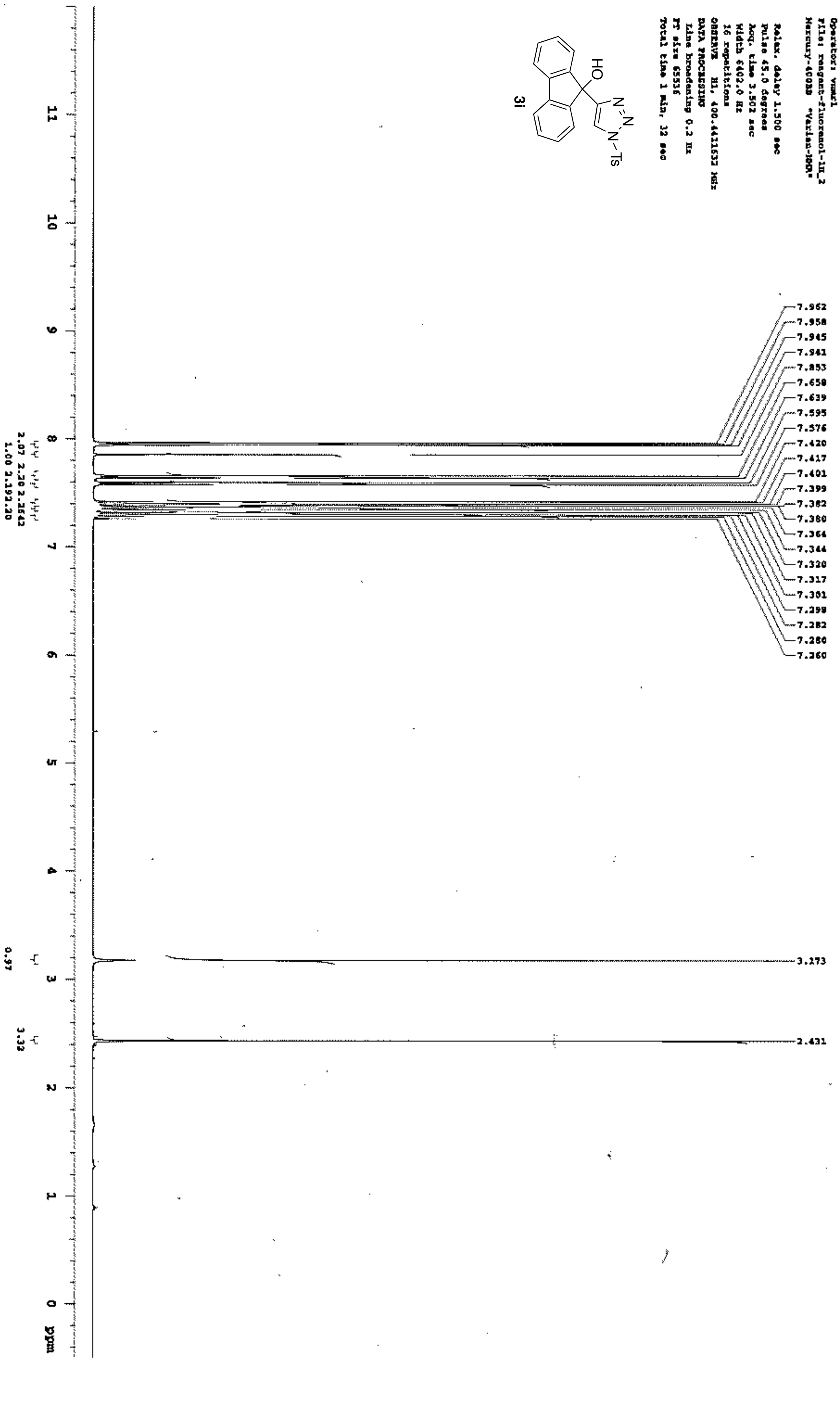

154.50
154.25
154.00
147.8
147.6
131.75
131.50
130.50
130.25
128.75
128.50
128.25
119.6
119.4
79.8
79.6
67.25
67.00
39.00
38.25
38.00
37.75
36.50
36.25
36.00
28.25
28.00
22.00

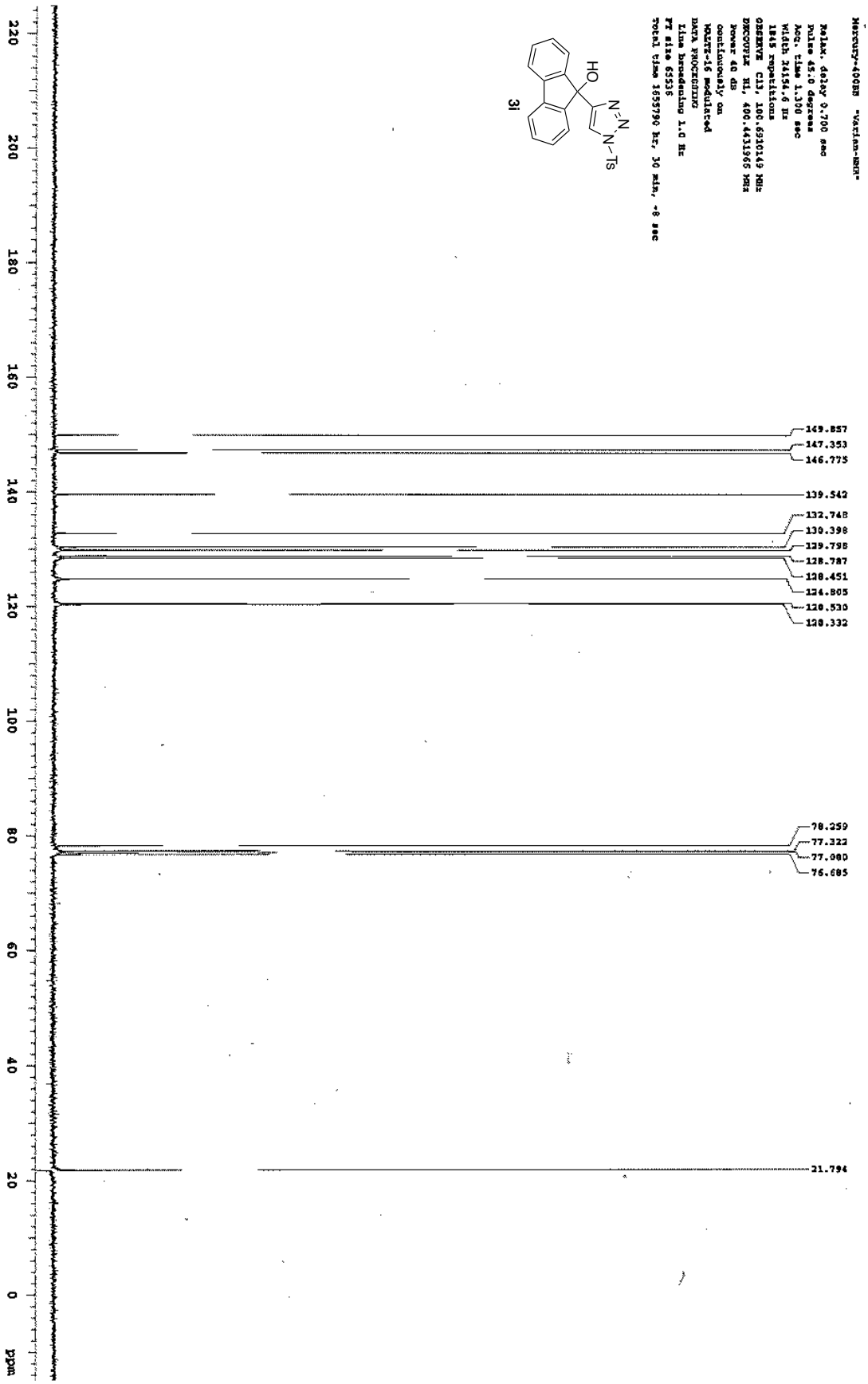

HO
N=N
N-Ts
N
Boc

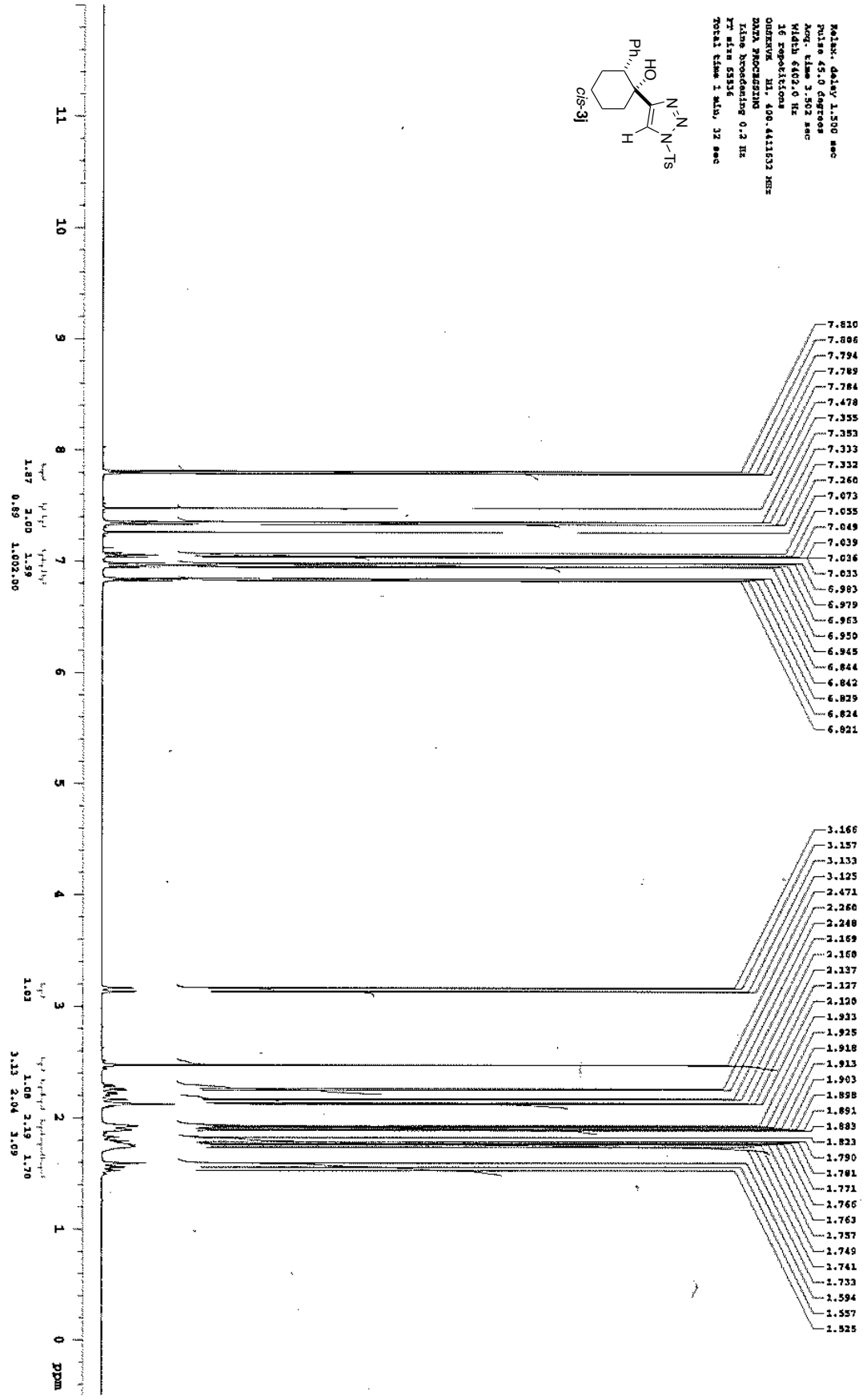
3h

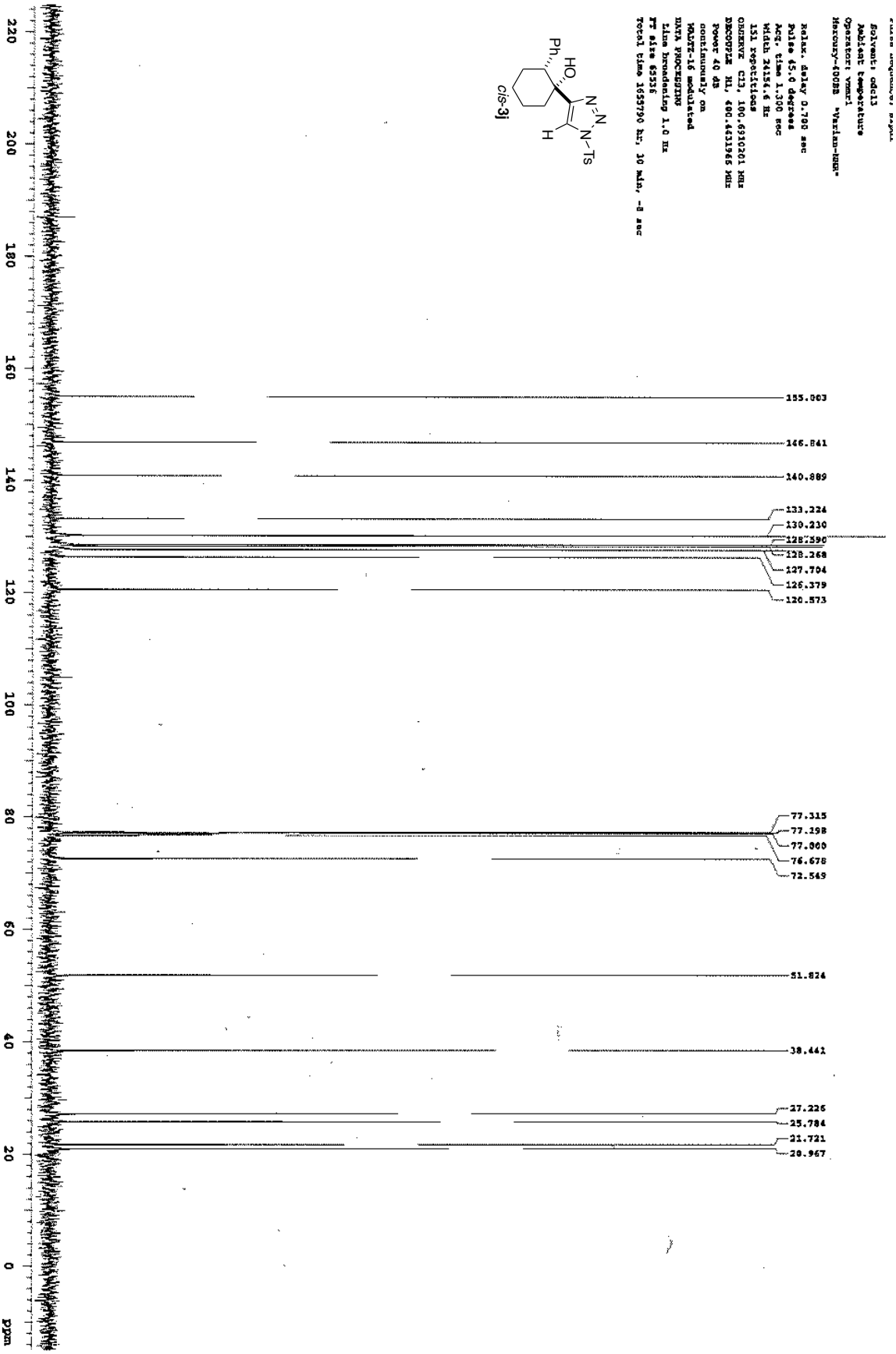
930875

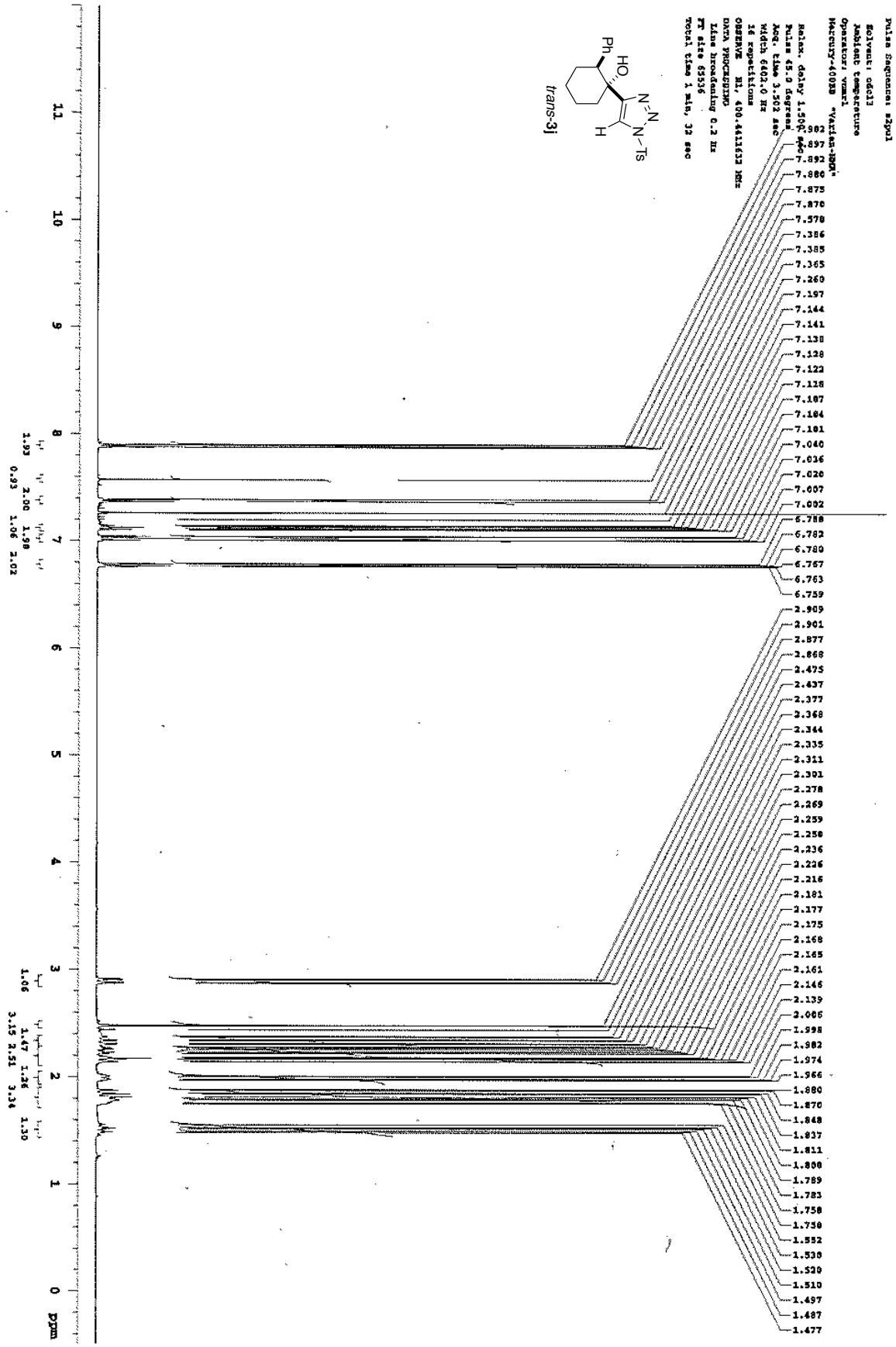

G:\(murakami)\funakosi\R-4N-6\k-funakoshi-R-4N-6(-60)-dept135-1.jdf
DFILE k-funakoshi-R-4N-6(-60)-dept135-
COMNT DEPT with decoupling
DATIM 19-07-2012 12:16:54
OBNUC 13C
EXMOD dept.ex2
OBFRQ 100.53 MHz
OBFIN 5.86 Hz
POINT 65536
FREQU 27654.87 Hz
SCANS 800
ACQTM 2.3698 sec
PD 2.0000 sec
PW1 8.00 usec
IRNUC 1H
CTEMP -60.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.21 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0
G:\(murakami)\funakosi\R-4N-6\k-funakoshi-R-4N-6(-60)-NNE-1.jdf
1.96
1.00
0.95
2.04
1.98
1.06
-0.02
1.02
0.95
-0.18
1.09
1.04
1.96
2.99
0.89
DFILE k-funakoshi-R-4N-6(-60)-NNE-1.jd
COMNT single pulse decoupled gated NO
DATIM 19-07-2012 11:15:44
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBFIN 5.86 Hz
POINT 65536
FREQU 27654.87 Hz
SCANS 897
ACQTM 2.3698 sec
PD 5.1000 sec
PW1 2.67 usec
IRNUC 1H
CTEMP -60.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.21 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0
154.402
154.150
147.778
131.608
130.525
128.565
119.532
99.567
79.758
77.319
77.206
77.000
76.685
67.135
39.120
38.083
36.252
36.080
28.163
22.039

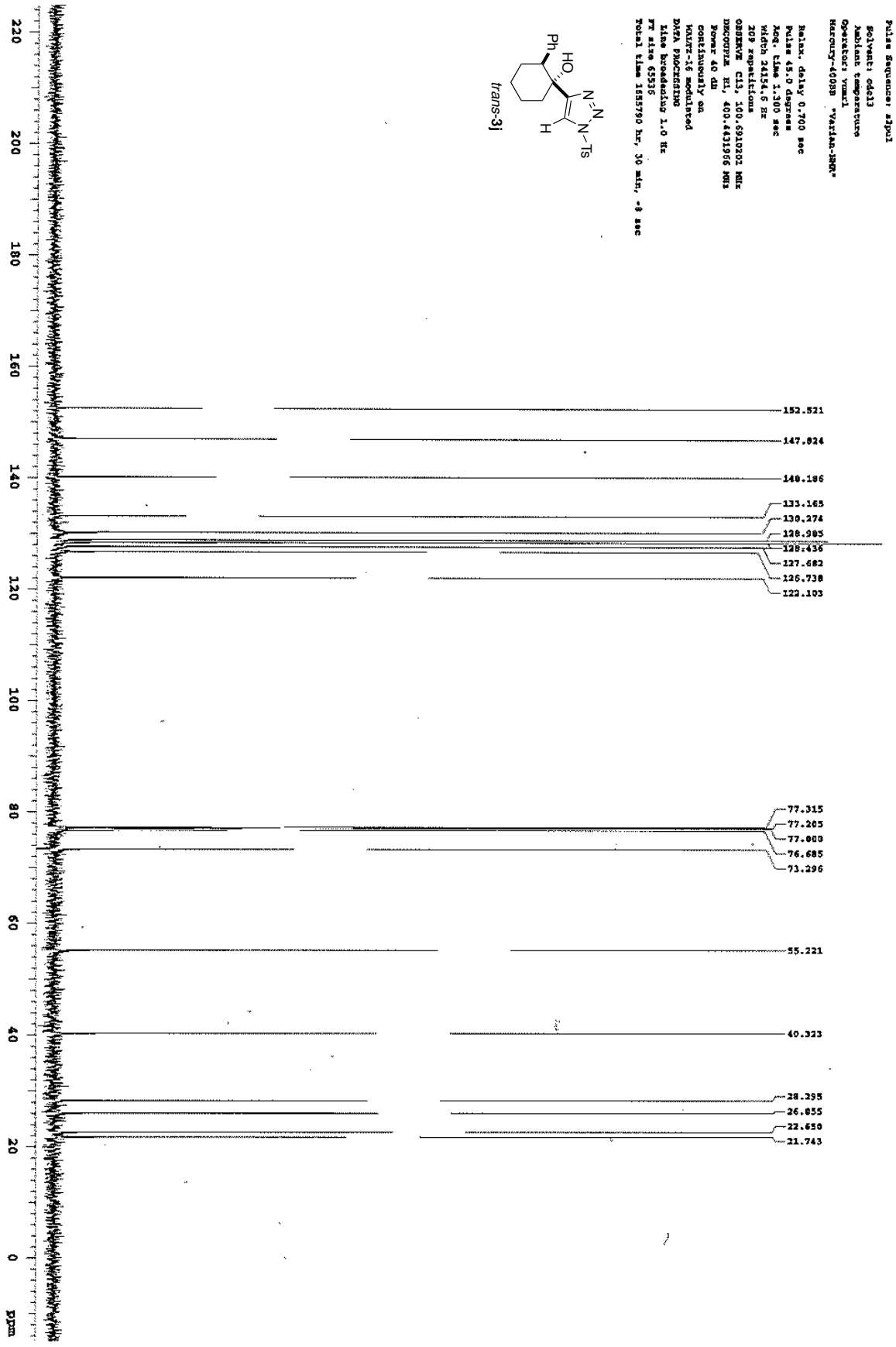

Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: reagent-fluorenol-1H_2
Mercury-400BB "Varian-NMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411637 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 12 sec
HO
N=N
N–Ts
3i
7.962
7.958
7.945
7.941
7.853
7.658
7.639
7.595
7.576
7.420
7.417
7.401
7.399
7.382
7.380
7.364
7.344
7.320
7.317
7.301
7.298
7.282
7.280
7.260
3.173
2.431
2.07
1.00
2.20
2.19
2.26
2.20
0.97
3.32
11
10
9
8
7
6
5
4
3
2
1
0
ppm

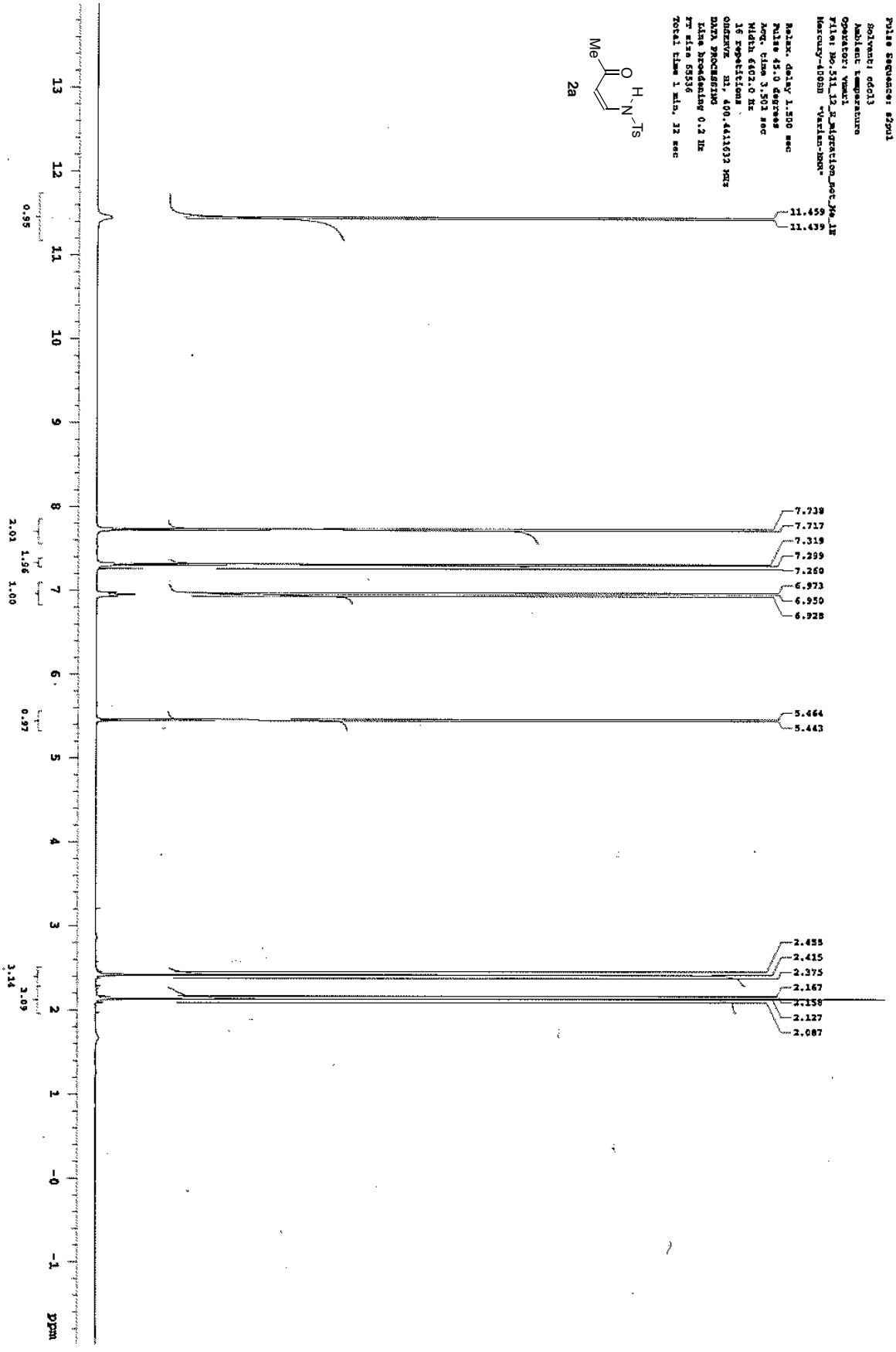

Mercury-400BB "varian-mnh"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1845 repetitions
OBSERVE C13, 100.6310149 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1655790 hr, 30 min, +8 sec
HO
N=N
N
Ts
3i
149.857
147.353
146.775
139.542
132.748
130.398
129.798
128.787
128.451
124.805
120.530
120.332
78.259
77.322
77.000
76.685
21.794
220
200
180
160
140
120
100
80
60
40
20
0
ppm

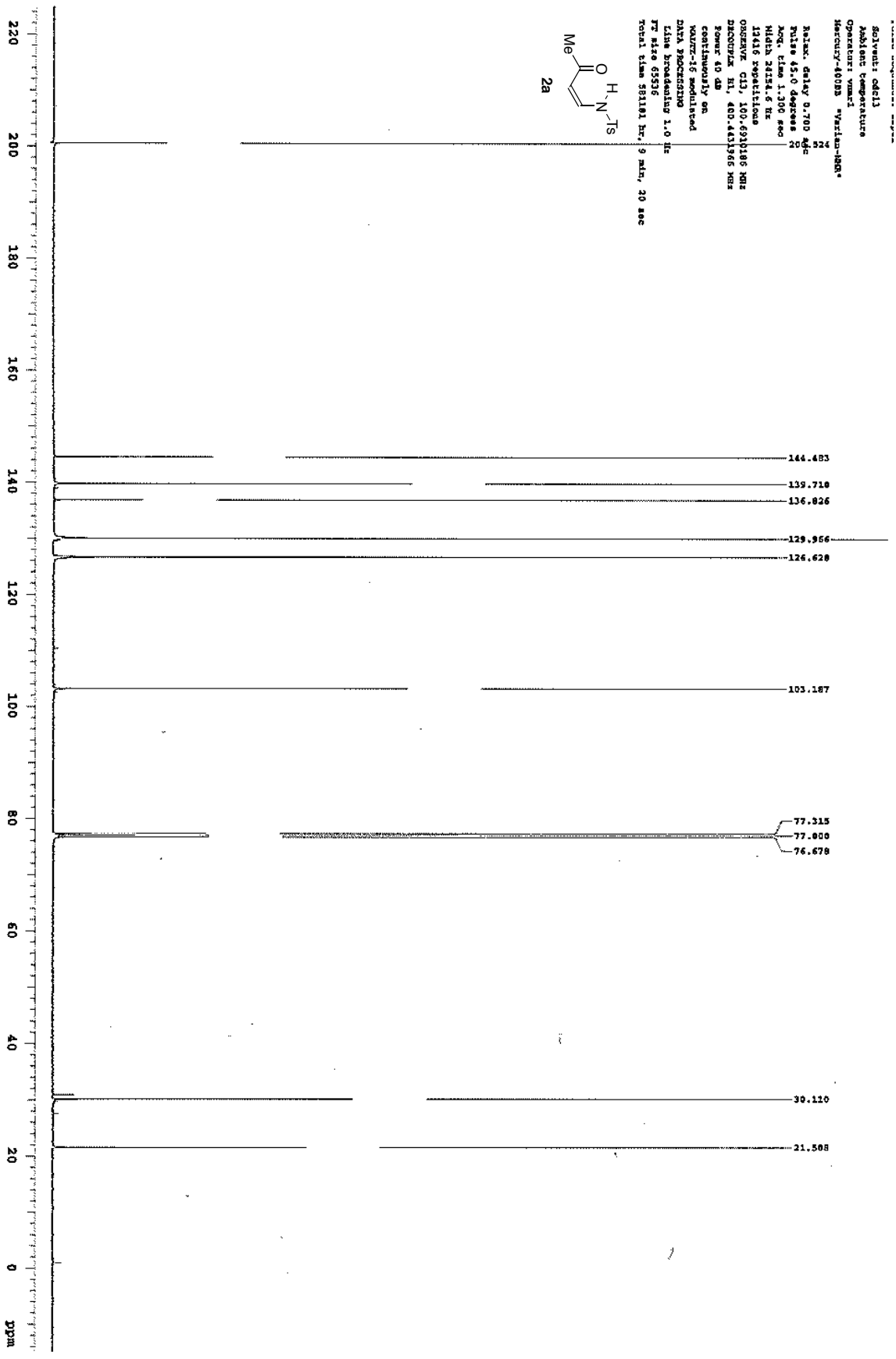

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.6 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
cis-3j
Ph
HO
N
N
Ts
H
7.810
7.806
7.794
7.789
7.784
7.478
7.355
7.353
7.333
7.332
7.260
7.073
7.055
7.049
7.039
7.036
7.033
6.983
6.979
6.963
6.950
6.945
6.844
6.842
6.829
6.824
6.821
3.166
3.157
3.133
3.125
2.471
2.260
2.248
2.169
2.160
2.137
2.127
2.120
1.933
1.925
1.918
1.913
1.903
1.898
1.891
1.883
1.823
1.790
1.781
1.771
1.766
1.763
1.757
1.749
1.741
1.733
1.594
1.557
1.525
1.87
3.00
1.99
1.002.00
1.03
3.13
2.04
3.09
1.08
2.19
1.70
0.89
ppm

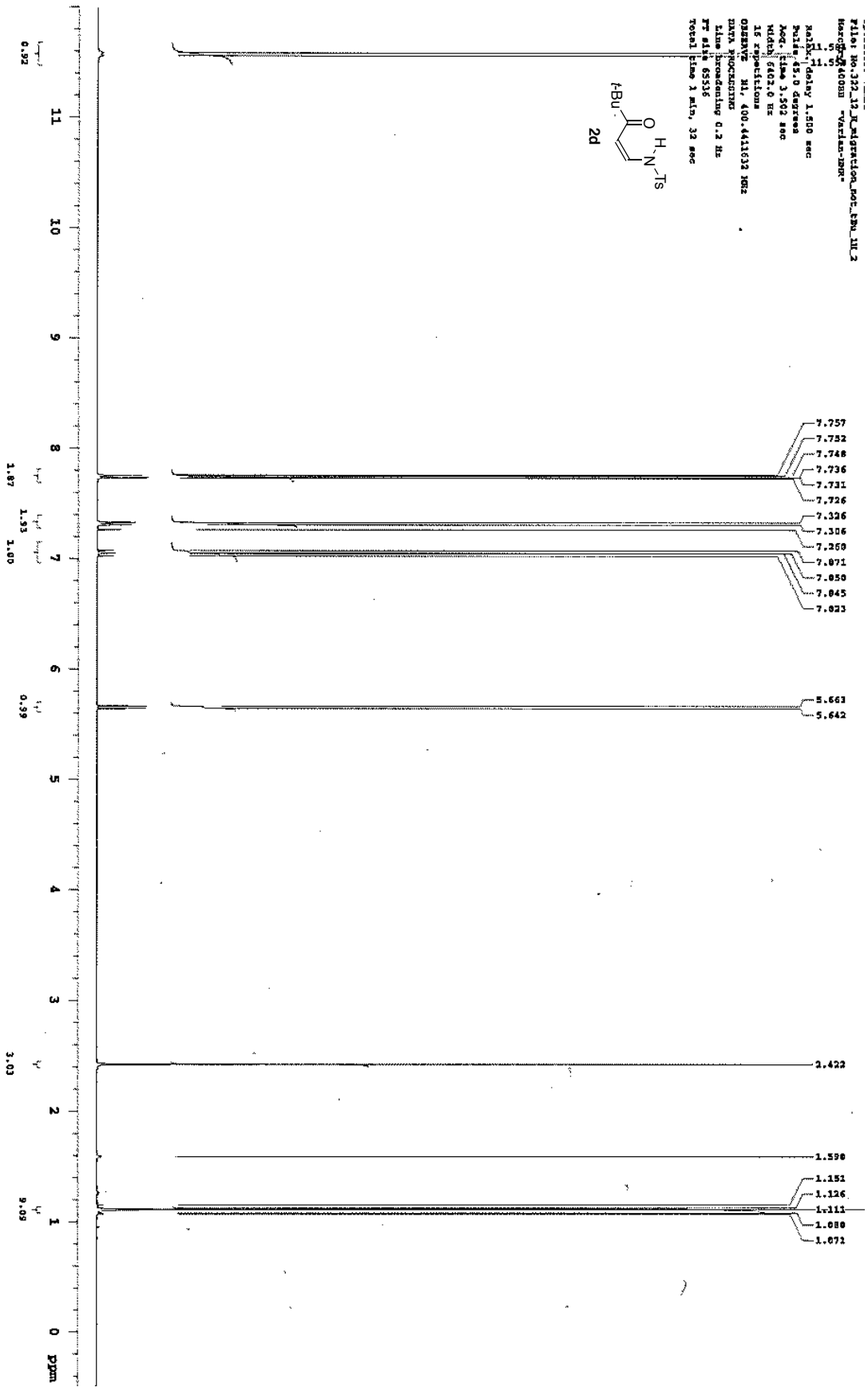

Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-MERC"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
151 repetitions
OBSERVE C13, 100.6916201 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1659796 hr, 30 min, -8 sec
cis-3j
HO
Ph
N
N
Ts
H
155.003
146.841
140.889
133.224
130.230
128.390
128.268
127.704
126.379
120.573
77.315
77.198
77.000
76.678
72.549
51.824
38.441
27.226
25.784
21.721
20.967
220
200
180
160
140
120
100
80
60
40
20
0
ppm

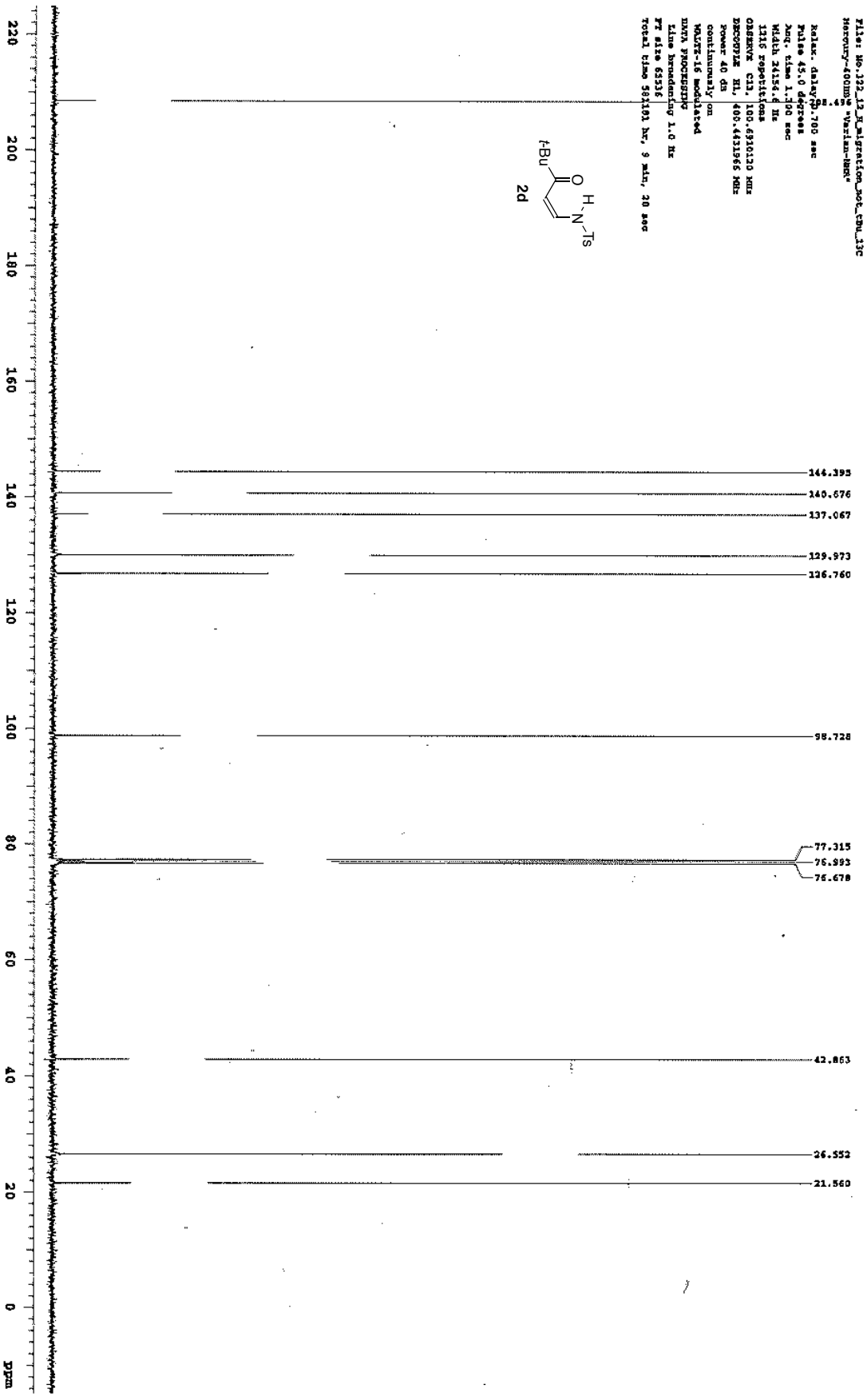

trans-3j

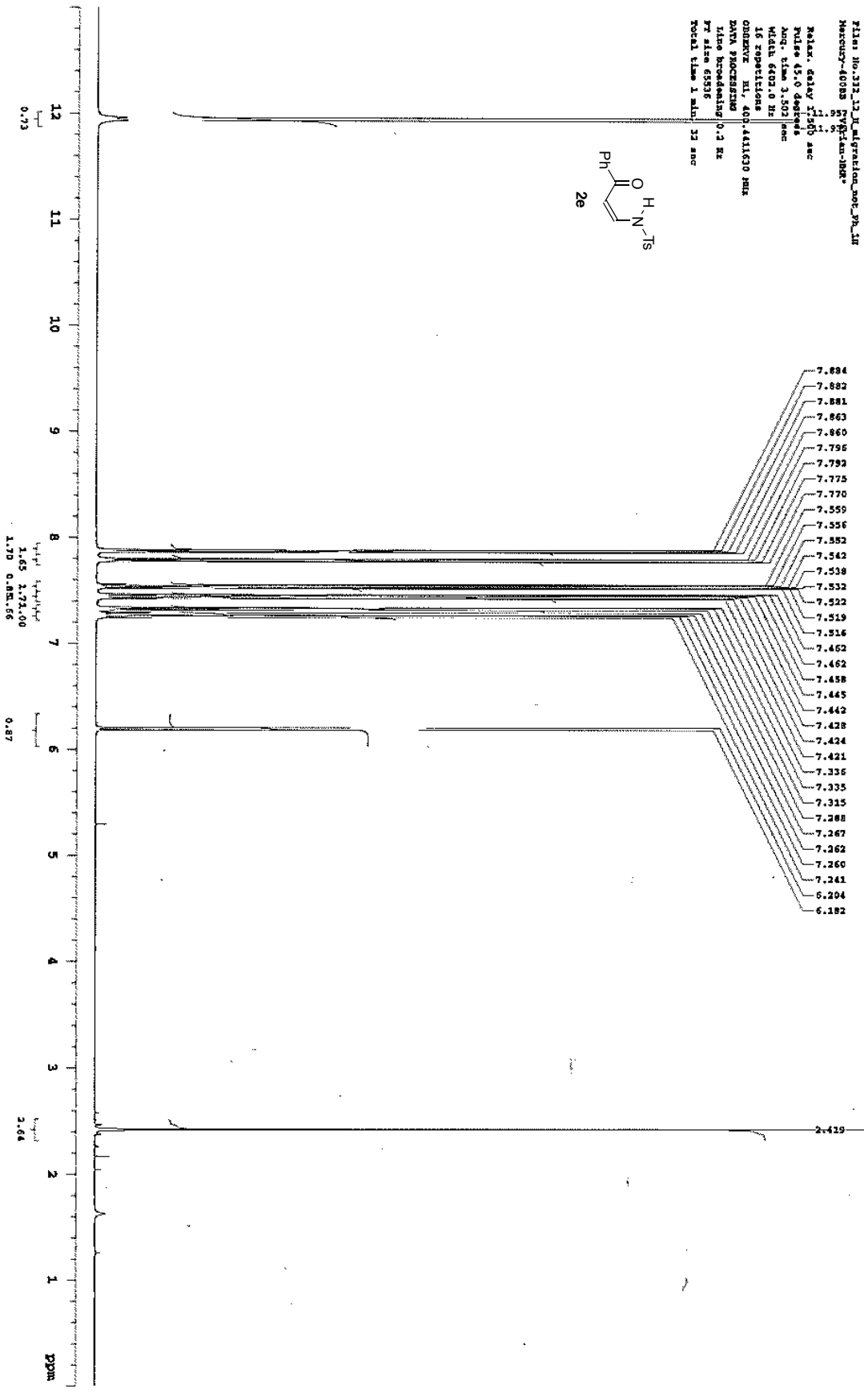

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.5 Hz
209 repetitions
OBSERVE C13, 100.6910202 MHz
DECOUPLE H1, 400.4431956 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1655790 hr, 30 min, -8 sec
Ph
HO
H
N
N
N
Ts
trans-3j
152.521
147.824
140.186
133.165
130.274
128.985
128.436
127.682
126.738
122.103
77.315
77.205
77.000
76.685
73.296
55.221
40.323
28.295
26.055
22.650
21.743
220
200
180
160
140
120
100
80
60
40
20
0
ppm

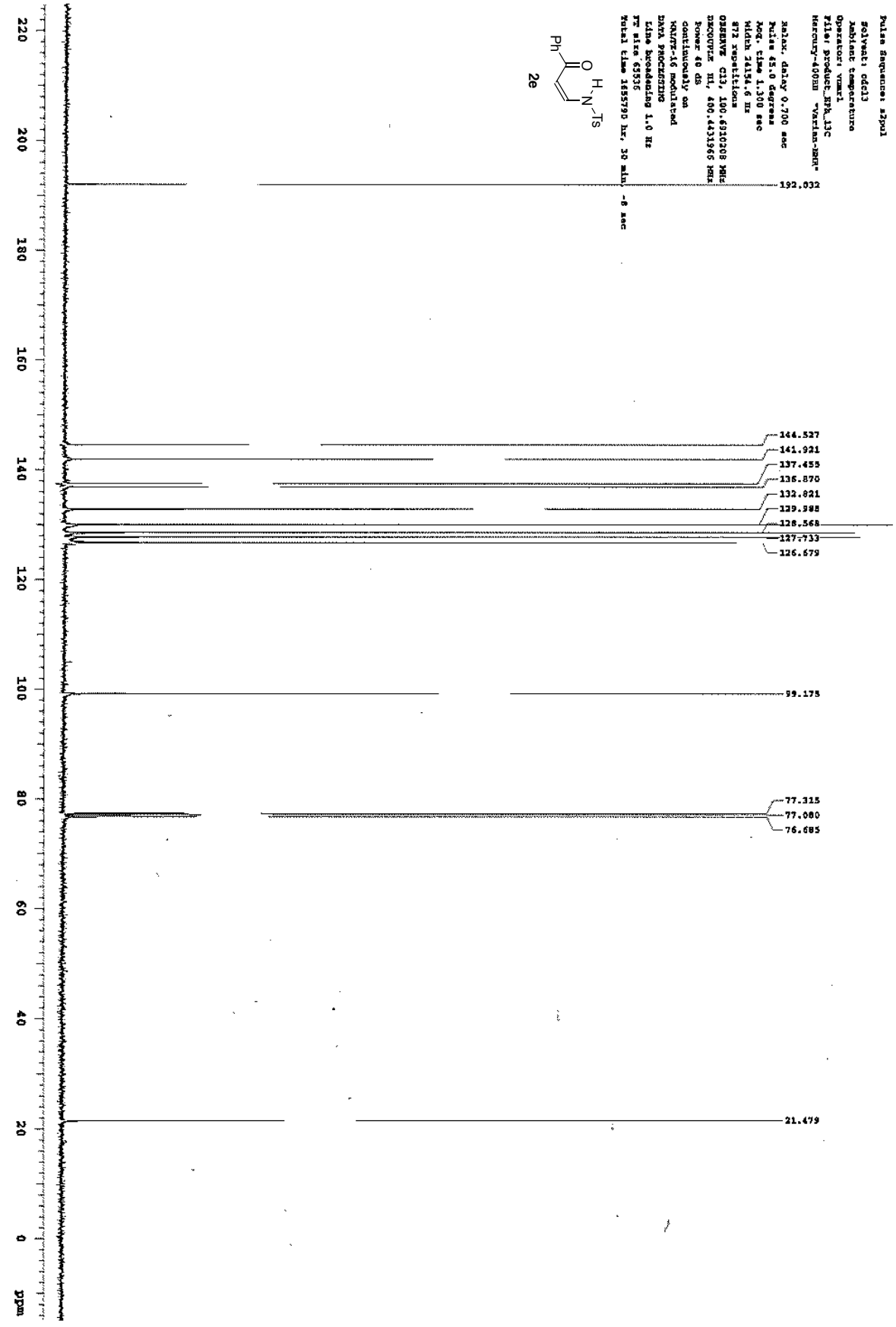

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
File: Mo-511_12_2_migration_pot_Me.fi
Mercury-400BB "Varian-NMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6602.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
Me
O
H
N
Ts
2a
11.459
11.439
7.738
7.717
7.319
7.299
7.260
6.973
6.950
6.928
5.464
5.443
2.455
2.415
2.375
2.167
2.158
2.127
2.087
0.95
2.01
1.96
1.00
0.97
3.14
3.09
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

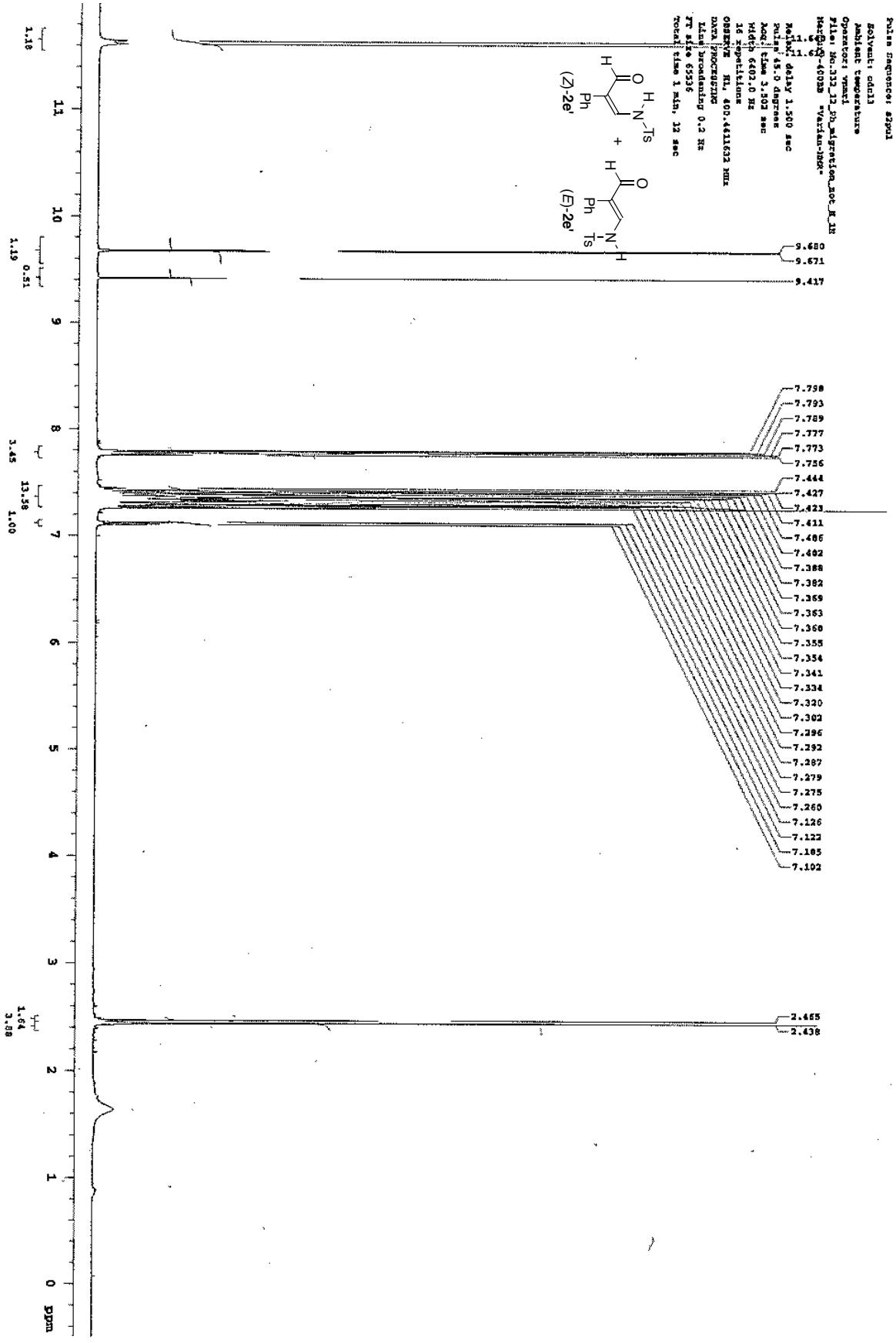

Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
12416 repetitions
OBSERVE C13, 100.6910186 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 58181 hr, 9 min, 20 sec
Me
O
H
N
Ts
2a
144.483
139.710
136.826
129.956
126.628
103.187
77.315
77.000
76.678
30.120
21.508
220
200
180
160
140
120
100
80
60
40
20
0
ppm

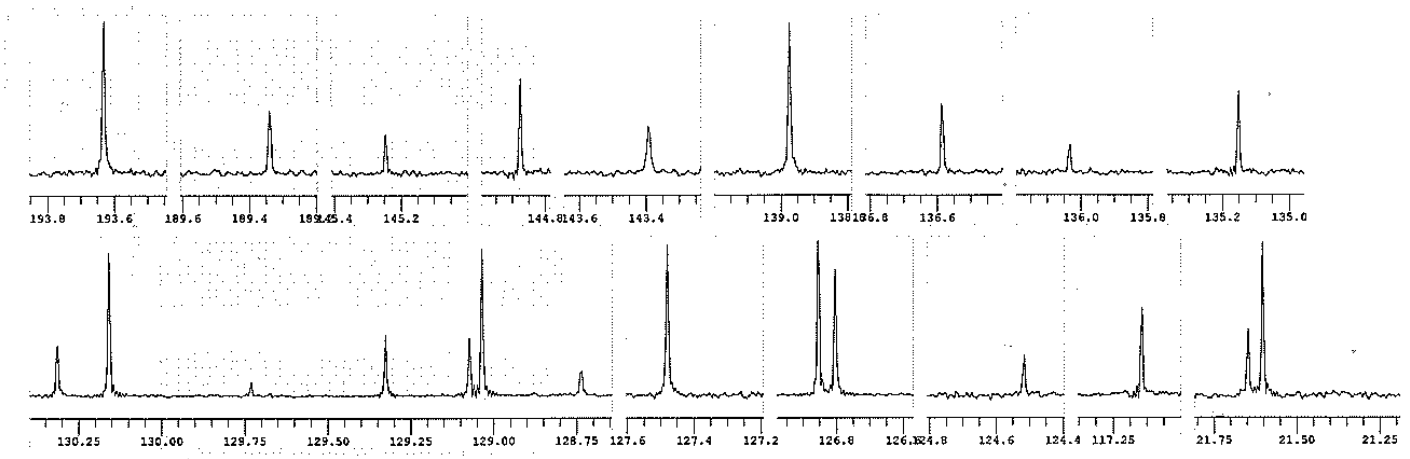

t-Bu
O
H
N
Ts
2d
7.757
7.752
7.748
7.736
7.731
7.726
7.326
7.306
7.269
7.071
7.050
7.045
7.023
5.663
5.642
2.422
1.590
1.151
1.126
1.111
1.080
1.071
0.92
1.87
1.93
1.05
0.99
3.03
9.09
ppm

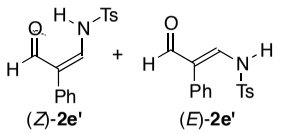

Operator: ymmc1
file: Mo-122-13_S_migration_oct_CDCl3_13C
Mercury-400MS "Varian-MNR"
98.69
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1115 repetitions
OBSERVE C13, 100.6910230 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
total time 58103 hr, 9 min, 20 sec
tBu
O
H
N
Ts
2d
144.395
140.676
137.067
129.973
126.760
98.728
77.315
76.993
76.678
42.863
26.552
21.560
220
200
180
160
140
120
100
80
60
40
20
0
ppm


2e
7.884
7.882
7.881
7.863
7.860
7.796
7.792
7.775
7.770
7.559
7.556
7.552
7.542
7.539
7.532
7.522
7.519
7.516
7.462
7.462
7.458
7.445
7.442
7.428
7.424
7.421
7.336
7.335
7.315
7.288
7.267
7.262
7.260
7.241
6.204
6.182
2.419
ppm

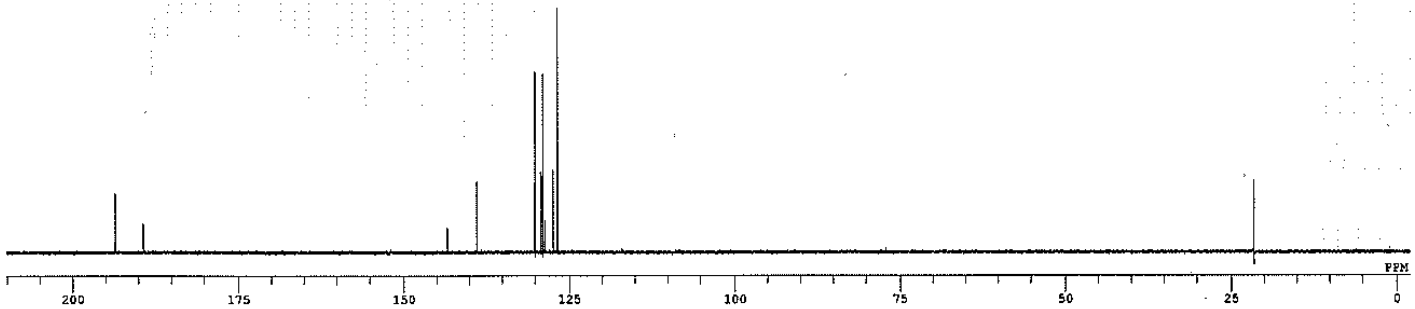

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmrl
File: product_RPA_13c
Mercury-400BB "varian-MERC"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
672 repetitions
OBSERVE C13, 100.6316309 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line Broadening 1.0 Hz
FT size 65536
Total time 1655790 hr, 30 min, -8 sec
Ph
O
H
N
Ts
2e
192.032
144.527
141.921
137.455
136.870
132.821
129.988
128.568
127.733
126.679
99.175
77.315
77.000
76.685
21.479
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmsd
File: No.332_72_Ph_migration_not_E_H
Mercury-400BB "Varian-INOVA"
11.67
11.67
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 12 sec
(Z)-2e'
(E)-2e'
9.680
9.671
9.417
7.798
7.793
7.789
7.777
7.773
7.756
7.444
7.427
7.423
7.411
7.406
7.402
7.388
7.382
7.369
7.363
7.360
7.355
7.354
7.341
7.334
7.320
7.302
7.296
7.292
7.287
7.279
7.275
7.260
7.126
7.122
7.105
7.102
2.465
2.438
1.10
1.19
0.51
3.45
13.58
1.00
1.64
3.58
ppm



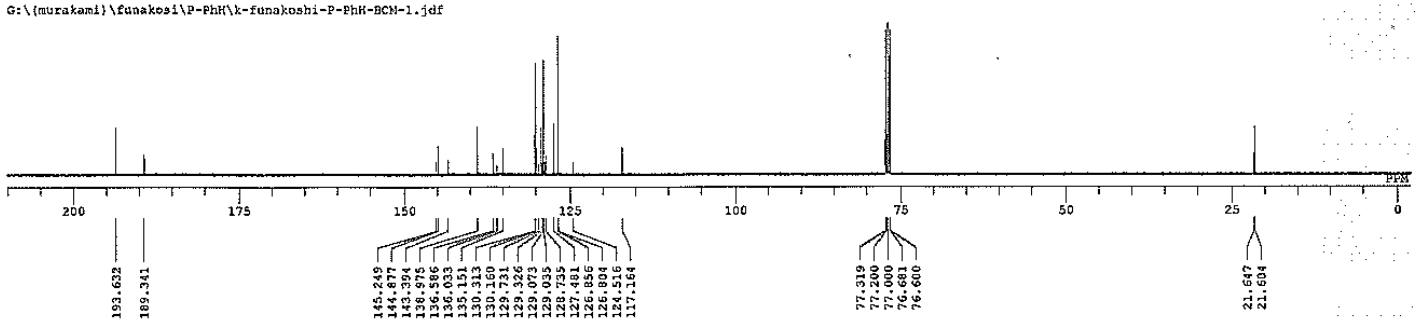


G:\(murakami)\funakosi\P-PhH\k-funakoshi-P-PhH-dept135-1.jdf

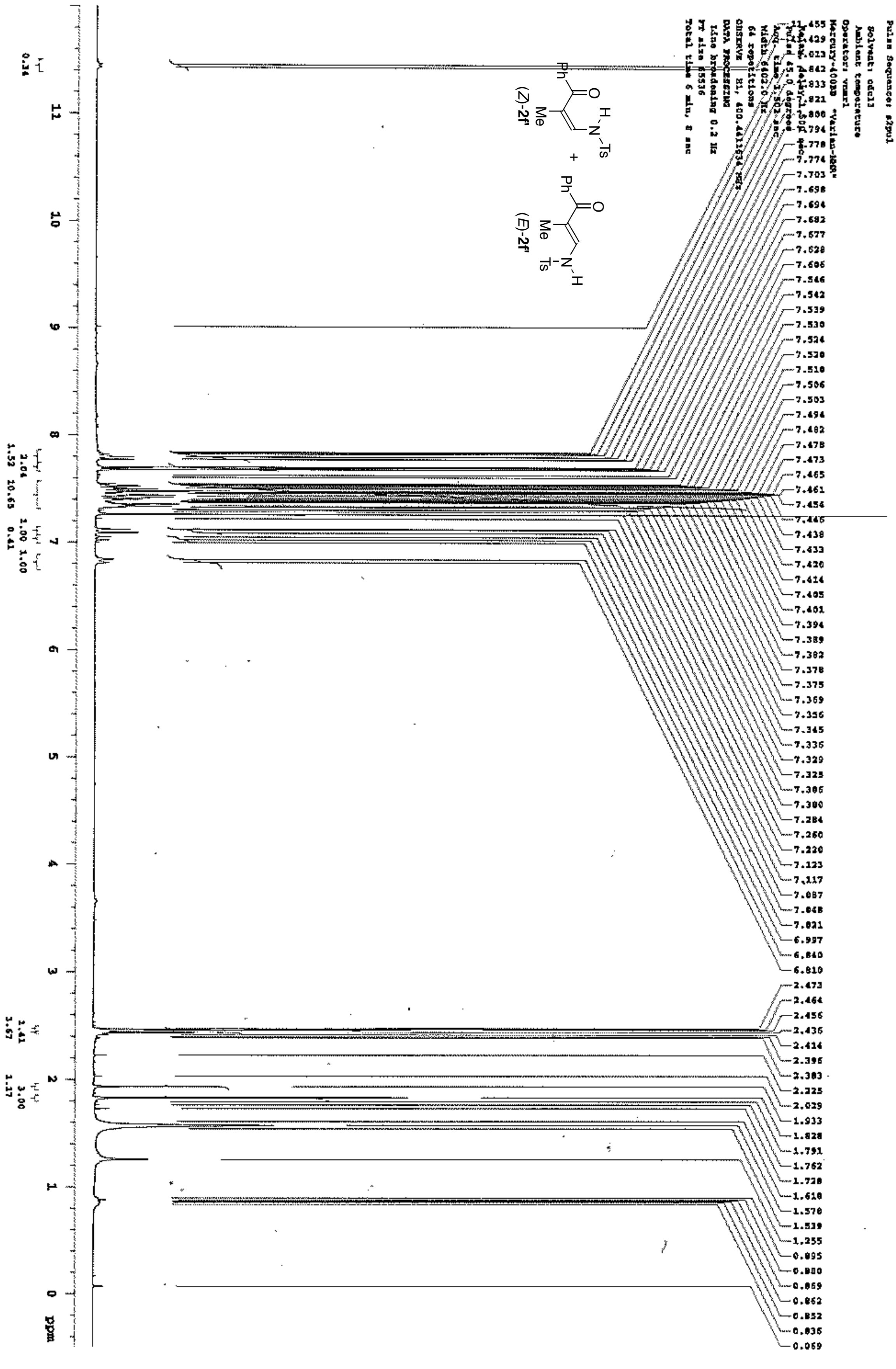


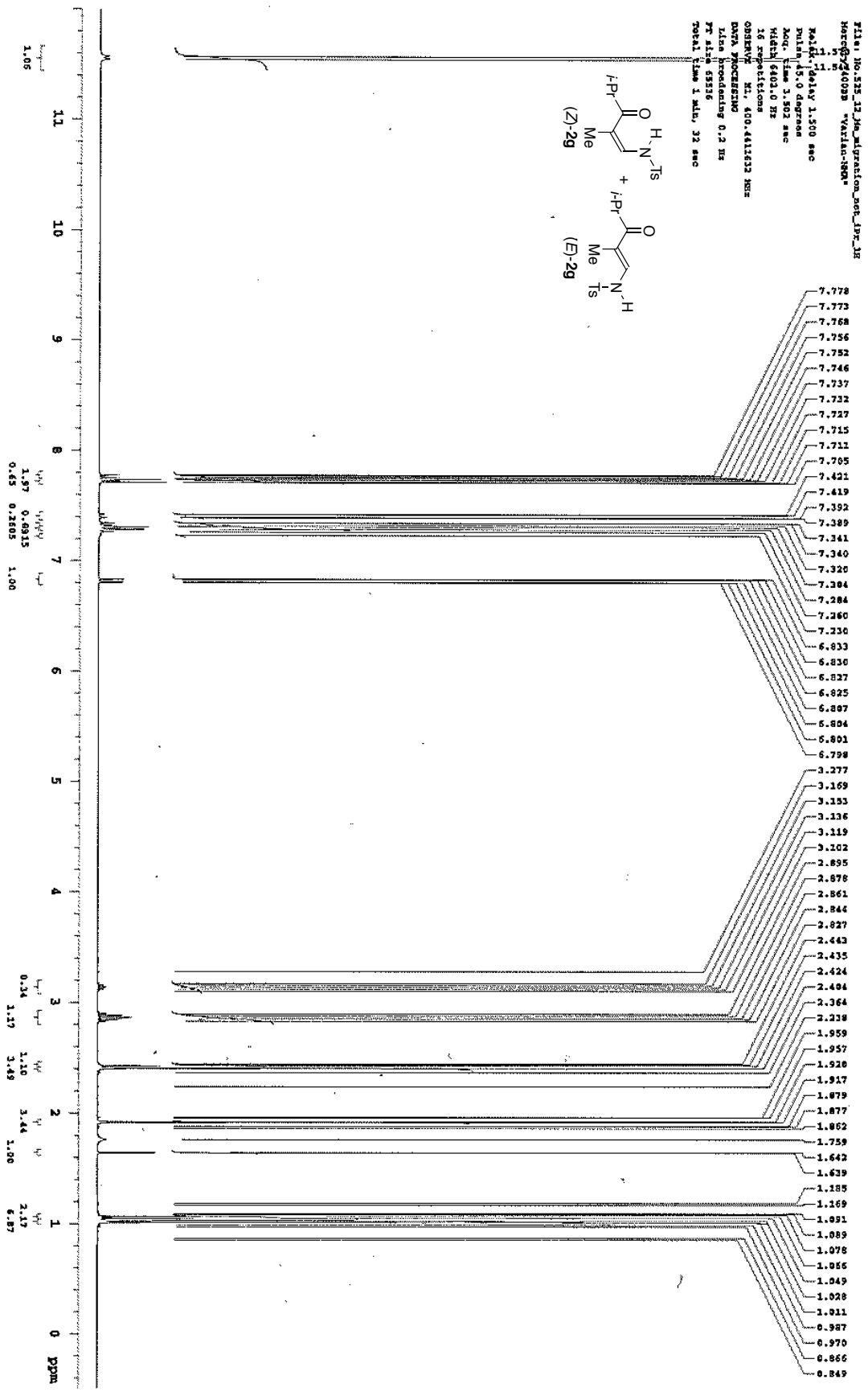

```
DFILE   k-funakoshi-P-PhH-dept135-1.jdf
COMNT   DEPT with decoupling
DATIM   01-08-2012 10:30:41
OBNUC   13C
EXMOD   dept.ex2
OBFRQ        100.53 MHz
OBFIN          5.86 Hz
POINT         65536
FREQU      31407.03 Hz
SCANS           216
ACQTM        1.0433 sec
PD           2.0000 sec
PW1             8.00 usec
IRNUC   1H
CTEMP          22.5 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.24 Hz
RGAIN            60
```

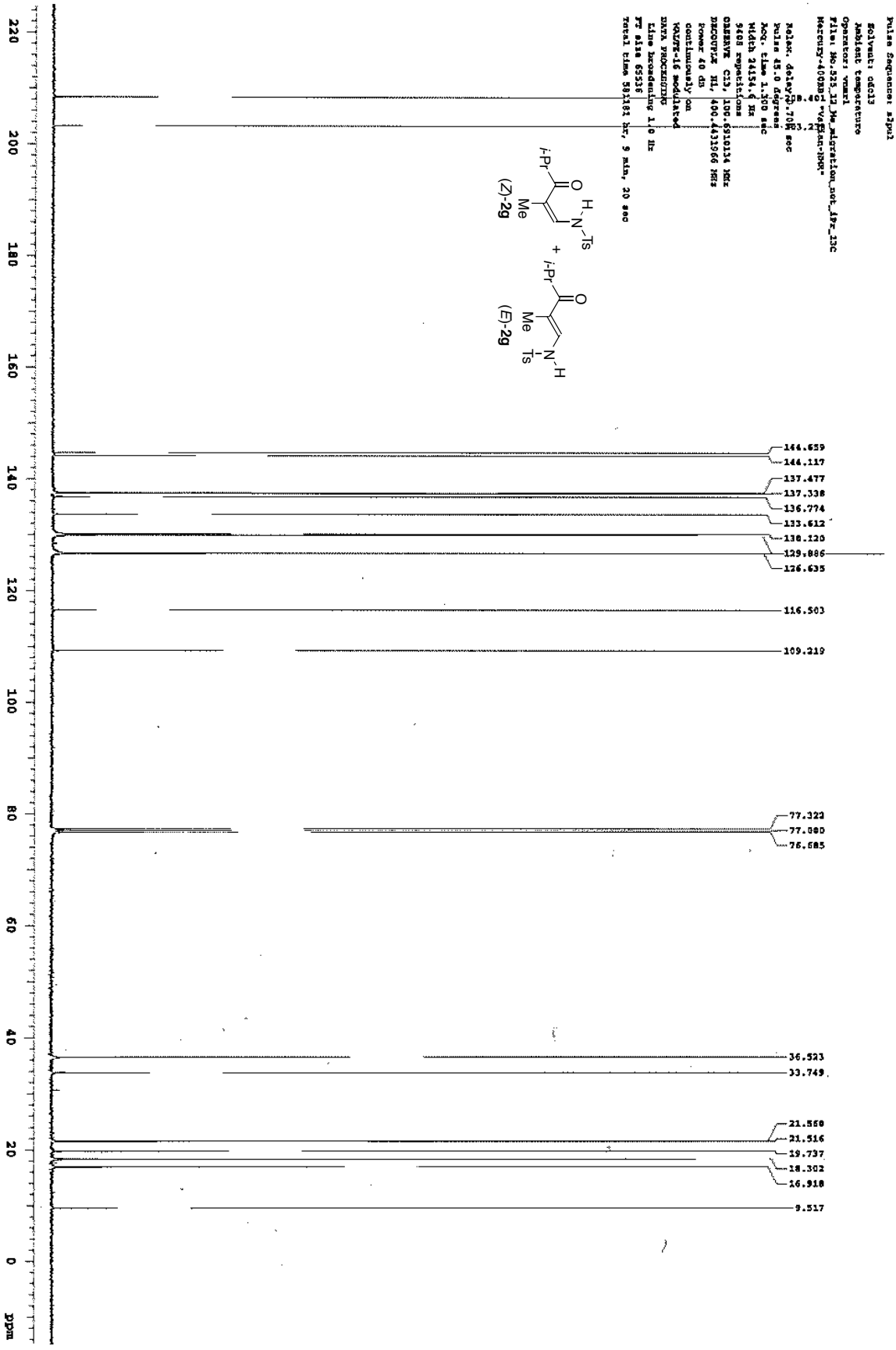
G:\(murakami)\funakosi\P-PhH\k-funakoshi-P-PhH-BCM-1.jdf

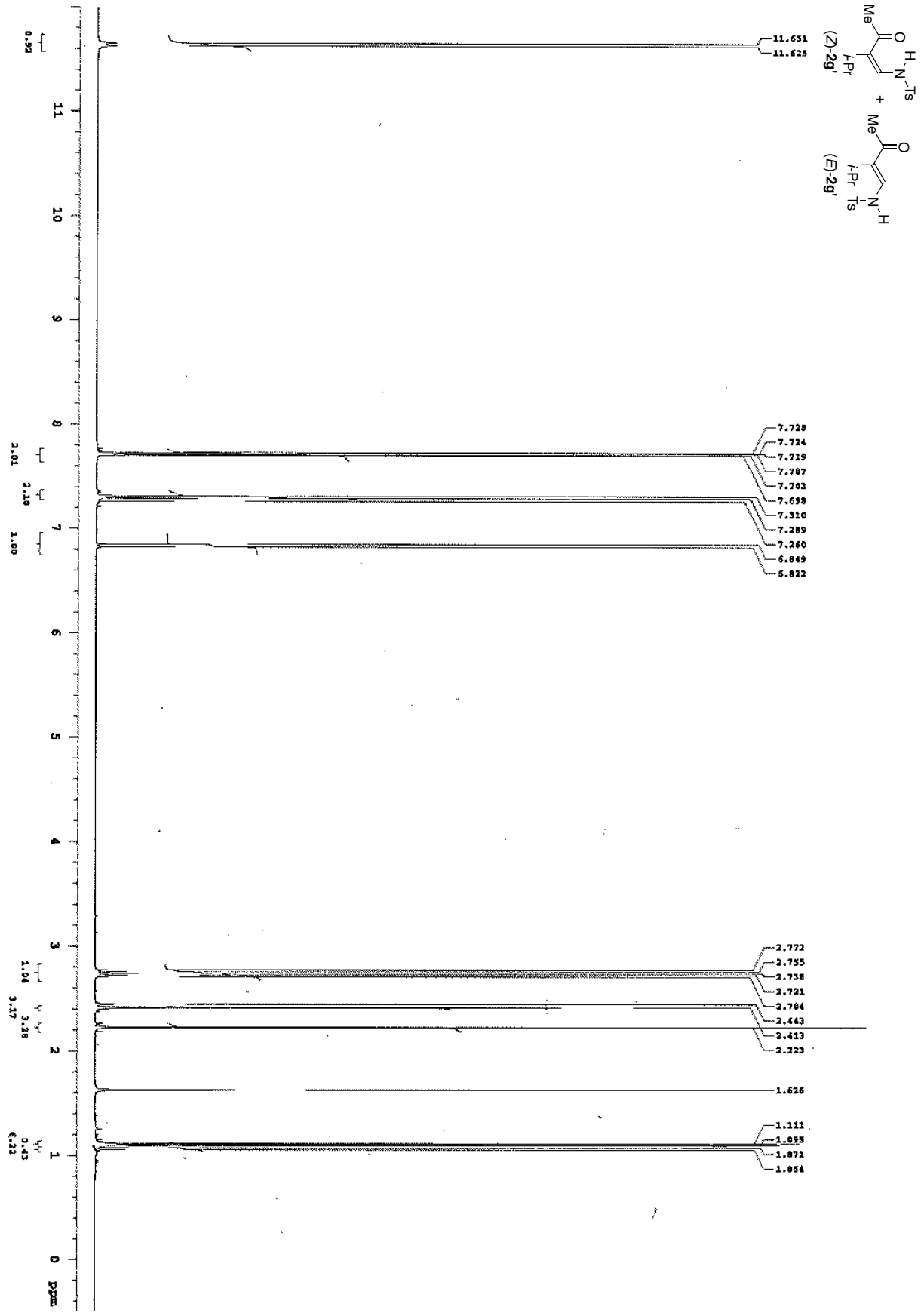


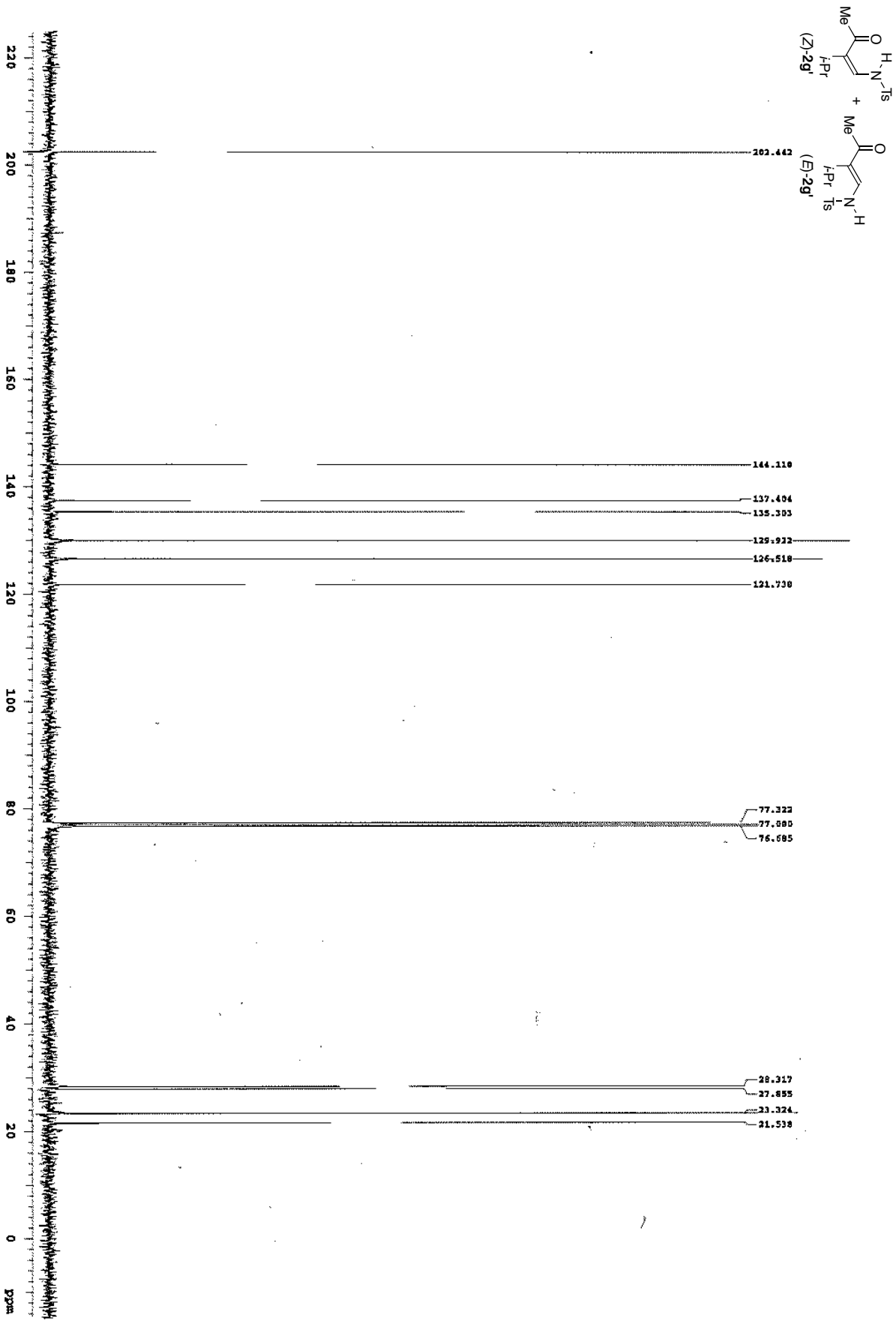

```
DFILE   k-funakoshi-P-PhH-BCM-1.jdf
COMNT   single pulse decoupled gated NOE
DATIM   01-08-2012 10:19:09
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ        100.53 MHz
OBFIN          5.86 Hz
POINT         65536
FREQU      31407.03 Hz
SCANS           526
ACQTM        1.0433 sec
PD           1.7000 sec
PW1             2.67 usec
IRNUC   1H
CTEMP          22.7 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.24 Hz
RGAIN            60
```

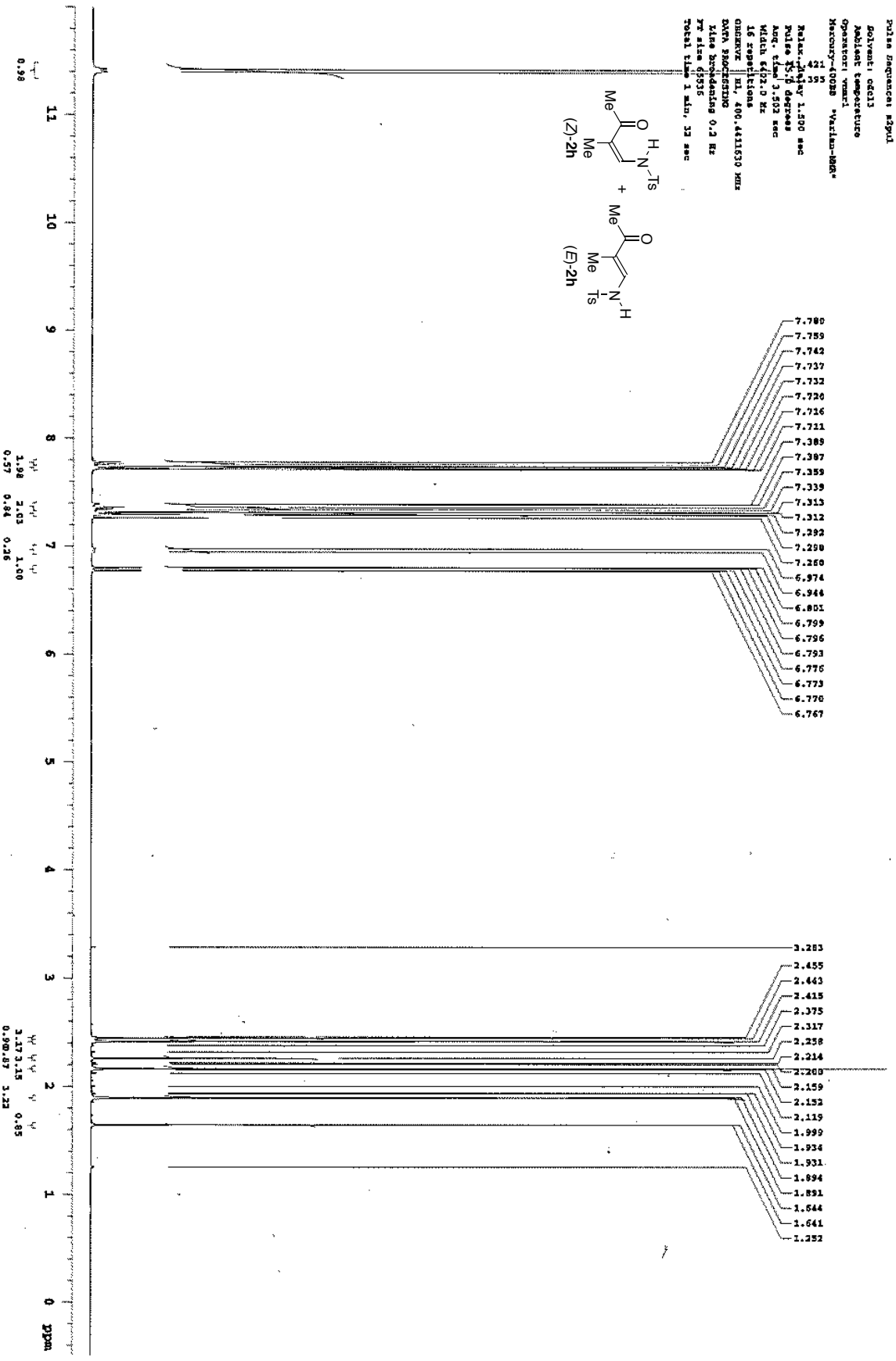

(Z)-2f'
(E)-2f'
ppm

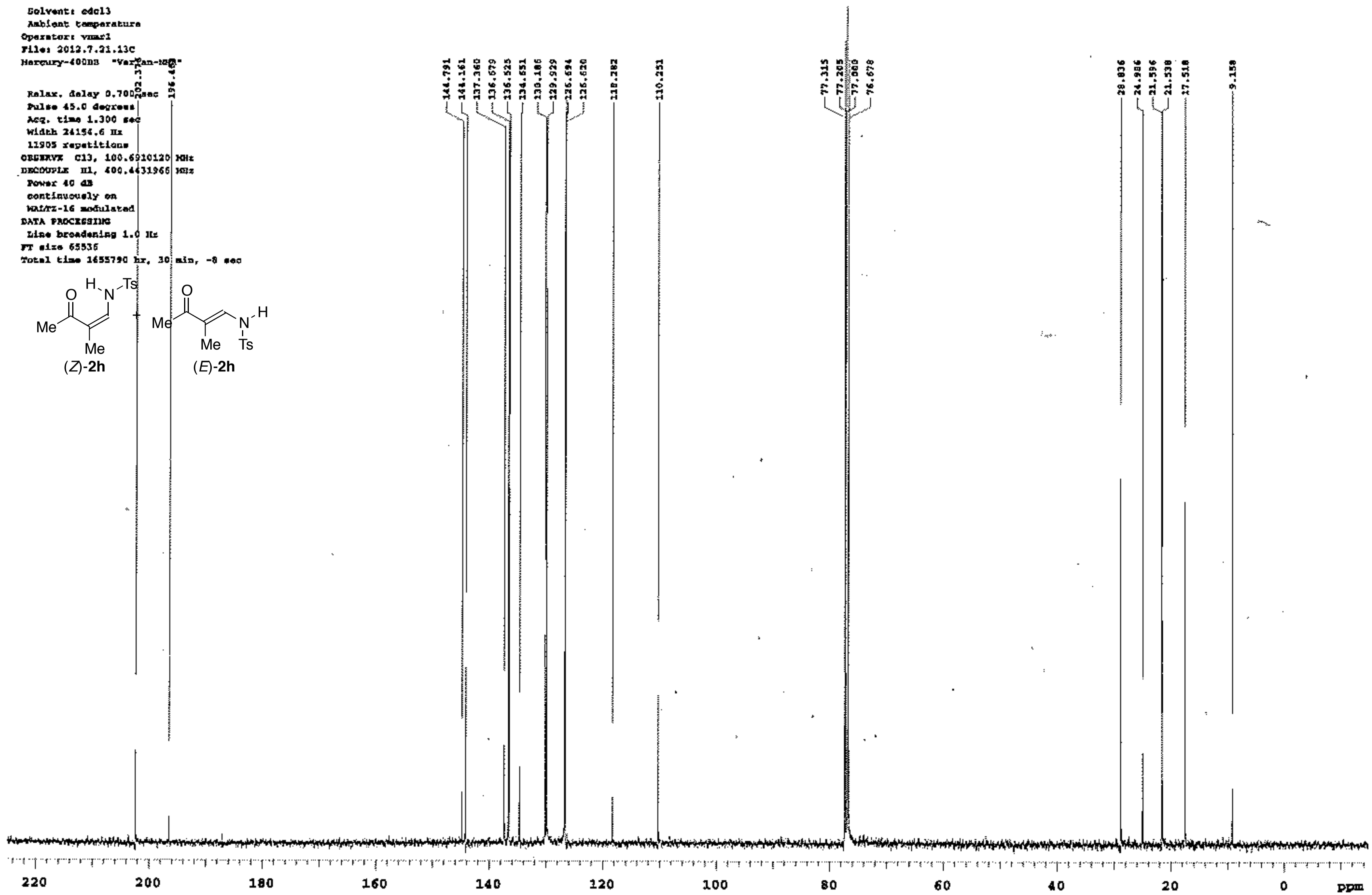

(Z)-2g
(E)-2g
7.778
7.773
7.768
7.756
7.752
7.746
7.737
7.732
7.727
7.715
7.711
7.705
7.421
7.419
7.392
7.389
7.341
7.340
7.320
7.304
7.284
7.260
7.230
6.833
6.830
6.827
6.825
6.807
6.804
6.801
6.798
3.277
3.169
3.153
3.136
3.119
3.102
2.895
2.878
2.861
2.844
2.827
2.443
2.435
2.424
2.404
2.364
2.238
1.959
1.957
1.920
1.917
1.879
1.877
1.862
1.759
1.642
1.639
1.185
1.169
1.091
1.089
1.078
1.056
1.049
1.028
1.011
0.987
0.970
0.866
0.849
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
File: Mo_323_33_jw_migration_not_4hz_13c
Mercury-400BB "Varian-NMR"
8.42 7.26 3.22
Relax. delay 5.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.4 Hz
9408 repetitions
OBSERVE C13, 100.6310134 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 58181 hr, 9 min, 20 sec
(Z)-2g
(E)-2g
144.659
144.117
137.477
137.338
136.774
133.612
130.120
129.886
126.635
116.503
109.219
77.322
77.000
76.685
36.523
33.749
21.550
21.516
19.737
18.302
16.918
9.517
220
200
180
160
140
120
100
80
60
40
20
0
ppm

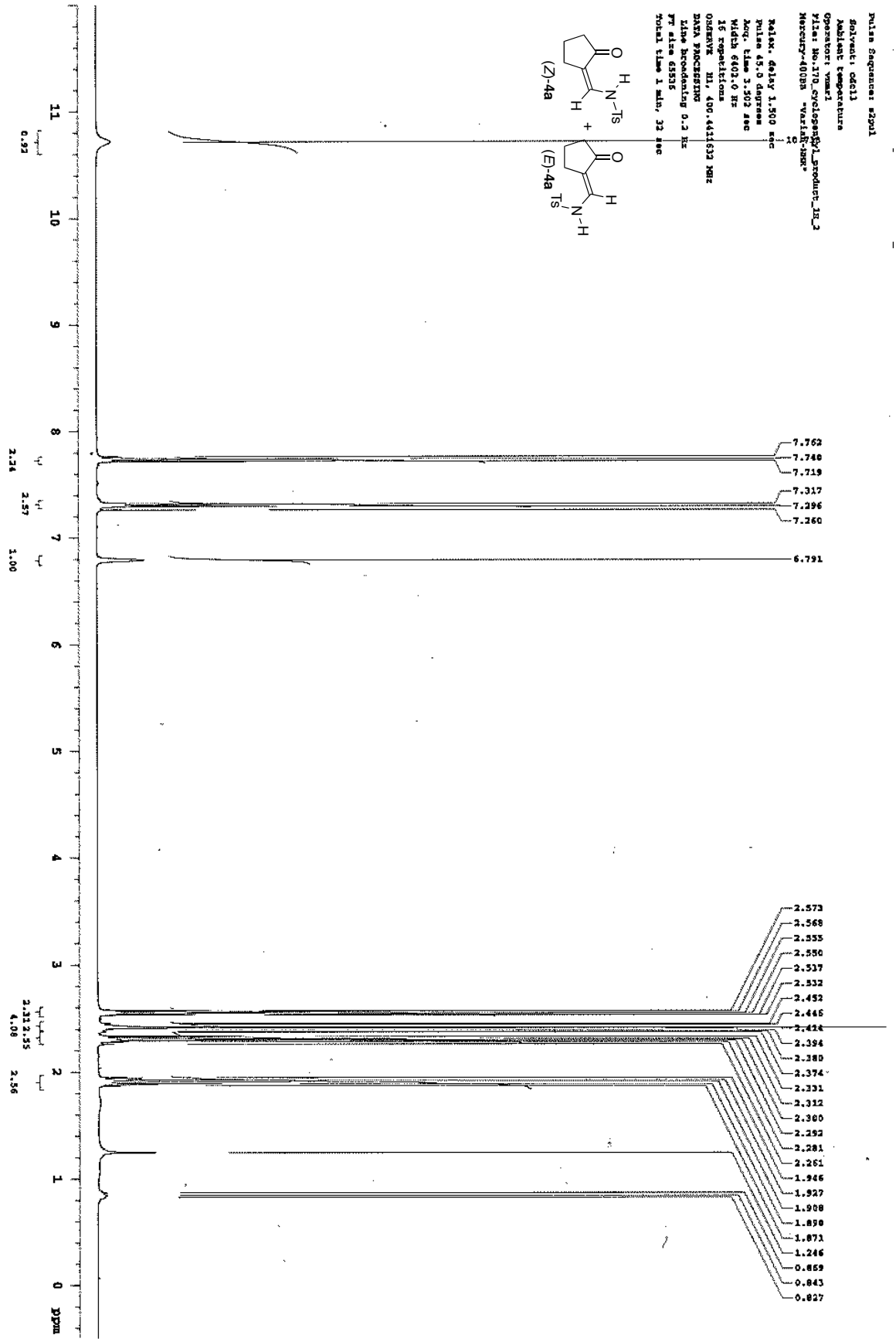

11.651
11.625
7.728
7.724
7.719
7.707
7.703
7.698
7.310
7.289
7.260
6.849
6.822
2.772
2.755
2.738
2.721
2.704
2.443
2.413
2.223
1.626
1.111
1.095
1.071
1.054
0.92
2.01
2.10
1.00
1.04
3.17
3.28
0.43
6.22
ppm
(Z)-2g'
(E)-2g'

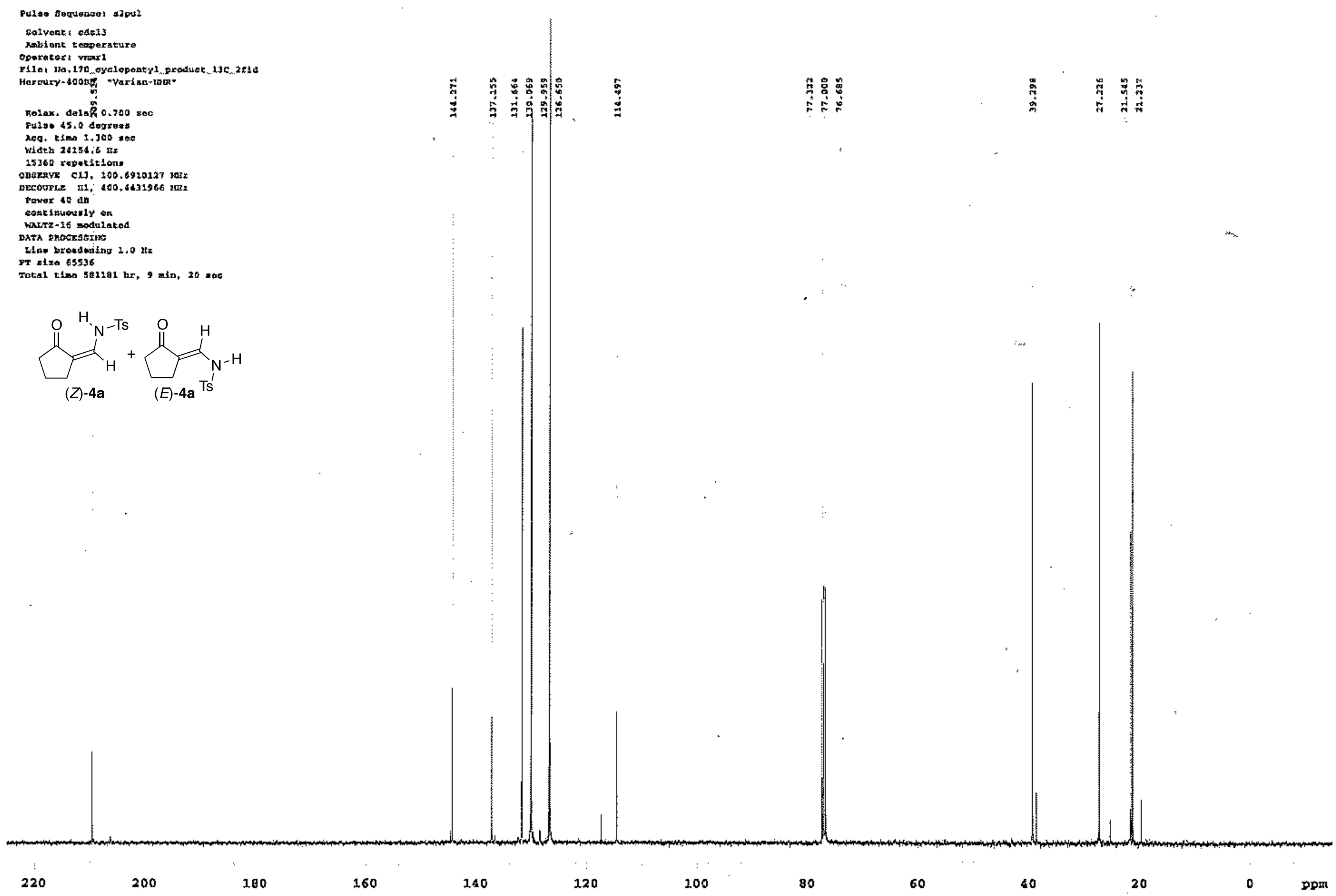

(Z)-2g'
+
(E)-2g'
202.442
144.110
137.404
135.303
129.922
126.518
121.730
77.322
77.080
76.685
28.317
27.855
23.324
21.538
ppm

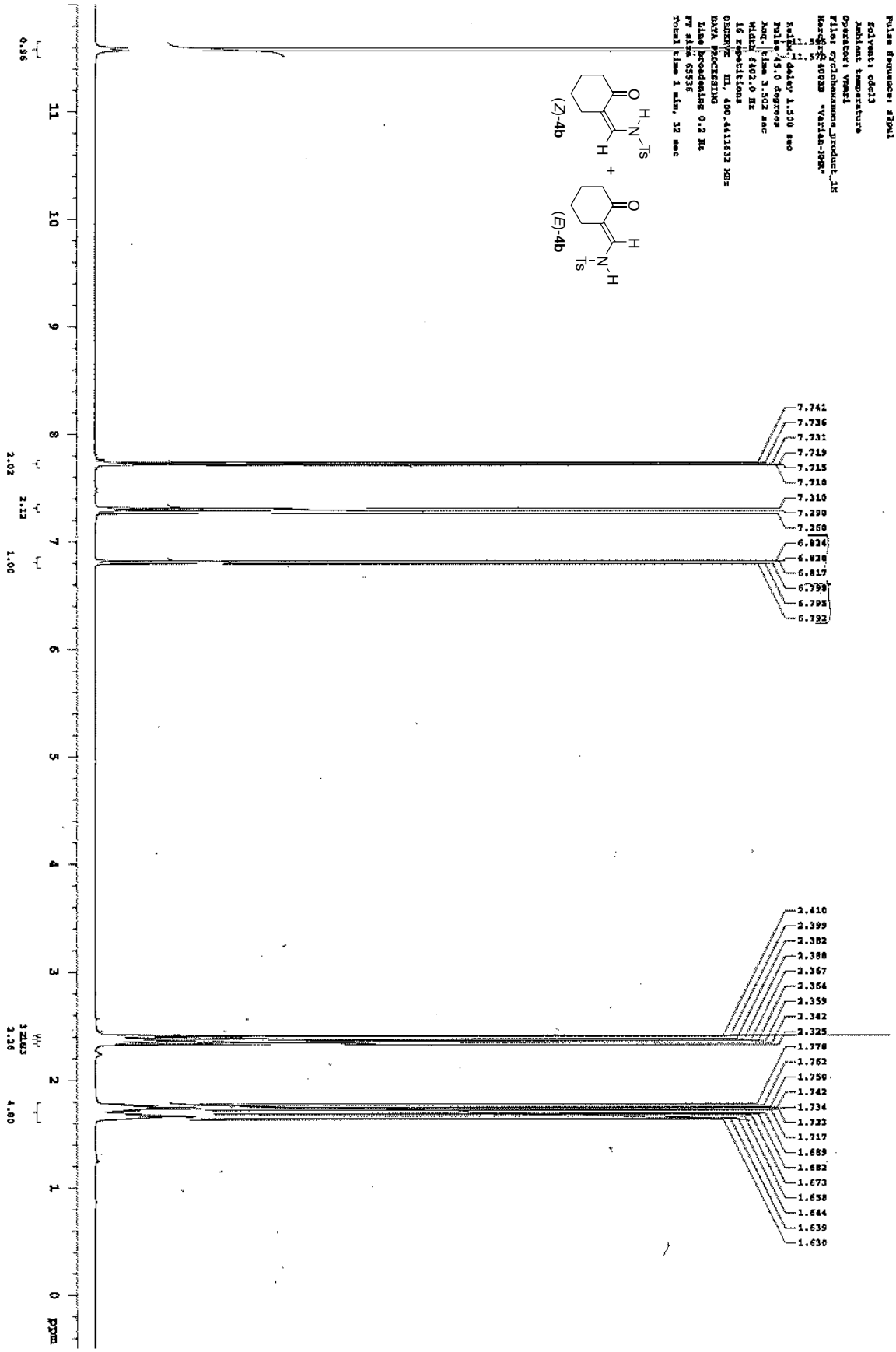

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmarl
Mercury-600BB "Varian-MMR"
421
395
Relax. delay 1.500 sec
Pulse 75.0 degrees
Acq. time 3.552 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 33 sec
(Z)-2h
(E)-2h
7.789
7.759
7.742
7.737
7.732
7.720
7.716
7.711
7.389
7.387
7.359
7.339
7.313
7.312
7.292
7.290
7.260
6.974
6.946
6.801
6.799
6.796
6.793
6.776
6.773
6.770
6.767
3.283
2.455
2.443
2.415
2.375
2.317
2.258
2.214
2.200
2.159
2.152
2.119
1.999
1.934
1.931
1.894
1.891
1.644
1.641
1.252
0.98
1.98
2.03
1.00
0.57
0.84
0.26
3.17
3.15
0.85
0.90
0.87
3.23
ppm

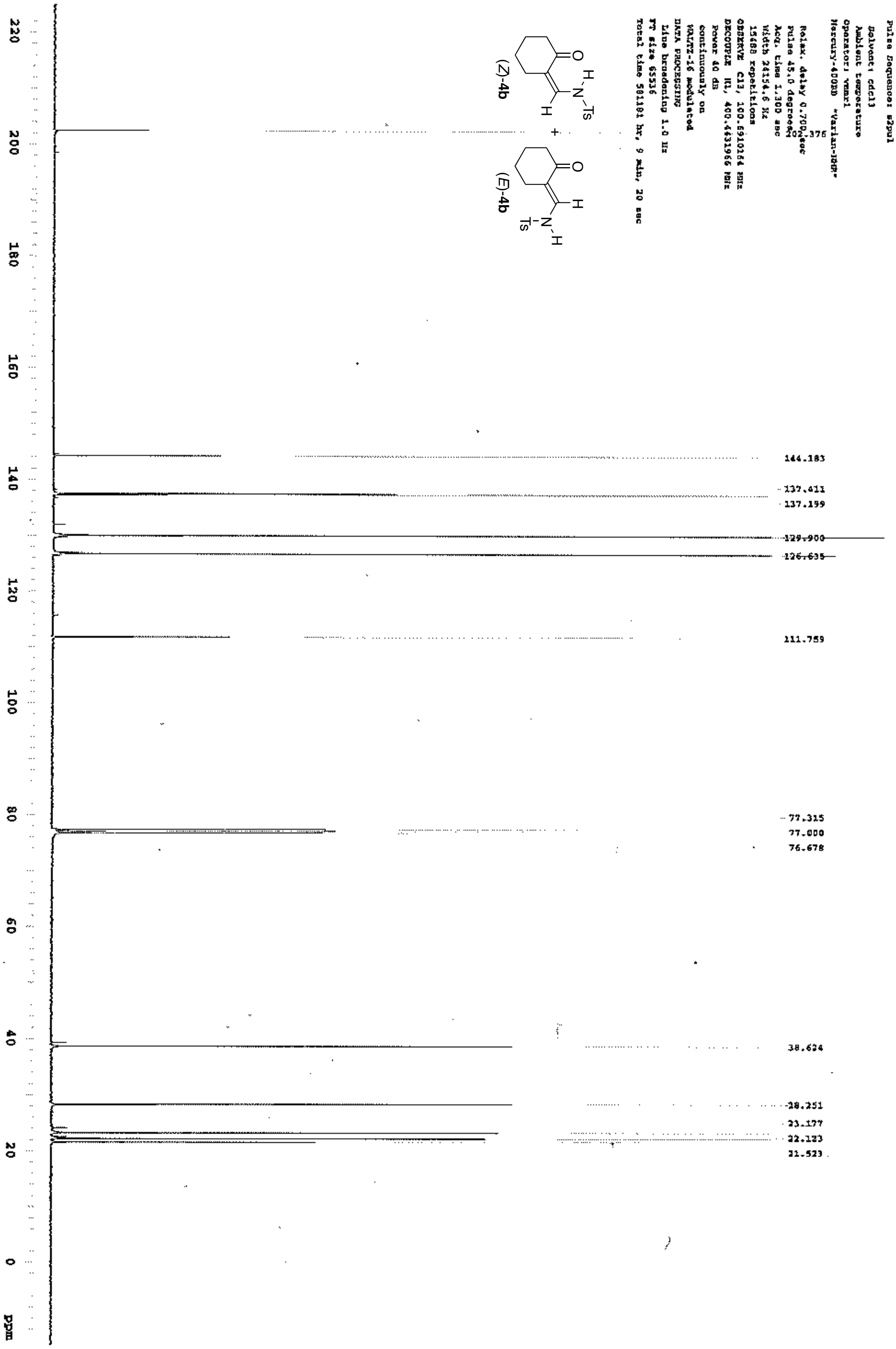


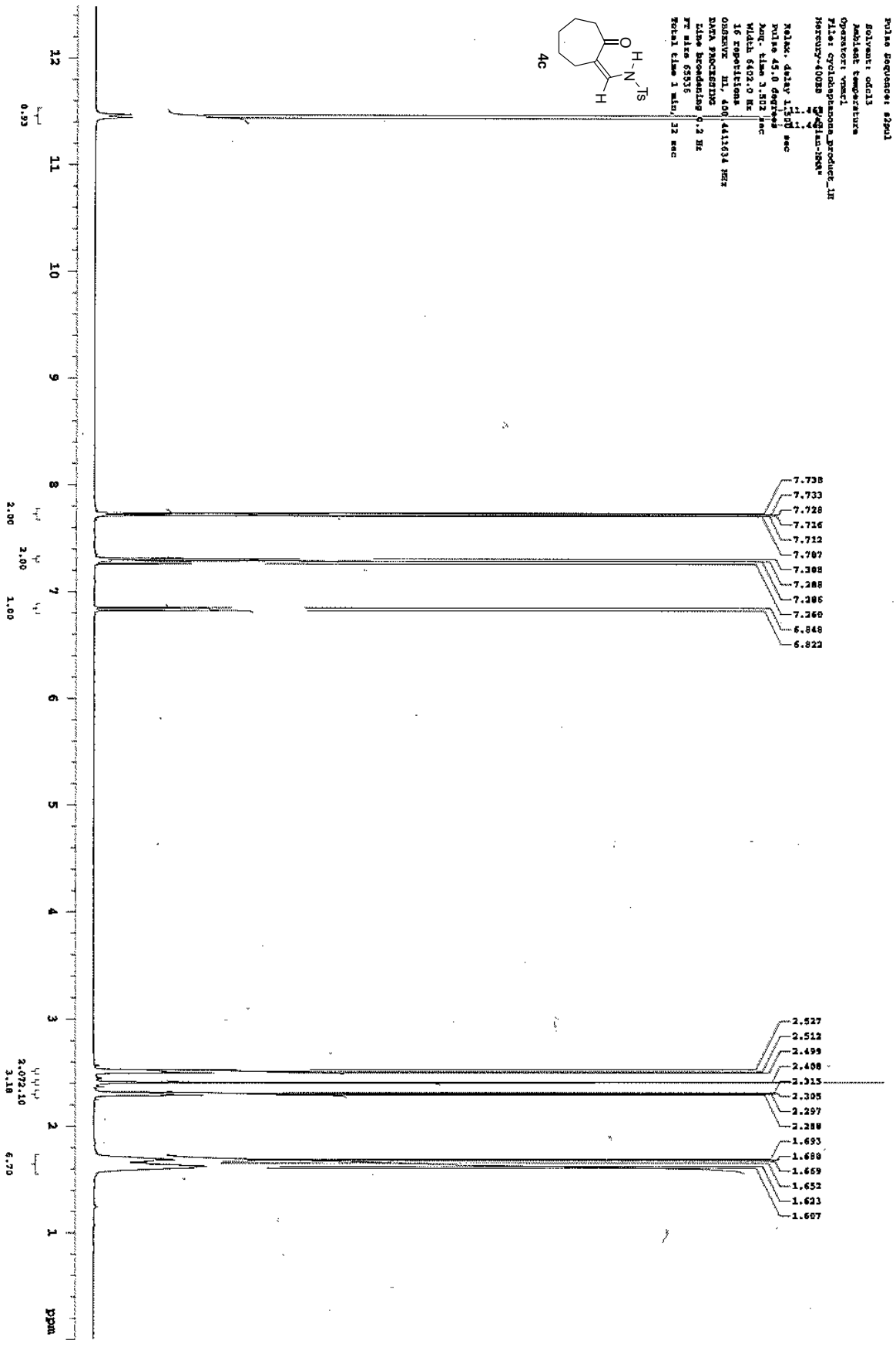
Solvent: cdcl3
Ambient temperature
Operator: vmarl
File: 2012.7.21.13C
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
11905 repetitions
OBSERVE C13, 100.6910120 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1655790 hr, 30 min, -8 sec
(Z)-2h
(E)-2h
202.37
196.46
144.791
144.161
137.360
136.679
136.525
134.651
130.186
129.929
125.694
125.620
118.282
110.251
77.315
77.205
77.000
76.678
28.836
24.986
21.596
21.538
17.518
9.158
220 200 180 160 140 120 100 80 60 40 20 0 ppm

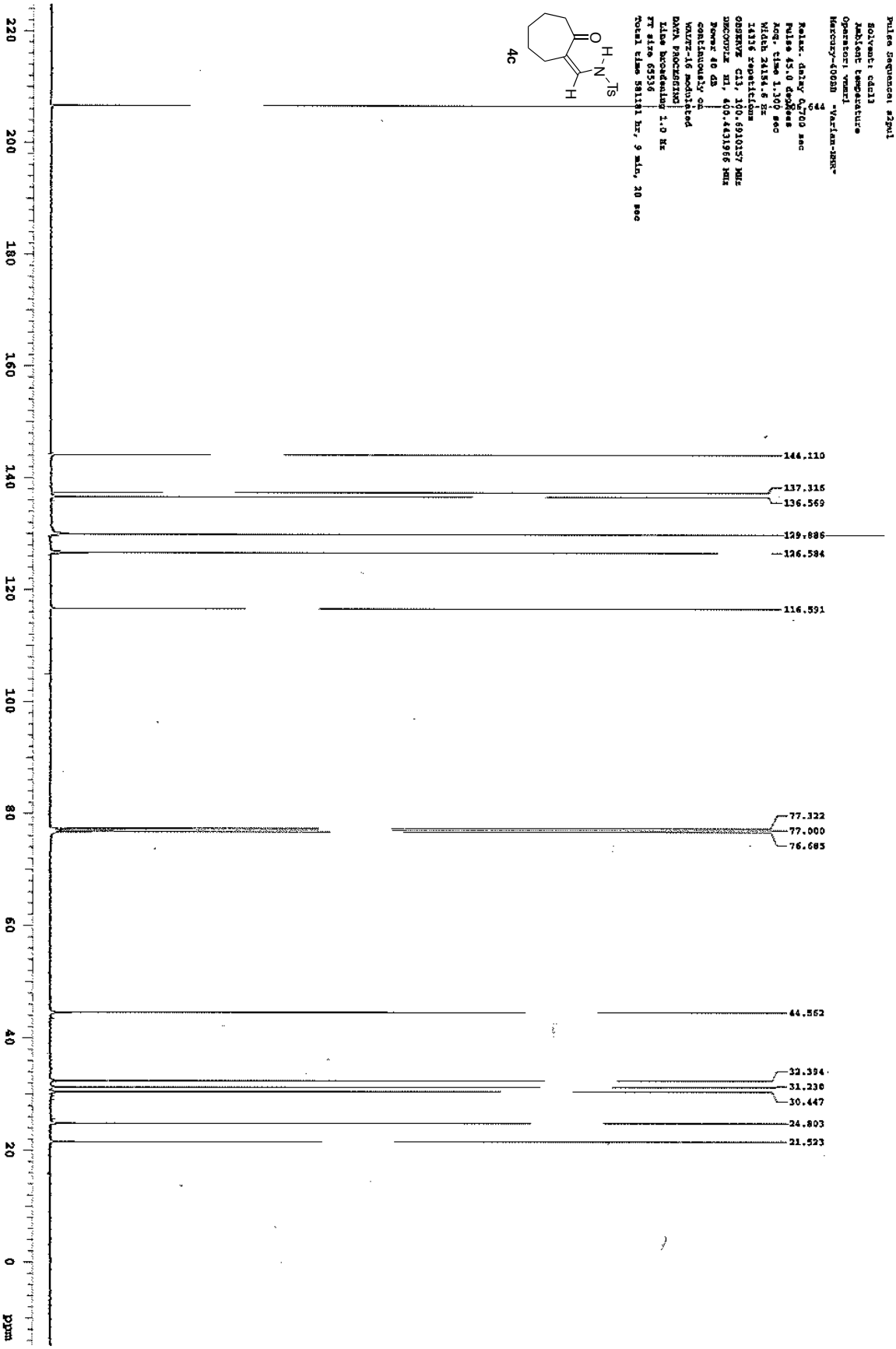

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmar1
File: No.170_cyclopentyl_product_1H_2
Mercury-400BB "varian-nmr"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411633 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
(Z)-4a
(E)-4a
7.762
7.740
7.719
7.317
7.296
7.260
6.791
2.573
2.568
2.555
2.550
2.537
2.532
2.452
2.445
2.414
2.394
2.389
2.374
2.331
2.312
2.300
2.292
2.281
2.261
1.946
1.927
1.908
1.850
1.871
1.246
0.859
0.843
0.827
0.92
2.24
2.57
1.00
2.11
2.55
4.08
2.56
ppm

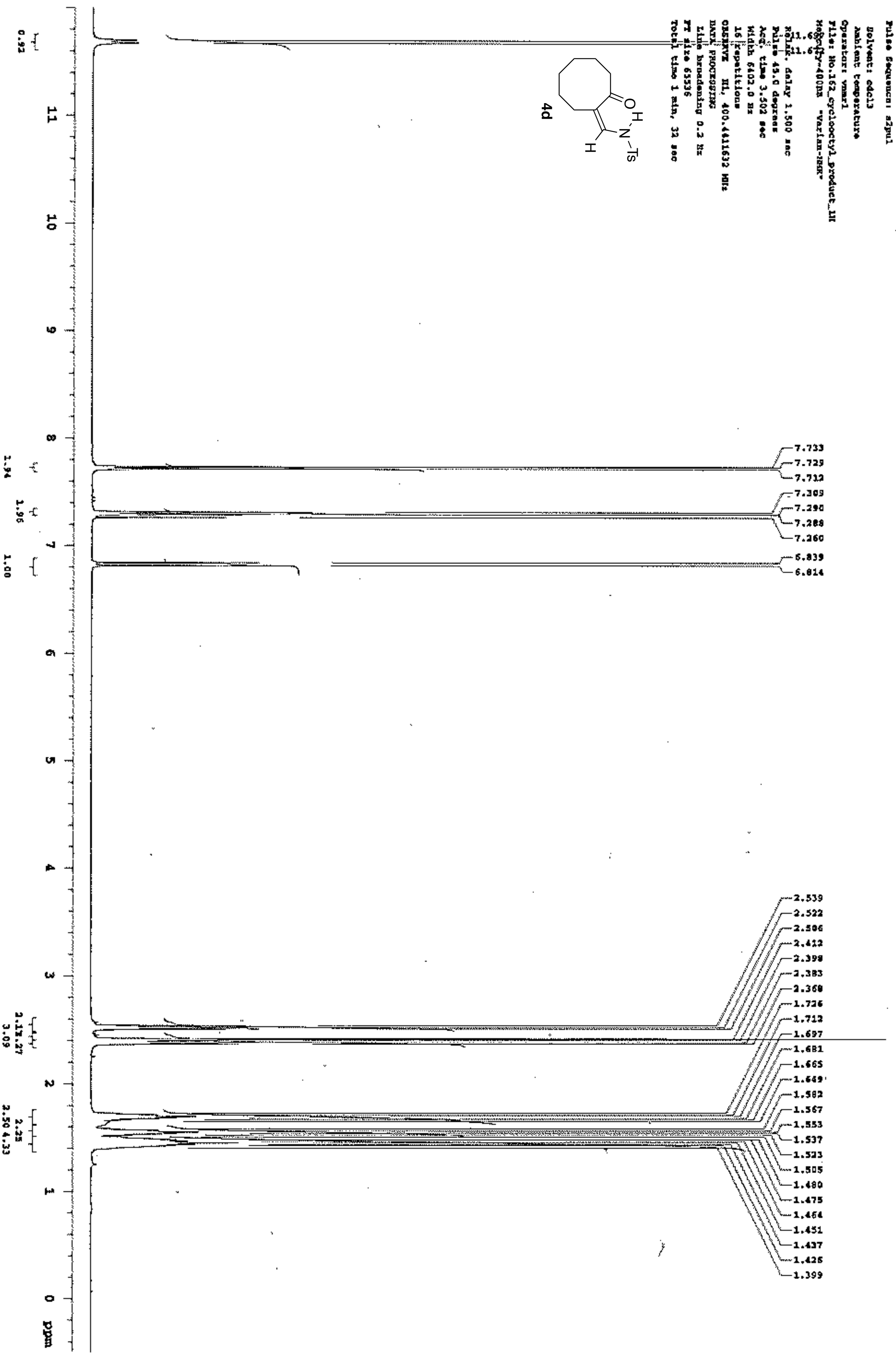

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: No.170_cyclopentyl_product_13C_2fid
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
15360 repetitions
OBSERVE C13, 100.6910127 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581181 hr, 9 min, 20 sec
(Z)-4a
(E)-4a
144.271
137.155
131.664
130.069
129.959
126.650
114.497
77.332
77.000
76.685
39.298
27.226
21.545
21.237
220
200
180
160
140
120
100
80
60
40
20
0
ppm

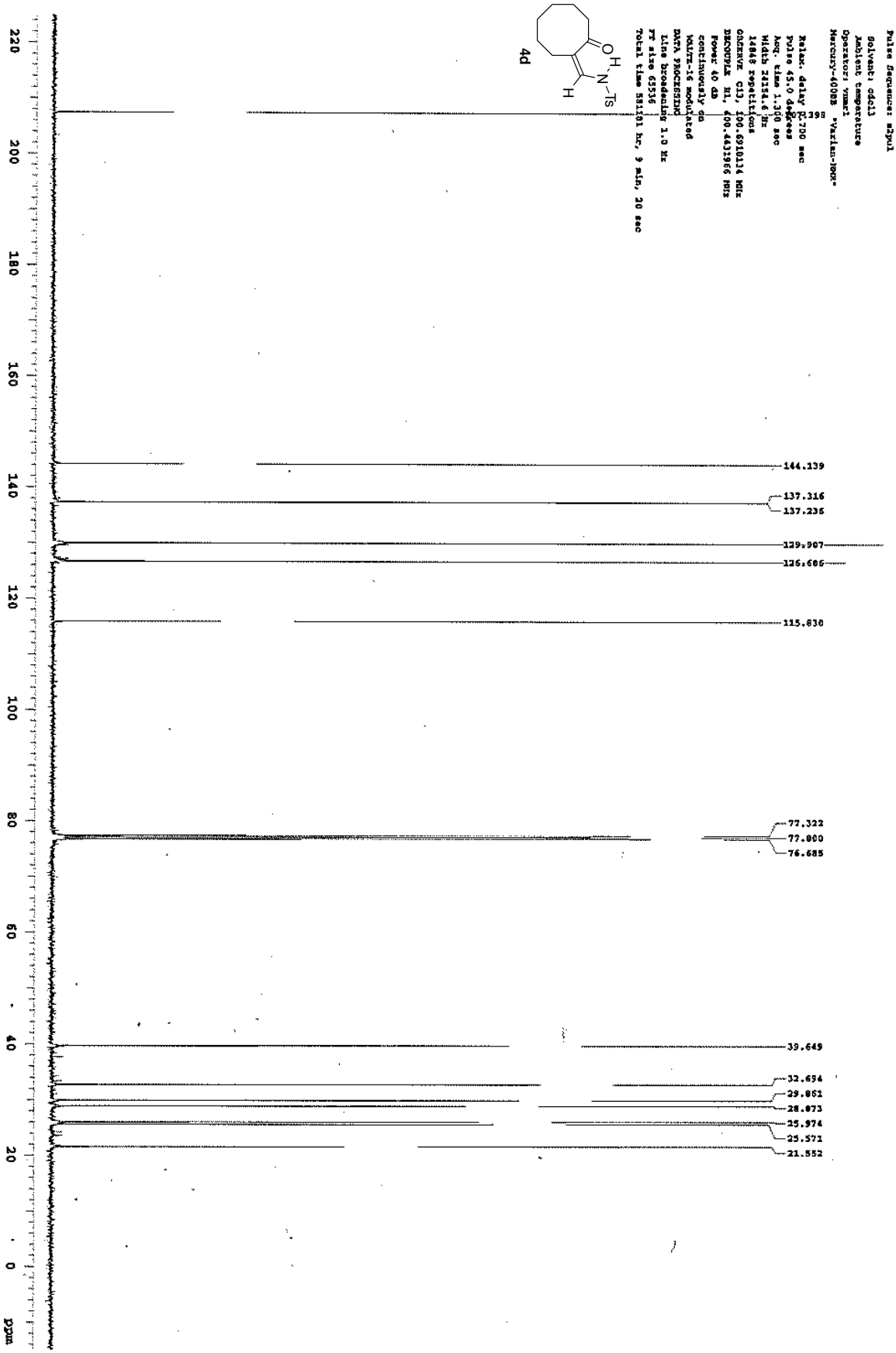

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: cyclohexanone_product_1H
Mercury-400BB "Varian-NMR"
11.5s
11.57
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.562 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
(Z)-4b
(E)-4b
7.741
7.736
7.731
7.719
7.715
7.710
7.310
7.290
7.260
6.820
6.820
6.817
6.798
6.795
6.792
2.410
2.399
2.382
2.380
2.367
2.364
2.359
2.342
2.325
1.778
1.762
1.750
1.742
1.734
1.723
1.717
1.689
1.682
1.673
1.658
1.644
1.639
1.630
0.96
2.02
2.23
1.00
3.26
4.80
ppm

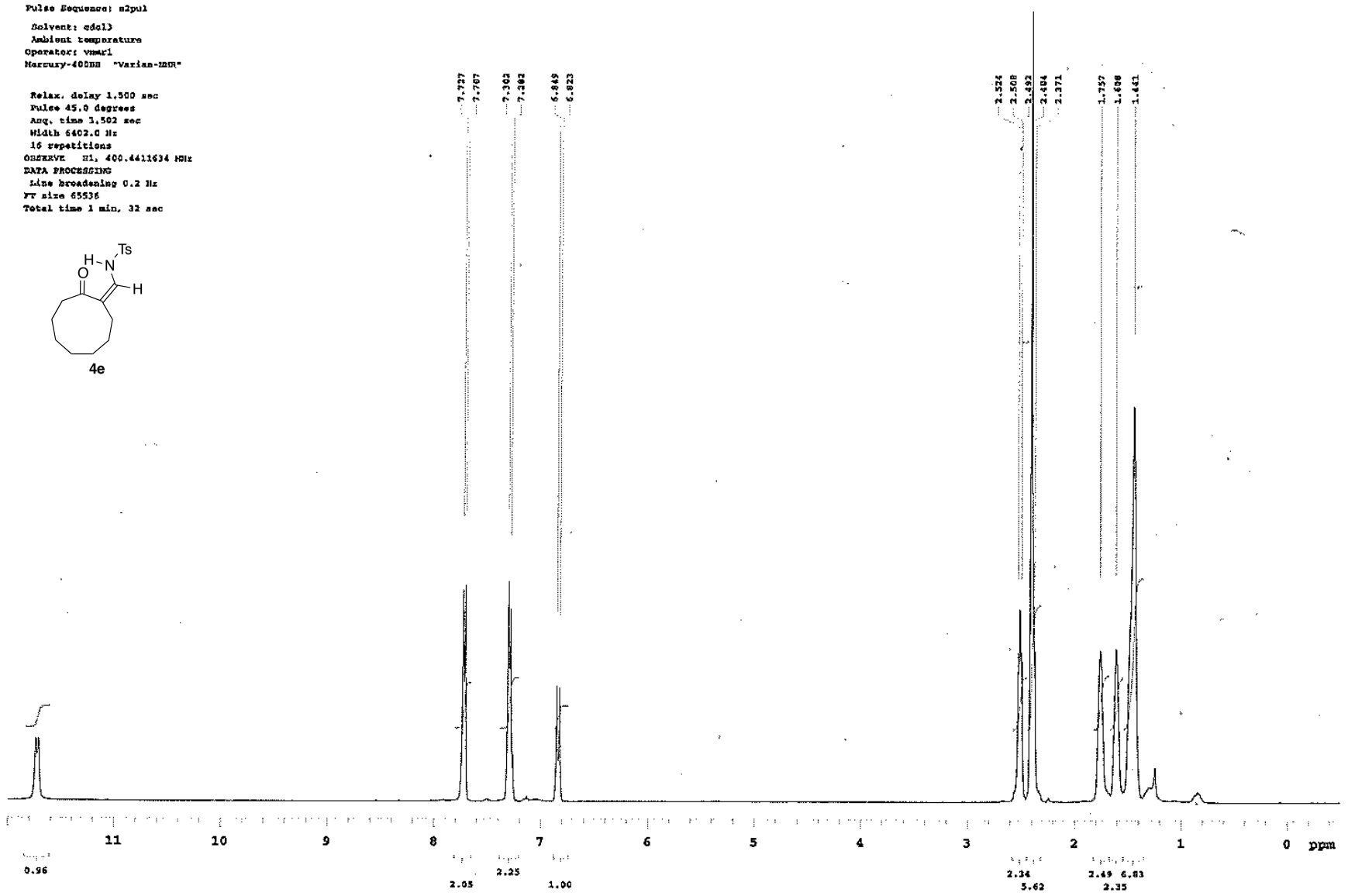

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-NMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
15488 repetitions
OBSERVE C13, 100.6910164 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581181 hr, 9 min, 20 sec
(Z)-4b
(E)-4b
Ts
202.376
144.183
137.411
137.199
129.900
126.635
111.759
77.315
77.000
76.678
38.624
28.251
23.177
22.123
21.523
220
200
180
160
140
120
100
80
60
40
20
0
ppm

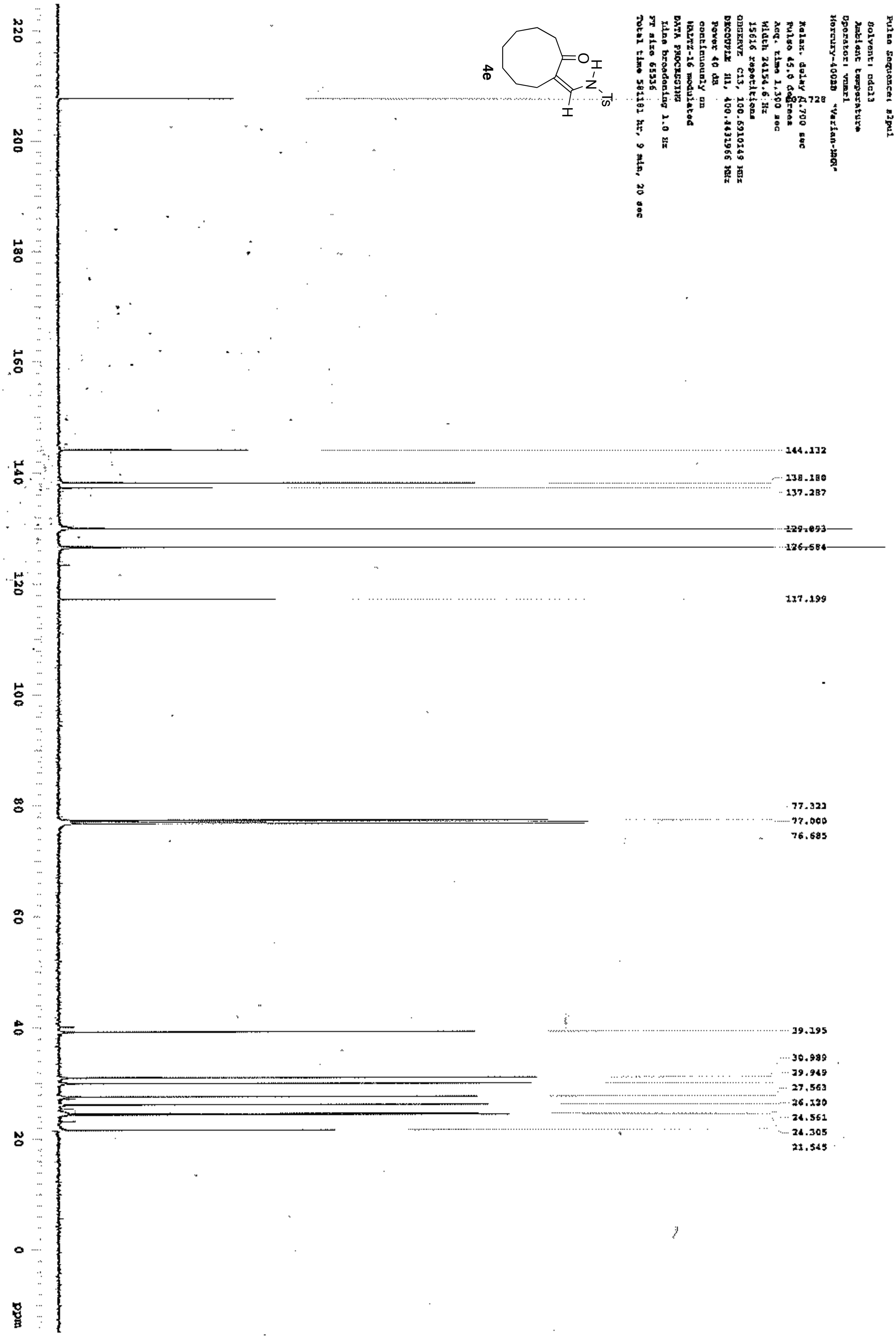

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
File: cyclohexanone_product_1H
Mercury-400BB
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411634 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
4c
7.738
7.733
7.728
7.716
7.712
7.707
7.308
7.288
7.286
7.269
6.848
6.822
2.527
2.512
2.499
2.488
2.315
2.305
2.297
2.288
1.693
1.680
1.669
1.652
1.623
1.607
0.93
2.00
2.00
1.00
2.072.10
3.10
6.70
ppm

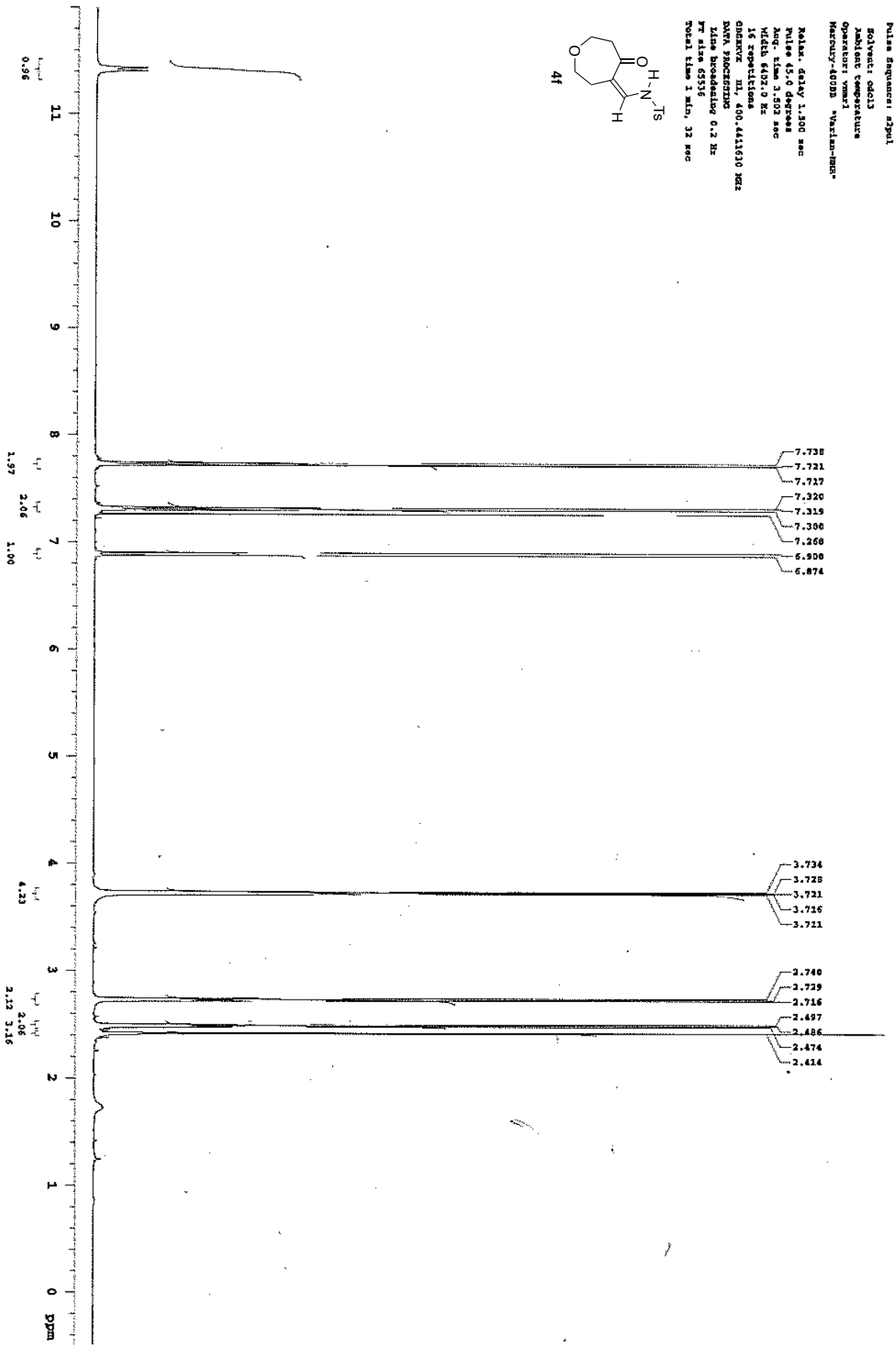

Pulse Sequence s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-MER"
644
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
14336 repetitions
OBSERVE C13, 100.6910157 MHz
DECOUPLE H1, 400.4431986 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line Broadening 1.0 Hz
FT size 65536
Total time 581181 hr, 9 min, 20 sec
4c
144.110
137.316
136.569
129.886
126.584
116.591
77.322
77.000
76.685
44.562
32.394
31.236
30.447
24.803
21.523
220
200
180
160
140
120
100
80
60
40
20
0
ppm

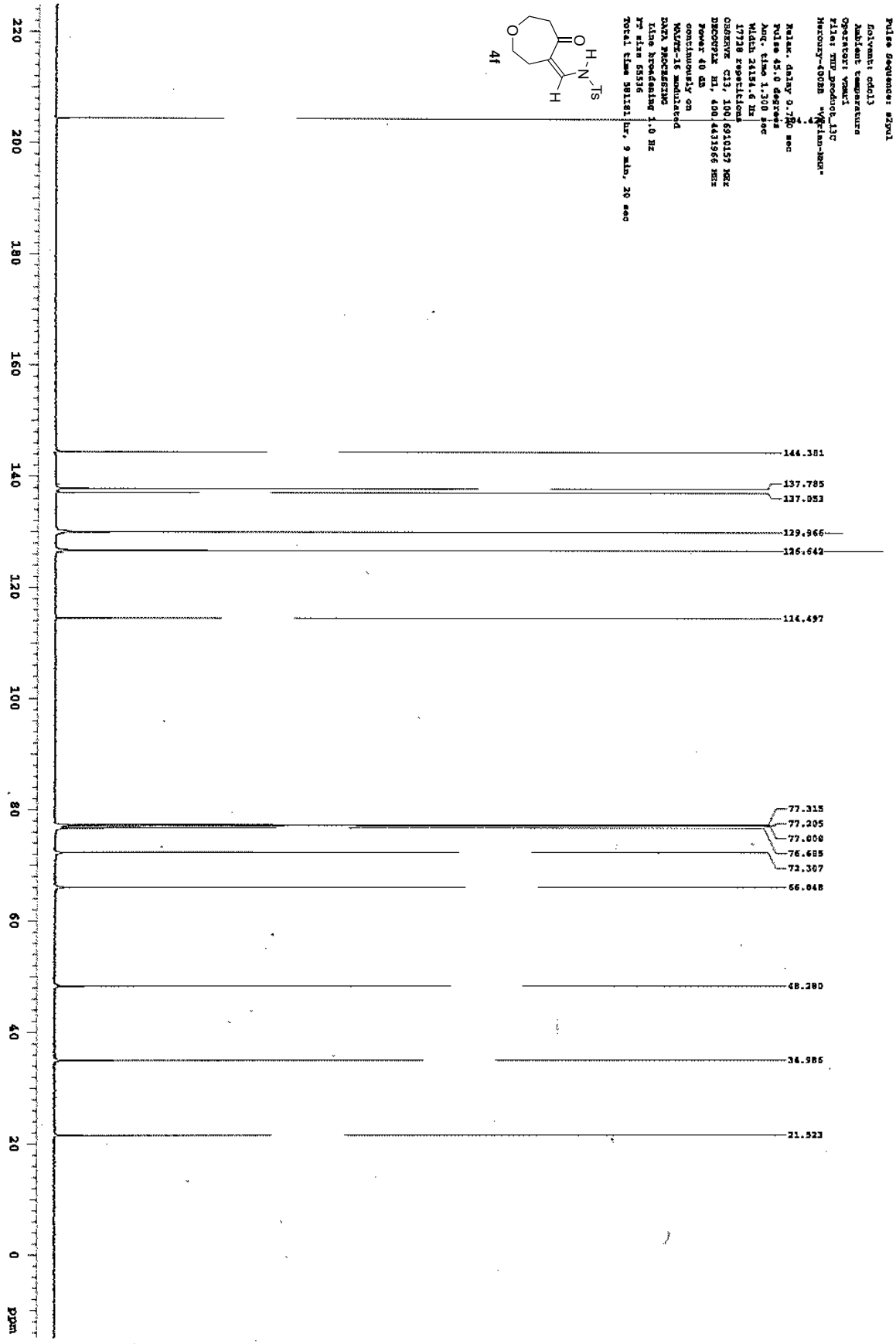

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: No.162_cyclooctyl_product_1H
Mercury-400BB "Varian-NMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
4d
Ts
H
O
N
7.733
7.729
7.712
7.309
7.290
7.288
7.260
6.839
6.814
2.539
2.522
2.506
2.412
2.398
2.383
2.368
1.726
1.712
1.697
1.681
1.665
1.649
1.582
1.567
1.553
1.537
1.523
1.505
1.480
1.475
1.464
1.451
1.437
1.426
1.399
0.92
1.94
1.96
1.00
3.09
2.25
4.33
ppm

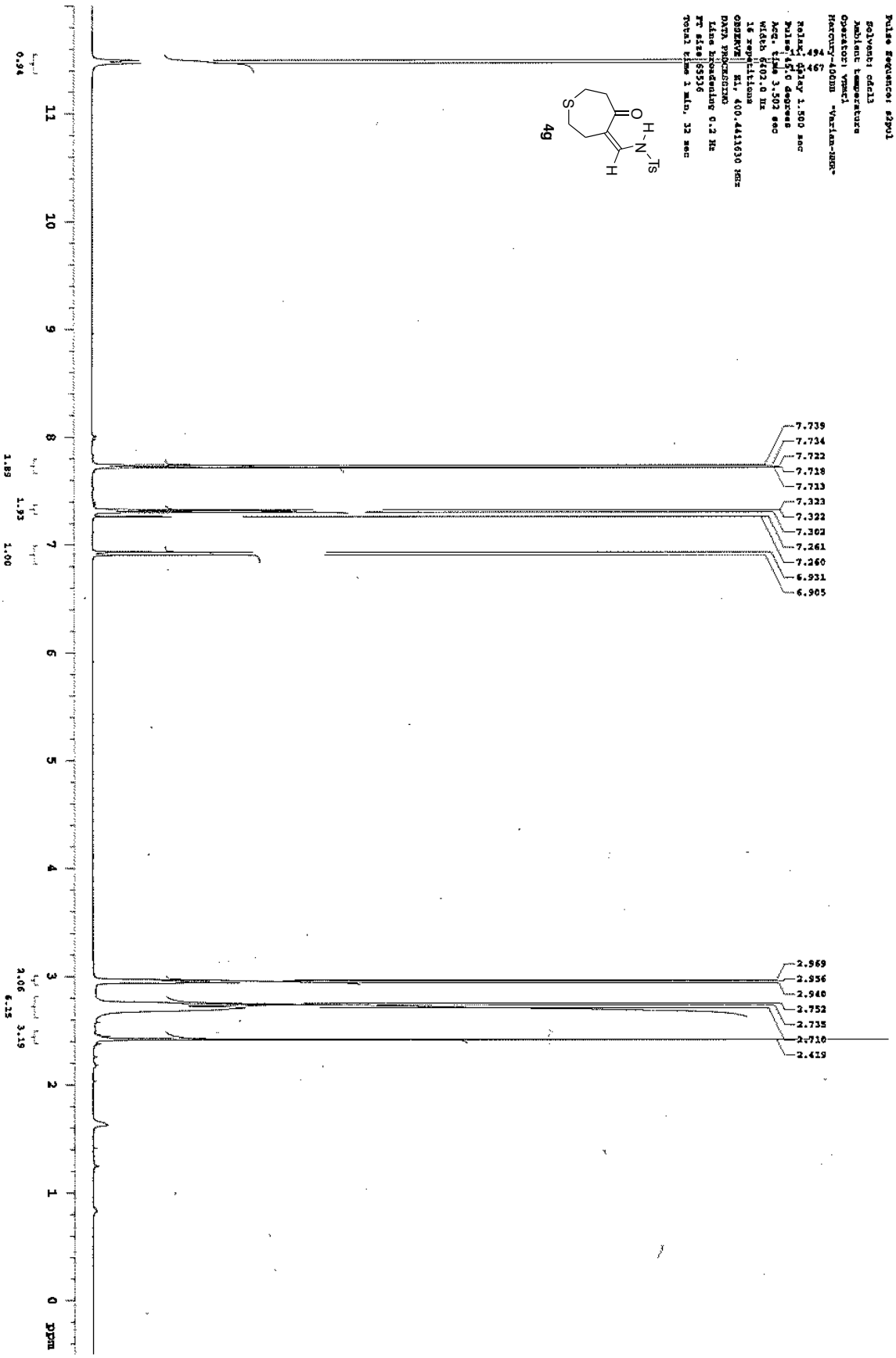

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmmr1
Mercury-400BB "Varian-MMM"
Relax. delay 1.700 sec
Pulse 45.0 degrees
Acq. time 1.360 sec
Width 24154.6 Hz
14048 repetitions
OBSERVE C13, 100.6910114 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Data Processing
Line broadening 1.0 Hz
FT size 65536
Total time 581101 hr, 9 min, 20 sec
4d
144.139
137.316
137.236
129.907
126.686
115.830
77.322
77.000
76.685
39.649
32.694
29.861
28.873
25.974
25.571
21.552
220
200
180
160
140
120
100
80
60
40
20
0
ppm

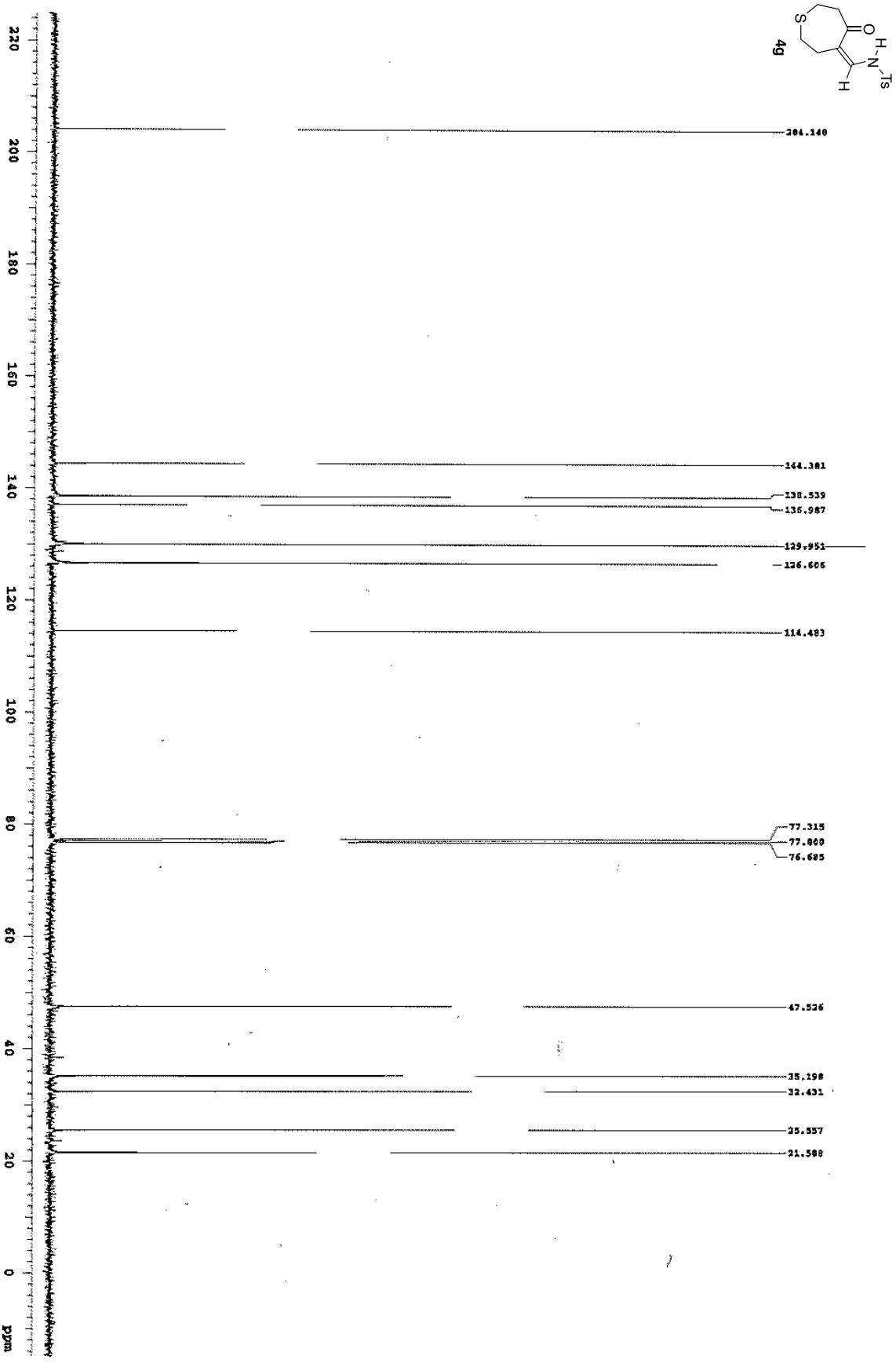

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-NMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 1.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411634 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
Ts
H
N
O
H
4e
7.727
7.707
7.302
7.282
6.849
6.823
2.524
2.508
2.492
2.404
2.371
1.757
1.698
1.441
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
2.05
2.25
1.00
2.34
5.62
2.49
6.83
2.35

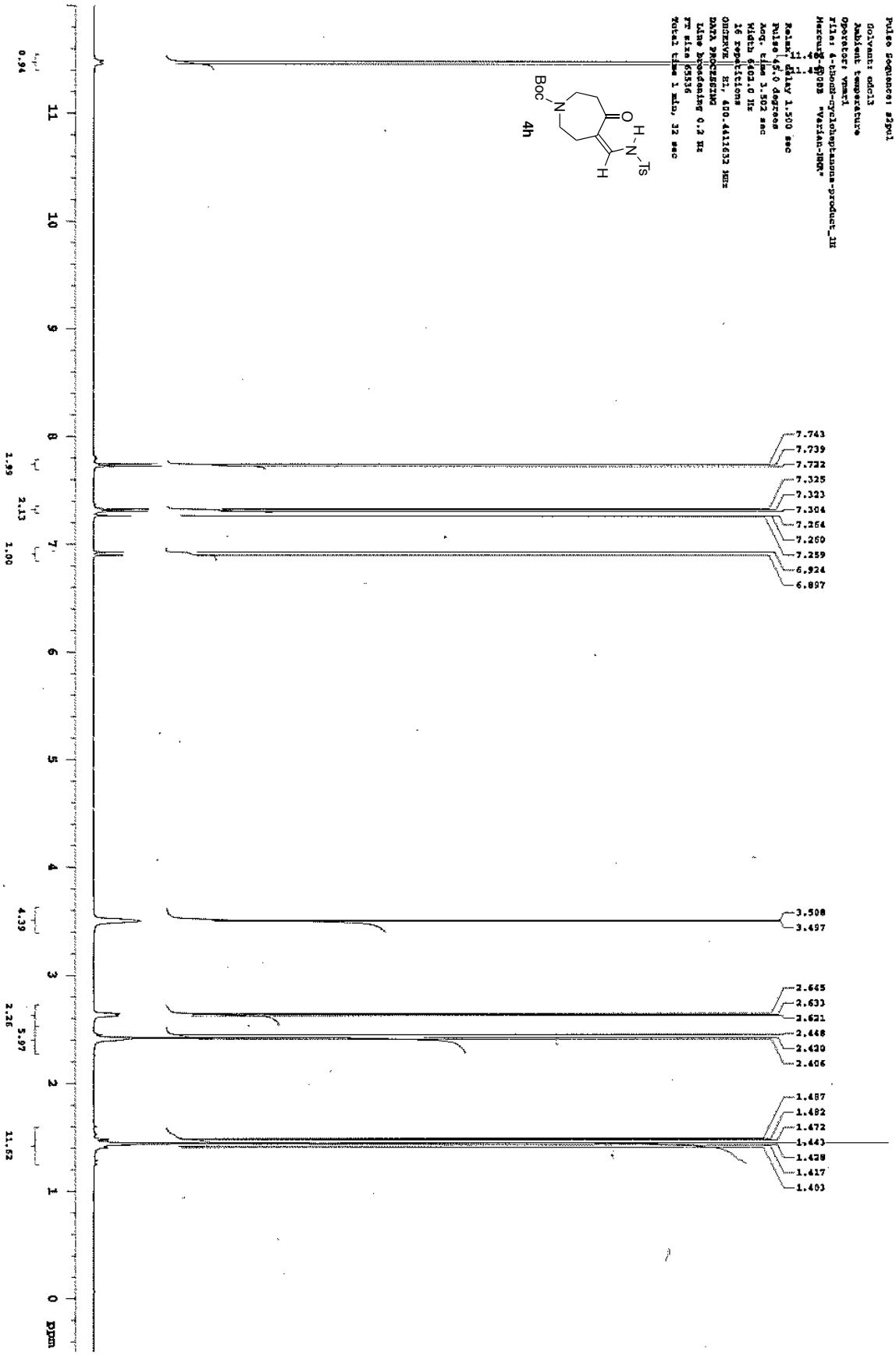

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-NMR"
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
15616 repetitions
OBSERVE C13, 100.5910149 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
H-N-Ts
O
H
4e
144.132
138.180
137.287
117.199
77.321
77.000
76.685
39.195
30.989
29.949
27.563
26.130
24.561
24.305
21.545
ppm

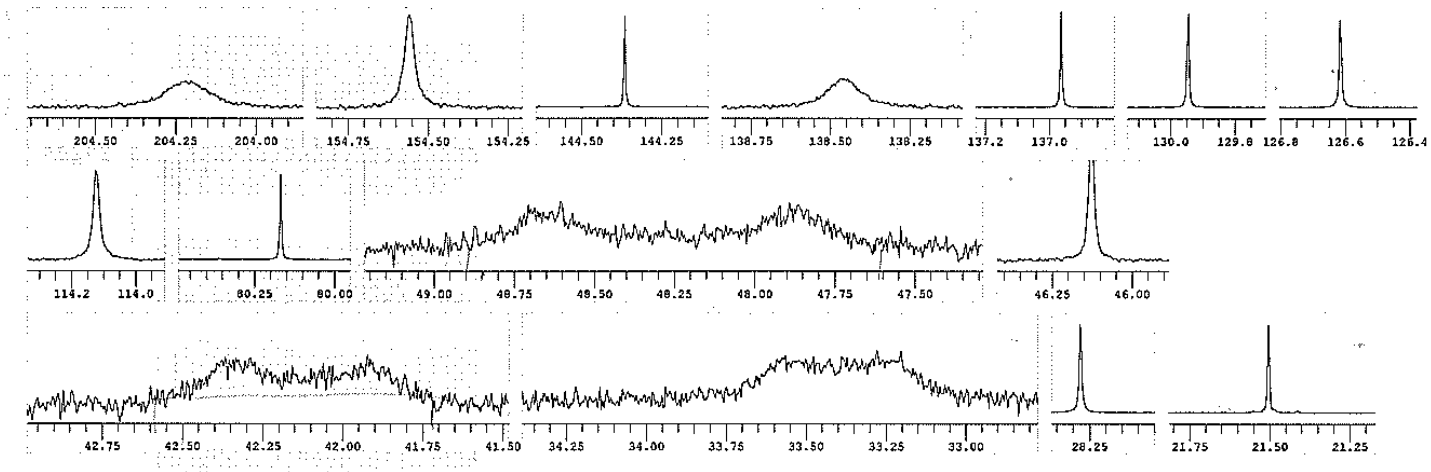

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-VNMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411610 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
4f
7.738
7.721
7.717
7.320
7.319
7.306
7.260
6.900
6.874
3.734
3.728
3.721
3.716
3.711
2.748
2.729
2.716
2.497
2.486
2.474
2.414
0.96
1.97
2.06
1.00
4.23
2.12
2.06
3.16
ppm

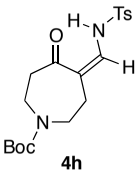

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmkc
File: rmp_product_13c
Mercury-400BB "varian-mkm"
Relax. delay 9.750 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
17730 repetitions
OBSERVE C13, 100.6910157 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 65536
Total time 581181 hr, 9 min, 20 sec
4f
144.381
137.785
137.053
129.966
126.642
114.497
77.315
77.205
77.000
76.685
72.307
56.048
48.280
34.985
21.523
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrj
Varian-400MR "Varian-MRH"
494.467
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
4g
7.739
7.734
7.722
7.718
7.713
7.323
7.322
7.302
7.261
7.260
6.931
6.905
2.969
2.956
2.940
2.752
2.735
2.710
2.419
0.94
1.89
1.93
1.00
2.06
6.25
3.19
ppm


4g
O
H
N
Ts
S
H
204.140
144.381
138.539
135.987
129.951
126.606
114.483
77.315
77.000
76.685
47.526
35.198
32.431
25.557
21.588
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Boc
N
O
H
N
Ts
H
4h
7.743
7.739
7.732
7.325
7.323
7.304
7.264
7.260
7.259
6.924
6.897
3.508
3.497
2.645
2.633
2.621
2.448
2.439
2.406
1.487
1.482
1.472
1.443
1.439
1.417
1.403
0.94
1.99
2.13
1.00
4.39
5.97
2.26
11.62
ppm

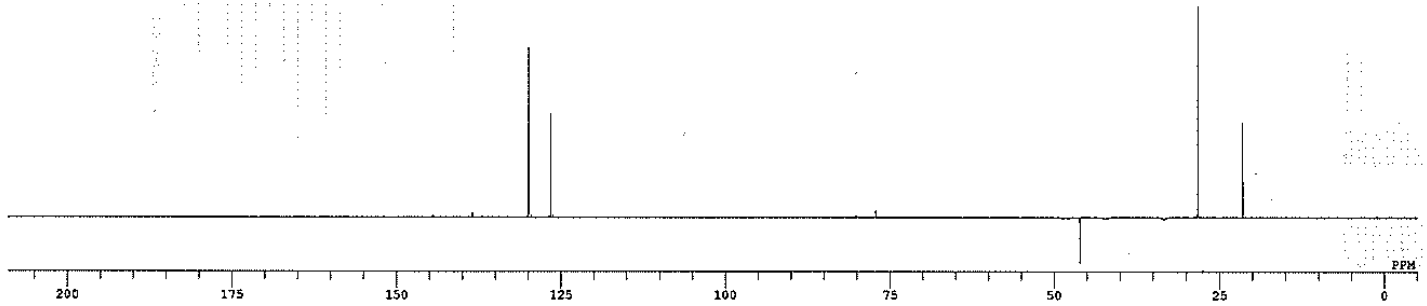




4h

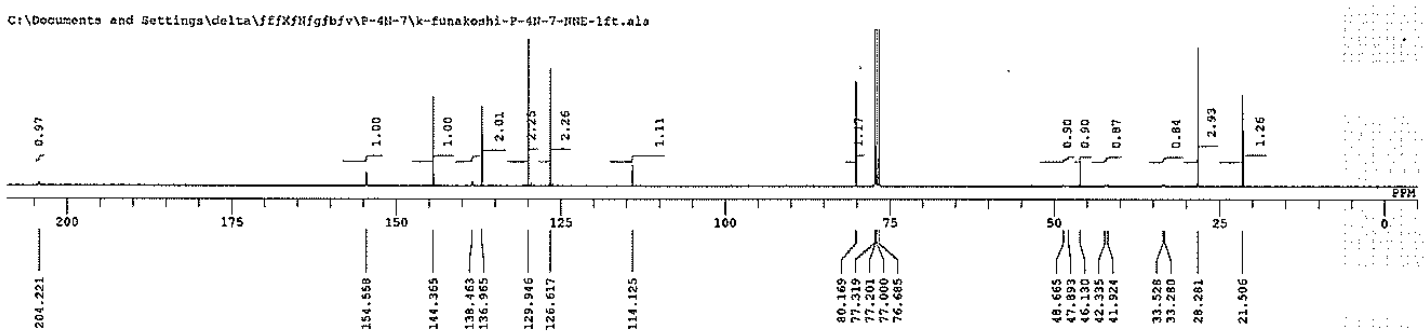
930874

C:\Documents and Settings\delta\ffXfNfgfbfv\P-4N-7\k-funakoshi-P-4N-7-dept135-1ft.als

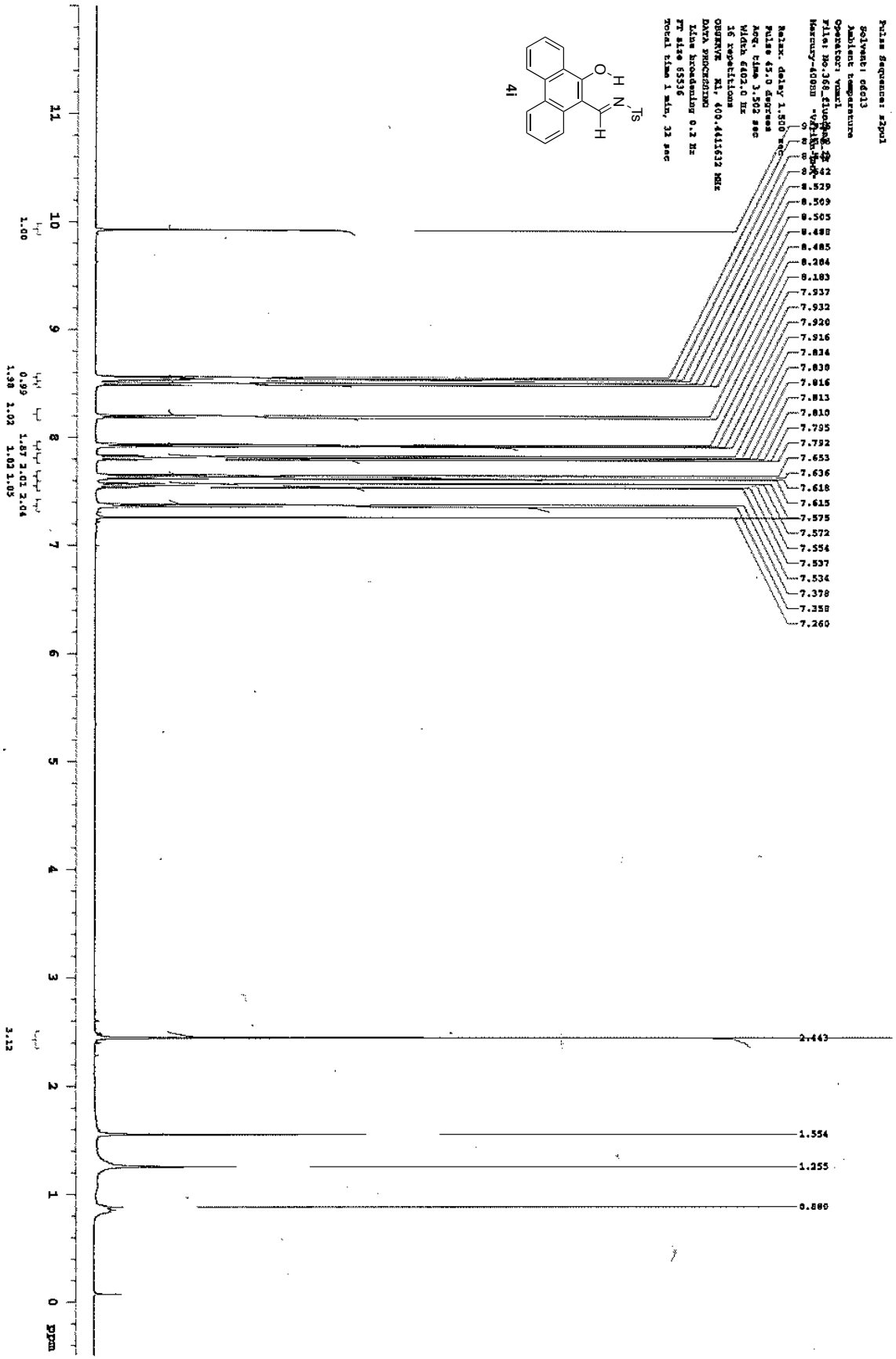


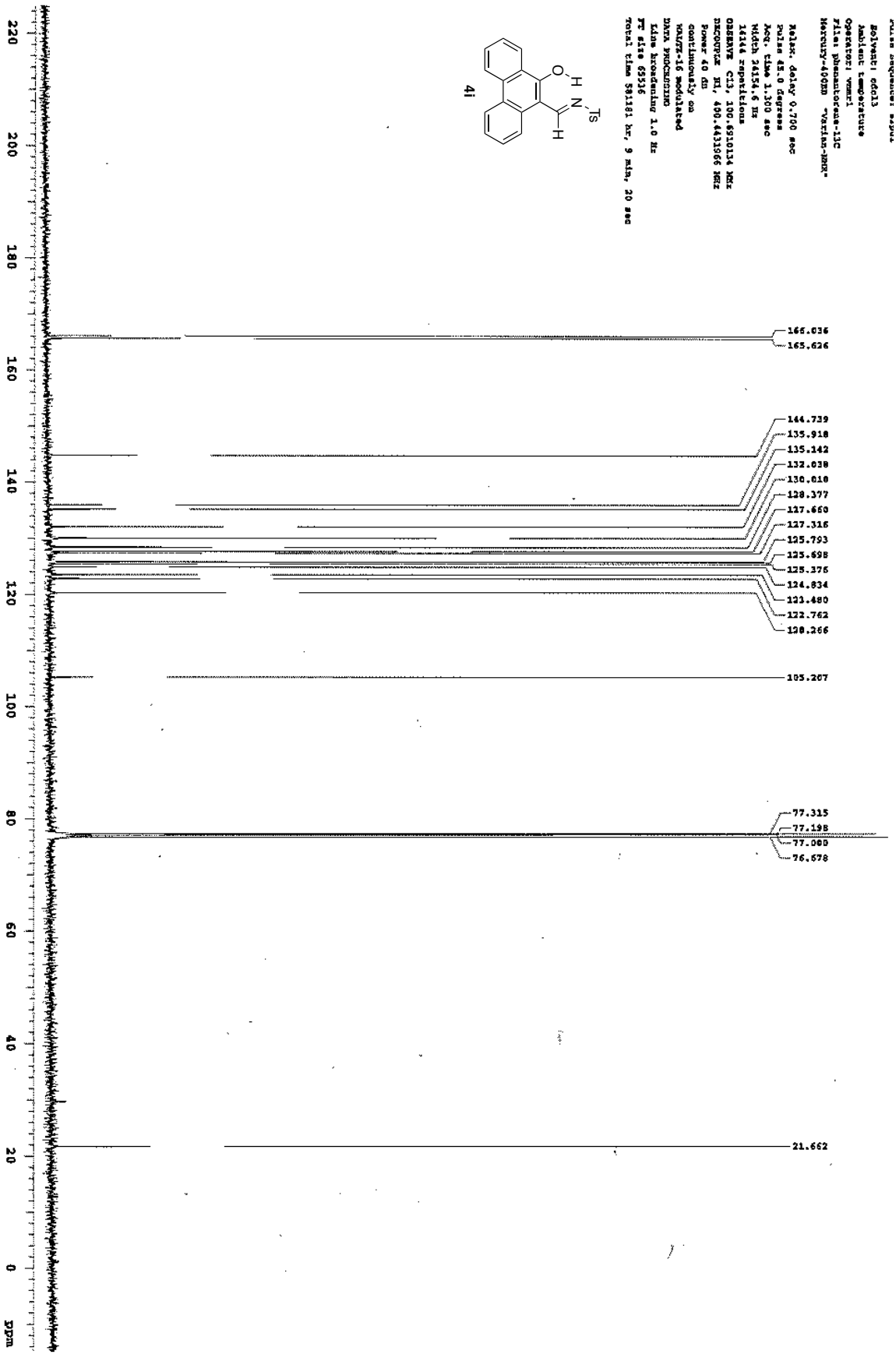

```
DFILE   k-funakoshi-P-4N-7-dept135-1ft.a
COMNT   DEPT with decoupling
DATIM   19-07-2012 08:20:29
OBNUC   13C
EXMOD   dept.ex2
OBFRQ         100.53 MHz
OBFIN           5.86 Hz
POINT          52428
FREQU       22123.56 Hz
SCANS           1200
ACQTM         2.3698 sec
PD            2.0000 sec
PW1              8.00 usec
IRNUC   1H
CTEMP           22.6 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.21 Hz
RGAIN             60
```

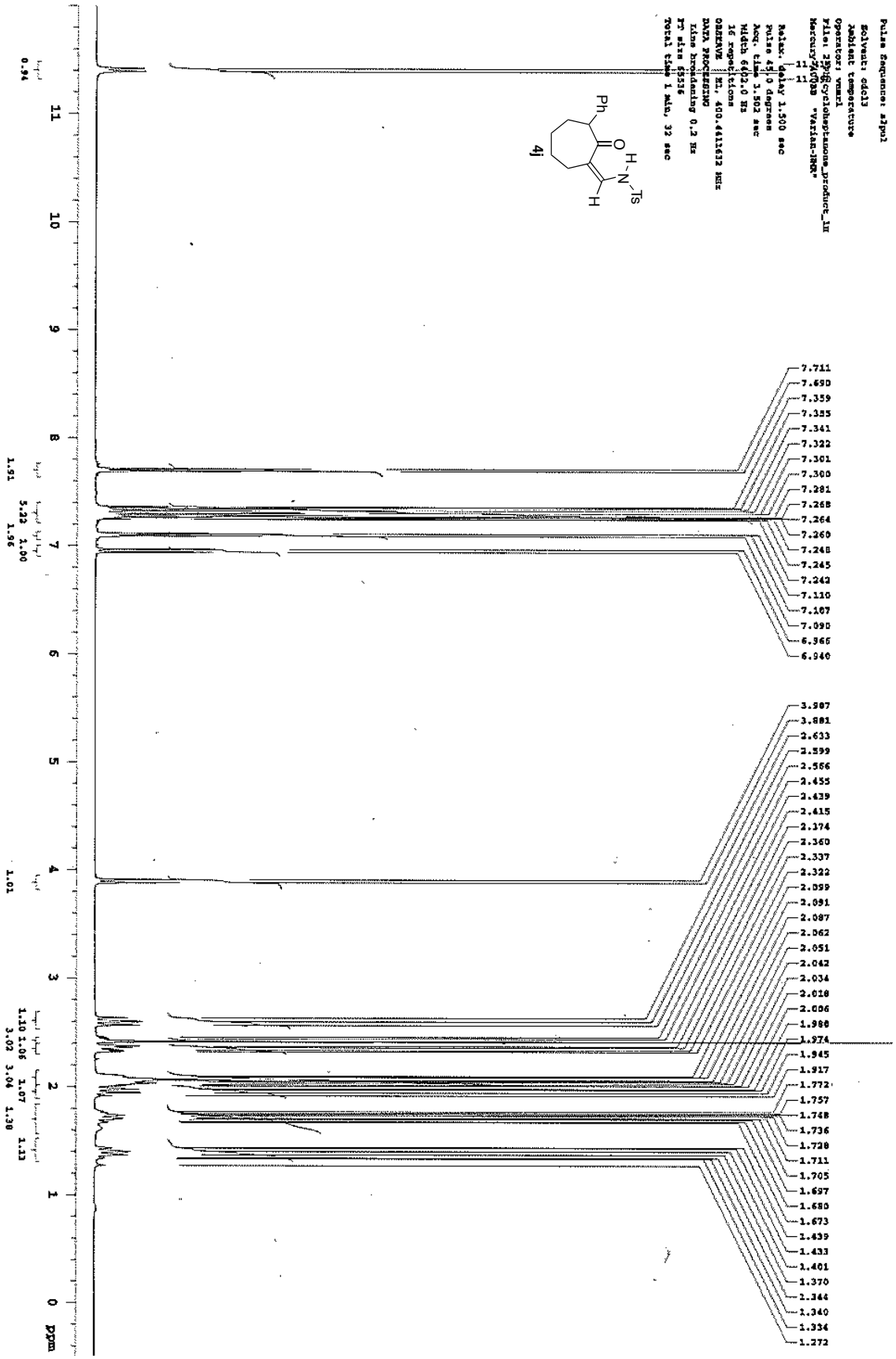
C:\Documents and Settings\delta\ffXfNfgfbfv\P-4N-7\k-funakoshi-P-4N-7-NNE-1ft.als

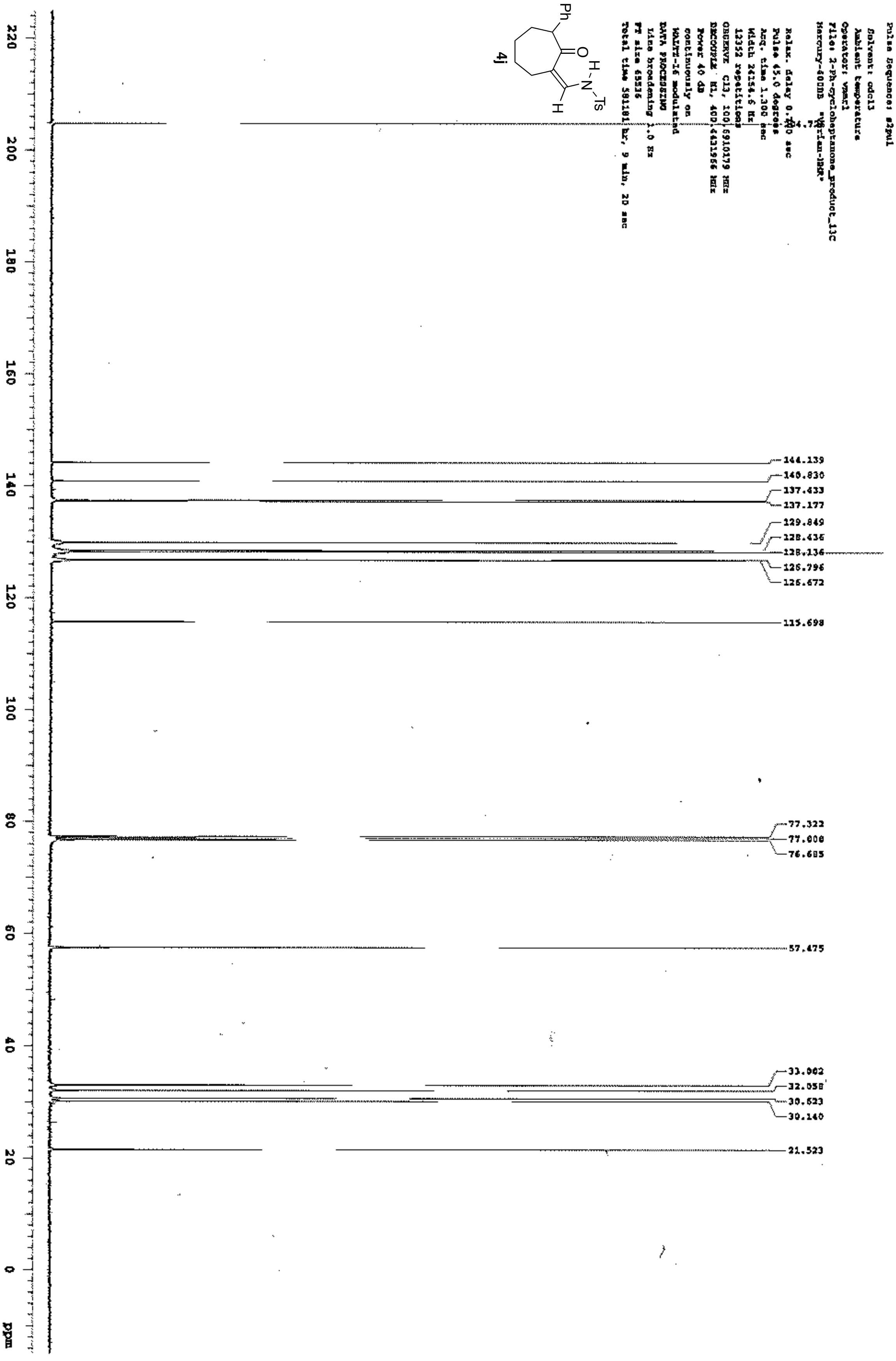


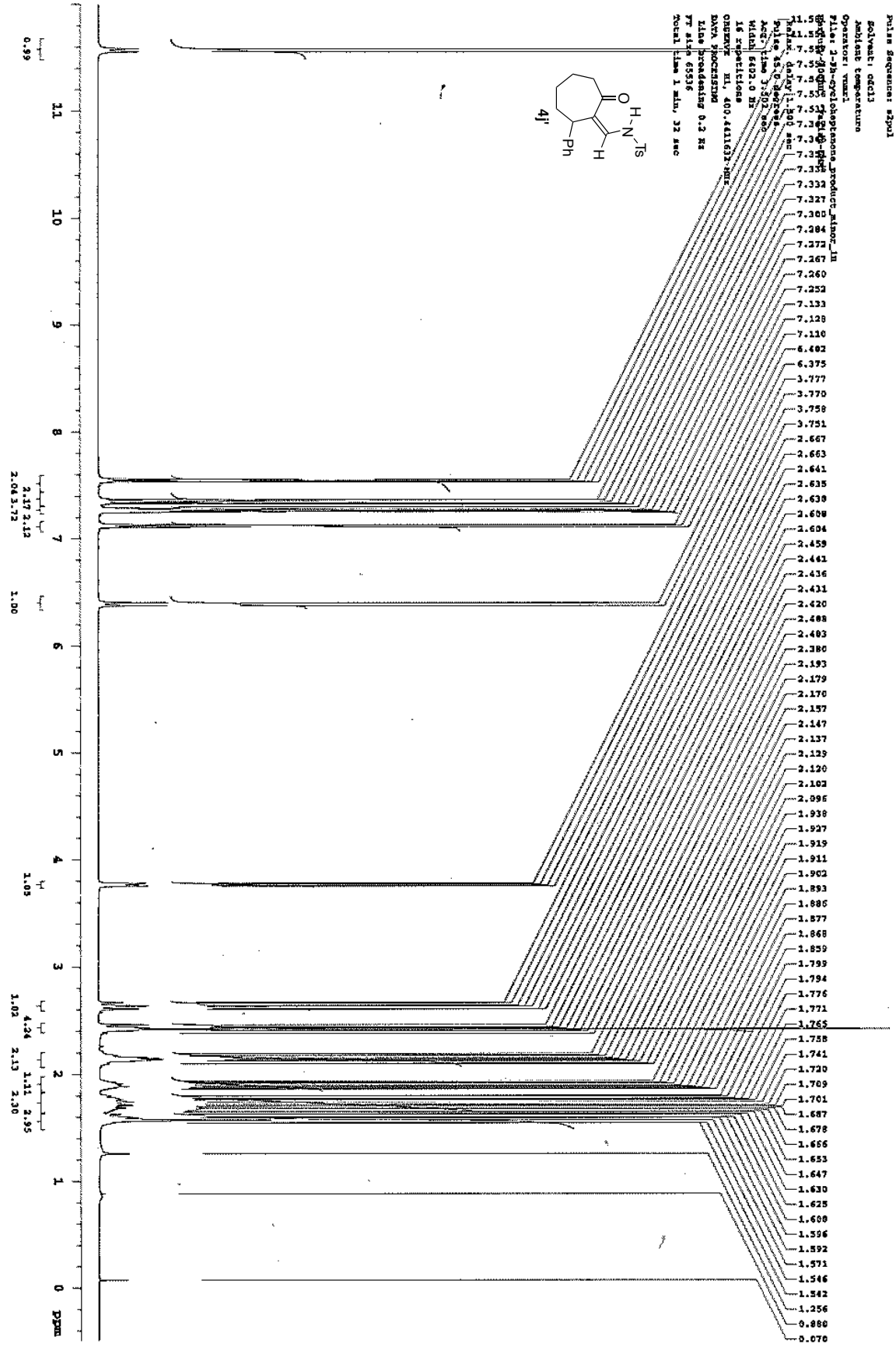

```
DFILE   k-funakoshi-P-4N-7-NNE-1ft.als
COMNT   single pulse decoupled gated NO
DATIM   19-07-2012 06:50:48
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         100.53 MHz
OBFIN           5.86 Hz
POINT          52428
FREQU       22123.56 Hz
SCANS           6800
ACQTM         2.3698 sec
PD            5.1000 sec
PW1              2.67 usec
IRNUC   1H
CTEMP           22.4 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.21 Hz
RGAIN             60
```

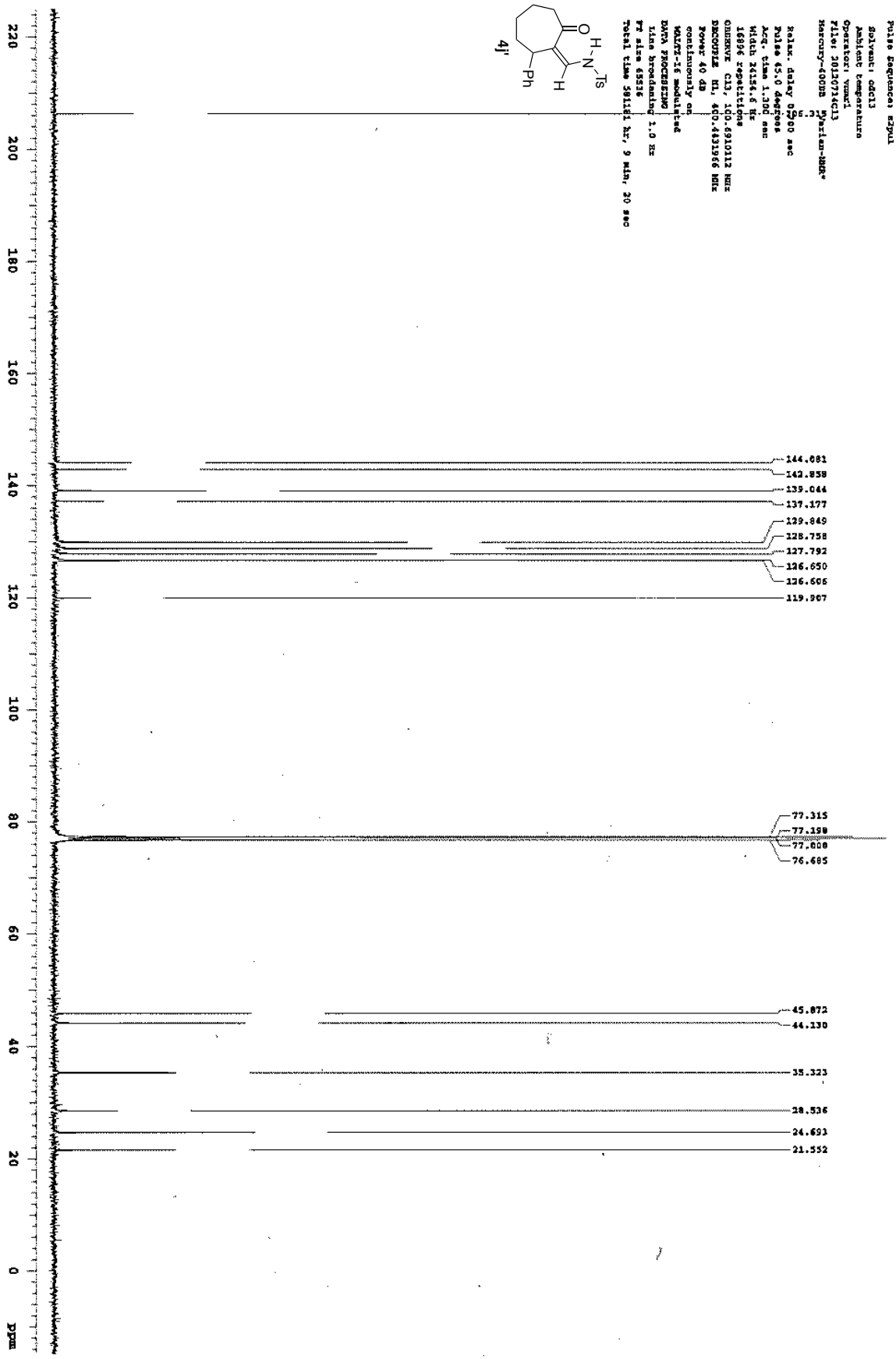

4i
Ts
H
N
O
H
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmmrl
File: No.368_tiuoqhuo_4i
Mercury-400BB: vnmrbg
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
9.042
8.529
8.509
8.505
8.498
8.485
8.204
8.183
7.937
7.932
7.920
7.916
7.834
7.830
7.816
7.813
7.810
7.795
7.792
7.653
7.636
7.618
7.615
7.575
7.572
7.554
7.537
7.534
7.378
7.358
7.260
2.443
1.554
1.255
0.880
1.00
0.99
1.98
1.02
1.87
2.01
2.04
1.03
1.05
3.12
ppm

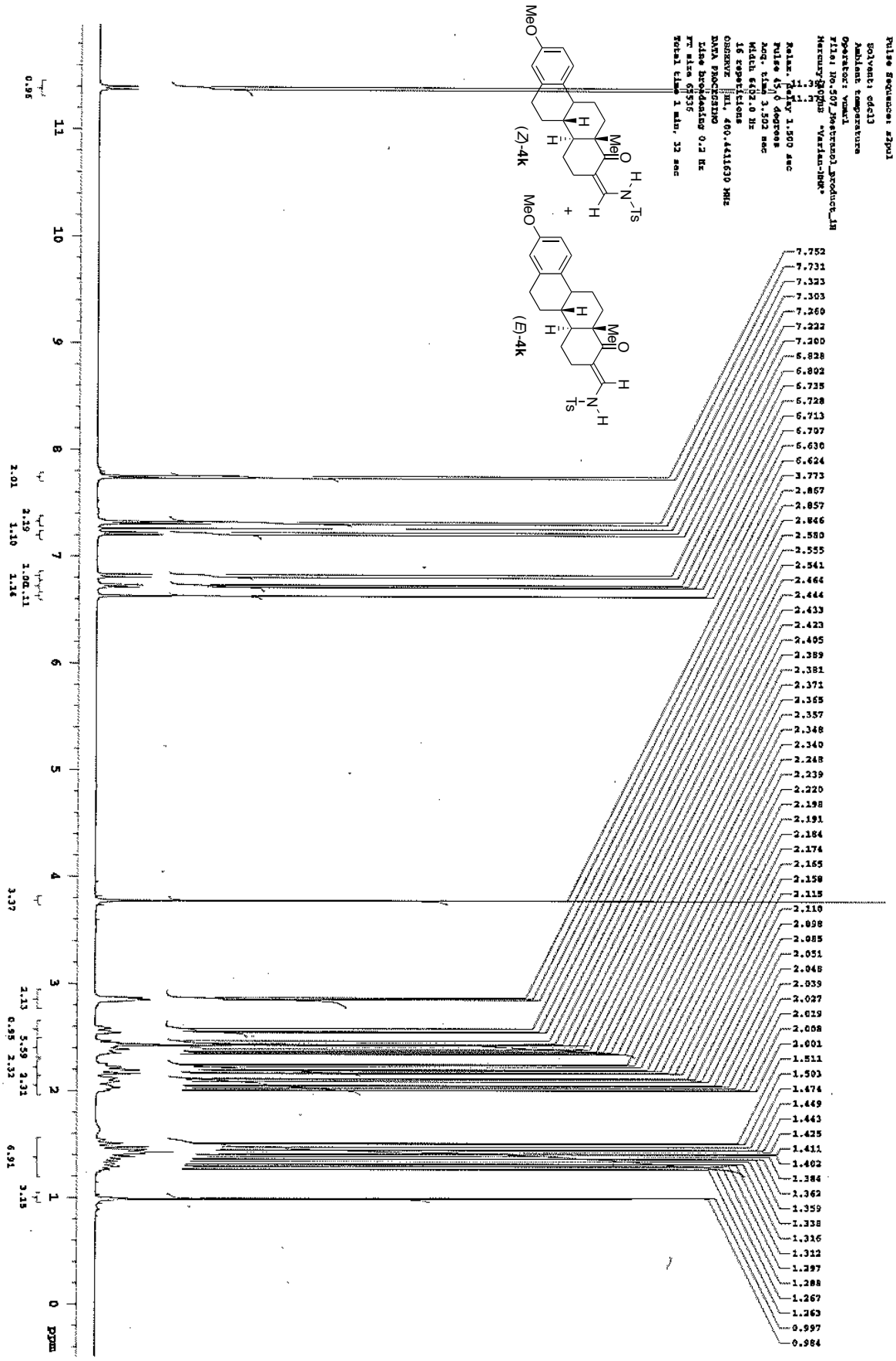

Pulse sequence: zgpg
Solvent: cdcl3
Ambient temperature
Operator: vmerl
File: phenanteroen-13c
Mercury-400BB "varian-mrm"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
16144 repetitions
OBSERVE C13, 100.6510134 MHz
DECOUPLE H1, 400.4433966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581181 hr, 9 min, 20 sec
4i
166.036
165.626
144.739
135.918
135.142
132.038
130.610
128.377
127.660
127.316
125.793
125.698
125.376
124.834
121.480
122.762
120.266
105.207
77.315
77.198
77.080
76.678
21.662
220
200
180
160
140
120
100
80
60
40
20
0
ppm

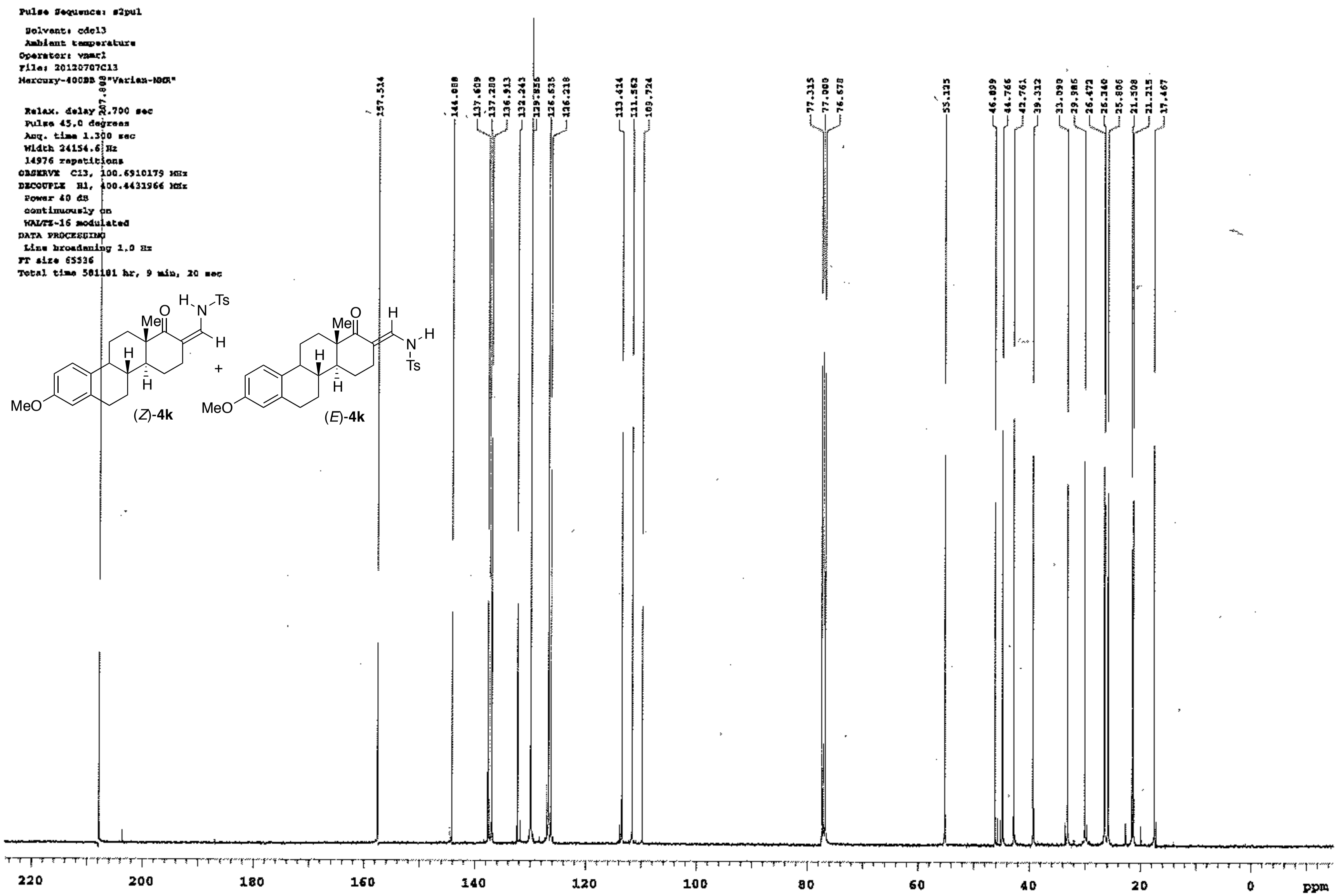

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
File: 2SP-8-cycloheptanone_product_1H
Mercury-400BB "Varian-NMR"
11
11
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4611632 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
Ph
O
H
N
Ts
H
4j
7.711
7.690
7.359
7.355
7.341
7.322
7.301
7.300
7.281
7.268
7.264
7.260
7.248
7.245
7.242
7.110
7.107
7.090
6.966
6.940
3.907
3.881
2.633
2.599
2.566
2.455
2.439
2.415
2.374
2.360
2.337
2.322
2.099
2.091
2.087
2.062
2.051
2.042
2.034
2.018
2.006
1.988
1.974
1.945
1.917
1.772
1.757
1.748
1.736
1.728
1.711
1.705
1.697
1.680
1.673
1.439
1.433
1.401
1.370
1.344
1.340
1.334
1.272
0.94
1.91
5.22
1.00
1.96
1.01
1.10
2.06
3.07
1.13
3.02
3.04
1.38
ppm

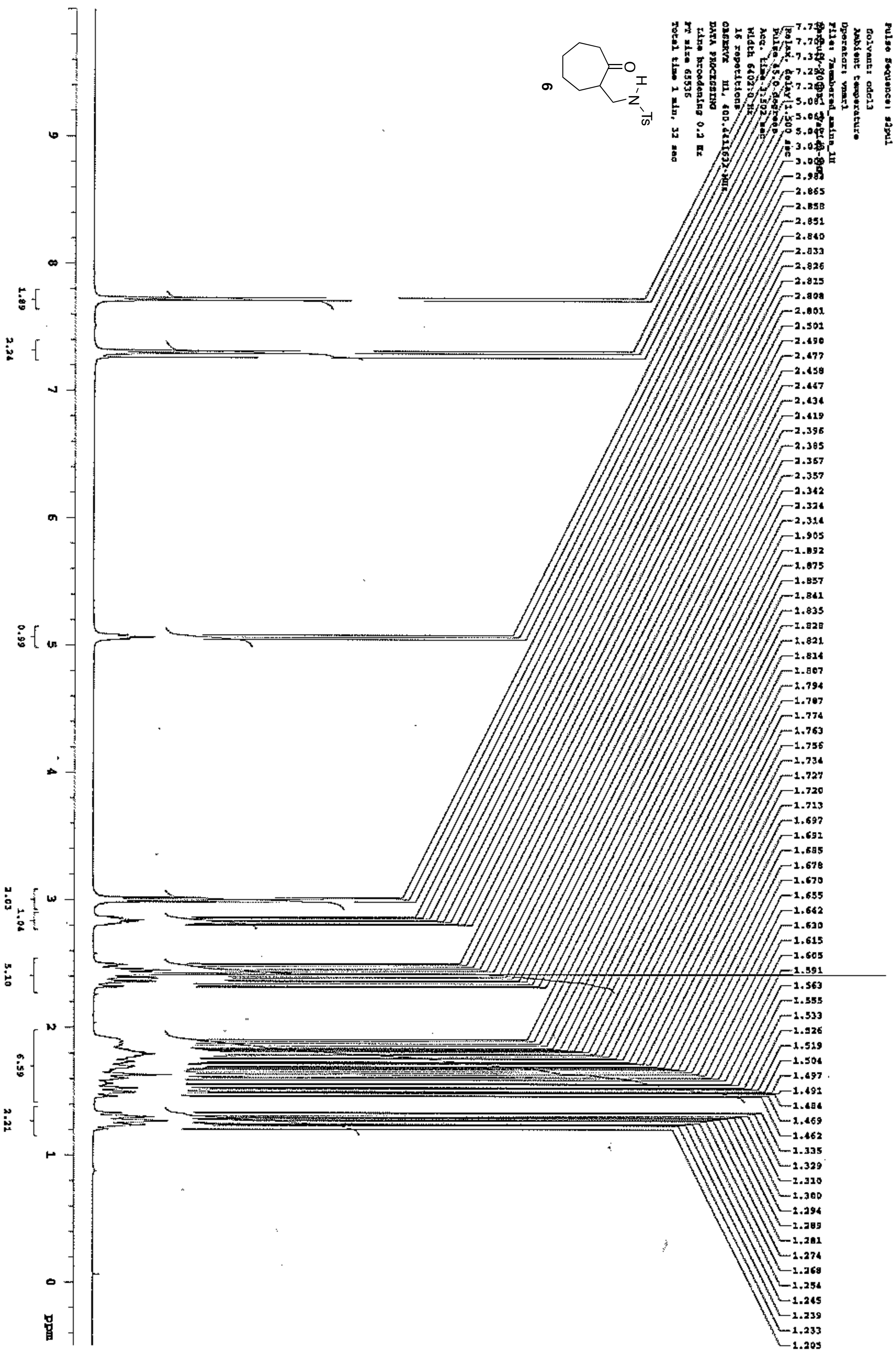

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: 2-Ph-cycloheptanone_product_13C
Mercury-400BB "Varian-NMR"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
12352 repetitions
OBSERVE C13, 100.6910179 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Ph
O
H
N
Ts
H
4j
144.139
140.830
137.433
137.177
129.849
128.436
128.136
126.796
126.672
115.698
77.322
77.000
76.685
57.475
33.002
32.058
30.623
30.140
21.523
220
200
180
160
140
120
100
80
60
40
20
0
ppm


4j'
O
H
N
Ts
Ph
H

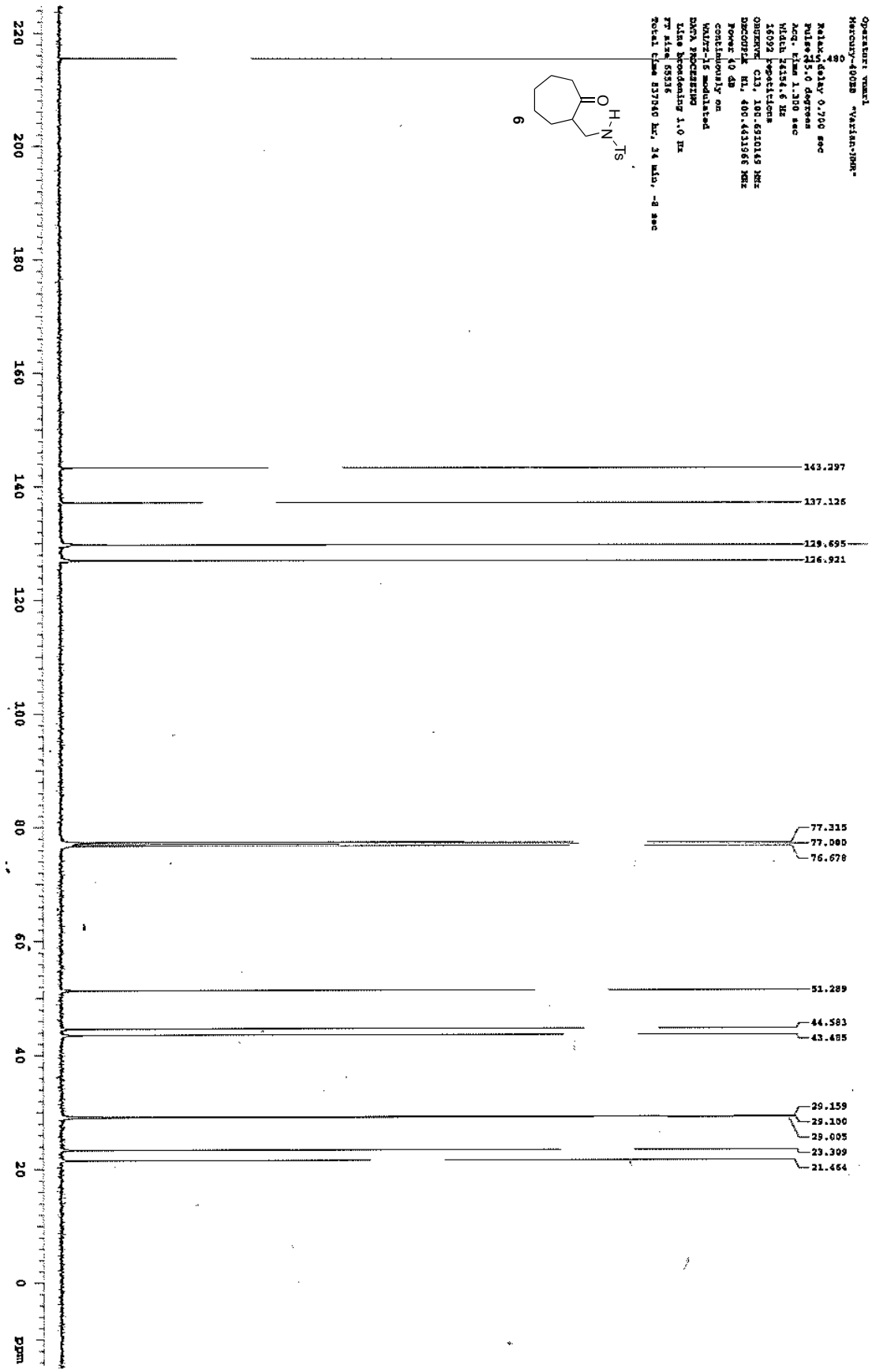

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: 20120714cl3
Mercury-400BB Agilent-NMR*
56.31
Solax. delay 0.000 sec
Pulse 45.0 degree
Acq. time 1.306 sec
Width 24154.6 Hz
16896 repetitions
OBSERVE C13, 100.6910112 MHz
DECOUPLE H1, 400.4831966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 5811:1 hr, 9 min, 30 sec
4j'
O
H
N
Ts
Ph
H
144.081
142.858
139.044
137.177
129.849
128.758
127.792
126.650
126.605
119.907
77.315
77.158
77.000
76.685
45.872
44.130
35.323
28.536
24.693
21.552
220
200
180
160
140
120
100
80
60
40
20
0
ppm

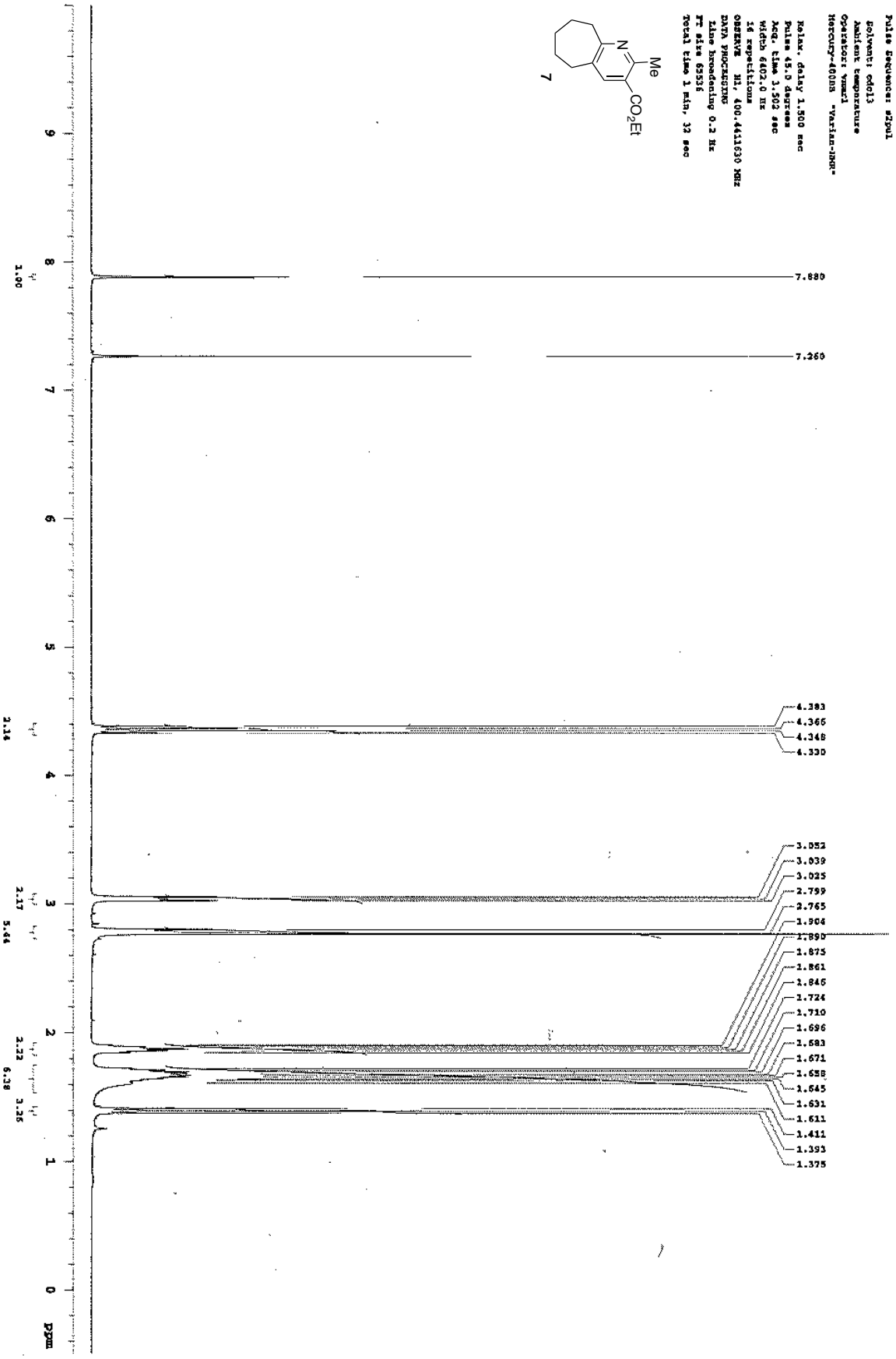

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vcmr1
File: No.507_Methanol_product_1H
Mercury-400BB "Varian-NMR"
11.37
1.37
Relax. Delay 1.500 sec
Pulse 45.0 degree
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 33 sec
MeO
MeO
(Z)-4k
(E)-4k
7.752
7.731
7.323
7.303
7.260
7.222
7.200
6.828
6.802
6.735
6.728
6.713
6.707
6.630
6.624
3.773
2.867
2.857
2.846
2.580
2.555
2.541
2.464
2.444
2.433
2.423
2.405
2.389
2.381
2.371
2.365
2.357
2.348
2.340
2.248
2.239
2.220
2.198
2.191
2.184
2.174
2.165
2.158
2.118
2.110
2.098
2.085
2.051
2.048
2.039
2.027
2.019
2.008
2.001
1.511
1.503
1.474
1.449
1.443
1.425
1.411
1.402
1.384
1.362
1.359
1.338
1.316
1.312
1.297
1.288
1.267
1.263
0.997
0.984
0.95
2.01
2.19
1.10
1.08
1.14
3.37
2.13
5.59
2.31
0.95
2.32
6.91
3.15
ppm

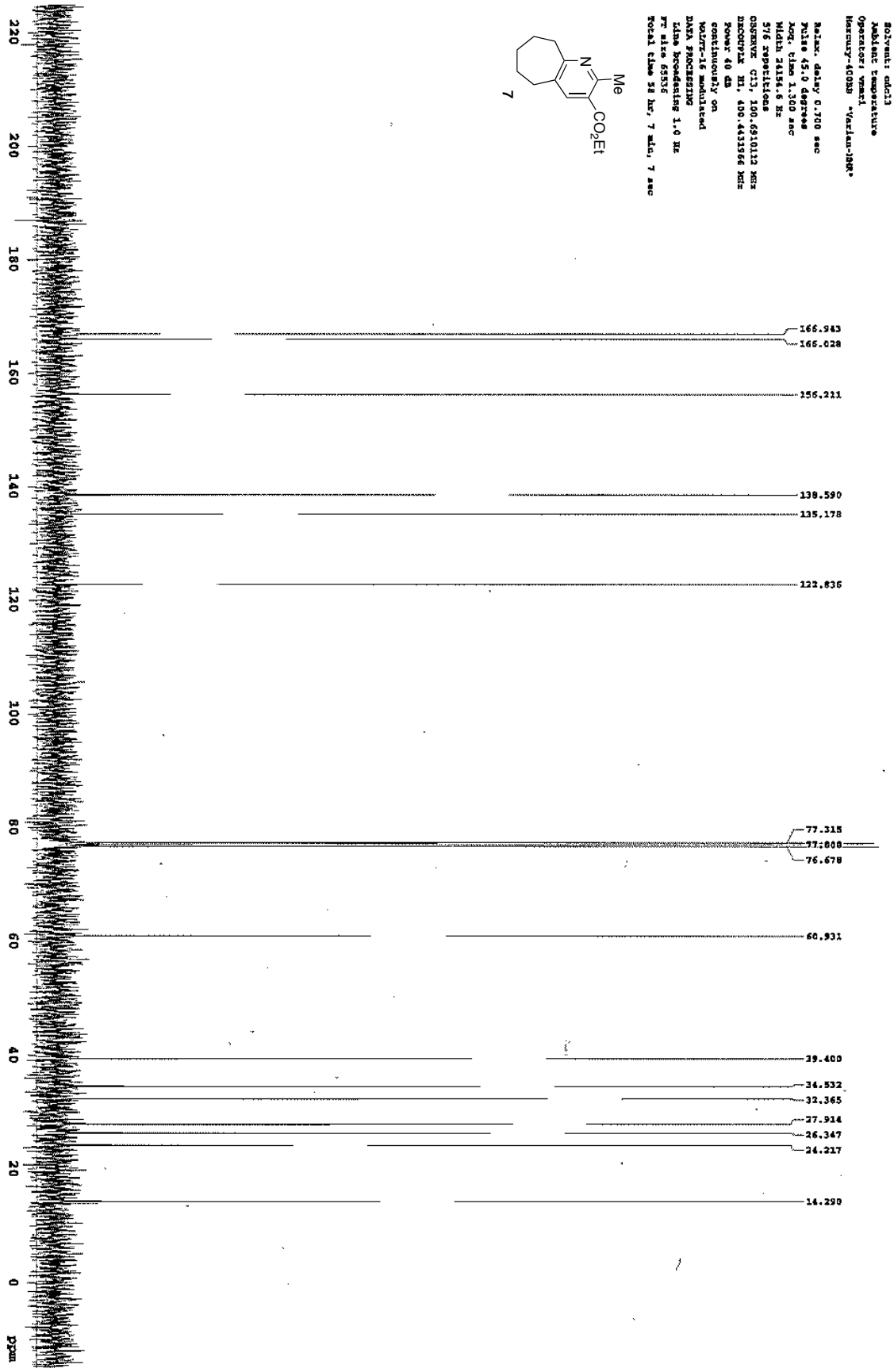

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
File: 20120707C13
Mercury-400BB "Varian-NMR"
Relax. delay 2.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
14976 repetitions
OBSERVE C13, 100.6910179 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581181 hr, 9 min, 20 sec
(Z)-4k
(E)-4k
157.514
144.088
137.609
137.280
136.913
132.243
126.635
126.218
113.414
111.562
109.724
77.315
77.000
76.678
55.125
46.899
44.766
42.761
39.312
33.090
29.985
26.472
26.340
25.806
21.508
21.215
17.467
ppm

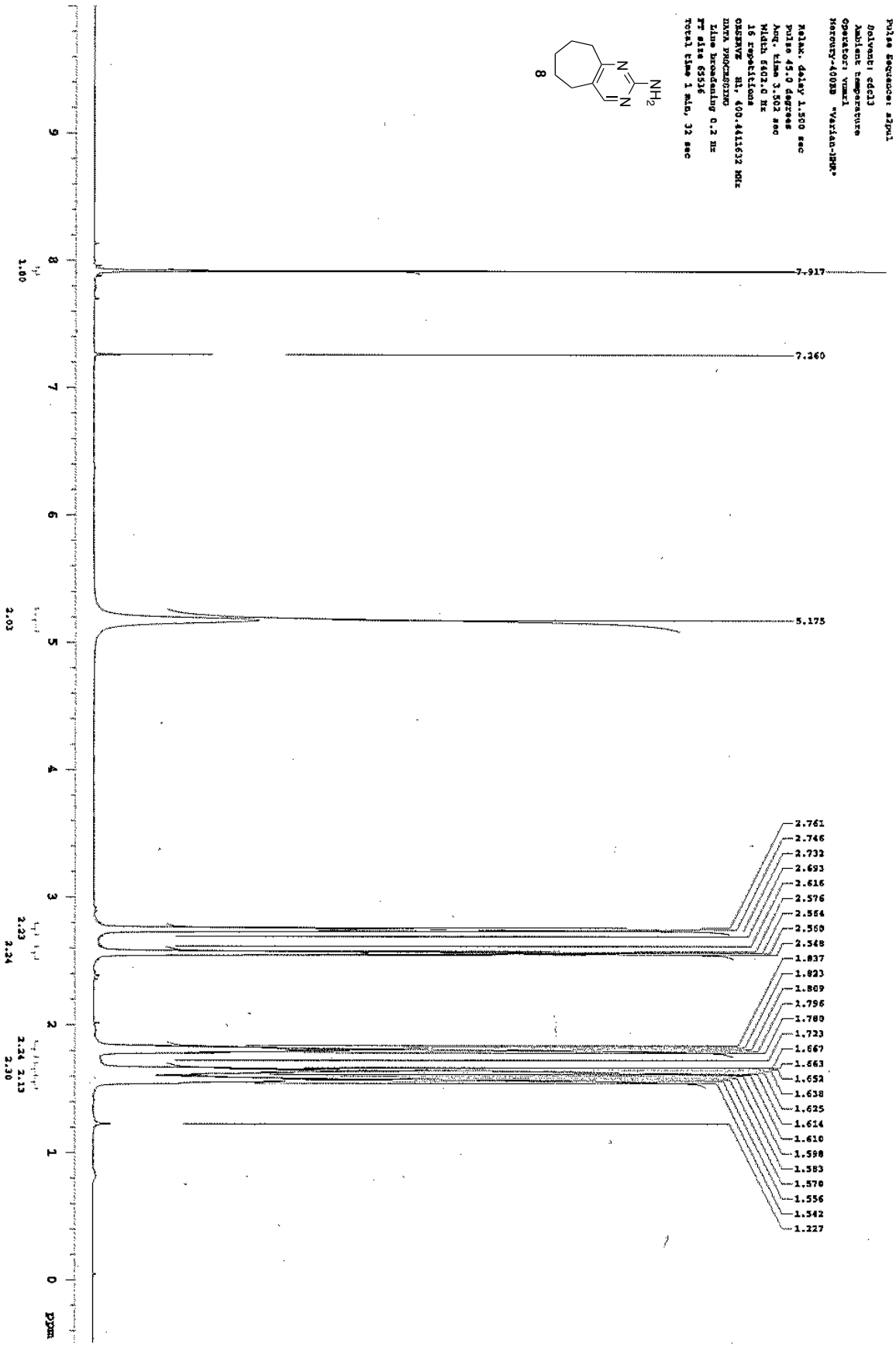


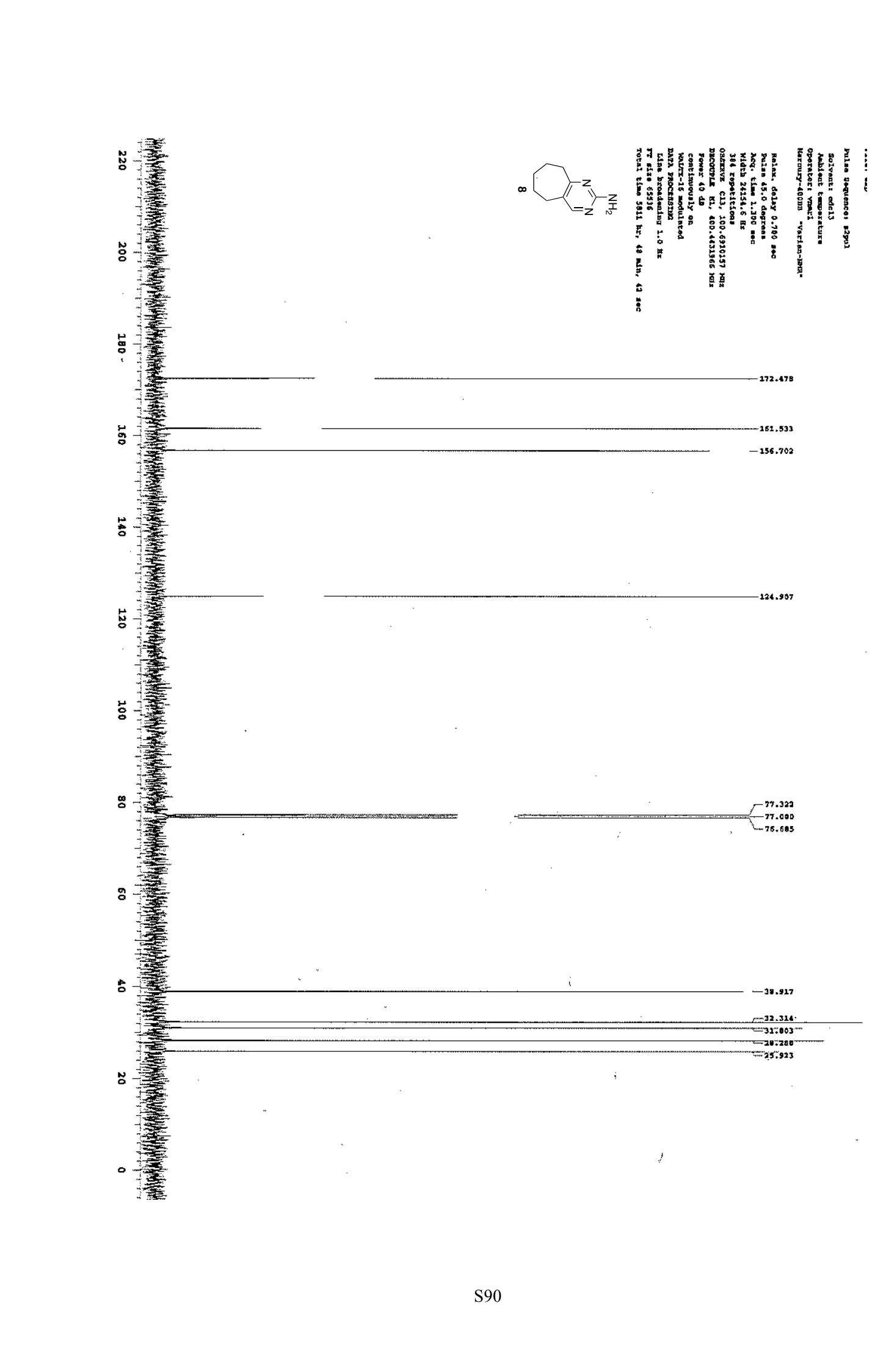
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Total time 1 min, 12 sec
O
H
N
Ts
6
2.865
2.858
2.851
2.840
2.833
2.826
2.815
2.808
2.801
2.501
2.490
2.477
2.458
2.447
2.434
2.419
2.396
2.385
2.367
2.357
2.342
2.324
2.314
1.905
1.892
1.875
1.857
1.841
1.835
1.828
1.821
1.814
1.807
1.794
1.787
1.774
1.763
1.756
1.734
1.727
1.720
1.713
1.697
1.691
1.685
1.678
1.670
1.655
1.642
1.630
1.615
1.605
1.591
1.563
1.555
1.533
1.526
1.519
1.504
1.497
1.491
1.484
1.469
1.462
1.335
1.329
1.310
1.300
1.294
1.289
1.281
1.274
1.268
1.254
1.245
1.239
1.233
1.205
1.89
2.24
0.99
2.03
1.04
5.10
6.59
2.21
9
8
7
6
5
4
3
2
1
0
ppm

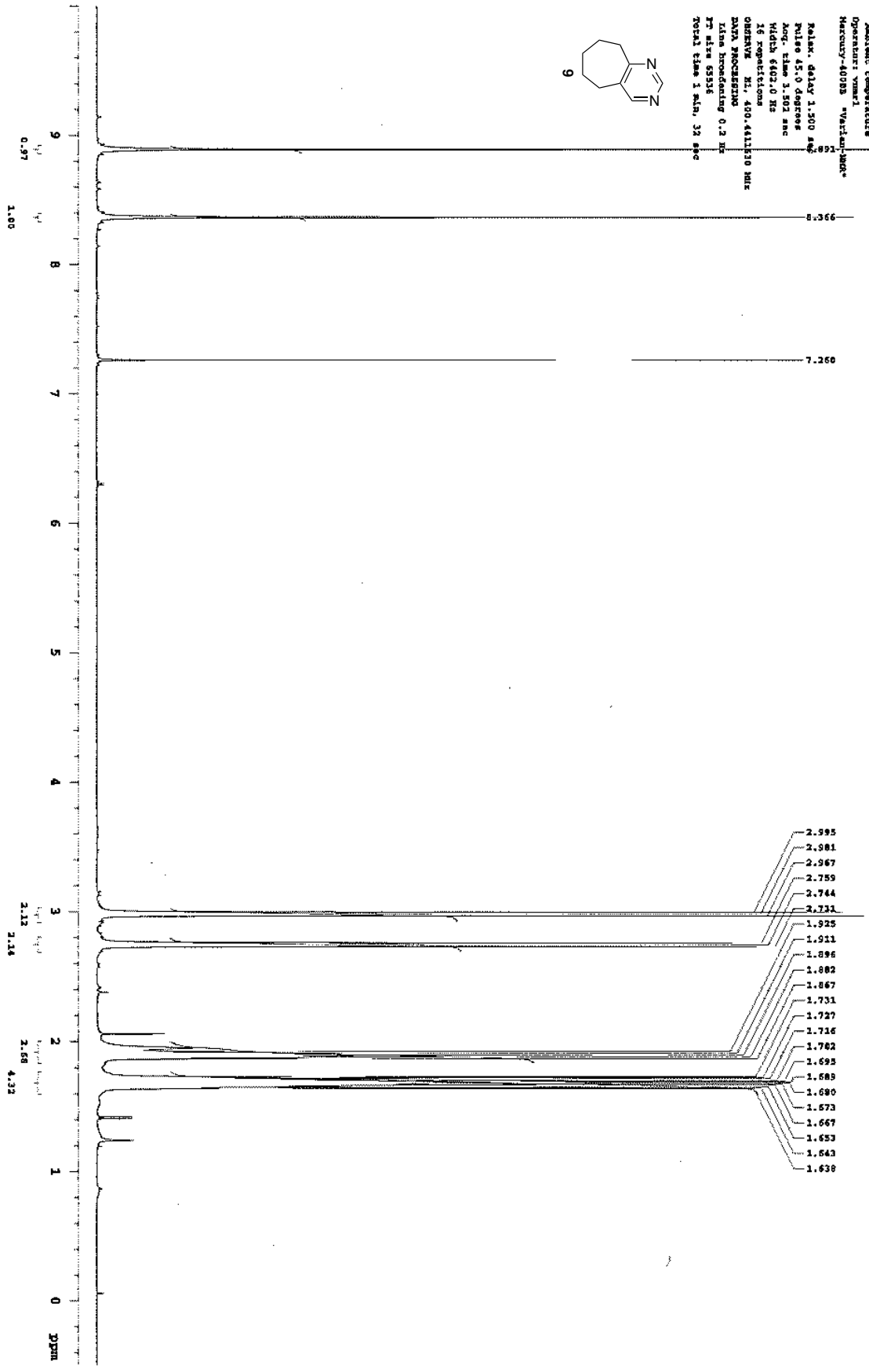

Solvent: cdcl3
Ambient temperature
Operator: vmarl
Mercury-400BB "Varian:NMR"
480
Relax.delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 16194.6 Hz
16092 repetitions
OBSERVE C13, 100.6310143 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 837840 hr, 24 min, -8 sec
6
143.297
137.125
129.695
126.921
77.315
77.000
76.678
51.289
44.583
43.485
29.159
29.100
29.005
23.309
21.464
220
200
180
160
140
120
100
80
60
40
20
0
ppm

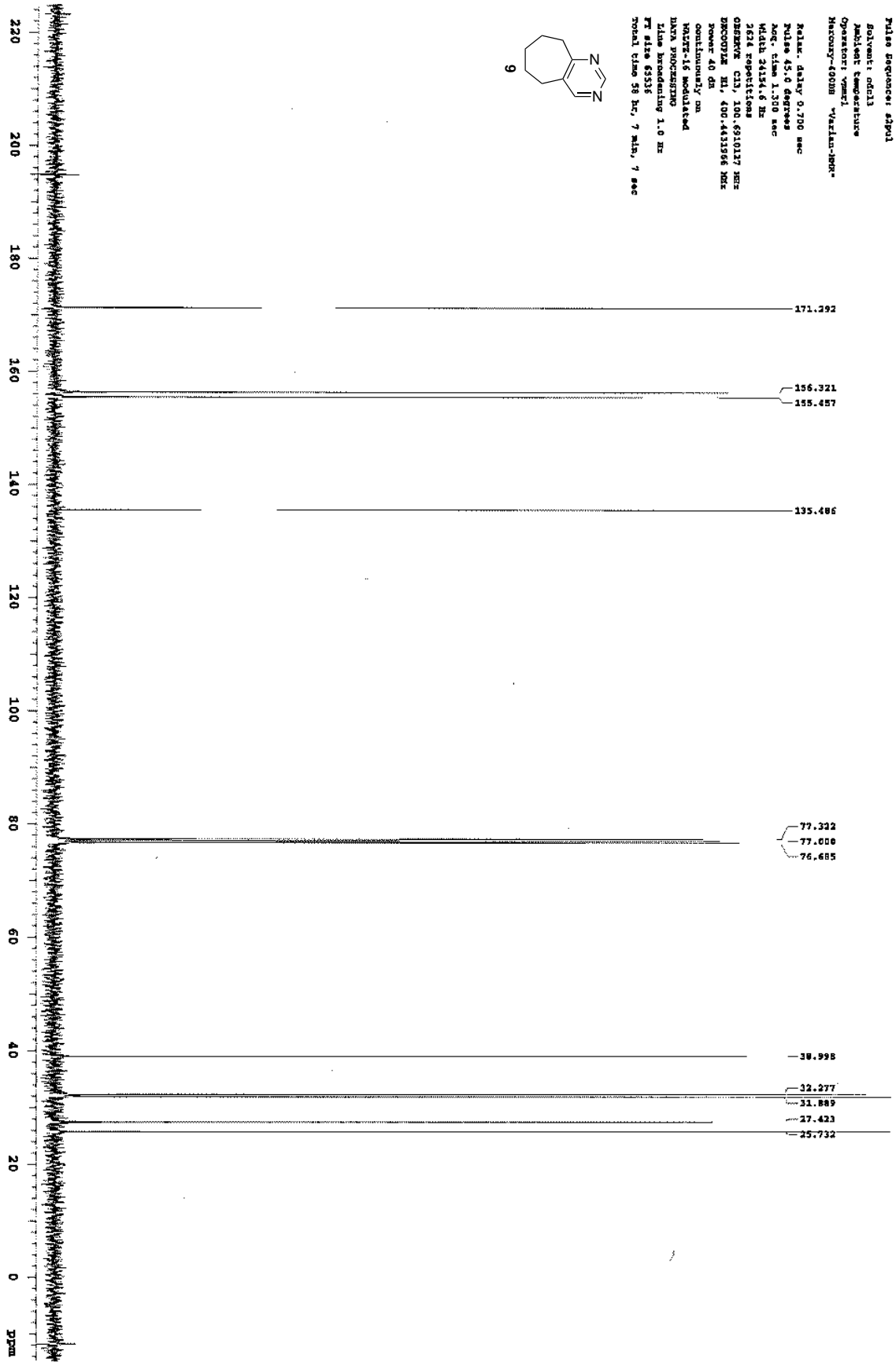

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-JHK"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 12 sec
Me
CO2Et
7
7.880
7.260
4.383
4.366
4.348
4.330
3.052
3.039
3.025
2.799
2.765
1.904
1.890
1.875
1.861
1.846
1.724
1.710
1.696
1.683
1.671
1.658
1.645
1.631
1.611
1.411
1.393
1.375
1.00
2.14
2.17
5.44
2.22
6.38
3.36
ppm

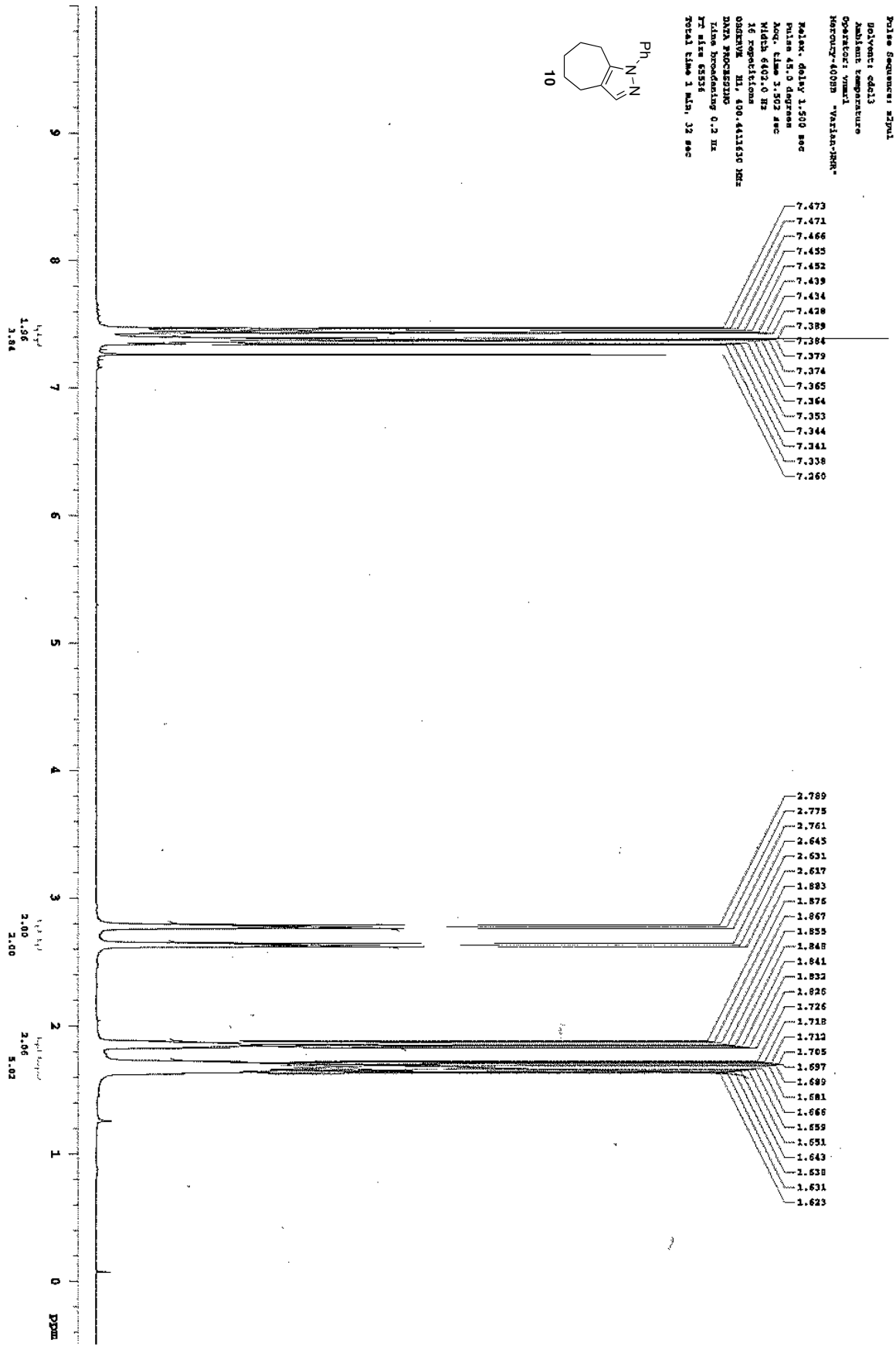

Solvent: cdcl3
Ambient temperature
Operator: vmsl
Mercury-400BB "Varian-300"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
576 repetitions
OBSERVE C13, 100.6910112 MHz
DECOUPLE H1, 400.4433566 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 65536
Total time 5h hr, 7 min, 7 sec
Me
N
CO2Et
7
166.983
166.028
156.211
138.590
135.178
122.836
77.315
77.000
76.678
60.931
39.400
34.532
32.365
27.914
26.347
24.217
14.290
220
200
180
160
140
120
100
80
60
40
20
0
ppm

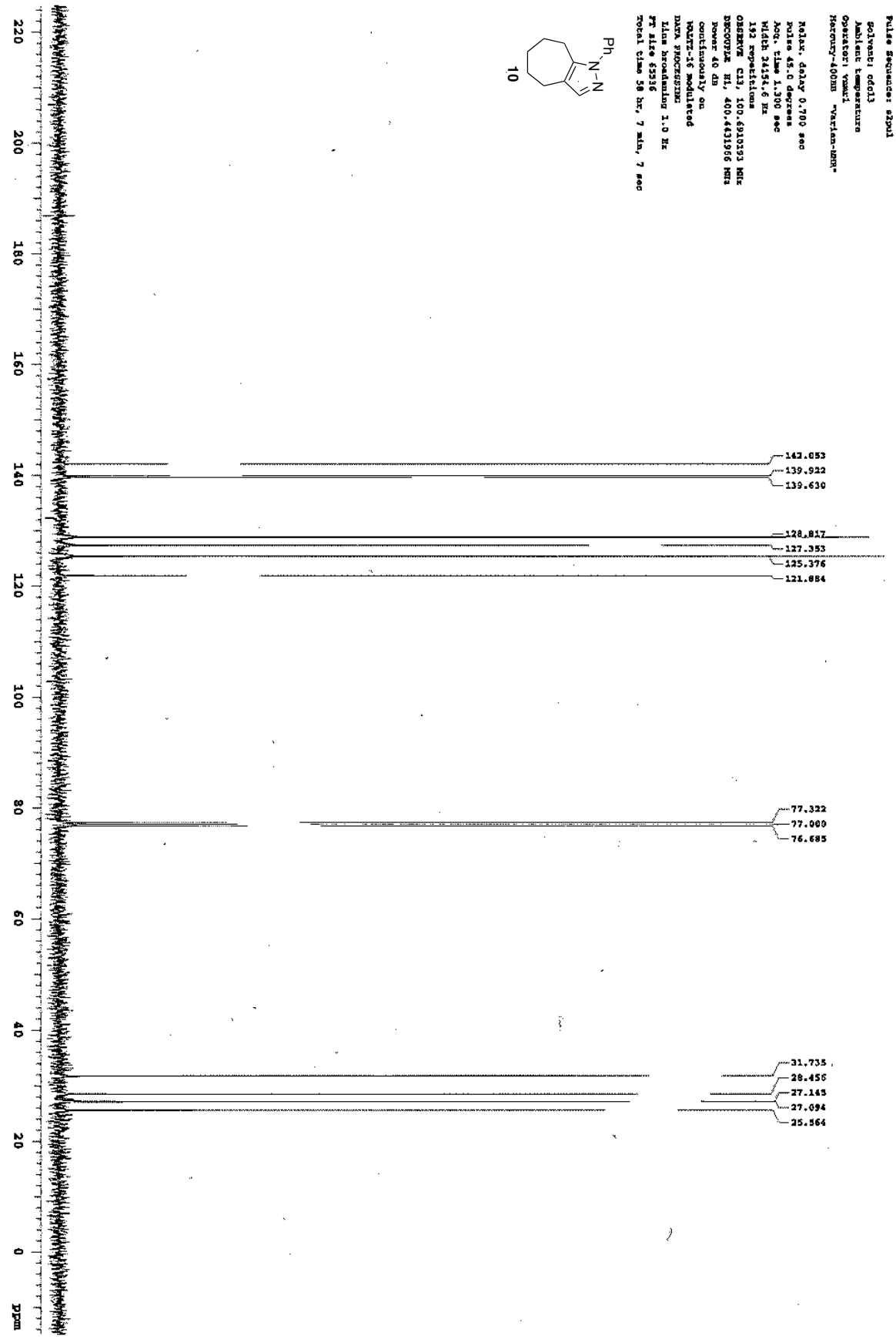

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-1HNB"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411532 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
8
NH2
7.917
7.260
5.175
2.761
2.746
2.732
2.693
2.616
2.576
2.564
2.560
2.548
1.837
1.823
1.809
1.796
1.780
1.733
1.667
1.663
1.652
1.638
1.625
1.614
1.610
1.598
1.583
1.570
1.556
1.542
1.227
1.00
2.03
2.23
2.24
2.24
2.13
2.30
ppm

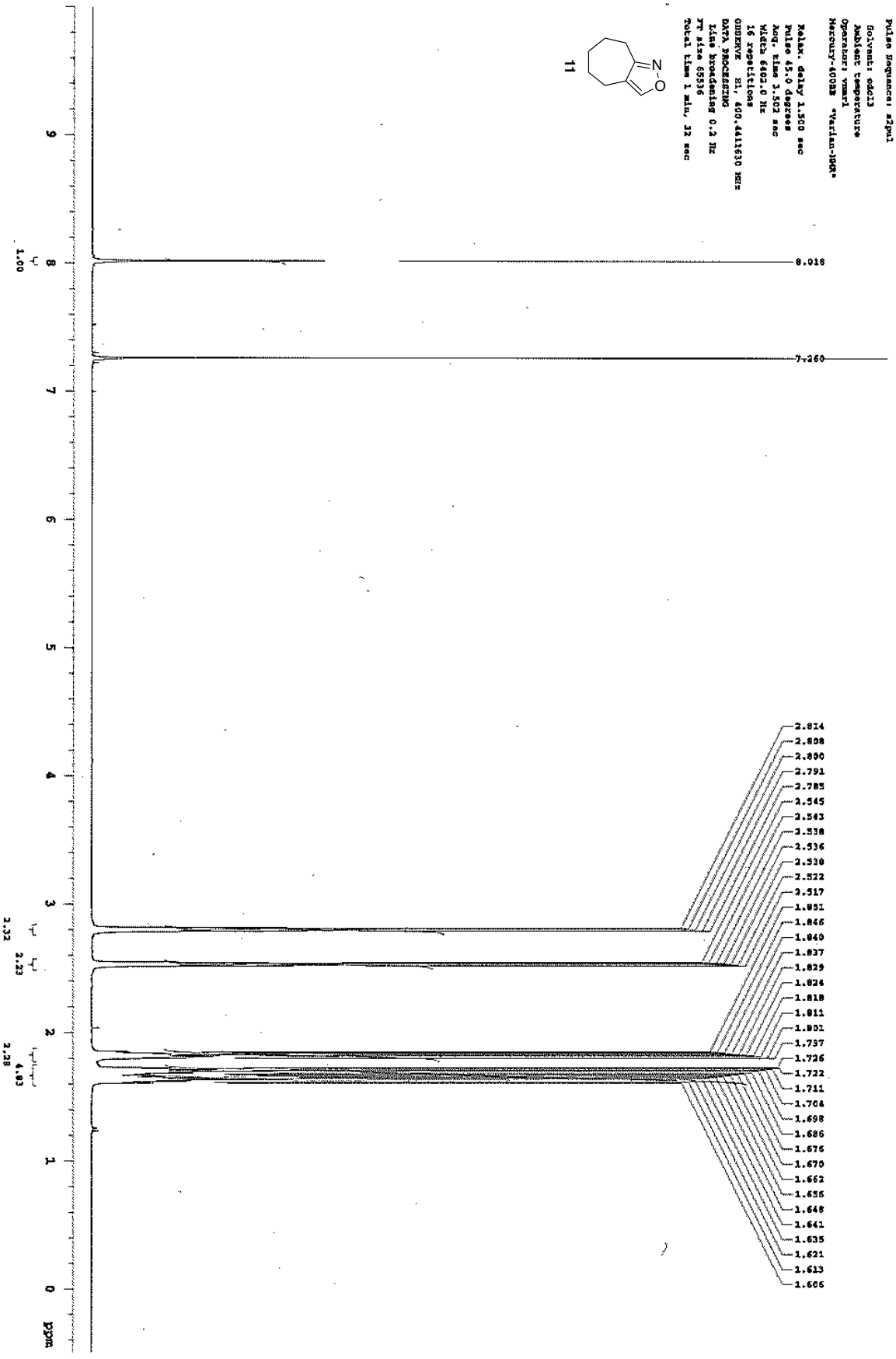

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmkl
Mercury-400BB "varian-2KH"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.5 Hz
384 repetitions
OBSERVE C13, 100.6930157 MHz
DECOUPLE H1, 400.4431965 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 5811 hr, 48 min, 42 sec
8
172.478
161.533
156.702
124.907
77.322
77.000
76.685
39.917
32.314
31.803
28.288
25.923
220
200
180
160
140
120
100
80
60
40
20
0

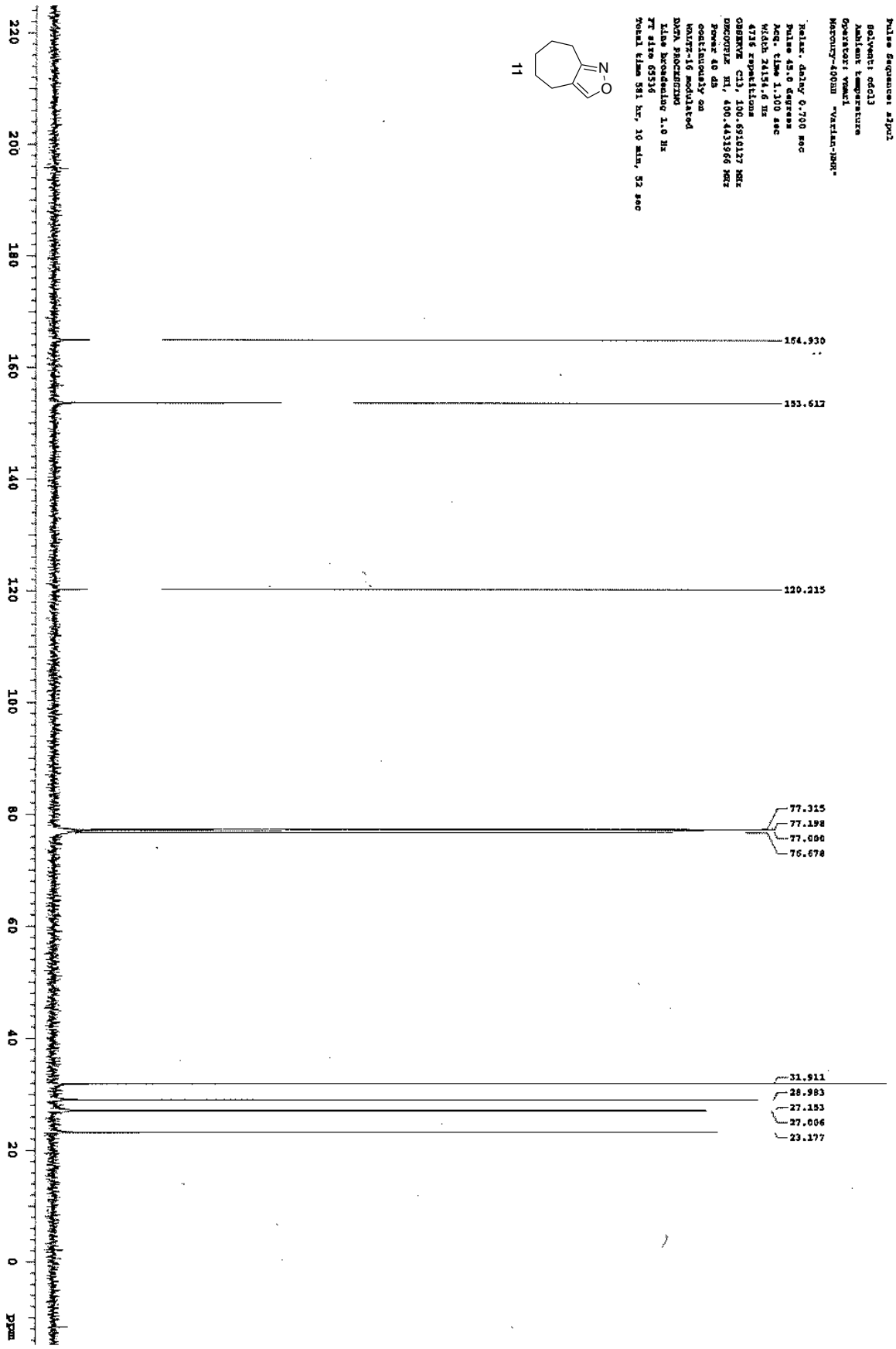

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "Varian-MR6"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6602.0 Hz
16 repetitions
OBSERVE H1, 400.4411530 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
9
8.851
8.366
7.260
2.995
2.981
2.967
2.759
2.744
2.731
1.925
1.911
1.896
1.882
1.867
1.731
1.727
1.716
1.702
1.695
1.689
1.680
1.673
1.667
1.653
1.643
1.638
0.97
1.00
2.12
2.14
2.68
4.32
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmsrl
Mercury-400BB "Varian-MMR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1624 repetitions
OBSERVE C13, 100.6910127 MHz
DECOUPLE H1, 400.4433566 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 58 hr, 7 min, 7 sec
9
171.292
156.321
155.457
135.486
77.322
77.000
76.685
38.998
32.277
31.889
27.423
25.732
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-NMR"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
Ph
N
N
10
7.473
7.471
7.466
7.455
7.452
7.439
7.434
7.428
7.389
7.384
7.379
7.374
7.365
7.364
7.353
7.344
7.341
7.338
7.260
2.789
2.775
2.761
2.645
2.631
2.617
1.883
1.875
1.867
1.855
1.848
1.841
1.832
1.826
1.726
1.718
1.712
1.705
1.697
1.689
1.681
1.666
1.659
1.651
1.643
1.638
1.631
1.623
1.96
3.84
2.00
2.00
2.06
5.02
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmrl
Mercury-400BB "varian-mrm"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
192 repetitions
OBSERVE C13, 100.6910193 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 58 hr, 7 min, 7 sec
Ph
N
N
10
143.653
139.922
139.630
128.817
127.353
125.376
121.884
77.322
77.000
76.685
31.735
28.456
27.143
27.094
25.564
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "Varian-400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 6402.0 Hz
16 repetitions
OBSERVE H1, 400.4411630 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 32 sec
11
8.918
7.260
2.814
2.808
2.800
2.791
2.785
2.545
2.543
2.538
2.536
2.530
2.522
2.517
1.851
1.846
1.840
1.837
1.829
1.824
1.818
1.811
1.801
1.737
1.726
1.722
1.711
1.704
1.698
1.686
1.676
1.670
1.662
1.656
1.648
1.641
1.635
1.621
1.613
1.605
1.00
2.32
2.23
4.03
2.28
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr1
Mercury-400BB "varian-bbs"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
4736 repetitions
OBSERVE C13, 100.6310127 MHz
DECOUPLE H1, 400.4431966 MHz
Power 40 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 581 hr, 10 min, 52 sec
11
N
O
164.930
153.612
120.215
77.315
77.198
77.080
76.678
31.911
28.983
27.153
27.006
23.177
220
200
180
160
140
120
100
80
60
40
20
0
ppm