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Supporting Information

to accompany

Isomerization-Cyclization Approach to the Synthesis of 2-Hydroxy-5-alkylidene-cyclopent-2-enones

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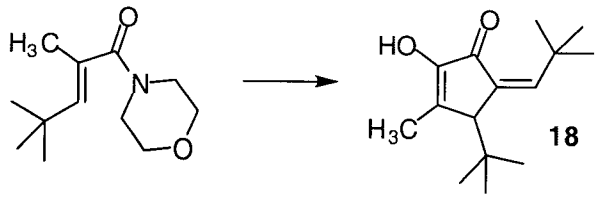

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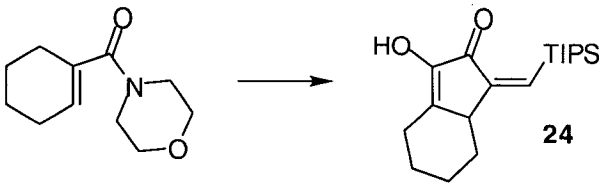
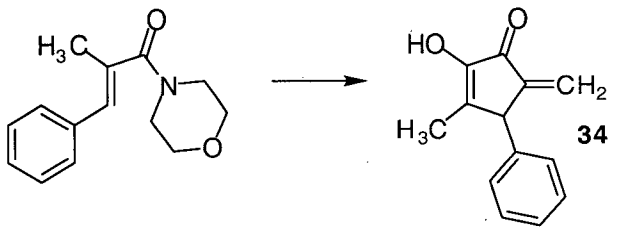
To a solution of ether **13** (2.23 mmol, 250 mg) in THF (3 ml) at -78° C was added *sec*-BuLi (1.64 ml, 2.13 mmol, 1.30 M in hexanes). After 15 min, a solution of 2,4,4-trimethyl-1-morpholin-4-yl-pent-2-en-1-one (0.71 mmol, 150 mg) in THF (1 ml) was added via cannula. After 30 min, the reaction mixture was quenched with pH 7 buffer. The reaction mixture was diluted with water and Et_2O. The aqueous phase was extracted with Et_2O (3 x) and the combined organic phases were washed with brine (1 x), dried over $MgSO_4$, and concentrated. The crude product was loaded onto a silica gel column (1% EtOAc in hexanes). After 8 h, the column was eluted with 1 – 2.5% EtOAc in hexanes to give 4-*tert*-butyl-5-(2,2-dimethyl-propylidene)-2-hydroxy-3-methyl-cyclopent-2-enone **18** (136 mg, 81% yield) as a pink solid: mp 133-136°C; R_f= 0.63 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.14 (s br, 1H), 5.89 (s br, 1H), 2.69 (s, 1H), 2.03 (s, 3H), 1.26 (s, 9H), 0.91 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.0, 153.0, 149.5, 138.3, 134.8, 56.6, 36.0, 33.5, 30.3, 29.2, 16.0; IR (neat) 3320 (br), 2960, 1680, 1615, 1410, 1350, 1195, 1095 cm^{-1}; EIMS *m/z* 232 (M^+, 2), 181 (12), 180 (100), 166 (15), 165 (91), 139 (13), 115 (24), 69 (14); HREIMS calcd for $C_{15}H_{24}O_2$ 236.1776, found 236.1765.



24

See procedure for cyclopentenone **18**: ether **14** (1.37 mmol, 350 mg); *sec*-BuLi (1.00 ml, 1.3 mmol, 1.30 M in hexanes); cyclohex-1-enyl-morpholin-4-yl-methanone (0.50 mmol, 97 mg); gave 3-hydroxy-1-[(triisopropyl-silanyl)-methylene]-1,4,5,6,7,7a-hexahydro-inden-2-one **24** (141 mg, 88% yield) as a white solid: mp 32-37°C; R_f = 0.59 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.07 (d, J = 0.9 Hz, 1H), 5.69 (s br, 1H), 3.00-2.94 (m, 1H), 2.87 (dd, J = 11.7, 5.5 Hz, 1H), 2.28 (m, 1H), 2.09 (ddd, J = 12.7, 5.5, 1.5 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.53 – 1.47 (m, 1H), 1.47 – 1.36 (m, 3H), 1.34 – 1.25 (m, 2H), 1.05 (d, J = 3.3 Hz, 12H), 1.02 (d, J = 3.3 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.6, 153.2, 147.9, 143.0, 134.3, 42.8, 33.2, 25.5, 25.4, 25.1, 19.3, 12.4; IR (neat) 3330 (br), 2930, 1680, 1655, 1615, 1400, 740 cm^{-1}; EIMS *m/z* 277 (M^+-isopropyl, 100), 145 (14), 117 (11), 115 (10), 75 (10), 74 (16), 69 (13), 68 (30); HREIMS calcd for $C_{19}H_{32}O_2Si$ (M^+ - isopropyl) 277.1624, found 277.1618.



To alkyne **28** (252 mg, 2.52 mmol) in THF (8 mL) at -78° C was added *sec*-BuLi (4.0 mL, 1.30 M, 5.04 mmol). The reaction mixture was stirred for 30 min and then a solution of 2-methyl-1-morpholin-4-yl-3-phenyl-propenone **2** (232 mg, 1.00 mmol) in THF (2 mL) was added via cannula and stirred at this temperature for an additional 30 min. The reaction was then quenched with pH 7 buffer and the aqueous phase was extracted with Et_2O (3 x). The combined organic extracts were washed with brine (1 x), dried over K_2CO_3, and concentrated. The crude product was loaded onto a silica gel column (1% EtOAc in hexanes) and allowed to stand for 12 h before it was eluted to give 2-hydroxy-3-methyl-5-methylene-4-phenyl-cyclopent-2-enone **34** (130 mg, 65% yield) as a white solid: mp 158-161°C; R_f = 0.62 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.33 (m, 3H), 7.11 (d, *J* = 6.6 Hz, 2H), 6.14 (d, *J* = 1.5 Hz, 1H), 5.19 (s, 1H), 4.22 (s, 1H), 1.85 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.4, 151.3, 145.0, 140.9, 139.8, 128.8, 128.0, 127.3, 118.3, 50.1, 12.0; IR (neat) 3320, 1685, 1630, 1415, 1360, 1200, 1100, 710 cm^{-1}; EIMS *m/z* 200 (M^+, 100), 142 (41), 129 (61), 128 (41), 115 (67), 88 (44); HREIMS calcd for $C_{13}H_{12}O_2$ 200.0837, found 200.0836.

5-(2,2-Dimethyl-propylidene)-2-hydroxy-3-methyl-4-phenyl-cyclopent-2-enone **16**: mp 186-188°C; R_f = 0.65 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.24 (m, 3H), 7.11 (dm, *J* = 7.1 Hz, 2H), 6.82 (s br, 1H), 5.85 (s br, 1H), 4.09 (s br, 1H), 1.79 (s, 3H), 1.25 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.7, 154.4, 152.0, 141.0, 137.8, 136.1, 128.6, 128.0, 127.0, 51.8, 33.5, 29.6, 11.9; IR (neat) 3335 (br), 2955, 1655, 1610, 1400, 1350, 1095, 695 cm^{-1}; EIMS *m/z* 256 (M^+, 37), 223 (26), 186 (100), 91 (38); HREIMS calcd for $C_{17}H_{20}O_2$ 256.1463, found 256.1464.

5-(2,2-Dimethyl-propylidene)-2-hydroxy-3,4-dimethyl-cyclopent-2-enone **17**: mp 109-114°C; R_f = 0.63 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.26 (s br, 1H), 6.01 (s br, 1H), 2.93 (qm, *J* = 7.1Hz, 1H), 1.94 (s, 3H), 1.27 (s, 9H), 1.16 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.9, 150.9, 150.8, 138.7, 136.3, 39.6, 33.3, 29.8, 18.3, 11.3; IR (neat) 3320 (br), 2960, 1680, 1615, 1410, 1350, 1195, 1095 cm^{-1}; EIMS *m/z* 194 (M^+, 63), 179 (27), 161 (40), 133 (25), 124 (100), 91 (20); HREIMS calcd for $C_{12}H_{18}O_2$ 194.1307, found 194.1290.

1-(2,2-Dimethyl-propylidene)-3-hydroxy-1,4,5,6,7,7a-hexahydro-inden-2-one **19**: mp 155-157°C; R_f = 0.60 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.47 (s br, 1H), 5.98 (s br, 1H), 2.95 (dd, *J* = 13.9, 4.2 Hz, 1H), 2.72 (dd, *J* = 12.7, 5.3 Hz, 1H), 2.14

(m, 1H), 2.03 (ddd, J = 12.7, 5.3, 1.5 Hz, 1H), 1.98 – 1.82 (m, 2H), 1.51 – 1.22 (m, 2H), 1.27 (s, 9H), 0.97 (qd, J = 12.2, 3.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.4, 150.7, 148.1, 141.0, 135.1, 41.7, 33.4, 33.3, 29.7, 25.3, 25.2, 24.9; IR (neat) 3330 (br), 2930, 1685, 1655, 1615, 1400, 1365, 1040 cm^{-1}; EIMS *m/z* 220 (M^+, 93), 187 (45), 150 (100), 122 (40), 91 (42), 79 (45); HREIMS calcd for $C_{14}H_{20}O_2$ 220.1463, found 220.1464.

5-(2,2-Dimethyl-propylidene)-2-hydroxy-3-methyl-cyclopent-2-enone **20**: mp 102-104°C; R_f = 0.65 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.60 (s, 1H), 5.82 (s, 1H), 3.15 (m, 2H), 2.04 (s, 3H), 1.18 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.0, 150.2, 145.2, 137.0, 128.7, 32.7, 31.5, 30.0, 13.8; IR (neat) 3320 (br), 2960, 1680, 1615, 1410, 1350, 1195, 1095 cm^{-1}; EIMS *m/z* 180 (M^+, 100), 165 (48), 139 (22), 119 (21), 117 (24), 110 (38), 91 (18), 81 (18); HREIMS calcd for $C_{11}H_{16}O_2$ 180.1150, found 180.1167.

2-Hydroxy-3-methyl-4-phenyl-5-[(triisopropyl-silanyl)-methylene]-cyclopent-2-enone **21**: mp 33-36°C; R_f = 0.59 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.20 (m, 3H), 7.15 – 7.05 (m, 2H), 5.89 (s, 1H), 5.62 (s, 1H), 4.19 (s, 1H), 1.81 (s, 3H), 1.42 – 1.32 (m, 3H), 0.98 – 0.90 (m, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 153.5, 152.1, 140.7, 140.7, 138.8, 128.8, 128.3, 127.3, 52.8, 19.2, 12.4, 12.2; IR (neat) 3320 (br), 2960, 1680, 1615, 1410, 1350, 1195, 1095 cm^{-1}; EIMS *m/z* 183 (M^+- $C_{10}H_{24}Si$, 100), 155 (44), 141 (34), 103 (23), 101 (29), 89 (24), 75 (71); HREIMS calcd for $C_{19}H_{25}O_2Si$ (M^+ - isopropyl) 313.1624, found 313.1618.

2-Hydroxy-3,4-dimethyl-5-[(triisopropyl-silanyl)-methylene]-cyclopent-2-enone **22**: mp 30-35°C; R_f = 0.56 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.09 (s, 1H), 5.60 (s br, 1H), 3.06 (q, J = 6.9 Hz, 1H), 1.98 (s, 3H), 1.50 – 1.35 (m, 3H), 1.22 (d, J = 6.9 Hz, 3H), 1.04 (d, J = 7.5 Hz, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.3, 154.3, 151.0, 141.5, 134.5, 40.6, 19.3, 18.0, 12.4, 11.7; IR (neat) 3330 (br), 2930, 1680, 1615, 1410, 1350, 1195, 1095, 740 cm^{-1}; EIMS *m/z* 294 (M^+, 4), 253 (22), 252 (76), 251 (100), 183 (22), 131 (44), 103 (37), 75 (64); HREIMS calcd for $C_{17}H_{30}O_2Si$ 294.2015, found 294.2018.

4-*tert*-Butyl-2-hydroxy-3-methyl-5-[(triisopropyl-silanyl)-methylene]-cyclopent-2-enone **23**: mp 31-34°C; R_f = 0.58 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.93 (s, 1H), 5.91 (s, 1H), 2.88 (s, 1H), 2.08 (s, 3H), 1.45 – 1.30 (m, 3H), 1.05 (d, J = 7.3 Hz, 12H), 1.01 (d, J = 7.3 Hz, 6H), 0.96 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.7, 152.7, 152.3, 140.5, 133.7, 57.5, 35.7, 29.2, 19.4, 16.0, 12.5; IR (neat) 3330 (br), 2930, 1680, 1615, 1410, 1350, 1195, 1095, 740 cm^{-1}; EIMS *m/z* 238 (11), 237 (40), 236 (10), 183 (8), 131 (9), 103 (8), 75 (23); HREIMS calcd for $C_{17}H_{29}O_2Si$ (M^+ - isopropyl) 293.1937, found 293.1919 .

3-Hydroxy-1-[(triisopropyl-silanyl)-methylene]-1,4,5,6,7,7a-hexahydro-inden-2-one **24**: mp 32-37°C; R_f = 0.56 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.07 (d, J = 0.9 Hz, 1H), 5.69 (s br, 1H), 3.00 – 2.94 (m, 1H), 2.87 (dd, J = 11.7, 5.4 Hz, 1H), 2.28 (m, 1H), 2.09 (ddd, J = 12.7, 5.7, 1.5 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.53 – 1.47 (m, 1H), 1.47 – 1.36 (m, 3H), 1.34 – 1.25 (m, 2H), 1.05 (d, J = 3.3 Hz, 12H), 1.02 (d, J = 3.3

Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.6, 153.2, 147.9, 143.0, 134.3, 42.8, 33.2, 25.5, 25.4, 25.1, 19.3, 12.4; IR (neat) 3330 (br), 2930, 1680, 1655, 1615, 1400, 740 cm^{-1}; EIMS *m/z* 277 (M^+- isopropyl, 100), 145 (14), 117 (11), 115 (10), 75 (10), 74 (16), 69 (13), 68 (30); HREIMS calcd for $C_{16}H_{25}O_2Si$ (M^+ - isopropyl) 277.1624, found 277.1618.

2-Hydroxy-3-methyl-5-[(triisopropyl-silanyl)-methylene]-cyclopent-2-enone **25**: 36-38°C; R_f = 0.60 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.10 (s, 1H), 5.53 (s br, 1H), 3.02 (s, 2H), 2.01 (s, 3H), 1.45 – 1.35 (m, 3H), 1.04 (d, *J* = 7.5 Hz, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.6, 151.6, 148.0, 137.4, 136.1, 36.8, 19.3,13.8, 12.4; IR (neat) 3330 (br), 2930, 1680, 1615, 1410, 1350, 1195, 1095, 740 cm^{-1}; EIMS *m/z* 237 (M^+ - isopropyl, 70), 197 (62), 183 (100), 155 (43), 141 (43), 101 (30), 89 (32), 75 (91); HREIMS calcd for $C_{13}H_{21}O_2Si$ (M^+ - *iso*-propyl) 237.1311, found 237.1307.

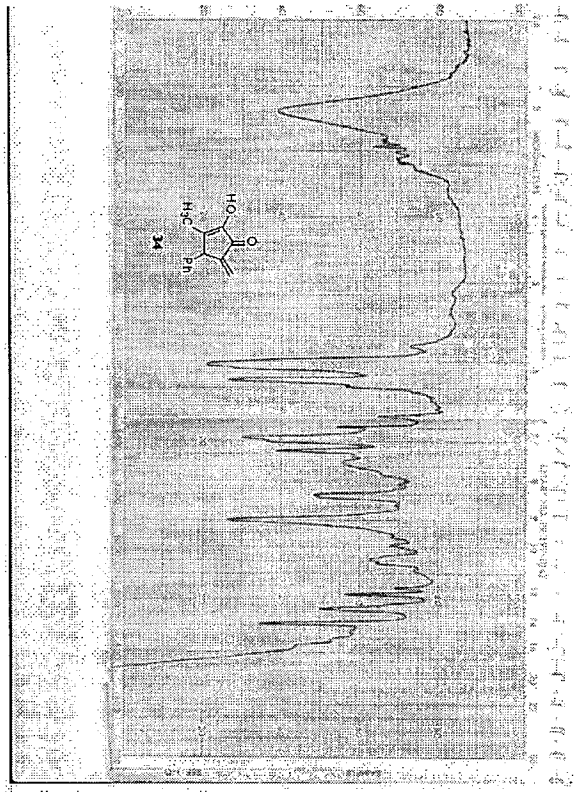
5-Benzylidene-2-hydroxy-3,4-dimethyl-cyclopent-2-enone **26**: mp >180°C dec.; R_f = 0.64 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.07 – 8.04 (m, 2H), 7.44 – 7.36 (m, 3H), 6.72 (s, 1H), 5.63 (s br, 1H), 3.18 (q, *J* = 7.0 Hz, 1H), 2.05 (s, 3H), 1.32 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.7, 150.6, 139.8, 138.4, 131.5, 131.2, 128.7, 128.2, 127.1, 48.8, 14.4, 11.5; IR (neat) 3275 (br), 1670, 1620, 1400, 1185, 1120, 690 cm^{-1}; EIMS *m/z* 214 (M^+, 100), 171 (73), 153 (32), 143 (33), 128 (52), 115 (45); HREIMS calcd for $C_{14}H_{14}O_2$ 214.0994, found 214.0989.

5-Benzylidene-2-hydroxy-3-methyl-4-phenyl-cyclopent-2-enone **27**: mp >200°C dec.; R_f = 0.65 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 – 7.91 (m, 2H), 7.40 – 7.21 (m, 5H), 7.20 – 7.16 (m, 3H), 6.49 (s, 1H), 5.82 (s, 1H), 4.25 (s, 1H), 1.84 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.8, 150.8, 140.9, 138.4, 135.4, 132.5, 132.4, 128.9 (2C); 128.7, 128.4, 128.3, 126.8, 46.3, 14.2; IR (neat) 3320 (br), 2960, 1680, 1615, 1410, 1350, 1195, 1095 cm^{-1}; EIMS *m/z* 276 (M^+, 100), 258 (24), 233 (54), 229 (28), 215 (37), 128 (23), 115 (29), 105 (27); HREIMS calcd for $C_{19}H_{16}O_2$ 276.1150, found 276.1160.

5-But-3-enylidene-2-hydroxy-3-methyl-4-phenyl-cyclopent-2-enone **31**: (*E / Z = 17 / 83*); *Z* Isomer: mp >140°C dec.; R_f = 0.60 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.22 (m, 3H), 7.10 – 7.05 (m, 2H), 6.50 – 6.30 (s br, 1H), 5.80 – 5.66 (m, 2H), 4.96 (dd, *J* = 3.6, 1.5 Hz, 1H), 4.91 (dd, *J* = 3.6, 1.5 Hz, 1H), 4.09 (s, 1H), 3.68 – 3.57 (m, 1H), 3.46 – 3.35 (m, 1H), 1.77 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.8, 151.8, 140.3, 139.6, 139.0, 136.7 (2C), 128.8, 128.0, 127.2, 116.0, 50.5, 31.8, 11.9; IR (neat) 3330, 1690, 1630, 1400 cm^{-1}; EIMS *m/z* 240 (M^+, 29), 183 (16), 167 (12), 91 (18), 69 (100); HREIMS calcd for $C_{16}H_{16}O_2$ 240.1150, found 240.1179.

2-Hydroxy-3,4-dimethyl-5-methylene-cyclopent-2-enone **32**: mp 87-89°C; R_f = 0.57 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.82 (s, 1H), 6.11 (d, *J* = 1.3 Hz, 1H), 5.38 (s, 1H), 3.13 (qm, *J* = 7.1 Hz, 1H), 2.02 (s, 3H), 1.25 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.2, 150.5, 145.9, 143.8, 116.0, 38.1, 17.4, 11.6; IR (neat) 3300, 2970, 1680, 1625, 1410, 1400, 1360, 1200, 1100 cm^{-1}; EIMS *m/z* 138 (M^+, 7), 136 (7), 124 (6), 123 (17), 119 (100), 95 (8); HREIMS calcd for $C_8H_{10}O_2$ 138.0681, found 138.0676.

3-Hydroxy-1-methylene-1,4,5,6,7,7a-hexahydro-inden-2-one **33**: mp 118-123°C; R_f = 0.56 (20% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.08 (s, 1H), 6.02 (s, 1H), 5.38 (s, 1H), 3.05 – 2.92 (m, 2H), 2.24 (m, 1H), 2.14 – 1.80 (m, 3H), 1.53 (qt, J = 13.4, 3.2 Hz, 1H), 1.03 (qdd (J = 12.9, 4.2, 3.4 Hz, 1H), 1.07 (qd, J = 12.0, 3.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.2, 147.7, 145.8, 144.6, 115.8, 40.5, 32.8, 25.6, 25.2, 24.9; IR (neat) 3310, 2940, 1680, 1645, 1400, 1205, 1100, 1075 cm^{-1}; EIMS *m/z* 164 (M^+, 100), 136 (39), 135 (33), 108 (34), 93 (38), 79 (50), 77 (30); HREIMS calcd for $C_{10}H_{12}O_2$ 164.0837, found 164.0819.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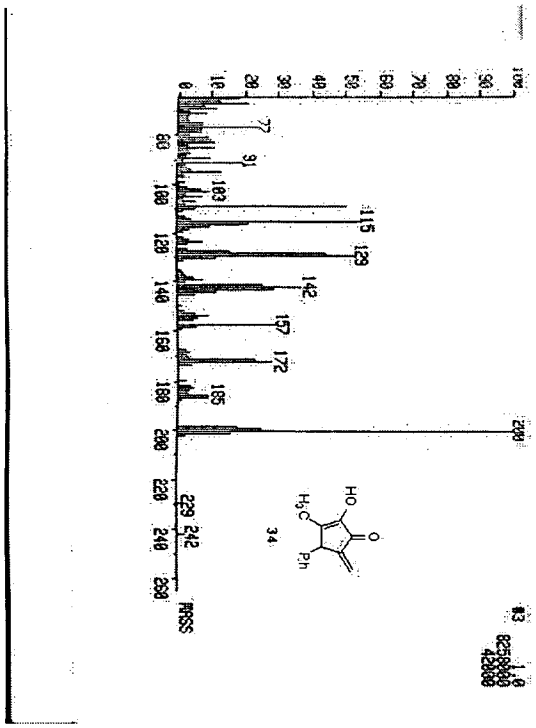
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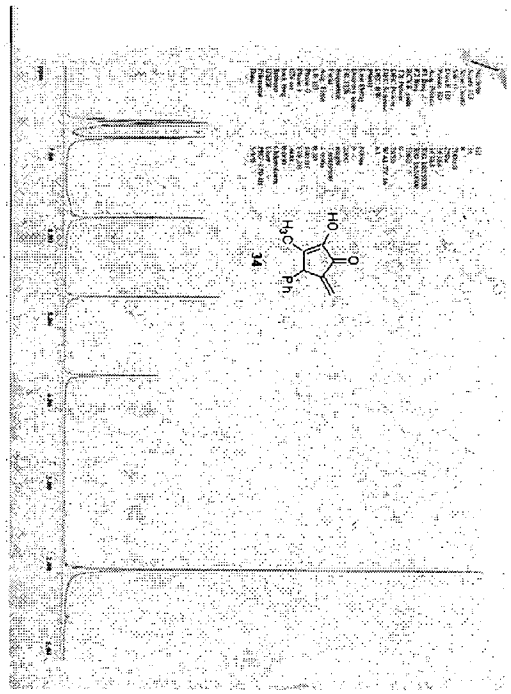
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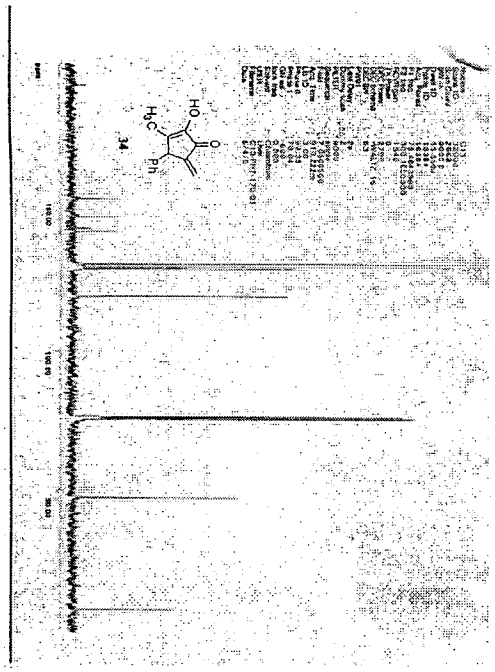
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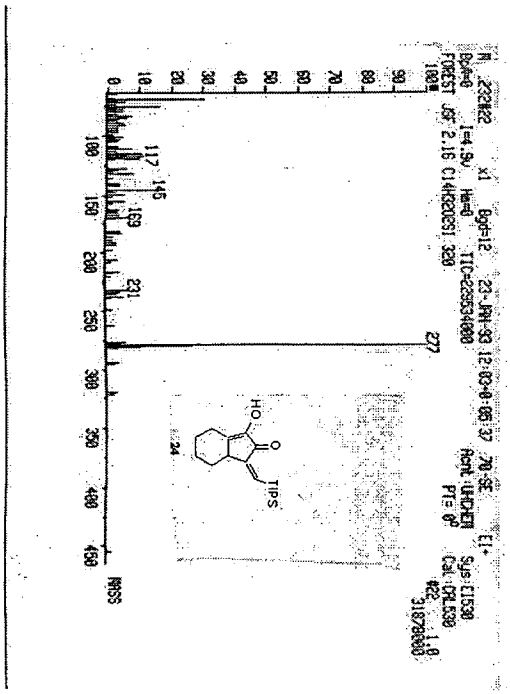
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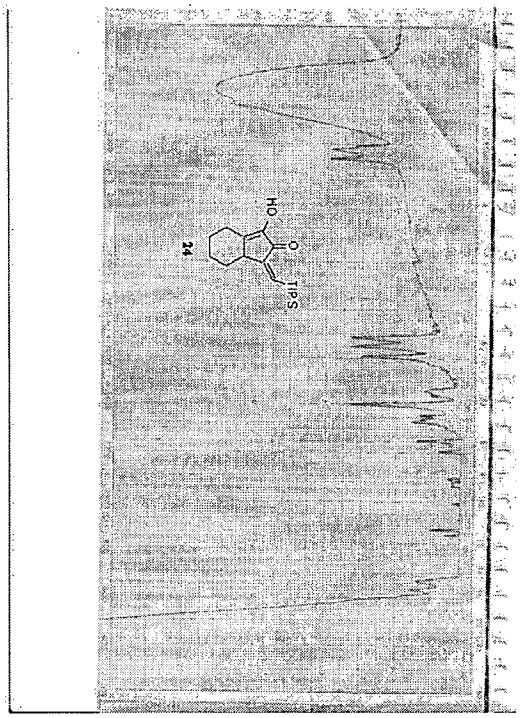
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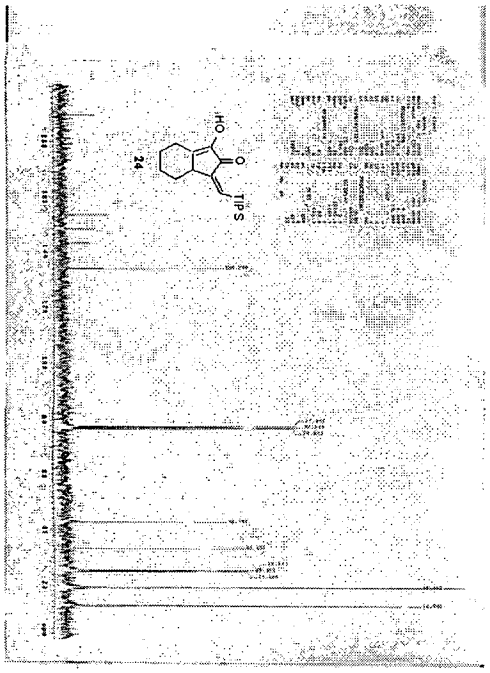
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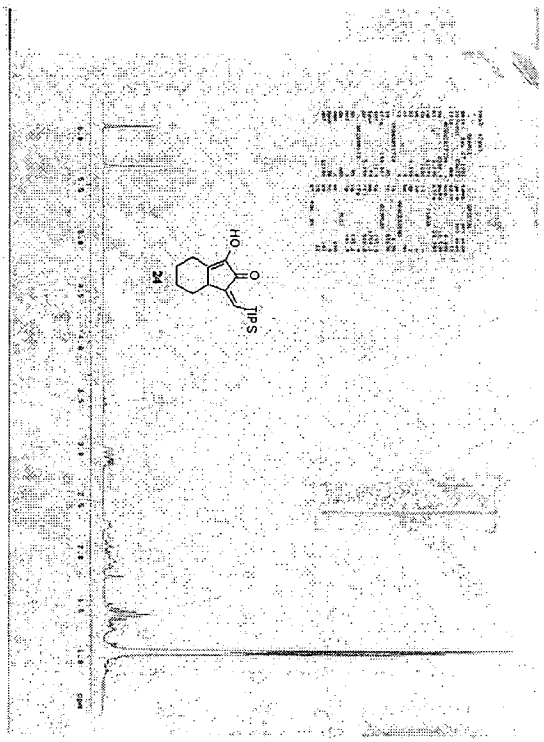
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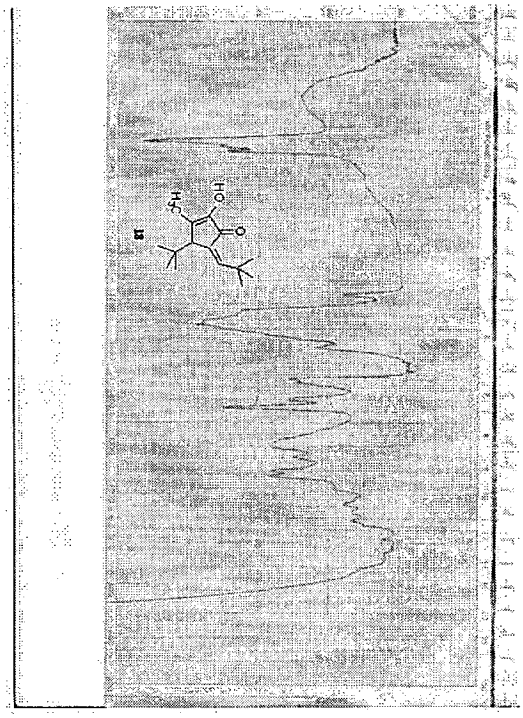
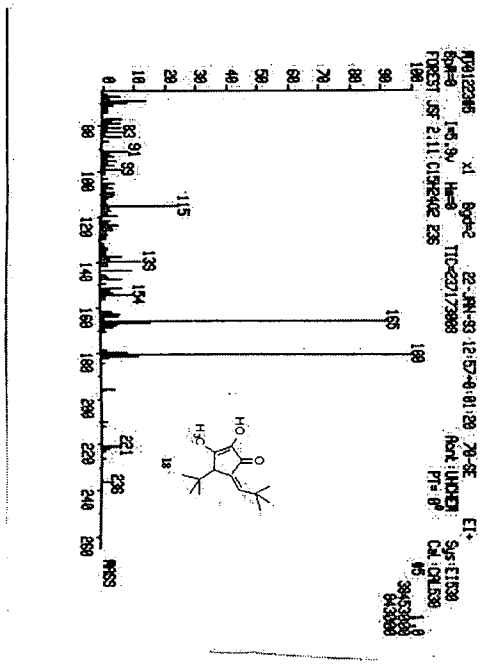
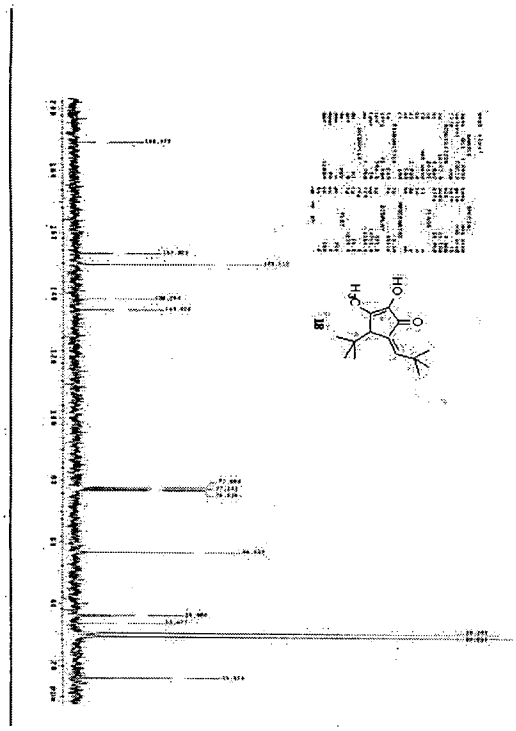
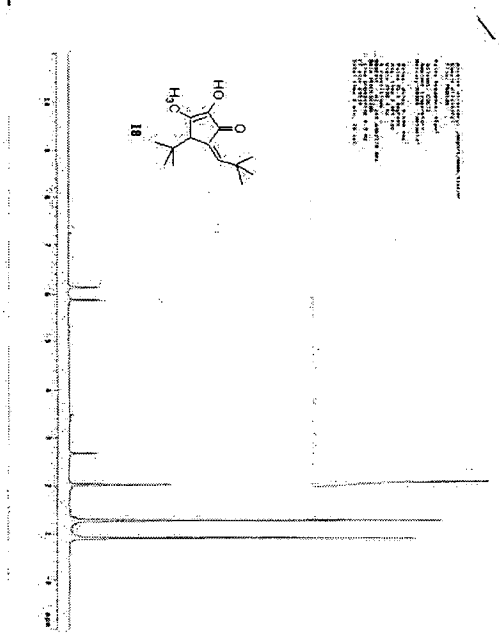
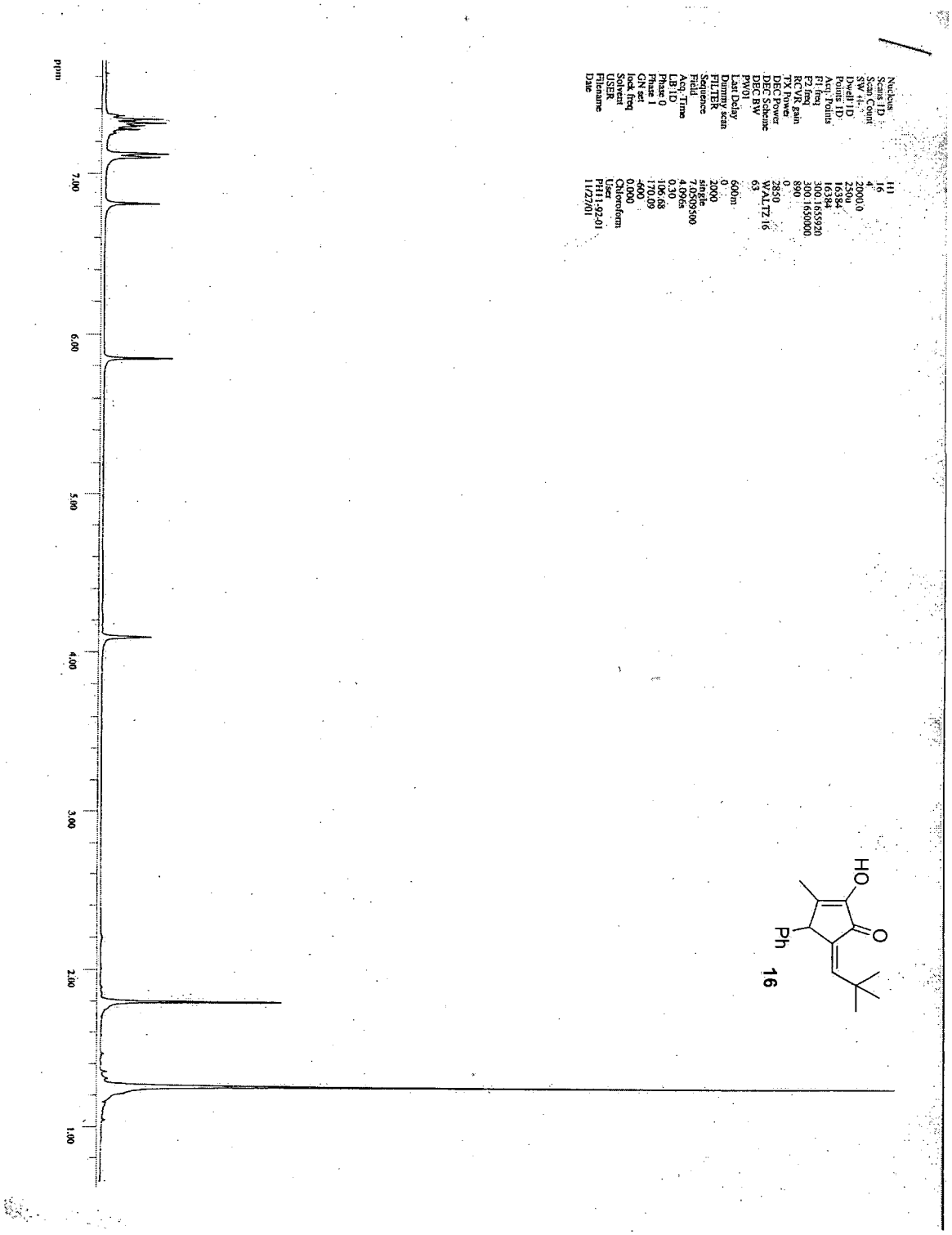
TIPS
24

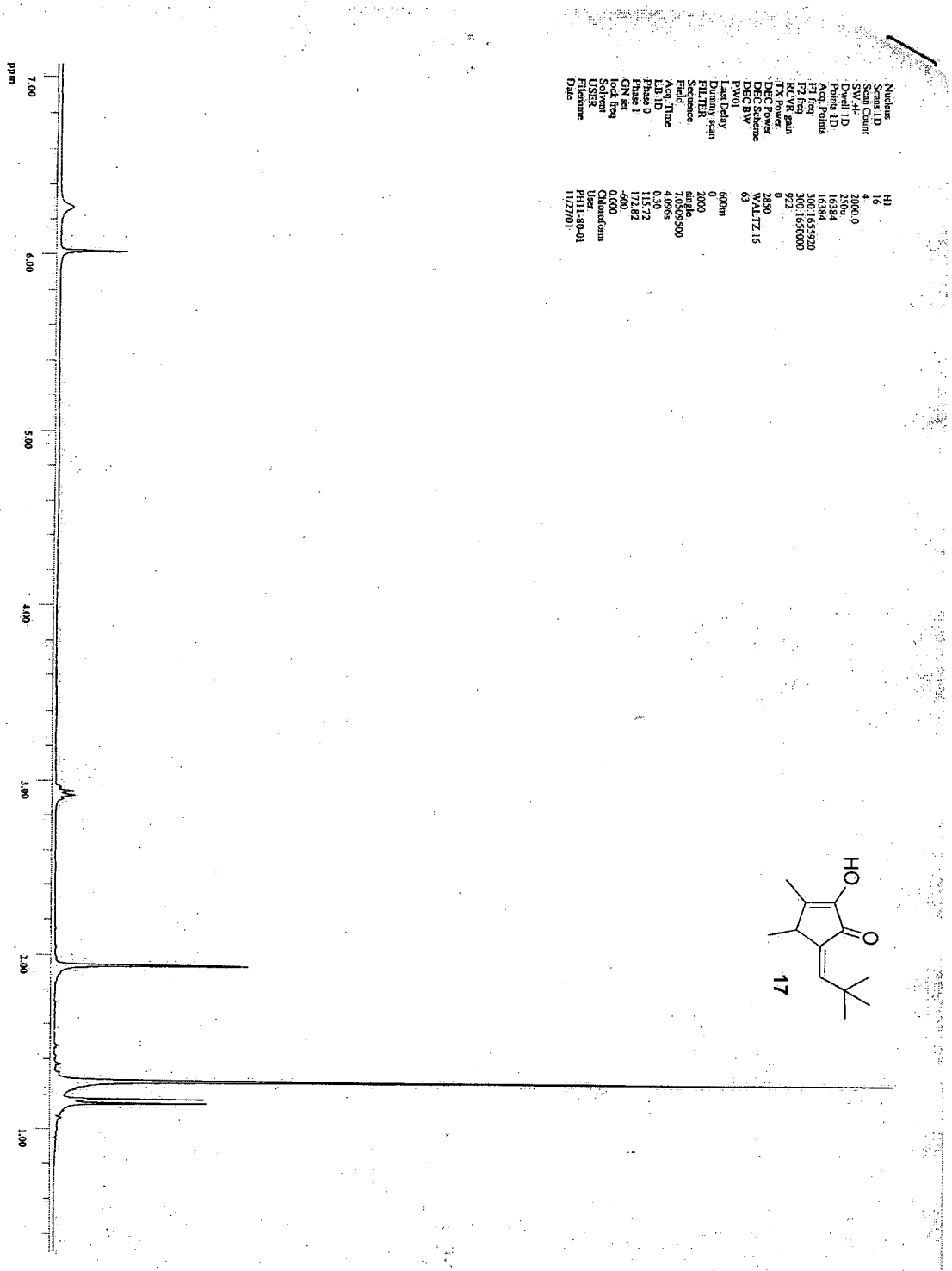


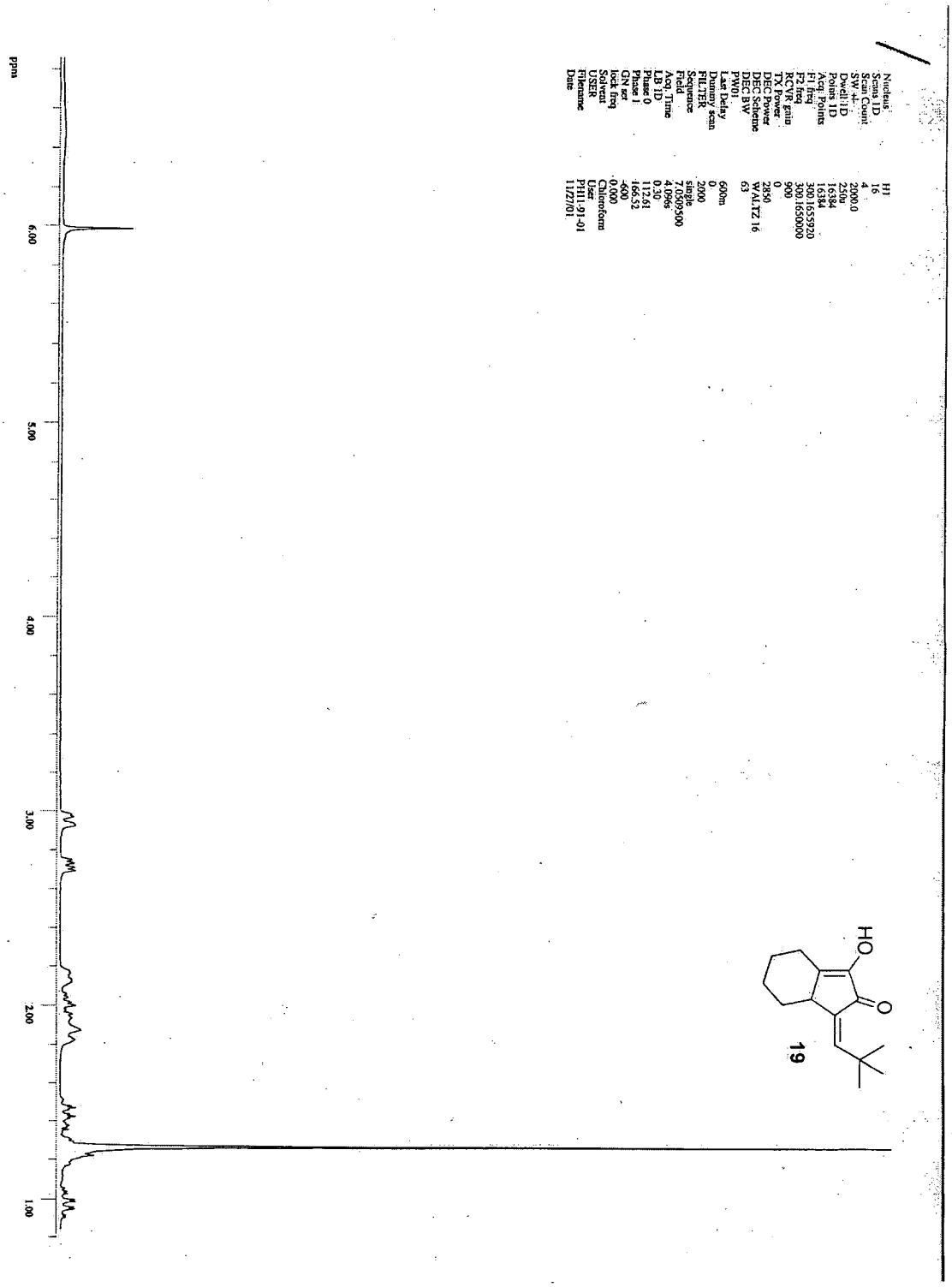


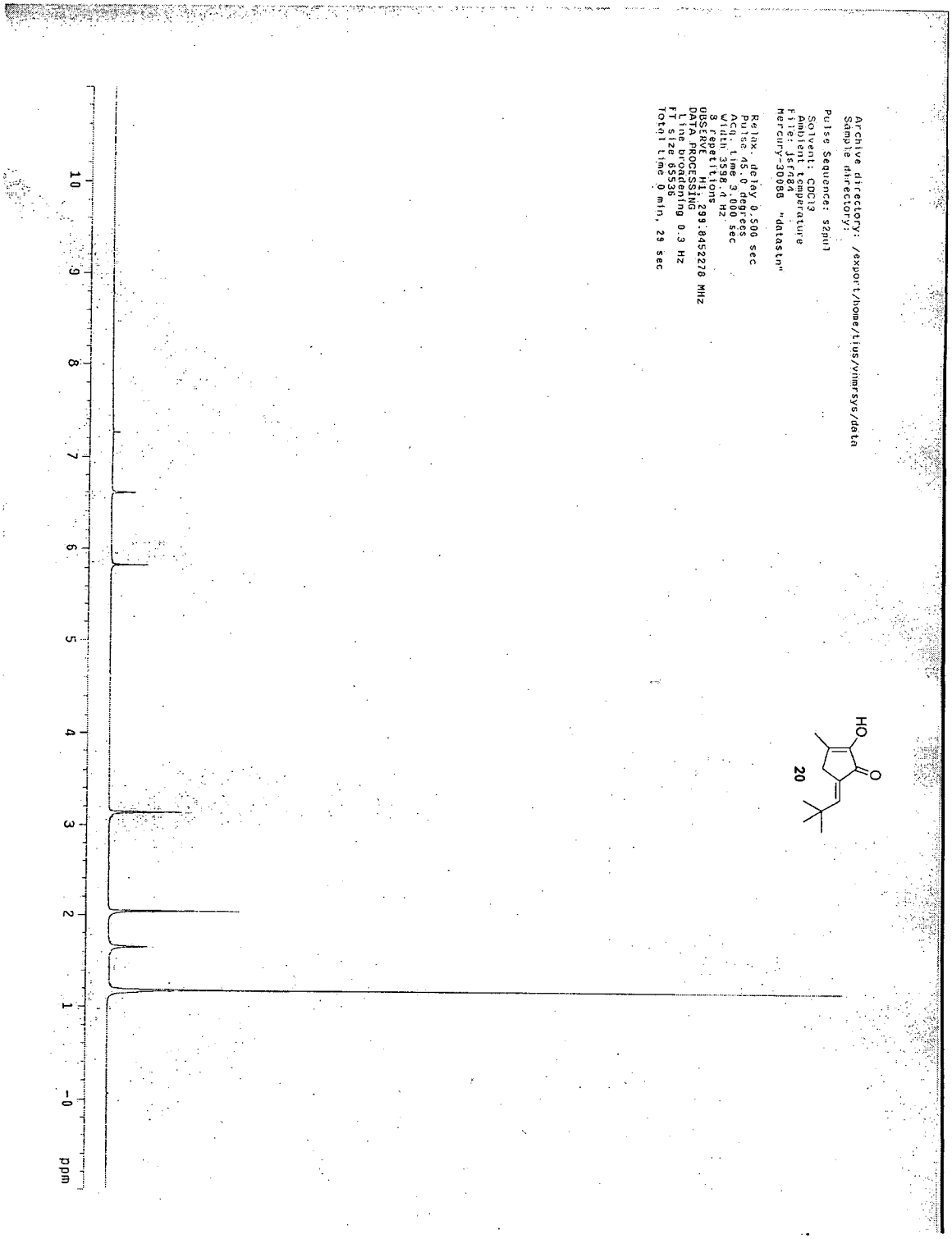


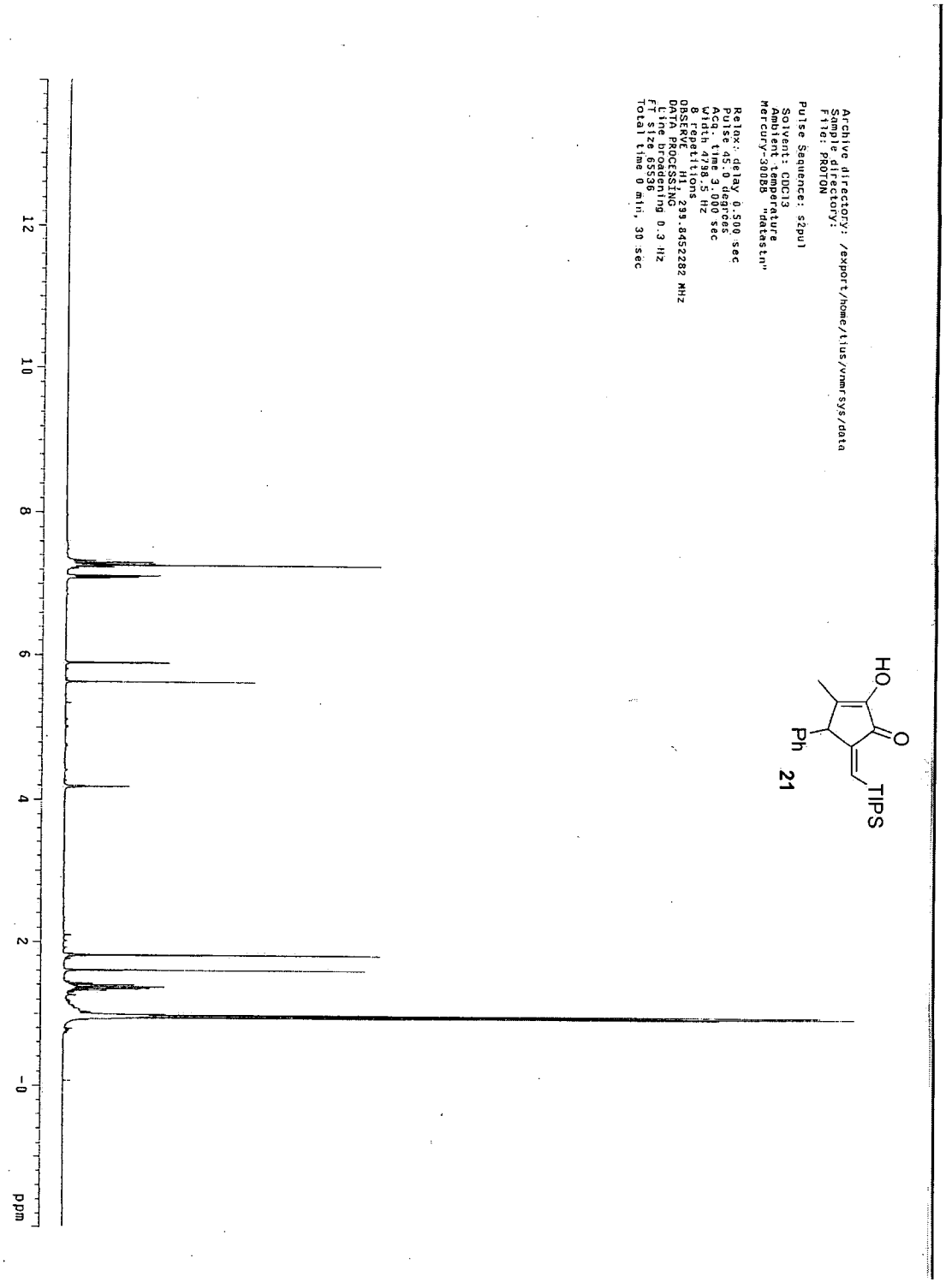


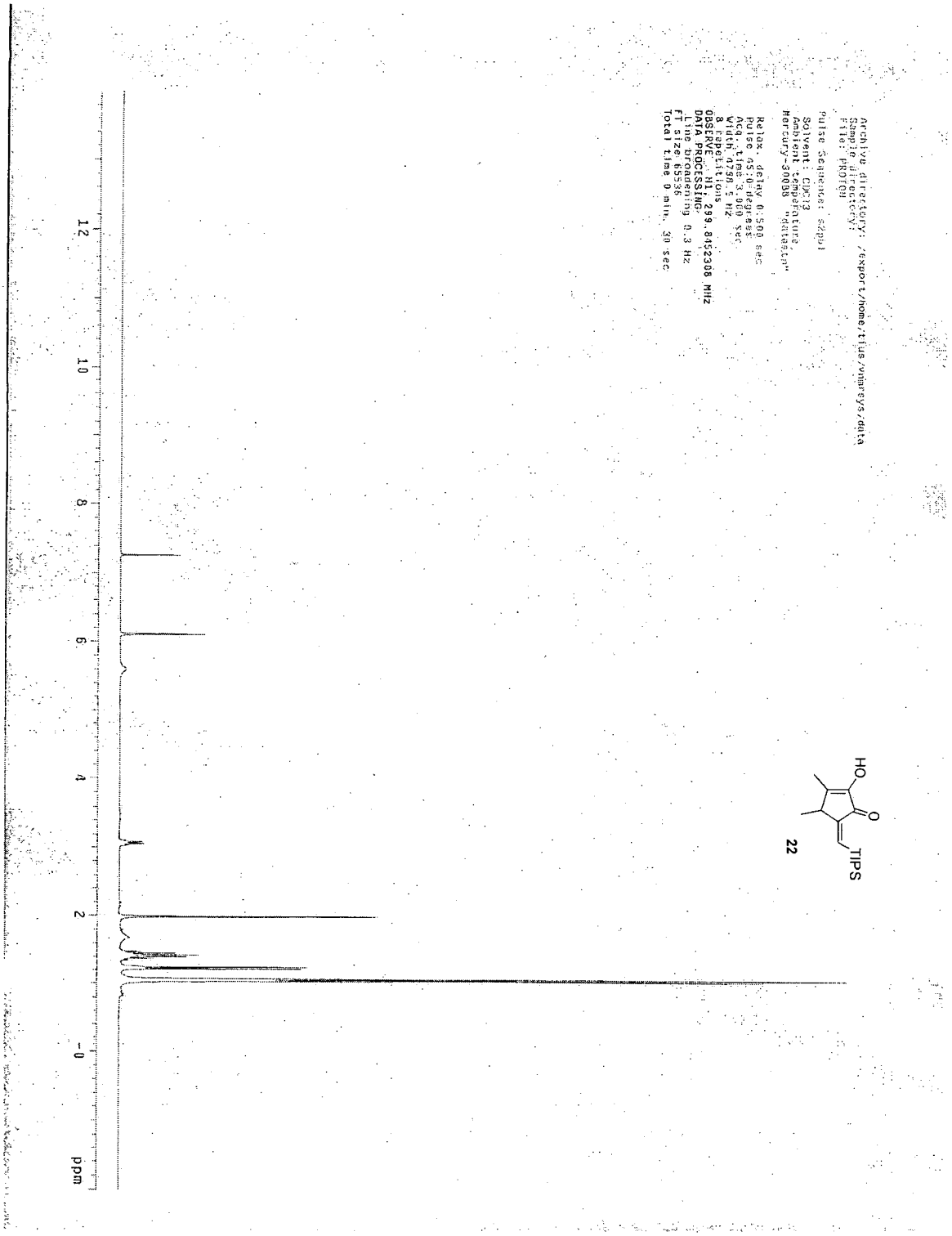

Nucleus H1
Scans ID 16
Solvent CDCl3
SW 2000.0
Dwell 1D 250u
Points 1D 16384
Acq. Points 16384
F1 freq 300.1655920
F2 freq 300.1650800
RCVR gain 890
TX Power 0
DEC Power 2850
DEC Scheme WALTZ16
DEC BW 65
PW90 600n
Last Delay 0
Dummy scan 2000
FILTER single
Sequence 7.0509500
Field 4.096s
Acq. Time 0.30
LB 1D 106.68
Phase 0 170.09
Phase 1 -600
ON set 0.000
Solvent Chloroform
USER User
Filename PH11-92-01
Date 11/27/01
HO
O
Ph
16
ppm
7.00
6.00
5.00
4.00
3.00
2.00
1.00

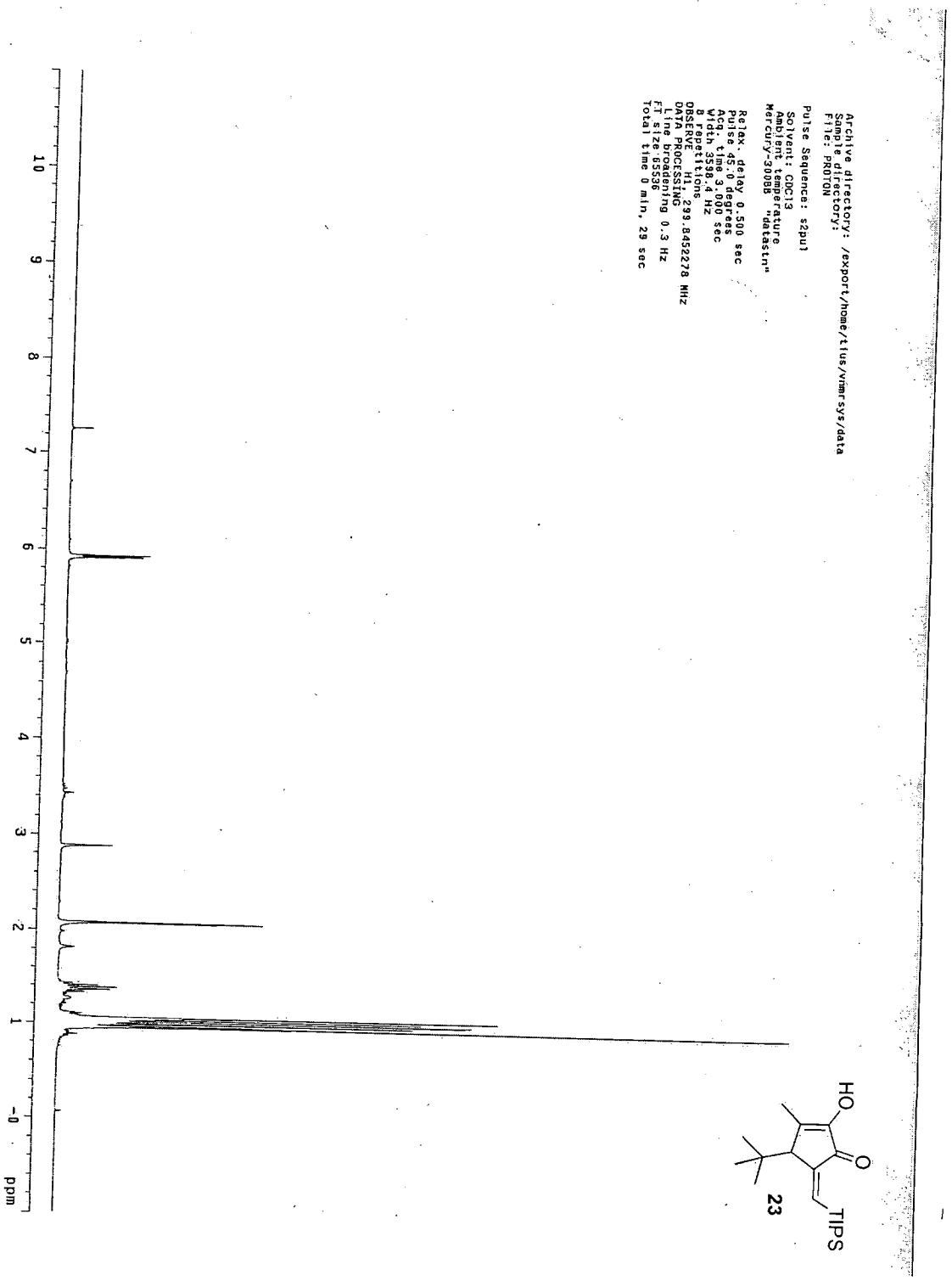

Nucleus H1
Scan ID 16
Scan Count 4
SW 4+ 2000.0
Dwell 1D 250u
Points 1D 16384
Acq. Points 16384
P1 freq 300.1655920
RCVR gain 300.1650000
TX Power 922
DEC Power 0
DEC Scheme 2850
DEC BW WALTZ16
PW01 63
Last Delay 600m
Dummy scan 0
FILTER 2000
Sequence single
Field 7050500
Acq. Time 4.0960s
D1 0.00s
Phase 0 115.72
Phase 1 172.82
GN set -600
lock freq 0.000
Solvent Chloroform
USER User
Filename PH11-80-01
Date 11/27/01
7.00
6.00
5.00
4.00
3.00
2.00
1.00
ppm
HO
O
17

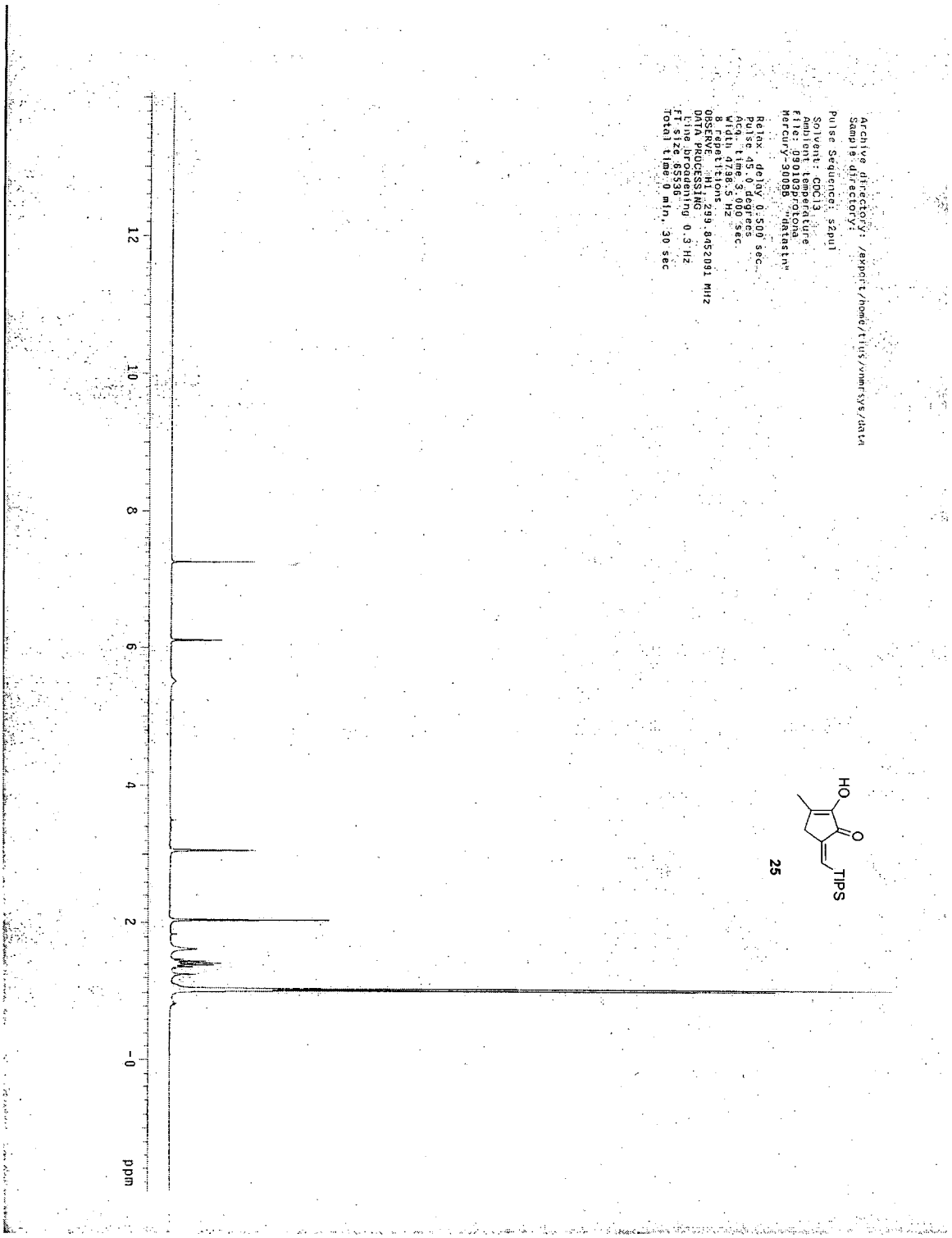

Nucleus 1H
Scans 1D 16
Scans Count 4
SW +/- 2000.0
Dwell 1D 250u
Points 1D 16384
Acq. Points 16384
F1 freq 300.1655920
F2 freq 300.1650000
RCVR gain 900
TX Power 0
DEC Power 2850
DEC Offset w WALTZ 16
DEC BW 63
PW01
Last Delay 600m
Dummy scan 0
FILTER 2000
Sequence single
Field 7.0509500
Acq. Time 4.096s
LB 1D 0.30
Phase 0 112.61
Phase 1 -160.52
GN offset 4000
lock freq -0.000
Solvent Chloroform
USER User
Filename PH11-91-01
Date 11/27/01
HO
O
19
ppm
6.00
5.00
4.00
3.00
2.00
1.00

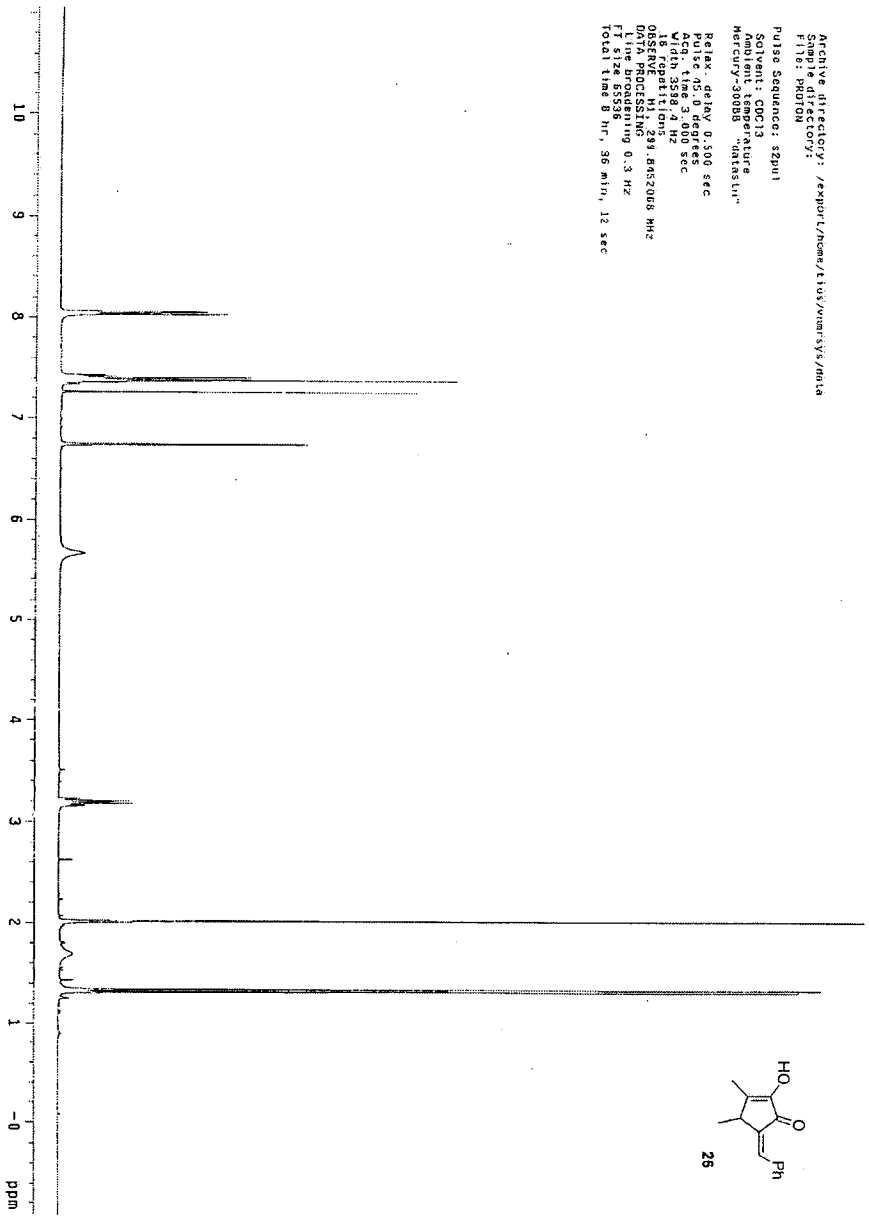

Archive directory: /export/home/tius/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: jsf08A
Mercury-300BB "data1n"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 3598.4 Hz
8 repetitions
OBSERVE H1, 299.8452270 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 29 sec
HO
O
20
10
9
8
7
6
5
4
3
2
1
0
ppm

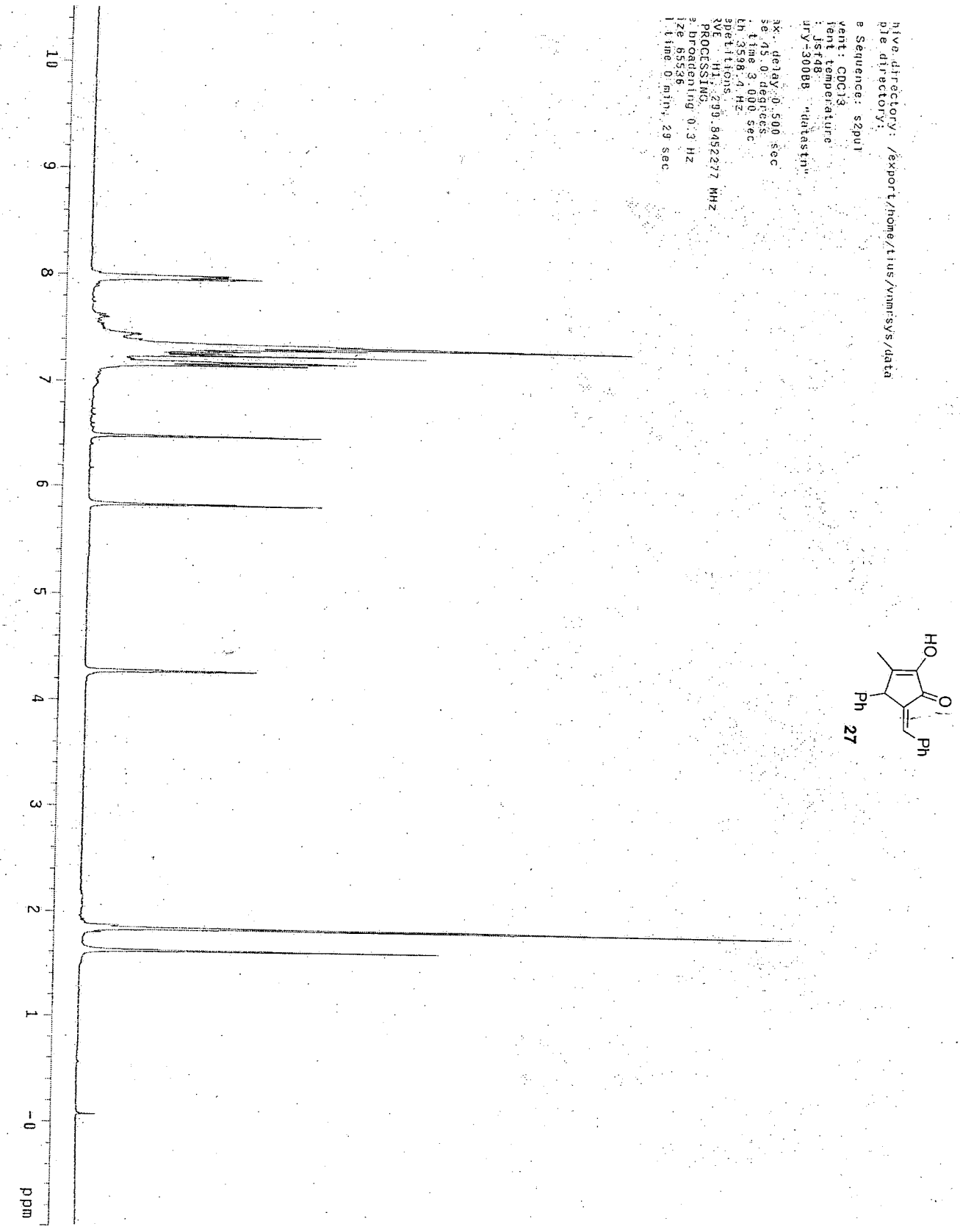

Archive directory: /export/home/tius/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "datastn"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 4798.5 Hz
8 repetitions
OBSERVE H1, 299.8452282 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 30 sec
HO
O
TIPS
Ph
21
12
10
8
6
4
2
-0
ppm

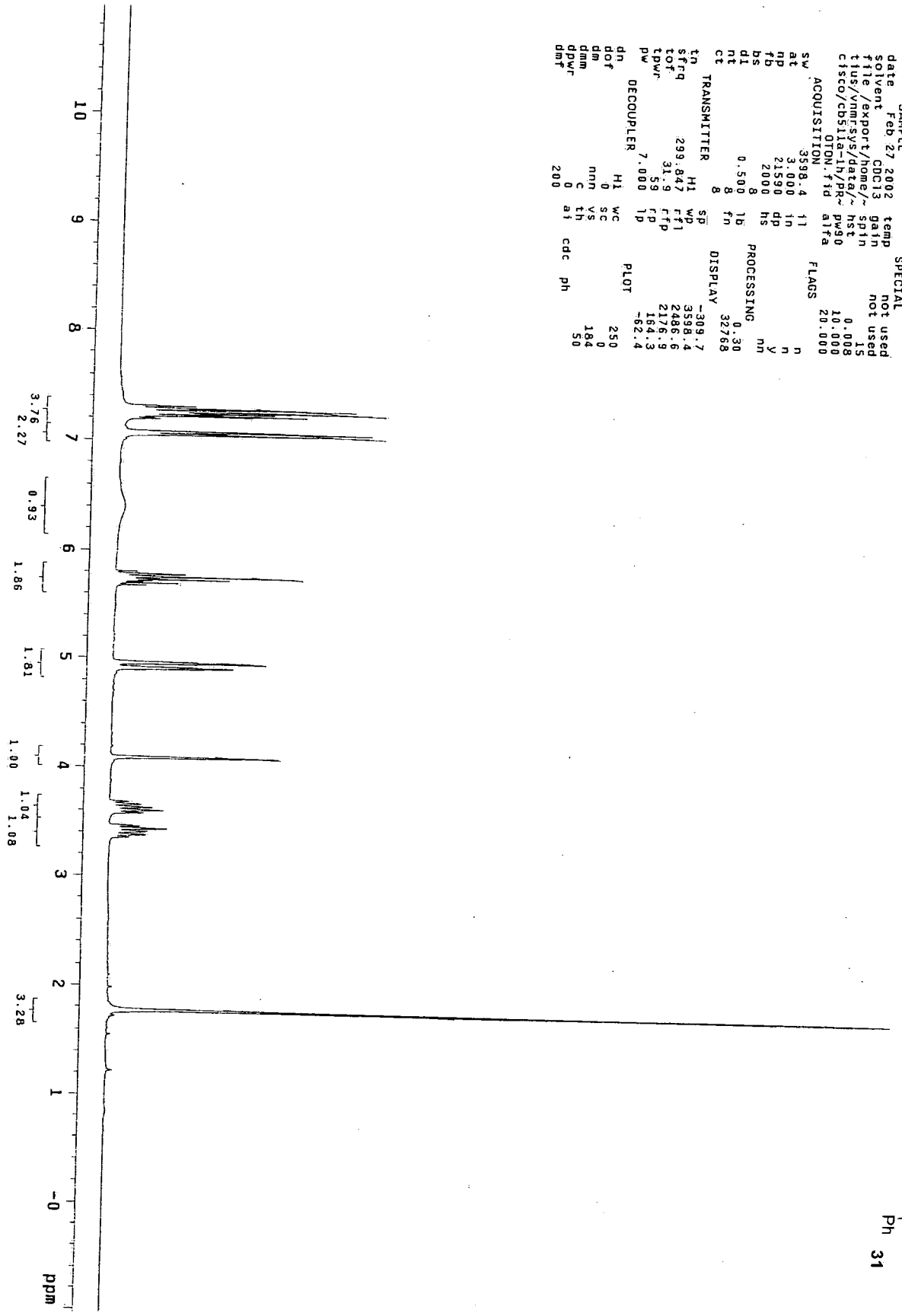

Archive directory: /export/home/tlu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "nmrlab"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 4789.5 Hz
8 repetitions
OBSERVE H1, 299.8452308 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 30 sec
HO
O
TIPS
22
12
10
8
6
4
2
-0
ppm

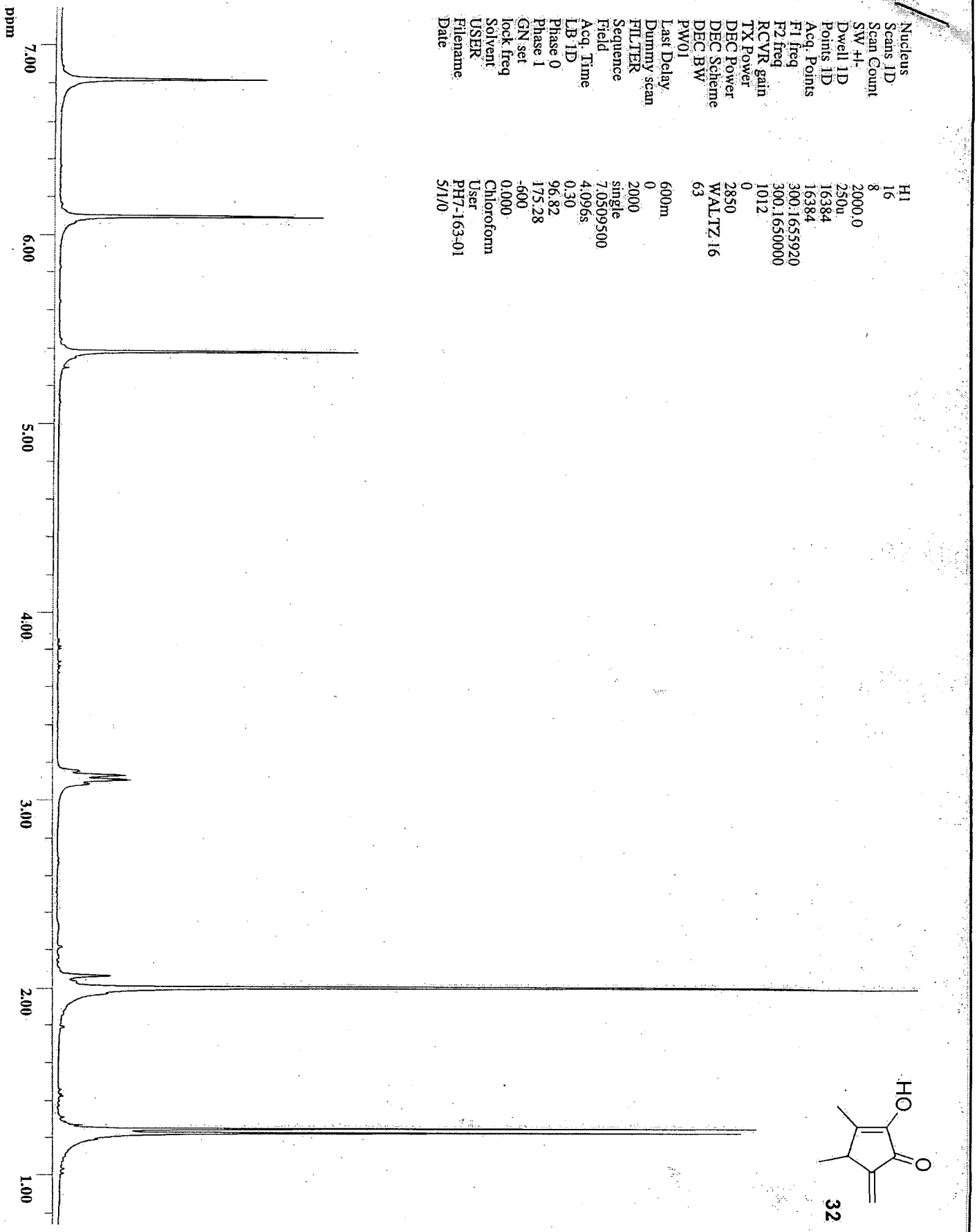


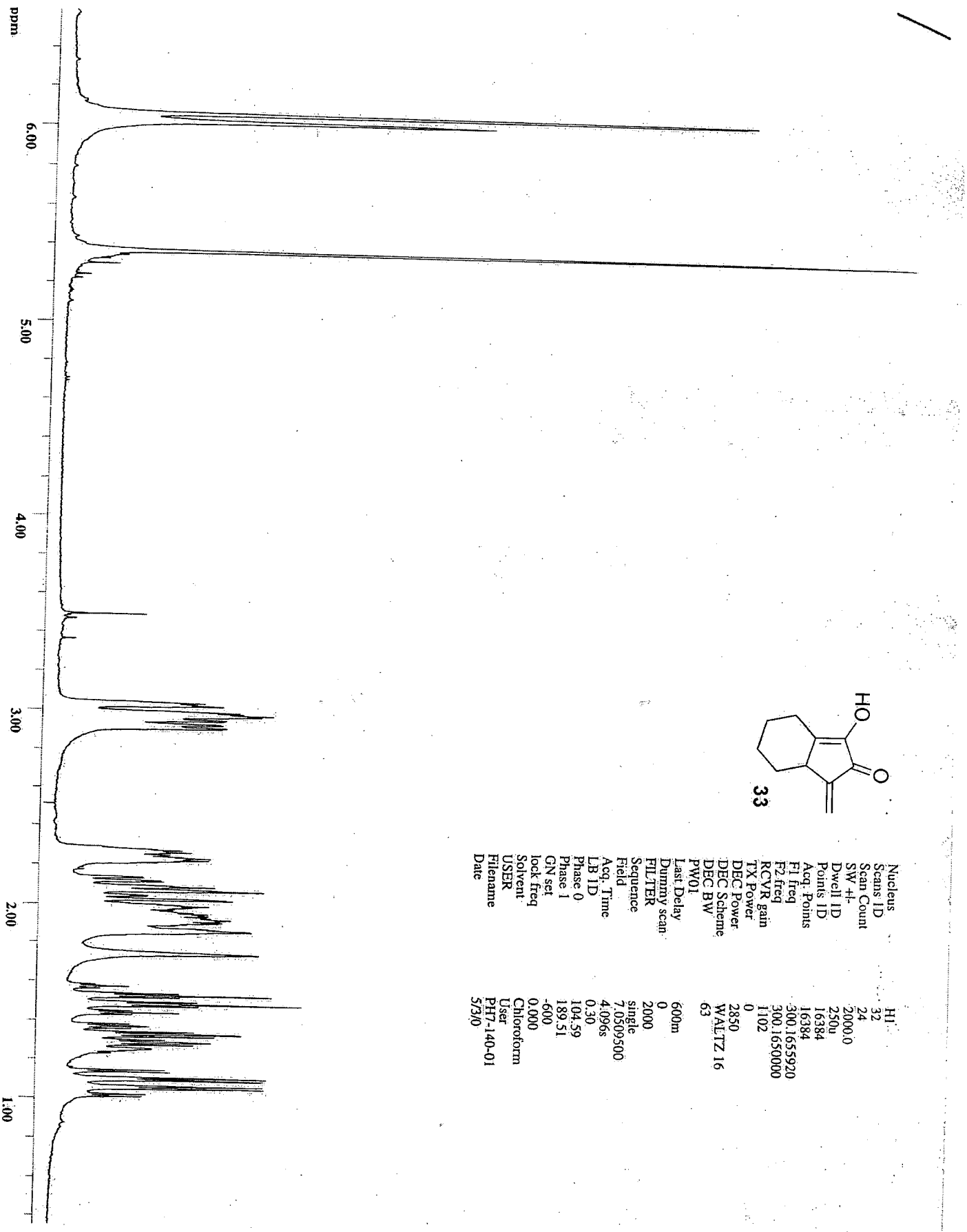



Archive directory: /export/home/tius/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "tantstn"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 3598.4 Hz
1 repetitions
OBSERVE H1, 299.8052068 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 8 hr, 36 min, 12 sec
10
9
8
7
6
5
4
3
2
1
-0
ppm
HO
O
Ph
26


hive directory: /export/home/tius/vnmrsys/data
ple directory:
e Sequence: s2pul
vent: CDCl3
ient temperature
: jsf48
ory=300BB "datastn"
ax. delay 0.500 sec
se 45.0 degrees
time 3.000 sec
th 3598.4 Hz
petitions
RVE H1, 299.8452277 MHz
PROCESSING
e broadening 0.3 Hz
ize 65536
l time 0 min, 29 sec
10
9
8
7
6
5
4
3
2
1
-0
ppm
HO
O
Ph
Ph
27




Nucleus H1
Scans 1D 16
Scan Count 8
SW +/- 2000.0
Dwell 1D 250u
Points 1D 16384
Acq. Points 16384
F1 freq 300.1655920
F2 freq 300.1650000
RCVR gain 1012
TX Power 0
DEC Power 2850
DEC Scheme WALTZ16
DEC BW 63
PW01
Last Delay 600m
Dummy scan 0
FILTER 2000
Sequence single
Field 7.0509500
Acq. Time 4.096s
LB 1D 0.30
Phase 0 96.82
Phase 1 175.28
GN set -600
lock freq 0.000
Solvent Chloroform
USER User
Filename PH7-163-01
Date 5/1/0
ppm
7.00
6.00
5.00
4.00
3.00
2.00
1.00
HO
O
32


ppm
6.00
5.00
4.00
3.00
2.00
1.00
HO
O
33
Nucleus H1
Scans ID 32
Scan Count 24
SW +/- 2000.0
Dwell ID 250u
Points ID 16384
Acq. Points 16384
F1 freq 300.1655920
F2 freq 300.1650000
RCVR gain 1102
TX Power 0
DEC Power 2850
DEC Scheme WALTZ 16
DEC BW 63
PW01
Last Delay 600m
Dummy scan 0
FILTER 2000
Sequence single
Field 7.0509500
Acq. Time 4.096s
LB ID 0.30
Phase 0 104.59
Phase 1 189.51
GN set -600
lock freq 0.000
Solvent Chloroform
USER User
Filename PH7-140-01
Date 5/3/0