

## Supporting Information

to accompany

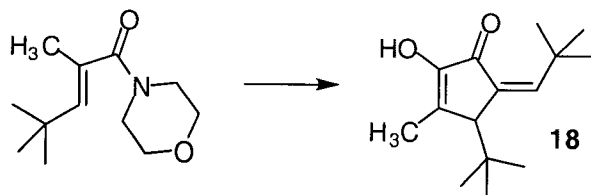
### Isomerization-Cyclization Approach to the Synthesis of 2- Hydroxy-5-alkylidene-cyclopent- 2-enones

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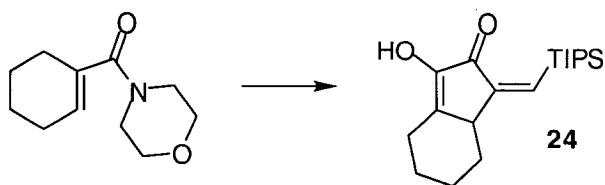
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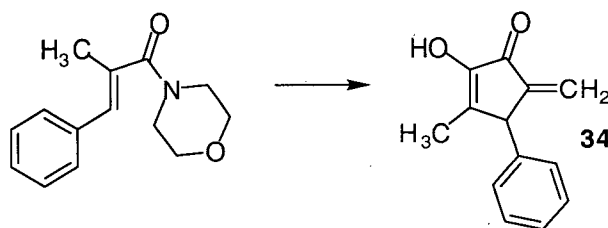
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To a solution of ether **13** (2.23 mmol, 250 mg) in THF (3 ml) at  $-78^{\circ}\text{C}$  was added *sec*-BuLi (1.64 ml, 2.13 mmol, 1.30 M in hexanes). After 15 min, a solution of 2,4,4-trimethyl-1-morpholin-4-yl-pent-2-en-1-one (0.71 mmol, 150 mg) 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<sub>2</sub>O. The aqueous phase was extracted with Et<sub>2</sub>O (3 x) and the combined organic phases were washed with brine (1 x), dried over MgSO<sub>4</sub>, 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  $133\text{--}136^{\circ}\text{C}$ ;  $R_f = 0.63$  (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  232 ( $\text{M}^+$ , 2), 181 (12), 180 (100), 166 (15), 165 (91), 139 (13), 115 (24), 69 (14); HREIMS calcd for C<sub>15</sub>H<sub>24</sub>O<sub>2</sub> 236.1776, found 236.1765.



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-en-1-yl-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  $32\text{--}37^{\circ}\text{C}$ ;  $R_f = 0.59$  (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  277 ( $\text{M}^+$  - isopropyl, 100), 145 (14), 117 (11), 115 (10), 75 (10), 74 (16), 69 (13), 68 (30); HREIMS calcd for C<sub>19</sub>H<sub>32</sub>O<sub>2</sub>Si ( $\text{M}^+$  - isopropyl) 277.1624, found 277.1618.



To alkyne **28** (252 mg, 2.52 mmol) in THF (8 mL) at  $-78^{\circ}\text{C}$  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  $\text{Et}_2\text{O}$  (3 x). The combined organic extracts were washed with brine (1 x), dried over  $\text{K}_2\text{CO}_3$ , 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  $158\text{--}161^{\circ}\text{C}$ ;  $R_f = 0.62$  (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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,  $710\text{ cm}^{-1}$ ; EIMS  $m/z$  200 ( $\text{M}^+$ , 100), 142 (41), 129 (61), 128 (41), 115 (67), 88 (44); HREIMS calcd for  $\text{C}_{13}\text{H}_{12}\text{O}_2$  200.0837, found 200.0836.

5-(2,2-Dimethyl-propylidene)-2-hydroxy-3-methyl-4-phenyl-cyclopent-2-enone **16**: mp  $186\text{--}188^{\circ}\text{C}$ ;  $R_f = 0.65$  (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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,  $695\text{ cm}^{-1}$ ; EIMS  $m/z$  256 ( $\text{M}^+$ , 37), 223 (26), 186 (100), 91 (38); HREIMS calcd for  $\text{C}_{17}\text{H}_{20}\text{O}_2$  256.1463, found 256.1464.

5-(2,2-Dimethyl-propylidene)-2-hydroxy-3,4-dimethyl-cyclopent-2-enone **17**: mp  $109\text{--}114^{\circ}\text{C}$ ;  $R_f = 0.63$  (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.26 (s br, 1H), 6.01 (s br, 1H), 2.93 (qm,  $J = 7.1$  Hz, 1H), 1.94 (s, 3H), 1.27 (s, 9H), 1.16 (d,  $J = 7.1$  Hz, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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,  $1095\text{ cm}^{-1}$ ; EIMS  $m/z$  194 ( $\text{M}^+$ , 63), 179 (27), 161 (40), 133 (25), 124 (100), 91 (20); HREIMS calcd for  $\text{C}_{12}\text{H}_{18}\text{O}_2$  194.1307, found 194.1290.

1-(2,2-Dimethyl-propylidene)-3-hydroxy-1,4,5,6,7,7a-hexahydro-inden-2-one **19**: mp  $155\text{--}157^{\circ}\text{C}$ ;  $R_f = 0.60$  (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  220 ( $\text{M}^+$ , 93), 187 (45), 150 (100), 122 (40), 91 (42), 79 (45); HREIMS calcd for  $\text{C}_{14}\text{H}_{20}\text{O}_2$  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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.60 (s, 1H), 5.82 (s, 1H), 3.15 (m, 2H), 2.04 (s, 3H), 1.18 (s, 9H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  180 ( $\text{M}^+$ , 100), 165 (48), 139 (22), 119 (21), 117 (24), 110 (38), 91 (18), 81 (18); HREIMS calcd for  $\text{C}_{11}\text{H}_{16}\text{O}_2$  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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  183 ( $\text{M}^+ - \text{C}_{10}\text{H}_{24}\text{Si}$ , 100), 155 (44), 141 (34), 103 (23), 101 (29), 89 (24), 75 (71); HREIMS calcd for  $\text{C}_{19}\text{H}_{25}\text{O}_2\text{Si}$  ( $\text{M}^+ - \text{isopropyl}$ ) 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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  294 ( $\text{M}^+$ , 4), 253 (22), 252 (76), 251 (100), 183 (22), 131 (44), 103 (37), 75 (64); HREIMS calcd for  $\text{C}_{17}\text{H}_{30}\text{O}_2\text{Si}$  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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  238 (11), 237 (40), 236 (10), 183 (8), 131 (9), 103 (8), 75 (23); HREIMS calcd for  $\text{C}_{17}\text{H}_{29}\text{O}_2\text{Si}$  ( $\text{M}^+ - \text{isopropyl}$ ) 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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  277 ( $\text{M}^+$  - isopropyl, 100), 145 (14), 117 (11), 115 (10), 75 (10), 74 (16), 69 (13), 68 (30); HREIMS calcd for  $\text{C}_{16}\text{H}_{25}\text{O}_2\text{Si}$  ( $\text{M}^+$  - 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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  237 ( $\text{M}^+$  - isopropyl, 70), 197 (62), 183 (100), 155 (43), 141 (43), 101 (30), 89 (32), 75 (91); HREIMS calcd for  $\text{C}_{13}\text{H}_{21}\text{O}_2\text{Si}$  ( $\text{M}^+$  - *iso*-propyl) 237.1311, found 237.1307.

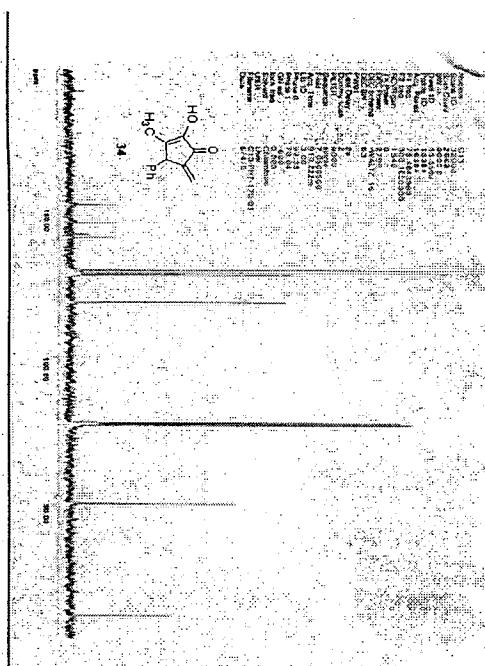
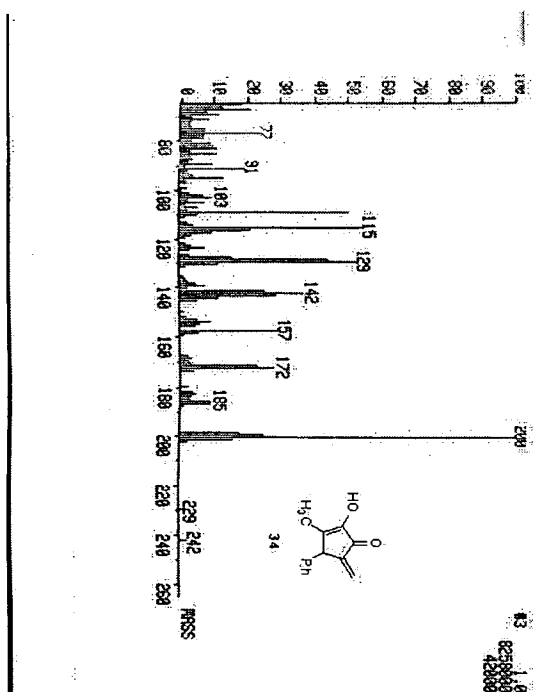
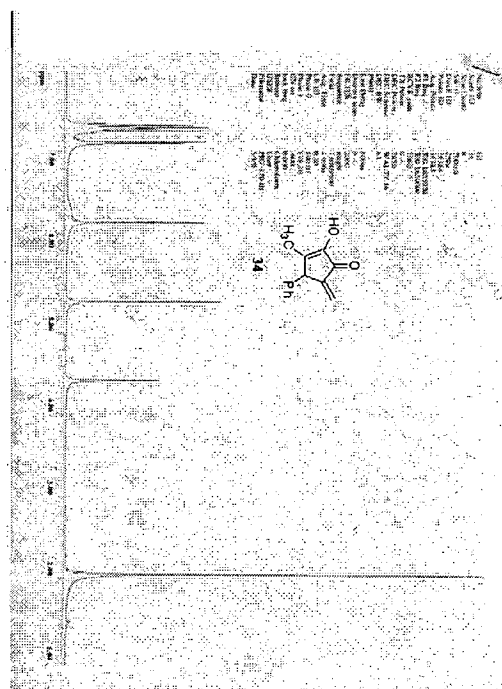
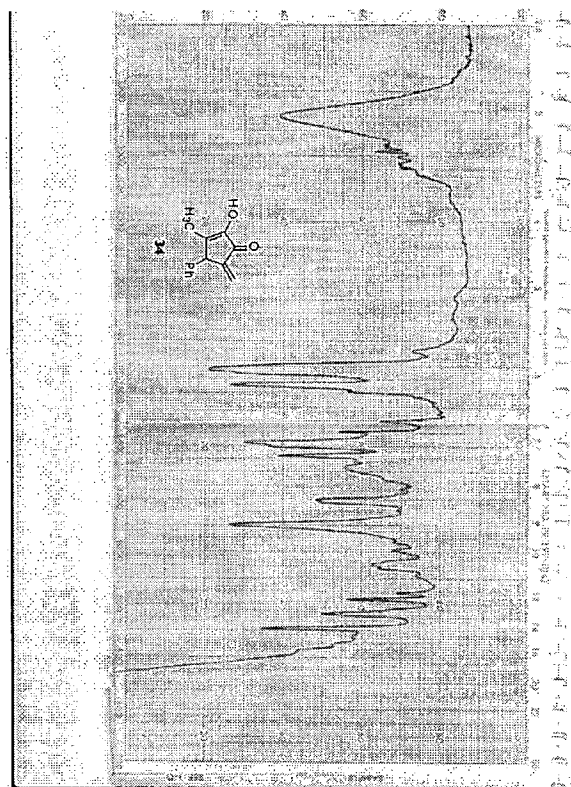
5-Benzylidene-2-hydroxy-3,4-dimethyl-cyclopent-2-enone **26**: mp >180°C dec.;  $R_f$  = 0.64 (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.07 – 8.04 (m, 2H), 7.44 – 7.36 (m, 3H), 6.72 (s, 1H), 5.63 (s br, 1H), 3.18 (q,  $J$  = 7.0 Hz, 1H), 2.05 (s, 3H), 1.32 (d,  $J$  = 7.0 Hz, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  214 ( $\text{M}^+$ , 100), 171 (73), 153 (32), 143 (33), 128 (52), 115 (45); HREIMS calcd for  $\text{C}_{14}\text{H}_{14}\text{O}_2$  214.0994, found 214.0989.

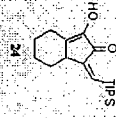
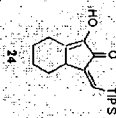
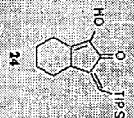
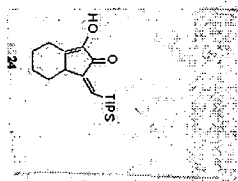
5-Benzylidene-2-hydroxy-3-methyl-4-phenyl-cyclopent-2-enone **27**: mp >200°C dec.;  $R_f$  = 0.65 (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 – 7.91 (m, 2H), 7.40 – 7.21 (m, 5H), 7.20 – 7.16 (m, 3H), 6.49 (s, 1H), 5.82 (s, 1H), 4.25 (s, 1H), 1.84 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  276 ( $\text{M}^+$ , 100), 258 (24), 233 (54), 229 (28), 215 (37), 128 (23), 115 (29), 105 (27); HREIMS calcd for  $\text{C}_{19}\text{H}_{16}\text{O}_2$  276.1150, found 276.1160.

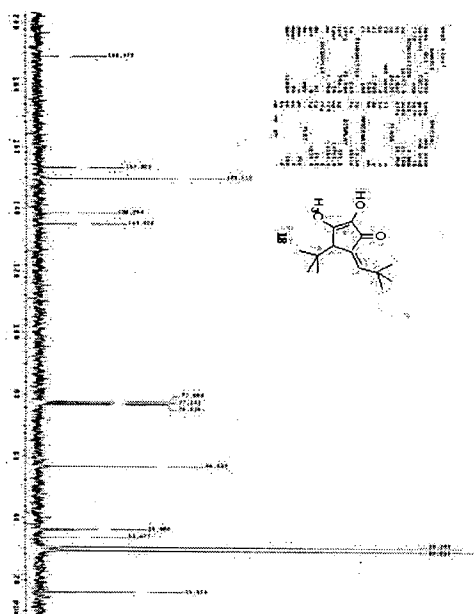
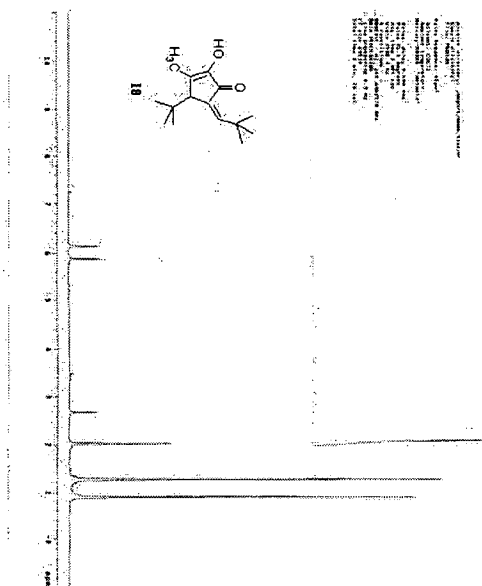
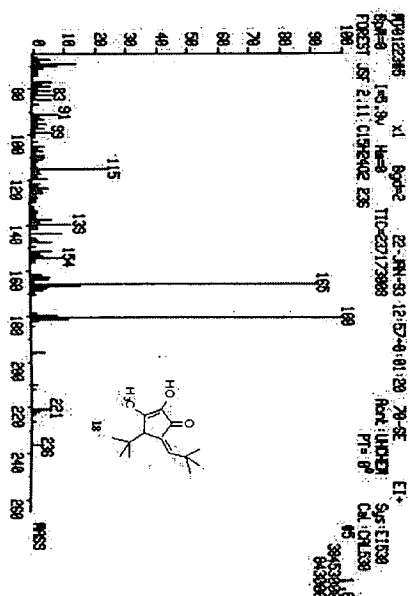
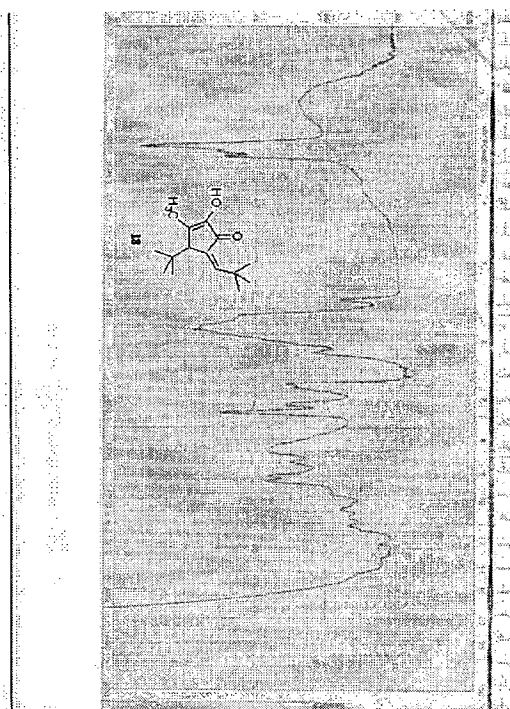
5-But-3-enylidene-2-hydroxy-3-methyl-4-phenyl-cyclopent-2-enone **31**: (*E* / *Z* = 17 / 83); *Z* Isomer: mp >140°C dec.;  $R_f$  = 0.60 (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.31 – 7.22 (m, 3H), 7.10 – 7.05 (m, 2H), 6.50 – 6.30 (s br, 1H), 5.80 – 5.66 (m, 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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  240 ( $\text{M}^+$ , 29), 183 (16), 167 (12), 91 (18), 69 (100); HREIMS calcd for  $\text{C}_{16}\text{H}_{16}\text{O}_2$  240.1150, found 240.1179.

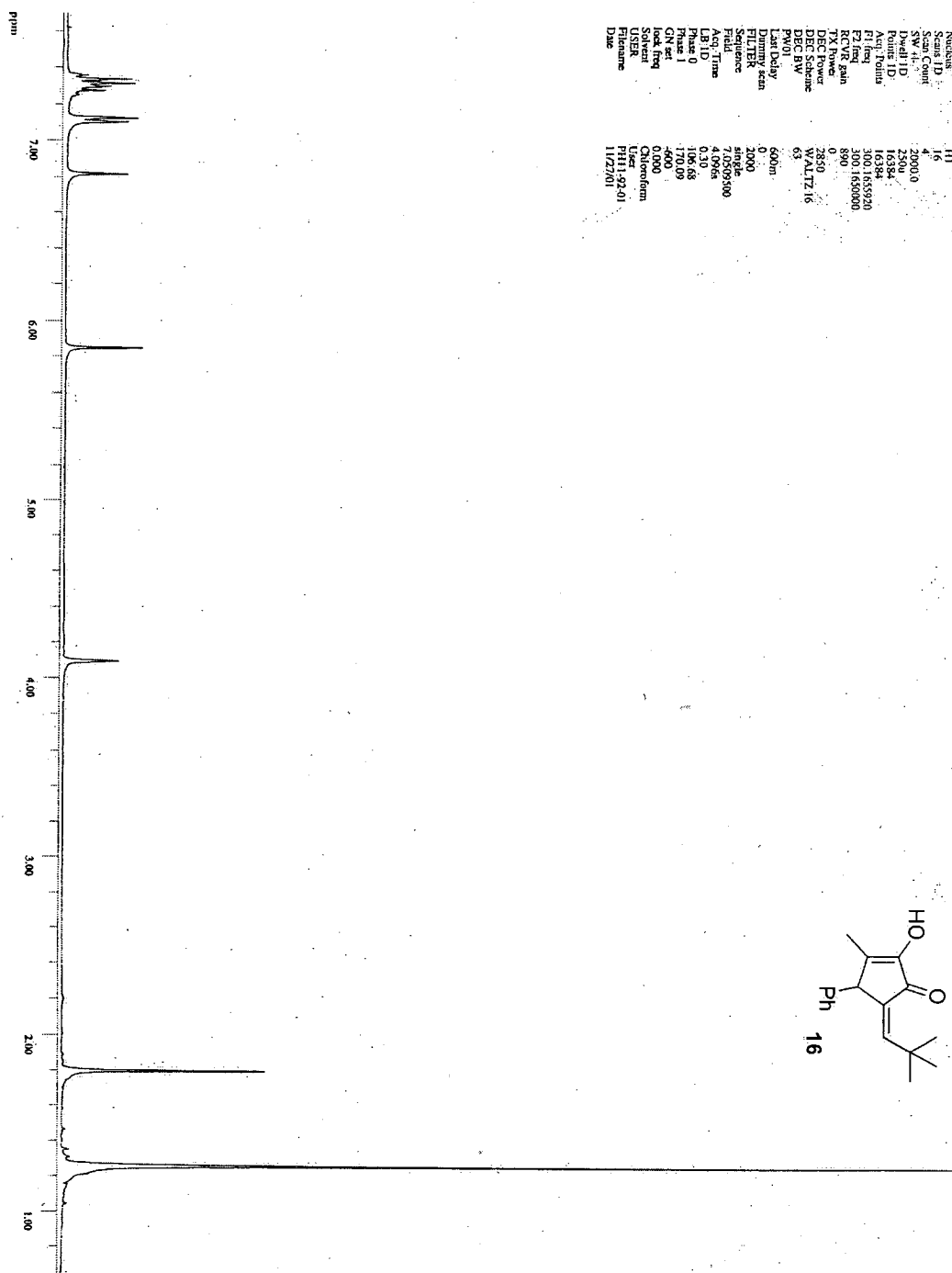
2-Hydroxy-3,4-dimethyl-5-methylene-cyclopent-2-enone **32**: mp 87–89°C;  $R_f$  = 0.57 (20% EtOAc in hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.82 (s, 1H), 6.11 (d,  $J$  = 1.3 Hz, 1H), 5.38 (s, 1H), 3.13 (qm,  $J$  = 7.1 Hz, 1H), 2.02 (s, 3H), 1.25 (d,  $J$  = 7.1 Hz, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  138 ( $\text{M}^+$ , 7), 136 (7), 124 (6), 123 (17), 119 (100), 95 (8); HREIMS calcd for  $\text{C}_8\text{H}_{10}\text{O}_2$  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);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  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);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  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  $\text{cm}^{-1}$ ; EIMS  $m/z$  164 ( $\text{M}^+$ , 100), 136 (39), 135 (33), 108 (34), 93 (38), 79 (50), 77 (30); HREIMS calcd for  $\text{C}_{10}\text{H}_{12}\text{O}_2$  164.0837, found 164.0819.

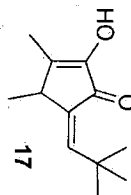


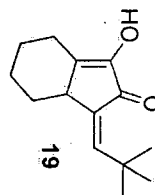


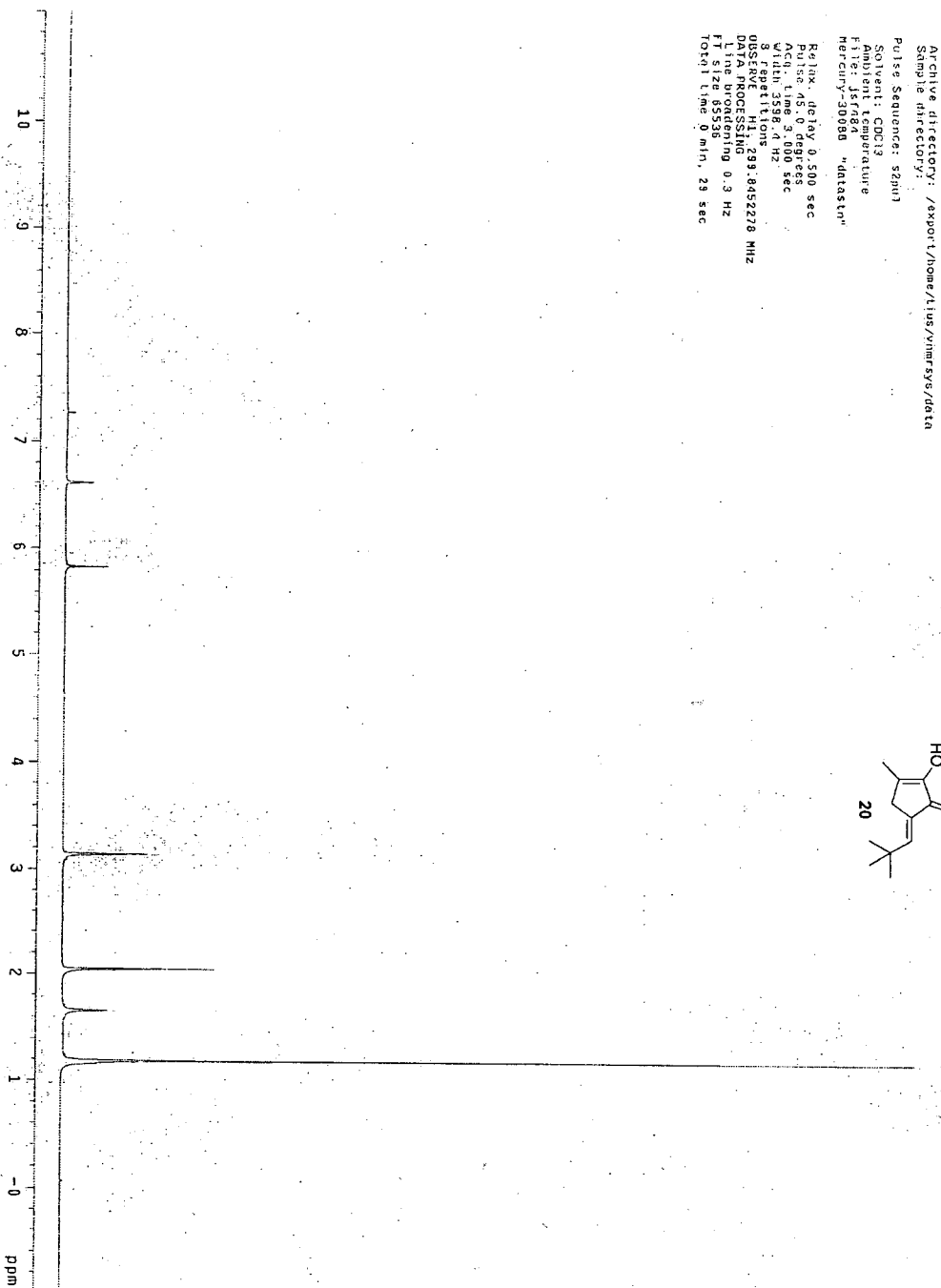


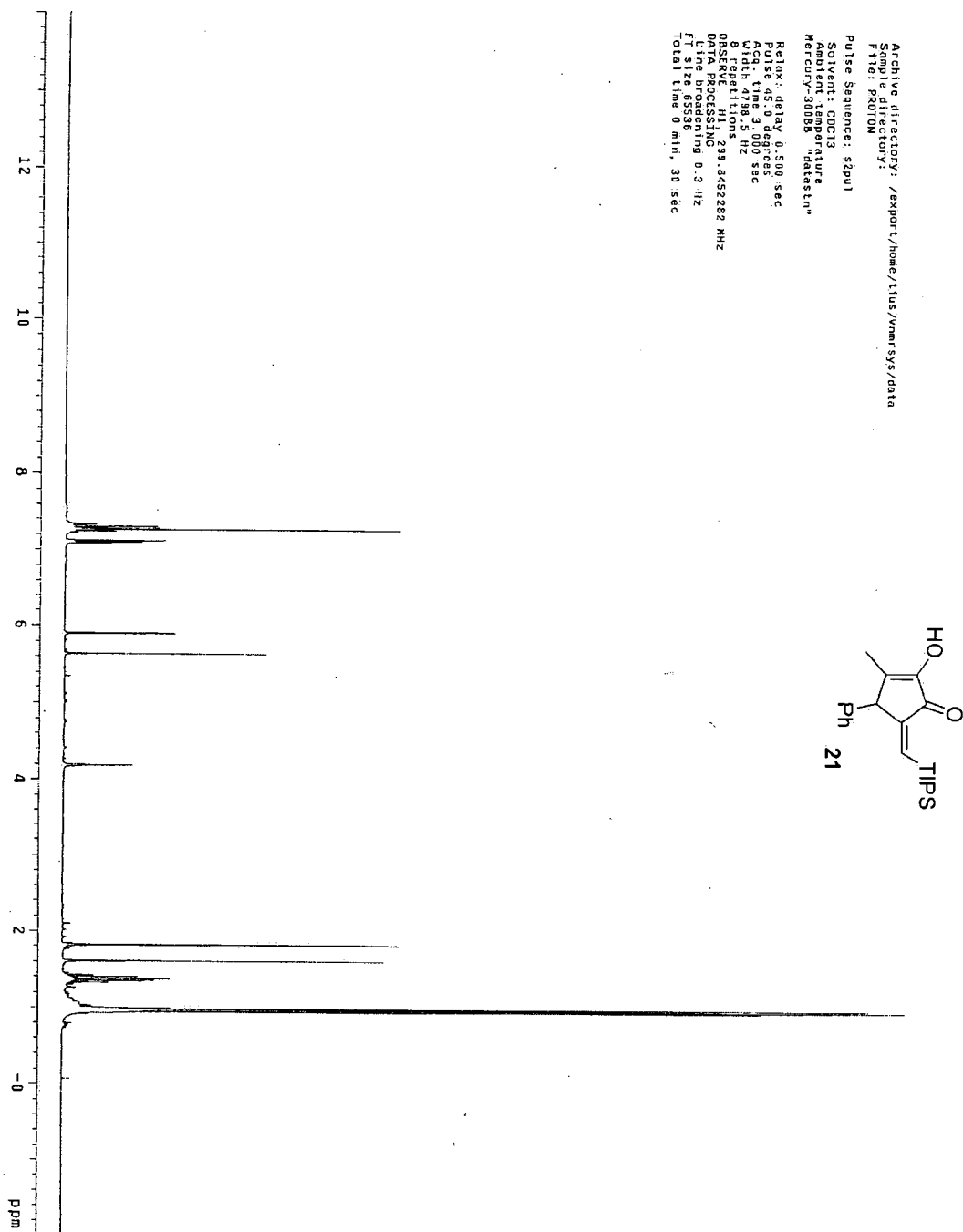


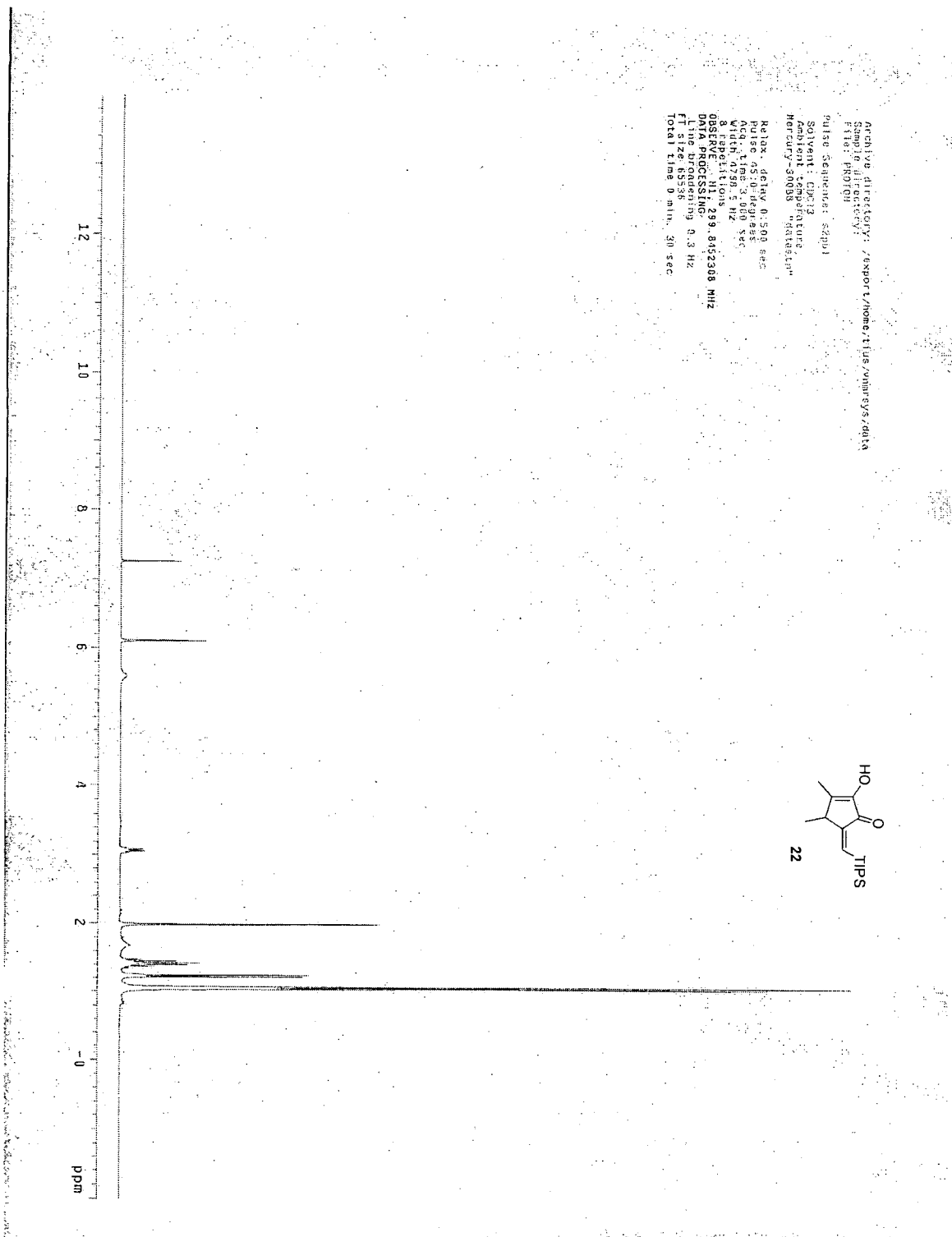
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 F2 (Hz): 300.1655920  
 RCVR gain: 890  
 F1 (Hz): 300.1655920  
 DEC Power: 2850  
 DEC Scheme: WALTZ16  
 DEC BW: 63  
 PWD: 600m  
 Scan Delay: 0  
 Delay Scan: 2000  
 FILTER: single  
 Sequence: 7.0509300  
 Field: 400MHz  
 Lab ID: 0.30  
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 GN ref: 400  
 Ref: 400  
 Solvent: CDCl3  
 User: PHL1-92-01  
 Filename: 11/22/01

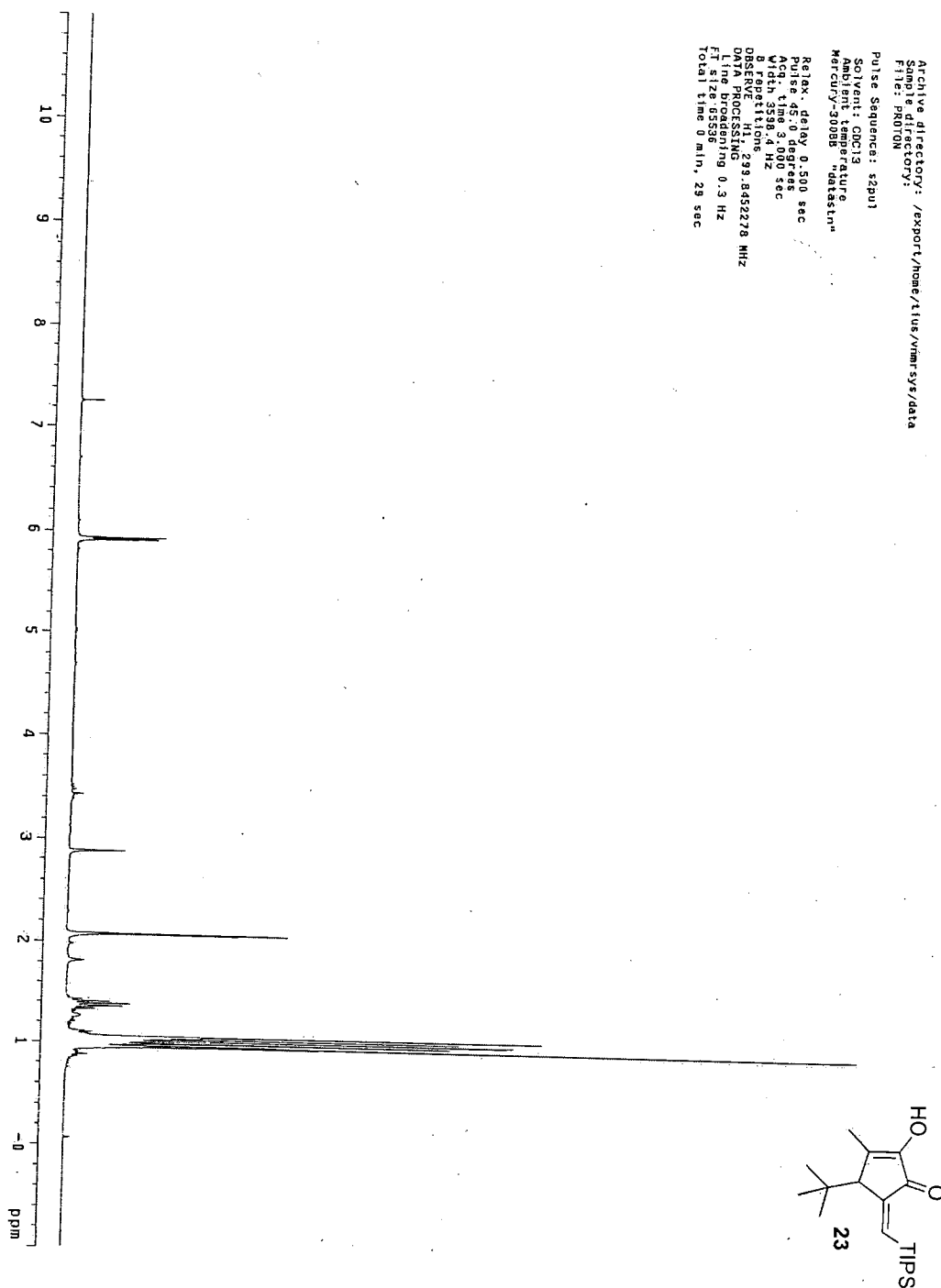


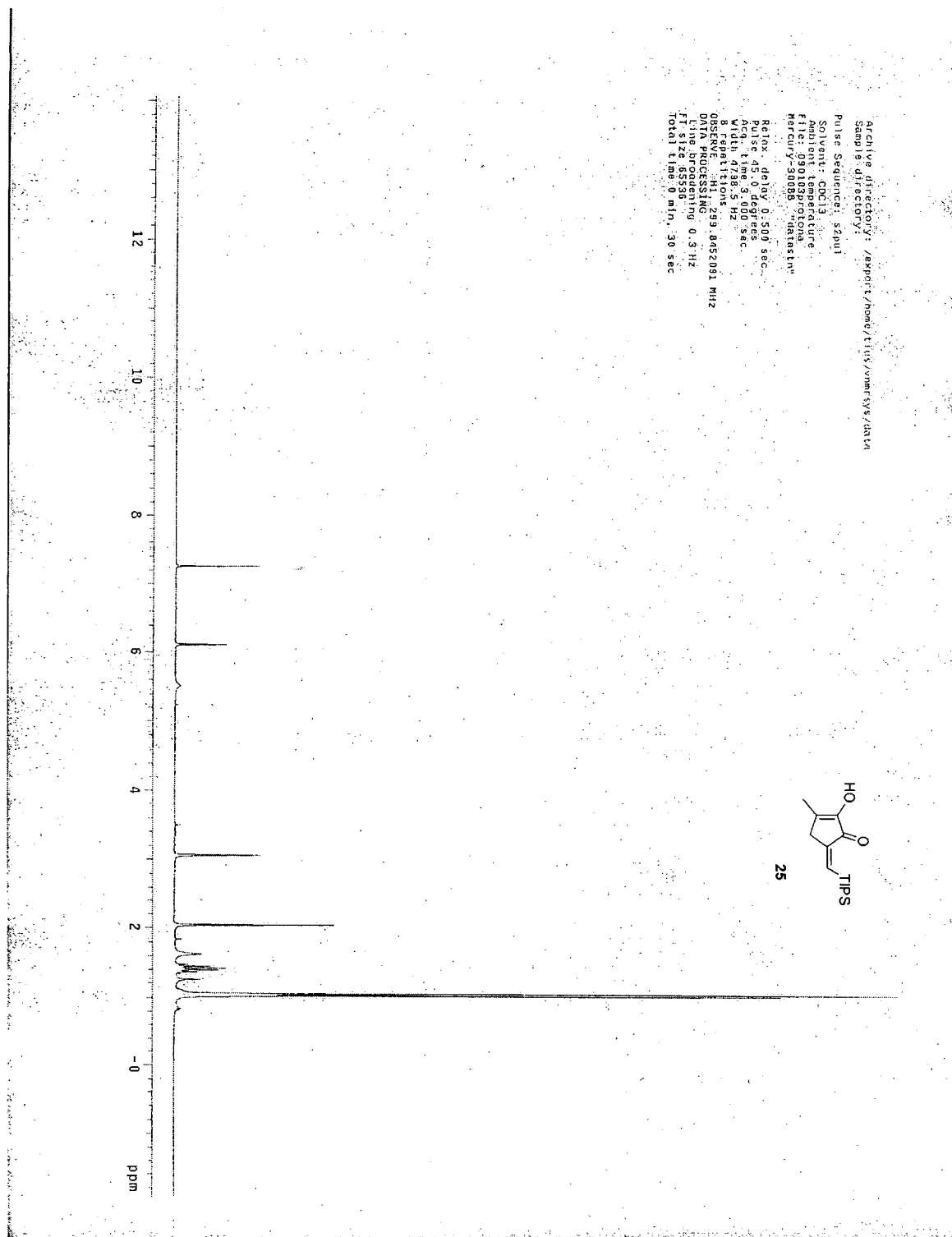


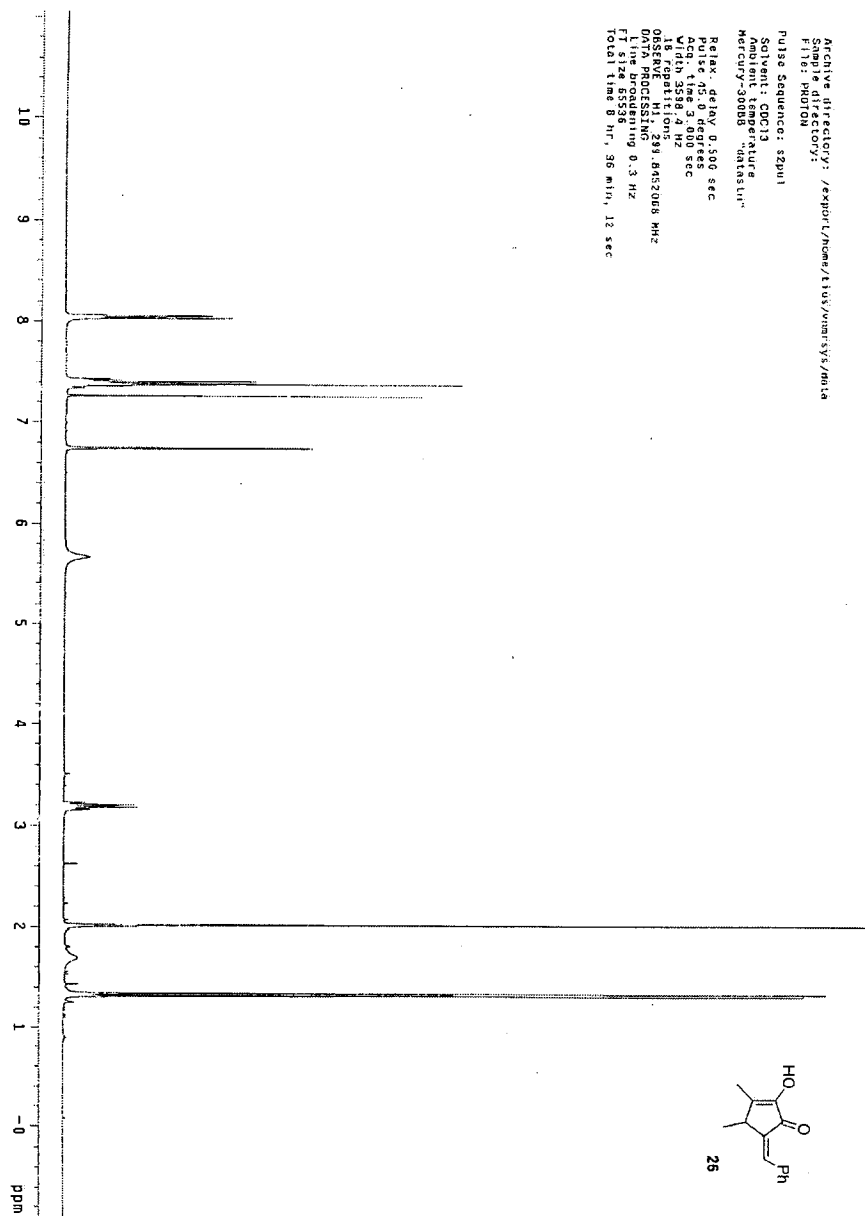


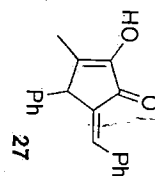




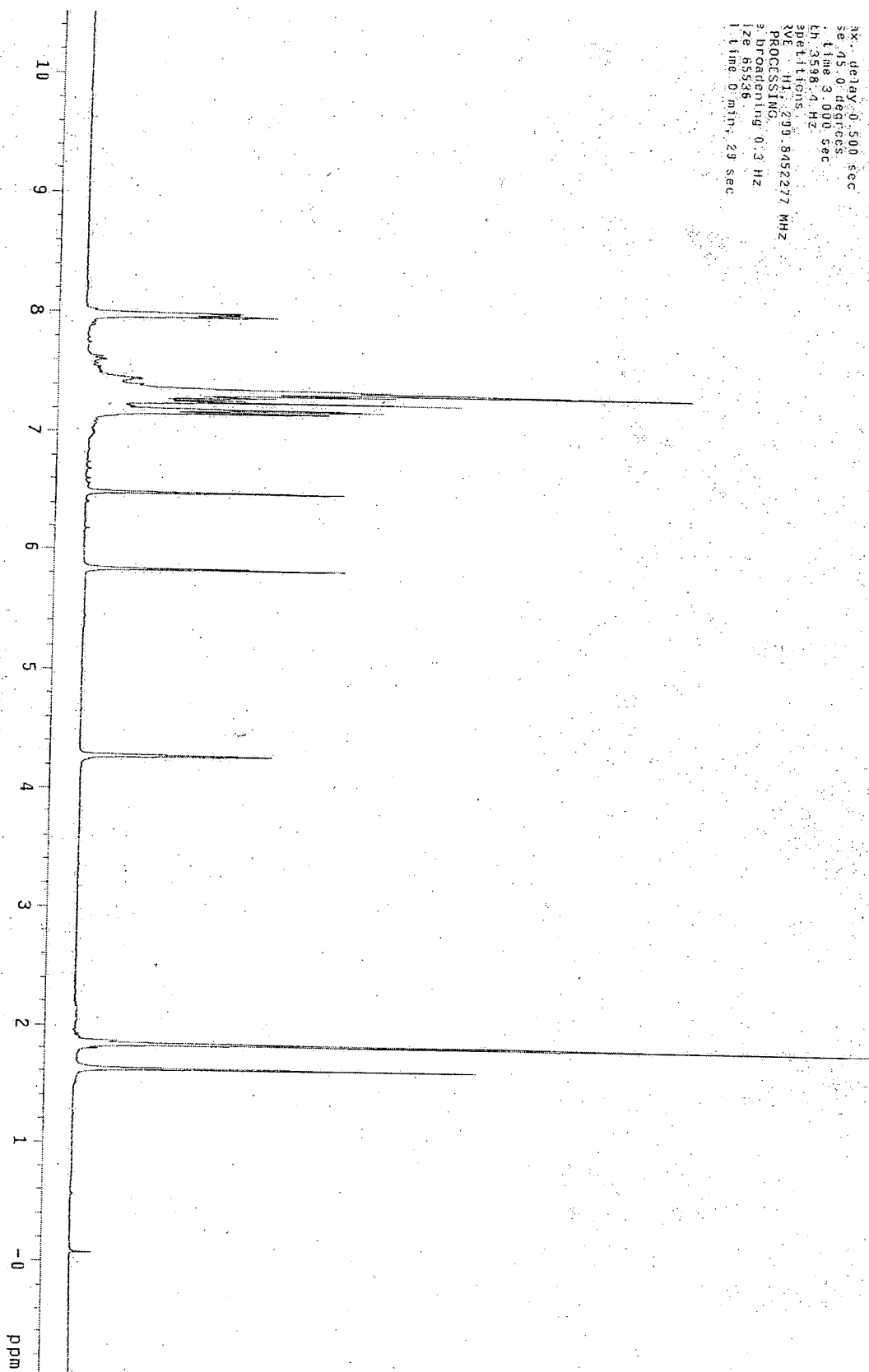






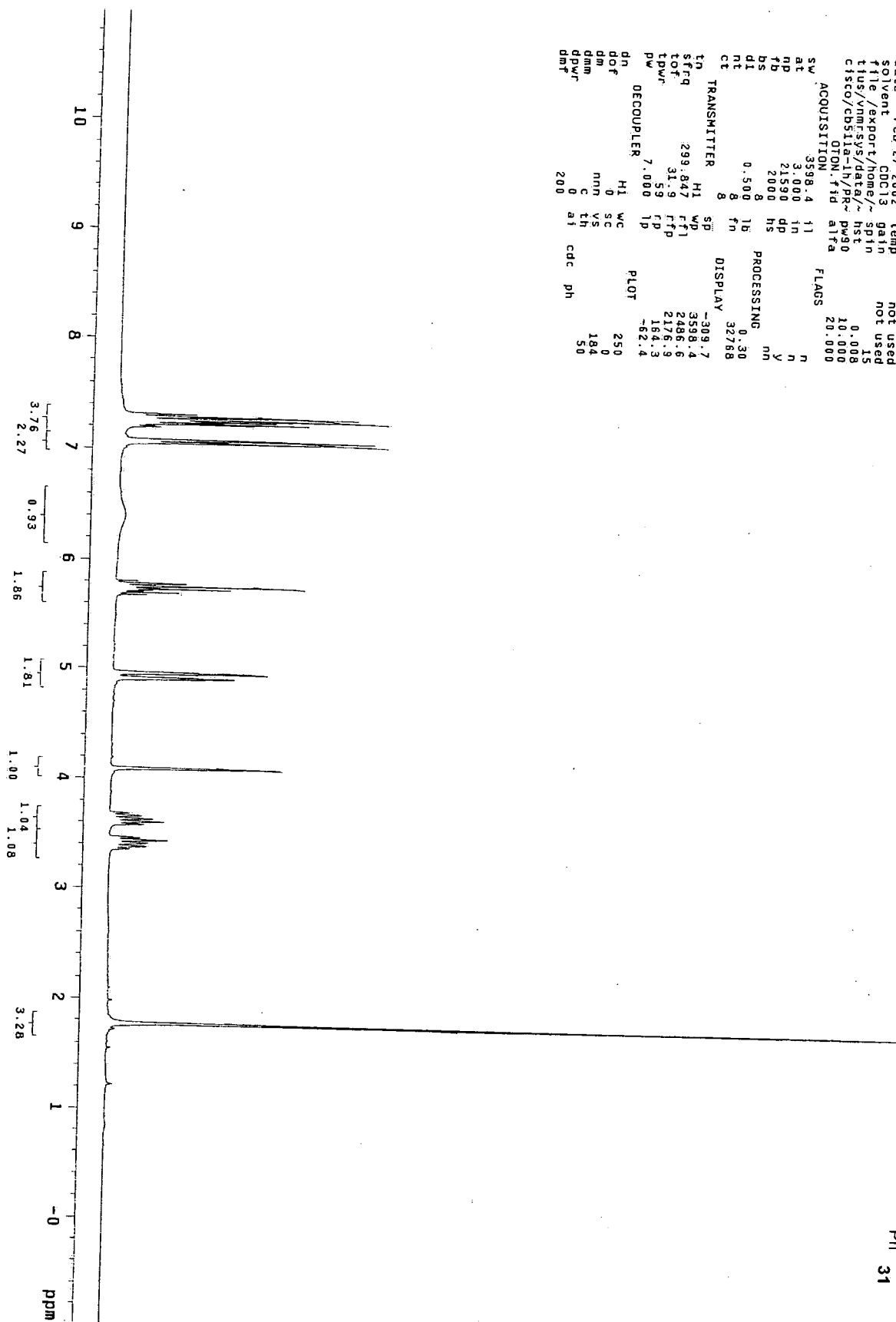


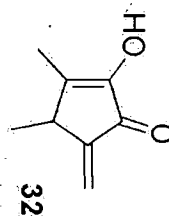
hve: directory: /export/home/tius/vnmr/sys/data  
 ple: directory:  
 e Sequence: szpul  
 vent: CDD13  
 tent: temperature  
 : J5f48  
 ury-300B8 "data51n"  
 ax: delay: 0.500 sec  
 se: 15.0 degrees  
 : time 3.000 sec  
 th: 3598.4 Hz  
 apert: 10.0  
 3VE: HI: 299.8452277 MHz  
 PROCESSING:  
 a: broadening: 0.3 Hz  
 ize: 65536  
 l: time 0 min, 29 sec



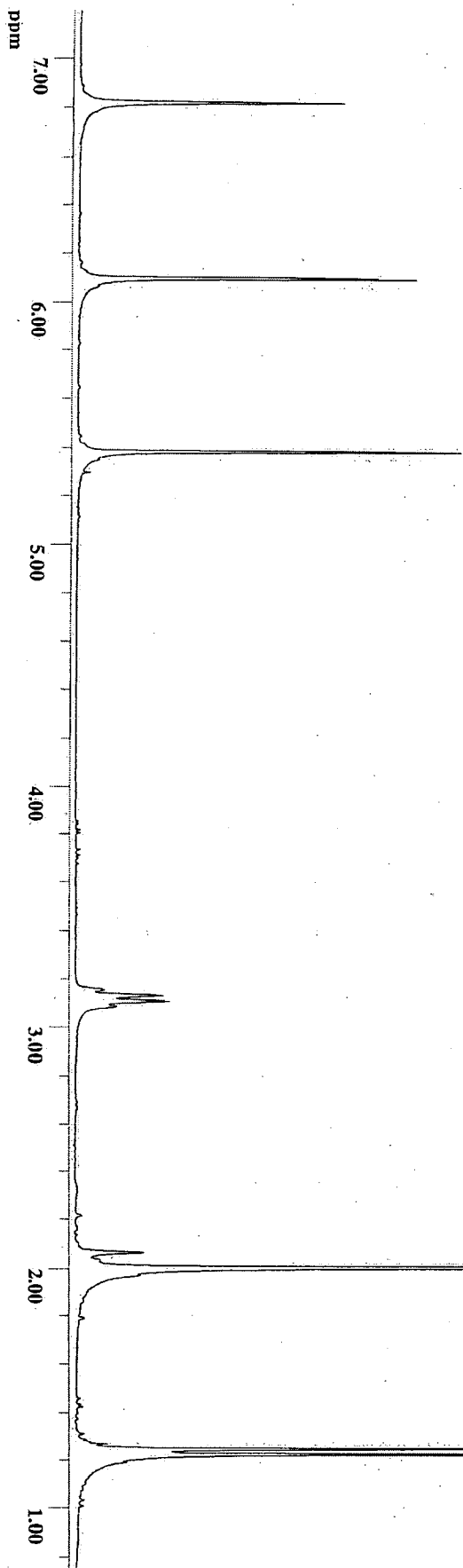
exptl s2pu1

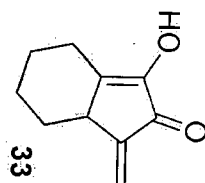
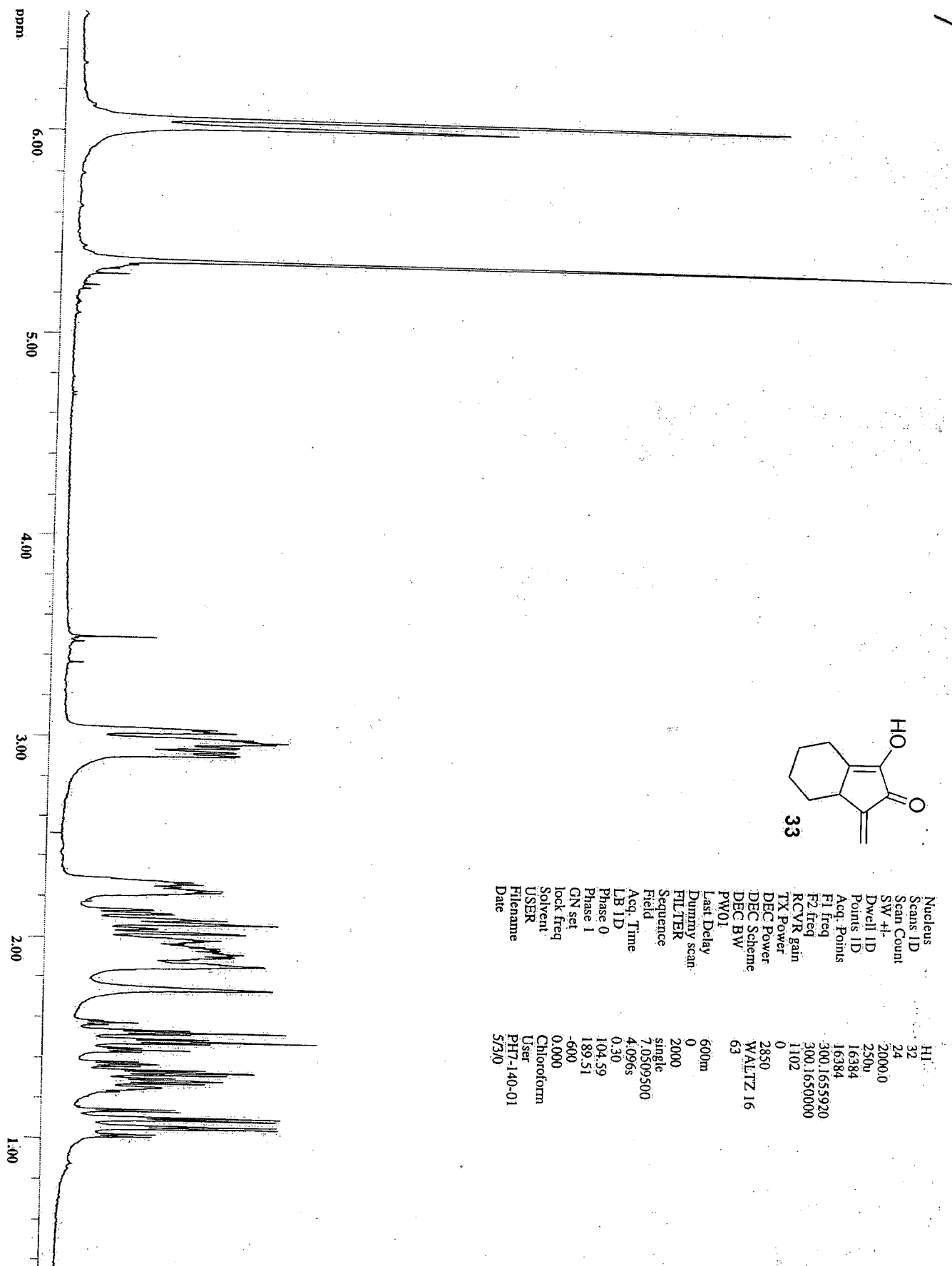
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|----------------------|----------------|---------|----------|
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| solvent              | CDCl3          | gain    | not used |
| file                 | /export/home/~ | spin    | IS       |
| titus/vnmrsvs/data/~ | hst            |         | 0.008    |
| cisco/cb51a-1h/pr~   | pw90           |         | 10.000   |
|                      | OTON fid       | alfa    | 20.000   |
| ACQUISITION          |                | FLAGS   |          |
| sw                   | 3598.4         | fl      | n        |
| at                   | 3.000          | in      | n        |
| np                   | 21590          | dp      | y        |
| fb                   | 2000           | hs      | n        |
| bs                   | 0.500          | lb      | 0.30     |
| dl                   | 8              | fm      | 32768    |
| nt                   | 8              |         |          |
| ct                   | 8              |         |          |
| TRANSMITTER          |                | SP      | DISPLAY  |
| tn                   | H1             | wp      | -309.7   |
| sfreq                | 299.847        | ft1     | 3598.4   |
| tof                  | 31.5           | ftp     | 2486.6   |
| tpwr                 | 55             | fp      | 2176.9   |
| pw                   | 7.000          | lp      | 164.3    |
| DECOUPLER            |                | PL0T    | -62.4    |
| dn                   | H1             | wc      | 250      |
| dof                  | 0              | sc      | 0        |
| dm                   | mm             | vs      | 184      |
| dmm                  | c              | th      | 50       |
| dpwr                 | 0              | at      | cdc      |
| dnt                  | 200            | ph      |          |





|             |             |
|-------------|-------------|
| Nucleus     | H1          |
| Scans ID    | 16          |
| Scan Count  | 8           |
| SW +/-      | 2000.0      |
| Dwell ID    | 250u        |
| Points ID   | 16384       |
| Acq. Points | 16384       |
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| F2 freq     | 300.1650000 |
| RCVR gain   | 1012        |
| TX Power    | 0           |
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| DEC Scheme  | WALTZ.16    |
| DEC BW      | 63          |
| PW01        |             |
| Last Delay  | 600m        |
| Dummy scan  | 0           |
| FILTER      | 2000        |
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| Field       | 7.0509500   |
| Acq. Time   | 4:09:56s    |
| LB ID       | 0.30        |
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| GN set      | -600        |
| lock freq   | 0.000       |
| Solvent     | Chloroform  |
| USER        | User        |
| Filename    | PH7-163-01  |
| Date        | 5/1/0       |





|             |             |
|-------------|-------------|
| Nucleus     | HL          |
| Scans ID    | 32          |
| Scan Count  | 24          |
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| Dwell ID    | 2500        |
| Points ID   | 16384       |
| Acq. Points | 16384       |
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| F2 freq     | 300.1650000 |
| RCVR gain   | 1102        |
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| DEC Power   | 2850        |
| DEC Scheme  | WALITZ 16   |
| DEC BW      | 63          |
| PW01        |             |
| Last Delay  | 600m        |
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| Field       | 7.0509500   |
| Acq. Time   | 4.0966      |
| LB 1D       | 0.30        |
| Phase 0     | 104.59      |
| CN set      | 189.51      |
| lock freq   | -600        |
| Solvent     | 0.000       |
| USER        | Chloroform  |
| Filename    | PHT-140-01  |
| Date        | 5/30        |