

Supporting information for:

A three component homo 3+2 dipolar cycloaddition. A diversity-oriented synthesis of tetrahydro-1,2-oxazines and FR900482 skeletal congeners.

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Experimental

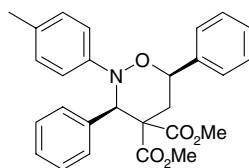
General

Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were obtained as thin films on NaCl plates using a Bruker Vector 33 FT-IR instrument. NMR experiments were performed on a Mercury 400 and Varian Inova 600 and 400 instruments and samples were obtained in CDCl₃ (referenced to 7.26 ppm for ¹H and 77.0 for ¹³C). Coupling constants (J) are in Hz. Mass spectra were obtained on a Finnigan MAT 8200 spectrometer at 70 eV.

Toluene and acetonitrile were distilled prior to use according to the standard procedures. All other reagents and solvents were used as purchased from Aldrich or Caledon. Reactions were checked for completion by TLC (EM Science, silica gel 60 F₂₅₄). Flash chromatography was performed using silica gel purchased from Silicycle Chemical Division Inc. (230-400 mesh)

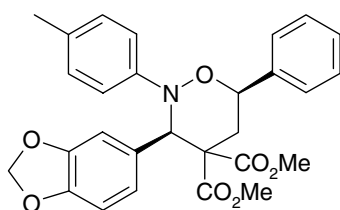
Typical Experimental Procedure for Preparation of Adducts

Yb(OTf)₃•xH₂O (10 mol %) was added to a solution of the hydroxylamine (0.65 mmol) and aldehyde (0.7 mmol) in toluene (4 mL) containing activated 4 Å molecular sieves. The solution was stirred under an argon atmosphere for 30 minutes at room temperature, after which the cyclopropane (0.5 mmol) was added. Solid cyclopropanes were added directly to the solution, while oils were added as a solution in toluene, 2 x 1mL. After the reaction was complete by TLC, the reaction mixture was filtered, and the filtrate was absorbed onto silica gel by reduced pressure removal of the solvent. Pure product was obtained after flash column chromatography on silica gel (eluent: EtOAc in hexanes). Samples could be recrystallized from CH₂Cl₂/hexanes if desired. For a few compounds, what appears to be the trans isomer was produced (<5%), but the minor isomer was removed by crystallization.



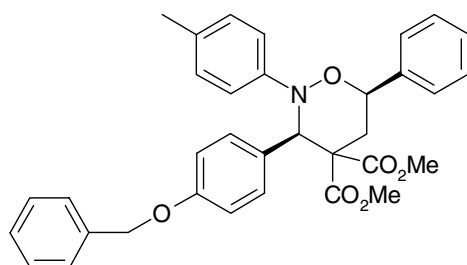
Compound 10

94 % yield, 8 hrs, colorless needles, m.p. 154-155 °C; R_f = 0.53 (30%, EtOAc in hexanes); ¹H-NMR (600 MHz, CDCl₃): δ = 7.62 (dd, J = 1.5, 8.4 Hz, 2H), 7.59 (d, J = 7.5 Hz, 2H), 7.48 (dd, J = 7.5, 7.5 Hz, 2H), 7.43-7.40 (m, 1H), 7.24-7.20 (m, 3 H), 7.03 (d, J = 8.7 Hz, 2H), 6.97 (d, J = 8.7 Hz, 2H), 5.78 (s, 1H), 5.07 (dd, J = 12.0, 3.0 Hz, 1H), 3.95, (s, 3H), 3.49 (s, 3H), 2.89 (dd, J = 14.4, 12.0 Hz, 1H), 2.81 (dd, J = 14.4, 2.4 Hz, 1H), 2.20 (s, 3H); ¹³C NMR (150 MHz, CDCl₃): δ = 170.1, 168.3, 146.2, 139.5, 135.1, 130.9, 130.5, 129.0, 128.6, 128.2, 128.0, 127.9, 126.4, 116.0, 78.7, 66.0, 59.5, 53.4, 52.5, 31.6, 20.5; IR (thin film): ν_{max} = 3062, 3028, 2950, 2923, 2855, 1742, 1508, 1437, 1258, 1232, 1177, 1085, 1049, 987, 952, 816, 743; HRMS calc'd for C₂₇H₂₇O₅N = 445.1890, found 445.1886; cis conformation confirmed by X-Ray diffraction.



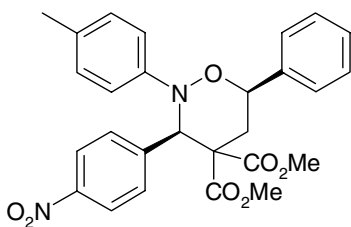
Compound 11

77 % yield, 20 hrs; colorless prisms, m.p. 200-201 °C; Rf = 0.40 (30%, EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 7.54 (d, J = 8.0 Hz, 2H), 7.45 (t, J = 6.8 Hz, 2H), 7.41-7.37 (m, 1H), 7.28 (d, J = 1.6 Hz, 1H), 7.00-6.92 (m, 5H), 6.62 (d, J = 8.0 Hz, 1H), 5.87 (app. s, 2H), 5.67 (s, 1H), 5.00 (dd, J = 12.0, 3.2 Hz, 1H), 3.91 (s, 3H), 3.53 (s, 3H), 2.83 (dd, J = 14.4, 11.6 Hz, 1H), 2.76 (dd, J = 14.4, 2.8 Hz, 1H), 2.20 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 170.1, 168.3, 147.2, 147.2, 146.1, 139.3, 131.0, 129.1, 128.7, 128.6, 128.3, 126.5, 124.3, 115.9, 110.7, 107.6, 100.9, 78.7, 65.4, 59.6, 53.4, 52.7, 31.4, 20.6; IR (thin film): ν_{max} = 3028, 2951, 2928, 2919, 1741, 1610, 1506, 1446, 1370, 1253, 1040, 935, 879, 818, 734, 699; HRMS calc'd for C₂₈H₂₇O₇N = 489.1788, found 489.1786.



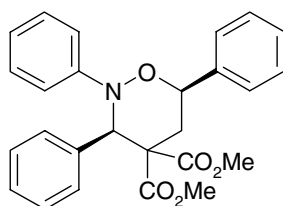
Compound 12

85 % yield, 7.5 hrs; colorless prisms, m.p. 170-172 °C; Rf = 0.56 (30%, EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 7.56 (d, J = 7.2 Hz, 2H), 7.51 (d, J = 8.8 Hz, 2H), 7.46 (t, J = 7.2 Hz, 2H), 7.41-7.31 (m, 6H), 6.99 (d, J = 8.8 Hz, 2H), 6.95 (d, J = 8.8 Hz, 2H), 6.80 (d, J = 8.8 Hz, 2H), 5.70 (s, 1H), 5.02 (dd, J = 11.6, 2.6 Hz, 1H), 4.95 (s, 2H), 3.92 (s, 3H), 3.48 (s, 3H), 2.84 (dd, J = 14.4, 11.6 Hz, 1H), 2.77 (dd, J = 14.4, 2.8 Hz, 1H), 2.19 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 170.2, 168.4, 158.4, 146.3, 139.6, 136.8, 131.7, 130.9, 129.1, 128.6, 128.5, 128.2, 128.0, 127.6, 127.4, 126.5, 116.0, 114.2, 78.7, 69.8, 65.5, 59.3, 53.4, 52.6, 31.5, 20.5; IR (thin film): ν_{max} = 3033, 2950, 2868, 1742, 1607, 1509, 1437, 1250, 1177, 1116, 1081, 1049, 953, 822, 742, 698; HRMS calc'd for C₃₄H₃₃O₆N = 551.2309, found 551.2308.



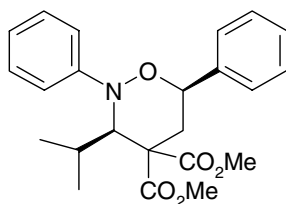
Compound 13

84 % yield, 20 hrs; yellow prisms, m.p. 181-182 °C; $R_f = 0.47$ (30%, EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 8.06$ (d, $J = 9.0$ Hz, 2H), 7.78 (d, $J = 9.0$ Hz, 2H), 7.54 (d, $J = 7.6$ Hz, 2H), 7.48 (t, $J = 7.2$ Hz, 2H), 7.44-7.39 (m, 1H), 6.96 (s, 4H), 5.85 (s, 1H), 5.07 (dd, $J = 12.0, 2.4$ Hz, 1H), 3.95 (s, 3H), 3.52 (s, 3H), 2.86 (dd, $J = 14.8, 2.8$ Hz, 1H), 2.76 (dd, $J = 14.8, 12.0$ Hz, 1H), 2.18 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.4, 167.9, 147.5, 145.7, 142.8, 138.9, 131.8, 131.4, 129.3, 128.7, 128.5, 126.4, 123.0, 115.9, 78.7, 65.8, 59.2, 53.7, 52.9, 31.5, 20.5$; IR (thin film): $\nu_{\text{max}} = 2954, 2922, 1742, 1521, 1456, 1436, 1348, 1260, 1232, 1210, 1178, 1050, 861, 822, 750$; HRMS calc'd for $\text{C}_{27}\text{H}_{26}\text{O}_7\text{N}_2 = 490.1740$, found 490.1742.



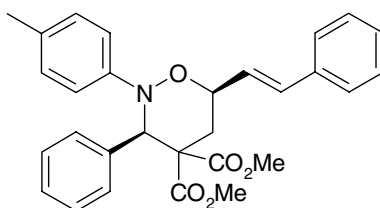
Compound 14

92 % yield, 15 hrs; colorless prisms, m.p. 169-171 °C; $R_f = 0.50$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.60$ -7.56 (m, 4H), 7.47 (t, $J = 7.4$ Hz, 2H), 7.42-7.38 (m, 1H), 7.22-7.09 (m, 7H), 6.82 (tt, $J = 7.2, 1.6$ Hz, 1H), 5.80 (s, 1H), 5.03 (dd, $J = 11.8, 2.8$ Hz, 1H), 3.93 (s, 3H), 3.48 (s, 3H), 2.87 (dd, $J = 11.8, 14.4$ Hz, 1H), 2.78 (dd, $J = 14.4, 2.8$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.0, 168.2, 148.5, 139.4, 135.0, 130.4, 128.6, 128.5, 128.3, 128.1, 128.0, 126.5, 121.6, 115.7, 78.8, 65.7, 59.5, 53.5, 52.6, 31.6$; IR (thin film): $\nu_{\text{max}} = 3032, 2954, 1740, 1652, 1598, 1558, 1540, 1493, 1455, 1233, 1051, 950, 882, 811, 752$; HRMS calc'd for $\text{C}_{26}\text{H}_{25}\text{O}_5\text{N} = 431.1731$, found 431.1733.



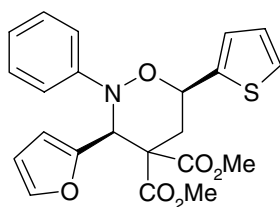
Compound 15

80 % yield, 19 hrs; colorless cubes, m.p. 90-91 °C; $R_f = 0.53$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.47$ (d, $J = 7.6$ Hz, 2H), 7.44-7.40 (m, 2H), 7.38-7.34 (m, 1H), 7.22 (dt, $J = 1.2, 8.8$ Hz, 2H), 7.04 (dd, $J = 8.8, 1.2$ Hz, 2H), 6.79 (dt, $J = 1.2, 7.4$ Hz, 1H), 4.81 (dd, $J = 9.0, 7.4$ Hz, 1H), 4.65 (d, $J = 7.6$ Hz, 1H), 3.80 (s, 3H), 3.76 (s, 3H), 2.72-2.63 (m, 2H), 2.44 (o, $J = 7.0$ Hz, 1H), 0.91 (d, $J = 7.0$ Hz, 3H), 0.86 (d, $J = 7.0$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.4, 169.5, 149.2, 139.5, 128.8, 128.5, 128.3, 126.4, 119.1, 112.2, 79.2, 66.5, 58.6, 53.1, 52.7, 33.3, 29.4, 22.2, 21.9$; IR (thin film): $\nu_{\text{max}} = 3063, 2956, 1738, 1597, 1494, 1436, 1244, 1058, 965, 750$; HRMS calc'd for $\text{C}_{23}\text{H}_{27}\text{O}_5\text{N} = 397.1889$, found 397.1890; Cis conformation confirmed by X-ray diffraction.



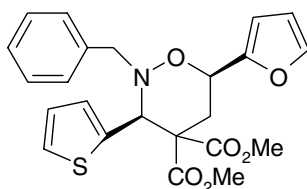
Compound 16

85 % yield, 8 hrs; colorless needles, m.p. 141-142 °C; $R_f = 0.36$ (20% EtOAc in hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): $\delta = 7.56-7.53$ (m, 2H), 7.51-7.49 (d, $J = 7.2$ Hz, 2H), 7.37 (dd, $J = 7.8, 7.8\text{Hz}$, 2H), 7.31-7.28 (m, 1H), 7.20-7.18 (m, 3H), 6.98 (d, $J = 8.1$ Hz, 2H), 6.95 (d, $J = 8.1$ Hz, 2H), 6.81 (d, $J = 16.2$ Hz, 1H), 6.41 (dd, $J = 16.2, 6.4$ Hz, 1H), 5.66 (s, 1H), 4.69-4.65 (m, 1H), 3.91 (s, 3H), 3.47 (s, 3H), 2.69 (dd, $J = 13.6, 11.6$ Hz, 1H), 2.66 (dd, $J = 13.6, 2.4$ Hz, 1H), 2.18 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.0, 168.3, 146.2, 136.6, 135.0, 132.5, 131.0, 130.5, 129.0, 128.6, 128.05, 128.01, 127.9, 127.0, 126.6, 116.1, 77.1, 66.1, 59.1, 53.4, 52.6, 30.6, 20.5$; IR (thin film): $\nu_{\text{max}} = 3057, 3026, 2951, 2928, 1741, 1612, 1508, 1438, 1239, 1200, 1075, 965, 816, 747, 698$; HRMS calc'd for $\text{C}_{29}\text{H}_{29}\text{O}_5\text{N} = 471.2047$, found 471.2034, Cis conformation confirmed by X-Ray diffraction.



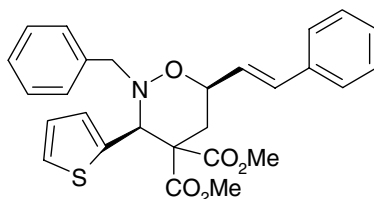
Compound 17

96 % yield, 18 hrs; colorless solid, m.p. 100-101 °C; $R_f = 0.47$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): $\delta = 7.37$ (d, $J = 5.1$ Hz, 1H), 7.25-7.18 (m, 4H), 7.09 (d, $J = 8.4$ Hz, 2H), 7.05 (dd, $J = 3.6, 5.1$ Hz, 1H), 6.93 (t, $J = 7.5$ Hz, 1H), 6.33 (d, $J = 3.0$ Hz, 1H), 6.21-6.20 (m, 1H), 5.84 (s, 1H), 5.27 (dd, $J = 11.4, 3.0$ Hz, 1H), 3.91 (s, 3H), 3.65 (s, 3H), 2.93 (dd, $J = 14.0, 3.6$ Hz, 1H), 2.86 (dd, $J = 14.0, 11.4$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.0, 167.8, 148.2, 141.8, 141.6, 128.5, 126.6, 125.7, 125.4, 122.4, 116.2, 111.1, 110.2, 74.7, 61.7, 58.1, 53.4, 52.9, 32.7$; IR (thin film): $\nu_{\text{max}} = 3006, 2955, 1742, 1597, 1541, 1493, 1436, 1241, 1082, 1034, 986, 961, 869, 804, 750$; HRMS calc'd for $\text{C}_{22}\text{H}_{21}\text{O}_6\text{NS} = 427.1088$, found 427.1093.



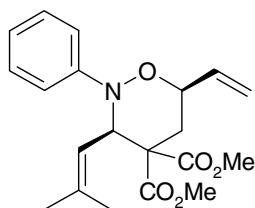
Compound 18

93 % yield, 15 hrs; colorless, low melting solid, m.p 55-58 °C ; $R_f = 0.50$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.46$ -7.45 (m, 1H), 7.35-7.28 (m, 4H), 7.24-7.22 (m, 2H), 7.02 (d, $J = 3.2$ Hz, 2H), 6.51 (d, $J = 3.0$ Hz, 1H), 6.40 (dd, $J = 3.0, 2.0$ Hz, 1H), 5.18 (s, 1H), 5.07 (dd, $J = 12.4, 2.4$ Hz, 1H), 4.01 (d, $J = 13.0$ Hz, 1H), 3.78 (s, 3H), 3.53 (d, $J = 13.0$ Hz, 1H), 3.49 (s, 3H), 2.93 (dd, 14.4 12.4 Hz, 1H), 2.64 (dd, $J = 14.4, 2.4$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.2, 168.0, 152.2, 142.8, 135.9, 133.6, 130.2, 129.2, 128.1, 127.4, 125.6, 110.3, 108.7, 71.7, 62.0, 59.2, 59.0, 53.1, 52.6, 28.5$; IR (thin film): $\nu_{\text{max}} = 3063, 3031, 2953, 2902, 1744, 1499, 1435, 1262, 1215, 1156, 1076, 1045, 1013, 962, 917, 887, 863, 742$; HRMS calc'd for $\text{C}_{23}\text{H}_{23}\text{O}_6\text{NS} = 441.1246$, found 441.1252.



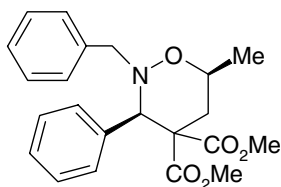
Compound 19

93 % yield, 15 hrs; colorless prisms, m.p. 161-162 °C; $R_f = 0.50$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.43$ (d, $J = 7.6$ Hz, 2H), 7.35-7.28 (m, 9H), 7.02 (dd, $J = 4.6, 3.4$ Hz, 1H), 6.98-6.97 (m, 1H), 6.73 (d, $J = 16.0$ Hz, 1H), 6.28 (dd, $J = 16.0, 6.2$ Hz, 1H), 5.18 (s, 1H), 4.66-4.60 (m, 1H), 3.99 (d, $J = 12.8$ Hz, 1H), 3.80 (s, 3H), 3.56 (d, $J = 12.8$ Hz, 1H), 3.48 (s, 3H), 2.59-2.48 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.3, 168.1, 136.5, 136.2, 133.7, 132.4, 130.3, 129.1, 128.5, 128.1, 127.9, 127.4, 127.4, 127.3, 126.7, 125.5, 76.6, 62.2, 59.2, 59.0, 53.0, 52.6, 30.7$; IR (thin film): $\nu_{\text{max}} = 3030, 2952, 1743, 1495, 1452, 1259, 1210, 1130, 1047, 955, 919, 747, 701$; HRMS calc'd for $\text{C}_{27}\text{H}_{27}\text{O}_5\text{NS} = 477.1610$, found 477.1577; Cis conformation confirmed by X-ray diffraction.



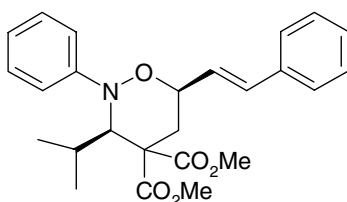
Compound 20

66 % yield, 17 hrs; colorless needles, m.p. 125-127 °C; $R_f = 0.58$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.23$ (t, $J = 7.2$ Hz, 2H), 7.06 (dd, $J = 7.2, 1.2$ Hz, 2H), 6.91 (tt, $J = 7.2, 1.2$ Hz, 1H), 5.96 (ddd, $J = 17.6, 10.8, 7.2$ Hz, 1H), 5.46-5.40 (m, 2H), 5.28 (dt, $J = 10.4, 1.2$ Hz, 1H), 5.23 (d, $J = 10.0$ Hz, 1H), 4.42-4.37 (m, 1H), 3.86 (s, 3H), 3.66 (s, 3H), 2.52 (dd, $J = 9.2, 1.6$ Hz, 1H), 2.26 (dd, $J = 14.4, 10.0$ Hz, 1H), 1.59 (s, 3H), 1.49 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.0, 168.6, 149.1, 139.7, 135.9, 128.4, 121.8, 117.2, 116.2, 115.0, 77.4, 61.6, 58.5, 53.2, 52.6, 30.9, 26.0, 18.6$; IR (thin film): $\nu_{\text{max}} = 2953, 1742, 1598, 1492, 1437, 1235, 1199, 1157, 1077, 1030, 935, 904, 757$; HRMS calc'd for $\text{C}_{20}\text{H}_{25}\text{O}_5\text{N} = 359.1733$, found 359.1735.



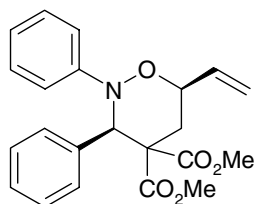
Compound 21

66 % yield, 17 hrs; colorless oil; $R_f = 0.43$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.52\text{-}7.50$ (m, 2H), 7.32-7.24 (m, 6H), 7.19 (d, $J = 6.4$ Hz, 2H), 4.75 (broad s, 1H), 4.07-3.96 (m, 1H), 3.85 (d, $J = 13.6$ Hz, 1H), 3.80 (s, 3H), 3.53 (d, $J = 13.6$ Hz, 1H), 3.34 (broad s, 3H), 2.38 (dd, $J = 14.2, 3.0$ Hz, 1H), 2.30 (dd, $J = 14.2, 11.4$ Hz, 1H), 1.27 (broad d, $J = 4.8$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.1, 168.6, 136.9, 135.0, 131.1, 128.7, 128.1, 128.2, 127.3, 72.1, 65.6, 59.2, 59.1, 52.9, 52.3, 32.1, 20.1$; IR (thin film): $\nu_{\text{max}} = 3030, 2953, 1744, 1495, 1454, 1259, 1203, 1116, 953, 777, 748$; HRMS calc'd for $\text{C}_{22}\text{H}_{25}\text{O}_5\text{N} = 383.1732$, found 383.1737.



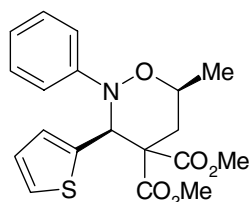
Compound 22

75 % yield, 15 hrs; colorless oil; $R_f = 0.60$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): $\delta = 7.44$ (d, $J = 7.2$ Hz, 2H), 7.35 (t, $J = 7.2$ Hz, 2H), 7.29-7.27 (m, 1H), 7.23 (dd, $J = 8.4, 7.2$ Hz, 2H), 7.04 (d, $J = 8.4$ Hz, 2H), 6.79 (t, $J = 7.2$ Hz, 1H), 6.74 (d, $J = 16.2$ Hz, 1H), 6.28 (dd, $J = 16.2, 6.0$ Hz, 1H), 4.59 (d, $J = 8.4$ Hz, 1H), 4.47-4.43 (m, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 2.59 (dd, $J = 14.4, 3.6$ Hz, 1H), 2.52 (dd, $J = 14.4, 11.4$ Hz, 1H), 2.38 (o, $J = 7.2$ Hz, 1H), 0.89 (d, $J = 6.6$ Hz, 3H), 0.82 (d, $J = 6.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.3, 169.5, 149.2, 136.3, 132.8, 128.7, 128.6, 128.0, 126.7, 126.6, 119.0, 112.1, 77.6, 66.3, 58.3, 53.1, 52.7, 32.2, 29.1, 22.0, 21.9$; IR (thin film): $\nu_{\text{max}} = 3060, 3028, 2991, 2954, 1739, 1597, 1494, 1451, 1435, 1242, 1198, 1175, 1065, 966, 748$; HRMS calc'd for $\text{C}_{25}\text{H}_{29}\text{O}_5\text{N} = 423.2045$, found 423.2043.



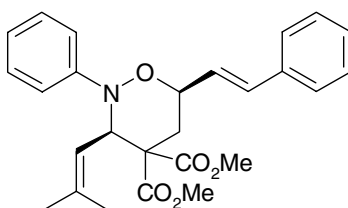
Compound 23

87 % yield, 17 hrs; colorless prisms, m.p. 138-139 °C; $R_f = 0.47$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.53\text{-}7.50$ (m, 2H), 7.21-7.13 (m, 5H), 7.08-7.06 (m, 2H), 6.84-6.80 (m, 1H), 6.08 (ddd, $J = 6.8, 10.8, 17.6$ Hz, 1H), 5.69 (s, 1H), 5.52 (dt, $J = 17.6, 1.4$ Hz, 1H), 5.36 (dt, $J = 10.8, 1.4$ Hz, 1H), 4.52-4.47 (m, 1H), 3.89 (s, 3H), 3.46 (s, 3H), 2.59-2.57 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.0, 168.2, 148.5, 135.9, 134.8, 130.4, 128.5, 128.0, 127.9, 121.6, 117.3, 115.8, 77.1, 65.8, 59.0, 53.4, 52.6, 30.3$; IR (thin film): $\nu_{\text{max}} = 3063, 3029, 2954, 1743, 1598, 1493, 1452, 1436, 1234, 1203, 1153, 1081, 1032, 991, 942, 915, 814, 756$; HRMS calc'd for $\text{C}_{22}\text{H}_{23}\text{O}_5\text{N} = 381.1576$ found 381.1576.



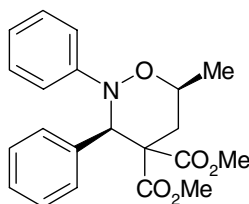
Compound 24

85 % yield, 15 hrs; colorless prisms, m.p. 148-149 °C; $R_f = 0.53$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.20\text{-}7.15$ (m, 3H), 7.11-7.09 (m, 2H), 6.90-6.86 (m, 3H), 6.82-6.80 (m, 1H), 6.02 (s, 1H), 4.17-4.10 (m, 1H), 3.87 (s, 3H), 3.56 (s, 3H), 2.53-2.42 (m, 2H), 1.45 (d, $J = 6.8$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.6, 168.0, 148.4, 134.0, 129.0, 128.4, 127.0, 125.3, 122.1, 116.1, 73.4, 63.9, 59.3, 53.4, 52.8, 32.6, 19.8$; IR (thin film): $\nu_{\text{max}} = 2983, 2953, 1740, 1597, 1494, 1432, 1386, 1249, 1155, 1127, 1082, 1045, 946, 896, 850, 759$; HRMS calc'd for $\text{C}_{19}\text{H}_{21}\text{O}_5\text{NS} = 375.1140$, found 375.1140; Cis conformation confirmed by X-ray diffraction.



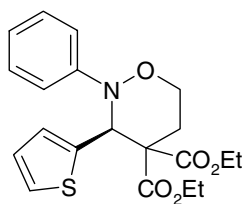
Compound 25

89 % yield, 2 hrs; colorless prisms, m.p. 132-134 °C; $R_f = 0.53$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): major diastereomer: $\delta = 7.40$ (d, $J = 7.6$ Hz, 2H), 7.34 (t, $J = 8.0$ Hz, 2H), 7.29-7.22 (m, 3H), 7.10 (dd, $J = 8.6$ Hz, 1.0 Hz, 2H), 6.93 (tt, $J = 7.6$ Hz, 1.0 Hz, 1 H), 6.74 (d, $J = 16.0$ Hz, 1H), 6.31 (dd, $J = 16.0$ Hz, 5.8 Hz, 1H), 5.50 (dt, $J = 10.5, 1.2$ Hz, 1H), 5.26 (d, $J = 10.5$ Hz, 1H), 4.61-4.56 (m, 1H), 3.88 (s, 3H), 3.67 (s, 3H), 2.58 (dd, $J = 14.0, 2.0$ Hz, 1H), 2.38 (dd, $J = 14.0, 12.0$ Hz, 1H), 1.61 (d, $J = 1.2$ Hz, 3H), 1.51 (d, $J = 1.2$ Hz, 3H); representative peaks for minor diastereomer: $\delta = 6.51$ (d, $J = 16.4$ Hz, 1H), 6.41 (dd, $J = 16.4, 5.2$ Hz, 1H), 5.55 (dt, $J = 10.5, 1.2$, 1H), 5.11 (d, $J = 10.5$, 1H), 4.98-4.92 (m, 1H), 3.68 (s, 3H), 3.59 (s, 3H), 2.87 (dd, $J = 14.4, 6.0$ Hz, 1H), 2.49 (dd, $J = 14.4, 2.4$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): major diastereomer: $\delta = 170.0, 168.7, 149.1, 139.8, 136.4, 132.5, 128.6, 128.4, 128.0, 127.0, 126.6, 121.9, 116.3, 115.1, 77.5, 61.6, 58.6, 53.2, 52.7, 31.3, 26.1, 18.7$; IR (thin film): $\nu_{\text{max}} = 3029, 2953, 1741, 1598, 1493, 1438, 1379, 1240, 1196, 1074, 1031, 967, 790, 753$; HRMS calc'd for $\text{C}_{26}\text{H}_{29}\text{O}_5\text{N} = 435.2045$, found 435.2040.



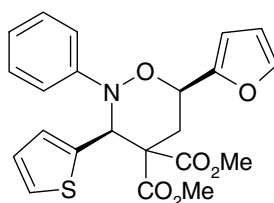
Compound 26

80 % yield, 20 hrs; colorless cubes, m.p. 194-195 °C; $R_f = 0.53$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.53$ -7.51 (m, 2H), 7.19-7.12 (m, 5H), 7.05 (dd, $J = 8.8, 1.2$ Hz, 2H), 6.80 (tt, $J = 6.4, 1.2$ Hz, 1H), 5.68 (s, 1H), 4.13-4.08 (m, 1H), 3.87 (s, 3H), 3.46 (s, 3H), 2.47-2.44 (m, 2H), 1.46 (d, $J = 6.4$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 170.2, 168.4, 148.6, 135.1, 130.4, 128.5, 128.0, 127.9, 121.3, 115.6, 73.2, 65.4, 59.1, 53.3, 52.5, 32.4, 19.9$; IR (thin film): $\nu_{\text{max}} = 3058, 2996, 2949, 1732, 1597, 1495, 1449, 1298, 1255, 1201, 1156, 1129, 975, 810, 753$; HRMS calc'd for $\text{C}_{21}\text{H}_{23}\text{O}_5\text{N} = 369.1576$, found 369.1572.



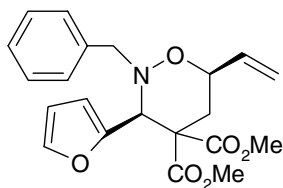
Compound 27

72 % yield, 40 hrs, (20 mol% Yb(OTf)₃ was used); colorless prisms, m.p. 93-94 °C; R_f = 0.58 (30% EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): δ 7.20-7.15 (m, 3H), 7.07 (d, J = 7.7, 2H), 6.90-6.85 (m, 2H), 6.80 (ddd, J= 5.2, 3.6, 0.8 Hz, 1H), 6.01 (s, 1H), 4.39-4.30 (m, 3H), 4.13-3.92 (m, 3H), 2.86 (ddd, J= 14.8, 13.6, 5.6 Hz, 1H); 2.49 (broad d, J = 14.8 Hz, 1H), 1.34 (t, J = 7.2 Hz, 3H), 1.10 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 168.9, 167.7, 148.4, 134.02, 129.2, 128.4, 126.8, 125.2, 122.1, 116.2, 67.6, 64.7, 62.3, 61.8, 58.4, 25.7, 14.1, 13.8; IR (thin film): ν_{max} = 2982, 2934, 2870, 1737, 1597, 1492, 1452, 1367, 1233, 1198, 1153, 1106, 1067, 1048, 1022, 757, 697; HRMS calc'd for C₂₀H₂₃O₅NS = 389.1297, found 389.1305.



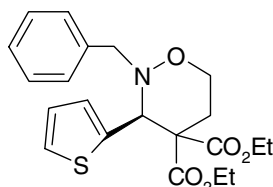
Compound 28

90% yield, 20 hrs; colorless prisms, m.p. 153-154 °C; R_f = 0.48 (30% EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 7.52-7.51 (m, 1H), 7.18 (d, J = 8.4 Hz, 2H), 7.15 (d, J = 2.4 Hz, 1H), 7.09 (d, J = 8.4 Hz, 2H), 6.98 (d, J = 5.2 Hz, 1H), 6.87 (t, J = 7.2 Hz, 1H), 6.84-6.82 (m, 1H), 6.59 (d, J = 3.2 Hz, 1H), 6.46-6.44 (m, 1H), 6.07 (s, 1H), 5.10 (dd, J = 12.8, 2.4 Hz, 1H), 3.91 (s, 3H), 3.59 (s, 3H), 3.10 (dd, J = 12.8, 14.4, 1H), 2.75 (dd, J = 14.4, 2.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 169.3, 167.7, 151.6, 148.1, 143.0, 133.8, 129.2, 128.5, 127.1, 125.4, 122.4, 116.2, 110.4, 109.1, 72.5, 64.1, 59.4, 53.5, 52.9, 28.6; IR (thin film): ν_{max} = 2955, 1741, 1596, 1493, 1434, 1267, 1246, 1210, 1175, 1152, 1089, 1069, 1045, 1014, 940, 916, 852, 753; HRMS calc'd for C₂₂H₂₁O₆NS = 427.1088, found 427.1093.



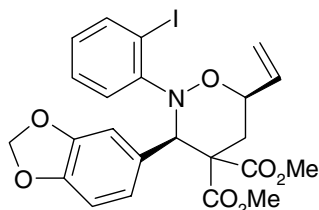
Compound 29

89 % yield, 15 hrs; colorless oil; $R_f = 0.52$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.43\text{-}7.42$ (m, 1H), 7.33-7.23 (m, 5H), 6.44 (d, $J = 3.2$ Hz, 1H), 6.39-6.37 (m, 1H), 5.88 (ddd, $J = 17.6, 10.6, 6.0$ Hz, 1H), 5.35 (dt, $J = 17.6, 1.2$ Hz, 1H), 5.22 (dt, $J = 10.6, 1.2$ Hz, 1H), 4.89 (s, 1H), 4.50-4.45 (m, 1H), 3.91 (d, $J = 13.0$ Hz, 1H), 3.75 (s, 3H), 3.55 (d, $J = 13.0$ Hz, 1H), 3.51 (s, 3H), 2.52 (ddd, $J = 14.0, 2.4, 1.2$ Hz, 1H), 2.29 (dd, $J = 14.0, 12.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 169.2, 168.3, 148.5, 142.2, 136.2, 129.0, 128.0, 127.2, 117.0, 111.7, 110.2, 76.3, 59.6, 59.0, 57.8, 52.9, 52.7, 30.8$; IR (thin film): $\nu_{\text{max}} = 3031, 2954, 1744, 1497, 1435, 1264, 1209, 1155, 1084, 1015, 931, 806, 743$; HRMS calc'd for $\text{C}_{21}\text{H}_{23}\text{O}_6\text{N} = 385.1525$, found 385.1529.



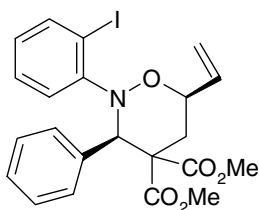
Compound 30

70 % yield, 39 hrs, (20 mol% $\text{Yb}(\text{OTf})_3$ was used); colorless oil; $R_f = 0.48$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.34\text{-}7.23$ (m, 6H), 7.01-6.99 (m, 2H), 5.25 (s, 1H), 4.37-4.20 (m, 2H), 4.07-3.85 (m, 4H), 3.76 (d, $J = 13.8$ Hz, 1H), 3.55 (d, $J = 13.8$ Hz, 1H), 2.66 (m, 1H), 2.33 (d, $J = 13.6$ Hz, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.04 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 168.9, 167.9, 136.8, 134.2, 130.0, 128.7, 128.1, 127.2, 127.1, 125.4, 67.0, 63.6, 61.9, 61.6, 59.3, 58.4, 25.3, 14.0, 13.7$; IR (thin film): $\nu_{\text{max}} = 2980, 2961, 2924, 2861, 1738, 1455, 1437, 1366, 1259, 1211, 1152, 1123, 1097, 1073, 1046, 1023, 997, 955, 860$; HRMS calc'd for $\text{C}_{21}\text{H}_{25}\text{O}_5\text{NS} = 403.1456$, found 403.1453.



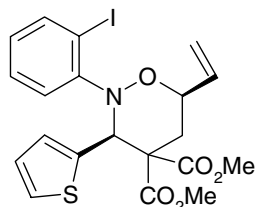
Precursor to compound 31

65 % yield, 20 hrs, 2:1 mixture of anisotropic isomers. Major isomer was isolated cleanly by recrystallization, colorless crystals, m.p. 139-140 °C; $R_f = 0.38$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): major isomer $\delta = 7.80$ (d, $J = 8.0$ Hz, 1H), 7.19 (broad s, 1H), 7.02 (t, $J = 7.6$ Hz, 1H), 6.86 (t, $J = 8.4$ Hz, 2H), 6.74 (t, $J = 7.6$ Hz, 1H), 6.65 (d, $J = 8.0$ Hz, 1H), 6.03 (ddd, $J = 16.4, 10.4, 4.8$ Hz, 1H), 5.94 (d, $J = 1.4$ Hz, 1H), 5.93 (d, $J = 1.4$ Hz, 1H), 5.46 (dt, $J = 16.4, 1.2$ Hz, 1H), 5.34 (s, 1H), 5.31 (dt, $J = 10.4, 1.2$ Hz, 1H), 4.96-4.88 (m, 1H), 3.90 (s, 3H), 3.48 (s, 3H), 2.62 (dd, $J = 14.4, 2.8$ Hz, 1H), 2.52 (dd, $J = 14.4, 12.0$ Hz, 1H); $^1\text{H-NMR}$ (400 MHz, CDCl_3): representative peaks for minor isomer $\delta = 5.83$ (s, 2H), 5.78 (ddd, $J = 16.8, 10.4, 6.0$ Hz, 1H), 5.65-5.59 (m, 1H), 5.21 (d, $J = 10.8$ Hz, 1H), 3.73 (s, 3H), 3.52 (s, 3H), 2.67 (dd, $J = 13.6, 3.2$ Hz, 1H), 1.98 (dd, $J = 13.8, 9.8$ Hz, 1H); IR (thin film): $\nu_{\text{max}} = 3127, 2953, 2897, 1741, 1661, 1641, 1550, 1530, 1488, 1466, 1442, 1250, 1039, 932, 800, 761$; HRMS calc'd for $\text{C}_{23}\text{H}_{22}\text{O}_7\text{NI} = 551.0440$, found: 551.0439.



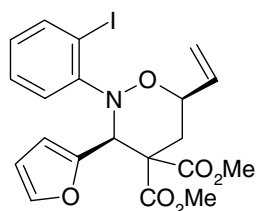
Precursor to compound 32

73 % yield, 22 hrs; 9:7 mixture of anisotropic isomers after one recrystallization; m.p. 120-128 °C; $R_f = 0.54$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): major isomer $\delta = 7.48$ (dd, $J = 1.6, 6.8$ Hz, 2H), 7.25-7.19 (m, 3H), 7.13 (ddd, $J = 1.6, 8.4, 8.4$ Hz, 1H), 7.09-7.04 (m, 3H, 2 H major, 1H minor), 6.95 (ddd, $J = 1.4, 8.4, 8.4$ Hz, 1H), 6.06 (ddd, $J = 5.2, 10.4, 16.8$, 1H), 5.50-5.44 (m, 1H major, 1H minor), 5.41-5.36 (m, 1H major, 1H minor), 5.31 (ddd, $J = 1.4, 1.4, 9.2$ Hz, 1H), 4.97-4.92 (m, 1H), 3.92 (s, 3H), 3.41 (s, 3H), 2.71-2.53 (m, 2H, major, 1H, minor), $^1\text{H-NMR}$ (400 MHz, CDCl_3): minor isomer $\delta = 7.78$ (dt, $J = 1.6, 8.0$ Hz, 2H), 7.31-7.29 (dd, $J = 1.6, 8.0$ Hz, 2H), 6.77-6.96 (m, 4H), 5.81 (ddd, $J = 6.4, 10.8, 16.8$, 1H), 5.68-5.63 (m, 1H), 5.22 (ddd, $J = 1.4, 1.4, 9.2$ Hz, 1H), 3.72 (s, 3H), 3.44 (s, 3H), 2.00 (dd, $J = 13.6, 10.0$ Hz, 1H), IR (thin film): $\nu_{\text{max}} = 3058, 3023, 2949, 2901, 1736, 1579, 1464, 1436, 1255, 1147, 1084, 1021, 989, 933, 766, 720$; HRMS calc'd for $\text{C}_{22}\text{H}_{22}\text{O}_5\text{NI} = 507.0543$, found 507.0542.



Precursor to compound 33

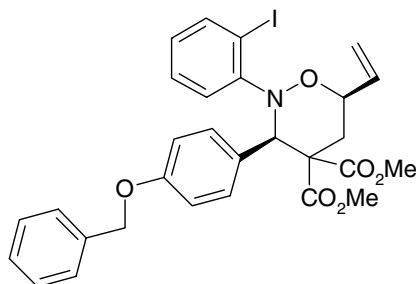
75 % yield of pure product isolated (85% of product recovered), 17 hrs; 3:1 mixture of anisotropic isomers, after one recrystallization the ratio was 5.5:1; colorless plates, m.p. 111-130 °C; Rf = 0.53 (30% EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): major isomer δ = 7.82 (dd, J = 8.0, 1.2 Hz, 1H), 7.01 (dt, J = 7.2, 1.2 Hz, 1H), 6.96 (m, 1H), 6.91 (dd, J = 5.0, 3.4 Hz, 1H), 6.85 (dd, J = 8.0, 1.2 Hz, 1H), 6.76 (dt, J = 1.2 Hz, 8.0 Hz, 1H), 6.00 (ddd, J = 17.0, 10.8, 6.0 Hz, 1H), 5.78 (s, 1H), 5.49 (dt, J = 17.0, 1.2 Hz, 1H), 5.32 (dt, J = 10.8, 1.2 Hz, 1H), 5.07-5.02 (m, 1H), 3.90 (s, 3H), 3.55 (s, 3H), 2.63 (dd, J = 14.6, 3.2 Hz, 1H), 2.51 (dd, J = 14.6, 11.8 Hz, 1H); ¹H-NMR (400 MHz, CDCl₃): representative peaks for minor isomer δ = 5.72 (s, 1H), 5.21 (dt, J = 10.4, 1.2 Hz, 1H), 3.77 (s, 3H), 3.53 (s, 3H), 2.78 (dd, J = 14.0, 4.2 Hz, 1H), 2.13 (dd, J = 14.0, 9.2 Hz, 1H); IR (thin film): ν_{max} = 3067, 2998, 2952, 2843, 1744, 1577, 1465, 1434, 1262, 1148, 1081, 1020, 988, 931, 904, 856, 760, 709; HRMS calc'd for C₂₀H₂₀O₅NSI = 513.0106, found 513.0099.



Precursor to compound 34

90 % yield, 17 hrs, 3.5:1 mixture of anisotropic isomers. Major isomer could be isolated cleanly by column chromatography, colorless prisms, m.p. 138-139 °C; Rf = 0.50 (30% EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): major isomer δ = 7.81 (dd, J = 7.8, 1.4 Hz, 1H), 7.32 (m, 1H), 7.10-7.05 (m, 1H), 6.80 (dt, J = 1.6, 8.0 Hz, 1H), 6.72 (dd, J = 8.0, 1.2 Hz, 1H), 6.29-6.26 (m, 2H), 5.96 (ddd, J = 17.2, 10.6, 6.4 Hz, 1H), 5.44 (dt, J = 17.2, 1.6 Hz, 1H), 5.41 (s, 1H), 5.28 (dt, J = 10.6, 1.6 Hz, 1H), 5.00-4.96 (m, 1H), 3.89 (s, 3H), 3.57 (s, 3H), 2.65 (ddd, J = 14.2, 2.4, 1.2 Hz, 1H), 2.38 (dd, J = 14.2, 11.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): major isomer δ = 168.9, 168.4, 148.8, 148.6, 141.8, 139.6, 135.9, 128.2, 127.6, 123.4, 117.4, 111.8, 110.2, 94.0, 77.3, 60.9, 57.7, 53.4, 52.8, 30.7; IR (thin film): ν_{max} = 2998, 2953, 1745, 1645, 1580, 1497, 1465, 1435, 1266, 1151, 1085, 1017, 991, 932, 905, 867, 807, 748; HRMS calc'd for C₂₀H₂₀O₆NI = 497.0332, found 497.0325. The Heck reaction was performed on both the major

and major/minor isomer mixture. The same product was obtained in 79% and 78% yield respectively.

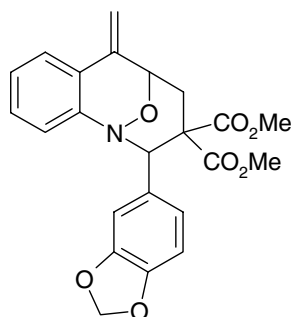


Precursor to compound 35

70 % yield, 5 hrs, 2.5:1 mixture of anisotropic isomers. After one recrystallization the minor isomer was favoured, 1:7.5. colorless prisms, m.p. 175-179 °C; R_f = 0.44 (30% EtOAc in hexanes); ¹H-NMR (400 MHz, CDCl₃): major isomer, representative peaks. δ = 6.09-6.01 (m, 1H), 5.47 (dt, J = 17.2, 1.2 Hz, 1H), 5.36 (s, 1H), 5.31 (dt, J = 10.8, 1.2 Hz, 1H), 5.01 (s, 2H), 3.91 (s, 3H), 3.42 (s, 3H), 2.64 (dd, J = 14.4, 2.8 Hz, 1H), 2.56 (dd, J = 14.4, 12.0 Hz); ¹H-NMR (400 MHz, CDCl₃): minor isomer, representative peaks. δ = 7.77 (dd, J = 8.0, 1.2 Hz, 1H), 7.21 (d, J = 8.4 Hz, 2H), 7.14 (dt, 1.6, 8.0 Hz, 1H), 7.09 (dd, J = 8.0, 1.2 Hz, 1H), 6.79-6.75 (m, 1H), 6.70 (d, J = 8.8 Hz, 2H), 5.81 (ddd, J = 17.2, 10.6, 6.4 Hz, 1H), 5.66 (m, 1H), 5.38 (s, 1H), 5.38 (dt, J = 17.2, 1.4 Hz, 1H), 5.21 (dt, J = 10.6, 1.2 Hz, 1H), 4.91 (d, J = 1.2 Hz, 2H), 3.72 (s, 3H), 3.45 (s, 3H), 2.68 (dd, J = 13.8, 3.4 Hz, 1H), 1.99 (dd, J = 13.8, 10.0 Hz, 1H); IR (thin film): ν_{max} = 3033, 2952, 1733, 1610, 1582, 1511, 1466, 1435, 1250, 1086, 1059, 1020, 933, 840, 740; HRMS calc'd for C₂₉H₂₈O₆NI = 613.0961, found 613.0961.

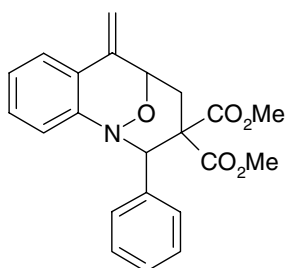
Typical Experimental Procedure for Heck Coupling

To a solution of the iodocycloadduct 4 (0.4 mmol) in CH₃CN (5 mL) was added triethylamine (2.0 mmol) and Pd(PPh₃)₄ (20 mol %). The reaction mixture was heated at reflux under an atmosphere of argon until TLC indicated complete consumption of the starting material. The reaction mixture was cooled to room temperature and directly absorbed onto silica gel by reduced pressure removal of the solvent. Pure product was obtained after flash column chromatography on silica gel (EtOAc and hexanes). Samples could be recrystallized from CH₂Cl₂/hexanes if desired



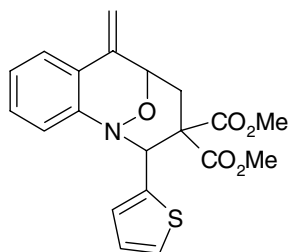
Compound 31

81 % yield, 16 hrs; yellow solid, decomposes at 66 °C; $R_f = 0.34$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.63$ (dd, $J = 8.0, 1.2$ Hz, 1H), 7.37 (d, $J = 1.6$ Hz, 1H), 7.29-7.25 (m, 1H), 7.10 (dt, $J = 1.2, 8.0$ Hz, 1H), 7.04 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.00 (dd, $J = 8.0, 1.2$ Hz, 1H), 6.78 (d, $J = 8.4$ Hz, 1H), 5.97 (d, $J = 1.4$ Hz, 1H), 5.96 (d, $J = 1.4$ Hz, 1H), 5.74 (s, 1H), 5.22 (s, 1H), 5.05 (s, 1H), 5.02-4.99 (m, 1H), 3.37 (s, 3H), 3.32 (s, 3H), 3.27 (dd, $J = 14.6, 6.0$ Hz, 1H), 2.53 (dd, $J = 14.6, 2.4$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.8, 168.8, 147.6, 147.1, 146.5, 138.2, 132.7, 129.1, 124.9, 123.2, 122.8, 122.4, 121.8, 109.4, 107.7, 107.6, 101.0, 71.6, 67.7, 52.9, 52.6, 52.0, 30.8$; IR (thin film): $\nu_{\text{max}} = 2952, 1737, 1651, 1490, 1442, 1252, 1039, 912, 760, 734$; HRMS calc'd for $\text{C}_{23}\text{H}_{21}\text{O}_7\text{N} = 423.1317$, found 423.1320.



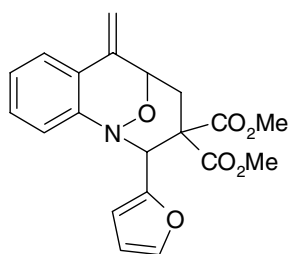
Compound 32

73 % yield, 18 hrs; yellow solid, decomposes at 60 °C; $R_f = 0.35$ (25 % EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.71$ -7.68 (m, 2H), 7.65 (dd, $J = 1.6, 8.0$ Hz, 1H), 7.38-7.31 (m, 3H), 7.27-7.25 (m, 1H), 7.12-7.08 (m, 1H), 7.01 (dd, $J = 1.2, 8.0$ Hz, 1H), 5.74 (d, $J = 0.8$ Hz, 1H), 5.31 (s, 1H), 5.06 (d, $J = 0.8$ Hz, 1H), 5.05-5.02 (m, 1H), 3.34 (s, 3H), 3.29 (dd, $J = 14.4, 6.4$ Hz, 1H), 3.27 (s, 3H), 2.54 (ddd, $J = 0.8, 2.8, 14.4$ Hz, 1H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): $\delta = 169.9, 168.9, 146.6, 139.0, 138.3, 129.1, 128.7, 128.2, 128.0, 124.8, 123.1, 122.8, 121.7, 107.5, 71.6, 68.3, 52.9, 52.5, 52.1, 30.9$; IR (thin film): $\nu_{\text{max}} = 3060, 3027, 2948, 2927, 2922, 1737, 1627, 1601, 1482, 1436, 1251, 1202, 1073, 1024, 953, 910, 759$; HRMS calc'd for $\text{C}_{22}\text{H}_{21}\text{O}_5\text{N} = 379.1420$, found 379.1421



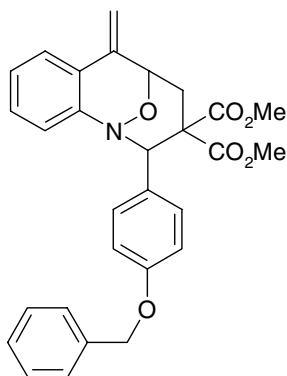
Compound 33

49 % yield, 42 hrs; colorless plates, m.p. 156-157 °C; $R_f = 0.42$ (30 % EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.65$ (dd, $J = 8.0, 1.2$ Hz, 1H), 7.31 (m, 1H), 7.28-7.26 (m, 1H), 7.14-7.07 (m, 3H), 6.97 (dd, $J = 4.8, 3.6$ Hz, 1H), 5.76 (s, 1H), 5.73 (s, 1H), 5.06 (s, 1H), 4.98 (d, $J = 6.4$ Hz, 1H), 3.42 (s, 3H), 3.38 (dd, $J = 14.4, 6.0$ Hz, 1H), 3.23 (s, 3H), 2.67 (dt, $J = 14.4, 1.2$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 168.9, 168.3, 145.0, 139.0, 137.7, 129.0, 127.2, 126.4, 125.4, 125.3, 123.9, 122.50, 122.47, 107.9, 71.8, 63.9, 52.82, 52.79, 51.5, 30.2$; IR (thin film): $\nu_{\text{max}} = 3072, 2952, 1740, 1455, 1435, 1312, 1250, 1205, 1072, 1047, 1025, 912, 779, 759, 729$; HRMS calc'd for $\text{C}_{20}\text{H}_{19}\text{O}_5\text{NS} = 385.0983$, found 385.0984.



Compound 34

79 % yield, 24 hrs; colorless needles, m.p. 152-154 °C; $R_f = 0.36$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.66$ (dt, $J = 8.0, 0.8$ Hz, 1H), 7.39 (m, 1H), 7.28 (t, $J = 8.0$ Hz, 1H), 7.13-7.18 (m, 2H), 6.59 (dd, $J = 3.4, 0.8$ Hz, 1H), 6.40-6.39 (m, 1H), 5.75 (s, 1H), 5.49 (s, 1H), 5.05 (s, 1H), 4.91 (d, $J = 6.4$ Hz, 1H), 3.48 (s, 3H), 3.25 (dd, $J = 14.6, 6.2$ Hz, 1H), 3.22 (s, 3H), 2.71 (dt, $J = 14.6, 1.2$ Hz, 1H), $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 168.8, 168.6, 151.6, 145.1, 141.7, 137.9, 129.0, 125.2, 124.0, 122.6, 122.4, 110.6, 109.2, 107.8, 72.1, 61.8, 53.0, 52.9, 50.2, 30.8$; IR (thin film): $\nu_{\text{max}} = 3151, 3124, 3084, 2998, 2953, 1826, 1738, 1651, 1636, 1456, 1434, 1255, 1203, 1182, 1078, 1023, 976, 920, 913, 882, 802, 758$; HRMS calc'd for $\text{C}_{20}\text{H}_{19}\text{O}_6\text{N} = 369.1212$, found 379.1209.



Compound 35

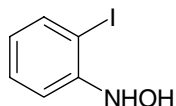
69 % yield, 20 hrs; orange, low melting solid, $R_f = 0.40$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.66\text{-}7.61$ (m, 3H), 7.44 (d, $J = 6.8$ Hz, 2H), 7.39 (t, $J = 7.2$ Hz, 2H), 7.35-7.31 (m, 1H), 7.29-7.25 (m, 1H), 7.10 (dt, $J = 1.2, 8.0$ Hz, 1H), 7.02 (dd, $J = 8.0, 1.2$ Hz, 1H), 6.96 (d, $J = 6.8$ Hz, 2H), 5.74 (s, 1H), 5.27 (s, 1H), 5.08 (s, 2H), 5.03 (s, 1H), 5.02 (dd, $J = 6.8, 1.6$ Hz, 1H), 3.32 (s, 3H), 3.30 (dd, $J = 14.6, 6.0$ Hz, 1H), 3.29 (s, 3H), 3.57 (dd, $J = 14.6, 2.2$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 169.8, 168.9, 158.3, 146.5, 138.3, 136.9, 131.3, 130.0, 129.0, 128.5, 127.9, 127.5, 124.8, 123.2, 122.7, 121.8, 114.5, 107.5, 71.6, 69.9, 67.5, 52.8, 52.5, 51.9, 30.7$; IR (thin film): $\nu_{\text{max}} = 3033, 2951, 1737, 1652, 1610, 1510, 1456, 1435, 1248, 1180, 1074, 1023, 957, 912, 840, 759$; HRMS calc'd for $\text{C}_{29}\text{H}_{27}\text{O}_6\text{N} = 485.1837$, found 485.1832.

Preparation of 2-iodophenylhydroxylamine

A modification of the procedure: V. Gautheron-Chapoulaud, S. U. Pandya, P. Cividino, G. Masson, S. Py, Y. Vallee, *Synlett* **2001**, 1281, was used to produce the desired hydroxylamine. The original procedure reduces nitro compounds to hydroxylamines in the presence of aldehydes to form nitrones. In this modification the aldehyde is omitted, allowing isolation of the hydroxylamine. The procedure is not optimized.

To an ice cold solution of 2-iodonitrobenzene (8.00 mmol) in 95% ethanol (50 mL) was added zinc powder (12.0 mmol). Acetic acid (24 mmol) was then added dropwise and the reaction stirred for 10 minutes TLC indicated that starting material still remained, but the reaction was filtered to remove the zinc, avoiding over reduction to the amine. The ethanol was removed under reduced pressure, and the crude material was dissolved in methylene chloride, and directly absorbed onto silica gel by reduced pressure removal of the solvent. Flash column chromatography (ethyl acetate in hexanes as eluent) produced pure product. The product is initially an orange crystalline solid and should be used immediately, as it turns brown after

approximately 12 hours at room temperature. The product could be stored in the freezer for a few days without deterioration.



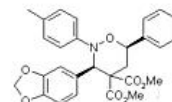
Compound 8d

64% yield, 10 mins; orange solid; $R_f = 0.50$ (30% EtOAc in hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.68$ (d, $J = 8.0$ Hz, 1H), 7.33 (t, $J = 7.4$ Hz, 1H), 7.24 (d, $J = 7.4$ Hz, 1H), 6.88 (broad s, 1H), 6.73 (t, $J = 7.4$ Hz, 1H), 5.86 (broad s, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 148.7, 138.5, 129.2, 123.7, 115.7, 82.4$; IR (thin film): $\nu_{\text{max}} = 3500\text{-}3200$ broad, 3259, 2926, 1652, 1583, 1464, 1395, 1287, 1006, 754; HRMS calc'd for $\text{C}_6\text{H}_6\text{ONI} = 234.9494$, found 234.9499.

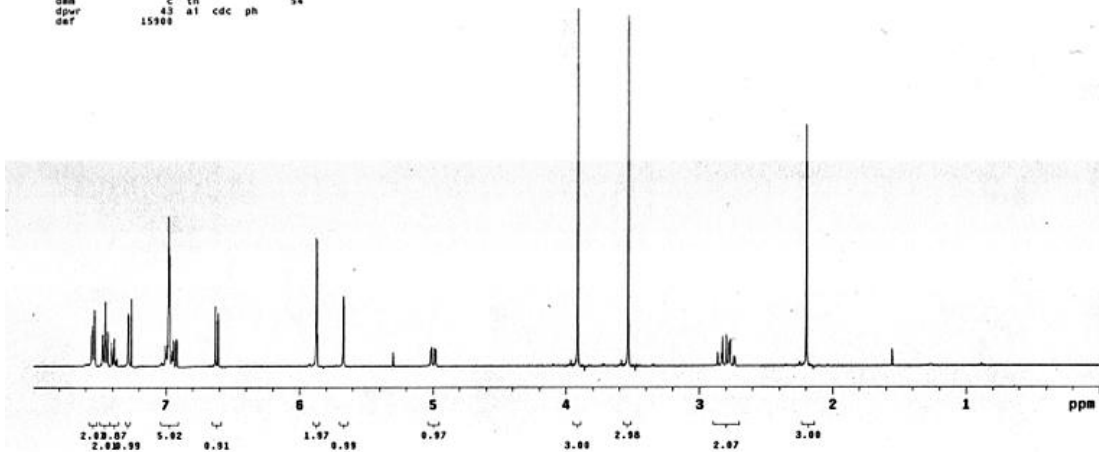
IV-3-25recryst
STANDARD 1H OBSERVE

exp12 s2pul

SAMPLE		SPECIAL	
date	Apr 16 2003	temp	not used
solvent	cdcl3	gain	not used
file /export/home/	spin	spin	20
nmr1/wakeup/auto-	hst	hst	0.000
18.08.03/0101	pw0	pw0	14.000
ACQUISITION	alfa	alfa	20.000
sw	6402.0	FLAGS	n
at	4.000	l1	n
np	51716	in	n
fb	not used	dp	y
bs	16	hs	nm
d1	1.000	PROCESSING	
nt	0	fn	not used
ct	0	DISPLAY	
TRANSMITTER	sp	sp	-0.3
tn	H1	wp	3196.0
sfrq	400.000	rf1	3704.0
tof	417.3	rfp	2994.6
tpwr	57	rfp	-149.0
pw	7.300	lp	-89.5
DECOUPLER	C13	wc	250
dn	0	sc	0
dof	0	vs	30
dm	mn	vs	30
dnn	57	vs	34
dpr	43	a1	cdc ph
daf	15900		



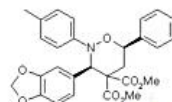
compound 11



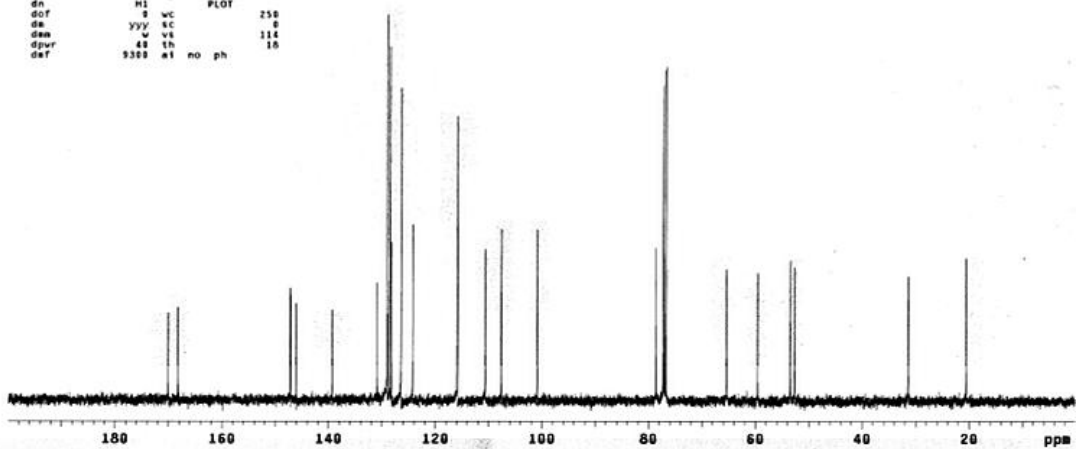
IV-3-25carbomh
STANDARD 1H OBSERVE

exp13 s2pul

SAMPLE		SPECIAL	
date	May 29 2003	temp	not used
solvent	cdcl3	gain	not used
file /export/home/	spin	spin	not used
nmr1/wakeup/auto-	hst	hst	0.000
18.08.03/0101	pw0	pw0	11.000
ACQUISITION	alfa	alfa	20.000
sw	25125.6	FLAGS	n
at	1.500	l1	n
np	60270	in	n
fb	13000	dp	y
bs	64	hs	nm
d1	1.000	PROCESSING	
nt	512	lb	1.00
ct	512	fn	not used
TRANSMITTER	C13	sp	10.1
tn	H1	wp	20100.0
sfrq	100.613	rf1	9270.4
tof	1550.0	rfp	7706.3
tpwr	57	rfp	-176.0
pw	5.000	lp	-290.7
DECOUPLER	H1	lp	
dn	0	wc	250
dof	0	sc	0
dm	xxx	vs	114
dnn	40	vs	114
dpr	40	th	10
daf	9300	a1	no ph



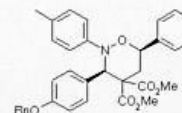
compound 11



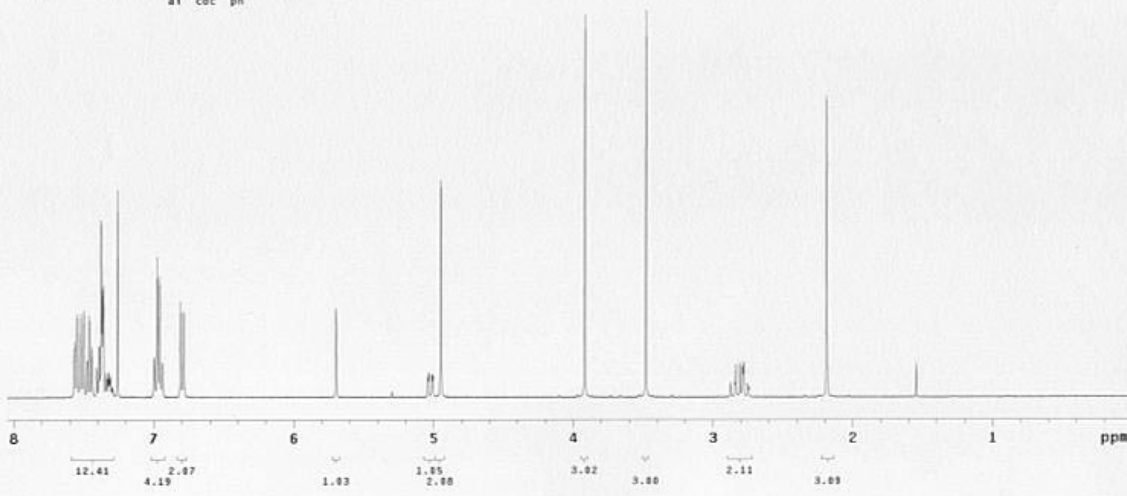
IV-3-35 (inova 400)

exp1 s2pu1

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SAMPLE SPECIAL
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solvent CDCl3 gain not used
file
ACQUISITION hst 0.000
sw 6395.9 pw90 14.500
at 3.744 a1fa 6.600
np 47692 FLAGS
fb 4000 l1 n
bs 16 in n
d1 1.000 dp y
nt 16 hs nn
ct 16 PROCESSING
TRANSMITTER fn not used
tn H1 DISPLAY
sfrq 399.762 sp -2.0
tof 399.0 wp 3210.9
tpwr 50 rfl 3605.0
pw 7.250 rfp 2992.3
DECOUPLER rp 103.7
dn C13 lp -25.1
dof 0 PLOT
da nnn wc 250
dwa c sc 0
dwr 32 vs 154
daf 17894 th 54
a1 cdc ph
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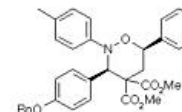
compound 12



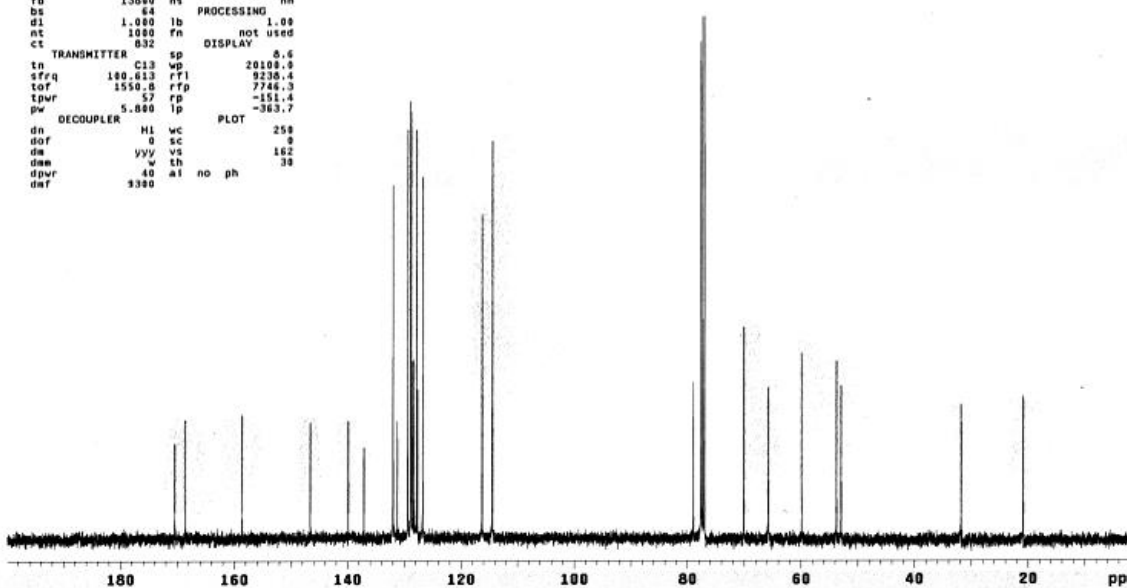
IV-3-35c
STANDARD 1H OBSERVE

exp11 s2pu1

```
SAMPLE SPECIAL
date Jun 11 2003 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin not used
vner1/vner1sys/deta hst 0.000
/lyoung/IV-3-35c/C- pw90 11.400
ARBON.fid a1fa 20.000
ACQUISITION FLAGS
sw 25125.6 l1 n
at 1.199 in n
np 68270 dp y
fb 13650 hs nn
bs 64 PROCESSING
d1 1.000 lb 1.00
nt 1000 fn not used
ct B32 DISPLAY
TRANSMITTER sp 0.6
tn C13 wp 20109.0
sfrq 100.613 rfl 9230.4
tof 1550.0 rfp 7746.3
tpwr 57 rp -151.4
pw 5.000 lp -363.7
DECOUPLER H1 wc 250
dof 0 sc 0
dwa yyv vs 162
dwr 40 al no ph
daf 5300
```



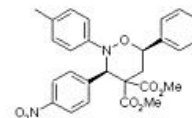
compound 12



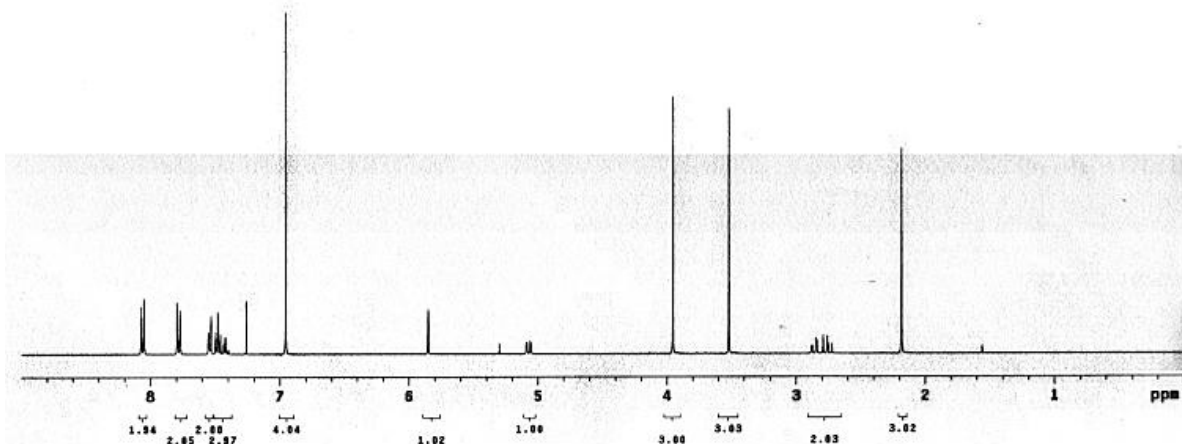
IV-3-37recryst
STANDARD IN OBSERVE

exp11 s2pul

SAMPLE		SPECIAL	
date	May 28 2003	temp	not used
solvent	cdc13	gain	not used
file	/export/home/~	spin	20
vnr1/vnr2/autoc	hst	hst	0.000
	28.05.03/0109	pw90	14.600
ACQUISITION		alfa	20.000
sv	6402.0	FLAGS	
at	4.000	l1	n
np	51216	ln	n
fb	not used	dp	y
bs	16	hc	nn
d1	1.000	PROCESSING	
nt	0	fn	not used
ct	0	DISPLAY	
TRANSMITTER		sp	-1.1
tr	H1	wp	3600.5
sfrq	400.050	rfl	3704.0
tof	417.3	rfp	2004.0
tpwr	57	rp	-153.0
pw	7.300	lp	-93.0
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
da	nnn	vs	19
daw	c	th	54
dpr	43	ai	cdc ph
def	15900		



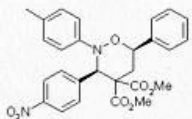
compound 13



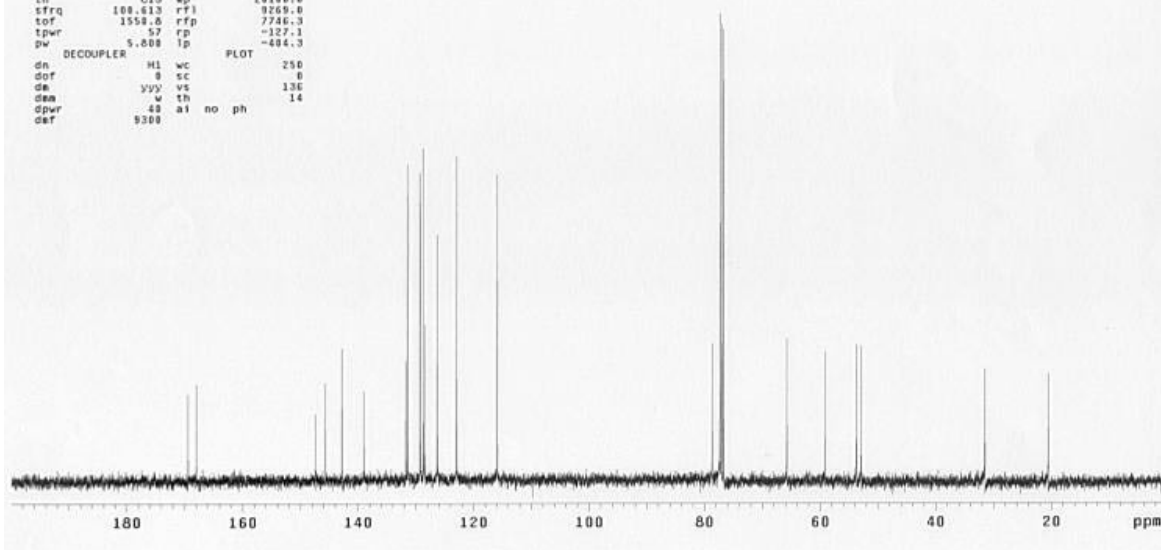
IV-3-37c
STANDARD IN OBSERVE

exp11 s2pul

SAMPLE		SPECIAL	
date	May 28 2003	temp	not used
solvent	cdc13	gain	not used
file	/export/home/~	spin	not used
vnr1/vnr2/autoc	hst	hst	0.000
/tyoung/IV-3-37c/C-	pw90	pw90	11.000
	ARBON.F1d	alfa	20.000
ACQUISITION		FLAGS	
lv	25225.6	l1	n
at	1.100	ln	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	
d1	1.000	lb	1.00
nt	512	fn	not used
ct	512	DISPLAY	
TRANSMITTER		sp	13.6
tr	C13	wp	20100.0
sfrq	100.613	rfl	9269.0
tof	1550.0	rfp	7746.3
tpwr	57	rp	-127.1
pw	5.000	lp	-404.3
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
da	yyy	vs	130
daw	w	th	14
dpr	43	ai	no ph
def	9300		



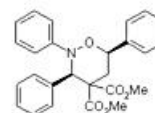
compound 13



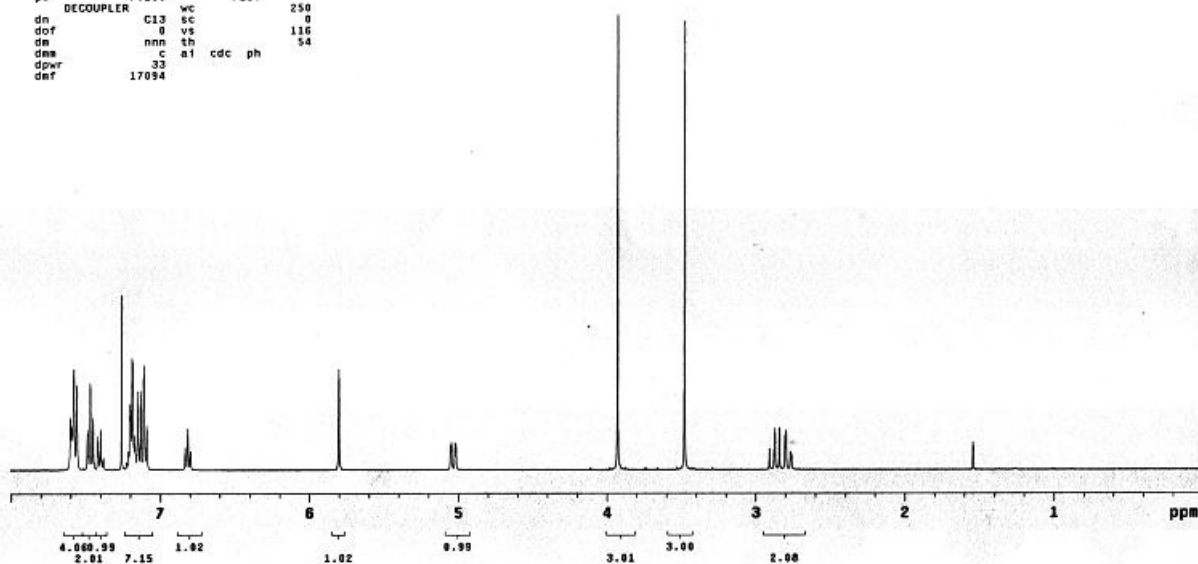
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IV-3-189
exp1 s2pu1
SAMPLE
date Jun 11 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin 29
vcorr/vmrcyc/data/~ hst 0.008
IV-3-189_11Jun2003- pw90 14.500
/PROTON.fid alfa 6.600
ACQUISITION FLAGS
sw 6395.9 f1 n
at 3.744 in n
np 47692 dp y
fb 4000 hs nn
bs 16 PROCESSING
dl 1.000 fn not used
nt 16 DISPLAY -2.0
ct 16 sp 3282.1
TRANSMITTER H1 rF1 3605.8
tn wp 2982.3
sfrq 399.762 rfp 183.4
tof 399.8 rp -23.3
tpwr 50 lp
pv 7.250 PLOT 250
DECOUPLER wc 0
dn C13 sc 116
dof 0 vs 54
dm nnn th
dms c a1 cdc ph
dpwr 32
def 17094

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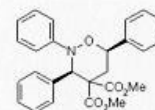
compound 14



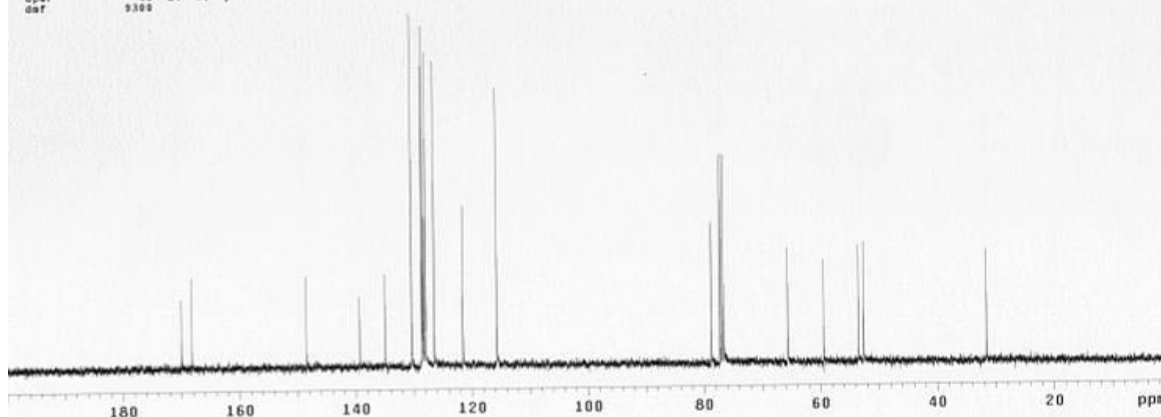
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IV-3-189C
STANDARD 1H OBSERVE
exp11 s2pu1
SAMPLE
date Jun 11 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
vcorr/vmrcyc/data/~ hst 0.008
/lyoung/IV-3-189c- pw90 11.600
CARBON.fid alfa 20.000
ACQUISITION FLAGS
sw 25125.6 f1 n
at 1.199 in n
np 69276 dp y
fb 13500 hs nn
bs 64 PROCESSING
dl 1.000 lb 1.00
nt 512 fn not used
ct 256 DISPLAY 7.0
TRANSMITTER C13 wp 20133.0
tn wf 9272.1
sfrq 100.613 rF1 7746.3
tof 1558.0 rfp -179.7
tpwr 57 rp -322.7
pv 5.890 lp
DECOUPLER H1 wc 250
dn 0 sc 0
dof yyy vs 64
dms w th 9
dm 40 a1 no ph
dpwr 9300
def

```

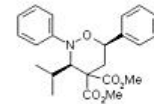


compound 14

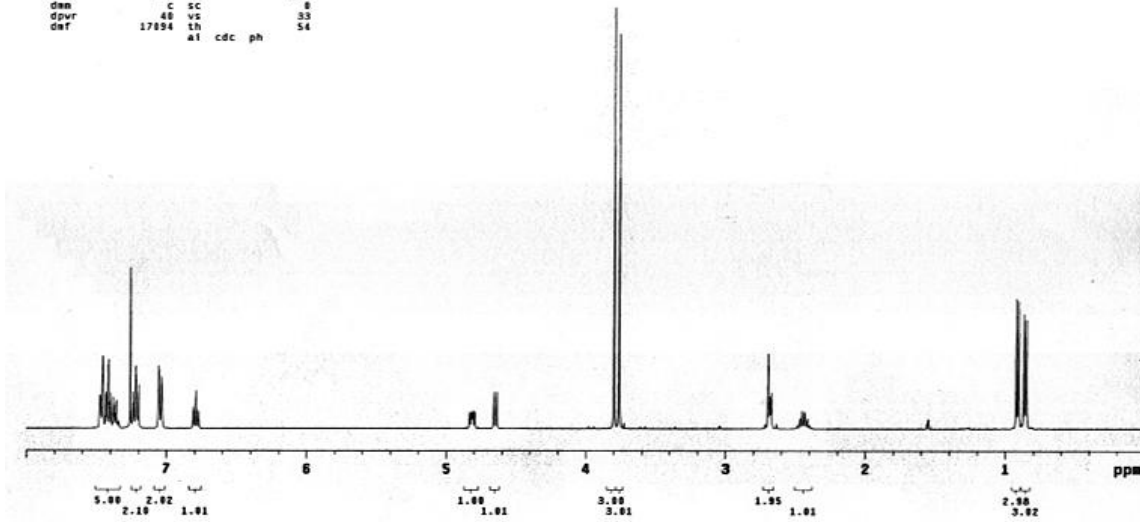


Iy-3-281 (Inova 400)
 expl s2pu1

SAMPLE		SPECIAL	
date	Jun 28 2003	temp	not used
solvent	CDCl3	gain	not used
file		exp	spin
ACQUISITION		ht	8.000
sw	6395.3	pwd8	9.700
at	3.744	atfa	6.600
fp	47892	FLAGS	
fb	6000	ll	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nm
ct	16	PROCESSING	
TRANSMITTER		fn	not used
tn	M1	DISPLAY	
sfrq	399.762	sp	-3.2
tof	399.8	wp	3202.1
tpwr	52	rfl	3607.0
pw	4.050	rfp	2902.3
DECOUPLER		rp	29.3
dn	C13	tp	-19.2
dof	0	PLOT	
dm	mn	vc	250
dsm	c	sc	0
dpwr	40	vs	33
def	17094	th	54
	al	cdc	ph



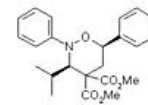
compound 15



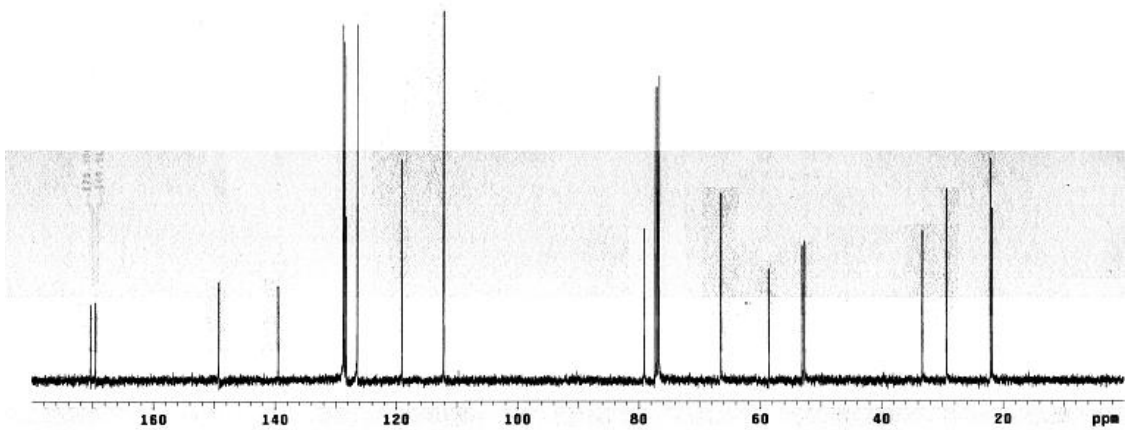
IY-3-201C
 STANDARD 1H OBSERVE

expl s2pu1

SAMPLE		SPECIAL	
date	Jun 28 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/-	spin	not used
nmr1/wkexp/auto-		ht	8.000
20.06.03/0203		pwd0	11.000
ACQUISITION		atfa	20.000
sw	25125.6	FLAGS	
at	1.100	ll	n
fp	60270	in	n
fb	12000	dp	y
bs	64	hs	nm
d1	1.000	PROCESSING	
nt	512	tb	1.00
ct	512	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	12.4
sfrq	100.613	wp	10002.6
tof	1550.0	rfl	9209.0
tpwr	57	rfp	7746.3
pw	5.000	rp	103.4
DECOUPLER		tp	-202.4
dn	H1	PLOT	
dof	0	vc	250
dm	yvy	sc	0
dsm	w	vs	99
dpwr	40	th	8
def	9300	al	no ph



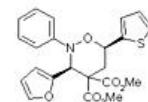
compound 15



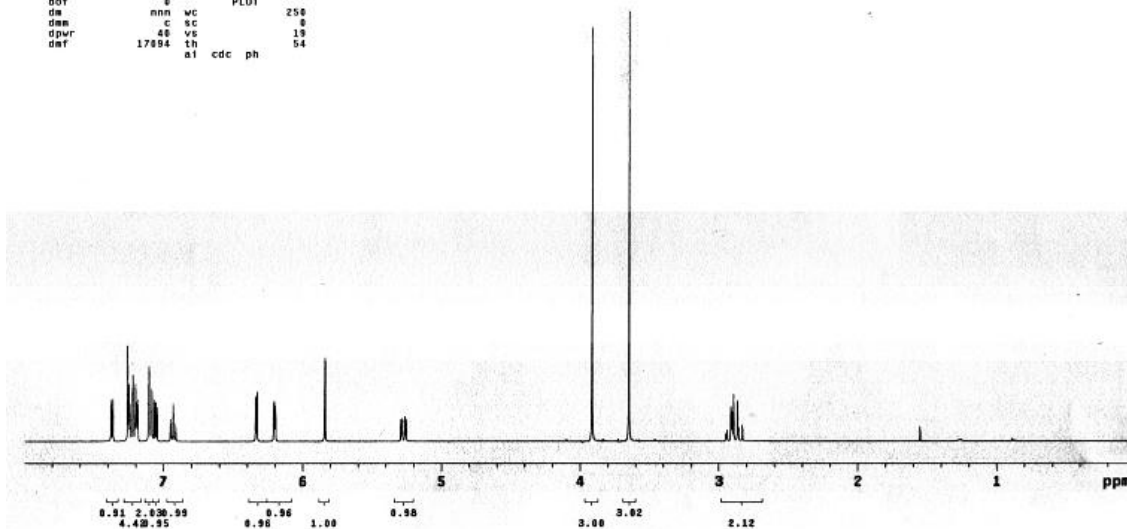
IV-3-199 rec Inova 400

expl s2pu1

```
SAMPLE SPECIAL
date Jul 5 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin 20
vmer/valkup/aut0.~ het 0.000
ACQUISITION dsc 0.000
sw 6395.9 pw90 9.700
at 3.744 alfa 6.600
np 4792 FLAGS
fb 4000 f1 n
bs 16 in n
d1 1.000 dp y
nt 16 hs nn
ct TRANSMITTER 16 fn PROCESSING not used
tn H1 DISPLAY 2.1
sfrq 309.762 sp 3193.6
tof 309.6 wp 3690.1
tpwr 4.859 rfp 2902.3
pw DECOUPLER rfp 61.2
dn C13 1p -9.7
dof 0 PLDT
dm nnn wc 250
dmm c sc 0
dpr 48 vc 19
dof 17894 th 54
cdc ph
```



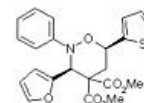
compound 17



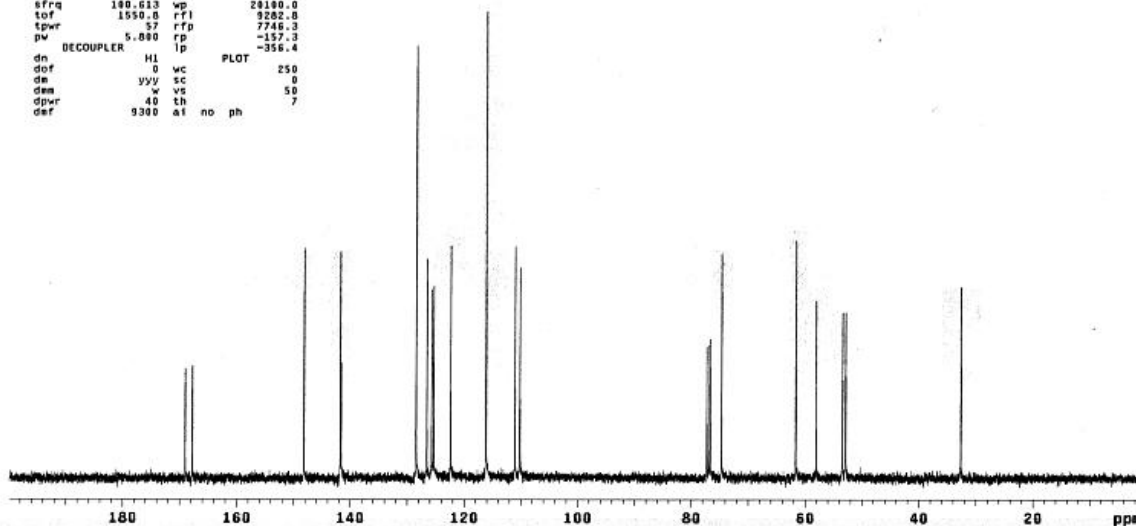
IV-3-199c
STANDARD IN OBSERVE

expl s2pu1

```
SAMPLE SPECIAL
date Jun 16 2003 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vmer/valkup/aut0.~ het 0.000
ACQUISITION dsc 11.600
sw 16.86.03/0703 pw90 20.000
at 25125.6 FLAGS
np 1.199 f1 n
fb 08279 in n
bs 13600 dp y
d1 64 hs nn
d1 1.000 dp PROCESSING 1.00
nt 512 fb fn not used
ct TRANSMITTER 126 fn DISPLAY 4.7
tn H1 DISPLAY 4.7
sfrq 100.613 sp 20100.0
tof 1550.8 wp 9282.0
tpwr 53 rfp 7746.3
pw DECOUPLER rfp -157.3
dn C13 1p -356.4
dof 0 PLDT
dm 0 wc 250
dmm yyy sc 0
dpr 40 vc 50
dof 9300 th 7
at no ph
```



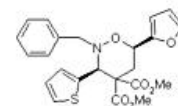
compound 17



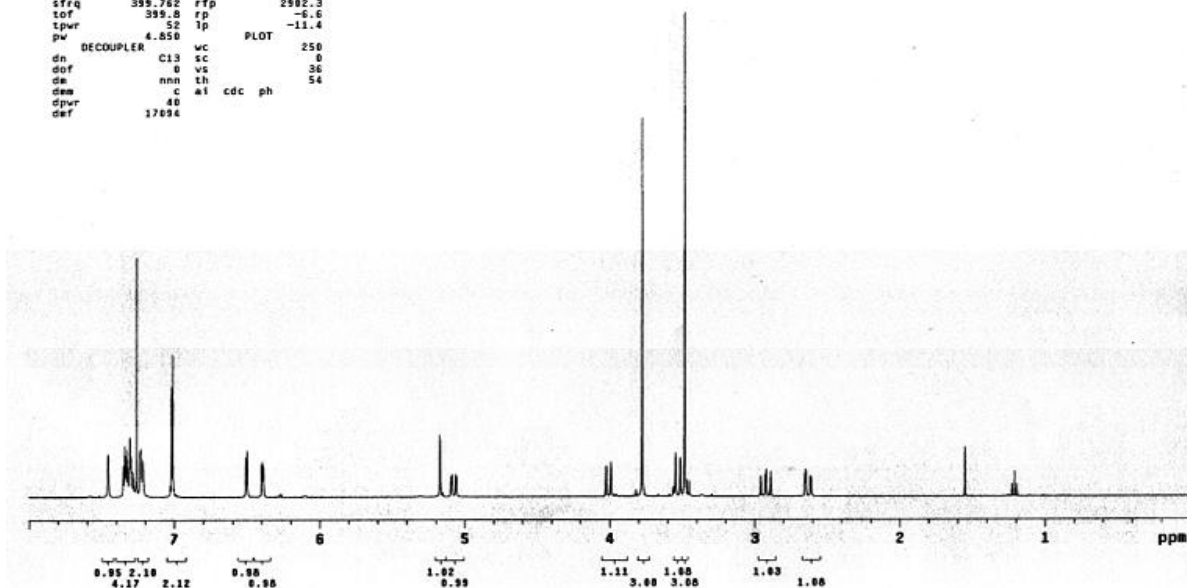
IY-4-95 solid Inova 400

exp1 s2pul

SAMPLE		SPECIAL	
date	Jul 31 2003	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/	spin	20
kerr/vnmr/sy/data/	hst	0.088	
IY-4-95solid_31jul	pv99	9.700	
2003/PROTD4.rfd	alfa	5.000	
ACQUISITION		FLAGS	
sw	6395.9	l1	n
at	3.744	in	n
np	47892	dp	y
fb	4800	hs	nn
bs	16		
d1	1.000	fn	not used
nt	16		
ct	16	sp	-3.8
TRANSMITTER		DISPLAY	
tn	H1	rfl	3292.1
sfrq	399.762	rff	3687.6
tof	399.8	rfp	2982.3
tpwr	52	lp	-6.6
pw	4.850		-11.4
DECOUPLER		PLOT	
dn	C13	sc	250
dof	0	vs	0
dm	nm1	lh	36
dwm	c	al	cdc ph
dpwr	40		54
def	17934		



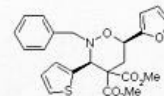
compound 18



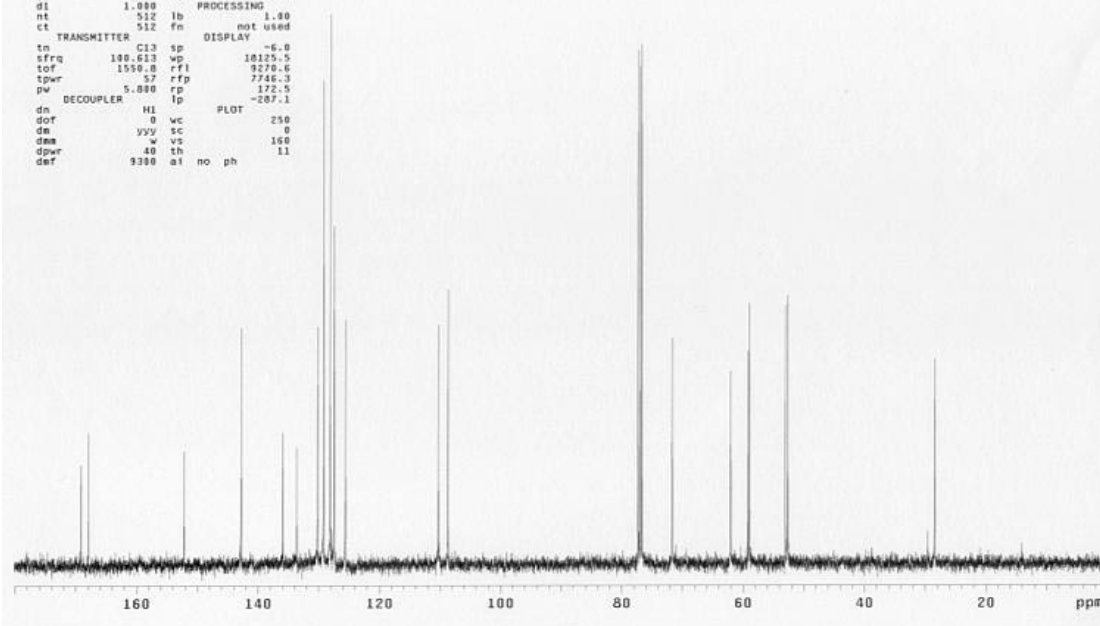
IY-4-95c
STANDARD 1H OBSERVE

exp11 s2pul

SAMPLE		SPECIAL	
date	Jul 13 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/	spin	not used
vnmr/wakeup/	auto	hst	0.088
13.97.03/9392	pv99	11.000	
ACQUISITION		alpha	
sv	25125.0		
at	1.199	l1	n
np	68270	in	n
fb	13800	dp	y
bs	64	hs	nn
d1	1.000		
nt	512	lb	1.00
ct	512	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-6.0
sfrq	100.613	wp	10125.5
tof	1550.8	rfl	9270.6
tpwr	57	rfp	7746.3
pw	5.000	rp	172.5
deco	HI	lp	-207.1
DECOUPLER		PLOT	
dn	0	vc	250
dof	yy	sc	0
dm	w	vs	160
dwm	40	lh	11
dpwr	9390	al	no ph
def			



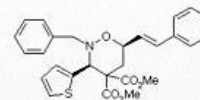
compound 18



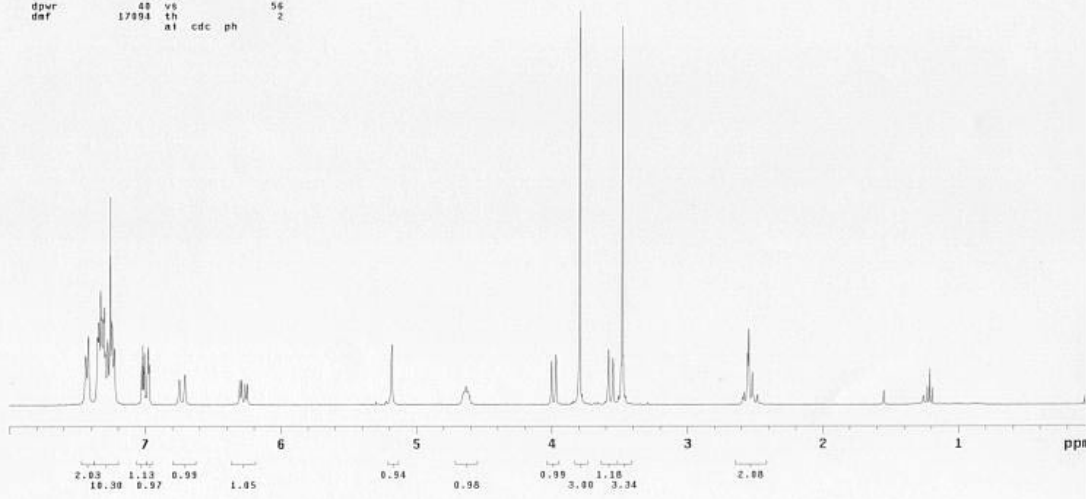
IV-4-91b Inova 400

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jul 12 2003	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		hst	0.000
sw	6395.0	pw90	9.700
at	3.744	alfa	6.600
np	47892	FLAGS	
fb	4800	ll	n
bs	16	fn	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	399.762	sp	-3.4
tof	399.8	wp	3302.1
tpwr	52	rfl	3607.2
pw	4.850	rfp	2302.3
DECOUPLER		rp	52.1
dn	C13	lp	-10.4
dof	0	PLOT	
dm	0	vc	250
dsm	C	sc	0
dpr	48	vs	56
daf	17904	th	2
	a1	cdc	ph



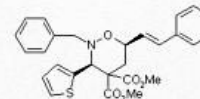
compound 19



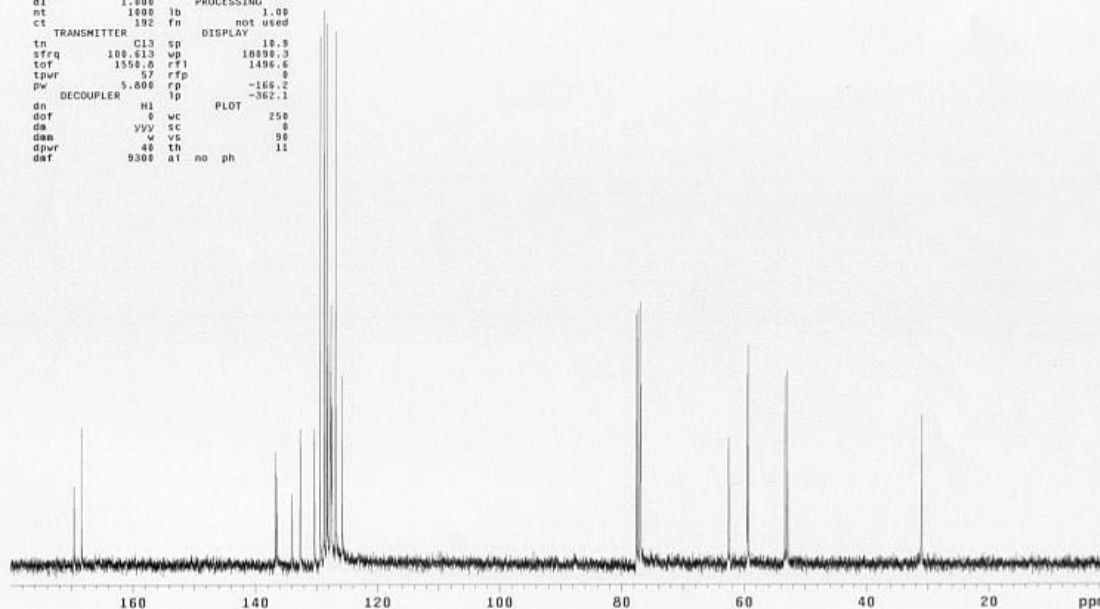
IV-4-91c2
STANDARD 1H OBSERVE

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jul 12 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/	spin	not used
nmr1/wakeup/auto	-	hst	0.000
acq	12.07.25/0102	pw90	11.000
ACQUISITION		alfa	20.000
sw	25125.6	FLAGS	
at	1.100	ll	n
np	60270	fn	n
fb	13000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	1000	lb	1.00
ct	100	fn	not used
TRANSMITTER		C13	sp
tn	H1	DISPLAY	
sfrq	100.613	wp	10000.3
tof	1550.0	rfl	1496.6
tpwr	57	rfp	0
pw	5.000	rp	-166.2
DECOUPLER		H1	lp
dn	0	PLOT	
dof	0	vc	250
dm	yyy	sc	0
dsm	v	vs	90
dpr	40	th	11
daf	9300	a1	no ph



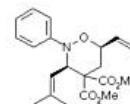
compound 19



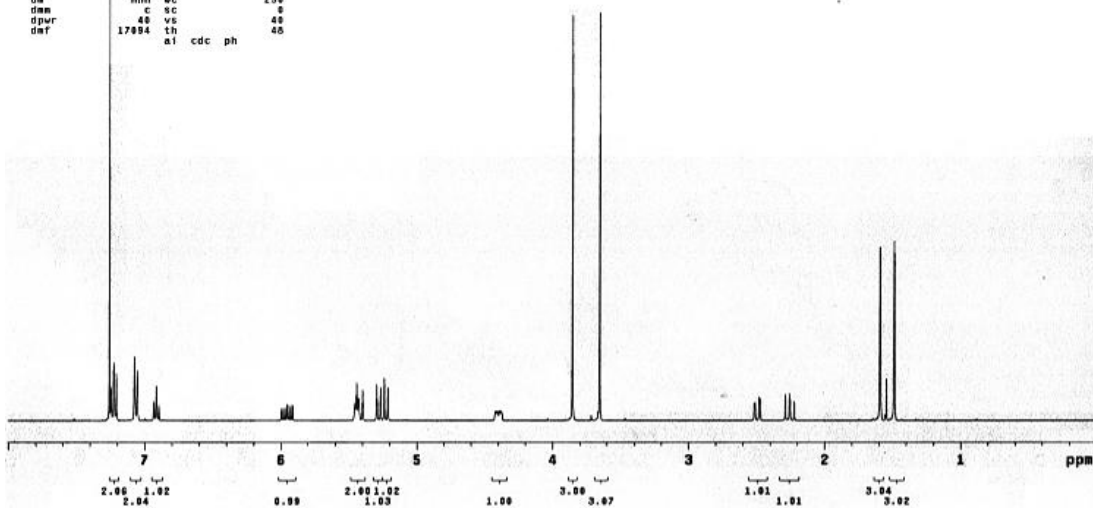
IY-4-37recd Inova 400

expl s2pu1

```
SAMPLE SPECIAL
date Jul 1 2003 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION hsc 0.000
sw 6395.9 pw90 9.700
at 3.744 alfa 6.600
np 47892 FLAGS n
fb 4000 ll n
bs 16 ln n
d1 1.000 dp y
nt 16 hs nn
ct 16 fn PROCESSING
tn H1 fn DISPLAY not used
sfrq 300.702 sp -2.0
tot 300.0 wp 3202.1
tpwr 52 rf1 3400.6
pw 4.000 rfp 2902.3
DECOUPLER C13 lp -25.2
dn 0 PLDT
dof 0 wc 250
dm c sc 0
dmm 40 vs 0
dpwr 17004 th 40
daf a1 cdc ph 40
```



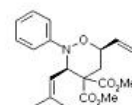
compound 20



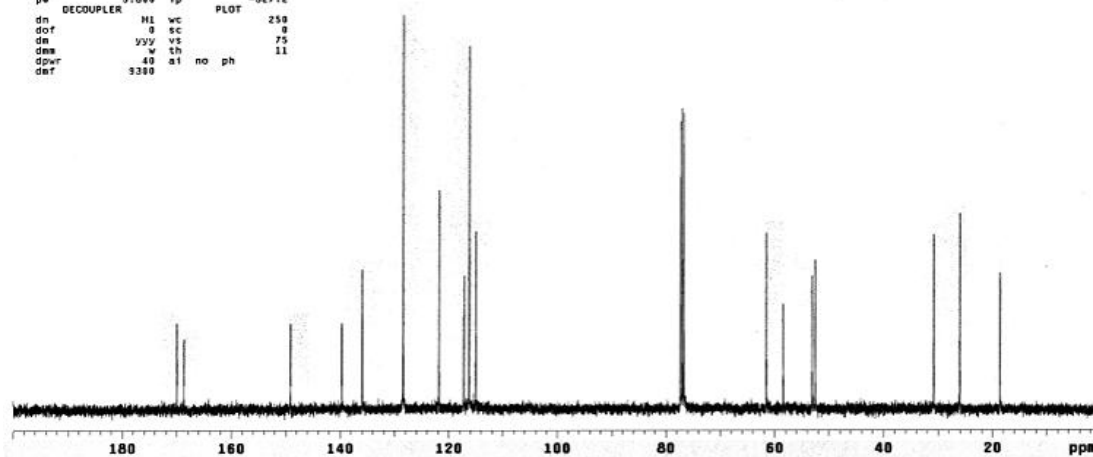
IY-4-37c
STANDARD 1H OBSERVE

expl s2pu1

```
SAMPLE SPECIAL
date Jun 27 2003 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin not used
vnmr1/vnmr/sys/data- hsc 0.000
/lyoung/IY-4-37c/c- pw90 11.000
ARBON.fid alfa 20.000
ACQUISITION hsc 0.000
sw 25125.6 ll n
at 1.199 ln n
np 40270 dp y
fb 13000 hs nn
bs 04 PROCESSING 1.00
d1 1.000 lb 1.00
nt 512 fn DISPLAY not used
ct 320
TRANSMITTER C13 sp 28100.3
sfrq 100.613 rf1 3269.5
tot 1500.0 rfp 7746.3
tpwr 57 rp -167.6
pw 5.000 lp -327.2
DECOUPLER H1 wc 250
dof 0 sc 0
dm 0 vs 75
dmm w th 11
dpwr 40 at no ph
daf 9380
```

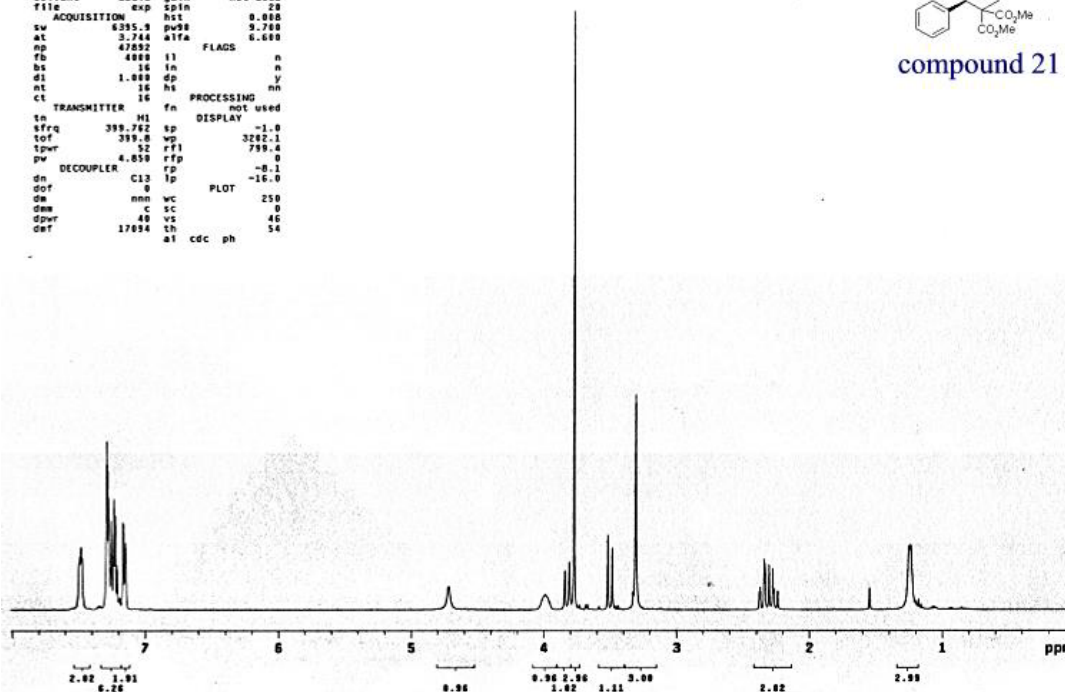
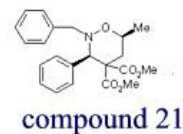


compound 20



1Y-4-121g
 expt s2pul

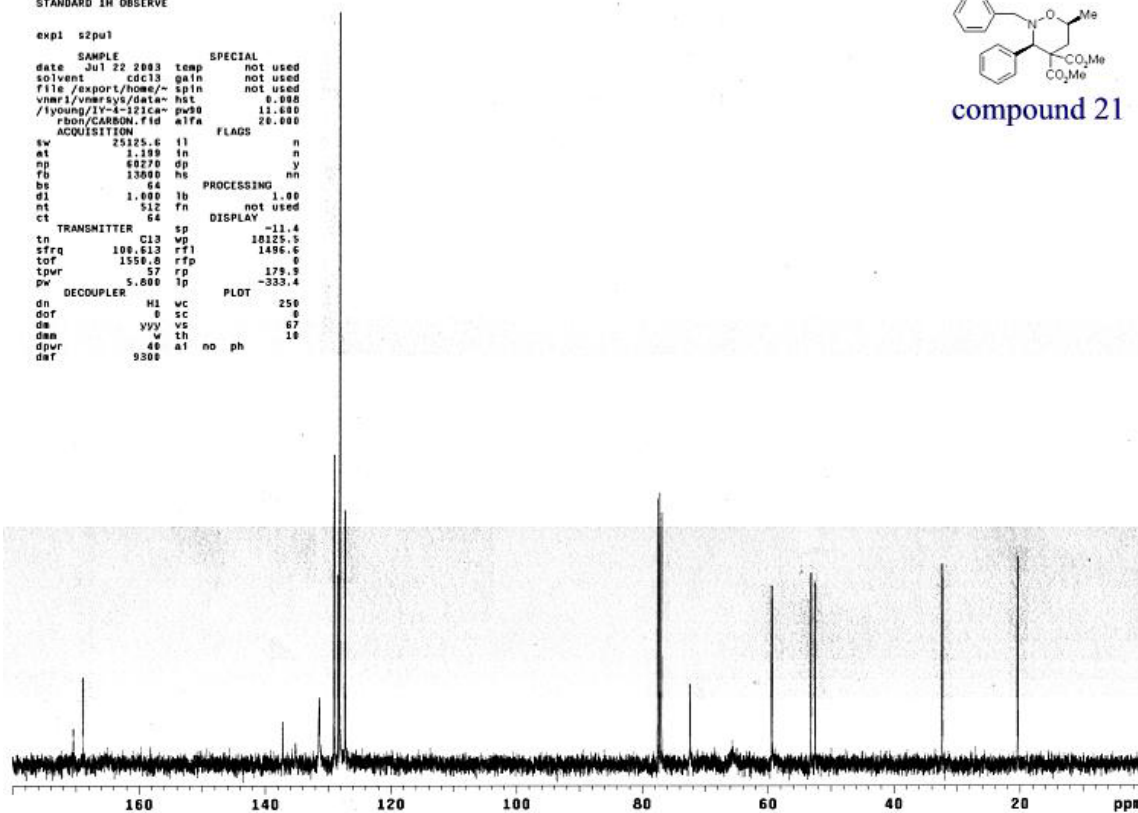
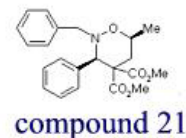
SAMPLE		SPECIAL	
date	Jul 31 2003	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION			
sw	4355.9	hst	0.000
at	3.744	alfa	9.700
np	47892	flfa	6.400
fb	4000	l1	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	mn
ct	16	PROCESSING	mn
TRANSMITTER			
tn	M1	fn	not used
DISPLAY			
sfrq	399.762	sp	-1.0
tor	399.8	wp	3282.1
tpwr	52	rfl	799.4
pw	4.859	rff	0
DECOUPLER			
dn	C13	lp	-16.0
dof	0	wc	250
dm	nm	sc	0
dms	C	sc	0
dpwr	40	vs	46
der	17094	th	54
	a1	cdc	ph



1Y-4-121carbon
 STANDARD IN OBSERVE

expt s2pul

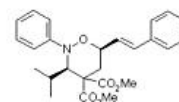
SAMPLE		SPECIAL	
date	Jul 22 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~sp1h	spin	not used
vnmr1/vnmr56/data~		hst	0.000
/young/1Y-4-121ca~		pw90	11.000
rbon/CARBON.fid		alfa	20.000
ACQUISITION			
sw	25125.6	l1	n
at	1.100	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	mn
d1	1.000	lb	1.00
nt	512	fn	not used
ct	64	DISPLAY	mn
TRANSMITTER			
tn	C13	sp	-11.4
sfrq	100.613	rfl	10125.5
tor	1550.0	rff	1496.6
tpwr	57	rp	179.9
pw	5.000	lp	-333.4
DECOUPLER			
dn	H1	vc	250
dof	0	sc	0
dm	yyv	vc	67
dms	w	th	10
dpwr	40	a1	no
der	9300		ph



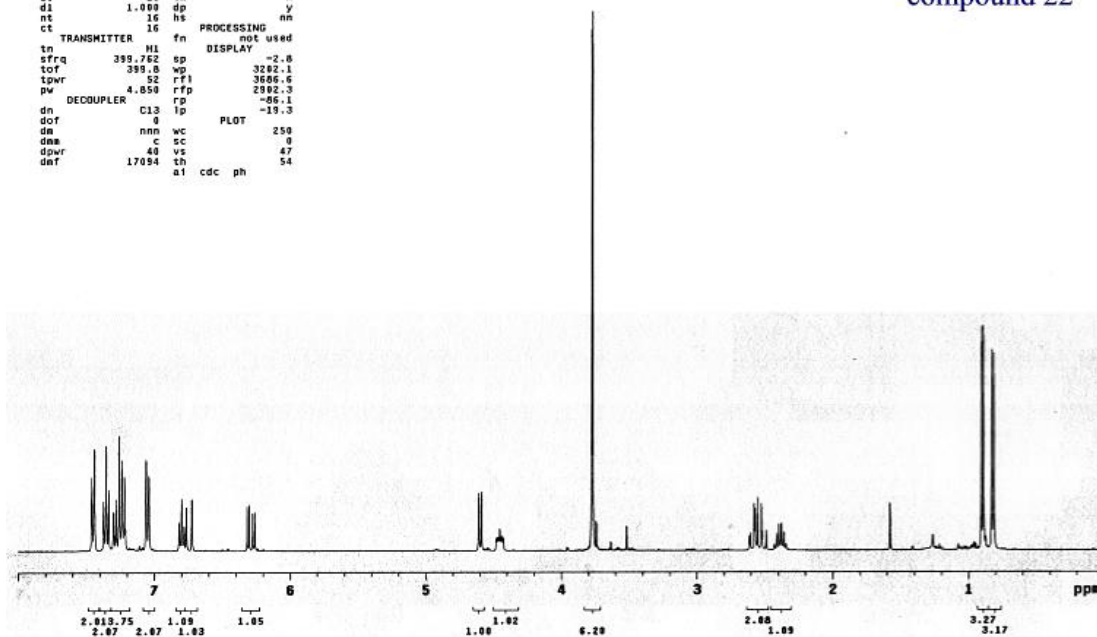
IV-4-29f Inova 400

expl s2pu1

SAMPLE		SPECIAL	
date	Jul 30 2003	temp	not used
solvent	CDCl3	gain	not used
file		exp	20
ACQUISITION			
sw	6385.3	hst	0.000
at	3.744	alfa	3.700
np	47000	flags	6.000
fb	4000	il	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct		fn	not used
PROCESSING			
tn	H1	display	-2.6
sfrq	399.762	sp	3282.1
tof	399.0	wp	3606.6
tpwr	52	rfl	2002.3
pw	4.050	rp	-06.1
DECOUPLER			
dn	C13	lp	-19.3
dof	0	plot	250
ds	nnn	wc	0
dss	c	sc	0
dpwr	40	vs	47
drt	17094	th	54
	a1	cdc	ph



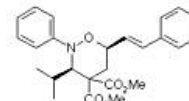
compound 22



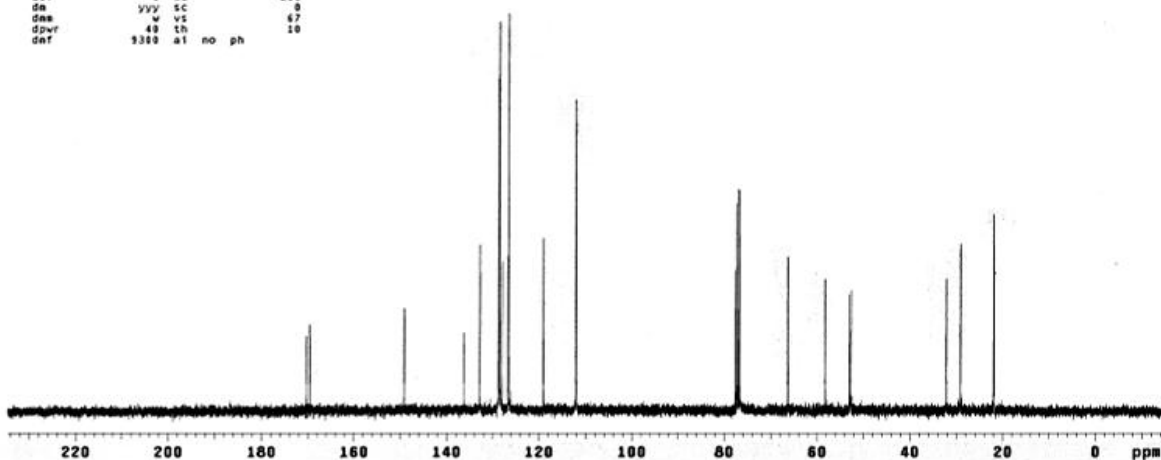
IV-4-29c
STANDARD 1H OBSERVE

expl s2pu1

SAMPLE		SPECIAL	
date	Jun 10 2003	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/	spn	not used
vnari/wakeup/auto_	hst	0.000	
dn	10.00	pr00	11.000
ACQUISITION			
sw	25125.6	flags	20.000
at	1.299	il	n
np	60270	in	n
fb	13000	dp	y
bs	64	ht	nn
d1	1.000	PROCESSING	
nt	512	lb	not used
ct	192	fn	1.00
TRANSMITTER			
tn	C13	sp	-1520.1
sfrq	100.613	wp	25125.6
tof	1550.0	rfl	3274.4
tpwr	57	rfl	7746.3
pw	5.000	rp	-173.0
DECOUPLER			
dn	H1	lp	-319.9
dof	0	wc	250
ds	yyy	sc	0
dss	w	vs	67
dpwr	40	th	10
drt	3300	a1	no ph



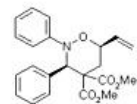
compound 22



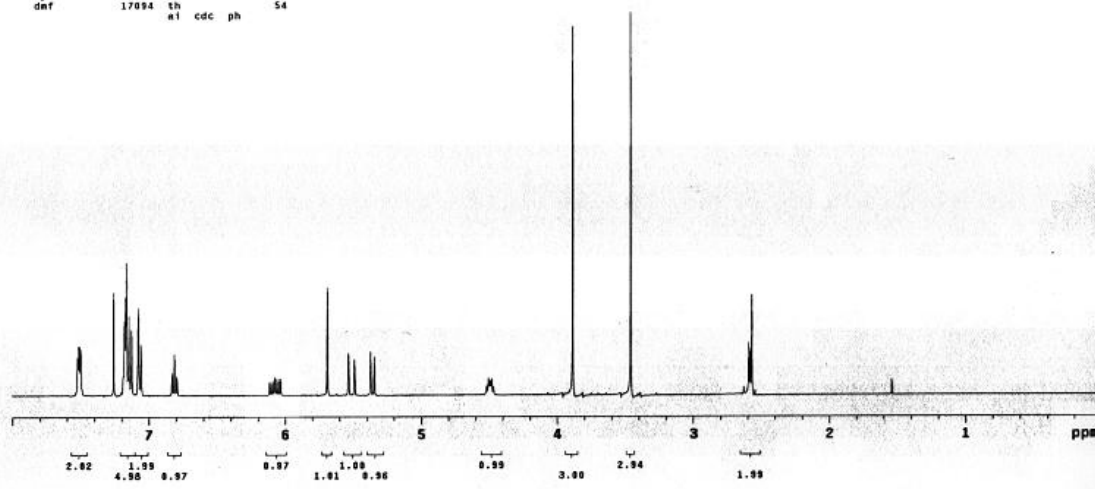
IY-4-35rec Inova 400

expl s2pu1

```
SAMPLE SPECIAL
date Jun 29 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
vnmr1/vmrsys/data/~ hst 8.000
/young/IY-4-35c/C- pw90 11.000
ARSON.Fid a1fa 20.000
ACQUISITION exp hst 0.000
sw 6395.9 pw90 9.700
at 3.744 a1fa 6.600
np 47800 FLAGS
fb 4000 f1 n
bs 16 in n
dl 1.000 dp y
nt 16 hs nn
ct 16 PROCESSING
TRANSMITTER fn not used
tn H1 DISPLAY -3.4
sfrq 399.702 sp 3202.1
tof 399.0 wp 3607.2
tpwr 52 rf1 2902.3
pv 4.050 rfp 52.0
DECOUPLER rp -49.3
dn C13 lp
dof 0 PLOT
dm nnn wc 250
dms c sc 0
dpr 40 vs 32
dnf 17094 th 54
a1 cdc ph
```



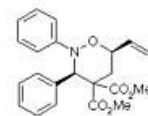
compound 23



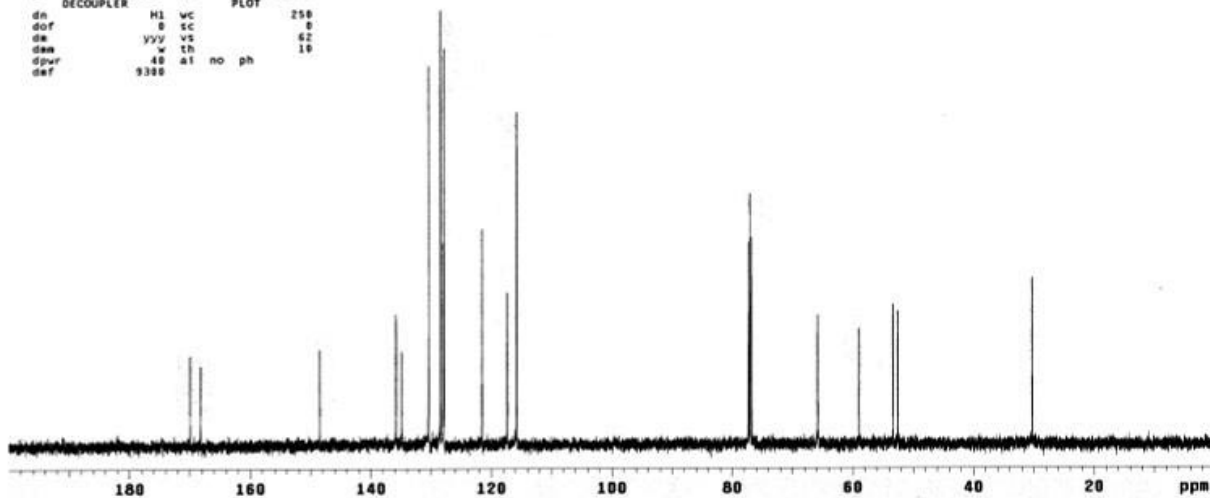
IY-4-35c
STANDARD IN OBSERVE

expl s2pu1

```
SAMPLE SPECIAL
date Jun 27 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
vnmr1/vmrsys/data/~ hst 8.000
/young/IY-4-35c/C- pw90 11.000
ARSON.Fid a1fa 20.000
ACQUISITION exp hst 0.000
sw 25125.6 f1 n
at 1.190 in n
np 60270 dp y
fb 13000 hs nn
bs 64 PROCESSING
dl 1.000 lb 1.00
nt 512 fn not used
ct 120 DISPLAY 8.6
TRANSMITTER sp 20186.0
tn C13 wp 9273.6
sfrq 100.613 rf1 7746.3
tof 1550.8 rfp -167.9
tpwr 57 rp -314.0
pv 5.000 lp
DECOUPLER H1 wc 250
dof 0 sc 0
dm yyy vs 62
dms w th 10
dpr 40 no ph
dnf 9300
```



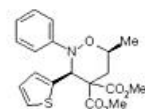
compound 23



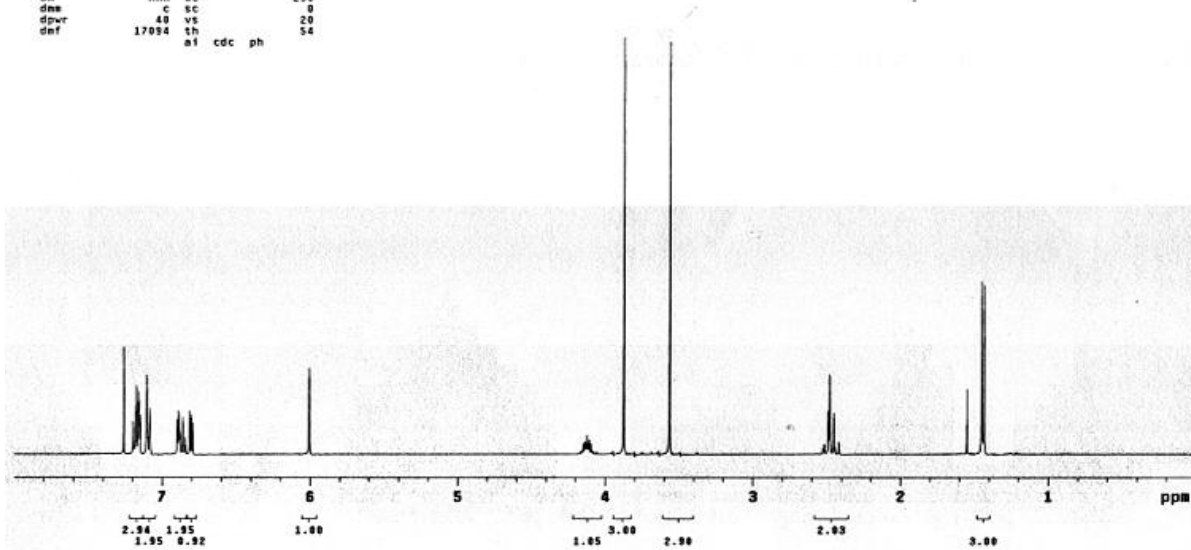
IV-4-27 rec Inova 400

expl szpu1

SAMPLE		SPECIAL	
date	Jul 3 2003	temp	not used
solvent	CDCl3	gain	not used
file		spn	28
ACQUISITION		ht	0.000
sw	6395.9	pw90	5.700
at	3.744	alFa	6.600
np	47892	FLAGS	
fb	4000	fl	n
bs	16	fn	n
d1	1.000	sp	y
nt	16	hs	nn
ct	16	PROCESSING	
TRANSMITTER		fn	not used
tn		H1	DISPLAY
sfrq	399.762	sp	3.0
tof	399.8	wp	3193.6
tpwr	52	rfl	3609.2
pw	4.650	rfp	2902.3
DECOUPLER		rp	146.0
dn	C13	lp	-19.0
dof	0	PLOT	
da	nnn	wc	250
dms	c	sc	0
dpr	40	vs	20
dnt	17094	th	54
	al	cdc	ph



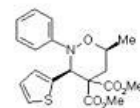
compound 24



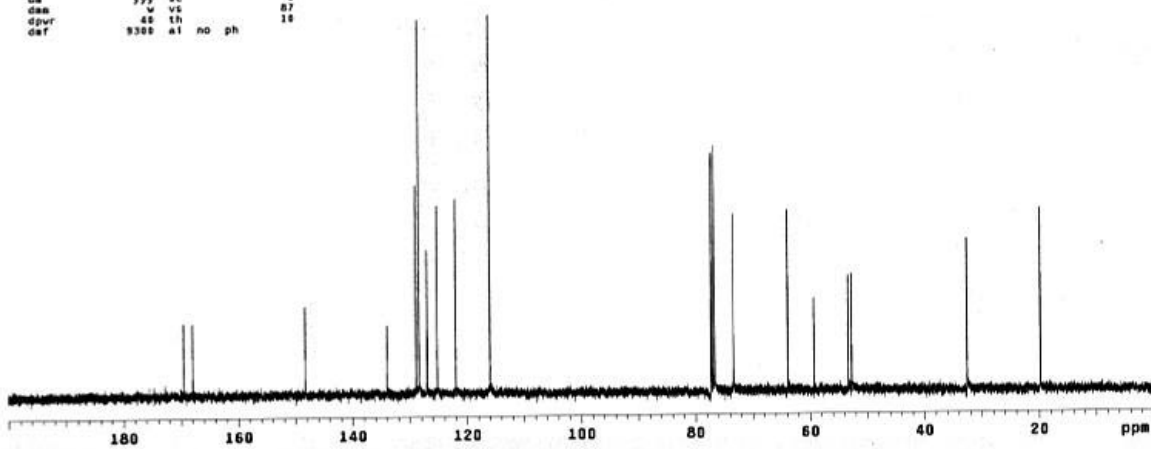
IV-4-27c
STANDARD IN OBSERVE

expl szpu1

SAMPLE		SPECIAL	
date	Jun 10 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spn	not used
vmer1/ve1kup/aufo	-	ht	0.000
10.06.03/4002	pu90	ht	11.000
ACQUISITION		alFa	20.000
sw	25125.6	FLAGS	
at	1.199	fl	n
np	60270	fn	n
fb	13000	sp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	512	th	1.00
ct	320	fn	not used
TRANSMITTER		DISPLAY	
tn		C13	sp
sfrq	100.613	wp	20139.9
tof	1550.0	rfl	9270.6
tpwr	57	rfp	7746.3
pw	5.000	rp	170.1
DECOUPLER		lp	-292.7
dn	H1	PLOT	
dof	0	wc	250
da	yyy	sc	0
dms	w	vs	07
dpr	40	th	10
dnt	9300	al	no
		ph	



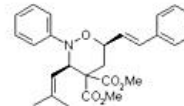
compound 24



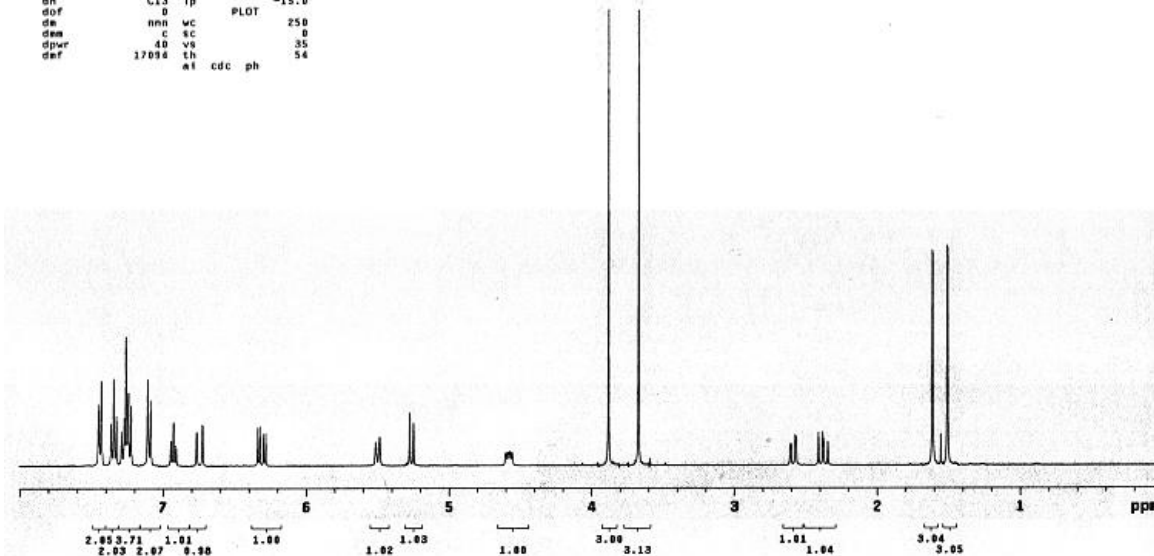
IY-4-31rcd

expl s2pu1

SAMPLE		SPECIAL	
date	Jul 17 2003	temp	not used
solvent	CDCl3	gain	not used
file	exp	spIn	20
ACQUISITION		hst	0.000
sw	639.5	pw90	9.700
at	3.744	alFa	6.600
np	47892	FLAGS	
fb	4000	l1	n
bs	10	ln	n
d1	1.000	dp	y
nt	8	hs	nn
ct	8	PROCESSING	
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	399.762	sp	-1.8
tof	399.8	wp	3202.1
tpwr	52	rfl	3685.6
pw	4.830	rfg	2892.3
DECOUPLER		rp	-57.2
dn	C13	lp	-15.0
dof	0	PLOT	
de	nnn	wc	250
dse	c	sc	0
dpr	40	vs	35
def	17090	th	54
	at	cdc	ph



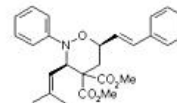
compound 25



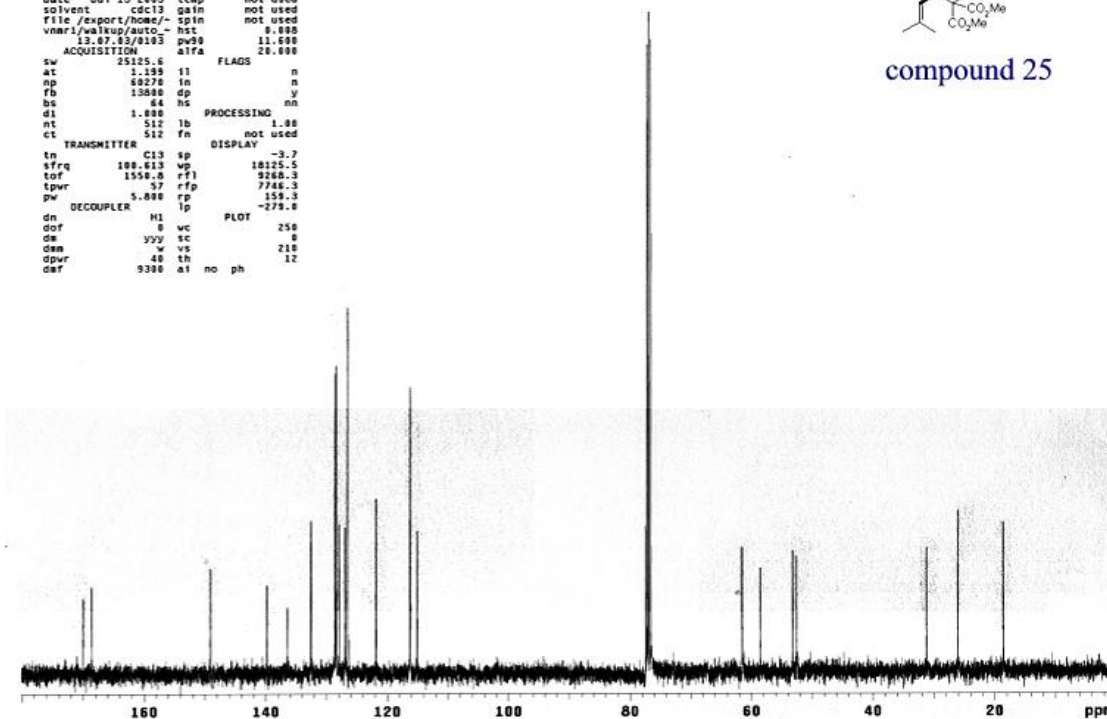
IY-4-31car
STANDARD 1H OBSERVE

expl s2pu1

SAMPLE		SPECIAL	
date	Jul 13 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spIn	not used
vmer1/wa1kw/auco	-	hst	0.000
13.07.05/0193		pw90	11.600
ACQUISITION		alFa	20.000
sw	25125.5	FLAGS	
at	1.195	l1	n
np	80270	ln	n
fb	13000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	512	lb	1.00
ct	512	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-3.7
sfrq	100.613	wp	10125.5
tof	1550.8	rfl	3260.3
tpwr	57	rfg	7746.3
pw	5.800	rp	159.3
DECOUPLER		lp	-279.8
dn	H1	PLOT	
dof	0	wc	250
dse	300	sc	0
dpr	w	vs	210
dpr	40	th	35
def	9300	at	no ph 12



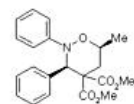
compound 25



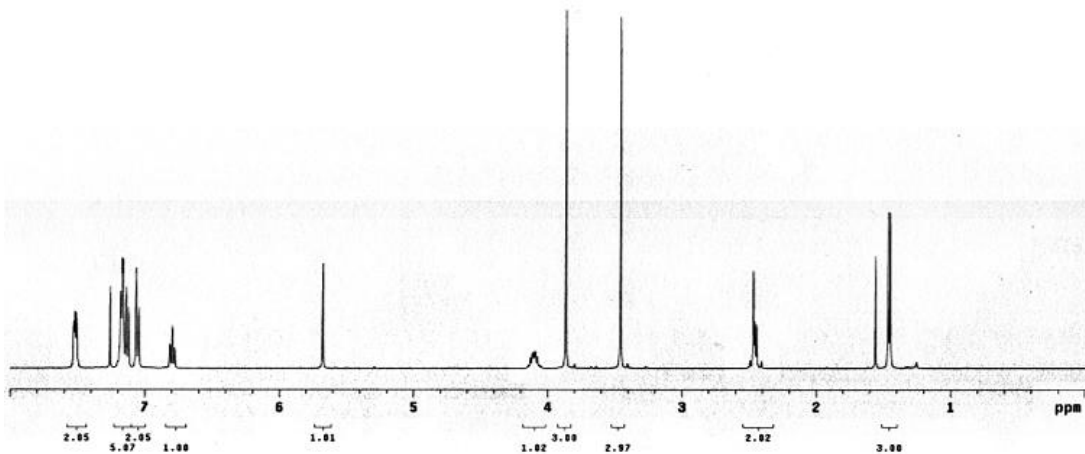
IV-4-23recInova 400

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jun 29 2003	temp	not used
solvent	CDCl3	gain	not used
file		exp	sp1n
ACQUISITION		hst	0.000
sw	4395.0	pu	9.700
at	3.744	alfa	6.000
np	47092	FLAGS	
fb	4000	l1	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	nn
TRANSMITTER	H1	fn	not used
tn		DISPLAY	
sfrq	399.762	sp	-3.6
tof	399.0	vp	3282.1
tpwr	52	rfl	3607.4
pw	4.850	rfd	2982.3
DECOUPLER	C13	rp	38.1
dn		lp	-19.7
dof	0	PLOT	
dm	nn	wc	250
dsm	c	sc	0
dpwr	40	vs	37
dof	17000	th	54
	al	cdc	ph



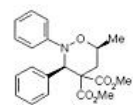
compound 26



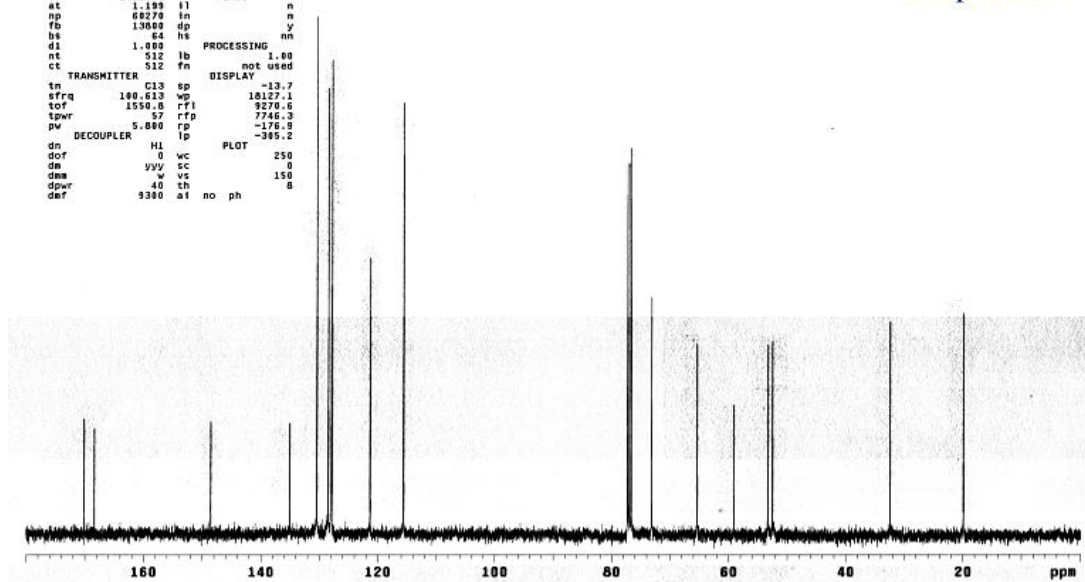
IV-4-23d
STANDARD 1H OBSERVE

exp11 s2pu1

SAMPLE		SPECIAL	
date	Jun 17 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/	sp1n	not used
vnmr1/wa/ku/au/c		hst	0.000
17.06.03/0103		pu	11.000
ACQUISITION		alfa	20.000
sw	25125.6	FLAGS	
at	1.199	l1	n
np	60270	in	n
fb	13000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	512	lb	1.00
ct	512	fn	not used
TRANSMITTER	C13	sp	not used
tn		DISPLAY	
sfrq	100.620	vp	-13.7
tof	1550.0	rfl	18127.1
tpwr	57	rfd	9270.6
pw	5.000	rp	7746.3
DECOUPLER	H1	lp	-175.0
dn		lp	-305.2
dof	0	PLOT	
dm	yy	sc	250
dsm	w	vs	150
dpwr	40	th	8
dof	9300	al	no
		ph	



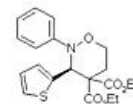
compound 26



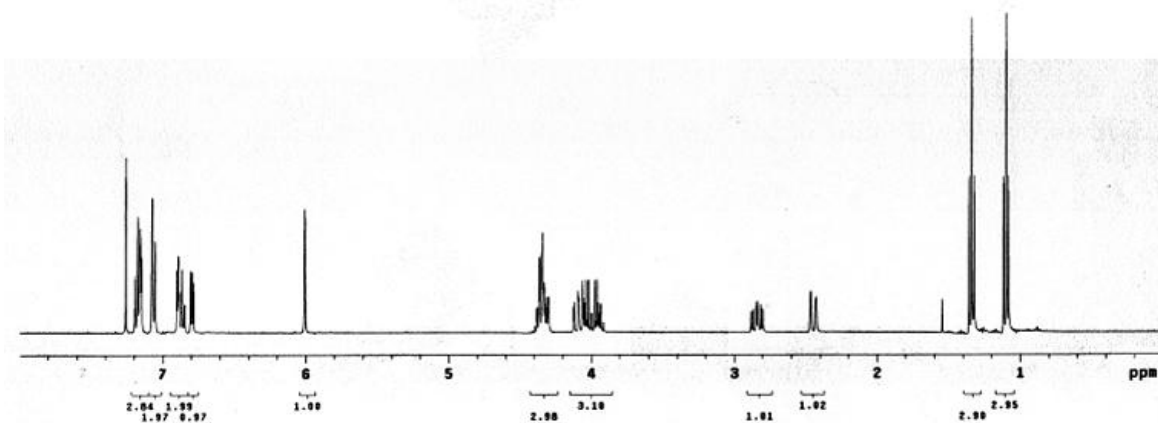
IY-4-53 rec Inova 400

expl s2pul

```
SAMPLE SPECIAL
date Jul 3 2003 temp not used
solvent CDCl3 gain not used
file exp spin 20
ACQUISITION hst 8.000
sw 6395.9 pw90 9.700
at 3.744 alfa 6.600
np 47032 FLAGS
fb 4800 ii n
bs 16 in n
d1 1.000 dp y
nt 16 hs n
ct 16 PROCESSING nn
TRANSMITTER fn not used
tn H1 DISPLAY 2.7
sfrq 399.762 sp 3193.6
tof 399.8 wp 3889.5
tpwr 52 rfl 2982.3
pw 4.859 rfp 136.1
DECOUPLER rp -7.4
dn 8 PLOT
dm nnn wc 250
dnn c sc 8
dpwr 48 vs 43
dnt 17894 th 54
dnt al cdc ph
```



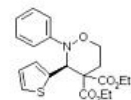
compound 27



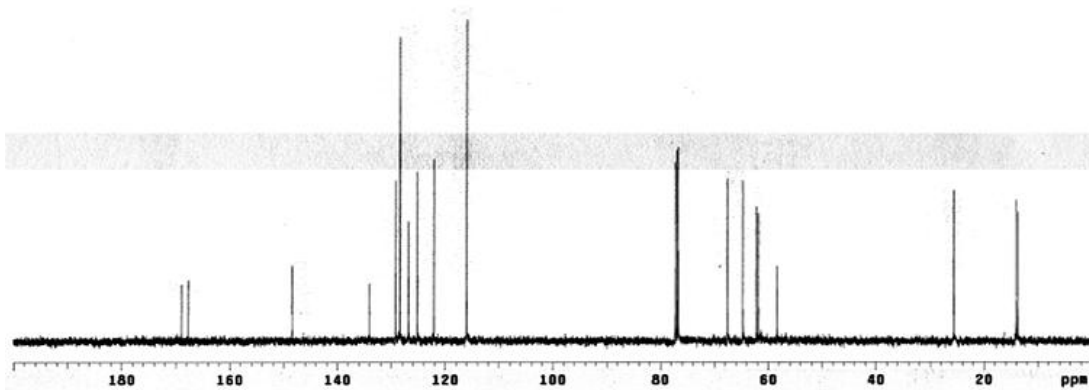
IY-4-53C
STANDARD IN OBSERVE

expl s2pul

```
SAMPLE SPECIAL
date Jul 3 2003 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/walrus/auto.- hst 8.000
83.87.85/0104 pw90 11.000
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 ii n
np 60270 in n
fo 12800 dp y
bs 64 hs n
d1 1.000 PROCESSING nn
nt 512 lb 1.00
ct 256 fn not used
TRANSMITTER C13 sp DISPLAY -9.1
tn H1 DISPLAY 20237.6
sfrq 100.613 wp 7746.3
tof 1558.8 rfl 171.7
tpwr 57 rfp -281.0
pw 5.000 rp
DECOUPLER H1 PLOT
dn 8 wc 250
dm YYY tc 0
dnn v vs 62
dpwr 48 th 18
dnt 9300 al no ph
```

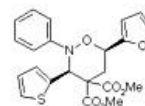


compound 27

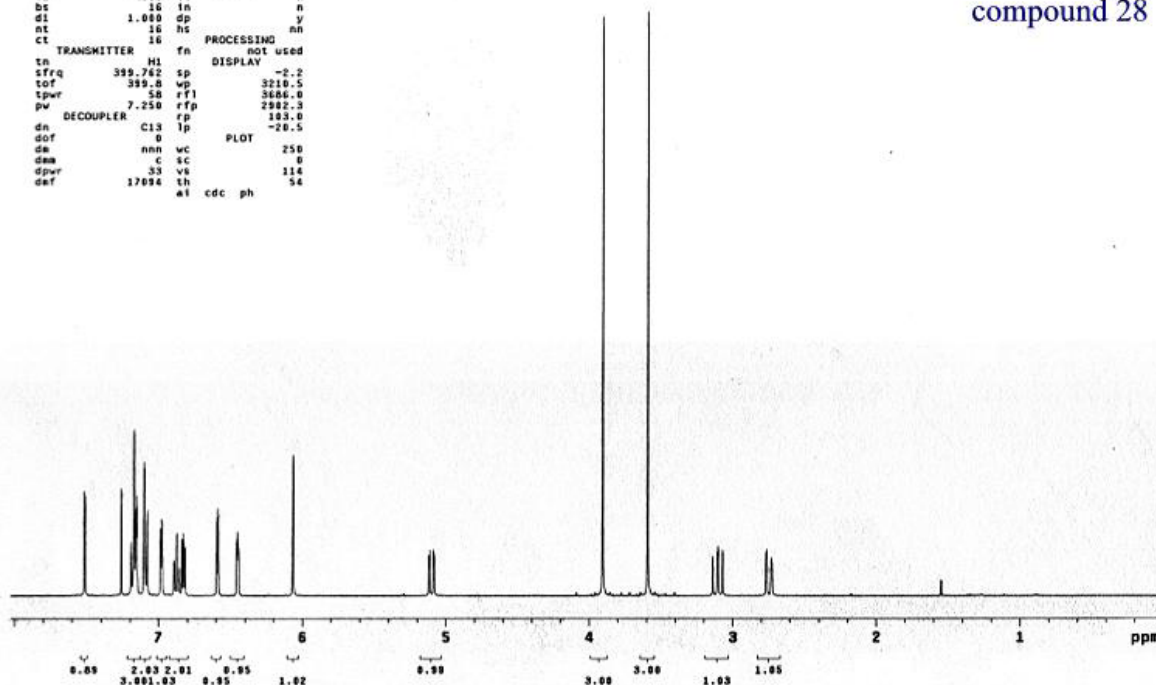


IY-3-193
exp1 s2pul

SAMPLE		SPECIAL	
date	Jun 11 2003	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		SPECIAL	
sw	4395.9	hst	0.000
at	3.744	alpha	6.600
np	47892	FLAGS	
Td	4000	il	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	nn
TRANSMITTER		SPECIAL	
tn		fn	not used
sfrq	399.762	sp	DISPLAY
tof	399.8	wp	-2.2
tpwr	7.250	rfl	3210.5
pw		rfg	3686.0
DECOUPLER		SPECIAL	
dn	C13	rp	103.0
dof	0	lp	-20.5
dm	nnn	vc	250
dms	c	sc	0
dpr	43	vs	114
daf	17094	th	54
	al	cdc	ph



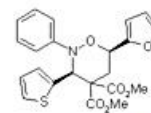
compound 28



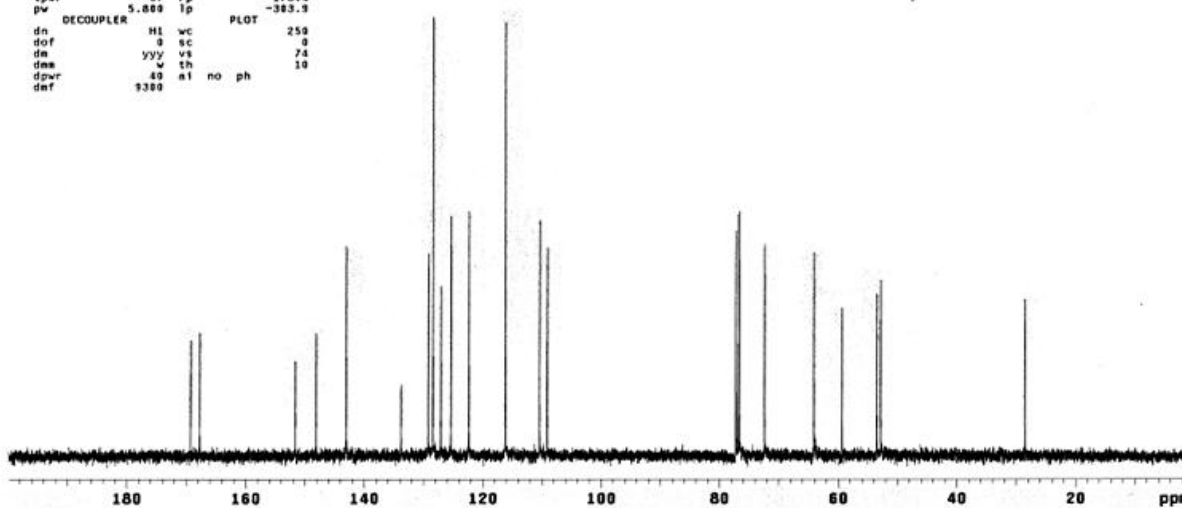
IY-3-193c
STANDARD IN OBSERVE

exp11 s2pul

SAMPLE		SPECIAL	
date	Jun 11 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/	spin	not used
vnmr1/vnmr5s/data/		hst	0.000
/lyoung/IY-3-193c/		pw30	11.000
CARBON.F1d		alpha	20.000
ACQUISITION		SPECIAL	
sw	25125.6	il	n
at	1.199	in	n
np	68278	dp	y
Td	13800	hs	nn
bs	64	PROCESSING	
d1	1.000	lb	1.00
nt	512	fn	not used
ct	192	DISPLAY	7.0
TRANSMITTER		SPECIAL	
tn	C13	sp	20100.0
sfrq	100.613	rfl	9272.0
tof	1559.8	rfg	7766.3
tpwr	5.7	rp	173.4
pw	5.000	lp	-383.3
DECOUPLER		SPECIAL	
dn	H1	vc	250
dof	0	sc	0
dm	yyy	vs	74
dms	w	th	10
dpr	43	ni	no
daf	9300	ph	



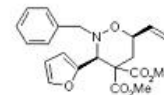
compound 28



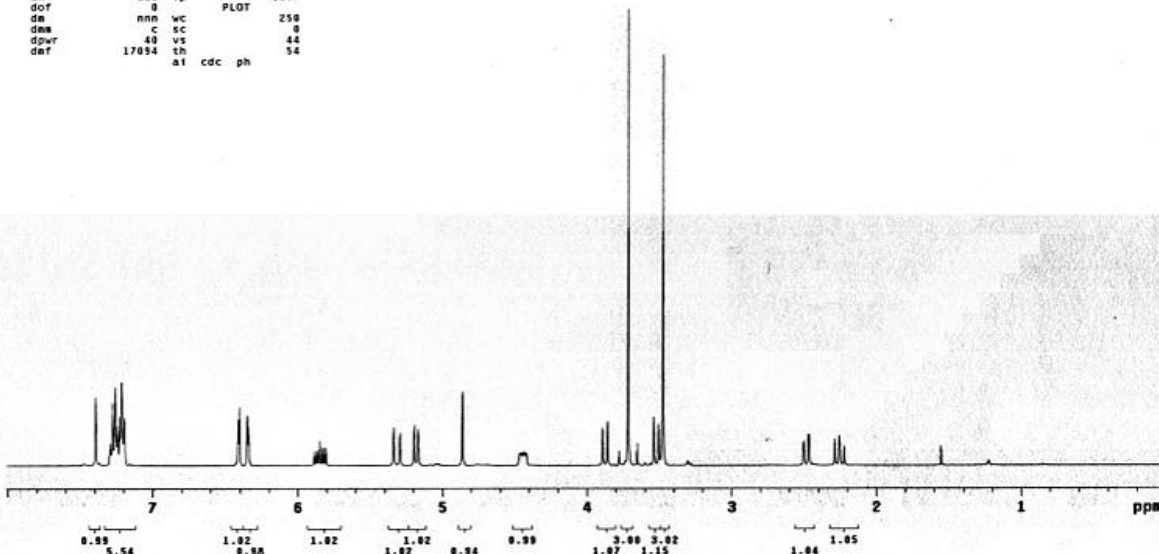
IV-4-93f Inova 400

exp1 s2pul

SAMPLE		SPECIAL	
date	Jul 30 2003	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		SPECIAL	
sw	8355.3	hst	0.000
at	3.744	pw90	3.700
np	47832	alpha	0.000
FLAGS			
fb	4000	l1	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	fn	not used
TRANSMITTER		PROCESSING	
tn	H1	sp	not used
sfrq	399.762	display	-1.0
tof	399.8	wp	3202.1
tpwr	52	rfl	799.4
pw	4.850	rfd	0
DECOUPLER		PLOT	
dn	C13	lp	-10.7
dof	0	wc	250
da	nnn	sc	0
dma	C	vs	44
dpwr	40	td	54
daf	17054	at	cdc ph



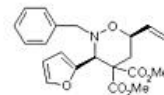
compound 29



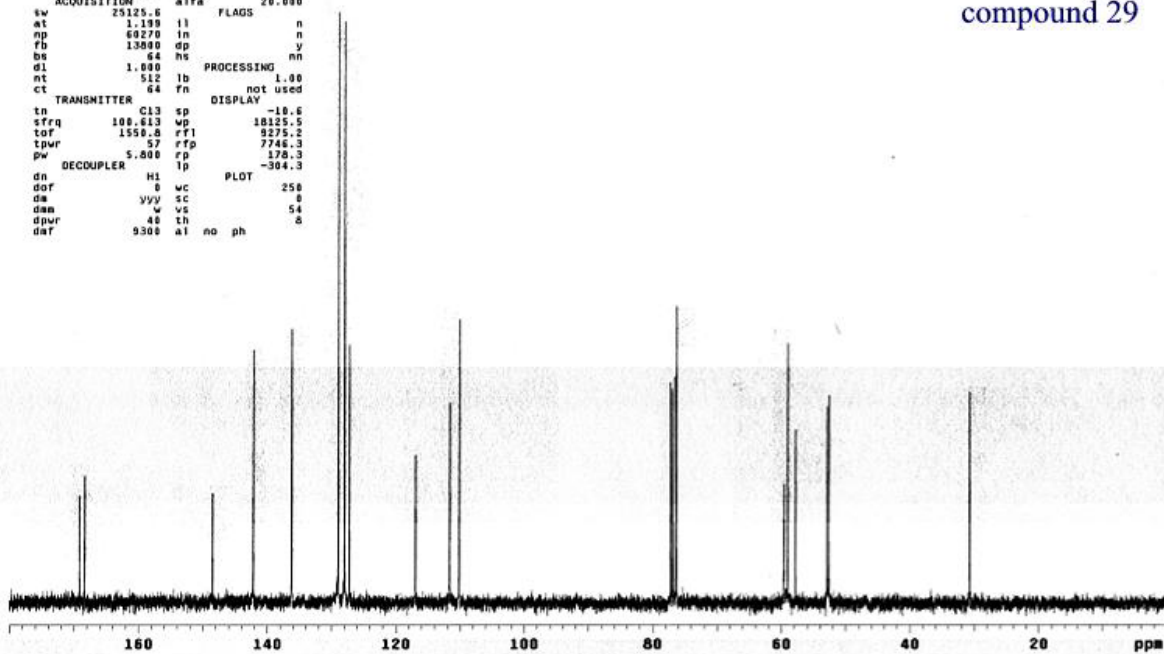
IV-4-93c
STANDARD 1H OBSERVE

exp1 s2pul

SAMPLE		SPECIAL	
date	Jul 13 2003	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
vnmr1/walrus/auto.		hst	0.000
13.07.03/9203		pw90	11.000
ACQUISITION		SPECIAL	
sw	25125.6	alpha	20.000
at	1.190	l1	n
np	60270	in	n
fb	13000	dp	y
bs	64	hs	nn
d1	1.000	dp	1.00
nt	512	lb	not used
ct	64	fn	not used
TRANSMITTER		PROCESSING	
tn	C13	sp	-10.0
sfrq	100.613	wp	10125.5
tof	1550.8	rfl	9275.2
tpwr	57	rfd	7746.3
pw	5.000	rfd	170.3
DECOUPLER		PLOT	
dn	H1	lp	-304.3
dof	0	wc	250
da	yyy	sc	0
dma	w	vs	54
dpwr	40	td	8
daf	9300	at	no ph



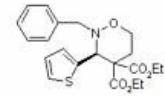
compound 29



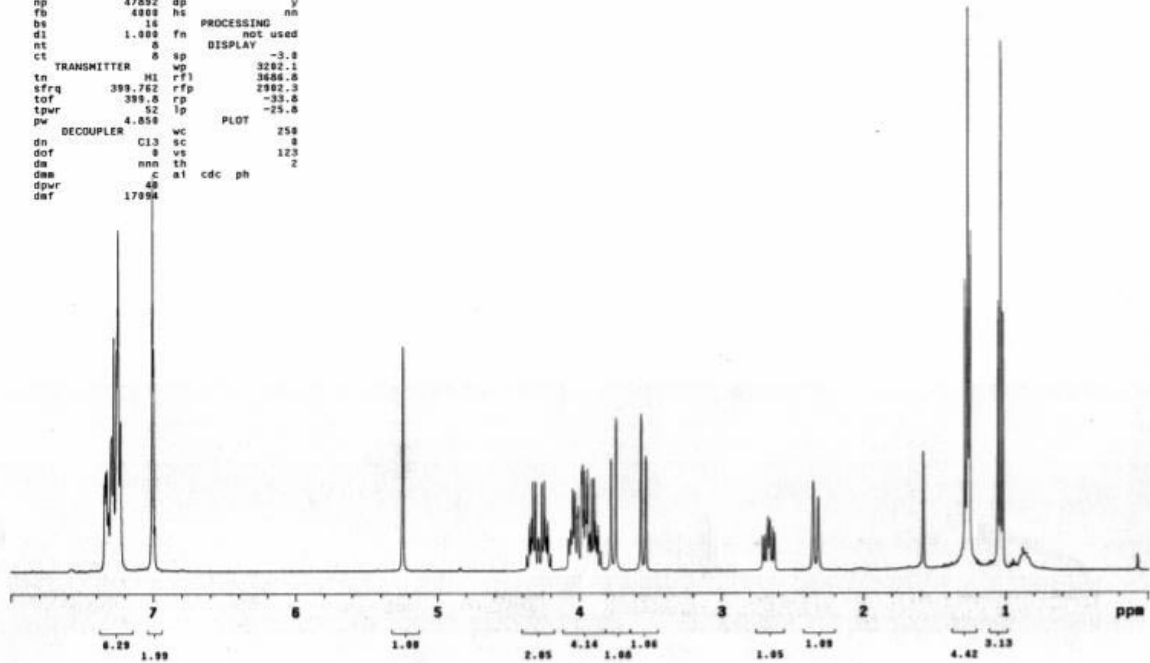
IV-4-97f Inova 400

expl s2pu1

```
SAMPLE SPECIAL
date Aug 3 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
vnr1/vnr2sys/data/~ hst 0.000
IV-4-97f_03Aug2003- pw98 9.700
/PROTON.fid a1fa 6.600
ACQUISITION FLAGS
sw 6395.9 f1 n
at 3.744 in n
np 47892 dp y
fb 4000 hs nn
bs 16 PROCESSING
d1 1.000 fn not used
nt 8 DISPLAY
ct 8 sp -3.0
TRANSMITTER HI wp 3282.1
tn HI rfl 3686.0
sfrq 399.762 rfp 2982.3
tof 399.8 rfp -33.0
tpwr 52 lp -25.0
pw 4.058 PLOT
dn DECOUPLER wc 250
dof C13 sc 8
dm 0 vs 123
dwm mm th 2
dpwr c a1 cdc ph 2
dnt 17094
```



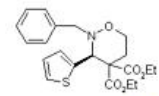
compound 30



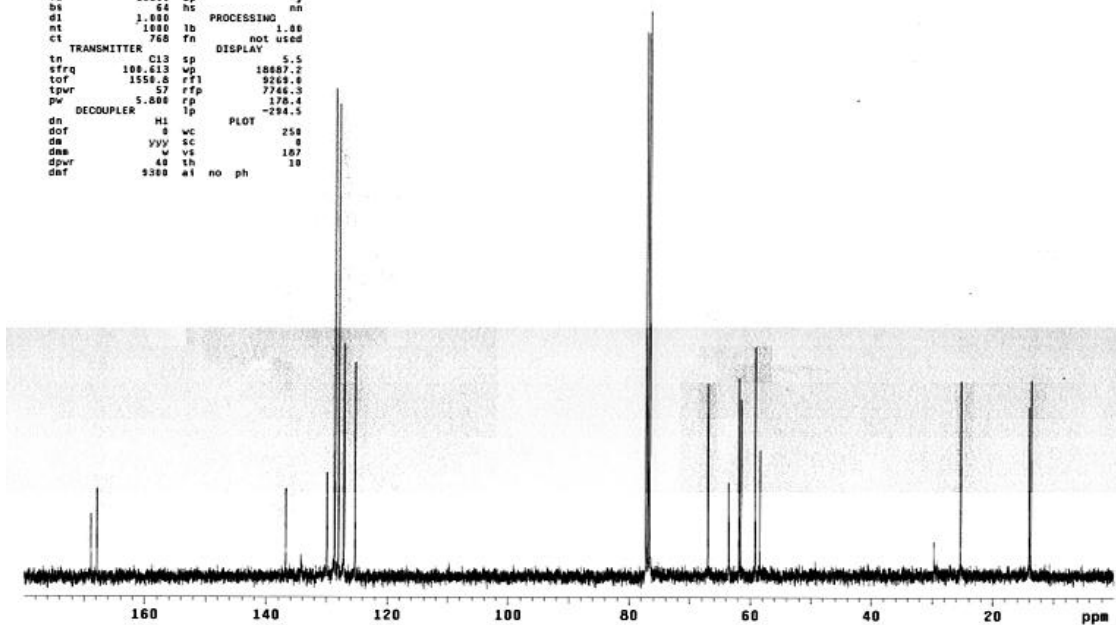
IV-4-97car
STANDARD 1H OBSERVE

expl s2pu1

```
SAMPLE SPECIAL
date Aug 3 2003 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin not used
vnr1/walkup/auto- hst 0.000
13.00.03/8293 pw98 11.000
ACQUISITION a1fa 20.000
sw 25125.6 f1 n
at 1.199 f1 n
np 68270 in n
fb 13000 dp y
bs 64 hs nn
d1 1.000 PROCESSING
nt 1000 lb not used
ct 760 fn DISPLAY
TRANSMITTER C13 sp
tn C13 sp 18887.2
sfrq 100.613 wp 9269.0
tof 1550.8 rfl 7746.3
tpwr 57 rfp 170.4
pw 5.008 lp -294.5
dn DECOUPLER H1 PLOT
dof 3 wc 250
dm yyy sc 0
dwm v vs 187
dpwr 40 th 10
dnt 9380 a1 no ph 10
```



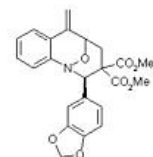
compound 30



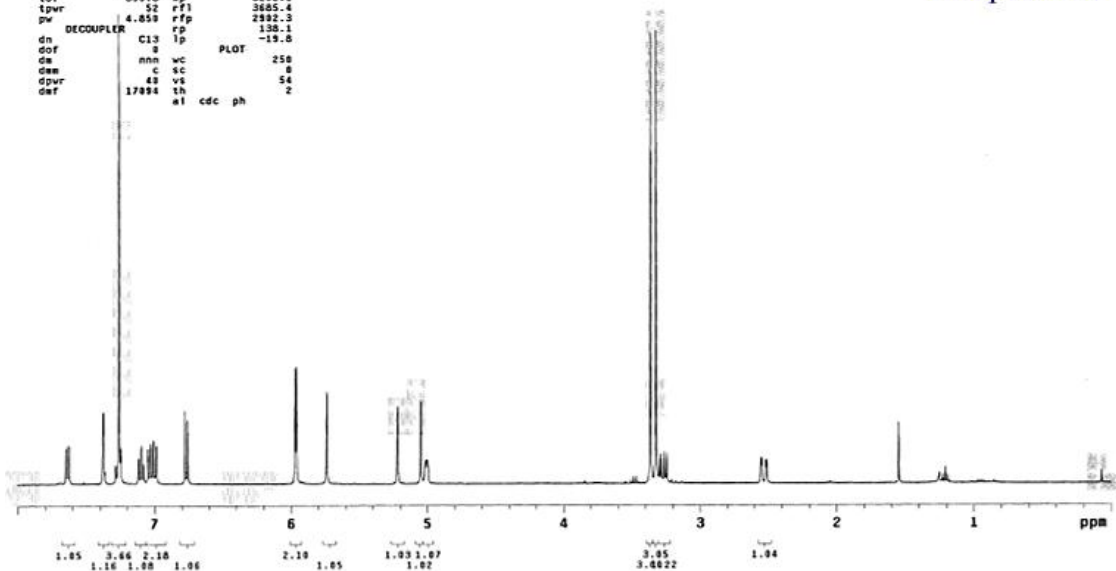
IV-4-177j Inova 400
 exp1 s2pul

```

SAMPLE          SPECIAL
date Sep 1 2003 temp not used
solvent cdc13 gain not used
file exp spin 20
ACQUISITION    hst 8.000
sw 6395.3 pvs9 5.700
at 3.744 alfa 6.600
np 47892 FLAGS n
fb 4809 i1 n
bs 16 in n
dl 1.000 dp y
nt 16 hs nn
ct 16 PROCESSING not used
TRANSMITTER H1 fn DISPLAY
tn 399.762 sp -1.6
sfrq 399.8 wp 3282.1
tpwr 52 rF1 3605.4
pw 4.050 rfp 2982.3
DECOUPLER C13 lp 130.1
dn 0 wc PLOT 250
dof 0 w 6
dem c sc 8
dpwr 40 vs 54
def 17094 ai cdc ph 2
  
```



Compound 31

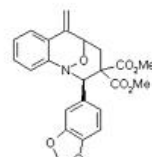


IV-4-177car
 STANDARD 1H OBSERVE

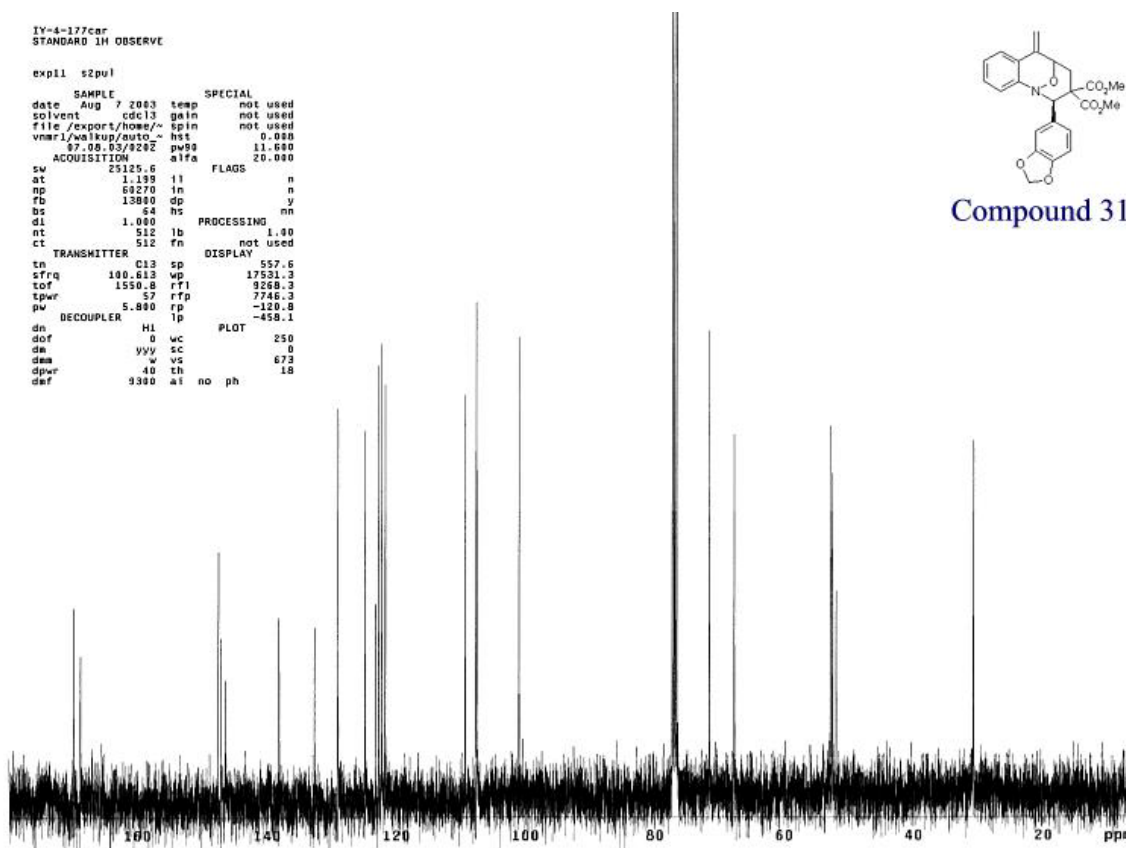
exp11 s2pul

```

SAMPLE          SPECIAL
date Aug 7 2003 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/walrup/otc- hst 0.000
$7.08.05/826Z pvs9 11.600
ACQUISITION    alfa 20.000
sw 25125.6 FLAGS n
at 1.199 i1 n
np 60270 in n
fb 13080 dp y
bs 64 hs nn
dl 1.000 PROCESSING 1.00
nt 512 lb fn not used
ct 512 fn DISPLAY 557.6
TRANSMITTER C13 sp 17531.3
tn 100.613 wp 9268.3
sfrq 1550.8 rF1 7746.3
tpwr 57 rfp -120.8
pw 5.800 rp -458.1
DECOUPLER HL lp PLOT 250
dn 0 wc 0
dof 0 w 673
dem vvy sc 18
dpwr 40 vs
def 9380 ai no ph
  
```



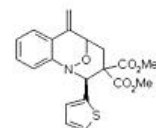
Compound 31



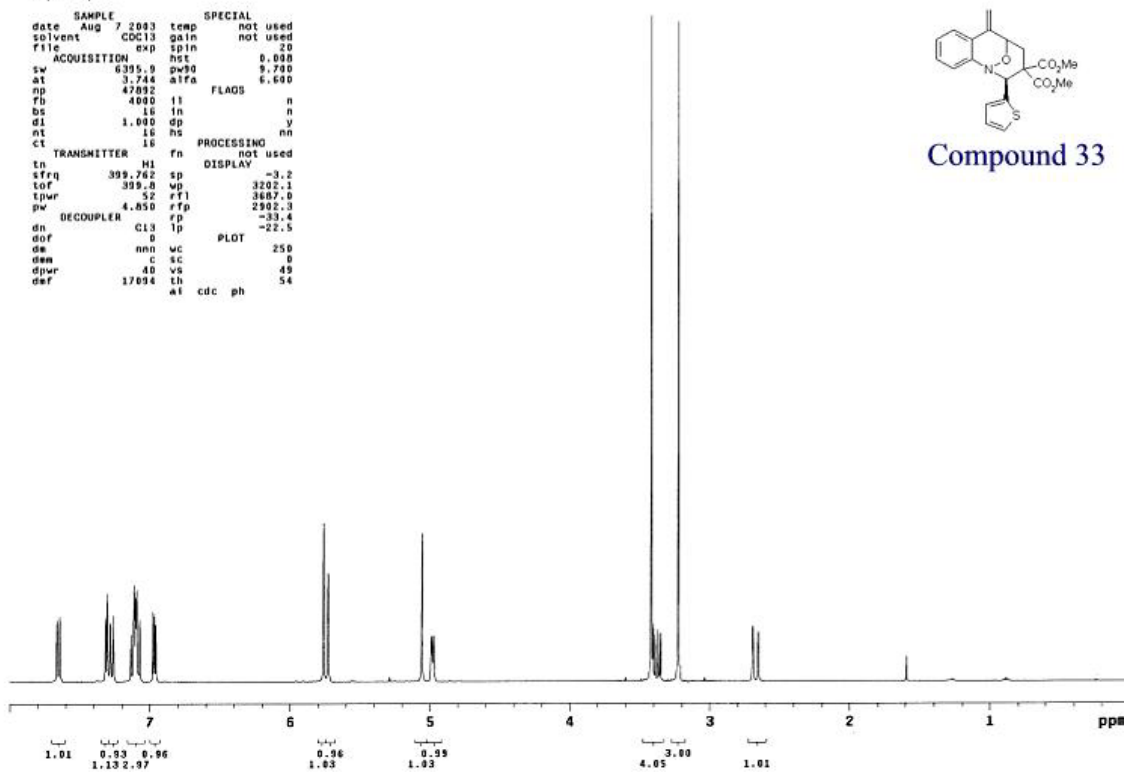
IY-4-173 rcd Inova 400

expl s2pul

```
SAMPLE SPECIAL
date Aug 7 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ACQUISITION exp hst 8.800
sw at 6395.9 pw90 9.700
np 47892 a1fa 6.600
fb 4000 f1 FLAGS n
bs 16 f1n n
d1 1.000 d9 y
ct 16 h5 nn
TRANSMITTER 16 fn PROCESSING not used
tn M1 sp DISPLAY -3.2
sfrq 399.762 wp 3202.1
tof 52 rf1 3607.0
pw 4.850 rfp 2902.3
DECOUPLER c13 fp -33.4
dn 0 lp -22.5
dof 0 uc PLOT 250
dem nnn c sc 0
dpm 40 vs 49
def 17094 th ai cdc ph 54
```



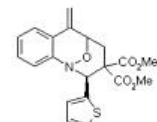
Compound 33



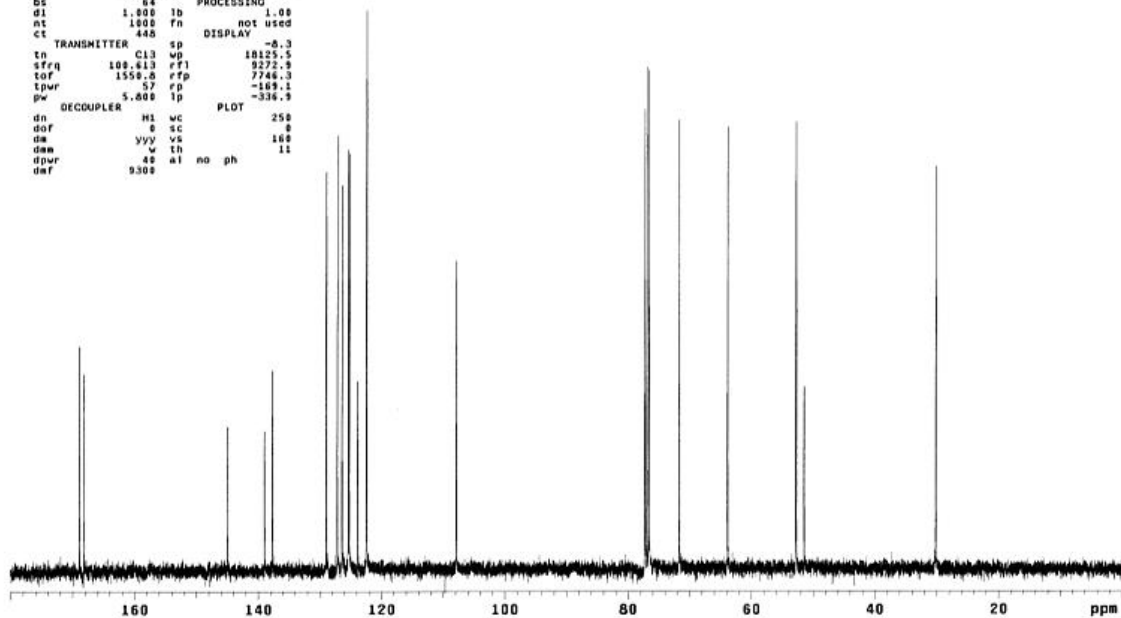
IY-4-173 rcd
STANDARD IN OBSERVE

expl s2pul

```
SAMPLE SPECIAL
date Aug 7 2003 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin not used
vnmr1/vnmrsys/data/~ hst 8.800
/young/IY-4-173ca-pw90 11.600
rred/CARBON.fid a1fa 20.000
ACQUISITION
sw 25125.6 f1 FLAGS n
at 1.199 f1n n
np 60270 d9 y
fb 13800 h5 nn
bs 64 PROCESSING 1.09
d1 1.000 f1b not used
ct 1800 f1n DISPLAY -0.3
TRANSMITTER 445 sp
tn C13 wp 18125.5
sfrq 100.613 rf1 9272.3
tof 1559.0 rfp 7746.3
pw 57 fp -169.1
DECOUPLER 5.600 lp -334.9
dn 0 uc PLOT 250
dof 0 sc 0
dem yyy vs 160
dpm w th 11
dpr 40 ai no ph
def 9302
```



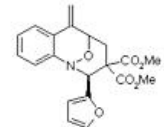
Compound 33



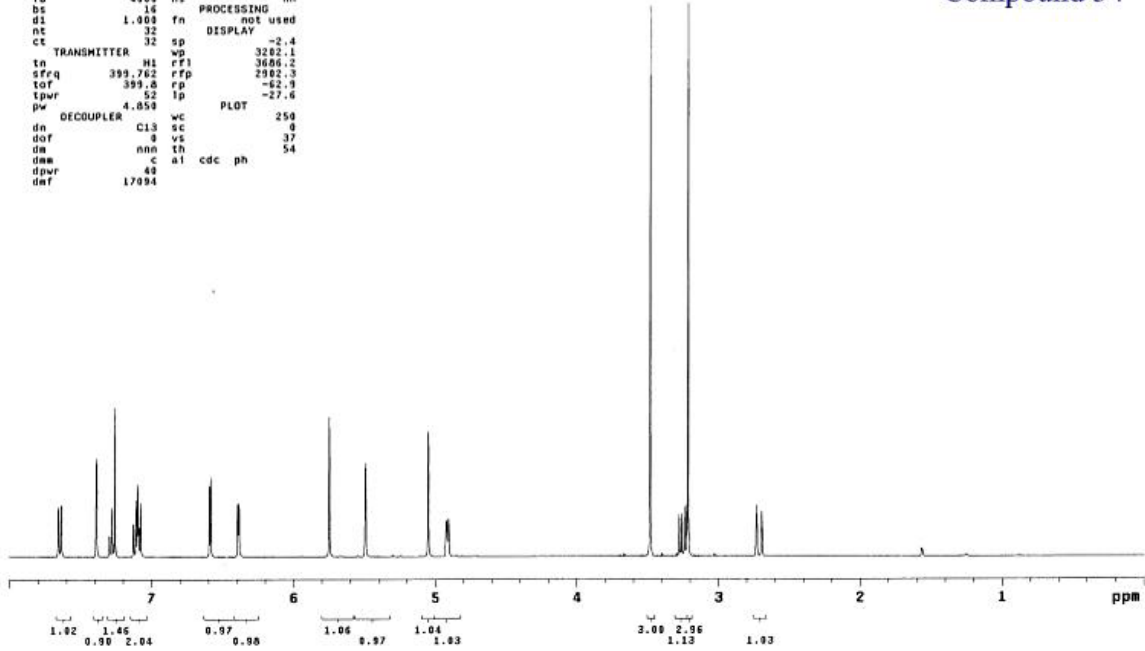
IV-4-125rcd Inova 400

expl s2pu1

```
SAMPLE SPECIAL
date Jul 27 2003 temp not used
solvent CDCl3 gain not used
file /export/home/~ sp in 28
kerr/vmer/sys/data/~ hst 0.000
IV-4-125rcd_27Jul2~ pw90 9.700
803/PROTON.fid alfa 6.600
ACQUISITION FLAGS
sv 6393.9 f1 n
at 3.744 f1 n
np 47892 dp y
fd 4003 hs nn
bs 16 PROCESSING
d1 1.000 f1 not used
nt 32 DISPLAY
ct 32 sp -2.4
TRANSMITTER wp 3282.1
tn H1 rfp 3686.2
sfrq 399.762 rfp 2902.3
tof 399.8 rfp -52.3
tpwr 52 lp -27.6
pw 4.850 PLOT 250
DECOUPLER C13 sc 0
dn 0 vs 37
dm nnn th 54
dwm c al cdc ph
dpwr 40
def 17094
```



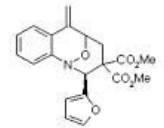
Compound 34



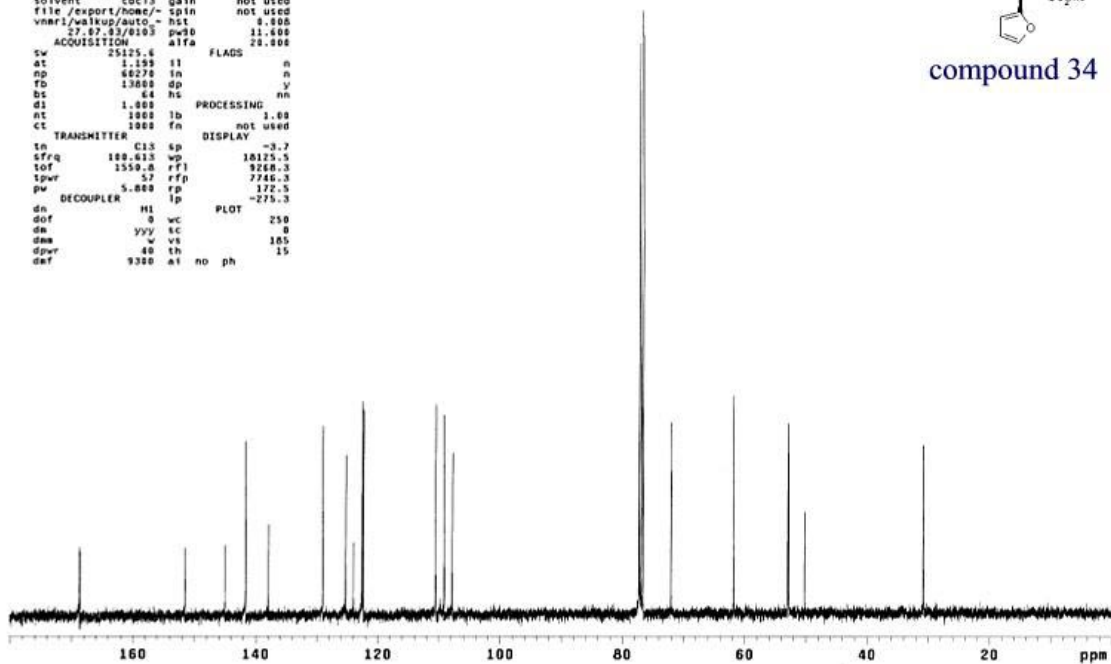
IV-4-125rcdcar
STANDARD IN OBSERVE

expl s2pu1

```
SAMPLE SPECIAL
date Jul 27 2003 temp not used
solvent cdcl3 gain not used
file /export/home/~ sp in 28
vmer/walkup/auto/~ hst 0.000
27.87_03/8103 pw90 11.600
ACQUISITION alfa 28.000
sv 25125.6 f1 n
at 1.199 f1 n
np 48276 dp y
fd 13800 hs nn
bs 64 hs PROCESSING
d1 1.000 f1 not used
nt 1000 f1 1.00
ct 1000 f1 DISPLAY
TRANSMITTER wp -3.7
tn C13 sp 18125.5
sfrq 1530.8 rfp 3268.3
tof 1530.8 rfp 7746.3
tpwr 57 rfp 172.5
pw 6.800 rfp -275.3
DECOUPLER HI PLOT 250
dn 0 vs 0
dm yyy sc 105
dwm w vs 15
dpwr 80 th
def 9300 al no ph
```



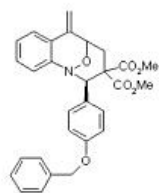
compound 34



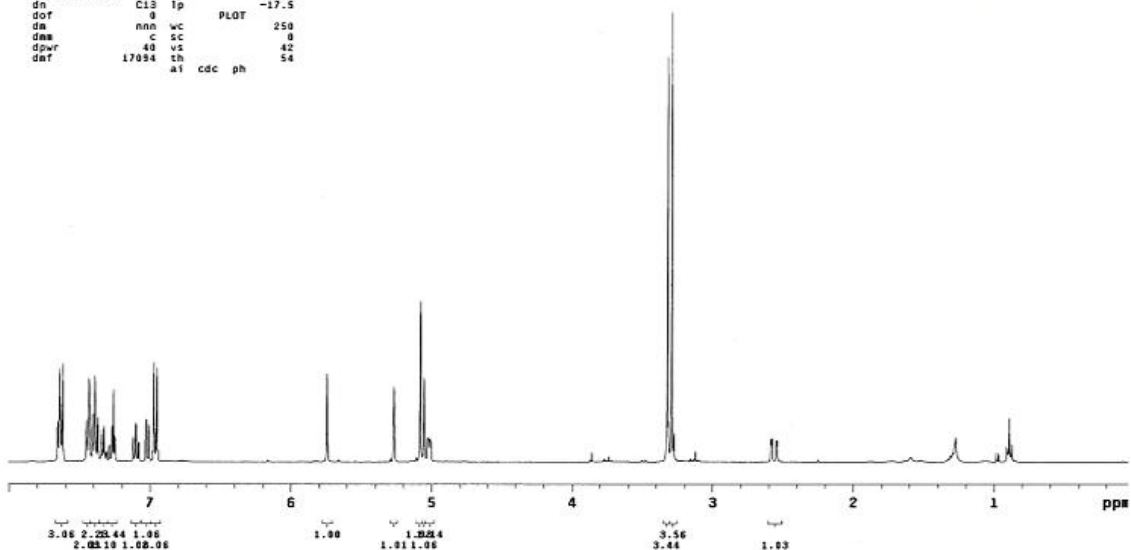
IY-4-177c Inova 400

expl s2pul

```
SAMPLE SPECIAL
date Sep 3 2003 temp not used
solvent cdcl3 gain not used
file /export/home/ spin not used
ACQUISITION hst 0.000
sw 6395.9 pw30 3.700
at 3.744 alfa 6.600
np 47800 FLADS
fb 4800 il n
bs 15 in n
d1 1.000 dp y
nt 15 hs nn
ct TRANSMITTER 18 fn PROCESSING not used
tn H1 sp DISPLAY -1.4
sfrq 399.762 wp 3282.1
tot 399.0 rfl 3685.3
tpwr 52 rfp 2982.3
pw DECOUPLER 4.859 rp 136.8
dn C13 lp -17.5
dof 0 PLOT 250
da nno wc 0
dwa c sc 0
dpwr 40 vs 42
dnt 17034 th 54
al cdc ph
```



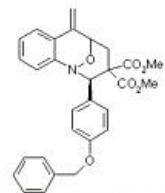
compound 35



IY-4-171f
STANDARD IN OBSERVE

expl s2pul

```
SAMPLE SPECIAL
date Sep 3 2003 temp not used
solvent cdcl3 gain not used
file /export/home/ spin not used
vnmr1/wakeup/auto_ hst 0.000
ACQUISITION hst 11.600
sw 25125.6 pw30 29.000
at 1.199 il n
np 60270 in n
fb 13000 dp y
bs 64 hs nn
d1 1.800 PROCESSING
nt 1800 lb not used
ct TRANSMITTER 704 fn DISPLAY 1.00
tn C13 sp DISPLAY -6.6
sfrq 100.613 wp 18125.5
tot 1559.6 rfl 9271.3
tpwr 57 rfp 7746.3
pw DECOUPLER 5.000 rp -160.6
dn H1 lp PLOT -331.0
dof 0 wc 250
da yyy sc 0
dwa w vs 181
dpwr 40 th 38
dnt 9300 al no ph
```



compound 35

