

Supporting Information

Stereoselective Construction of Functionalized Tetra and Pentacyclic Hybrid Coumarinopyranpyrazole / Pyrimidinedione / Coumarin Scaffolds Using Solid State Melt Reaction

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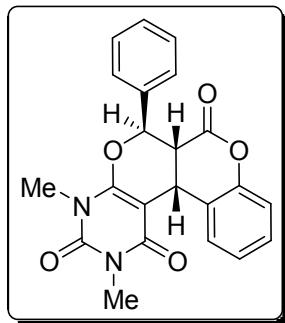
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Experimental Section

General Remarks: Melting points were recorded on a Superfit (India) capillary melting point apparatus and were uncorrected. IR spectra were recorded on a Bruker-FT-IR spectrometer using solid samples as KBr plates. For compounds ¹H NMR (300 MHz, CDCl₃) and ¹³C NMR (75 MHz, 100 MHz, CDCl₃) spectra were recorded in deuteriochloroform (CDCl₃) on a Bruker 300 MHz spectrometer using tetramethylsilane (TMS, δ = 0) as an internal standard at room temperature. Mass spectra were recorded on Electrospray Ionisation Method with Thermo Finnigan mass spectrometer. The X-ray diffraction measurements were carried on a Bruker AXS SMART APEX 2 diffractometer.

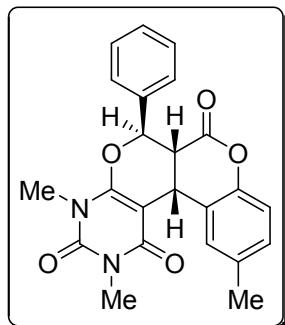
Representative procedure for the synthesis of tetracyclic coumarinopyranpyrimidinediones: A mixture of (*E*)-2-formylphenyl cinnamate (**2a**, 0.252g, 1mmol) and *N,N*-dimethylbarbituric acid (**3a**, 0.156g, 1mmol) was placed in a round bottom flask and melted at 180 °C for 1 h. After completion of the reaction as indicated by TLC, the crude product was washed with 5 mL of ethylacetate and hexane mixture (1:49 ratio) which successfully provided the pure product **5a** in 98% yield, as colorless solid.

4,6-dimethyl-9-phenyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5a**):**



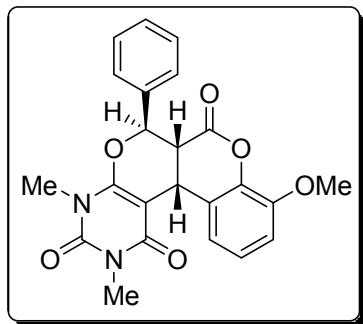
Yield: 98%; Colourless solid; mp:238-240 °C; ¹H NMR (300 MHz, CDCl₃): δ 3.32 - 3.33 (m, 1H), 3.37 (s, 3H), 3.44 (s, 3H), 4.60 (d, *J*=4.8Hz, 1H), 5.30 (d, *J* = 9.6Hz, 1H), 7.07-7.46 (m, 9H); ¹³C NMR (75 MHz): δ 28.3, 28.9, 30.5, 45.4, 77.8, 86.1, 116.9, 123.5, 125.4, 126.4, 128.0, 129.1, 129.3, 130.0, 134.4, 149.3, 150.7, 156.2, 162.8, 164.5; IR (KBr): 1725, 1703, 1642 cm⁻¹; MS (m/z): 391 (M⁺+1); Elemental Analysis for C₂₂H₁₈N₂O₅: Calculated: C, 67.69; H, 4.65; N, 7.18; Found: 67.58; H, 4.56; N, 7.26.

4,6,16-trimethyl-9-phenyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5b):



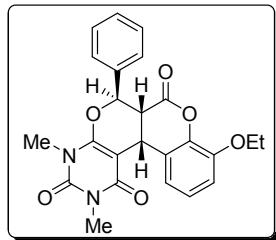
Yield: 96%; mp: 249-251°C; ¹H NMR (300 MHz, CDCl₃): δ 2.33 (s, 3H), 3.26 (dd, *J*=4.8, 9.9 Hz, 1H), 3.37 (s, 3H), 3.47 (s, 3H), 4.59 (d, *J*=4.2 Hz, 1H), 5.25 (d, *J*=9.9 Hz, 1H), 6.98-7.44 (m, 8H); ¹³C NMR (75 MHz): δ 21.0, 28.4, 28.9, 30.5, 45.6, 77.8, 86.1, 116.7, 123.4, 126.5, 129.1, 129.8, 129.9, 130.0, 134.4, 135.2, 147.2, 150.8, 156.2, 162.8, 164.7; IR (KBr): 1732, 1709, 1632 cm⁻¹; MS (m/z): 405 (M⁺+1); Elemental Analysis for C₂₃H₂₀N₂O₅: Calculated: C 68.31; H, 4.98; N, 6.93; Found: C 68.22; H, 4.86; N, 7.04.

14-methoxy-4,6-dimethyl-9-phenyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5c):



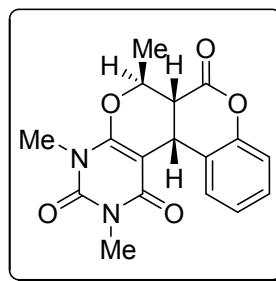
Yield: 93%; mp: 250-252°C; ¹H NMR (300 MHz, CDCl₃): δ 3.29-3.33 (m, 1H), 3.38 (s, 3H), 3.43 (s, 3H), 3.91 (s, 3H), 4.59 (d, *J*=4.8 Hz, 1H), 5.35 (d, *J*=9.6 Hz, 1H), 6.85-7.45 (m, 8H); ¹³C NMR (75 MHz): δ 28.3, 28.9, 30.6, 45.1, 56.2, 77.7, 86.1, 111.9, 121.0, 124.6, 125.1, 126.4, 128.0, 128.6, 129.1, 129.9, 134.5, 138.6, 147.6, 150.7, 156.2, 162.7, 163.9; IR (KBr): 1748, 1710, 1644 cm⁻¹; MS (m/z): 421 (M⁺+1); Elemental Analysis for C₂₃H₂₀N₂O₆: Calculated: C 65.71; H, 4.79; N, 6.66; Found: C 65.71; H, 4.79; N, 6.66.

14-ethoxy-4,6-dimethyl-9-phenyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5d)



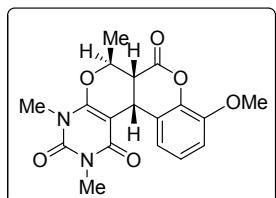
Yield: 91%; mp: 252-254 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.46 (t, $J=6.9\text{Hz}$, 3H), 3.27-3.32 (m, 1H), 3.37 (s, 3H), 3.43 (s, 3H), 4.13 (q, $J=6.3\text{Hz}$, 2H), 4.57 (d, $J=4.5\text{Hz}$, 1H), 5.36 (d, $J=9.3\text{ Hz}$ 1H); 6.82-7.44 (m, 8H); ^{13}C NMR (75 MHz): δ 14.7, 28.3, 28.9, 30.6, 45.1, 65.0, 77.7, 86.1, 113.4, 121.0, 124.7, 125.1, 126.4, 129.1, 129.9, 134.5, 139.0, 146.9, 150.8, 156.1, 162.7, 164.2; IR (KBr): 1726, 1709, 1637 cm^{-1} ; MS (m/z): 435 ($M^{+}+1$); Elemental Analysis for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_6$: Calculated: C 66.35; H, 5.10; N, 6.45; Found: C 66.28; H, 5.02; N, 6.56.

4,6,9-trimethyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5e):



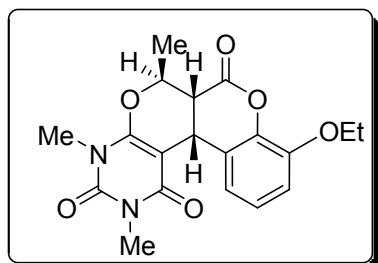
Yield: 92%; Colourless solid; mp: 244 – 246 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.57 (d, $J=6.3\text{Hz}$, 3H), 2.96 (dd, $J=5.1,9.9\text{Hz}$, 1H), 3.36 (s, 3H), 3.43 (s, 3H), 4.37-4.43 (m, 1H), 4.58 (d, $J=4.8\text{Hz}$, 1H), 7.03-7.31 (m, 4H); ^{13}C NMR (75 MHz): δ 18.8, 28.3, 28.7, 30.0, 44.7, 72.2, 85.7, 116.9, 123.7, 125.4, 129.0, 129.5, 149.1, 150.8, 156.0, 162.9, 165.6; IR (neat): ν 1785, 1576, 1530 cm^{-1} ; MS (m/z): 329 ($M^{+}+1$); Elemental Analysis for $\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_5$: Calculated: C 62.19; H, 4.91; N, 8.53; Found: C 62.08; H, 4.82; N, 8.45.

14-methoxy-4,6,9-trimethyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5f):



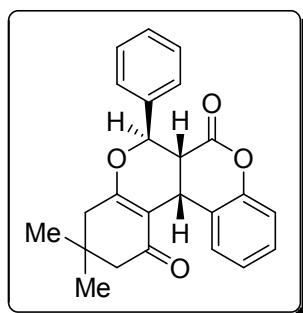
Yield: 90%; Colourless solid; mp: 228 – 230 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.59 (d, $J=6.3\text{Hz}$, 3H), 2.97 (dd, $J=4.8, 10.2\text{Hz}$, 1H), 3.37 (s, 3H), 3.44 (s, 3H), 3.90 (s, 3H), 4.38–4.48 (m, 1H), 4.59 (d, $J=4.8\text{ Hz}$, 1H), 6.74–7.12 (m, 3H); ^{13}C NMR (75 MHz): δ 28.3, 28.7, 30.2, 44.5, 56.1, 72.2, 85.7, 111.6, 118.6, 120.4, 124.8, 125.2, 138.4, 147.4, 150.7, 156.0, 162.8, 165.0; IR (neat): ν 1729, 1578, 1546 cm^{-1} ; MS (m/z): 359 (M^++1); Elemental Analysis for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_6$: Calculated: C 60.33; H, 5.06; N, 7.82; Found: C 60.24; H, 4.96; N, 7.94.

14-ethoxy-4,6,9-trimethyl-8,12-dioxa-4,6-diazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-3,5,11-trione (5g):



Yield: 91%; Colourless solid; mp: 231 – 233 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.46 (t, $J=6.9\text{Hz}$, 3H), 1.58 (d, $J=6\text{ Hz}$, 3H), 2.94 (dd, $J=4.8, 9.9\text{ Hz}$, 1H), 3.37 (s, 3H), 3.42 (s, 3H), 4.10 (q, $J=6.9\text{ Hz}$, 2H), 4.38–4.47 (m, 1H), 4.57 (d, $J=4.5\text{ Hz}$, 1H), 6.72 (d, $J=7.8\text{Hz}$, 1H), 6.87 (d, $J=8.4\text{Hz}$, 1H), 7.05 (t, $J=7.8\text{Hz}$, 1H); ^{13}C NMR (75 MHz): δ 14.7, 18.9, 28.3, 28.7, 30.2, 44.6, 64.9, 72.2, 85.7, 113.1, 120.4, 124.8, 125.2, 138.8, 146.8, 150.8, 156.0, 162.8, 165.2; IR (neat): ν 1719, 1583, 1549 cm^{-1} ; MS (m/z): 373 (M^++1); Elemental Analysis for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{O}_6$: Calculated: C 61.28; H, 5.41; N, 7.52; Found: C 61.19; H, 5.30; N, 7.63.

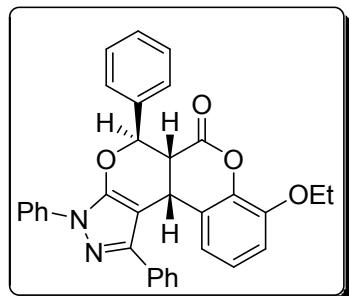
Methyl 4,6-dimethyl-12-[(4-methylbenzene)sulfonyl]-9-(3,4-dimethoxyphenyl)-3,5-dioxa-8-oxa-4,6,12-triazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-10-carboxylate (5h):



Yield: 90%; Colourless solid; mp: 198 – 200 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.16(s, 3H), 1.18 (s, 3H), 2.40 (d, $J = 15.6\text{ Hz}$, 2H), 2.50 (d, $J=16.8\text{Hz}$, 2H), 3.18(dd, $J=5.1, 9.6\text{ Hz}$, 1H),

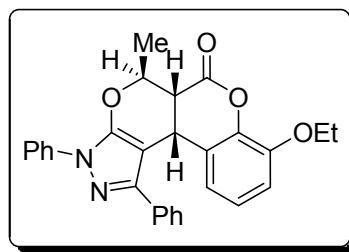
4.52 (d, $J=3.9$ Hz, 1H), 5.04 (t, $J=9.9$ Hz, 1H); 6.97-7.41 (m, 9H); ^{13}C NMR (75 MHz): δ 28.4, 29.6, 32.3, 42.3, 45.5, 50.7, 75.5, 109.4, 116.9, 124.5, 125.2, 125.9, 126.6, 128.0, 128.5, 128.8, 129.5, 135.6, 149.5, 165.2, 170.5, 197.2; IR (neat): ν 1743, 1598, 1529 cm^{-1} ; MS (m/z): 375 (M^++1).

11,14,16-triphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-9-one (8a):



Yield: 97%; Colourless solid; mp: 228 – 230 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.47 (t, $J=6.9$ Hz, 3H), 3.49 (dd, $J=4.5, 5.4$ Hz, 1H), 4.13 (q, $J=6.9$ Hz, 1H), 4.78 (s, 1H), 5.39 (d, $J=5.7$ Hz, 1H), 6.70-7.88 (m, 18H); $^{13}\text{CNMR}$ (CDCl_3 , 75 MHz): δ 14.8, 29.6, 32.2, 46.6, 65.0, 94.0, 113.6, 120.6, 121.4, 124.9, 125.2, 126.2, 126.6, 126.7, 128.3, 128.6, 128.9, 129.0, 129.5, 133.1, 135.8, 138.3, 139.3, 147.1, 148.1, 149.7, 165.1; IR (neat): ν 1734, 1583, 1537 cm^{-1} ; MS (m/z): 516 (M^++1); Elemental Analysis for $\text{C}_{33}\text{H}_{26}\text{N}_2\text{O}_4$: Calculated: C 77.03; H, 5.09; N, 5.44; Found: C 76.98; H, 5.00; N, 5.35.

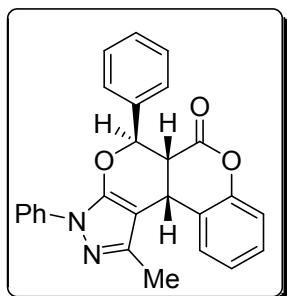
6-ethoxy-11-methyl-14,16-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-9-one (8b):



Yield: 90%; Colourless solid; mp: 226 – 228 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.47 (t, $J=6.0$ Hz, 1H), 1.59 (t, $J=6.0$ Hz, 3H), 3.19-3.22 (m, 1H), 4.11 (q, $J=6.3$ Hz, 1H), 4.44 (d, $J=9.6$ Hz, 1H), 4.82 (d, $J=4.2$ Hz, 1H), 6.68-7.87 (m, 13H); $^{13}\text{CNMR}$ (CDCl_3 , 75 MHz): δ 14.7, 19.1, 32.0, 45.9, 64.9, 71.4, 93.5, 113.2, 120.7, 121.1, 125.0, 125.4, 126.2, 126.3, 128.2, 128.7, 129.0, 133.2, 138.4, 139.0, 147.0, 147.8, 149.8, 166.3; IR (neat): ν 1720, 1569,

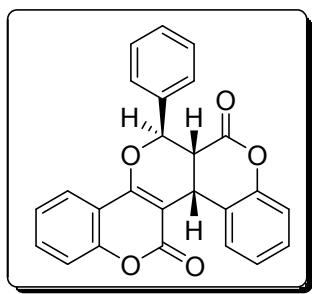
1544cm^{-1} ; MS (m/z): 454 (M^++1); Elemental Analysis for $C_{28}H_{24}N_2O_4$: Calculated: C 74.32; H, 5.35; N, 6.19; Found: C 74.24; H, 5.26; N, 6.28.

16-methyl-11,14-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-9-one(8c):



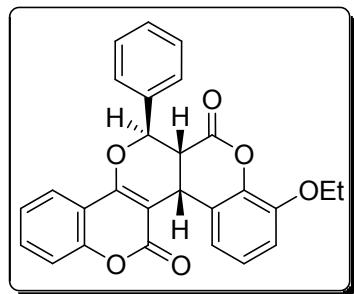
Yield: 92%; Colourless solid; mp: 233 – 235 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.64 (s, 1H), 1.98 (bs, 3H), 3.37 (s, 1H), 4.03 (bs, 1H), 7.10-7.80 (m, 14H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 13.8, 29.5, 44.7, 77.7, 94.3, 117.2, 120.5, 122.9, 124.6, 125.2, 125.8, 128.8, 129.0, 129.4, 129.8, 137.2, 138.2, 146.3, 148.8, 150.2, 166.3; IR (neat): ν 1733, 1567, 1539 cm^{-1} ; MS (m/z): 409 (M^++1); Elemental Analysis for $C_{26}H_{20}N_2O_3$: Calculated: C 76.45; H, 4.94; N, 6.86; Found: C 76.55; H, 4.99; N, 6.98.

12-phenyl-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaene-10,22-dione (10a):



Yield: 96%; Colourless solid; mp: 219 – 221 °C; ^1H NMR (300 MHz, CDCl_3): δ 3.33 (t, $J = 11.1\text{Hz}$, 1H), 4.50, (d, $J=13.2\text{Hz}$, 1H), 5.35 (d, $J=10.2\text{Hz}$, 1H), 7.09-7.83 (m, 13H); ^{13}C NMR (75 MHz): δ 33.1, 46.7, 78.9, 99.6, 116.7, 117.0, 123.2, 124.2, 124.7, 125.5, 128.2, 128.6, 129.0, 129.5, 132.7, 132.8, 136.5, 151.3, 152.9, 161.7, 163.0, 166.3; IR (neat): ν 1735, 1572, 1540 cm^{-1} ; MS (m/z): 397 (M^++1).

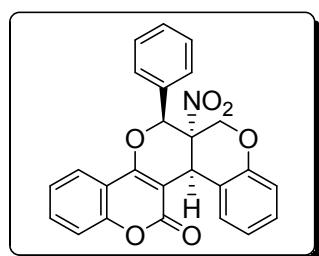
7-ethoxy-12-phenyl-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaene-10,22-dione (10b):



Yield: 92%; Colourless solid; mp: 222 – 224 °C; ^1H NMR (300 MHz, CDCl_3): δ 1.46 (t, $J=6.9\text{Hz}$, 3H), 3.40 (dd, $J = 4.2, 8.7\text{Hz}$, 1H), 4.14 (q, $J=6.6\text{Hz}$, 2H), 4.66 (d, $J=6.0\text{Hz}$, 1H), 5.38 (d, $J=12.3\text{Hz}$, 1H), 6.86-7.82(m, 12H); ^{13}C NMR (75 MHz): δ 14.7, 31.4, 45.0, 65.0, 75.9, 99.6, 113.2, 113.6, 114.7, 116.8, 120.8, 123.0, 124.1, 124.3, 125.2, 126.6, 128.2, 128.5, 129.0, 129.7, 132.7, 135.2, 139.2, 147.1, 153.0, 160.5; IR (neat): ν 1699, 1589, 1523 cm^{-1} ; MS (m/z): 441 (M^++1).

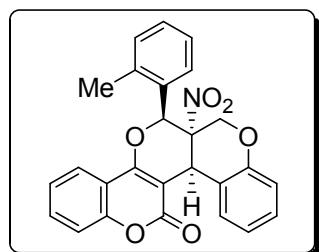
Representative procedure for synthesis of pentacyclic coumarinopyrancoumarins: A mixture of *O*-alkylated product (**11a**, 0.36g, 0.841 mmol) and 4-hydroxy-2*H*-chromen-2-one (**6b**, 0.12g, 0.841 mmol) was placed in a round bottom flask and melted at 180 °C for 1 h. After completion of the reaction as indicated by TLC, the crude product was washed with 5 mL of ethylacetate and hexane mixture (1:49 ratio) which successfully provided the pure product **13a** as colorless solid.

12-phenyl-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (13a**)**



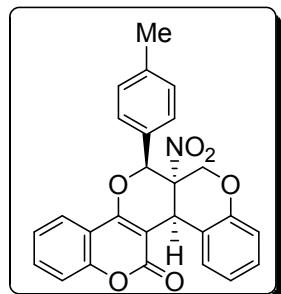
Yield: 90%; Colourless solid; mp: 228-230°C; ^1H NMR (300 MHz, CDCl_3): δ 4.54 (d, $J = 12.6\text{Hz}$, 1H), 4.77 (d, $J = 12.6\text{Hz}$, 1H), 5.24 (s, 1H), 5.83 (s, 1H), 6.63-7.83 (m, 13H); ^{13}C NMR (75 MHz): δ 38.1, 60.8, 79.9, 99.7, 116.7, 117.0, 123.3, 124.3, 124.7, 125.5, 128.2, 128.6, 129.0, 132.7, 132.8, 136.5, 156.3, 158.8, 161.7, 162.0, 164.3; IR (neat): ν 1692, 1580, 1538 cm^{-1} ; MS (m/z): 442 (M^++1); Elemental Analysis for $\text{C}_{26}\text{H}_{19}\text{NO}_6$: Calculated: C 70.74; H, 4.34; N, 3.17; Found: C 70.62; H, 4.24; N, 3.28.

12-(2-methylphenyl)-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (13a)



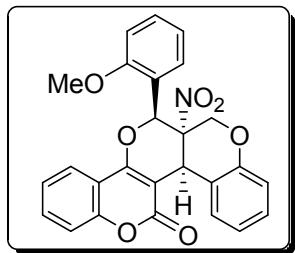
Yield: 92%; Colourless solid; mp: 239-241°C; ¹H NMR (300 MHz, CDCl₃): δ 2.42 (s, 3H), 4.70 (d, *J* = 12.3 Hz, 1H), 4.87 (d, *J* = 12.3 Hz, 1H), 5.39 (s, 1H), 6.33 (s, 1H), 6.49-7.77 (m, 12H); ¹³C NMR (75 MHz): δ 19.5, 37.1, 63.7, 77.7, 86.0, 102.6, 114.3, 116.0, 116.6, 116.9, 121.3, 123.1, 124.2, 125.7, 127.0, 129.2, 129.6, 130.7, 131.0, 132.3, 132.8, 136.6, 151.9, 152.5, 158.7, 161.2; IR (neat): ν 1687, 1582, 1535cm⁻¹; MS (m/z): 442 (M⁺+1); Elemental Analysis for C₂₆H₁₉NO₆: Calculated: C 70.74; H, 4.34; N, 3.17; Found: C 70.62; H, 4.24; N, 3.28.

12-(4-methylphenyl)-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (13b):



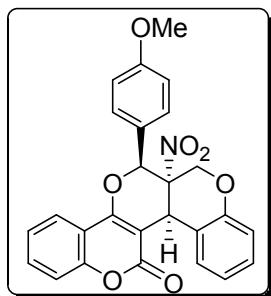
Yield: 90%; Colourless solid; mp: 241 -243 °C; ¹H NMR (300 MHz, CDCl₃): δ 2.37 (s, 3H), 4.54 (d, *J* = 12.9 Hz, 1H), 4.78 (d, *J* = 12.9 Hz, 1H), 5.24 (s, 1H), 5.82(s, 1H), 6.62-7.83 (m, 12H); ¹³C NMR (75 MHz): δ 21.2, 30.9, 38.3, 62.1, 81.2, 86.6, 103.2, 114.3, 116.0, 116.2, 116.6, 121.0, 122.9, 124.2, 126.6, 128.4, 129.4, 129.5, 132.8, 133.6, 140.2, 152.2, 152.5, 158.2, 160.5; IR (neat): ν 1684, 1569, 1598cm⁻¹; MS (m/z): 442 (M⁺+1); Elemental Analysis for C₂₆H₁₉NO₆: Calculated: C 70.74; H, 4.34; N, 3.17; Found: C 70.64; H, 4.26; N, 3.28.

12-(2-methoxyphenyl)-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (13c):



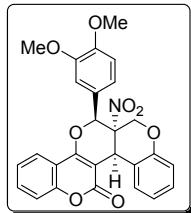
Yield: 89%; Colourless solid; mp: 236-238 °C; ¹H NMR (300 MHz, CDCl₃): δ 3.90 (s, 3H), 4.54 (d, *J* = 11.7 Hz, 1H), 4.86 (d, *J* = 11.7 Hz, 1H), 5.38 (s, 1H), 6.29-7.80 (m, 12H); ¹³C NMR (75 MHz): δ 34.7, 55.5, 65.4, 74.8, 85.8, 101.5, 110.0, 114.4, 115.7, 116.7, 118.3, 119.7, 121.4, 121.7, 123.2, 124.2, 127.1, 128.6, 130.1, 130.4, 132.7, 151.6, 152.7, 157.0, 158.9, 162.2; IR (neat): ν 1679, 1593, 1529 cm⁻¹; MS (m/z): 458 (M⁺+1); Elemental Analysis for C₂₆H₁₉NO₇: Calculated: C 68.27; H, 4.19; N, 3.06; Found: C 68.38; H, 4.28; N, 3.14.

12-(4-dimethoxyphenyl)-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11.0^{3,8.0^{15,20]}}] docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (13d)}



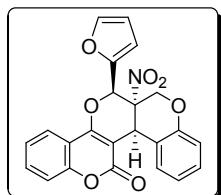
Yield: 90%; Colourless solid; mp: 245 -247°C; ¹H NMR (300 MHz, CDCl₃): δ 3.81 (s, 3H), 4.54 (d, *J* = 12.9 Hz, 1H), 4.77 (d, *J*=12.6 Hz, 1H), 5.23 (s, 1H), 5.82 (s, 1H), 6.62-7.83 (m, 12H); ¹³C NMR (75 MHz): δ 38.2, 55.3, 62.2, 81.1, 86.6, 103.1, 114.1, 114.3, 116.1, 116.2, 116.6, 121.0, 122.9, 123.3, 124.2, 128.1, 129.5, 132.8, 133.4, 152.1, 152.5, 158.3, 160.8; IR (neat): ν 1687, 1583, 1522cm⁻¹; MS (m/z): 459 (M⁺+1); Elemental Analysis for C₂₆H₁₉NO₇: Calculated: C 68.27; H, 4.19; N, 3.06; Found: C 68.15; H, 4.08; N, 3.14.

12-(3,4-dimethoxyphenyl)-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11.0^{3,8.0^{15,20]}}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (13e)}



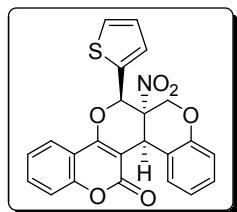
Yield: 92%; Colourless solid; mp: 189-191 °C; ^1H NMR (300 MHz, CDCl_3): δ 3.80 (s, 3H), 3.90(s, 3H), 4.56 (d, $J = 12.6\text{Hz}$, 1H), 4.78 (d, $J = 15\text{Hz}$, 1H), 5.24 (s, 1H), 5.83 (s, 1H), 6.64 – 7.83 (m, 11H); ^{13}C NMR (75 MHz): δ 38.1, 55.9, 56.0, 62.4, 81.3, 86.6, 109.6, 111.0, 114.2, 116.2, 116.3, 116.6, 119.8, 121.1, 122.9, 123.7, 124.2, 129.6, 132.8, 133.2, 149.1, 150.4, 152.2, 152.5, 160.6; IR (neat): ν 1691, 1574, 1526 cm^{-1} ; MS (m/z): 488 ($M^{++}1$); Elemental Analysis for $\text{C}_{27}\text{H}_{21}\text{NO}_8$: Calculated: C 66.53; H, 4.34; N, 2.87; Found: C 66.42; H, 4.23; N, 2.98.

12-(furan-2-yl)-11-nitro-9,13,21-trioxapentacyclo[12.8.0.0^{2,11.0^{3,8.0^{15,20]}}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (16a)}



Yield: 88%; Colourless solid; mp: 159-161 °C; ^1H NMR (300 MHz, CDCl_3): δ 4.72 (d, $J = 12.9\text{Hz}$, 1H), 4.97 (d, $J = 12.9\text{ Hz}$, 1H), 5.19 (s, 1H), 6.05 (s, 1H), 6.29 – 7.80 (m, 11H); ^{13}C NMR (75 MHz): δ 37.1, 64.6, 74.8, 86.5, 102.1, 110.7, 112.4, 114.2, 116.3, 116.6, 117.6, 121.5, 123.0, 124.2, 129.2, 131.9, 132.8, 144.2, 144.7, 152.1, 152.5, 158.2, 160.9; IR (neat): ν 1689, 1584, 1537 cm^{-1} ; MS (m/z): 418 ($M^{++}1$); Elemental Analysis for $\text{C}_{23}\text{H}_{15}\text{NO}_7$: Calculated: C 66.19; H, 3.62; N, 3.36; Found: C 66.08; H, 3.50; N, 3.44.

11-nitro-12-(thiophen-2-yl)-9,13,21-trioxapentacyclo[12.8.0.0^{2,11.0^{3,8.0^{15,20]}}]docosa-1(14),3,5,7,15(20),16,18-heptaen-22-one (16b)}

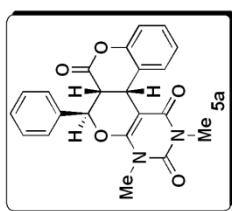


Yield: 86%; Colourless solid; mp: 145-147 °C; ^1H NMR (300 MHz, CDCl_3): δ 4.56 (d, $J = 13.2\text{Hz}$, 1H), 4.94 (d, $J = 12.9\text{ Hz}$, 1H), 5.16 (s, 1H), 6.15 (s, 1H), 6.74 - 7.84 (m, 11H); ^{13}C

NMR (75 MHz): δ 39.3, 62.5, 78.4, 87.3, 103.4, 114.0, 116.0, 116.5, 116.6, 121.1, 122.9, 124.3, 127.4, 127.6, 128.1, 129.9, 132.6, 132.9, 133.9, 152.5, 152.5, 157.9, 160.0; IR (neat): ν 1678, 1589, 1517 cm⁻¹; MS (m/z): 434 (M⁺+1); Elemental Analysis for C₂₃H₁₅NO₆S: Calculated: C 63.73; H, 3.49; N, 3.23; Found: C 63.61; H, 3.38; N, 3.34.



3.284
3.301
3.316
3.330
3.373
3.440
4.588
4.604
5.280
5.312
7.068
7.092
7.118
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Current Data Parameters
NAME VM-I-94
EXPNO 7
PROCNO 1

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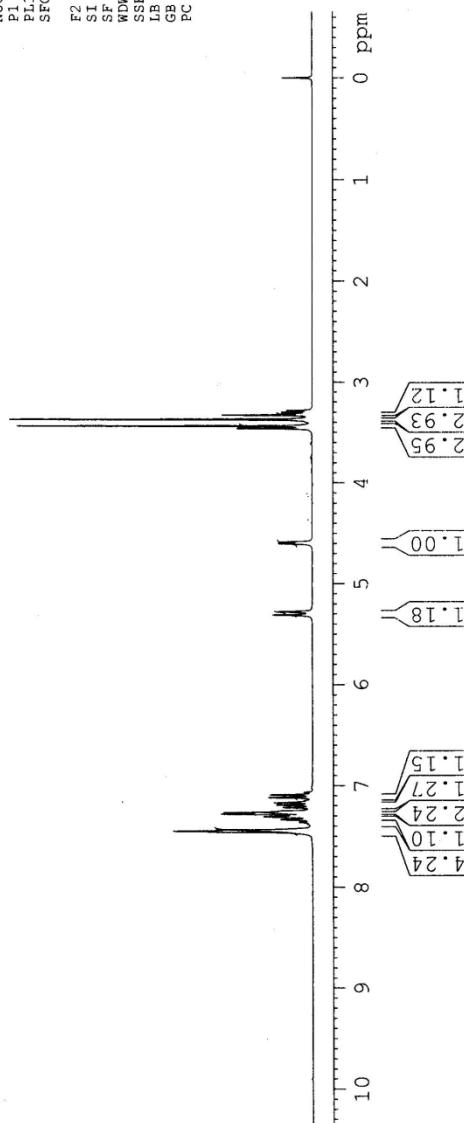
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D1 1.0000000 sec
TDO 1

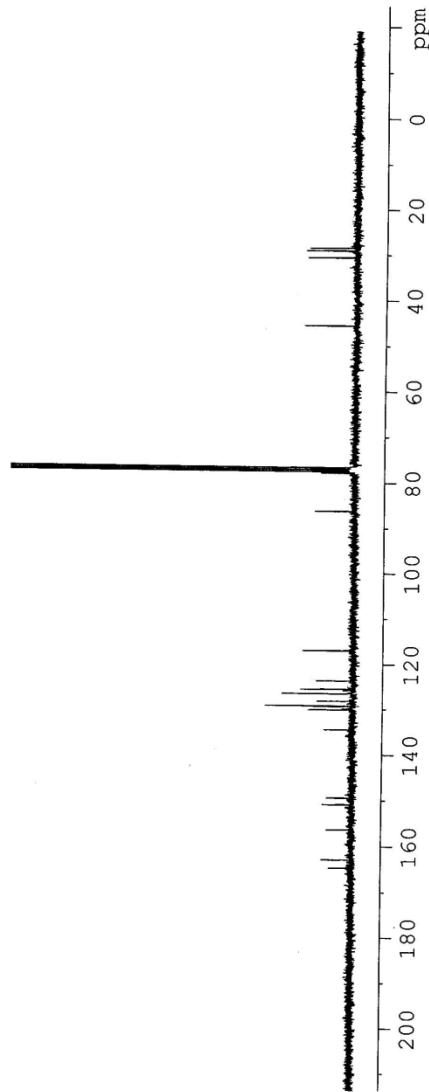
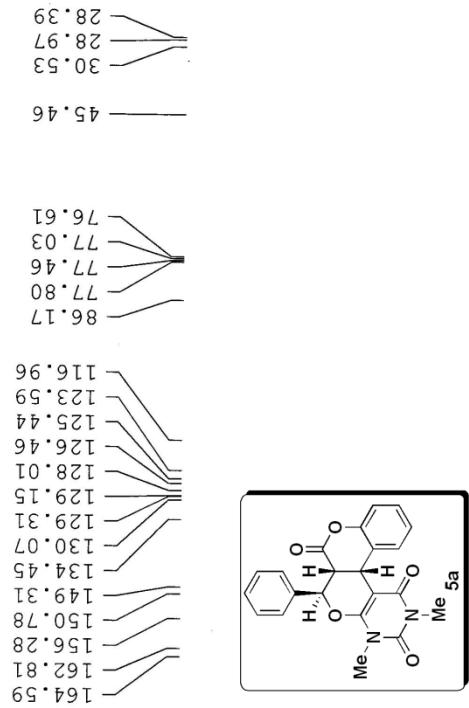
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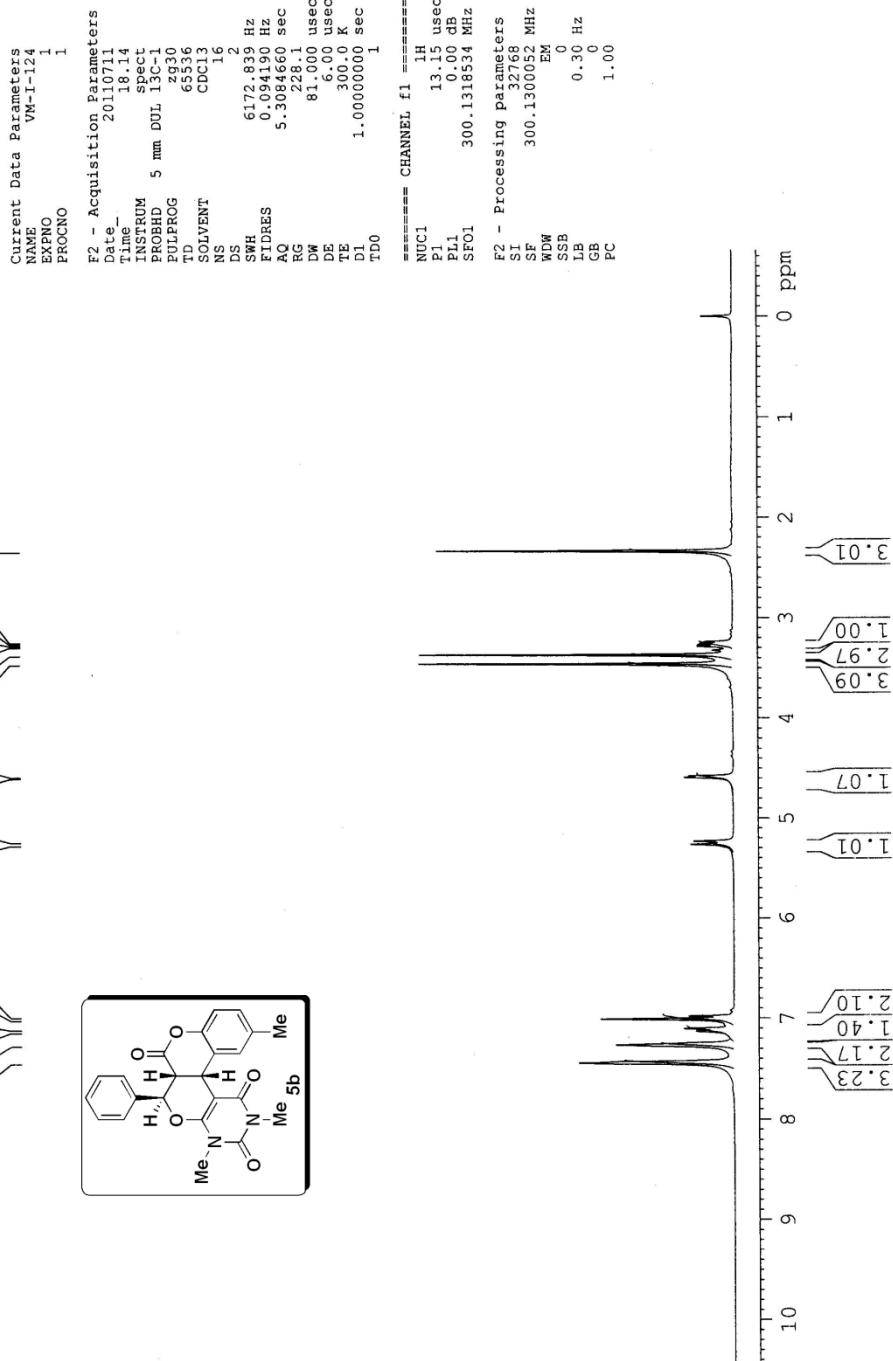
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F2 - Processing Parameters

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GB 0
PC 1.00

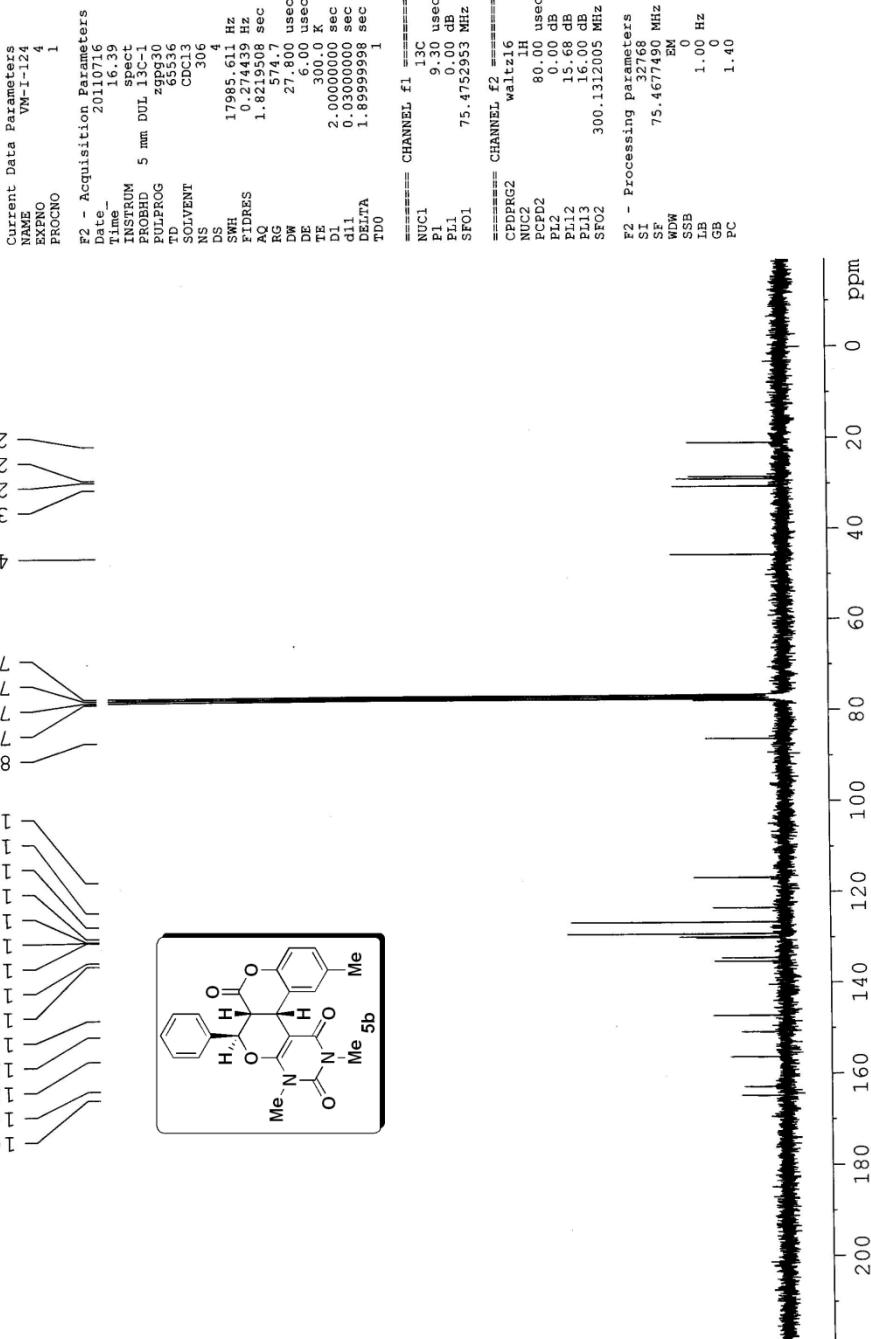
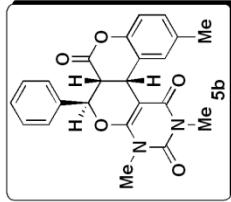


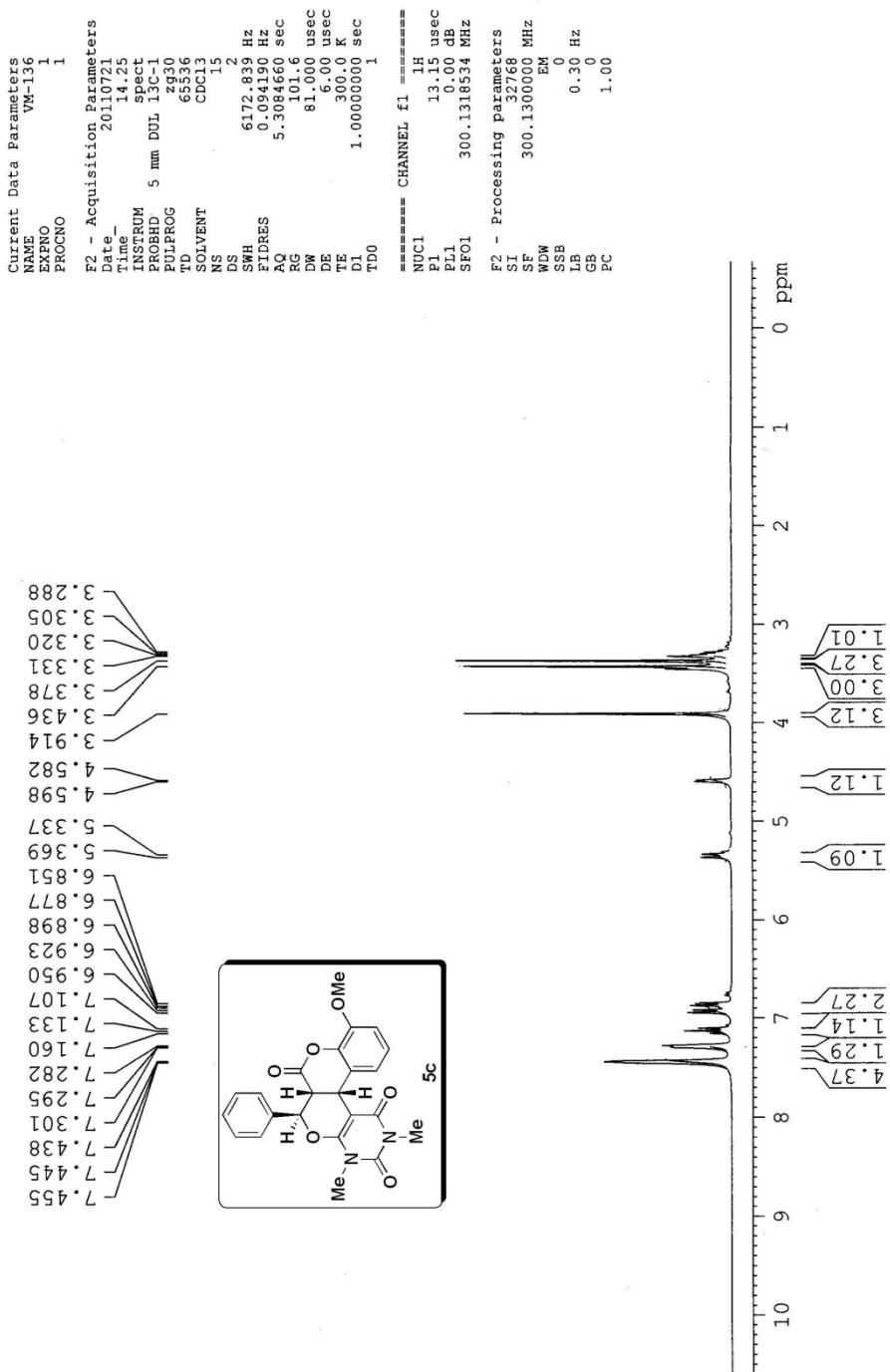


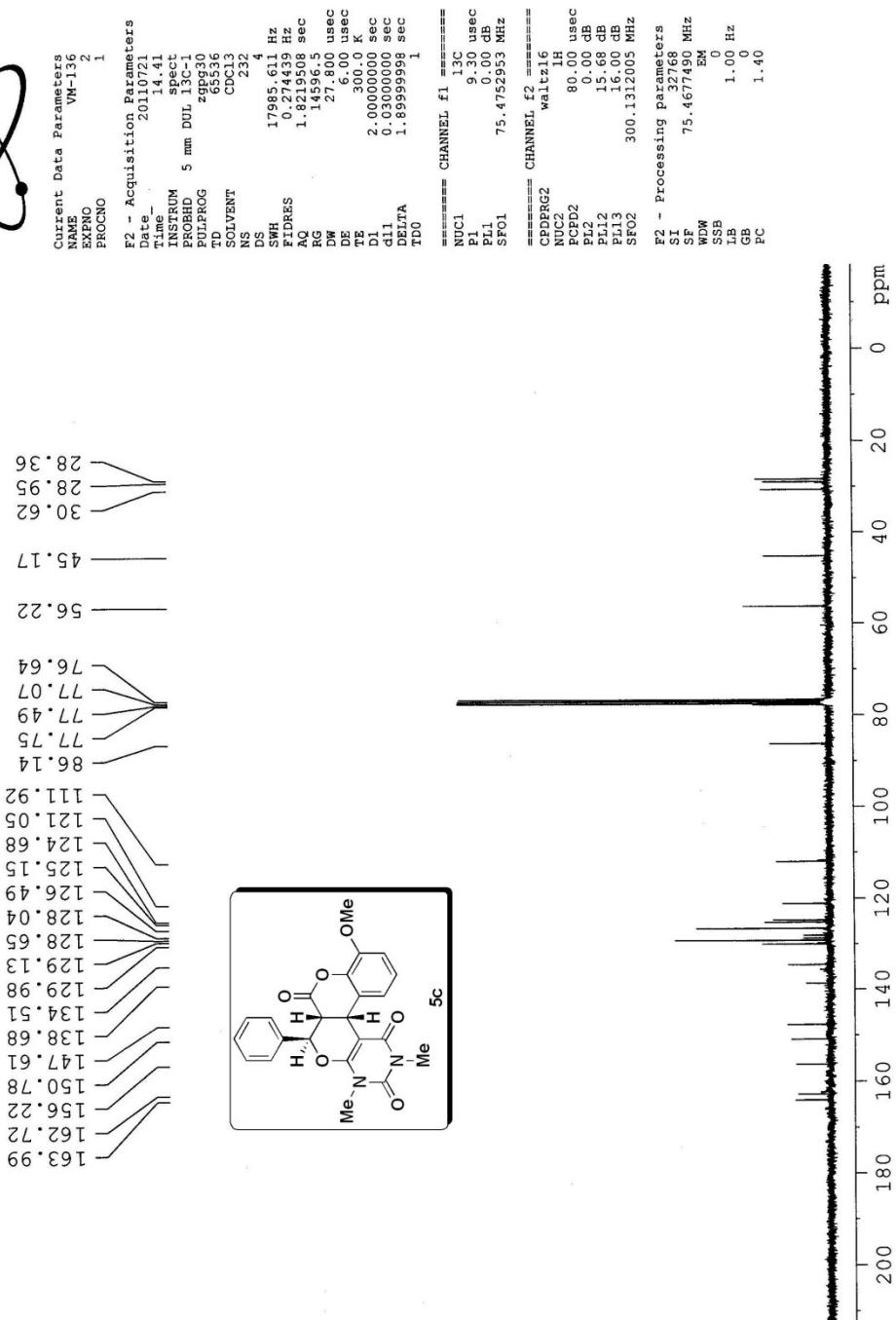




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Current Data Parameters
NAME VM-I-105
EXPNO 1
PROCNO 1

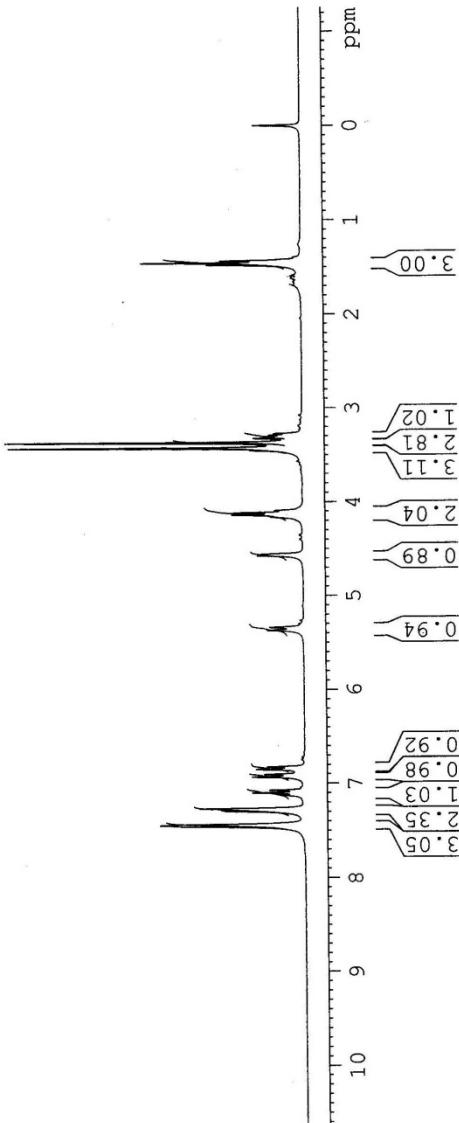
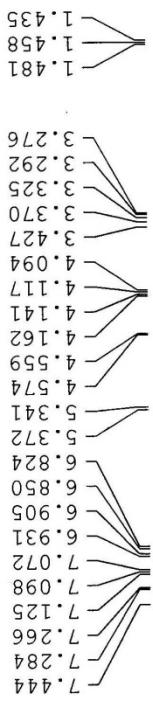
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Time 20.11

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TD 65536
SOLVENT CDCl3
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TDO

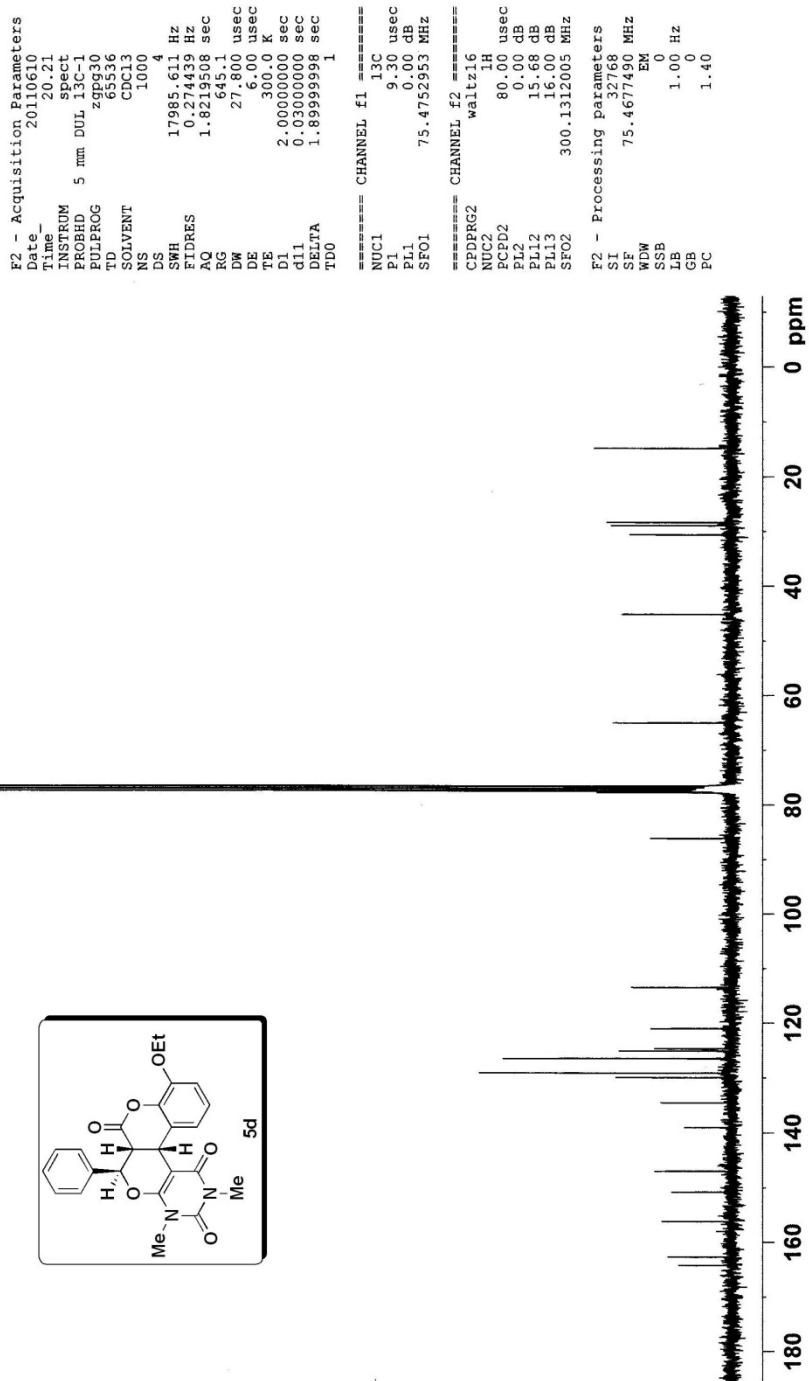
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F2 - Processing Parameters
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SF 300.1300048 MHz
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters
NAME VM-I-105
EXPNO 2
PROCNO 1





Current Data Parameters

NAME VM-I-Me- nmba
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

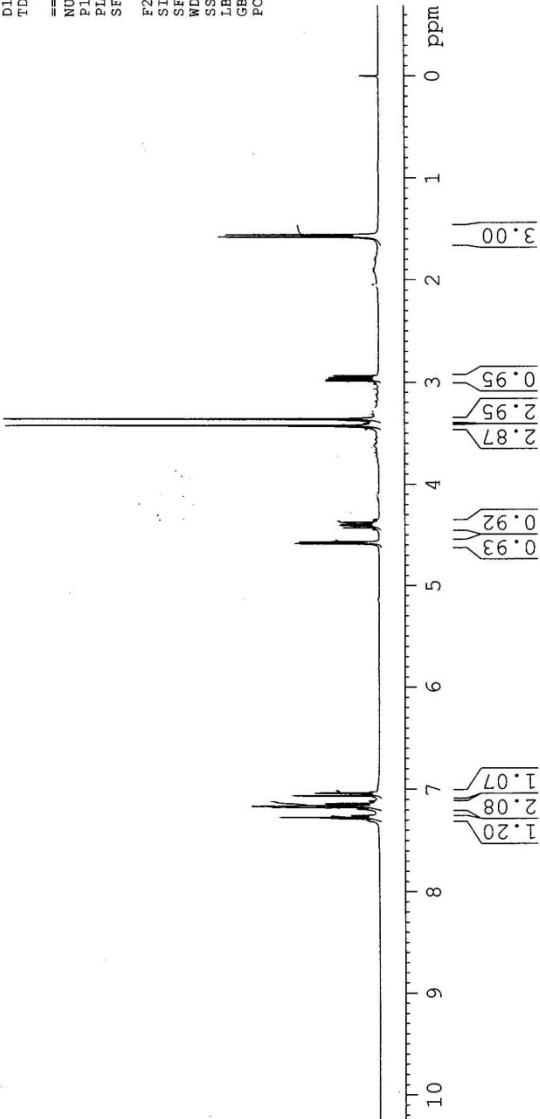
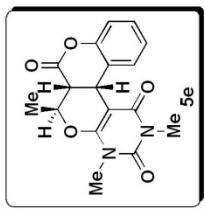
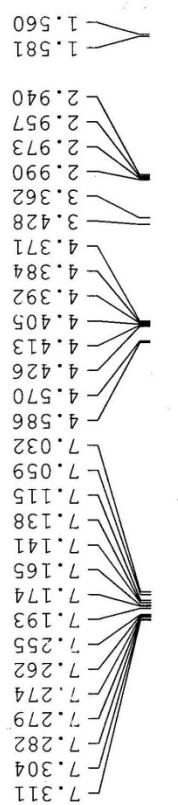
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TD 2930
SOLVENT CDCl3
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SWH 6172.83 Hz
FIDRES 0.094190 Hz
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RG 161.3
DW 81.0000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====

NUC1 1H
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PL1 0.00 dB
SF01 300.1318534 MHz

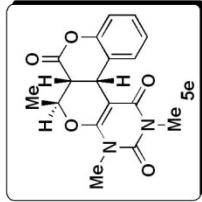
F2 - Processing Parameters

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LB 0.30 Hz
GB 0
PC 1.00





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 30.09
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 28.34
 18.83



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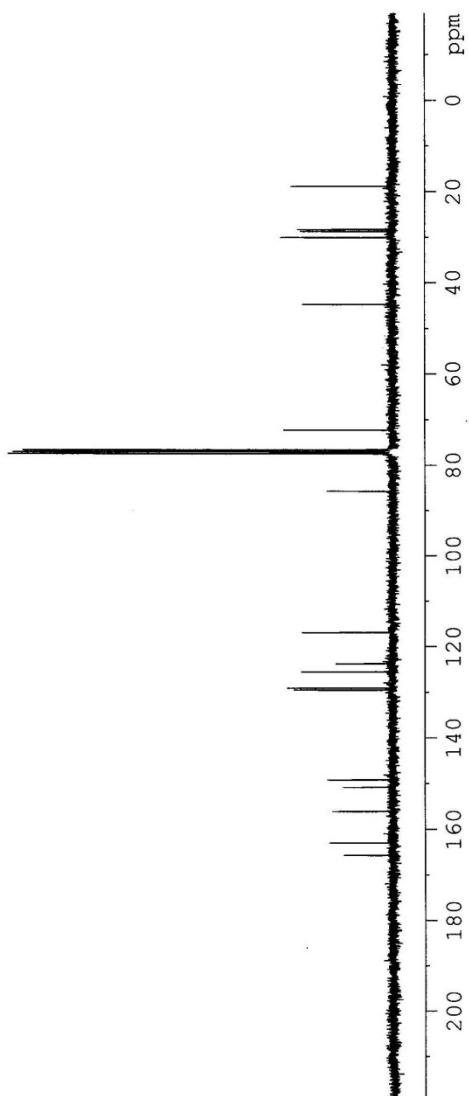
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EXPNO     2
PROCNO   1

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Time_     15.45
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PULPROG zpg30
TD      6536
SOLVENT  CDCl3
NS       194
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FIDRES  0.274439 Hz
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DE      6.00 usec
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TEC     2.0000000 sec
D1      0.0300000 sec
DETA    1.8999998 sec
TBO     1

===== CHANNEL F1 =====
NUC1      13C
P1        9.30 usec
PL1      75.4752553 MHz
SF01

===== CHANNEL F2 =====
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NUC2      1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     15.68 dB
PL13     16.00 dB
SF02    300.1112005 MHz

==== Processing parameters ====
SI      32768
SF      75.467790 MHz
WMW
SSB
EM      0
LB      1.00 Hz
GB      0
PC      1.40
  
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The Bruker logo consists of the word "BRUKER" in a bold, sans-serif font, with two stylized atomic orbits forming a circle around the letters.

| Current Data Parameters | |
|-------------------------|----------|
| NAME | VM-I-151 |
| EXPNO | 1 |
| PROCNO | 1 |

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=====
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parameters
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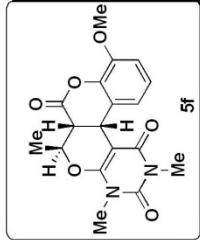
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                                      CDCL13
COLEVNT:                                16
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E:                                     300.0 K
E:                                     1.0000000 sec
D0:                                     1

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UCL1:                                0.00 dB
UCL1:                                300.1318534 MHz
UFO1:                                32768
UFO1:                                EM
UFO1:                                300.13100000 MHz
SB:                                     0
SB:                                     0.30 Hz
SB:                                     1.00 C

```

The figure shows a proton NMR spectrum with the x-axis representing chemical shift (δ) in ppm, ranging from 0 to 10. The spectrum exhibits several distinct signals. Key peaks are annotated with their corresponding chemical shift values:

- Peak at $\delta = 0.62$ ppm (labeled 0.62)
- Peak at $\delta = 1.04$ ppm (labeled 1.04)
- Peak at $\delta = 1.17$ ppm (labeled 1.17)
- Peak at $\delta = 1.18$ ppm (labeled 1.18)
- Peak at $\delta = 1.19$ ppm (labeled 1.19)
- Peak at $\delta = 1.20$ ppm (labeled 1.20)
- Peak at $\delta = 1.21$ ppm (labeled 1.21)
- Peak at $\delta = 1.22$ ppm (labeled 1.22)
- Peak at $\delta = 1.23$ ppm (labeled 1.23)
- Peak at $\delta = 1.24$ ppm (labeled 1.24)
- Peak at $\delta = 1.25$ ppm (labeled 1.25)
- Peak at $\delta = 1.26$ ppm (labeled 1.26)
- Peak at $\delta = 1.27$ ppm (labeled 1.27)
- Peak at $\delta = 1.28$ ppm (labeled 1.28)
- Peak at $\delta = 1.29$ ppm (labeled 1.29)
- Peak at $\delta = 1.30$ ppm (labeled 1.30)
- Peak at $\delta = 1.31$ ppm (labeled 1.31)
- Peak at $\delta = 1.32$ ppm (labeled 1.32)
- Peak at $\delta = 1.33$ ppm (labeled 1.33)
- Peak at $\delta = 1.34$ ppm (labeled 1.34)
- Peak at $\delta = 1.35$ ppm (labeled 1.35)
- Peak at $\delta = 1.36$ ppm (labeled 1.36)
- Peak at $\delta = 1.37$ ppm (labeled 1.37)
- Peak at $\delta = 1.38$ ppm (labeled 1.38)
- Peak at $\delta = 1.39$ ppm (labeled 1.39)
- Peak at $\delta = 1.40$ ppm (labeled 1.40)
- Peak at $\delta = 1.41$ ppm (labeled 1.41)
- Peak at $\delta = 1.42$ ppm (labeled 1.42)
- Peak at $\delta = 1.43$ ppm (labeled 1.43)
- Peak at $\delta = 1.44$ ppm (labeled 1.44)
- Peak at $\delta = 1.45$ ppm (labeled 1.45)
- Peak at $\delta = 1.46$ ppm (labeled 1.46)
- Peak at $\delta = 1.47$ ppm (labeled 1.47)
- Peak at $\delta = 1.48$ ppm (labeled 1.48)
- Peak at $\delta = 1.49$ ppm (labeled 1.49)
- Peak at $\delta = 1.50$ ppm (labeled 1.50)
- Peak at $\delta = 1.51$ ppm (labeled 1.51)
- Peak at $\delta = 1.52$ ppm (labeled 1.52)
- Peak at $\delta = 1.53$ ppm (labeled 1.53)
- Peak at $\delta = 1.54$ ppm (labeled 1.54)
- Peak at $\delta = 1.55$ ppm (labeled 1.55)
- Peak at $\delta = 1.56$ ppm (labeled 1.56)
- Peak at $\delta = 1.57$ ppm (labeled 1.57)
- Peak at $\delta = 1.58$ ppm (labeled 1.58)
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- Peak at $\delta = 1.61$ ppm (labeled 1.61)
- Peak at $\delta = 1.62$ ppm (labeled 1.62)
- Peak at $\delta = 1.63$ ppm (labeled 1.63)
- Peak at $\delta = 1.64$ ppm (labeled 1.64)
- Peak at $\delta = 1.65$ ppm (labeled 1.65)
- Peak at $\delta = 1.66$ ppm (labeled 1.66)
- Peak at $\delta = 1.67$ ppm (labeled 1.67)
- Peak at $\delta = 1.68$ ppm (labeled 1.68)
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- Peak at $\delta = 1.76$ ppm (labeled 1.76)
- Peak at $\delta = 1.77$ ppm (labeled 1.77)
- Peak at $\delta = 1.78$ ppm (labeled 1.78)
- Peak at $\delta = 1.79$ ppm (labeled 1.79)
- Peak at $\delta = 1.80$ ppm (labeled 1.80)
- Peak at $\delta = 1.81$ ppm (labeled 1.81)
- Peak at $\delta = 1.82$ ppm (labeled 1.82)
- Peak at $\delta = 1.83$ ppm (labeled 1.83)
- Peak at $\delta = 1.84$ ppm (labeled 1.84)
- Peak at $\delta = 1.85$ ppm (labeled 1.85)
- Peak at $\delta = 1.86$ ppm (labeled 1.86)
- Peak at $\delta = 1.87$ ppm (labeled 1.87)
- Peak at $\delta = 1.88$ ppm (labeled 1.88)
- Peak at $\delta = 1.89$ ppm (labeled 1.89)
- Peak at $\delta = 1.90$ ppm (labeled 1.90)
- Peak at $\delta = 1.91$ ppm (labeled 1.91)
- Peak at $\delta = 1.92$ ppm (labeled 1.92)
- Peak at $\delta = 1.93$ ppm (labeled 1.93)
- Peak at $\delta = 1.94$ ppm (labeled 1.94)
- Peak at $\delta = 1.95$ ppm (labeled 1.95)
- Peak at $\delta = 1.96$ ppm (labeled 1.96)
- Peak at $\delta = 1.97$ ppm (labeled 1.97)
- Peak at $\delta = 1.98$ ppm (labeled 1.98)
- Peak at $\delta = 1.99$ ppm (labeled 1.99)
- Peak at $\delta = 2.00$ ppm (labeled 2.00)



6.741
6.767
6.879
6.906
7.066
7.093
7.119
7.282

- 4.601
 - 4.479
 - 4.458
 - 4.437
 - 4.424
 - 4.403
 - 3.905
 - 3.438
 - 3.373
 - 2.993
 - 2.977
 - 2.959
 - 2.943

1.599 1.578



Current Data Parameters

NAME VM-I-151
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date 20110723
Time 17:09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 113
DS 4
SWH 17985.611 Hz
FIDRES 0.27439 Hz
AQ 1.8815908 sec
RG 20612.5
DW 27.00 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
d1.1 0.0300000 sec
DELT1A 1.8999998 sec
TD0 1

===== CHANNEL f1 =====

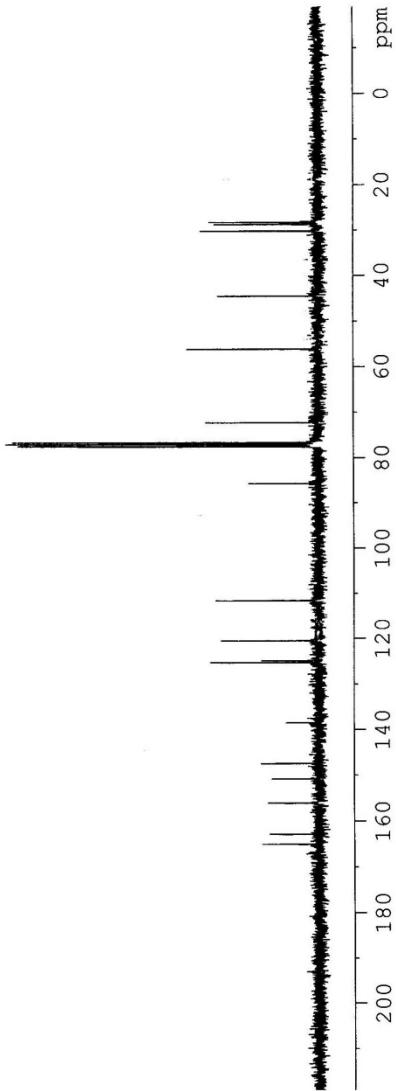
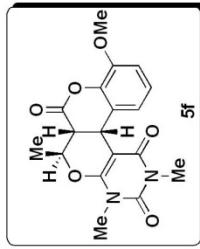
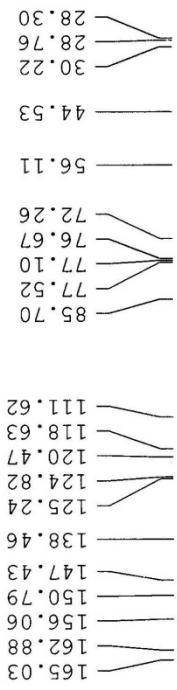
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SPO1 75.4152953 MHz

===== CHANNEL f2 =====

CPDRG2
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SPO2 300.131205 MHz

F2 - Processing parameters

SI 32768
SF 75.467790 MHz
WM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

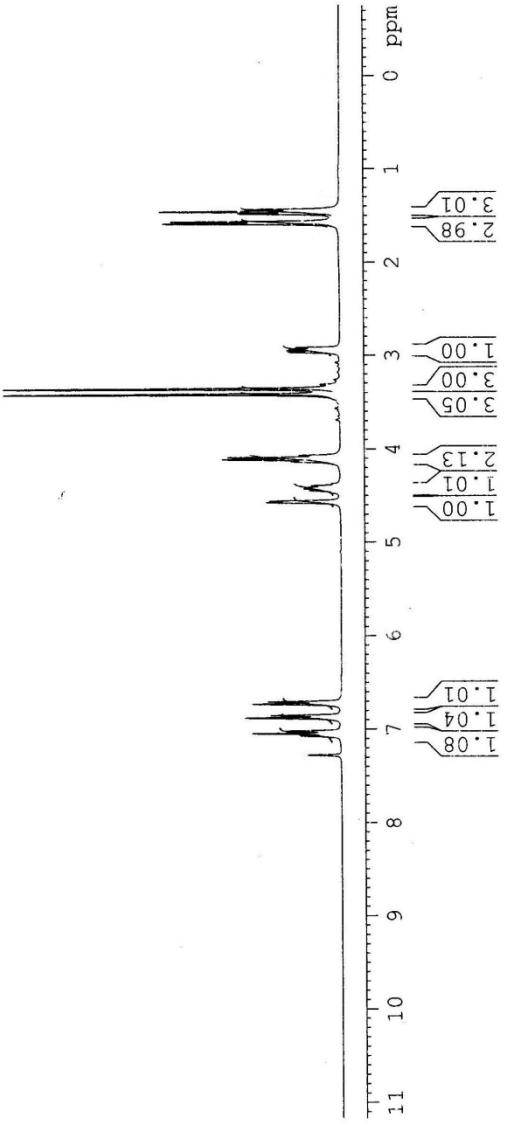
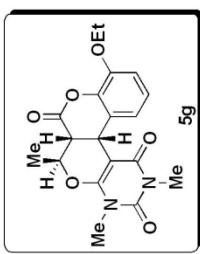


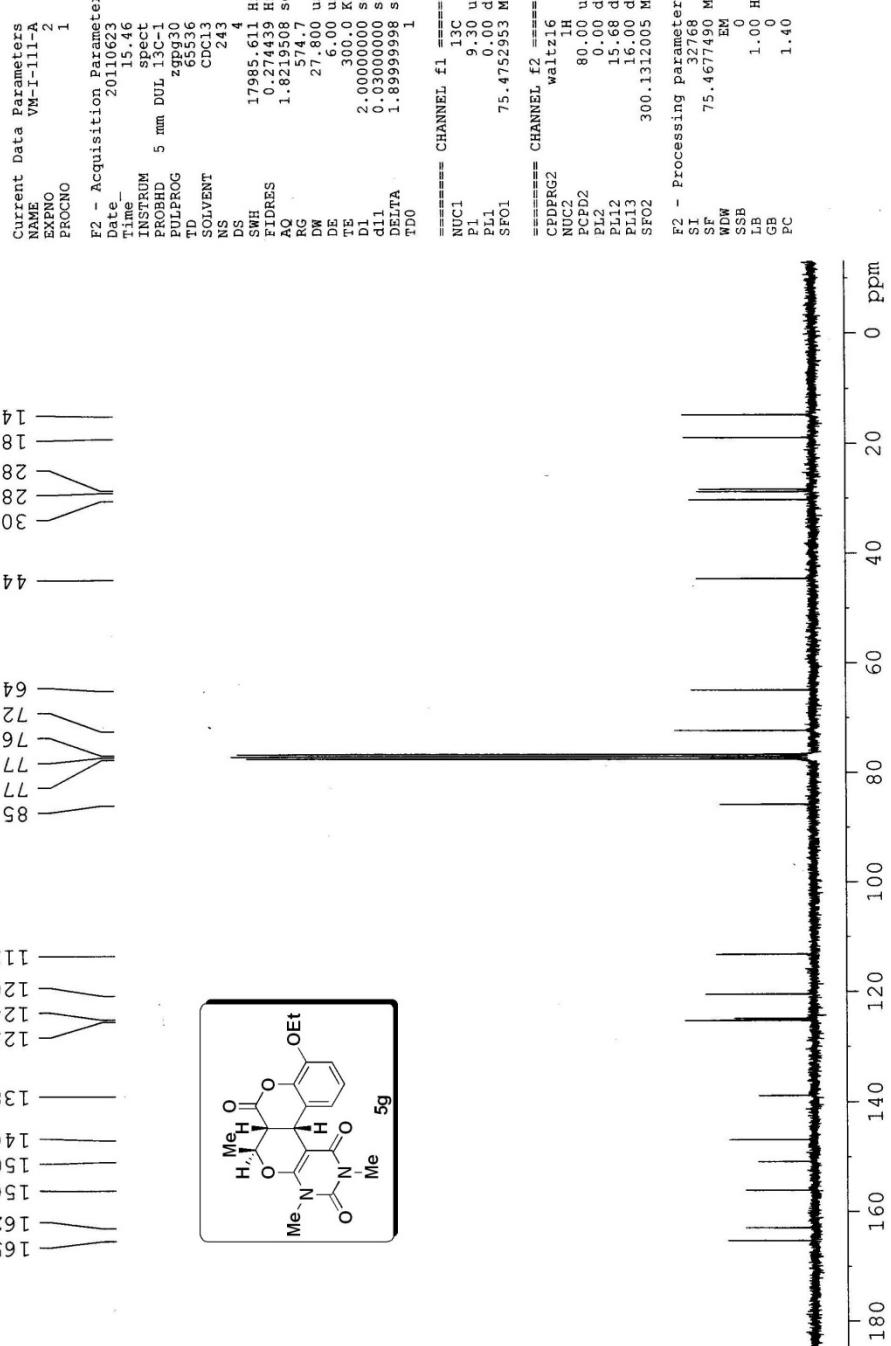


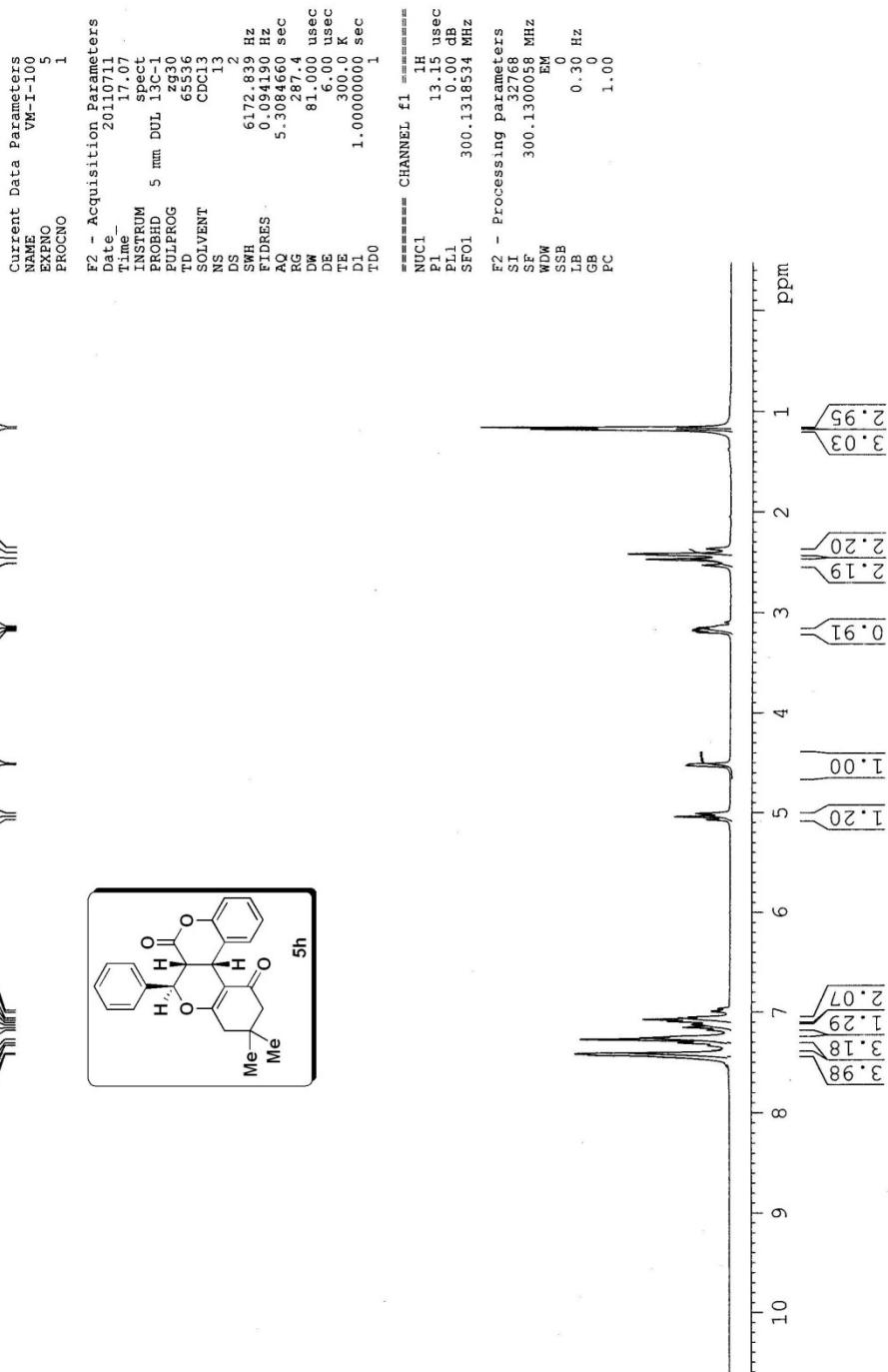
Current Data Parameters
NAME: 1M-1-11-A
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date: 2010-07-3
Time: 15:34
INSTRUM: spect
PROBID: 5 mm DUL 13C-1
PULPROG: 2930
TD: 65316
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 6172.839 Hz
FIDRES: 0.091150 Hz
AQ: 5.3084650 sec
RG: 143.7
DW: 81.000 usec
DE: 6.00 usec
TE: 300.0 K
D1: 1.0000000 sec
T1D0:

==== CHANNEL F1 =====
NUC1: 1H
P1: 13.15 usec.
PL1: 0.00 dB
SFO1: 300.1318534 MHz
F2 - Processing Parameters
SI: 32768
SF: 300.1300013 MHz
WDW:
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00





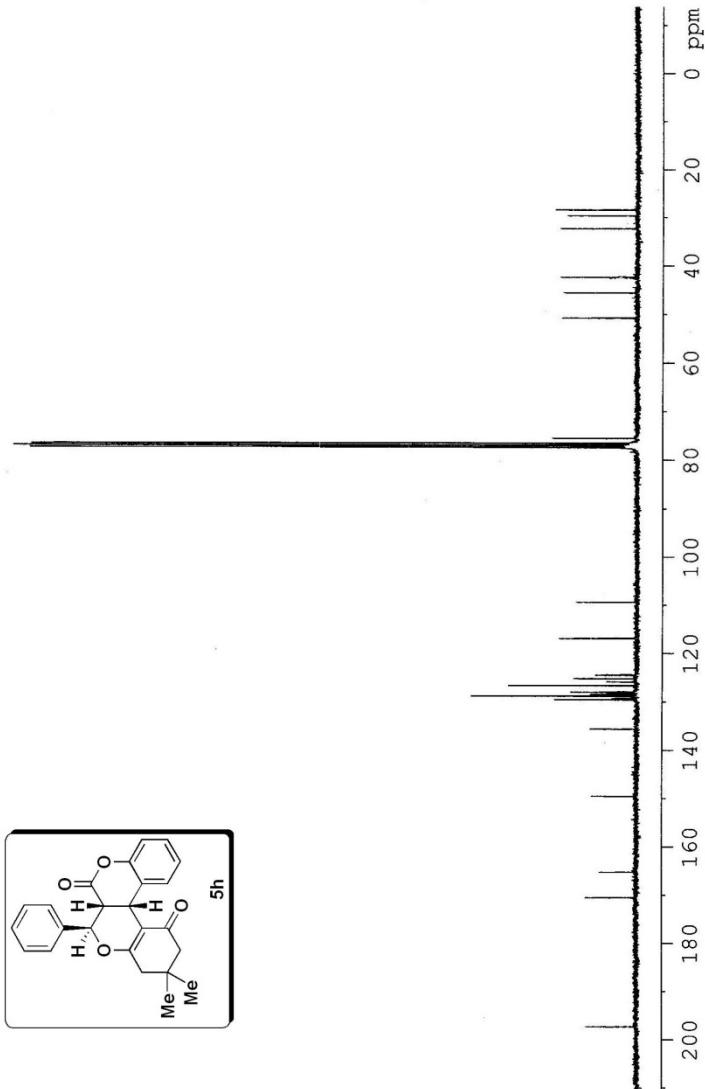
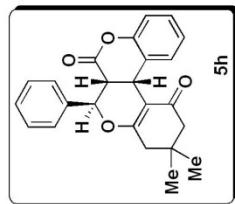
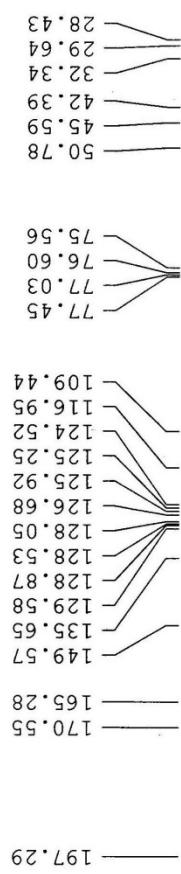




Current Data Parameters
NAME VM-T-110
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date 20110716
Time 21.43
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10.18
DS 4
SWH 17985.611 Hz
ETRINES 0.224439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.1800 ussec
DE 6.00 usec
TE 300.0 K
D1 2.000000 sec
d11 0.0300000 sec
DETA 1.6993998 sec
TDO 1

===== CHANNEL f1 =====
NUCL 13C
P1 9.30 usec
PL1 0.00 dB
SFOL 75.4752933 MHz
===== CHANNEL f2 =====
CPDPFG2 walt16
NUCL 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFOL2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.467450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



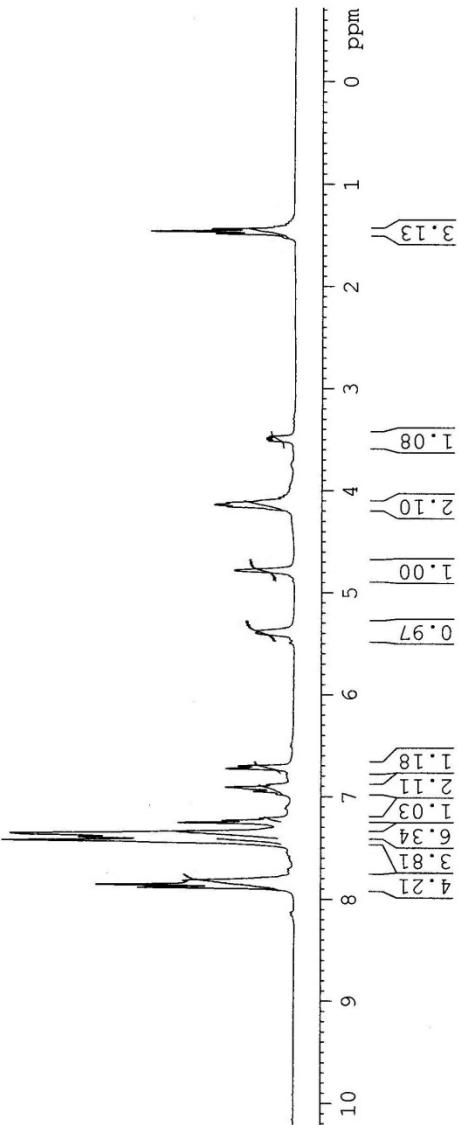
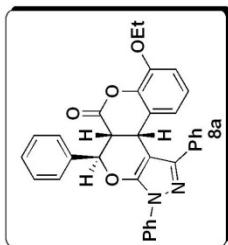


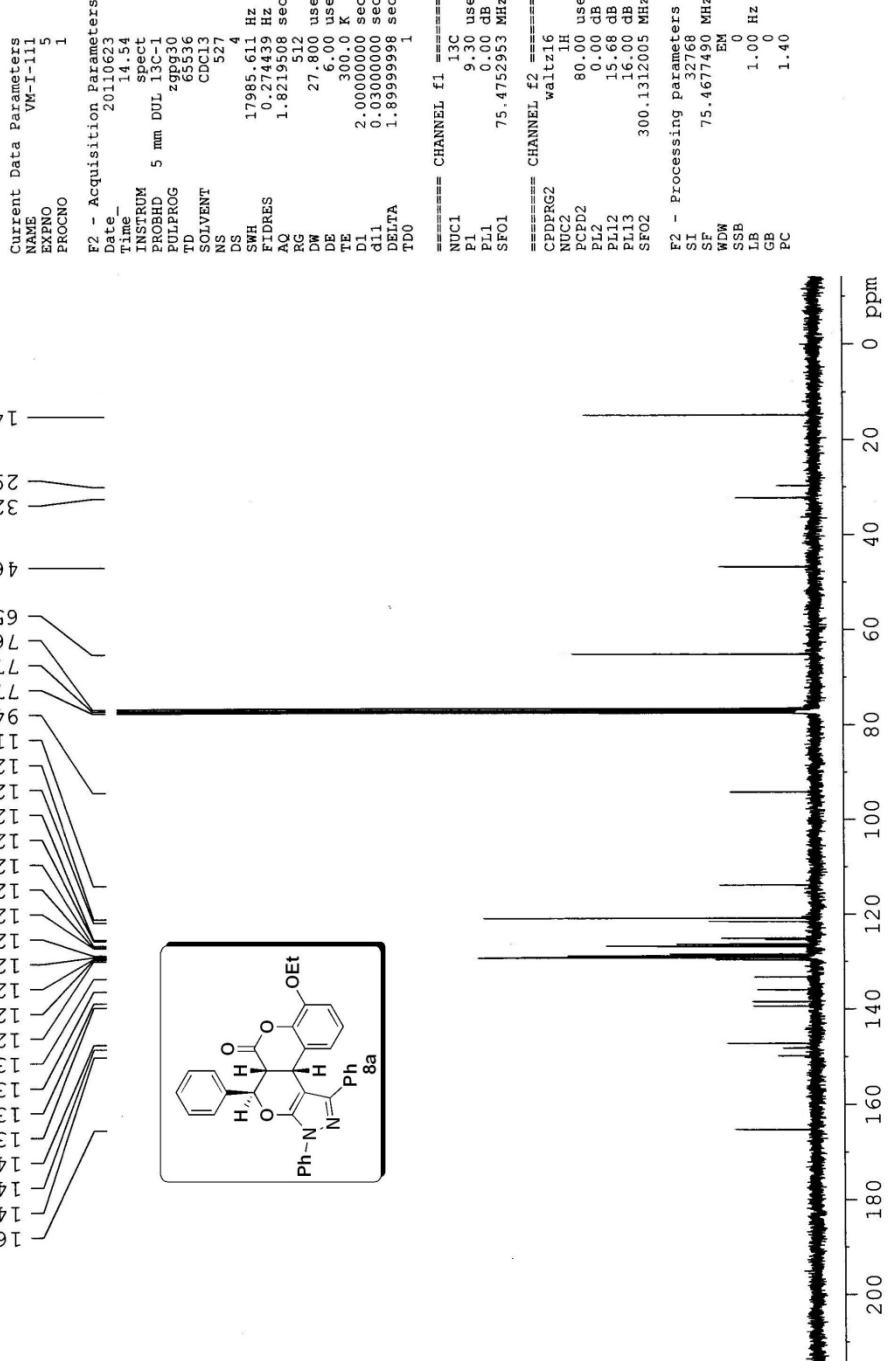
Current Data Parameters
NAME VM-I-111
EXPNO 4
PROCNO 1

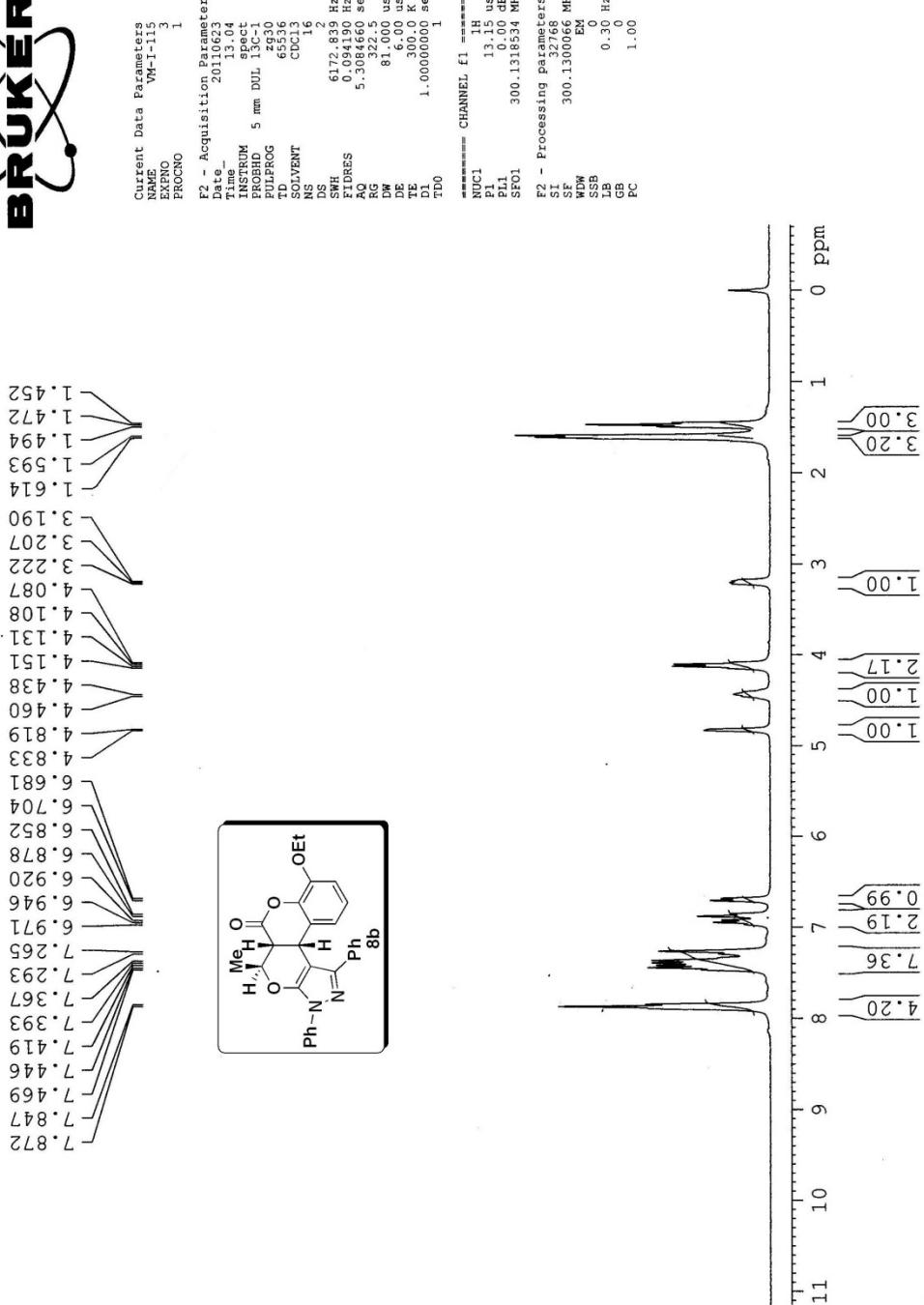
F2 - Acquisition Parameters
Date 20110623
Time 14.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG 2930
TD 6536
SOLVENT CDCl3
NS 16
DS 1
SWH 6172.899 Hz
F1RES 0.094190 Hz
AQ 5.308460 sec
RG 101
DW 81.00 usc
DE 6.00 usc
TE 300.0 K
D1 1.000000 sec
TDO 1.

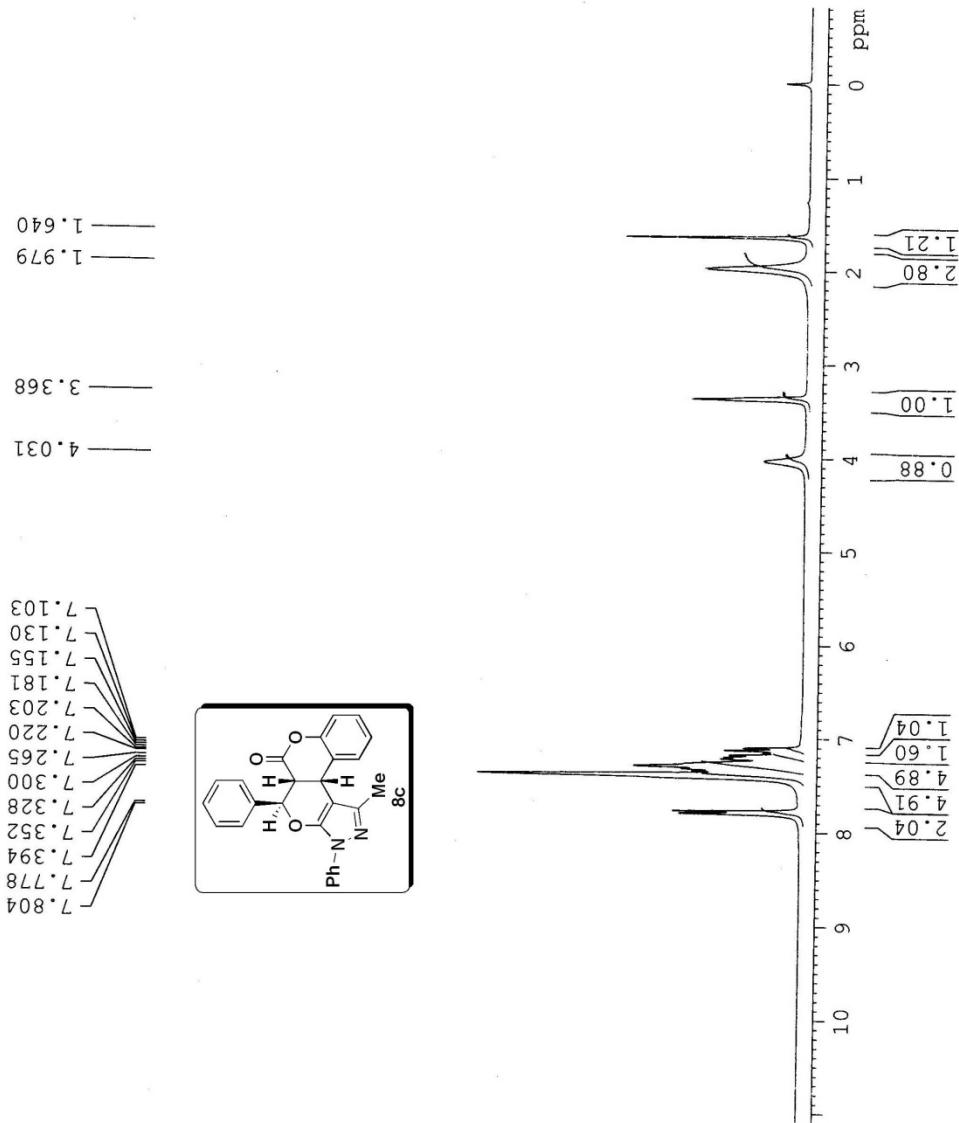
===== CHANNEL f1 =====
NUC1 1H
P1 13.15 usc
PL1 0.00 dB
SF01 300.131854 MHz

F2 - Processing parameters
SI 32768
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00









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| | | |
|---------|------|------------|
| Current | Data | Parameters |
| NAME | | DK-UK |
| EXPNO | | 2 |

```

F2 - Acquisition Parameters
Date: 20110624
Time: 13:34
INSTRUM spect
PROBOD 5 mm DUL 13C-1
PULPROG zgpp30
TD 65536
TE 1000
SOLVENT CDCl3
NS 4
DS 17995.11 Hz
SWH 0.274439 Hz
FIDRES 1.821508 sec
AQ 362
RG 27.800 ussec
DW 6.000 ussec
DE 30.0 K
TE 300.0 sec
D1 2.0000000 sec
D11 0.03000000 sec
DETA 1.8999998 sec

```

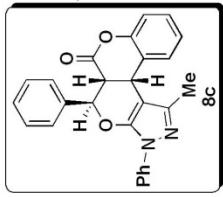
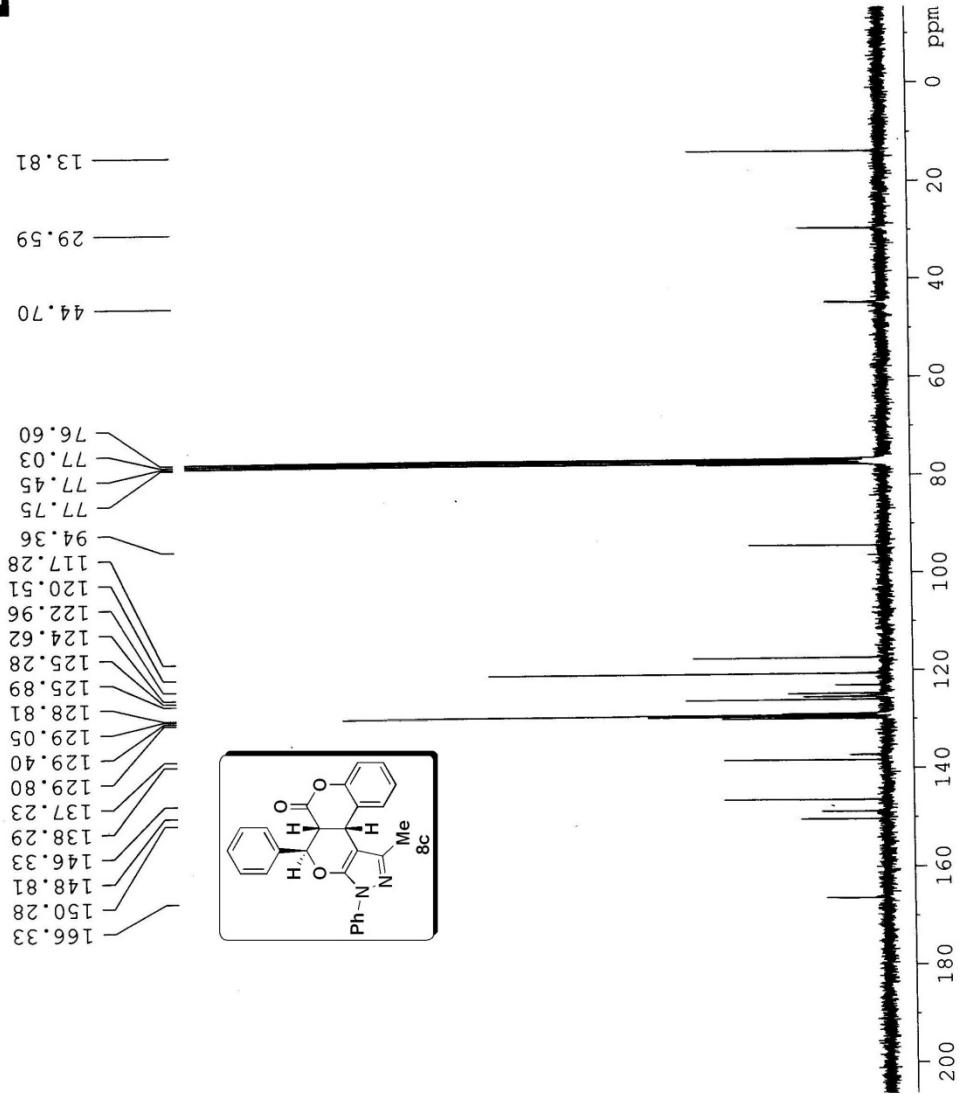
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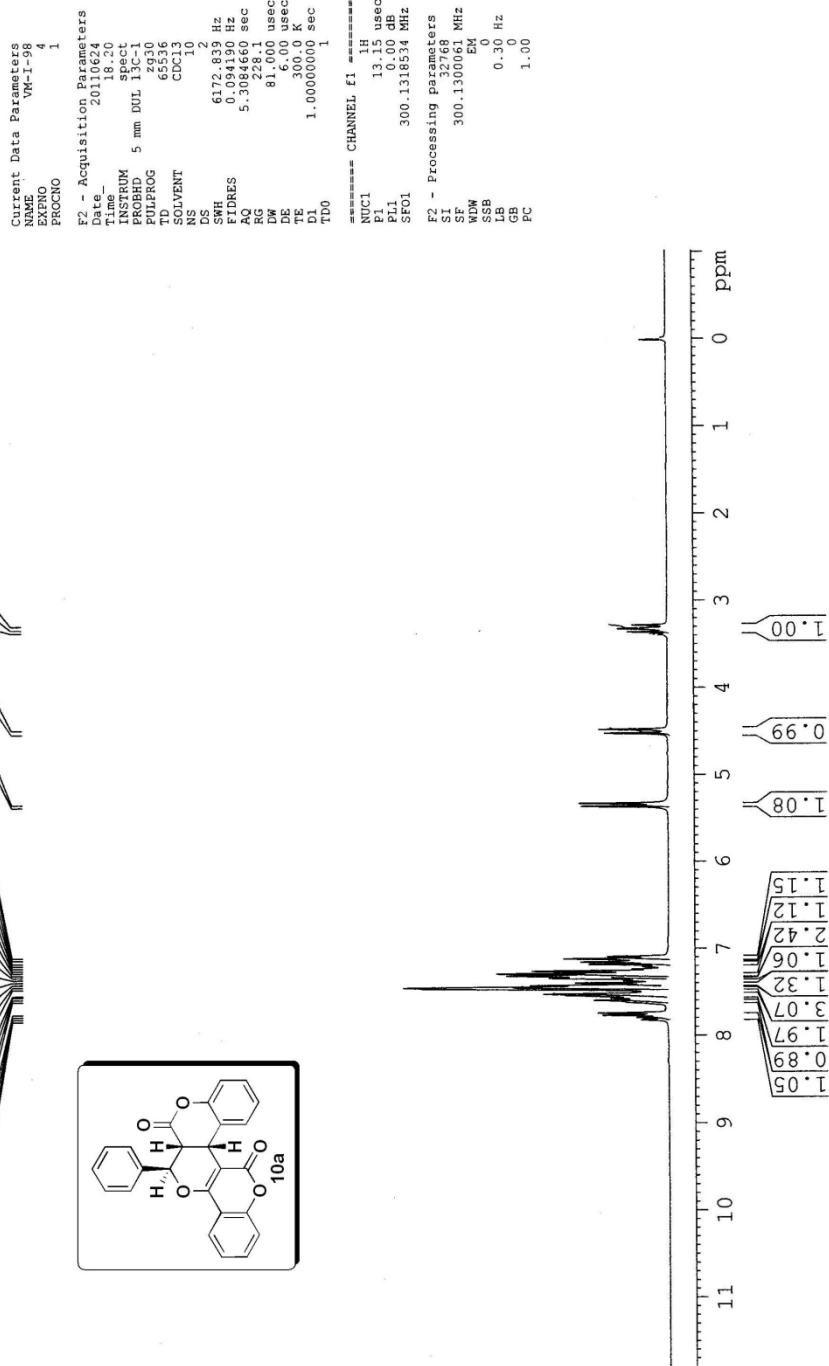
=====
 CHANNEL f1 =====
 NUC1          13C    9.30  usec
 P1            0.00  dB
 P1L           0.00  dB
 SFO1          75.4752953 MHz

=====
 CHANNEL f2 =====
 CDPDR22      Walz16
 NUC2          1H     80.00  usec
 PCBD2         0.00  dB
 P12           15.68  dB
 P112          16.00  dB
 P113          300.1312005 MHz

F2 - Processing parameters
SI             37768
SF             75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB           0
PC           1.40

```







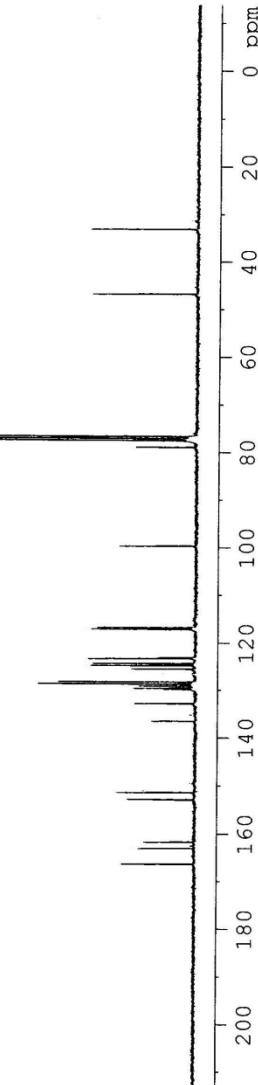
Current Data Parameters
NAME VR-I-98
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

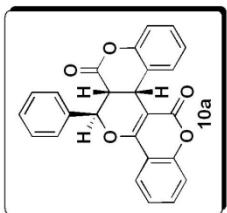
Date 20110630
Time 16:00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpp30
TD 65336
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.2274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.000 usec
DE 6.000 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0300000 sec
DETA 1.8999998 sec
TDO 1.

===== CHANNEL f1 =====

NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SF01 75.475253 MHz
===== CHANNEL f2 =====
CDPPRG2
NUC2 1H
RPBD2 80.00 usec
P22 0.00 dB
P12 15.68 dB
P13 16.00 dB
SF02 300.1312005 MHz

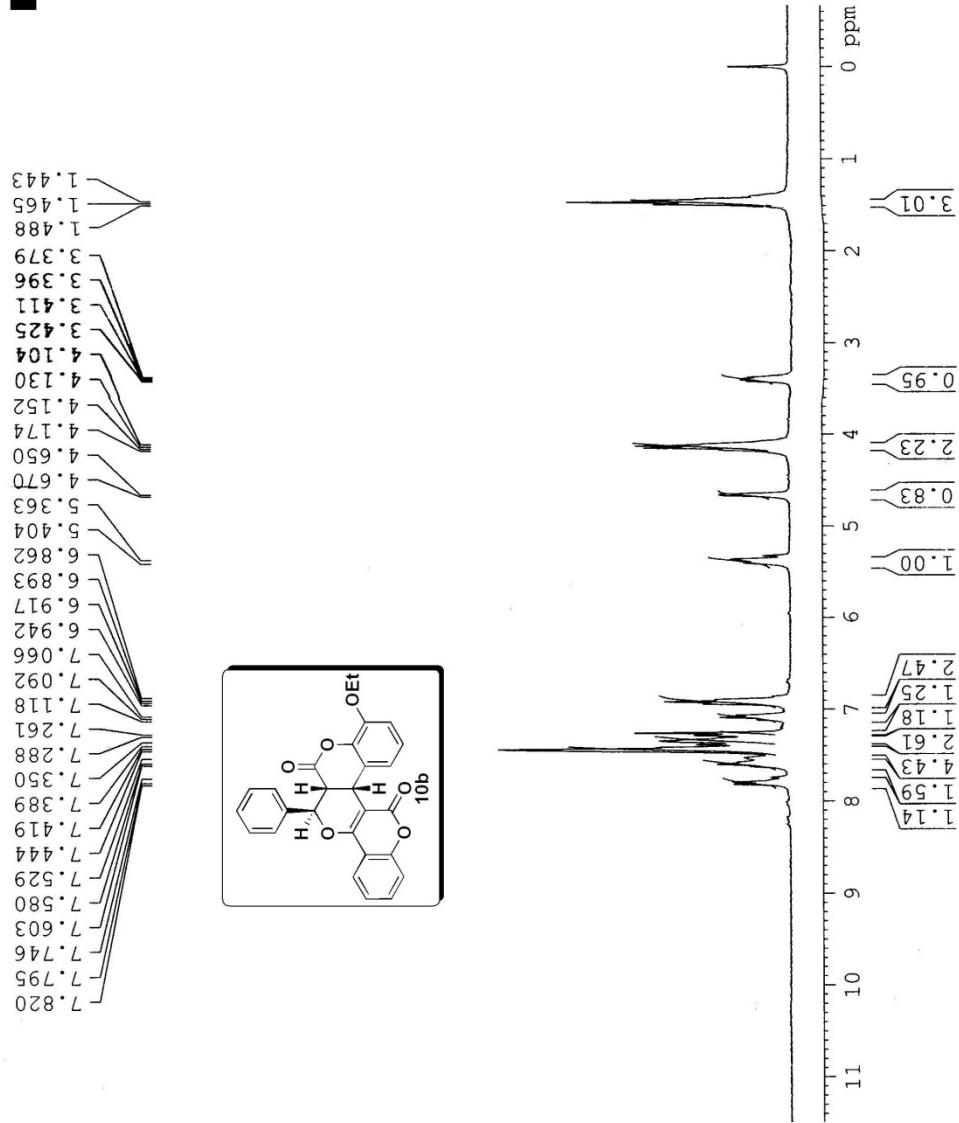


166.32
163.02
161.70
152.85
151.38
150.50
136.50
132.84
132.79
129.57
129.08
128.65
128.22
125.50
124.73
124.29
123.27
117.06
116.70
99.65
78.93
77.50
77.08
76.65



F2 - Processing Parameters

SI 32768
SP 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





Current Data Parameters
NAME VM-OET-COM
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date 20110627
Time 16.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.224439 Hz
AQ 1.822950 sec
RG 574.7
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.000000 sec
d11 0.0300000 sec
DETA 1.8999998 sec
TD0 1

===== CHANNEL f1 =====

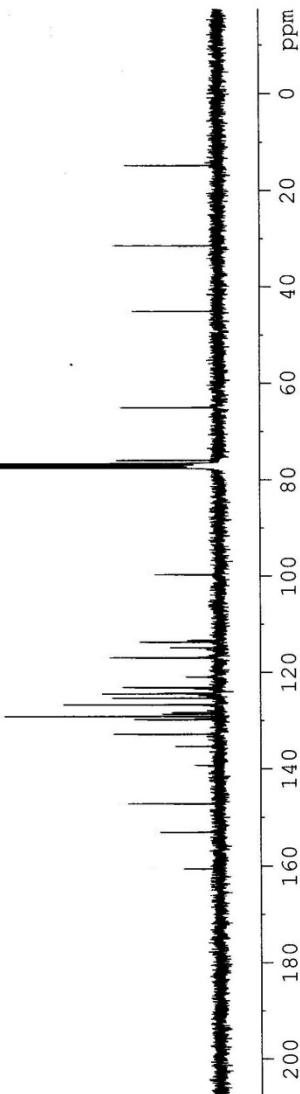
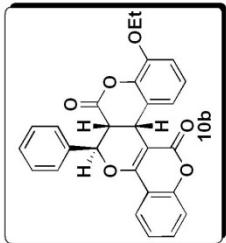
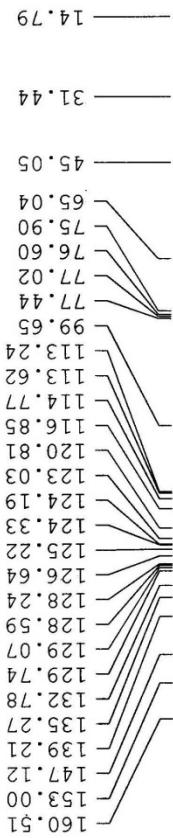
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.472953 MHz

===== CHANNEL f2 =====

CPDRG2 w11z16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SF02 300.1312005 MHz

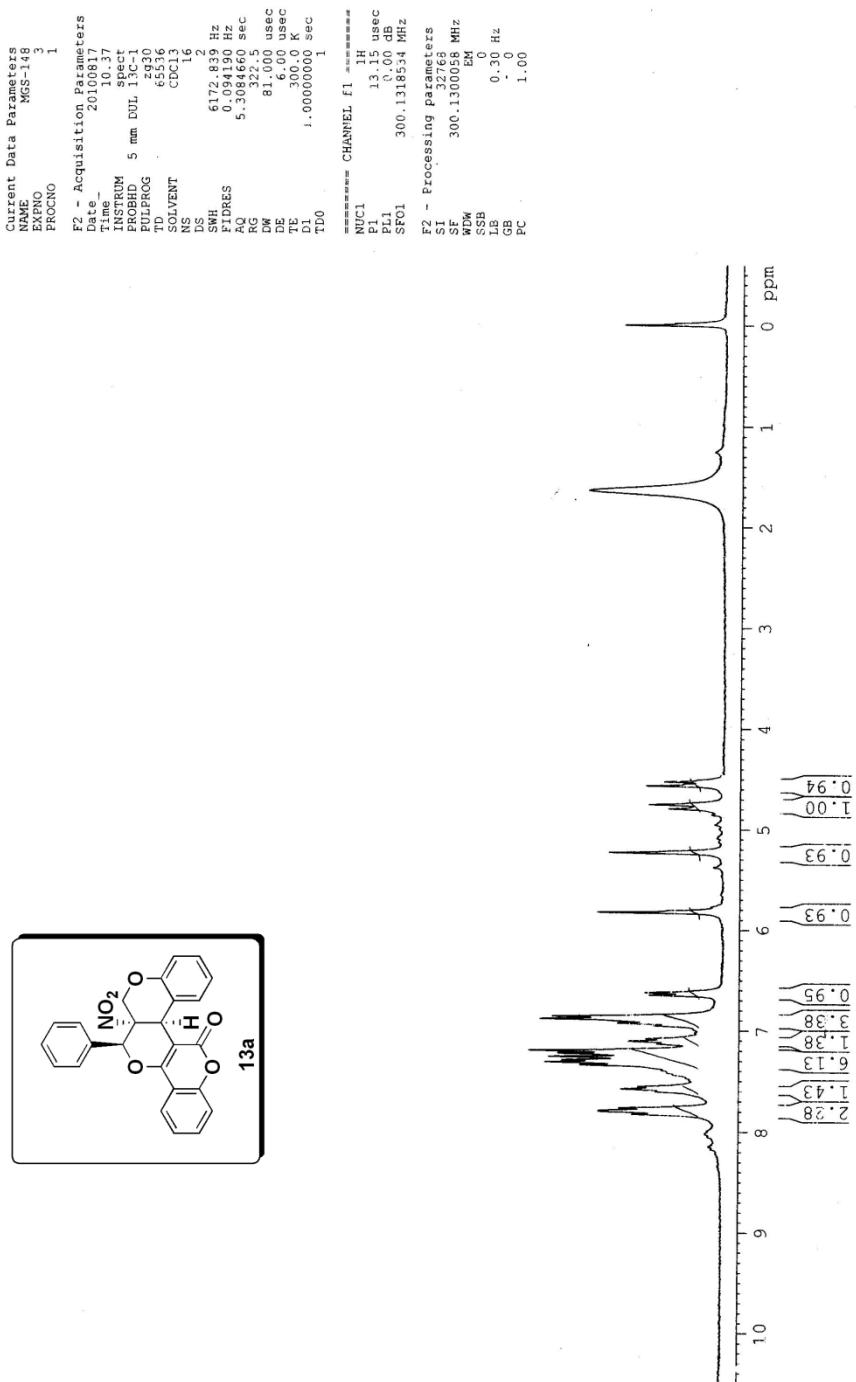
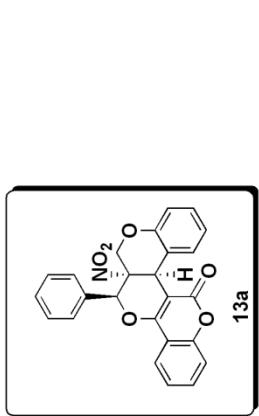
F2 - Processing Parameters

SI 3768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





4.527
4.569
4.755
4.758
4.798
5.236
5.829
6.026
6.652
6.865
6.890
6.931
6.956
7.083
7.109
7.133
7.206
7.231
7.266
7.297
7.320
7.346
7.556
7.582
7.604
7.769
7.798
7.831





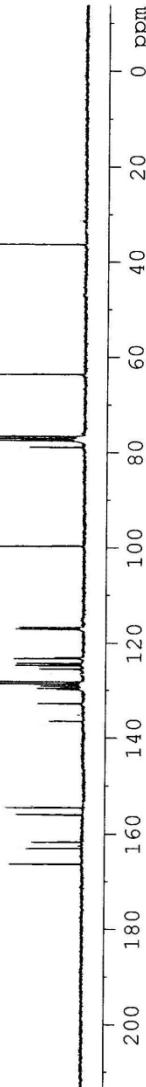
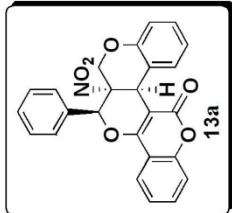
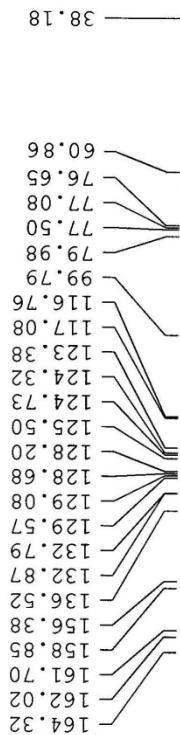
Current Data Parameters
NAME MGS-18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date 20100817
Time 16.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpp30
TD 65336
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.2274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.000 usec
DE 6.000 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.3000000 sec
DETA 1.8999998 sec
TDO 1

===== CHANNEL f1 =====

NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SF01 75.475253 MHz
===== CHANNEL f2 =====
CDPPRG2
NUC2 1H
RPBD2 80.00 usec
P22 0.00 dB
P12 15.68 dB
P13 16.00 dB
SF02 300.1312005 MHz

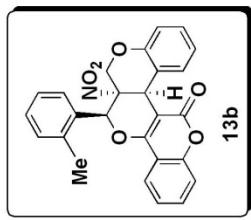




Bruker

— 2.424 —

4.685
4.726
4.833
4.894
4.894
5.322
5.333
6.496
6.523
6.894
6.920
6.943
7.025
7.048
7.102
7.133
7.210
7.262
7.344
7.371
7.506
7.556
7.581
7.749
7.775

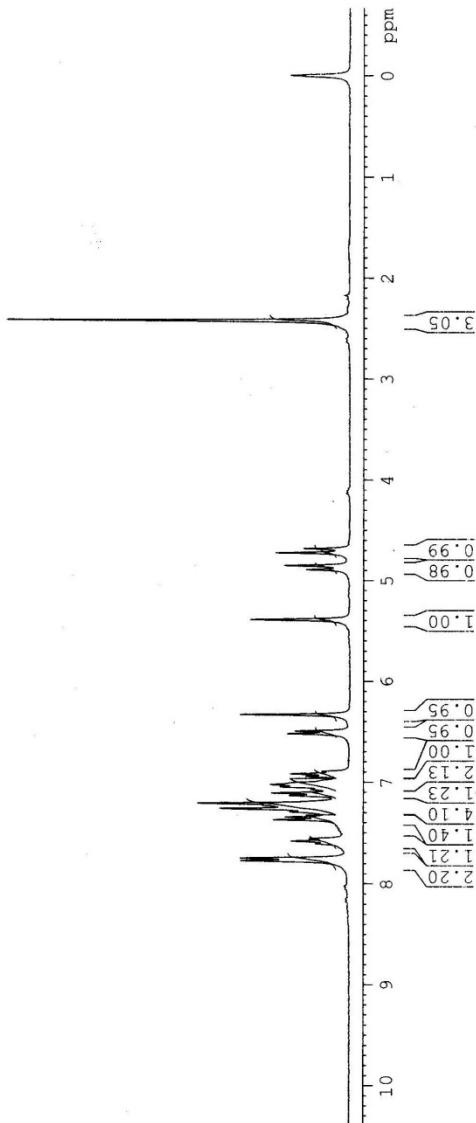


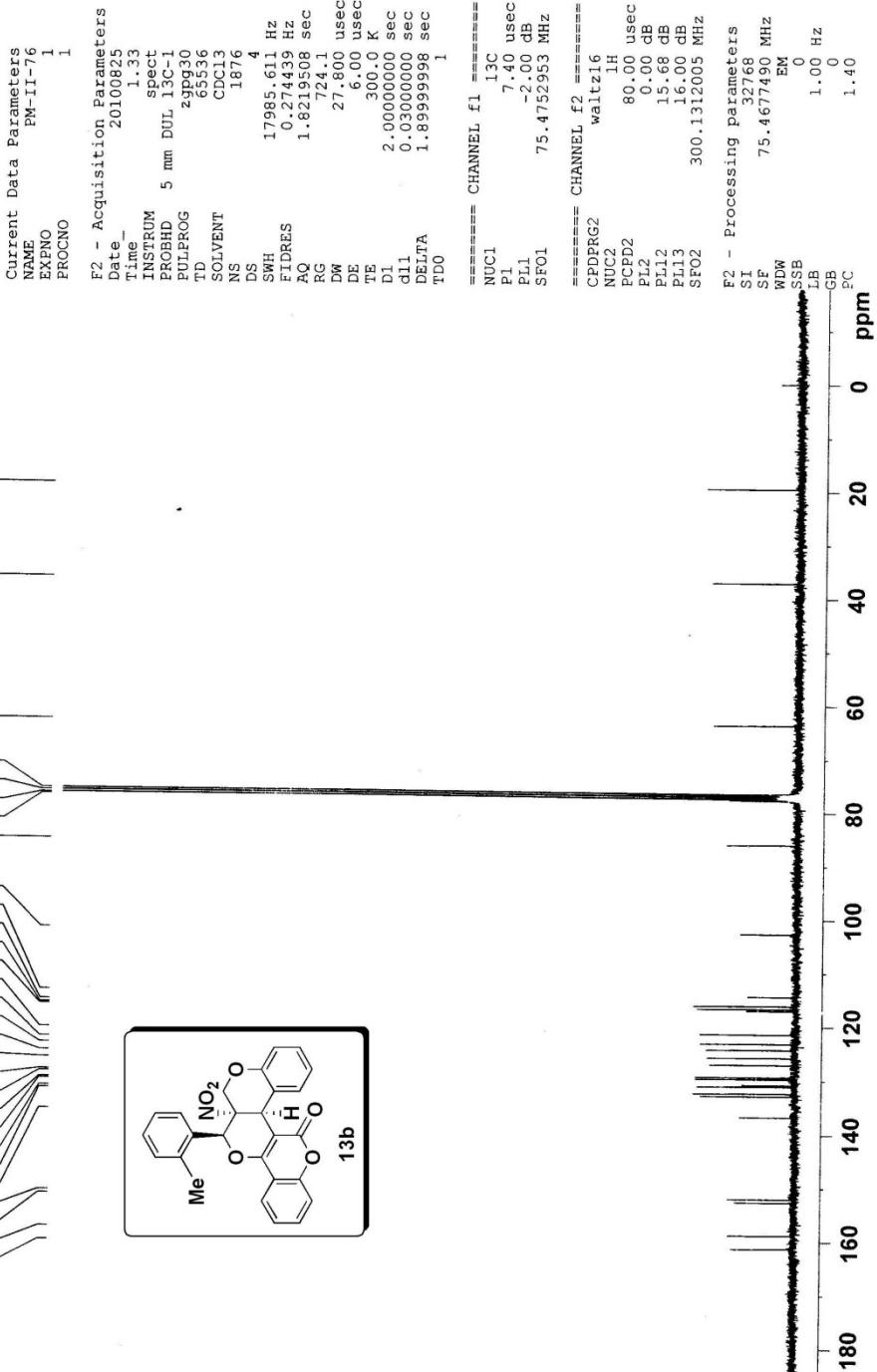
Current Data Parameters
NAME PH-II-72
EXPNO 2
PROCNO 1

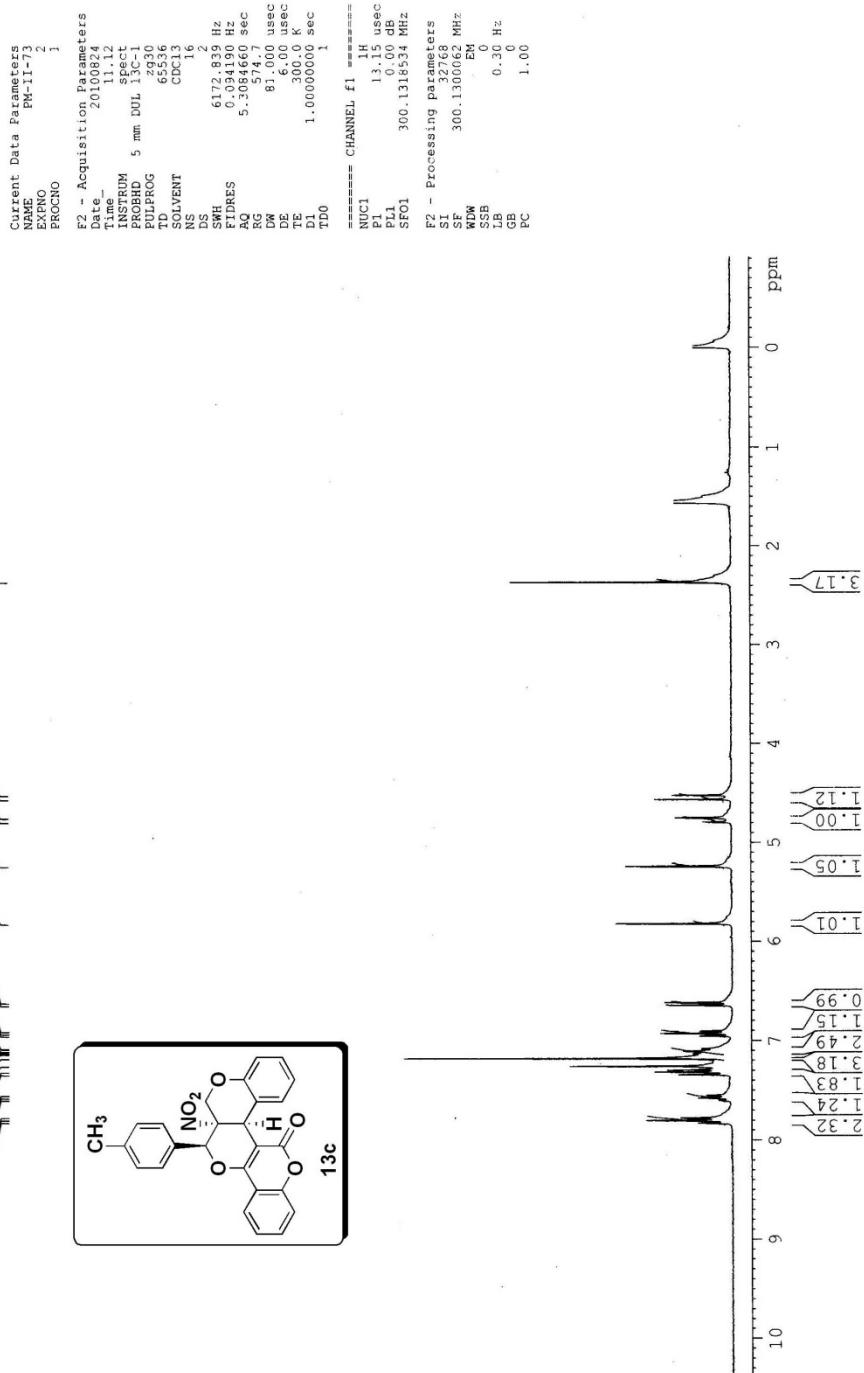
F2 - Acquisition Parameters
Date 20100814
Time 11:22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 6536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.304460 sec
RG 406.4
DW 81.000 usec
DE 6.000 usec
TE 300.0 K
D1 1.0000000 sec
TDO

===== CHANNEL f1 =====
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing Parameters
SI 32768
SF 300.1390062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00









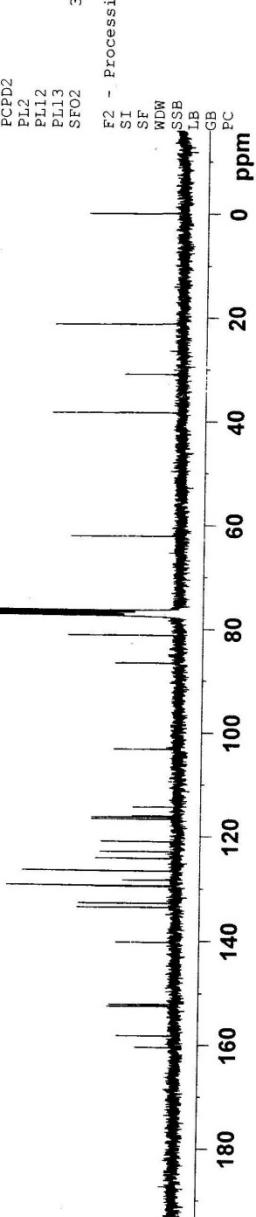
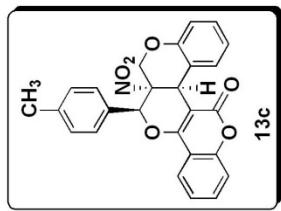
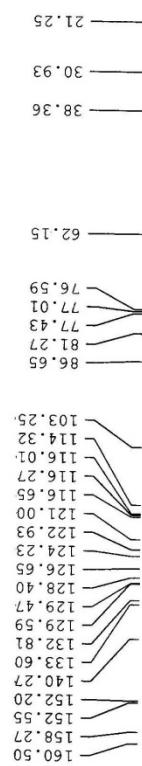
Current Data Parameters
NAME PM-II-73
EXPNO 3
PROCNO 1

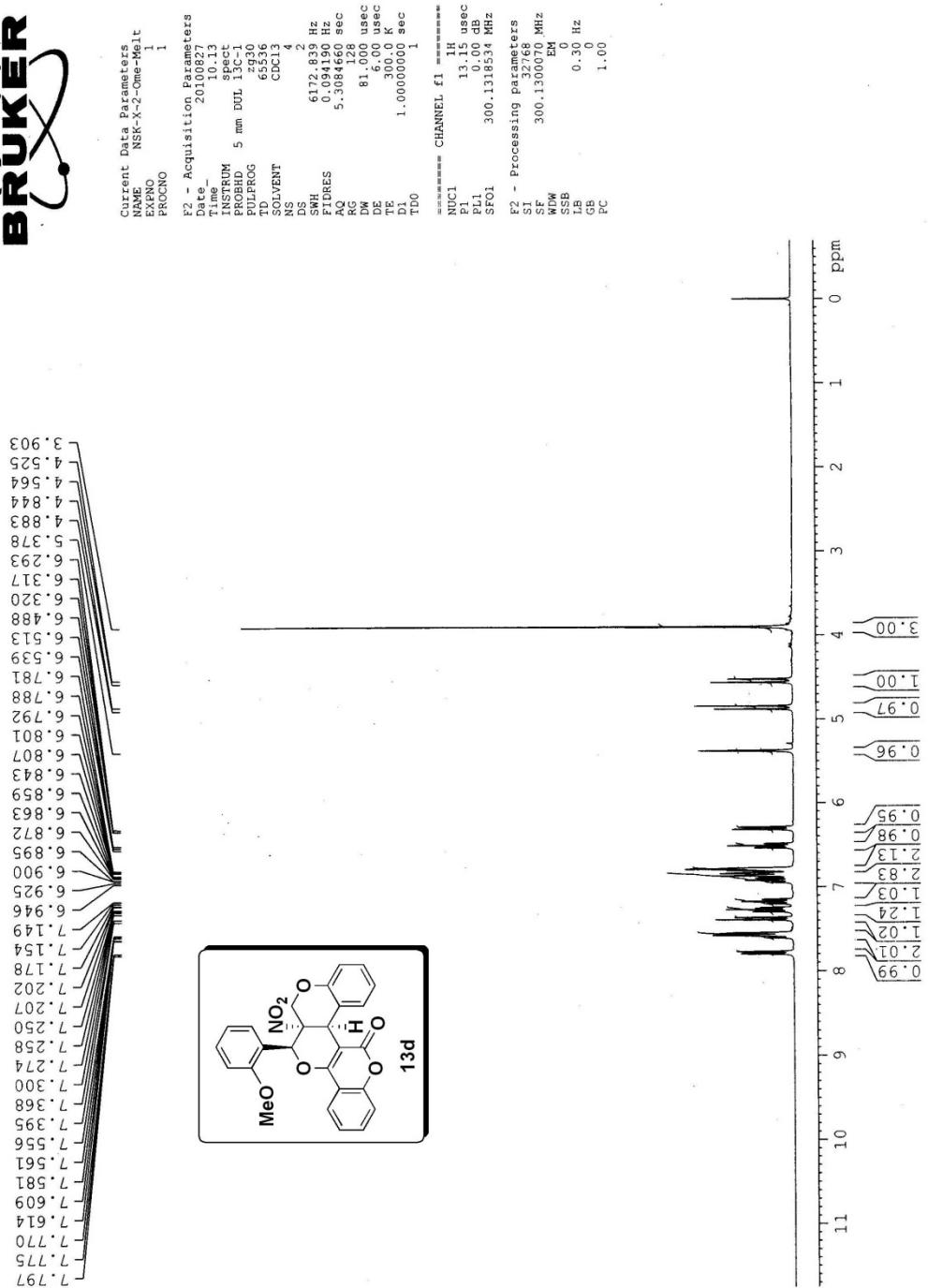
F2 - Acquisition Parameters
Date 20100825
Time 7:48
INSTRUM 5 mm DUL 13C-
PROBHD 29P930
TD 65536
SOLVENT CDCl3
NS 5488
DS 4
SWH 17985.611 Hz
ETDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0300000 sec
DETA 1.8939998 sec
TDDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SF01 75.4752953 MHz

===== CHANNEL f2 =====
CDPRG2 wait216
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SF02 300.1312005 MHz

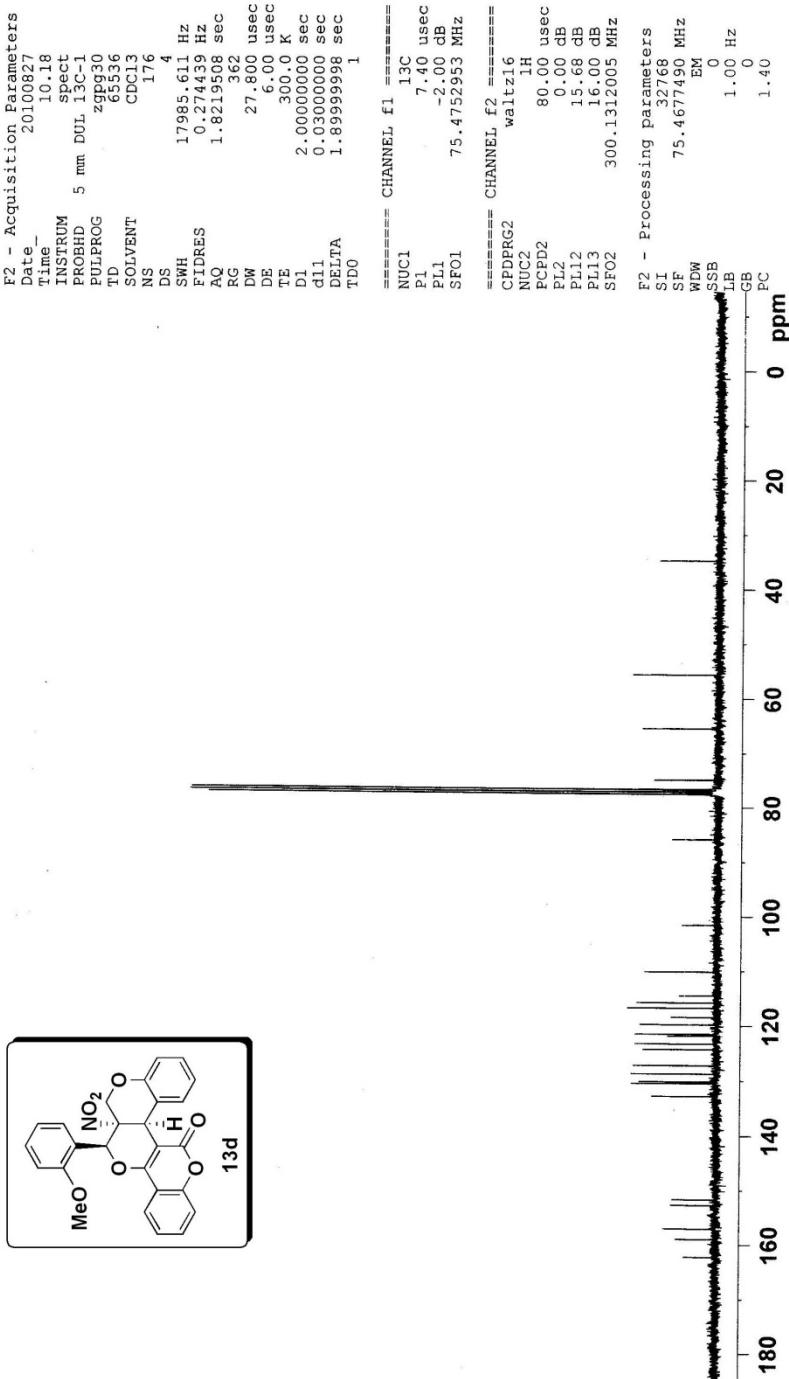
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW SSB
LB 0
EM 1.00 Hz
GB 0
PC 1.40







Current Data Parameters
NAME NSK-X-2-Ome-Melt
EXPNO 2
PROCNO 1



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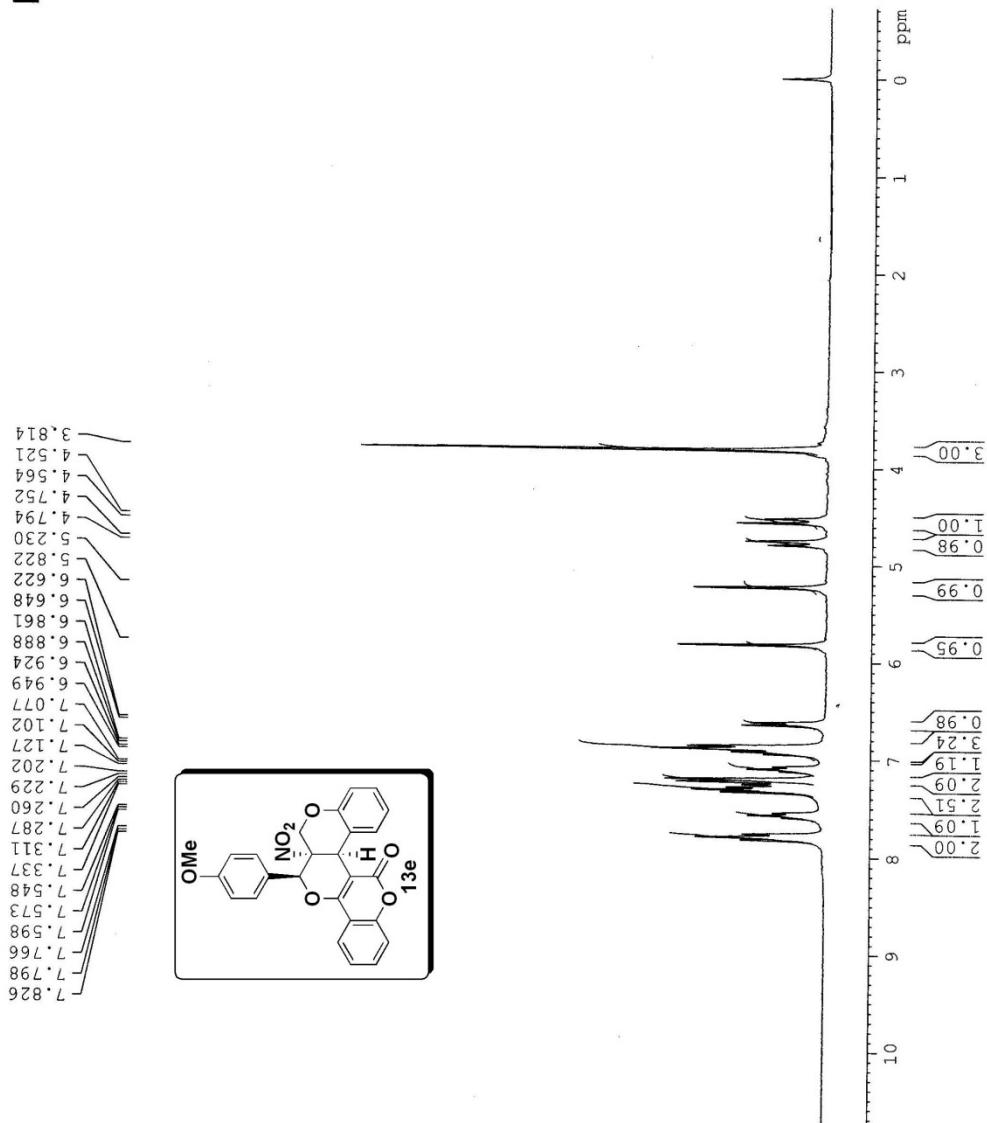
```

Current Data Parameters          F2 - Acquisition Parameters
NAME          EM-II-4ONE          Date      2010/09/19
EXPNO         2                  Time      14:21:19
INSTRUM       FIDRES           INSTRUM   5 mm DUL 13C-1
              AQ                 PULPROG  29.10
              SWB                SOLVENT  CDCl3
              NS                 2
              SWH                6172.839 Hz
              FIDRES            0.034660 sec
             AQ                  5.034660 sec
              RG                 14.57
              DW                 8.000 usec
              DE                 6.000 usec
              TE                 300.0 K
              T1                 1.0000000 sec
              T90                1

===== CHANNEL f1 =====
NUC1          P1                 13.15 usec
P1L          0.00 dB
SP01         300.13186334 MHz

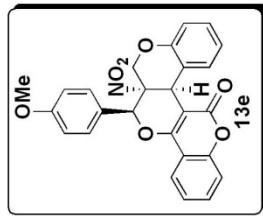
F2 - Processing parameters
SEI          300.13100000 MHz
WWD          0.30 Hz
SSB          0.00
LB          1.00
GB          0.00
GP          0.00

```





38.21
55.39
62.24
86.65
81.14
77.46
77.04
76.61
103.16
114.18
114.32
116.11
116.27
116.65
122.01
122.95
123.30
124.24
128.14
129.58
133.44
135.16
152.55
158.84
160.84



Current Data Parameters
NAME PM-II-4OME
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date 20100819
Time 16.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CNC13
NS 152
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8215508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.8999998 sec
TDOO 1

===== CHANNEL f1 =====

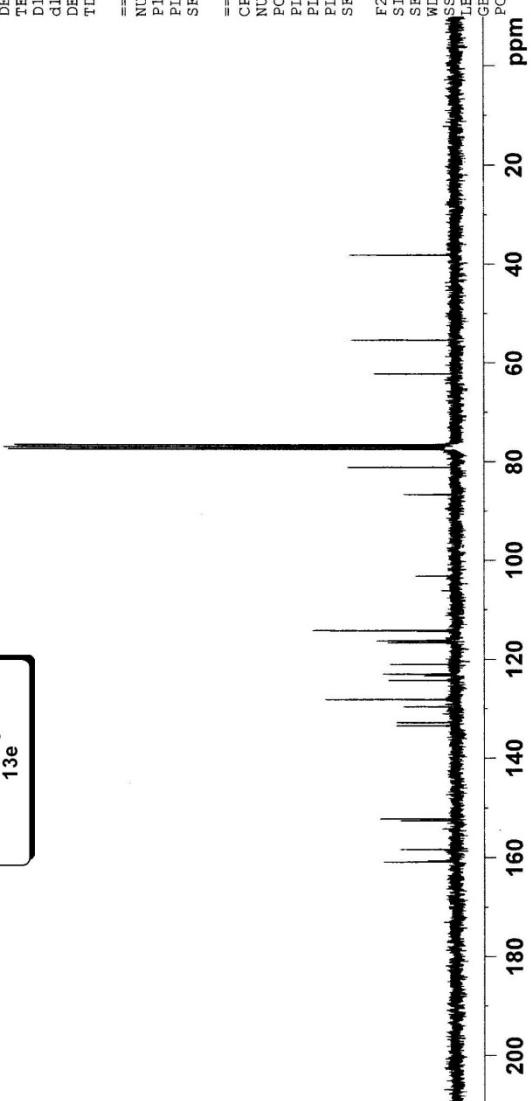
NUC1 13C
PL1 7.40 usec
PL1 2.00 dB
SP01 75.4752953 MHz

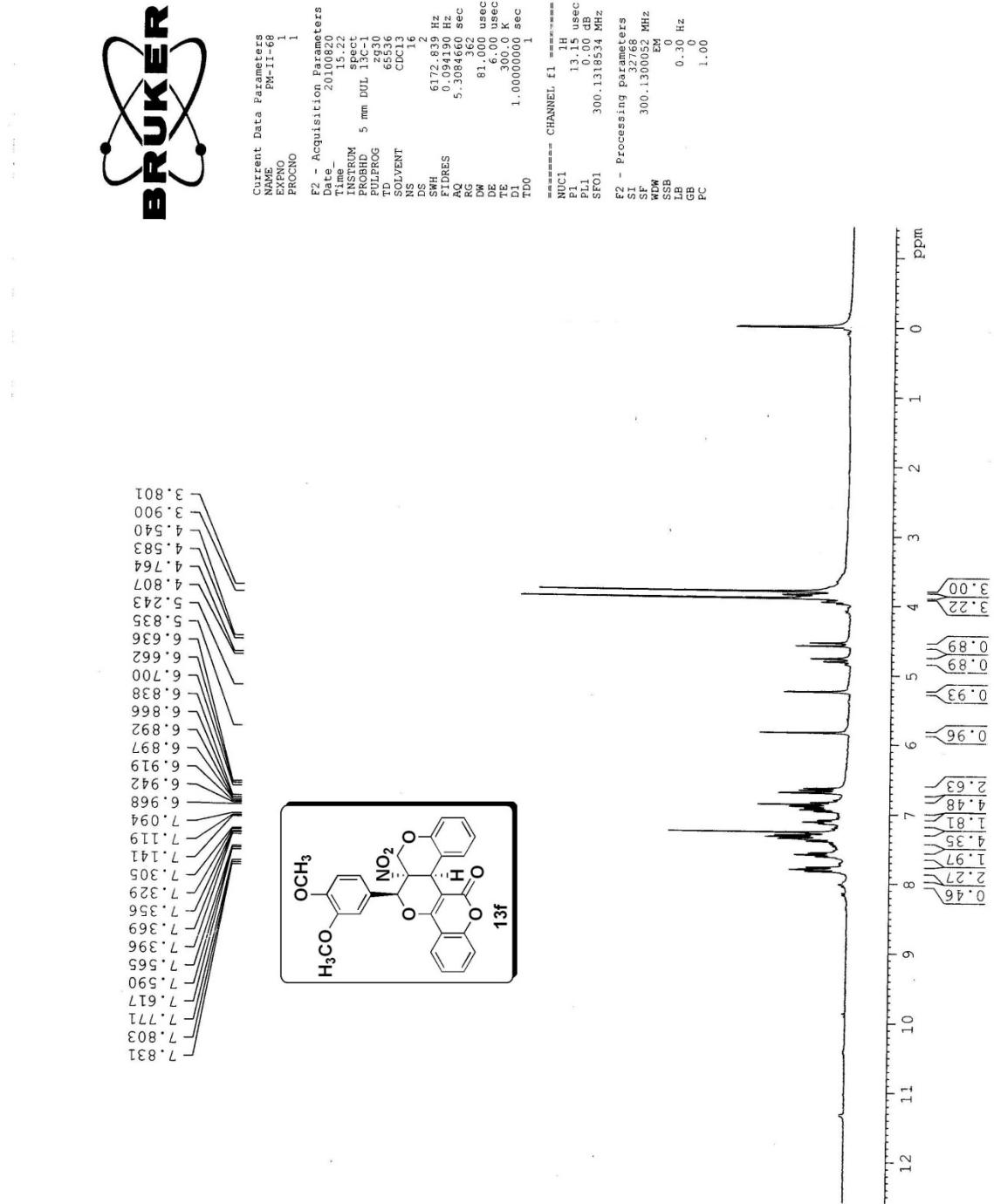
===== CHANNEL f2 =====

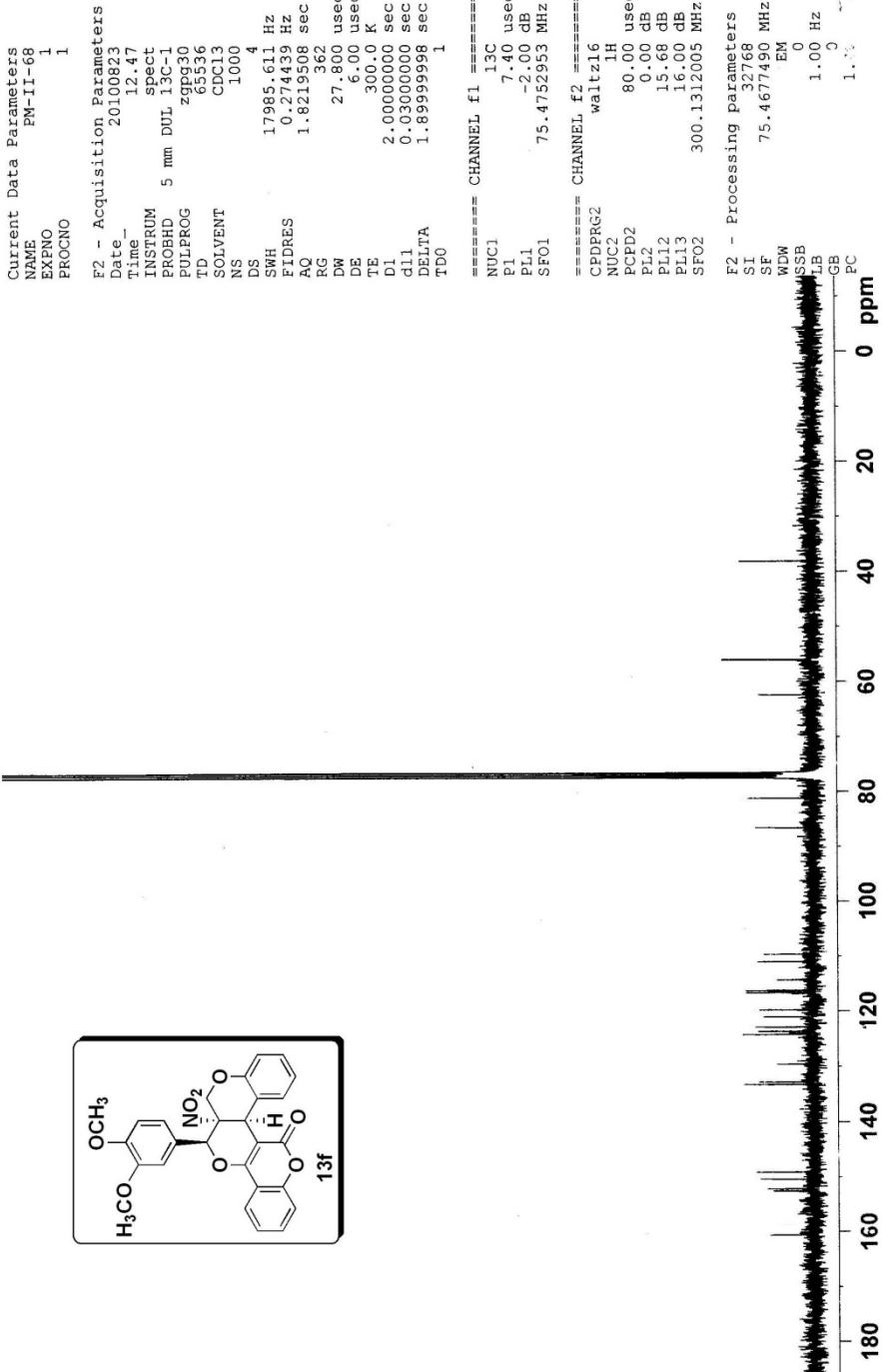
CDDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SP02 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677490 MHz
WID 0
SSB EM
LB 1.00 Hz
GB 0
PC 1.40



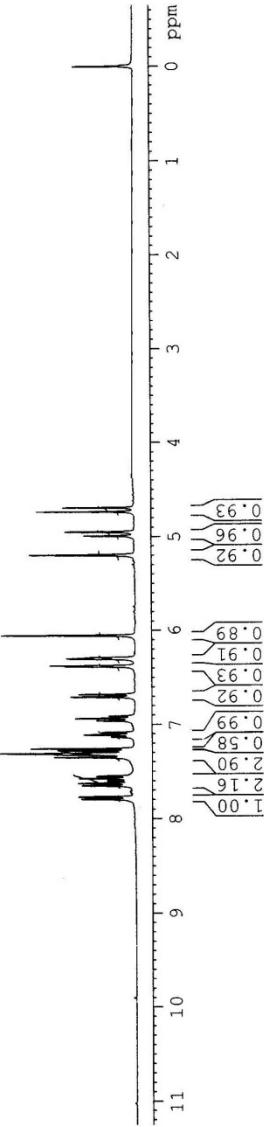






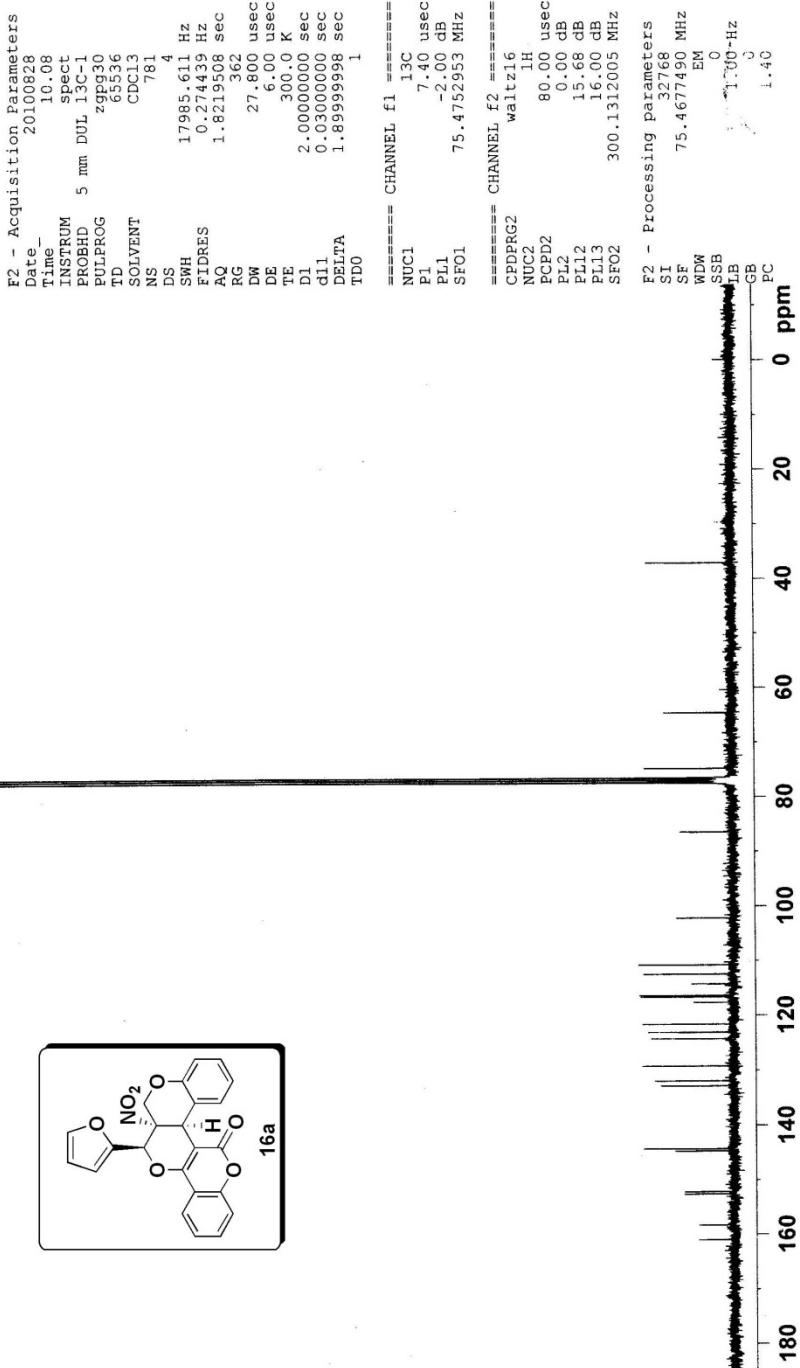
Current Data Parameters-Melt
NAME NSK-X-Furfury-Melt
EXNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20100827
Time 10.04
INSTRUM spect
PROBID 5 mm DUL 13C-1
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 6172.839 Hz
ETRINES 0.004180 Hz
AQ 5.304460 sec
RG 228.1
DW 81.000 usec
DE 6.000 usec
TE 300.0 K
D1 1.000000 sec
TDO 1.000000 sec



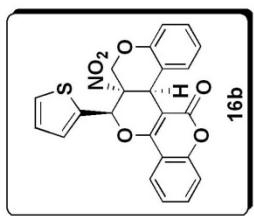


Current Data Parameters
NAME NSK-X-Furyl-mel
EXPNO 1
PROCNO 1





7.840
7.812
7.779
7.609
7.584
7.559
7.457
7.450
7.333
7.312
7.262
7.170
7.145
7.039
6.993
6.969
6.944
6.769
6.742
6.149
5.161
4.968
4.925
4.586
4.542



```

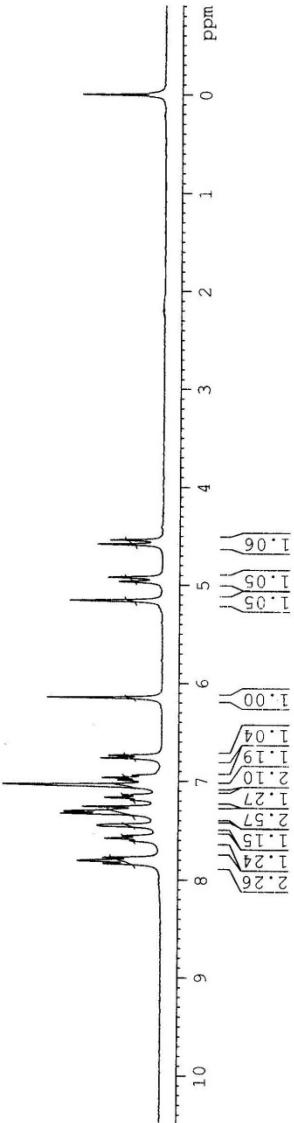
Current Data Parameters
NAME      PN-II-5
EXPTNO    1
PROCNO   1

F2 - Acquisition Parameters
Date        20100824
Time       16.28
INSTRUM   spect
PROBID    5 mm DUL 13C-1
PULPROG  zg30
TD        65536
SOLVENT   CDCl3
NS         13
DS         2
SWH       6177.839 Hz
FIDRES   0.004190 Hz
AQ        5.308460 sec
RG        456.1
DW        81.000 usec
DE        6.000 usec
TE        300.0 K
D1        1.000000 sec
TDCO

===== CHANNEL f1 =====
NUC1      1H
P1        13.15 usec
PL1      0.0 df
SF01    300.1318334 MHz

F2 - Processing Parameters
SI        32768
SF        300.1300058 MHz
WDW
SSB
LB        0.30 Hz
GB        0
PC        1.00

```





Current Data Parameters

NAME PM-II-75

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date 20100824

Time 23.09

INSTRUM spect

BPROBID 5 mm DUL 13C-1

PULPROG zppg30

TD 65536

SOLVENT CDCl3

NS 1253

DS 4

SWH 17985.61 Hz

ETDRES 0.274439 Hz

AQ 1.8219508 sec

RG 724.1

DE 27.800 usec

TE 6.00 usec

D1 300.0 K

c11 2.0000000 sec

DELTA 0.0300000 sec

TDDA 1.8999998 sec

TDO 1

===== CHANNEL f1 =====

NUC1 13C

PL 7.40 usec

PL1 -2.00 dB

SFO1 75.472953 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 80.00 usec

PL2 0.00 dB

PL12 15.68 dB

PL13 16.00 dB

SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768

SF 75.4677490 MHz

WDW EM

SSB Hz

LB

GB

FC

1.40



Peak labels (ppm): 160.02, 157.99, 152.55, 152.51, 133.55, 132.91, 132.65, 128.15, 127.62, 124.31, 122.98, 121.18, 116.63, 116.52, 116.08, 114.09, 103.44, 87.39, 62.52, 78.45, 77.44, 77.02, 76.60.