Supporting Information

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

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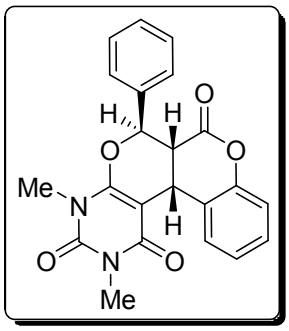
Contents **page**

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 ^{1}H NMR (300 MHz, $CDCl_3$) and ^{13}C NMR (75 MHz, 100 MHz, $CDCl_3$) spectra were recorded in deuterochloroform ($CDCl_3$) 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.

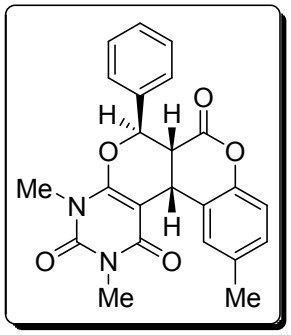
Representative procedure for the synthesis of tetracyclic coumarinopyranpyrimidinediones: A mixture of (*E*)-2-formylphenyl cinnamate (**2a**, 0.252g, 1mmol) and *N*,*N*-dimethylbarbituric acid (**3a**, 0.156g, 1mmol) was placed in a round bottom flask and melted at 180 ºC for 1 h. After completion of 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):



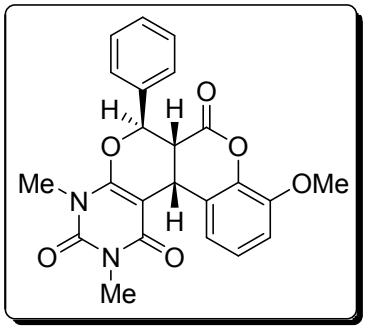
Yield: 98%; Colourless solid; mp:238-240 ˚C;^{1}H NMR (300 MHz,$CDCl_3$): δ 3.32 - 3.33 (m, 1H), 3.37 (s, 3H), 3.44 (s, 3H), 4.60 (d, *J*=4.8Hz, 1H), 5.30 (d, *J* = 9.6Hz,1H), 7.07-7.46 (m, 9H); ^{13}C NMR (75 MHz): δ 28.3, 28.9, 30.5, 45.4, 77.8, 86.1, 116.9, 123.5, 125.4, 126.4, 128.0,129.1, 129.3, 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 $C_{22}H_{18}N_2O_5$: 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):



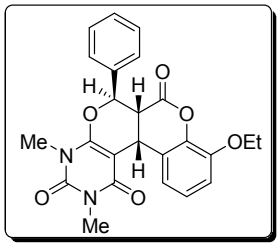
Yield: 96%; mp: 249-251˚C;^{1}H NMR (300 MHz,$CDCl_3$): δ 2.33 (s, 3H), 3.26 (dd, *J* =4.8,9.9 Hz, 1H), 3.37 (s, 3H), 3.47 (s, 3H), 4.59 (d, *J*=4.2 Hz, 1H), 5.25 (d, *J*=9.9 Hz, 1H), 6.98-7.44 (m, 8H); ^{13}C NMR (75 MHz): δ 21.0, 28.4, 28.9, 30.5, 45.6, 77.8, 86.1, 116.7, 123.4, 126.5, 129.1, 129.8, 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 $C_{23}H_{20}N_2O_5$: 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):



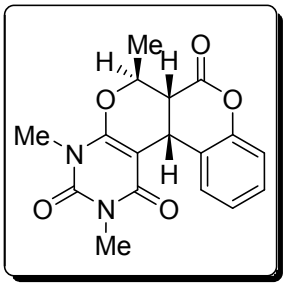
Yield: 93%; mp: 250-252˚C;^{1}H NMR (300 MHz,$CDCl_3$): δ 3.29-3.33 (m, 1H), 3.38 (s, 3H), 3.43 (s, 3H), 3.91 (s, 3H), 4.59 (d,*J*= 4.8 Hz, 1H), 5.35 (d, *J*=9.6Hz, 1H), 6.85-7.45 (m, 8H); ^{13}C NMR (75 MHz): δ 28.3, 28.9, 30.6, 45.1, 56.2, 77.7, 86.1, 111.9, 121.0, 124.6, 125.1, 126.4, 128.0, 128.6, 129.1, 129.9, 134.5, 138.6, 147.6, 150.7, 156. 2, 162.7, 163.9; IR (KBr): 1748, 1710, 1644cm^{-1}; MS (m/z): 421 (M^++1); Elemental Analysis for $C_{23}H_{20}N_2O_6$: 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)



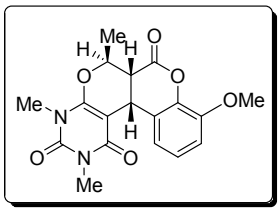
Yield: 91%; mp: 252-254 ˚C;^{1}H NMR (300 MHz,$CDCl_3$): δ 1.46 (t,*J*=6.9Hz, 3H), 3.27-3.32 (m, 1H),3.37(s, 3H), 3.43 (s, 3H), 4.13 (q, *J*=6.3Hz,2H), 4.57 (d, *J*=4.5Hz, 1H), 5.36 (d, *J*=9.3 Hz 1H); 6.82-7.44 (m, 8H); ^{13}C NMR (75 MHz): δ 14.7, 28.3, 28.9, 30.6, 45.1, 65.0, 77.7, 86.1, 113.4, 121.0, 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, 1637cm^{-1}; MS (m/z): 435 (M^{+}+1); Elemental Analysis for $C_{24}H_{22}N_2O_6$: 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):



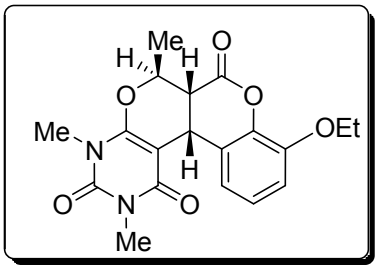
Yield: 92%; Colourless solid; mp: 244 – 246 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.57 (d,*J*=6.3Hz, 3H), 2.96 (dd, *J* =5.1,9.9Hz, 1H), 3.36 (s, 3H), 3.43 (s, 3H), 4.37-4.43 (m, 1H), 4.58 (d,*J*=4.8Hz, 1H), 7.03-7.31 (m, 4H); ^{13}C NMR (75 MHz): δ 18.8, 28.3, 28.7, 30.0, 44.7, 72.2, 85.7, 116.9, 123.7, 125.4, 129.0, 129.5, 149.1, 150.8, 156.0, 162.9, 165.6; IR (neat): *v*1785, 1576, 1530 cm^{-1}; MS (m/z): 329 (M^{+}+1); Elemental Analysis for $C_{17}H_{16}N_2O_5$; 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):



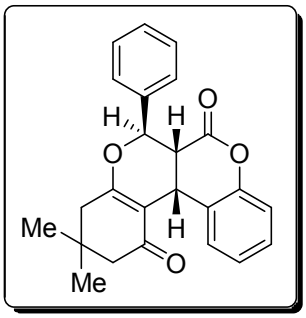
Yield: 90%; Colourless solid; mp: 228 – 230 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.59 (d,*J*=6.3Hz, 3H), 2.97 (dd, *J* =4.8, 10.2Hz, 1H), 3.37 (s, 3H), 3.44 (s,3H), 3.90 (s, 3H), 4.38-4.48 (m, 1H), 4.59 (d, *J*=4.8 Hz, 1H), 6.74-7.12 (m, 3H); ^{13}C NMR (75 MHz): δ 28.3, 28.7, 30.2, 44.5, 56.1, 72.2, 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 $C_{18}H_{18}N_2O_6$: 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; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.46 (t, *J* = 6.9Hz, 3H), 1.58 (d, *J* = 6 Hz, 3H), 2.94 (dd, *J* = 4.8, 9.9 Hz, 1H), 3.37 (s, 3H), 3.42 (s, 3H), 4.10 (q, *J*=6.9 Hz, 2H), 4.38-4.47 (m, 1H), 4.57 (d, *J*=4.5 Hz, 1H), 6.72 (d, *J*=7.8Hz, 1H), 6.87 (d,*J*=8.4Hz, 1H), 7.05 (t,*J*=7.8Hz, 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 $C_{19}H_{20}N_2O_6$: Calculated: C 61.28; H, 5.41; N, 7.52; Found: C 61.19; H, 5.30; N, 7.63.

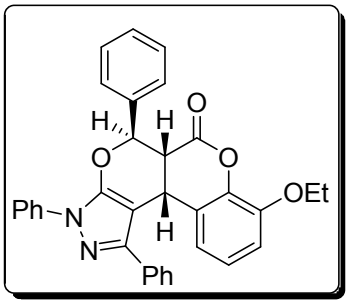
Methyl 4,6-dimethyl-12-[(4-methylbenzene)sulfonyl]-9-(3,4-dimethoxyphenyl)-3,5-dioxo-8-oxa-4,6,12-triazatetracyclo[8.8.0.0^{2,7}.0^{13,18}]octadeca-2(7),13,15,17-tetraene-10-carboxylate (5h):



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

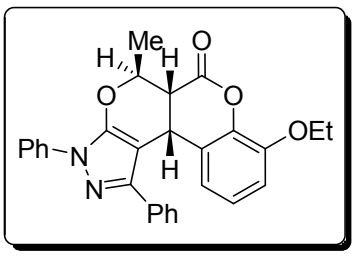
4.52 (d, *J*=3.9Hz, 1H), 5.04 (t, *J*= 9.9 Hz, 1H); 6.97-7.41 (m, 9H); ^{13}C NMR (75 MHz): δ 28.4, 29.6, 32.3, 42.3, 45.5, 50.7, 75.5, 109.4, 116.9, 124.5, 125.2, 125.9, 126.6, 128.0, 128.5, 128.8, 129.5, 135.6, 149.5, 165.2, 170.5, 197.2; IR (neat): ν 1743, 1598, 1529 cm^{-1}; MS (m/z): 375 (M^++1).

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; ^{1}H 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}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 $C_{33}H_{26}N_2O_4$: Calculated: C 77.03; H, 5.09; N, 5.44; Found: C 76.98; H, 5.00; N, 5.35.

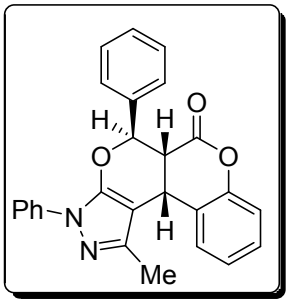
6-ethoxy-11-methyl-14,16-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-9-one (8b):



Yield: 90%; Colourless solid; mp: 226 – 228 ˚C; ^{1}H 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}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,

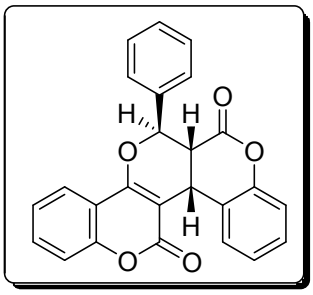
1544cm^{-1}; MS (m/z): 454 (M^++1); Elemental Analysis for $C_{28}H_{24}N_2O_4$: Calculated: C 74.32; H, 5.35; N, 6.19; Found: C 74.24; H, 5.26; N, 6.28.

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):



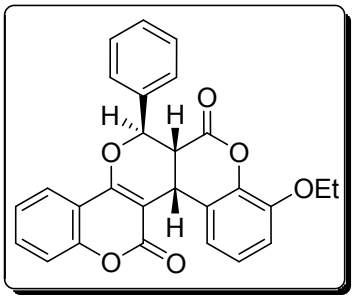
Yield: 92%; Colourless solid; mp: 233 – 235 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.64 (s, 1H), 1.98 (bs, 3H), 3.37 (s, 1H), 4.03 (bs, 1H), 7.10-7.80 (m, 14H); ^{13}CNMR ($CDCl_3$, 75 MHz): δ 13.8, 29.5, 44.7, 77.7, 94.3, 117.2, 120.5, 122.9, 124.6, 125.2, 125.8, 128.8, 129.0, 129.4, 129.8, 137.2, 138.2, 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; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.33 (t, *J* = 11.1Hz, 1H), 4.50, (d, *J*=13.2Hz, 1H), 5.35 (d, *J*=10.2Hz, 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).

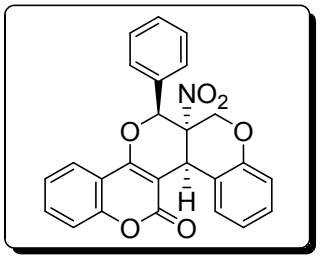
7-ethoxy-12-phenyl-9,13,21-trioxapentacyclo[12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaene-10,22-dione (10b):



Yield: 92%; Colourless solid; mp: 222 – 224 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.46 (t,*J*=6.9Hz, 3H), 3.40 (dd, *J* = 4.2, 8.7Hz, 1H), 4.14 (q, *J*=6.6Hz, 2H), 4.66 (d, *J*=6.0Hz, 1H),5.38 (d, *J*=12.3Hz,1H), 6.86-7.82(m, 12H); ^{13}C NMR (75 MHz): δ14.7, 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, 1589,1523cm^{-1}; MS (m/z): 441 (M^++1).

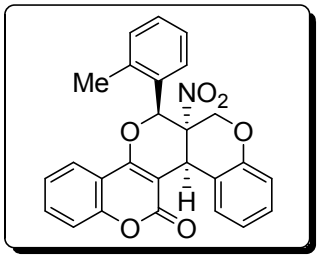
Representative procedure for synthesis of pentacyclic coumarinopyrancoumarins: A mixture of *O*-alkylated product (**11a**, 0.36g, 0.841 mmol**)** and 4-hydroxy-2*H*-chromen-2-one (**6b**, 0.12g, 0.841mmol) was placed in a round bottom flask and melted at 180 ºC for 1 h. After completion of the reaction 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)



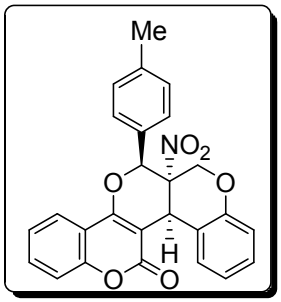
Yield: 90%; Colourless solid; mp: 228-230˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.54 (d, *J* = 12.6Hz, 1H), 4.77 (d, *J* = 12.6Hz, 1H), 5.24 (s, 1H), 5.83 (s, 1H), 6.63-7.83 (m, 13H); ^{13}C NMR (75 MHz): δ 38.1, 60.8, 79.9, 99.7, 116.7, 117.0, 123.3, 124.3, 124.7, 125.5, 128.2, 128.6, 129.0, 132.7, 132.8, 136.5, 156.3, 158.8, 161.7, 162.0, 164.3; IR (neat): ν 1692, 1580, 1538cm^{-1}; MS (m/z): 442 (M^++1); Elemental Analysis for $C_{26}H_{19}NO_6$: 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)



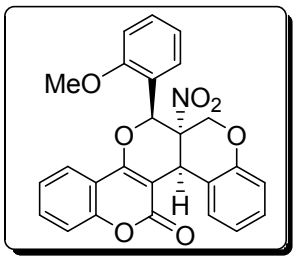
Yield: 92%; Colourless solid; mp: 239-241˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.42 (s, 3H), 4.70 (d, *J* = 12.3 Hz, 1H), 4.87 (d, *J* = 12.3 Hz, 1H), 5.39 (s, 1H), 6.33 (s, 1H), 6.49-7.77 (m, 12H); ^{13}C NMR (75 MHz): δ 19.5, 37.1, 63.7, 77.7, 86.0, 102.6, 114.3, 116.0, 116.6, 116.9, 121.3, 123.1, 124.2, 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 $C_{26}H_{19}NO_6$: 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):



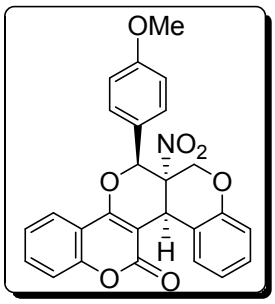
Yield: 90%; Colourless solid; mp: 241 -243 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.37 (s, 3H), 4.54 (d, *J* = 12.9 Hz, 1H), 4.78 (d, *J* = 12.9 Hz, 1H), 5.24 (s, 1H), 5.82(s, 1H), 6.62-7.83 (m, 12H); ^{13}C NMR (75 MHz): δ 21.2, 30.9, 38.3, 62.1, 81.2, 86.6, 103.2, 114.3, 116.0, 116.2, 116.6, 121.0, 122.9, 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 $C_{26}H_{19}NO_6$: 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):



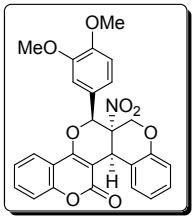
Yield: 89%; Colourless solid; mp: 236-238 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.90 (s, 3H), 4.54 (d, *J* = 11.7 Hz, 1H), 4.86 (d, *J* = 11.7 Hz, 1H), 5.38 (s, 1H), 6.29-7.80 (m, 12H); ^{13}C NMR (75 MHz): δ 34.7, 55.5, 65.4, 74.8, 85.8, 101.5, 110.0, 114.4, 115.7, 116.7, 118.3, 119.7, 121.4, 121.7, 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 $C_{26}H_{19}NO_7$: 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)



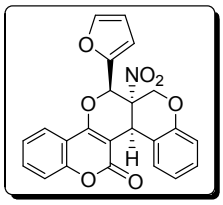
Yield: 90%; Colourless solid; mp: 245 -247˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.81 (s, 3H), 4.54 (d, *J* = 12.9 Hz, 1H), 4.77 (d, *J* =12.6 Hz, 1H), 5.23 (s, 1H), 5.82 (s, 1H), 6.62-7.83 (m, 12H); ^{13}C NMR (75 MHz): δ 38.2, 55.3, 62.2, 81.1, 86.6, 103.1, 114.1, 114.3, 116.1, 116.2, 116.6, 121.0, 122.9, 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 $C_{26}H_{19}NO_7$: 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)



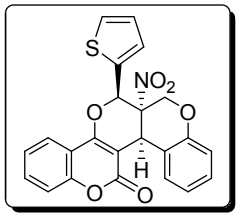
Yield: 92%; Colourless solid; mp: 189-191 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.80 (s, 3H), 3.90(s, 3H), 4.56 (d, *J* = 12.6Hz, 1H), 4.78 (d, *J* = 15Hz, 1H), 5.24 (s, 1H), 5.83 (s, 1H), 6.64 – 7.83 (m, 11H).; ^{13}C NMR (75 MHz): δ 38.1, 55.9, 56.0, 62.4, 81.3, 86.6, 109.6, 111.0, 114.2, 116.2, 116.3, 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, 1526cm^{-1}; MS (m/z): 488 (M^{+}+1); Elemental Analysis for $C_{27}H_{21}NO_8$: 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)



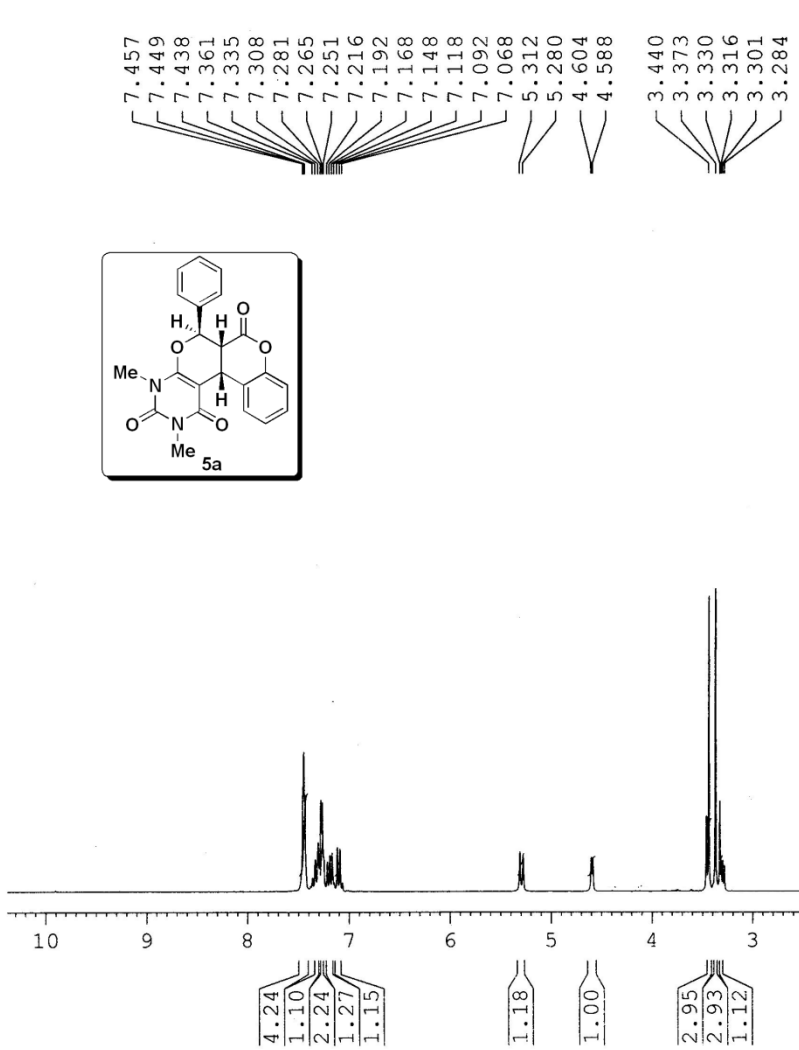
Yield: 88%; Colourless solid; mp: 159-161 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.72 (d, *J* = 12.9Hz, 1H), 4.97 (d, *J* = 12.9 Hz, 1H), 5.19 (s, 1H), 6.05 (s, 1H), 6.29 – 7.80 (m, 11H); ^{13}C NMR (75 MHz): δ 37.1, 64.6, 74.8, 86.5, 102.1, 110.7, 112.4, 114.2, 116.3, 116.6, 117.6, 121.5, 123.0, 124.2, 129.2, 131.9, 132.8, 144.2, 144.7, 152.1, 152.5, 158.2, 160.9; IR (neat): ν 1689, 1584, 1537cm^{-1}; MS (m/z): 418 (M^{+}+1); Elemental Analysis for $C_{23}H_{15}NO_7$: 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; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.56 (d, *J* = 13.2Hz, 1H), 4.94 (d, *J* = 12.9 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, 1517cm^{-1}; MS (m/z): 434 (M^++1); Elemental Analysis for $C_{23}H_{15}NO_6S$: Calculated: C 63.73; H, 3.49; N, 3.23; Found: C 63.61; H, 3.38; N, 3.34.



BRUKER

Current Data Parameters
NAME VM-I-94
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
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Time 17.46
INSTRUM spect
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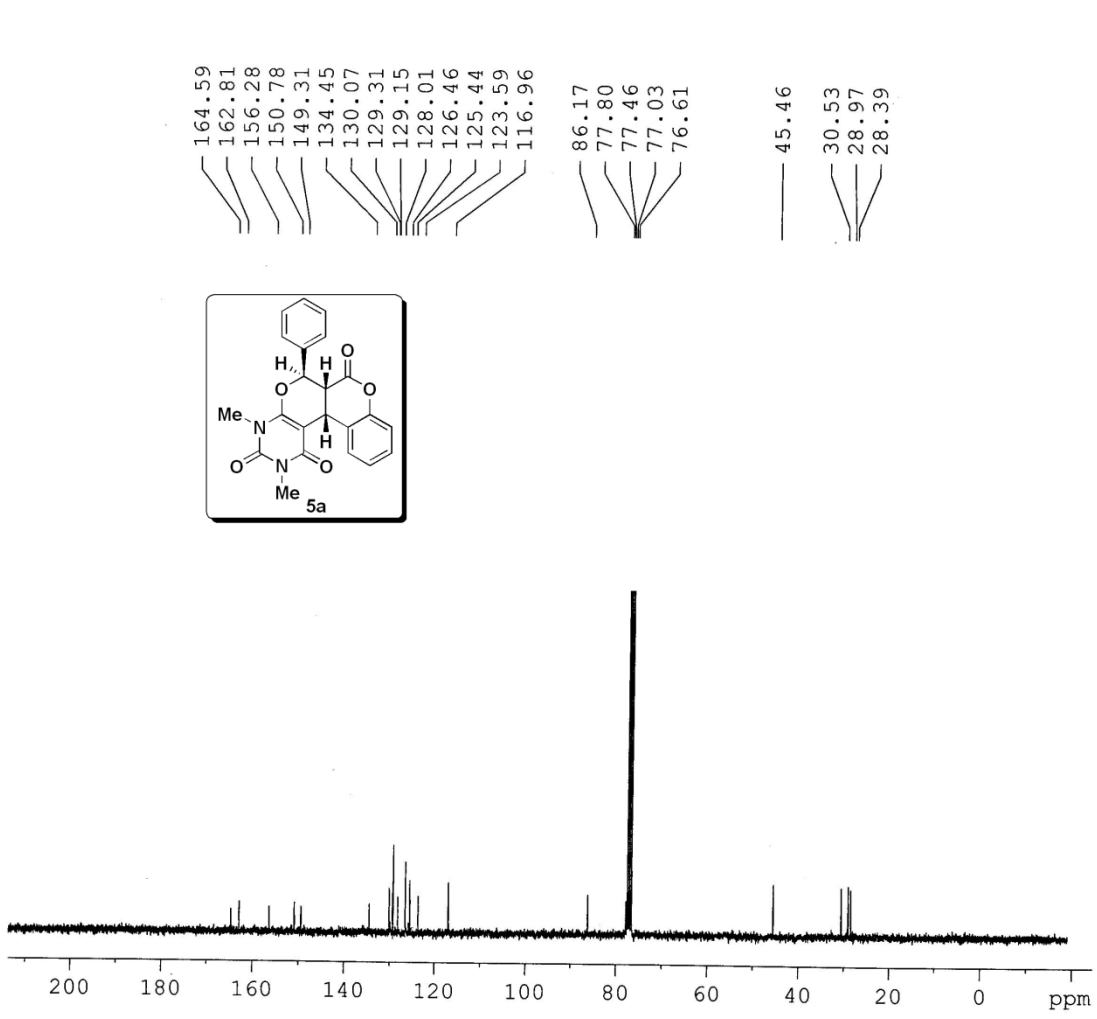
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME VM-I-94
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110711
Time 17.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 241
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40

Current Data Parameters
NAME VM-I-124
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110711
Time 18.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.336
3.240
3.256
3.273
3.289
3.371
3.461
4.579
4.593
5.233
5.266
6.980
7.008
7.104
7.131
7.265
7.443
3.23
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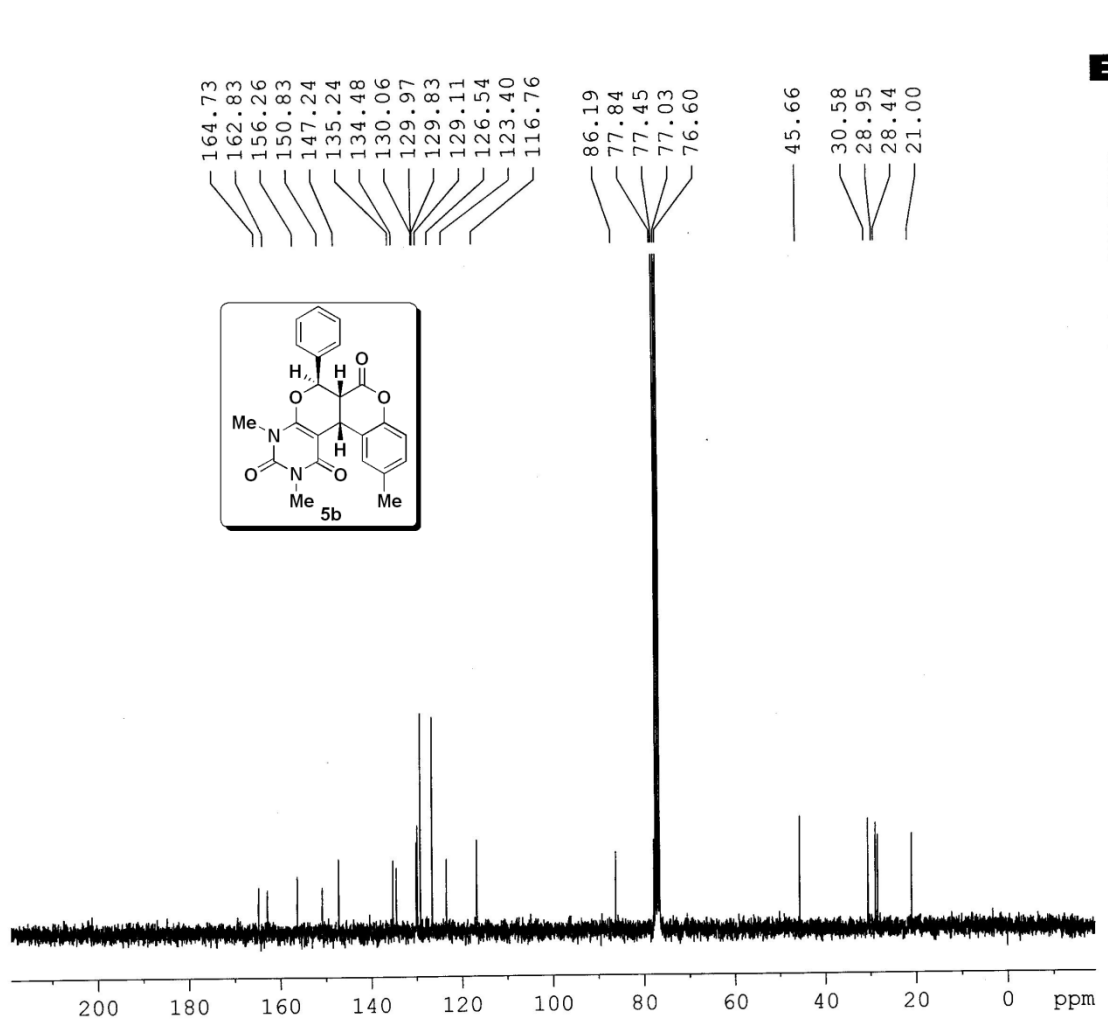
2.10
1.01
1.07
3.09
2.97
1.00
3.01
ppm
5b



BRUKER

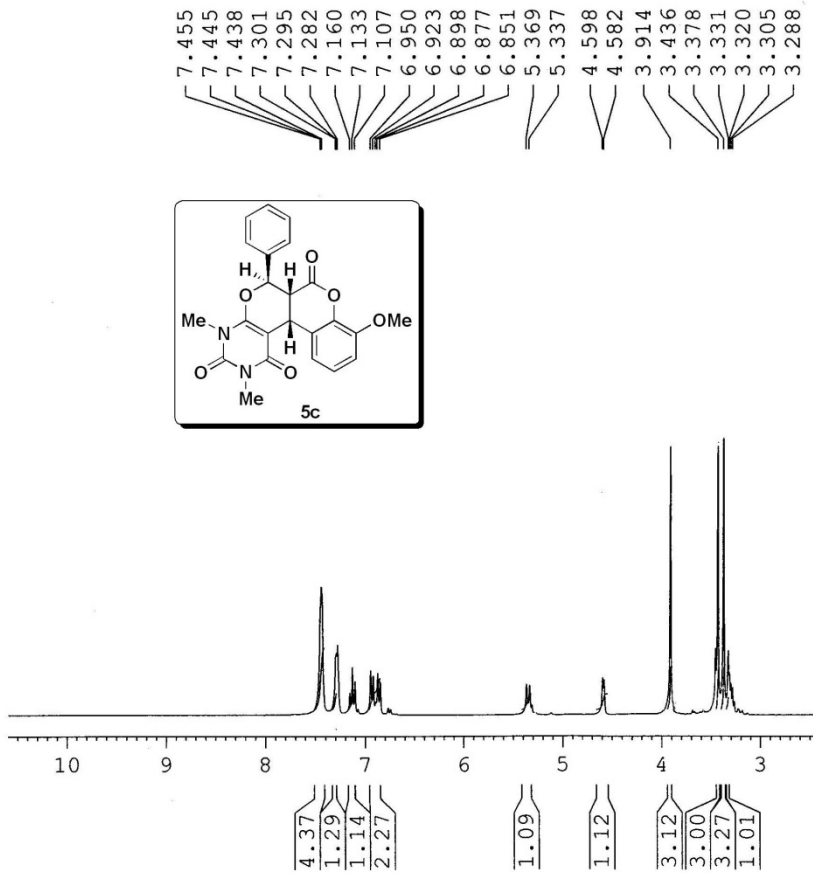
Current Data Parameters
NAME VM-I-124
EXPNO 4
PROCNO 1

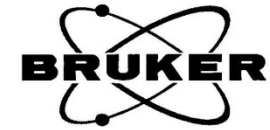
F2 - Acquisition Parameters
Date_ 20110716
Time 16.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 306
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 574.7
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40



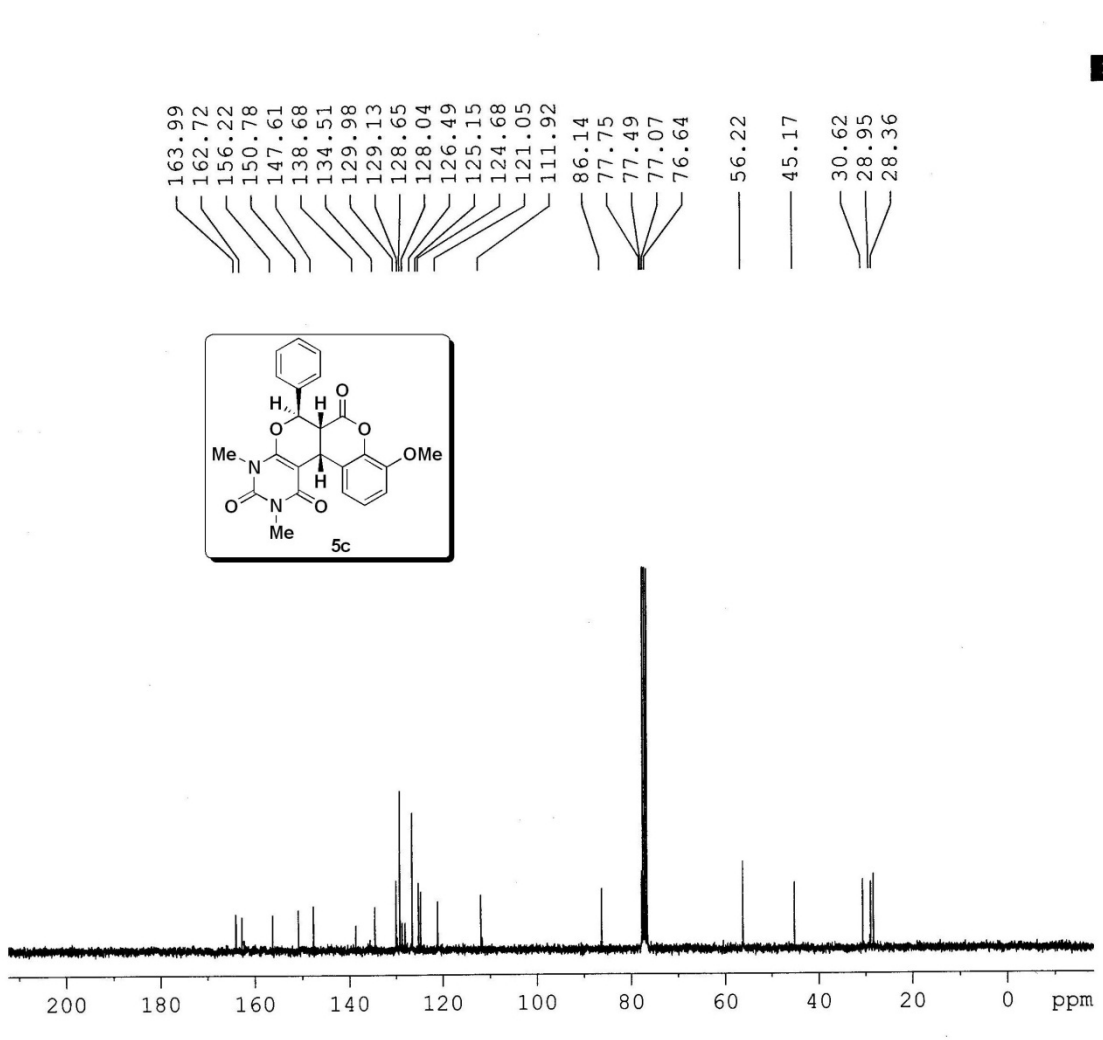


Current Data Parameters
NAME VM-136
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110721
Time 14.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

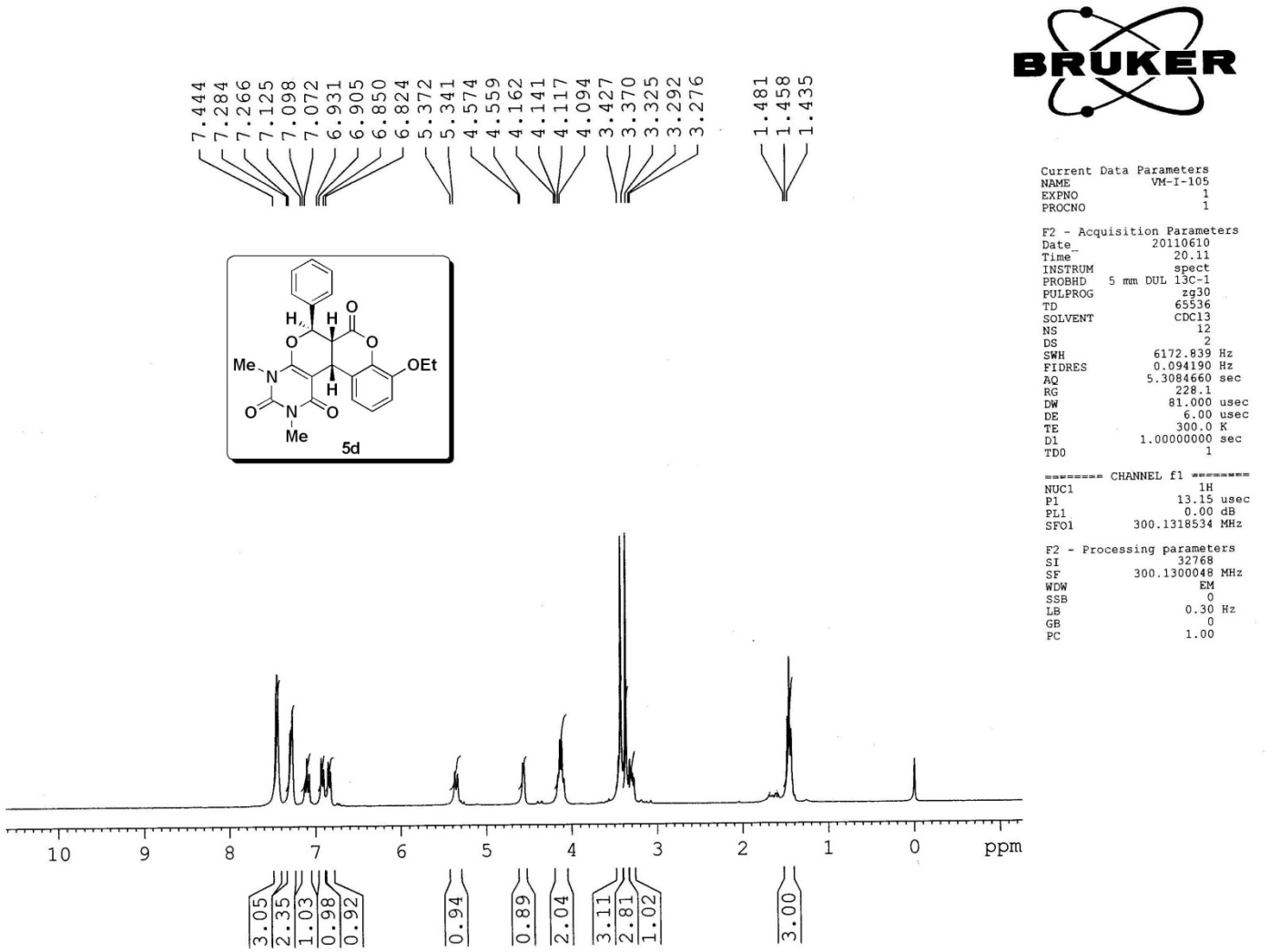
Current Data Parameters
NAME VM-136
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110721
Time 14.41
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 232
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

Current Data Parameters

NAME	VM-I-105
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

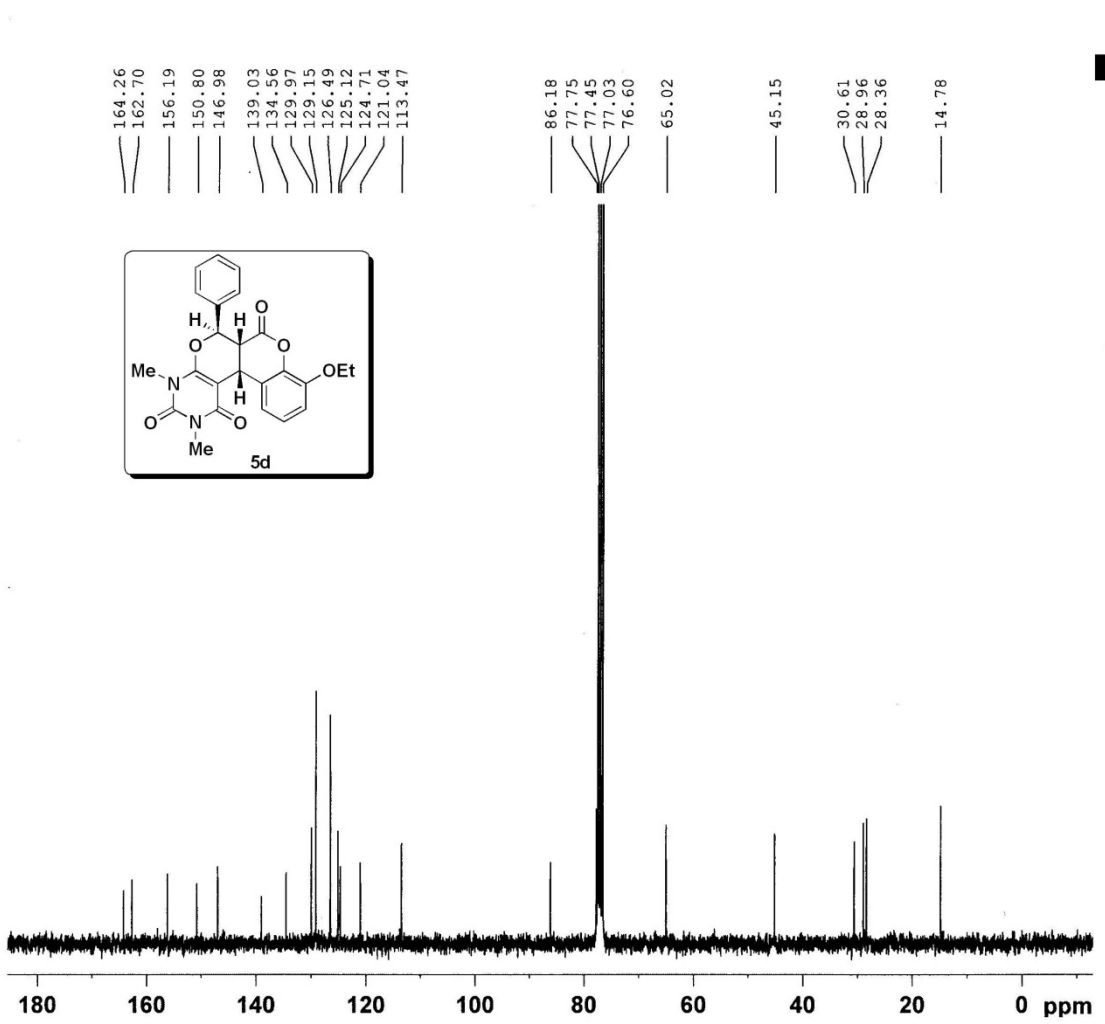
Date_	20110610
Time	20.11
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	12
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	228.1
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	13.15 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300048 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

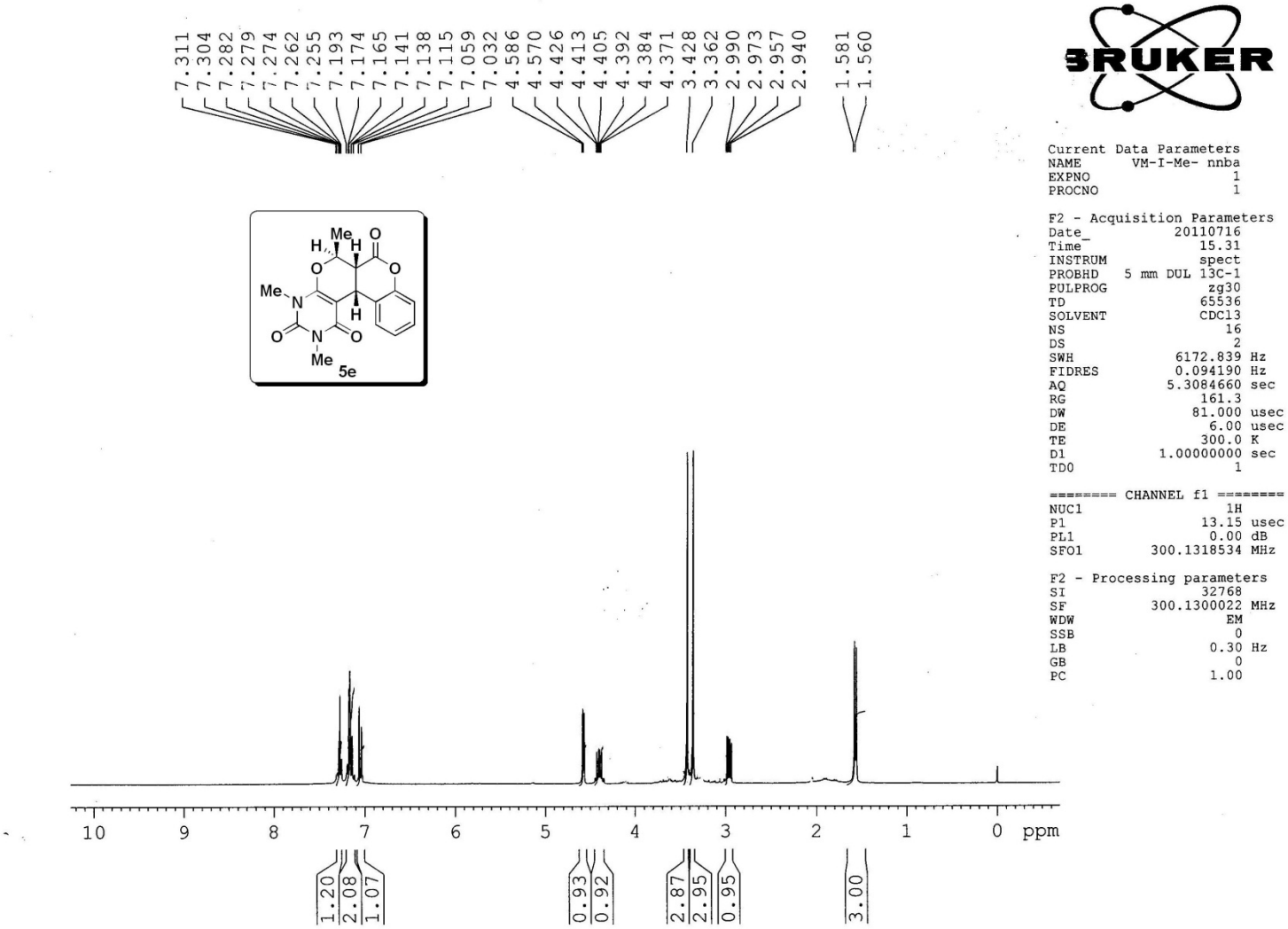
Current Data Parameters
NAME VM-I-105
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110610
Time 20.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40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7.141
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5e
10
9
8
7
6
5
4
3
2
1
0
ppm
1.20
2.08
1.07
0.93
0.92
2.87
2.95
0.95
3.00
BRUKER
Current Data Parameters
NAME VM-I-Me- nnba
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110716
Time 15.31
INSTRUM spect
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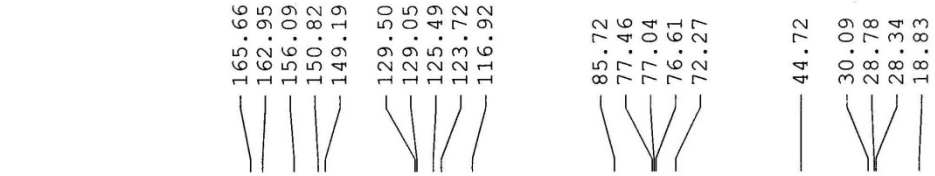
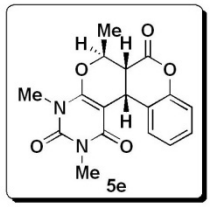
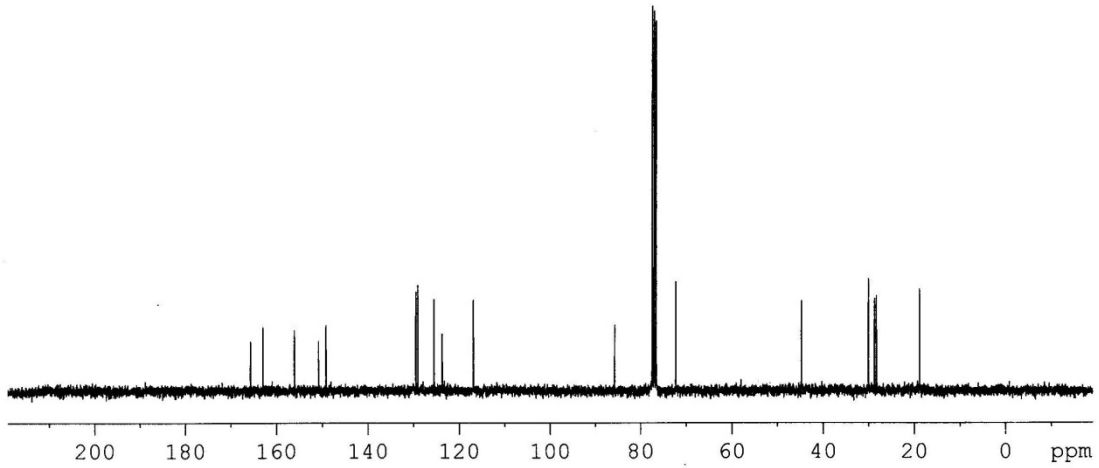
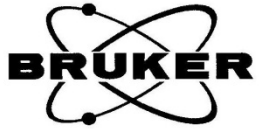
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300022 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00









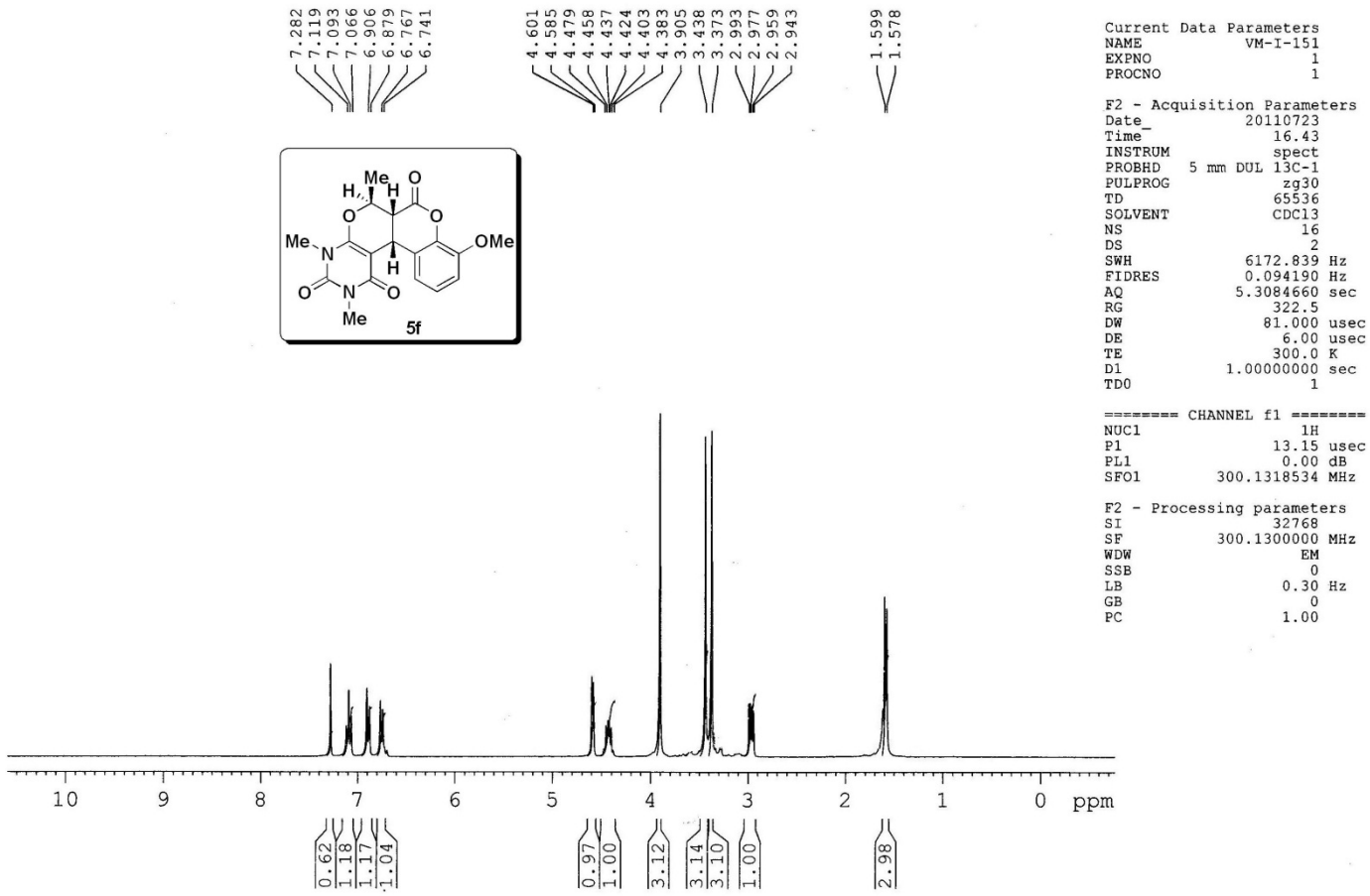
Current Data Parameters
NAME VM-I-Me- nnba
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110716
Time 15.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 194
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

Current Data Parameters

NAME	VM-I-151
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

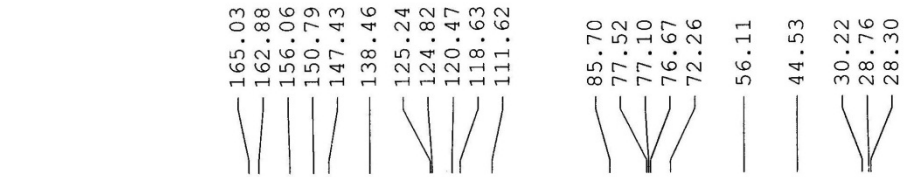
Date_	20110723
Time	16.43
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	322.5
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

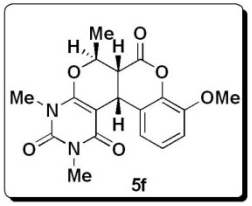
======== CHANNEL f1 ========

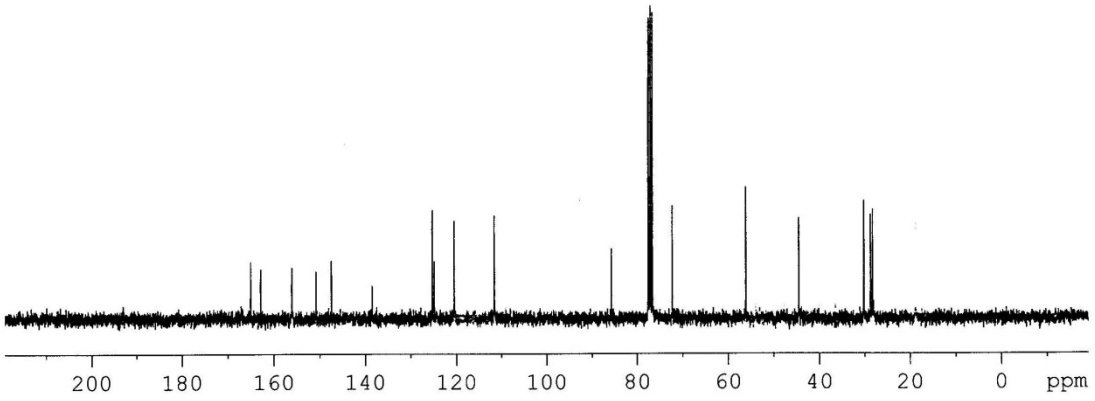
NUC1	1H
P1	13.15 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00







BRUKER

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	0.274439 Hz
AQ	1.8219508 sec
RG	20642.5
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

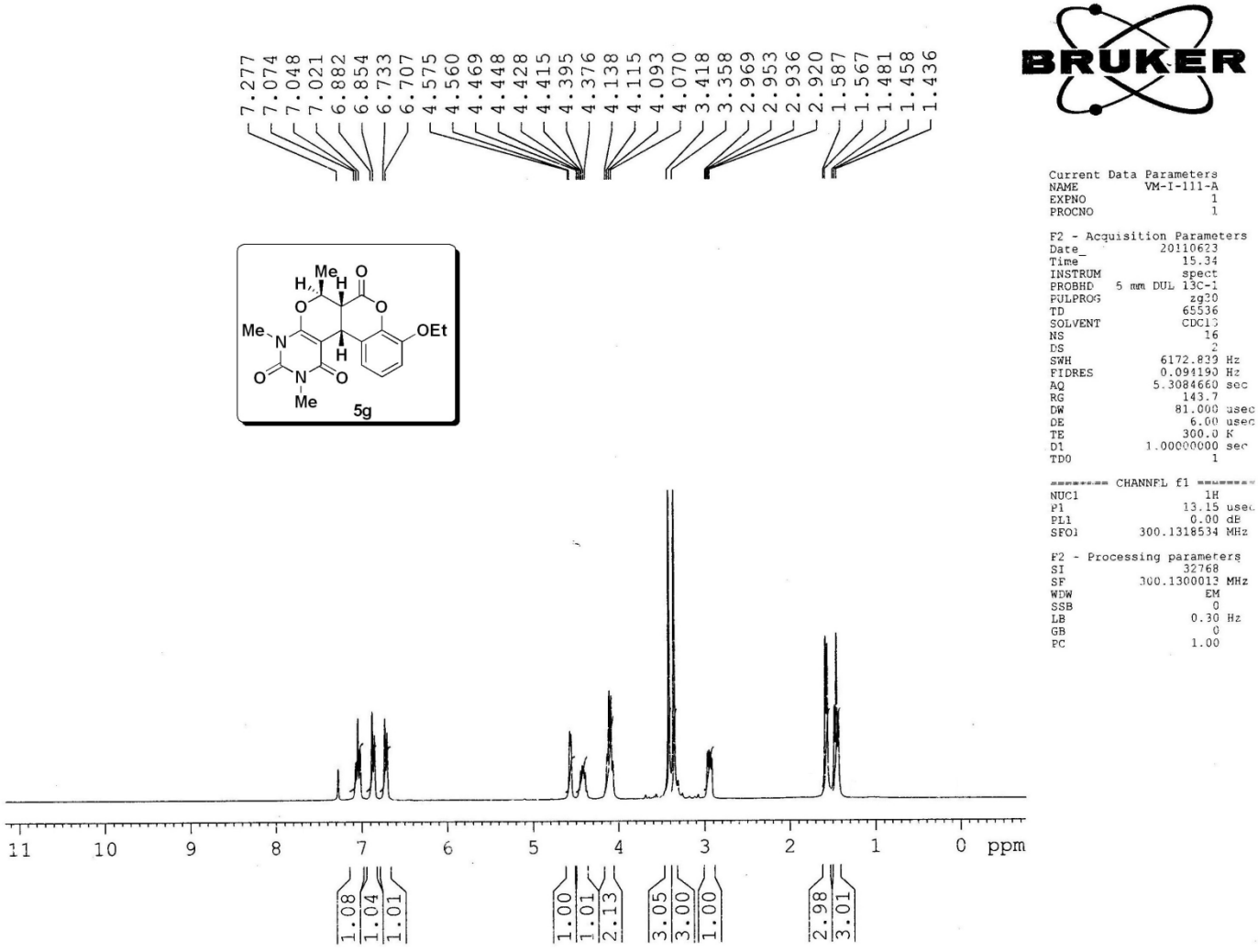
NUC1	13C
P1	9.30 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

======== 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	0
LB	1.00 Hz
GB	0
PC	1.40



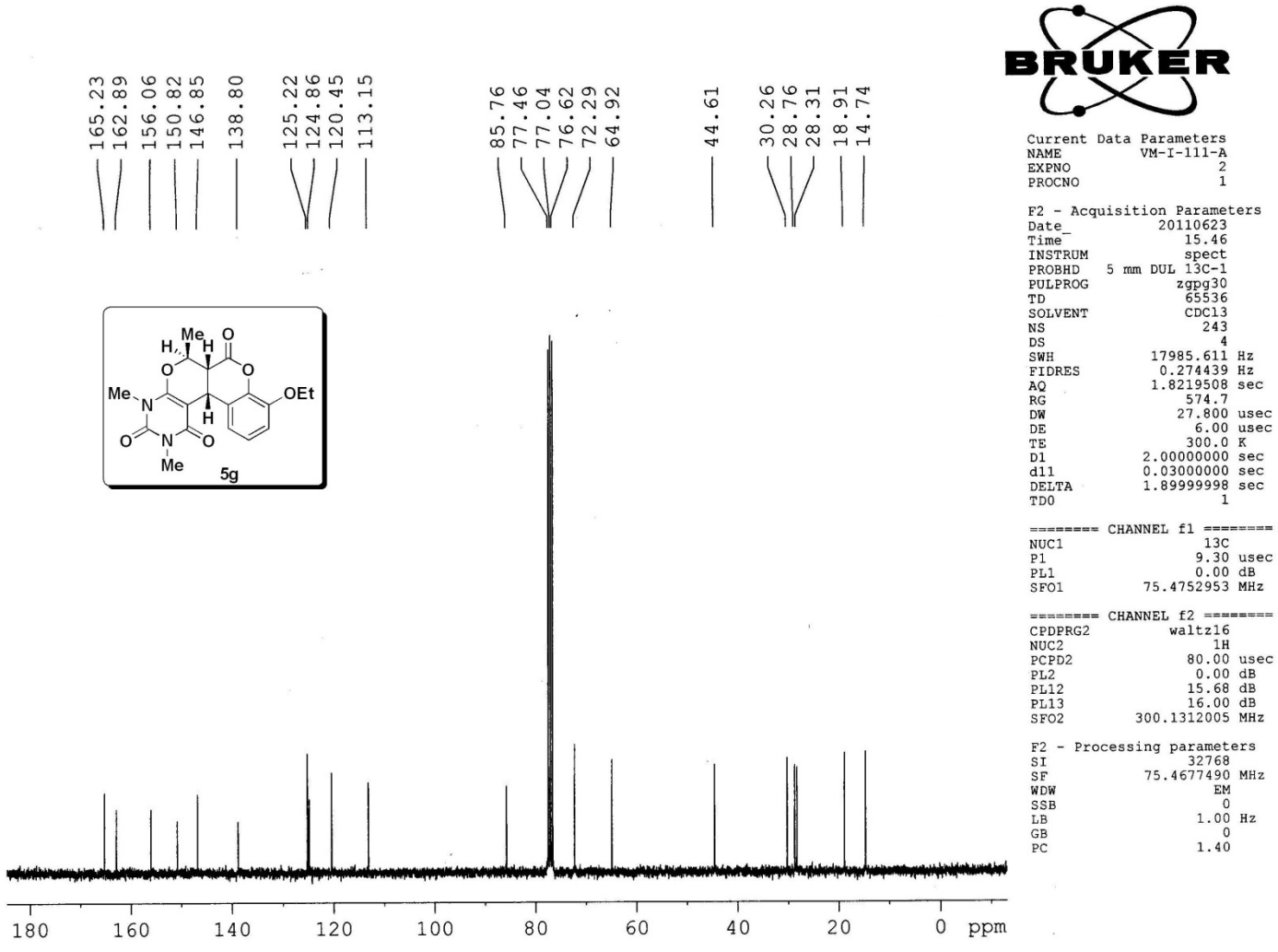
BRUKER

```
Current Data Parameters
NAME                  VM-I-111-A
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20110623
Time                       15.34
INSTRUM                    spect
PROBHD     5 mm DUL 13C-1
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     6172.839 Hz
FIDRES                  0.094190 Hz
AQ                      5.3084660 sec
RG                         143.7
DW                        81.000 usec
DE                         6.00 usec
TE                        300.0 K
D1                      1.00000000 sec
TD0                            1

======== 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                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



BRUKER

Current Data Parameters

NAME	VM-I-111-A
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20110623
Time	15.46
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	243
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	574.7
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

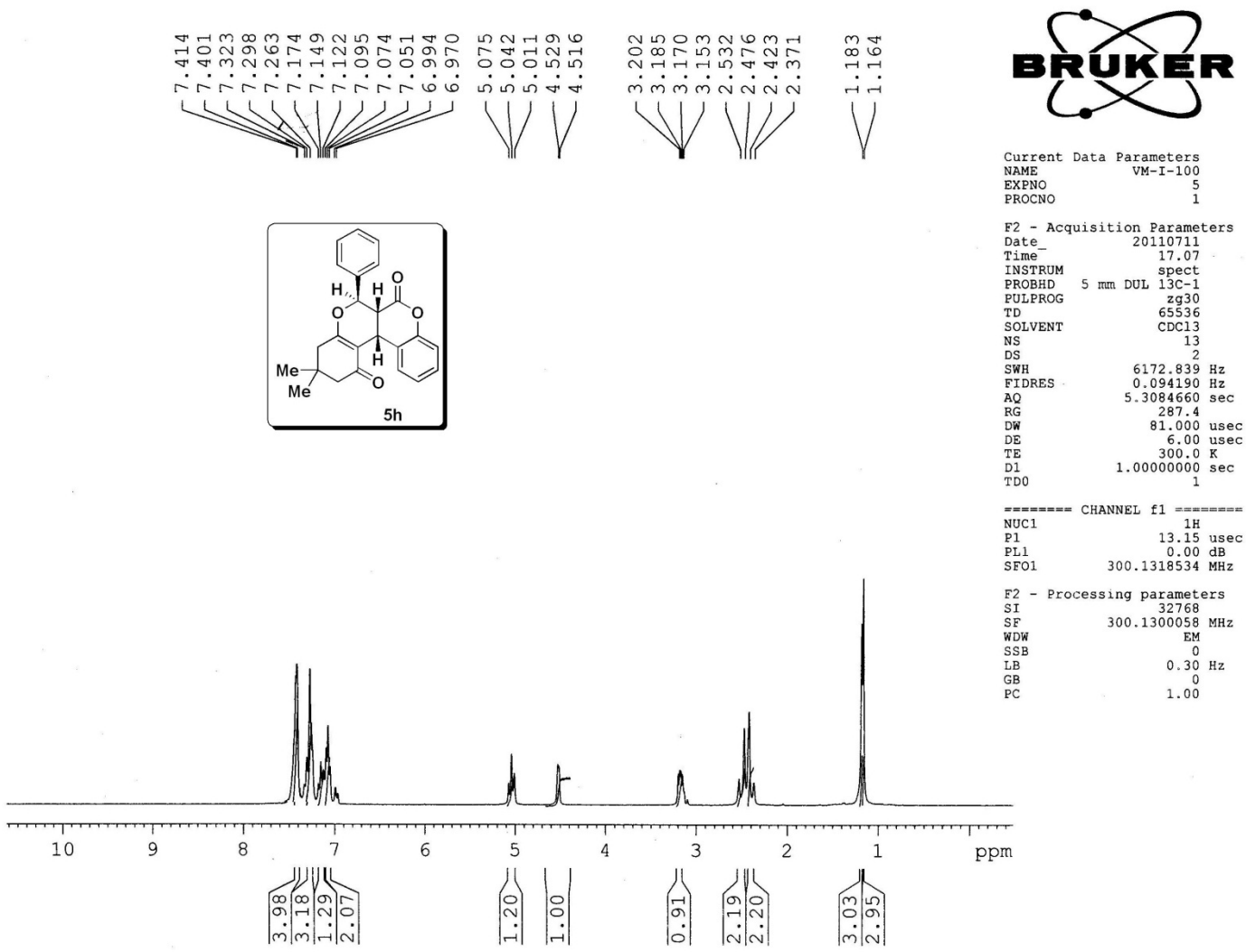
NUC1	13C
P1	9.30 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

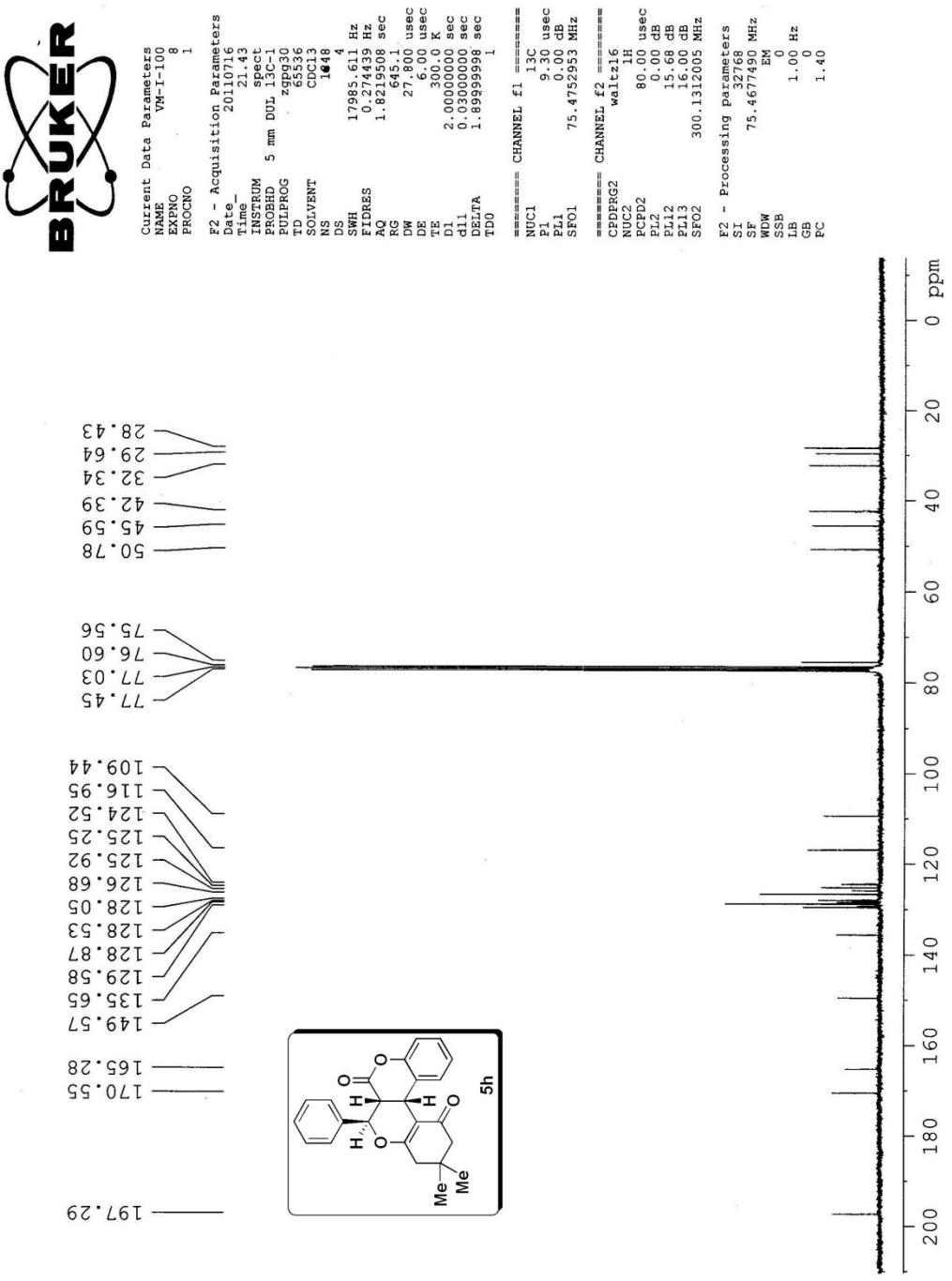
======== CHANNEL f2 ========

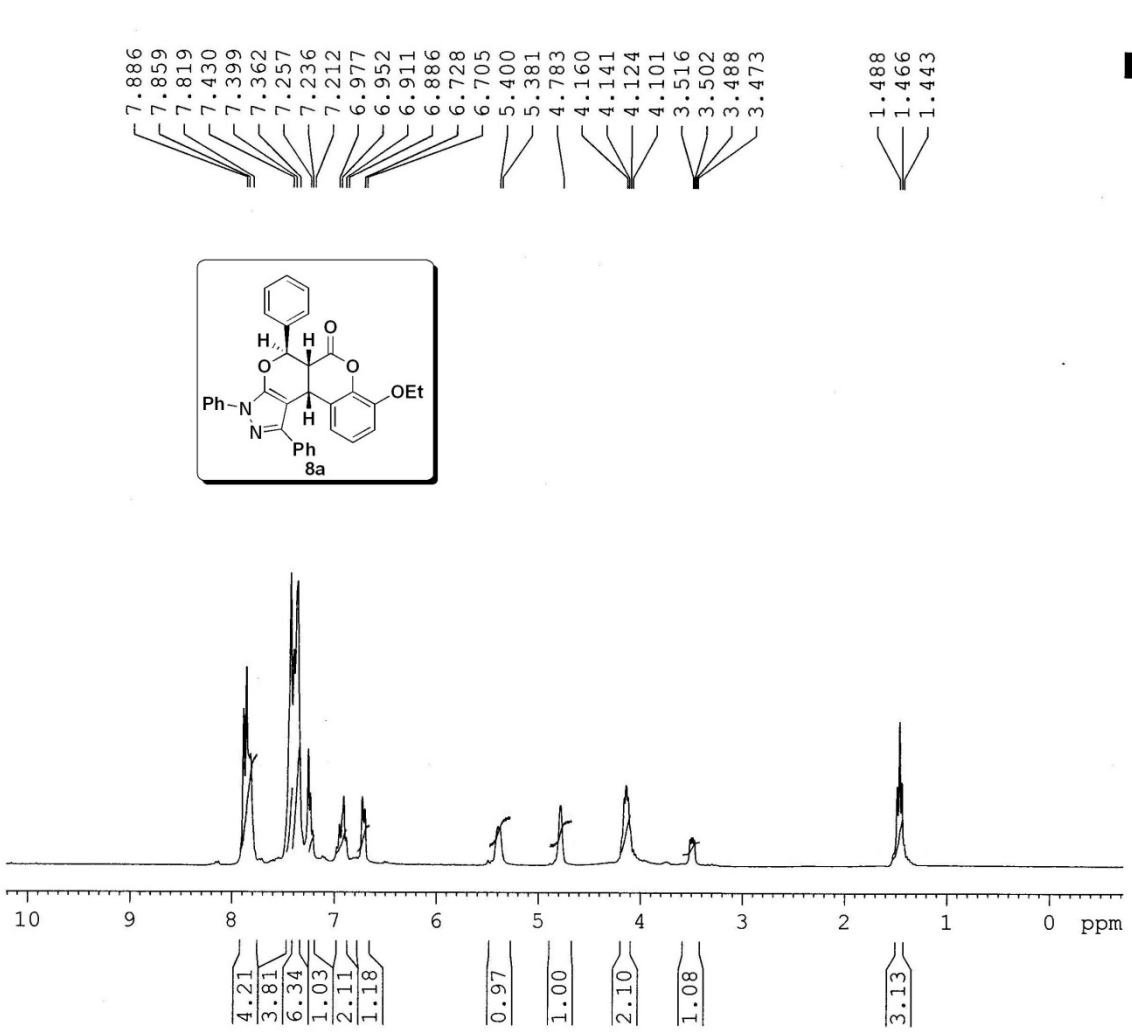
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

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


7.414
7.401
7.323
7.298
7.263
7.174
7.149
7.122
7.095
7.074
7.051
6.994
6.970
5.075
5.042
5.011
4.529
4.516
3.202
3.185
3.170
3.153
2.532
2.476
2.423
2.371
1.183
1.164
BRUKER
Current Data Parameters
NAME VM-I-100
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110711
Time 17.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
5h
10
9
8
7
6
5
4
3
2
1
ppm
3.98
3.18
1.29
2.07
1.20
1.00
0.91
2.19
2.20
3.03
2.95


Current Data Parameters
NAME VM-I-100
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 1048
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40
28.43
29.64
32.34
42.39
45.59
50.78
75.56
76.60
77.03
77.45
109.44
116.95
124.52
125.25
125.92
126.68
128.05
128.53
128.87
129.58
135.65
149.57
165.28
170.55
197.29
Me
Me
5h
200
180
160
140
120
100
80
60
40
20
0
ppm



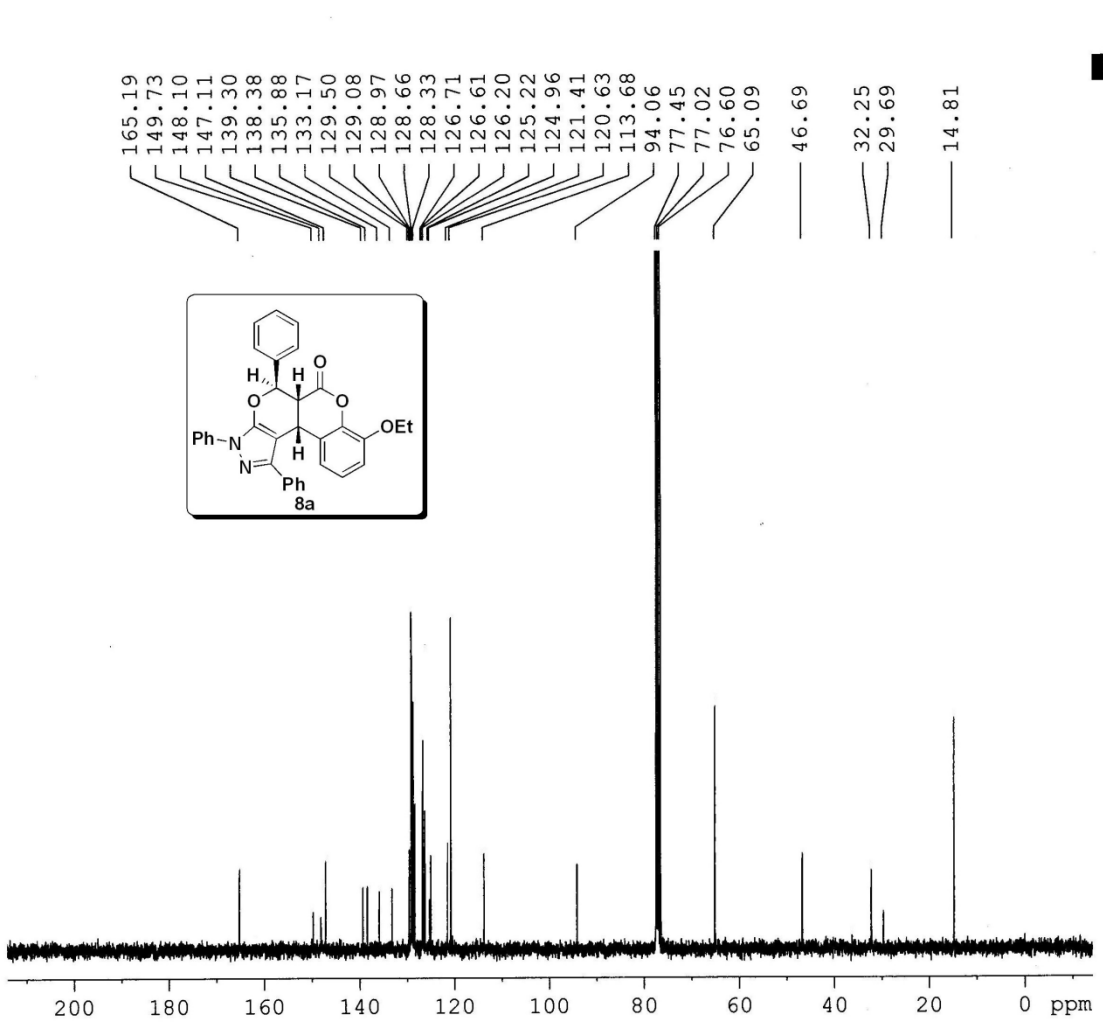
BRUKER

```
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                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          16
DS                           2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084660 sec
RG                         181
DW                    81.000 usec
DE                     6.00 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                    13.15 usec
PL1                    0.00 dB
SFO1           300.1318534 MHz

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



BRUKER

Current Data Parameters

NAME	VM-I-111
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20110623
Time	14.54
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	527
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	512
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

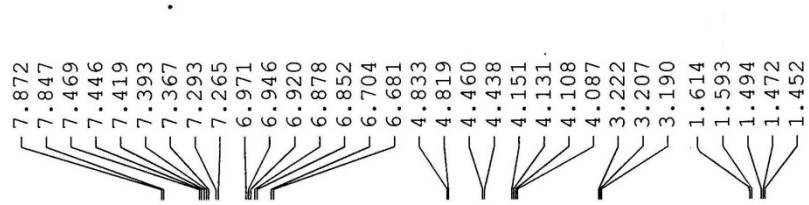
NUC1	13C
P1	9.30 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

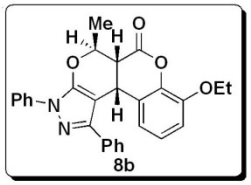
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

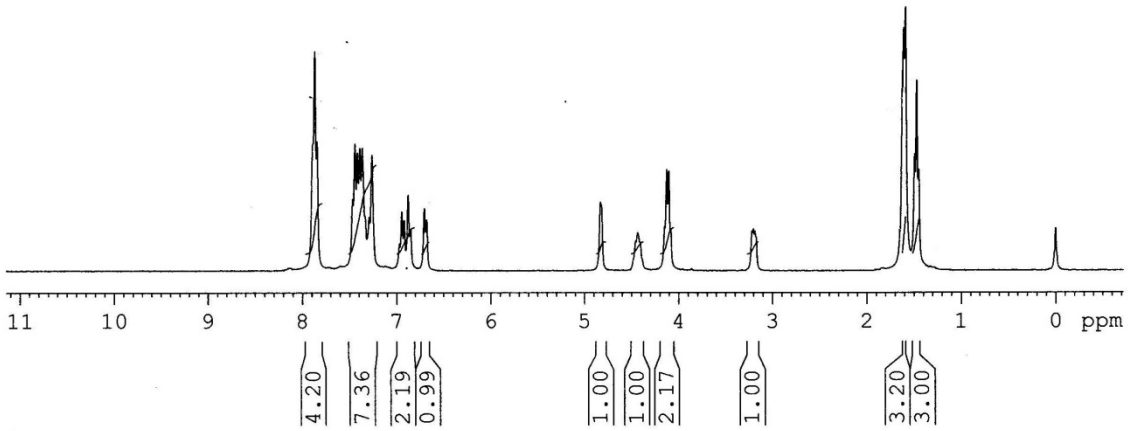
F2 - Processing parameters

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





8b

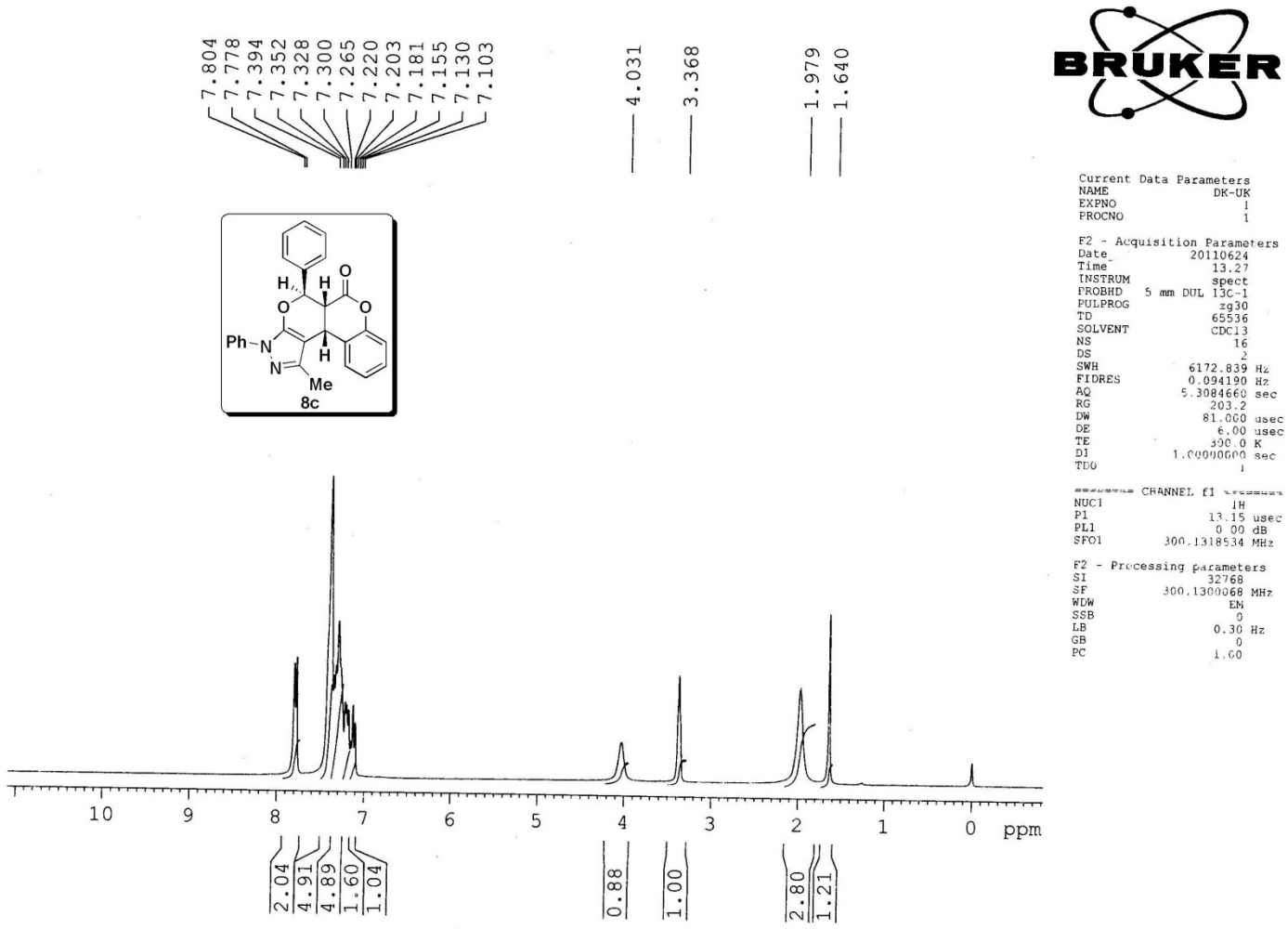



```
Current Data Parameters
NAME                  VM-I-115
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_              20110623
Time                   13.04
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       16
DS                        2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    322.5
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.15 usec
PL1                    0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                       32768
SF            300.1300066 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.00
```



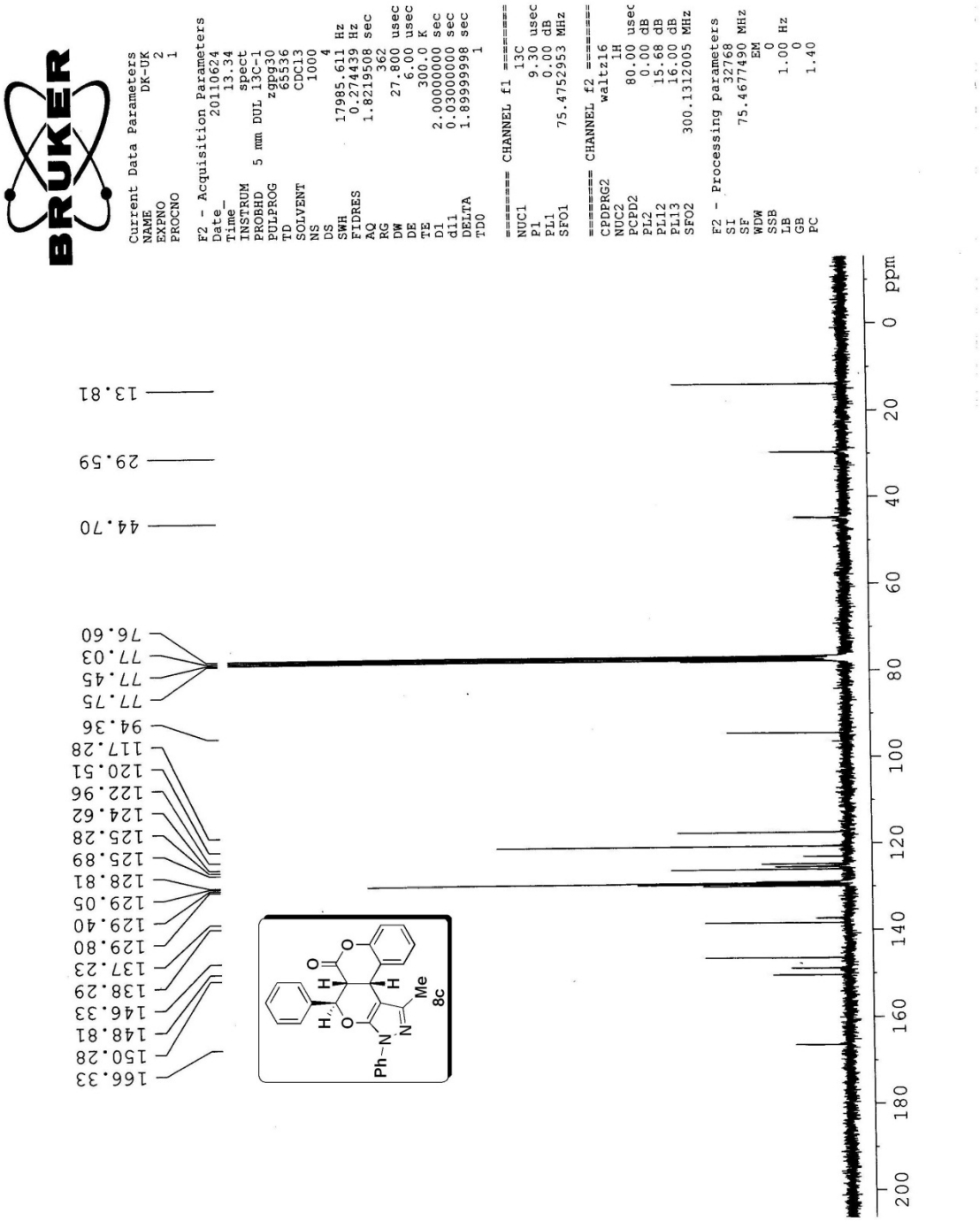
BRUKER

Current Data Parameters
NAME DK-UK
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20110624
Time 13.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME DK-UK
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110624
Time_ 13.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40
13.81
29.59
44.70
76.60
77.03
77.45
77.75
94.36
117.28
120.51
122.96
124.62
125.28
125.89
128.81
129.05
129.40
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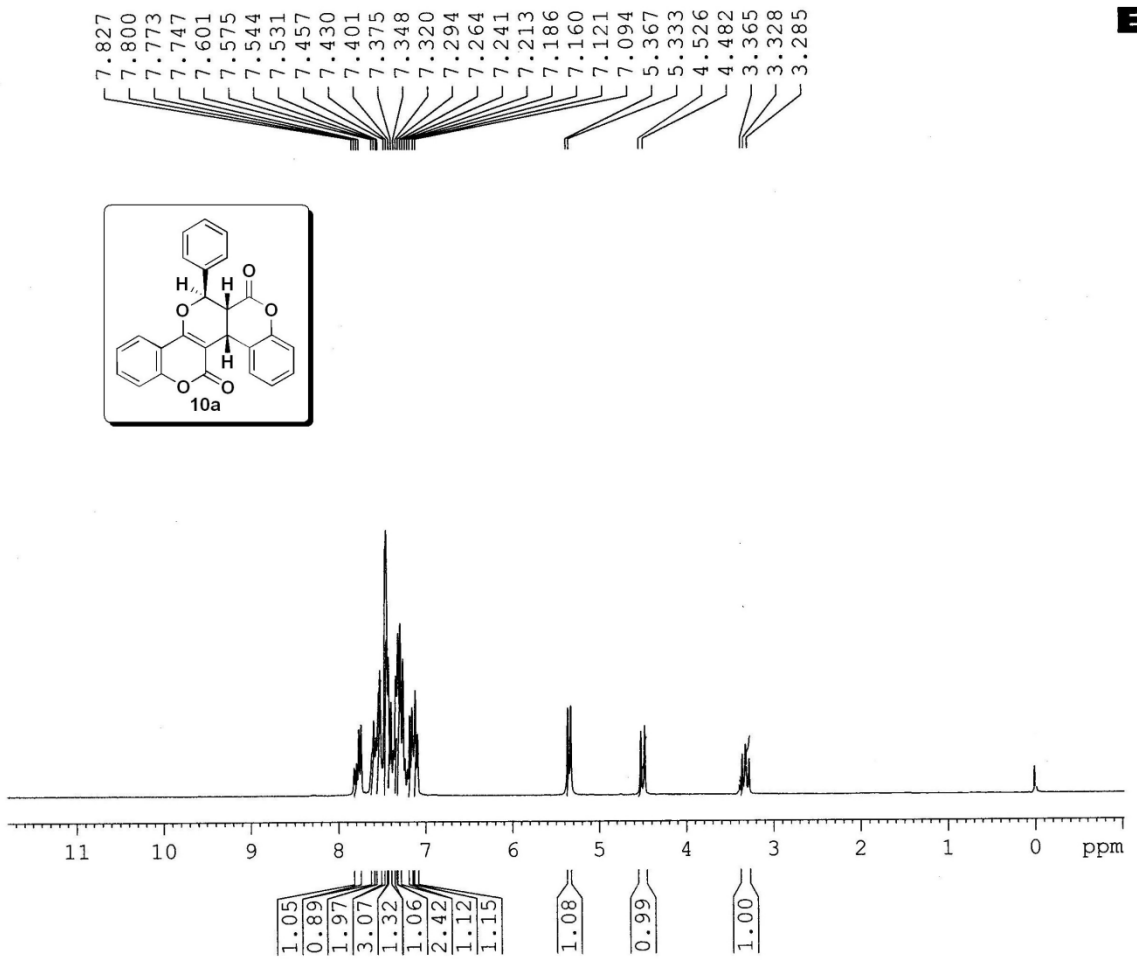
137.23
138.29
146.33
148.81
150.28
166.33
Ph–N
N
Me
8c
ppm



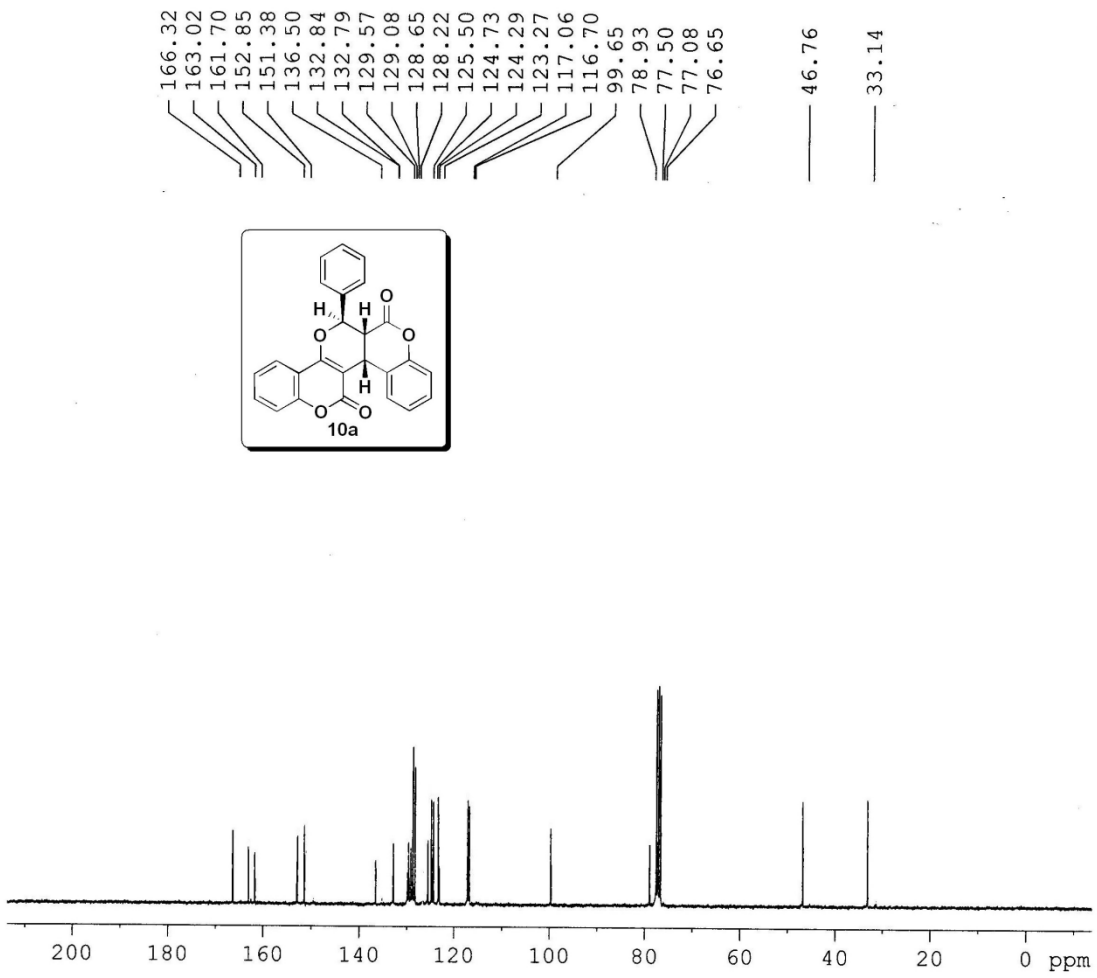
BRUKER

Current Data Parameters
NAME VM-I-98
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110624
Time 18.20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

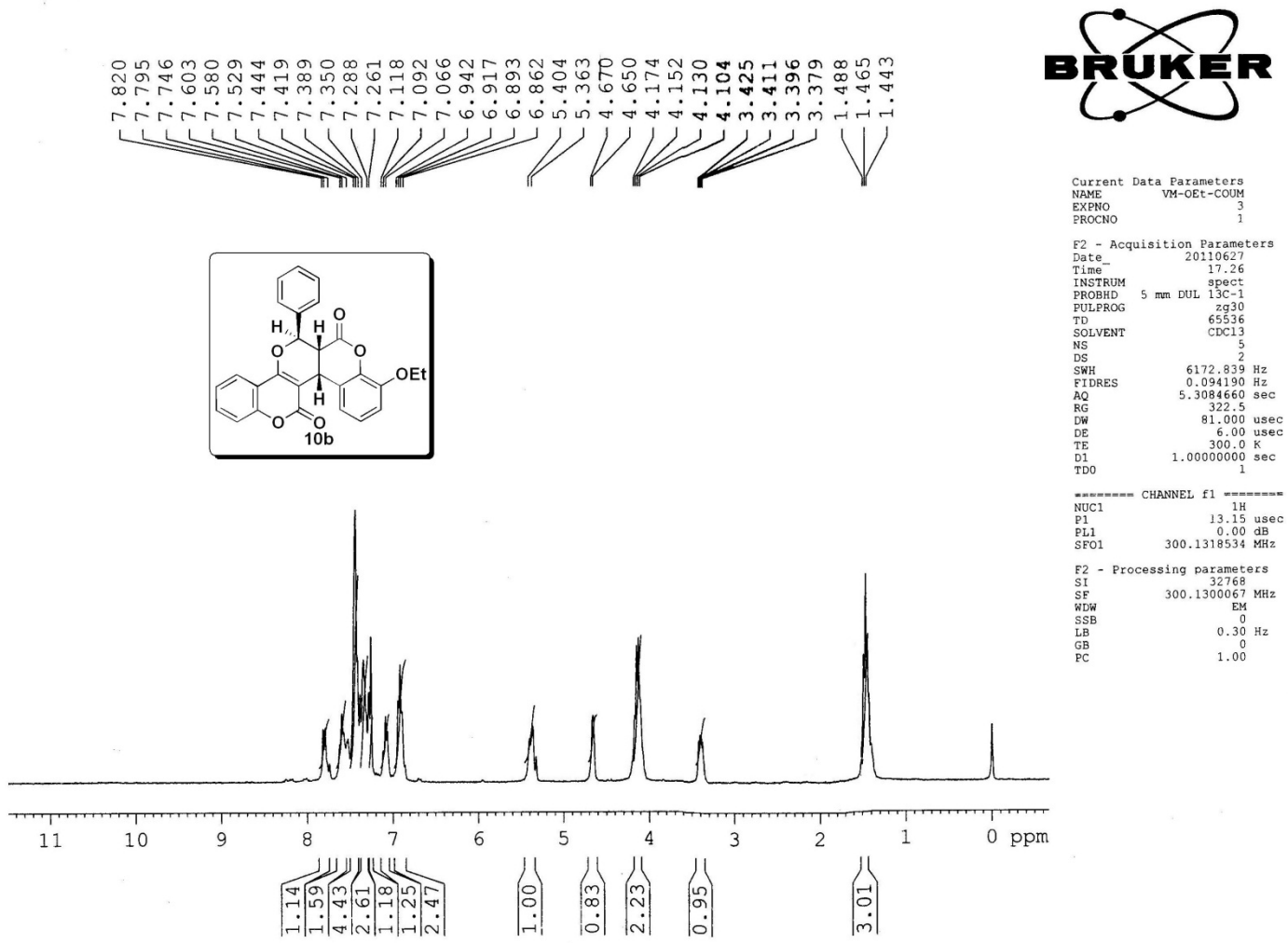
======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

```
Current Data Parameters
NAME                  VM-I-98
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110630
Time                  16.00
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1000
DS                       4
SWH              17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                     512
DW                  27.800 usec
DE                   6.00 usec
TE                  300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                    13C
P1                   9.30 usec
PL1                  0.00 dB
SFO1            75.4752953 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                         0
LB                   1.00 Hz
GB                          0
PC                       1.40
```



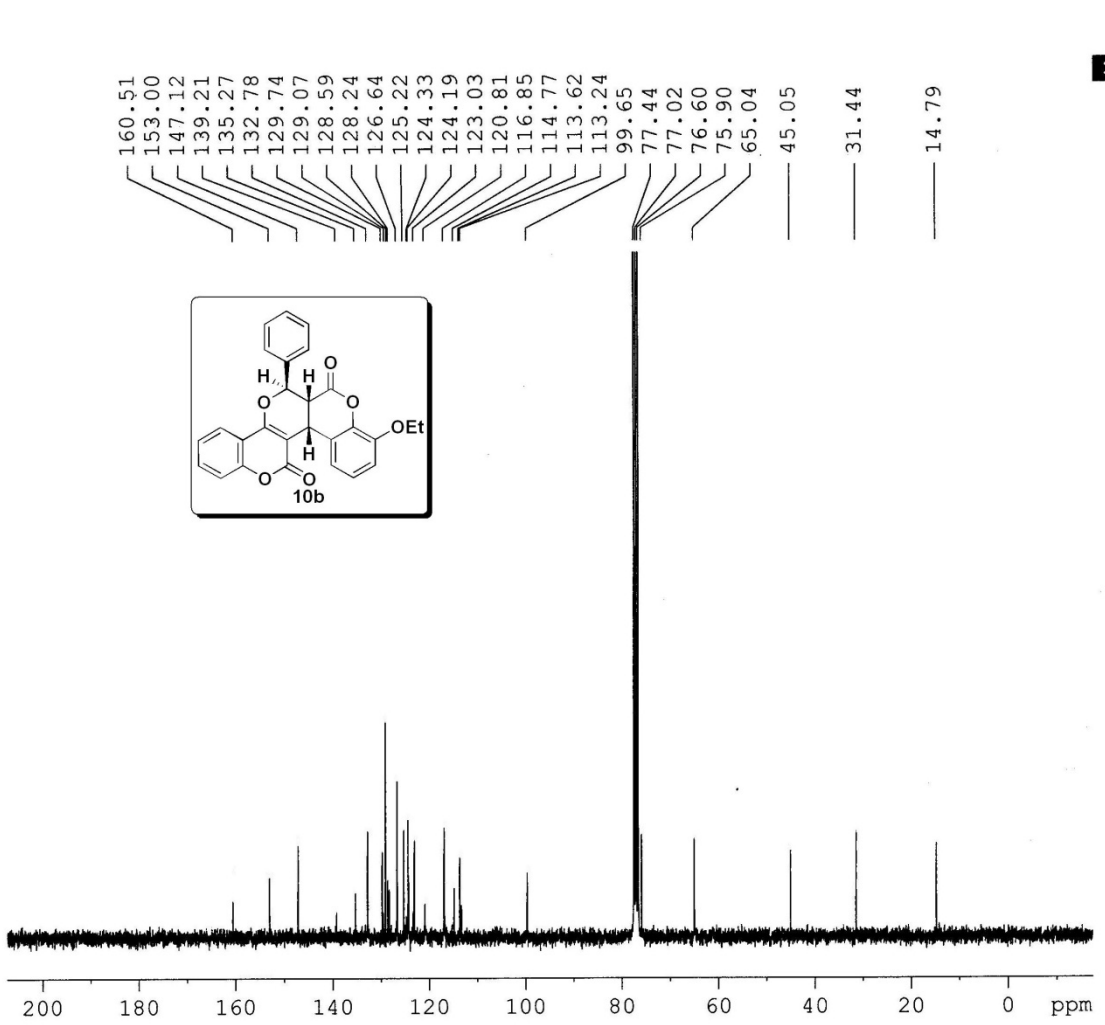
BRUKER

Current Data Parameters
NAME VM-OEt-COUM
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110627
Time 17.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

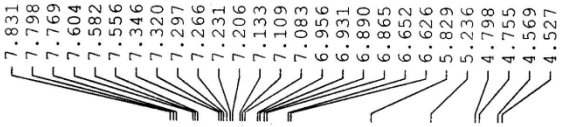
Current Data Parameters
NAME VM-OEt-COUM
EXPNO 2
PROCNO 1

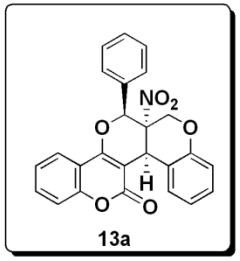
F2 - Acquisition Parameters
Date_ 20110627
Time 16.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 574.7
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 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

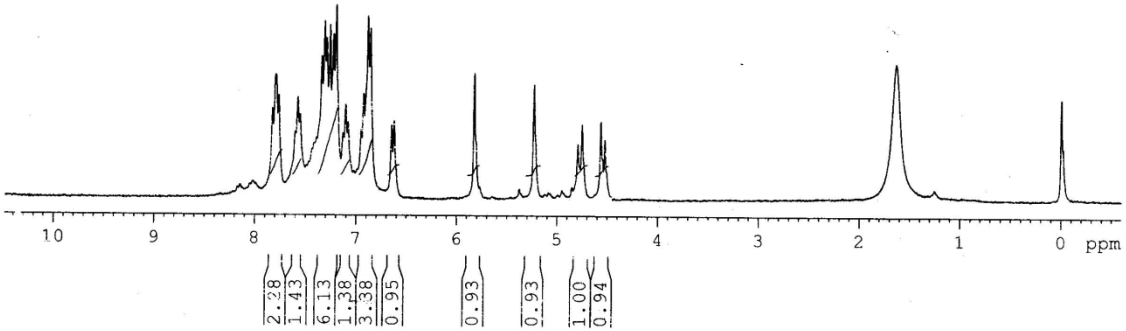
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

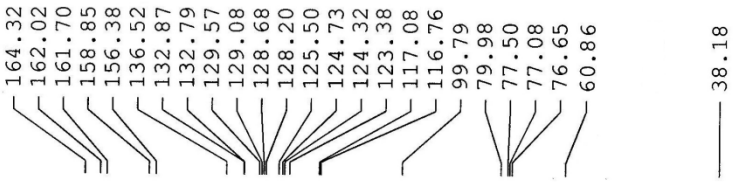


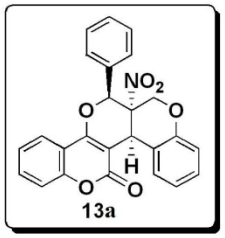


13a

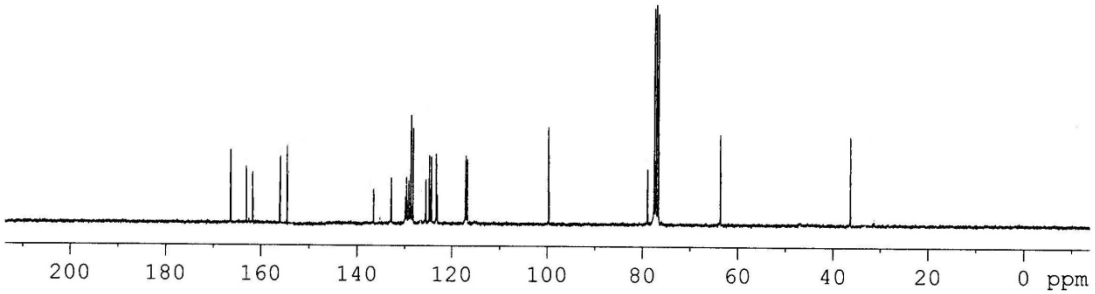

```
Current Data Parameters
NAME                  MGS-148
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20100817
Time                   10.37
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         16
DS                          2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                     322.5
DW                    81.000 usec
DE                     6.00 usec
TE                    300.0 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.15 usec
PL1                      0.00 dB
SFO1          300.1318534 MHz

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





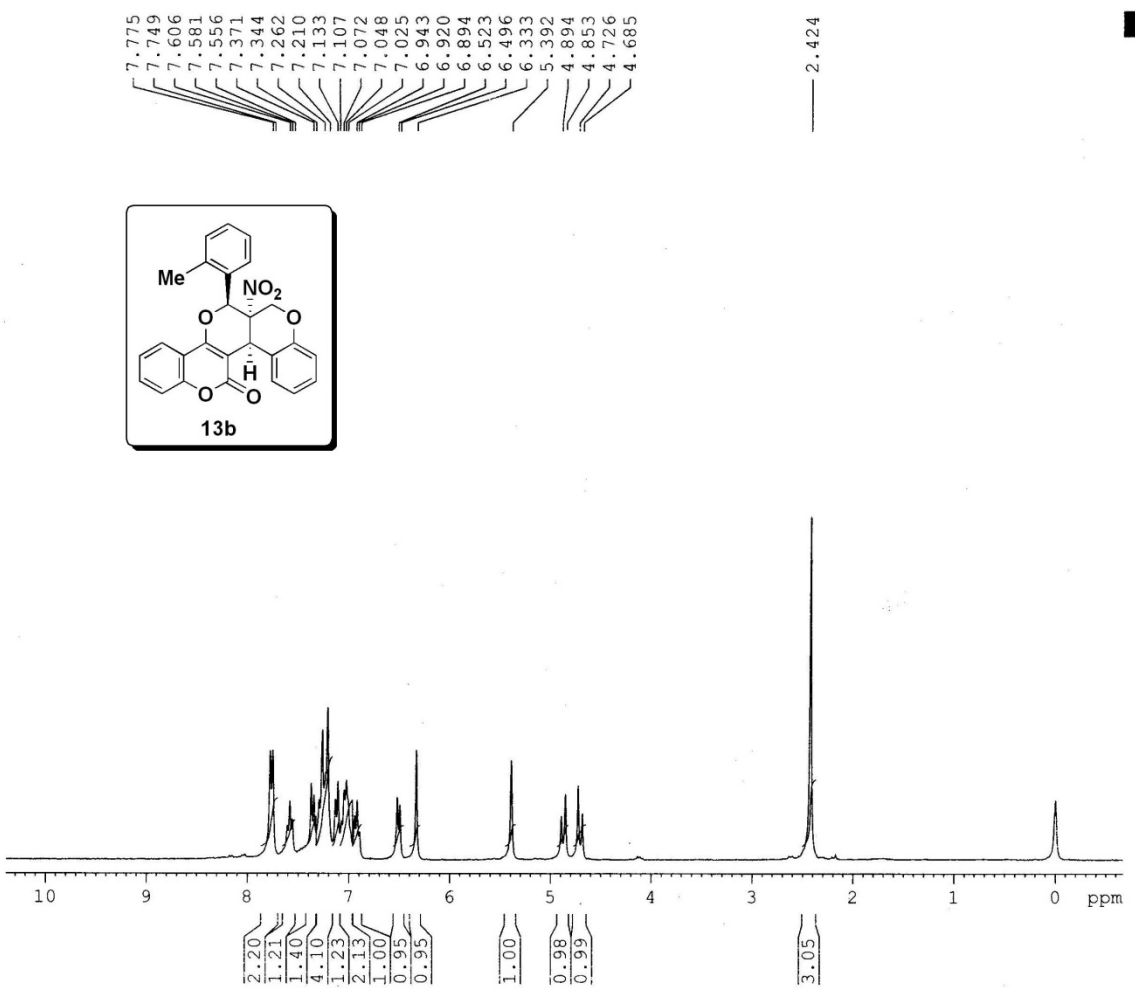


13a



BRUKER

```
Current Data Parameters
NAME                  . MGS-148
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20100817
Time                      16.00
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1000
DS                            4
SWH                  17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                          512
DW                       27.800 usec
DE                        6.00 usec
TE                       300.0 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.30 usec
PL1                        0.00 dB
SFO1                75.4752953 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                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```



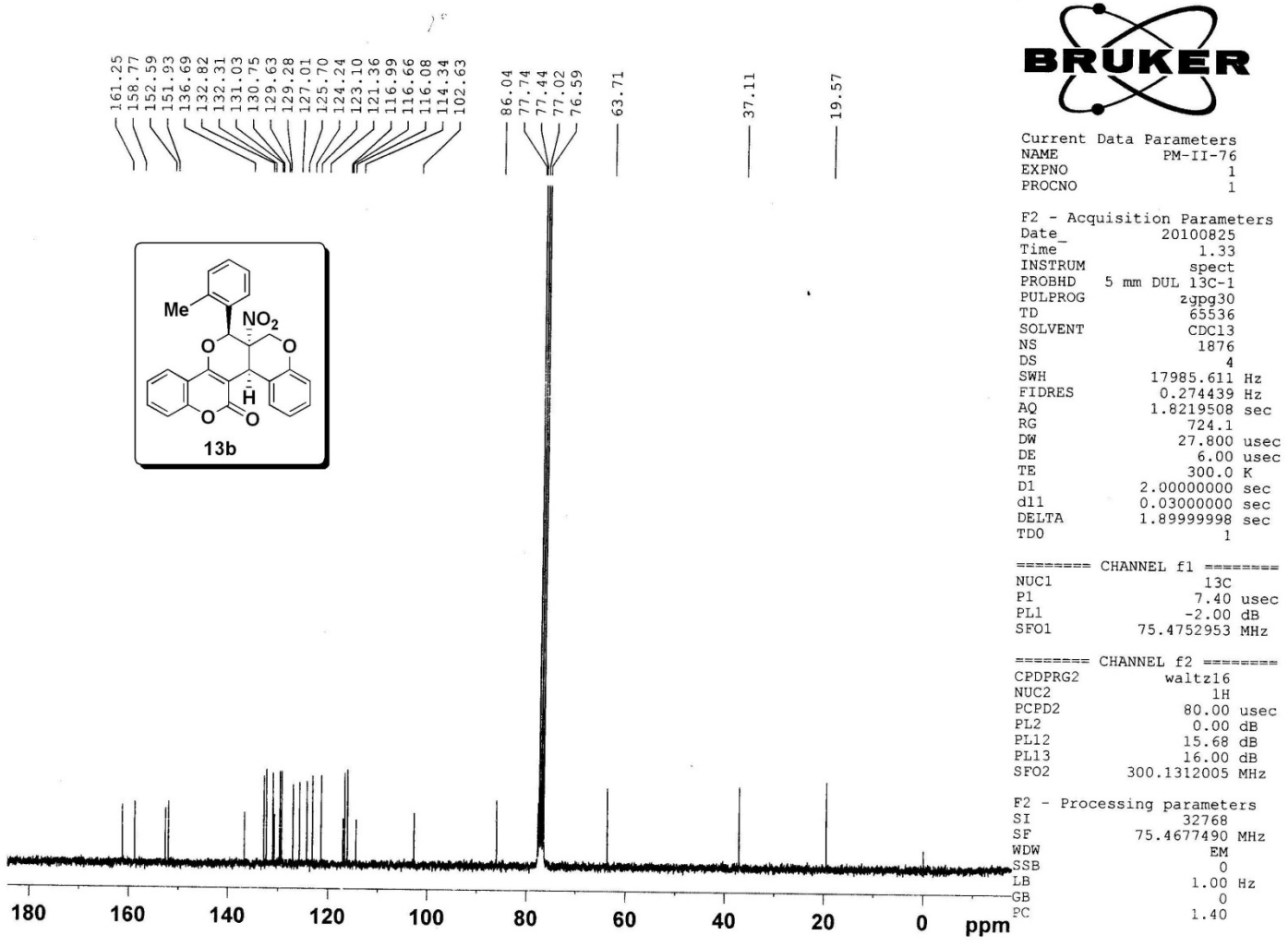
BRUKER

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

F2 - Acquisition Parameters
Date_ 20100824
Time 11.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 406.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters

NAME	PM-II-76
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20100825
Time	1.33
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1876
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	724.1
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

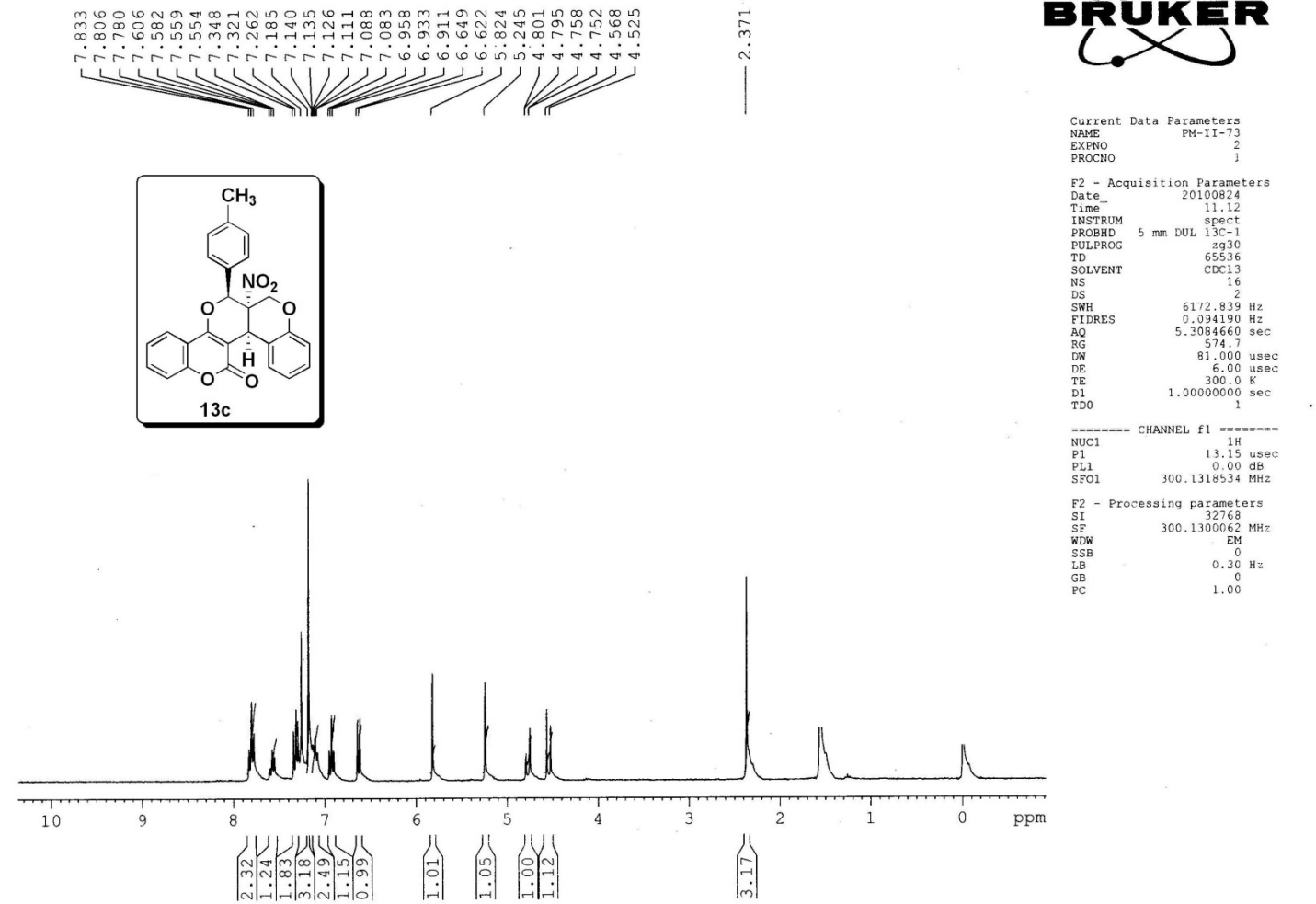
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

======== 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	0
LB	1.00 Hz
GB	0
PC	1.40



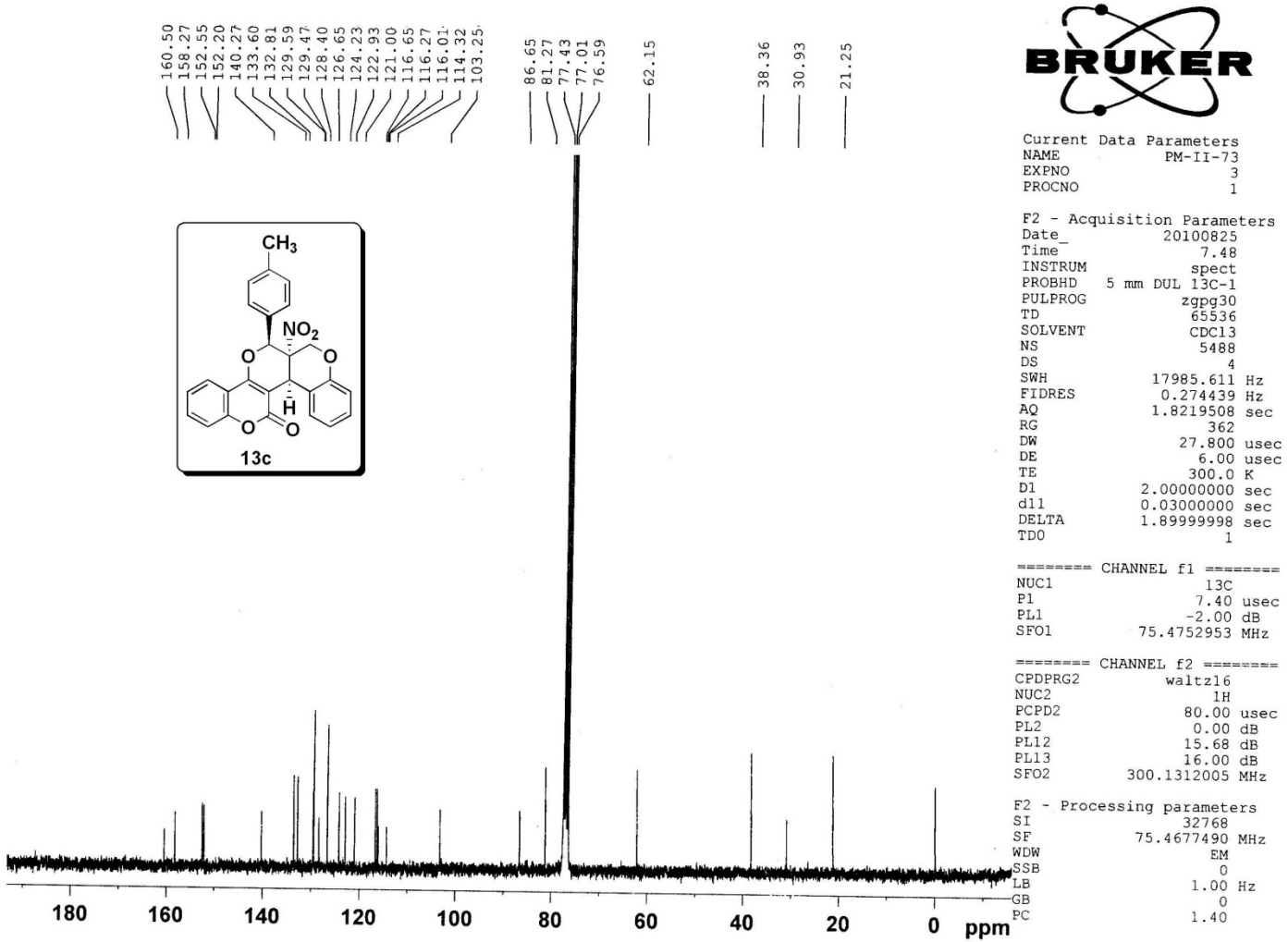
BRUKER

Current Data Parameters
NAME PM-II-73
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100824
Time 11.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 574.7
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters

NAME	PM-II-73
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20100825
Time	7.48
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	5488
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	362
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

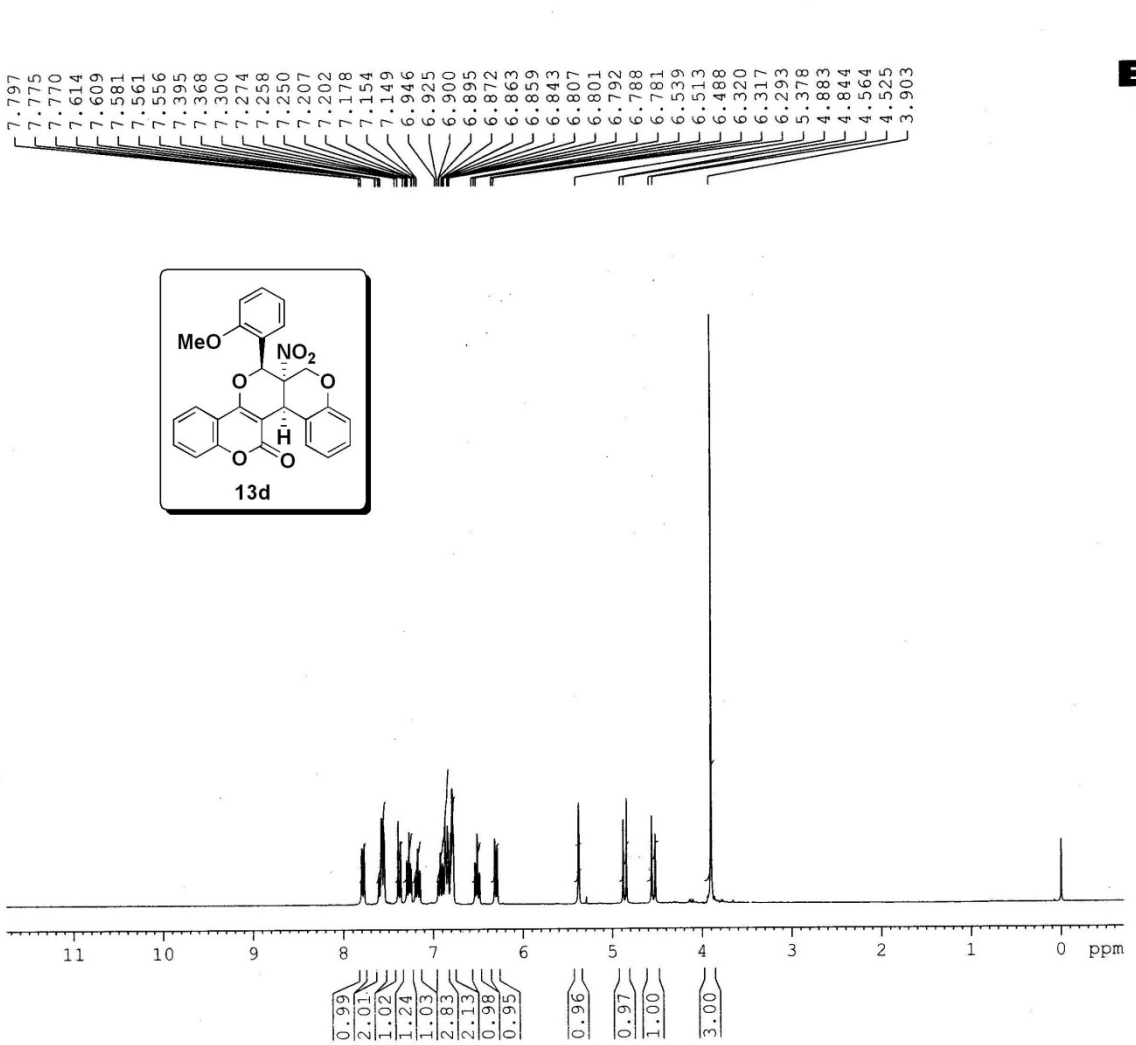
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

======== 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

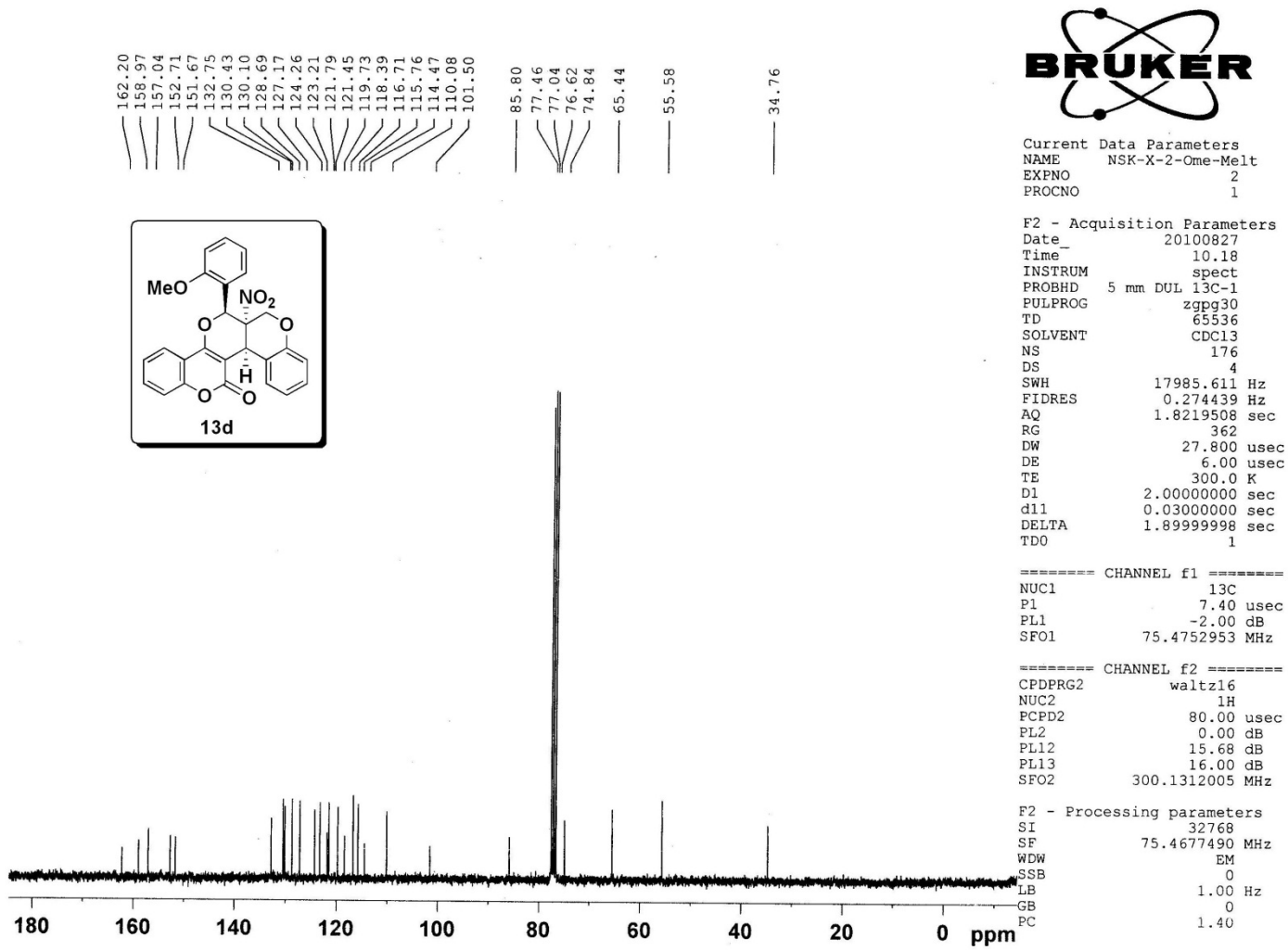
F2 - Processing parameters

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



BRUKER

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

F2 - Acquisition Parameters
Date_             20100827
Time                 10.13
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    128
DW                 81.000 usec
DE                  6.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.15 usec
PL1                 0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300070 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```



BRUKER

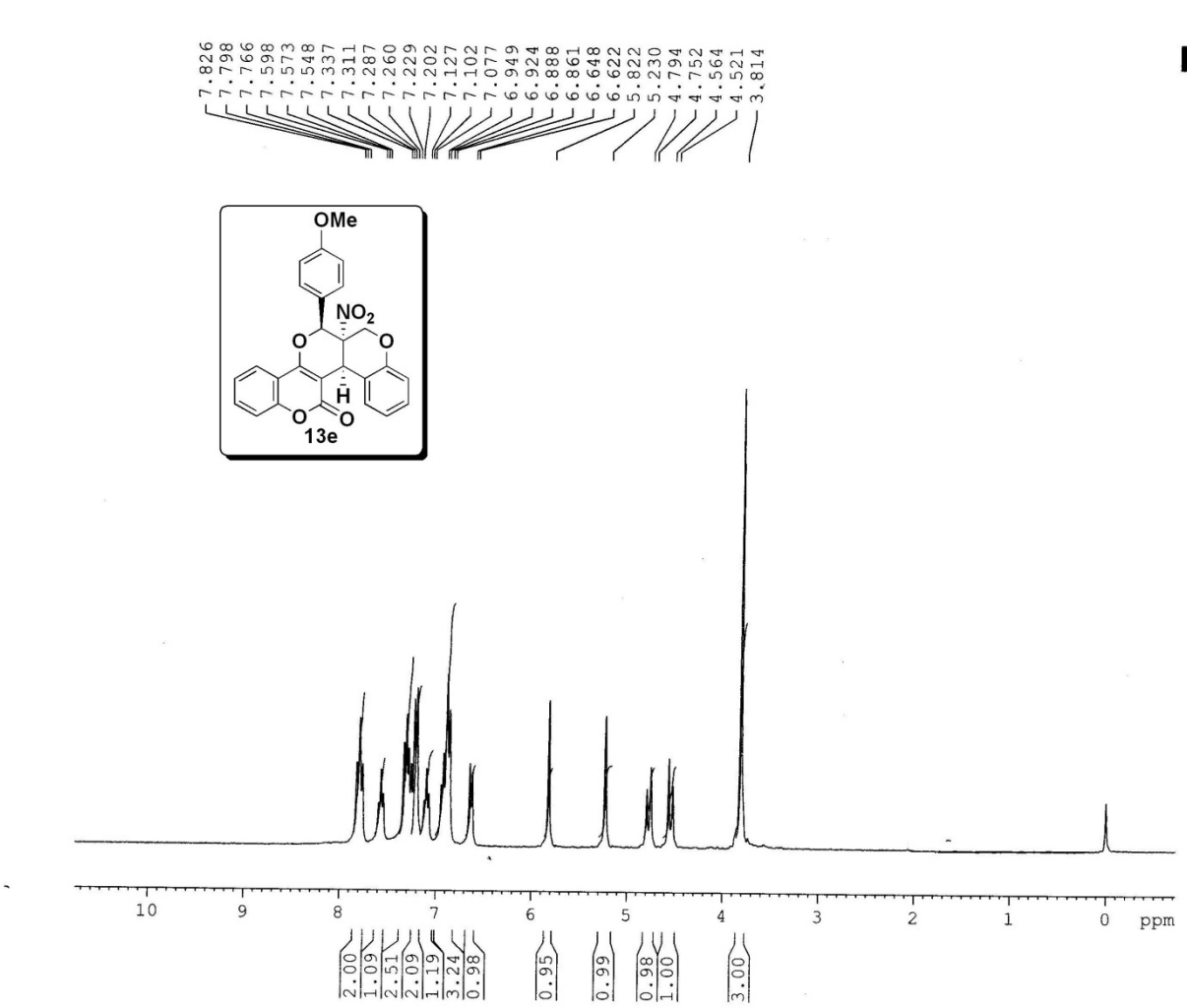
Current Data Parameters
NAME NSK-X-2-Ome-Melt
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100827
Time 10.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 176
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

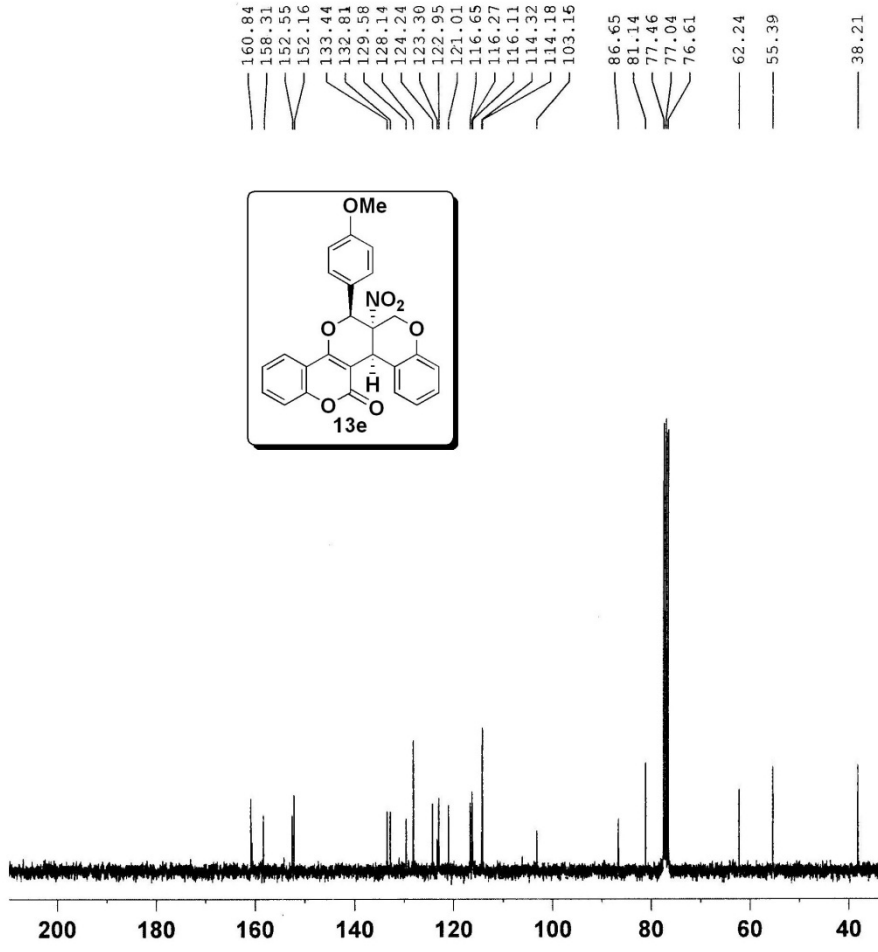
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

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



BRUKER

```
Current Data Parameters
NAME          PM-II-4OME
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100819
Time               14.23
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                     2
DS                     2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                 143.7
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.15 usec
PL1                0.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300065 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



BRUKER

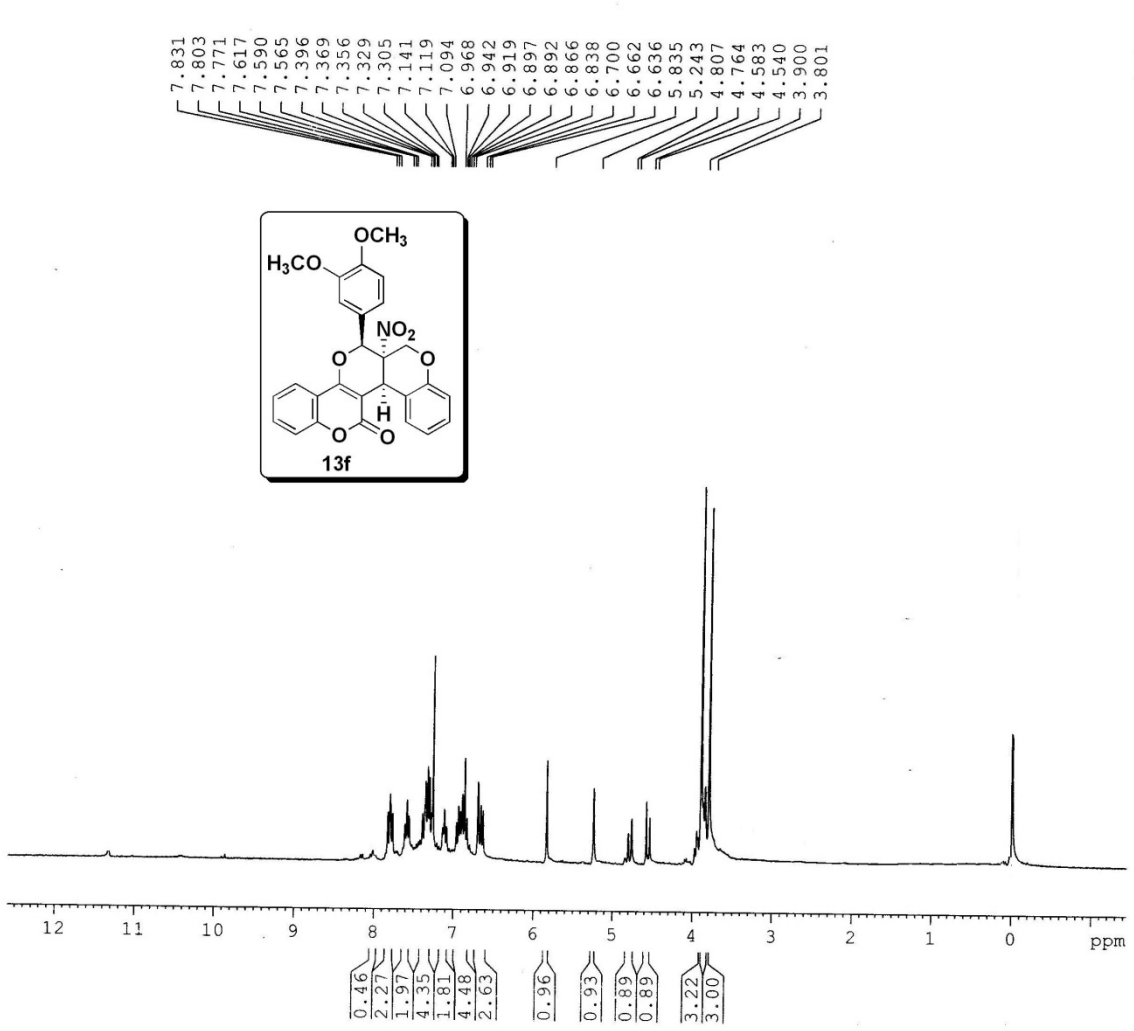
Current Data Parameters
NAME PM-II-4OME
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100819
Time 16.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 152
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40



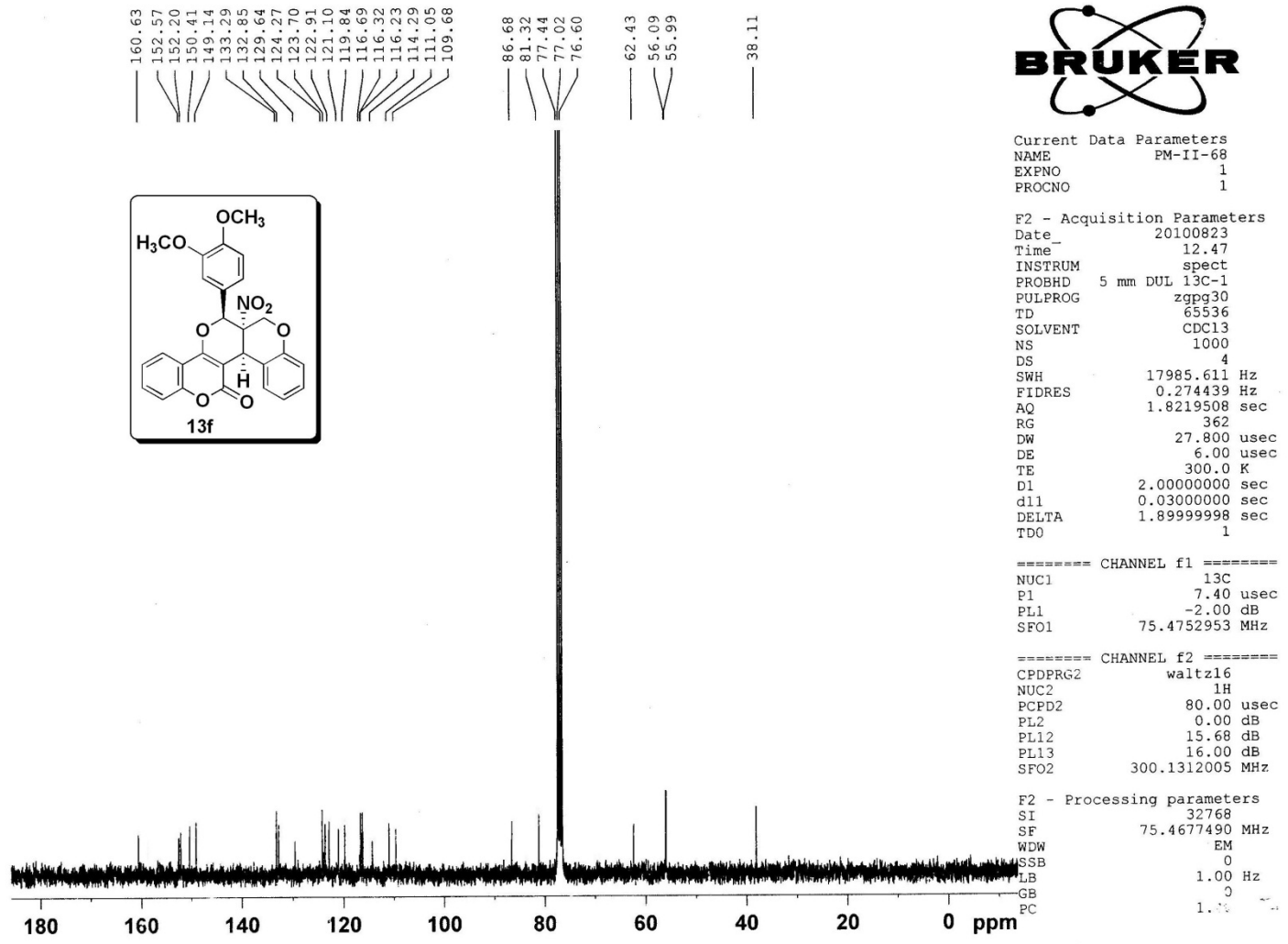
BRUKER

Current Data Parameters
NAME PM-II-68
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100820
Time 15.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

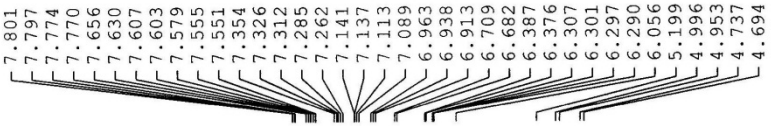
Current Data Parameters
NAME PM-II-68
EXPNO 1
PROCNO 1

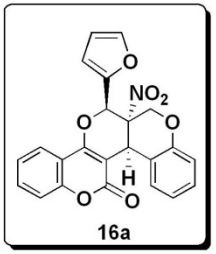
F2 - Acquisition Parameters
Date_ 20100823
Time 12.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.

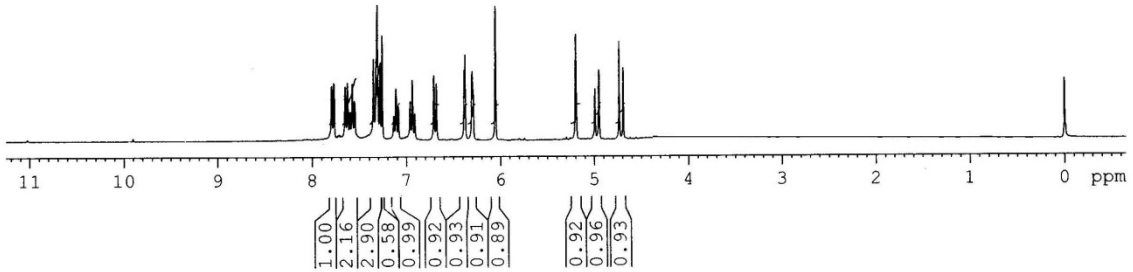


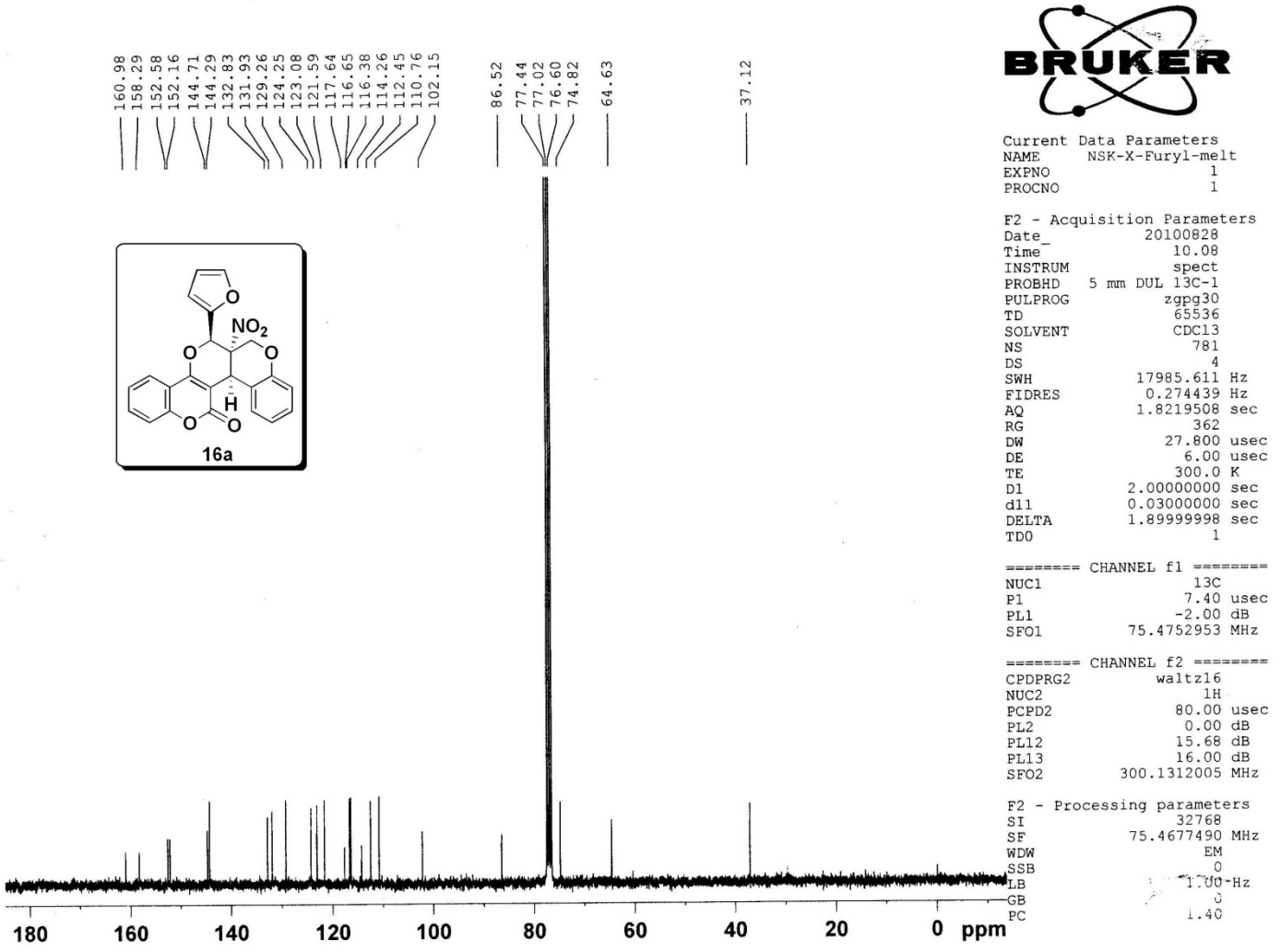


16a

BRUKER

```
Current Data Parameters
NAME      NSK-X-Fufuryl-Melt
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20100827
Time                   10.04
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       13
DS                        2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    228.1
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.15 usec
PL1                    0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF            300.1300059 MHz
WDW                          EM
SSB                           0
LB                     0.30 Hz
GB                            0
PC                     1.00
```





BRUKER

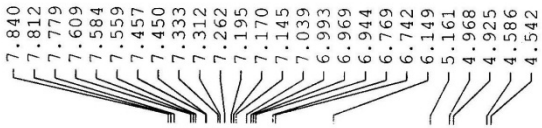
Current Data Parameters
NAME NSK-X-Furyl-melt
EXPNO 1
PROCNO 1

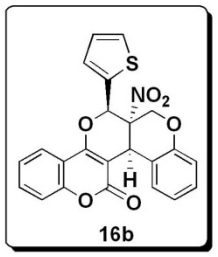
F2 - Acquisition Parameters
Date_ 20100828
Time 10.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 781
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 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 0
LB 1.00 Hz
GB 0
PC 1.40

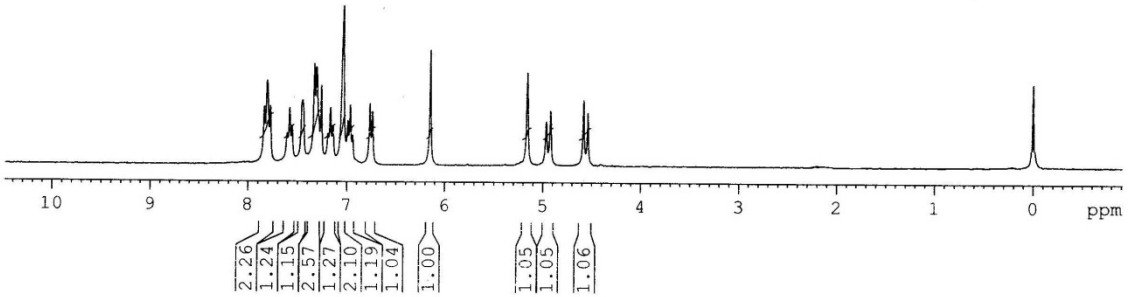


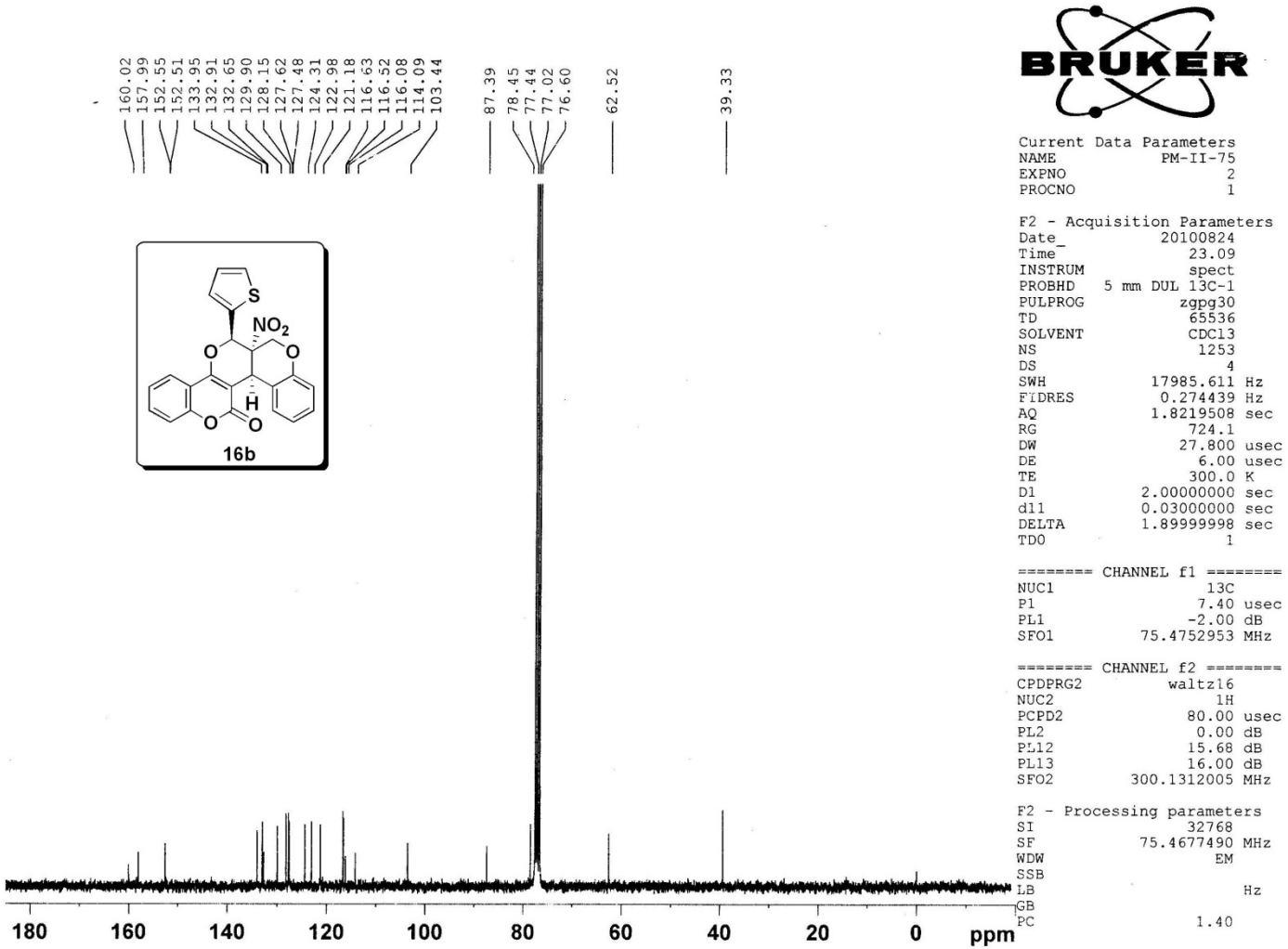


16b

BRUKER

```
Current Data Parameters
NAME                  PM-II-75
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20100824
Time                   16.28
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        13
DS                         2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                     456.1
DW                  81.000 usec
DE                   6.00 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                       1H
P1                  13.15 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

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





BRUKER

Current Data Parameters
NAME PM-II-75
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100824
Time 23.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1253
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 724.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 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
LB Hz
GB
PC 1.40