Electronic Supplementary Material (ESI) for Chemical Communications

Supplementary Information

A multicomponent cascade reaction for the synthesis of novel chromenopyranpyrazole scaffolds

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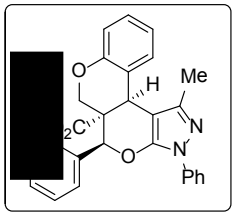
Experimental Section

General Remarks: Melting points were recorded on a Superfit (India) capillary melting point apparatus and were uncorrected. IR spectra were recorded on a Perkin Elmer-FTIR spectrometer using solid samples as KBr plates. For compounds ^{1}H NMR (300 MHz , $CDCl_3$) and^{13}C NMR (75 MHz and 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 Bruker and Jeol mass spectrometer. The X-ray diffraction measurements were carried on a Bruker AXS SMART APEX 2 diffractometer.

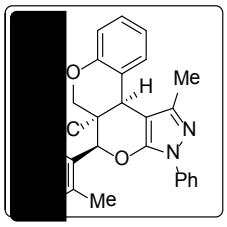
Representative procedure for the synthesis of methyl 16-methyl-11,14-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5a): A mixture of (*E*)-methyl-2-((2-formylphenoxy)methyl)-3-phenylacrylate (**3a**, 1mmol), ethyl/methyl acetoacetate (**1**, 1mmol) and phenyl hydrazine (**2**, 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** as colorless solid.

Methyl 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-pentaene-10-carboxylate (5a):



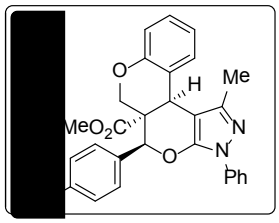
Yield = 95%; reaction time = 1 h; m.p. 246-248°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.09 (s, 3H), 3.43 (s, 3H), 4.35 (d, *J* = 11.4 Hz, 1H), 4.57 (dd, *J* = 1.2, 11.4 Hz, 1H), 4.78 (s, 1H), 5.59 (s, 1H), 6.77-7.74 (m, 14H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.28, 37.17, 48.14, 52.50, 60.63, 84.21, 98.62, 116.81, 118.66, 119.54, 120.23, 125.69, 126.68, 128.62, 129.06, 129.13, 129.31, 132.06, 134.68, 138.35, 147.22, 147.34, 153.04, 171.19; ^{13}C NMR (DEPT 135, 75 MHz, $CDCl_3$) = δ 15.33, 37.16, 52.53, 60.64, 84.21, 116.83, 119.56, 120.21, 125.70, 126.69, 128.64, 129.08, 129.15, 129.33, 132.10; IR (KBr) = ν 1735, 1596, 1510 cm^{-1}; HRMS (ESI) Calcd for $C_{28}H_{24}N_2O_4$ 452.1736; Found: 453.1816 (M^++1).

Methyl 16-methyl-11-(2-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo [8.7.0.0^{2,7}.0^{13,17}] heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5b)



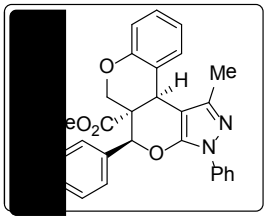
Yield = 92%; reaction time = 1 h; m.p. 258-260°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.12 (s, 3H), 2.39 (s, 3H), 3.47 (s, 3H), 4.31 (d, J = 11.4 Hz, 1H), 4.57 (dd, J = 1.5, 11.4 Hz, 1H), 4.72 (s, 1H), 5.55 (s, 1H), 6.67 - 7.73 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.26, 37.12, 48.09, 52.65, 60.47, 83.43, 98.64, 116.87, 118.45, 119.65, 120.30, 124.79, 125.84, 126.87, 129.11, 129.21, 129.52, 129.94, 132.02, 134.73, 136.68, 138.24, 146.95, 147.21, 152.94, 171.03; IR (KBr) = ν 1737, 1593, 1517 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{26}N_2O_4$ 466.1893; Found 466.1893.

Methyl 16-methyl-11-(4-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo [8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5c)



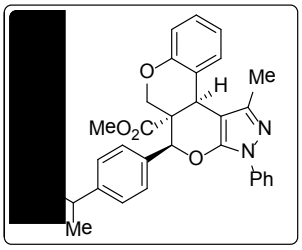
Yield = 95%; reaction time = 1 h; m.p. 264-268°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.11 (s, 3H), 2.38 (s, 3H), 3.47 (s, 3H), 4.31 (d, J = 11.4 Hz, 1H), 4.57 (dd, J = 1.5, 11.4 Hz, 1H), 4.71 (s, 1H), 5.54 (s, 1H), 6.66 - 7.78 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.25, 21.26, 37.17, 48.14, 52.49, 60.71, 84.15, 98.58, 116.81, 118.71, 119.50, 120.19, 125.64, 126.60, 129.04, 129.11, 129.28, 131.68, 132.03, 138.38, 139.20, 147.20, 147.47, 153.08, 171.23; IR (KBr) = ν 1733, 1596, 1512 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{26}N_2O_4$ 466.1893; Found 466.1893.

Methyl 16-methyl-11-(4-ethylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo [8.7.0.0^{2,7}.0^{13,17}] heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5d)



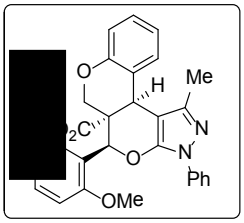
Yield = 92%; reaction time = 1 h; m.p. 221-223°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.26 (t, 3H, J = 7.5 Hz), 2.08 (s, 3H), 2.69 (q, J = 7.5 Hz, 2H), 3.44 (s, 3H), 4.34 (d, J = 11.1 Hz, 1H), 4.58 (dd, 1H, J = 1.2, 11.1 Hz), 4.76 (s, 1H), 5.57 (s, 1H), 6.77-7.75 (m, 13H); ^{13}C NMR (100 MHz, $CDCl_3$) = δ 15.25, 15.40, 28.59, 37.17, 48.18, 52.45, 60.73, 84.16, 98.58, 116.81, 118.74, 119.59, 120.21, 125.63, 126.66, 128.05, 129.03, 129.09, 131.89, 132.02, 138.40, 145.49, 147.19, 147.48, 153.10, 171.23; IR (KBr) = ν 1728, 1585, 1519 cm^{-1}; HRMS (m/z) Calcd for $C_{30}H_{28}N_2O_4$ 480.2047; Found: 480.2047.

Methyl 16-methyl-14-phenyl-11-[4-(propan-2-yl)phenyl]-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5e):



Yield = 94%; reaction time = 1 h; m.p. 226-228°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.26 (d, J = 6.9 Hz, 6H), 2.06 (s, 3H), 2.93 (sep, J = 7.2 Hz, 1H), 3.41 (s, 3H), 4.33 (d, J = 11.1 Hz, 1H), 4.57 (d, J = 11.1 Hz, 1H), 4.74 (s, 1H), 5.56 (s, 1H), 6.76-7.73 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.24, 23.89, 23.94, 33.90, 37.14, 48.20, 52.44, 60.74, 84.15, 98.63, 116.83, 118.75, 119.53, 120.29, 125.71, 126.63, 126.66, 129.06, 129.12, 131.99, 132.04, 138.33, 147.23, 147.52, 150.13, 153.11, 171.21; IR (KBr) = ν 1720, 1582, 1516 cm^{-1}; HRMS (m/z) Calcd for $C_{31}H_{30}N_2O_4$ 494.2206; Found: 494.2206.

Methyl 11-(2-methoxyphenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.02,7.013,17]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5f)



Yield = 93%; reaction time = 1 h; m.p. 232-234°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.14 (s, 3H), 3.42 (s, 3H), 3.78 (s, 3H), 4.38 (d, J = 11.1 Hz, 1H), 4.67 (d, J = 11.1 Hz, 1H), 4.91 (s, 1H), 6.01 (s, 1H), 6.73 - 7.73 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.48, 36.21, 47.54, 52.31, 55.38, 61.96, 98.73, 110.11, 116.77, 119.60, 119.81, 120.17, 120.66, 123.71, 125.52, 127.97, 128.68, 128.99, 130.10, 132.16, 138.45, 147.23, 147.66, 153.01, 156.43, 170.02; IR (KBr) = ν 1716, 1599, 1488 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{26}N_2O_5$ 482.1842; Found: 482.1842.

Crystal data for **methyl 11-(2-methoxyphenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.02,7.013,17]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate 5f**: Empirical formula, $C_{29}H_{26}N_2O_5$; Formula weight, 482.53; crystal color, colorless; Single crystal X-ray structure of the molecule shown in ORTEP diagram (Figure 1). Detailed X-ray crystallographic data is available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (for Chromenopyranpyrazole **5f** CCDC # **780638**).

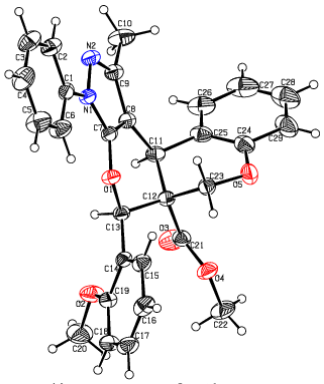
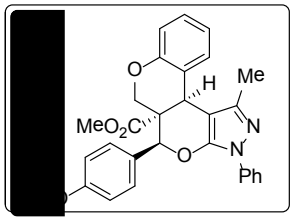


Figure 1. ORTEP diagram of Chromenopyranpyrazole **5f**

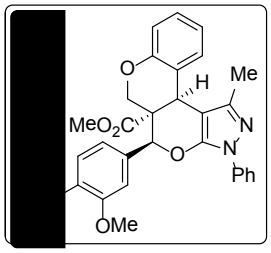
Methyl 11-(4-methoxyphenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo [8.7.0.02,7.013,17]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5g)



Yield = 94%; reaction time = 1 h; m.p. 224-228°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.07 (s, 3H), 3.44 (s, 3H), 3.83 (s, 3H), 4.33 (d, J = 11.4 Hz, 1H), 4.59 (dd, J = 1.8, 11.4 Hz, 1H), 4.74 (s, 1H), 5.54 (s, 1H), 6.77-7.74 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.26, 37.14,

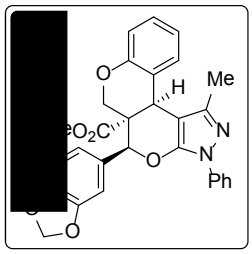
48.21, 52.54, 55.34, 60.71, 84.01, 98.59, 113.98, 116.80, 118.73, 119.51, 120.20, 125.64, 126.69, 128.02, 129.04, 129.11, 132.02, 138.40, 147.21, 147.51, 153.08, 160.21, 171.31; IR (KBr) = ν 1715, 1597, 1490 cm^{-1}; HRMS (ESI) Calcd for $C_{29}H_{26}N_2O_5$ 482.1842; Found: 483.1916 (M^++1).

Methyl 11-(3,4-dimethoxyphenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5h)



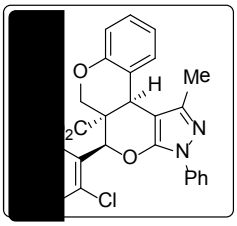
Yield = 95%; reaction time = 1 h; m.p. 192-194°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.07 (s, 3H), 3.45 (s, 3H), 3.88 (s, 3H), 3.91 (s, 3H), 4.34 (d, J = 11.4 Hz, 1H), 4.62 (d, J = 11.4 Hz, 1H), 4.74 (s, 1H), 5.54 (s, 1H), 6.77 - 7.75 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.24, 37.20, 48.20, 52.57, 55.95, 56.08, 60.76, 84.04, 98.58, 109.85, 110.99, 116.80, 118.64, 119.44, 119.56, 120.08, 125.66, 127.02, 129.05, 129.16, 132.01, 138.39, 147.23, 147.42, 148.98, 149.70, 153.00, 171.37; IR (KBr) = ν 1721, 1523, 1497 cm^{-1}; HRMS (m/z) Calcd for $C_{30}H_{28}N_2O_6$ 512.1947; Found: 512.1947.

Methyl 11-(2*H*-1,3-benzodioxol-5-yl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5i)



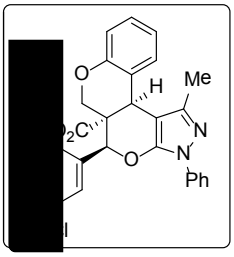
Yield = 93%; reaction time = 1 h; m.p. 194-196°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.07 (s, 3H), 3.47 (s, 3H), 4.31 (d, J = 11.4 Hz, 1H), 4.61 (dd, J = 1.5 Hz, J = 11.1 Hz, 1H), 4.73 (s, 1H), 5.50 (s, 1H), 6.01 (s, 2H), 6.74 - 7.72 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.25, 37.12, 48.24, 52.60, 60.68, 84.06, 98.59, 101.49, 107.03, 108.22, 116.82, 118.62, 119.53, 120.23, 120.60, 125.71, 128.33, 129.07, 129.15, 132.00, 138.33, 147.18, 147.31, 148.05, 148.32, 153.02, 171.20; IR (KBr) = ν 1718, 1529, 1493 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{24}N_2O_6$ 496.1633; Found: 496.1633.

Methyl 16-methyl-11-(2-chlorophenyl)-14-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5j)



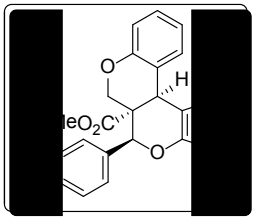
Yield = 95%; reaction time = 1 h; m.p. 230-232°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.16 (s, 3H), 3.49 (s, 3H), 4.45 (d, J = 10.8 Hz, 1H), 4.73 (d, J = 10.5 Hz, 1H), 4.94 (s, 1H), 6.10 (s, 1H), 6.73 - 7.70 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.44, 36.20, 47.65, 52.93, 61.94, 79.73, 98.85, 116.78, 119.63, 119.85, 120.28, 125.73, 127.03, 128.80, 129.05, 129.66, 130.30, 132.04, 133.16, 138.31, 147.25, 152.77, 169.66; IR (KBr) = ν 1735, 1596, 1517 cm^{-1}; HRMS (m/z) Calcd for $C_{28}H_{23}ClN_2O_4$ 486.1346; Found: 486.1345.

Methyl 16-methyl-11-(3-chlorophenyl)-14-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5k)



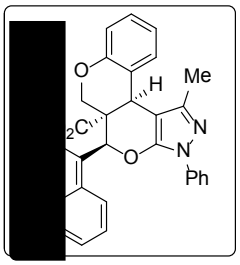
Yield = 92%; reaction time = 1 h; m.p. 238-240°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.09 (s, 3H), 3.48 (s, 3H), 4.32 (d, J = 11.4 Hz, 1H), 4.53 (dd, J = 1.8, 11.4 Hz, 1H), 4.77 (s, 1H), 5.55 (s, 1H), 6.77 - 7.72 (m, 13H); ^{13}C NMR (100 MHz, $CDCl_3$) = δ 15.26, 37.12, 48.24, 52.62, 60.67, 84.07, 98.59, 101.49, 107.03, 108.22, 116.83, 118.60, 119.53, 120.25, 120.60, 125.72, 128.32, 129.08, 129.15, 132.01, 147.20, 147.30, 148.05, 148.32, 153.01, 171.21; IR (KBr) = ν 1738, 1602, 1518 cm^{-1}; HRMS (m/z) Calcd for $C_{28}H_{23}ClN_2O_4$ 486.1346; Found: 486.1346.

Methyl 16-methyl-11-(4-chlorophenyl)-14-diphenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5l)



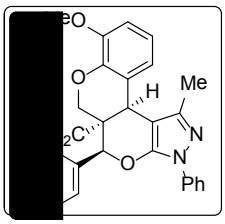
Yield = 94%; Reaction time = 1 h; m.p. 246-248°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.00 (s, 3H), 3.39 (s, 3H), 4.25 (d, J = 10.8 Hz, 1H), 4.44 (d, J = 10.8 Hz, 1H), 4.68 (s, 1H), 5.50 (s, 1H), 6.70 - 7.63 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.18, 37.21, 48.08, 52.62, 60.53, 83.52, 98.60, 116.85, 119.64, 120.29, 125.84, 128.07, 128.89, 129.07, 129.21, 129.23, 131.94, 131.97, 133.22, 138.24, 147.09, 147.23, 152.96, 171.11 ppm; IR (KBr) = ν 1734, 1598, 1516 cm^{-1}; HRMS (m/z) Calcd for $C_{28}H_{23}ClN_2O_4$ 486.1346; Found: 486.1345..

Methyl 16-methyl-11-(naphthalen-1-yl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo [8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5m)



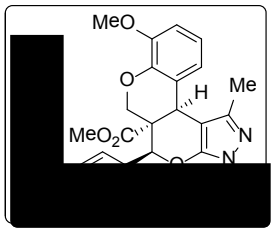
Yield = 95%; reaction time = 1 h; m.p. 234-236°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.17 (s, 3H), 2.82 (s, 3H), 4.51 (d, J = 10.8 Hz, 1H), 4.76 (d, J = 10.5 Hz, 1H), 5.03 (s, 1H), 6.43 (s, 1H), 6.74 - 7.93 (m, 16H); ^{13}C NMR (100 MHz, $CDCl_3$) = δ 15.55, 37.02, 47.95, 52.31, 61.65, 99.02, 116.83, 119.29, 119.70, 120.17, 122.51, 125.13, 125.55, 125.66, 125.88, 126.56, 128.94, 129.06, 129.82, 130.55, 130.77, 132.29, 133.57, 138.39, 147.33, 147.71, 152.95, 170.44; IR (KBr) = ν 1735, 1595, 1512 cm^{-1}; HRMS (m/z) Calcd for $C_{32}H_{26}N_2O_4$: 502.1893; Found: 502.1892.

Methyl 6-methoxy-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-pentaene-10-carboxylate (5n)



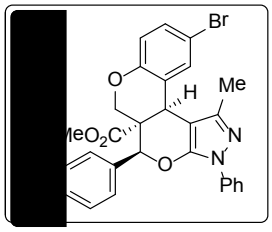
Yield = 93%; reaction time = 1 h; m.p. 242-244°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.08 (s, 3H), 3.41 (s, 3H), 3.81 (s, 3H), 4.39 (d, J = 11.1 Hz, 1H), 4.72 – 4.79 (m, 2H), 5.58 (s, 1H), 6.76-7.70 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.37, 37.06, 48.19, 52.59, 56.02, 61.19, 84.35, 98.70, 111.07, 119.23, 119.68, 120.40, 124.06, 125.80, 126.80, 128.67, 129.15, 129.43, 134.76, 138.46, 142.70, 147.29, 147.46, 148.43, 171.26; IR (KBr) = ν 1716, 1592, 1485 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{26}N_2O_5$ 482.1842; Found: 482.1842.

Methyl 6-methoxy-11-(4-methoxyphenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}] heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5o)



Yield = 95%; reaction time = 1 h; m.p. 248-250°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.06 (s, 3H), 3.42 (s, 3H), 3.81 (s, 3H), 3.83 (s, 3H), 4.38 (d, J = 11.7 Hz, 1H), 4.74 – 4.78 (m , 2H), 5.54 (s, 1H), 6.75 - 7.73 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) = δ 15.23, 36.93, 48.16, 52.50, 55.32, 55.92, 61.16, 84.04, 98.54, 110.96, 113.92, 119.07, 119.65, 120.26, 123.92, 125.63, 126.68, 128.03, 129.01, 138.39, 142.62, 147.16, 147.52, 148.32, 160.23, 171.27; IR (KBr) = ν 1721, 1596, 1483 cm^{-1}; HRMS (m/z) Calcd for $C_{30}H_{28}N_2O_6$ 512.1947; Found: 512.1947.

Methyl 4-bromo-11-(4-methoxyphenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}] heptadeca-2,4,6,13(17),15-pentaene-10-carboxylate (5p)

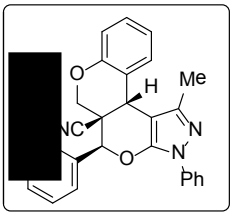


Yield = 93%; reaction time = 1 h; m.p. 268-270°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.11 (s, 3H), 2.38 (s, 3H), 3.47 (s, 3H), 4.30 (d, J = 11.4 Hz, 1H), 4.57 (dd, J = 1.5, 11.4 Hz, 1H), 4.71 (s, 1H), 5.54 (s, 1H), 6.66 - 7.74 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.28, 21.26, 36.97, 47.85, 52.64, 60.96, 84.01, 97.94, 111.30, 118.73, 120.19, 120.94, 125.75, 126.56, 129.06, 129.34, 131.44, 131.94, 134.26, 138.29, 139.33, 146.95, 147.45, 152.28,

170.89; IR (KBr) = ν 1738, 1626, 1522 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{25}BrN_2O_4$: 544.0997; Found: 544.0997.

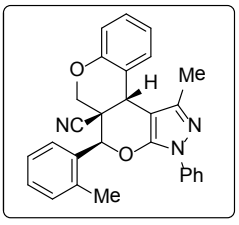
Representative procedure for the synthesis of 16-methyl-11,14-diphenyl-8,12-dioxa-14,15-diazatetracyclo [8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile 8a: A mixture of (*E*)-2-((2-formylphenoxy)methyl)-3-phenylacrylonitrile (**6a**, 1mmol), ethyl/methyl acetoacetate (**1**, 1mmol) and phenyl hydrazine (**2**, 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 **8a** as colorless solid.

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-pentaene-10-carbonitrile (8a)



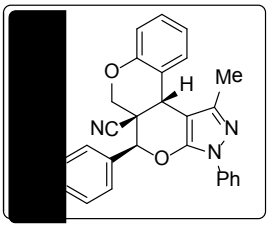
Yield = 94%; reaction time = 1 h; m.p. 220-224°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.59 (s, 3H), 4.11 (d, J = 11.4 Hz, 1H), 4.25 (d, J = 11.4 Hz, 1H), 4.56 (s, 1H), 5.33 (s, 1H), 6.95 - 7.76 (m, 14H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 14.03, 36.55, 39.31, 66.99, 77.93, 97.40, 116.96, 117.38, 120.44, 122.40, 122.77, 125.99, 127.73, 128.91, 129.01, 129.08, 129.92, 130.03, 133.53, 138.27, 146.64, 148.66, 150.99; ^{13}C NMR (DEPT 135, 75 MHz, $CDCl_3$) = δ 14.03, 36.55, 66.99, 77.93, 117.38, 120.45, 122.78, 126.00, 127.74, 128.92, 129.02, 129.08, 129.92, 130.03 ;IR (KBr) = ν 2242, 1597, 1514 cm^{-1}; HRMS (ESI) Calcd for $C_{27}H_{21}N_3O_2$ 419.1634; Found: 420.1713 (M^++1).

16-methyl-11-(2-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8b)



Yield = 91%; reaction time = 1 h; m.p. 254-256°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.29 (s, 3H), 2.59 (s, 3H), 4.05 (d, *J* = 11.4 Hz, 1H), 4.32 (d, *J* = 11.7 Hz, 1H), 4.56 (s, 1H), 5.52 (s, 1H), 6.87 - 8.02 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 13.94, 18.94, 37.12, 39.47, 67.13, 74.39, 97.37, 117.10, 117.84, 120.47, 122.06, 122.63, 125.90, 126.86, 127.70, 129.00, 129.06, 129.84, 129.91, 131.04, 131.88, 137.66, 138.29, 146.73, 149.17, 150.52; IR (KBr) = ν 2249, 1590, 1517 cm^{-1}; HRMS (m/z) Calcd for $C_{28}H_{23}N_3O_2$ 433.1790; Found: 433.1790.

16-methyl-11-(4-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8c)



Yield = 92%; reaction time = 1 h; m.p. 208-210°C; ^{1}H NMR (300 MHz, $CDCl_3$) = *δ* 2.43 (s, 3H), 2.58 (s, 3H), 4.12 (d, *J* = 11.4 Hz, 1H), 4.25 (d, *J* = 11.4 Hz, 1H), 4.55 (s, 1H), 5.30 (s, 1H), 6.94 - 7.76 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = *δ* 13.99, 21.34, 36.54, 39.35, 67.06, 77.89, 97.36, 117.08, 117.35, 120.40, 122.44, 122.71, 125.91, 127.62, 128.95, 129.03, 129.56, 129.90, 130.57, 138.31, 140.05, 146.61, 148.77, 151.04; IR (KBr) = ν 2233, 1599, 1518 cm^{-1}; HRMS (ESI) Calcd for $C_{28}H_{23}N_3O_2$ 433.1790; Found: 434.1864 (M^++1).

Crystal data for 16-methyl-11-(4-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8c): Empirical formula, $C_{28}H_{23}N_3O_2$; Formula weight, 433.50; crystal color, colorless; Single crystal X-ray structure of the molecule shown in ORTEP diagram (Figure 2). Detailed X-ray crystallographic data is available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (for Chromenopyranpyrazole **8c** CCDC # **780639**).

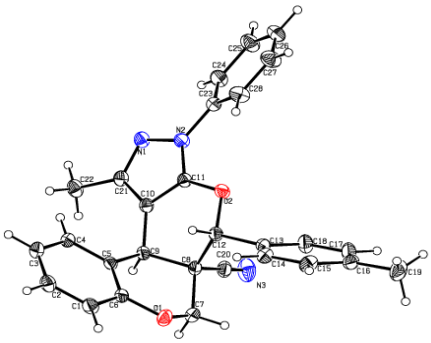
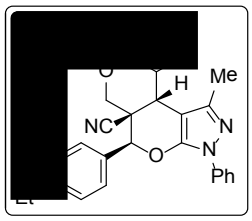


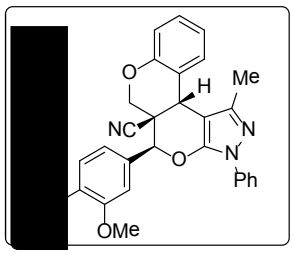
Figure 2. ORTEP diagram of Chromenopyranpyrazole 8**c**

16-methyl-11-(4-ethylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8d)



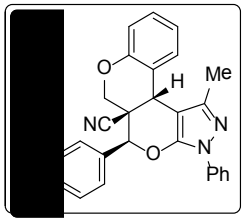
Yield = 94%; Reaction time = 1 h; m.p. 228-230°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.29 (t, 3H, J = 7.8 Hz), 2.58 (s, 3H), 2.72 (q, J = 7.5 Hz, 2H), 4.12 (d, J = 11.4 Hz, 1H), 4.24 (d, J = 11.4 Hz, 1H), 4.55 (s, 1H), 5.30 (s, 1H), 6.94 - 7.76 (m, 13 H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 14.02, 15.39, 28.71, 36.50, 39.35, 67.04, 77.91, 97.39, 117.12, 117.37, 120.42, 122.43, 122.72, 125.94, 127.69, 128.39, 128.97, 129.06, 129.91, 130.76, 138.29, 146.27, 146.63, 148.78, 151.03; IR (KBr) = ν 2247, 1585, 1528 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{25}N_3O_2$: 447.1947; Found: 447.1946.

16-methyl-11-(3,4-dimethoxyphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8e)



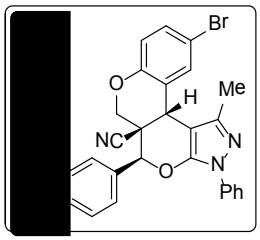
Yield = 95%; reaction time = 1 h; m.p. 214-216°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.59 (s, 3H), 3.93 (s, 3H), 3.95 (s, 3H), 4.13 (d, J = 11.4 Hz, 1H), 4.26 (d, J = 11.4 Hz, 1H), 4.56 (s, 1H), 5.28 (s, 1H), 6.94 - 7.78 (m, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 13.99, 36.49, 39.35, 55.98, 56.10, 67.10, 77.43, 97.39, 110.40, 111.06, 117.31, 120.26, 120.67, 122.45, 122.73, 125.78, 125.89, 128.95, 129.03, 129.90, 138.35, 146.62, 148.79, 149.27, 150.33, 151.02; IR (KBr) = ν 2236, 1589, 1527 cm^{-1}; HRMS (m/z) Calcd for $C_{29}H_{25}N_3O_4$ 479.1845; Found: 479.1844.

16-methyl-11-(4-chlorophenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8f)



Yield = 92%; reaction time = 1 h; m.p. 236-238°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.58 (s, 3H), 4.08 (d, J = 11.4 Hz, 1H), 4.24 (d, J = 11.4 Hz, 1H), 4.55 (s, 1H), 5.31 (s, 1H), 6.94 – 7.73 (m, 13 H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 14.04, 36.48, 39.22, 66.89, 77.55, 97.43, 116.71, 117.42, 120.42, 120.43, 122.31, 122.92, 126.10, 129.12, 129.22, 129.92, 132.00, 136.12, 138.18, 146.64, 148.37, 150.87; IR (KBr) = ν 2225, 1597, 1514 cm^{-1}; HRMS (m/z) Calcd for $C_{27}H_{20}ClN_3O_2$: Calculated 453.1244; Found: 453.1244.

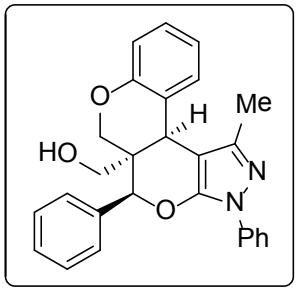
4-bromo-16-methyl-11-(4-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaene-10-carbonitrile (8g)



Yield = 93%; reaction time = 1 h; m.p. 238-240°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.43 (s, 3H), 2.58 (s, 3H), 4.12 (d, J = 11.4 Hz, 1H), 4.23 (d, J = 11.4 Hz, 1H), 4.52 (s, 1H), 5.24 (s, 1H), 6.84 – 7.76 (m, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$) = δ 13.98, 21.34, 36.36, 39.10, 67.13, 77.93, 96.73, 114.89, 116.72, 119.17, 120.46, 120.58, 124.49, 126.06, 127.58, 129.06, 129.63, 130.28, 131.99, 132.67, 138.20, 140.22, 146.43, 150.17; IR (KBr) = ν 2242, 1595, 1516 cm^{-1}; HRMS (m/z) Calcd for $C_{28}H_{22}BrN_3O_2$ 511.0895; Found: 511.0895.

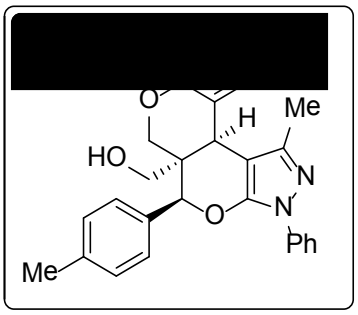
A representative procedure for the synthesis of {16-methyl-11,14-diphenyl-8,12-dioxa-14,15diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-10-yl}methanol (9a): A suspension of lithium aluminium hydride (0.03 g, 0.88 mmol) in dry THF (5 mL) was cooled at 0°C. To this suspension was added drop wise solution of the chromenopyranpyrazole **5a** (0.20 g, 0.44 mmol) in dry THF (5 mL). The resulting mixture was allowed to stirr at room temperature for 0.5 h. After the completion of reaction, as indicated by TLC, the reaction mixture was quenched with methanol and the separated precipitate was filtered through celite. The filtrate was evaporated under reduced pressure and the colurless solid (0.17 g) thus obtained was taken for next step without purification.

{16-Methyl-11,14-diphenyl-8,12-dioxa-14,15diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-10-yl}methanol (9a)



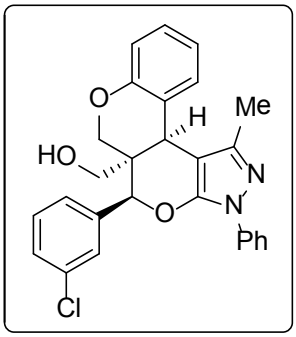
Yield = 91%; reaction time = 0.5 h; m.p. 190-191°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.71 (bs, 1H), 2.05 (s, 3H), 3.25 (d, J = 11.4Hz, 1H), 3.51 (d, J = 11.1 Hz, 1H), 3.84 (d, J = 11.4Hz, 1H), 4.29 – 4.34 (m, 1H), 5.54 (s, 1H), 6.66 – 7.65 (m, 14H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.56, 33.77, 38.49, 61.62, 62.47, 83.94, 98.98, 116.66, 119.64, 120.07, 120.24, 125.48, 127.60, 128.45, 128.68, 128.75, 128.57, 132.82, 135.62, 135.45, 147.56, 147.95, 152.94; ^{13}C NMR (DEPT 135, 75 MHz, $CDCl_3$) = δ 15.60, 33.71, 61.57, 62.45, 83.92, 116.66, 119.65, 120.25, 125.51, 127.60, 128.47, 128.69, 128.76, 128.99, 132.85; IR (KBr) = ν 1358, 1597, 3448 cm^{-1}; MS (m/z): 425 (M^++1); Elemental Analysis for $C_{27}H_{24}N_2O_3$: Calculated: C, 76.39; H, 5.70; N, 6.60; Found: C, 76.45; H, 5.63; N, 6.71.

[16-methyl-11-(4-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-10-yl]methanol (9b)



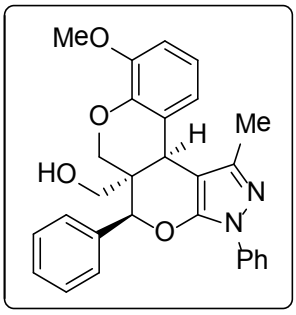
Yield = 93%; reaction time = 0.5 h; m.p. 195-197°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.82 (bs, 1H), 2.03 (s, 3H), 2.32 (s, 3H), 3.23 (d, J = 11.4 Hz, 1H), 3.48 (d, J = 11.4 Hz, 1H), 3.84 (d, J = 11.4 Hz, 1H), 4.26-4.30 (m, 2H), 6.65-7.63 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.56,21.24, 33.77, 38.49, 61.61, 62.48, 83.98, 99.00, 116.64, 119.60, 120.10, 120.23, 125.45,127.50, 128.64, 128.96, 129.16, 132.61, 132.84, 138.44, 138.61, 147.58, 148.07, 152.97; IR (KBr) = ν 1356, 1594, 3452 cm^{-1}; MS (m/z): 439 (M^++1); Elemental Analysis for $C_{28}H_{26}N_2O_3$: Calculated: C, 76.69; H, 5.98; N, 6.39; Found: C, 76.74; H, 5.87; N, 6.45.

[11-(3-chlorophenyl)-16-methyl-14-phenyl-8,12-dioxa-14,15-diazatetracyclo[8.7.0.$0^{2,7}$.$0^{13,17}$]heptadeca-2,4,6,13(17),15-pentaen-10-yl]methanol (9c)



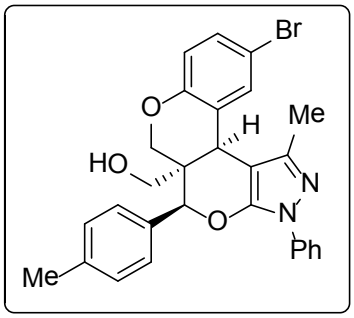
Yield = 90%; reaction time = 0.5 h; m.p. 192-193°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.90 (bs, 1H), 2.05 (s, 3H), 3.23 (d, J = 11.1 Hz, 1H), 3.52 (d, J = 10.8Hz, 1H), 3.78 (d, J = 11.4Hz, 1H), 4.28 (d, J = 11.4 Hz, 1H), 4.36 (s, 1H), 4.53 (s, 1H), 6.66-7.59 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.55, 33.61, 38.43, 61.51, 62.49, 83.07, 99.03, 116.69, 119.75, 119.95, 120.31, 125.63, 125.77, 127.59, 127.79, 128.74, 128.91, 129.02, 129.67, 132.78, 134.45, 137.63, 138.33, 147.58, 152.80; IR (KBr) = ν 1351, 1589, 3443 cm^{-1}; MS (m/z): 459 (M^{+}+1); Elemental Analysis for $C_{27}H_{23}ClN_2O_3$: Calculated: C, 70.66; H, 5.05; N, 6.10; Found: C, 70.71; H, 5.00; N, 6.18.

[6-methoxy-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-10-yl]methanol (9d)



Yield = 93%; reaction time = 0.5 h; m.p. 194-195°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 1.79 (bs, 1H), 2.05 (s, 3H), 3.23 (d, J = 11.1 Hz, 1H), 3.52 (d, J = 11.1 Hz, 1H), 3.73 (s, 3H), 3.97 (d, J = 11.4 Hz, 1H), 4.33-4.38 (m, 2H), 5.56 (s, 1H), 6.69-7.64 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.62, 33.52, 38.33, 55.89, 61.63, 62.97, 83.85, 98.95, 110.47, 119.22, 120.27, 120.92, 124.73, 125.49, 127.57, 128.39, 128.76, 128.97, 135.51, 138.42, 142.24, 147.53, 147.95, 148.19; IR (KBr) = ν 1349, 1586, 3456 cm^{-1}; MS (m/z): 455 (M^{+}+1); Elemental Analysis for $C_{28}H_{26}N_2O_4$: Calculated: C, 73.99; H, 5.77; N, 6.16; Found: C, 73.88; H, 5.86; N, 6.23.

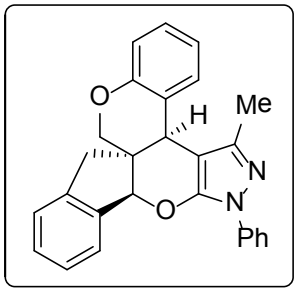
[4-bromo-16-methyl-11-(4-methylphenyl)-14-phenyl-8,12-dioxa-14,15-diazatetra cyclo[8.7.0.0^{2,7}.0^{13,17}]heptadeca-2,4,6,13(17),15-pentaen-10-yl]methanol (9e)



Yield = 90%; reaction time = 0.5 h; m.p. 194-196°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.24 (bs, 1H) 2.07 (s, 3H), 2.34 (s, 3H), 3.29 (d, J = 11.4 Hz, 1H), 3.54 (d, J = 11.4 Hz, 1H), 3.88 (d, J = 11.4 Hz, 1H), 4.29-4.33 (m, 2H), 5.52 (s, 1H), 6.68-7.66 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.60, 21.23, 33.88, 38.53, 60.39, 61.77, 84.02, 89.95, 116.66, 118.55, 119.61, 120.13, 125.37, 127.49, 128.66, 128.95, 129.19, 132.62, 132.83, 1338.58, 138.67, 147.52, 147.99, 152.92; IR (KBr) = ν 1361, 1593, 3449 cm^{-1}; MS (m/z): 518 (M^++1); Elemental Analysis for $C_{28}H_{25}BrN_2O_3$: Calculated: C, 65.00; H, 4.87; N, 5.41; Found: C, 65.04; H, 4.82; N, 5.46.

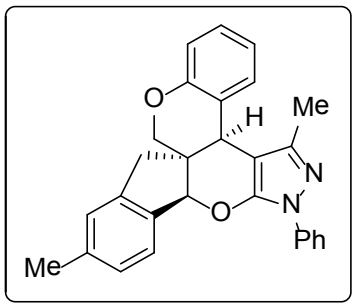
A representative procedure for the synthesis of 14-methyl-12-phenyl-10,23-dioxa-12,13-diazahexacyclo[14.8.0.0^{1,9}.0^{3,8}.0^{11,15}.0^{17,22}]tetracosa-3(8),4,6,11(15),13,17,19,21-octaene (10a): To a solution of chromenopyranpyrazole alcohol **9a** (0.10 g, 0.22 mmol) in dichloroethane (4ml), trifluoroacetic acid (15 equivalent) was added at room temperature and the resulting solution was kept under reflux condition for 15 h. After the completion of reaction as indicated by TLC, a saturated solution of sodium hydrogen carbonate (5 mL) was added slowly to the mixture and extrated using ethyl acetate. The organic layer was dried over Na_2SO_4 and the crude product thus obtained was purified using column chromatography to afford the desired product **10a** as a colourless solid.

14-methyl-12-phenyl-10,23-dioxa-12,13diazahexacyclo[14.8.0.0^{1,9}.0^{3,8}.0^{11,15}.0^{17,22}] tetra cosa-3(8),4,6,11(15),13,17,19,21-octaene (10a)



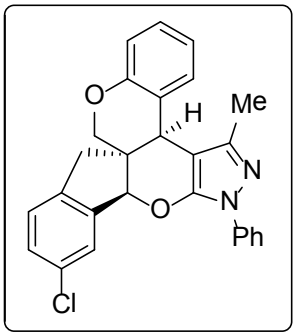
Yield = 85%; reaction time = 15 h; m.p. 220-222°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.09 (s, 3H), 3.83 (d, J = 11.7 Hz, 1H), 4.10 (d, J = 11.1 Hz, 1H), 4.21 (s, 1H), 4.29 (d, J = 11.7 Hz, 1H), 4.46 (d, J = 11.7 Hz, 1H), 5.46 (s, 1H), 6.72 – 7.64 (m, 13 H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.54, 34.93, 37.48, 61.57, 66.84, 83.92, 98.55, 116.97, 118.74, 120.35, 125.77, 127.29, 127.59, 128.98, 129.03, 129.29, 129.55, 132.70, 134.35, 138.21, 147.37, 147.62, 152.36; ^{13}C NMR (DEPT 135, 75 MHz, $CDCl_3$) = δ 15.55, 34.92, 61.55, 66.84, 83.91, 116.97, 120.35, 125.78, 127.29, 127.59, 128.46, 128.98, 129.04, 129.29, 129.55, 132.71; IR (KBr) = ν 1355, 1594, cm^{-1}; HRMS (m/z) Calcd for $C_{27}H_{22}N_2O_2$: 406.1681; Found: 406.1681.

5,14-dimethyl-12-phenyl-10,23-dioxa-12,13-diazahexacyclo[14.8.0.0^{1,9}.0^{3,8}.0^{11,15}.0^{17,22}] tetracosa-3(8),4,6,11(15),13,17,19,21-octaene (10b)



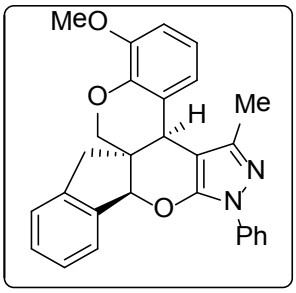
Yield = 80%; reaction time = 15 h; m.p. 225-227°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.08 (s, 3H), 2.34 (s, 3H), 3.83 (d, J = 11.7 Hz, 1H), 4.11 (d, J = 11.7 Hz, 1H), 4.21 (s, 1H), 4.27 (d, J = 12 Hz, 1H), 4.44 (d, J = 11.4 Hz, 1H), 5.42 (s, 1H), 6.72-7.64 (m, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.62, 21.19, 34.91, 37.43, 61.61, 66.94, 83.84, 98.54, 116.95, 118.81, 120.23, 125.65, 127.18, 129.01, 129.24, 129.63, 131.31, 132.74, 138.31, 139.59, 147.35, 147.71, 152.38; IR (KBr) = ν 1357, 1599 cm^{-1}; MS (m/z): 421 (M^++1); Elemental Analysis for $C_{28}H_{24}N_2O_2$: Calculated: C, 79.98; H, 5.75; N, 6.66; Found: C, 79.89; H, 5.66; N, 6.72.

6-chloro-14-methyl-12-phenyl-10,23-dioxa-12,13-diazahexacyclo[14.8.0.0^{1,9}.0^{3,8}.0^{11,15}. 0^{17,22}]tetracosa-3(8),4,6,11(15),13,17,19,21-octaene (10c)



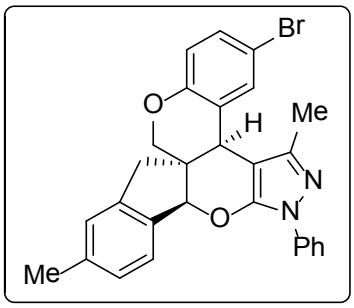
Yield = 83%; reaction time = 15 h; m.p. 228-230°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.09 (s, 3H), 3.83 (dd, J = 1.5, 11.7 Hz, 1H), 4.19 (s, 1H), 4.31 (d, J = 12 Hz, 1H), 4.43 (d, J = 11.7 Hz, 1H), 5.41 (s, 1H), 6.73-7.61 (m, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.58, 34.98, 37.53, 61.43, 66.71, 83.30, 98.55, 117.03, 118.55, 120.25, 120.37, 125.45, 125.59, 129.89, 127.52, 129.03, 129.09, 129.37, 129.79, 130.24, 132.66, 135.17, 136.36, 147.35, 152.24; 1357, 1599; MS (m/z): 441 (M^++1); Elemental Analysis for $C_{27}H_{22}N_2O_2$: Calculated: C, 73.55; H, 4.80; N, 6.35; Found: C, 73.62; H, 4.75; N, 6.44.

21-methoxy-14-methyl-12-phenyl-10,23-dioxa-12,13-diazahexacyclo[14.8.0.0^{1,9}.0^{3,8}.0^{11,15}.0^{17,22}]tetracosa-3(8),4,6,11(15),13,17,19,21-octaene (10d)



Yield = 78%; reaction time = 15 h; m.p. 234-236°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.07 (s, 3H), 3.77-3.81 (m, 4H), 4.23-4.51 (m, 4H), 5.46 (s, 1H), 6.74-7.63 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.65, 34.73, 37.33, 55.92, 62.00, 66.90, 83.82, 98.49, 110.99, 119.58, 119.99, 120.35, 124.47, 125.75, 127.27, 128.97, 129.09, 129.60, 134.27, 138.25, 141.70, 147.33, 147.59, 148.34; IR (KBr) = ν 1349, 1610 cm^{-1}; MS (m/z): 437 (M^++1); Elemental Analysis for $C_{28}H_{24}N_2O_3$: Calculated: C, 77.04; H, 5.54; N, 6.42; Found: C, 77.10; H, 5.48; N, 6.50.

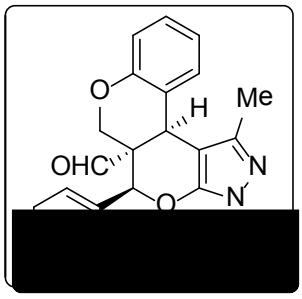
19-bromo-5,14-dimethyl-12-phenyl-10,23-dioxa-12,13-diazahexacyclo[14.8.0.0^{1,9}.0^{3,8}.0^{11,15}.0^{17,22}]tetracosa-3(8),4,6,11(15),13,17,19,21-octaene (10e)



Yield = 80%; reaction time = 15 h; m.p. 230-232°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.08 (s, 3H), 2.33 (s, 3H), 3.82 (d, J = 12 Hz, 1H), 4.08-4.28 (m, 3H), 4.43 (d, J = 11.4 Hz, 1H), 5.42 (s, 1H), 6.72-7.62 (m, 11H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.56, 21.20, 34.88, 37.43, 61.59, 66.93, 83.87, 98.58, 116.96, 118.76, 120.28, 120.34, 125.76, 127.18, 129.04, 129.27, 129.64, 131.27, 132.74, 138.18, 139.61, 147.39, 147.74, 152.38; IR (KBr) = ν 1358, 1585 cm^{-1}; MS (m/z): 500 (M^{+}+1); Elemental Analysis for $C_{28}H_{23}BrN_2O_2$: Calculated: C, 67.34; H, 4.64; N, 5.61; Found: C, 67.31; H, 4.67; N, 5.59.

A representative procedure for the synthesis of 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-pentaene-10-carbaldehyde (11): To a solution of chromenopyranpyrazole alcohol **9a** (0.10 g, 0.22 mmol) in DMSO (10 ml), IBX (1.5 equiv) was added at room temperature and stirred well at rt for 6 h. After the completion of reaction as indicated by TLC, the reaction mixture was poured into cold water and extrated using ethyl acetate. The organic layer was dried over Na_2SO_4 and the crude product thus obtained was purified using column chromatography to afford the desired product **11** as a colourless solid.

16-methyl-11,14-diphenyl-8,12-dioxa-14,15 diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}] hep- tadeca-2,4,6,13(17),15-pentaene-10-carbaldehyde (11)

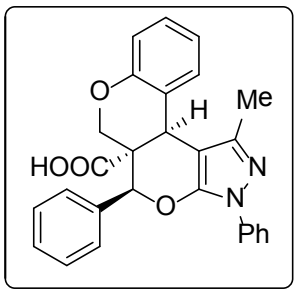


Yield = 90%; reaction time = 6 h; m.p. 180-182°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.18 (s, 3H), 4.49 (d, J = 11.4 Hz, 1H), 4.69 – 4.73 (m, 2H), 5.51 (s, 1H), 6.76 – 7.75 (m, 14H), 9.46 (s, 1H) ; ^{13}C NMR (75 MHz, $CDCl_3$) = δ 15.56, 35.00, 49.54, 61.14, 82.90, 98.26, 116.89, 118.90, 120.13, 120.31, 125.79, 127.23, 128.94, 129.06, 129.39, 132.27, 133.72, 138.28,

147.31, 147.38, 153.13, 202.43; IR (KBr) = ν 1512, 1597, 1728 cm^{-1}; MS (m/z): 423 (M^++1); Elemental Analysis for $C_{27}H_{22}N_2O_3$: Calculated: C, 76.76; H, 5.25; N, 6.63; Found: C, 76.71; H, 5.33; N, 6.59.

A representative procedure for the synthesis of 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-pentaene-10-carboxylic acid (12): To a solution of chromenopyranpyrazole **5a** (0.20 g, 0.44 mmol) in methanol (4ml), aquoues KOH solution (5 equivalent in 4ml water) was added at room temperature and the resulting solution was kept under reflux condition for 24 h. After the completion of reaction as indicated by TLC, a solution of dil. HCl was added slowly to the mixture and extrated using ethyl acetate. The organic layer was dried over Na_2SO_4 and the crude product thus obtained was purified using column chromatography to afford the desired product **12** as a colourless solid.

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-pentaene-10-carboxylic acid (12)

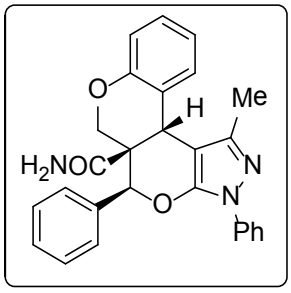


Yield = 93%; reaction time = 24 h; m.p. 228-230°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.06 (s, 3H), 4.11 – 4.23 (m, 3H), 4.69 (s, 1H), 5.52 (s, 1H), 6.57 – 7.76 (m, 14H); ^{13}C NMR (75 MHz, $CDCl_3$ $CDCl_3$; 3 drops DMSO-d_6) = δ 13.36, 14.36, 20.15, 59.34, 98.23, 115.53, 118.21, 119.13, 119.35, 124.59, 126.38, 127.38, 127.66, 127.74, 128.14, 131.37, 134.91, 137.65, 146.12, 146.90, 152.40, 169.97; IR (KBr) = ν 1730, 1561, 1489 cm^{-1}; MS (m/z): 439 (M^++1); Elemental Analysis for $C_{27}H_{22}N_2O_4$: Calculated: C, 73.96; H, 5.06; N, 6.39; Found: C, 73.91; H, 5.14; N, 6.33.

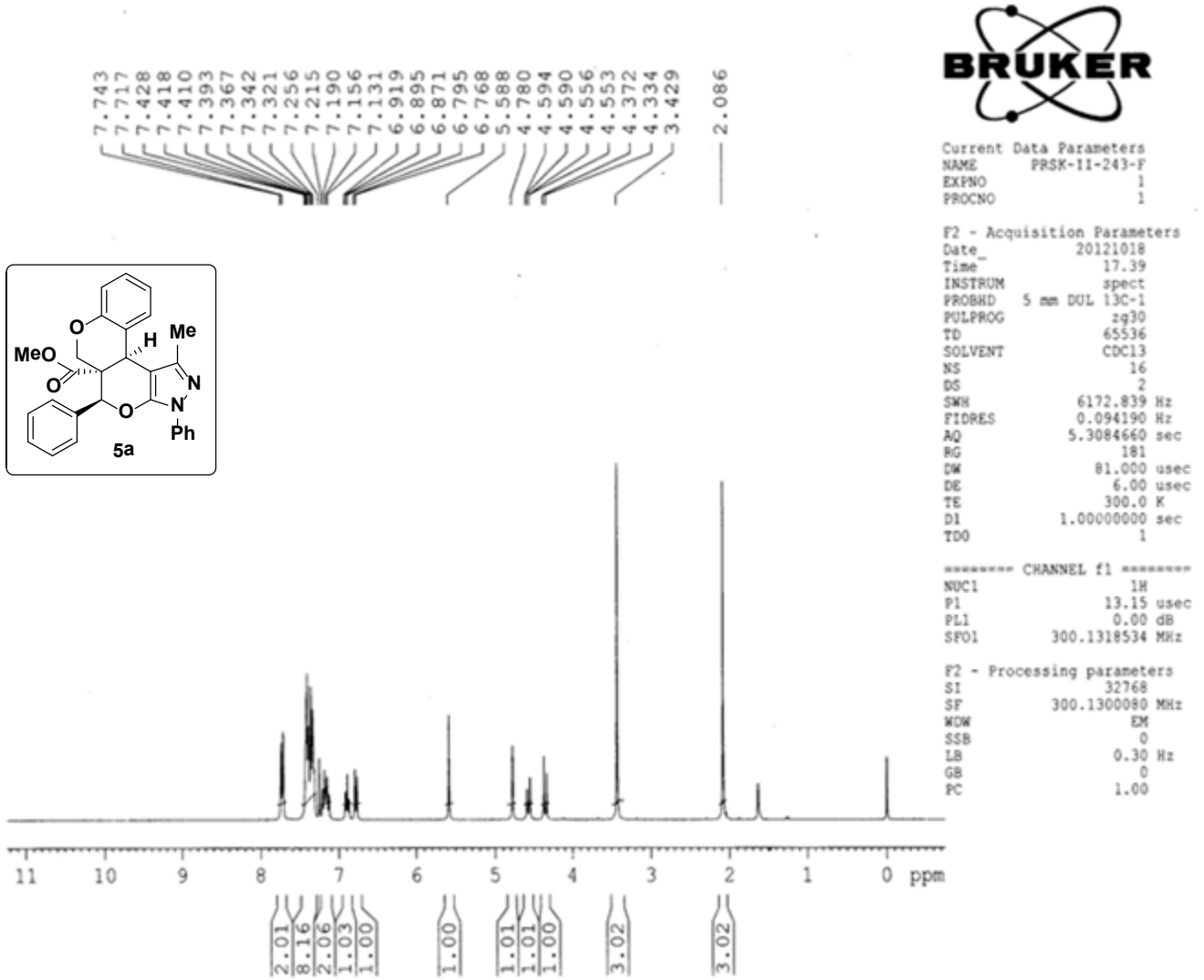
A representative procedure for the synthesis of 16-methyl-11,14-diphenyl-8,12-dioxa-14,15 diazatetracyclo[8.7.0.0^{2,7}.0^{13,17}] heptadeca-2,4,6,13(17),15-pentaene-10-carbox amide (13): To a solution of chromenopyranpyrazole **8a** (0.20 g, 0.47 mmol) in methanol (4ml), aquoues KOH solution (5 equivalent in 4ml water) was added at room temperature and the resulting solution was kept under reflux condition for 24 h. After the completion of

reaction as indicated by TLC, a solution of dil. HCl was added slowly to the mixture and extrated using ethyl acetate. The organic layer was dried over Na_2SO_4 and the crude product thus obtained was purified using column chromatography to afford the desired product **13** as a colourless solid.

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-pentaene-10-carboxamide (13)



Yield = 90%; reaction time = 24 h; m.p. 218-220°C; ^{1}H NMR (300 MHz, $CDCl_3$) = δ 2.57 (s, 3H), 4.23 – 4.48 (m, 3H), 5.61 (s, 1H), 5.74 (s, 1H), 5.83 (s, 1H), 6.97 – 7.79 (m, 14H); ^{13}C NMR (75 MHz, $CDCl_3$) = δ 14.41, 31.58, 34.51, 67.89, 80.11, 99.10, 117.38, 120.16, 121.72, 125.97, 127.48, 128.50, 128.56, 129.110, 129.17, 130.24, 134.99, 138.25, 146.64, 148.63, 152.12, 171.25; IR (KBr) = ν 1605, 1660, 3456cm^{-1}; MS (m/z): 438 (M^++1); Elemental Analysis for $C_{27}H_{23}N_3O_3$: Calculated: C, 74.12; H, 5.30; N, 9.60; Found: C, 70.20; H, 5.26; N, 9.71.



BRUKER

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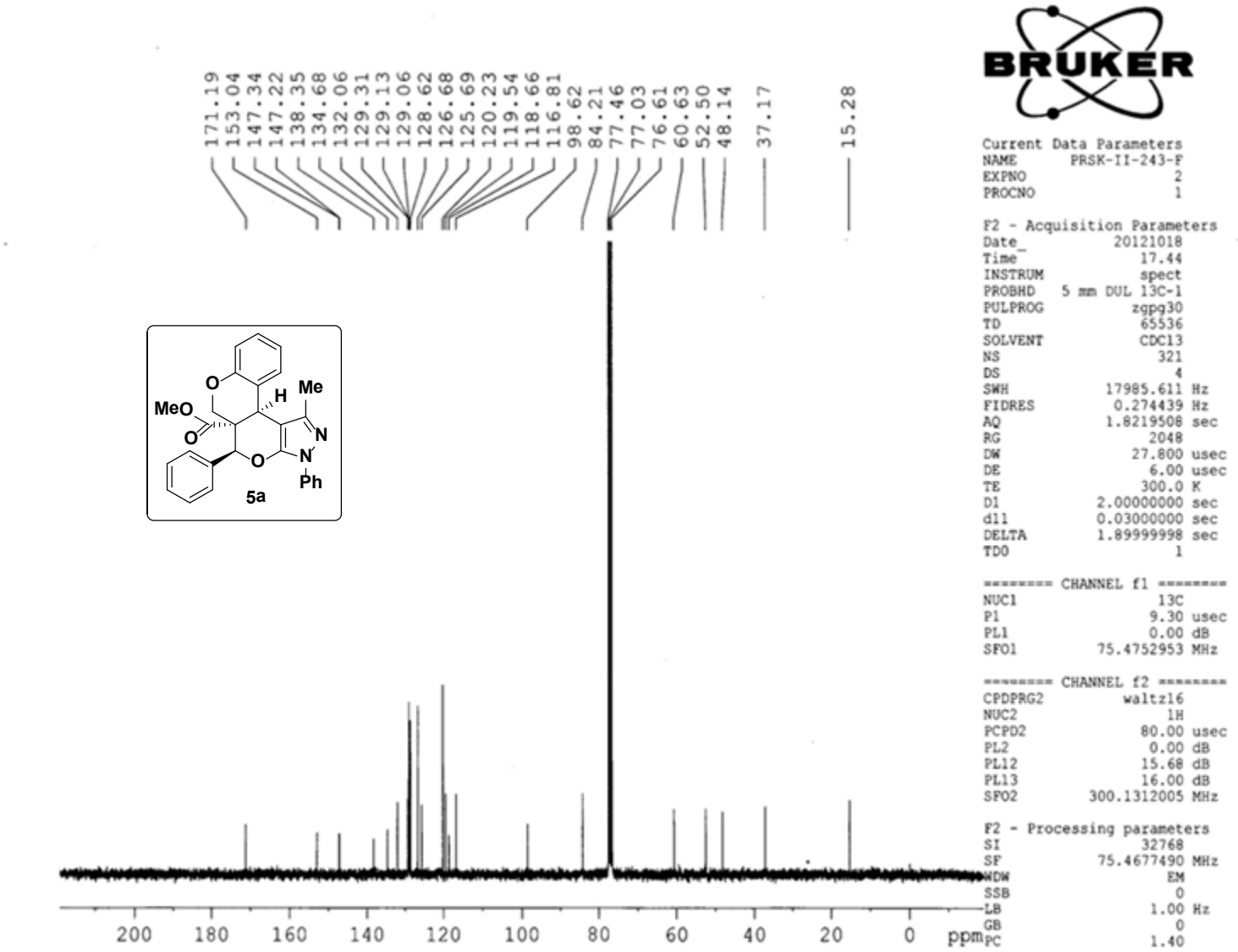

BRUKER

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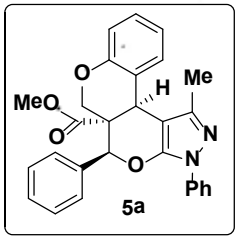

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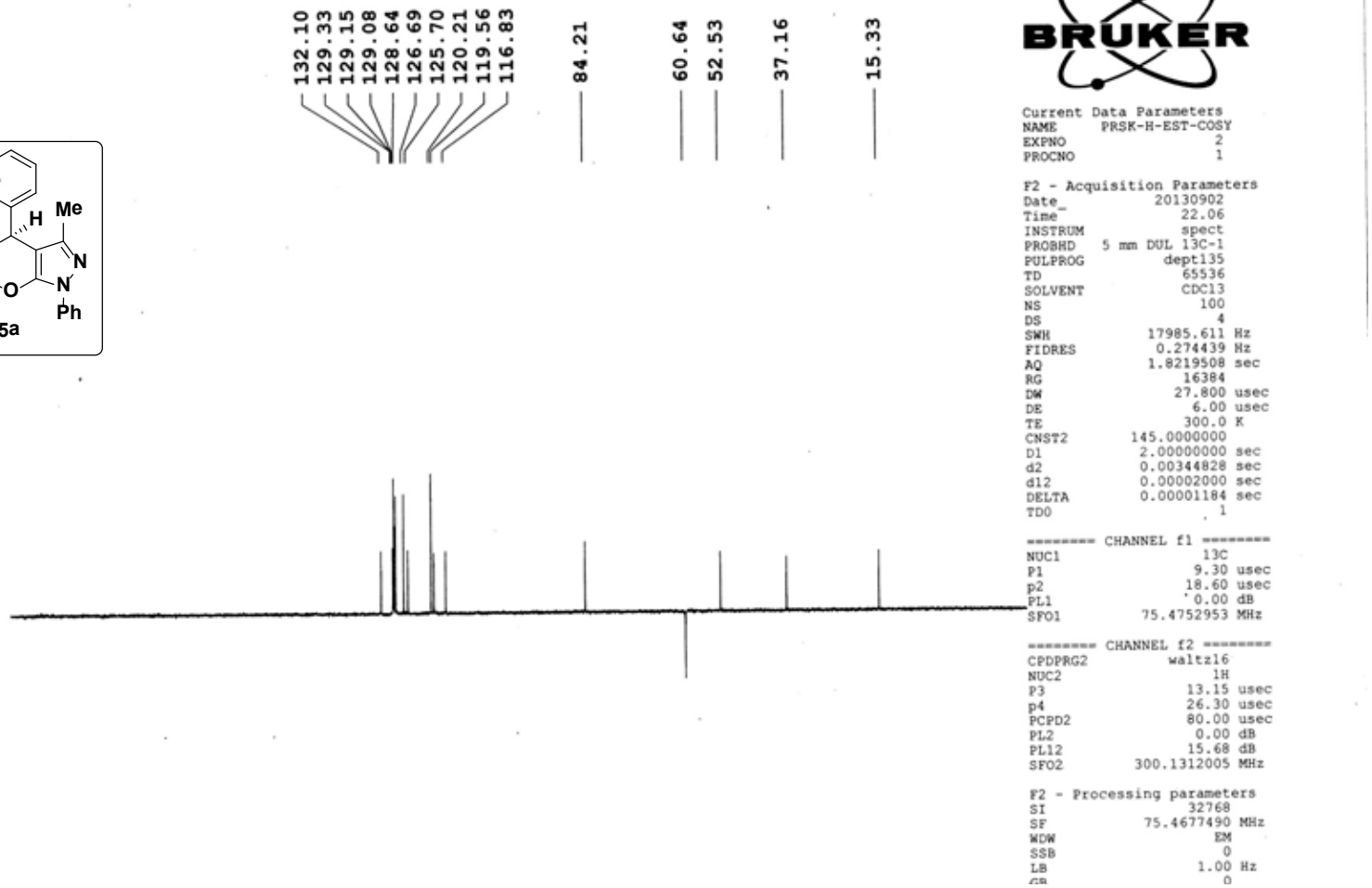

BRUKER

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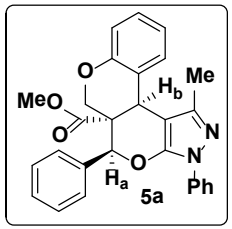

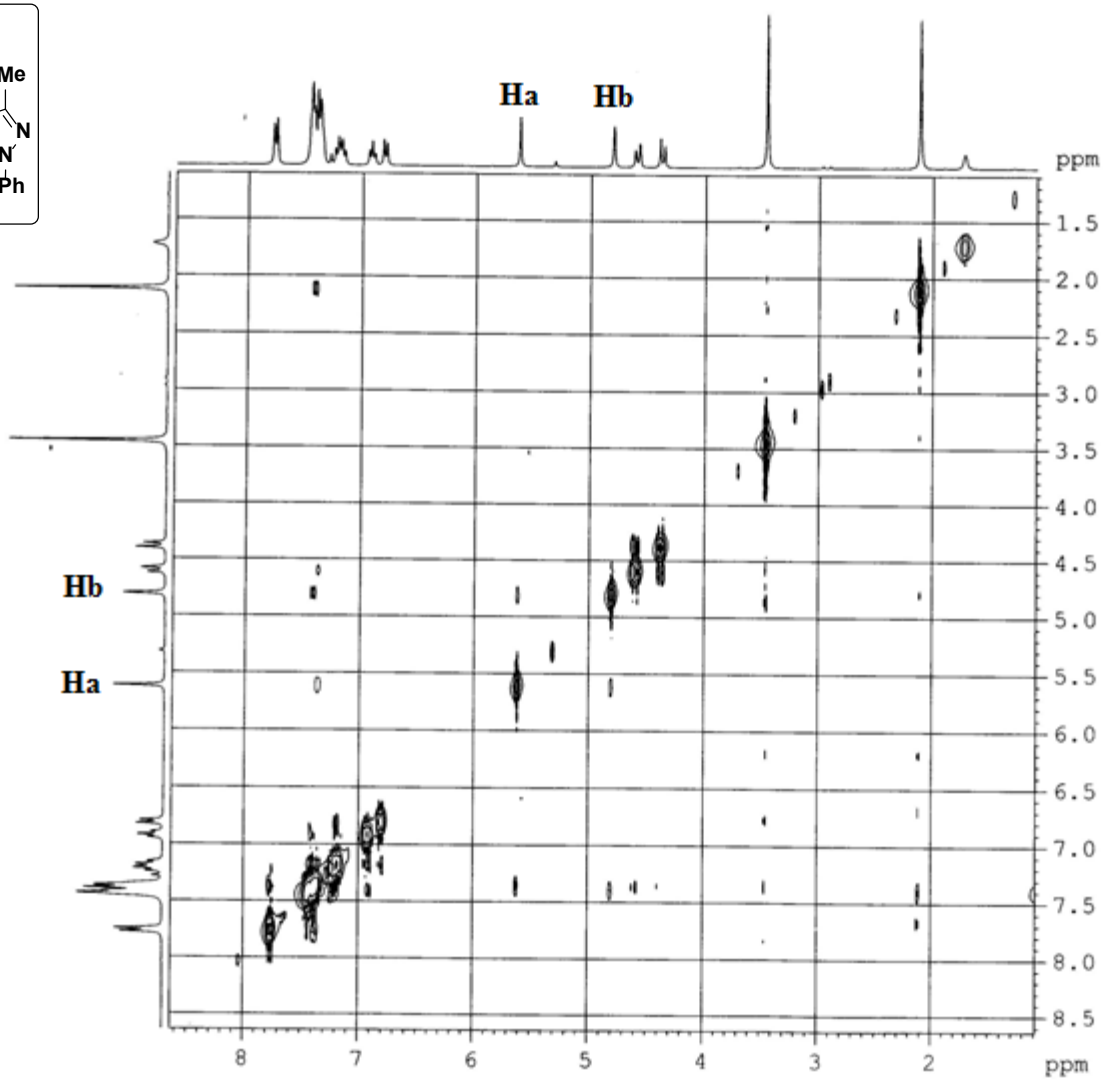

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F2 - Acquisition Parameters
Date_          20130902
Time_             13.04
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         noesyph
TD                2048
SOLVENT           CDCl3
NS                  16
DS                   4
SWH           2264.493 Hz
FIDRES        1.105709 Hz
AQ            0.4522484 sec
RG                  64
DW              220.800 usec
DE                6.00 usec
TE               300.0 K
d0            0.00020406 sec
D1            1.88367295 sec
D8            0.30000001 sec
IN0           0.00044160 sec
ST1CNT               128

======== CHANNEL f1 ========
NUC1                 1H
P1                13.15 usec
PL1                0.00 dB
SFO1        300.1314602 MHz

F1 - Acquisition parameters
NDO                  1
TD                 124
SFO1        300.1315 MHz
FIDRES     18.262039 Hz
SW             7.545 ppm
FnMODE     States-TPPI

F2 - Processing parameters
SI                1024
SF        300.1300000 MHz
WDW               QSINE
SSB                  2
LB                0.00 Hz
GB                   0
PC                1.00

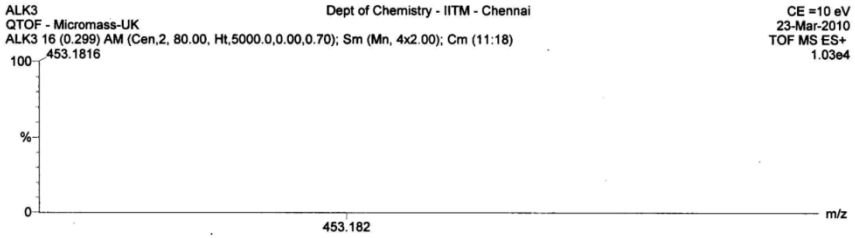
F1 - Processing parameters
SI                1024
MC2        States-TPPI
SF        300.1300000 MHz
WDW               QSINE
SSB                  2
LB                0.00 Hz
GB                   0
```

NOESY spectrum of compound **5a**

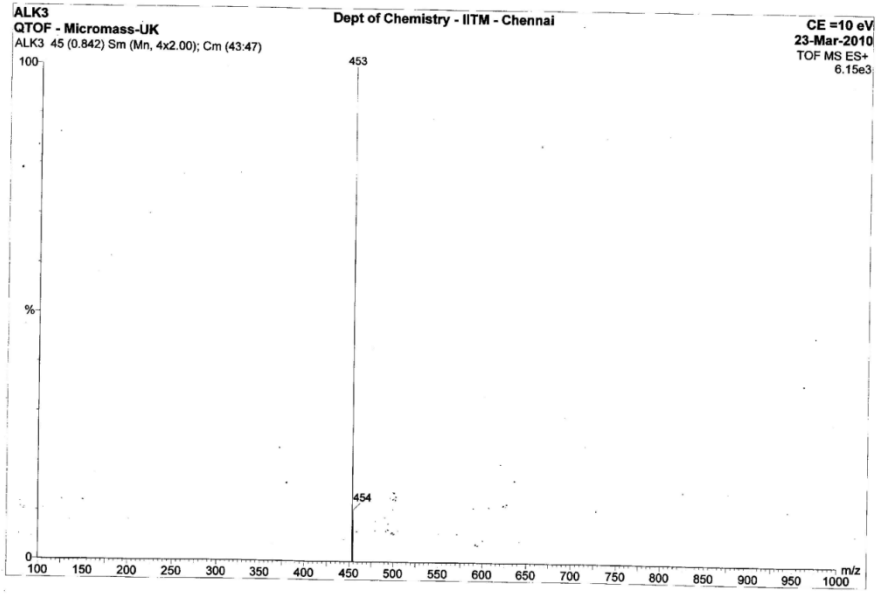
Elemental Composition Report

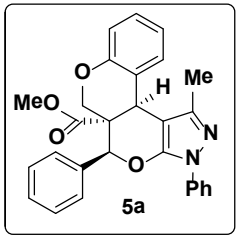
Single Mass Analysis
Tolerance = 200.0 mDa / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

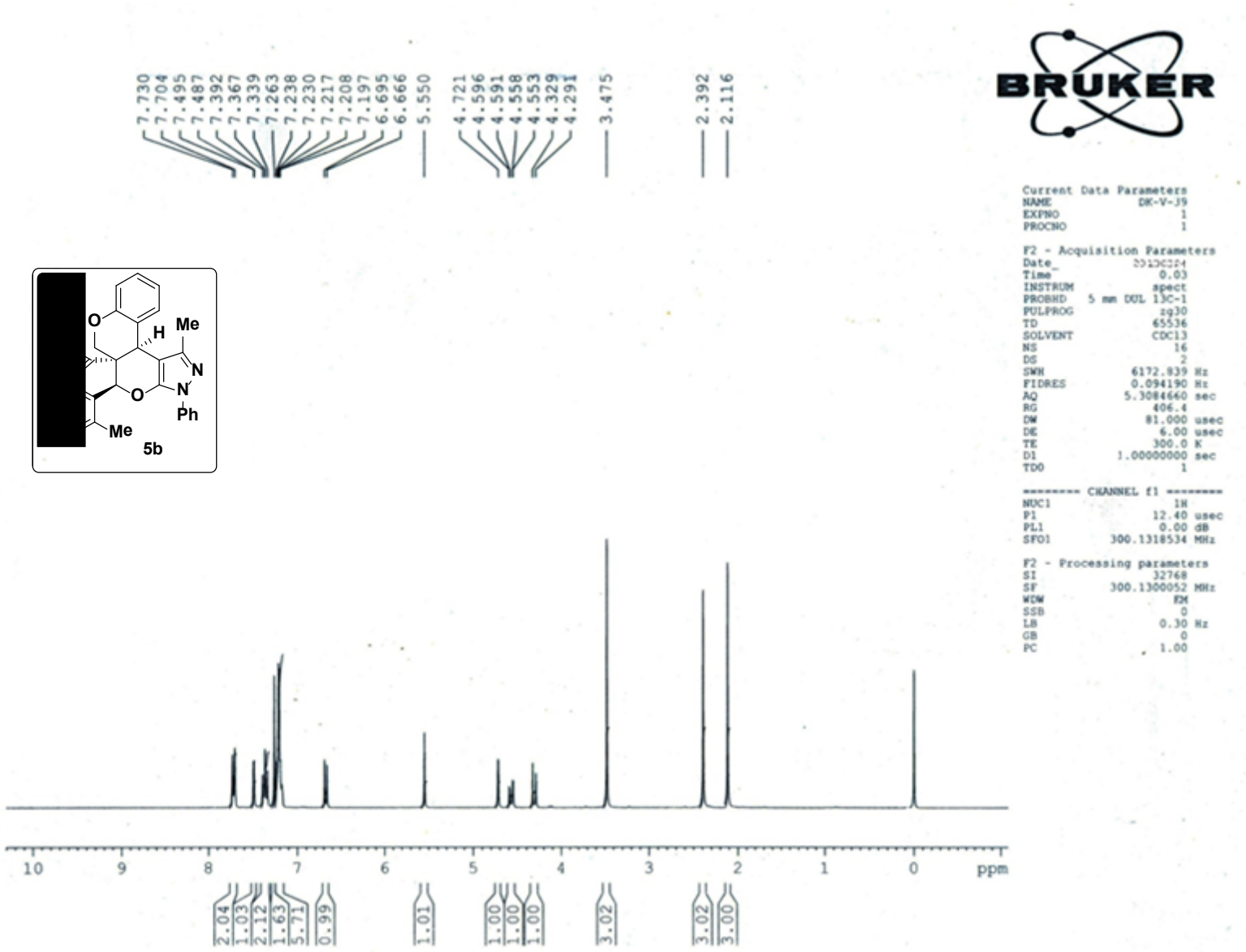
Monoisotopic Mass, Odd and Even Electron Ions
10 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

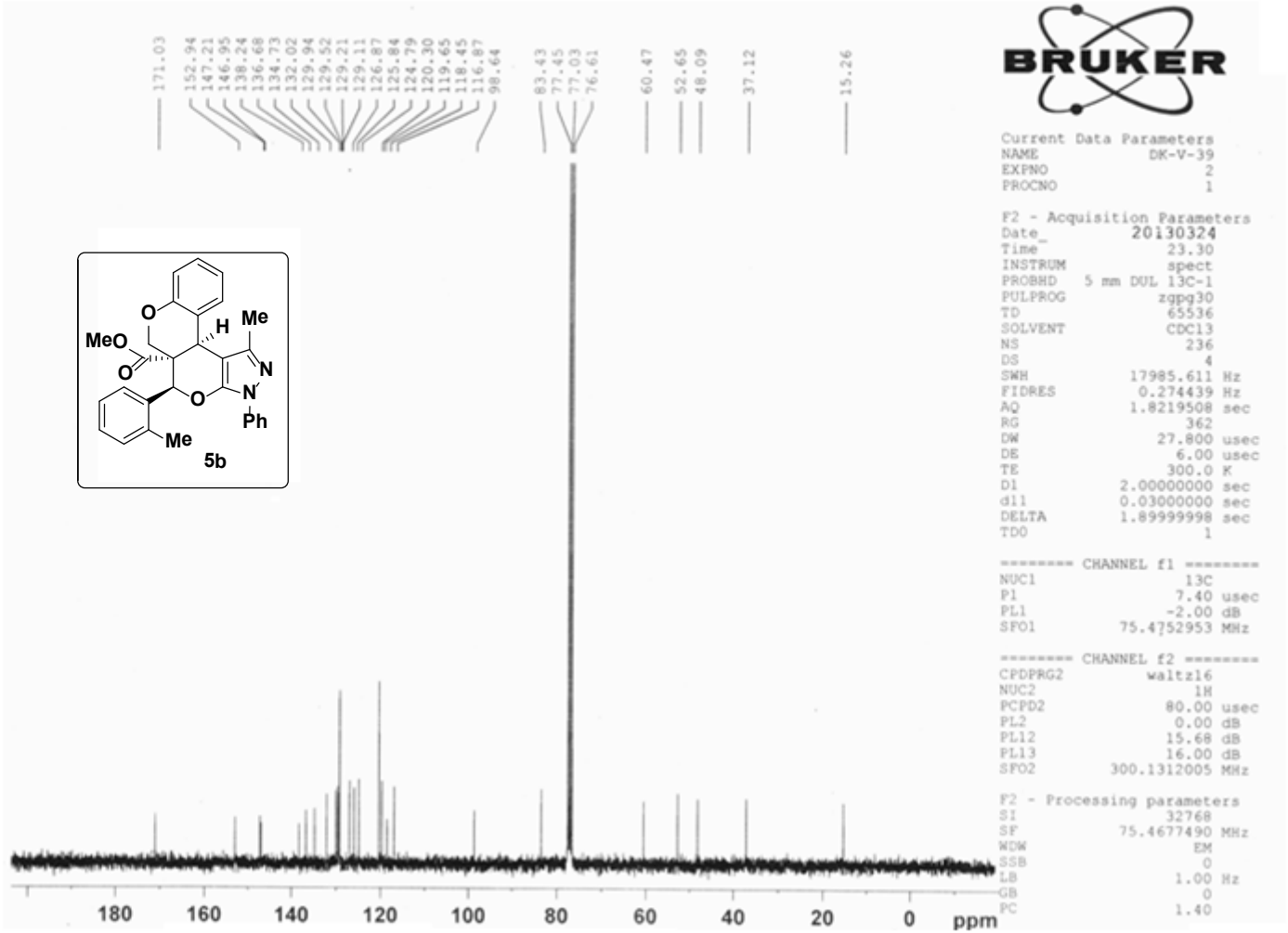


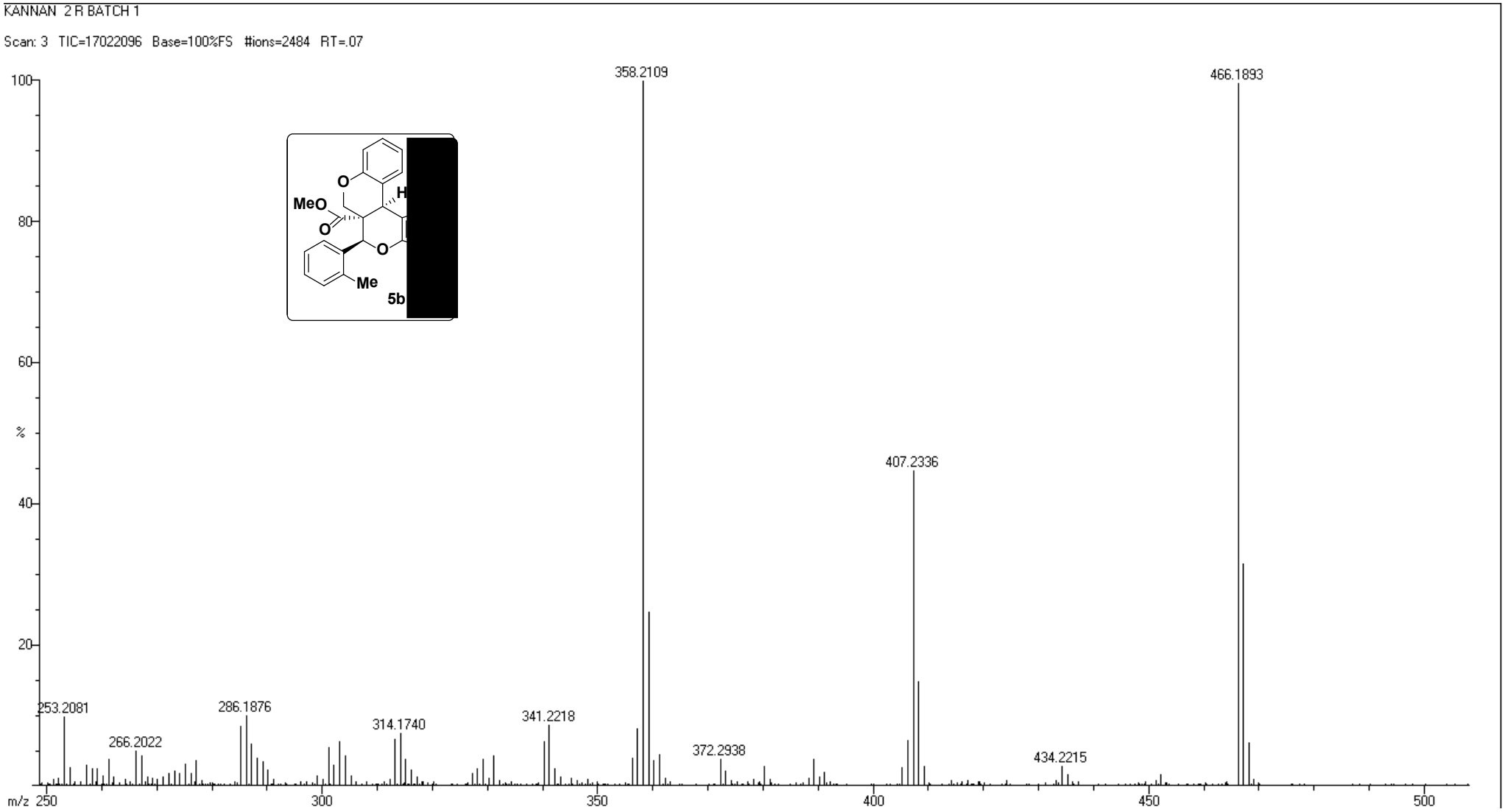
Minimum:				-1.5		
Maximum:		200.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
453.1816	453.1814	0.2	0.5	17.5	1	C28 H25 N2 O4

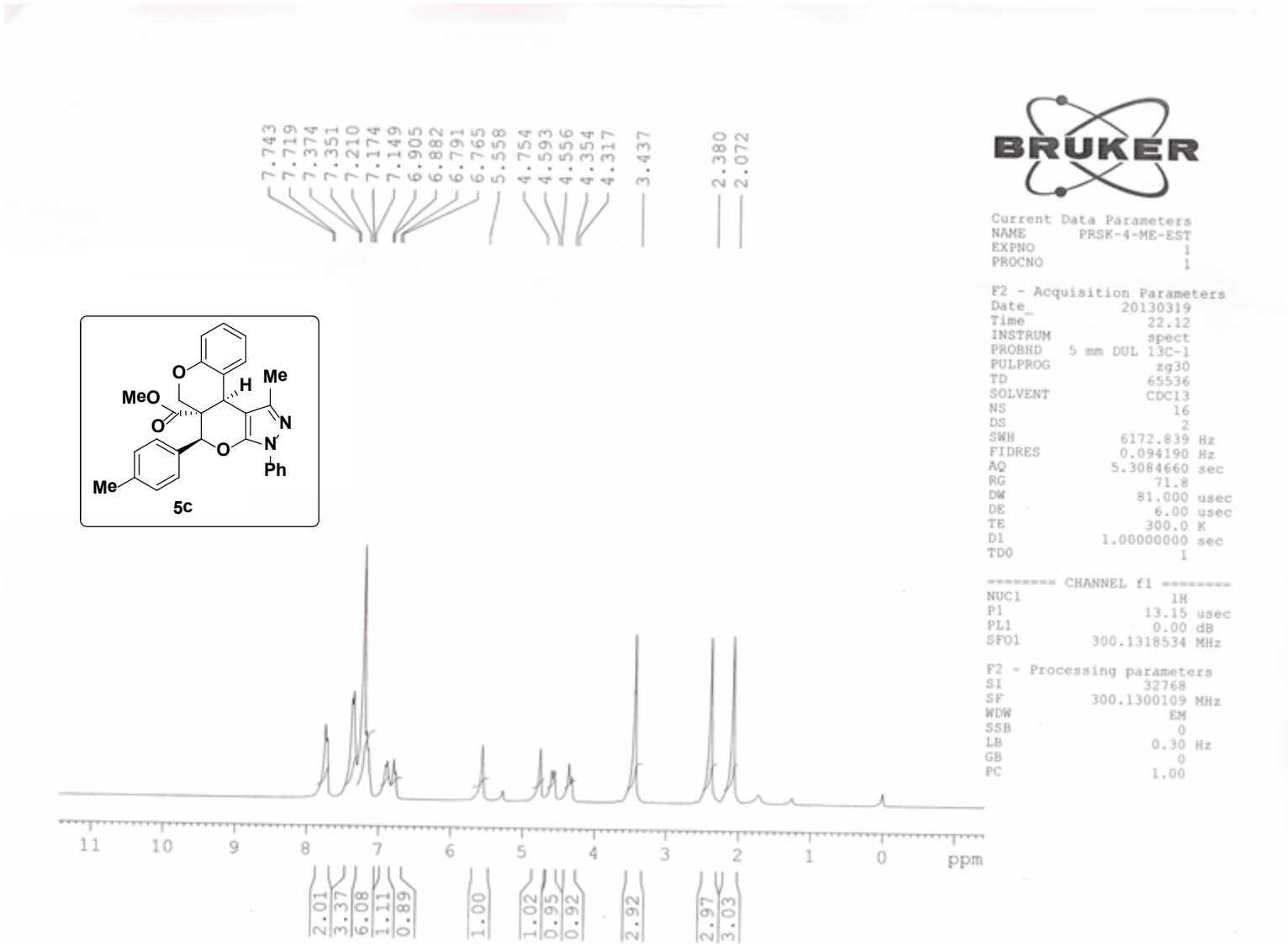


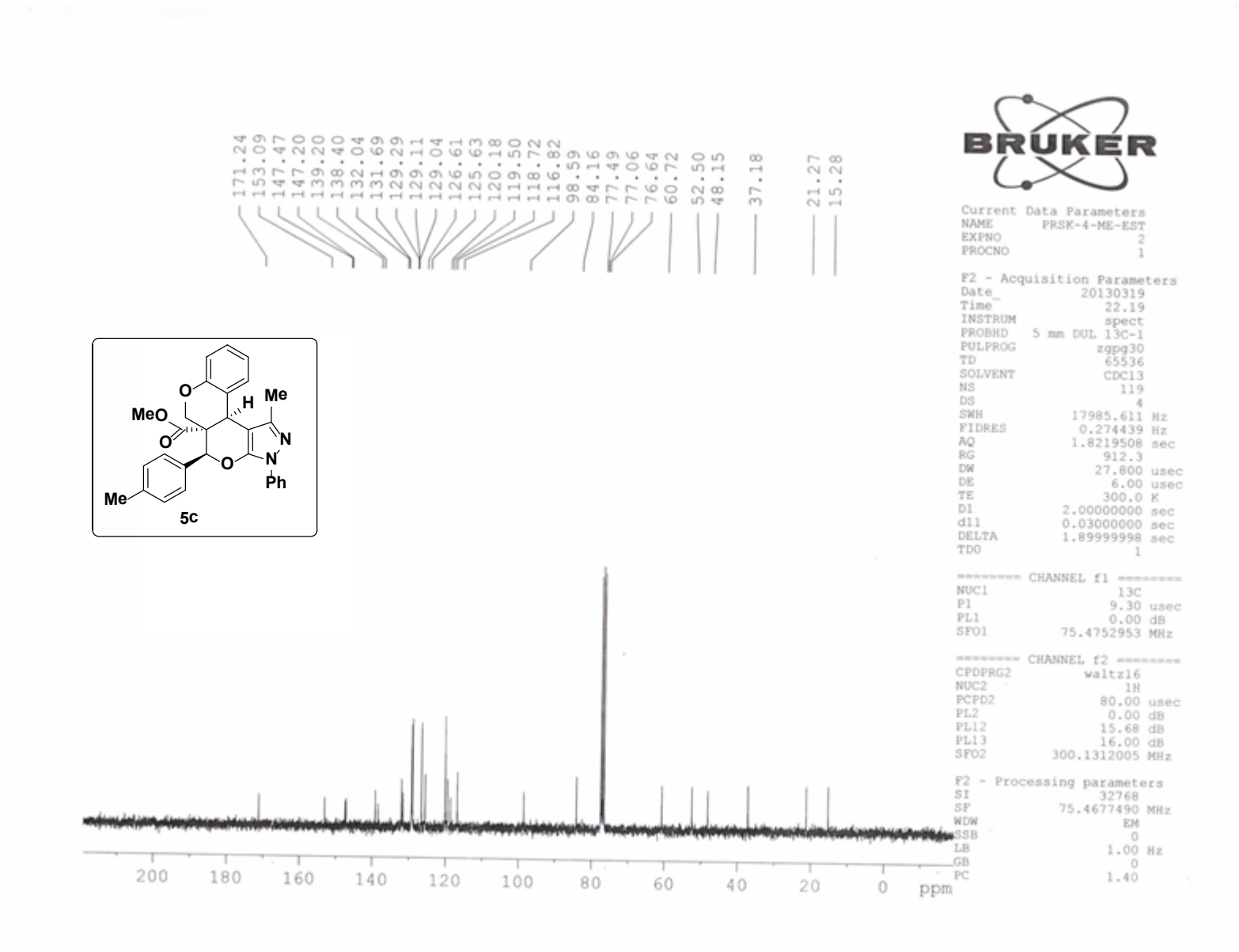


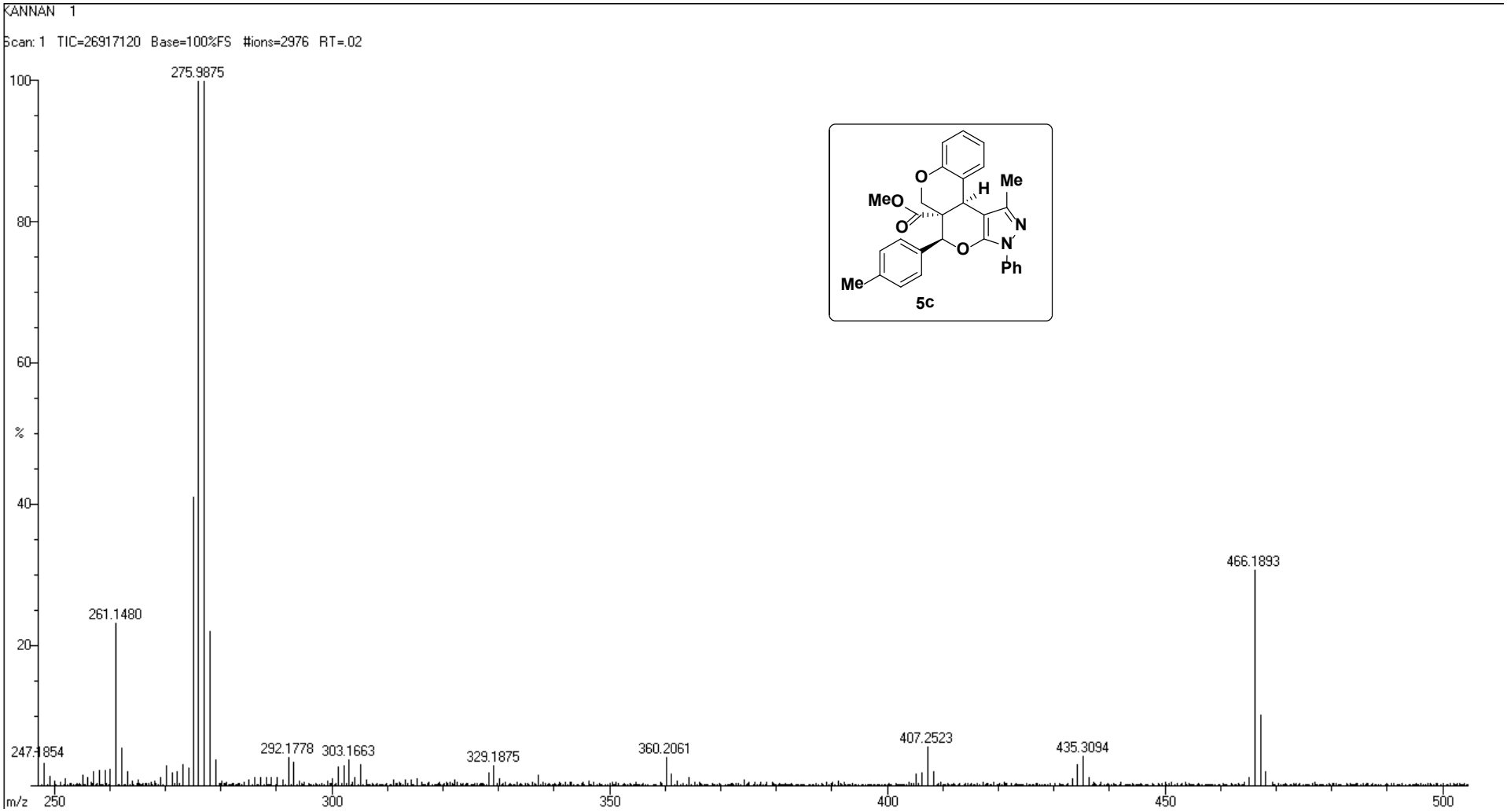



171.03
152.94
147.21
146.95
138.24
136.68
134.73
132.02
129.94
129.52
129.21
129.11
126.87
125.84
124.79
120.30
119.65
118.45
116.87
98.64
83.43
77.45
77.03
76.61
60.47
52.65
48.09
37.12
15.26
MeO
O
O
H
Me
N
N
Ph
O
Me
5b
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME DK-V-39
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130324
Time 23.30
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 236
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

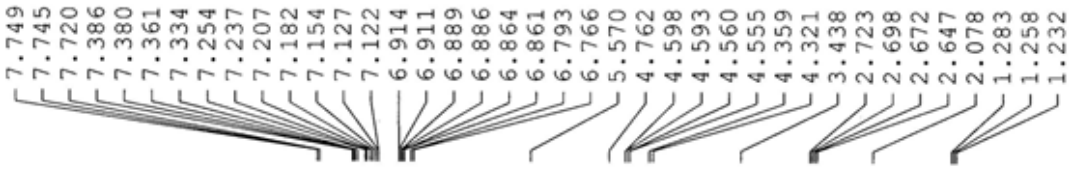


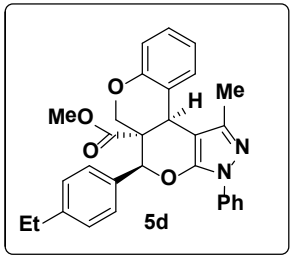


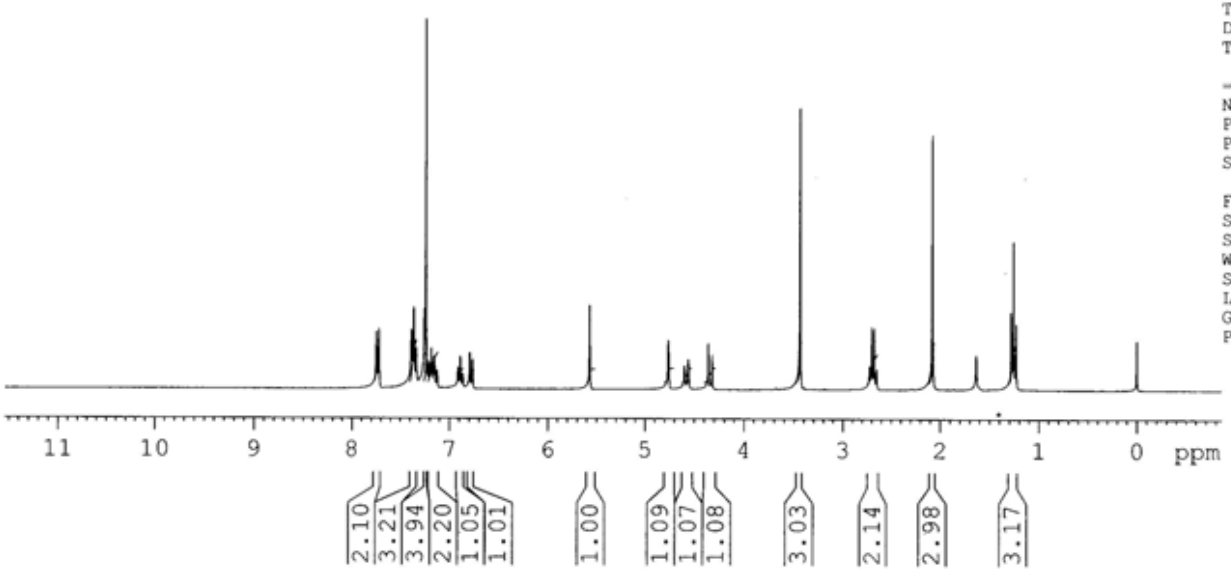



KANNAN 1
Scan: 1 TIC=26917120 Base=100%FS #ions=2976 RT=.02
100
80
60
%
40
20
275.9875
261.1480
247.1854
292.1778
303.1663
329.1875
360.2061
407.2523
435.3094
466.1893
m/z
250
300
350
400
450
500
MeO
O
O
H
Me
N
N
Ph
O
Me
5c

2





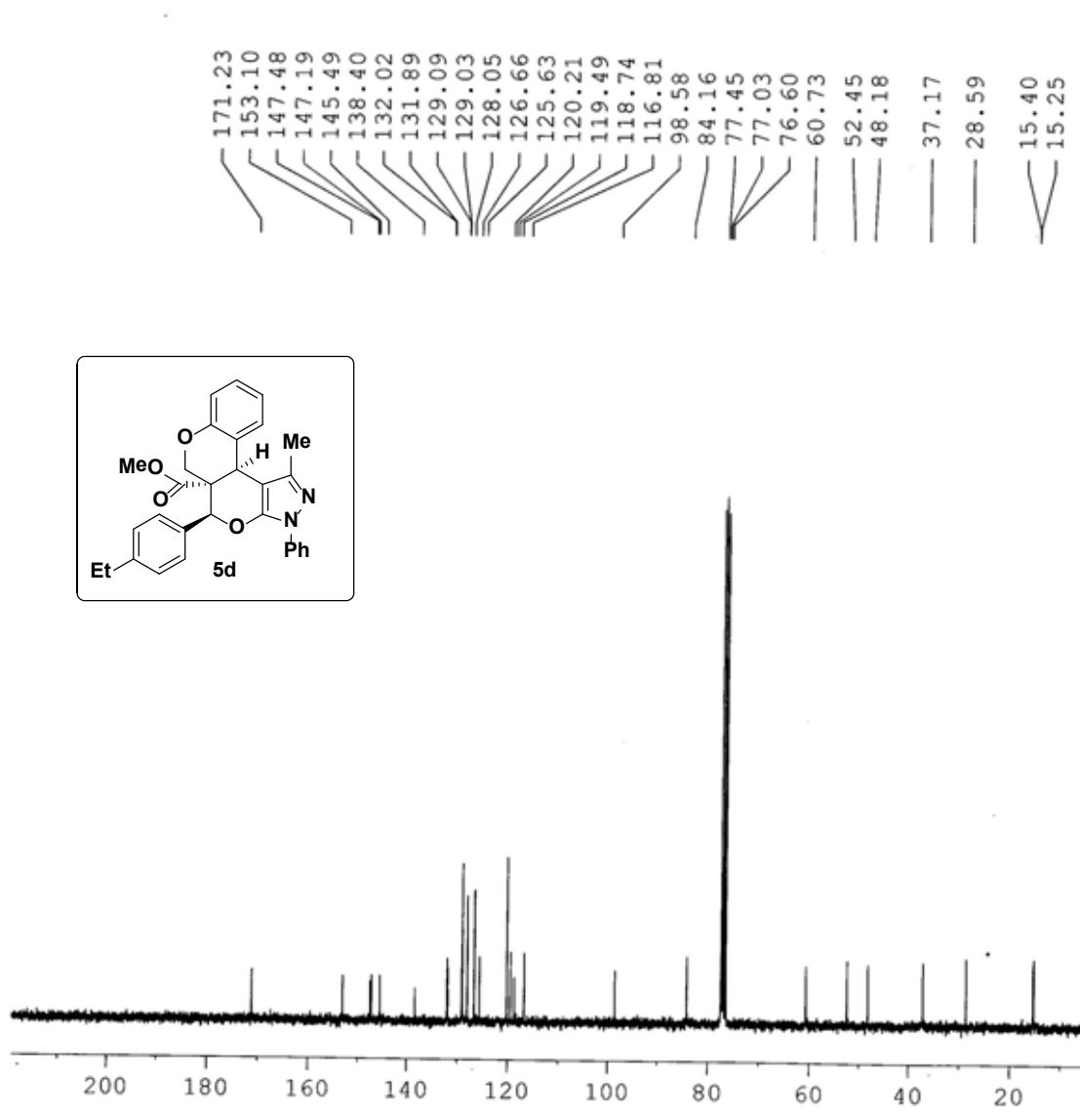



```
Current Data Parameters
NAME                  PRSK-4-ET
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130319
Time                      20.55
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                            7
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                  300.1300082 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```



BRUKER

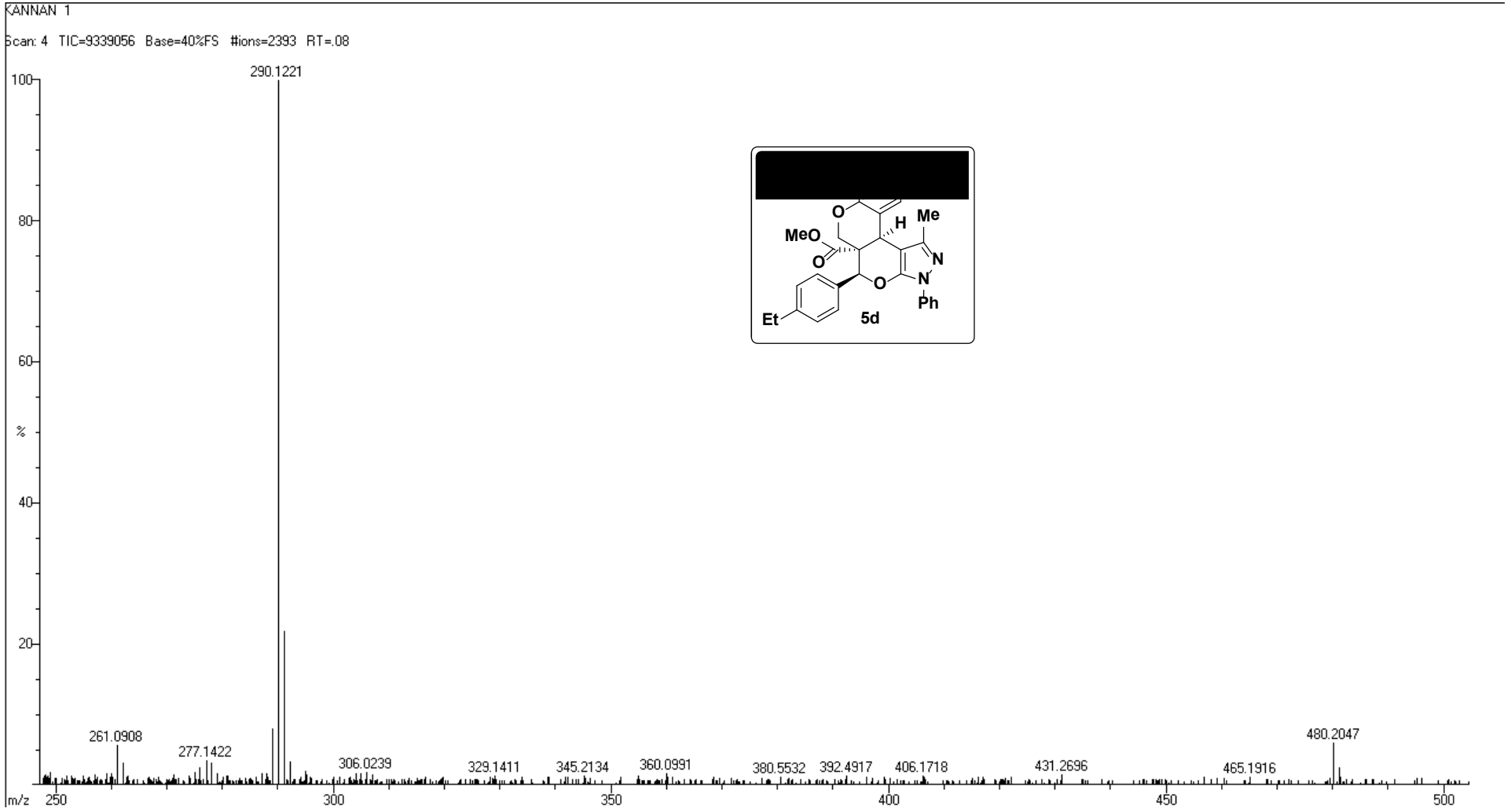
Current Data Parameters
NAME PRSK-4-ET
EXPNO 2
PROCNO 1

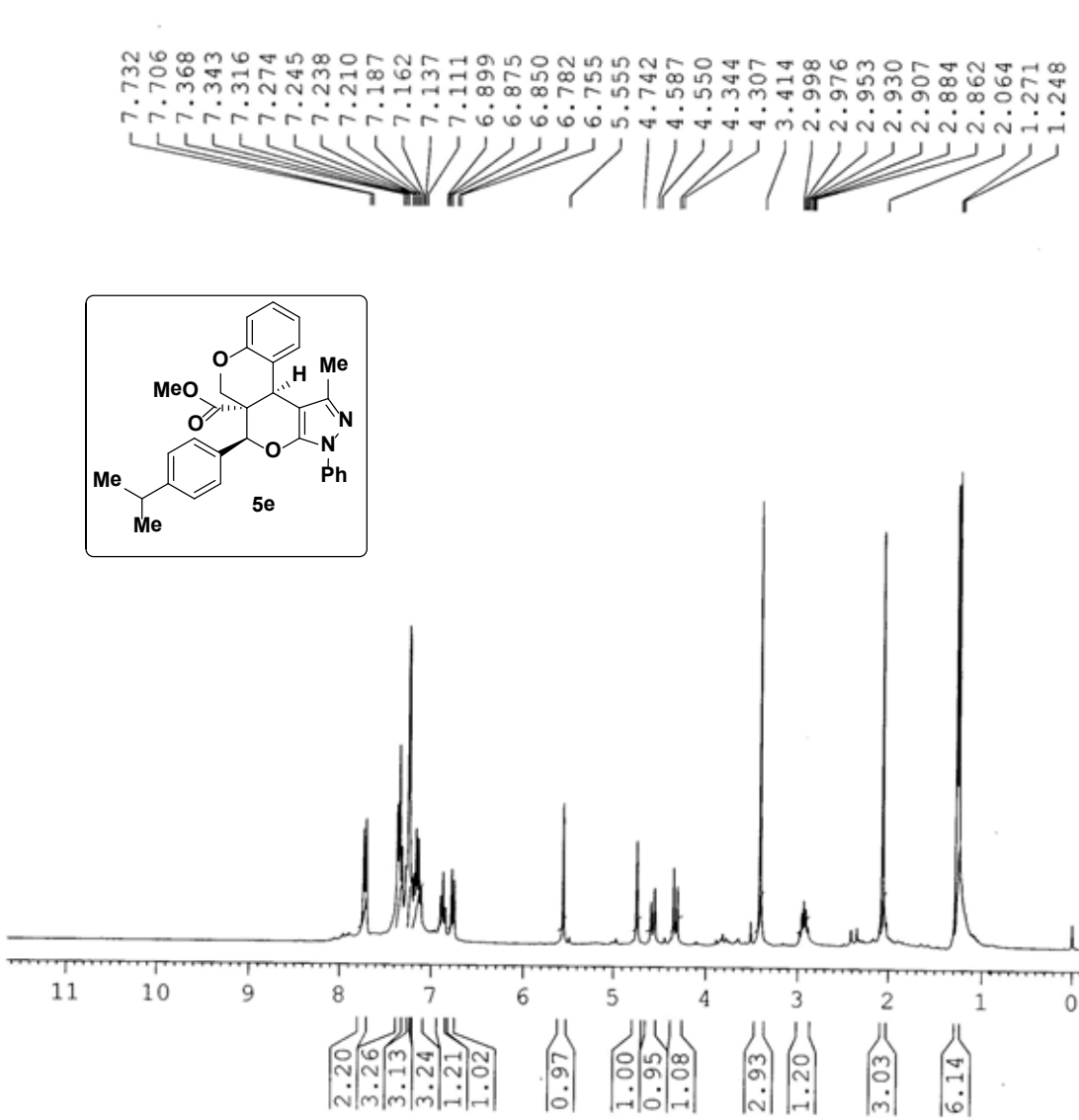
F2 - Acquisition Parameters
Date_ 20130319
Time 20.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 437
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1824.6
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 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


KANNAN 1
Scan: 4 TIC=9339056 Base=40%FS #ions=2393 RT=.08
290.1221
100
80
60
%
40
20
261.0908
277.1422
306.0239
329.1411
345.2134
360.0991
380.5532
392.4917
406.1718
431.2696
465.1916
480.2047
m/z
250
300
350
400
450
500
O
H
Me
MeO
O
N
O
N
Ph
Et
5d



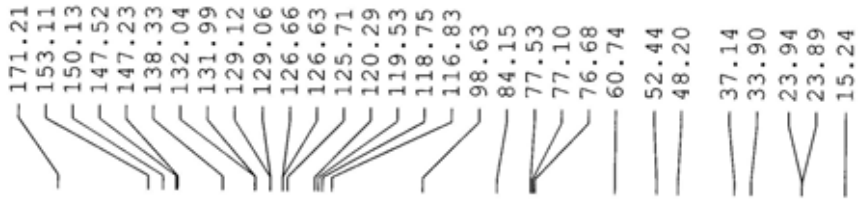
BRUKER

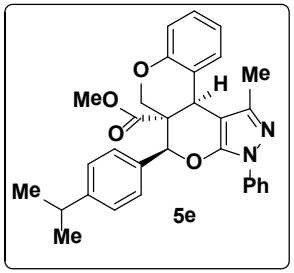
Current Data Parameters
NAME PRSK-4-ISO-EST
EXPNO 1
PROCNO 1

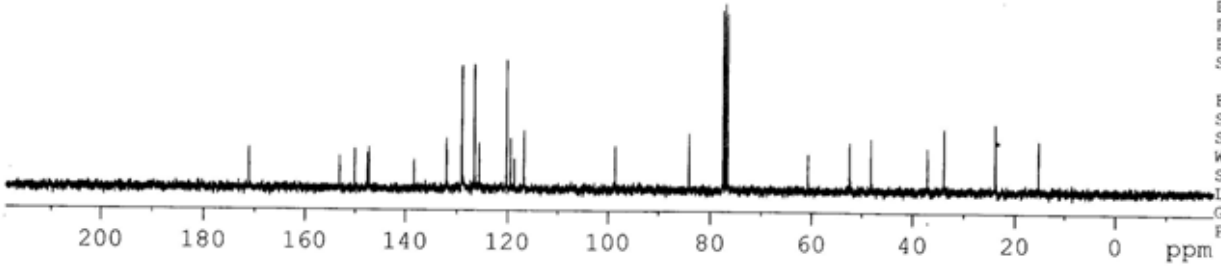
F2 - Acquisition Parameters
Date_ 20130323
Time 18.48
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 40.3
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

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

F2 - Processing parameters
SI 32768
SF 300.1300143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







BRUKER

Current Data Parameters
NAME PRSK-4-ISO-EST
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20130323
Time	18.52
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	50
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	812.7
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 ========

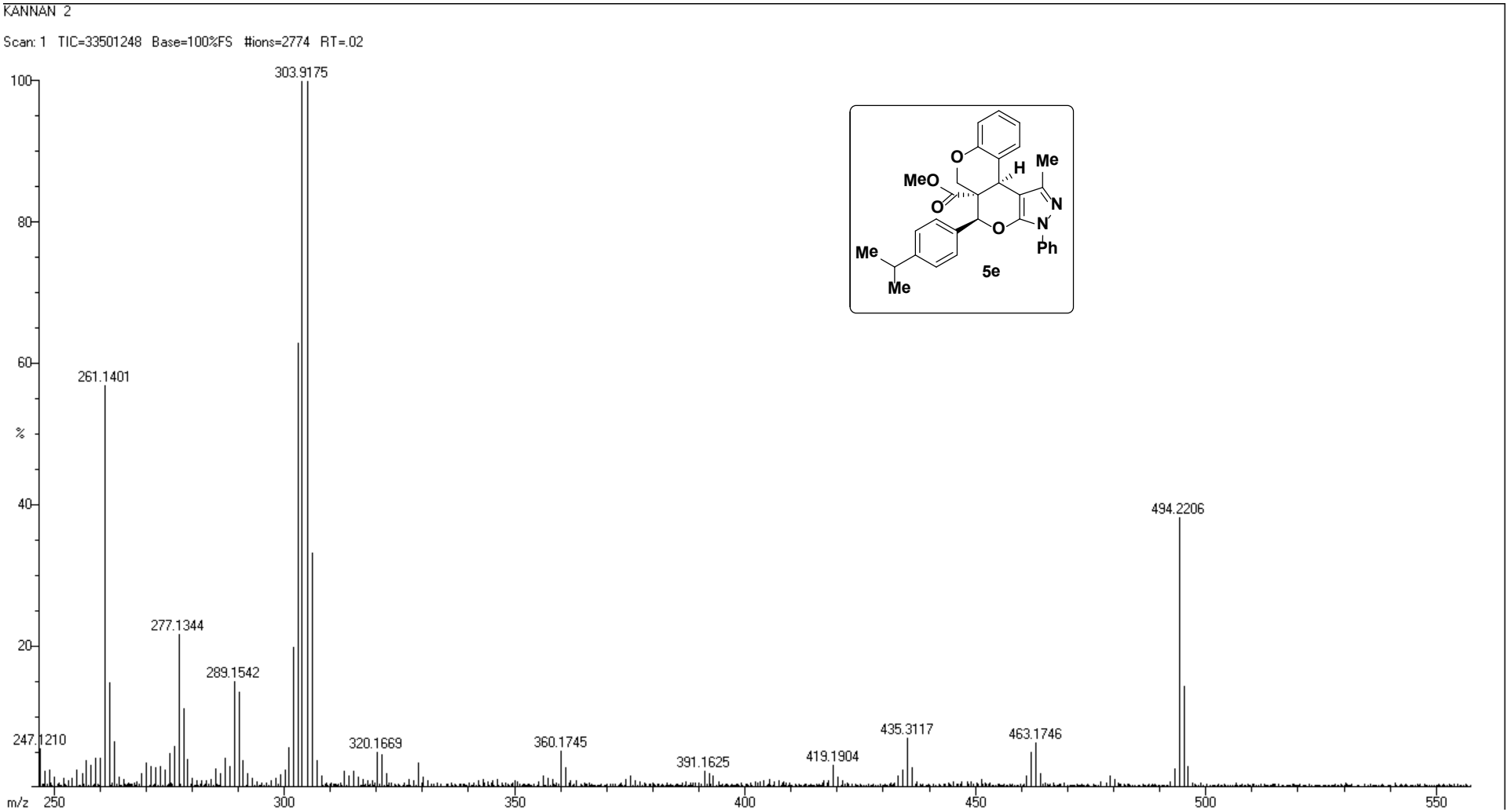
NUC1	13C
P1	9.30 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

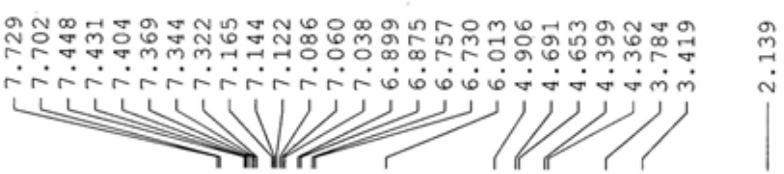
======== CHANNEL f2 ========

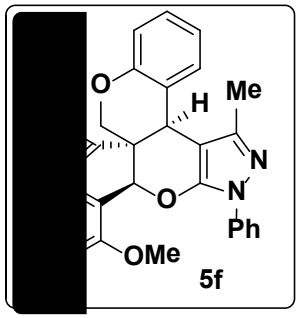
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

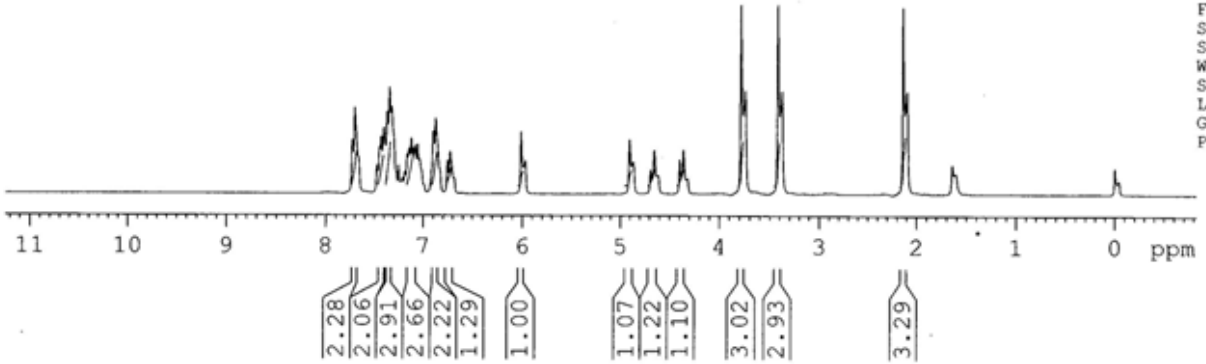
F2 - Processing parameters

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







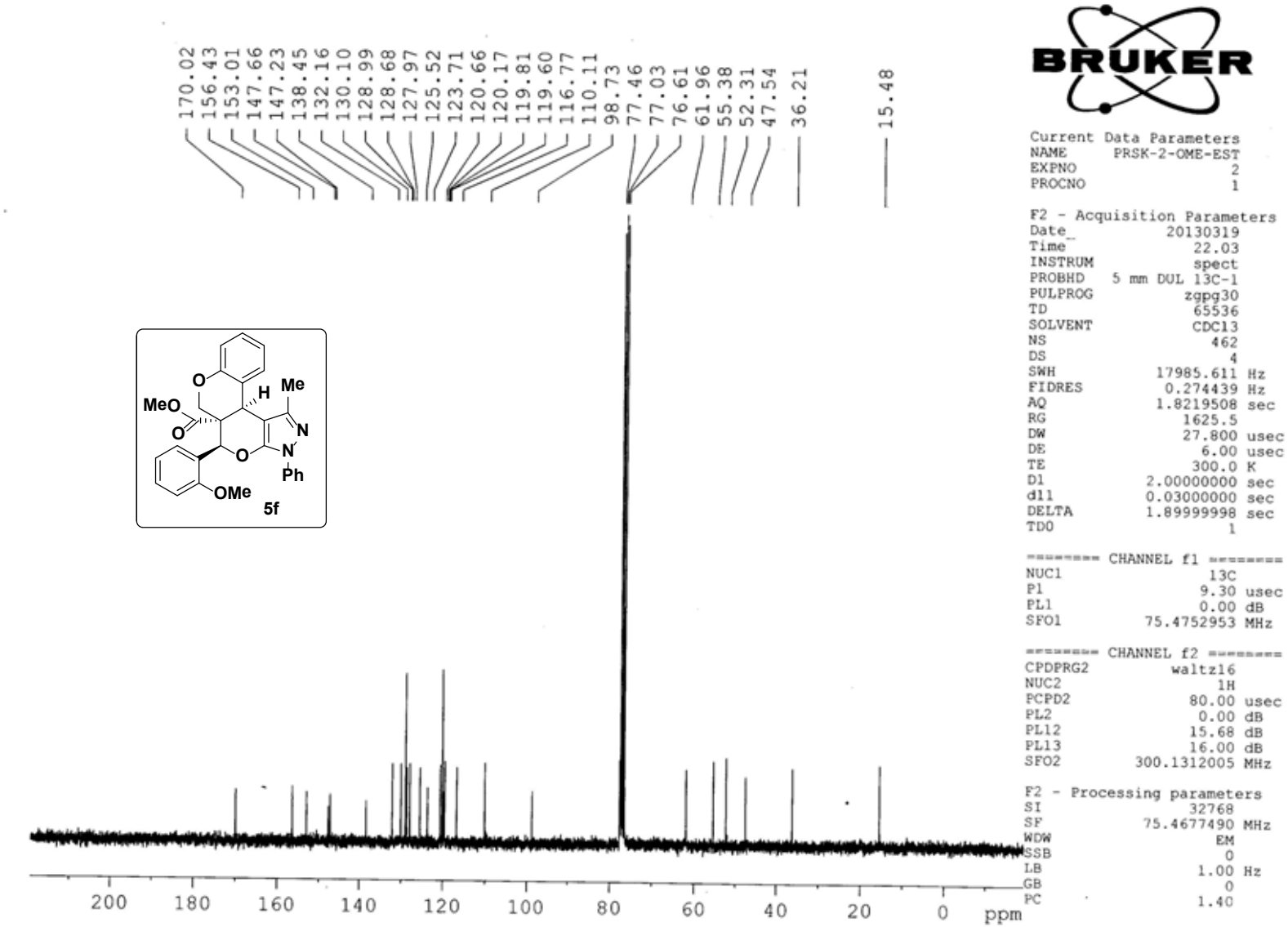



```
Current Data Parameters
NAME      PRSK-2-OME-EST
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130319
Time               21.33
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                   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           300.1300181 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



BRUKER

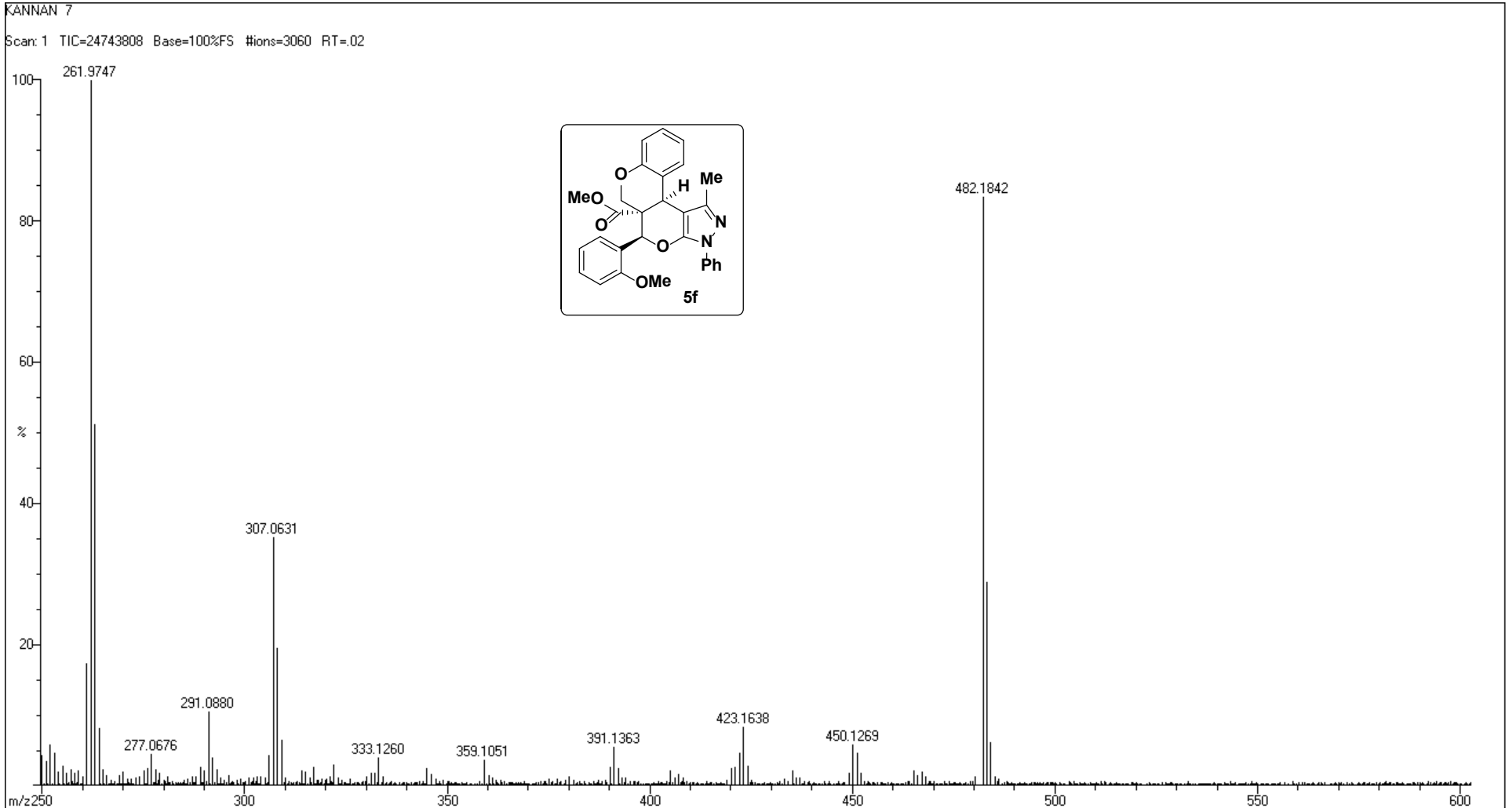
Current Data Parameters
NAME PRSK-2-OME-EST
EXPNO 2
PROCNO 1

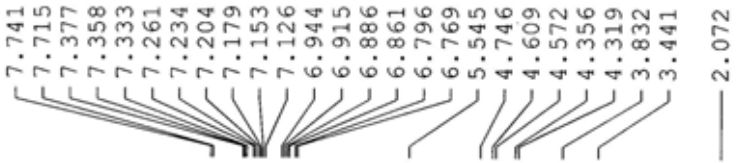
F2 - Acquisition Parameters
Date_ 20130319
Time 22.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 462
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

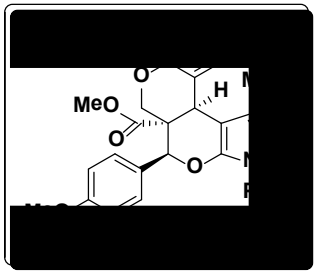
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

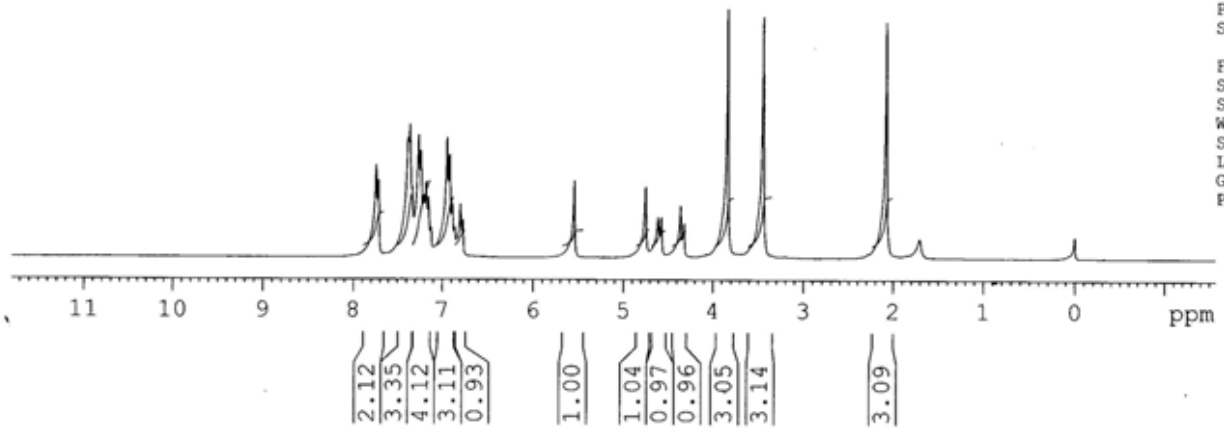
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

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









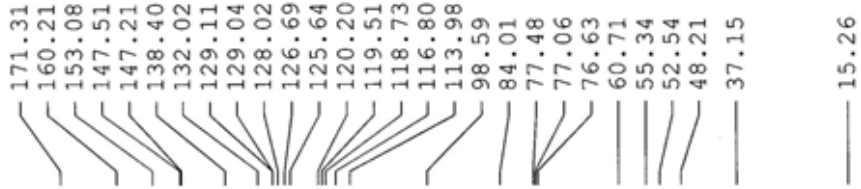
BRUKER

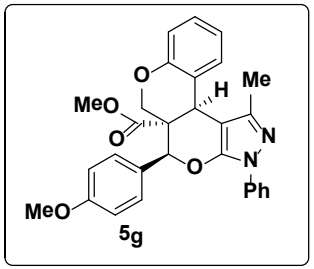
```
Current Data Parameters
NAME      PRSK-4-OME-EST
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130319
Time               22.29
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                  80.6
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

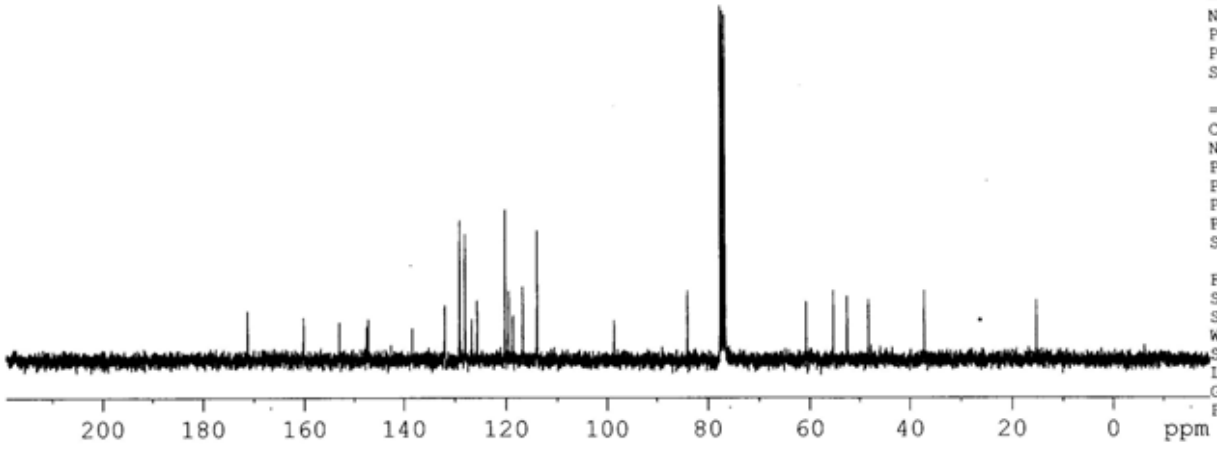
======== CHANNEL f1 ========
NUC1                  1H
P1                 13.15 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300085 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





5g



BRUKER

```
Current Data Parameters
NAME      PRSK-4-OME-EST
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130319
Time               22.34
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  144
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                1448.2
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

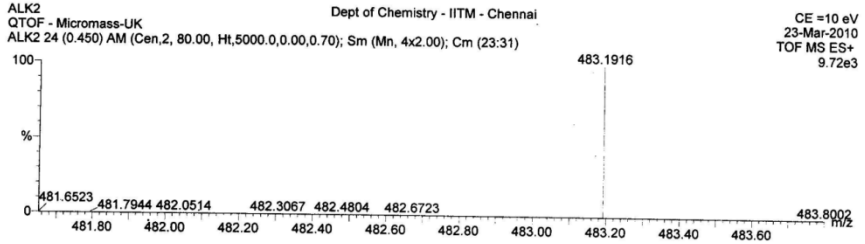
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.68 dB
PL13               16.00 dB
SFO2         300.1312005 MHz

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

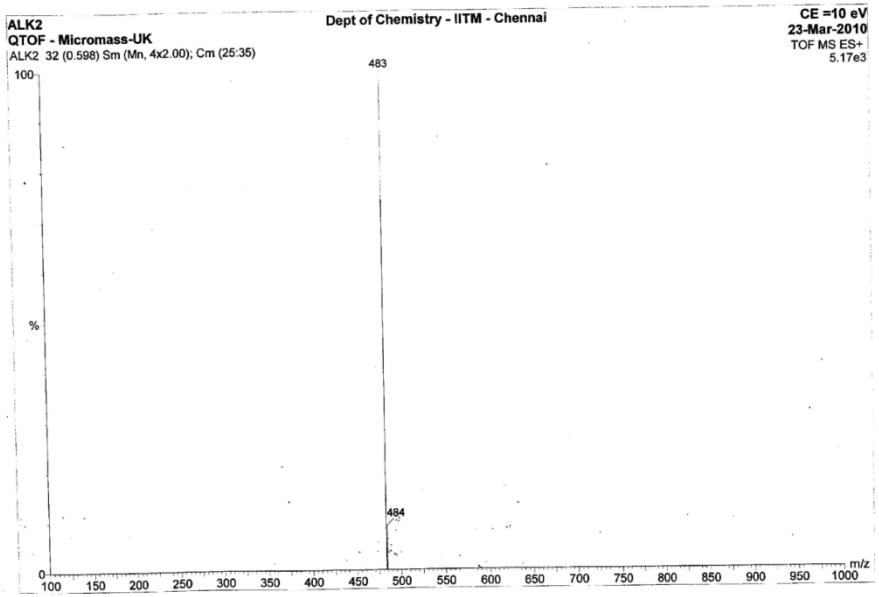
Elemental Composition Report

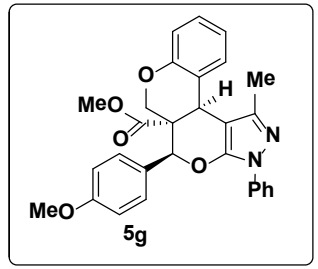
Single Mass Analysis
Tolerance = 200.0 mDa / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

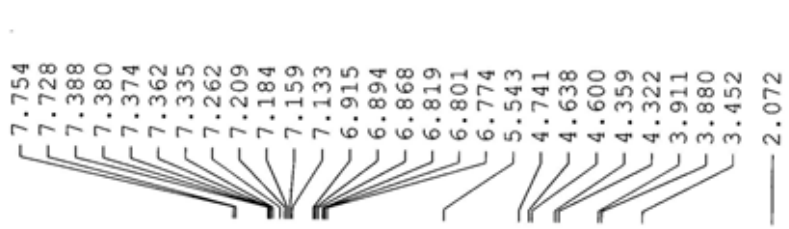


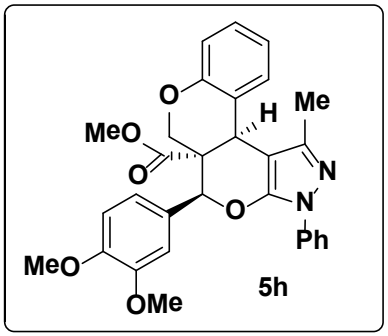
Minimum:				-1.5		
Maximum:		200.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
483.1916	483.1920	-0.4	-0.9	17.5	1	C29 H27 N2 O5

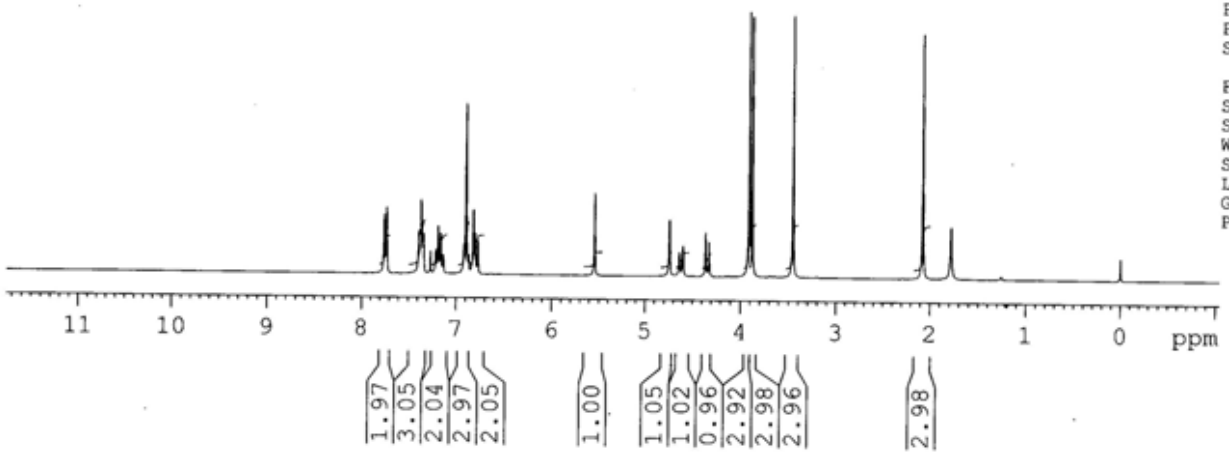




5g





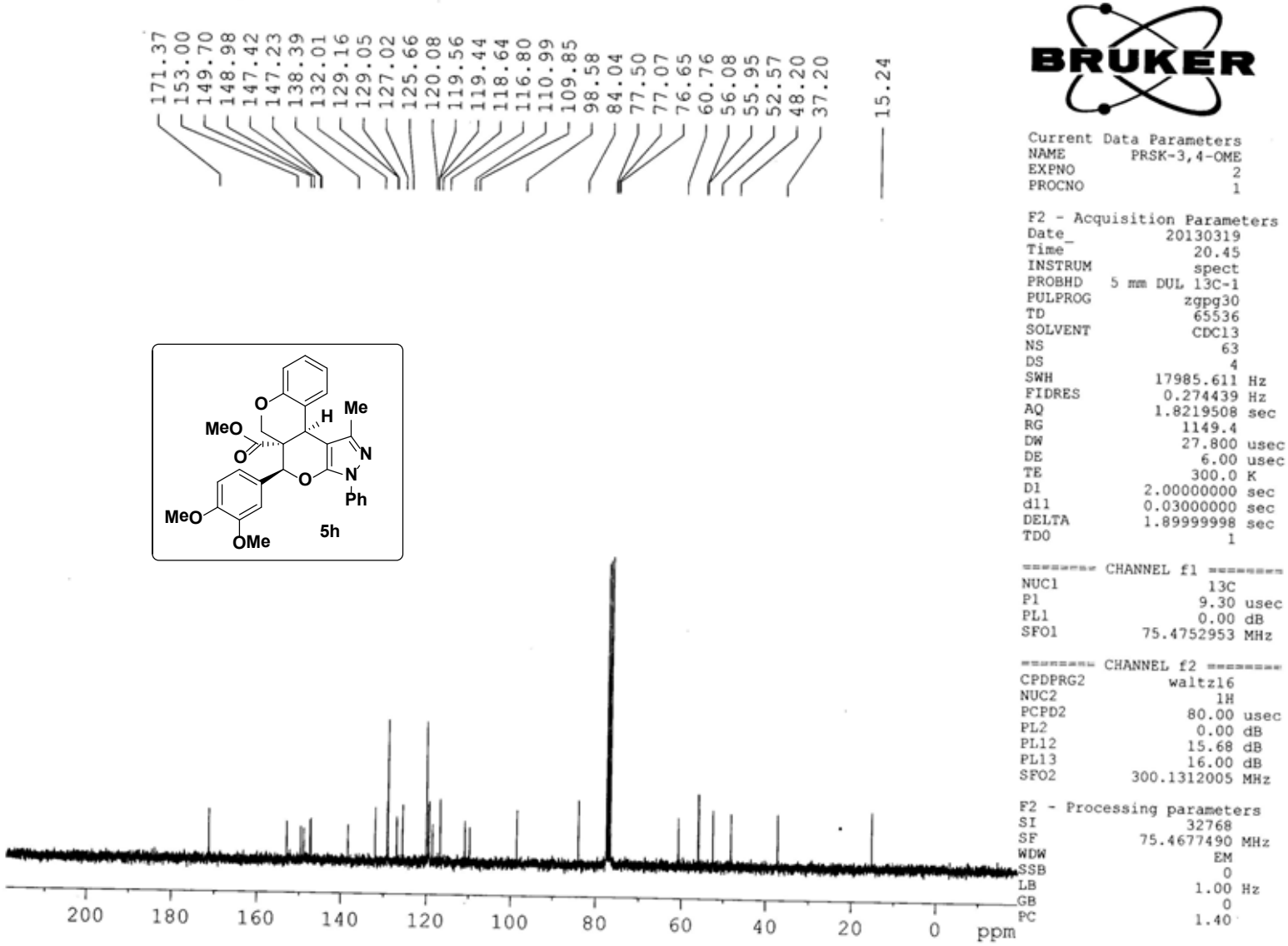


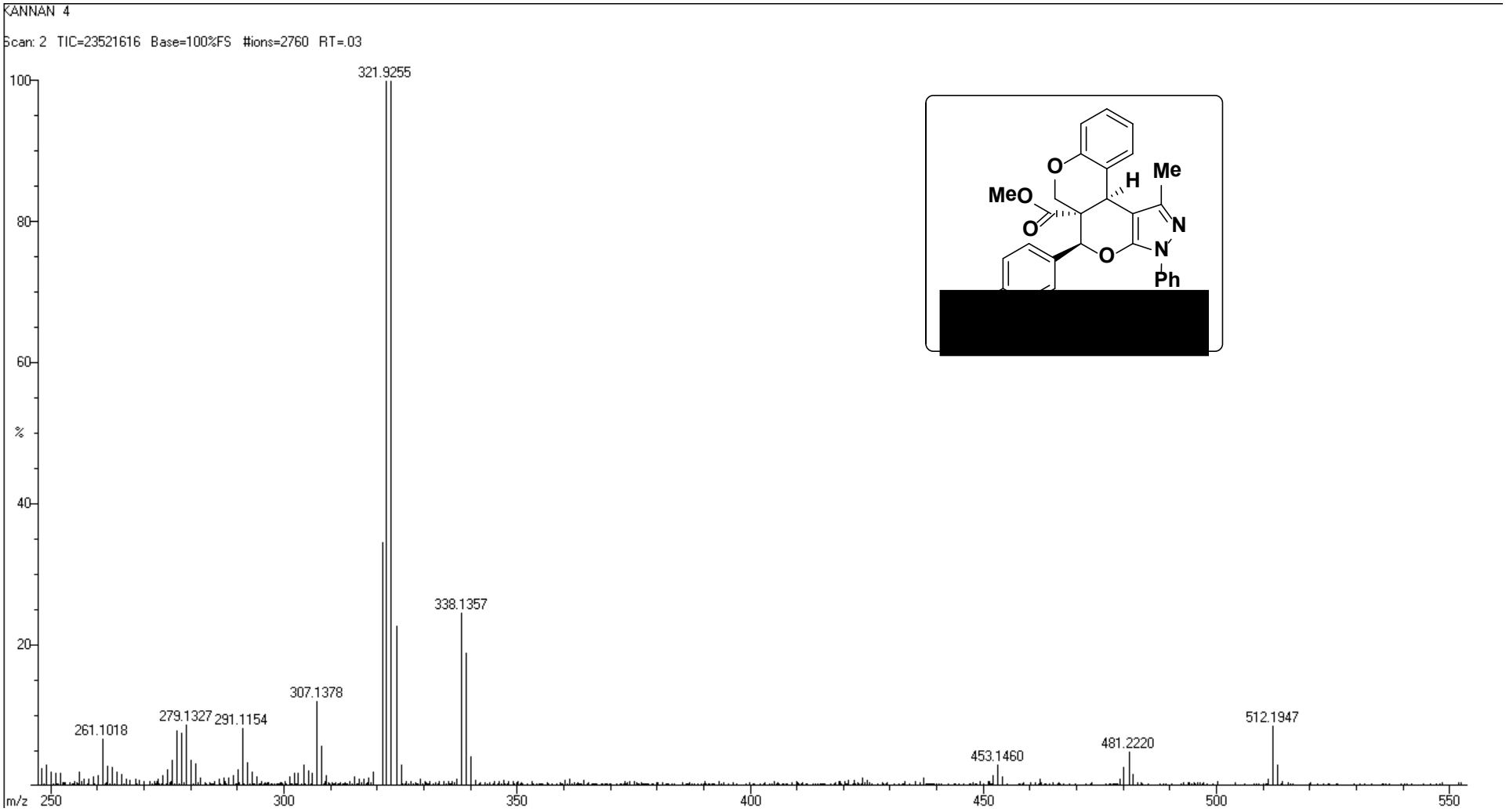

```
Current Data Parameters
NAME          PRSK-3,4-OME
EXPNO                    1
PROCNO                   1

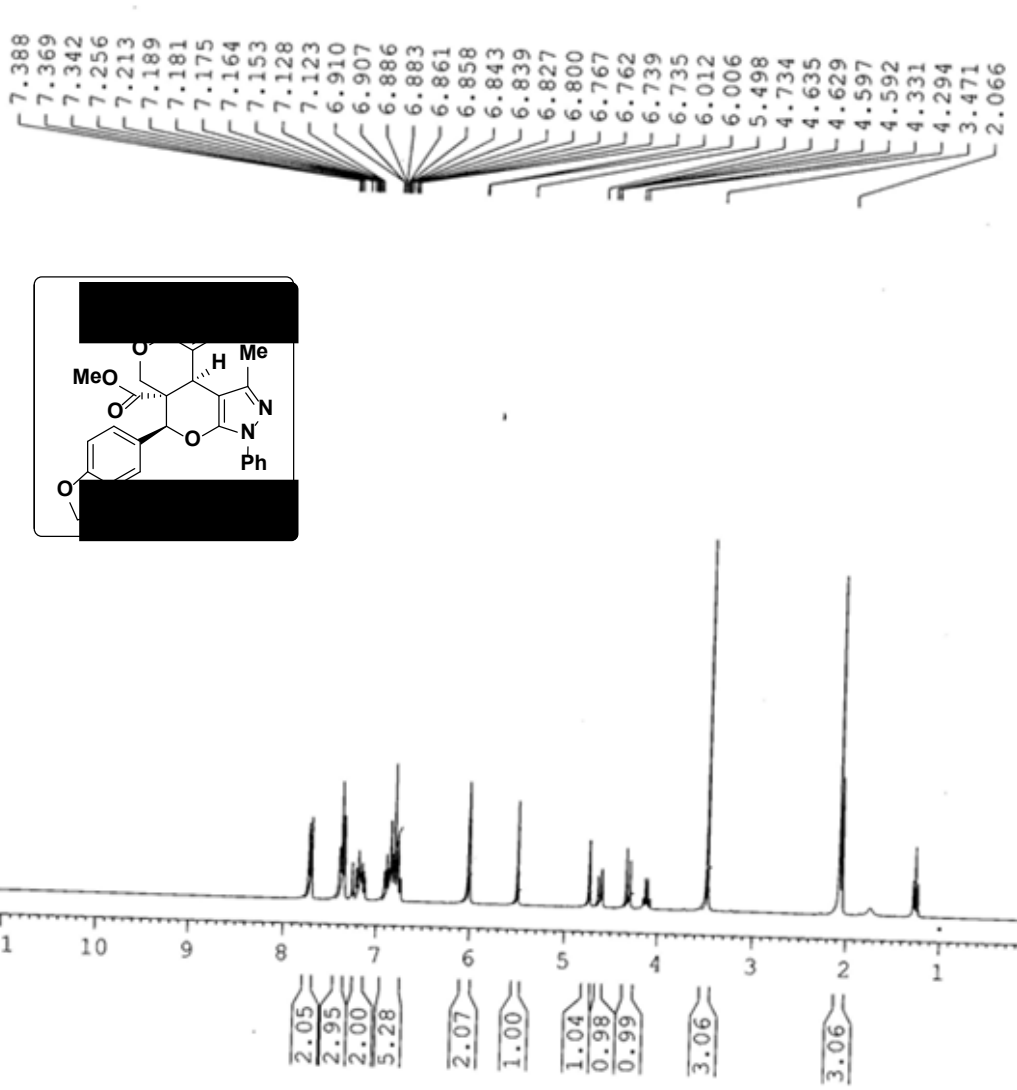
F2 - Acquisition Parameters
Date_             20130319
Time                 20.42
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                   71.8
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
```


171.37
153.00
149.70
148.98
147.42
147.23
138.39
132.01
129.16
129.05
127.02
125.66
120.08
119.56
119.44
118.64
116.80
110.99
109.85
98.58
84.04
77.50
77.07
76.65
60.76
56.08
55.95
52.57
48.20
37.20
15.24
MeO
O
O
H
Me
N
N
Ph
O
MeO
OMe
5h
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME PRSK-3,4-OME
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130319
Time 20.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 63
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1149.4
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





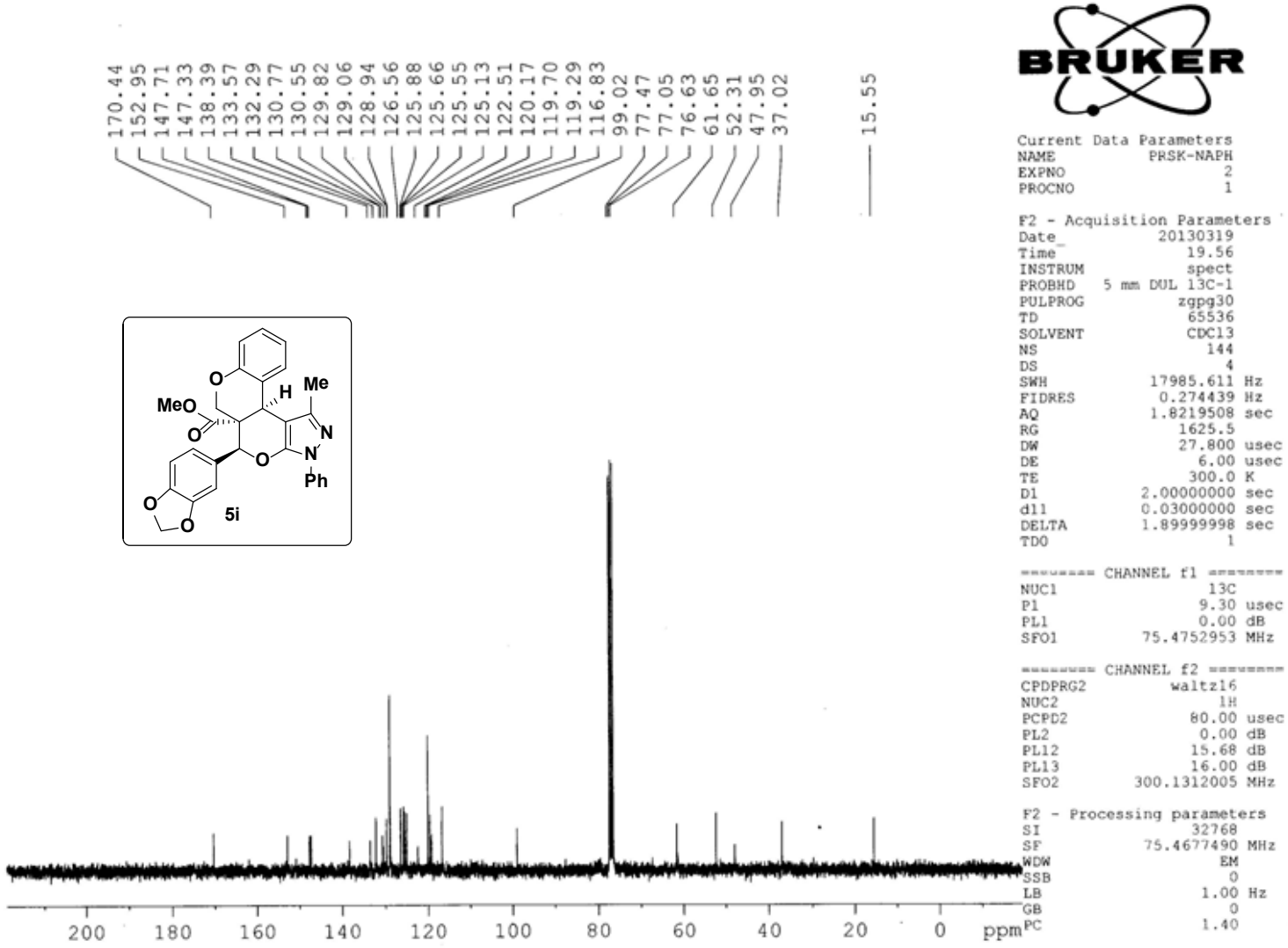
BRUKER

Current Data Parameters
NAME PRSK-PIP-EST
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130319
Time 22.49
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 80.6
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 300.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

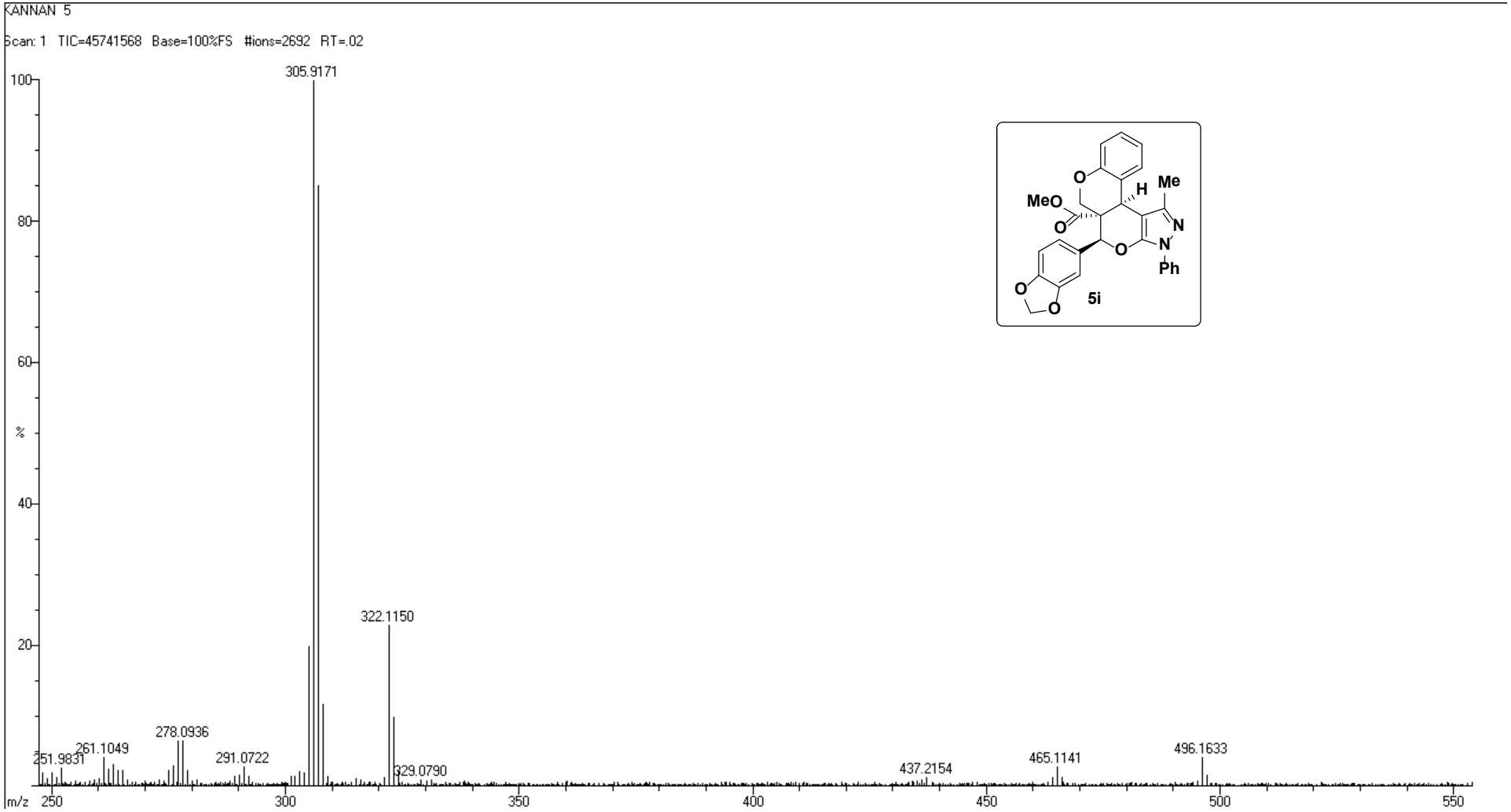
Current Data Parameters
NAME PRSK-NAPH
EXPNO 2
PROCNO 1

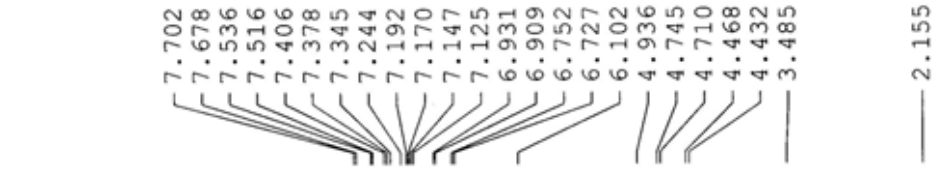
F2 - Acquisition Parameters
Date_ 20130319
Time 19.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 144
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

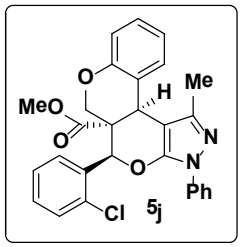
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

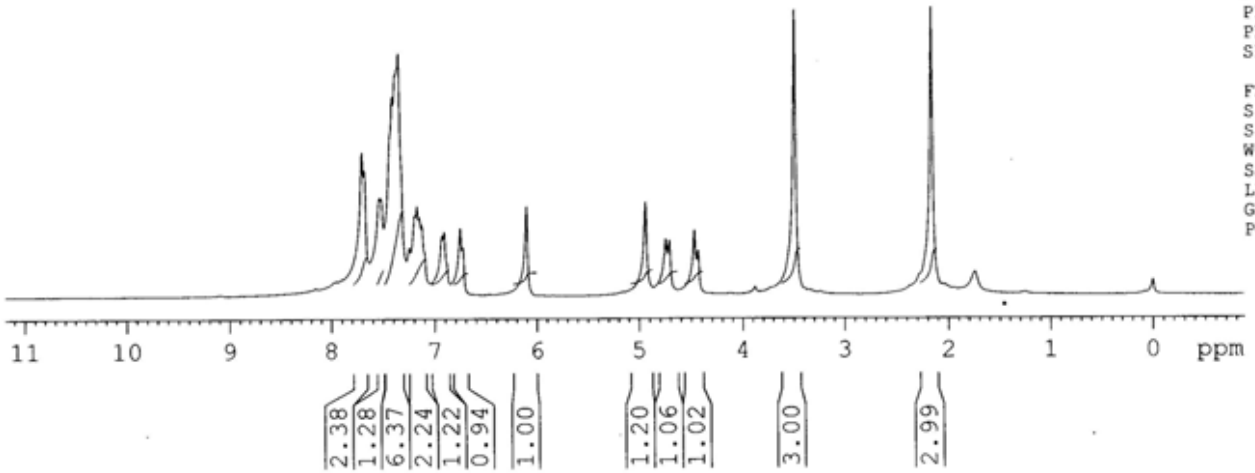
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

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


KANNAN 5
Scan: 1 TIC=45741568 Base=100%FS #ions=2692 RT=.02
305.9171
322.1150
278.0936
261.1049
251.9831
291.0722
329.0790
437.2154
465.1141
496.1633
%
m/z
MeO
Me
H
N
Ph
5i





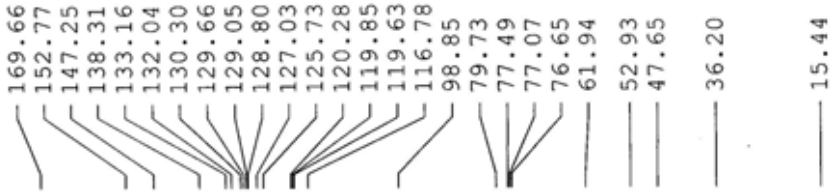


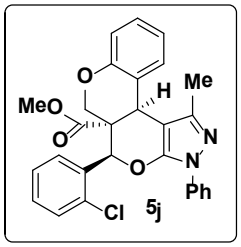

```
Current Data Parameters
NAME        PRSK-2-CL-EST
EXPNO                   1
PROCNO                  1

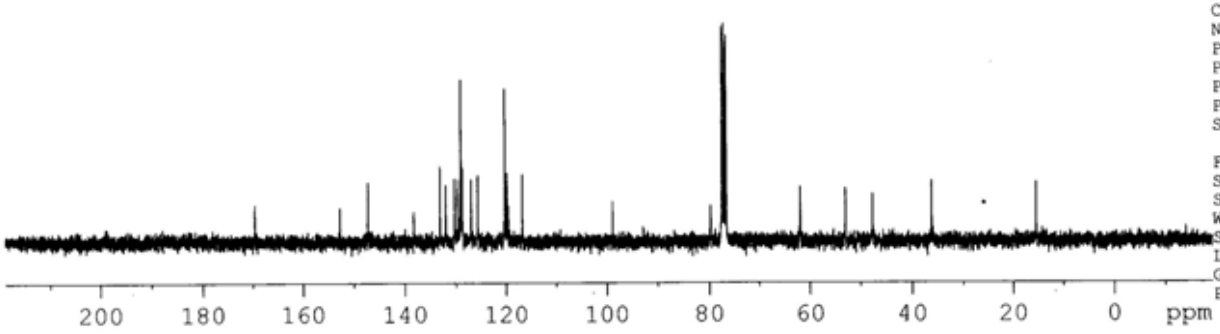
F2 - Acquisition Parameters
Date_            20130323
Time                18.15
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                   71.8
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           300.1300110 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```







Current Data Parameters

NAME	PRSK-2-CL-EST
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20130323	
Time	18.20	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	100	
DS	4	
SWH	17985.611	Hz
FIDRES	0.274439	Hz
AQ	1.8219508	sec
RG	912.3	
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 ========

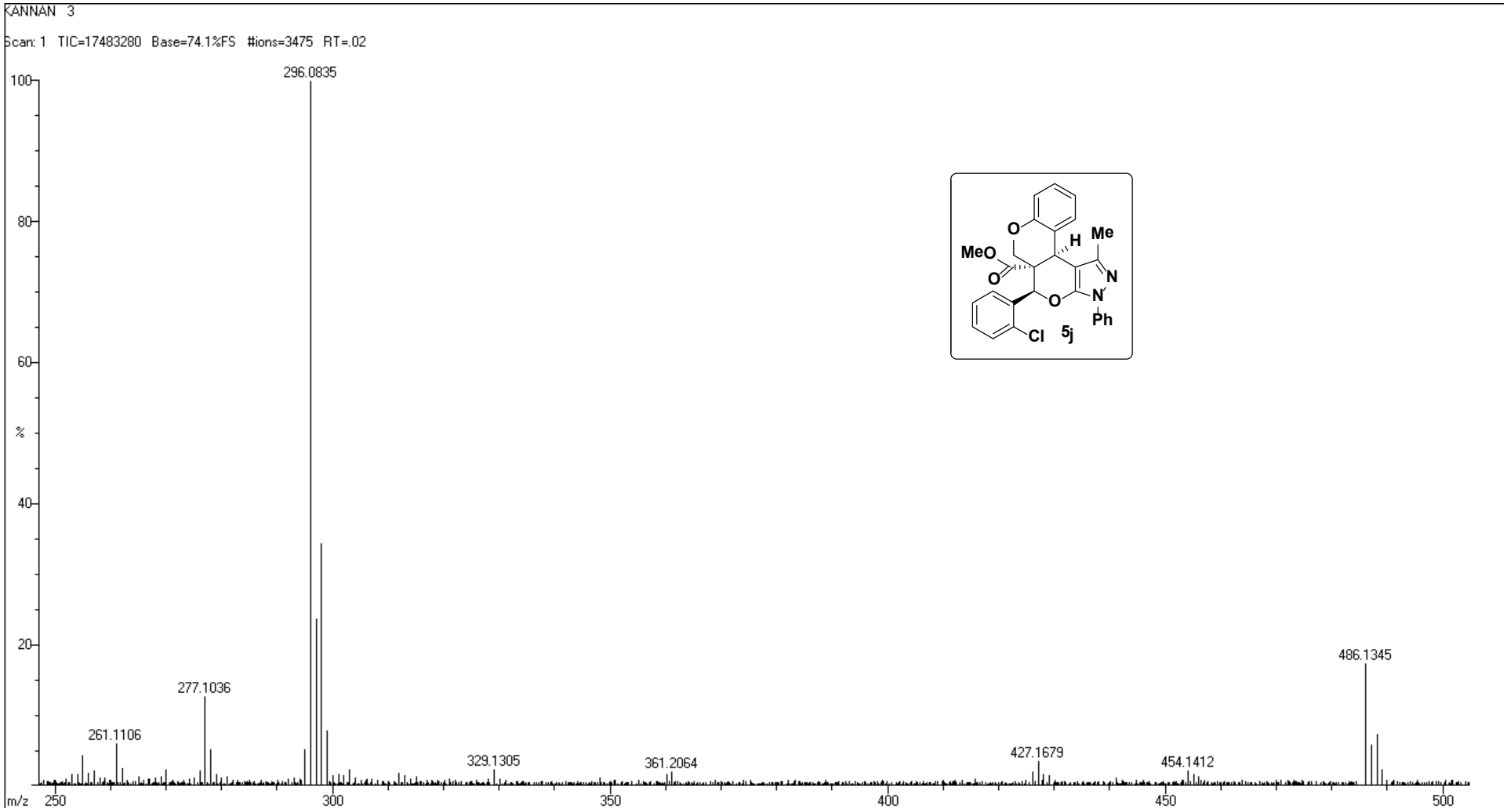
NUC1	13C	
P1	9.30	usec
PL1	0.00	dB
SFO1	75.4752953	MHz

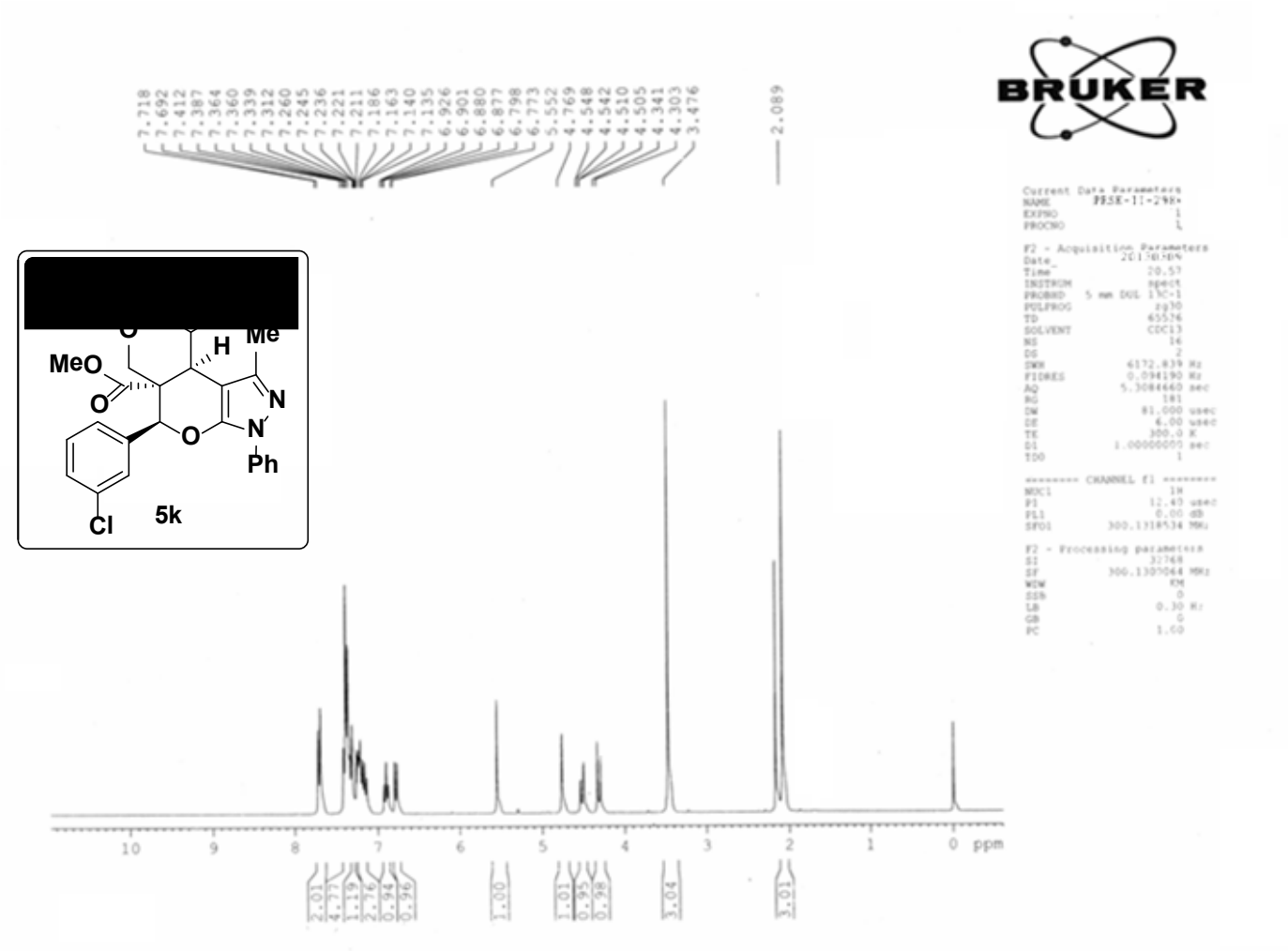
======== CHANNEL f2 ========

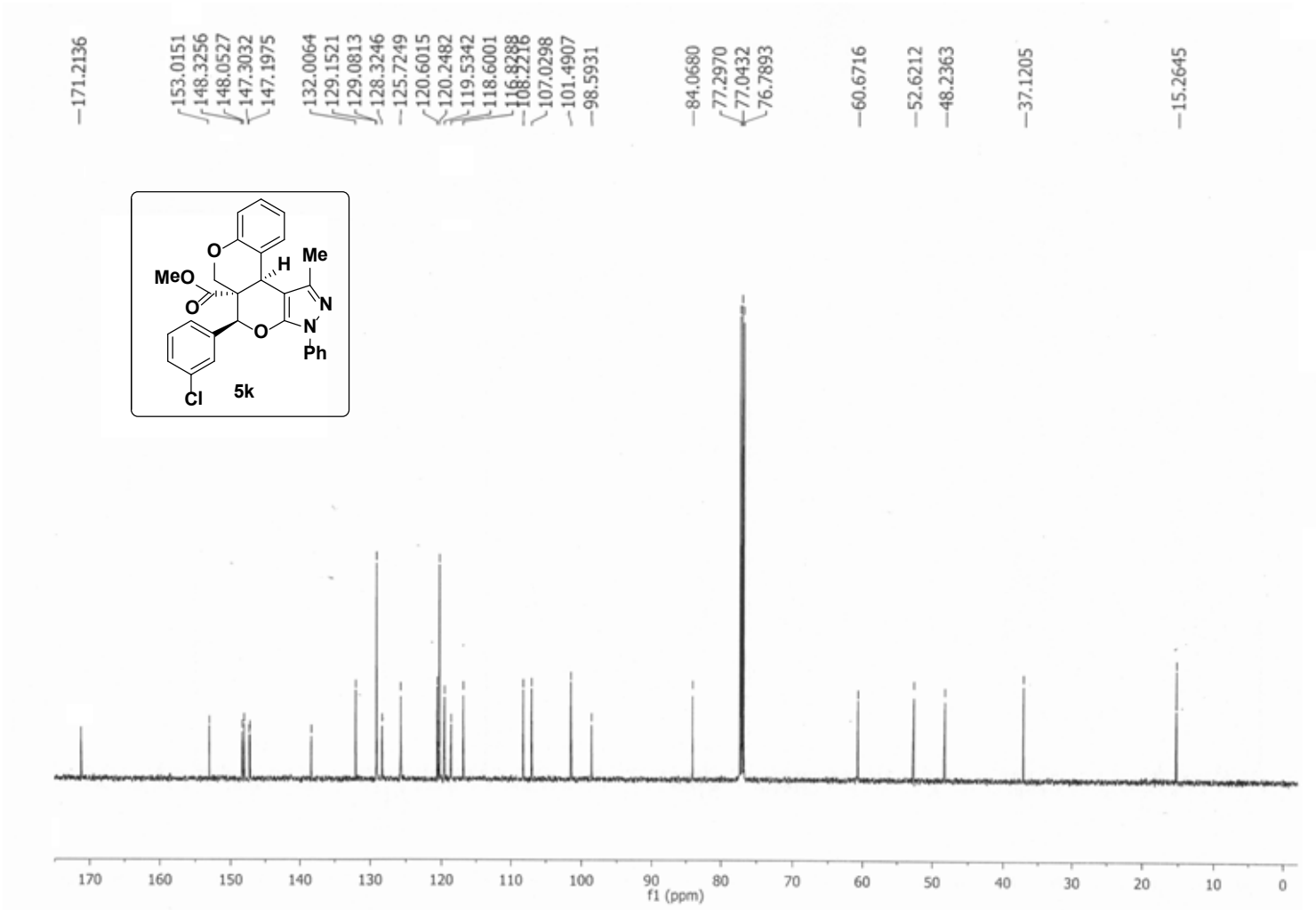
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	0.00	dB
PL12	15.68	dB
PL13	16.00	dB
SFO2	300.1312005	MHz

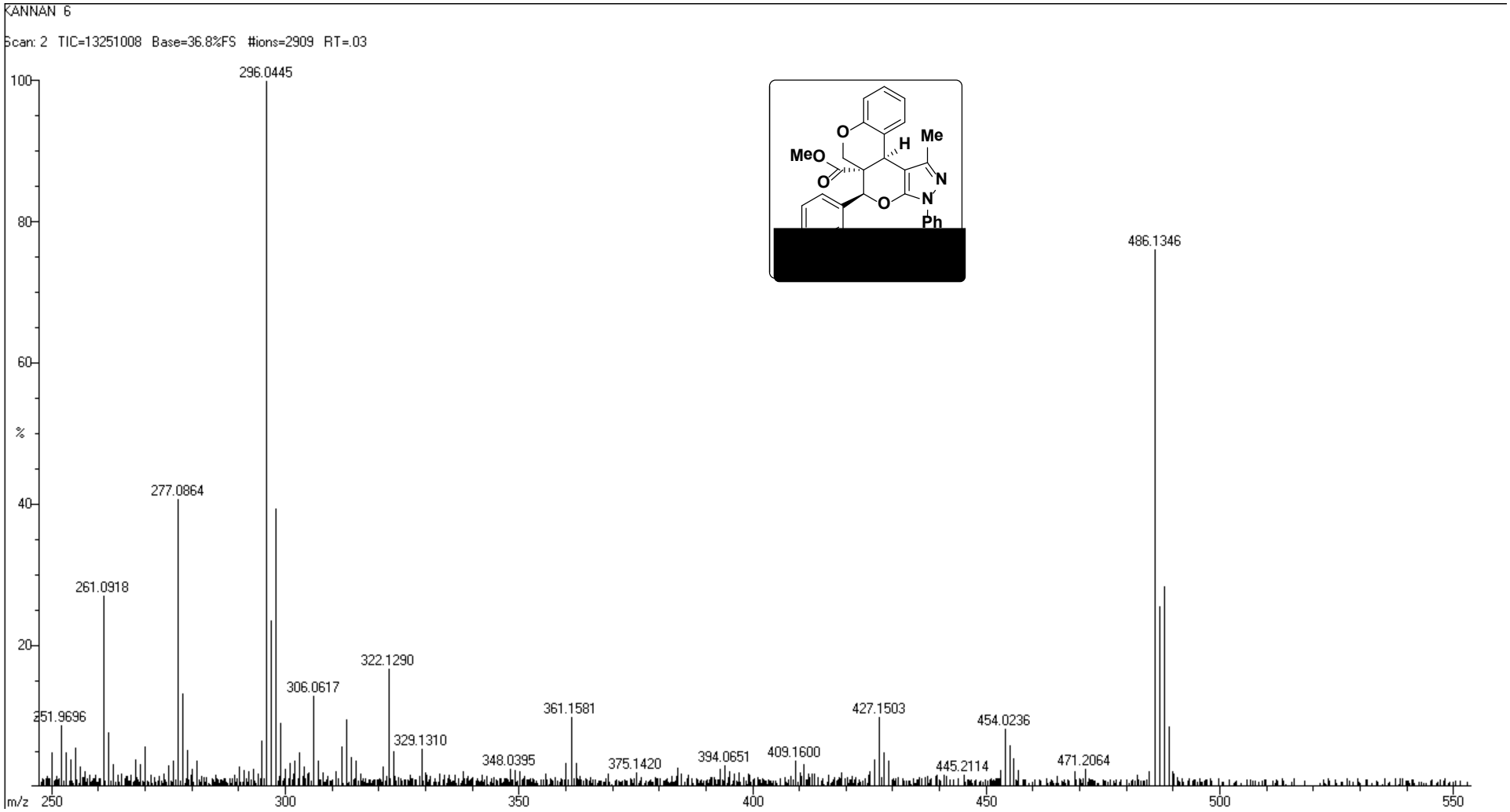
F2 - Processing parameters

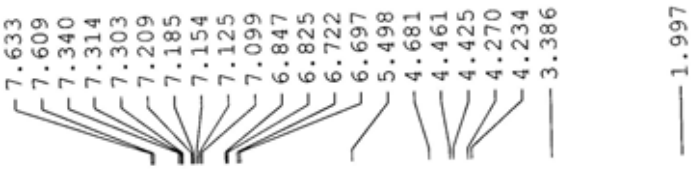
SI	32768	
SF	75.4677490	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

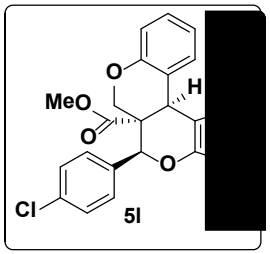


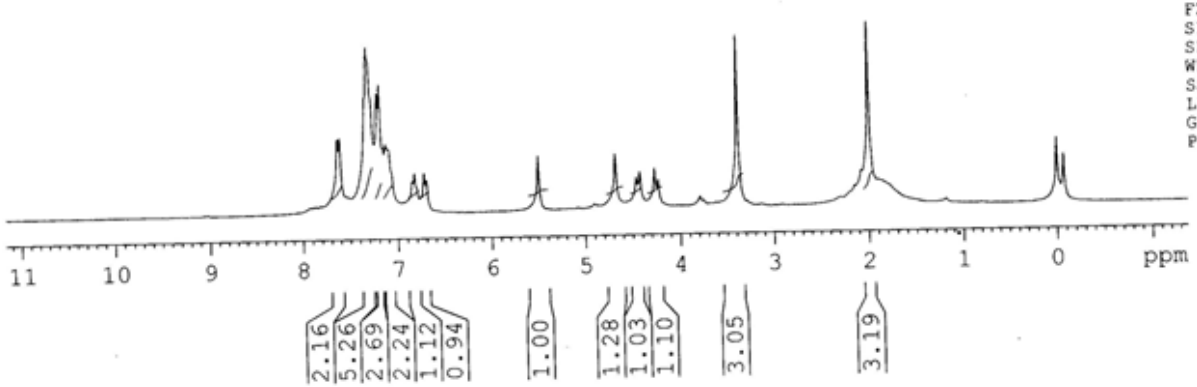










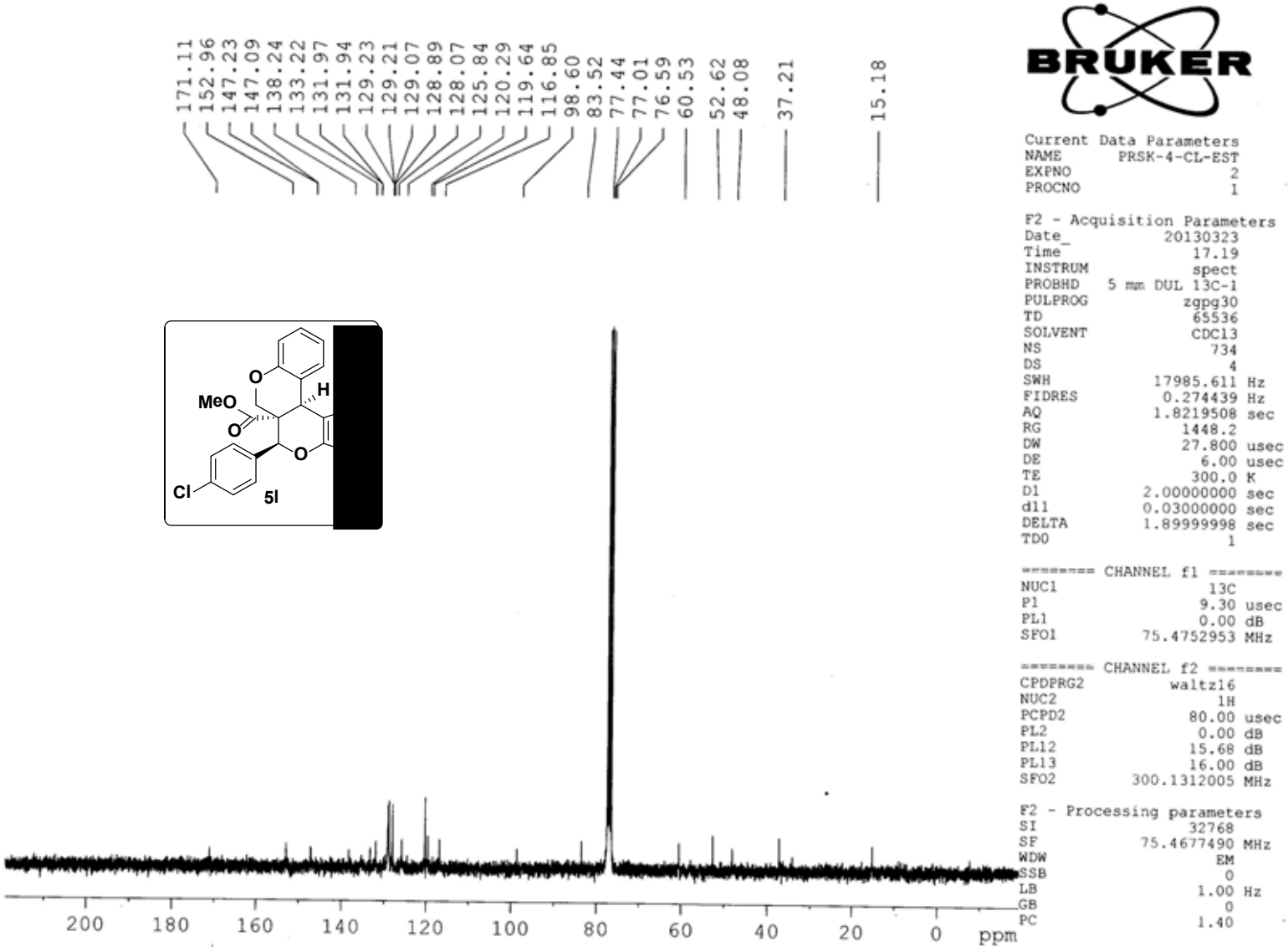



```
Current Data Parameters
NAME        PRSK-4-CL-EST
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130323
Time                18.07
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                  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           300.1300298 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```



BRUKER

Current Data Parameters
NAME PRSK-4-CL-EST
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20130323	
Time	17.19	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	734	
DS	4	
SWH	17985.611	Hz
FIDRES	0.274439	Hz
AQ	1.8219508	sec
RG	1448.2	
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 ========

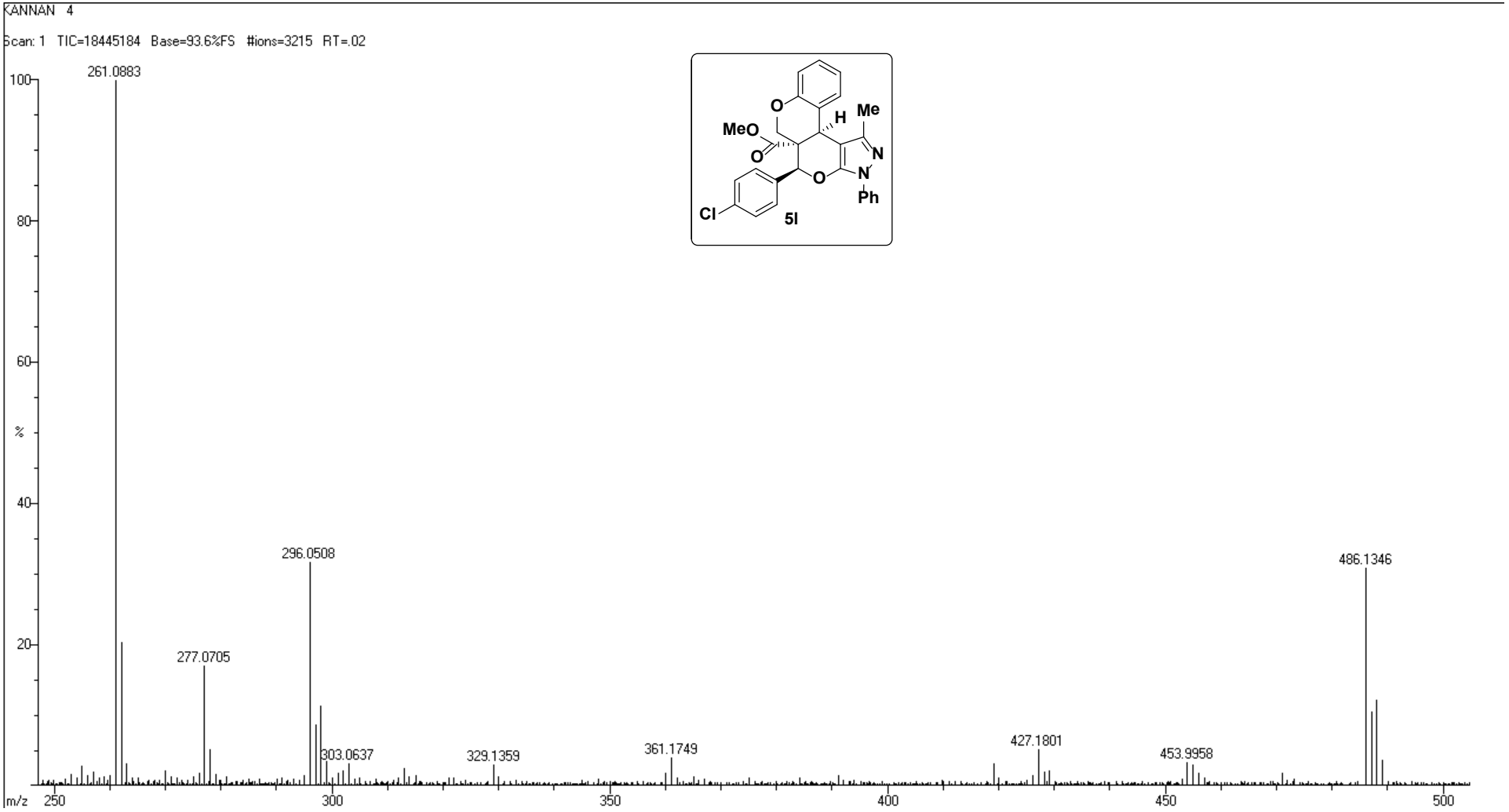
NUC1	13C	
P1	9.30	usec
PL1	0.00	dB
SFO1	75.4752953	MHz

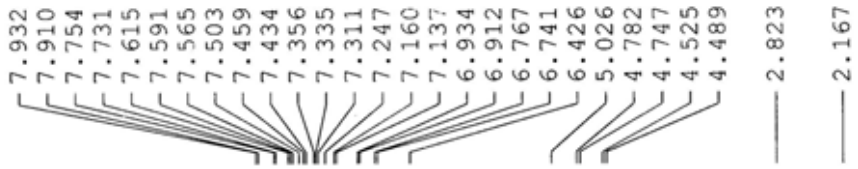
======== CHANNEL f2 ========

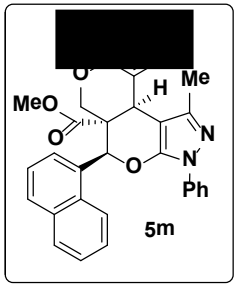
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	0.00	dB
PL12	15.68	dB
PL13	16.00	dB
SFO2	300.1312005	MHz

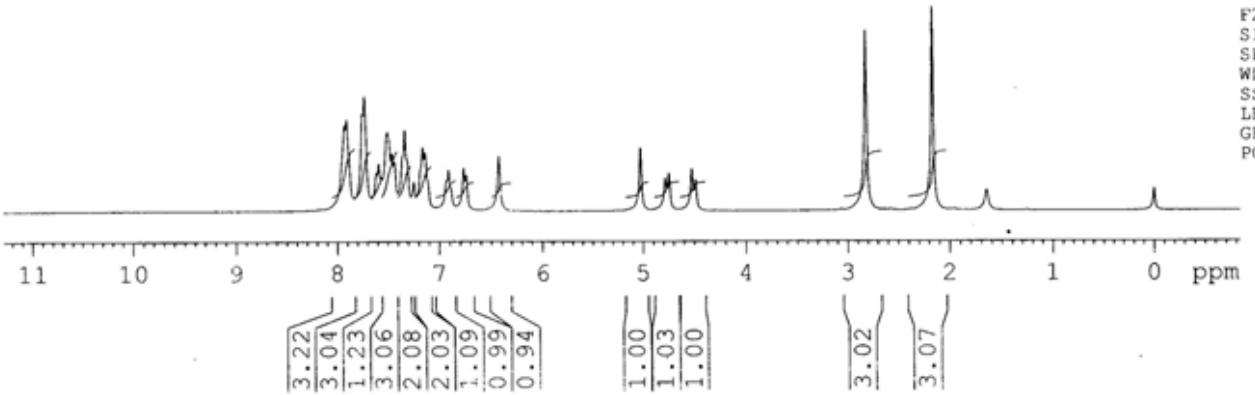
F2 - Processing parameters

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







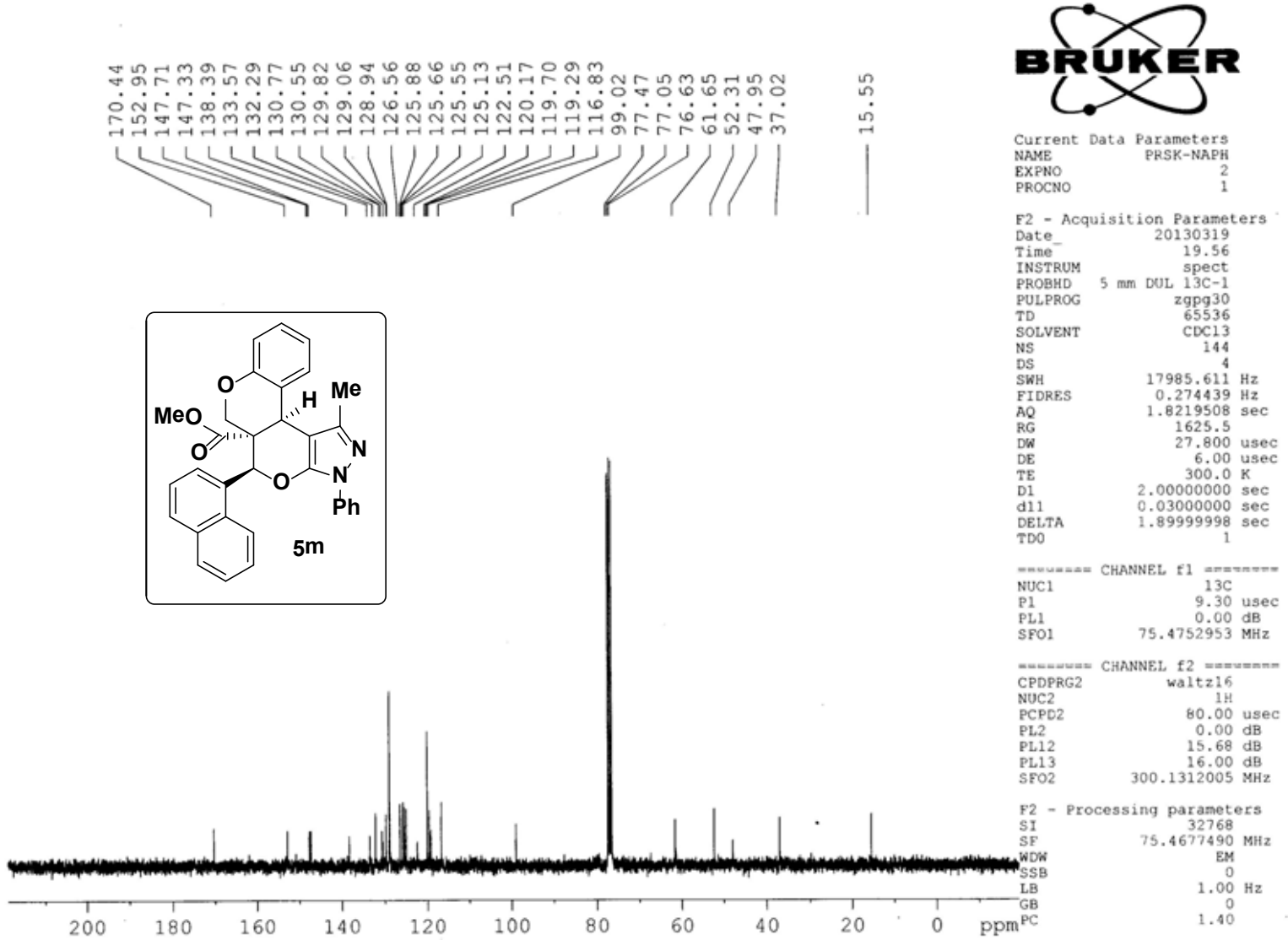



```
Current Data Parameters
NAME                 PRSK-NAPH
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130319
Time                   19.51
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                      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            300.1300115 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



BRUKER

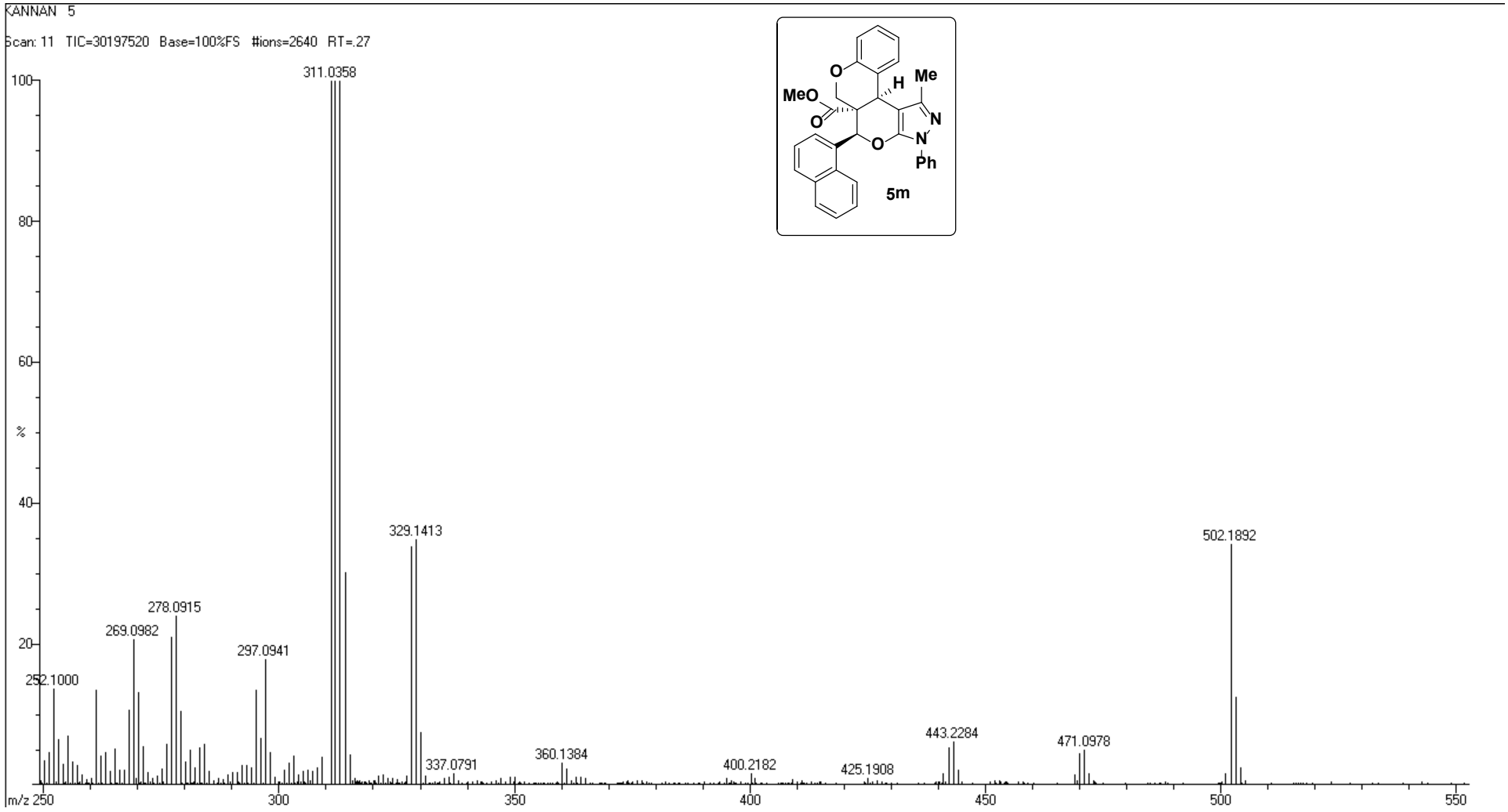
Current Data Parameters
NAME PRSK-NAPH
EXPNO 2
PROCNO 1

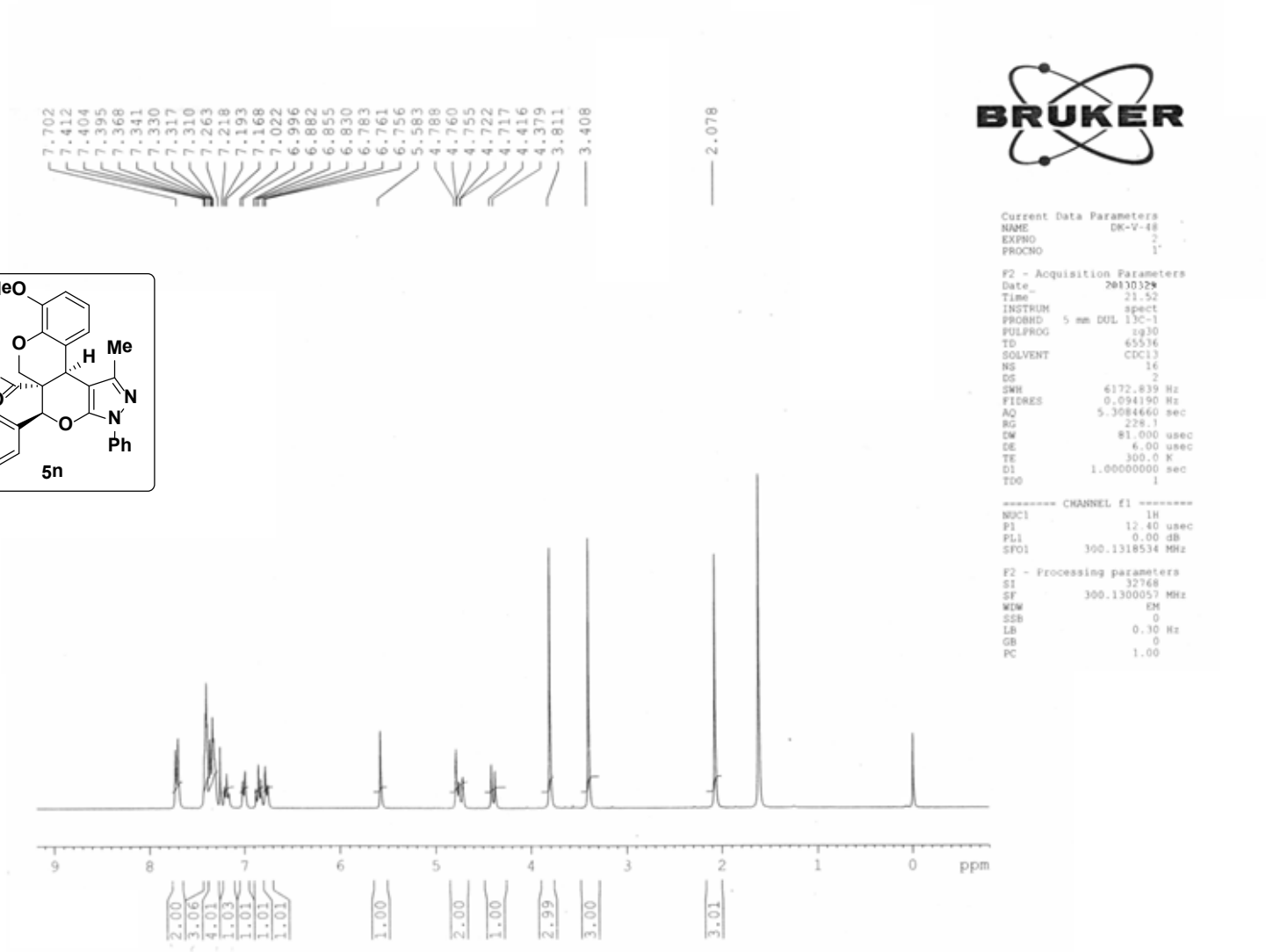
F2 - Acquisition Parameters
Date_ 20130319
Time 19.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 144
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

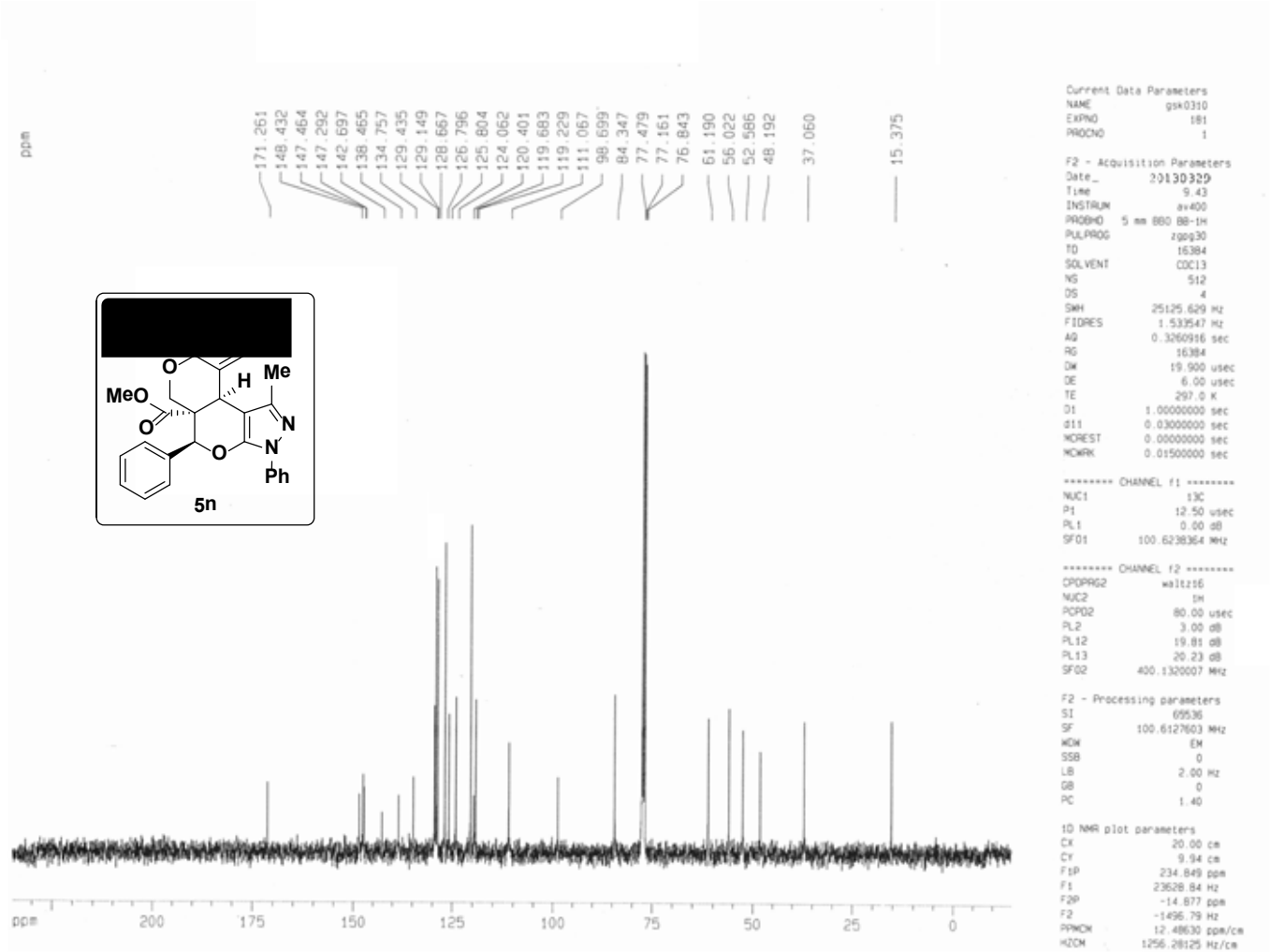
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== 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








```
Current Data Parameters
NAME                  gsk0310
EXPNO                     181
PROCNO                      1

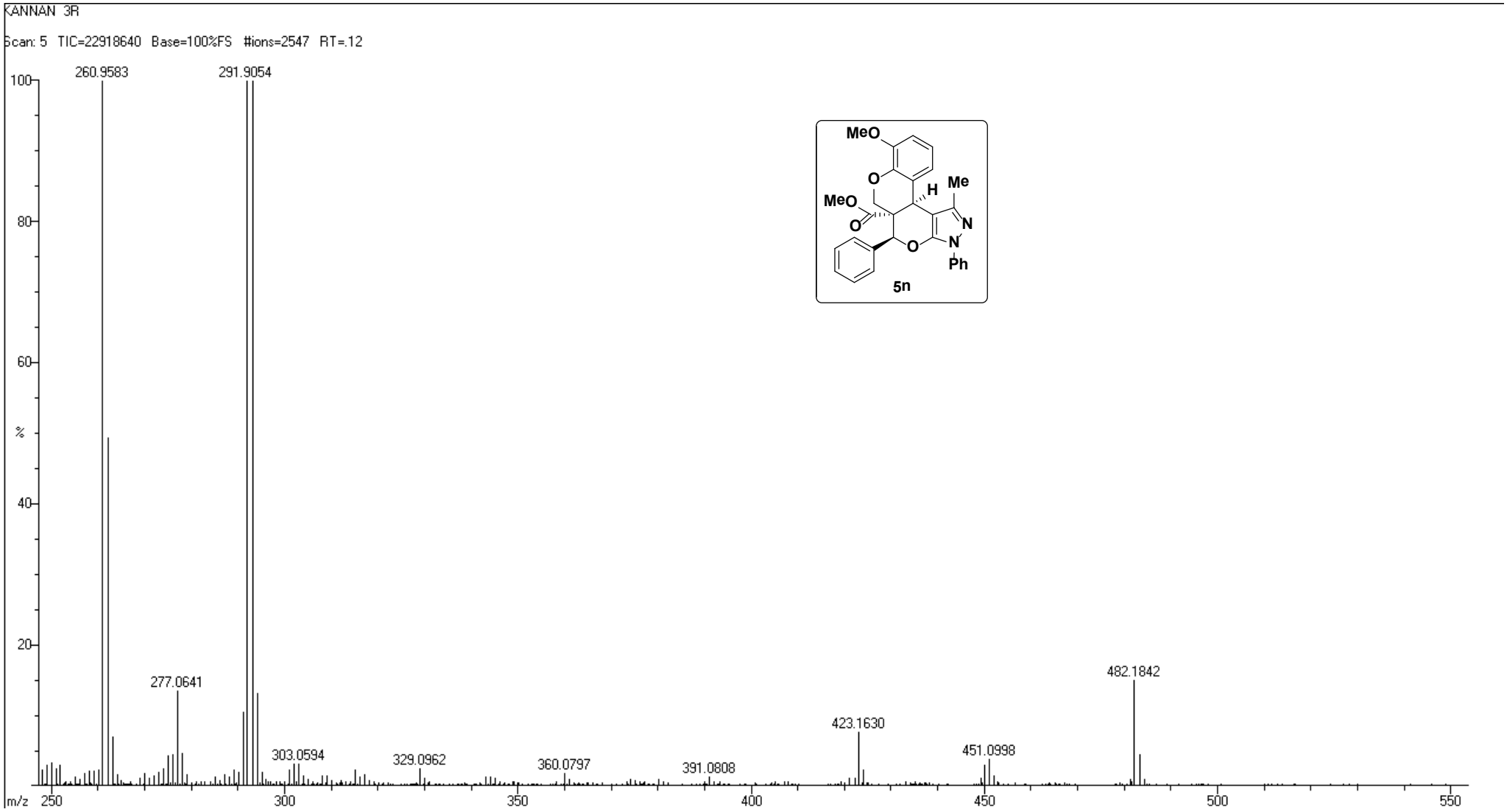
F2 - Acquisition Parameters
Date_              20130329
Time                    9.43
INSTRUM                av400
PROBHD  5 mm BBO BB-1H
PULPROG               zgpg30
TD                     16384
SOLVENT                CDCl3
NS                       512
DS                         4
SWH                25125.629 Hz
FIDRES              1.533547 Hz
AQ                  0.3260916 sec
RG                     16384
DW                  19.900 usec
DE                   6.00 usec
TE                   297.0 K
D1                  1.00000000 sec
d11                 0.03000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

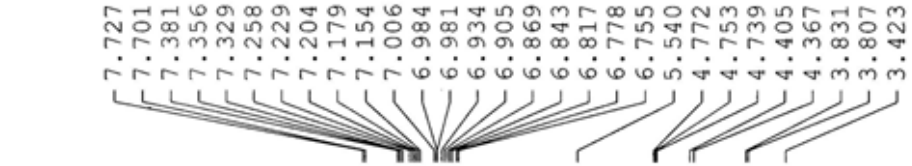
======== CHANNEL f1 ========
NUC1                      13C
P1                   12.50 usec
PL1                   0.00 dB
SFO1             100.6238364 MHz

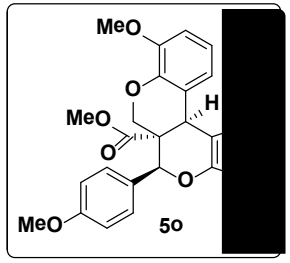
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                80.00 usec
PL2                   3.00 dB
PL12                 19.81 dB
PL13                 20.23 dB
SFO2             400.1320007 MHz

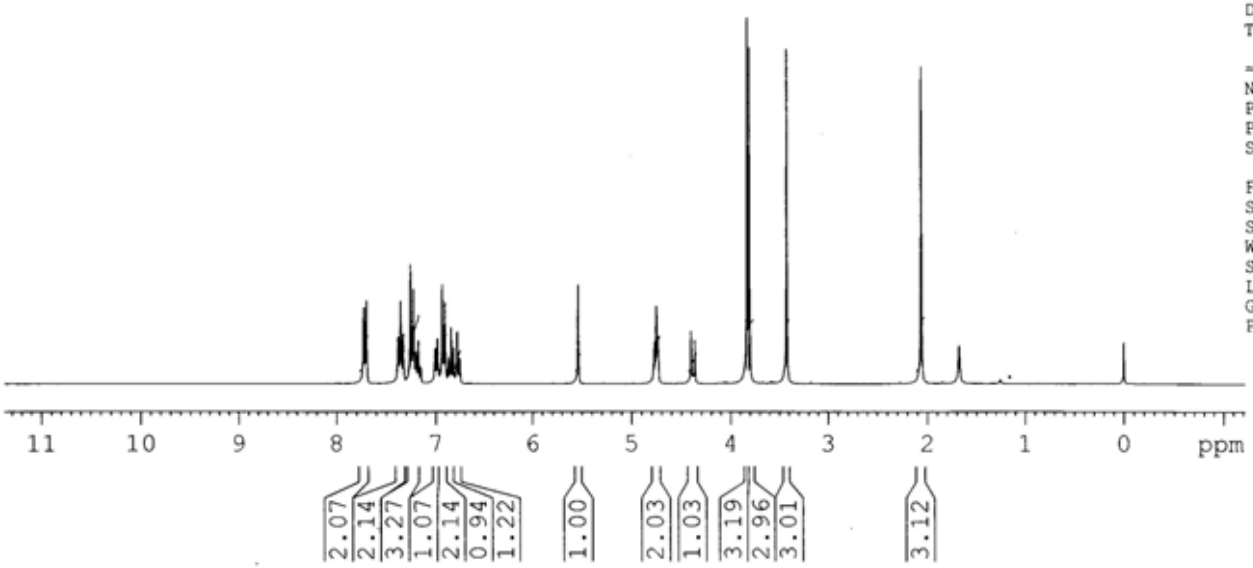
F2 - Processing parameters
SI                       65536
SF               100.6127603 MHz
WDW                       EM
SSB                        0
LB                    2.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    9.94 cm
F1P                 234.849 ppm
F1                  23628.84 Hz
F2P                 -14.877 ppm
F2                  -1496.79 Hz
PPMCM              12.48630 ppm/cm
HZCM               1256.28125 Hz/cm
```


KANNAN 3R
Scan: 5 TIC=22918640 Base=100%FS #ions=2547 RT=.12
260.9583
291.9054
277.0641
303.0594
329.0962
360.0797
391.0808
423.1630
451.0998
482.1842
100
80
60
%
40
20
m/z
250
300
350
400
450
500
550
MeO
O
H
Me
MeO
O
N
N
O
Ph
5n





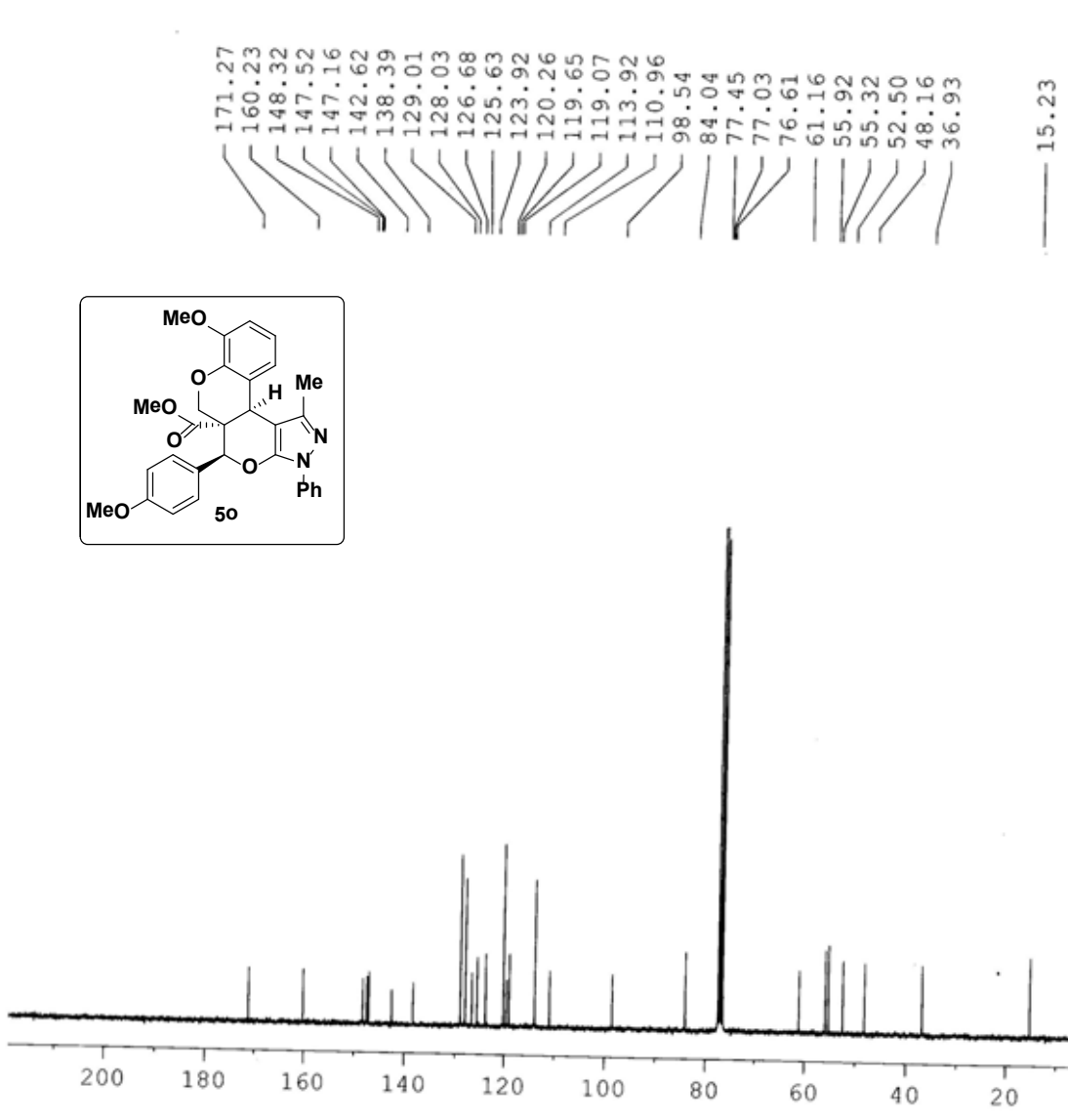



```
Current Data Parameters
NAME      prsk-3-OMe-2-OMe
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130324
Time                 23.38
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                   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            300.1300071 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



BRUKER

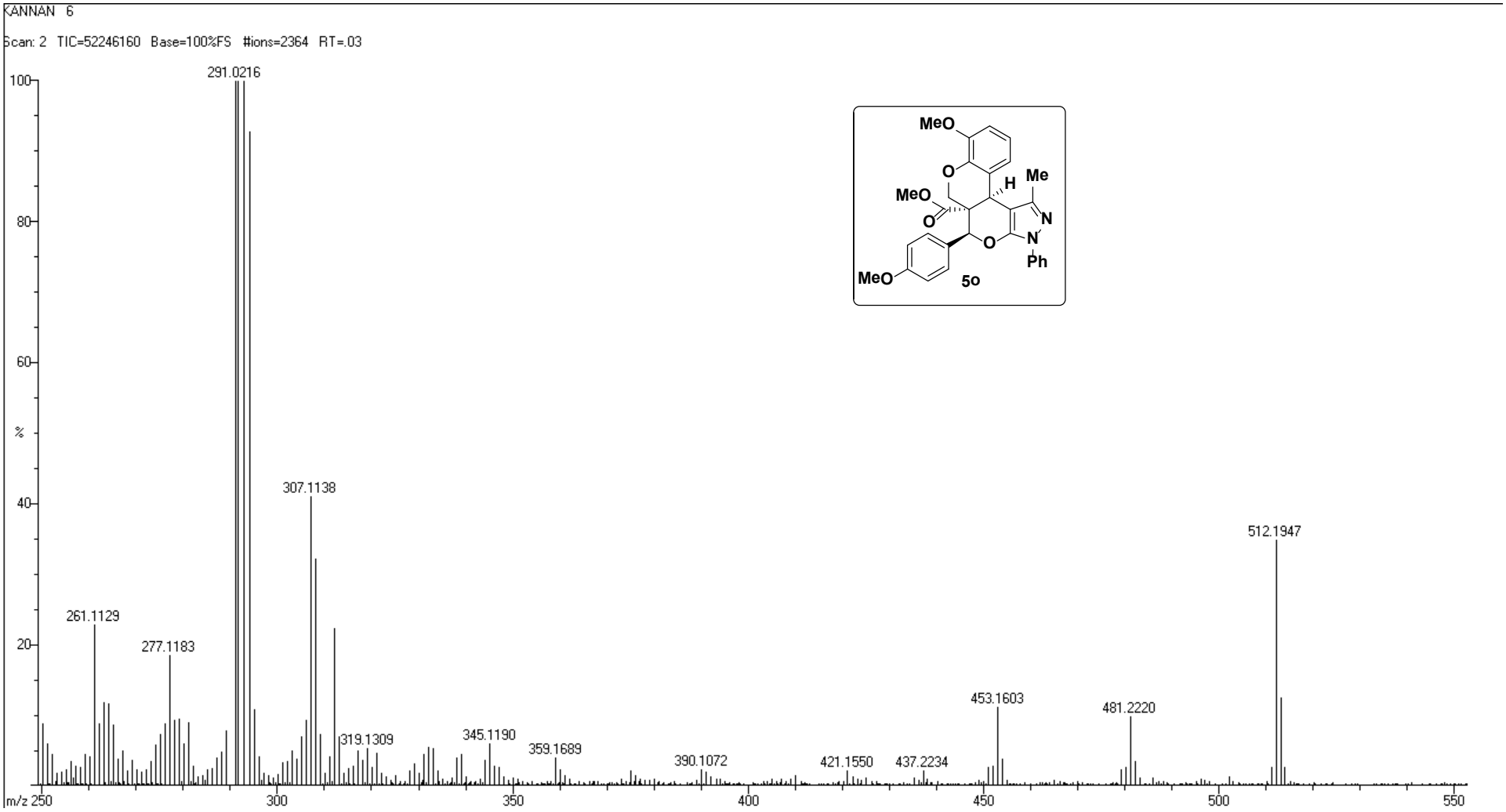
Current Data Parameters
NAME prsk-3-OMe-2-OMe
EXPNO 2
PROCNO 1

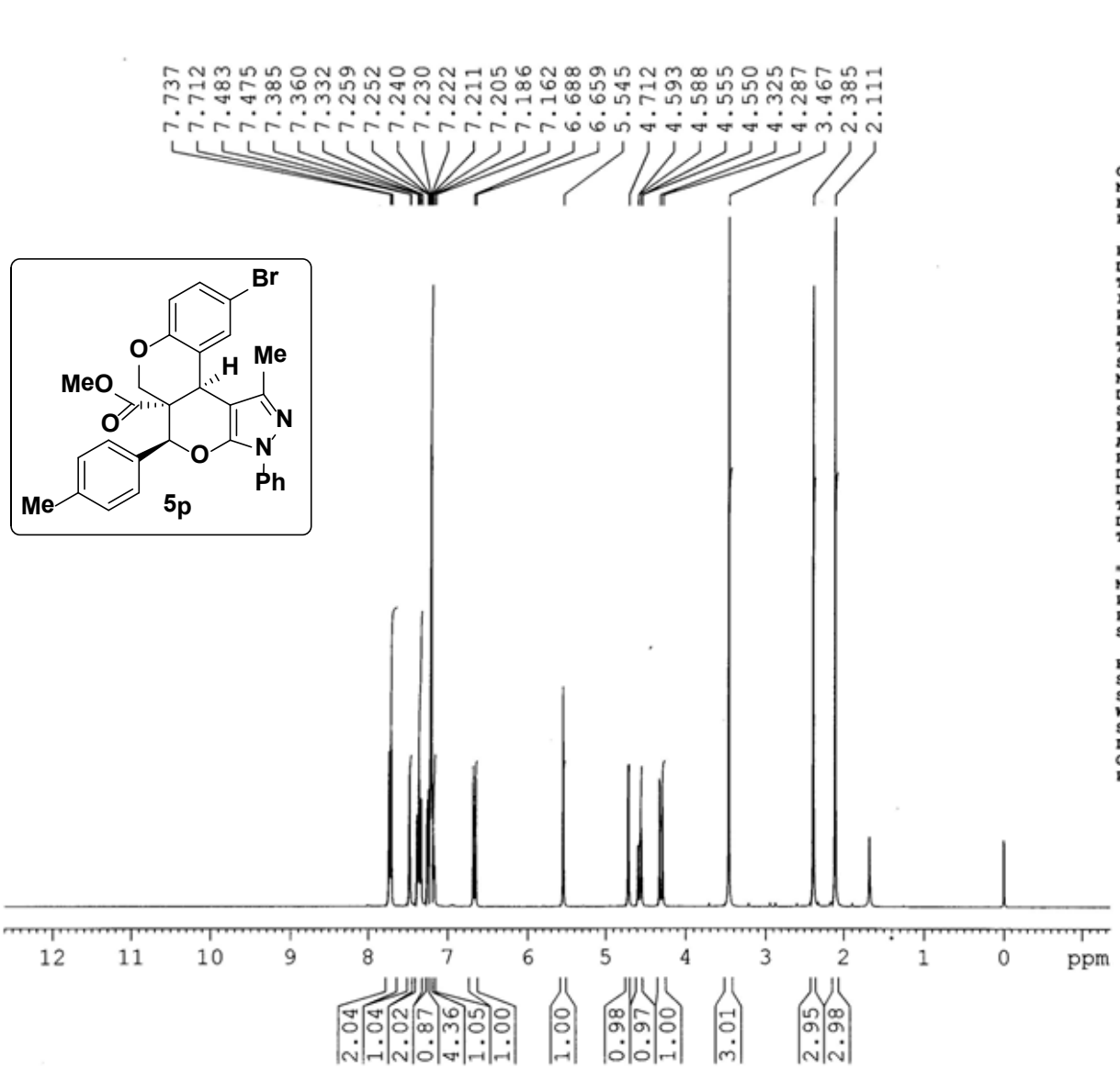
F2 - Acquisition Parameters
Date_ 20130325
Time 0.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 908
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2298.8
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 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


KANNAN 6
Scan: 2 TIC=52246160 Base=100%FS #ions=2364 RT=.03
291.0216
307.1138
261.1129
277.1183
319.1309
345.1190
359.1689
390.1072
421.1550
437.2234
453.1603
481.2220
512.1947
%
m/z
MeO
O
H
Me
MeO
O
N
N
O
Ph
MeO
5o



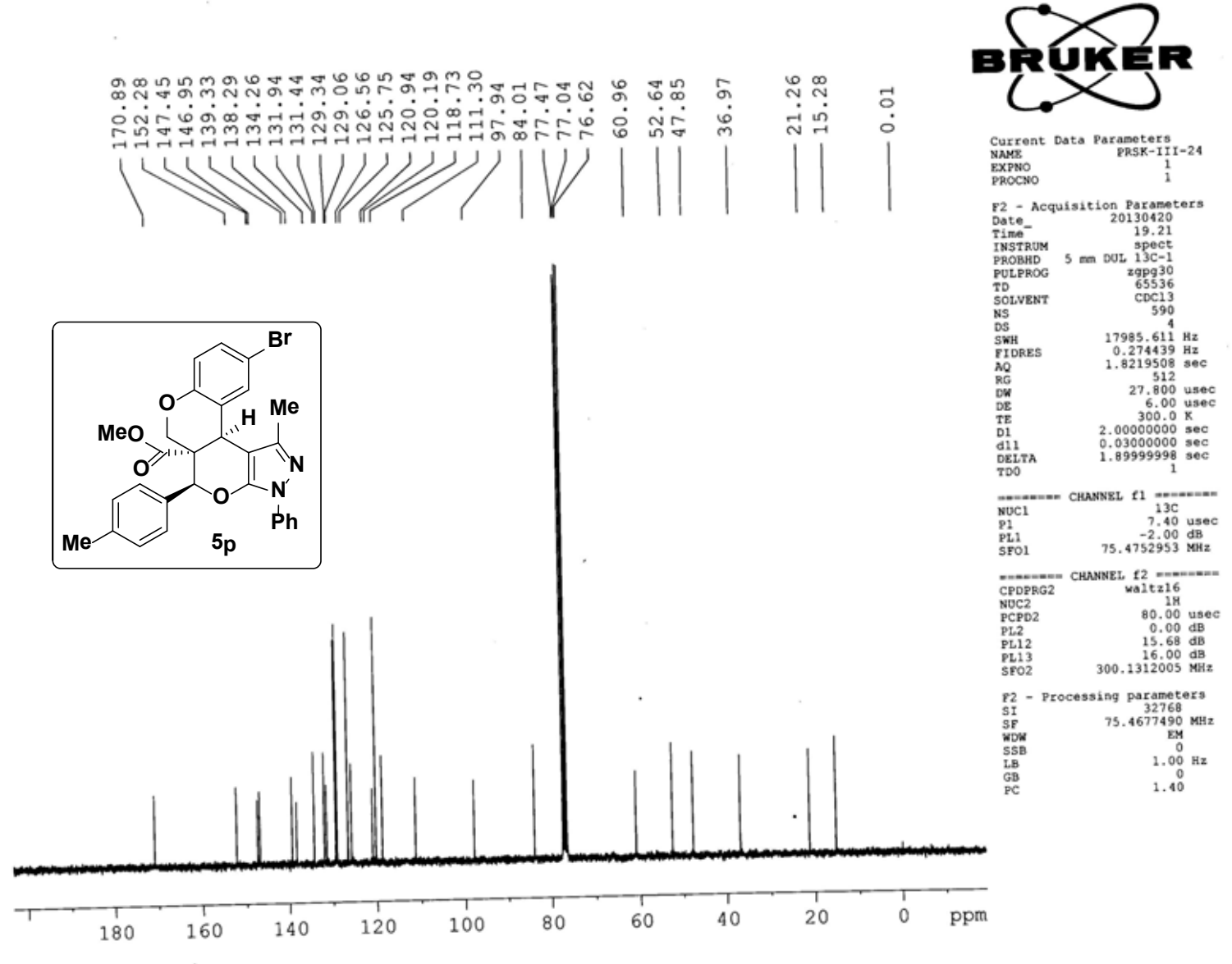
BRUKER

```
Current Data Parameters
NAME                  PRSK-III-24
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20130501
Time                        17.29
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                           90.5
DW                          81.000 usec
DE                           6.00 usec
TE                          300.0 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          12.40 usec
PL1                          0.00 dB
SFO1                 300.1318534 MHz

F2 - Processing parameters
SI                          32768
SF                   300.1300082 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



BRUKER

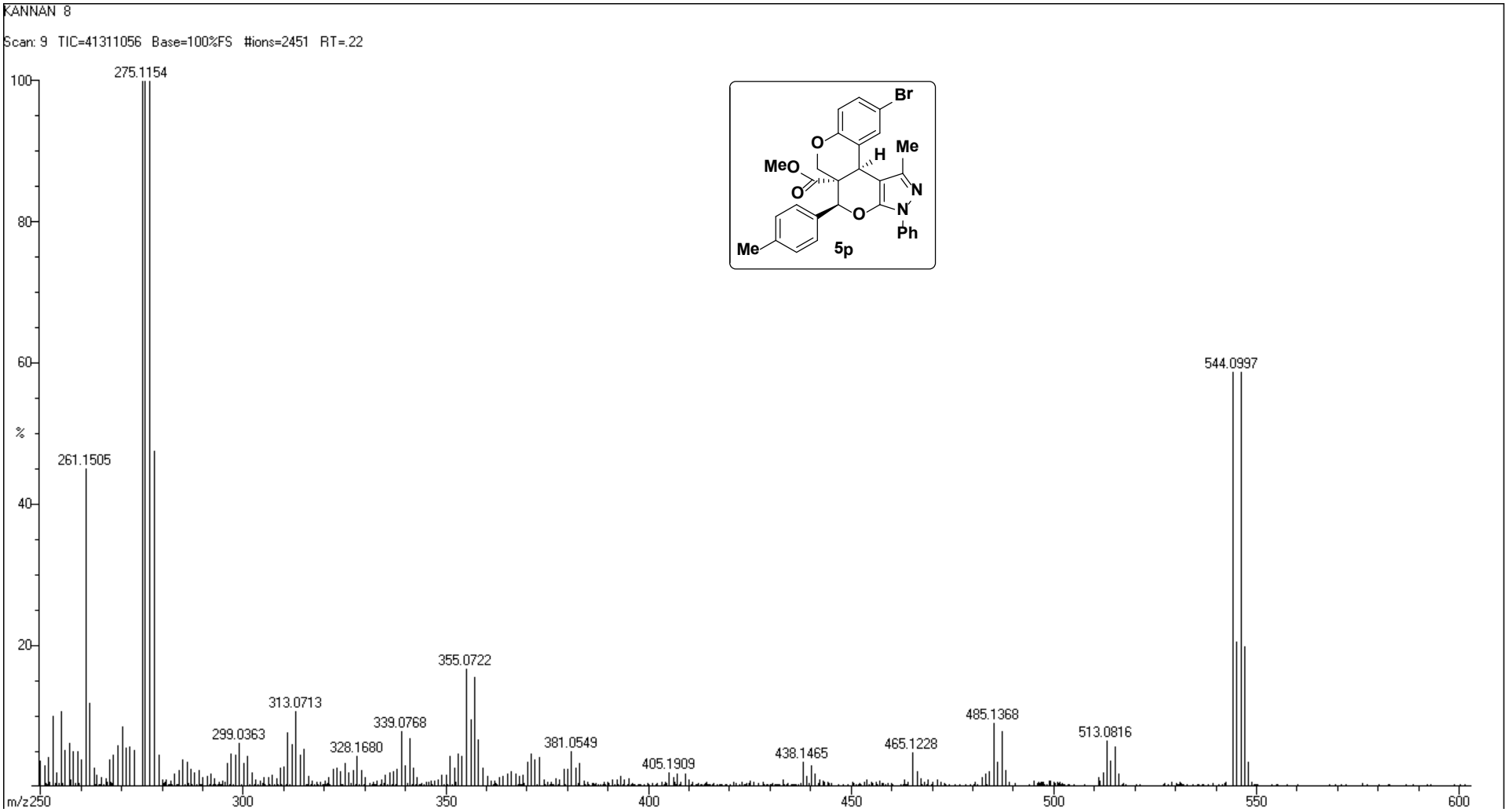
Current Data Parameters
NAME PRSK-III-24
EXPNO 1
PROCNO 1

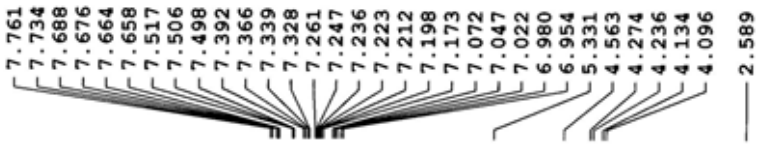
F2 - Acquisition Parameters
Date_ 20130420
Time 19.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 590
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

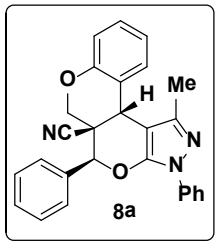
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

======== 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

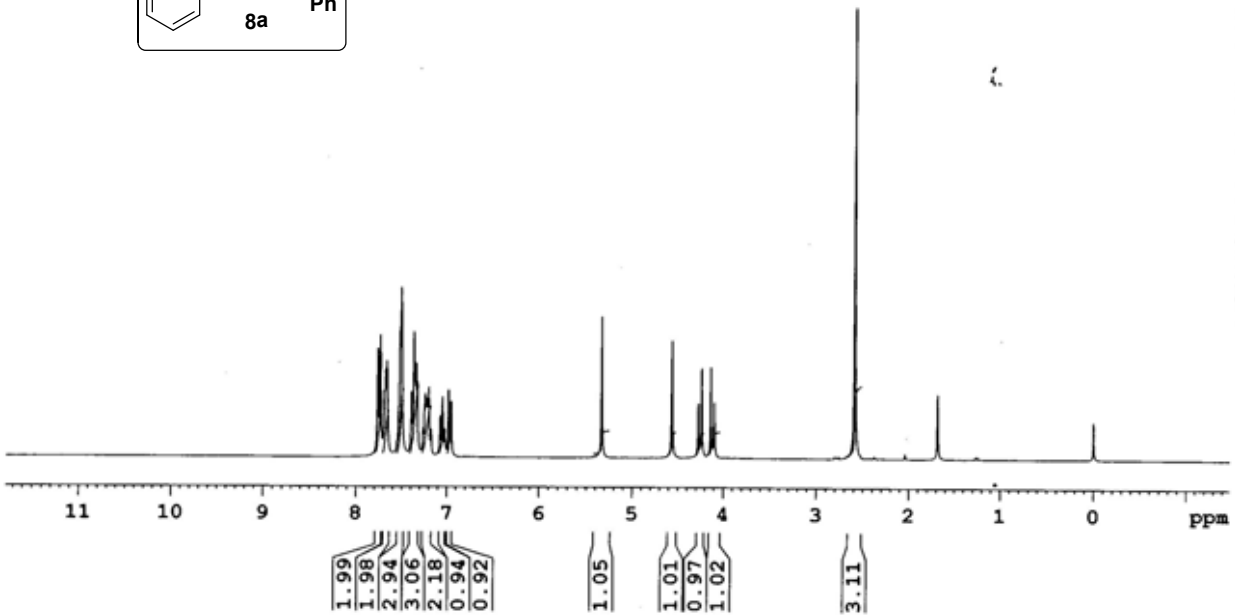
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







8a

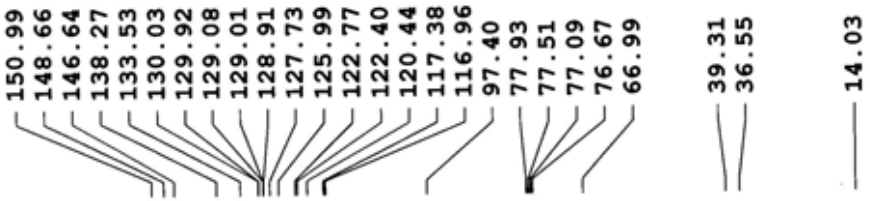


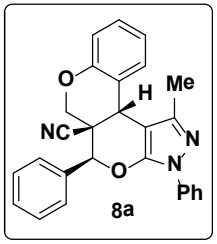

```
Current Data Parameters
NAME                  PRSK-H-CN
EXPNO                         1
PROCNO                        1

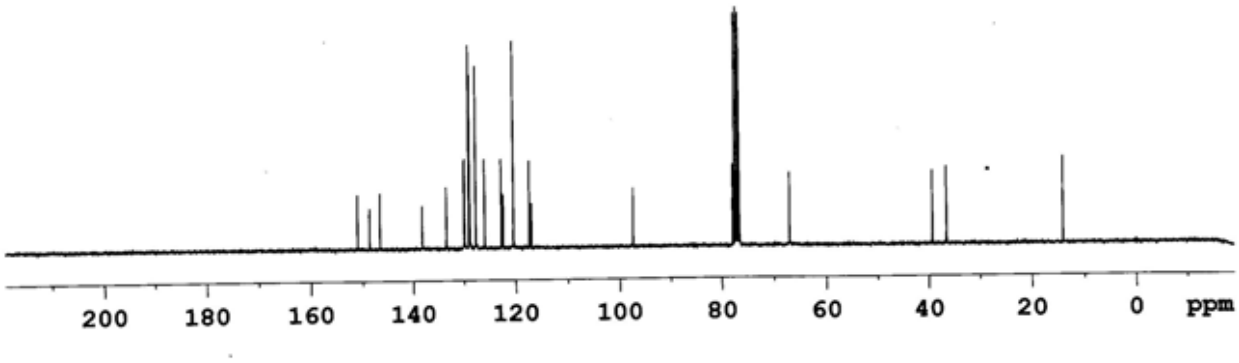
F2 - Acquisition Parameters
Date_                  20130402
Time                      14.36
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           11
DS                            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

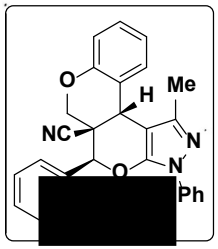
======== CHANNEL f1 ========
NUC1                         1H
P1                        12.40 usec
PL1                        0.00 dB
SFO1                 300.1318534 MHz

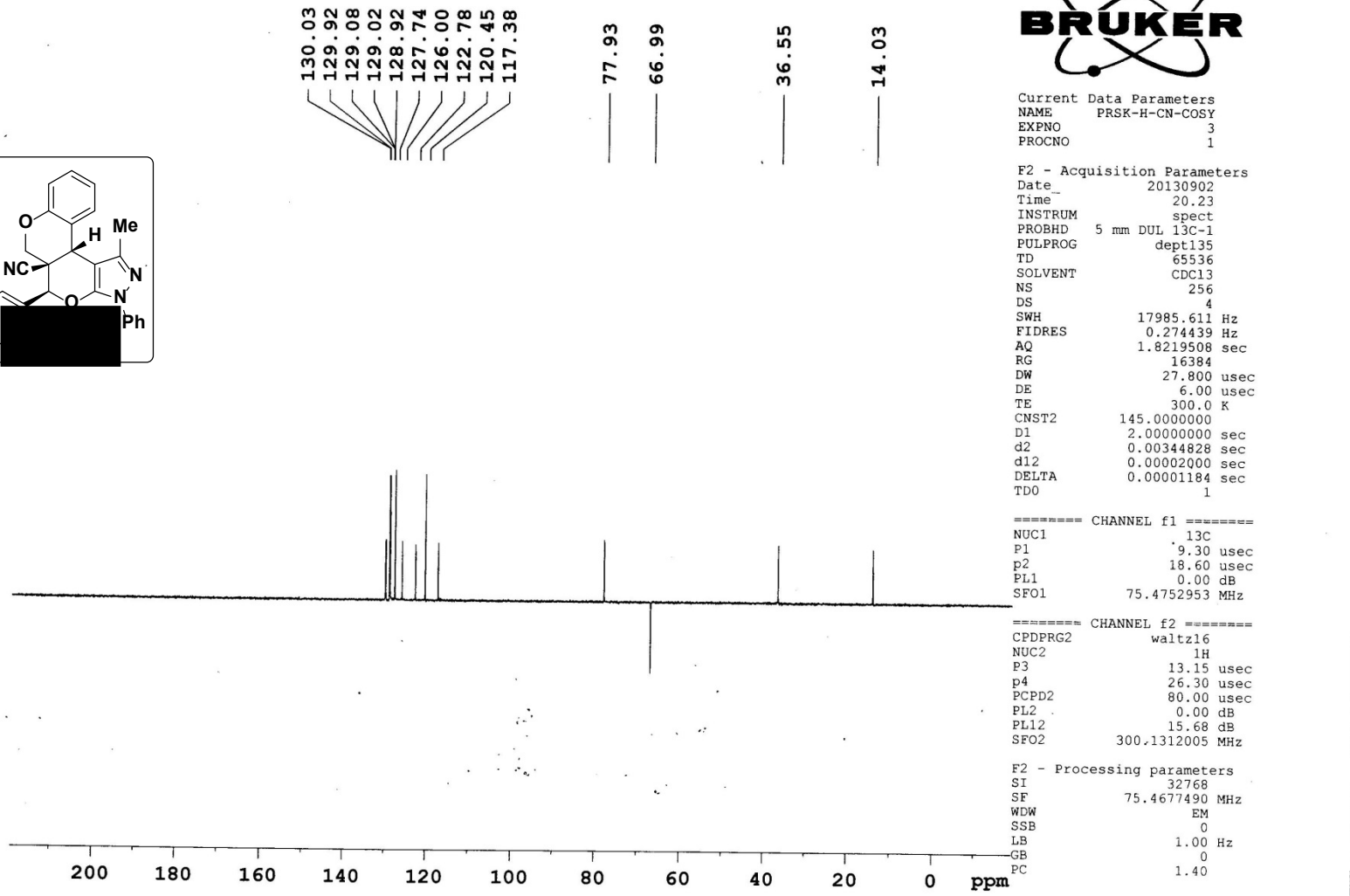
F2 - Processing parameters
SI                        32768
SF                   300.1300098 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```











Current Data Parameters
NAME PRSK-H-CN-COSY
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_	20130902
Time	20.23
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	dept135
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	16384
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
D1	2.00000000 sec
d2	0.00344828 sec
d12	0.00002000 sec
DELTA	0.00001184 sec
TD0	1

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

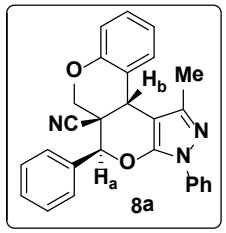
NUC1	13C
P1	9.30 usec
p2	18.60 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

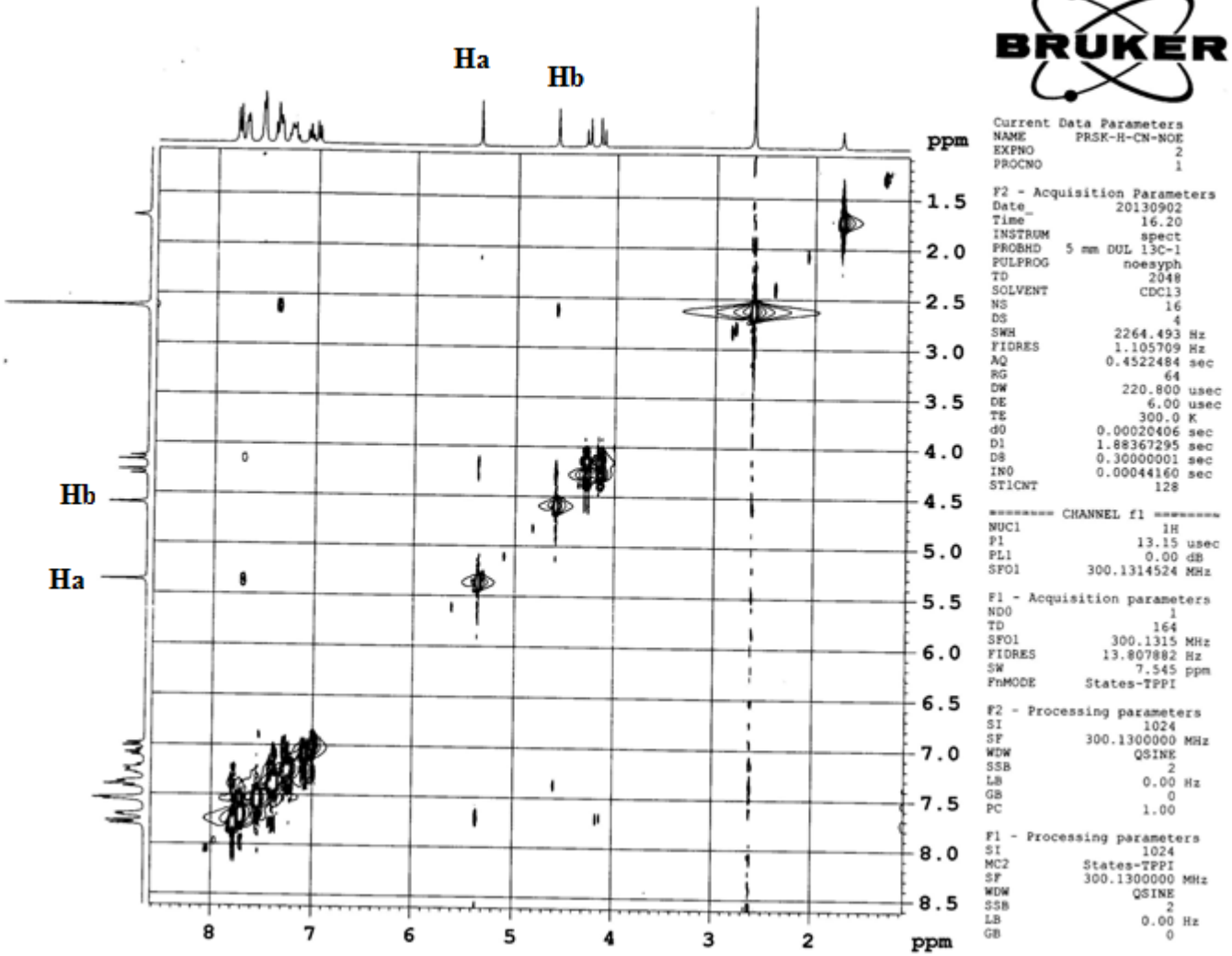
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
P3	13.15 usec
p4	26.30 usec
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 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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```
Current Data Parameters
NAME          PRSK-H-CN-NOE
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130902
Time                  16.20
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG           noesyph
TD                   2048
SOLVENT              CDCl3
NS                      16
DS                       4
SWH               2264.493 Hz
FIDRES            1.105709 Hz
AQ                0.4522484 sec
RG                      64
DW                 220.800 usec
DE                   6.00 usec
TE                  300.0 K
d0                0.00020406 sec
D1                1.88367295 sec
D8                0.30000001 sec
IN0               0.00044160 sec
ST1CNT                 128

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.15 usec
PL1                  0.00 dB
SFO1          300.1314524 MHz

F1 - Acquisition parameters
ND0                      1
TD                     164
SFO1          300.1315 MHz
FIDRES        13.807882 Hz
SW                 7.545 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                    1024
SF            300.1300000 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
PC                    1.00

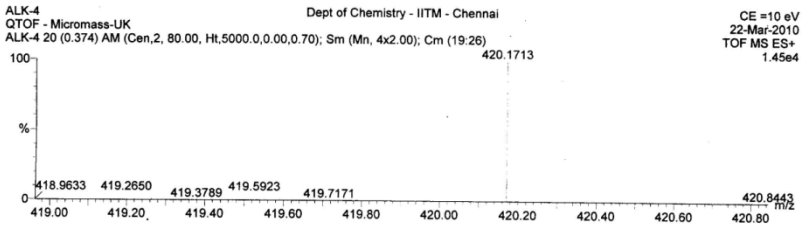
F1 - Processing parameters
SI                    1024
MC2           States-TPPI
SF            300.1300000 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
```

NOESY spectrum of compound **8a**

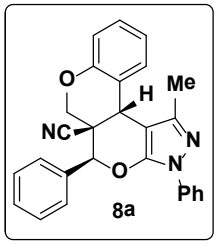
Elemental Composition Report

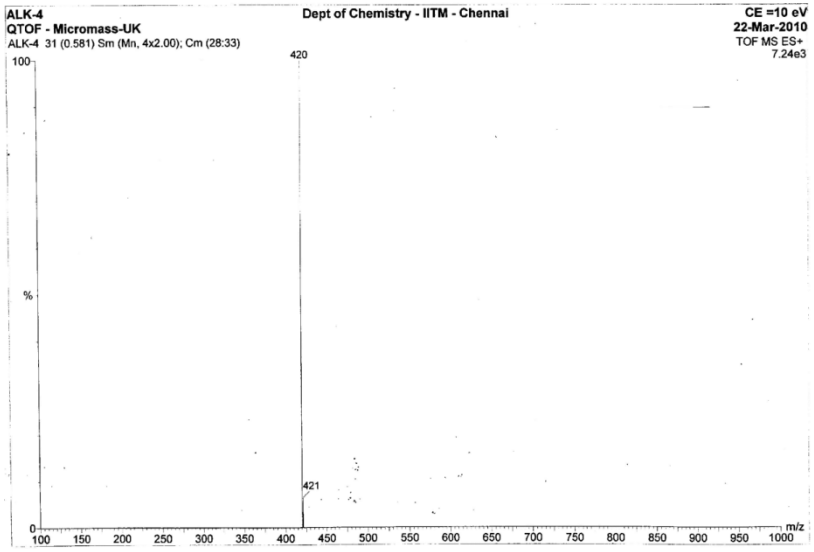
Single Mass Analysis
Tolerance = 200.0 mDa / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

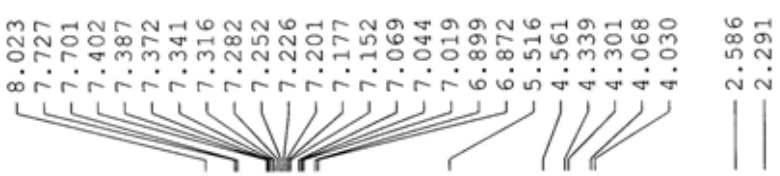
Monoisotopic Mass, Odd and Even Electron Ions
4 formula(e) evaluated with 1 results within limits (all results (up to.1000) for each mass)

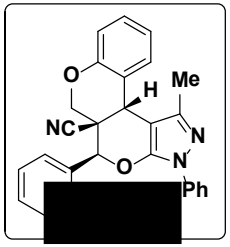


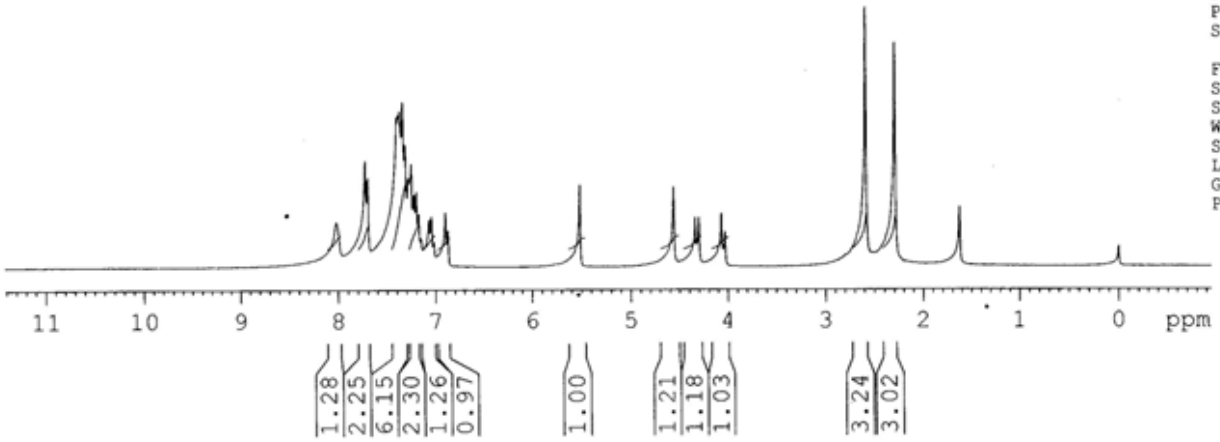
Minimum:				-1.5		
Maximum:		200.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
420.1713	420.1712	0.1	0.3	18.5	1	C27 H22 N3 O2









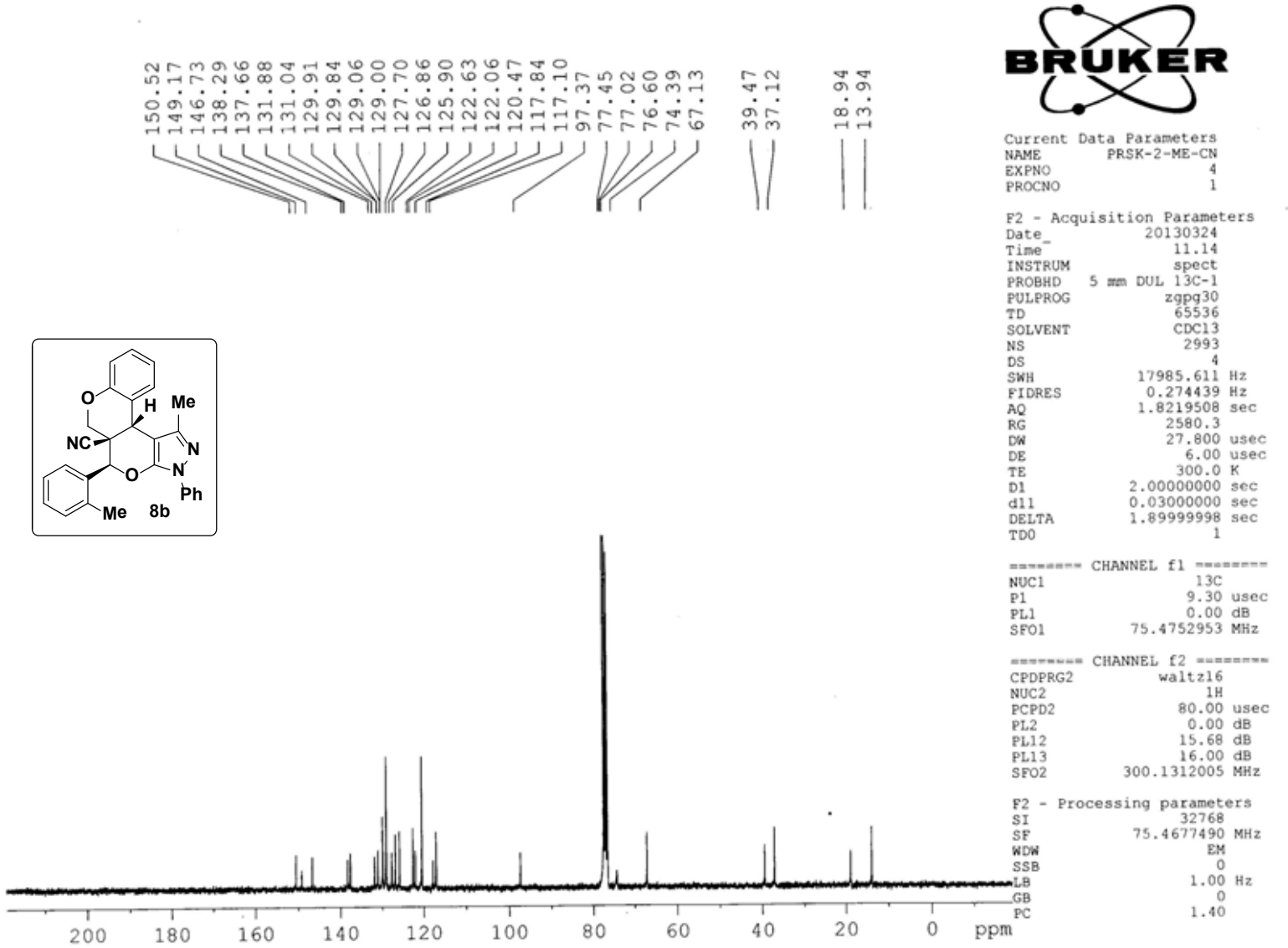


Current Data Parameters
NAME PRSK-2-ME-CN
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130324
Time 7.59
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 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 300.1300084 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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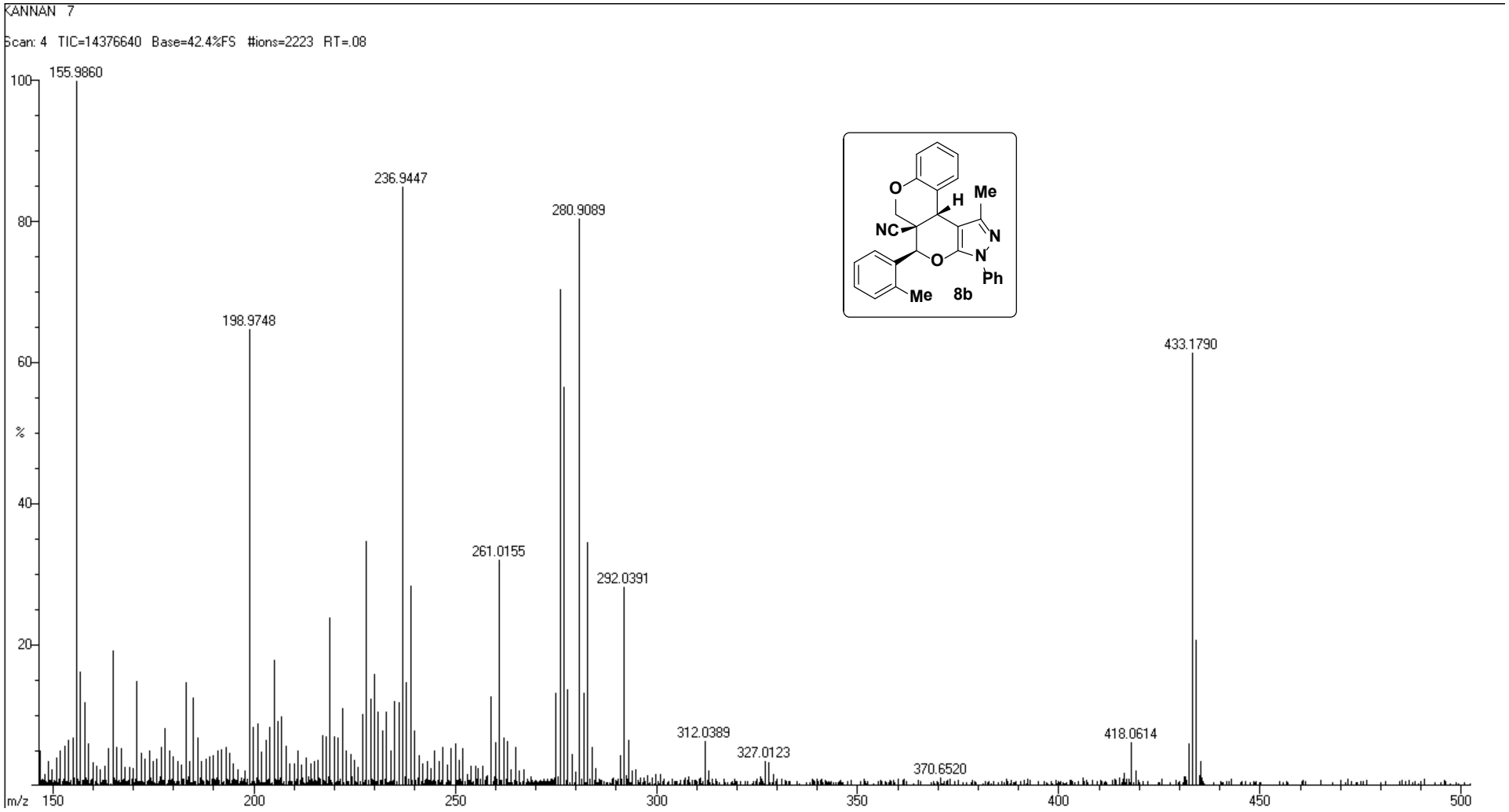
Current Data Parameters
NAME PRSK-2-ME-CN
EXPNO 4
PROCNO 1

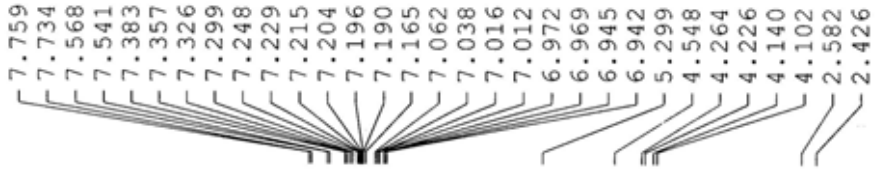
F2 - Acquisition Parameters
Date_ 20130324
Time 11.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2993
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2580.3
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

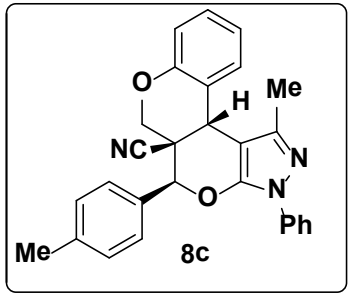
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

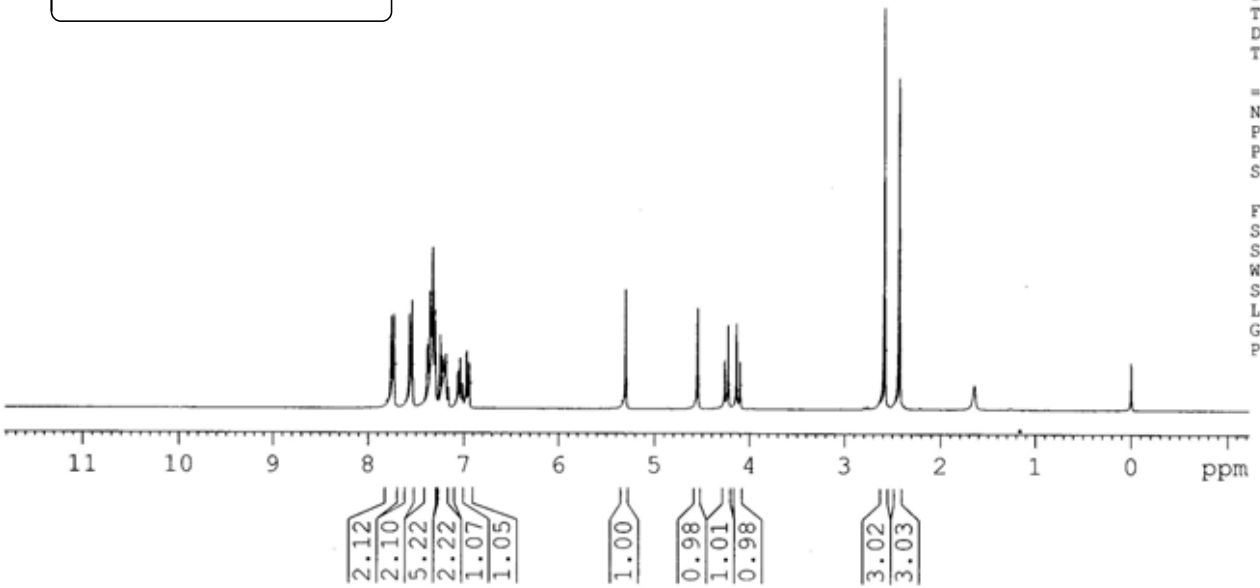
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

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







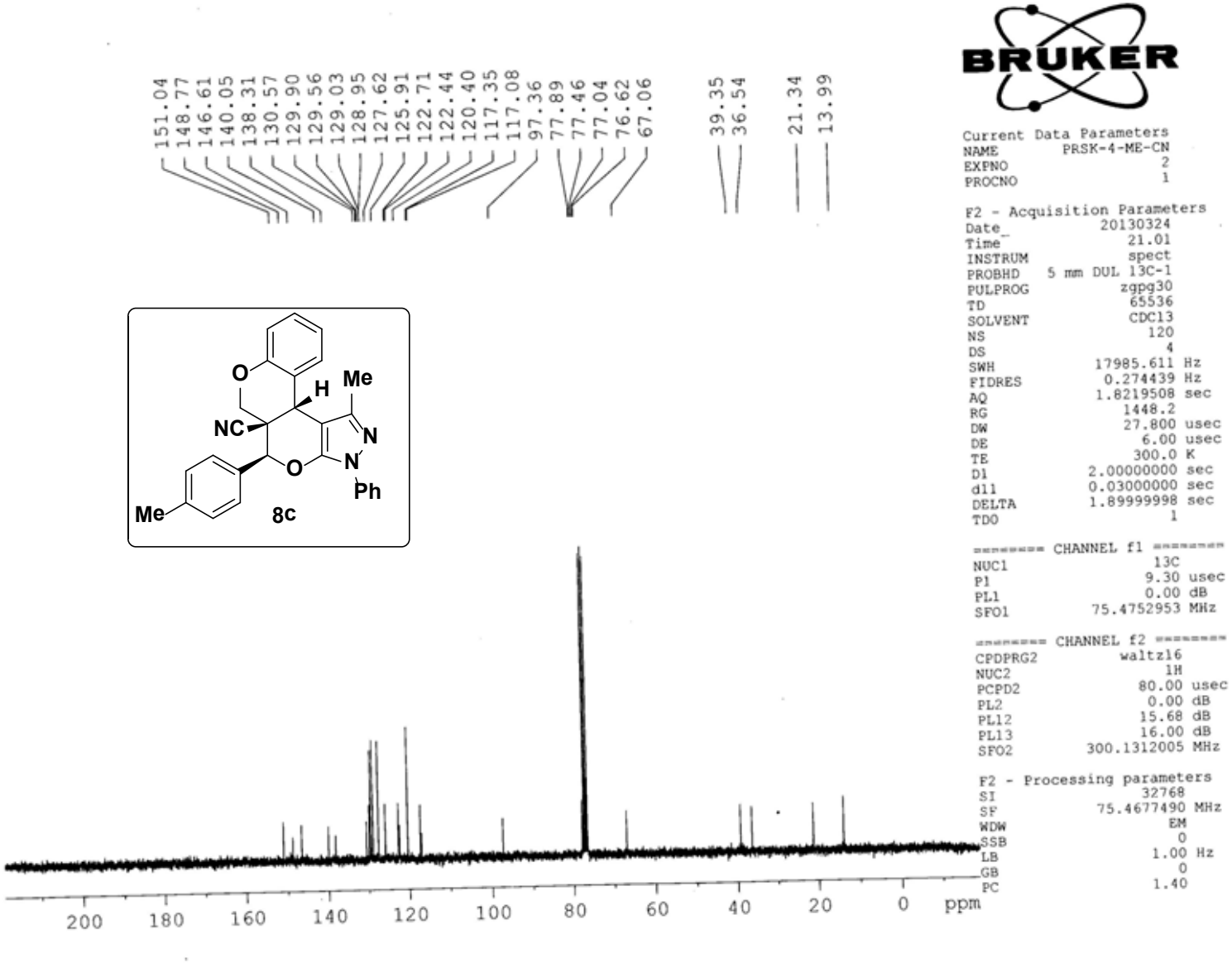



```
Current Data Parameters
NAME          PRSK-4-ME-CN
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130324
Time                 20.59
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       9
DS                       2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                     114
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            300.1300104 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



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Current Data Parameters
NAME PRSK-4-ME-CN
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130324
Time 21.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 120
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1448.2
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

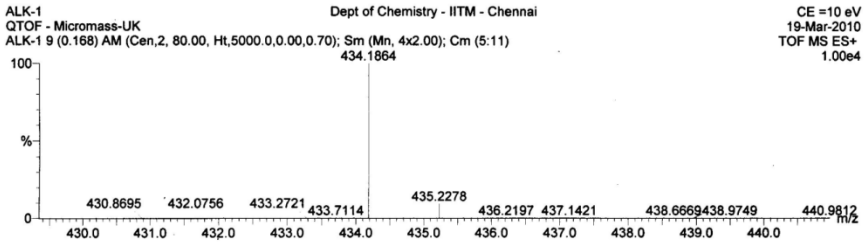
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

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

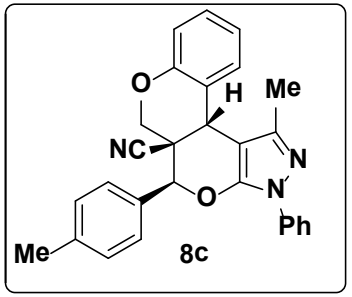
Elemental Composition Report **Page 1**

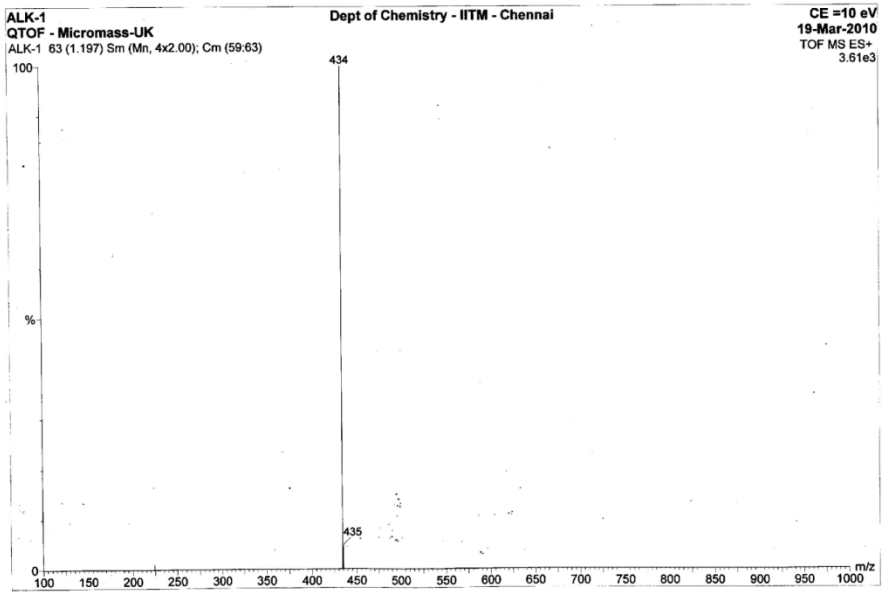
Single Mass Analysis
Tolerance = 200.0 mDa / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

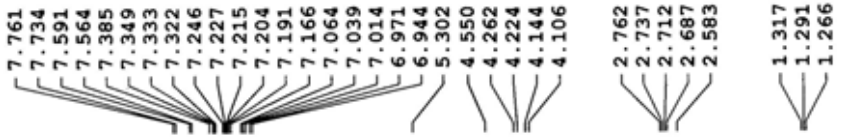
Monoisotopic Mass, Odd and Even Electron Ions
4 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

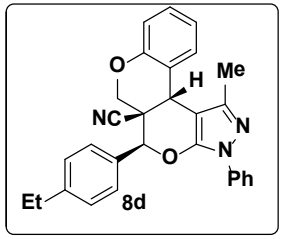


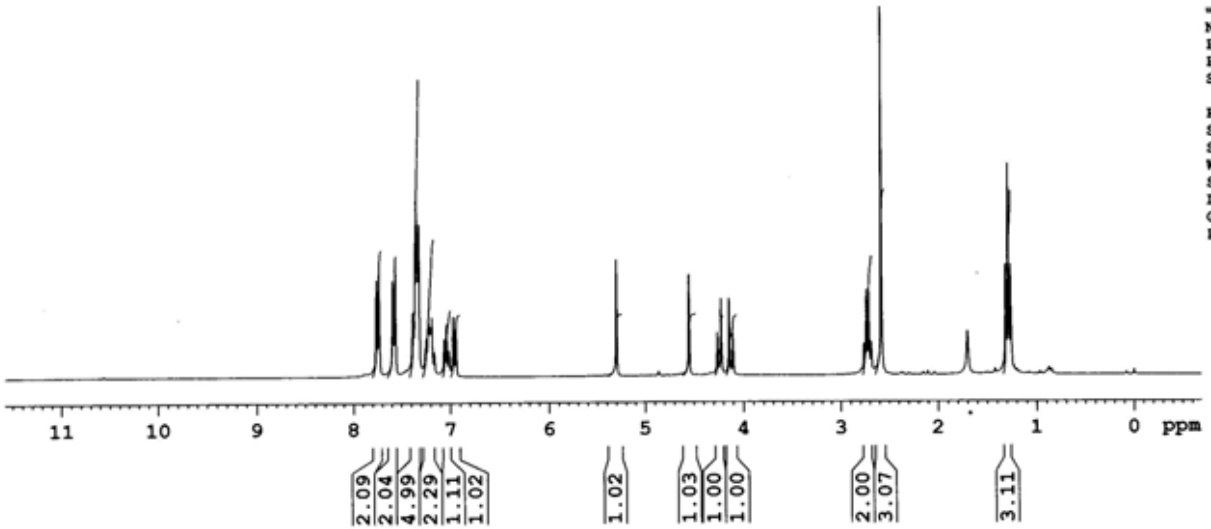
Minimum:				-1.5		
Maximum:		200.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
434.1864	434.1869	-0.5	-1.1	18.5	1	C28 H24 N3 O2









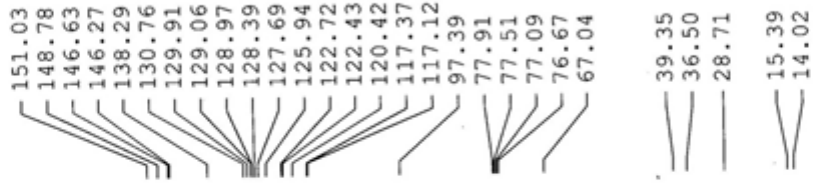


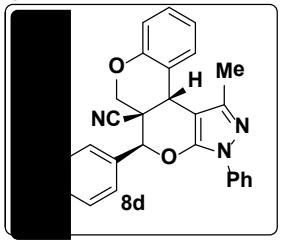

```
Current Data Parameters
NAME                  PRSK-4-ET
EXPNO                         1
PROCNO                        1

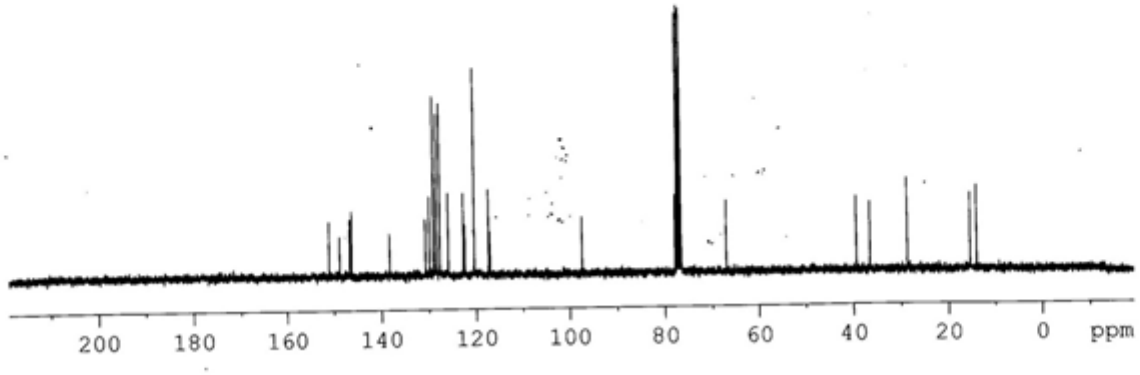
F2 - Acquisition Parameters
Date_                  20130510
Time                      08.12
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                    6172.839 Hz
FIDRES                 0.094190 Hz
AQ                     5.3084660 sec
RG                          90.5
DW                        81.000 usec
DE                         6.00 usec
TE                        300.0 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        12.40 usec
PL1                        0.00 dB
SFO1                 300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF                   300.1300102 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```







BRUKER

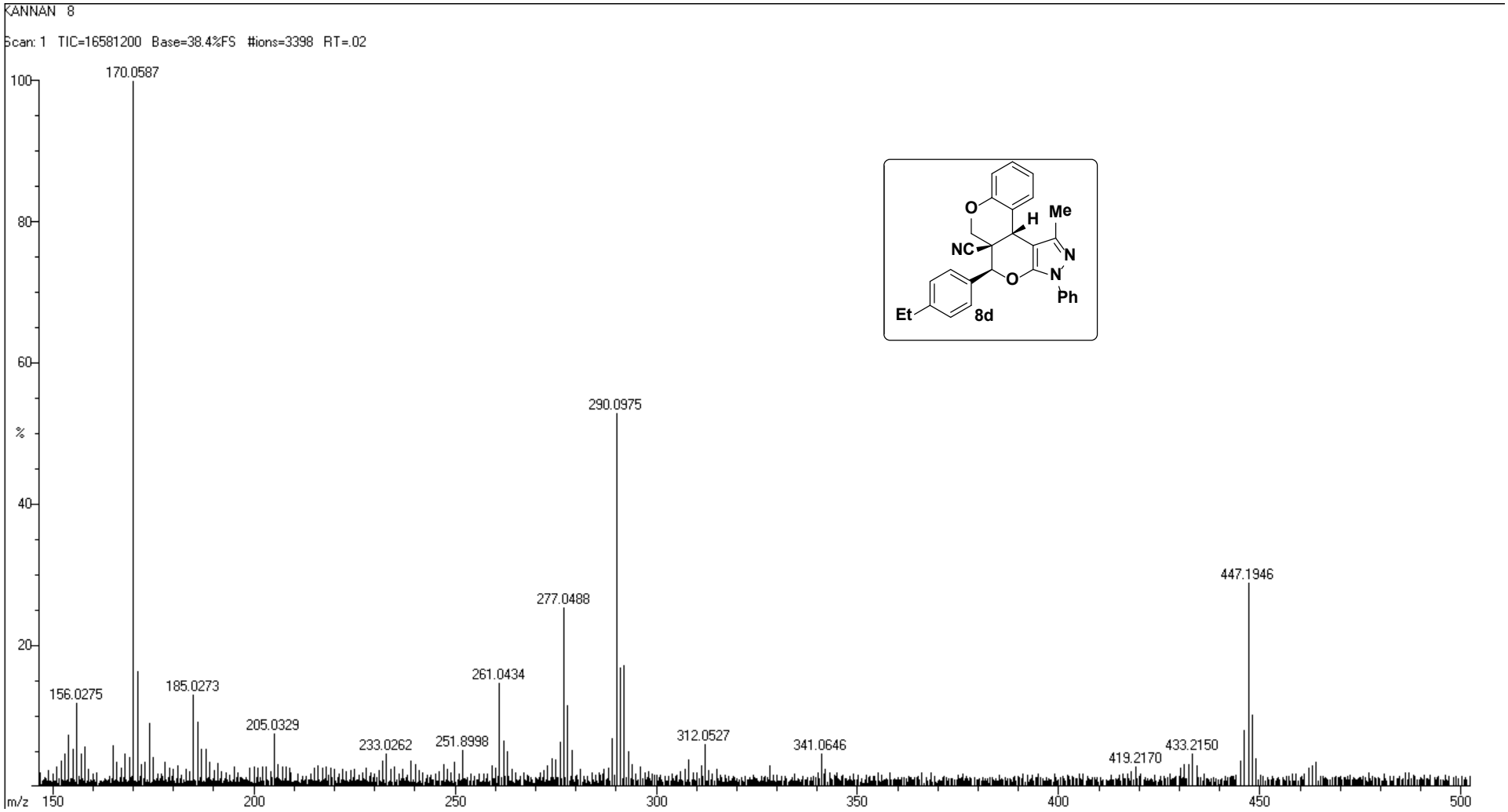
Current Data Parameters
NAME PRSK-4-ET-~~BUT~~
EXPNO 1
PROCNO 1

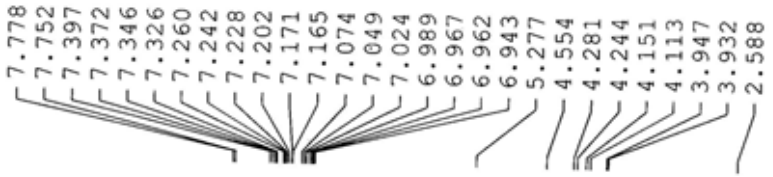
F2 - Acquisition Parameters
Date_ 20130405
Time 19.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 150
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 912.3
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

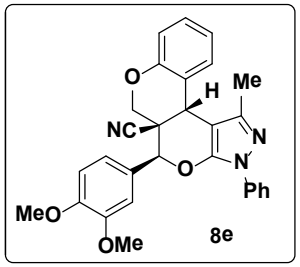
======== CHANNEL f1 ========
NUC1 13C
P1 8.10 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

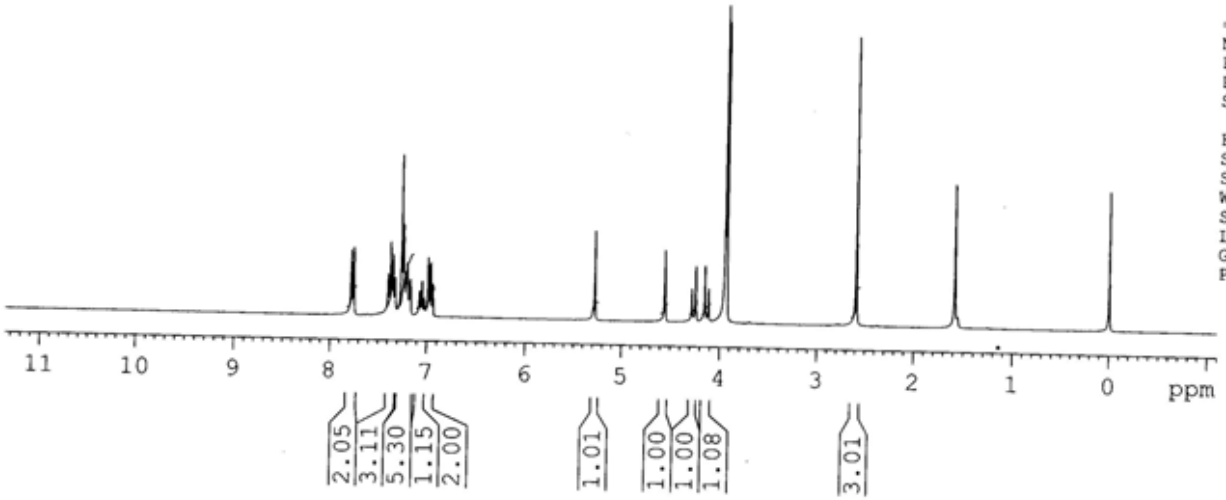
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.19 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

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







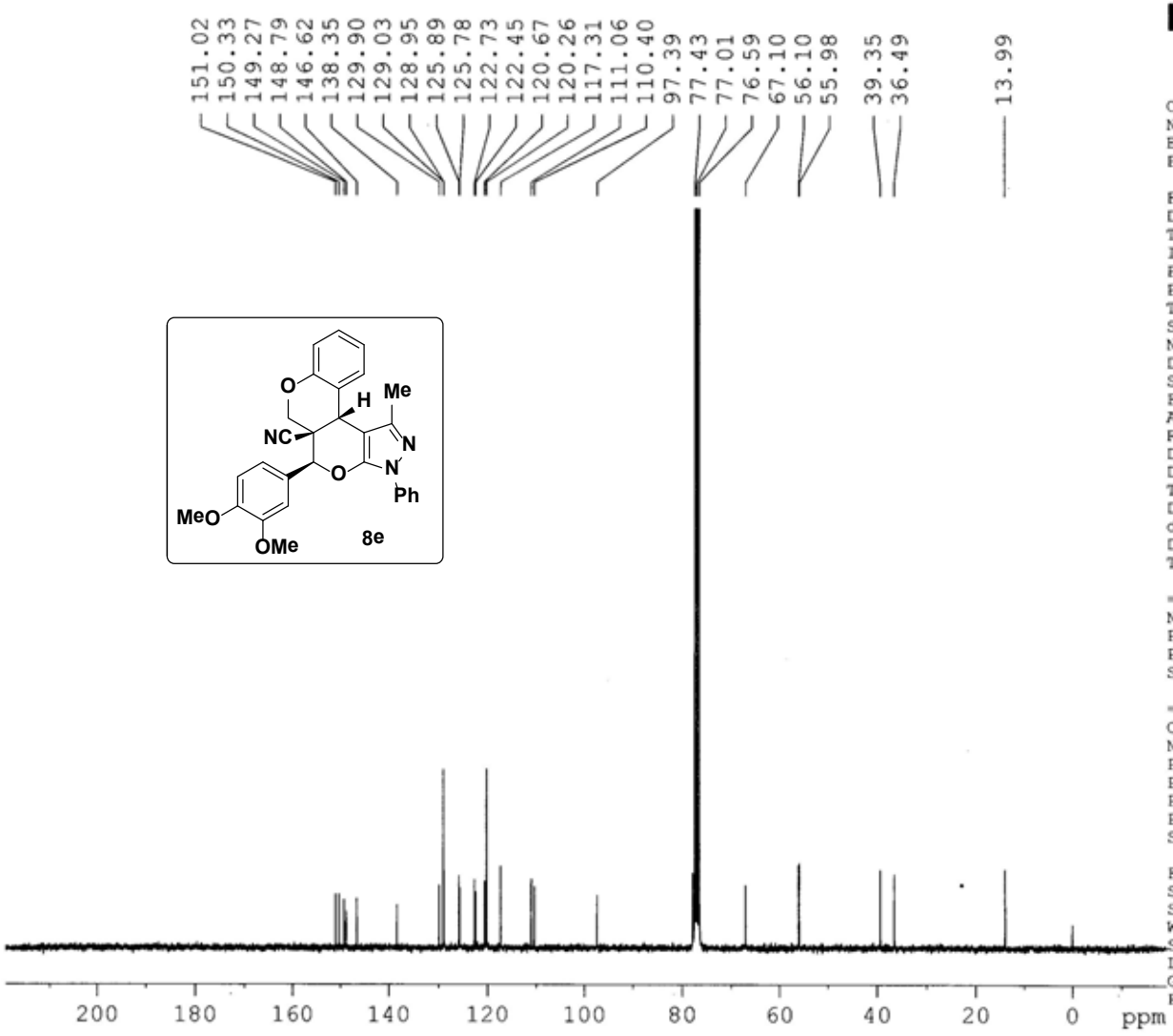



```
Current Data Parameters
NAME      PRSK-3,4-DI-OME-CN
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20130320
Time                   0.32
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                      256
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

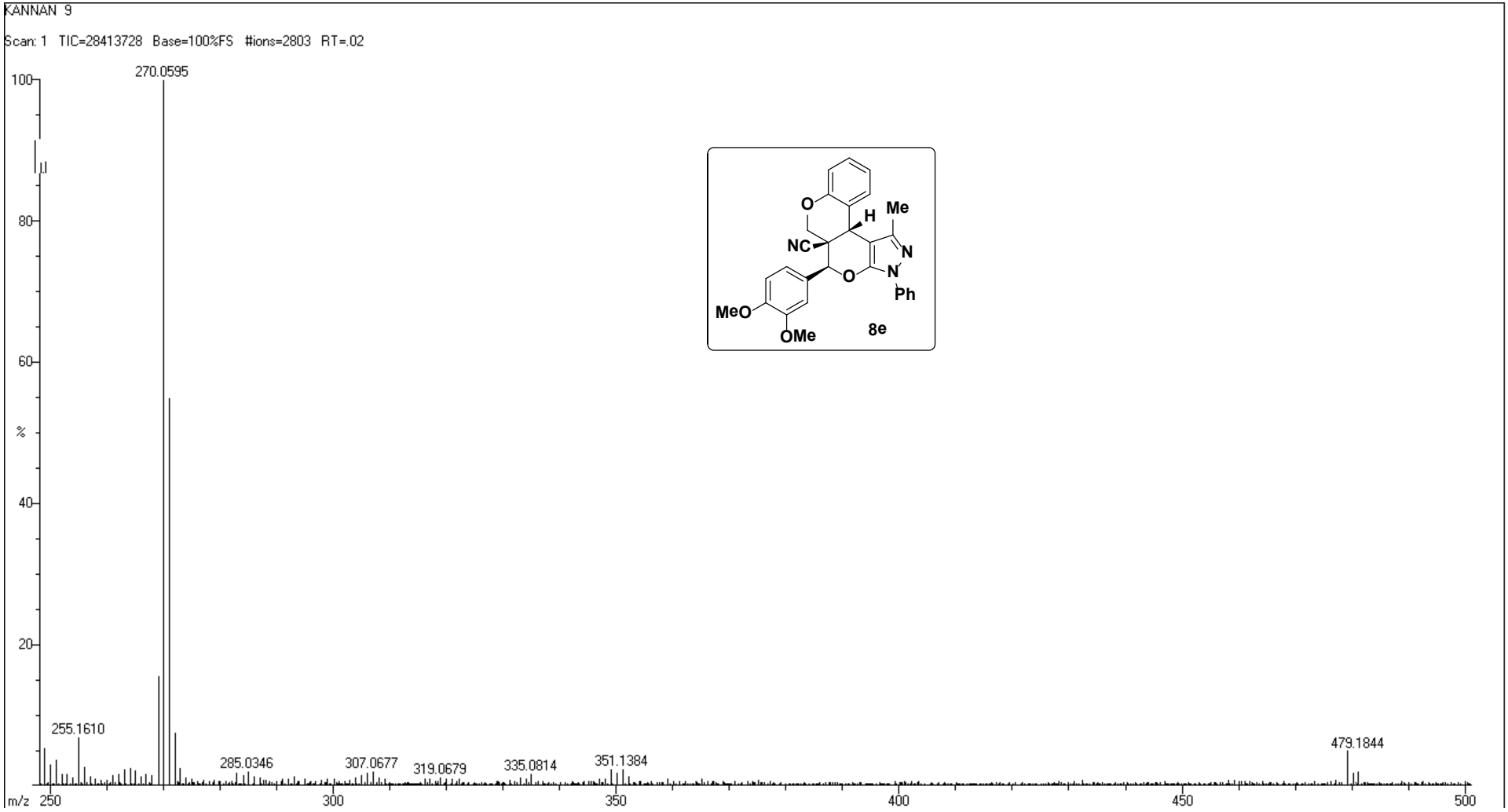
Current Data Parameters
NAME PRSK-3,4-DI-OME-CN
EXPNO 2
PROCNO 1

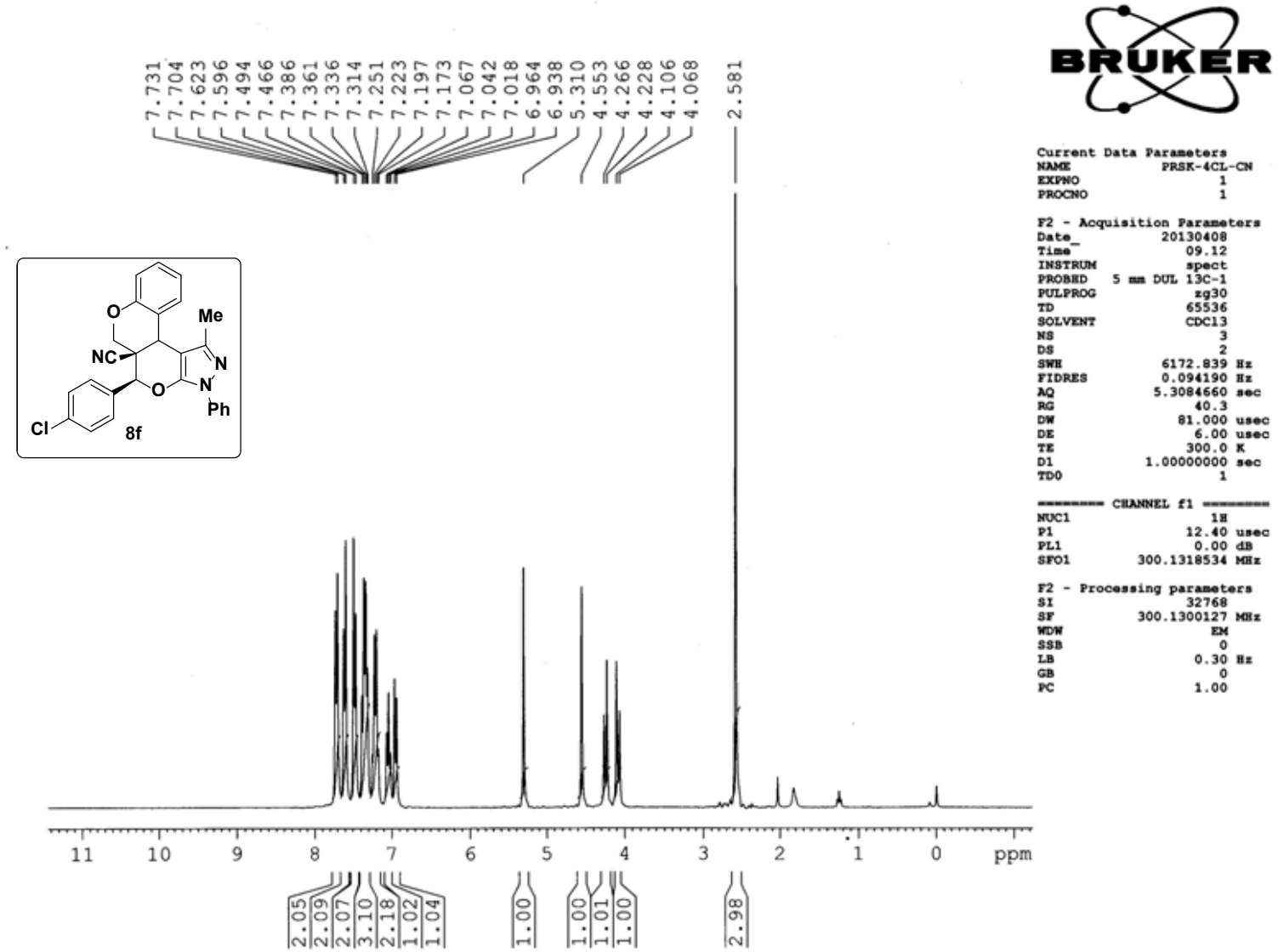
F2 - Acquisition Parameters
Date_ 20130320
Time 6.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 5150
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1448.2
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 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





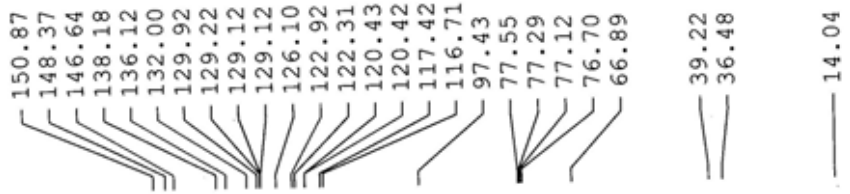
BRUKER

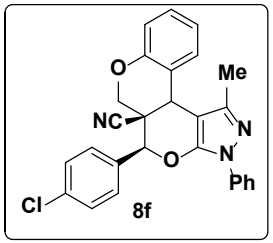
Current Data Parameters
NAME PRSK-4CL-CN
EXPNO 1
PROCNO 1

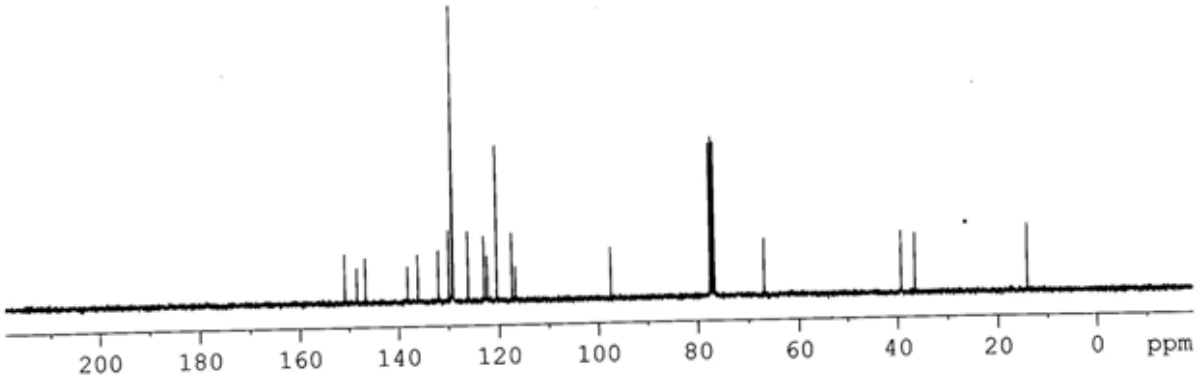
F2 - Acquisition Parameters
Date_ 20130408
Time 09.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 40.3
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.40 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







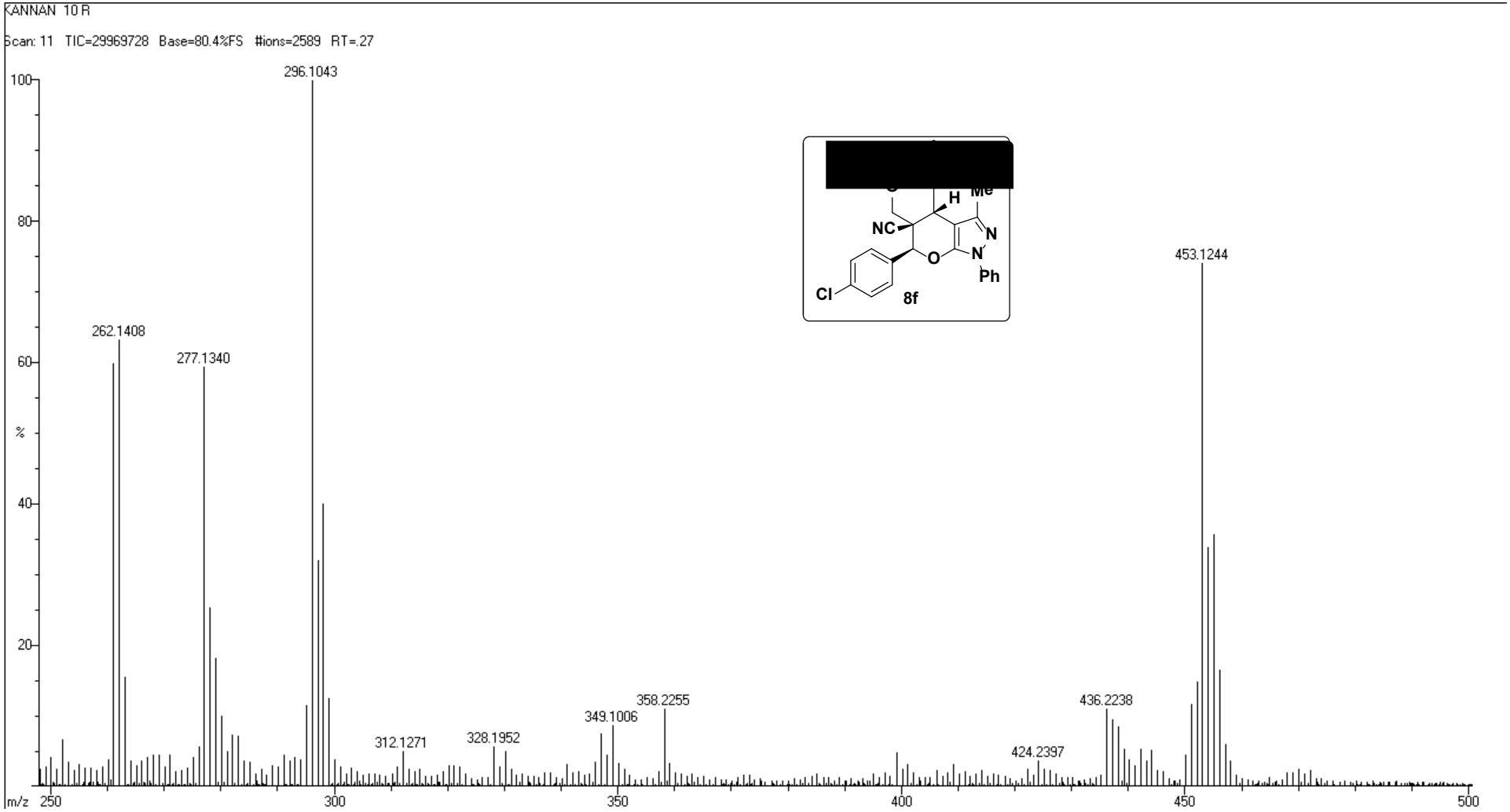

```
Current Data Parameters
NAME                  PRSK-4CL-CN
EXPNO                           2
PROCNO                          1

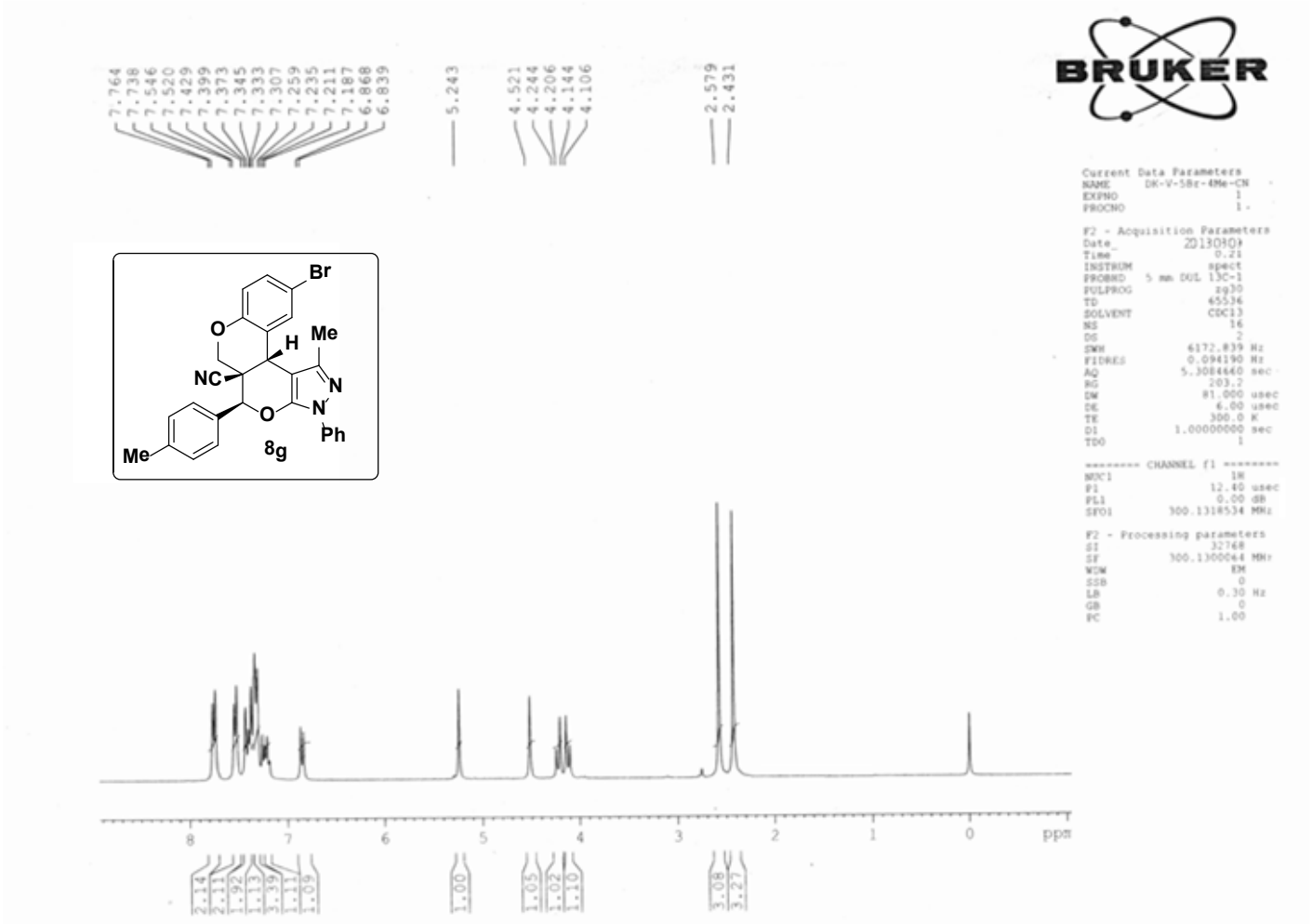
F2 - Acquisition Parameters
Date_                    20130408
Time                        09.06
INSTRUM                     spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            80
DS                             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

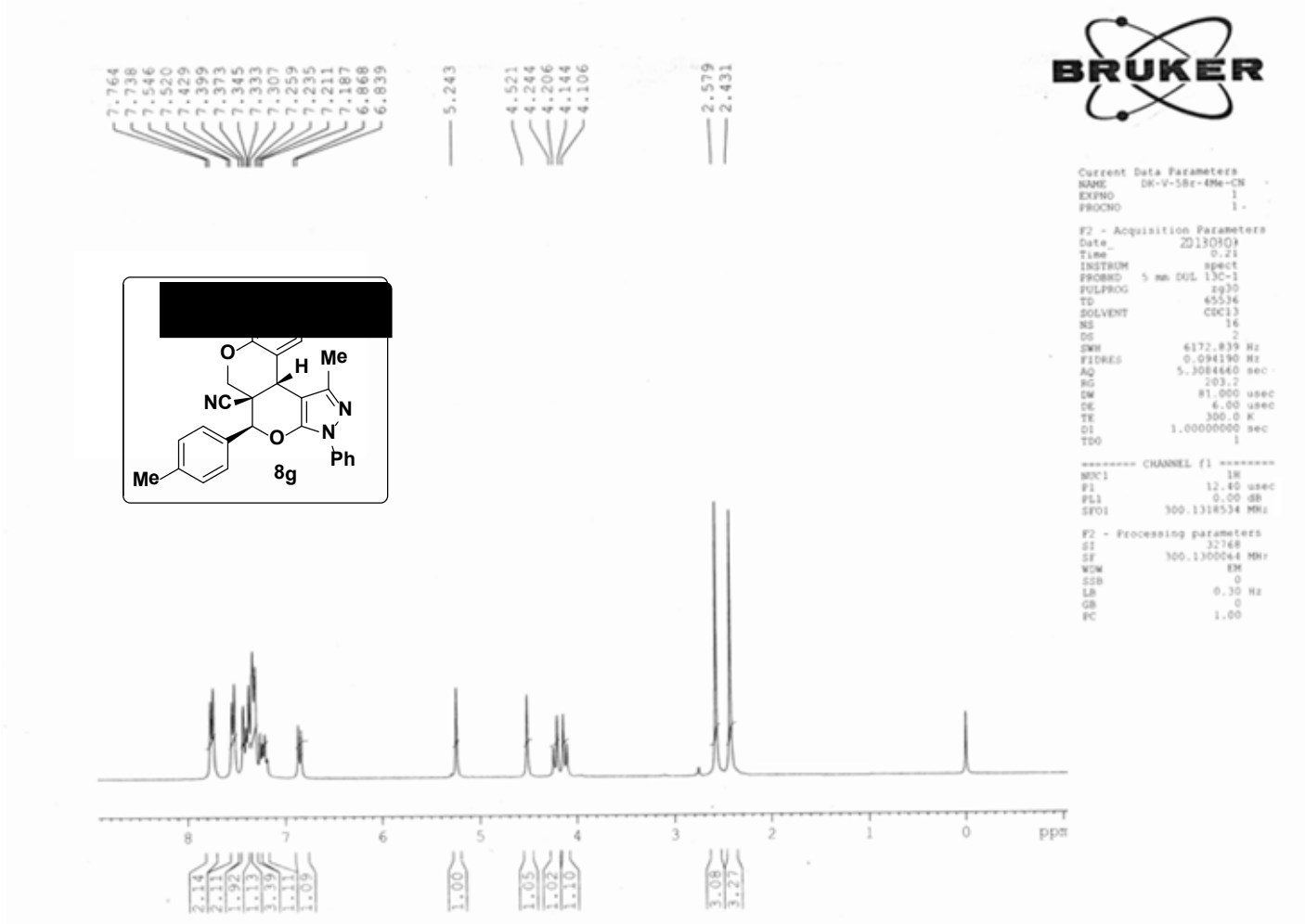
======== CHANNEL f1 ========
NUC1                         13C
P1                         8.10 usec
PL1                       -2.00 dB
SFO1                 75.4752953 MHz

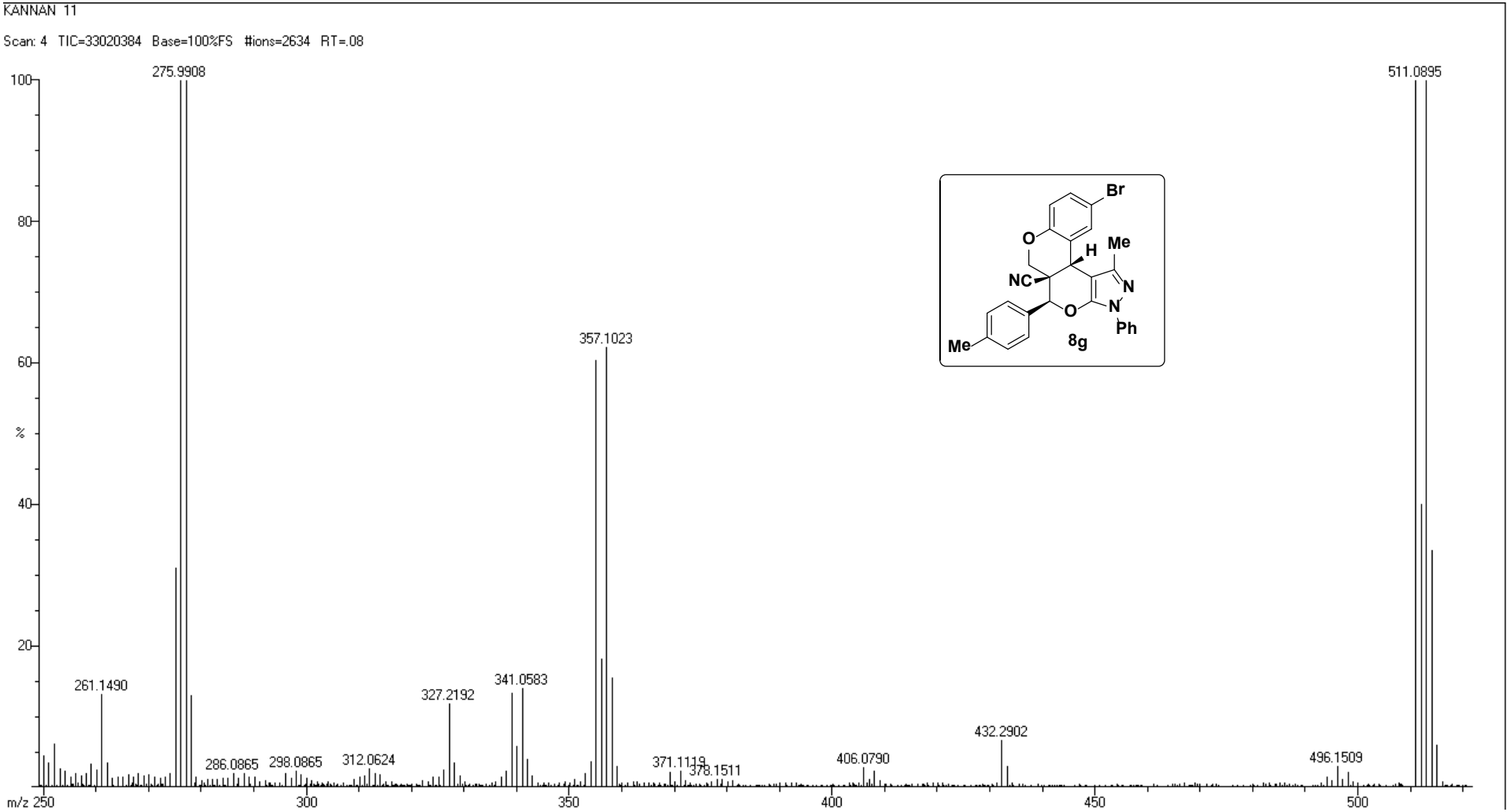
======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                        0.00 dB
PL12                      16.19 dB
PL13                      16.00 dB
SFO2                300.1312005 MHz

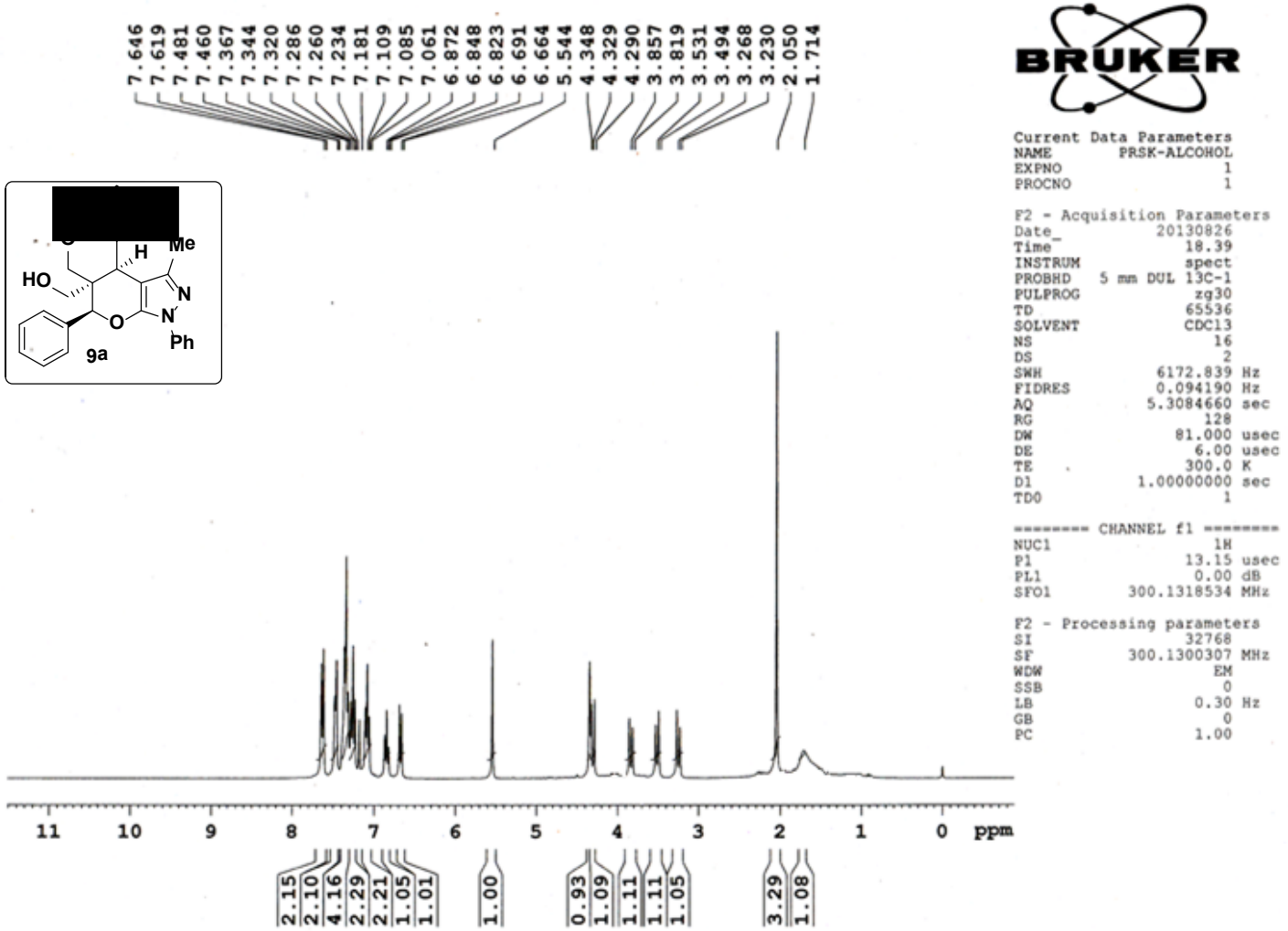
F2 - Processing parameters
SI                           32768
SF                   75.4677490 MHz
WDW                             EM
SSB                              0
LB                        1.00 Hz
GB                               0
PC                            1.40
```






7.764
7.738
7.546
7.520
7.429
7.399
7.373
7.345
7.333
7.307
7.259
7.235
7.211
7.187
6.868
6.839
5.243
4.521
4.244
4.206
4.144
4.106
2.579
2.431
BRUKER
Current Data Parameters
NAME DK-V-5Br-4Me-CN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130103
Time 0.21
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 5.3084440 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 12.40 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300044 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
H
Me
NC
N
N
O
Ph
Me
8g
8
7
6
5
4
3
2
1
0
ppm
2.14
2.11
1.92
1.13
3.39
1.11
1.09
1.00
1.05
1.02
1.10
3.08
3.27


KANNAN 11
Scan: 4 TIC=33020384 Base=100%FS #ions=2634 RT=.08
100
80
60
40
20
%
m/z 250
300
350
400
450
500
275.9908
511.0895
357.1023
261.1490
327.2192
341.0583
286.0865
298.0865
312.0624
371.1119
378.1511
406.0790
432.2902
496.1509
Br
O
H
Me
NC
N
N
O
Ph
Me
8g



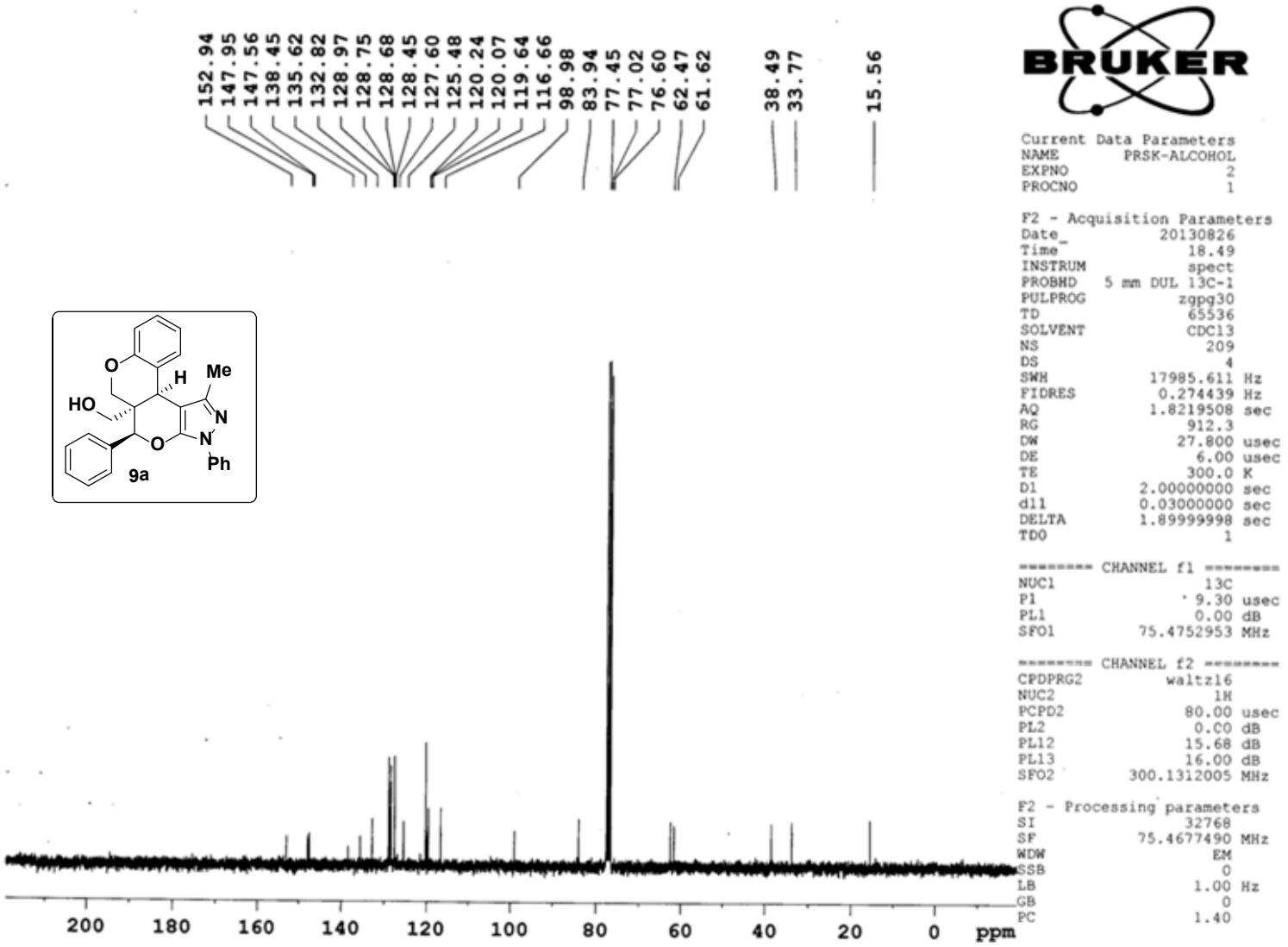
BRUKER

```
Current Data Parameters
NAME        PRSK-ALCOHOL
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130826
Time              18.39
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                 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         300.1300307 MHz
WDW                 EM
SSB                  0
LB              0.30 Hz
GB                   0
PC              1.00
```



BRUKER

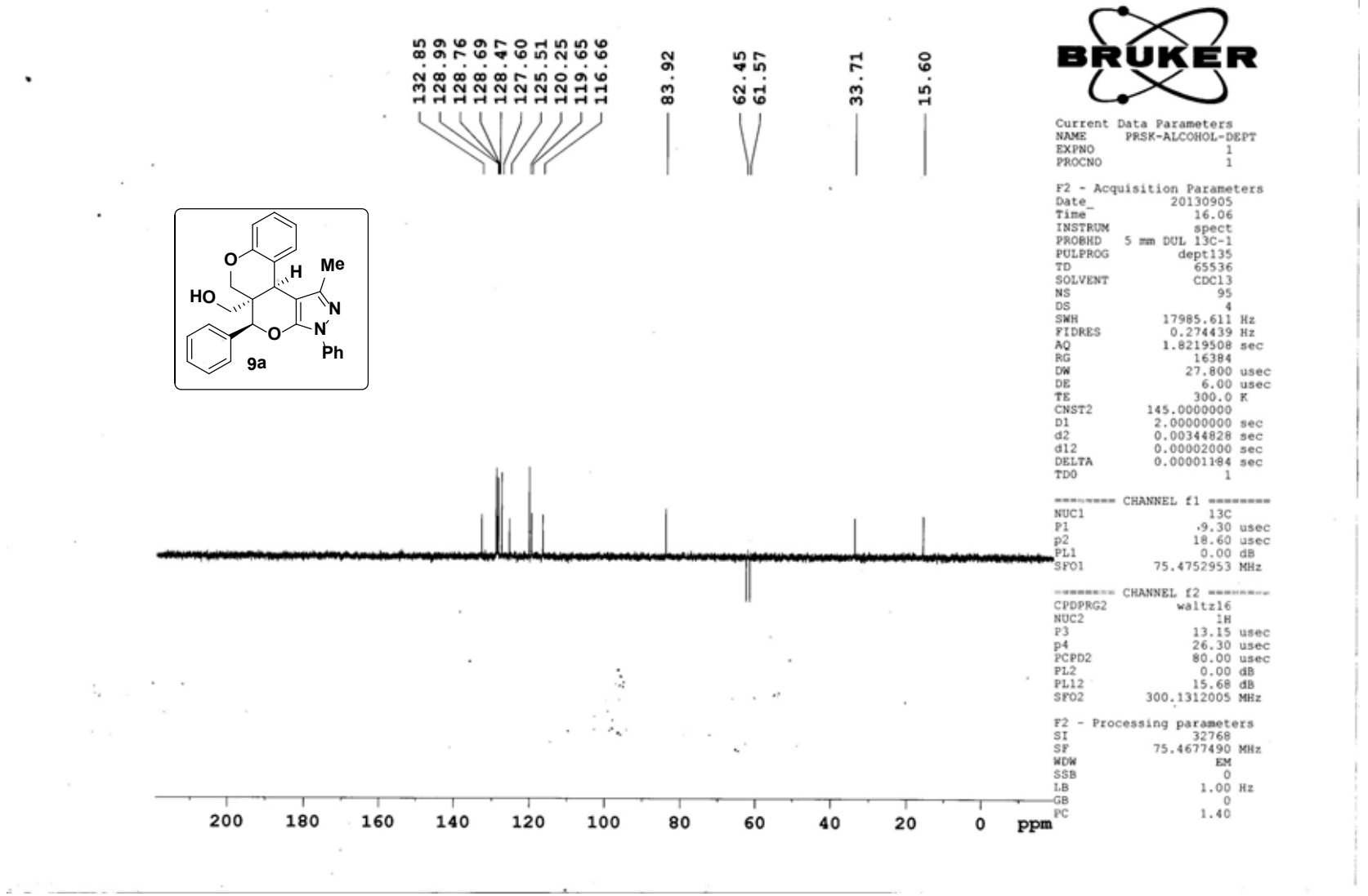
Current Data Parameters
NAME PRSK-ALCOHOL
EXPNO 2
PROCNO 1

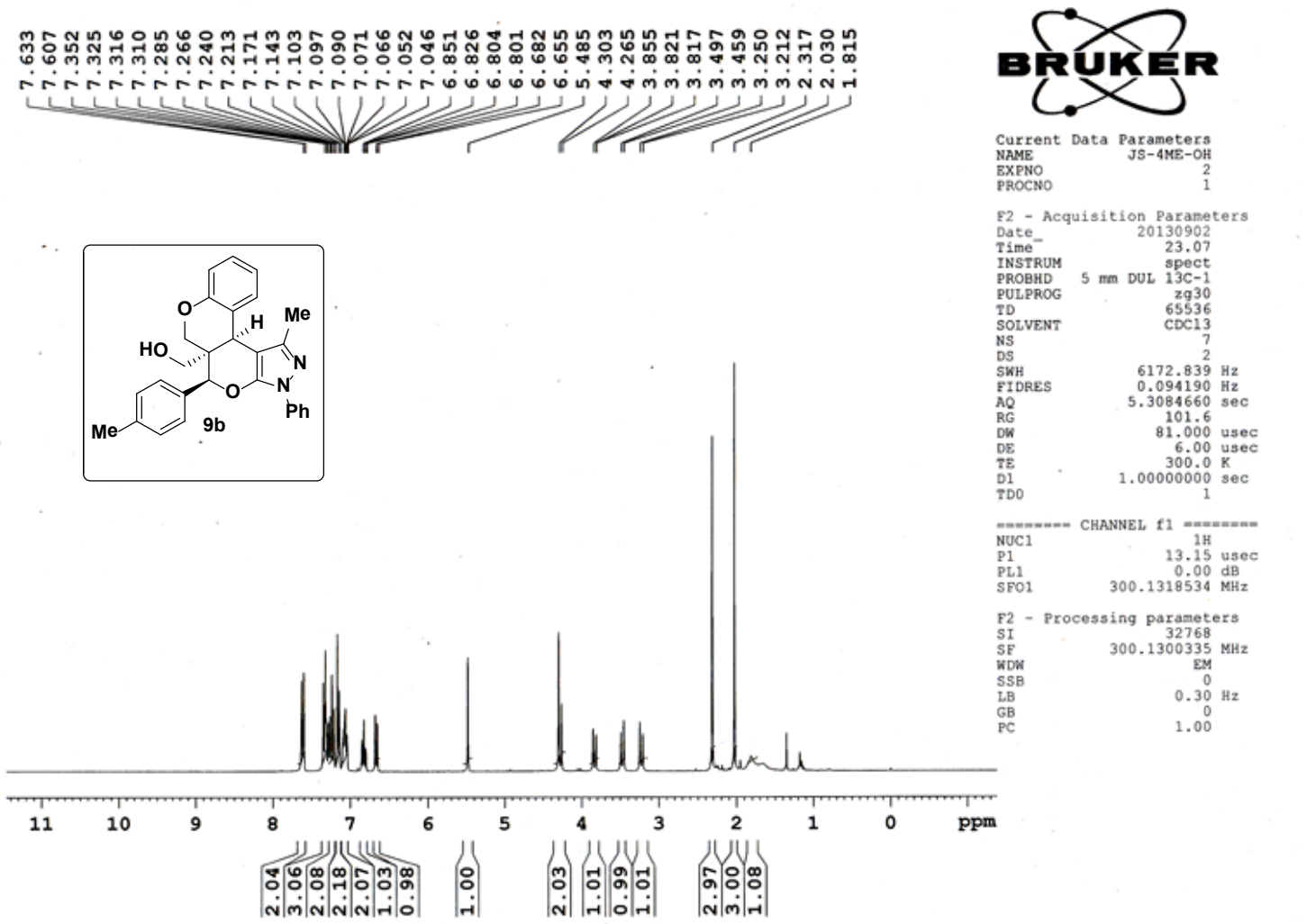
F2 - Acquisition Parameters
Date_ 20130826
Time 18.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 209
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 912.3
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 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





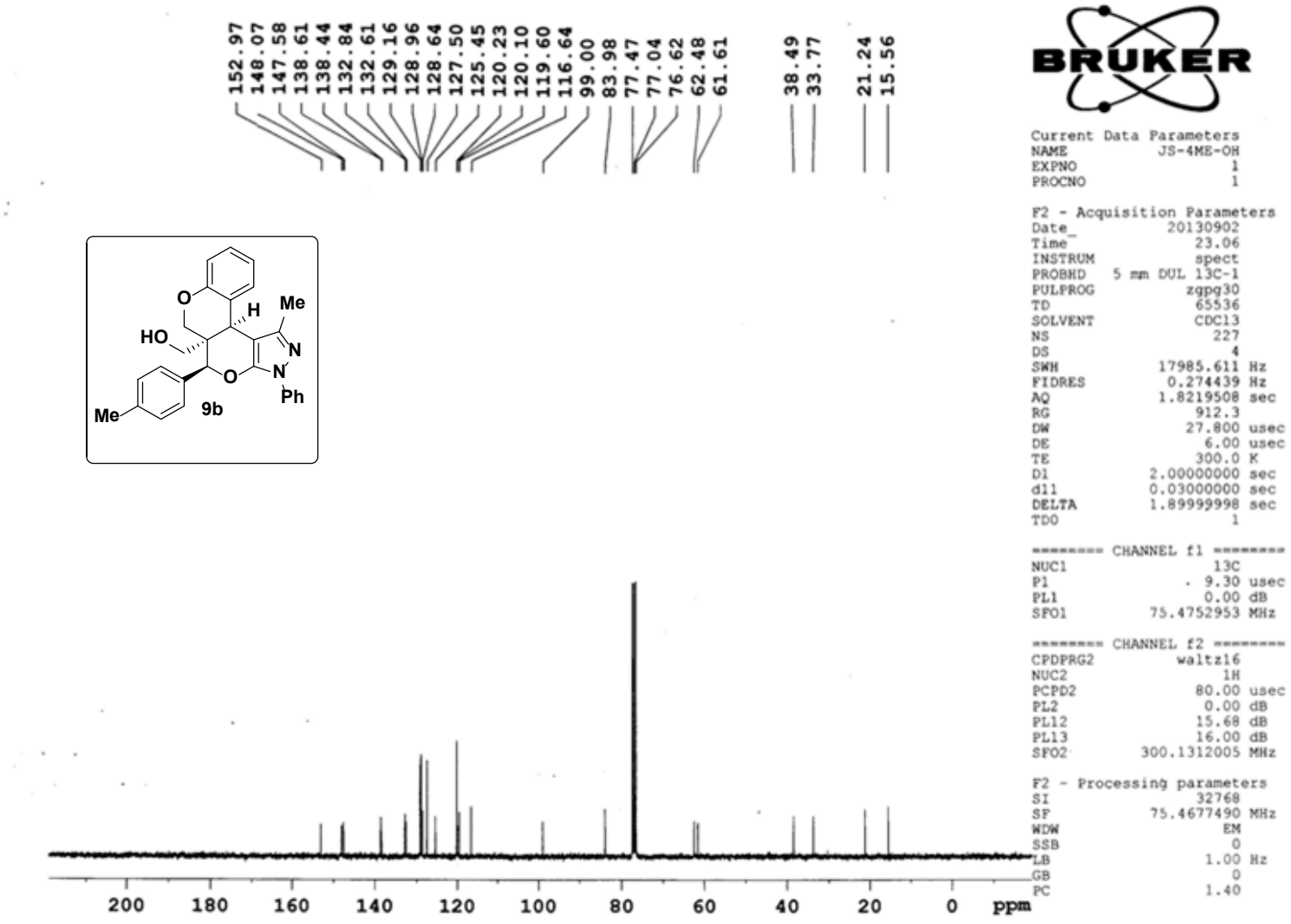
BRUKER

```
Current Data Parameters
NAME                  JS-4ME-OH
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130902
Time                      23.07
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                            7
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                  300.1300335 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



BRUKER

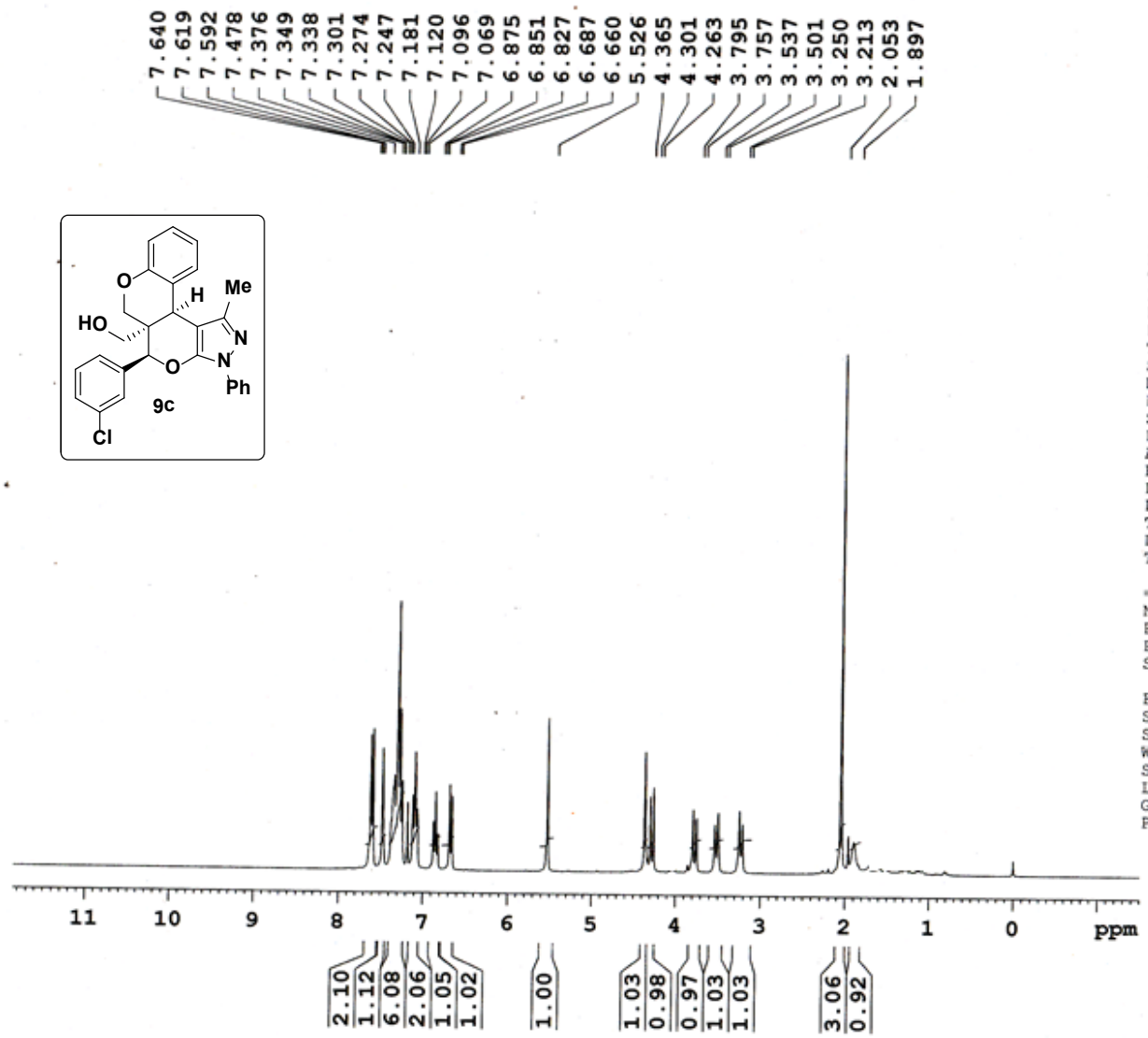
```
Current Data Parameters
NAME                  JS-4ME-OH
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130902
Time                      23.06
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDC13
NS                          227
DS                            4
SWH                 17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                        912.3
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
```



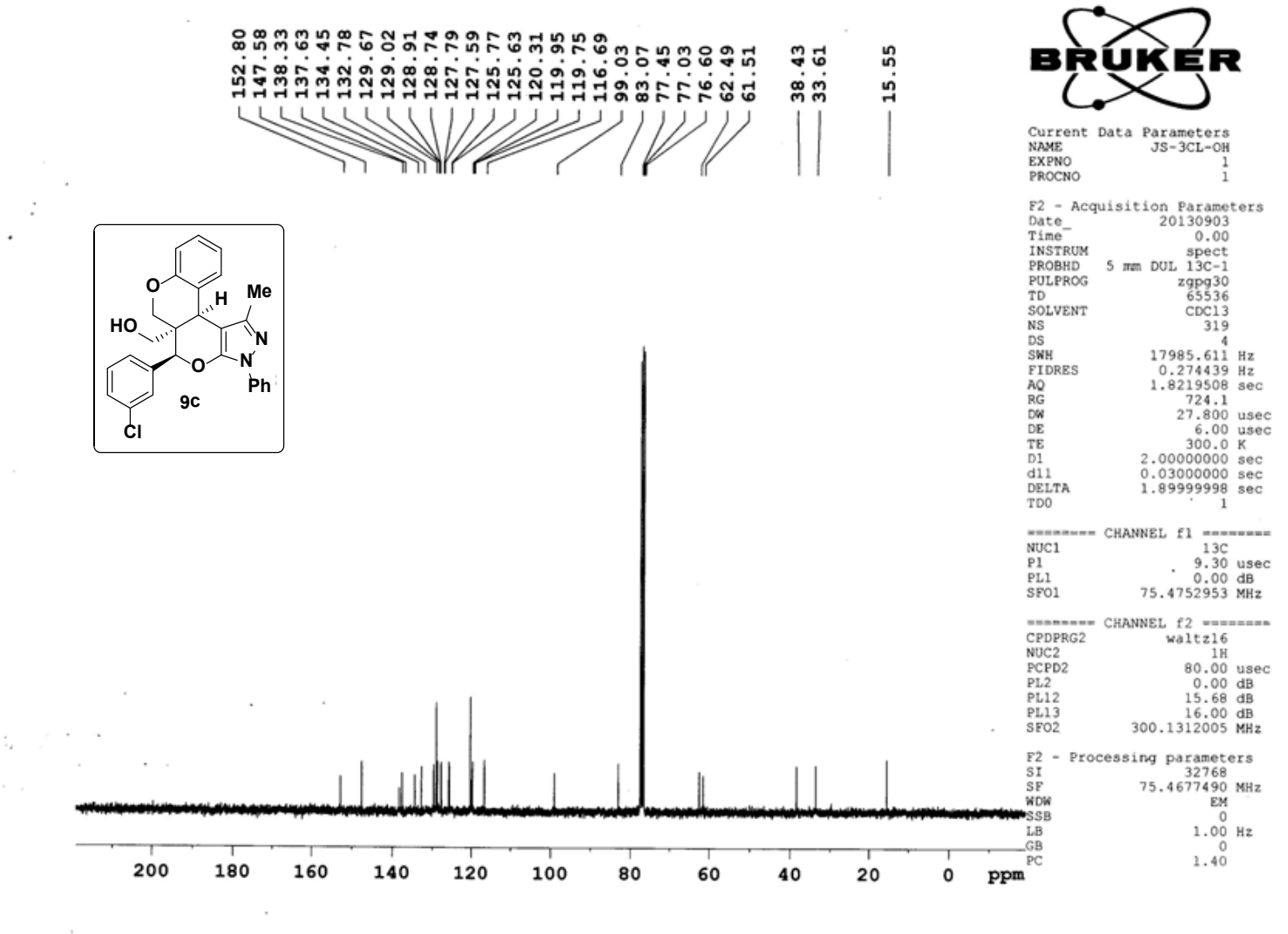
BRUKER

Current Data Parameters
NAME JS-3CL-OH
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 0.16
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 300.1300307 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

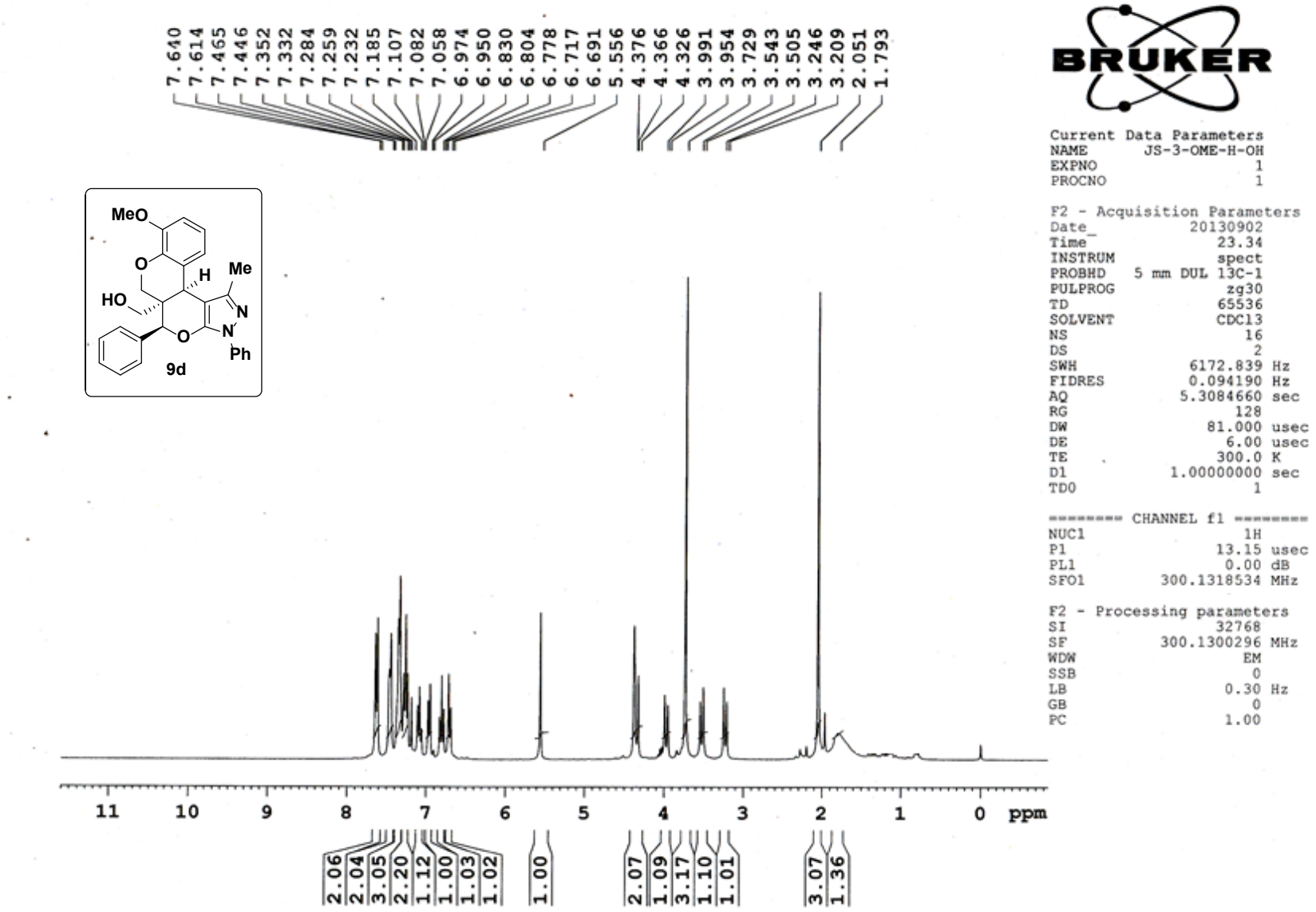
Current Data Parameters
NAME JS-3CL-OH
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 0.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 319
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 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



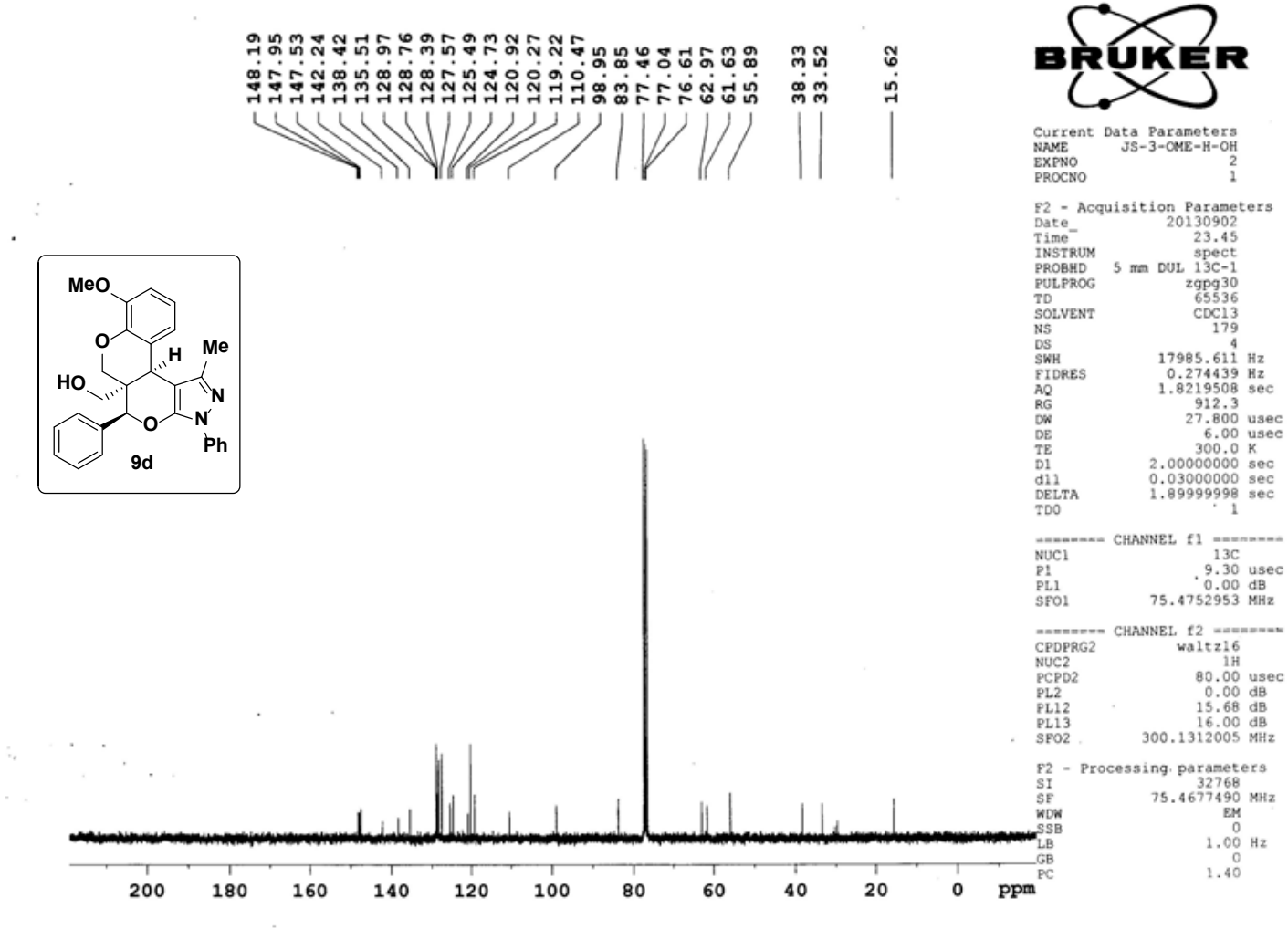
BRUKER

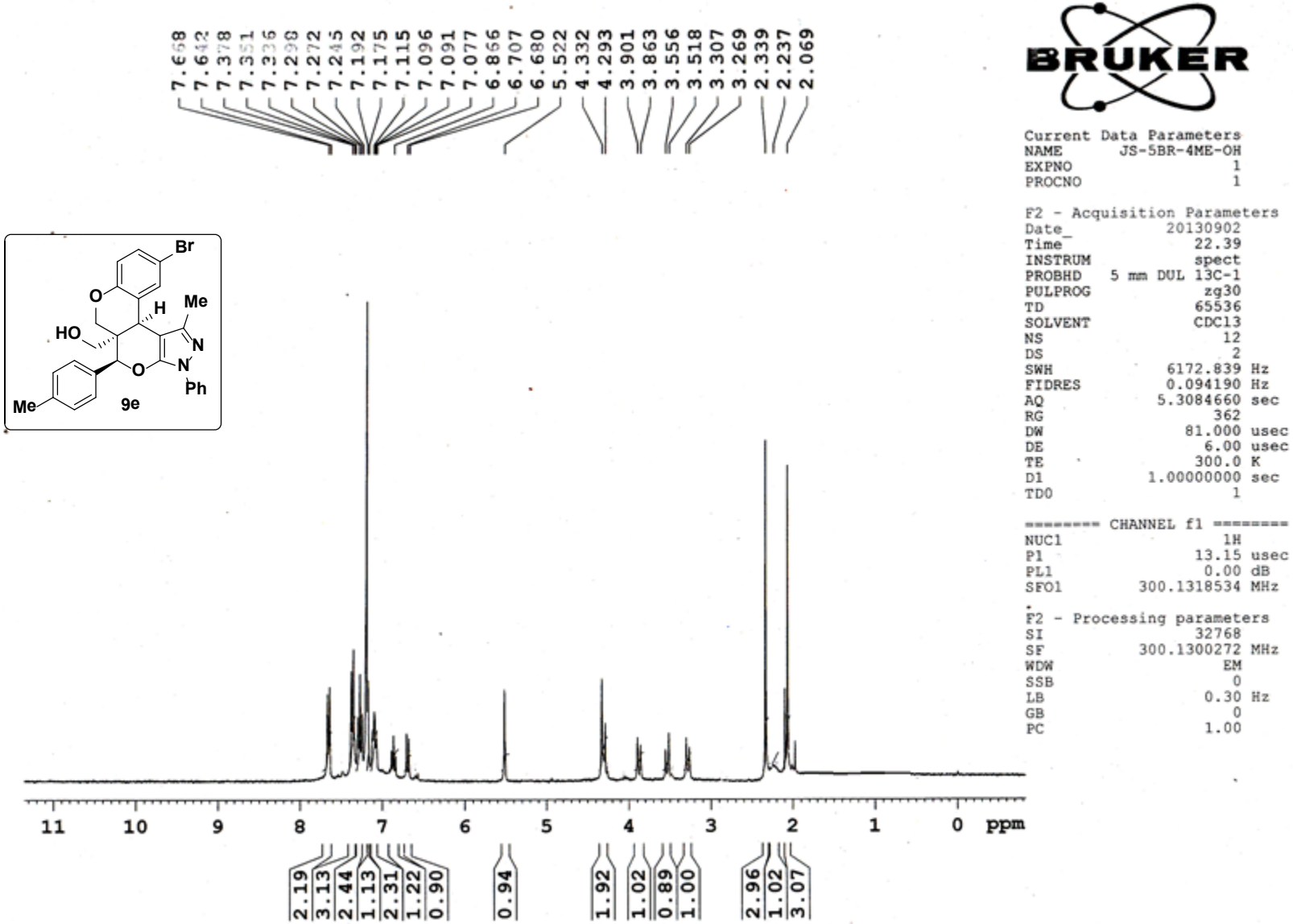
```
Current Data Parameters
NAME       JS-3-OME-H-OH
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130902
Time              23.34
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                 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       300.1300296 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



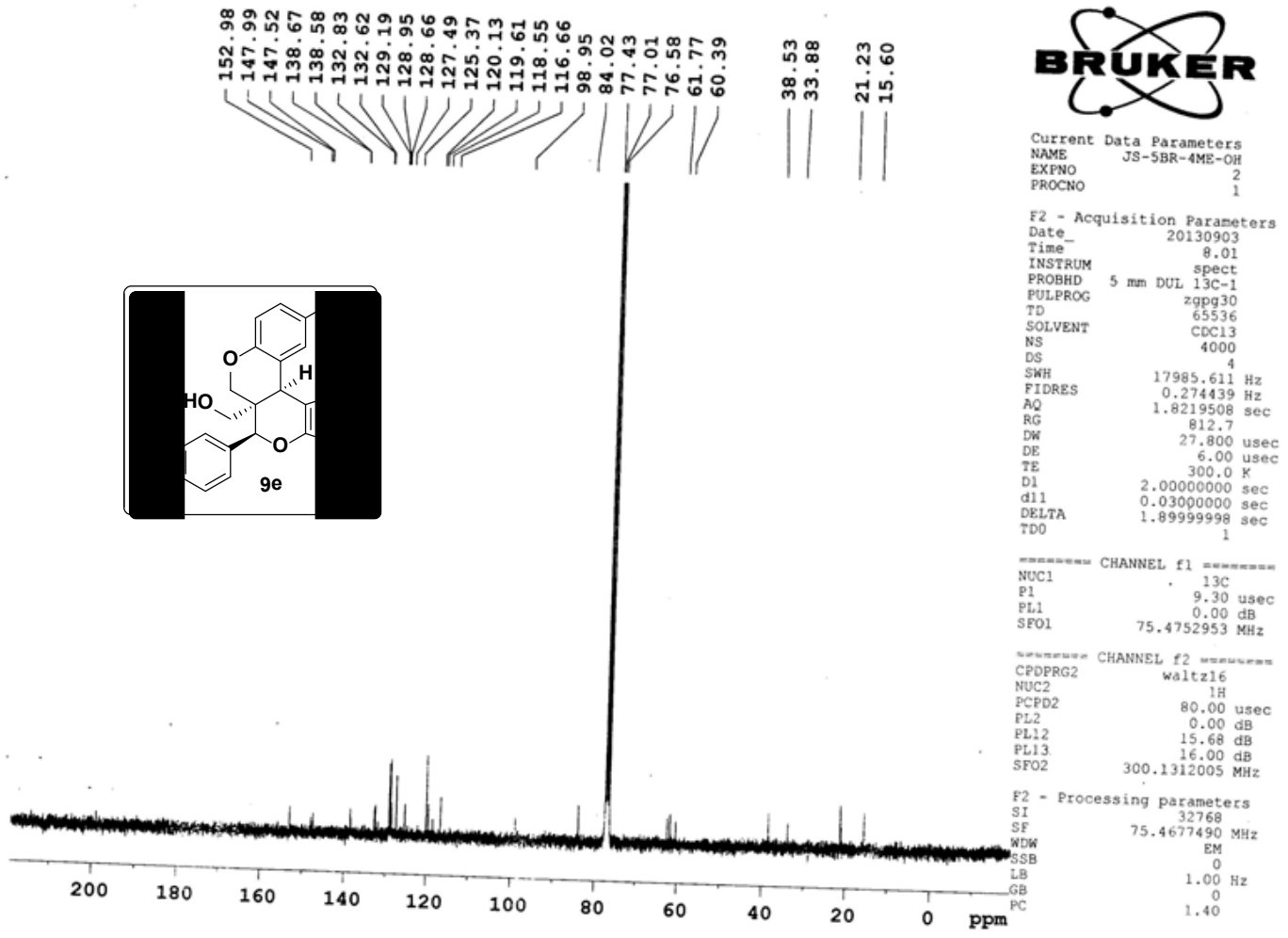



```
Current Data Parameters
NAME       JS-5BR-4ME-OH
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130902
Time              22.39
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                 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         300.1300272 MHz
WDW                  EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 1.00
```



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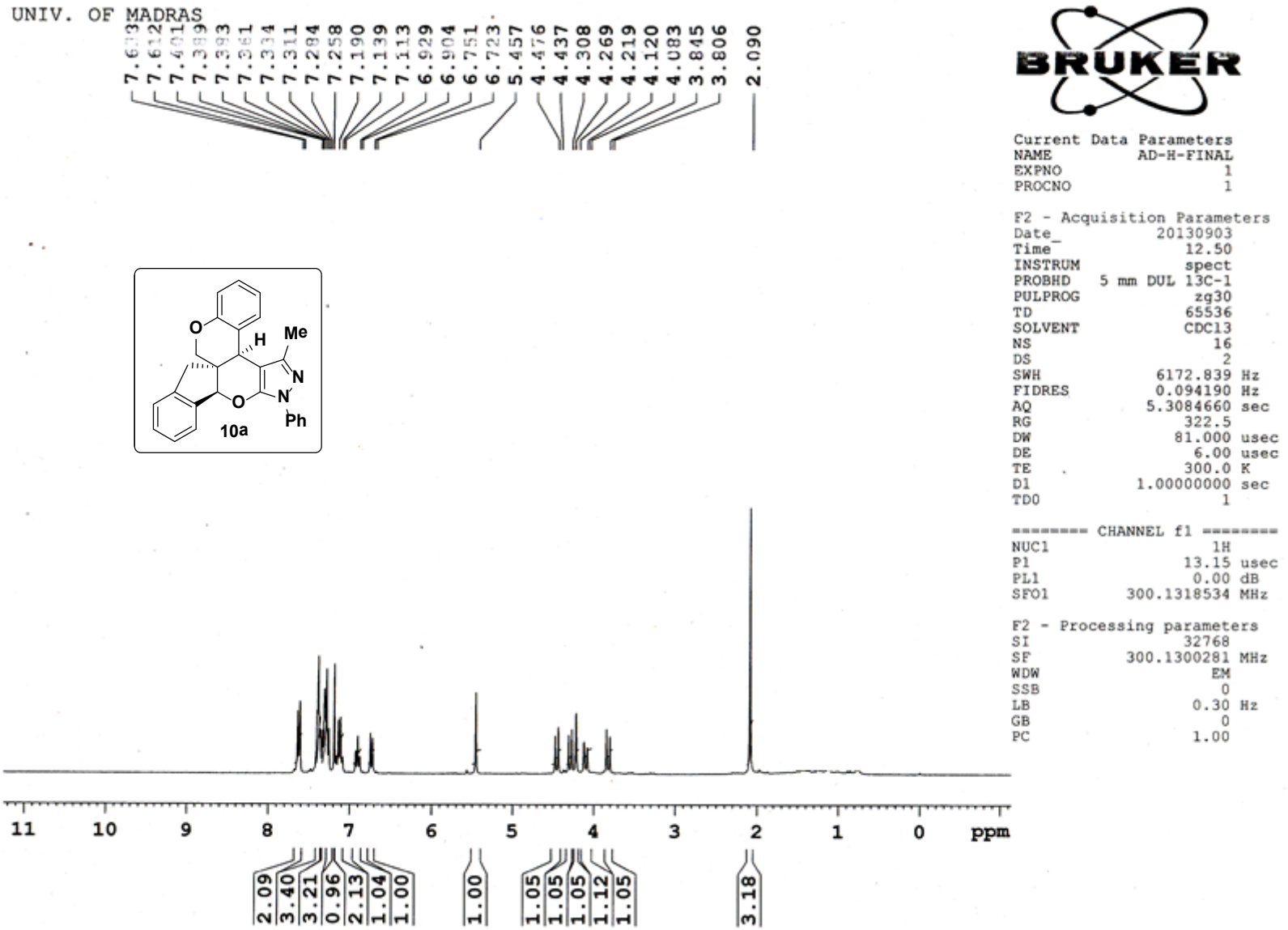
Current Data Parameters
NAME JS-5BR-4ME-OH
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 8.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 812.7
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



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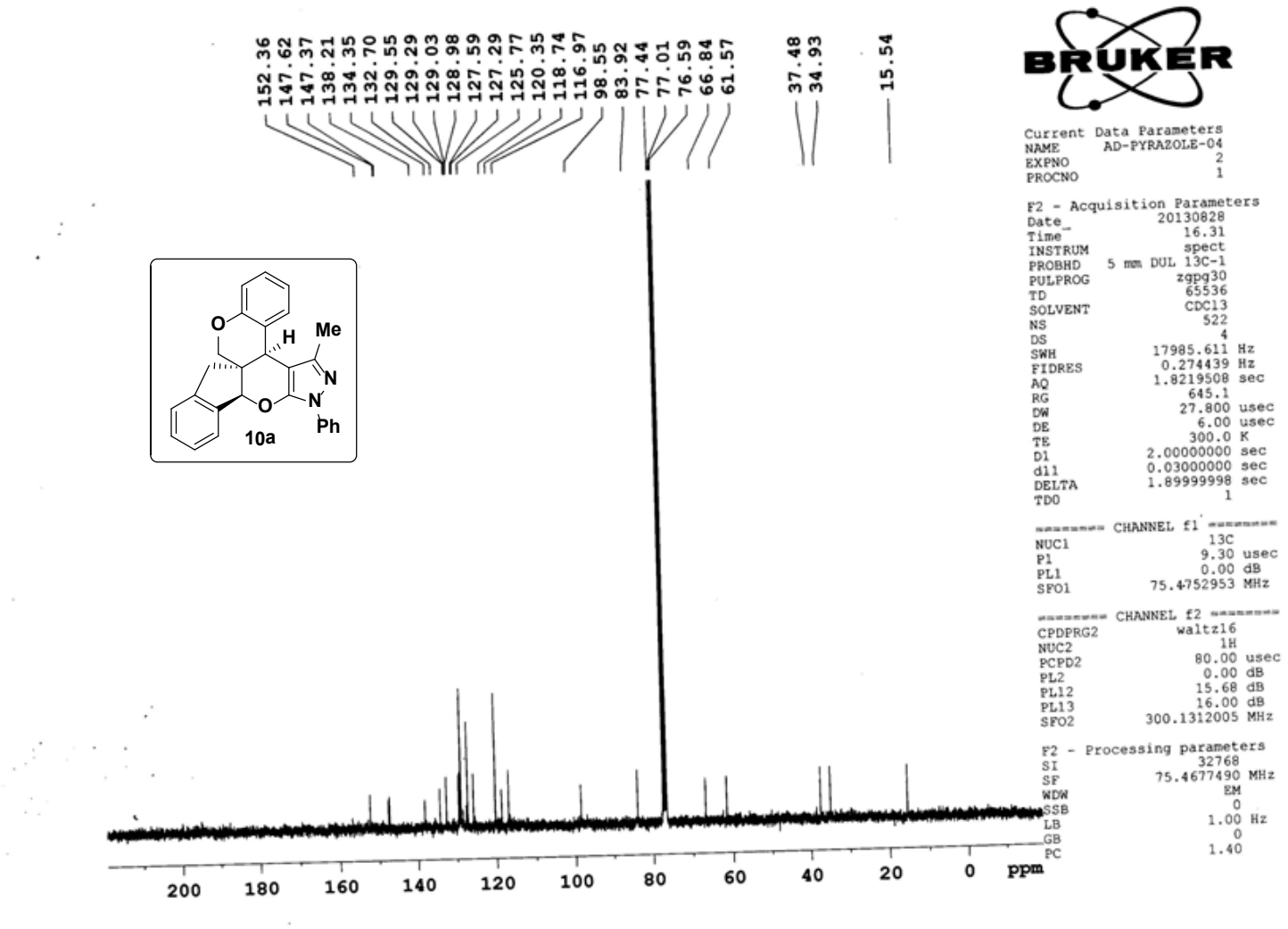
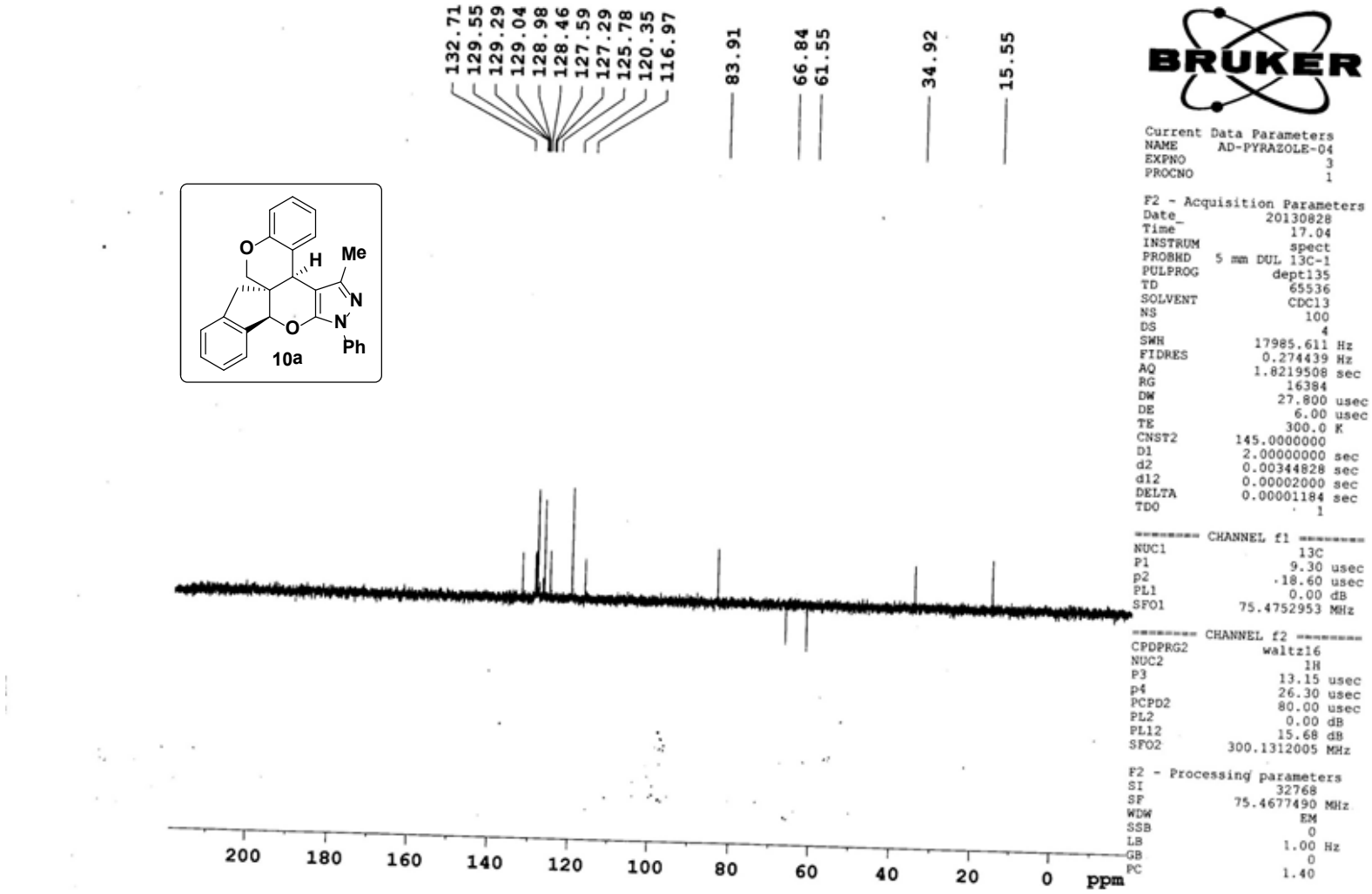
Current Data Parameters
NAME AD-H-FINAL
EXPNO 1
PROCNO 1

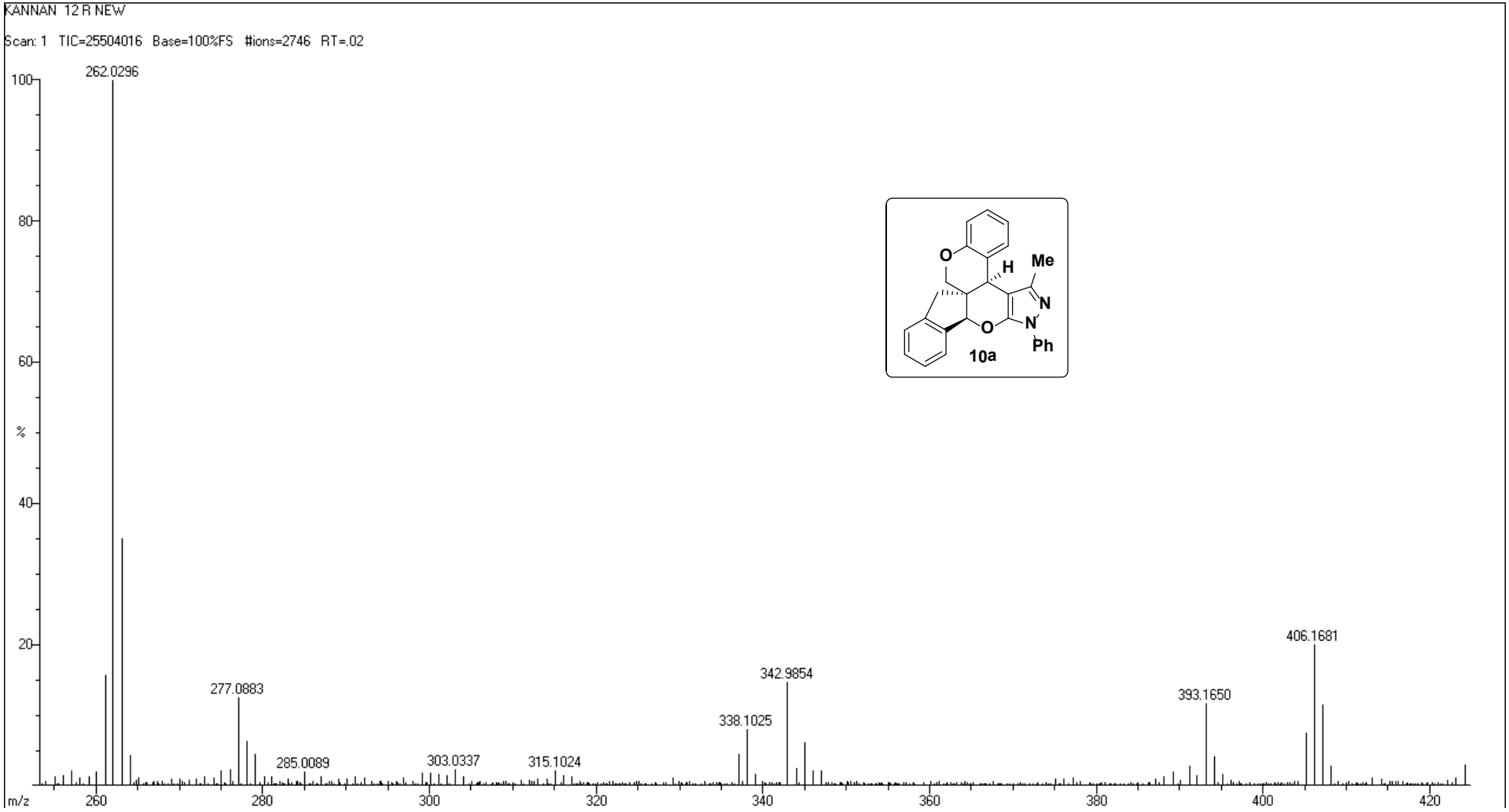
F2 - Acquisition Parameters
Date_ 20130903
Time 12.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

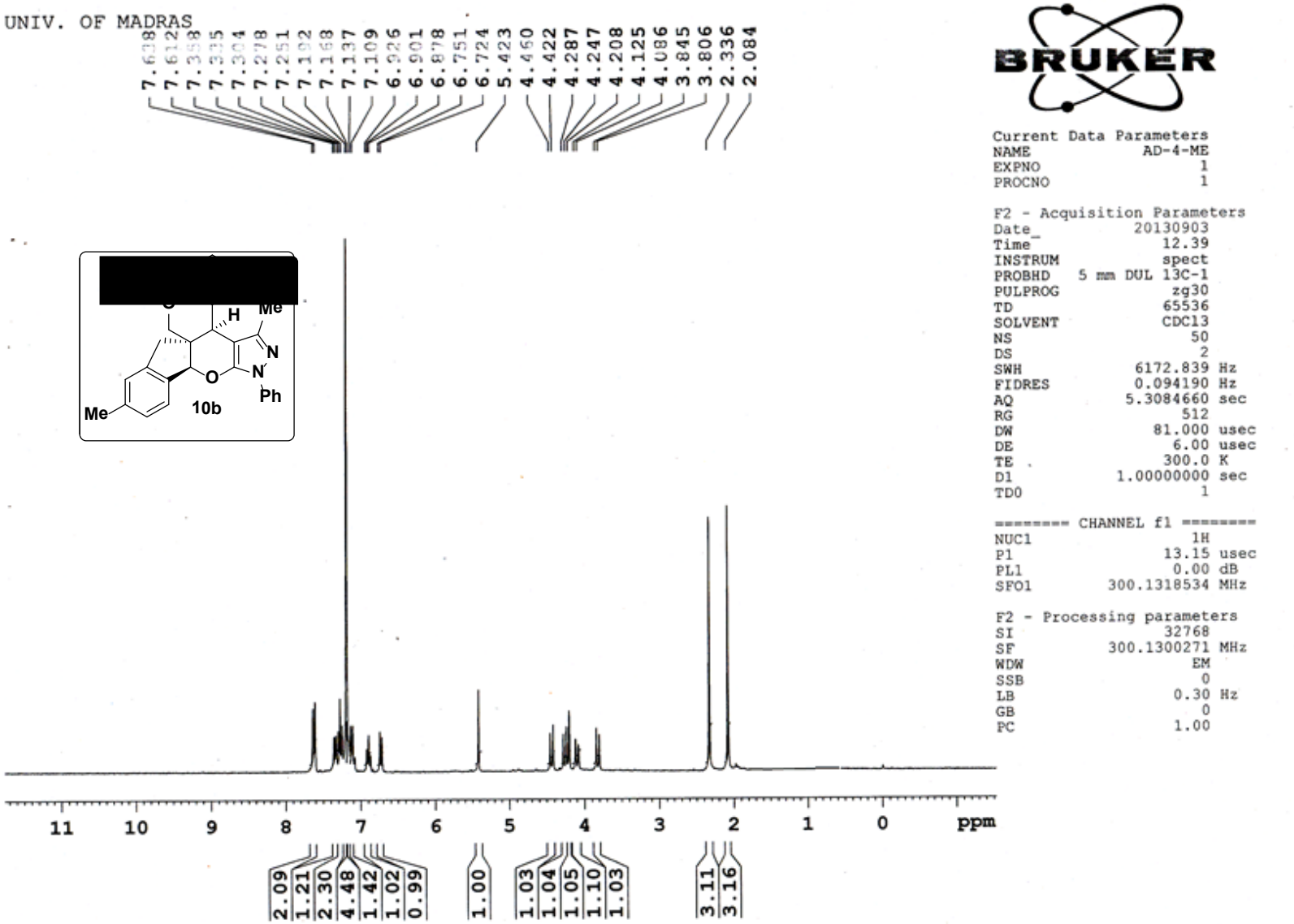
======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300281 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00









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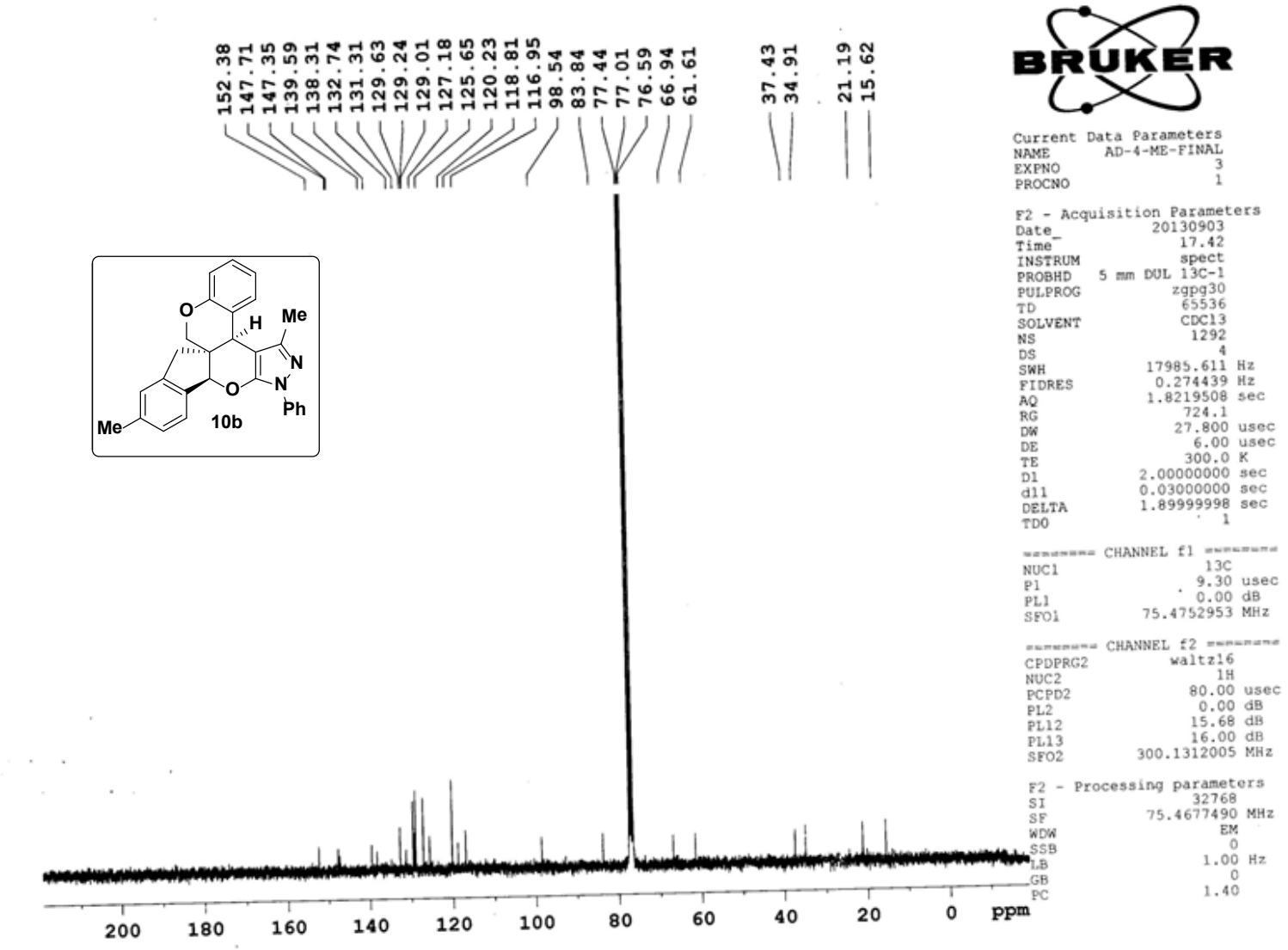
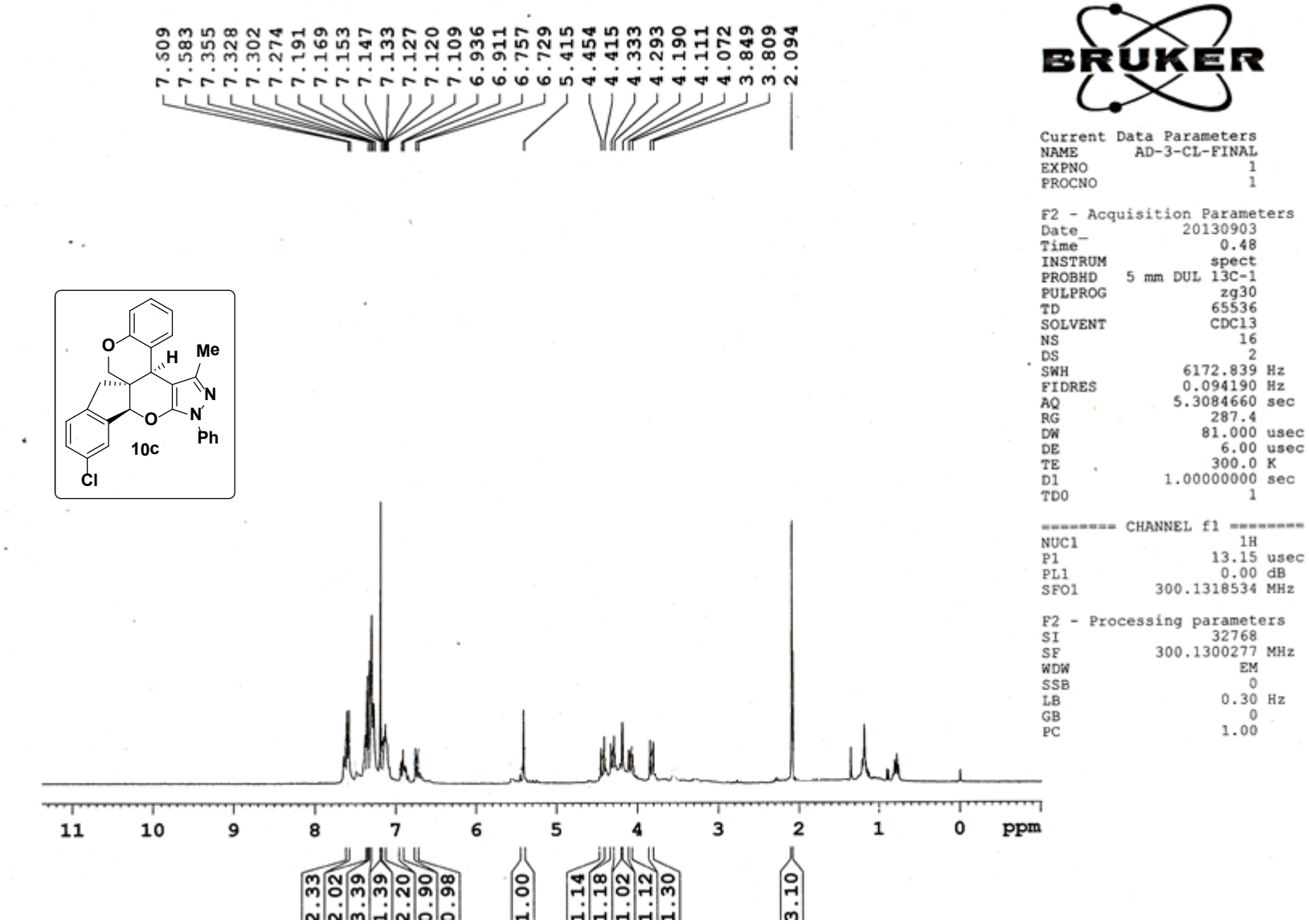
Current Data Parameters
NAME AD-4-ME
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 12.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 50
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 512
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 300.1300271 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





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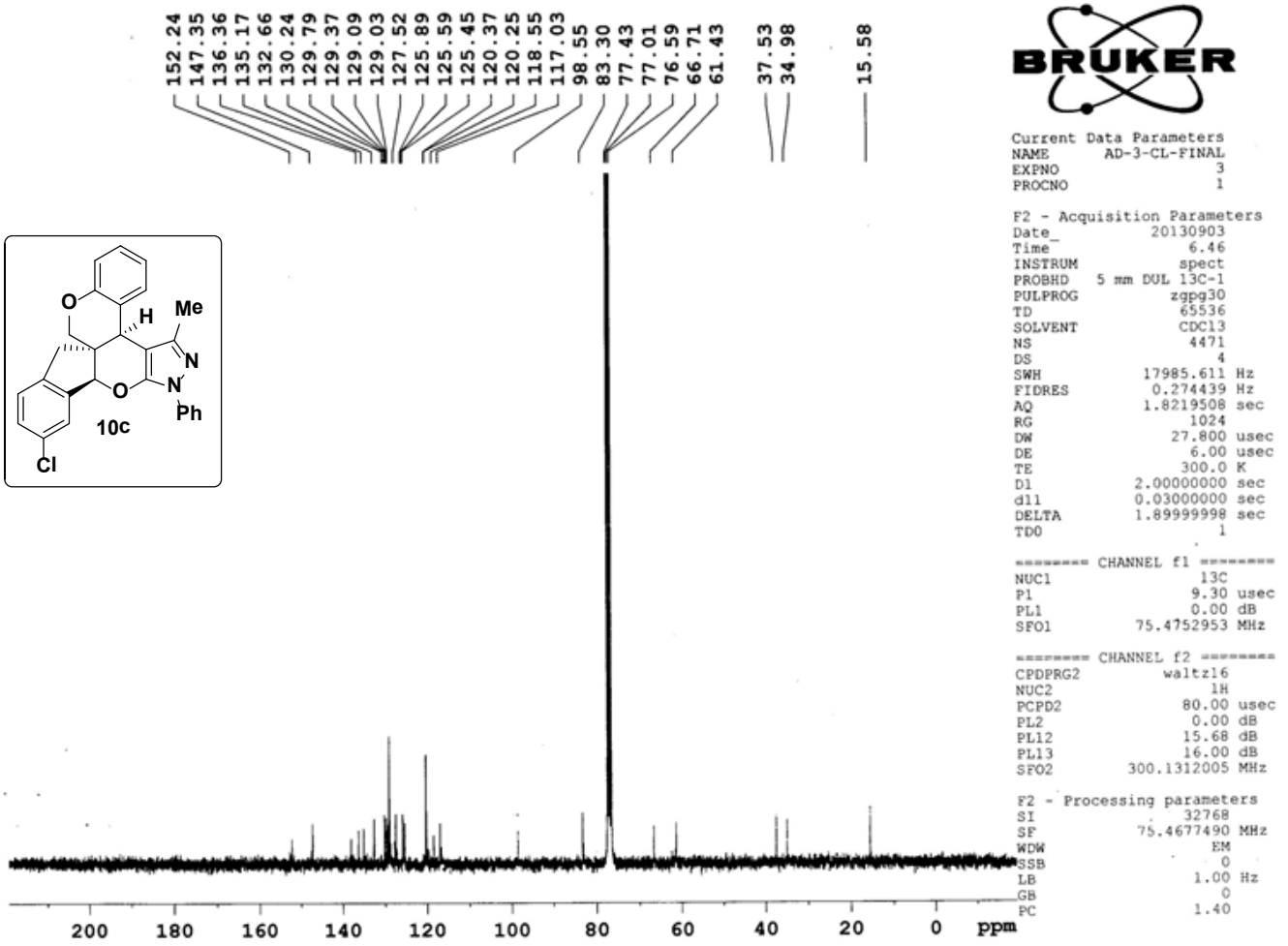
Current Data Parameters
NAME AD-3-CL-FINAL
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 0.48
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 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 300.1300277 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

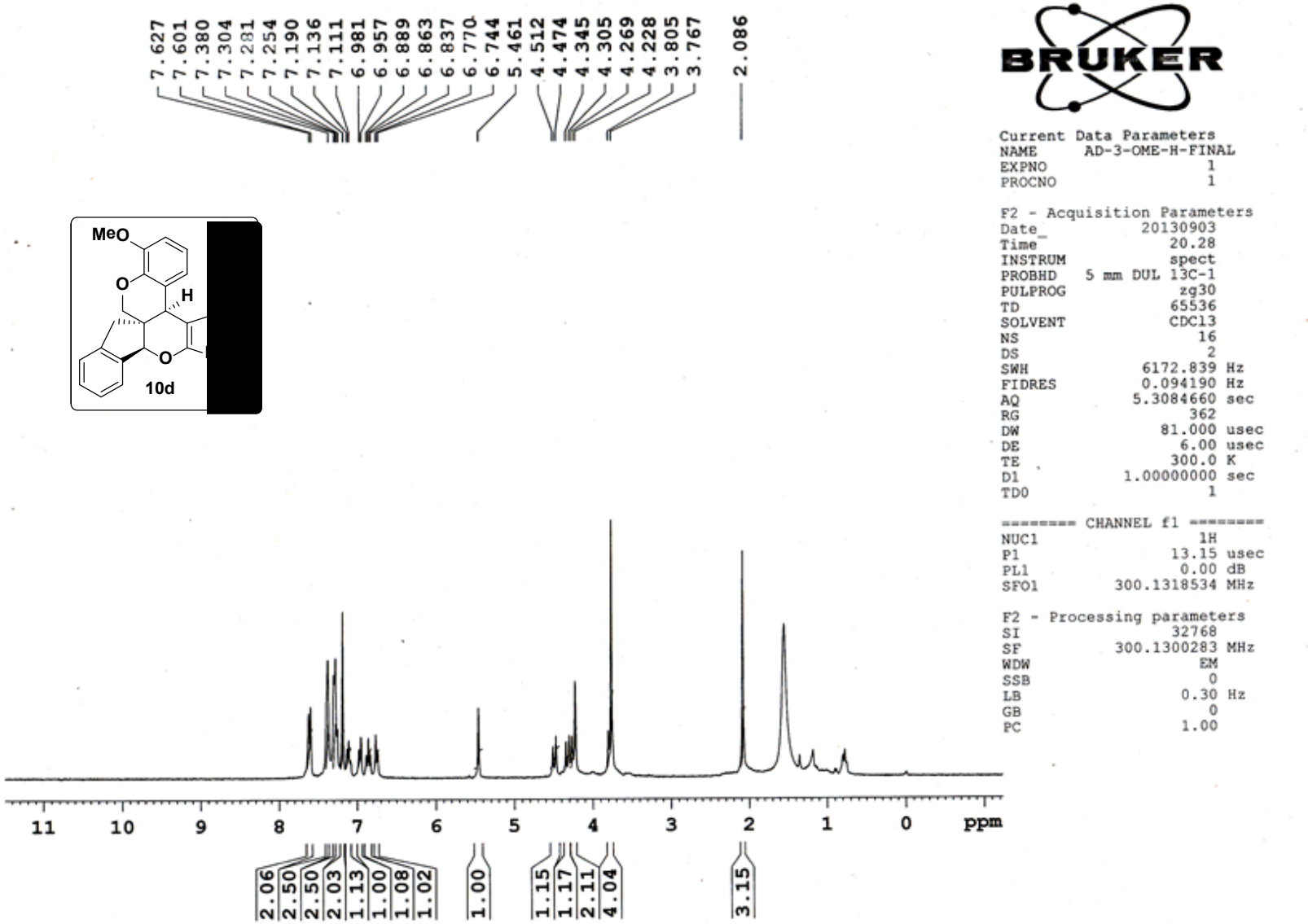
Current Data Parameters
NAME AD-3-CL-FINAL
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 6.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4471
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1024
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



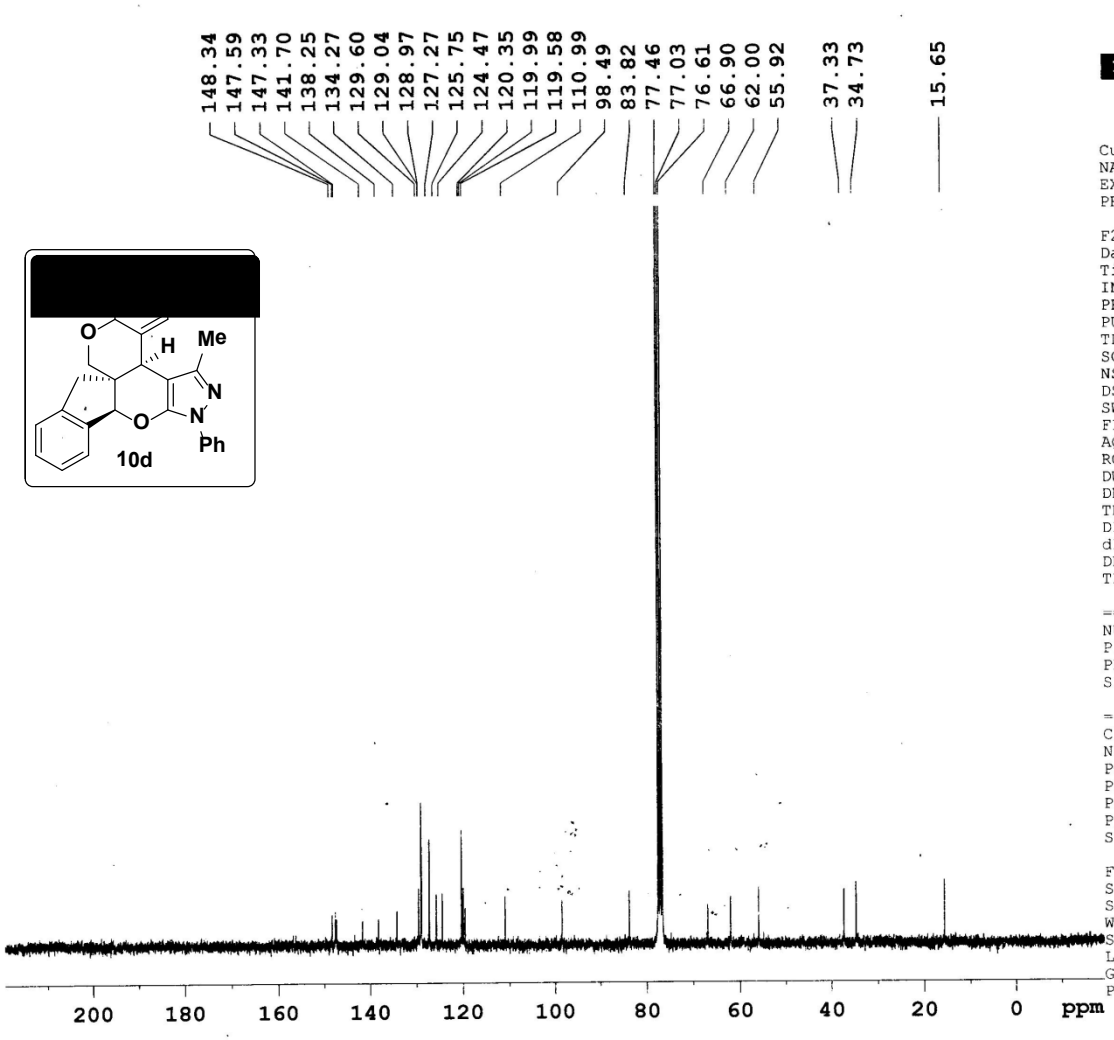
BRUKER

Current Data Parameters
NAME AD-3-OME-H-FINAL
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 20.28
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 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 300.1300283 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

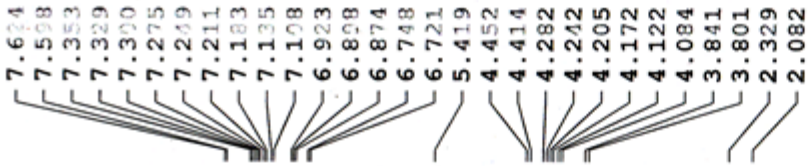
```
Current Data Parameters
NAME                  3-OME-H-F
EXPNO                         1
PROCNO                        1

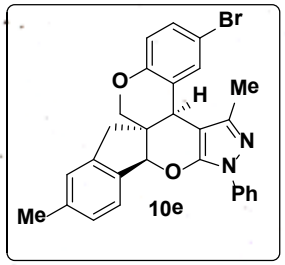
F2 - Acquisition Parameters
Date_                  20130903
Time                      22.21
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDC13
NS                         1024
DS                            4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                  1.8219508 sec
RG                        812.7
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

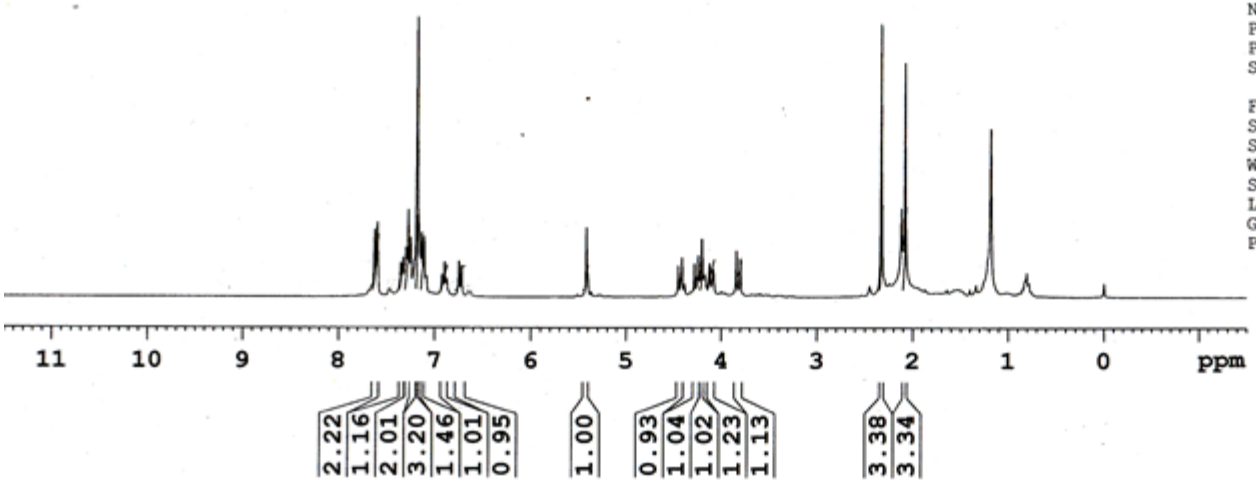
======== CHANNEL f1 ========
NUC1                        13C
P1                       9.30 usec
PL1                      0.00 dB
SFO1               75.4752953 MHz

======== 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
```







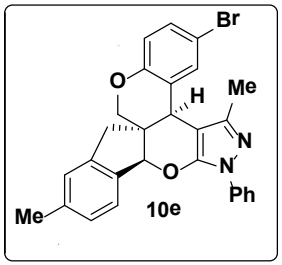
BRUKER

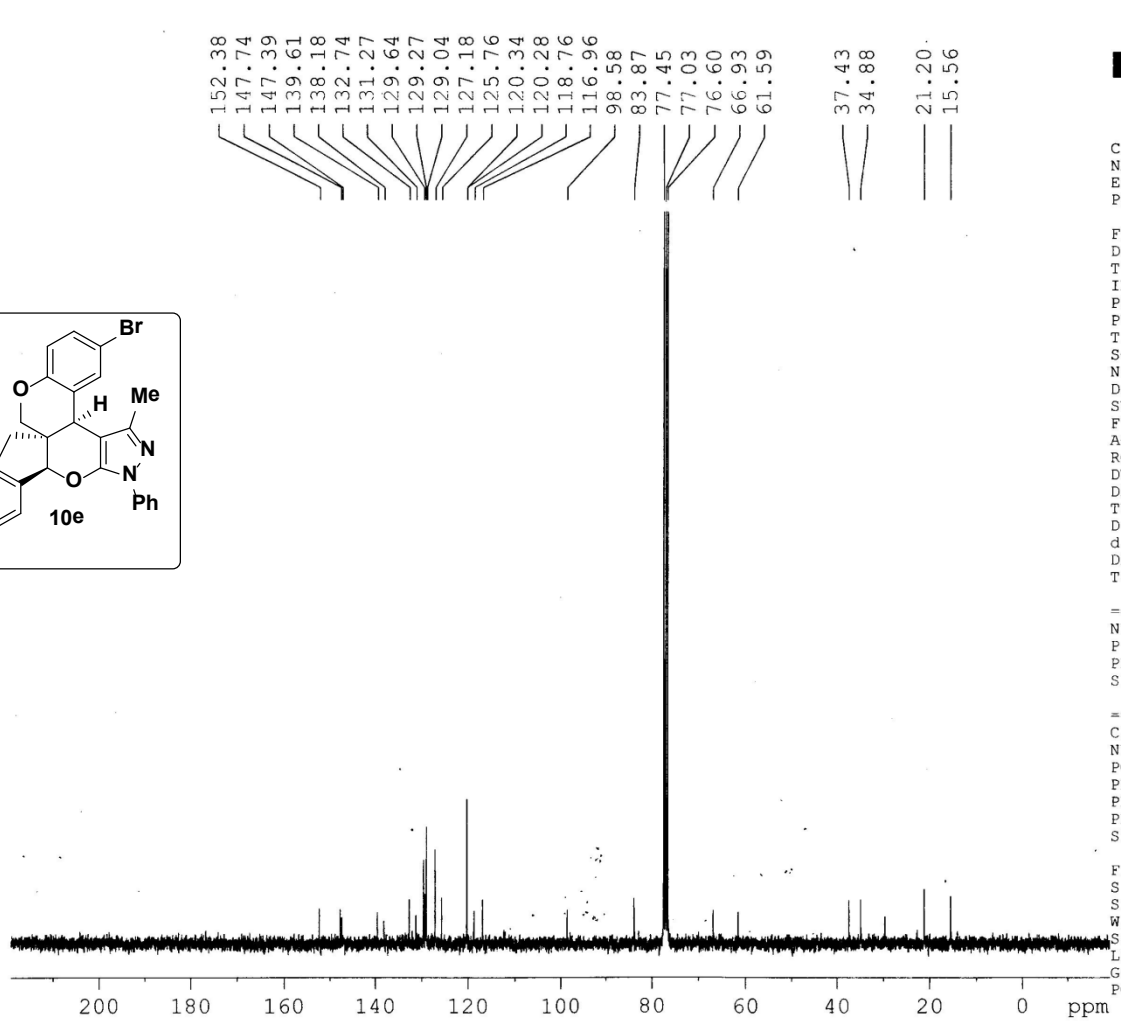
```
Current Data Parameters
NAME      AD-5BR-4-ME-FINAL
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130904
Time                 11.16
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            300.1300294 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```





BRUKER

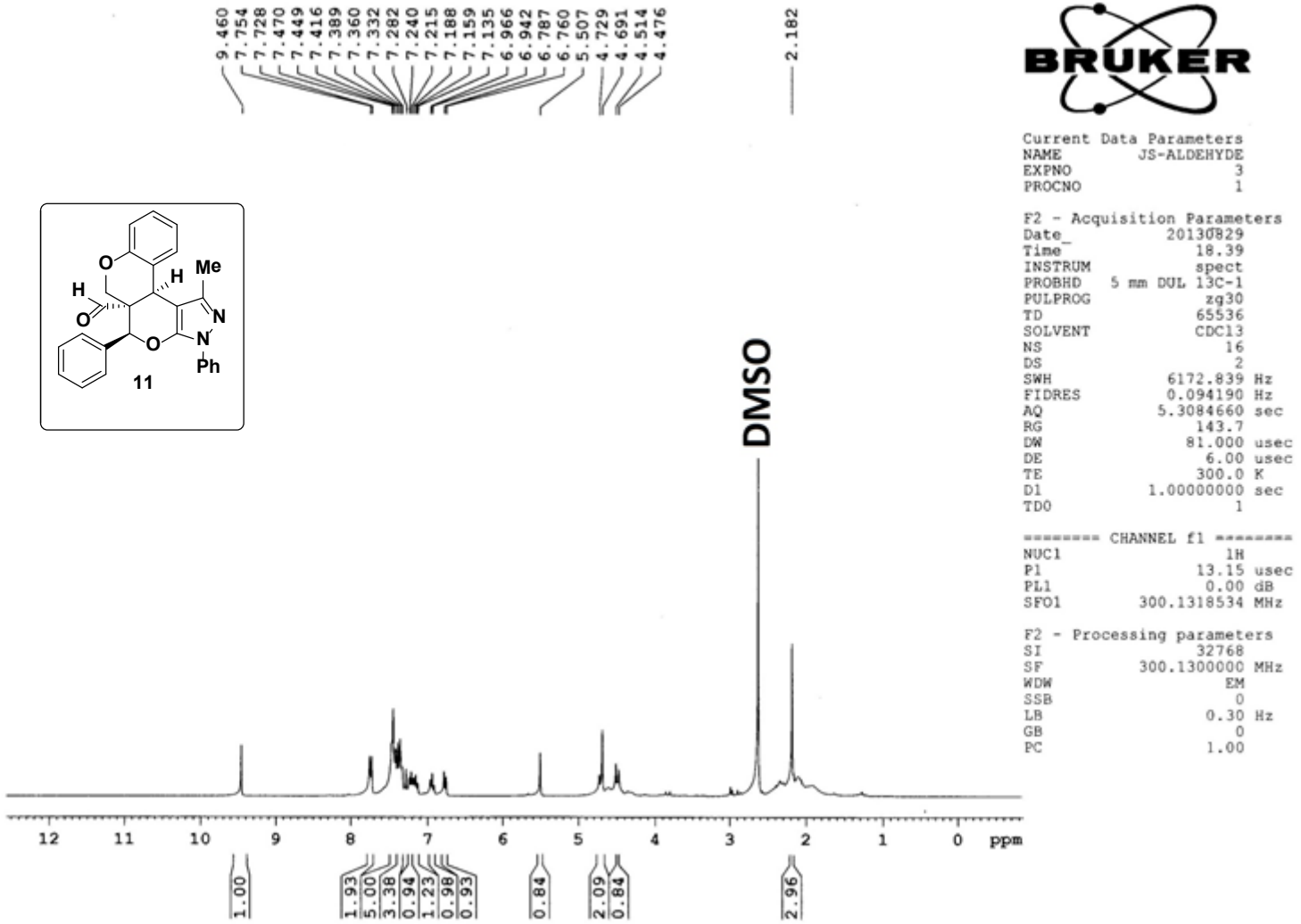
```
Current Data Parameters
NAME      AD-5BR-4-ME-FINAL
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130904
Time                 11.24
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     400
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                    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
```



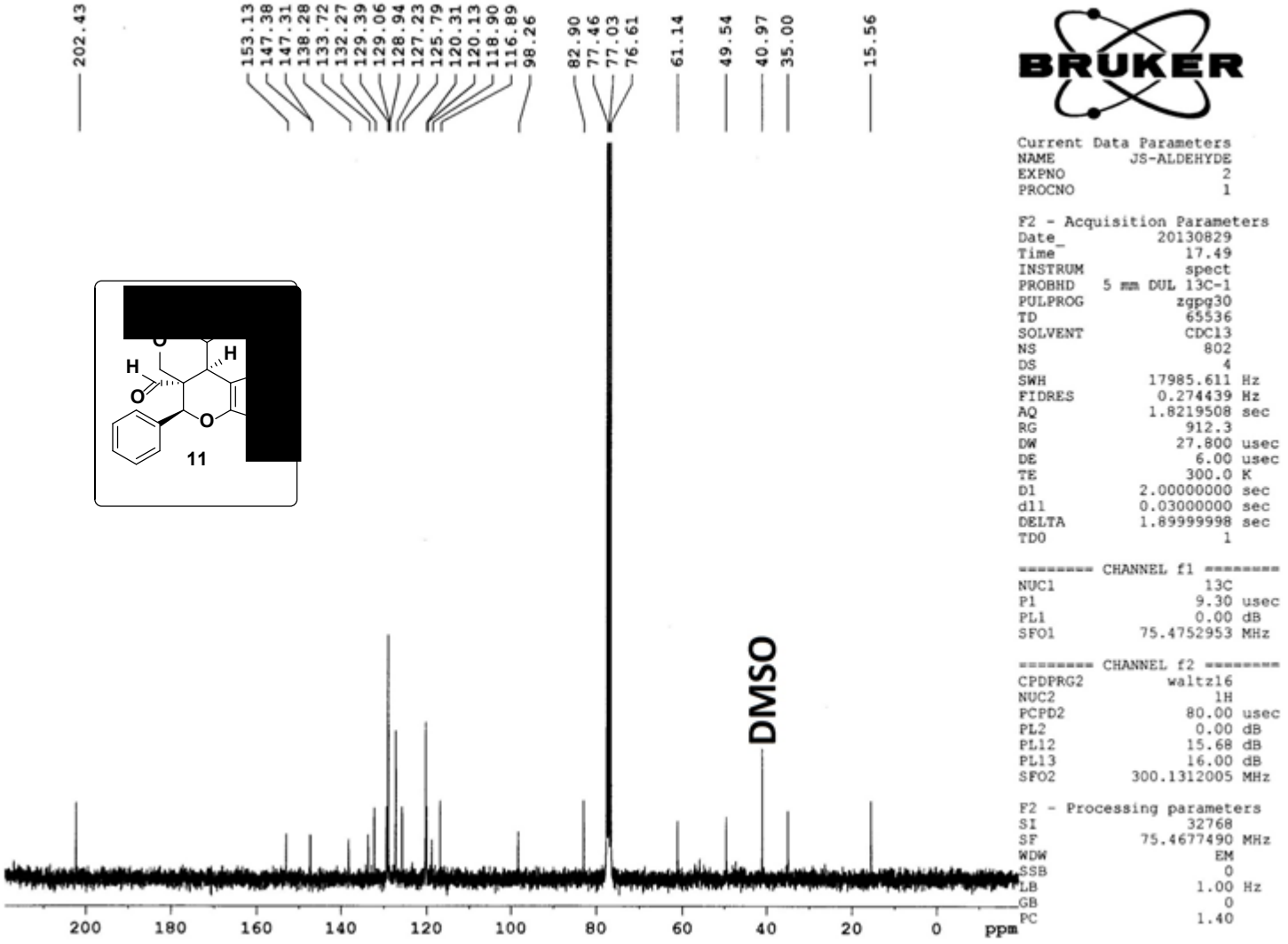
BRUKER

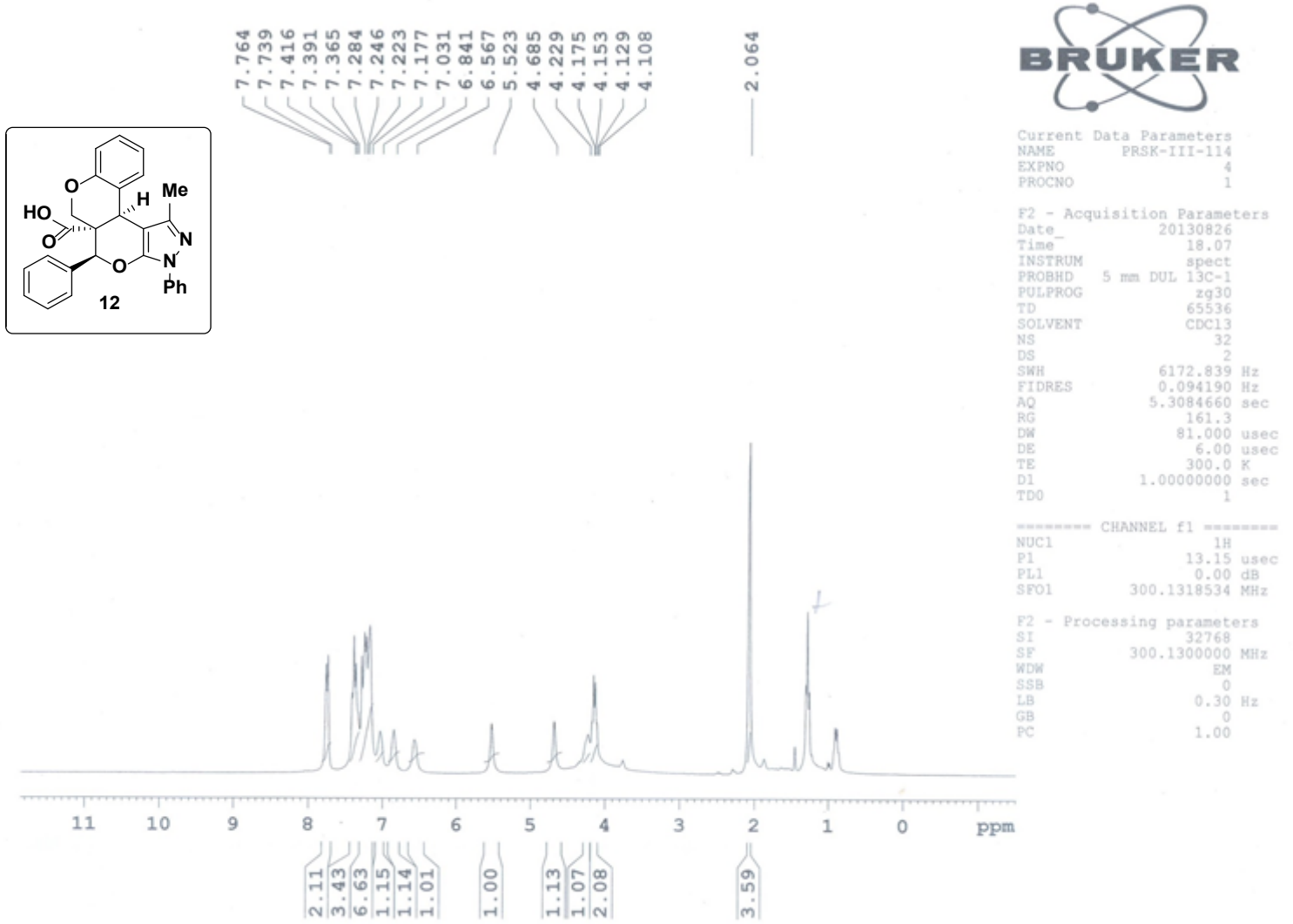
```
Current Data Parameters
NAME          JS-ALDEHYDE
EXPNO                   3
PROCNO                  1

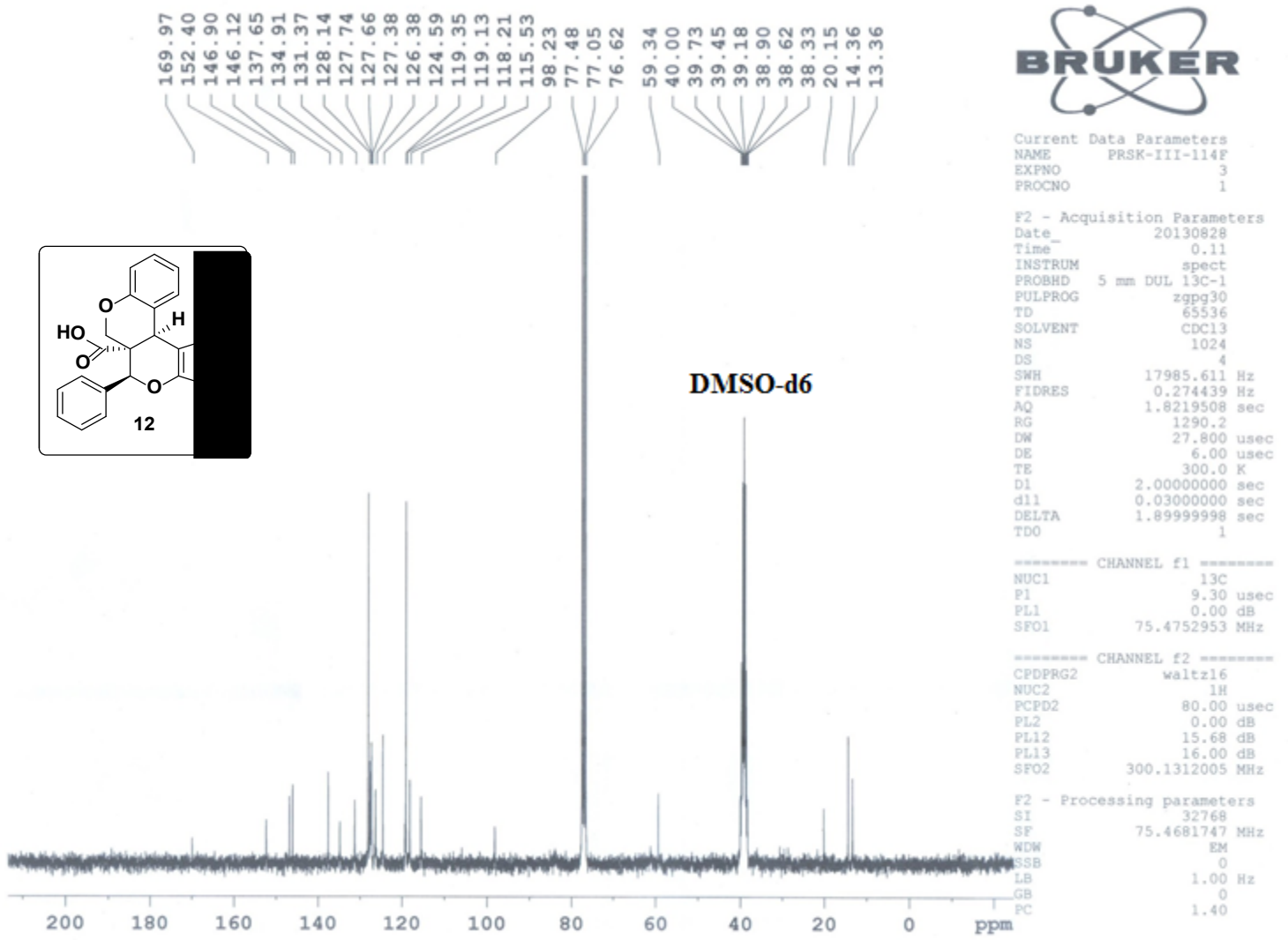
F2 - Acquisition Parameters
Date_            20130829
Time                18.39
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           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            300.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```







BRUKER

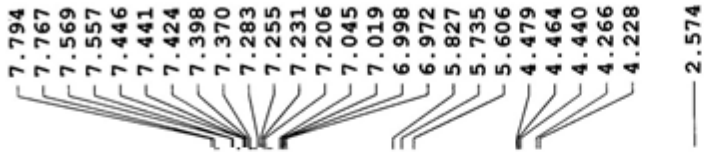
```
Current Data Parameters
NAME       PRSK-III-114F
EXPNO                 3
PROCNO                1

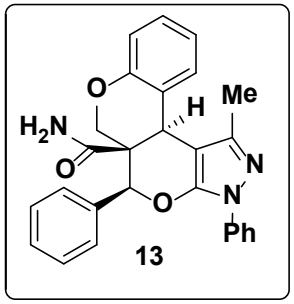
F2 - Acquisition Parameters
Date_          20130828
Time               0.11
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG               1290.2
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

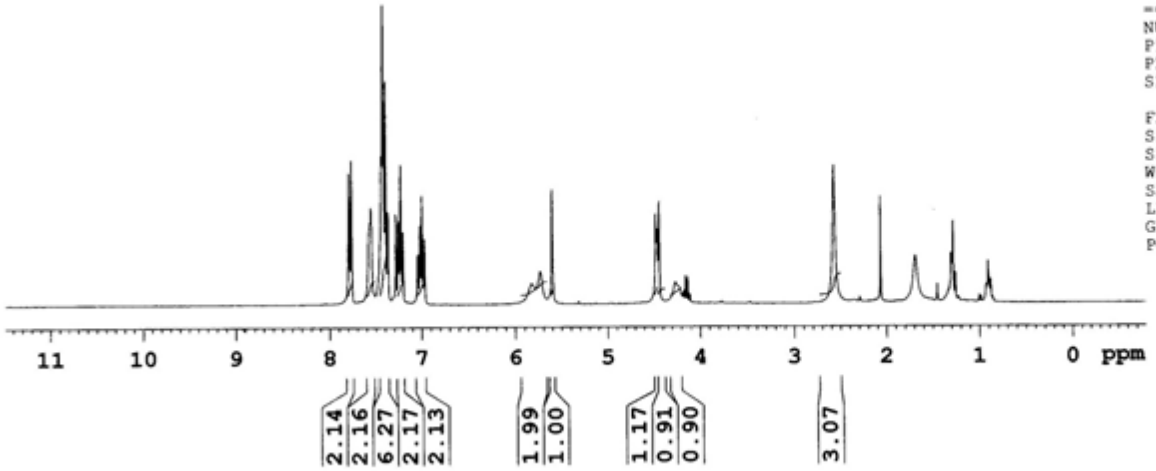
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             15.68 dB
PL13             16.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF         75.4681747 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```





13




```
Current Data Parameters
NAME      PRSK-III-118-CN
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130830
Time                12.20
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                 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          300.1300000 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```