Benzopentalenonaphthalenones from the Intramolecular Capture of a Merocyanine Derived from a Naphthopyran

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SUPPLEMENTARY INFORMATION

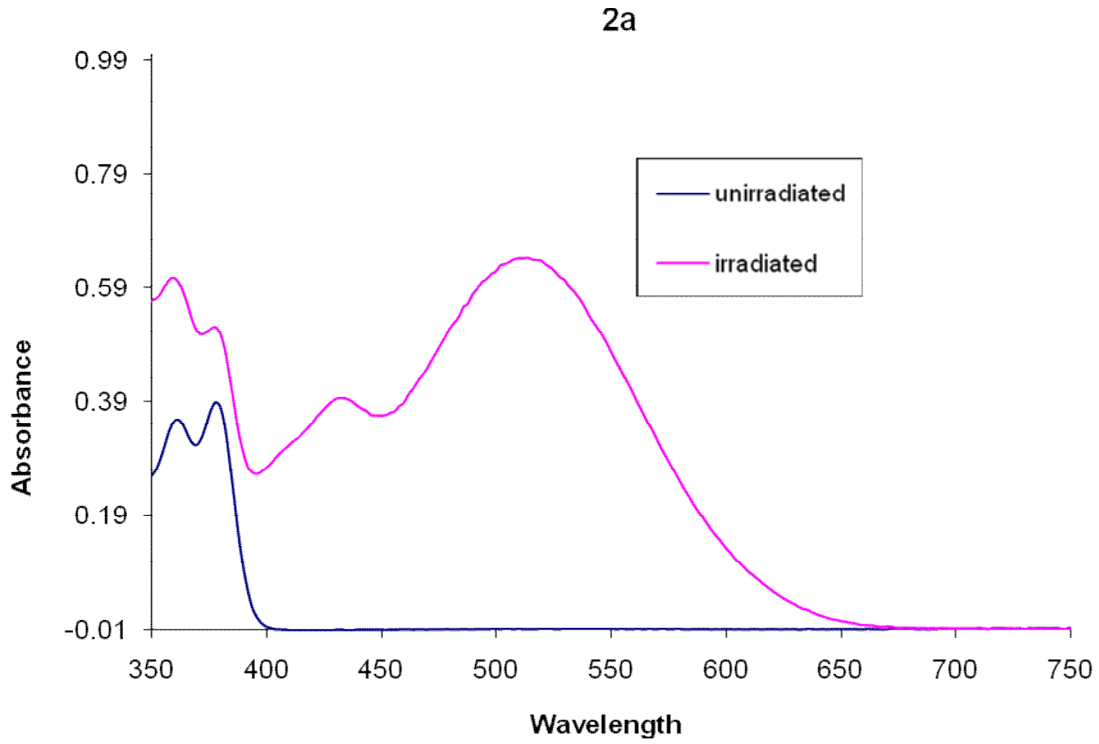
Equipment

Unless otherwise stated, reagents were used as supplied. NMR spectra were recorded on a Bruker Avance 400 MHz spectrophotometer (^{1}H NMR 400 MHz, ^{13}C NMR 100 MHz) for sample solutions in $CDCl_3$ with tetramethylsilane as an internal reference. The crystal structure determination was carried out at 150 K on a Bruker-Nonius Apex X8 diffractometer equipped with an Apex II CCD detector and using graphite monochromated Mo-Kα radiation from a FR591 rotating anode generator. The structure was solved by direct methods and refined using SHELXL-97. FT-IR spectra were recorded on a Perkin Elmer Spectrum One spectrophotometer system equipped with a golden gate ATR attachment (neat sample). UV-visible spectra were recorded for spectroscopic grade CH_2Cl_2 solutions of the samples (10 mm path length quartz cuvette, PTFE capped, *ca*. $1 \times 10^{-4} - 10^{-6}$ moldm^{-3}) using a Cary 50 Probe spectrophotometer equipped with a single cell Peltier temperature controlled (20 ^{o}C) stirred cell attachment with activating irradiation provided by an Oriel 150 Watt xenon arc lamp source (Newport 66906), xenon ozone free arc lamp (Newport 6255), distilled water liquid filter (Newport 6177), multiple filter holder (Newport 62020), UG11 filter (Newport FSO-UG11), fibre optic coupler (Newport 77799) and liquid light guide (Newport 77557). All compounds were homogeneous by TLC (Merck TLC Aluminium sheets, silica gel 60 F_{254}) using a range of eluent systems of differing polarity. Mass spectra were recorded at the National EPSRC Mass Spectrometry Service Centre, Swansea.

^{1}H and ^{13}C NMR, infrared and high resolution mss spectra of all new compounds follow the crystal structure data tables for compound **5a**.

Supplementary Material (ESI) for Chemical Communications

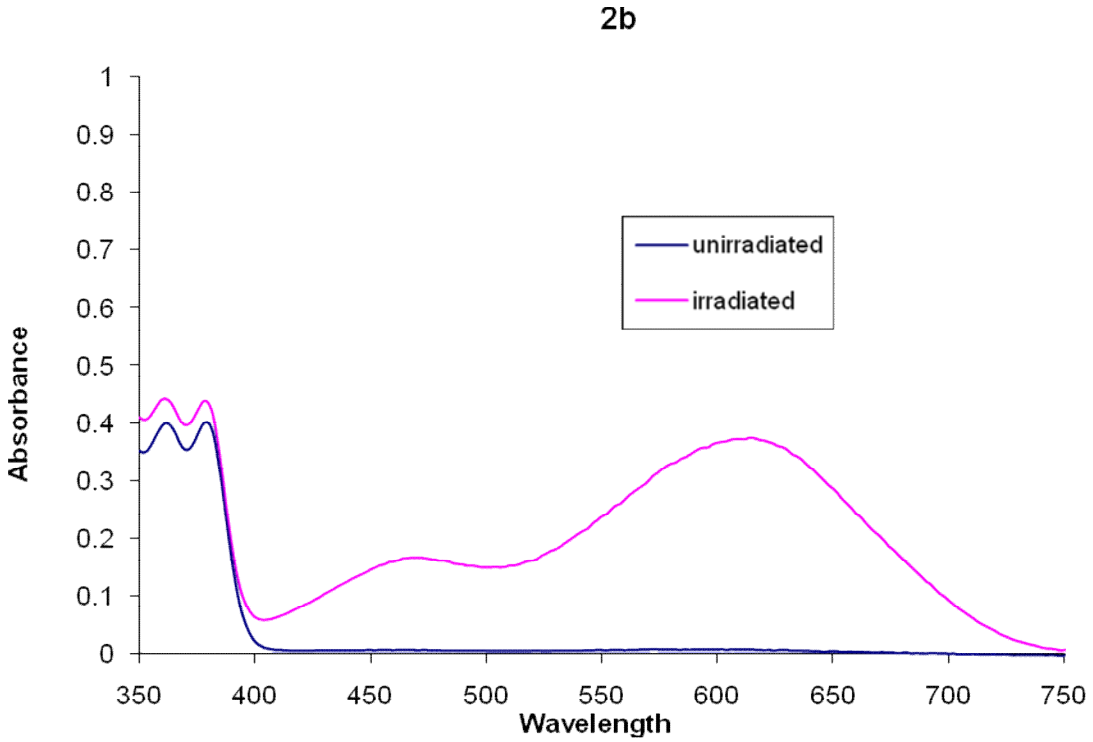
Ethyl 9-methoxy-2,2-bis(4-methoxyphenyl)-2*H*-naphtho[1,2-*b*]pyran-5-carboxylate **2a** was obtained according to the literature procedure and was identical in all respects to authentic material.[†] λ_{max} after irradiation = 514, 432 nm (CH_2Cl_2).



[†] C. D. Gabbutt, J. D. Hepworth, B. M. Heron, D. A. Thomas, C. Kilner, S. M. Partington, *Heterocycles*, **2004**, *63*, 567 – 582.

Preparation of Ethyl 2,2-bis(4-dimethylaminophenyl)-9-methoxy-2*H*-naphtho[1,2-*b*]pyran-5-carboxylate 2b

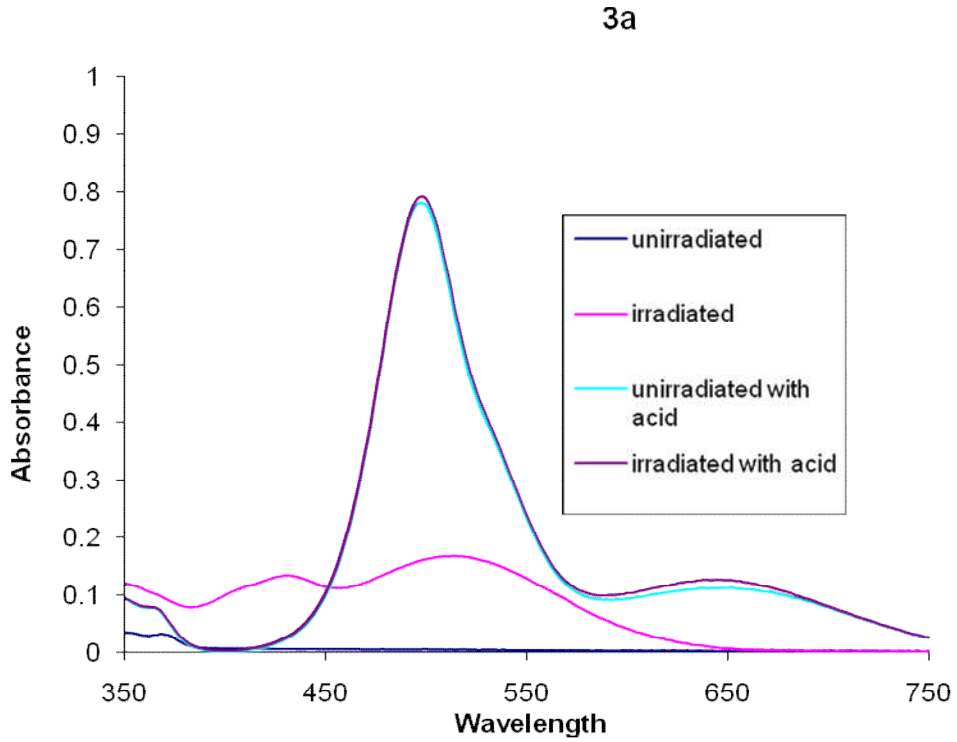
A suspension of ethyl 4-hydroxy-6-methoxynaphthalene-2-carboxylate 3.0 g (12.2 mmol), 1,1-bis(4-dimethylaminophenyl)prop-2-yn-1-ol 3.60 g (12.2 mmol) and Sasol Pural MG-30 (magnesium aluminium hydroxy carbonate, 30 % Mg content on silica) (10 g) in toluene (120 mL) were heated under reflux until TLC examination of the mixture indicated that no propynol remained. The cooled solution was filtered and the spent MG-30 catalyst was washed with toluene (2 × 30 mL). Removal of the combined toluene washings and reaction solvent gave a dark brown gum that was eluted from silica with 2% ethyl acetate in toluene to afford the title compound **2b** as colourless microcrystals (3.85g), 60.5 %, mp = 185 – 186 ^{o}C, ν_{max} 1694.1, 1607.0, 1513.1, 1435.8, 1350.4, 1287.8, 1194.6, 1167.2, 997.0, 809.9 cm^{-1}, λ_{max} after irradiation = 614, 472 nm (CH_2Cl_2), δ_H 1.41 (3H, t, *J* = 7.1 Hz, $CH_2C\underline{H}_3$), 2.90 (12H, s NMe_2), 3.93 (3H, s, OMe), 4.37 (2H, q, *J* = 7.1 Hz, $C\underline{H}_2CH_3$), 6.15 (1H, d, *J* = 10.1 Hz, 3-H), 6.34 (4H, m, Ar-H), 7.09 (1H, dd, *J* = 8.9, 2.5 Hz, 8-H), 7.34 (4H, m, Ar-H), 7.58 1H, d, *J* = 2.5 Hz, 10-H), 7.62 (1H, d, *J* = 10.1 Hz, 4-H), 7.66 (1H, d, *J* = 8.9 Hz, 7-H), 7.98 (1H, s, 6-H), δ_C 14.68, 40.74, 55.81, 61.02, 63.76, 82.83, 100.91, 112.08, 115.85, 119.73, 121.51, 122.61, 124.47, 128.33, 128.52, 129.39, 130.70, 133.44, 148.17, 149.99, 159.58, 167.56. Found $[M+H]^+$ = 523.2591, $C_{33}H_{34}N_2O_4$ requires $[M+H]^+$ = 523.2587.



General method for the preparation of the 5-[1,1-bis(4-methoxyphenyl)-1-hydroxymethyl] substituted naphthopyrans

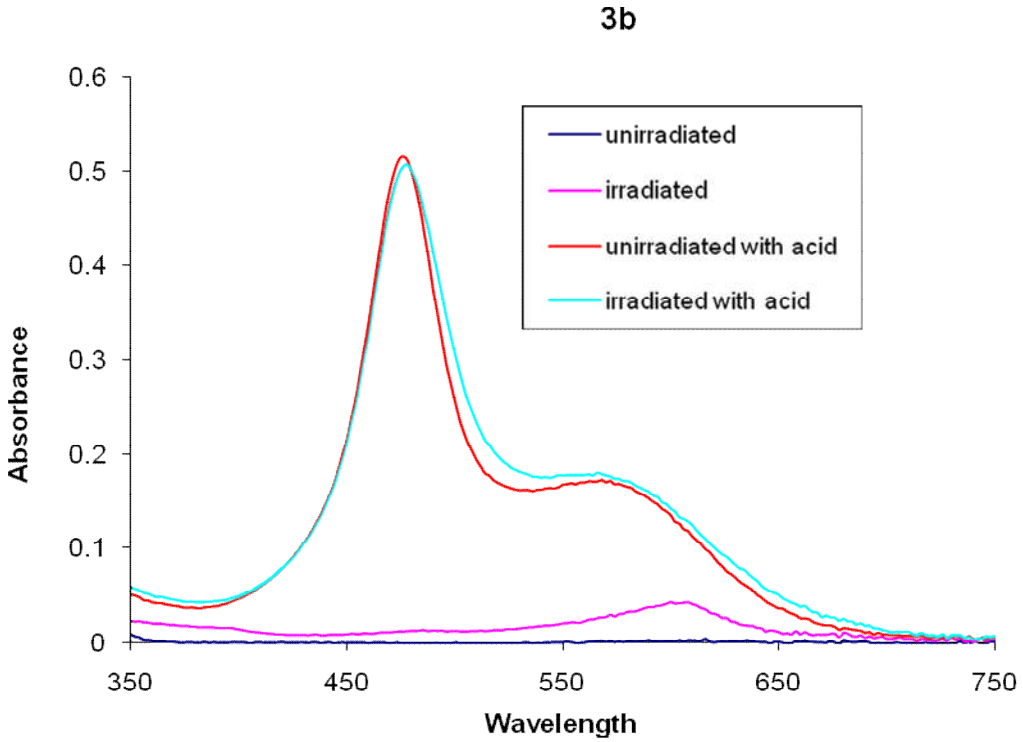
n-Butyllithium 10.1 mL (16.1 mmol, 1.6 M in hexanes) was added via syringe to a cold (~ -70 ^{o}C) stirred solution of 4-bromoanisole 3.01 g (16.1 mmol) in anhydrous THF (60 mL) under nitrogen. After 2 hours the naphthopyran (4 mmol) was added in a single portion and the solution was allowed to stir and warm to rt overnight. The reaction mixture was quenched with water (30 mL) and the layers separated. The aqueous phase was extracted with EtOAc (2 × 30 mL) and the combined organic layers were washed with water (50 mL), dried (anhyd. Na_2SO_4) and evaporated to afford the crude product as an off-white powder which was purified by column chromatography. The following compounds were obtained in this manner:

1. 9-Methoxy-5-[1,1-bis(4-methoxyphenyl)-1-hydroxymethyl]-2,2-bis(4-methoxyphenyl)-2*H*-naphtho[1,2-*b*]pyran **3a** as colourless microcrystals (2.13g) 79 % after elution from silica with 40% EtOAc in hexane and recrystallisation from acetone and methanol, mp = 181 – 182 ^{o}C, ν_{max} 3446.8, 1607.1, 1504.9, 1297.7, 1246.7, 1171.9, 1032.3, 944.4, 824.9, 585.9 cm^{-1}, λ_{max} after irradiation = 514, 429 nm (CH_2Cl_2) then upon addition of acid λ_{max} = 498 nm, $\varepsilon_{max} = 7.87 \times 10^4$ $mol^{-1}dm^3cm^{-1}$, λ_{max} = 648 nm, $\varepsilon_{max} = 1.26 \times 10^4$ $mol^{-1}dm^3cm^{-1}$ (CH_2Cl_2 + 2μL $MeSO_3H$), δ_H 2.96 (1H, s, OH), 3.76 (6H, s, OMe), 3.82 (6H, s, OMe), 3.92 (3H, s, OMe), 5.78 (1H, d, *J* = 10.1 Hz, 3-H), 6.68 (1H, s, 6-H), 6.82 (8H, m, Ar-H), 6.89 (1H, d, *J* = 10.1 Hz, 4-H), 7.03 (1H, dd, *J* = 9.2, 2.4 Hz, 8-H), 7.14 (4H, m, Ar-H), 7.32 (4H, m, Ar-H), 7.39 (1H, d, *J* = 9.2 Hz, 7-H), 7.56 (1H, d, *J* = 2.4 Hz, 10-H), δ_C 55.47, 55.51, 55.76, 81.80, 82.36, 100.86, 113.44, 113.55, 116.52, 119.02, 121.86, 124.10, 126.20, 126.84, 128.28, 128.57, 129.24, 130.04, 137.53, 138.29, 139.52, 148.58, 158.35, 158.80, 159.07. Found $[M+Na]^+$ = 689.2503 $C_{43}H_{38}O_7$ requires $[M+Na]^+$ = 689.2510.



2. 2,2-Bis(4-dimethylaminophenyl)-9-methoxy-5-[1,1-bis(4-methoxyphenyl)-1-hydroxymethyl]-2*H*-naphtho[1,2-*b*]pyran **3b** as pale green microcrystals (1.64 g), 59 % after elution from silica with 10% ethyl acetate in toluene and recrystallisation from CH_2Cl_2 and hexane, mp = 136 – 138 ^{o}C, ν_{max} 3489.9, 1604.5, 1506.4, 1348.7, 1245.0, 1169.4, 1030.9, 996.5, 816.2, 547.2 cm^{-1}, λ_{max} after irradiation = 615, 495 nm (CH_2Cl_2) then upon addition of acid λ_{max} = 476 nm, ε_{max} = 5.11 × 10^4 $mol^{-1}dm^3cm^{-1}$, λ_{max} = 568 nm, ε_{max} = 1.72 × 10^4 $mol^{-1}dm^3cm^{-1}$ (CH_2Cl_2 + 2μL $MeSO_3H$), δ_H 2.96 (6H, s, NMe_2), 2.99 (6H, s, NMe_2), 3.77 (3H, s, OMe), 3.78 (3H, s, OMe), 3.92 (3H, s, OMe), 5.69 (1H, s, OH), 5.87 (1H, d, *J* = 10.1 Hz, 3-H), 6.12 (1H, d, *J* = 10.1 Hz, 4-H), 6.63 (2H, m, Ar-H), 6.74 (2H, m, Ar-H), 6.81 (4H, m, Ar-H), 7.09 (1H, dd, *J* = 8.9, 2.5 Hz, 8-H), 7.10 (1H, s, 6-H), 7.23 (2H, m, Ar-H), 7.29 (2H, m, Ar-H), 7.37 (4H, m, Ar-H), 7.44 (1H, d, *J* = 2.5 Hz, 10-H), 7.63 (1H, d, *J* = 8.9 Hz, 7-H), δ_C 40.44, 55.23, 55.26, 55.37, 77.53, 91.80, 99.87,

111.86, 112.08, 113.14, 113.26, 114.26, 119.00, 121.36, 123.02, 125.09, 128.81, 129.15, 129.23, 129.41, 130.46, 131.17, 136.21, 138.27, 143.15, 144.76, 149.02, 157.25, 158.71, 158.87. Found $[M+H]^+$ = 693.3320 $C_{45}H_{44}N_2O_5$ requires $[M+H]^+$ = 693.3323.

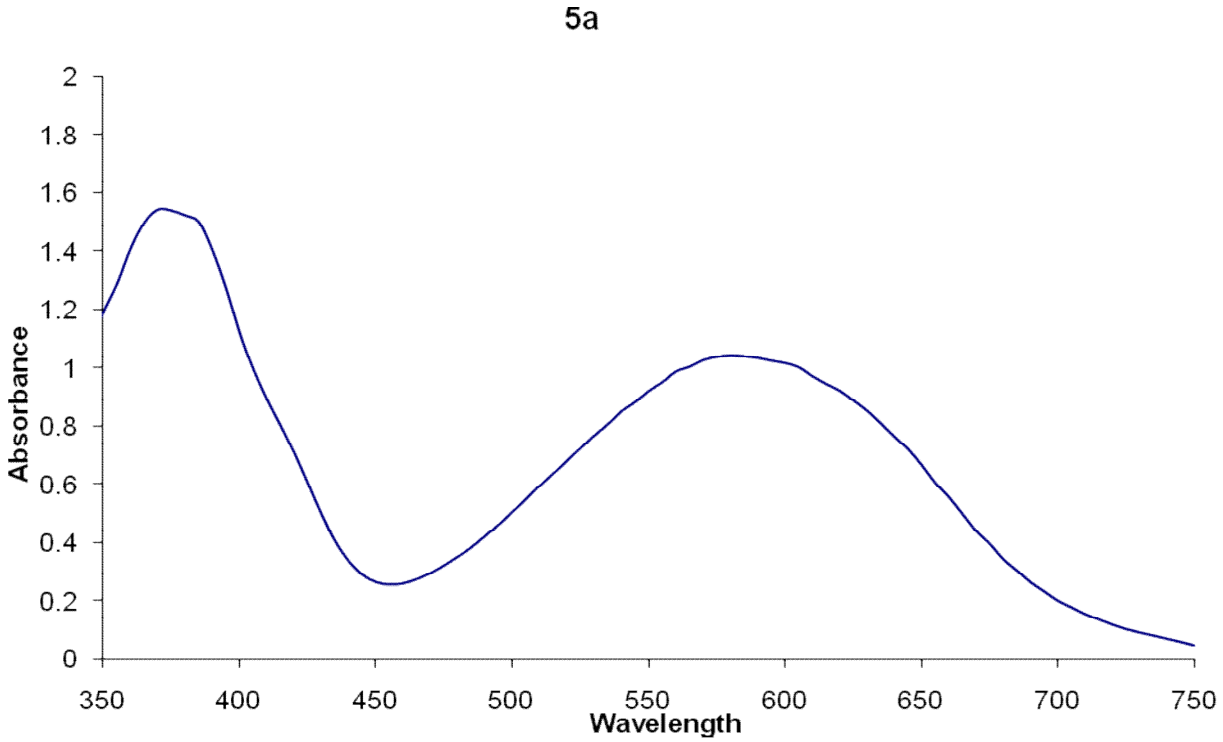


General method for the intramolecular capture of the merocyanine form of a naphthopyran

A solution of the 5-[1,1-bis(4-methoxyphenyl)-1-hydroxymethyl]-2*H*-naphtho[1,2-*b*]pyran (0.75 mmol) in toluene (30 mL) containing a catalytic amount of 4-TsOH 20 mg (0.1 mmol) was heated until TLC examination of the reaction mixture indicated that no naphthopyran remained (see individual examples for temperature and time). The cooled solution was diluted with EtOAc (20 mL) and the mixture washed with water (2 × 30 mL). Removal of the dried (Na_2SO_4) solvent

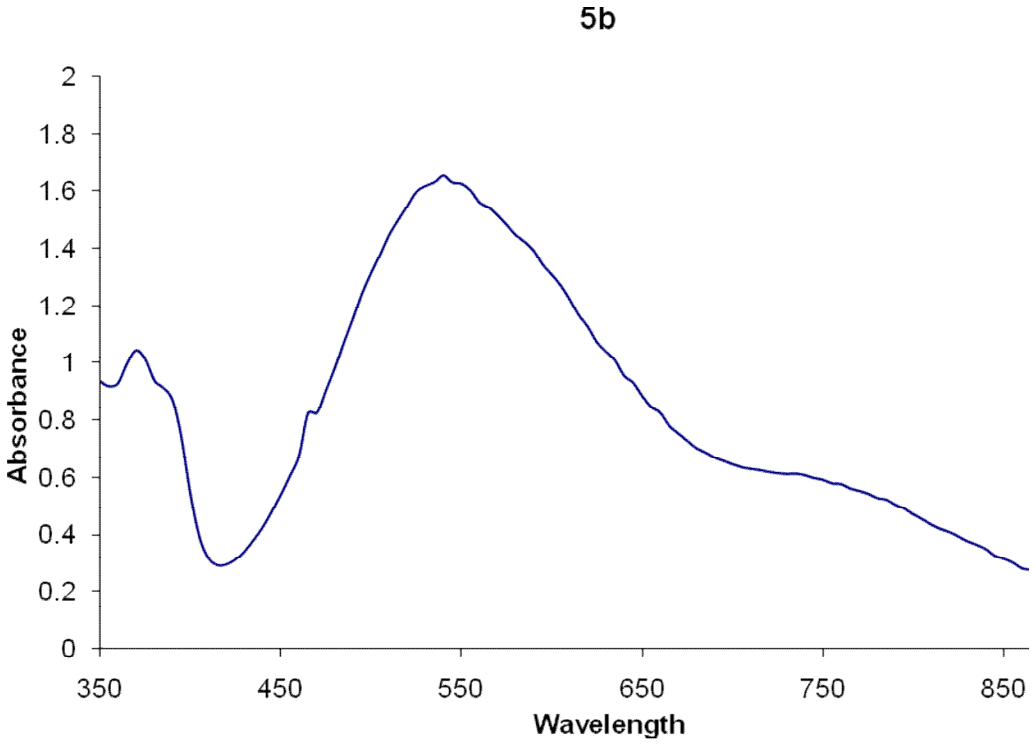
gave a deep purple solid that was eluted from silica. The following compounds were obtained in this fashion:

1. From heating **3a** at 60 ^{o}C for 20 h. 3,7-Dimethoxy-11,11,12-tris(4-methoxyphenyl)-11*H*-benzo[5,6]pentaleno[1,2-*b*]naphthalene-5-one **5a** as deep purple microcrystals 0.37 g (76 %) after elution from silica with 5% ethyl acetate in toluene and recrystallisation from acetone and methanol, mp = 252 – 255 ^{o}C, ν_{max} 3002.7, 2949.2, 2831.4, 1583.4, 1505.3, 1491.4, 1244.1, 1222.9, 1175.1, 1030.5, 824.8, 787.2, 551.9 cm^{-1}, λ_{max} = 586 nm, $\varepsilon_{max} = 1.06 \times 10^4$ $mol^{-1}dm^3cm^{-1}$, λ_{max} = 376 nm, $\varepsilon_{max} = 1.59 \times 10^4$ $mol^{-1}dm^3cm^{-1}$ (CH_2Cl_2), δ_H 3.75 (6H, s, OMe), 3.78 (3H, s, OMe), 3.92 (3H, s, OMe), 3.95 (3H, s, OMe), 6.42 (1H, s, 10-H), 6.71 (6H, m, Ar-H), 6.75 (1H, dd, *J* = 8.2, 2.6 Hz, 2-H), 6.95 (3H, m, Ar-H), 7.11 (1H, d, *J* = 8.4, 2.8 Hz, 8-H), 7.19 (5H, m, Ar-H), 7.80 (1H, d, *J* = 2.8 Hz, 6-H), 8.51 (1H, d, *J* = 2.6 Hz, 4-H), δ_C 55.66, 56.13, 56.35, 58.66, 110.15, 113.72, 113.94, 115.54, 116.33, 120.66, 122.28, 122.80, 126.58, 130.17, 130.21, 130.56, 131.93, 131.99, 132.16, 133.21, 136.52, 140.85, 144.10, 150.53, 156.95, 158.65, 159.50, 159.91, 160.34, 160.46, 184.16. Found $[M+H]^+$ = 647.2419 $C_{43}H_{34}O_6$ requires $[M+H]^+$ = 647.2428.

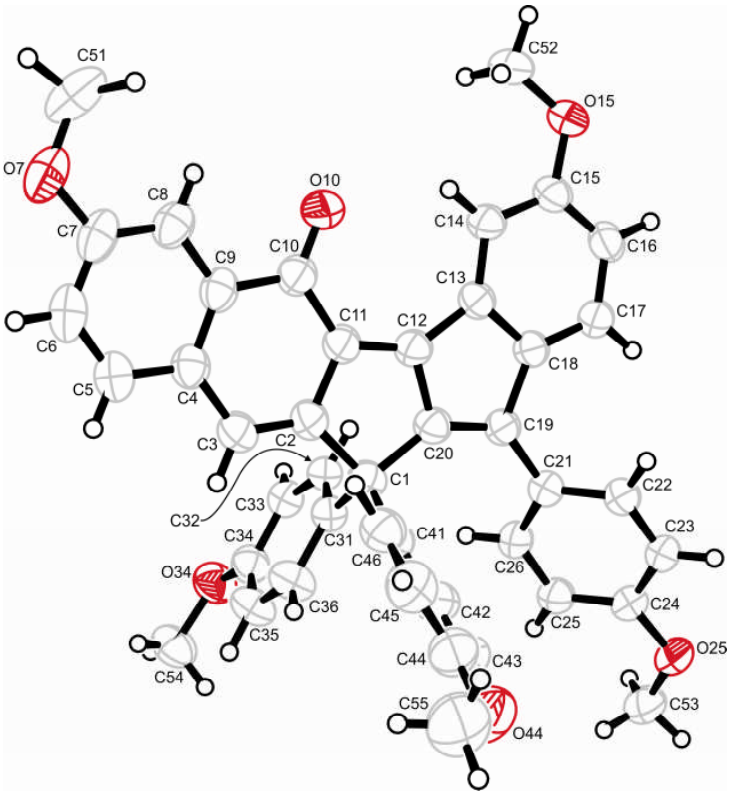


2. From heating **3b** at 100 ^{o}C for 48 h. 3-Dimethylamino-12-(4-dimethylaminophenyl)-7-methoxy-11,11-bis(4-methoxyphenyl)-11*H*-benzo[5,6]pentaleno[1,2-*b*]naphthalene-5-one **5b** as deep purple microcrystals 0.29 g (58 %) after elution from silica with 10% ethyl acetate, 10% hexane in toluene and recrystallisation from acetone and methanol, mp = 266 – 269 ^{o}C, ν_{max} 2901.7, 2833.8, 1600.2, 1586.6, 1504.9, 1489.4, 1243.8, 1430.8, 1284.1, 1243.8, 1178.1, 1033.1, 832.9, 817.1, 801.2 cm^{-1}, λ_{max} = 545 nm, ε_{max} = 1.61 × 10^{4} mol^{-1}dm^{3}cm^{-1}, λ_{max} = 370 nm, ε_{max} = 1.04 × 10^{4} mol^{-1}dm^{3}cm^{-1} (CH_2Cl_2), δ_H 2.95 (6H, s, NMe_2), 3.12 (6H, s, NMe_2), 3.75 (6H, s, OMe), 3.92 (3H, s, OMe), 6.41 (1H, s, 10-H), 6.45 (1H, dd, *J* = 8.5, 2.5 Hz, 2-H), 6.49 (2H, m, Ar-H), 6.70 (4H, m, Ar-H), 7.01 (2H, m, Ar-H), 7.05 (1H, d, *J* = 8.5 Hz, 1-H), 7.08 (1H, dd, *J* = 8.5, 2.5 Hz, 8-H), 7.21 (1H, d, *J* = 8.5 Hz, 9-H), 7.25 (4H, m, Ar-H), 7.82 (1H, d, *J* = 2.5 Hz, 6-H), 8.53 (1H, d, *J* = 2.5 Hz, 4-H), δ_C 40.24, 41.01, 55.21, 55.69, 58.44, 109.32, 111.42, 111.61,

113.17, 115.28, 118.64, 121.59, 121.99, 123.19, 129.54, 129.79, 129.88, 130.11, 131.92, 132.38, 132.97, 136.52, 138.16, 143.33, 147.46, 150.23, 150.81, 157.49, 158.04, 158.69, 162.82, 183.11. Found $[M+H]^+$ = 673.3054 $C_{45}H_{40}N_2O_4$ requires $[M+H]^+$ = 673.3061.



Crystal data and structure refinement for 5a



View of **5a**. X-ray structure with thermal ellipsoids scaled at the 50% probability level

Table 1. Crystal data and structure refinement for **5a**.

Formula	$C_{43}H_{34}O_6$
Formula weight	646.7
Size	0.38 x 0.29 x 0.21 mm
Crystal morphology	Red block
Temperature	150(2) K
Wavelength	0.71073 Å [Mo-K_α]
Crystal system	Monoclinic
Space group	*C*2/*c*
Unit cell dimensions	a = 23.5830(17) Å $\alpha = 90°$
	b = 16.0599(13) Å $\beta = 96.983(3)°$
	c = 17.8227(15) Å $\gamma = 90°$
Volume	6700.1(9) $Å^3$
Z	8
Density (calculated)	1.282 Mg/m^3
Absorption coefficient	0.085 mm^{-1}
F(000)	2720
Data collection range	$1.98 \le \theta \le 27.45°$
Index ranges	$-30 \le h \le 25$, $-20 \le k \le 20$, $-23 \le l \le 23$
Reflections collected	51816
Independent reflections	7460 [R(int) = 0.0785]
Observed reflections	3901 [$I > 2\sigma(I)$]
Absorption correction	none
Refinement method	Full
Data / restraints / parameters	7460 / 0 / 447
Goodness of fit	1.033
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0611, wR_2 = 0.1379
R indices (all data)	R_1 = 0.1310, wR_2 = 0.1930
Largest diff. peak and hole	0.320 and -0.235e.$Å^{-3}$

Table 2. Atomic co-ordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^4) with standard uncertainties (s.u.s) in parentheses. U_{eq} is defined as $^1/_3$ of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	670.6(12)	7757.9(17)	-596.3(15)	492(7)
C(2)	342.7(12)	8549.2(17)	-931.7(15)	500(7)
C(3)	-119.5(13)	8592.3(18)	-1458.0(16)	536(7)
C(4)	-364.8(13)	9403.0(19)	-1693.6(16)	530(7)
C(5)	-859.0(14)	9474(2)	-2212.8(17)	622(8)
C(6)	-1088.0(15)	10240(2)	-2434.2(17)	677(9)
O(7)	-1079.8(11)	11706.1(16)	-2406.5(13)	763(7)
C(7)	-825.0(15)	10969(2)	-2142.9(17)	632(9)
C(8)	-342.9(14)	10928(2)	-1627.2(17)	577(8)
C(9)	-113.2(13)	10149.4(18)	-1397.1(15)	517(7)
O(10)	656.3(10)	10769.6(13)	-592.1(13)	691(6)
C(10)	403.6(14)	10131.0(19)	-838.3(16)	542(7)
C(11)	612.9(13)	9294.6(17)	-597.6(15)	497(7)
C(12)	1062.4(12)	9082.6(16)	-74.3(15)	483(7)
C(13)	1497.1(12)	9460.0(17)	472.0(15)	469(7)
C(14)	1628.7(13)	10289.1(17)	644.8(15)	500(7)
O(15)	2290.6(10)	11243.4(12)	1373.8(11)	638(6)
C(15)	2105.5(14)	10447.5(17)	1173.0(16)	527(7)
C(16)	2424.6(13)	9797.9(18)	1528.2(15)	525(7)
C(17)	2283.1(12)	8969.5(17)	1351.8(15)	481(7)
C(18)	1820.2(12)	8793.2(16)	819.2(14)	458(7)
C(19)	1591.9(12)	7988.3(16)	483.7(14)	454(6)
C(20)	1140.6(12)	8182.0(16)	-42.9(15)	477(7)
C(21)	1851.1(12)	7176.6(16)	722.3(15)	466(7)
C(22)	1996.4(13)	6995.4(17)	1494.3(15)	527(7)
C(23)	2229.2(14)	6242.7(17)	1736.4(16)	551(8)
C(24)	2337.3(13)	5638.1(17)	1215.3(16)	522(7)
O(25)	2560.4(10)	4905.5(12)	1521.8(11)	619(6)
C(25)	2211.5(13)	5801.2(17)	443.8(16)	505(7)
C(26)	1972.5(13)	6563.1(17)	207.6(16)	503(7)
C(31)	903.3(12)	7319.9(16)	-1263.0(15)	472(7)
C(32)	1377.1(13)	7636.8(17)	-1562.1(15)	501(7)
C(33)	1555.5(13)	7311.7(17)	-2215.7(16)	516(7)
O(34)	1472.4(10)	6367.6(13)	-3229.1(12)	650(6)
C(34)	1261.7(13)	6647.6(18)	-2583.0(16)	537(7)
C(35)	796.7(14)	6317(2)	-2293.9(18)	632(8)
C(36)	616.3(14)	6657.5(19)	-1640.6(18)	615(8)

Table 2. (continued)

C(41)	296.9(13)	7199.9(18)	-155.2(16)	525(7)
C(42)	488.7(14)	6405.5(19)	84.8(18)	627(8)
C(43)	172.3(16)	5900(2)	506(2)	732(10)
O(44)	-630.4(13)	5600.2(19)	1105.9(18)	1039(9)
C(44)	-345.5(17)	6169(2)	704(2)	746(10)
C(45)	-540.5(16)	6956(2)	488.8(19)	727(10)
C(46)	-213.7(15)	7462(2)	65.0(18)	627(8)
C(51)	-786.1(18)	12462(3)	-2156(3)	967(14)
C(52)	1984.6(17)	11920.4(19)	991.2(19)	749(10)
C(53)	2704.5(17)	4270.4(19)	1022.8(19)	701(9)
C(54)	1150.3(16)	5732(2)	-3648.2(19)	719(10)
C(55)	-1208(2)	5753(3)	1172(3)	1096(15)

Table 3. Anisotropic displacement parameters ($Å^2$ x 10^3). The anisotropic displacement factor exponent takes the form:

$$-2\pi^2[h^2a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}]$$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	53.9(18)	44.9(15)	47.9(15)	-6.2(12)	2.9(13)	-0.7(13)
C(2)	55.3(18)	50.2(16)	43.9(15)	-3.4(13)	3.1(13)	0.7(13)
C(3)	58.9(19)	52.5(17)	48.9(16)	-6.8(13)	4.6(14)	-0.2(14)
C(4)	54.8(19)	60.6(19)	43.7(15)	-1.0(14)	6.2(13)	5.2(14)
C(5)	59(2)	77(2)	49.1(17)	-0.2(16)	2.0(15)	2.8(16)
C(6)	61(2)	96(3)	44.9(17)	7.8(18)	1.3(15)	13.2(19)
O(7)	86.8(17)	80.0(17)	61.8(14)	21.5(12)	8.5(12)	23.7(14)
C(7)	68(2)	77(2)	47.3(17)	16.3(16)	13.9(16)	20.9(18)
C(8)	64(2)	60.9(19)	49.7(17)	6.4(14)	12.0(15)	11.6(15)
C(9)	55.1(18)	59.4(18)	41.3(15)	3.7(13)	8.6(13)	9.2(14)
O(10)	81.1(17)	46.2(12)	74.4(15)	-4.5(11)	-13.8(12)	6.1(11)
C(10)	65(2)	51.0(17)	46.2(16)	-2.2(14)	5.7(14)	8.3(15)
C(11)	56.9(19)	47.4(16)	44.2(15)	-3.1(13)	4.4(13)	4.8(13)
C(12)	57.0(18)	43.1(15)	44.9(15)	-1.4(12)	6.6(13)	2.3(13)
C(13)	54.6(18)	46.1(15)	40.4(14)	-4.8(12)	7.2(12)	1.7(13)
C(14)	63.0(19)	40.9(15)	46.7(16)	-3.9(12)	8.4(14)	2.9(13)
O(15)	87.8(16)	45.8(12)	55.1(12)	-7.9(10)	-2.6(11)	-7.2(11)
C(15)	69(2)	45.1(16)	44.4(15)	-7.4(13)	8.1(14)	-3.9(14)
C(16)	58.2(19)	56.6(18)	41.6(15)	-5.2(13)	1.8(13)	-5.2(14)
C(17)	54.5(18)	45.7(15)	43.6(15)	1.1(12)	4.5(13)	2.2(13)
C(18)	55.5(18)	43.9(15)	38.5(14)	-1.1(12)	7.1(12)	-0.6(12)
C(19)	56.4(18)	41.8(15)	38.3(14)	-1.8(11)	7.0(12)	1.1(12)
C(20)	57.1(18)	42.5(15)	44.1(15)	-2.3(12)	8.1(13)	-0.3(13)
C(21)	51.8(17)	44.5(15)	43.1(15)	1.7(12)	3.7(12)	-2.1(12)
C(22)	70(2)	45.8(16)	42.0(15)	-2.3(13)	4.8(13)	-2.9(14)
C(23)	75(2)	47.5(17)	41.0(15)	5.8(13)	1.6(14)	-6.6(14)
C(24)	63(2)	42.0(15)	50.4(17)	6.0(13)	1.6(14)	-2.2(13)
O(25)	85.2(16)	42.4(11)	56.1(12)	8.5(9)	0.5(11)	3.9(10)
C(25)	61.0(19)	43.4(16)	47.3(16)	-1.3(12)	6.8(13)	0.0(13)
C(26)	60.8(19)	48.5(16)	41.1(15)	2.5(12)	3.6(13)	0.1(13)
C(31)	52.6(18)	41.2(15)	46.1(15)	-1.0(12)	-0.7(13)	1.3(12)
C(32)	61.2(19)	39.5(15)	47.0(16)	-0.8(12)	-3.8(13)	-0.3(13)
C(33)	58.1(19)	48.8(16)	47.3(16)	2.8(13)	3.4(13)	-0.9(13)
O(34)	76.1(15)	61.3(13)	58.5(13)	-14.6(10)	11.8(11)	-0.4(11)
C(34)	61(2)	51.3(17)	48.3(16)	-6.0(13)	5.0(14)	5.9(14)

Table 3. (continued)

C(35)	68(2)	57.3(19)	64(2)	-19.6(16)	7.2(16)	-7.7(16)
C(36)	62(2)	58.6(19)	65(2)	-14.0(16)	12.8(16)	-10.6(15)
C(41)	61(2)	48.5(16)	47.6(16)	-7.9(13)	3.1(14)	-3.3(14)
C(42)	61(2)	55.3(19)	71(2)	0.7(16)	5.0(16)	-3.7(15)
C(43)	78(3)	60(2)	80(2)	12.9(18)	5(2)	-7.3(18)
O(44)	88(2)	108(2)	119(2)	31.5(18)	26.6(18)	-19.5(16)
C(44)	78(3)	77(2)	69(2)	10.1(19)	10.4(19)	-17(2)
C(45)	72(2)	80(2)	68(2)	-3.1(19)	17.8(18)	-6.1(19)
C(46)	73(2)	56.9(19)	58.8(19)	-5.7(15)	8.9(16)	-0.4(16)
C(51)	90(3)	79(3)	125(4)	47(3)	28(3)	19(2)
C(52)	116(3)	41.1(17)	65(2)	-7.4(15)	0(2)	-7.6(18)
C(53)	96(3)	45.5(17)	68(2)	4.0(16)	4.2(19)	8.8(17)
C(54)	80(2)	68(2)	67(2)	-25.3(17)	7.3(18)	-0.7(18)
C(55)	104(4)	129(4)	101(3)	16(3)	32(3)	-24(3)

Table 4. Hydrogen atom co-ordinates (x 10^3) and isotropic displacement parameters ($Å^2$ x 10^2) with s.u.s in parentheses.

	x	y	z	U_{eq}
H(3)	-286.	8096.	-1676.	64.
H(5)	-1041.	8982.	-2418.	75.
H(6)	-1425.	10271.	-2785.	81.
H(8)	-166.	11425.	-1427.	69.
H(14)	1403.	10730.	413.	60.
H(16)	2742.	9919.	1894.	63.
H(17)	2503.	8530.	1596.	58.
H(22)	1932.	7405.	1859.	63.
H(23)	2316.	6136.	2262.	66.
H(25)	2289.	5395.	83.	61.
H(26)	1889.	6670.	-318.	60.
H(32)	1583.	8086.	-1313.	60.
H(33)	1878.	7542.	-2412.	62.
H(35)	598.	5858.	-2537.	76.
H(36)	290.	6431.	-1451.	74.
H(42)	844.	6210.	-45.	75.
H(43)	312.	5365.	659.	88.
H(45)	-893.	7152.	627.	87.
H(46)	-349.	8004.	-75.	75.
H(51a)	-759.	12500.	-1604.	145.
H(51b)	-998.	12942.	-2383.	145.
H(51c)	-401.	12457.	-2310.	145.
H(52a)	2005.	11876.	447.	112.
H(52b)	2155.	12449.	1179.	112.
H(52c)	1584.	11900.	1086.	112.
H(53a)	2365.	4116.	679.	105.
H(53b)	2847.	3782.	1316.	105.
H(53c)	3001.	4476.	729.	105.
H(54a)	1144.	5231.	-3335.	108.
H(54b)	1327.	5601.	-4104.	108.
H(54c)	759.	5927.	-3793.	108.
H(55a)	-1239.	6241.	1496.	164.
H(55b)	-1373.	5267.	1397.	164.
H(55c)	-1414.	5860.	670.	164.

Table 5. Interatomic distances (Å) with s.u.s in parentheses.

C(1)-C(41)	1.538(4)	C(1)-C(31)	1.538(4)
C(1)-C(20)	1.549(4)	C(1)-C(2)	1.568(4)
C(2)-C(3)	1.351(4)	C(2)-C(11)	1.450(4)
C(3)-C(4)	1.465(4)	C(4)-C(5)	1.402(4)
C(4)-C(9)	1.411(4)	C(5)-C(6)	1.382(5)
C(6)-C(7)	1.395(5)	O(7)-C(7)	1.383(4)
O(7)-C(51)	1.441(5)	C(7)-C(8)	1.374(4)
C(8)-C(9)	1.404(4)	C(9)-C(10)	1.478(4)
O(10)-C(10)	1.239(3)	C(10)-C(11)	1.477(4)
C(11)-C(12)	1.367(4)	C(12)-C(13)	1.457(4)
C(12)-C(20)	1.458(4)	C(13)-C(14)	1.393(4)
C(13)-C(18)	1.413(4)	C(14)-C(15)	1.399(4)
O(15)-C(15)	1.384(3)	O(15)-C(52)	1.431(4)
C(15)-C(16)	1.393(4)	C(16)-C(17)	1.398(4)
C(17)-C(18)	1.386(4)	C(18)-C(19)	1.497(4)
C(19)-C(20)	1.367(4)	C(19)-C(21)	1.480(4)
C(21)-C(26)	1.399(4)	C(21)-C(22)	1.407(4)
C(22)-C(23)	1.375(4)	C(23)-C(24)	1.389(4)
C(24)-O(25)	1.375(3)	C(24)-C(25)	1.396(4)
O(25)-C(53)	1.421(4)	C(25)-C(26)	1.391(4)
C(31)-C(36)	1.390(4)	C(31)-C(32)	1.392(4)
C(32)-C(33)	1.388(4)	C(33)-C(34)	1.391(4)
O(34)-C(34)	1.384(3)	O(34)-C(54)	1.428(4)
C(34)-C(35)	1.374(4)	C(35)-C(36)	1.398(4)
C(41)-C(46)	1.377(4)	C(41)-C(42)	1.403(4)
C(42)-C(43)	1.383(5)	C(43)-C(44)	1.381(5)
O(44)-C(44)	1.385(4)	O(44)-C(55)	1.403(5)
C(44)-C(45)	1.382(5)	C(45)-C(46)	1.402(5)

Table 6. Angles between interatomic vectors (°) with s.u.s in parentheses.

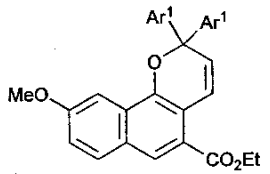
C(41)-C(1)-C(31)	114.1(2)	C(41)-C(1)-C(20)	109.9(2)
C(31)-C(1)-C(20)	113.5(2)	C(41)-C(1)-C(2)	112.4(2)
C(31)-C(1)-C(2)	106.4(2)	C(20)-C(1)-C(2)	99.7(2)
C(3)-C(2)-C(11)	121.4(3)	C(3)-C(2)-C(1)	128.7(3)
C(11)-C(2)-C(1)	109.9(2)	C(2)-C(3)-C(4)	120.1(3)
C(5)-C(4)-C(9)	117.1(3)	C(5)-C(4)-C(3)	121.9(3)
C(9)-C(4)-C(3)	120.9(3)	C(6)-C(5)-C(4)	121.7(3)
C(5)-C(6)-C(7)	120.0(3)	C(7)-O(7)-C(51)	116.3(3)
C(8)-C(7)-O(7)	123.9(3)	C(8)-C(7)-C(6)	120.2(3)
O(7)-C(7)-C(6)	115.9(3)	C(7)-C(8)-C(9)	119.8(3)
C(8)-C(9)-C(4)	121.2(3)	C(8)-C(9)-C(10)	118.2(3)
C(4)-C(9)-C(10)	120.7(3)	O(10)-C(10)-C(11)	121.4(3)
O(10)-C(10)-C(9)	122.9(3)	C(11)-C(10)-C(9)	115.7(3)
C(12)-C(11)-C(2)	109.9(2)	C(12)-C(11)-C(10)	129.0(3)
C(2)-C(11)-C(10)	121.1(3)	C(11)-C(12)-C(13)	140.9(3)
C(11)-C(12)-C(20)	110.9(2)	C(13)-C(12)-C(20)	108.1(2)
C(14)-C(13)-C(18)	122.3(3)	C(14)-C(13)-C(12)	131.7(3)
C(18)-C(13)-C(12)	106.0(2)	C(13)-C(14)-C(15)	117.5(3)
C(15)-O(15)-C(52)	117.0(2)	O(15)-C(15)-C(16)	116.0(3)
O(15)-C(15)-C(14)	123.0(3)	C(16)-C(15)-C(14)	121.0(3)
C(15)-C(16)-C(17)	120.6(3)	C(18)-C(17)-C(16)	119.7(3)
C(17)-C(18)-C(13)	118.9(2)	C(17)-C(18)-C(19)	131.7(3)
C(13)-C(18)-C(19)	109.3(2)	C(20)-C(19)-C(21)	131.1(2)
C(20)-C(19)-C(18)	106.9(2)	C(21)-C(19)-C(18)	122.0(2)
C(19)-C(20)-C(12)	109.7(2)	C(19)-C(20)-C(1)	140.8(2)
C(12)-C(20)-C(1)	109.5(2)	C(26)-C(21)-C(22)	116.8(2)
C(26)-C(21)-C(19)	122.8(2)	C(22)-C(21)-C(19)	120.4(2)
C(23)-C(22)-C(21)	121.9(3)	C(22)-C(23)-C(24)	120.2(3)
O(25)-C(24)-C(23)	115.2(2)	O(25)-C(24)-C(25)	125.3(3)
C(23)-C(24)-C(25)	119.6(3)	C(24)-O(25)-C(53)	118.3(2)
C(26)-C(25)-C(24)	119.5(3)	C(25)-C(26)-C(21)	121.9(3)
C(36)-C(31)-C(32)	117.4(3)	C(36)-C(31)-C(1)	121.7(3)
C(32)-C(31)-C(1)	120.5(2)	C(33)-C(32)-C(31)	121.6(3)
C(32)-C(33)-C(34)	119.9(3)	C(34)-O(34)-C(54)	116.5(2)
C(35)-C(34)-O(34)	124.8(3)	C(35)-C(34)-C(33)	119.6(3)
O(34)-C(34)-C(33)	115.6(3)	C(34)-C(35)-C(36)	119.9(3)
C(31)-C(36)-C(35)	121.5(3)	C(46)-C(41)-C(42)	116.8(3)
C(46)-C(41)-C(1)	122.9(3)	C(42)-C(41)-C(1)	120.1(3)
C(43)-C(42)-C(41)	121.6(3)	C(44)-C(43)-C(42)	120.5(3)
C(44)-O(44)-C(55)	117.7(3)	C(43)-C(44)-C(45)	119.3(3)
C(43)-C(44)-O(44)	115.2(3)	C(45)-C(44)-O(44)	125.5(4)
C(44)-C(45)-C(46)	119.5(3)	C(41)-C(46)-C(45)	122.2(3)

Table 7. Torsion angles (°) with s.u.s in parentheses.

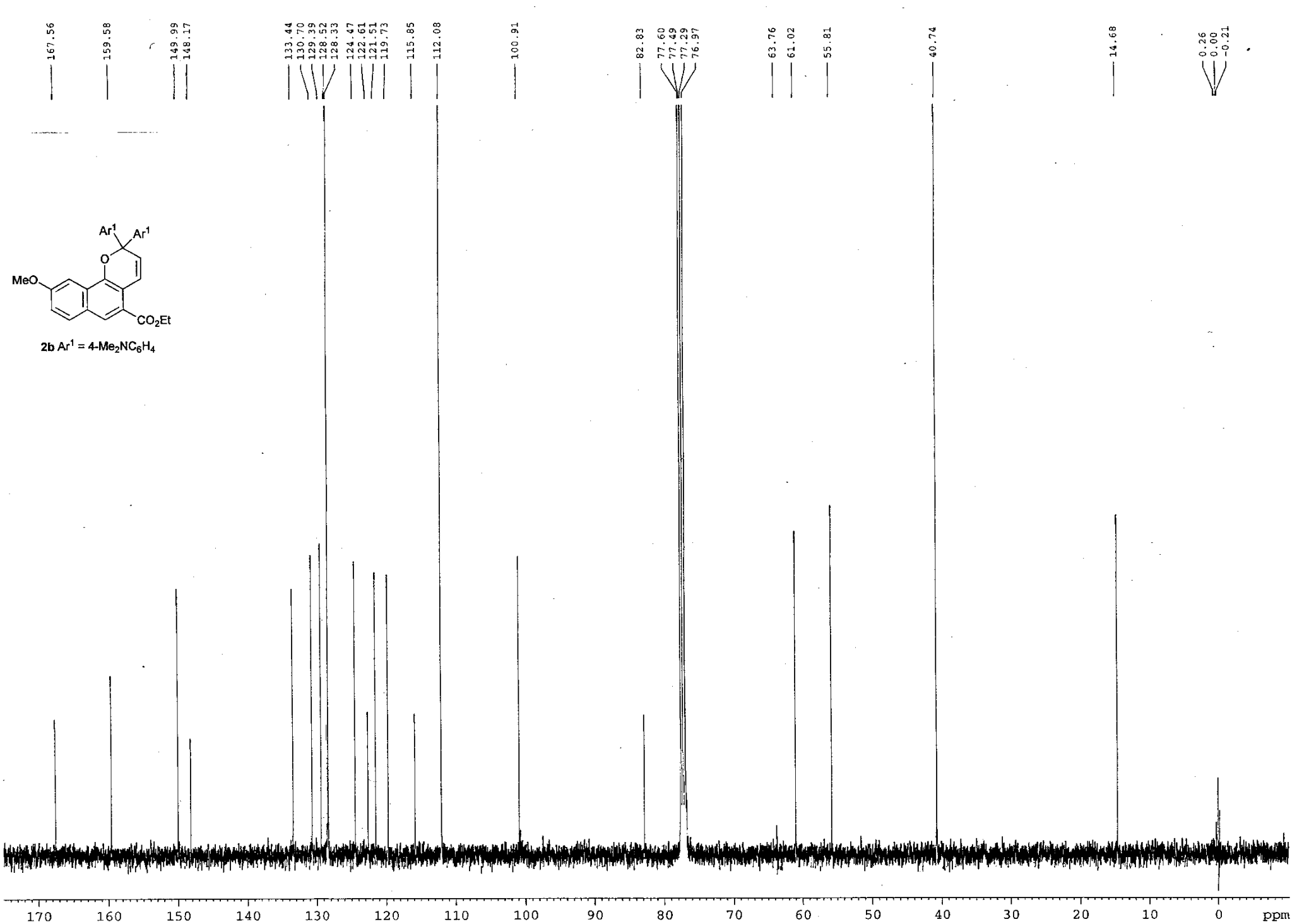
C(41)-C(1)-C(2)-C(3)	-63.9(4)	C(31)-C(1)-C(2)-C(3)	61.6(4)
C(20)-C(1)-C(2)-C(3)	179.7(3)	C(41)-C(1)-C(2)-C(11)	116.8(3)
C(31)-C(1)-C(2)-C(11)	-117.7(2)	C(20)-C(1)-C(2)-C(11)	0.5(3)
C(11)-C(2)-C(3)-C(4)	-1.1(4)	C(1)-C(2)-C(3)-C(4)	179.7(3)
C(2)-C(3)-C(4)-C(5)	-177.1(3)	C(2)-C(3)-C(4)-C(9)	2.2(4)
C(9)-C(4)-C(5)-C(6)	0.8(4)	C(3)-C(4)-C(5)-C(6)	-179.9(3)
C(4)-C(5)-C(6)-C(7)	0.3(5)	C(51)-O(7)-C(7)-C(8)	4.8(4)
C(51)-O(7)-C(7)-C(6)	-175.2(3)	C(5)-C(6)-C(7)-C(8)	-1.0(5)
C(5)-C(6)-C(7)-O(7)	179.0(3)	O(7)-C(7)-C(8)-C(9)	-179.6(3)
C(6)-C(7)-C(8)-C(9)	0.4(4)	C(7)-C(8)-C(9)-C(4)	0.8(4)
C(7)-C(8)-C(9)-C(10)	-179.6(3)	C(5)-C(4)-C(9)-C(8)	-1.4(4)
C(3)-C(4)-C(9)-C(8)	179.3(3)	C(5)-C(4)-C(9)-C(10)	179.1(3)
C(3)-C(4)-C(9)-C(10)	-0.2(4)	C(8)-C(9)-C(10)-O(10)	-3.4(4)
C(4)-C(9)-C(10)-O(10)	176.1(3)	C(8)-C(9)-C(10)-C(11)	177.9(3)
C(4)-C(9)-C(10)-C(11)	-2.6(4)	C(3)-C(2)-C(11)-C(12)	178.9(3)
C(1)-C(2)-C(11)-C(12)	-1.8(3)	C(3)-C(2)-C(11)-C(10)	-2.0(4)
C(1)-C(2)-C(11)-C(10)	177.4(2)	O(10)-C(10)-C(11)-C(12)	3.9(5)
C(9)-C(10)-C(11)-C(12)	-177.3(3)	O(10)-C(10)-C(11)-C(2)	-175.0(3)
C(9)-C(10)-C(11)-C(2)	3.7(4)	C(2)-C(11)-C(12)-C(13)	-177.6(3)
C(10)-C(11)-C(12)-C(13)	3.3(6)	C(2)-C(11)-C(12)-C(20)	2.4(3)
C(10)-C(11)-C(12)-C(20)	-176.7(3)	C(11)-C(12)-C(13)-C(14)	-3.2(6)
C(20)-C(12)-C(13)-C(14)	176.8(3)	C(11)-C(12)-C(13)-C(18)	179.6(4)
C(20)-C(12)-C(13)-C(18)	-0.4(3)	C(18)-C(13)-C(14)-C(15)	1.1(4)
C(12)-C(13)-C(14)-C(15)	-175.7(3)	C(52)-O(15)-C(15)-C(16)	177.3(3)
C(52)-O(15)-C(15)-C(14)	-2.5(4)	C(13)-C(14)-C(15)-O(15)	177.9(3)
C(13)-C(14)-C(15)-C(16)	-1.9(4)	O(15)-C(15)-C(16)-C(17)	-178.4(3)
C(14)-C(15)-C(16)-C(17)	1.5(4)	C(15)-C(16)-C(17)-C(18)	-0.1(4)
C(16)-C(17)-C(18)-C(13)	-0.7(4)	C(16)-C(17)-C(18)-C(19)	175.6(3)
C(14)-C(13)-C(18)-C(17)	0.2(4)	C(12)-C(13)-C(18)-C(17)	177.7(2)
C(14)-C(13)-C(18)-C(19)	-176.9(2)	C(12)-C(13)-C(18)-C(19)	0.6(3)
C(17)-C(18)-C(19)-C(20)	-177.2(3)	C(13)-C(18)-C(19)-C(20)	-0.6(3)
C(17)-C(18)-C(19)-C(21)	2.1(4)	C(13)-C(18)-C(19)-C(21)	178.7(2)
C(21)-C(19)-C(20)-C(12)	-178.9(3)	C(18)-C(19)-C(20)-C(12)	0.4(3)
C(21)-C(19)-C(20)-C(1)	4.2(6)	C(18)-C(19)-C(20)-C(1)	-176.5(3)
C(11)-C(12)-C(20)-C(19)	180.0(2)	C(13)-C(12)-C(20)-C(19)	0.0(3)
C(11)-C(12)-C(20)-C(1)	-2.1(3)	C(13)-C(12)-C(20)-C(1)	177.9(2)
C(41)-C(1)-C(20)-C(19)	59.6(5)	C(31)-C(1)-C(20)-C(19)	-69.5(4)
C(2)-C(1)-C(20)-C(19)	177.8(3)	C(41)-C(1)-C(20)-C(12)	-117.3(3)
C(31)-C(1)-C(20)-C(12)	113.6(3)	C(2)-C(1)-C(20)-C(12)	0.9(3)
C(20)-C(19)-C(21)-C(26)	45.7(5)	C(18)-C(19)-C(21)-C(26)	-133.5(3)
C(20)-C(19)-C(21)-C(22)	-135.4(3)	C(18)-C(19)-C(21)-C(22)	45.4(4)
C(26)-C(21)-C(22)-C(23)	-2.1(4)	C(19)-C(21)-C(22)-C(23)	178.9(3)
C(21)-C(22)-C(23)-C(24)	1.1(5)	C(22)-C(23)-C(24)-O(25)	-178.9(3)

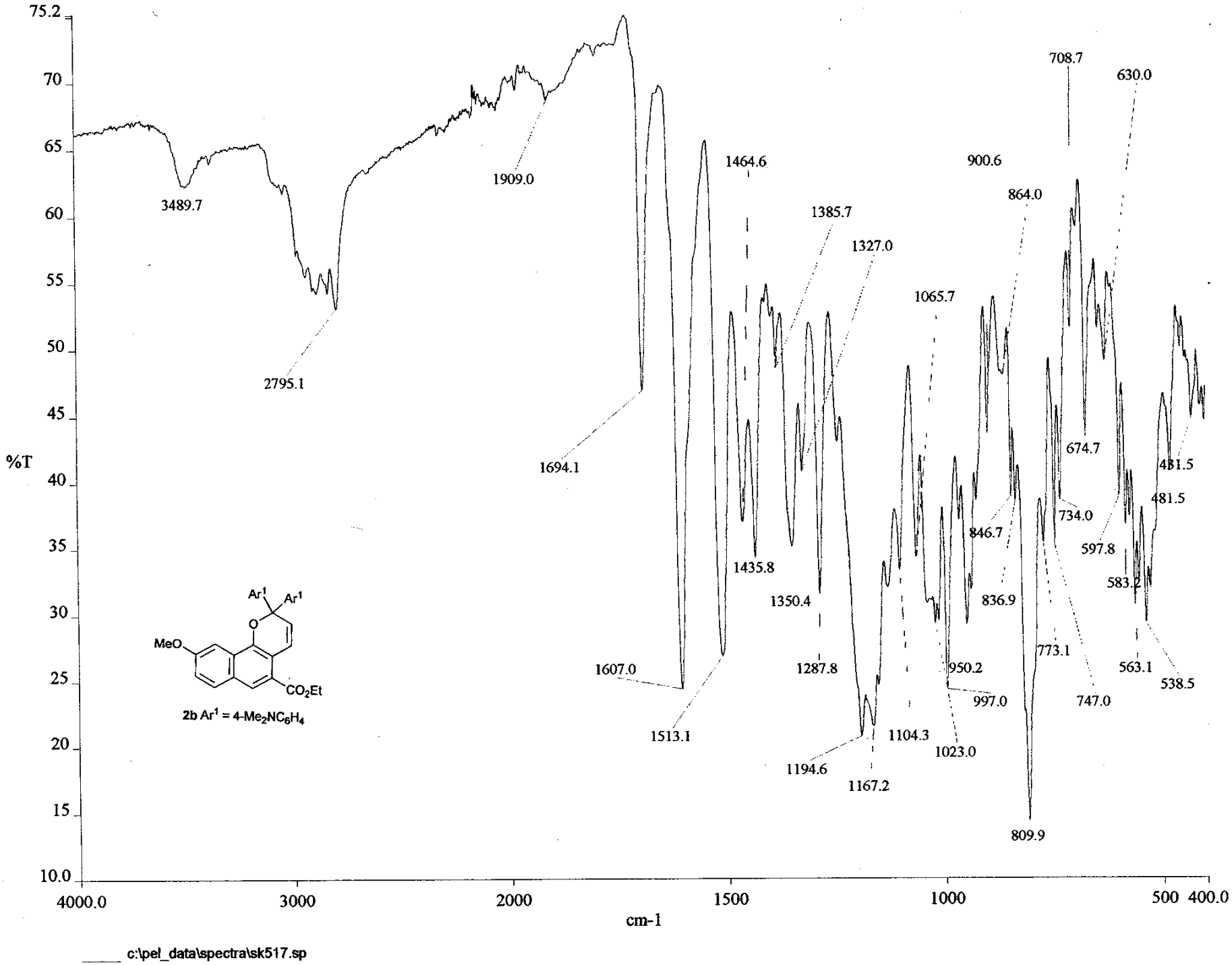
Table 7. (continued)

C(22)-C(23)-C(24)-C(25)	0.4(5)	C(23)-C(24)-O(25)-C(53)	-177.4(3)
C(25)-C(24)-O(25)-C(53)	3.4(4)	O(25)-C(24)-C(25)-C(26)	178.3(3)
C(23)-C(24)-C(25)-C(26)	-0.9(4)	C(24)-C(25)-C(26)-C(21)	-0.2(4)
C(22)-C(21)-C(26)-C(25)	1.6(4)	C(19)-C(21)-C(26)-C(25)	-179.4(3)
C(41)-C(1)-C(31)-C(36)	27.5(4)	C(20)-C(1)-C(31)-C(36)	154.5(3)
C(2)-C(1)-C(31)-C(36)	-97.0(3)	C(41)-C(1)-C(31)-C(32)	-159.8(3)
C(20)-C(1)-C(31)-C(32)	-32.8(4)	C(2)-C(1)-C(31)-C(32)	75.7(3)
C(36)-C(31)-C(32)-C(33)	0.5(4)	C(1)-C(31)-C(32)-C(33)	-172.5(2)
C(31)-C(32)-C(33)-C(34)	-0.7(4)	C(54)-O(34)-C(34)-C(35)	5.1(4)
C(54)-O(34)-C(34)-C(33)	-175.1(3)	C(32)-C(33)-C(34)-C(35)	-0.1(4)
C(32)-C(33)-C(34)-O(34)	-180.0(3)	O(34)-C(34)-C(35)-C(36)	-179.2(3)
C(33)-C(34)-C(35)-C(36)	1.0(5)	C(32)-C(31)-C(36)-C(35)	0.3(5)
C(1)-C(31)-C(36)-C(35)	173.3(3)	C(34)-C(35)-C(36)-C(31)	-1.1(5)
C(31)-C(1)-C(41)-C(46)	-135.3(3)	C(20)-C(1)-C(41)-C(46)	96.0(3)
C(2)-C(1)-C(41)-C(46)	-14.1(4)	C(31)-C(1)-C(41)-C(42)	49.0(4)
C(20)-C(1)-C(41)-C(42)	-79.7(3)	C(2)-C(1)-C(41)-C(42)	170.2(3)
C(46)-C(41)-C(42)-C(43)	1.7(5)	C(1)-C(41)-C(42)-C(43)	177.7(3)
C(41)-C(42)-C(43)-C(44)	-0.2(5)	C(42)-C(43)-C(44)-C(45)	-1.2(6)
C(42)-C(43)-C(44)-O(44)	178.2(3)	C(55)-O(44)-C(44)-C(43)	-165.6(4)
C(55)-O(44)-C(44)-C(45)	13.7(6)	C(43)-C(44)-C(45)-C(46)	0.9(5)
O(44)-C(44)-C(45)-C(46)	-178.4(3)	C(42)-C(41)-C(46)-C(45)	-2.0(5)
C(1)-C(41)-C(46)-C(45)	-177.8(3)	C(44)-C(45)-C(46)-C(41)	0.7(5)



2b $Ar^1 = 4\text{-}Me_2NC_6H_4$

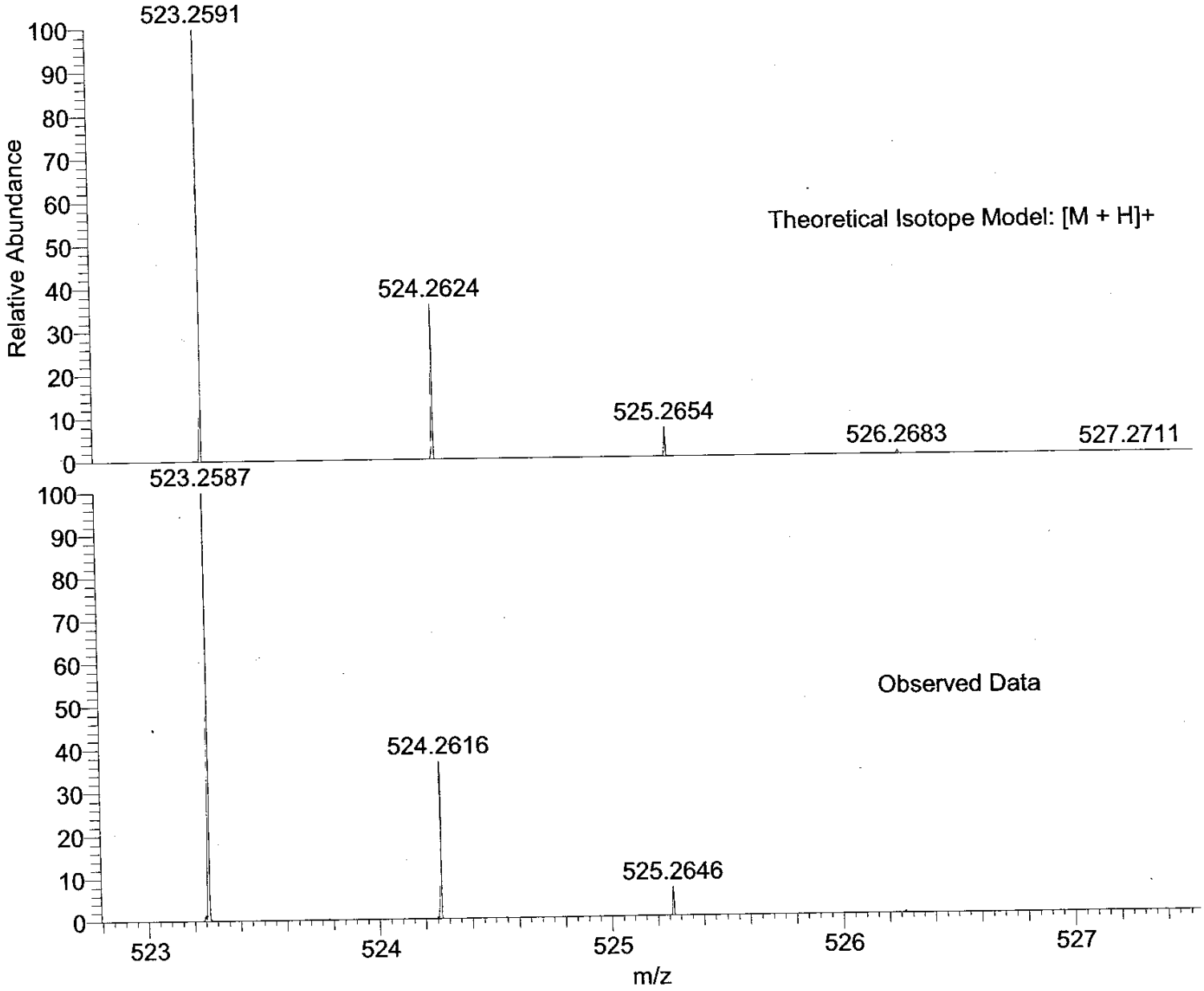




SK517 MW=522?
(DCM)/MeOH + NH4OAc

EPSRC National Centre Swansea
LTQ Orbitrap XL

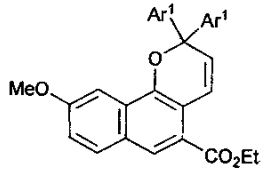
Christopher S. Kershaw
12/02/2010 15:41:52



NL:
1.61E4

$C_{33}H_{34}O_4N_2H$:
$C_{33}H_{35}O_4N_2$
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

NL:
2.59E7
LEEHER113-OM-HNESP#9-14
RT: 0.87-1.19 AV: 5 T: FTMS
+ p NSI Full ms
[120.00-2000.00]

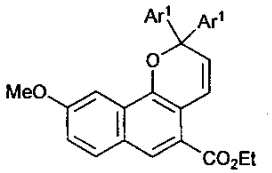


2b $Ar^1 = 4\text{-}Me_2NC_6H_4$

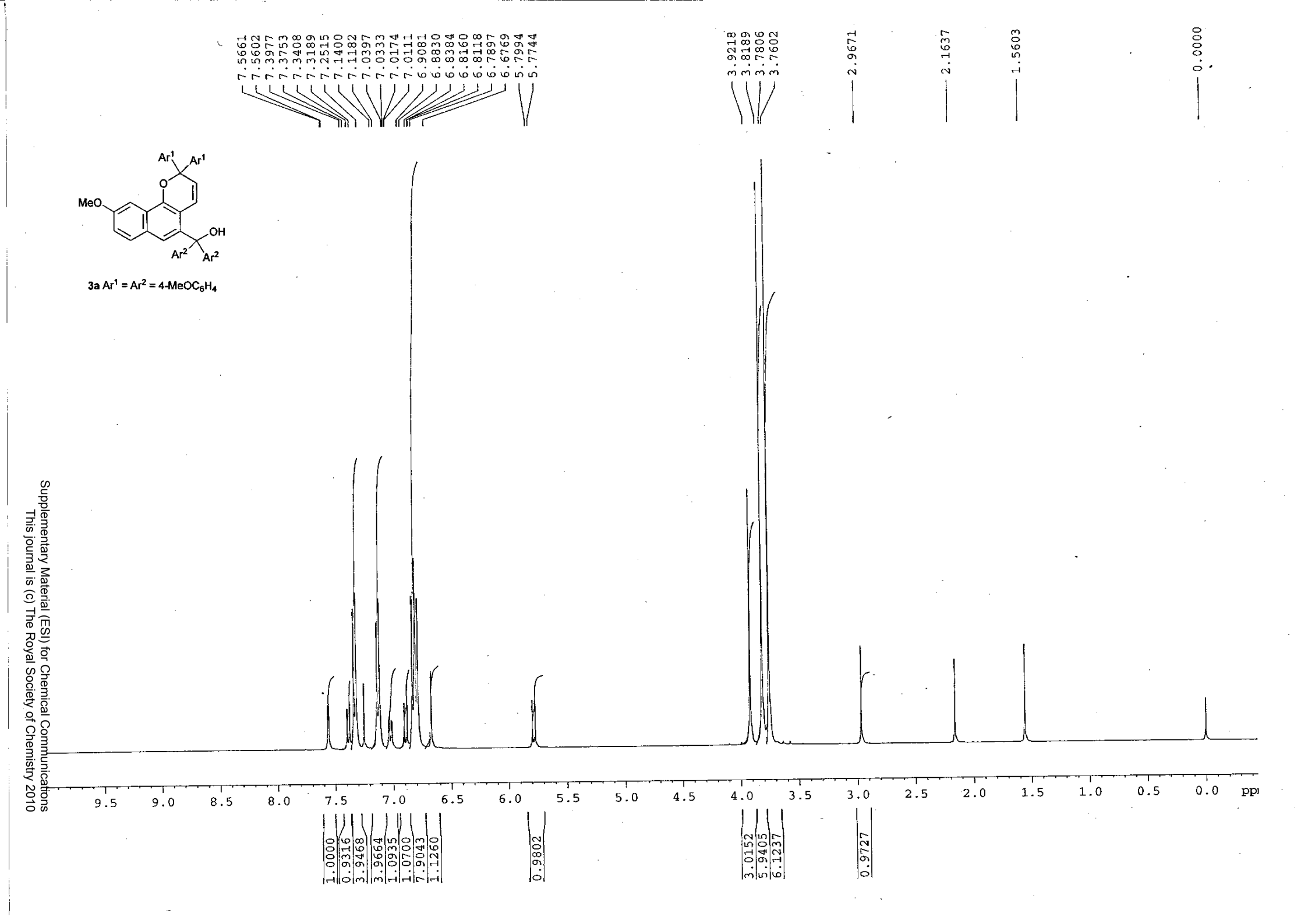
Isotope:	Min. .. Max.
14 N	0....12
16 O	0....14
12 C	0....60
1 H	0....80
23 Na	0....0
Tolerance Window:	+- 5.00 ppm
Db/Ring Equiv:	-2.. 100
Fits:	200

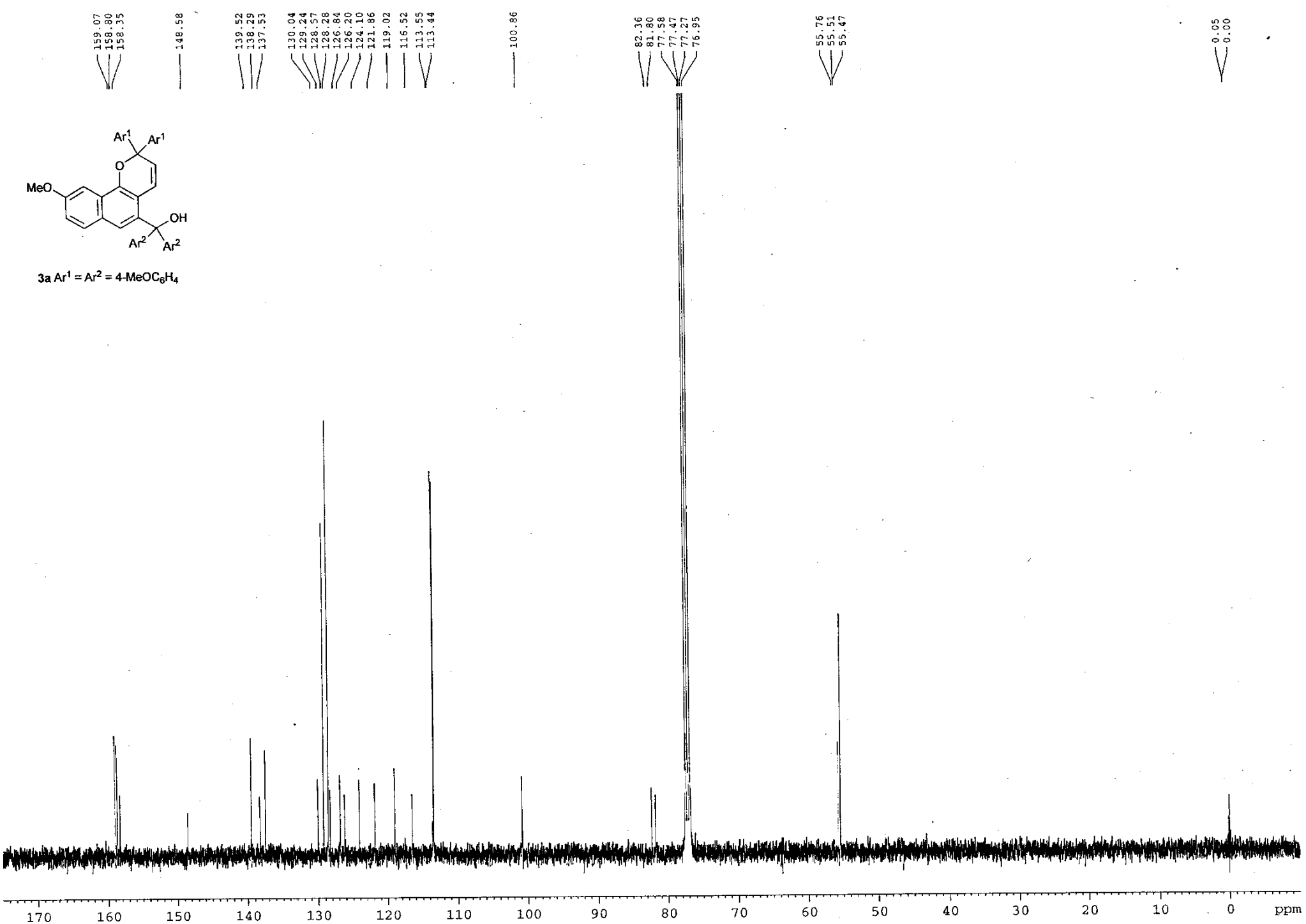
N-Rule: Do not use
Charge: 1

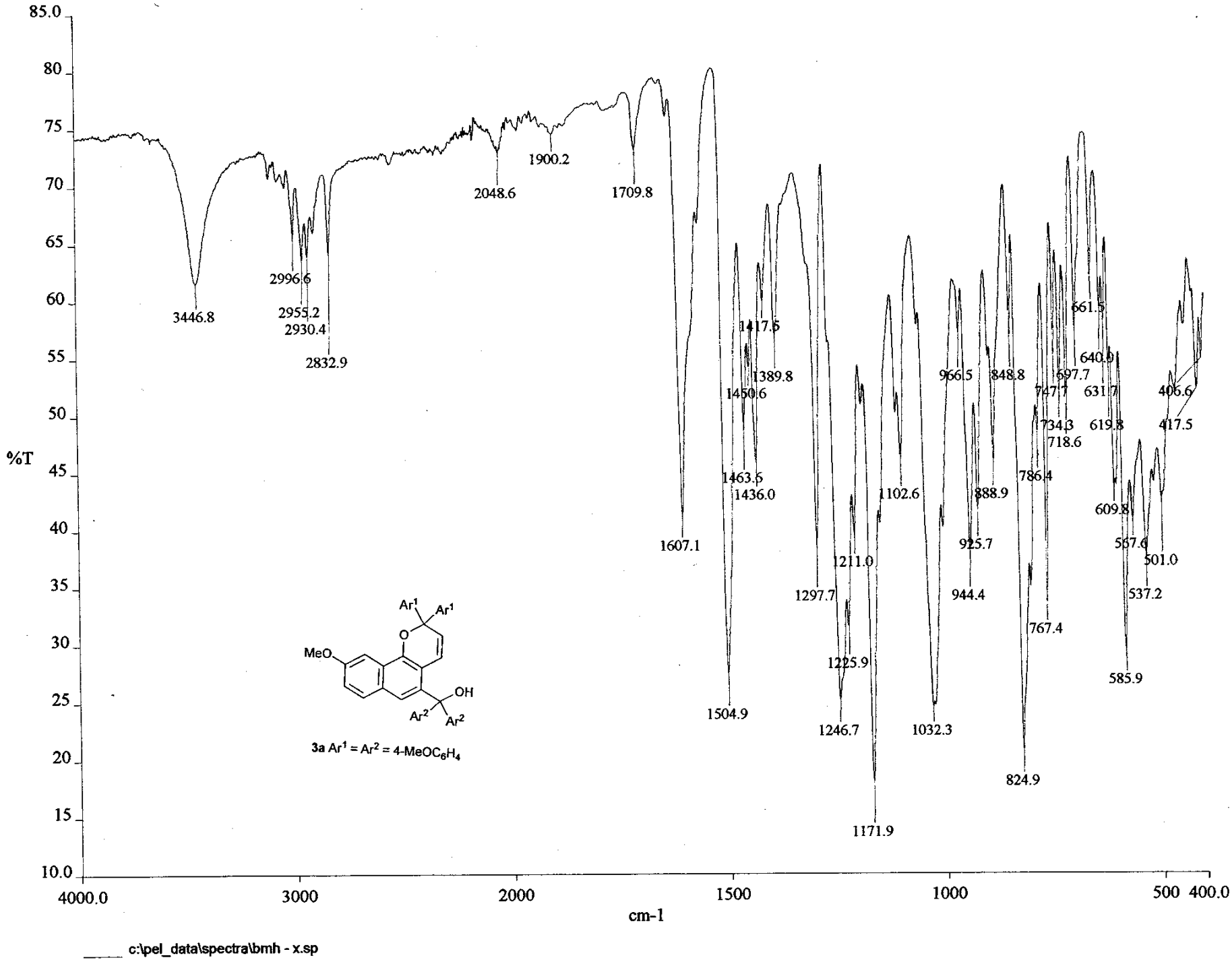
Mass	Theoretical Mass	Delta [ppm]	RDB	Composition
523.2587	523.2583	0.8	0.0	$C_{18}H_{41}O_{14}N_3$
	523.2583	0.8	5.5	$C_{17}H_{35}O_9N_{10}$
	523.2591	-0.8	17.5	$C_{33}H_{35}O_4N_2$
	523.2578	1.7	18.0	$C_{31}H_{33}O_3N_5$
	523.2596	-1.8	5.0	$C_{19}H_{37}O_{10}N_7$
	523.2570	3.3	0.5	$C_{16}H_{39}O_{13}N_6$
	523.2605	-3.4	22.5	$C_{34}H_{31}N_6$
	523.2565	4.3	13.0	$C_{30}H_{37}O_7N_1$
	523.2564	4.3	18.5	$C_{29}H_{31}O_2N_8$
	523.2610	-4.4	10.0	$C_{20}H_{33}O_6N_{11}$
	523.2610	-4.4	4.5	$C_{21}H_{39}O_{11}N_4$



2b Ar^1 = 4-$Me_2NC_6H_4$





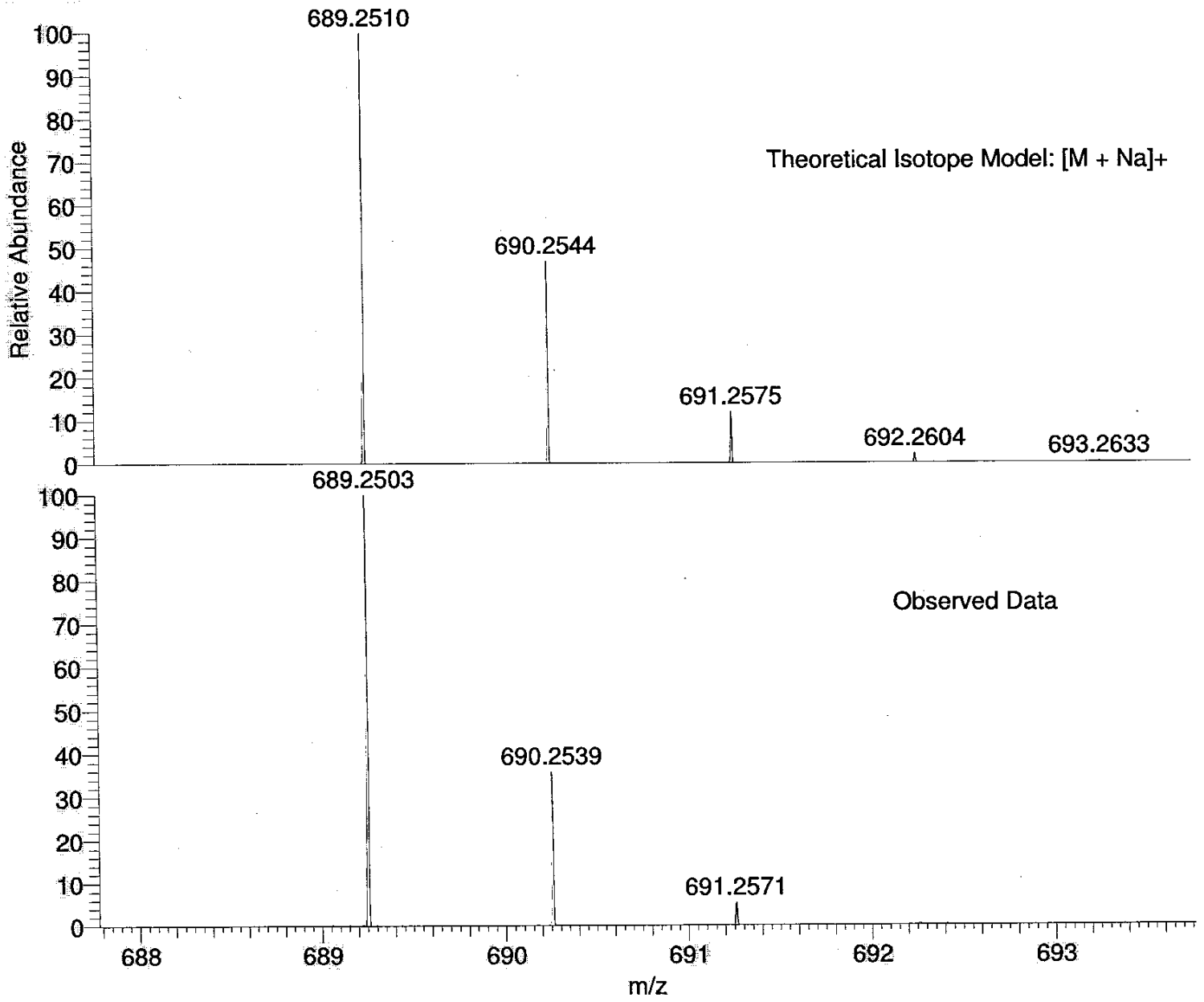


BMH-3A MW=666?
(DCM)/MeOH + NH4OAc

EPSRC National Centre Swansea
LTQ Orbitrap XL

Christopher S. Kershaw
18/05/2010 13:04:26

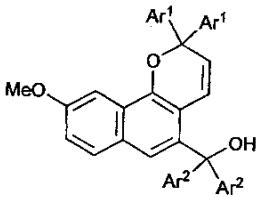
SM: 7G



NL:
1.45E4

$C_{43}H_{38}O_7Na$:
$C_{43}H_{38}O_7Na_1$
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

NL:
2.46E5
LEEHER126-OC-HNESP#9-13
RT: 0.87-1.19 AV: 5 T: FTMS
+ p NSI Full ms
[120.00-2000.00]



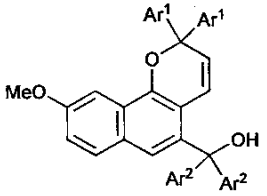
3a $Ar^1 = Ar^2 = 4\text{-}MeOC_6H_4$

Isotope:	Min. .. Max.
14 N	0....10
16 O	0....12
12 C	0....60
1 H	0....80
23 Na	0....1
Tolerance Window:	+- 5.00 ppm
Db/Ring Equiv:	-3.. 100
Fits:	100

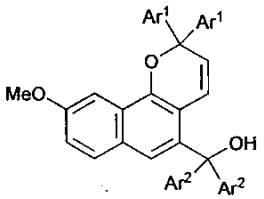
N-Rule: Do not use

Charge: 1

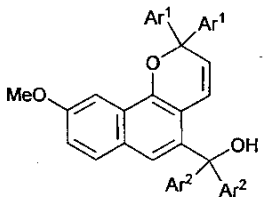
Mass	Theoretical Mass	Delta [ppm]	RDB	Composition
689.2503	689.2501	0.2	12.5	$C_{27}H_{38}O_{12}N_8Na_1$
	689.2507	-0.6	28.5	$C_{41}H_{33}O_5N_6$
	689.2496	1.0	25.0	$C_{41}H_{36}O_6N_3Na_1$
	689.2510	-1.0	30.0	$C_{42}H_{32}O_2N_7Na_1$
	689.2496	1.0	30.5	$C_{40}H_{30}O_1N_{10}Na_1$
	689.2510	-1.0	24.5	$C_{43}H_{38}O_7Na_1$
	689.2494	1.4	23.5	$C_{40}H_{37}O_9N_2$
	689.2494	1.4	29.0	$C_{39}H_{31}O_4N_9$
	689.2520	-2.5	33.5	$C_{42}H_{29}O_1N_{10}$
	689.2520	-2.5	28.0	$C_{43}H_{35}O_6N_3$
	689.2483	2.9	25.5	$C_{39}H_{34}O_5N_6Na_1$
	689.2523	-2.9	29.5	$C_{44}H_{34}O_3N_4Na_1$
	689.2525	-3.3	15.5	$C_{29}H_{37}O_{12}N_8$
	689.2480	3.3	24.0	$C_{38}H_{35}O_8N_5$
	689.2528	-3.7	17.0	$C_{30}H_{36}O_9N_9Na_1$
	689.2475	4.1	36.5	$C_{52}H_{33}O_2$
	689.2534	-4.5	33.0	$C_{44}H_{31}O_2N_7$
	689.2534	-4.5	27.5	$C_{45}H_{37}O_7$
	689.2470	4.9	20.5	$C_{38}H_{38}O_9N_2Na_1$
	689.2469	4.9	26.0	$C_{37}H_{32}O_4N_9Na_1$
	689.2537	-4.9	29.0	$C_{46}H_{36}O_4N_1Na_1$



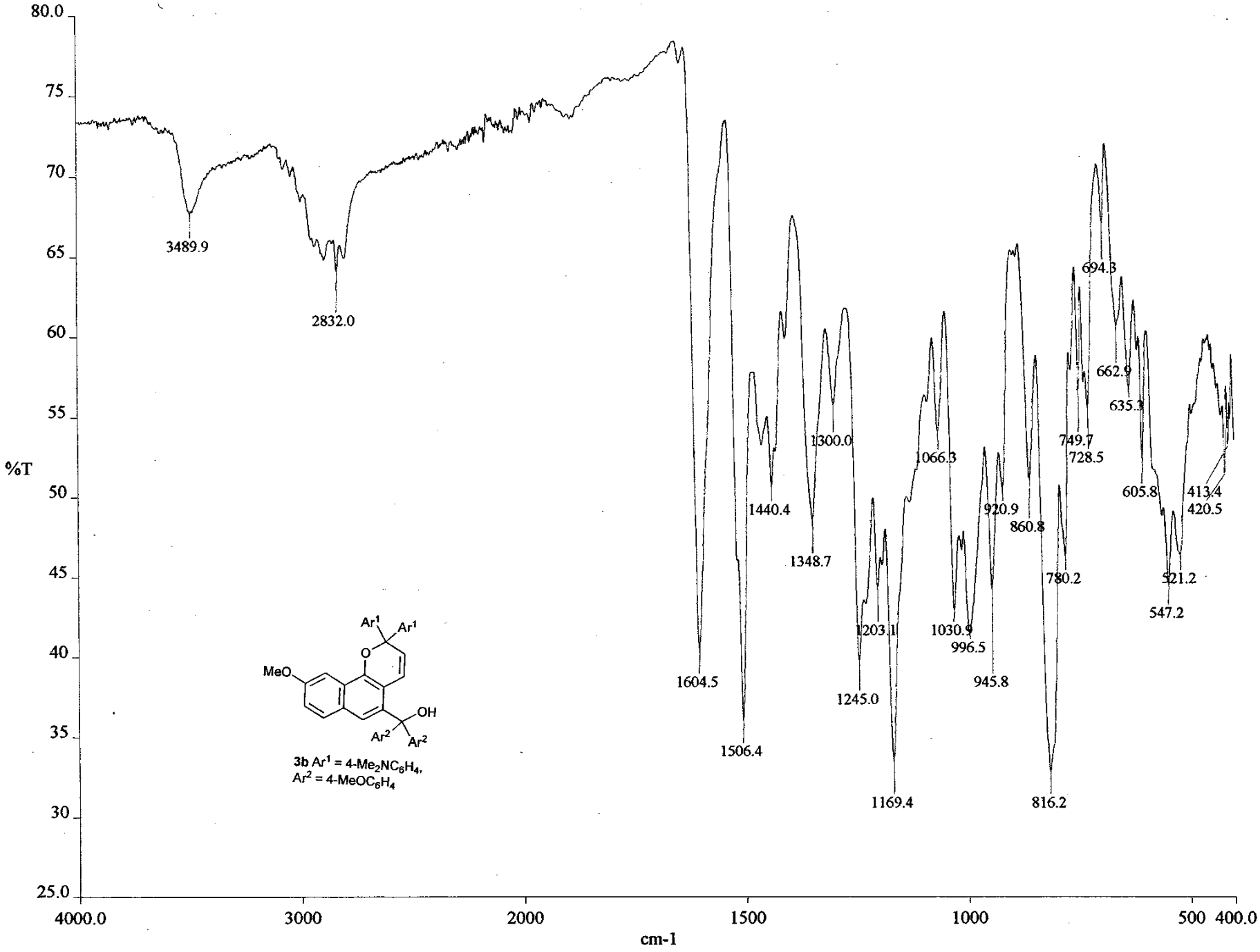
3a $Ar^1 = Ar^2 = 4\text{-}MeOC_6H_4$



3b $Ar^1 = 4\text{-}Me_2NC_6H_4$, $Ar^2 = 4\text{-}MeOC_6H_4$



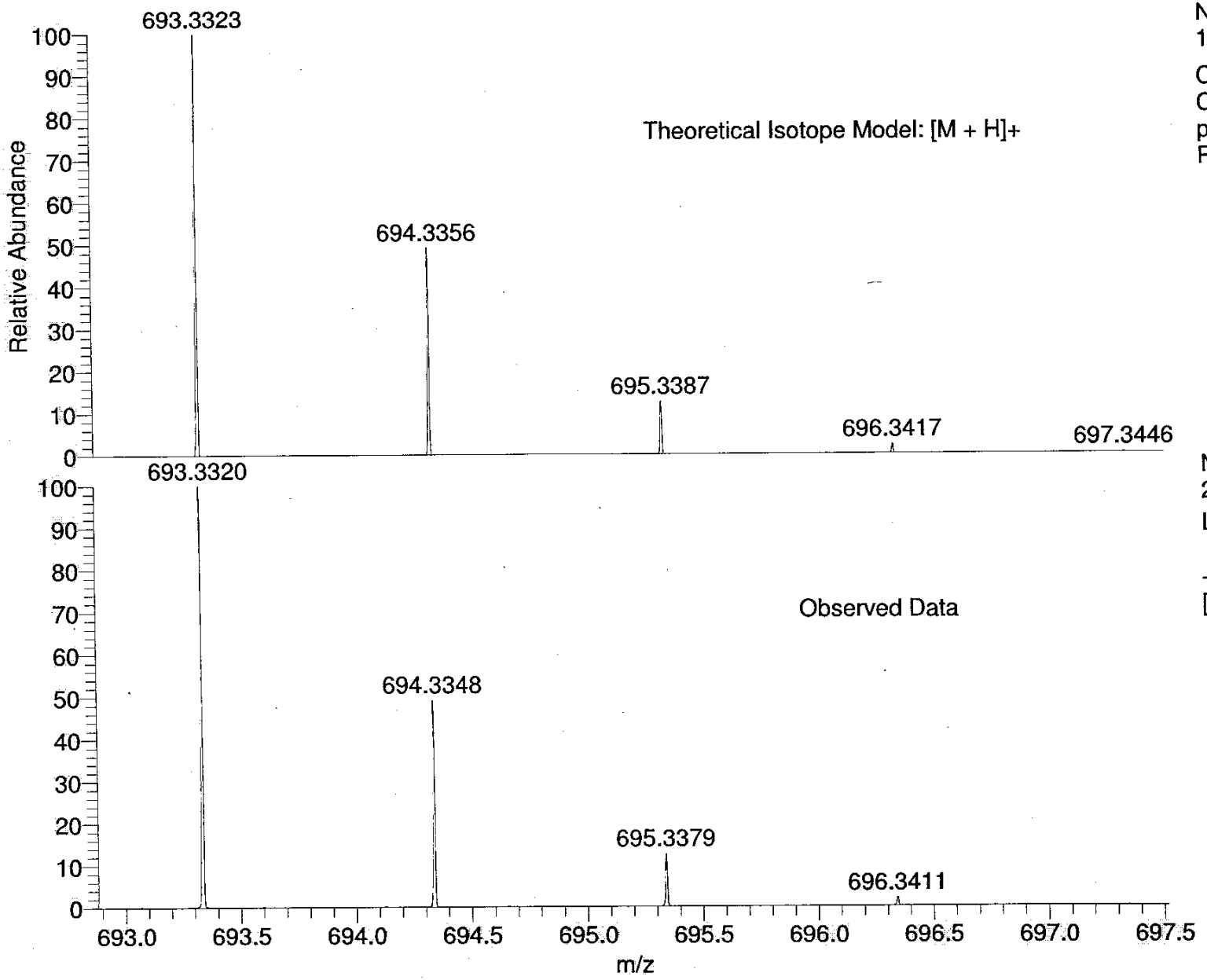
3b $Ar^1 = 4\text{-}Me_2NC_6H_4$, $Ar^2 = 4\text{-}MeOC_6H_4$

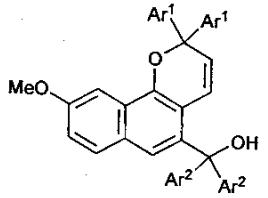


BMH-3B MW=692?
(DCM)/MeOH + NH4OAc

EPSRC National Centre Swansea
LTQ Orbitrap XL

Christopher S. Kershaw
18/05/2010 13:07:31



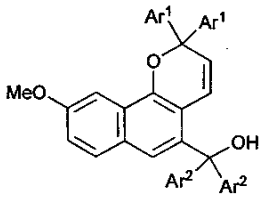


3b $Ar^1 = 4\text{-}Me_2NC_6H_4$, $Ar^2 = 4\text{-}MeOC_6H_4$

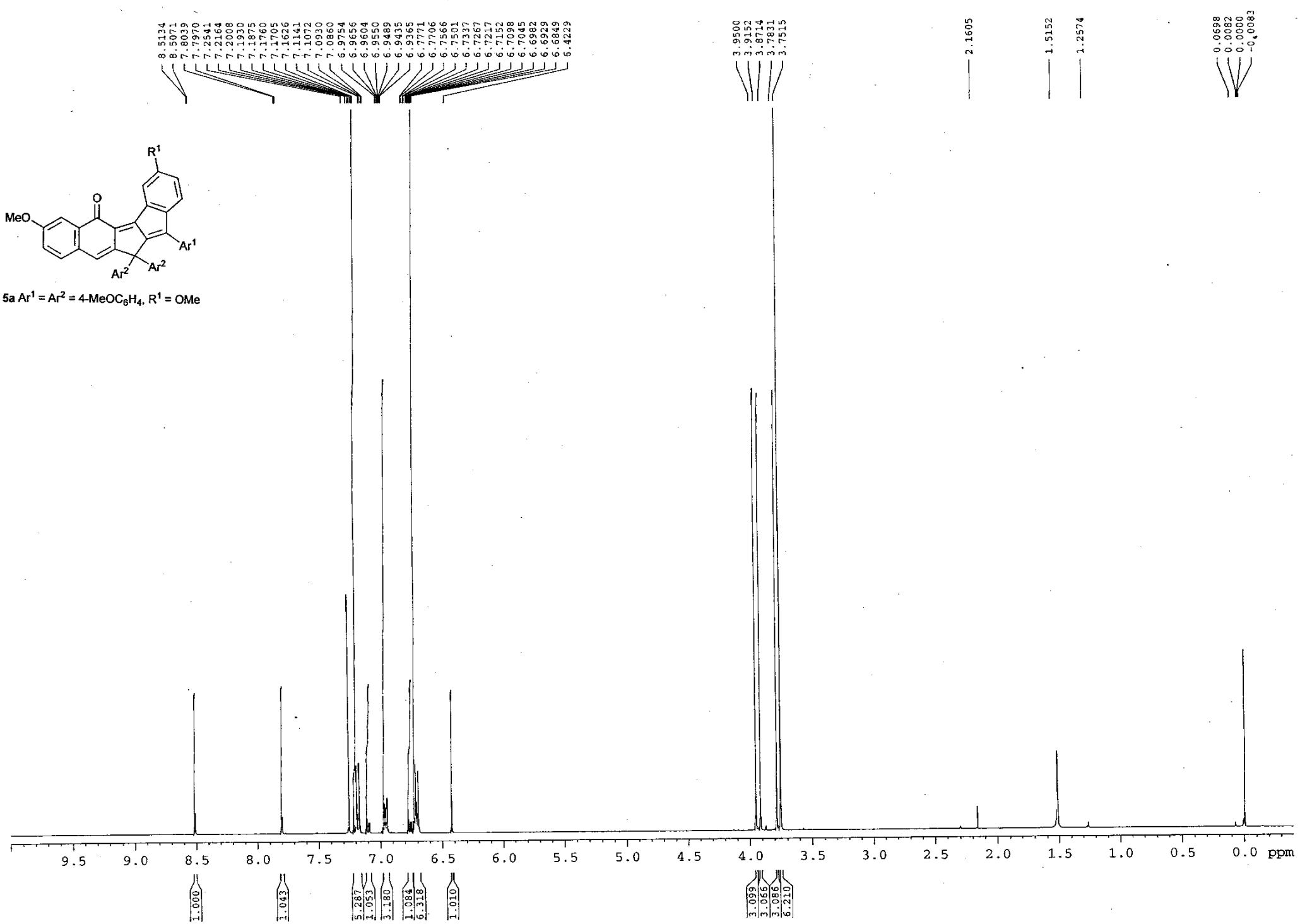
Isotope:	Min. .. Max.
14 N	0....10
16 O	0....12
12 C	0....60
1 H	0....80
23 Na	0....0
Tolerance Window:	+- 5.00 ppm
Db/Ring Equiv:	-3.. 100
Fits:	100

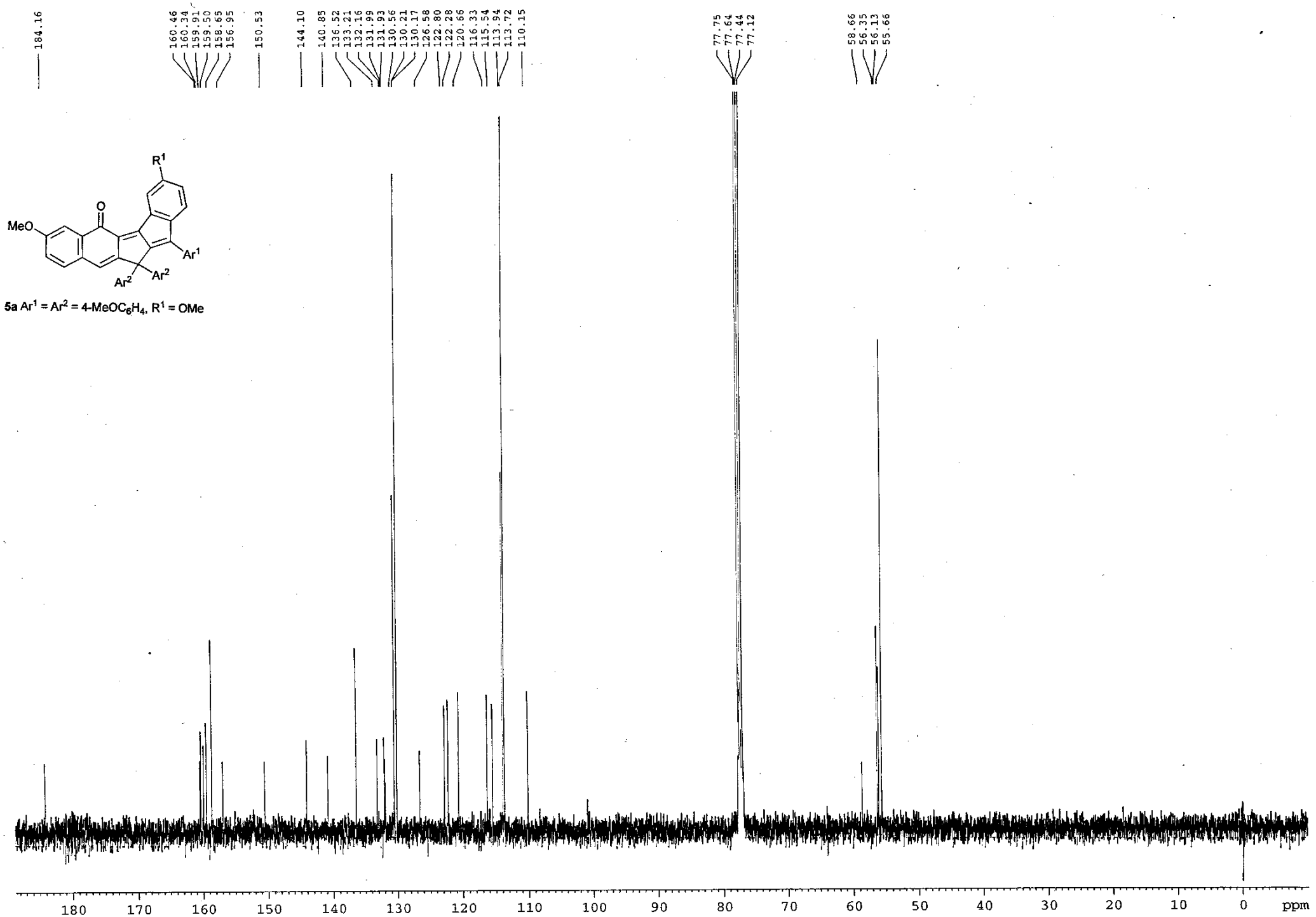
N-Rule:	Do not use
Charge:	1

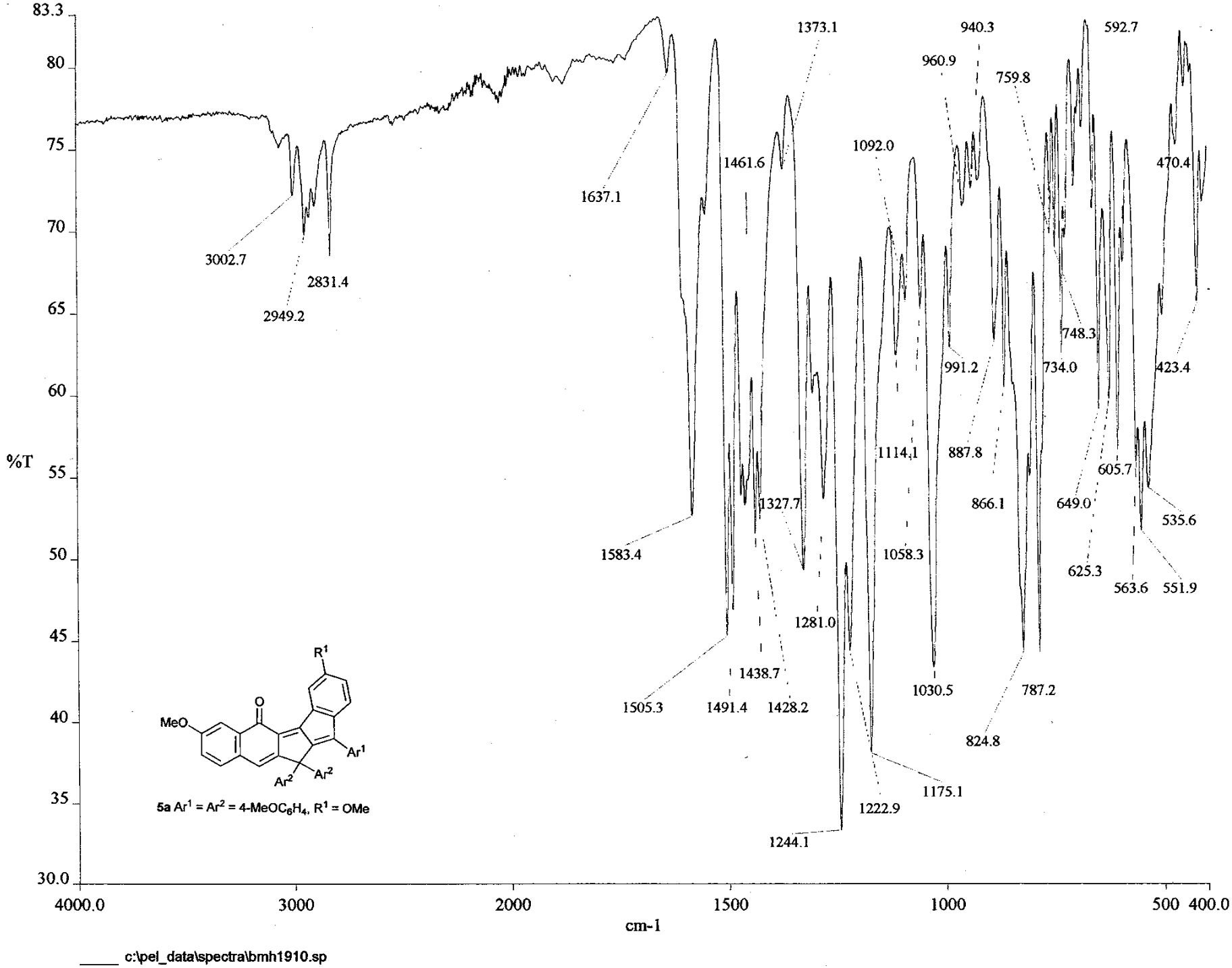
Mass	Theoretical Mass	Delta [ppm]	RDB	Composition
693.3320	693.3323	-0.4	30.0	$C_{44}H_{39}N_9$
	693.3323	-0.4	24.5	$C_{45}H_{45}O_5N_2$
	693.3315	0.8	12.5	$C_{29}H_{45}O_{10}N_{10}$
	693.3328	-1.2	12.0	$C_{31}H_{47}O_{11}N_7$
	693.3310	1.5	25.0	$C_{42}H_{43}O_4N_5$
	693.3336	-2.4	29.5	$C_{46}H_{41}O_1N_6$
	693.3341	-3.1	11.5	$C_{33}H_{49}O_{12}N_4$
	693.3296	3.4	20.0	$C_{42}H_{47}O_8N_1$
	693.3296	3.4	25.5	$C_{41}H_{41}O_3N_8$
	693.3350	-4.3	29.0	$C_{48}H_{43}O_2N_3$



3b Ar^1 = 4-$Me_2NC_6H_4$, Ar^2 = 4-$MeOC_6H_4$





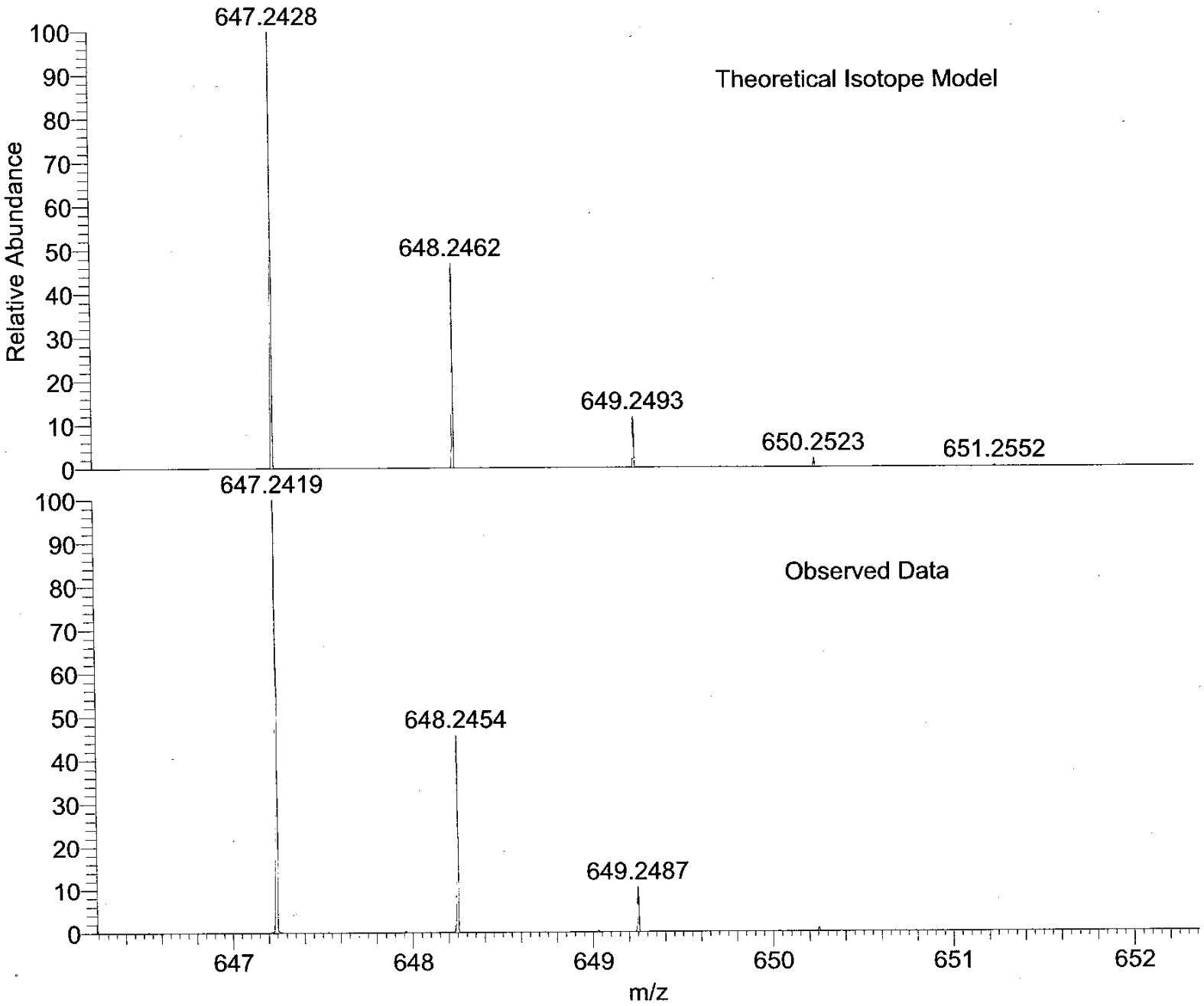


BMH1910 MW=648?
(DCM)/MeCN

EPSRC National Centre Swansea
LTQ Orbitrap XL

Dr. B M Heron
21/01/2010 17:00:02

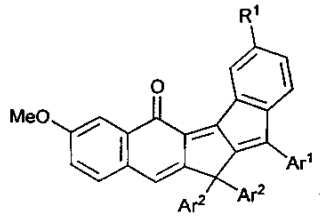
SM: 7G



NL:
1.45E4

$C_{43}H_{34}O_6H$:
$C_{43}H_{35}O_6$
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

NL:
1.07E6
LEEHER108-OC-HNESP#11-16 RT: 1.24-1.67 AV: 6 T: FTMS + p NSI Full ms [120.00-2000.00]

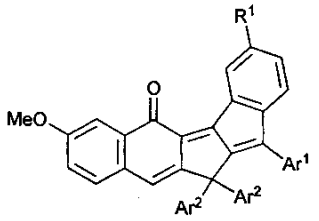


5a $Ar^1 = Ar^2 = 4\text{-}MeOC_6H_4$, $R^1 = OMe$

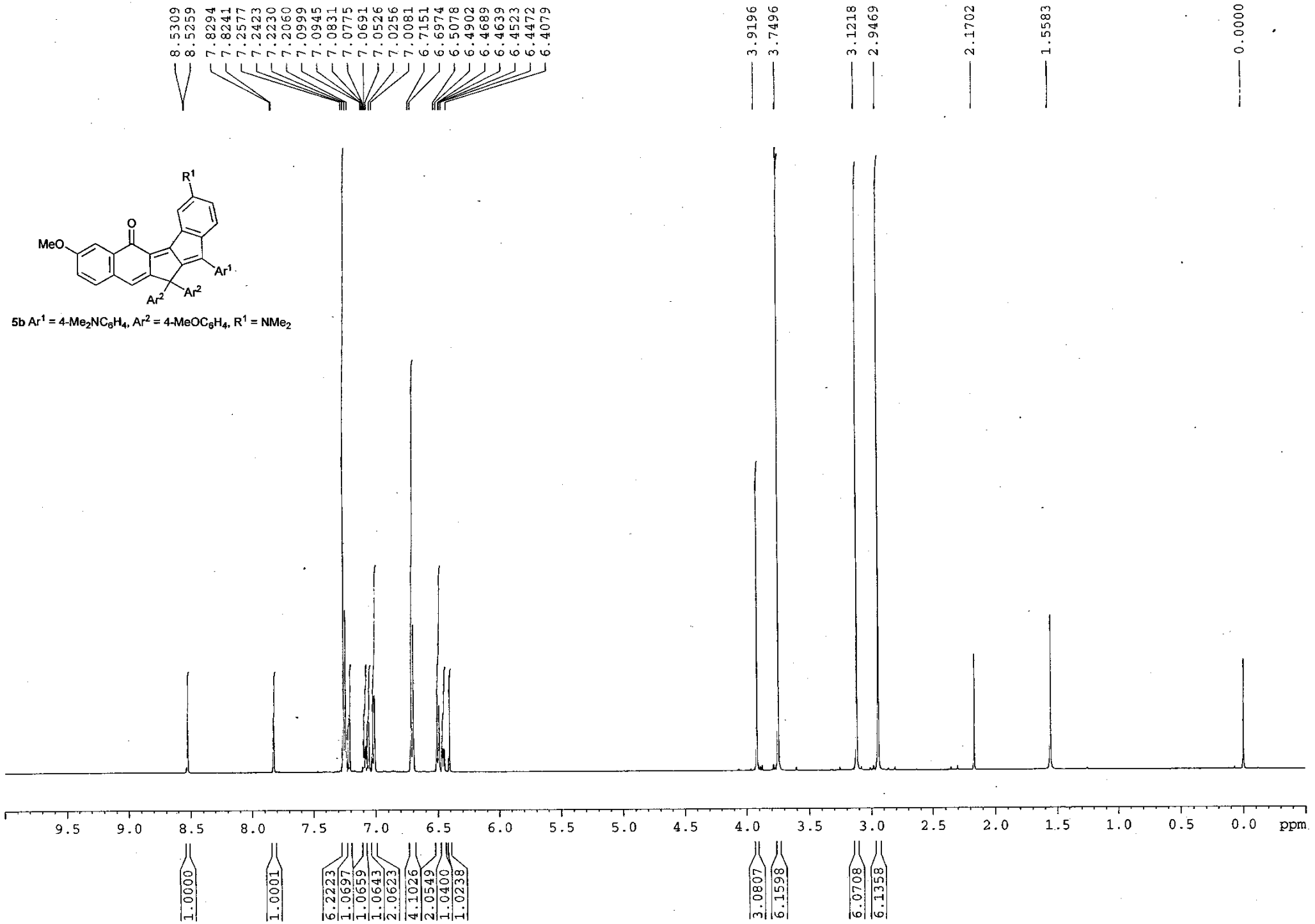
Isotope:	Min. .. Max.
14 N	0....10
16 O	0....15
12 C	0....50
1 H	0....70
23 Na	0....0
Tolerance Window:	+- 5.00 ppm
Db/Ring Equiv:	-2.. 100
Fits:	100

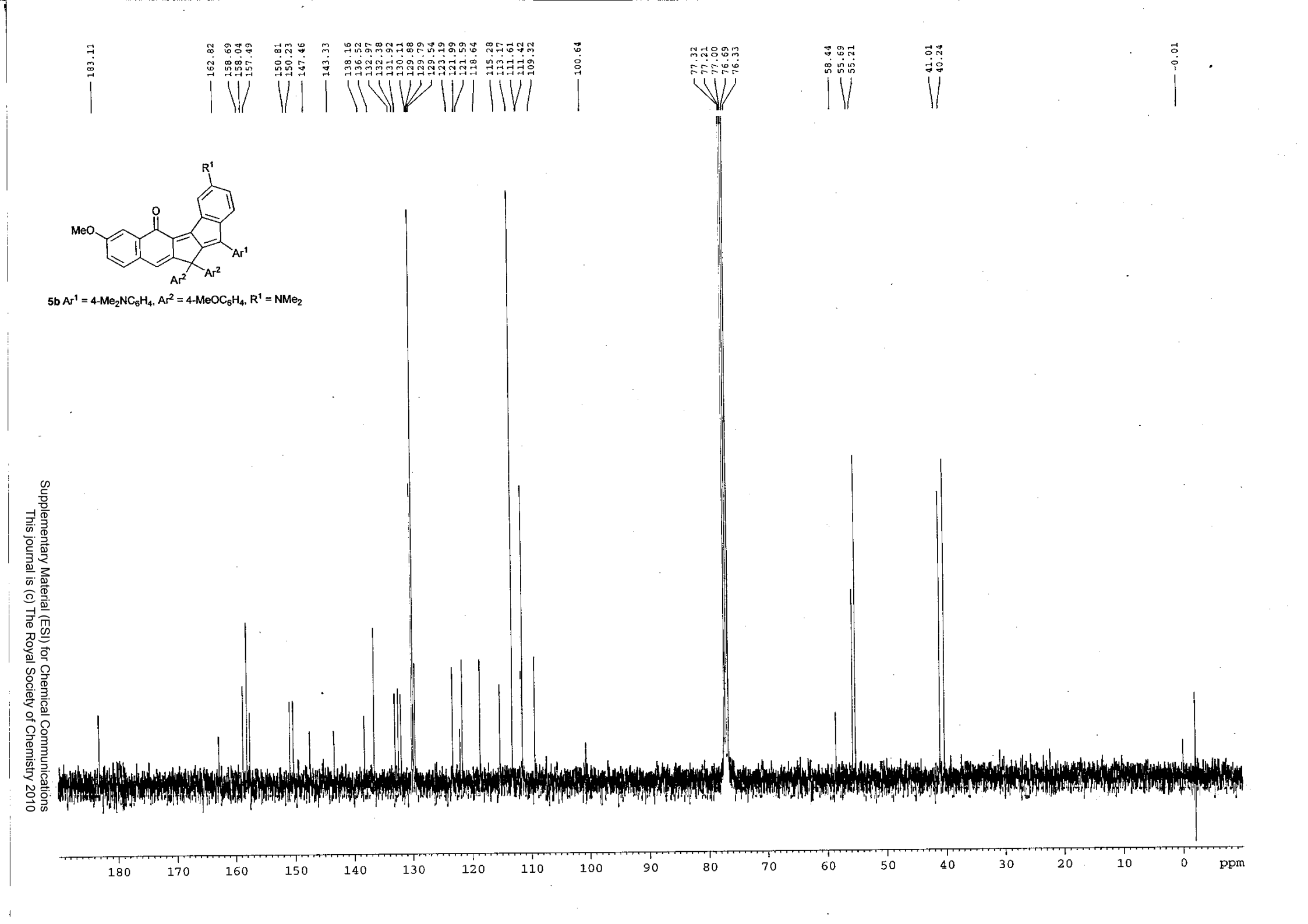
N-Rule: Do not use
Charge: 1

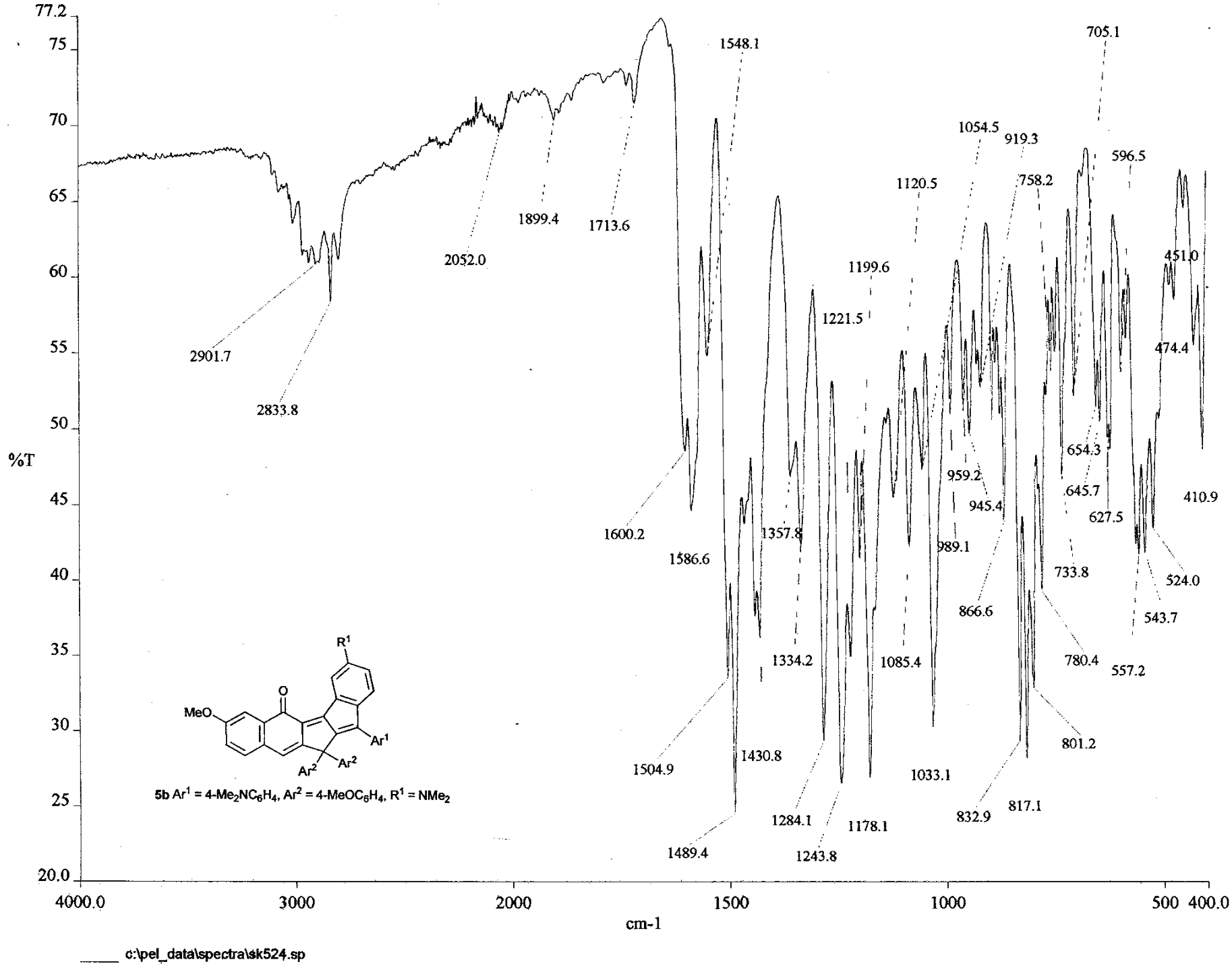
Mass	Theoretical Mass	Delta [ppm]	RDB	Composition
647.2419	647.2420	-0.1	14.5	$C_{27} H_{35} O_{11} N_8$
	647.2415	0.7	27.0	$C_{41} H_{33} O_5 N_3$
	647.2415	0.7	32.5	$C_{40} H_{27} N_{10}$
	647.2428	-1.4	32.0	$C_{42} H_{29} O_1 N_7$
	647.2428	-1.4	26.5	$C_{43} H_{35} O_6$
	647.2406	1.9	9.5	$C_{26} H_{39} O_{15} N_4$
	647.2433	-2.2	14.0	$C_{29} H_{37} O_{12} N_5$
	647.2401	2.7	27.5	$C_{39} H_{31} O_4 N_6$
	647.2442	-3.5	31.5	$C_{44} H_{31} O_2 N_4$
	647.2393	4.0	10.0	$C_{24} H_{37} O_{14} N_7$
	647.2447	-4.3	19.0	$C_{30} H_{33} O_8 N_9$
	647.2447	-4.3	13.5	$C_{31} H_{39} O_{13} N_2$
	647.2388	4.8	22.5	$C_{38} H_{35} O_8 N_2$
	647.2388	4.8	28.0	$C_{37} H_{29} O_3 N_9$



5a $Ar^1 = Ar^2 = 4\text{-}MeOC_6H_4$, $R^1 = OMe$



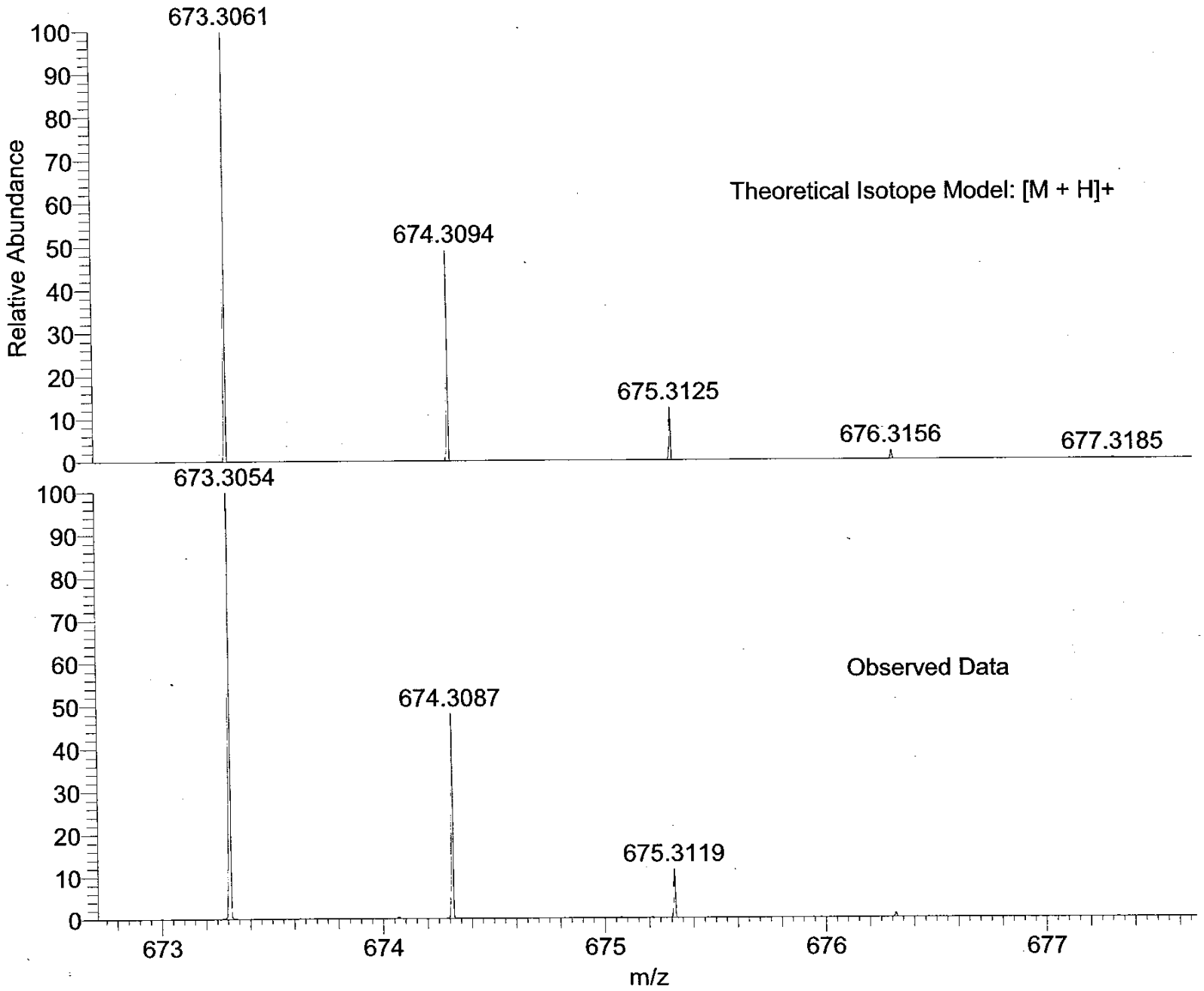




SK524 MW=672?
(DCM)/MeOH + NH4OAc
SM: 7G

EPSRC National Centre Swansea
LTQ Orbitrap XL

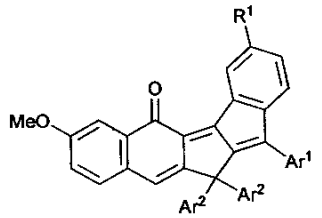
Christopher S. Kershaw
08/03/2010 17:25:06



NL:
1.41E4

$C_{45}H_{40}O_4N_2$ H:
$C_{45}H_{41}O_4N_2$
p (gss, s /p:40) Chrg 1
R: 100000 Res .Pwr . @FWHM

NL:
2.43E6
LEEHER116-OM-HNESP#8-12
RT: 0.80-1.12 AV: 5 T: FTMS
+ p NSI Full ms
[120.00-2000.00]

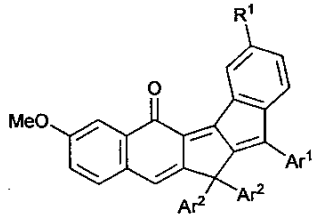


5b $Ar^1 = 4\text{-}Me_2NC_6H_4$, $Ar^2 = 4\text{-}MeOC_6H_4$, $R^1 = NMe_2$

Isotope:	Min. .. Max.
14 N	0....12
16 O	0....14
12 C	0....70
1 H	0....80
23 Na	0....0
Tolerance Window:	+- 5.00 ppm
Db/Ring Equiv:	-2.. 100
Fits:	150

N-Rule: Do not use
Charge: 1

Mass	Theoretical Mass	Delta [ppm]	RDB	Composition
673.3054	673.3053	0.2	9.0	$C_{30}H_{47}O_{14}N_3$
	673.3052	0.2	14.5	$C_{29}H_{41}O_9N_{10}$
	673.3047	1.0	27.0	$C_{43}H_{39}O_3N_5$
	673.3061	-1.0	26.5	$C_{45}H_{41}O_4N_2$
	673.3066	-1.8	14.0	$C_{31}H_{43}O_{10}N_7$
	673.3039	2.2	9.5	$C_{28}H_{45}O_{13}N_6$
	673.3034	3.0	22.0	$C_{42}H_{43}O_7N_1$
	673.3034	3.0	27.5	$C_{41}H_{37}O_2N_8$
	673.3074	-3.0	31.5	$C_{46}H_{37}N_6$
	673.3079	-3.8	19.0	$C_{32}H_{39}O_6N_{11}$
	673.3079	-3.8	13.5	$C_{33}H_{45}O_{11}N_4$
	673.3026	4.2	10.0	$C_{26}H_{43}O_{12}N_9$
	673.3021	5.0	22.5	$C_{40}H_{41}O_6N_4$
	673.3021	5.0	28.0	$C_{39}H_{35}O_1N_{11}$
	673.3088	-5.0	31.0	$C_{48}H_{39}O_1N_3$



5b Ar^1 = 4-$Me_2NC_6H_4$, Ar^2 = 4-$MeOC_6H_4$, R^1 = NMe_2