A Cu(I)-Catalyzed C-H α-Amination of Aryl Ketones. Direct Synthesis of Imidazolinones

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Supporting Information

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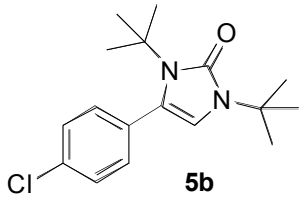
General Methods. All commercially available reagents and solvents were used without further purification. Chromatography was performed on silica gel (200-400 mesh) column. ^{1}H NMR spectra were recorded on 300 MHz NMR spectrometer and ^{13}C NMR spectra were recorded on 100 MHz NMR spectrometer. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected.

Spectroscopic and analytic data

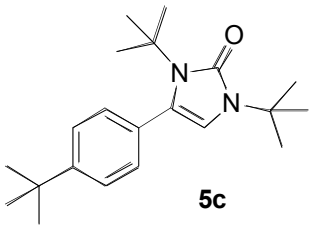
Table 1, Entry 2



5b

White solid; mp. 131-132 ^{o}C; IR (film) 1674 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (d, J = 8.4 Hz, 2H), 7.23 (d, J = 8.4 Hz, 2H), 6.03 (s, 1H), 1.53 (s, 9H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.8, 133.9, 133.4, 131.7, 128.2, 121.6, 108.6, 58.5, 54.8, 30.4, 28.3; HRMS Calcd for $C_{17}H_{23}ClN_2O$ (M^+): 306.1499. Found: 306.1506.

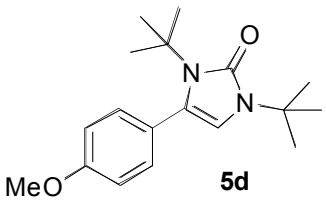
Table 1, Entry 3



5c

White solid; mp. 65-66 ^{o}C; IR (film) 1678 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, J = 8.4 Hz, 2H), 7.23 (d, J = 8.4 Hz, 2H), 6.03 (s, 1H), 1.53 (s, 9H), 1.42 (s, 9H), 1.32 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.7, 151.0, 131.9, 130.3, 124.8, 122.8, 108.1, 58.2, 54.6, 34.7, 31.5, 30.4, 28.3; HRMS Calcd for $C_{21}H_{32}N_2O$ (M^+): 328.2515. Found: 328.2520.

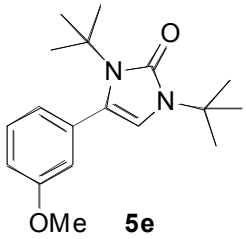
Table 1, Entry 4



5d

White solid; mp. 78-79 ^{o}C; IR (film) 1676 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, J = 8.7 Hz, 2H), 6.84 (d, J = 8.7 Hz, 2H), 6.01 (s, 1H), 3.82 (s, 3H), 1.53 (s, 9H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.4, 153.6, 132.0, 127.0, 122.4, 113.3, 107.9, 58.2, 55.4, 54.6, 30.4, 28.3; Anal. Calcd for $C_{18}H_{26}N_2O_2$: C, 71.49; H, 8.67; N, 9.26. Found: C, 71.19; H, 8.55; N, 9.12.

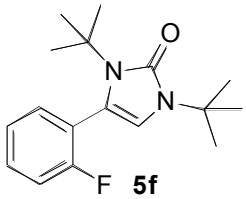
Table 1, Entry 5



5e

White solid; mp. 101-102 ^{o}C; IR (film) 1678 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25-7.19 (m, 1H), 6.93-6.89 (m, 1H), 6.86-6.83 (m, 2H), 6.05 (s, 1H), 3.81 (s, 3H), 1.53 (s, 9H), 1.44 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 153.8, 136.2, 128.8, 123.1, 122.7, 116.1, 113.3, 108.2, 58.4, 55.4, 54.7, 30.2, 28.3; Anal. Calcd for $C_{18}H_{26}N_2O_2$: C, 71.49; H, 8.67; N, 9.26. Found: C, 71.34; H, 8.47; N, 9.12.

Table 1, Entry 6

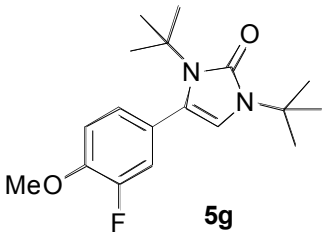


5f

White solid; mp. 110-111 ^{o}C; IR (film) 1669 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.27 (m, 2H), 7.16-7.00 (m, 2H), 6.12 (s, 1H), 1.55 (s, 9H), 1.43 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.8, 159.6, 153.5, 132.61, 132.58, 130.5, 130.3, 123.93, 123.88, 123.0,

122.8, 115.8, 115.5, 115.2, 108.9, 58.3, 54.9, 29.4, 28.3; Anal. Calcd for $C_{17}H_{23}FN_2O$: C, 70.32; H, 7.98; N, 9.65. Found: C, 70.21; H, 7.76; N, 9.44.

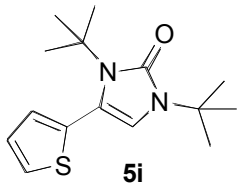
Table 1, Entry 7



5g

White solid; mp. 94-95 ^oC; IR (film) 1677 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.07-7.00 (m, 2H), 6.92-6.86 (m, 1H), 6.03 (s, 1H), 3.90 (s, 3H), 1.52 (s, 9H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.6, 153.2, 149.9, 147.6, 147.5, 127.6, 127.5, 126.8, 126.7, 121.4, 118.7, 118.5, 112.6, 108.4, 58.4, 56.4, 54.8, 30.4, 28.3; HRMS Calcd for $C_{18}H_{25}FN_2O_2$ (M^+): 320.1900. Found: 320.1903.

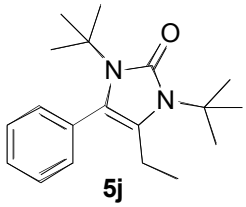
Table 1, Entry 9



5i

Yellow solid; mp. 119-120 ^oC; IR (film) 1672 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.32 (dd, J = 5.1, 1.5 Hz, 1H), 7.00-6.94 (m, 2H), 6.24 (s, 1H), 1.52 (s, 9H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.2, 135.0, 130.2, 126.9, 126.5, 113.7, 110.7, 58.4, 54.9, 29.9, 28.3; Anal. Calcd for $C_{15}H_{22}N_2OS$: C, 64.71; H, 7.96; N, 10.06. Found: C, 64.89; H, 7.91; N, 9.90.

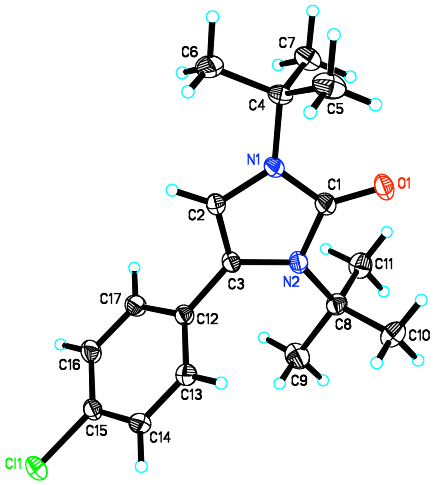
Table 1, Entry 10



5j

White solid; mp. 72-73 ^{o}C; IR (film) 1670 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.32 (m, 3H), 7.29-7.25 (m, 2H), 2.26 (q, *J* = 7.2 Hz, 2H), 1.69 (s, 9H), 1.34 (s, 9H), 0.90 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.0, 134.9, 131.9, 128.1, 127.9, 123.3, 120.7, 58.2, 58.0, 30.3, 29.5, 19.4, 15.6; Anal. Calcd for $C_{19}H_{28}N_2O$: C, 75.96; H, 9.39; N, 9.32. Found: C, 75.87; H, 9.29; N, 9.19.

The X-ray structure and data of **5b**



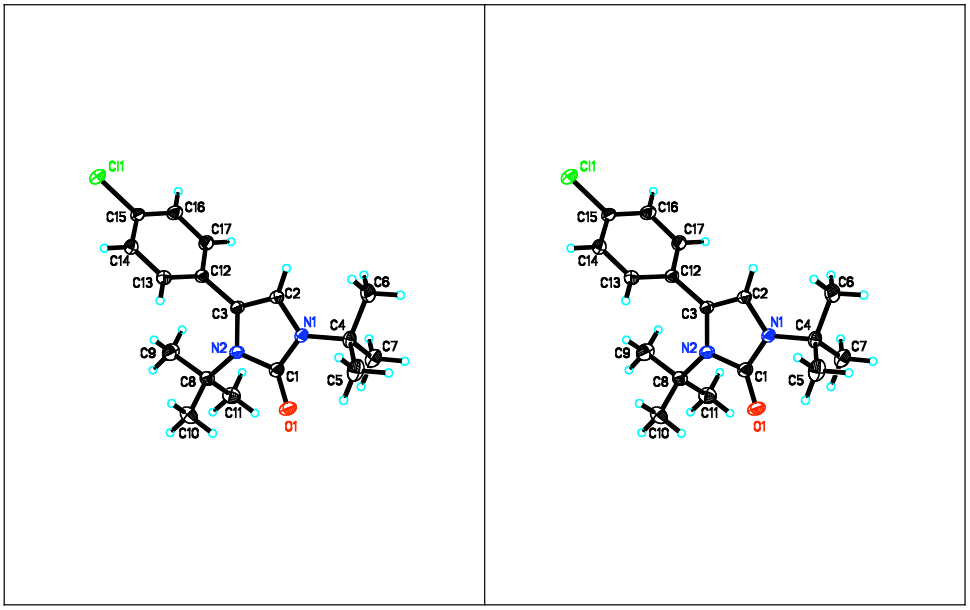


Table 1. Crystal data and structure refinement for **5b**.

Identification code	ys186_0m	
Empirical formula	C17 H23 Cl N2 O	
Formula weight	306.82	
Temperature	80(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 5.8924(4) Å	α= 90°.
	b = 20.8589(13) Å	β= 97.309(4)°.
	c = 13.5893(8) Å	γ = 90°.
Volume	1656.68(18) $Å^3$	
Z	4	
Density (calculated)	1.230 Mg/m^3	
Absorption coefficient	0.232 mm^{-1}	
F(000)	656	
Crystal size	0.49 x 0.43 x 0.09 mm^3	
Theta range for data collection	1.95 to 33.30°.	
Index ranges	-9<=h<=8, -32<=k<=32, -20<=l<=20	
Reflections collected	24630	
Independent reflections	6336 [R(int) = 0.0436]	
Completeness to theta = 33.30°	99.3 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9792 and 0.8959	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6336 / 0 / 191	
Goodness-of-fit on F^2	1.037	
Final R indices [I>2sigma(I)]	R1 = 0.0455, wR2 = 0.1208	
R indices (all data)	R1 = 0.0787, wR2 = 0.1440	
Largest diff. peak and hole	0.423 and -0.311 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ys186_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	4150(1)	5047(1)	8683(1)	32(1)
O(1)	10367(2)	7280(1)	3711(1)	31(1)
N(1)	10011(2)	7618(1)	5324(1)	19(1)
N(2)	8470(2)	6685(1)	4835(1)	21(1)
C(1)	9697(2)	7204(1)	4533(1)	22(1)
C(2)	9038(2)	7356(1)	6113(1)	19(1)
C(3)	8089(2)	6788(1)	5824(1)	18(1)
C(4)	11022(2)	8270(1)	5274(1)	20(1)
C(5)	13524(2)	8207(1)	5094(1)	31(1)
C(6)	10874(2)	8613(1)	6253(1)	31(1)
C(7)	9650(2)	8645(1)	4436(1)	26(1)
C(8)	7433(2)	6210(1)	4083(1)	21(1)
C(9)	5869(2)	5740(1)	4527(1)	32(1)
C(10)	9368(2)	5835(1)	3699(1)	28(1)
C(11)	5994(2)	6566(1)	3238(1)	28(1)
C(12)	7084(2)	6343(1)	6497(1)	18(1)
C(13)	8172(2)	5771(1)	6808(1)	20(1)
C(14)	7276(2)	5370(1)	7472(1)	21(1)
C(15)	5273(2)	5549(1)	7840(1)	21(1)
C(16)	4171(2)	6116(1)	7559(1)	24(1)
C(17)	5081(2)	6511(1)	6885(1)	22(1)

Table 3. Bond lengths [Å] and angles [°] for ys186_0m.

Cl(1)-C(15)	1.7434(13)
O(1)-C(1)	1.2408(15)
N(1)-C(1)	1.3726(17)
N(1)-C(2)	1.3910(15)
N(1)-C(4)	1.4892(16)
N(2)-C(1)	1.3922(16)
N(2)-C(3)	1.4080(15)
N(2)-C(8)	1.4959(16)
C(2)-C(3)	1.3469(17)
C(3)-C(12)	1.4791(16)
C(4)-C(6)	1.5229(19)
C(4)-C(7)	1.5262(18)
C(4)-C(5)	1.5306(17)
C(8)-C(9)	1.5223(18)
C(8)-C(10)	1.5297(18)
C(8)-C(11)	1.5308(18)
C(12)-C(13)	1.3934(17)
C(12)-C(17)	1.3973(16)
C(13)-C(14)	1.3838(17)
C(14)-C(15)	1.3904(17)
C(15)-C(16)	1.3798(18)
C(16)-C(17)	1.3896(18)

C(1)-N(1)-C(2)	109.24(10)
C(1)-N(1)-C(4)	123.64(10)
C(2)-N(1)-C(4)	126.83(10)
C(1)-N(2)-C(3)	108.31(10)
C(1)-N(2)-C(8)	119.73(10)
C(3)-N(2)-C(8)	130.72(10)
O(1)-C(1)-N(1)	126.66(12)
O(1)-C(1)-N(2)	126.97(12)
N(1)-C(1)-N(2)	106.37(10)
C(3)-C(2)-N(1)	108.46(10)
C(2)-C(3)-N(2)	107.61(10)

C(2)-C(3)-C(12)	123.83(11)
N(2)-C(3)-C(12)	128.23(11)
N(1)-C(4)-C(6)	108.80(10)
N(1)-C(4)-C(7)	108.98(10)
C(6)-C(4)-C(7)	109.05(11)
N(1)-C(4)-C(5)	109.12(10)
C(6)-C(4)-C(5)	110.08(11)
C(7)-C(4)-C(5)	110.77(10)
N(2)-C(8)-C(9)	111.85(10)
N(2)-C(8)-C(10)	108.34(10)
C(9)-C(8)-C(10)	108.66(12)
N(2)-C(8)-C(11)	109.31(11)
C(9)-C(8)-C(11)	107.74(11)
C(10)-C(8)-C(11)	110.96(11)
C(13)-C(12)-C(17)	118.56(11)
C(13)-C(12)-C(3)	121.34(10)
C(17)-C(12)-C(3)	119.97(11)
C(14)-C(13)-C(12)	121.13(11)
C(13)-C(14)-C(15)	118.93(12)
C(16)-C(15)-C(14)	121.43(11)
C(16)-C(15)-Cl(1)	119.50(10)
C(14)-C(15)-Cl(1)	119.07(10)
C(15)-C(16)-C(17)	118.93(11)
C(16)-C(17)-C(12)	121.01(12)

Symmetry transformations used to generate equivalent atoms:

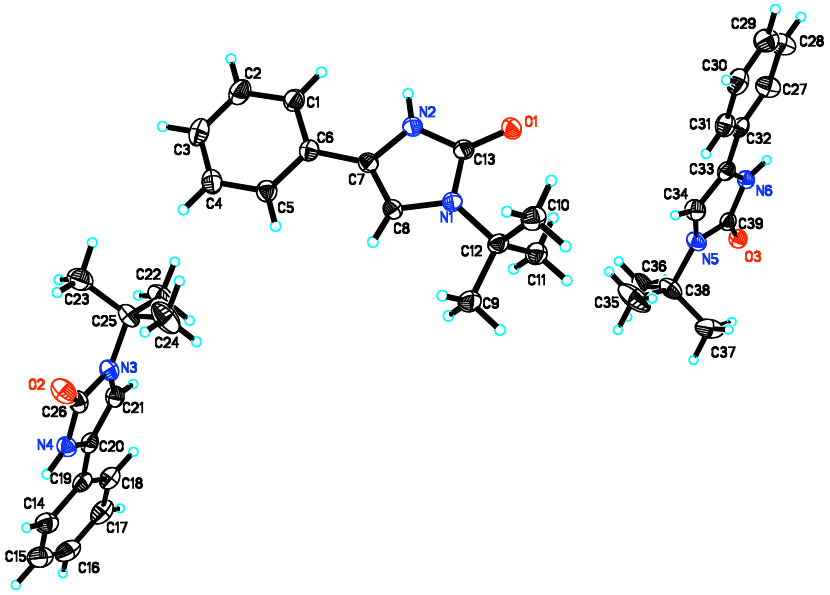
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for ys186_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	46(1)	27(1)	25(1)	5(1)	16(1)	-5(1)
O(1)	41(1)	32(1)	22(1)	-2(1)	16(1)	-11(1)
N(1)	22(1)	19(1)	18(1)	0(1)	5(1)	-5(1)
N(2)	26(1)	22(1)	17(1)	-3(1)	7(1)	-7(1)
C(1)	25(1)	22(1)	21(1)	-1(1)	7(1)	-5(1)
C(2)	21(1)	20(1)	16(1)	0(1)	5(1)	-1(1)
C(3)	18(1)	19(1)	16(1)	1(1)	4(1)	-1(1)
C(4)	19(1)	17(1)	24(1)	2(1)	2(1)	-4(1)
C(5)	18(1)	28(1)	46(1)	7(1)	4(1)	-2(1)
C(6)	40(1)	22(1)	29(1)	-4(1)	3(1)	-10(1)
C(7)	21(1)	26(1)	30(1)	7(1)	4(1)	0(1)
C(8)	22(1)	21(1)	20(1)	-4(1)	3(1)	-4(1)
C(9)	37(1)	32(1)	25(1)	-4(1)	3(1)	-18(1)
C(10)	29(1)	26(1)	28(1)	-6(1)	2(1)	3(1)
C(11)	27(1)	32(1)	24(1)	-1(1)	0(1)	2(1)
C(12)	18(1)	18(1)	17(1)	0(1)	3(1)	-2(1)
C(13)	21(1)	20(1)	20(1)	0(1)	5(1)	0(1)
C(14)	26(1)	18(1)	20(1)	2(1)	3(1)	1(1)
C(15)	28(1)	20(1)	16(1)	0(1)	5(1)	-5(1)
C(16)	23(1)	26(1)	24(1)	2(1)	10(1)	1(1)
C(17)	22(1)	22(1)	24(1)	4(1)	7(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for ys186_0m.

	x	y	z	U(eq)
H(2A)	9041	7546	6749	22
H(5A)	13602	7986	4463	46
H(5B)	14369	7959	5635	46
H(5C)	14204	8634	5070	46
H(6A)	9267	8652	6360	46
H(6B)	11552	9041	6231	46
H(6C)	11709	8367	6797	46
H(7A)	8059	8684	4569	39
H(7B)	9697	8419	3807	39
H(7C)	10313	9074	4395	39
H(9A)	4616	5974	4773	47
H(9B)	6746	5508	5077	47
H(9C)	5240	5434	4016	47
H(10A)	10386	6132	3409	42
H(10B)	8721	5529	3191	42
H(10C)	10238	5602	4249	42
H(11A)	4764	6802	3501	42
H(11B)	5329	6257	2740	42
H(11C)	6967	6868	2930	42
H(13A)	9551	5656	6560	24
H(14A)	8019	4979	7674	26
H(16A)	2811	6234	7821	28
H(17A)	4330	6902	6685	27

The X-ray structure and data of **6**



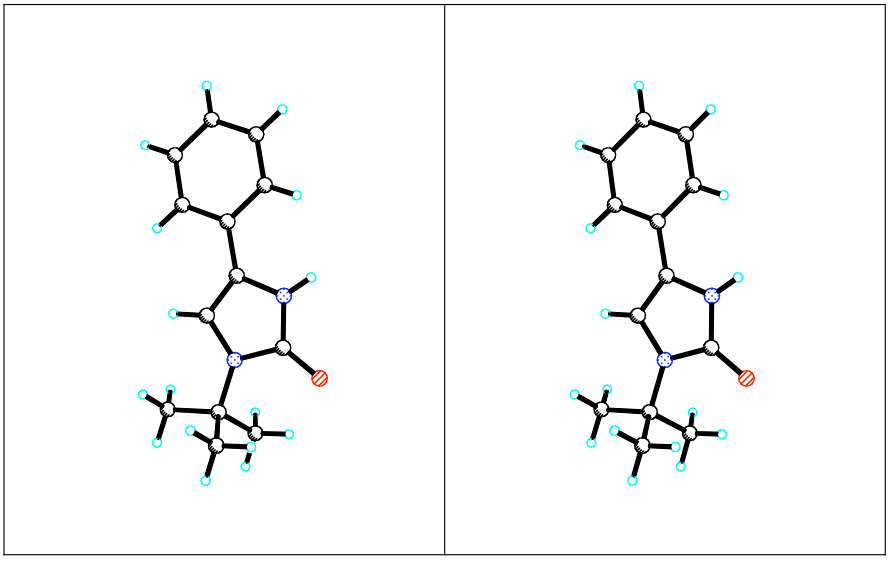


Table 1. Crystal data and structure refinement for **6**.

Identification code	ys191r_0m	
Empirical formula	C39 H48 N6 O3	
Formula weight	648.83	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.3603(5) Å	α= 90°.
	b = 19.5772(9) Å	β= 91.330(3)°.
	c = 18.0785(8) Å	γ = 90°.
Volume	3665.8(3) Å^3	
Z	4	
Density (calculated)	1.176 Mg/m^3	
Absorption coefficient	0.076 mm^{-1}	
F(000)	1392	
Crystal size	0.41 x 0.20 x 0.16 mm^3	
Theta range for data collection	2.08 to 36.32°.	
Index ranges	-17<=h<=17, -32<=k<=32, -30<=l<=30	
Reflections collected	75621	
Independent reflections	17760 [R(int) = 0.0674]	
Completeness to theta = 36.32°	99.9 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9878 and 0.9699	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	17760 / 0 / 433	
Goodness-of-fit on F^2	1.000	
Final R indices [I>2sigma(I)]	R1 = 0.0586, wR2 = 0.1255	
R indices (all data)	R1 = 0.1378, wR2 = 0.1554	
Largest diff. peak and hole	0.337 and -0.301 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ys191r_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	1030(1)	5495(1)	5654(1)	29(1)
O(2)	8718(1)	1387(1)	2794(1)	34(1)
O(3)	2077(1)	10950(1)	2442(1)	27(1)
N(1)	2934(1)	4847(1)	5751(1)	22(1)
N(2)	1355(1)	4459(1)	5048(1)	24(1)
N(3)	8166(1)	2191(1)	1873(1)	25(1)
N(4)	10141(1)	1800(1)	1918(1)	25(1)
N(5)	2432(1)	9905(1)	3047(1)	28(1)
N(6)	600(1)	10427(1)	3213(1)	23(1)
C(1)	1177(1)	3233(1)	4106(1)	28(1)
C(2)	1172(1)	2691(1)	3613(1)	34(1)
C(3)	2257(1)	2290(1)	3528(1)	34(1)
C(4)	3357(1)	2427(1)	3956(1)	29(1)
C(5)	3377(1)	2968(1)	4450(1)	25(1)
C(6)	2291(1)	3387(1)	4526(1)	22(1)
C(7)	2357(1)	3987(1)	5005(1)	22(1)
C(8)	3324(1)	4227(1)	5449(1)	21(1)
C(9)	5078(1)	5044(1)	6318(1)	29(1)
C(10)	3074(1)	5345(1)	6993(1)	30(1)
C(11)	3737(1)	6022(1)	5879(1)	28(1)
C(12)	3702(1)	5316(1)	6238(1)	23(1)
C(13)	1699(1)	4986(1)	5499(1)	23(1)
C(14)	12280(1)	2007(1)	883(1)	34(1)
C(15)	13275(1)	2136(1)	398(1)	41(1)
C(16)	13172(1)	2653(1)	-119(1)	41(1)
C(17)	12064(1)	3049(1)	-151(1)	39(1)
C(18)	11069(1)	2926(1)	326(1)	32(1)
C(19)	11154(1)	2401(1)	849(1)	27(1)
C(20)	10076(1)	2262(1)	1332(1)	24(1)
C(21)	8848(1)	2499(1)	1309(1)	26(1)
C(22)	6203(1)	2813(1)	1513(1)	41(1)

C(23)	6098(1)	1593(1)	1889(1)	43(1)
C(24)	6646(2)	2514(1)	2826(1)	52(1)
C(25)	6768(1)	2274(1)	2028(1)	29(1)
C(26)	8984(1)	1753(1)	2251(1)	26(1)
C(27)	-1685(1)	9984(1)	4064(1)	35(1)
C(28)	-2685(1)	9789(1)	4519(1)	43(1)
C(29)	-2529(1)	9246(1)	5003(1)	40(1)
C(30)	-1366(1)	8905(1)	5043(1)	36(1)
C(31)	-366(1)	9102(1)	4599(1)	31(1)
C(32)	-507(1)	9640(1)	4098(1)	25(1)
C(33)	567(1)	9829(1)	3633(1)	24(1)
C(34)	1703(1)	9511(1)	3527(1)	30(1)
C(35)	4198(2)	9068(1)	3063(1)	64(1)
C(36)	3791(2)	9799(1)	1957(1)	47(1)
C(37)	4641(1)	10321(1)	3141(1)	45(1)
C(38)	3769(1)	9774(1)	2800(1)	34(1)
C(39)	1734(1)	10476(1)	2858(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for ys191r_0m.

O(1)-C(13)	1.2498(14)
O(2)-C(26)	1.2507(14)
O(3)-C(39)	1.2512(14)
N(1)-C(13)	1.3754(14)
N(1)-C(8)	1.3939(14)
N(1)-C(12)	1.4896(14)
N(2)-C(13)	1.3583(14)
N(2)-C(7)	1.3924(14)
N(3)-C(26)	1.3761(14)
N(3)-C(21)	1.3913(15)
N(3)-C(25)	1.4900(15)
N(4)-C(26)	1.3566(15)
N(4)-C(20)	1.3946(14)
N(5)-C(39)	1.3704(14)
N(5)-C(34)	1.3956(16)
N(5)-C(38)	1.4880(16)
N(6)-C(39)	1.3549(15)
N(6)-C(33)	1.3948(14)
C(1)-C(2)	1.3846(17)
C(1)-C(6)	1.4000(15)
C(2)-C(3)	1.3831(19)
C(3)-C(4)	1.3883(18)
C(4)-C(5)	1.3844(16)
C(5)-C(6)	1.4026(16)
C(6)-C(7)	1.4599(15)
C(7)-C(8)	1.3532(15)
C(9)-C(12)	1.5249(16)
C(10)-C(12)	1.5271(16)
C(11)-C(12)	1.5271(16)
C(14)-C(15)	1.3913(19)
C(14)-C(19)	1.3983(18)
C(15)-C(16)	1.381(2)
C(16)-C(17)	1.386(2)
C(17)-C(18)	1.3798(18)

C(18)-C(19)	1.3975(17)
C(19)-C(20)	1.4596(17)
C(20)-C(21)	1.3531(17)
C(22)-C(25)	1.5157(17)
C(23)-C(25)	1.5214(19)
C(24)-C(25)	1.5255(18)
C(27)-C(28)	1.3920(19)
C(27)-C(32)	1.3949(18)
C(28)-C(29)	1.383(2)
C(29)-C(30)	1.378(2)
C(30)-C(31)	1.3815(19)
C(31)-C(32)	1.3935(17)
C(32)-C(33)	1.4576(17)
C(33)-C(34)	1.3495(17)
C(35)-C(38)	1.5232(19)
C(36)-C(38)	1.5253(19)
C(37)-C(38)	1.523(2)

C(13)-N(1)-C(8)	108.54(9)
C(13)-N(1)-C(12)	123.63(9)
C(8)-N(1)-C(12)	127.77(9)
C(13)-N(2)-C(7)	110.53(9)
C(26)-N(3)-C(21)	108.44(10)
C(26)-N(3)-C(25)	124.41(10)
C(21)-N(3)-C(25)	127.04(9)
C(26)-N(4)-C(20)	110.71(9)
C(39)-N(5)-C(34)	108.39(10)
C(39)-N(5)-C(38)	123.78(10)
C(34)-N(5)-C(38)	127.75(10)
C(39)-N(6)-C(33)	110.49(9)
C(2)-C(1)-C(6)	120.38(11)
C(3)-C(2)-C(1)	121.00(11)
C(2)-C(3)-C(4)	119.17(11)
C(5)-C(4)-C(3)	120.45(12)
C(4)-C(5)-C(6)	120.73(11)
C(1)-C(6)-C(5)	118.23(10)

C(1)-C(6)-C(7)	121.51(10)
C(5)-C(6)-C(7)	120.19(10)
C(8)-C(7)-N(2)	106.27(9)
C(8)-C(7)-C(6)	130.91(10)
N(2)-C(7)-C(6)	122.75(9)
C(7)-C(8)-N(1)	108.49(9)
N(1)-C(12)-C(9)	109.02(9)
N(1)-C(12)-C(10)	108.55(9)
C(9)-C(12)-C(10)	110.16(9)
N(1)-C(12)-C(11)	108.94(9)
C(9)-C(12)-C(11)	108.95(10)
C(10)-C(12)-C(11)	111.18(10)
O(1)-C(13)-N(2)	126.98(10)
O(1)-C(13)-N(1)	126.87(10)
N(2)-C(13)-N(1)	106.16(9)
C(15)-C(14)-C(19)	120.11(13)
C(16)-C(15)-C(14)	120.87(13)
C(15)-C(16)-C(17)	119.23(13)
C(18)-C(17)-C(16)	120.51(13)
C(17)-C(18)-C(19)	120.92(13)
C(18)-C(19)-C(14)	118.36(12)
C(18)-C(19)-C(20)	120.24(11)
C(14)-C(19)-C(20)	121.38(11)
C(21)-C(20)-N(4)	105.90(10)
C(21)-C(20)-C(19)	130.60(11)
N(4)-C(20)-C(19)	123.43(10)
C(20)-C(21)-N(3)	108.83(10)
N(3)-C(25)-C(22)	108.93(10)
N(3)-C(25)-C(23)	108.37(10)
C(22)-C(25)-C(23)	109.87(11)
N(3)-C(25)-C(24)	108.26(10)
C(22)-C(25)-C(24)	109.07(11)
C(23)-C(25)-C(24)	112.26(12)
O(2)-C(26)-N(4)	126.90(11)
O(2)-C(26)-N(3)	126.98(11)
N(4)-C(26)-N(3)	106.12(10)

C(28)-C(27)-C(32)	120.22(12)
C(29)-C(28)-C(27)	120.54(13)
C(30)-C(29)-C(28)	119.63(13)
C(29)-C(30)-C(31)	120.05(12)
C(30)-C(31)-C(32)	121.37(12)
C(31)-C(32)-C(27)	118.18(11)
C(31)-C(32)-C(33)	119.80(11)
C(27)-C(32)-C(33)	122.03(11)
C(34)-C(33)-N(6)	106.09(10)
C(34)-C(33)-C(32)	130.11(11)
N(6)-C(33)-C(32)	123.77(10)
C(33)-C(34)-N(5)	108.64(10)
N(5)-C(38)-C(37)	107.65(11)
N(5)-C(38)-C(35)	109.27(11)
C(37)-C(38)-C(35)	110.23(13)
N(5)-C(38)-C(36)	109.30(11)
C(37)-C(38)-C(36)	111.09(12)
C(35)-C(38)-C(36)	109.27(13)
O(3)-C(39)-N(6)	126.82(10)
O(3)-C(39)-N(5)	126.79(11)
N(6)-C(39)-N(5)	106.39(10)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for ys191r_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	23(1)	30(1)	34(1)	-10(1)	-5(1)	6(1)
O(2)	34(1)	36(1)	31(1)	11(1)	4(1)	9(1)
O(3)	28(1)	24(1)	28(1)	2(1)	0(1)	2(1)
N(1)	20(1)	23(1)	23(1)	-2(1)	-3(1)	2(1)
N(2)	20(1)	26(1)	26(1)	-5(1)	-5(1)	3(1)
N(3)	27(1)	24(1)	25(1)	2(1)	1(1)	4(1)
N(4)	25(1)	25(1)	26(1)	1(1)	-2(1)	3(1)
N(5)	25(1)	25(1)	33(1)	4(1)	1(1)	5(1)
N(6)	24(1)	22(1)	24(1)	0(1)	-2(1)	4(1)
C(1)	28(1)	25(1)	30(1)	-1(1)	-3(1)	0(1)
C(2)	39(1)	29(1)	33(1)	-5(1)	-8(1)	-4(1)
C(3)	47(1)	24(1)	30(1)	-6(1)	1(1)	-4(1)
C(4)	34(1)	23(1)	31(1)	-1(1)	6(1)	2(1)
C(5)	26(1)	23(1)	25(1)	0(1)	2(1)	0(1)
C(6)	26(1)	20(1)	20(1)	1(1)	2(1)	-1(1)
C(7)	21(1)	22(1)	21(1)	1(1)	0(1)	1(1)
C(8)	21(1)	21(1)	23(1)	1(1)	0(1)	2(1)
C(9)	25(1)	29(1)	34(1)	-2(1)	-8(1)	1(1)
C(10)	31(1)	36(1)	23(1)	-2(1)	-1(1)	-3(1)
C(11)	28(1)	25(1)	32(1)	1(1)	-1(1)	-1(1)
C(12)	21(1)	23(1)	23(1)	-1(1)	-4(1)	-1(1)
C(13)	21(1)	26(1)	23(1)	-3(1)	-3(1)	1(1)
C(14)	30(1)	39(1)	32(1)	-3(1)	-2(1)	-2(1)
C(15)	30(1)	52(1)	40(1)	-8(1)	1(1)	-4(1)
C(16)	36(1)	50(1)	36(1)	-9(1)	7(1)	-17(1)
C(17)	47(1)	36(1)	32(1)	-3(1)	4(1)	-15(1)
C(18)	37(1)	29(1)	31(1)	-1(1)	1(1)	-7(1)
C(19)	29(1)	27(1)	24(1)	-6(1)	-2(1)	-6(1)
C(20)	29(1)	21(1)	23(1)	-3(1)	-3(1)	-2(1)
C(21)	31(1)	22(1)	24(1)	1(1)	-1(1)	1(1)
C(22)	35(1)	41(1)	47(1)	13(1)	2(1)	13(1)

C(23)	29(1)	38(1)	62(1)	9(1)	6(1)	2(1)
C(24)	54(1)	68(1)	34(1)	-5(1)	9(1)	26(1)
C(25)	29(1)	31(1)	29(1)	3(1)	5(1)	10(1)
C(26)	30(1)	24(1)	24(1)	0(1)	-1(1)	4(1)
C(27)	31(1)	45(1)	29(1)	6(1)	-1(1)	2(1)
C(28)	30(1)	64(1)	36(1)	5(1)	2(1)	4(1)
C(29)	39(1)	51(1)	28(1)	-3(1)	7(1)	-9(1)
C(30)	48(1)	32(1)	28(1)	-1(1)	3(1)	-6(1)
C(31)	38(1)	26(1)	28(1)	0(1)	0(1)	-1(1)
C(32)	29(1)	26(1)	21(1)	-4(1)	-4(1)	-3(1)
C(33)	28(1)	22(1)	22(1)	0(1)	-5(1)	-1(1)
C(34)	31(1)	25(1)	33(1)	6(1)	-1(1)	3(1)
C(35)	49(1)	52(1)	92(1)	32(1)	26(1)	28(1)
C(36)	48(1)	55(1)	39(1)	-6(1)	9(1)	18(1)
C(37)	26(1)	66(1)	44(1)	6(1)	0(1)	1(1)
C(38)	29(1)	34(1)	40(1)	8(1)	7(1)	11(1)
C(39)	25(1)	22(1)	23(1)	-3(1)	-4(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for ys191r_0m.

	x	y	z	U(eq)
H(2A)	603	4420	4815	29
H(4B)	10837	1570	2053	31
H(6A)	-28	10729	3183	28
H(1A)	420	3501	4159	33
H(2B)	411	2594	3329	40
H(3A)	2251	1926	3181	40
H(4A)	4101	2148	3910	35
H(5A)	4134	3055	4741	30
H(8A)	4132	4010	5539	26
H(9A)	5471	5029	5830	44
H(9B)	5061	4583	6529	44
H(9C)	5585	5346	6645	44
H(10A)	3066	4886	7211	45
H(10B)	2186	5512	6935	45
H(10C)	3567	5654	7319	45
H(11A)	4148	5989	5398	42
H(11B)	4231	6336	6199	42
H(11C)	2853	6194	5811	42
H(14A)	12366	1651	1238	40
H(15A)	14034	1864	423	49
H(16A)	13854	2737	-450	49
H(17A)	11988	3407	-503	46
H(18A)	10316	3202	299	39
H(21A)	8507	2822	965	31
H(22A)	6276	2661	999	61
H(22B)	6675	3242	1584	61
H(22C)	5291	2883	1624	61
H(23A)	6199	1459	1371	64
H(23B)	5178	1637	1993	64
H(23C)	6485	1244	2212	64

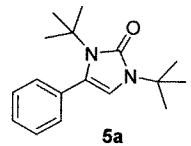
H(24A)	7093	2952	2891	78
H(24B)	7036	2174	3161	78
H(24C)	5732	2570	2939	78
H(27A)	-1805	10354	3729	42
H(28A)	-3481	10029	4497	52
H(29A)	-3220	9110	5307	47
H(30A)	-1253	8533	5376	43
H(31A)	435	8867	4635	37
H(34A)	1963	9089	3742	36
H(35A)	3630	8721	2841	96
H(35B)	4153	9045	3603	96
H(35C)	5088	8986	2914	96
H(36A)	3230	9439	1751	71
H(36B)	4676	9727	1793	71
H(36C)	3482	10245	1785	71
H(37A)	4354	10772	2969	68
H(37B)	5532	10243	2993	68
H(37C)	4596	10300	3682	68

STANDARD 1H OBSERVE

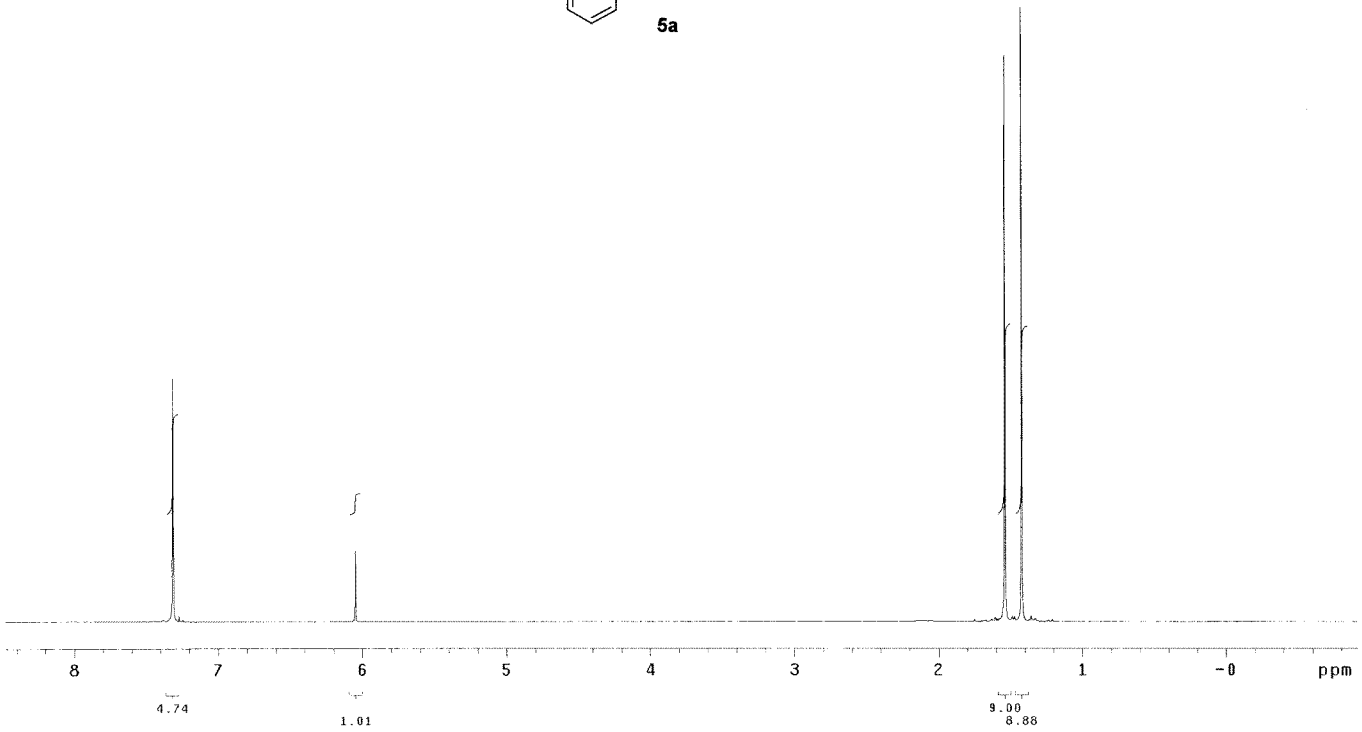
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234A-P2-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 289.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 1



5a

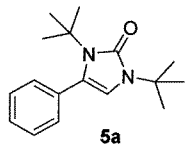


13C OBSERVE

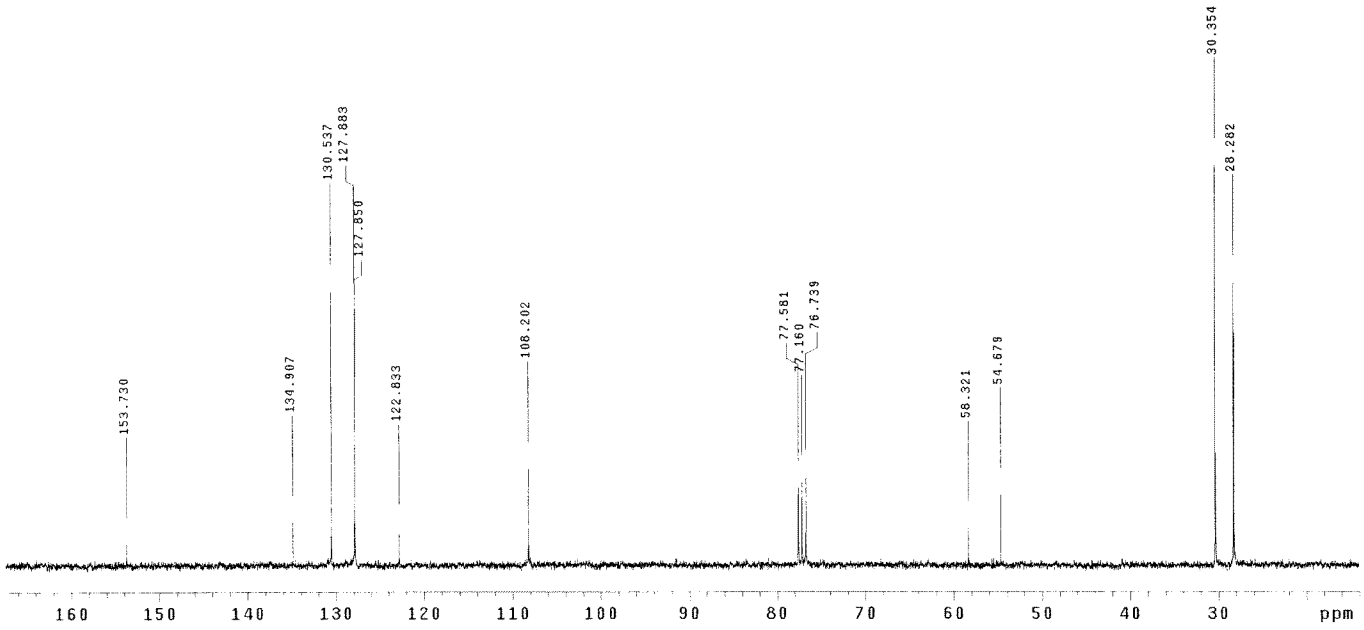
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234A-P2-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
64 repetitions
OBSERVE C13, 75.4233263 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec

Table 1, Entry 1



5a

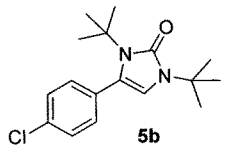


STANDARD 1H OBSERVE

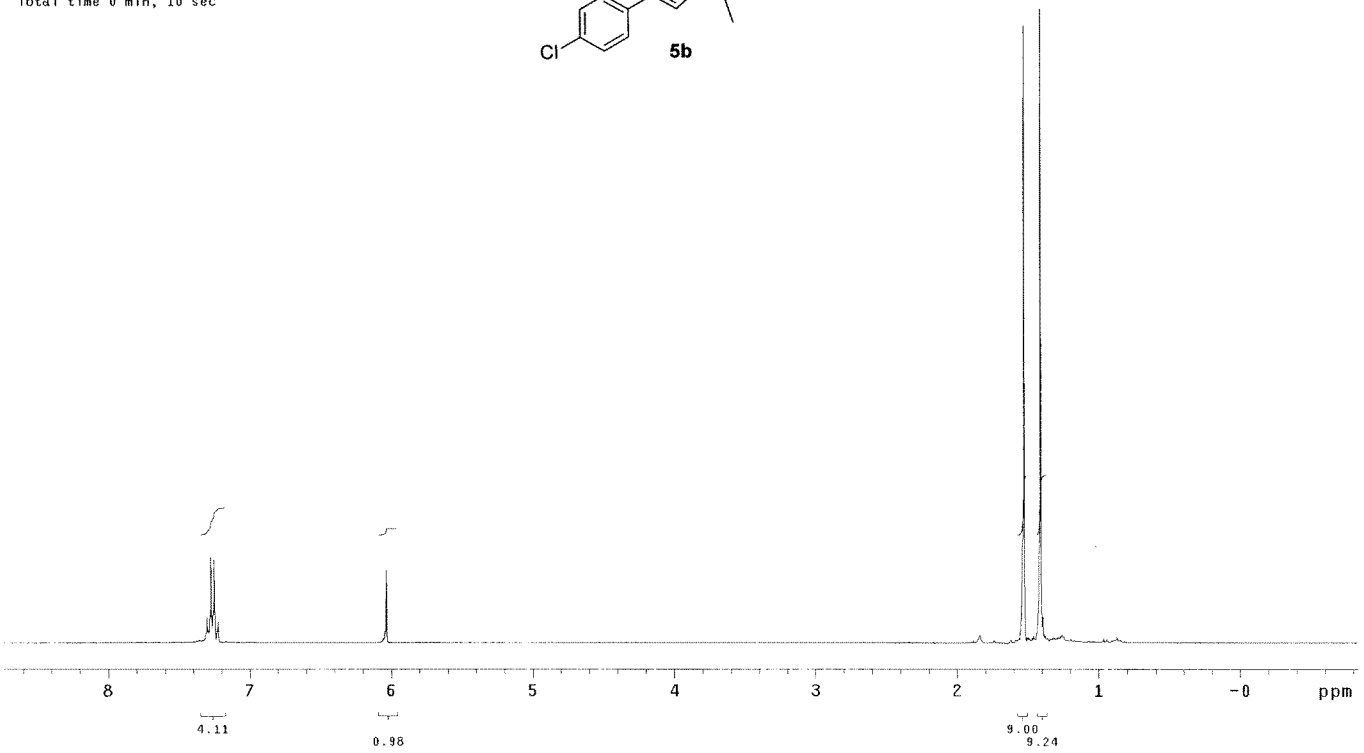
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234B-H3
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
2 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 10 sec

Table 1, Entry 2



5b

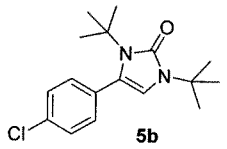


13C OBSERVE

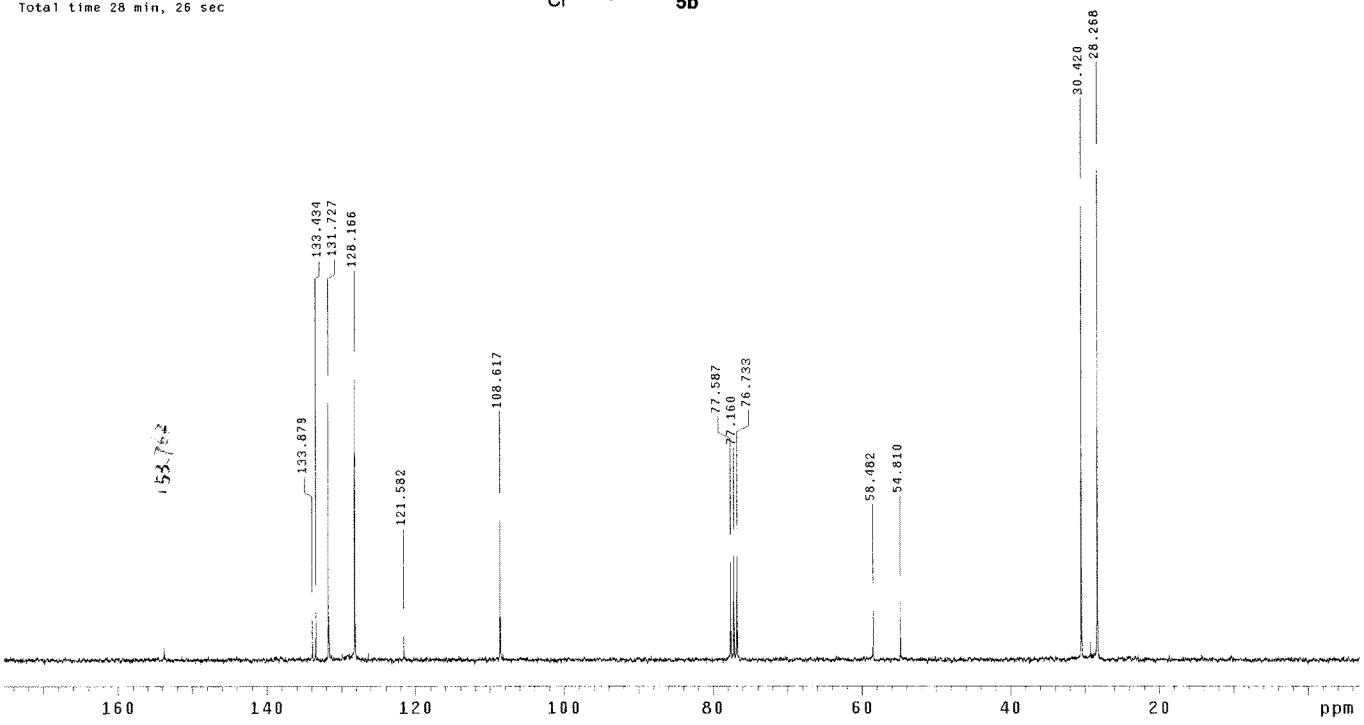
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234B-C3
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
800 repetitions
OBSERVE C13, 75.4750857 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 28 min, 26 sec

Table 1, Entry 2



5b

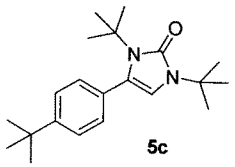


STANDARD 1H OBSERVE

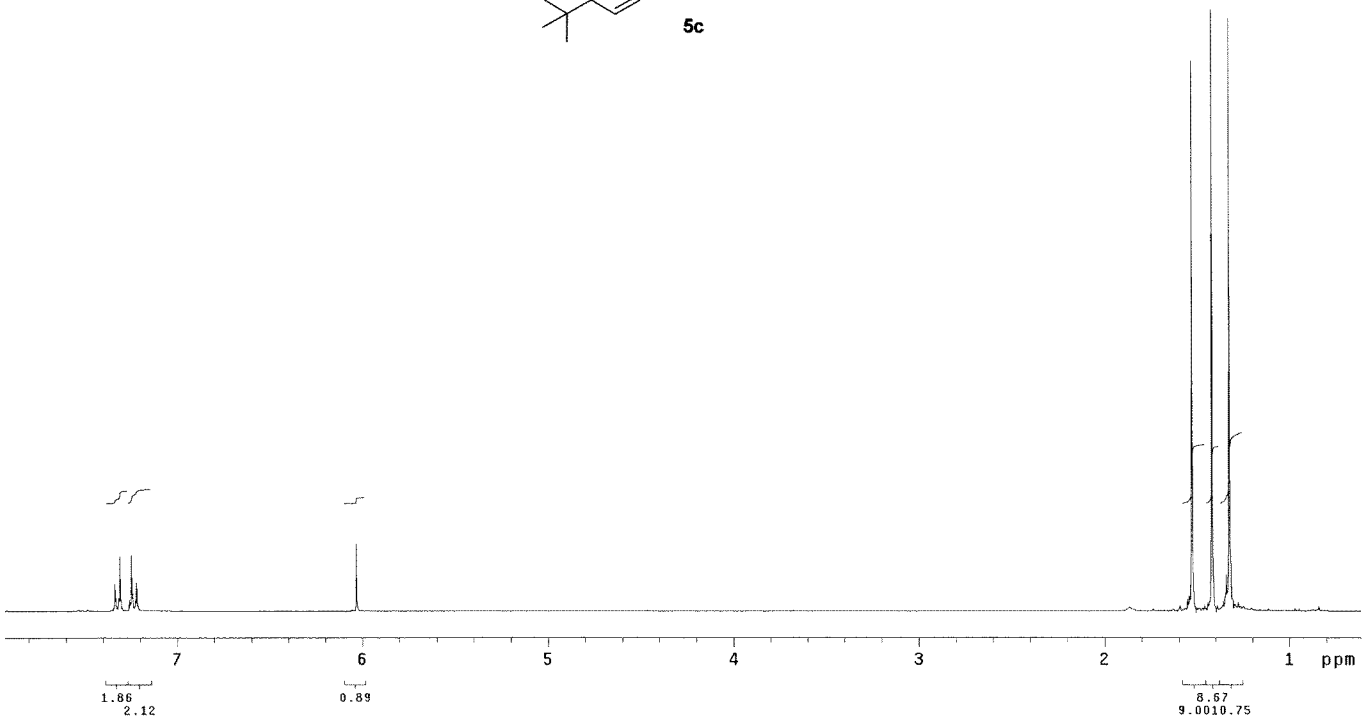
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234F-H
INOVA-500 "epoxide"

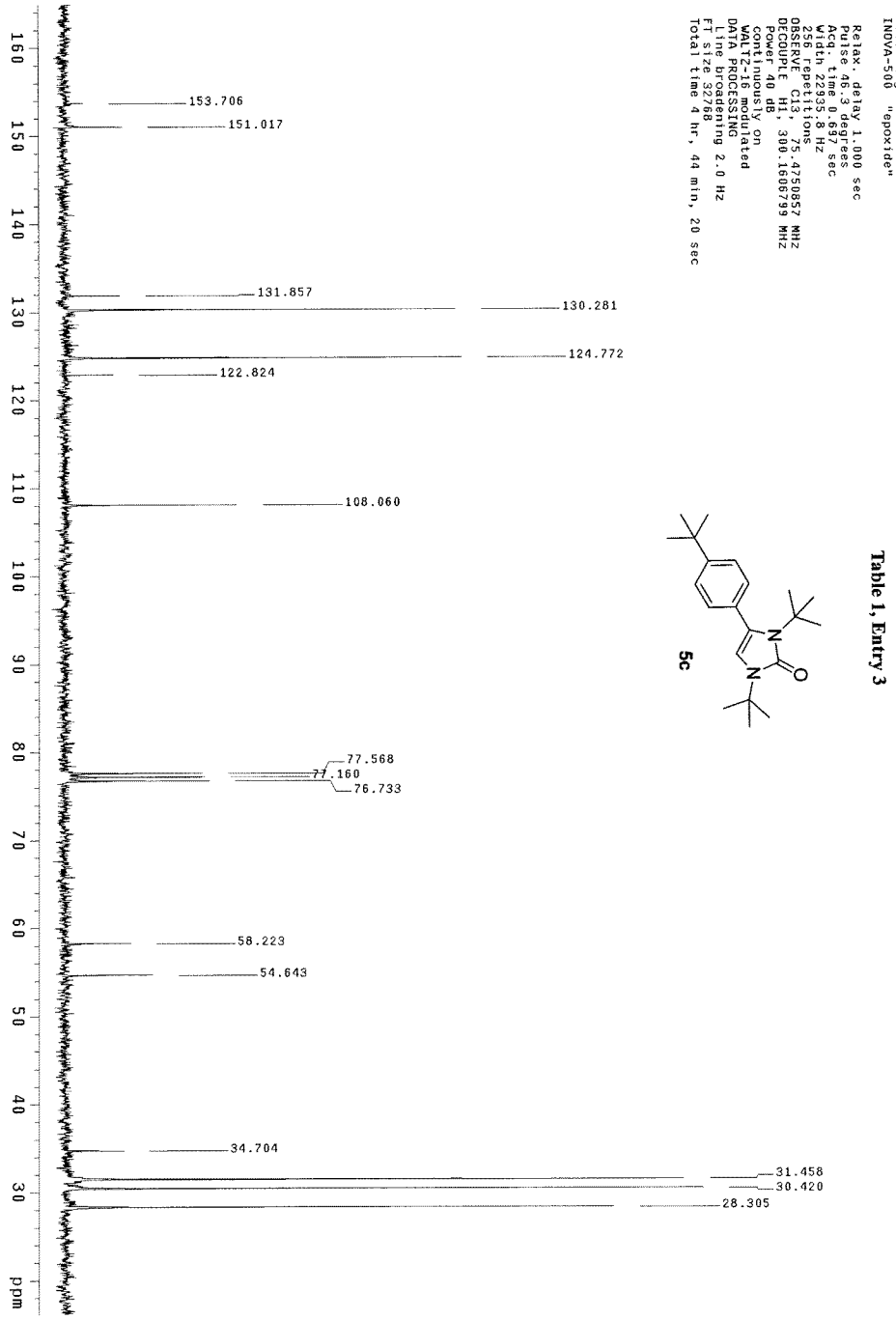
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
2 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 10 sec

Table 1, Entry 3



5c



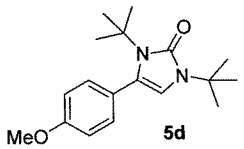

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234f-C13
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
256 repetitions
OBSERVE C13, 75.4750857 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 4 hr, 44 min, 20 sec
153.706
151.017
131.857
130.281
124.772
122.824
108.060
77.568
77.160
76.733
58.223
54.643
34.704
31.458
30.420
28.305
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm
5c
Table 1, Entry 3

STANDARD 1H OBSERVE

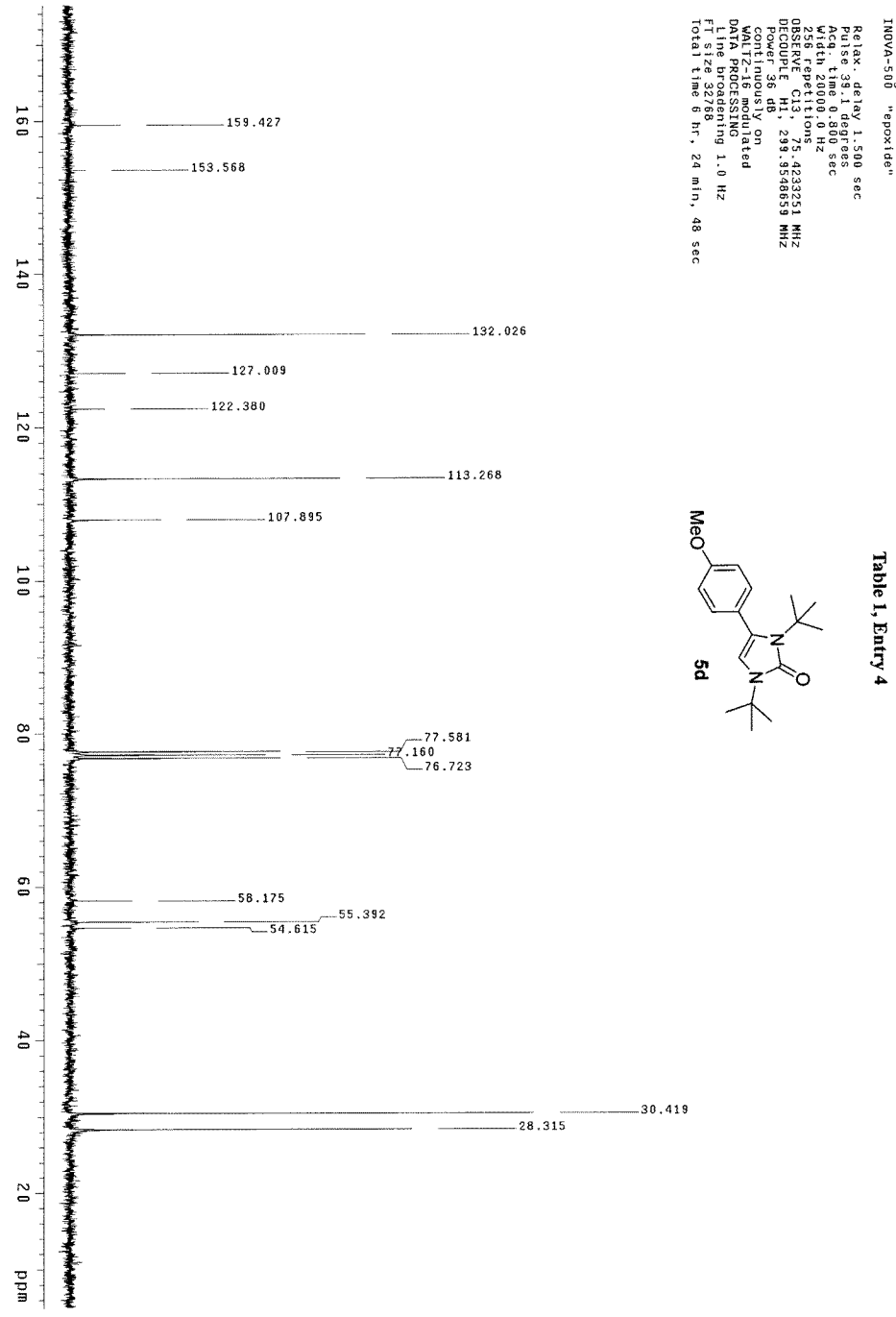
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234E-H2
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 4



5d



5d

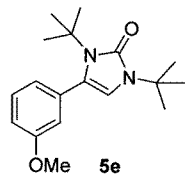
Table 1, Entry 4

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237H-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533695 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 5



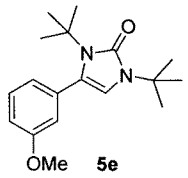
5e

13C OBSERVE

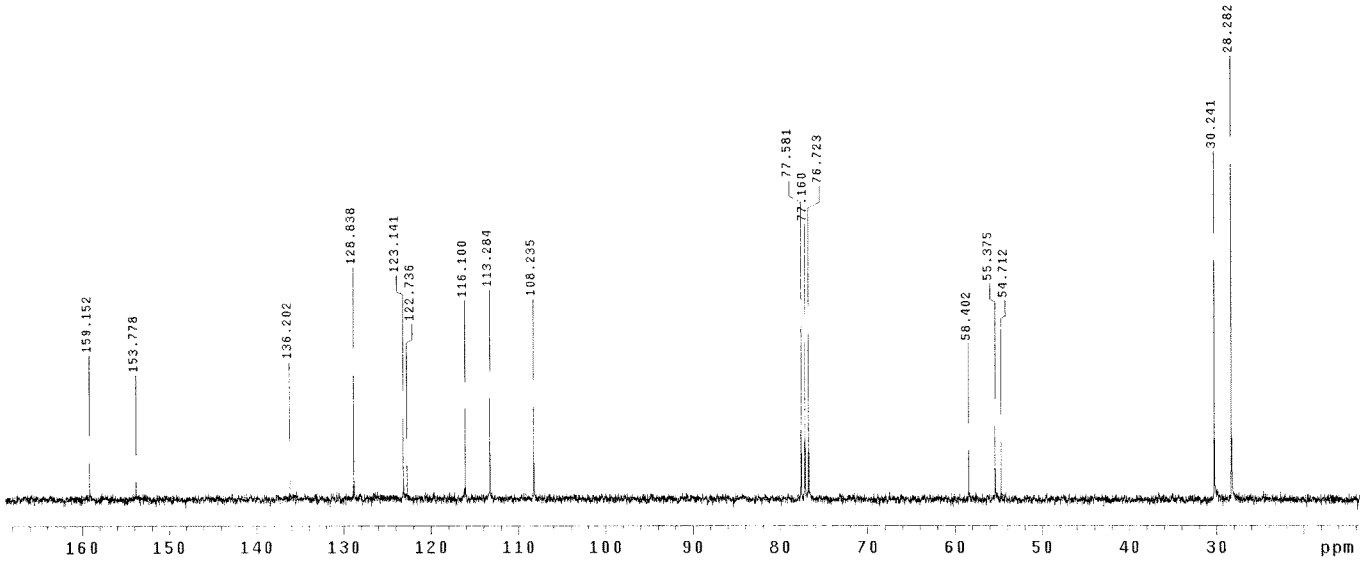
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237H-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
192 repetitions
OBSERVE C13, 75.4233251 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec

Table 1, Entry 5



5e

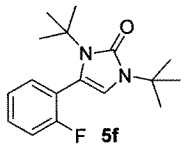


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234J-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 6



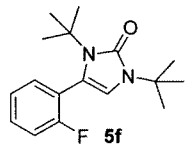
5f

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234J-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
480 repetitions
OBSERVE C13, 75.4233239 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec

Table 1, Entry 6



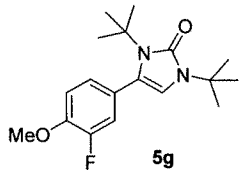
5f

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg12370-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533696 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 7



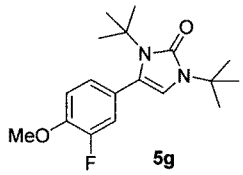
5g

13C OBSERVE

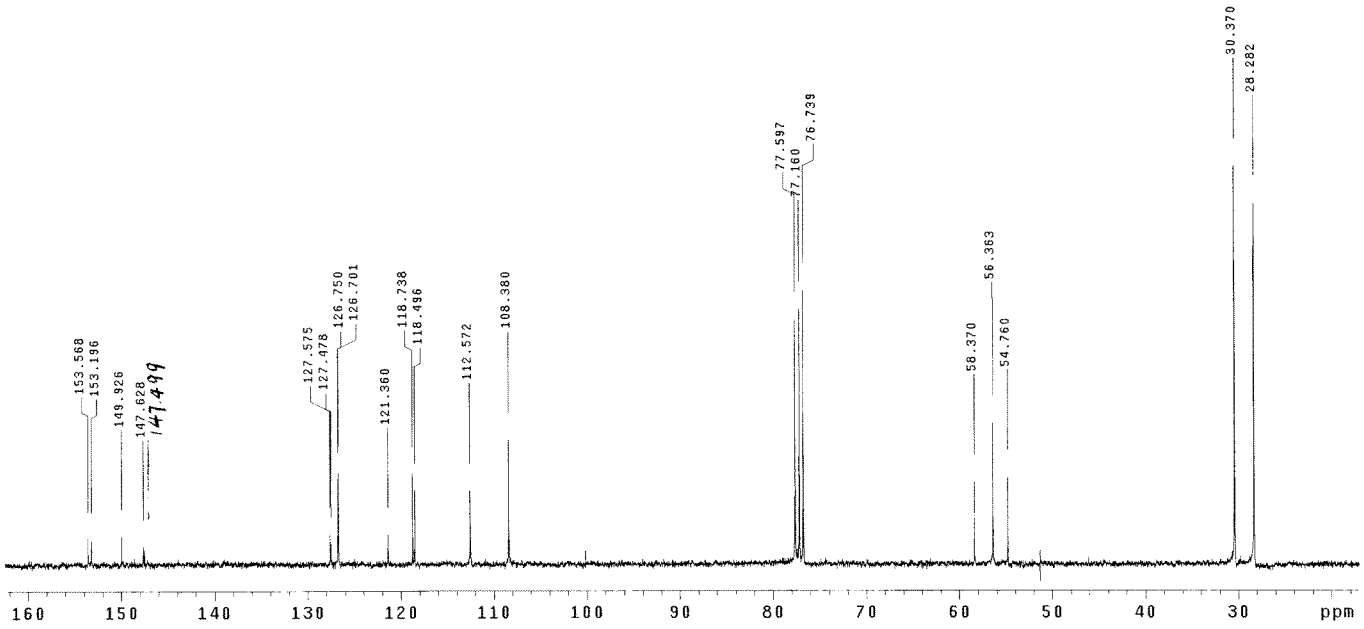
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237D-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
1568 repetitions
OBSERVE C13, 75.4233239 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec

Table 1, Entry 7



5g

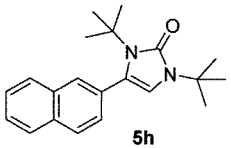


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237C-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533718 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 8



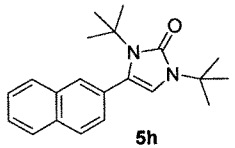
5h

13C OBSERVE

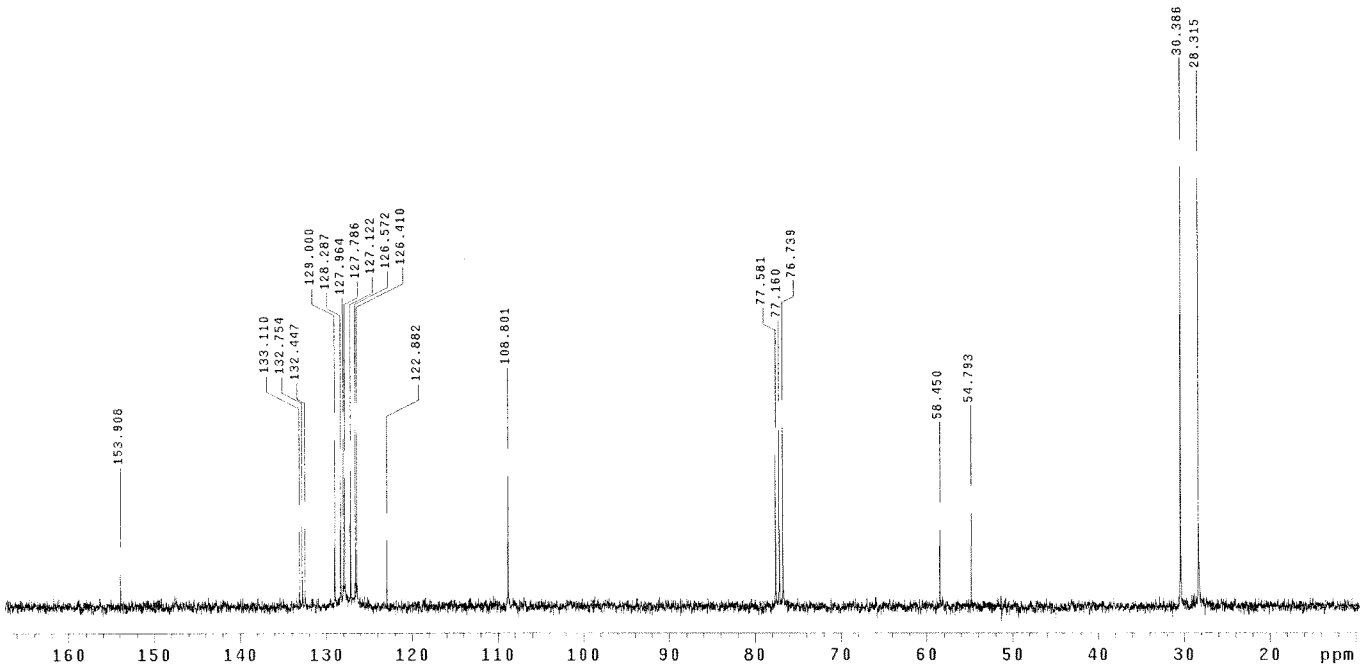
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237C-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
224 repetitions
OBSERVE C13, 75.4233251 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec

Table 1, Entry 8



5h

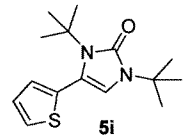


STANDARD 1H OBSERVE

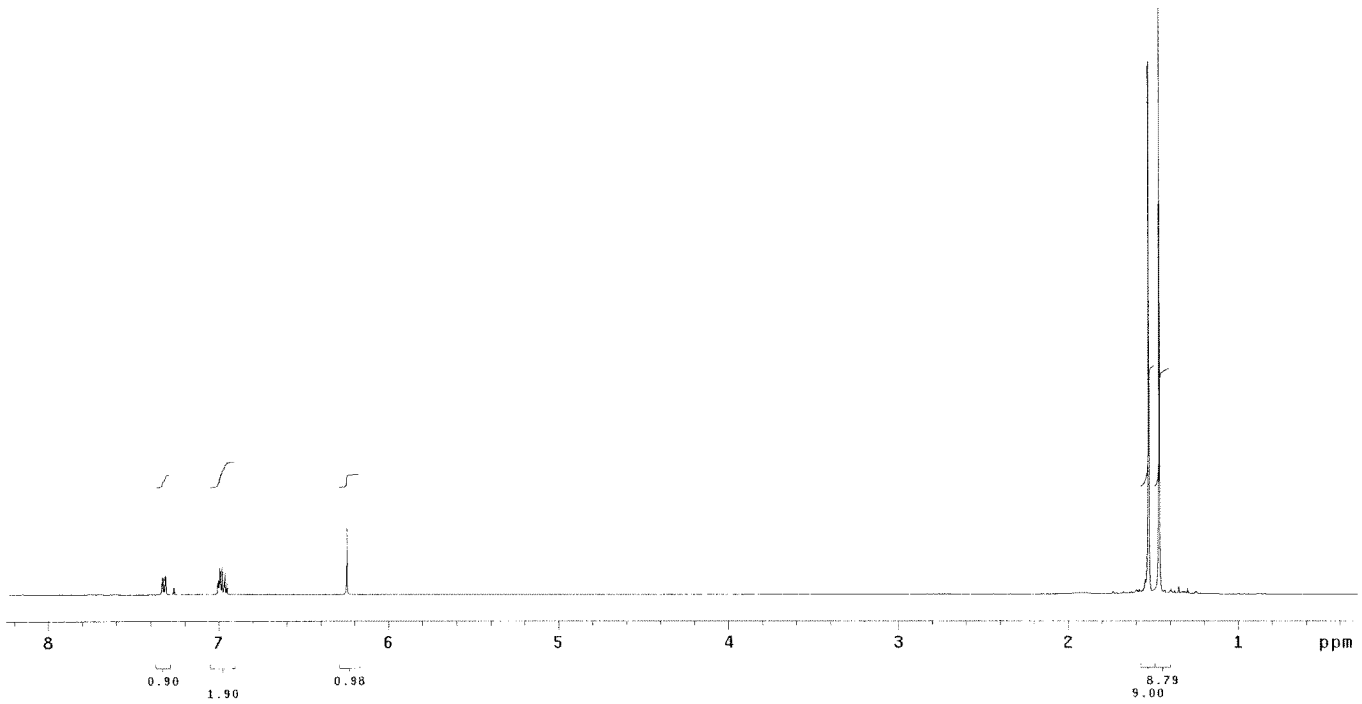
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237M-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533691 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 9



5i

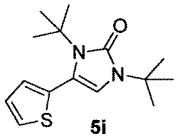


13C OBSERVE

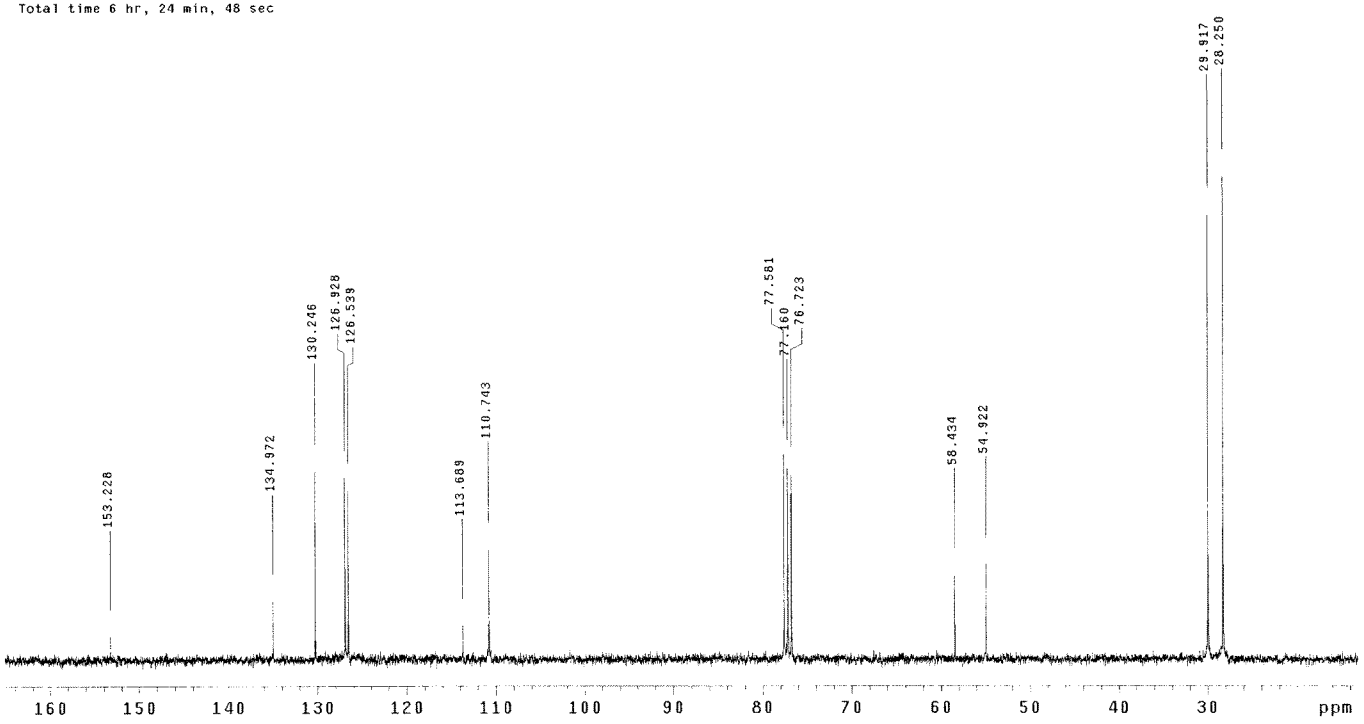
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1237M-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
208 repetitions
OBSERVE C13, 75.4233263 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec

Table 1, Entry 9



5i

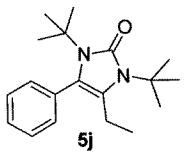


STANDARD 1H OBSERVE

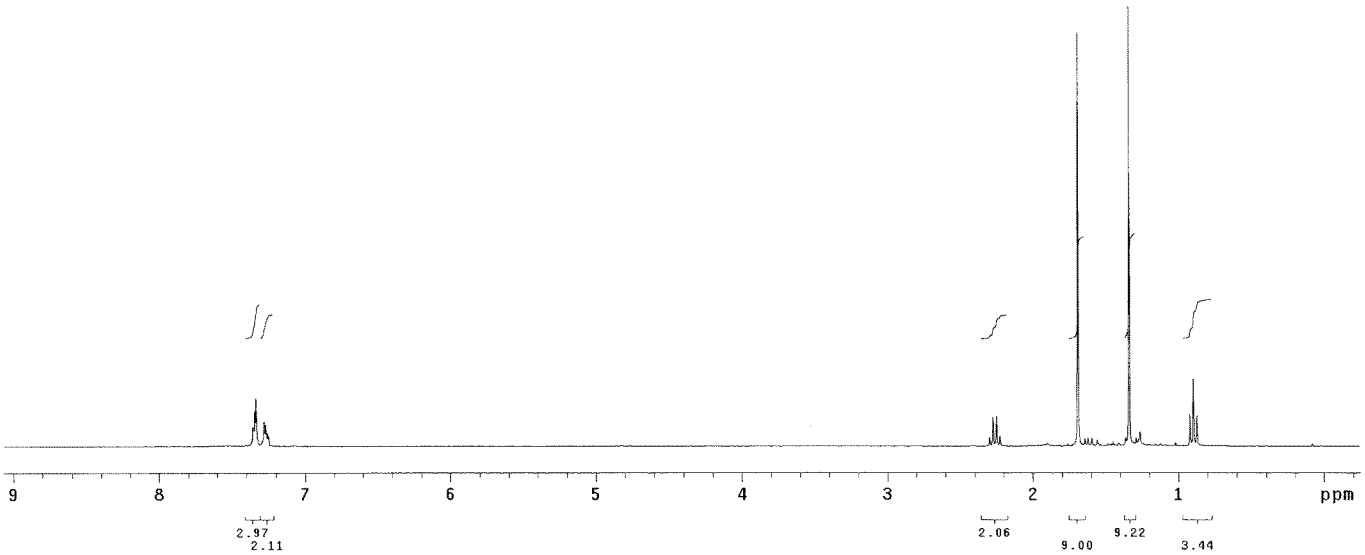
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234D-H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
2 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 14 sec

Table 1, Entry 10



5j

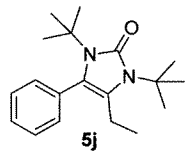


13C OBSERVE

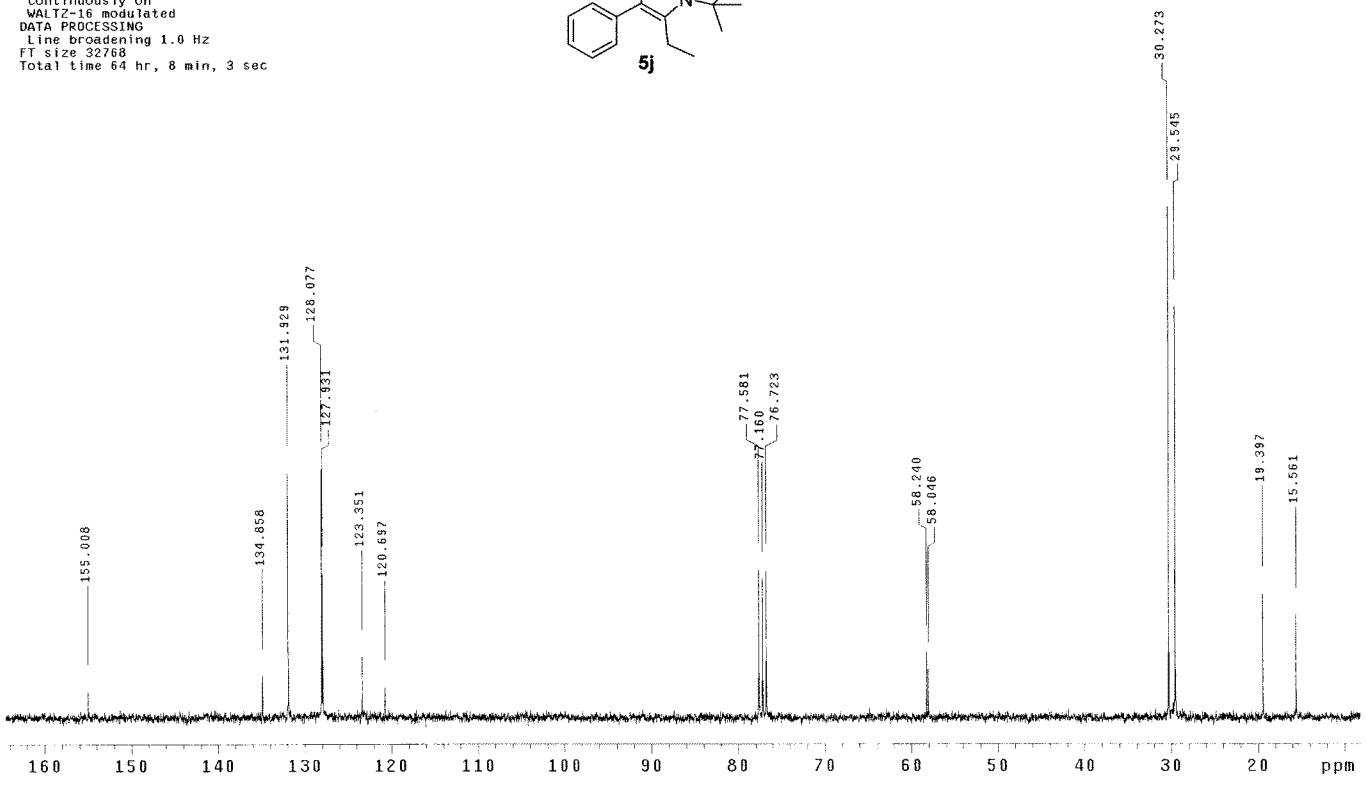
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: zbg1234D-C13
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
256 repetitions
OBSERVE C13, 75.4233251 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 64 hr, 8 min, 3 sec

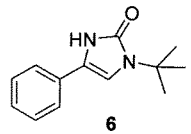
Table 1, Entry 10



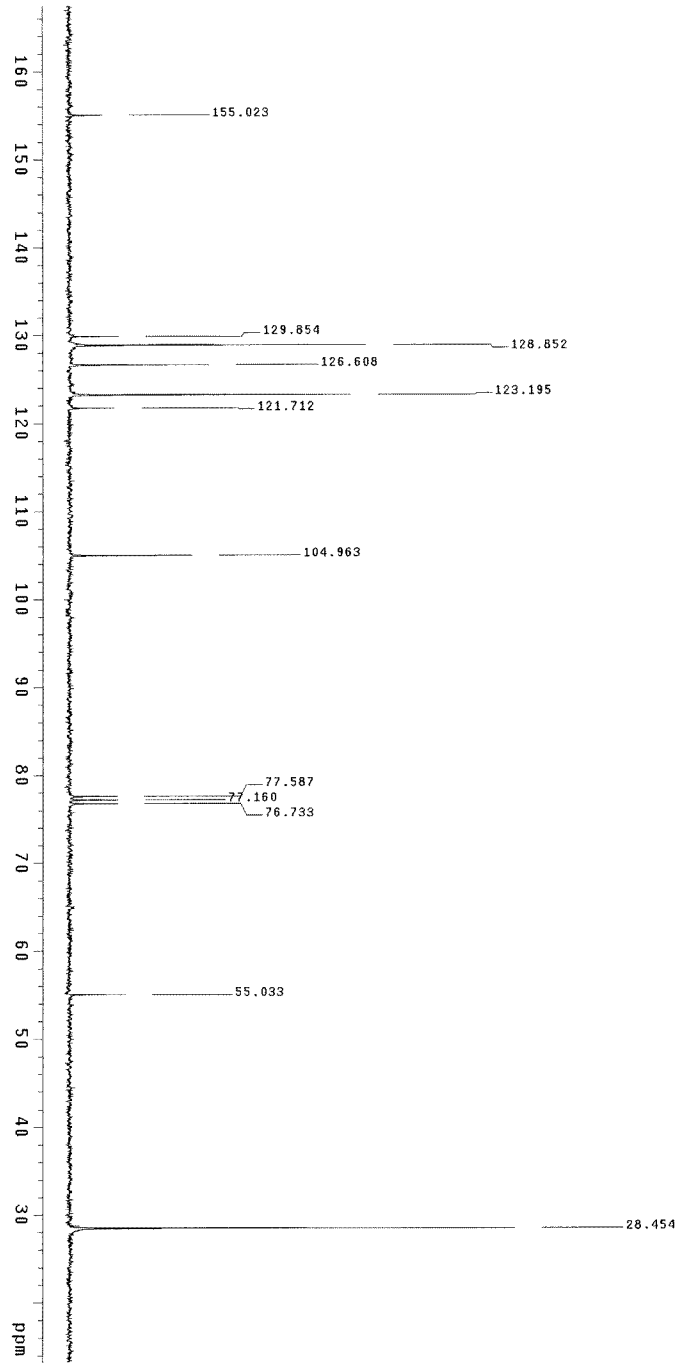
5j

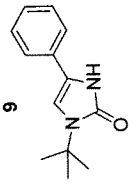


Scheme 2



6


155.023
129.854
128.852
126.608
123.195
121.712
104.963
77.587
77.160
76.733
55.033
28.454
ppm



6

Scheme 2