Supporting Information

Lewis Acid-Catalyzed Rearrangement of Vinylcyclopropenes for the Construction of Naphthalene and Indene Skeletons

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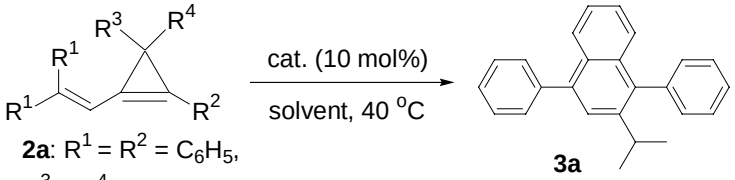
CONTENTS

General Remarks. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury vx-300 spectrometer for solution in $CDCl_3$ with tetramethylsilane (TMS) as an internal standard; *J*-values are in Hz. Mass spectra were recorded by EI, and HRMS was measured on a Micromass GCT mass spectrometer. CHN microanalyses were recorded on a Elementar Vario EL III analyzer. THF and toluene were distilled from Na under Ar atmosphere. 1,2-Dichloroethane was distilled from CaH_2 under Ar atmosphere. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with Huanghai GF_{254} silica gel coated plates. Flash column chromatography was carried out using 300-400 mesh silica gel at increased pressure.

Optimization for the Formation of Products 3a and 4a:

Table SI-I. Optimization for the formation of naphthalene **3a**.



2a: $R^1 = R^2 = C_6H_5$, $R^3 = R^4 = Me$

3a

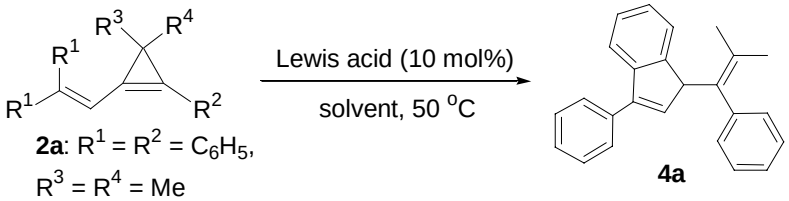
entry[a]	solvent	cat.	time (h)	yield (%)[b]
1	DCE	none	120	NR
2	**DCE**	**BF_3·OEt_2**	**5**	**60**
3	DCE	TfOH	5	56
4	DCE	HBr	5	56
5	DCE	CF_3CO_2H	5	48[c]
6	DCE	CH_3CO_2H	5	NR
7	DMF	BF_3·OEt_2	120	NR
8	Toluene	BF_3·OEt_2	5	53
9	THF	BF_3·OEt_2	120	NR
10	Hexane	BF_3·OEt_2	10	48
11	Ethanol	BF_3·OEt_2	120	NR

[a] All reactions were carried out using **2a** (0.2 mmol) in the presence of the listed catalysts (10 mol%) and solvent (5.0 mL).

[b] Isolated yields.

[c] Along with the corresonding indene product in 24% yield.

Table SI-II. Optimization for the formation of indene **4a**.



2a: $R^1 = R^2 = C_6H_5$, $R^3 = R^4 = Me$

4a

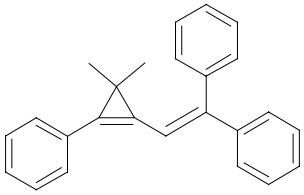
entry[a]	solvent	Lewis acid	time (h)	yield (%)[a]
1	**DCE**	**$Cu(OTf)_2$**	**5**	**98**
2	DCE	$Sc(OTf)_3$	5	70
3	DCE	$Sn(OTf)_2$	5	70
4	DCE	$Eu(OTf)_3$	5	74
5	DCE	$Zr(OTf)_4$	5	70
6	DMF	$Cu(OTf)_2$	120	NR
7	Toluene	$Cu(OTf)_2$	5	72
8	THF	$Cu(OTf)_2$	5	52
9	Hexane	$Cu(OTf)_2$	5	62
10	Ethanol	$Cu(OTf)_2$	5	67

[a] All reactions were carried out using **2a** (0.2 mmol) in the presence of the listed catalysts (10 mol%) and solvent (5.0 mL).
[b] Isolated yields.

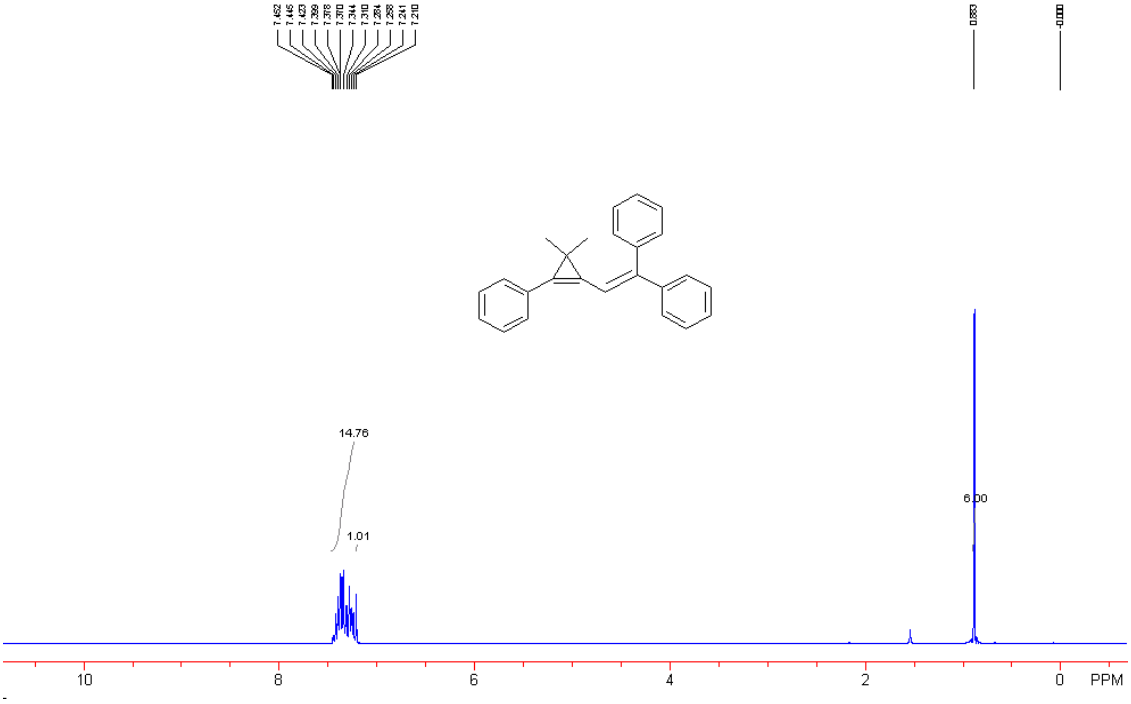
Typical Reaction Procedure for the Preparation of Vinylcyclopropenes: A mixture of Bu_4NHSO_4 (0.4 mmol) and powered NaOH (160 mg, 4 mmol) was added to a toluene (5.0 mL) solution containing vinylidenecyclopropane **1a** (0.2 mmol). The mixture was vigorously stirred at 60 ^oC for about 5 hrs. Flash column chromatography of the resulting mixture on silica gel gave the product **2a** (92%) as a yellow oil.

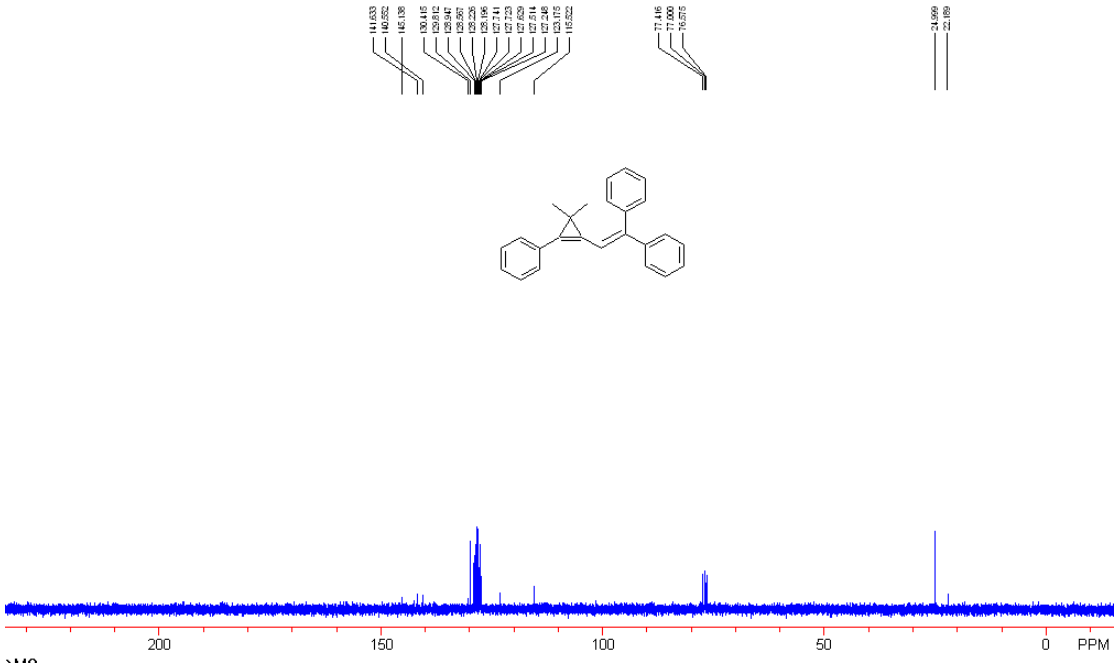
Typical Reaction Procedure for the Rearrangement of Vinylcyclopropenes 2 to Naphthalenes 3: BF_3 OEt_2 (10 mol%) was added to a 1,2-dichloroethane (5.0 mL) solution containing vinylcyclopropene **2a** (0.2 mmol). The mixture was vigorously stirred at 40 ^oC for about 5 hrs. Flash column chromatography of the resulting mixture on silica gel gave the product **3a** (75%) as a white solid.

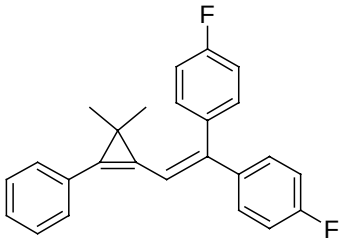
Typical Reaction Procedure for the Rearrangement of Vinylcyclopropenes 2 to Indenes 4: $Cu(OTf)_2$ (10 mol%) was added to a 1,2-dichloroethane (5.0 mL) solution containing vinylcyclopropene **2a** (0.2 mmol). The mixture was vigorously stirred at 50 ^oC for about 5 hrs. Flash column chromatography of the resulting mixture on silica gel gave the product **4a** (98%) as a yellow solid.



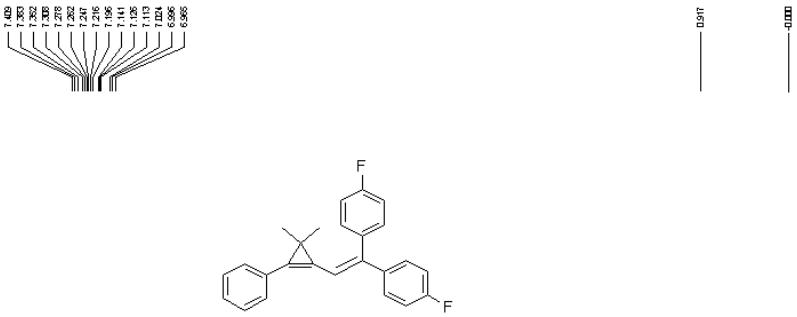
Compound **2a**. A yellow oil. IR (CH_2Cl_2): ν 3075, 3056, 3024, 2959, 2923, 2854, 1771, 1595, 1494, 1485, 1444, 1362, 1072, 1029, 878 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.88 (6H, s, CH_3), 7.21 (1H, s, =CH), 7.24-7.45 (15H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.2, 25.0, 115.5, 123.2, 127.2, 127.5, 127.6, 127.72, 127.74, 128.20, 128.23, 128.6, 128.9, 129.8, 130.4, 140.6, 141.6, 145.1; MS (EI) *m/z* (%): 322 (21) [M^+], 307 (100), 292 (28), 229 (33), 215 (24); HRMS (EI) Calcd. for $C_{25}H_{22}$ (M^+) requires 322.1722, Found: 322.1718.



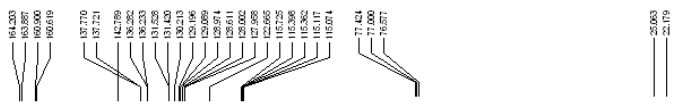


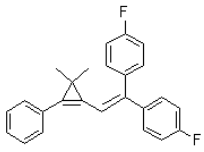


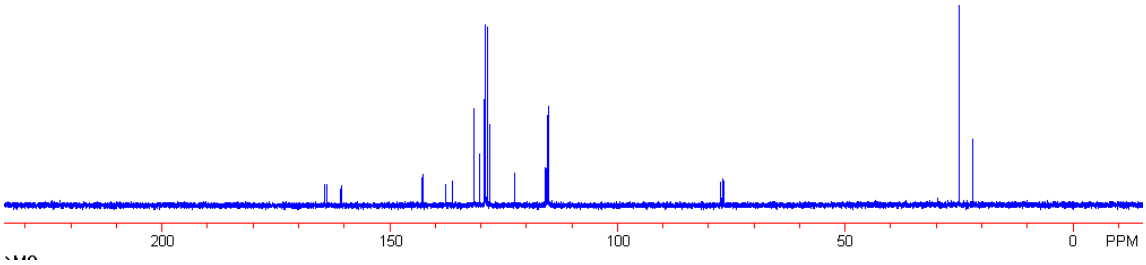
Compound **2b**. A yellow solid, m.p. 118.3-118.8 °C. IR (CH_2Cl_2): ν 2955, 2910, 2846, 1601, 1509, 1232, 1158, 834 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.92 (6H, s, CH_3), 6.97-7.41 (14H, m, =CH + ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.2, 25.1, 115.2 (d, J_{C-F} = 21.6 Hz), 115.3 (d, J_{C-F} = 21.1 Hz), 115.7, 122.7, 127.96, 128.00, 128.6, 129.0, 129.1 (d, J_{C-F} = 8.0 Hz), 130.2, 131.5 (d, J_{C-F} = 8.1 Hz), 136.3 (d, J_{C-F} = 3.7 Hz), 137.7 (d, J_{C-F} = 3.7 Hz), 142.8, 162.3 (d, J_{C-F} = 245.1 Hz), 162.6 (d, J_{C-F} = 247.7 Hz); MS (EI) *m/z* (%): 358 (21) [M^+], 343 (100), 131 (61); Anal. Calcd. for $C_{25}H_{20}F_2$ requires C, 83.77; H, 5.62%. Found: C, 84.00; H, 5.37%.

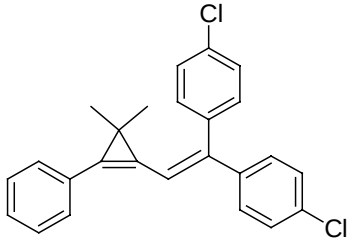


14.29
6.00
10
8
6
4
2
0
PPM

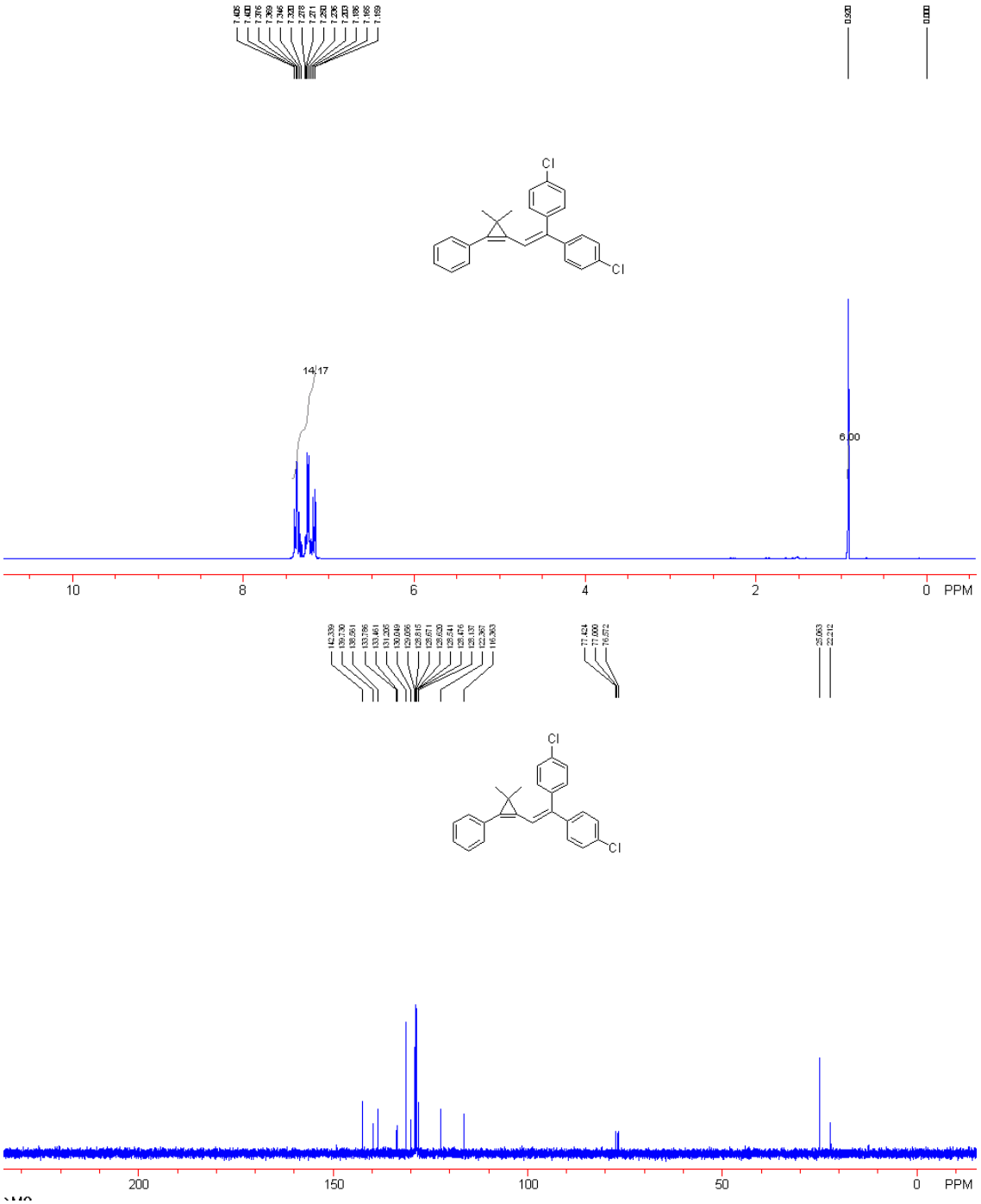


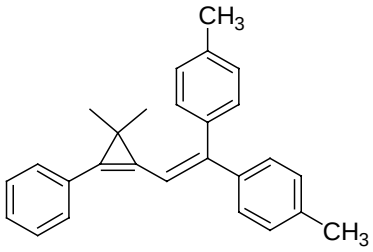



200
150
100
50
0
PPM

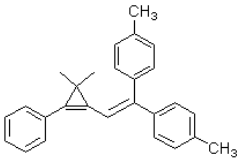


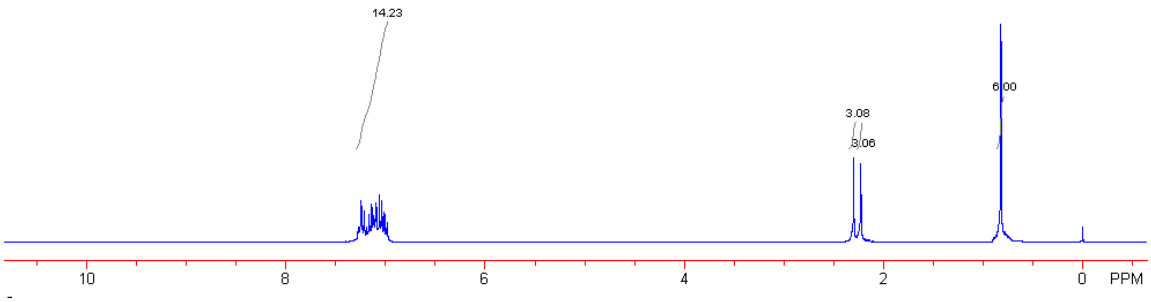
Compound **2c**. A yellow oil. IR (CH_2Cl_2): ν 2924, 2854, 1492, 1485, 1462, 1092, 1015, 828 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.92 (6H, s, CH_3), 7.16-7.41 (14H, m, =CH + ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.2, 25.1, 116.4, 122.4, 128.1, 128.48, 128.54, 128.6, 128.7, 128.8, 129.1, 130.0, 131.2, 133.5, 133.8, 138.6, 139.7, 142.3; MS (EI) *m/z* (%): 390 (19) [M^+], 375 (49), 355 (100), 340 (66), 325 (45); HRMS (EI) Calcd. for $C_{25}H_{20}Cl_2$ (M^+) requires 390.0942, Found: 390.0938.

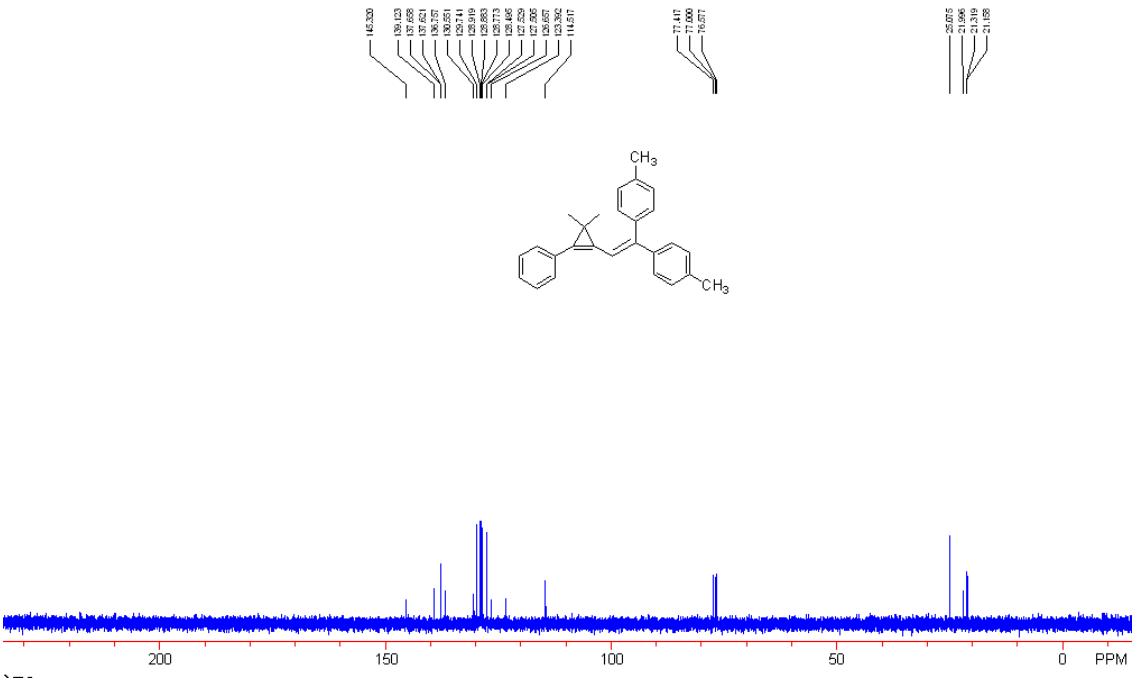


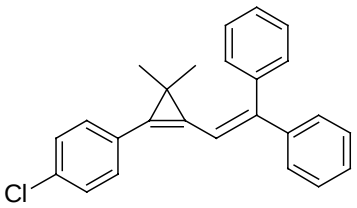


Compound **2d**. A yellow oil. IR (CH_2Cl_2): ν 3023, 2958, 2921, 2853, 1486, 1446, 1361, 1275, 1261, 817 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.82 (6H, s, CH_3), 2.23 (3H, s, CH_3), 2.30 (3H, s, CH_3), 6.98-7.27 (14H, m, =CH + ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.2, 21.3, 22.0, 25.1, 114.5, 123.4, 126.7, 127.51, 127.53, 128.5, 128.8, 128.88, 128.92, 129.7, 130.6, 136.8, 137.6, 137.7, 139.1, 145.3; MS (EI) *m/z* (%): 350 (27) [M^+], 335 (100), 320 (33), 219 (26); HRMS (EI) Calcd. for $C_{27}H_{26}$ (M^+) requires 350.2023, Found: 350.2020.

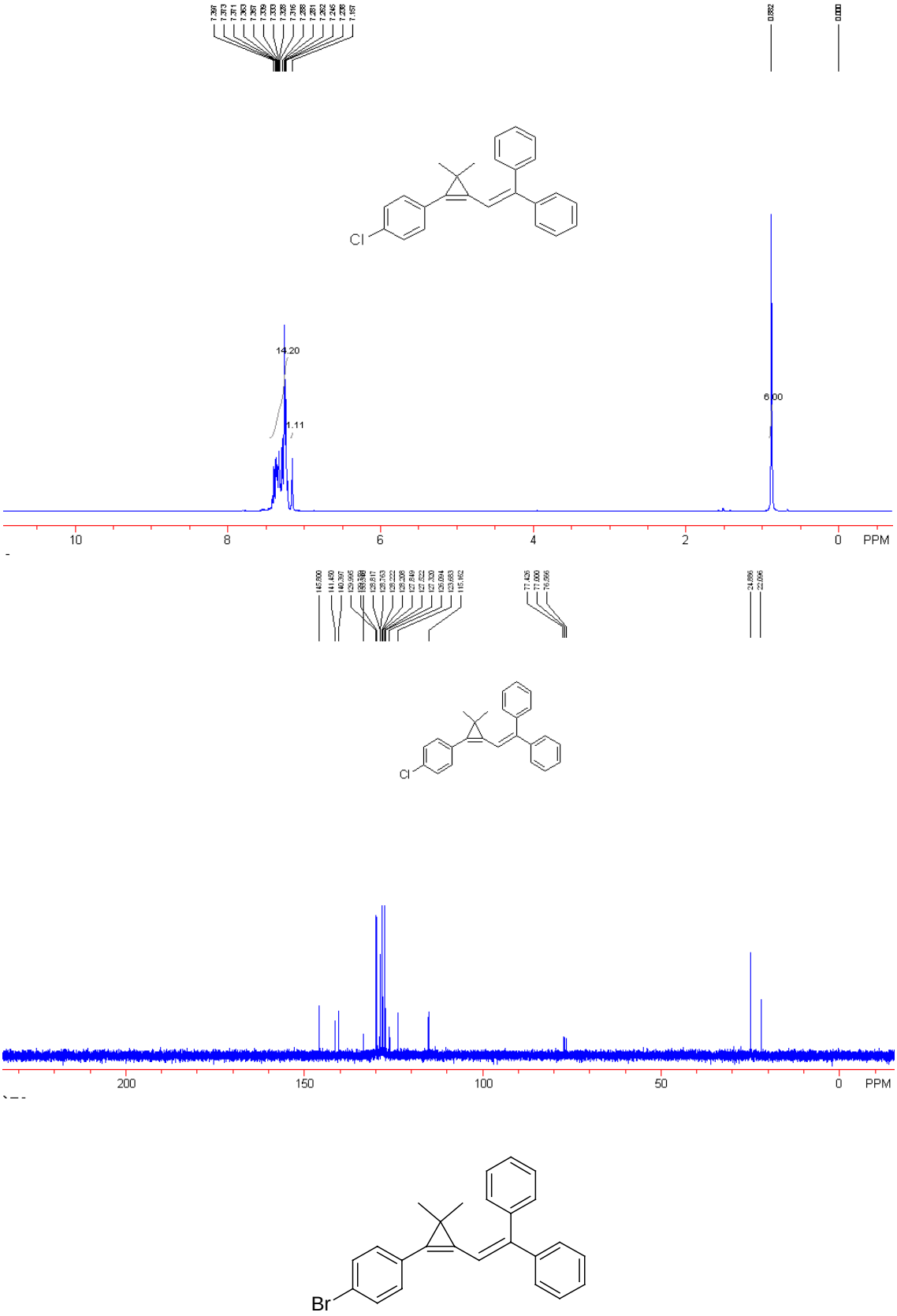






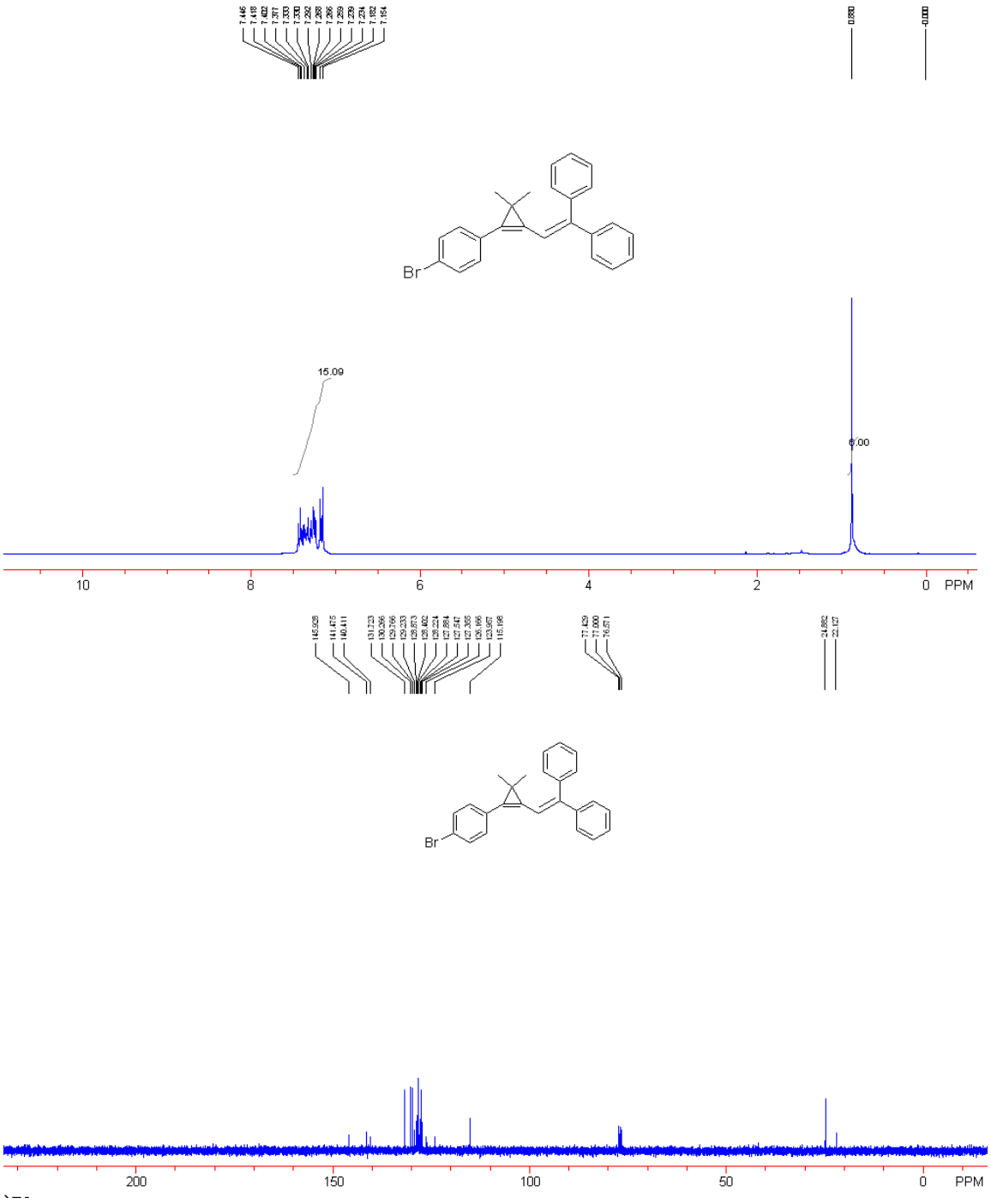


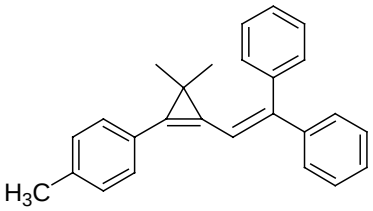
Compound **2e**. A yellow oil. IR (CH_2Cl_2): ν 3057, 3025, 2961, 2934, 2872, 2854, 1763, 1736, 1662, 1588, 1484, 1445, 1400, 1362, 1278, 828 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.88 (6H, s, CH_3), 7.16 (1H, s, =CH) 7.24-7.39 (14H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.1, 24.9, 115.2, 123.7, 126.1, 127.3, 127.5, 127.8, 128.21, 128.22, 128.76, 128.82, 129.8, 130.0, 133.3, 140.4, 141.5, 145.8; MS (EI) *m/z* (%): 356 (23) [M^+], 341 (100), 291 (41), 229 (40); HRMS (EI) Calcd. for $C_{25}H_{21}Cl$ (M^+) requires 356.1332, Found: 356.1305.



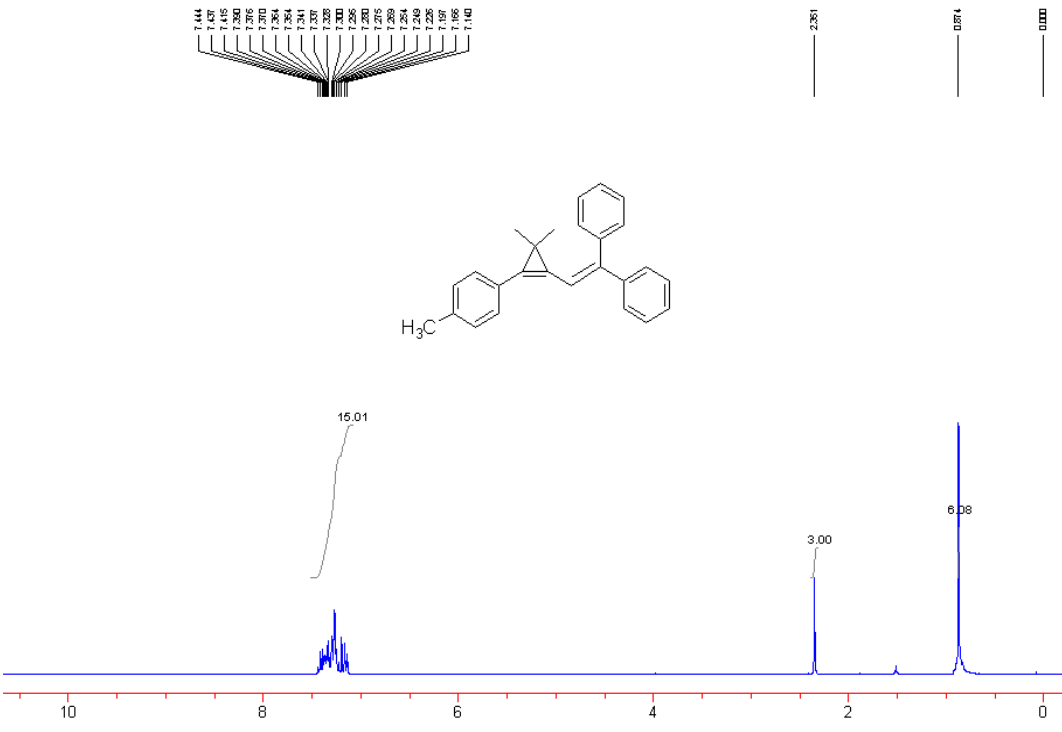
Compound **2f**. A yellow oil. IR (CH_2Cl_2): ν 3079, 3058, 3025, 2924, 2854, 1756, 1596, 1494,

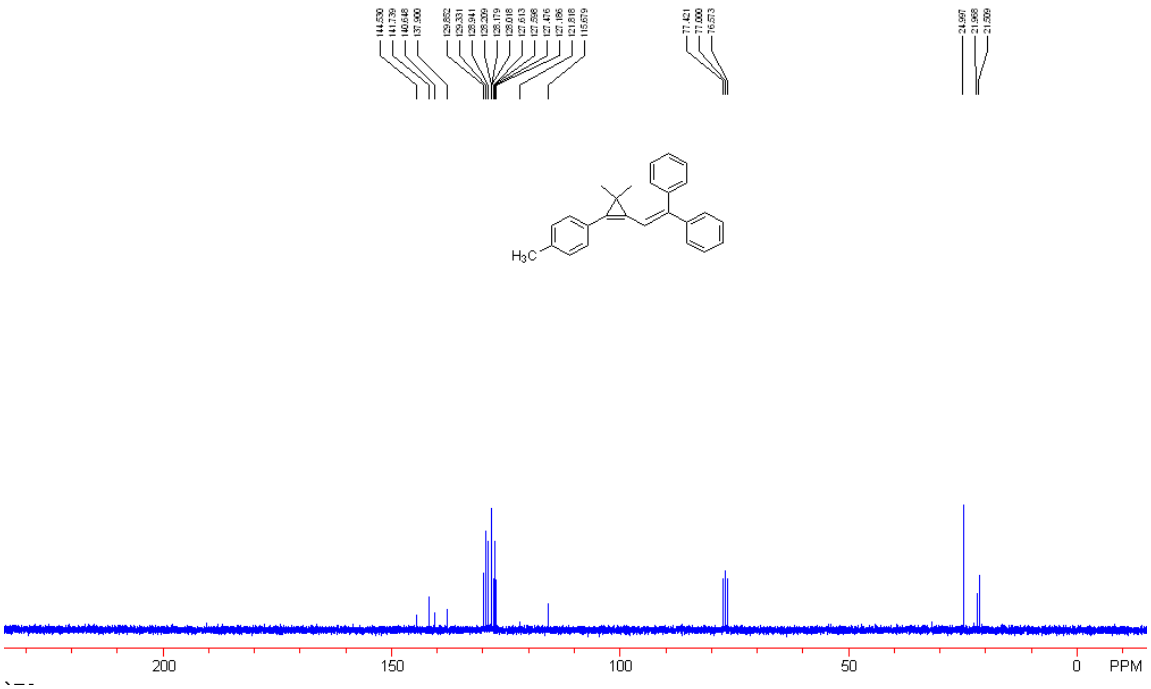
1480, 1444, 1396, 1362, 1068, 1009, 823 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 0.88 (6H, s, CH_3), 7.15-7.45 (15H, m, =CH + ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.1, 24.9, 115.2, 124.0, 126.2, 127.4, 127.5, 127.9, 128.2, 128.4, 128.9, 129.2, 129.8, 130.3, 131.7, 140.4, 141.5, 145.9; MS (EI) *m/z* (%): 400 (13) [M^+], 385 (66), 291 (41), 306 (100), 291 (62), 229 (36); HRMS (EI) Calcd. for $C_{25}H_{21}Br$ (M^+) requires 400.0827, Found: 400.0850.

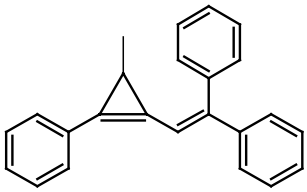




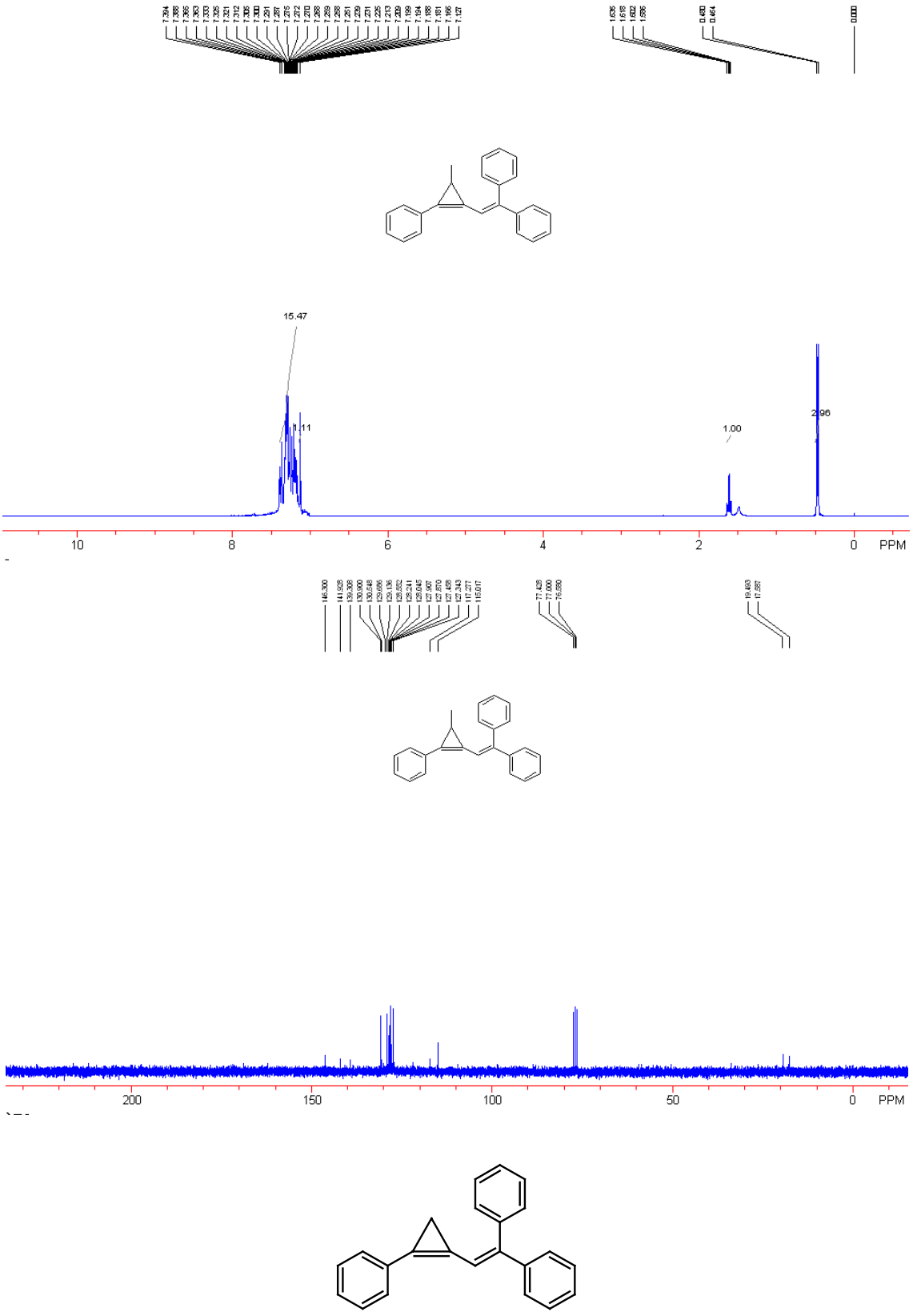
Compound **2g**. A yellow oil. IR (CH_2Cl_2): ν 3022, 2957, 2923, 2854, 1738, 1504, 1444, 1361, 1229, 1217, 968, 817 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.87 (6H, s, CH_3), 2.35 (3H, s, CH_3), 7.14-7.44 (15H, m, =CH + ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.5, 22.0, 25.0, 115.7, 121.8, 127.2, 127.5, 127.60, 127.61, 128.0, 128.18, 128.21, 128.9, 129.3, 129.9, 137.9, 140.6, 141.7, 144.5; MS (EI) *m/z* (%): 336 (23) [M^+], 321 (100), 229 (27), 145 (63); HRMS (EI) Calcd. for $C_{26}H_{24}$ (M^+) requires 336.1878, Found: 336.1841.





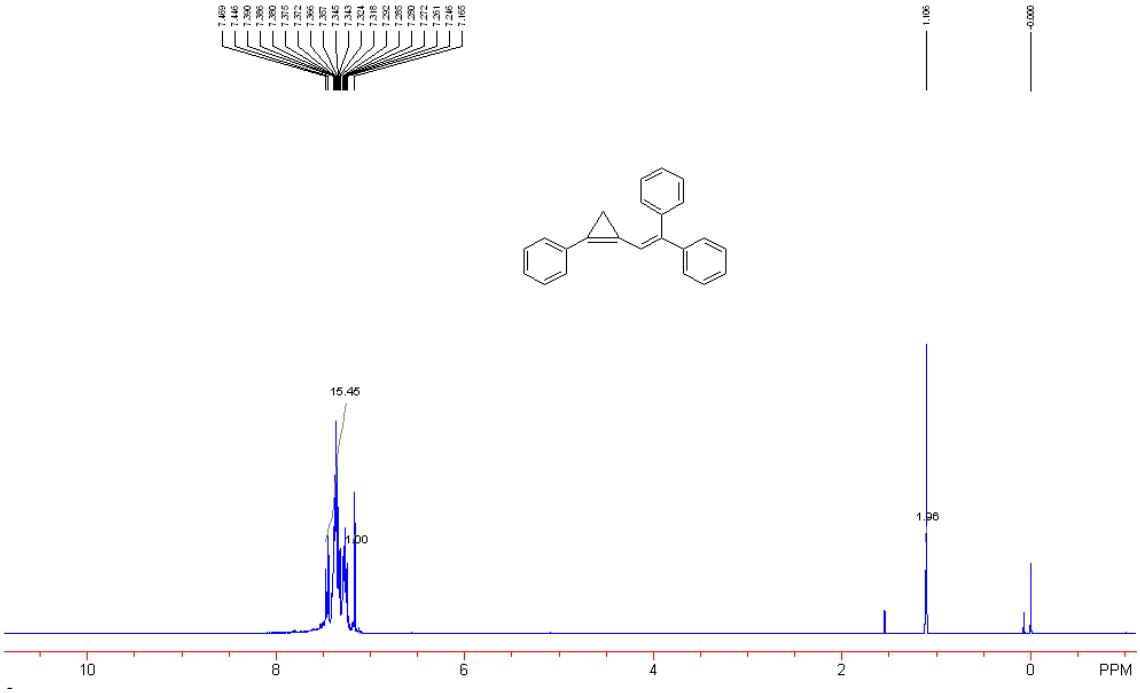


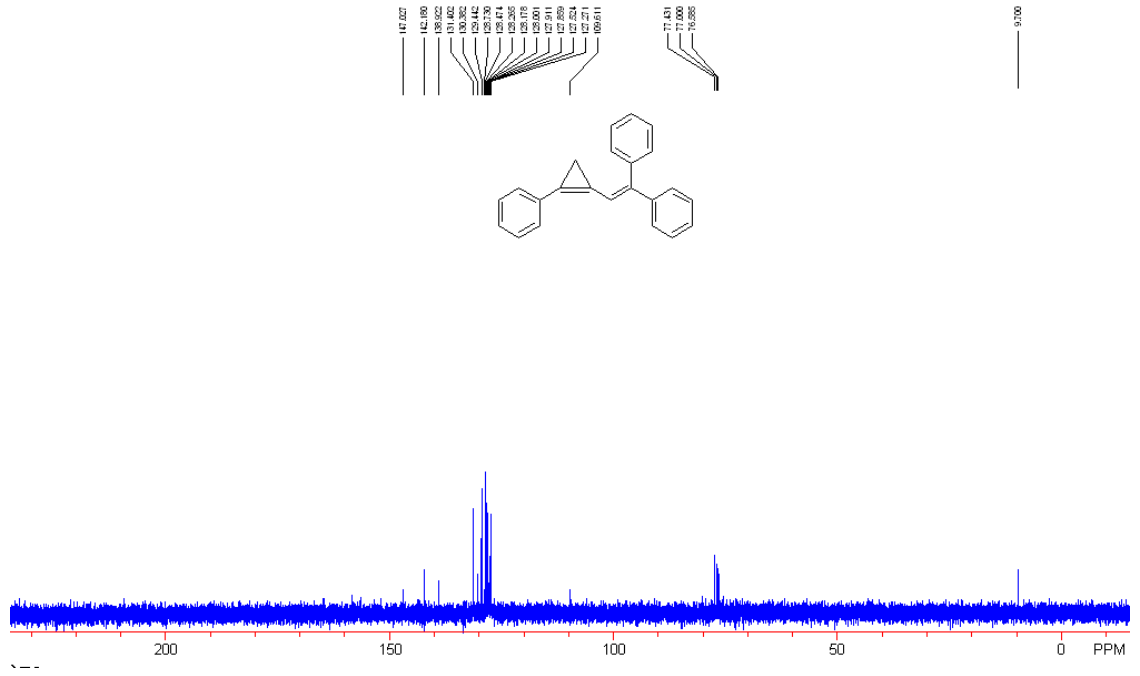
Compound **2h**. A yellow oil. IR (CH_2Cl_2): ν 3051, 3019, 2955, 2854, 1661, 1598, 1494, 1445, 1277, 764 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 0.47 (3H, d, *J* = 4.8 Hz, CH_3), 1.61 (1H, q, *J* = 4.8 Hz), 7.13 (1H, s, =CH), 7.17-7.39 (15H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 17.6, 19.5, 115.0, 117.3, 127.3, 127.5, 127.87, 127.91, 128.0, 128.2, 128.6, 129.1, 129.7, 130.5, 130.9, 139.3, 141.9, 146.3; MS (EI) *m/z* (%): 308 (27) [M^+], 293 (100), 215 (50); HRMS (EI) Calcd. for $C_{24}H_{20}$ (M^+) requires 308.1565, Found: 308.1572.

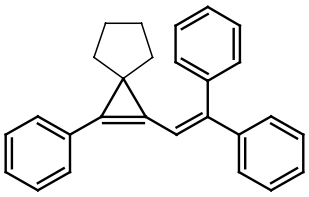


Compound **2i**. A yellow oil. IR (CH_2Cl_2): ν 3051, 3019, 2955, 2923, 2846, 1667, 1588, 1445,

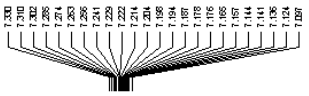
1399, 1362, 1277, 1090, 1012, 973, 828, 764 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.11 (2H, s, CH_2), 7.17 (1H, s, =CH), 7.25-7.47 (15H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 9.7, 115.1, 127.3, 127.5, 127.86, 127.91, 128.0, 128.2, 128.3, 128.5, 128.7, 129.4, 130.4, 131.4, 138.9, 142.2, 147.0; MS (EI) *m/z* (%): 294 (95) [M^+], 279 (46), 217 (69), 215 (100), 202 (44), 103 (84); HRMS (EI) Calcd. for $C_{23}H_{18}$ (M^+) requires 294.1409, Found: 294.1404.

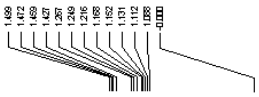


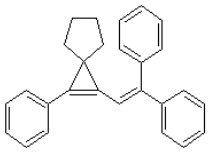


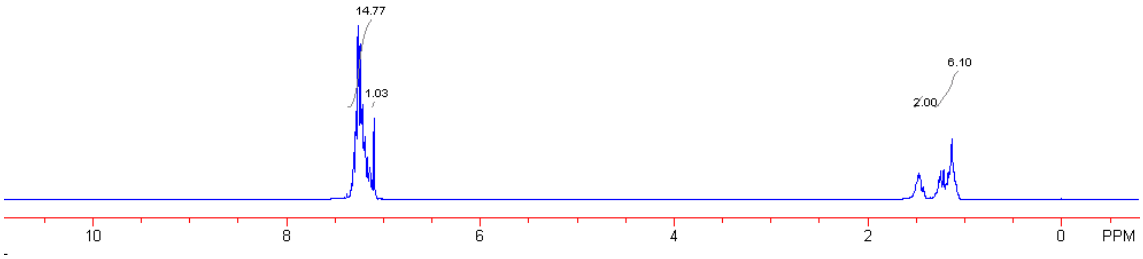


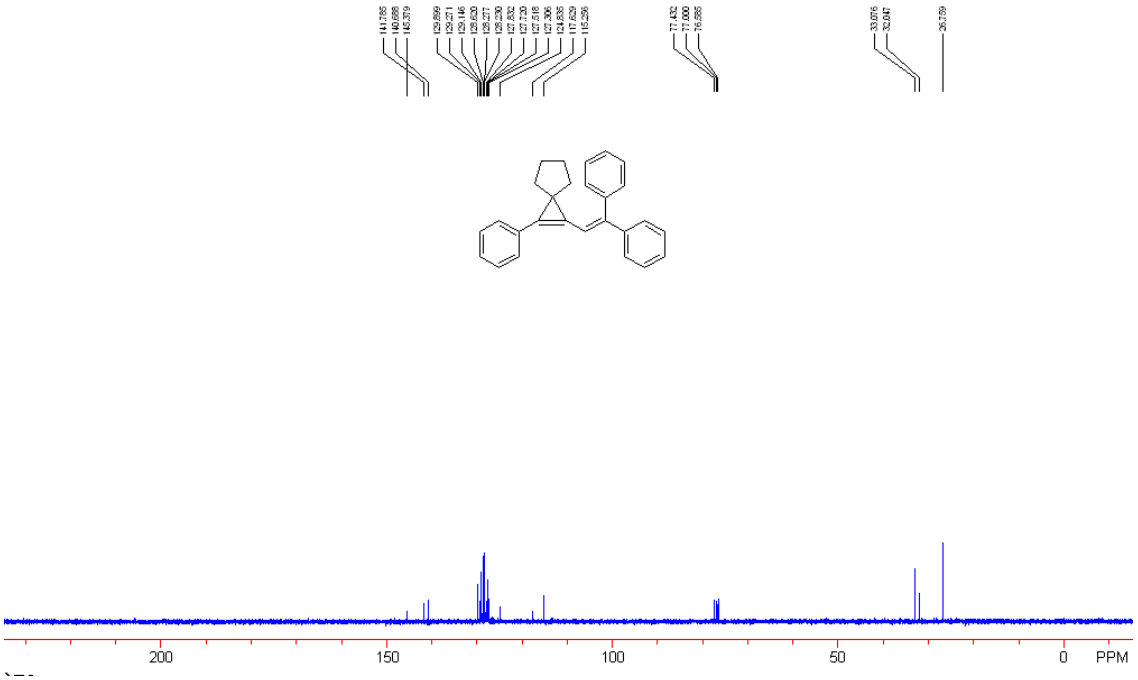
Compound **2j**. A yellow oil. IR (CH_2Cl_2): ν 3051, 3019, 2955, 2923, 2854, 1661, 1598, 1494, 1445, 1277, 764 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.09-1.27 (6H, m), 1.43-1.50 (2H, m), 7.10 (1H, s, =CH), 7.13-7.33 (15H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.8, 32.0, 33.1, 115.3, 117.6, 124.8, 127.3, 127.5, 127.7, 127.8, 128.2, 128.3, 128.6, 129.1, 129.3, 129.9, 140.7, 141.8, 145.4; MS (EI) *m/z* (%): 348 (100) [M^+], 305 (58), 215 (43); HRMS (EI) Calcd. for $C_{27}H_{24}$ (M^+) requires 348.1878, Found: 348.1840.

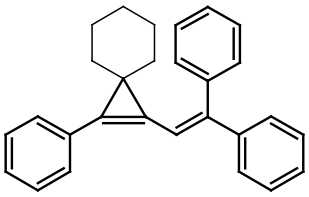




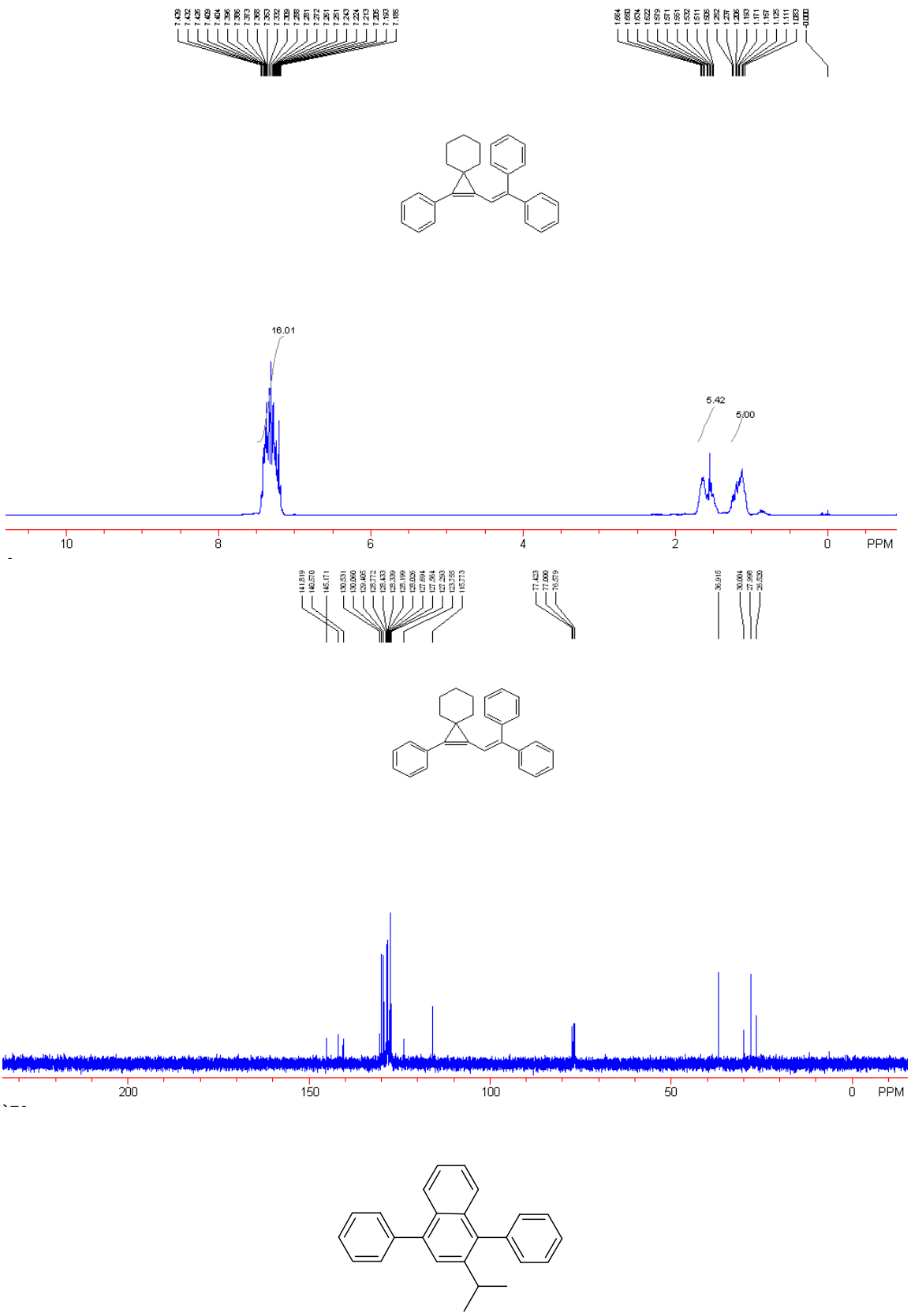






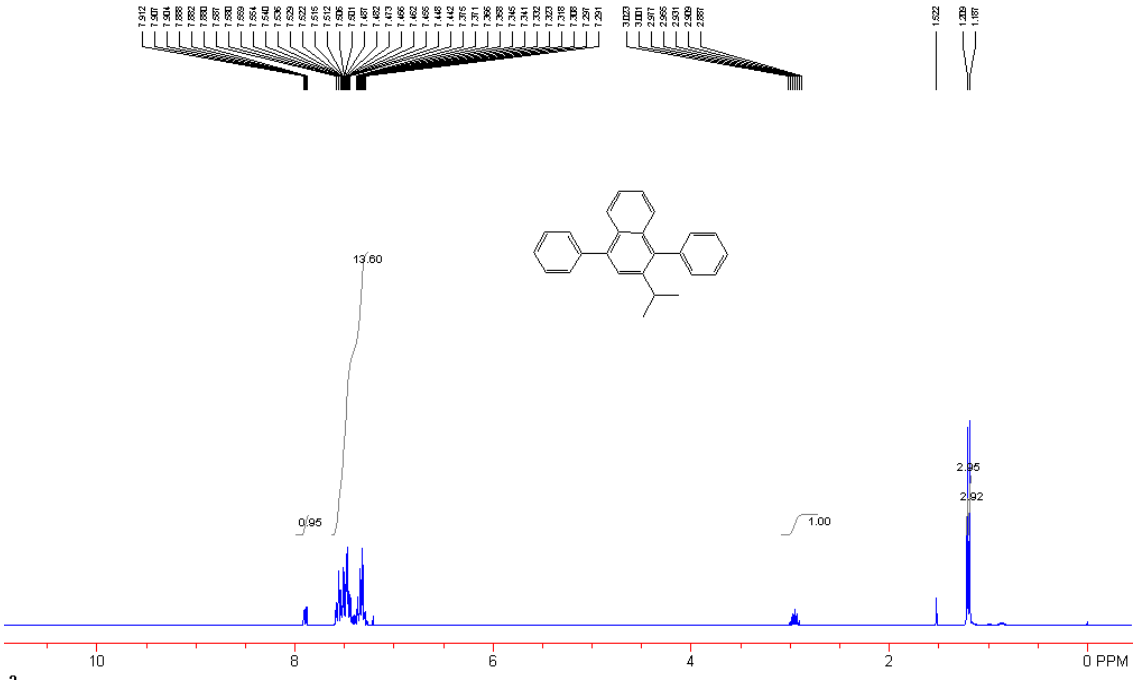


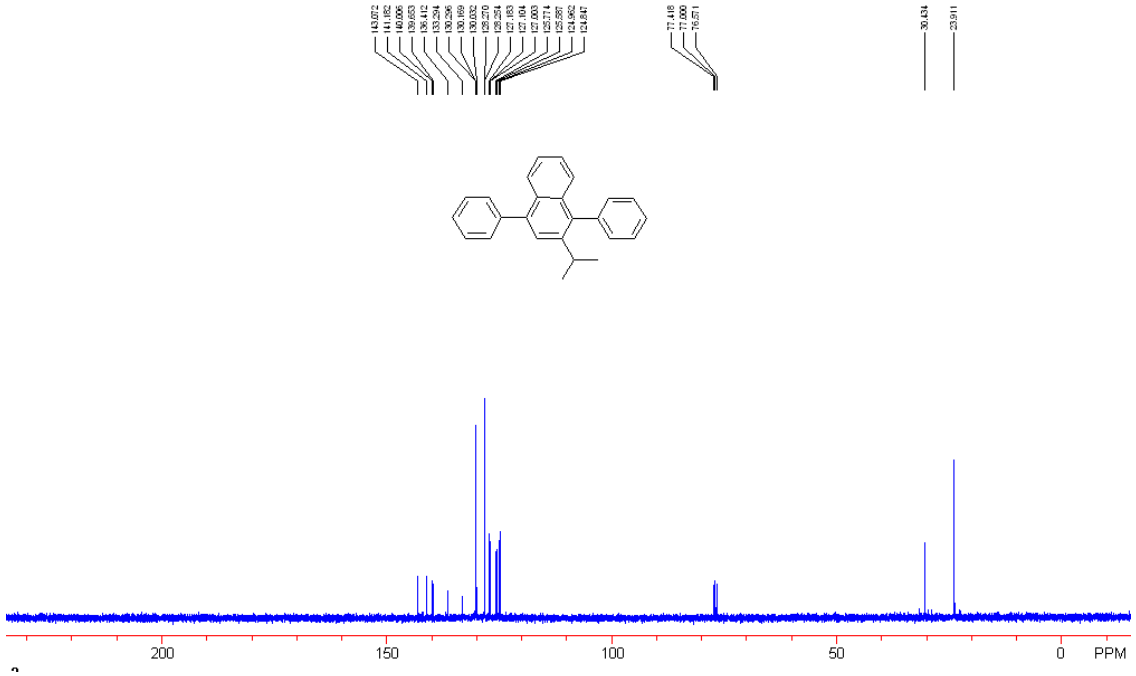
Compound **2k**. A yellow oil. IR (CH_2Cl_2): ν 3060, 2922, 2851, 1487, 1444, 1074, 763 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.08-1.25 (5H, m), 1.51-1.66 (5H, m), 7.19-7.44 (16H, m, =CH + ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.5, 28.0, 30.0, 36.9, 115.8, 123.8, 127.3, 127.6, 127.7, 128.0, 128.2, 128.3, 128.4, 128.8, 129.4, 130.1, 130.5, 140.6, 141.8, 145.2; MS (EI) *m/z* (%): 362 (100) [M^+], 319 (18), 305 (24); HRMS (EI) Calcd. for $C_{28}H_{26}$ (M^+) requires 362.2035, Found: 362.2036.

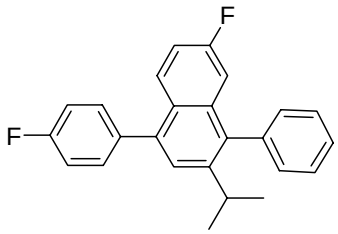


Compound **3a**. A white solid, m.p. 151.8-152.7 °C; IR (CH_2Cl_2): ν 3057, 2961, 2927, 2867,

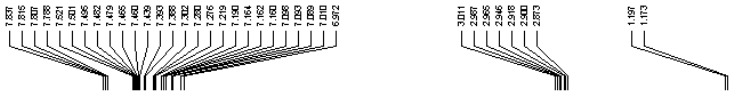
1590, 1496, 1387, 1072, 1056, 893, 765, 741 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.20 (6H, d, *J* = 6.6 Hz, CH_3), 2.96 (1H, hept, *J* = 6.6 Hz, CH), 7.31-7.59 (14H, m, ArH), 7.88-7.91 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.9, 30.4, 124.8, 125.0, 125.6, 125.8, 127.0, 127.1, 127.2, 128.25, 128.27, 130.0, 130.2, 130.3, 133.3, 136.4, 139.7, 140.0, 141.2, 143.1; MS (EI) *m/z* (%): 322 (100) [M^+], 307 (54); HRMS (EI) Calcd. for $C_{25}H_{22}$ (M^+) requires 322.1722, Found: 322.1714.

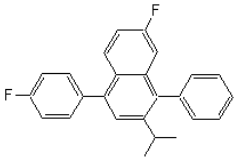


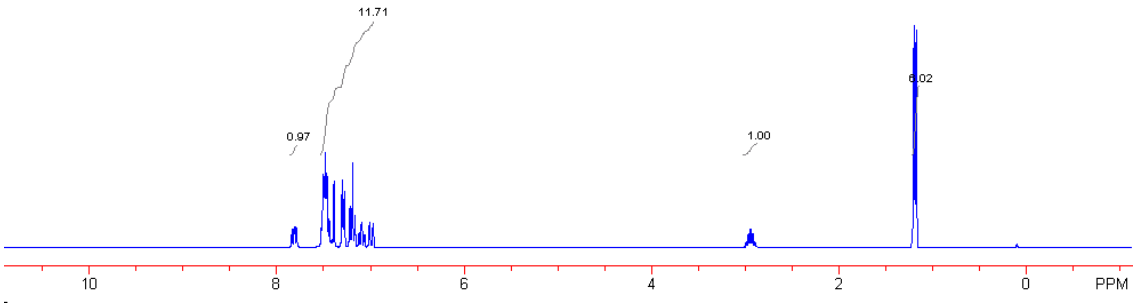


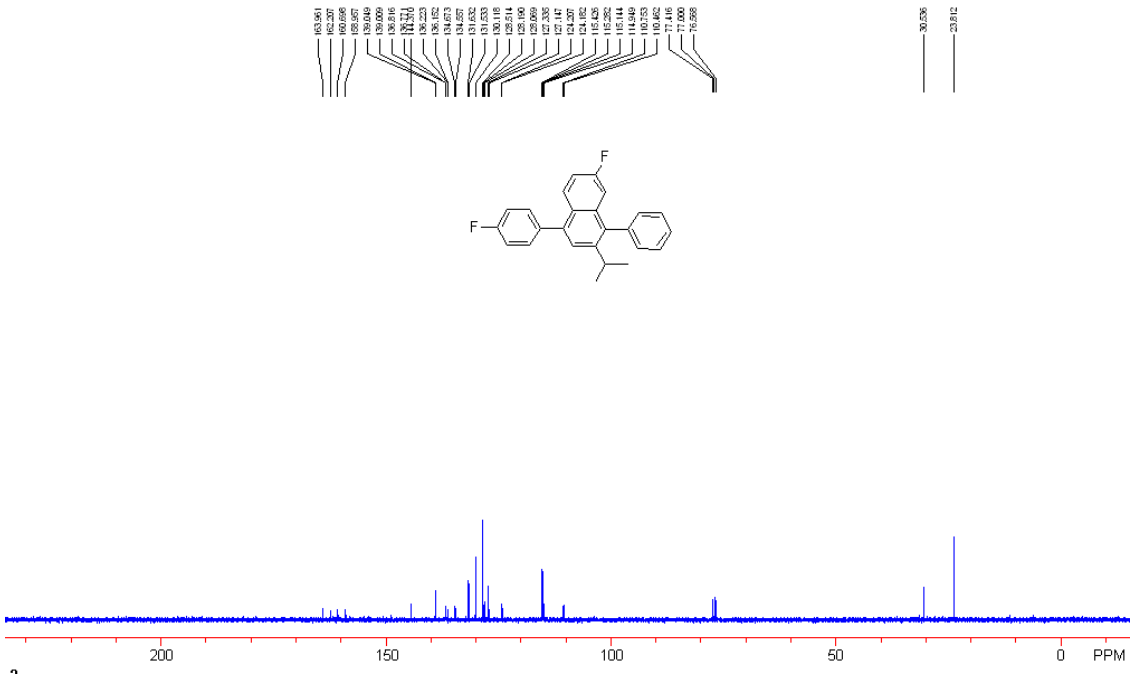


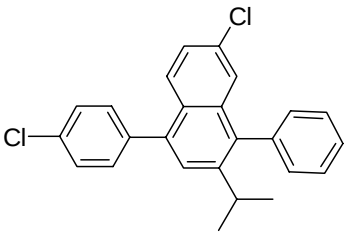
Compound **3b**. A white solid, m.p. 157.4-158.2 °C; IR (CH_2Cl_2): ν 3058, 3019, 2963, 2853, 1624, 1519, 1506, 1428, 1223, 841, 757 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.19 (6H, d, J = 7.2 Hz, CH_3), 2.94 (1H, hept, J = 7.2 Hz, CH), 6.97-7.52 (12H, m, ArH), 7.79-7.84 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.8, 30.5, 110.6 (d, J_{C-F} = 21.8 Hz), 115.1 (d, J_{C-F} = 25.4 Hz), 115.3 (d, J_{C-F} = 21.2 Hz), 124.2, 127.1, 127.3, 128.1 (d, J_{C-F} = 9.4 Hz), 128.5, 129.3 (d, J_{C-F} = 3.9 Hz), 130.1, 131.6 (d, J_{C-F} = 8.1 Hz), 134.6 (d, J_{C-F} = 8.5 Hz), 134.9 (d, J_{C-F} = 2.9 Hz), 136.8 (d, J_{C-F} = 3.8 Hz), 139.0, 144.4, 160.6 (d, J_{C-F} = 244.1 Hz), 162.3 (d, J_{C-F} = 245.5Hz); MS (EI) *m/z* (%): 358 (100) [M^+], 343 (29); HRMS (EI) Calcd. for $C_{25}H_{20}F_2$ (M^+) requires 358.1533, Found: 358.1532.



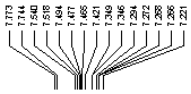


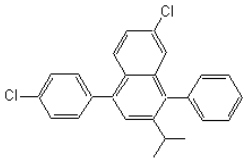


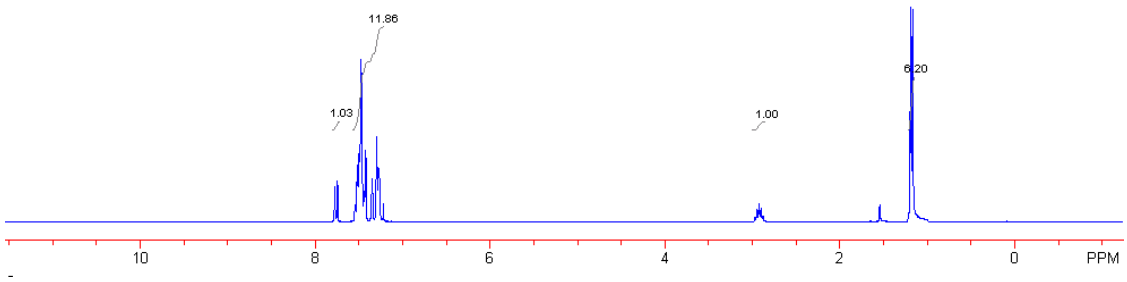


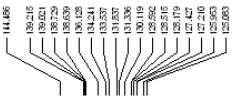


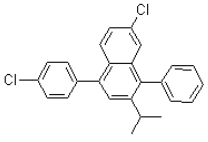
Compound **3c**. A white solid, m.p. 197.8-198.5 °C; IR (CH_2Cl_2): ν 3058, 2961, 2925, 2868, 1604, 1502, 1092, 1015, 834, 759, 705 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.18 (6H, d, *J* = 6.6 Hz, CH_3), 2.93 (1H, hept, *J* = 6.6 Hz, CH), 7.23-7.53 (12H, m, ArH), 7.75-7.78 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.8, 30.5, 125.1, 126.0, 127.2, 127.4, 128.2, 128.5, 128.6, 130.1, 131.3, 131.8, 133.5, 134.2, 136.1, 138.6, 138.7, 139.0, 139.2, 144.5; MS (EI) *m/z* (%): 390 (100) [M^+]; HRMS (EI) Calcd. for $C_{25}H_{20}Cl_2$ (M^+) requires 390.0942, Found: 390.0959.

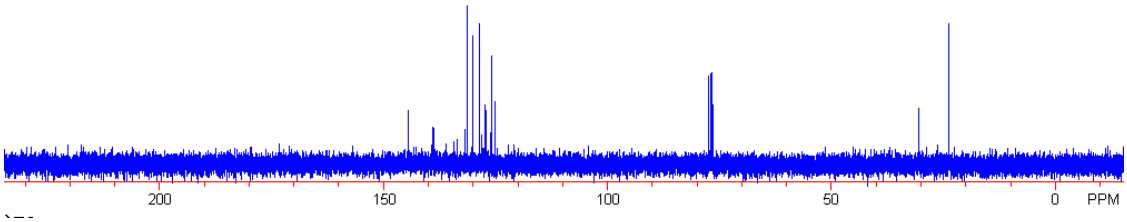


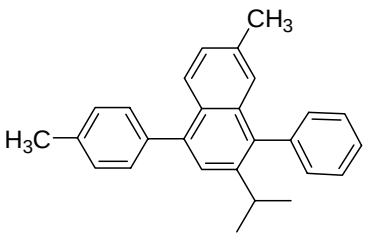

Cl
Cl


11.86
1.03
1.00
6.20
10
8
6
4
2
0
PPM

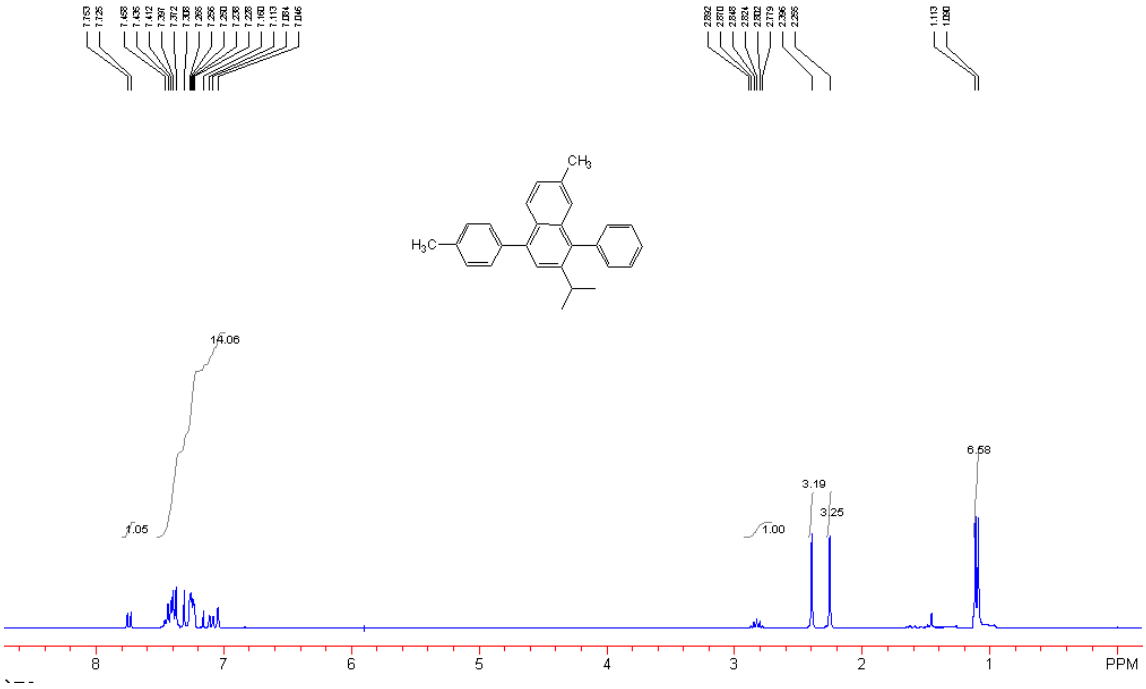


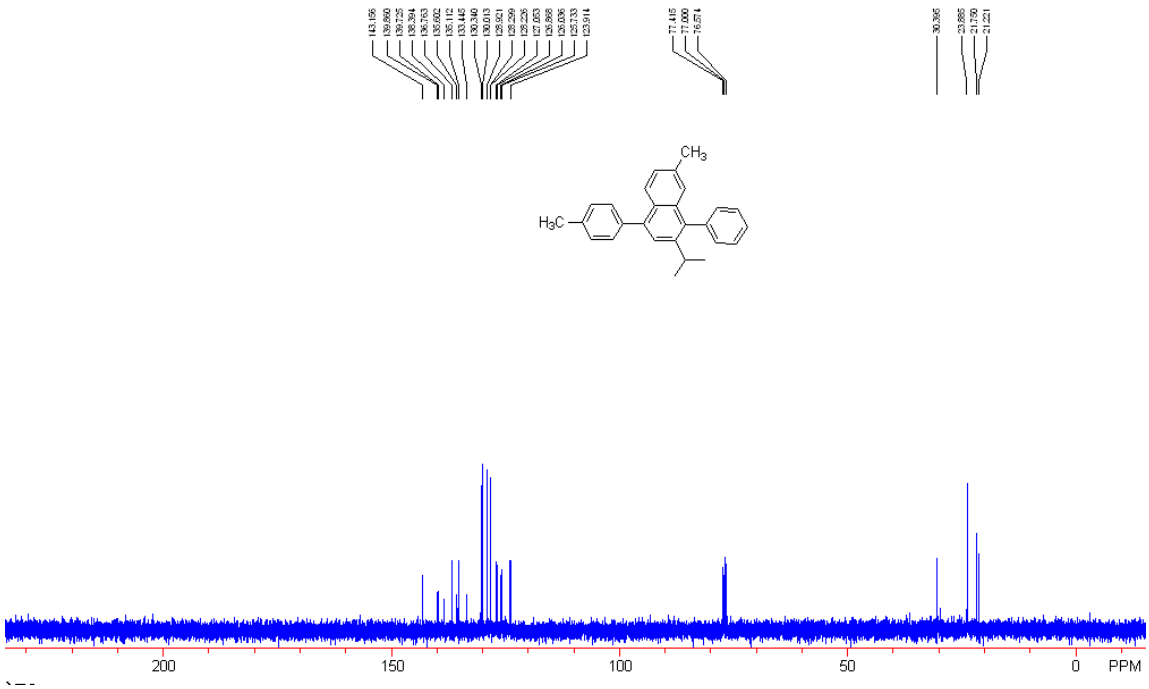

Cl
Cl

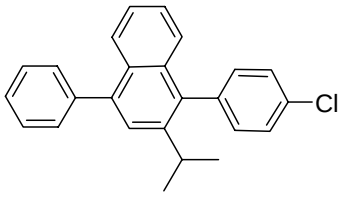

200
150
100
50
0
PPM


CH3
H3C

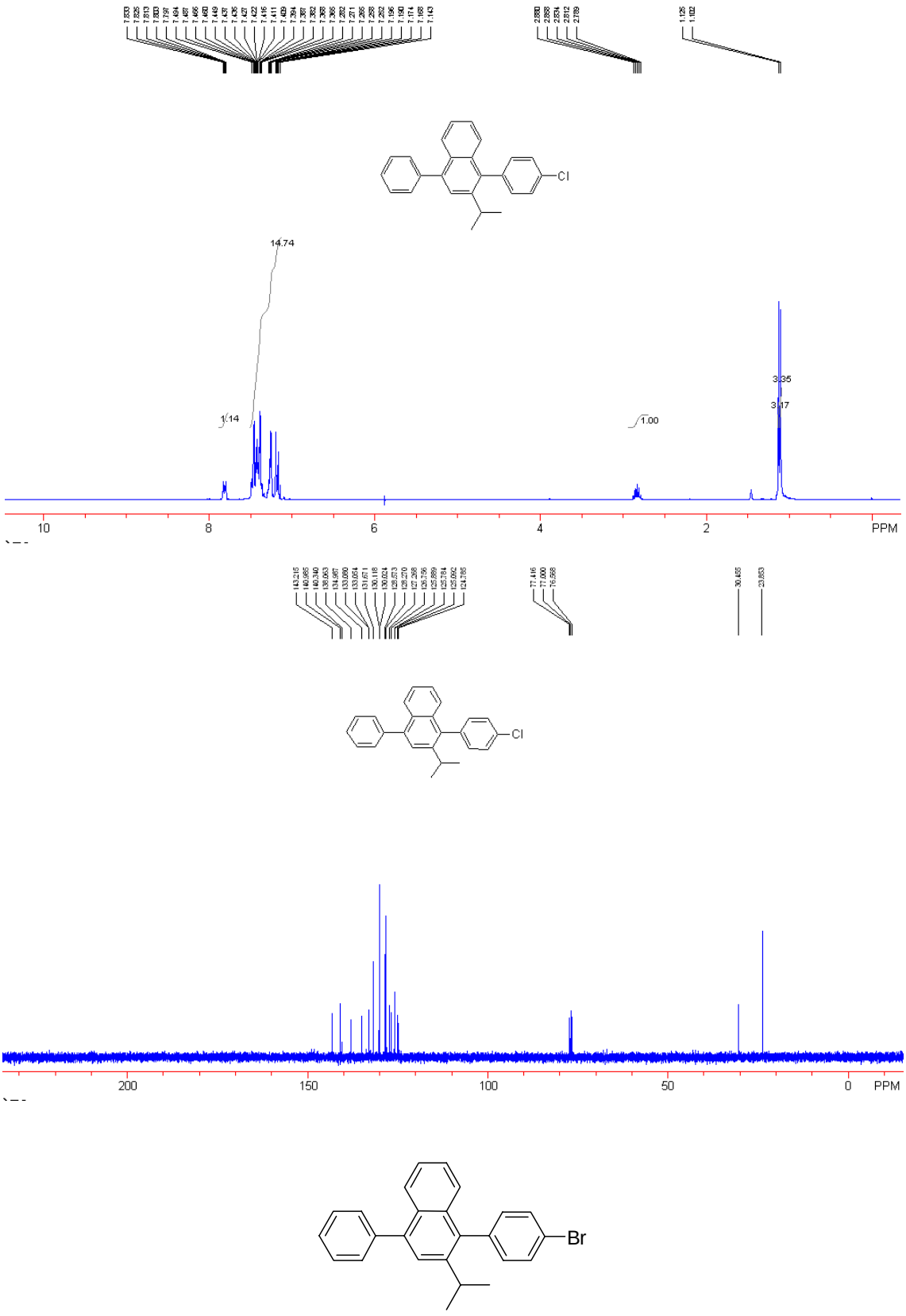
Compound **3d**. A white solid, m.p. 144.4-145.6 °C; IR (CH_2Cl_2): ν 3052, 3022, 2959, 2855, 1507, 1460, 1441, 1376, 1362, 1053, 825, 754 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.10 (6H, d, *J* = 7.2 Hz, CH_3), 2.93 (1H, hept, *J* = 7.2 Hz, CH), 2.36 (3H, s, CH_3), 2.50 (3H, s, CH_3), 7.15-7.56 (12H, m, ArH), 7.84 (1H, d, *J* = 8.4 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 21.2, 21.8, 23.9, 30.4, 123.9, 125.7, 126.0, 126.9, 127.1, 128.2, 128.3, 128.9, 130.0, 130.3, 133.4, 135.1, 135.6, 136.8, 138.4, 139.7, 139.9, 143.2; MS (EI) *m/z* (%): 350 (100) [M^+], 335 (24); Anal. Calcd. for $C_{27}H_{26}$ requires C, 92.52; H, 7.48%. Found: C, 92.49; H, 7.42%.





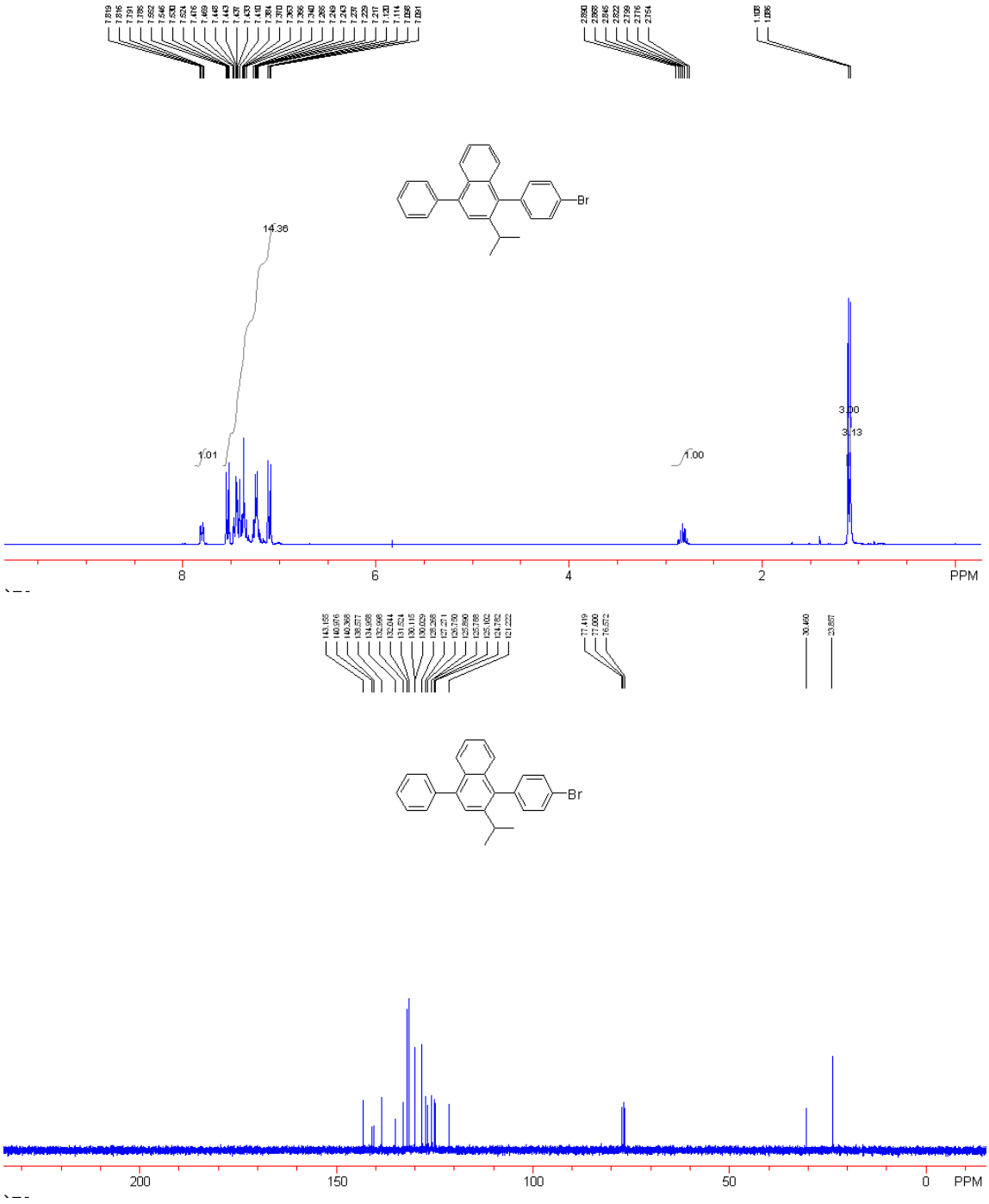


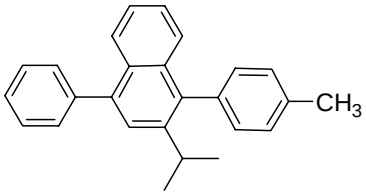
Compound **3e**. A white solid, m.p. 185.6-186.4 °C; IR (CH_2Cl_2): ν 3063, 2961, 2926, 2868, 1591, 1491, 1388, 1089, 1017, 826, 766 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.23 (6H, d, *J* = 7.2 Hz, CH_3), 2.83 (1H, hept, *J* = 7.2 Hz, CH), 7.28-7.52 (13H, m, ArH), 7.80-7.83 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.8, 30.4, 124.8, 125.1, 125.8, 125.9, 126.8, 127.3, 128.3, 128.6, 130.0, 130.1, 131.7, 133.05, 133.08, 135.0, 138.1, 140.3, 141.0, 143.2; MS (EI) *m/z* (%): 356 (100) [M^+], 341 (23); HRMS (EI) Calcd. for $C_{25}H_{21}Cl$ (M^+) requires 356.1332, Found: 356.1326.



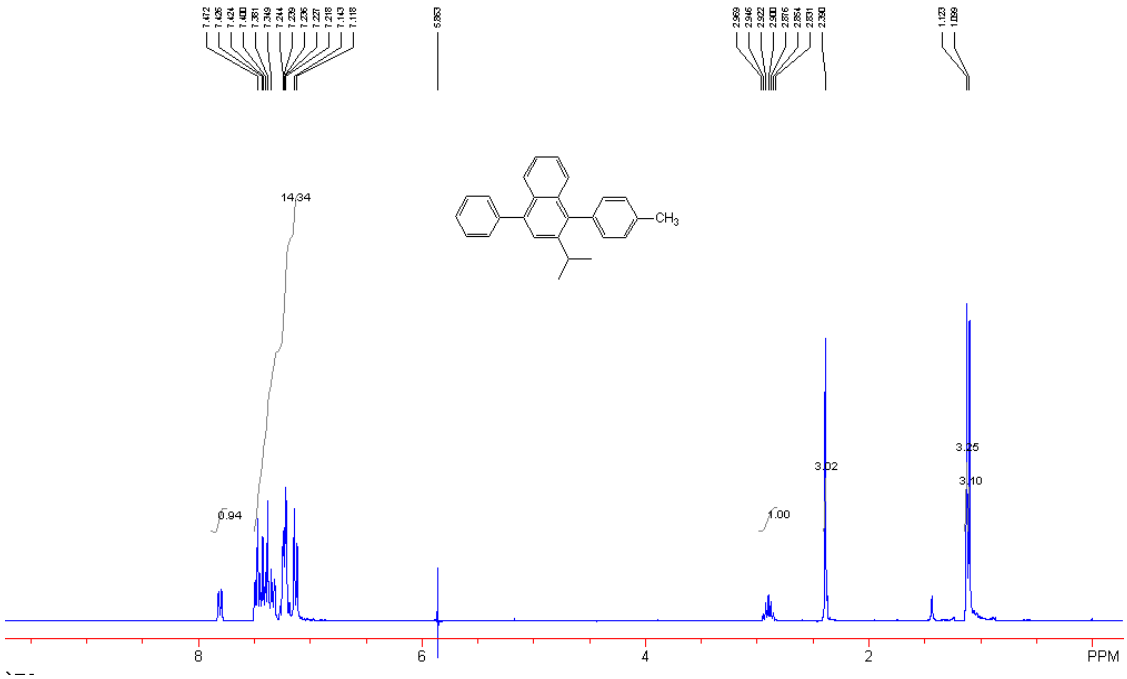
Compound **3f**. A white solid, m.p. 193.1-194.6 °C; IR (CH_2Cl_2): ν 3057, 2961, 2926, 2868,

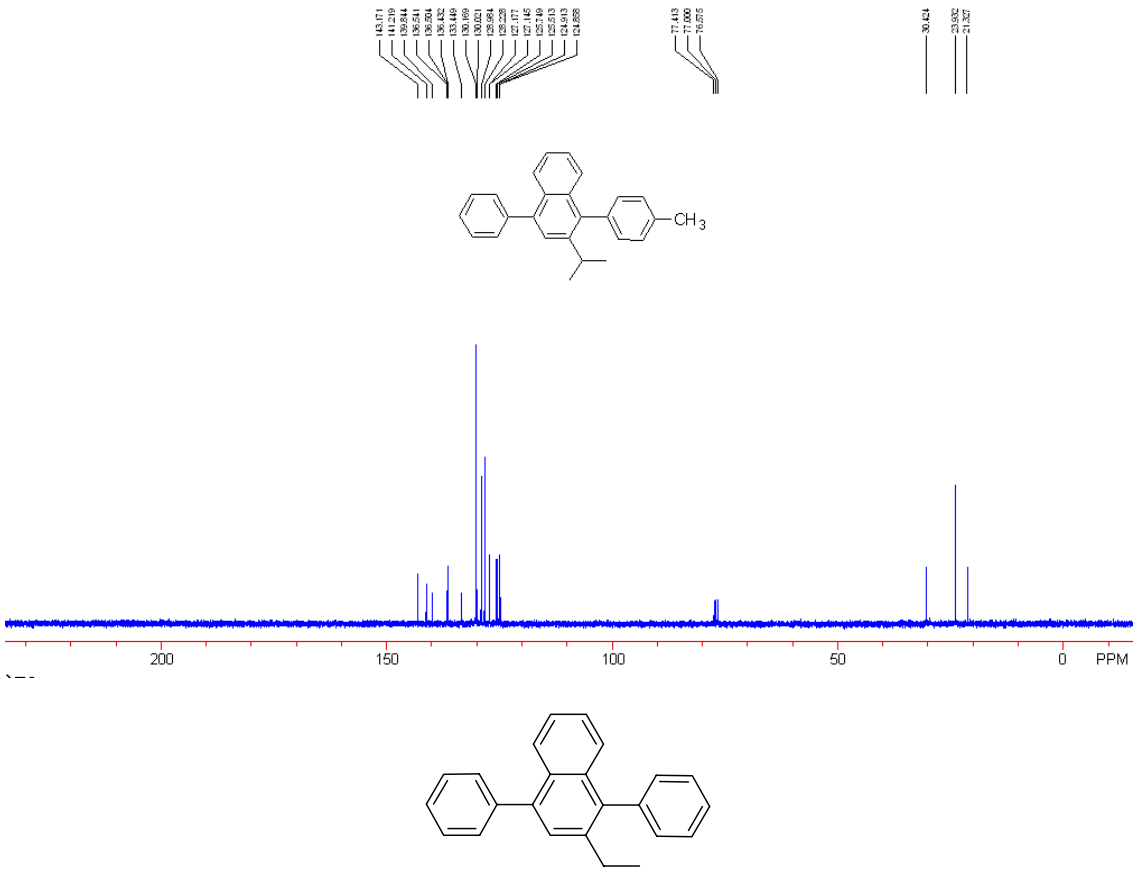
1591, 1491, 1388, 1071, 1012, 823, 765 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.27 (6H, d, *J* = 6.9 Hz, CH_3), 2.82 (1H, hept, *J* = 6.9 Hz, CH), 7.10-7.56 (13H, m, ArH), 7.79-7.82 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.8, 30.4, 121.2, 124.8, 125.1, 125.8, 125.9, 126.7, 127.3, 128.3, 130.0, 130.1, 131.5, 132.0, 133.0, 134.9, 138.6, 140.4, 141.0, 143.1; MS (EI) *m/z* (%): 400 (41) [M^+], 306 (100); HRMS (EI) Calcd. for $C_{31}H_{26}$ (M^+) requires 400.0827, Found: 400.0793.



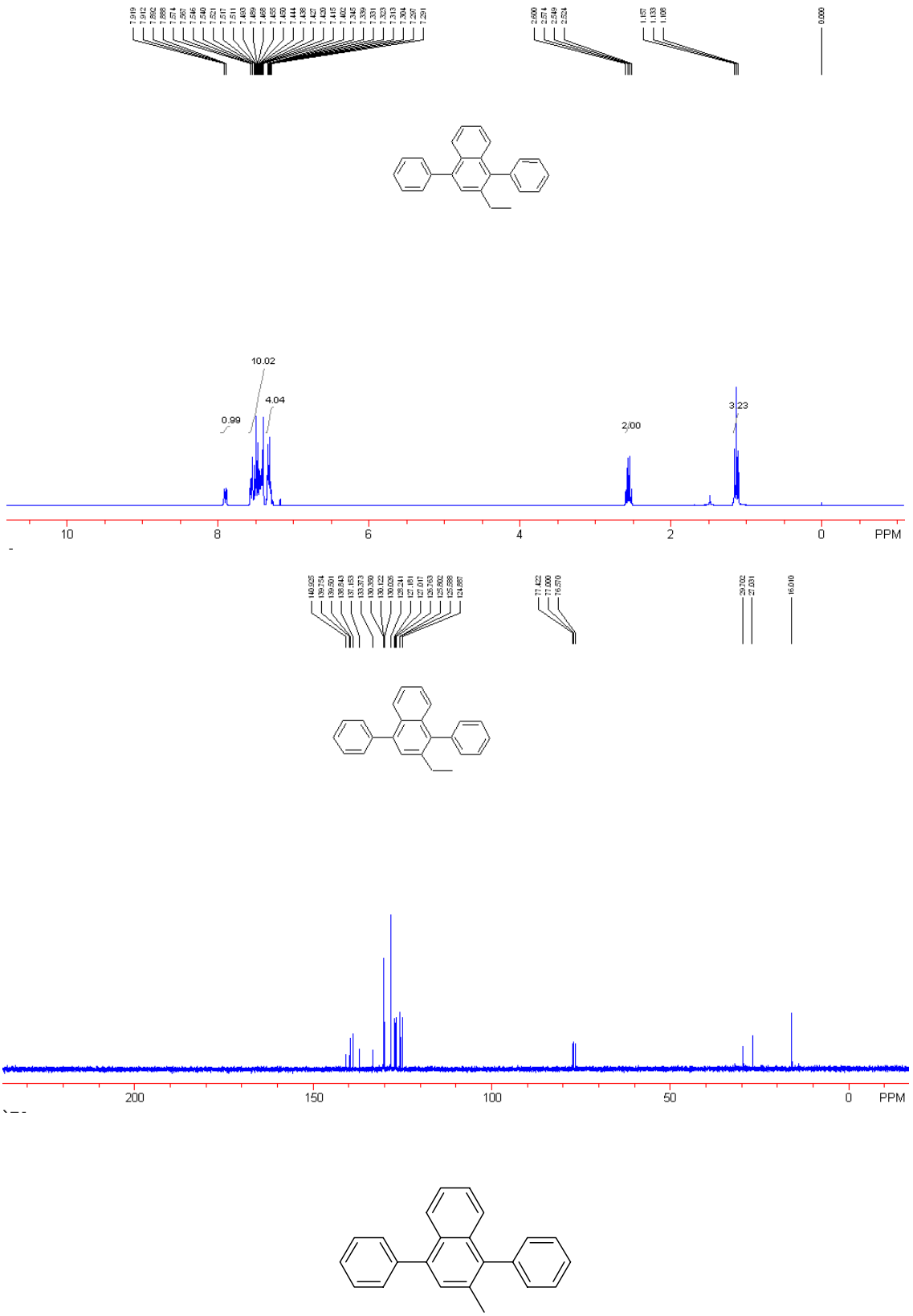


Compound **3g**. A white solid, m.p. 146.3-145.8 °C; IR (CH_2Cl_2): ν 3023, 2960, 2925, 2867, 1515, 1459, 1387, 1056, 893, 817, 775, 765 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.11 (6H, d, *J* = 7.2 Hz, CH_3), 2.51 (3H, s, CH_3), 2.90 (1H, hept, *J* = 7.2 Hz, CH), 7.24-7.62 (13H, m, ArH), 7.91-7.94 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 21.3, 23.9, 30.4, 124.86, 124.91, 125.5, 125.7, 127.1, 127.2, 128.2, 129.0, 130.0, 130.2, 133.4, 136.4, 136.50, 136.54, 139.8, 141.2, 143.2; MS (EI) *m/z* (%): 336 (100) [M^+], 321 (42); Anal. Calcd. for $C_{26}H_{24}$ requires C, 92.81; H, 7.19%. Found: C, 92.54; H, 6.92%.



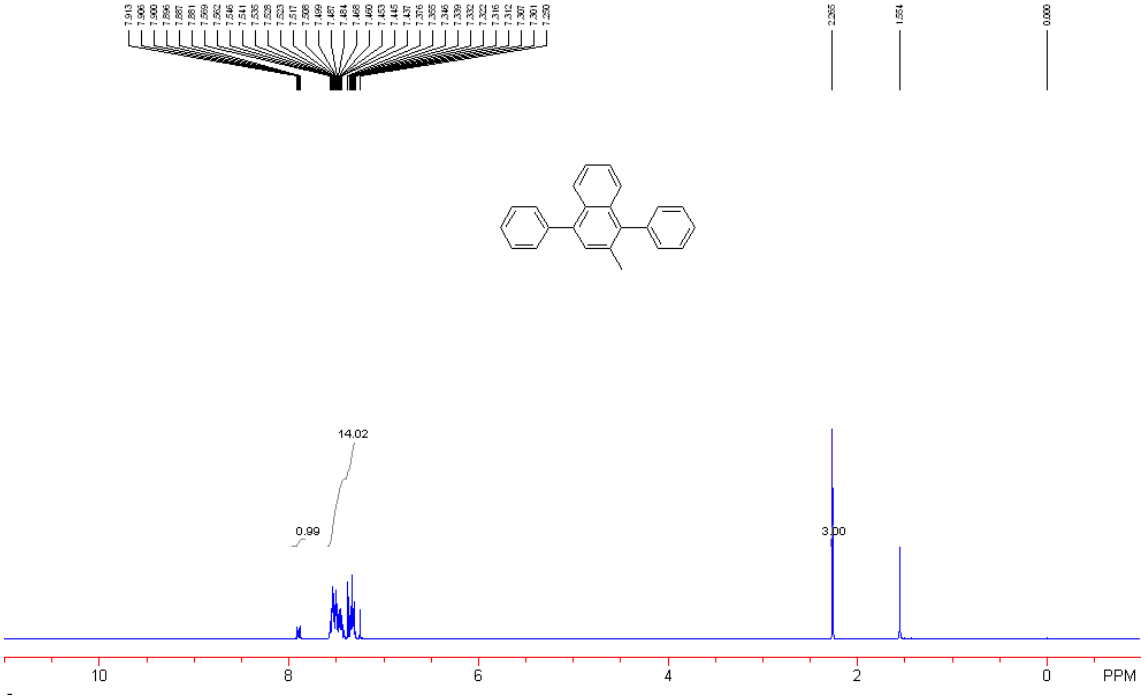


Compound **3h**. A white solid, m.p. 109.7-110.6 °C; IR (CH_2Cl_2): ν 3058, 3025, 2959, 2925, 2854, 1601, 1496, 1456, 1441, 1381, 1289, 1244, 891, 773, 760, 741 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.13 (3H, t, *J* = 7.5 Hz, CH_3), 2.56 (2H, q, *J* = 7.5 Hz, CH_2), 7.29-7.35 (4H, m, ArH), 7.40-7.57 (10H, m, ArH), 7.89-7.92 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 16.0, 27.0, 124.9, 125.6, 125.8, 126.8, 127.0, 127.2, 128.2, 130.0, 130.1, 130.4, 133.4, 137.2, 138.8, 139.5, 139.8, 140.9; MS (EI) *m/z* (%): 308 (100) [M^+], 293 (15), 215 (21); Anal. Calcd. for $C_{24}H_{20}$ requires C, 93.46; H, 6.54%. Found: C, 93.07; H, 6.69%.

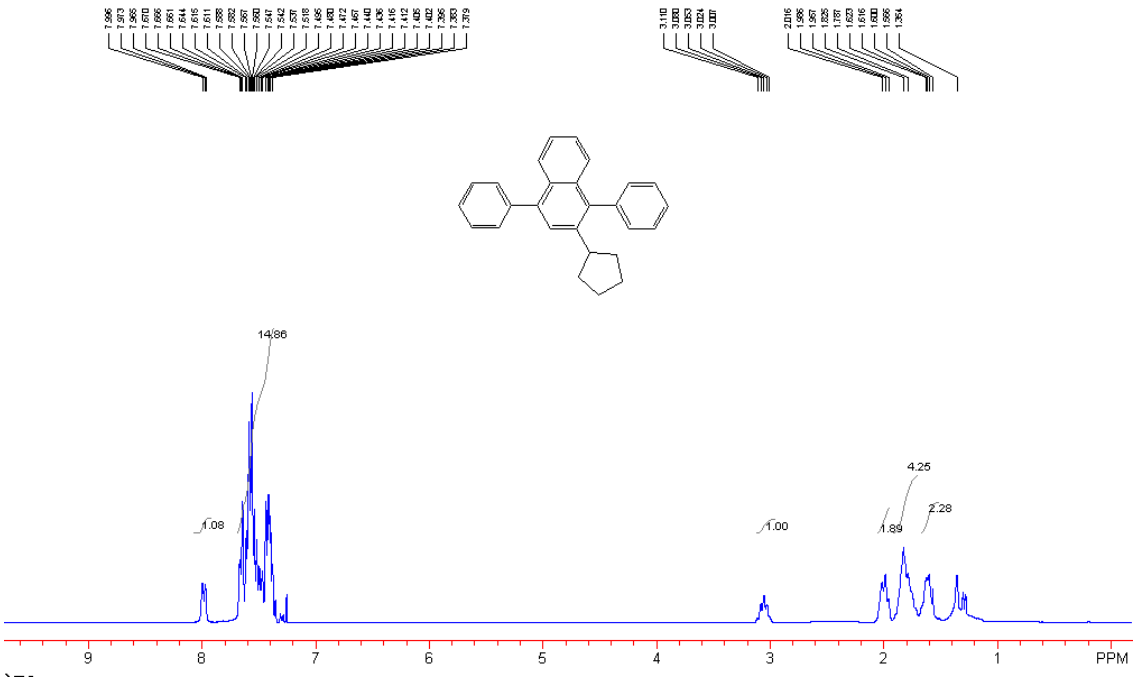


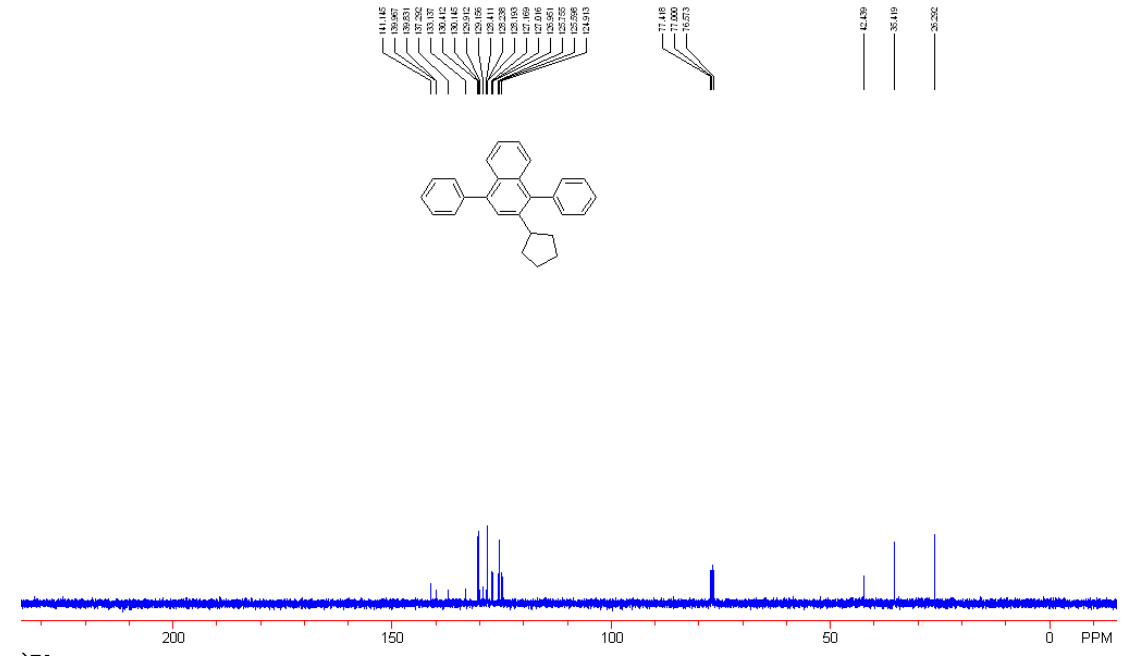
Compound **3i**.A white solid, m.p. 129.2-129.5 °C; IR (CH_2Cl_2): ν 3056, 3025, 2920, 2859,

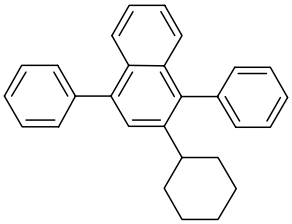
1591, 1496, 1441, 1387, 1373, 1072, 1030, 886, 774, 744 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.27 (3H, s, CH_3), 7.30-7.57 (14H, m, ArH), 7.88-7.91 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.8, 124.8, 125.6, 125.8, 126.5, 127.0, 127.2, 128.2, 128.4, 129.7, 130.06, 130.10, 130.2, 132.6, 133.3, 137.8, 139.4, 139.8, 140.8; MS (EI) *m/z* (%): 294 (100) [M^+], 279 (30), 215 (24), 202 (15); Anal. Calcd. for $C_{23}H_{18}$ requires C, 93.84; H, 6.16%. Found: C, 93.55; H, 6.43%.



Compound **3j**. A white solid, m.p. 137.7-138.8 °C; IR (CH_2Cl_2): ν 3057, 3024, 2955, 2866, 1592, 1496, 1441, 1380, 768 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.57-2.02 (8H, m), 3.05-3.08 (1H, m), 7.38-7.57 (14H, m, ArH), 7.97-7.99 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.3, 35.4, 42.4, 124.9, 125.6, 125.8, 126.95, 127.02, 127.2, 128.19, 128.24, 128.4, 129.2, 129.9, 130.1, 130.4, 133.1, 137.3, 139.8, 140.0, 141.1; MS (EI) *m/z* (%): 348 (100) [M^+]; Anal. Calcd. for $C_{27}H_{24}$ requires C, 93.06%; H, 6.94%. Found: C, 92.75%; H, 6.67%.

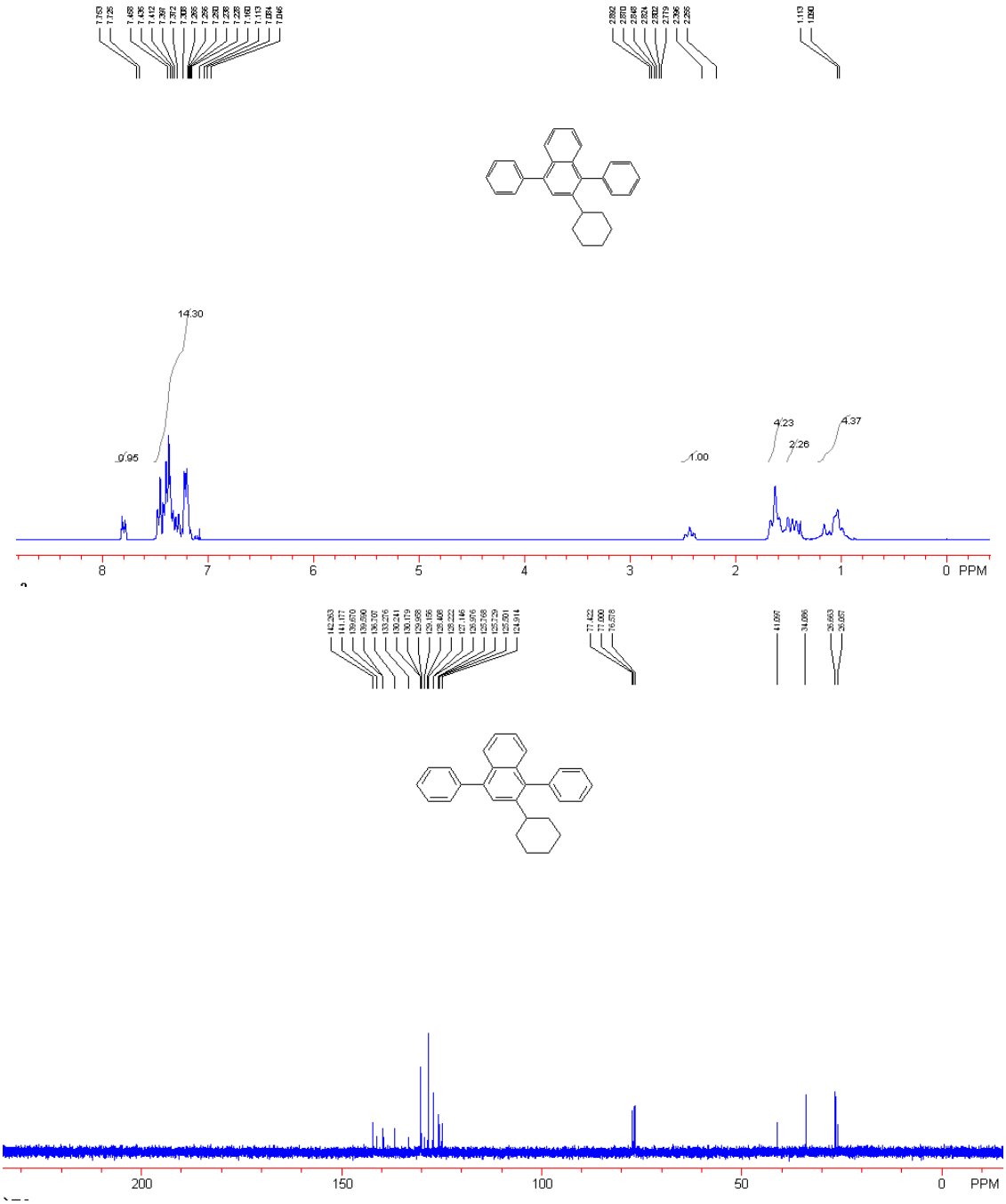


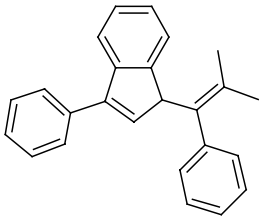




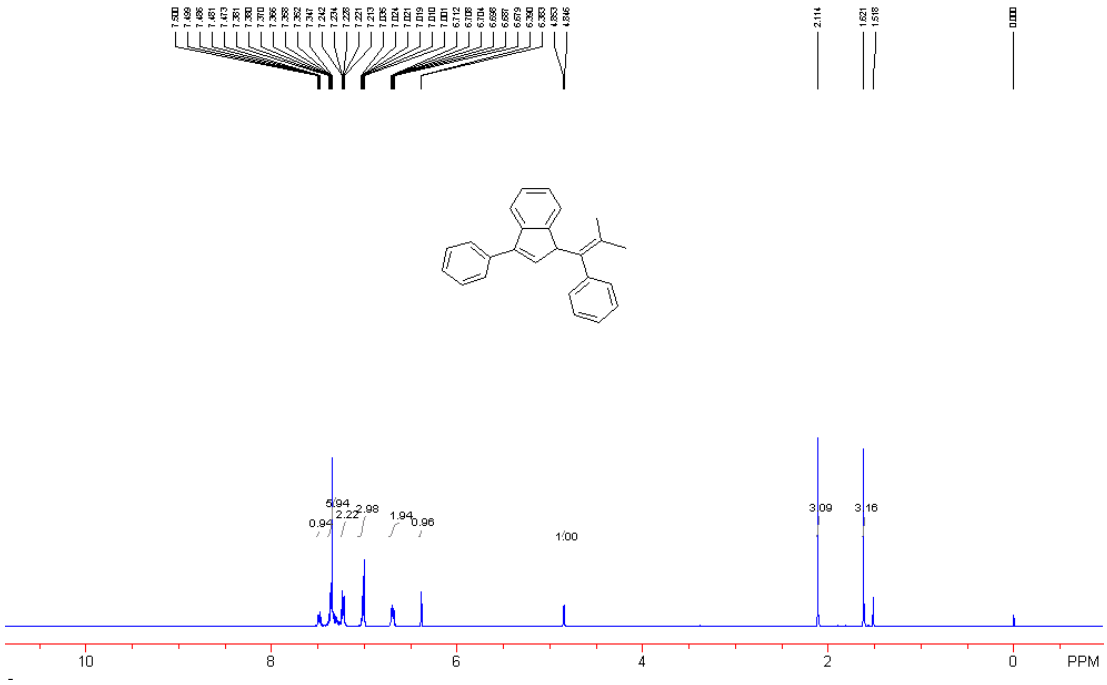
Compound **3k**. A white solid, m.p. 153.4-154.7 °C; IR (CH_2Cl_2): ν 3057, 3026, 2925, 2850,

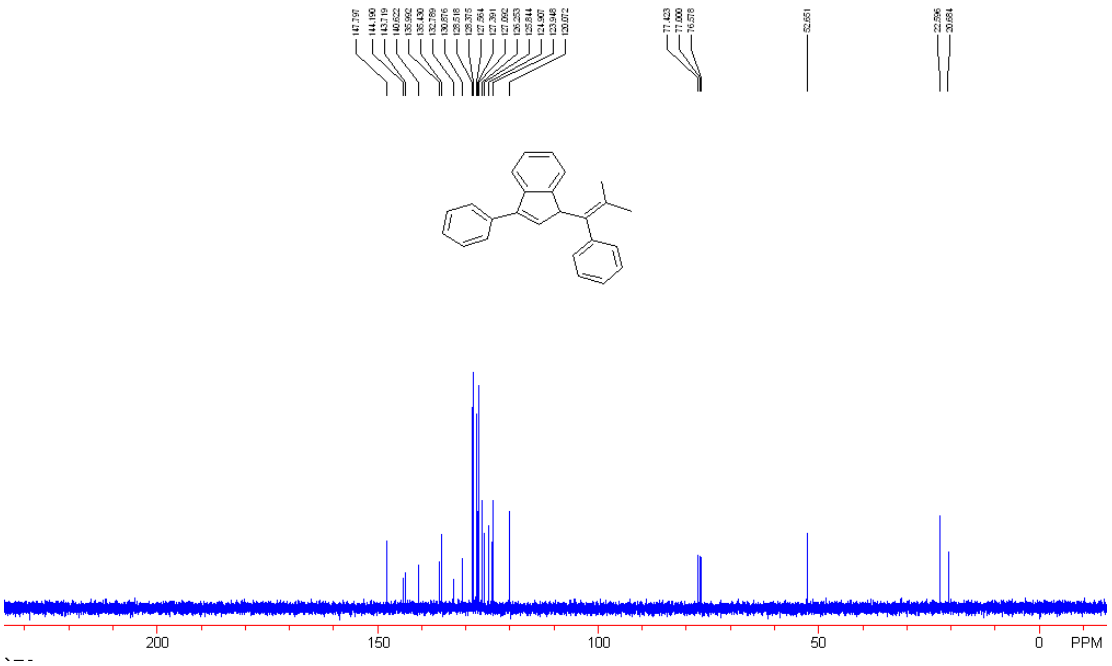
1587, 1496, 1443, 1381, 1261, 1184, 1072, 977, 768 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.21-1.85 (10H, m), 2.57-2.65 (1H, m), 7.36-7.66 (14H, m, ArH), 7.96-7.99 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.1, 26.7, 34.1, 41.1, 124.9, 125.5, 125.7, 125.8, 127.0, 127.1, 128.2, 129.2, 130.0, 130.18, 130.24, 133.3, 136.7, 139.6, 139.7, 141.2, 142.3; MS (EI) *m/z* (%): 362 (100) [M^+]; HRMS (EI) Calcd. for $C_{28}H_{26}$ (M^+) requires 362.2035, Found: 362.2014.

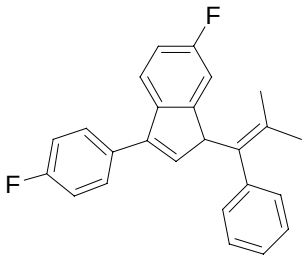




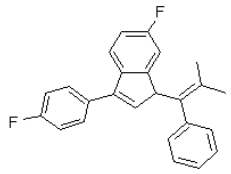
Compound **4a**. A yellow solid, m.p. 97.3-98.1 °C. IR (CH_2Cl_2): ν 3057, 3015, 2956, 2925, 2854, 1752, 1734, 1660, 1594, 1490, 1445, 1362, 1268, 1180, 1072, 1022, 965, 765 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.62 (3H, s, CH_3), 2.11 (3H, s, CH_3), 4.85 (1H, d, *J* = 1.8 Hz, CH), 6.39 (1H, d, *J* = 1.8 Hz, =CH), 6.68-6.71 (2H, m, ArH), 7.00-7.03 (3H, m, ArH), 7.21-7.24 (2H, m, ArH), 7.35-7.38 (6H, m, ArH), 7.47-7.50 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 22.6, 52.7, 120.1, 123.9, 124.9, 125.8, 126.3, 127.1, 127.4, 127.6, 128.4, 128.5, 130.9, 132.8, 135.4, 136.0, 140.6, 143.7, 144.2, 147.8; MS (EI) *m/z* (%): 322 (82) [M^+], 307 (100), 229 (23), 131 (39), 91 (37); Anal. Calcd. for $C_{25}H_{22}$ requires C, 93.12; H, 6.88%. Found: C, 92.75; H, 5.37%.

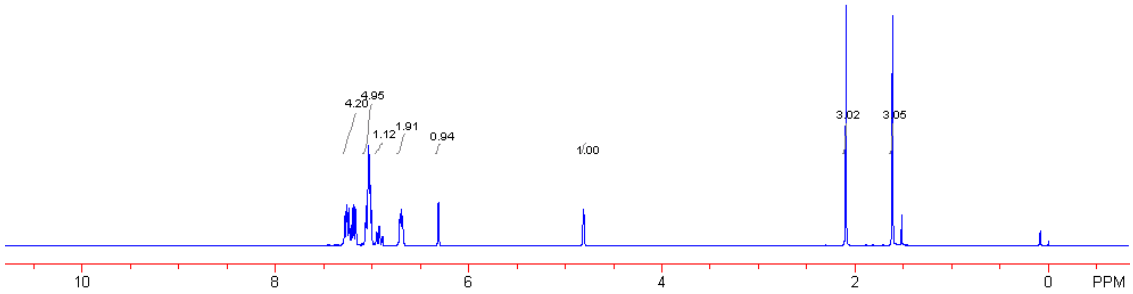


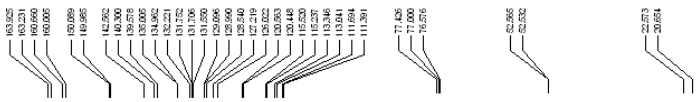


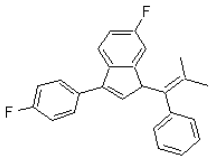


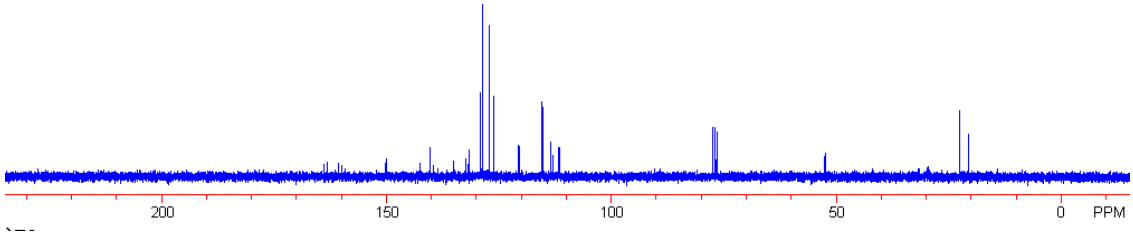
Compound **4b**. A yellow oil. IR (CH_2Cl_2): ν 3051, 3015, 2955, 2926, 2855, 1604, 1587, 1575, 1504, 1471, 1268, 1221, 1157, 826, 756 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.61 (3H, s, CH_3), 2.10 (3H, s, CH_3), 4.81 (1H, d, J = 2.4 Hz, CH), 6.31 (1H, d, J = 2.4 Hz, =CH), 6.69-7.71 (2H, m, ArH), 6.89-6.95 (1H, m, ArH), 7.01-7.06 (5H, m, ArH), 7.17-7.28 (3H, m, ArH), 7.47-7.50 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 22.6, 52.5 (d, J_{C-F} = 2.5 Hz), 111.5 (d, J_{C-F} = 22.7 Hz), 113.2 (d, J_{C-F} = 22.9 Hz), 115.4 (d, J_{C-F} = 21.2 Hz), 120.5 (d, J_{C-F} = 8.6 Hz), 126.0, 127.2, 128.5, 129.0 (d, J_{C-F} = 8.0 Hz), 131.6, 131.7 (d, J_{C-F} = 3.5 Hz), 132.2, 135.0 (d, J_{C-F} = 3.2 Hz), 139.6, 140.3, 142.6, 150.0 (d, J_{C-F} = 7.8 Hz), 161.6 (d, J_{C-F} = 242.0 Hz), 162.3 (d, J_{C-F} = 244.9 Hz); MS (EI) *m/z* (%): 358 (31) [M^+], 343 (28), 131 (100), 91 (37); HRMS (EI) Calcd. for $C_{25}H_{20}F_2$ (M^+) requires 358.1533, Found: 358.1550.

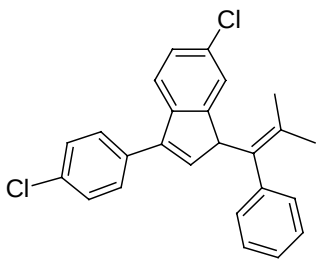

F
F


4.20
4.95
1.12
1.91
0.94
1.00
3.02
3.05
10
8
6
4
2
0
PPM

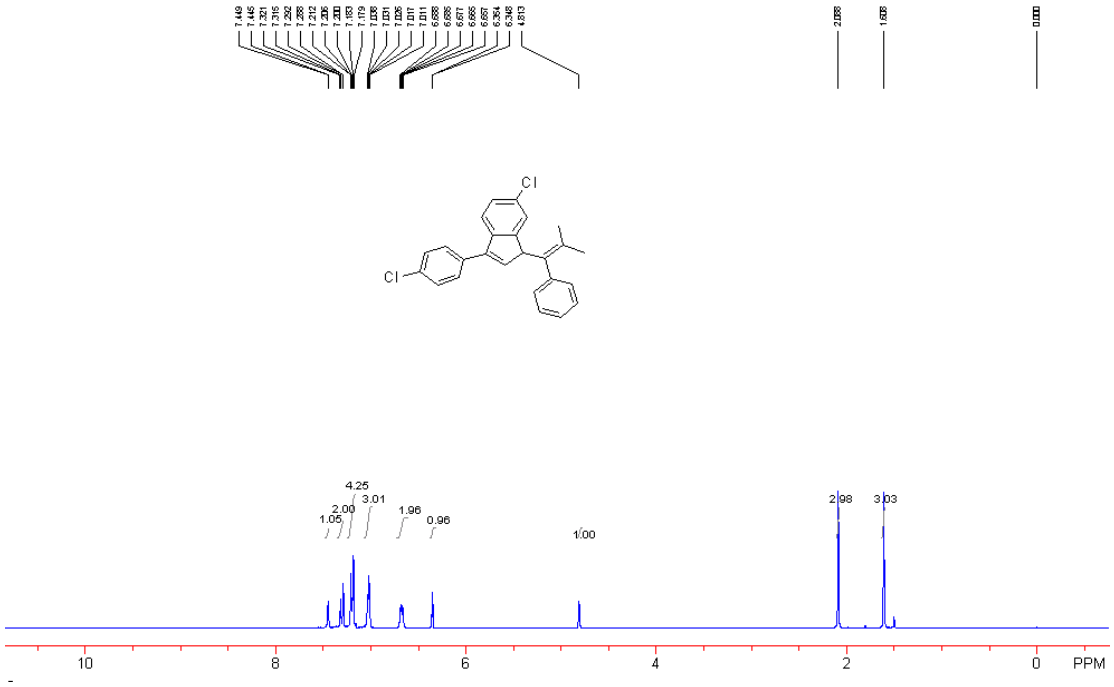


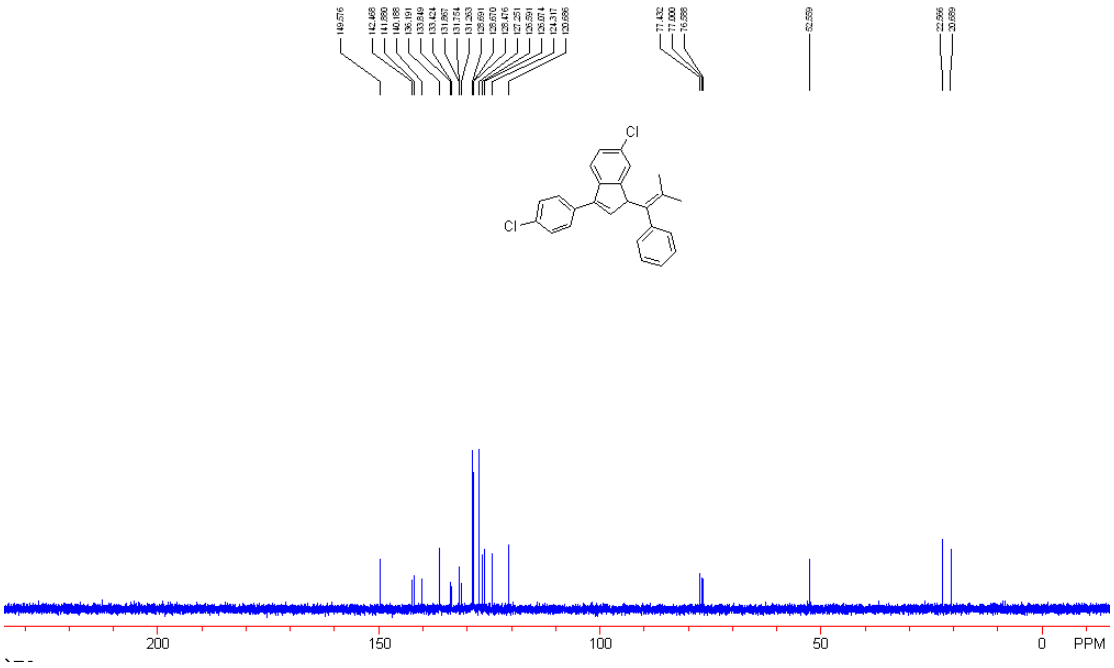

F
F

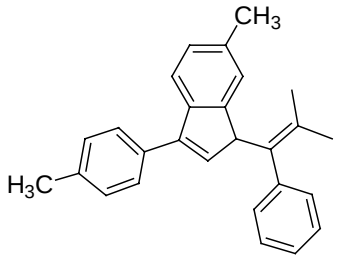

200
150
100
50
0
PPM


Cl
Cl

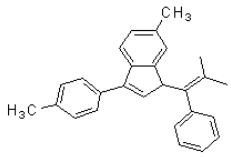
Compound **4c**. A yellow oil. IR (CH_2Cl_2): ν 3071, 3045, 3019, 2949, 2923, 2853, 1744, 1731, 1664, 1594, 1490, 1457, 1440, 1408, 1396 1373, 1093, 1015, 760 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.61 (3H, s, CH_3), 2.09 (3H, s, CH_3), 4.81 (1H, d, *J* = 1.8 Hz, CH), 6.35 (1H, d, *J* = 1.8 Hz, =CH), 6.66-6.69 (2H, m, ArH), 7.01-7.04 (3H, m, ArH), 7.18-7.21 (4H, m, ArH), 7.29-7.32 (2H, m, ArH), 7.44-7.45 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 22.6, 52.6, 120.7, 124.3, 126.1, 126.6, 127.3, 128.5, 128.67, 128.69, 131.3, 131.8, 131.9, 133.4, 133.8, 136.2, 140.2, 141.9, 142.5, 149.6; MS (EI) *m/z* (%): 390 (12) [M^+], 375 (11), 131 (100), 91 (55); HRMS (EI) Calcd. for $C_{25}H_{20}Cl_2$ (M^+) requires 390.0942, Found: 390.0942.

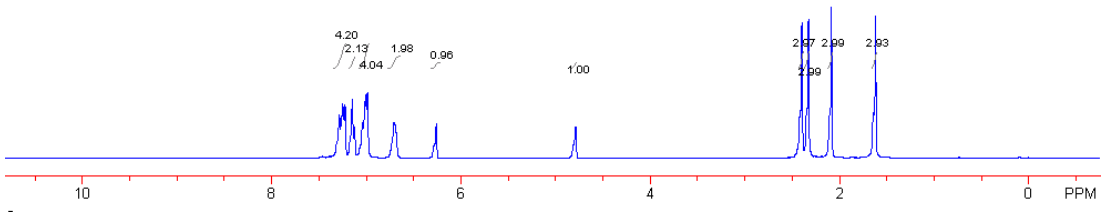


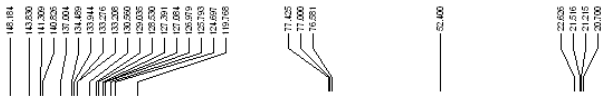


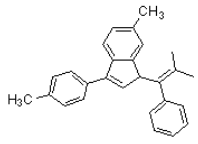


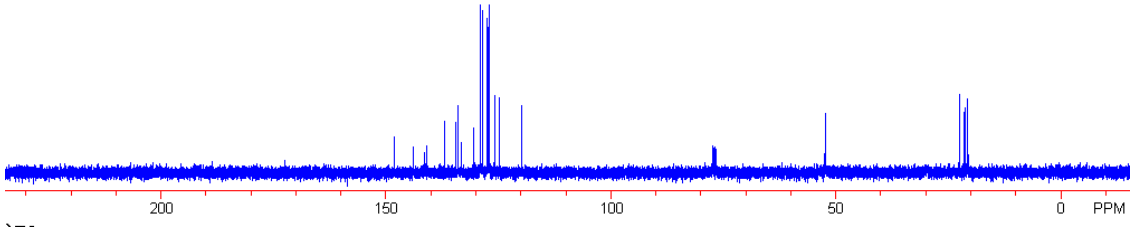
Compound **4d**. A yellow oil. IR (CH_2Cl_2): ν 3052, 3015, 2985, 2917, 2860, 1749, 1656, 1601, 1568, 1509, 1491, 1441, 1338, 1284, 1181 823, 815, 717 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.62 (3H, s, CH_3), 2.08 (3H, s, CH_3), 2.33 (3H, s , CH_3), 2.40 (3H, s , CH_3), 4.79 (1H, d, *J* = 1.8 Hz, CH), 6.26 (1H, d, *J* = 1.8 Hz, =CH), 6.68-6.71 (2H, m, ArH), 6.99-7.04 (4H, m, ArH), 7.12-7.15 (2H, m, ArH), 7.22-7.28 (4H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 21.2, 21.5, 22.6, 52.4, 119.8, 124.7, 125.8, 127.0, 127.1, 127.4, 128.5, 129.0, 130.6, 133.2, 133.3, 133.9, 134.5, 137.0, 140.8, 141.3, 143.8, 148.2; MS (EI) *m/z* (%): 350 (62) [M^+], 335 (100), 320 (28), 219 (28); HRMS (EI) Calcd. for $C_{27}H_{26}$ (M^+) requires 350.2035, Found: 350.2038.

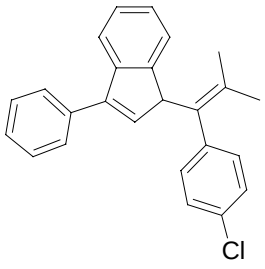

CH3
H3C


4.20
2.13
4.04
1.98
0.96
1.00
2.97
2.99
2.93
2.99
10
8
6
4
2
0
PPM

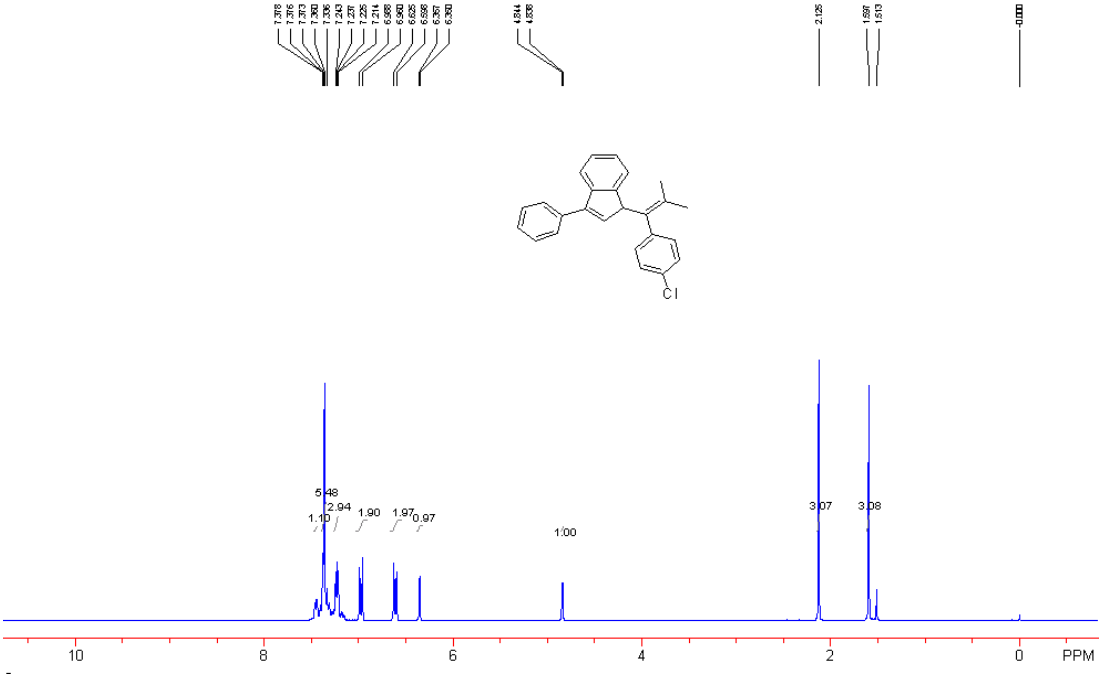


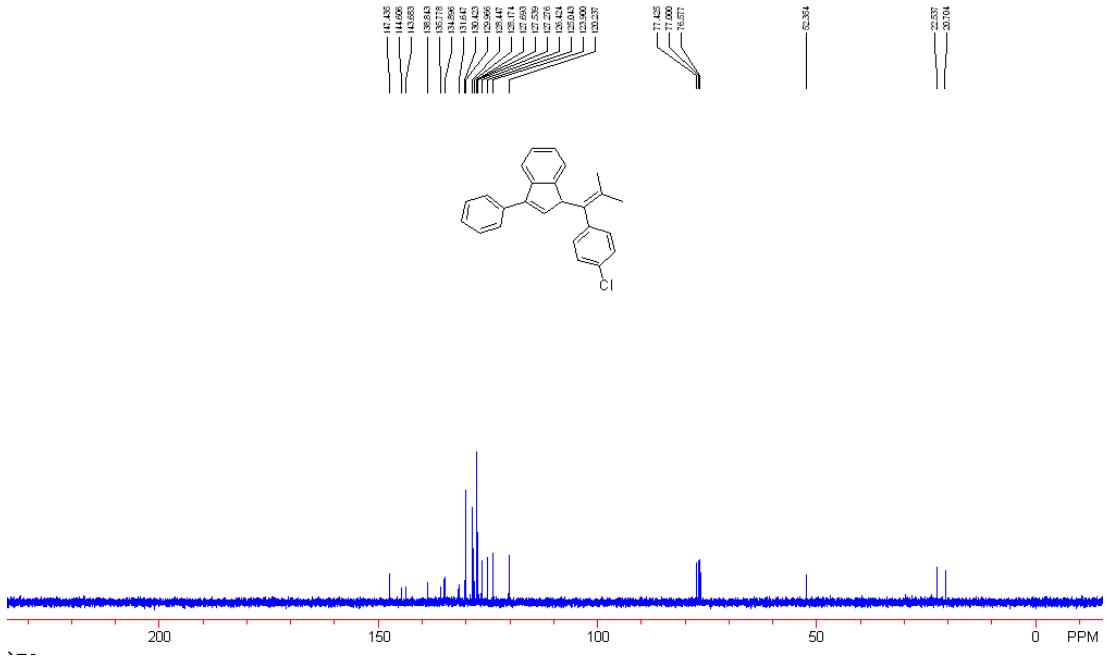

CH3
H3C

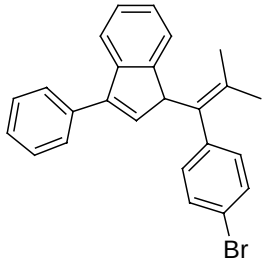

200
150
100
50
0
PPM


Cl

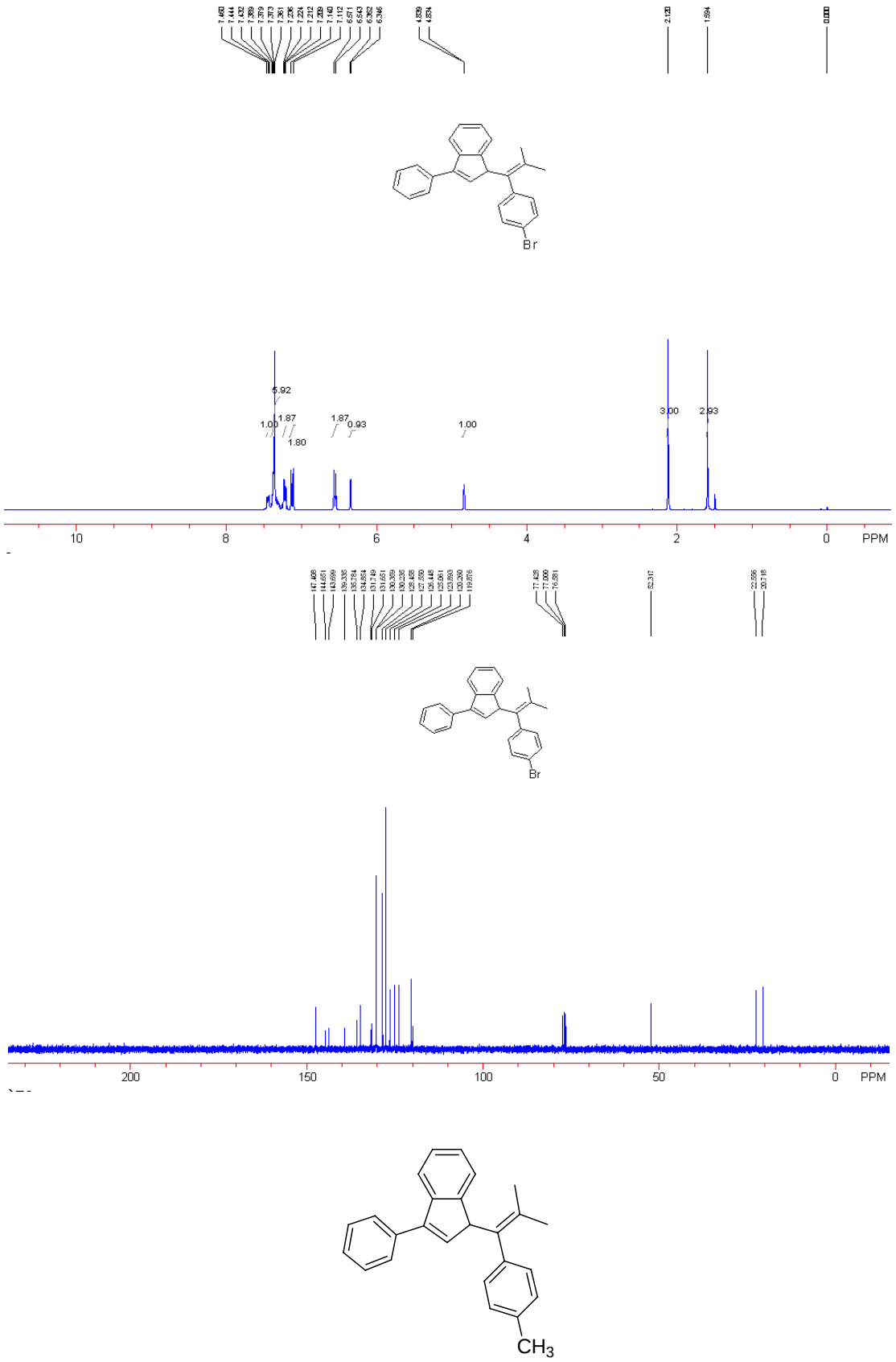
Compound **4e**. A yellow oil. IR (CH_2Cl_2): ν 3060, 3025, 2978, 2924, 2856, 1594, 1489, 1450, 1094, 1082, 1015, 820, 765 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.60 (3H, s, CH_3), 2.13 (3H, s, CH_3), 4.84 (1H, d, *J* = 1.8 Hz, CH), 6.35 (1H, d, *J* = 1.8 Hz, =CH), 6.61 (2H, d, *J* = 8.1 Hz, ArH), 6.98 (2H, d, *J* = 8.1 Hz, ArH), 7.22-7.24 (3H, m, ArH), 7.36-7.38 (5H, m, ArH), 7.44-7.47 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 22.5, 52.4, 120.2, 123.9, 125.0, 126.4, 127.3, 127.5, 127.7, 128.2, 128.4, 130.0, 130.4, 131.6, 134.9, 135.8, 138.8, 143.7, 144.6, 147.4; MS (EI) *m/z* (%): 356 (56) [M^+], 341 (88), 191 (45), 165 (100); HRMS (EI) Calcd. for $C_{25}H_{21}Cl$ (M^+) requires 356.1340, Found: 356.1332.





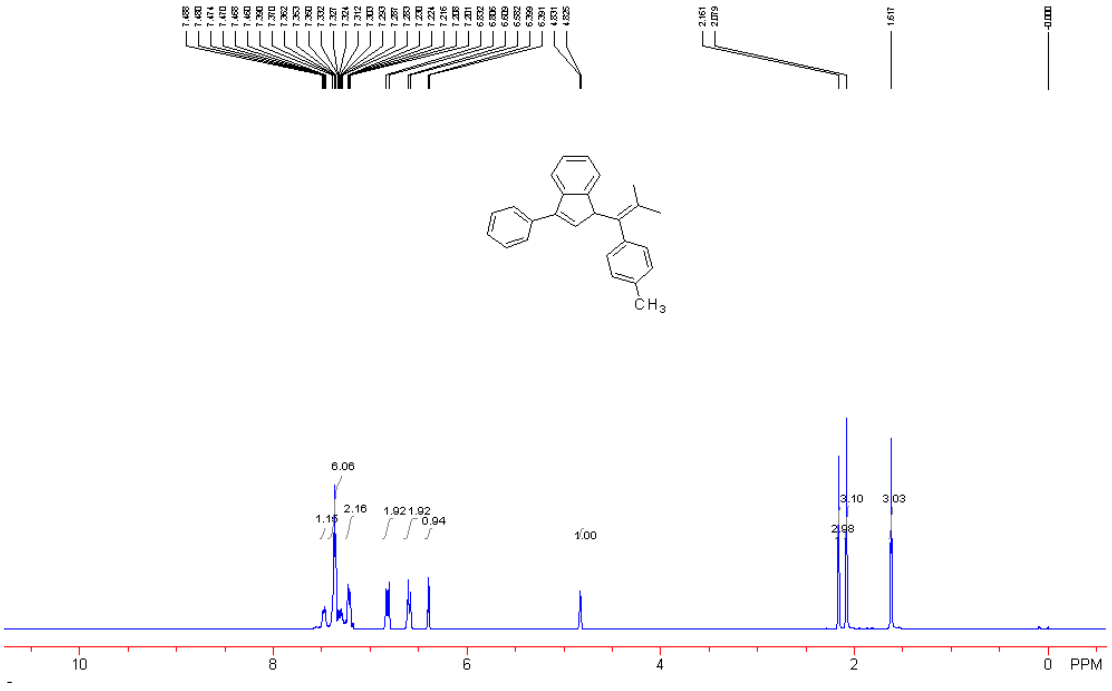


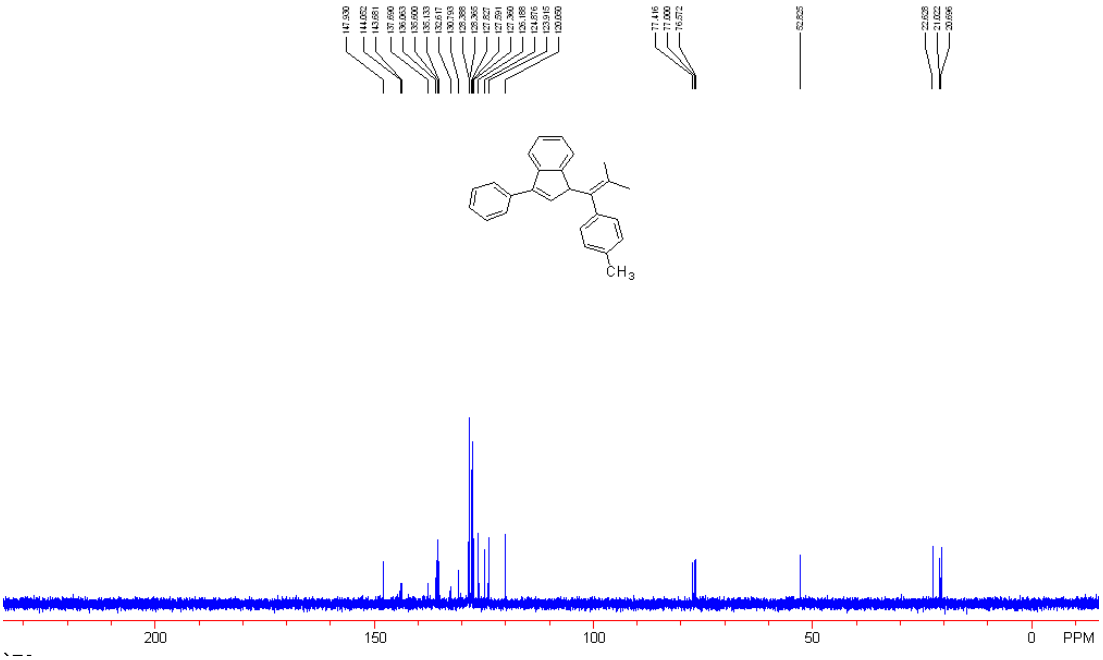
Compound **4f**. A yellow oil. IR (CH_2Cl_2): ν 3060, 3019, 2981, 2918, 2853, 1581, 1486, 1458, 1389, 1265, 1096, 1011, 821, 762 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.59 (3H, s, CH_3), 2.12 (3H, s, CH_3), 4.84 (1H, d, *J* = 1.8 Hz, CH), 6.35 (1H, d, *J* = 1.8 Hz, =CH), 6.56 (2H, d, *J* = 8.1 Hz, ArH), 7.13 (2H, d, *J* = 8.1 Hz, ArH), 7.21-7.24 (2H, m, ArH), 7.36-7.39 (6H, m, ArH), 7.43-7.46 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 22.6, 52.3, 119.9, 120.3, 123.9, 125.1, 126.4, 127.6, 128.5, 130.2, 130.4, 131.65, 131.75, 134.9, 135.8, 139.3, 143.7, 144.7, 147.4; MS (EI) *m/z* (%): 400 (31) [M^+], 385 (35), 191 (45), 306 (40), 191 (46), 130 (100); HRMS (EI) Calcd. for $C_{25}H_{21}Br$ (M^+) requires 400.0827, Found: 400.0858.

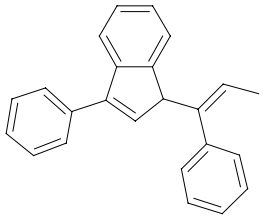


Compound **4g**. A yellow oil. IR (CH_2Cl_2): ν 3058, 3026, 2974, 2955, 2916, 2856, 1509, 1488,

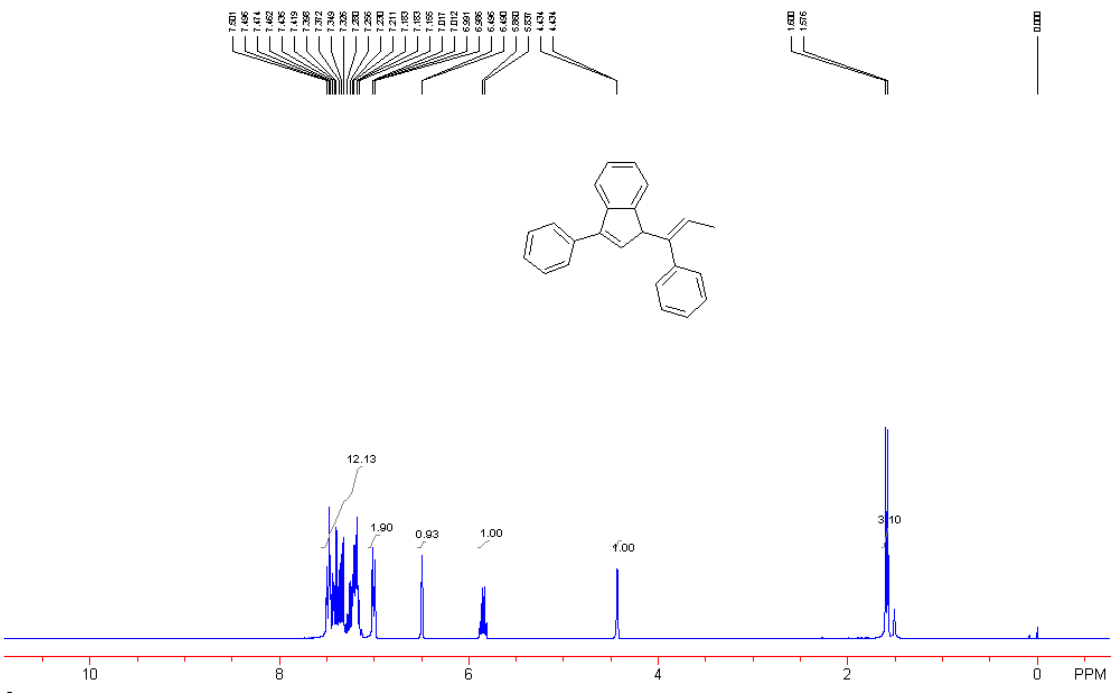
1456, 1443, 816 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.62 (3H, s, CH_3), 2.08 (3H, s, CH_3), 2.16 (3H, s, CH_3), 4.83 (1H, d, *J* = 1.8 Hz, CH), 6.40 (1H, d, *J* = 1.8 Hz, =CH), 6.60 (2H, d, *J* = 8.1 Hz, ArH), 6.82 (2H, d, *J* = 8.1 Hz, ArH), 7.20-7.23 (2H, m, ArH), 7.28-7.39 (6H, m, ArH), 7.46-7.49 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.7, 21.0, 22.6, 52.8, 120.1, 123.9, 124.9, 126.2, 127.4, 127.6, 127.8, 128.37, 128.39, 130.8, 132.6, 135.1, 135.6, 136.1, 137.7, 143.7, 144.1, 147.9; MS (EI) *m/z* (%): 336 (25) [M^+], 321 (34), 145 (100); HRMS (EI) Calcd. for $C_{26}H_{24}$ (M^+) requires 336.1878, Found: 336.1878.

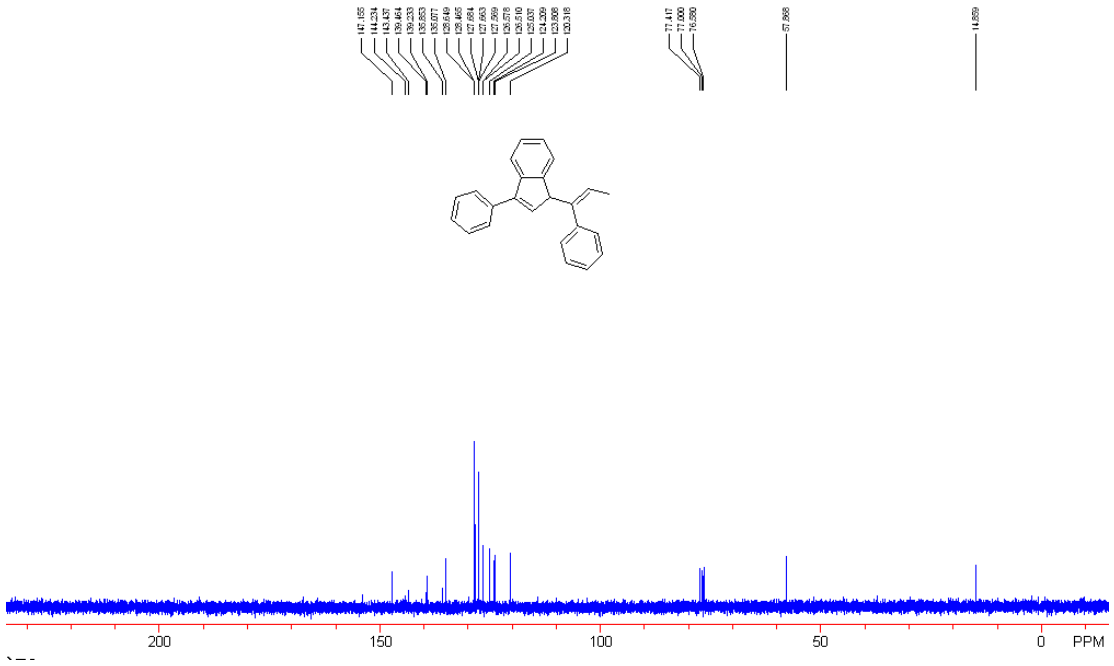


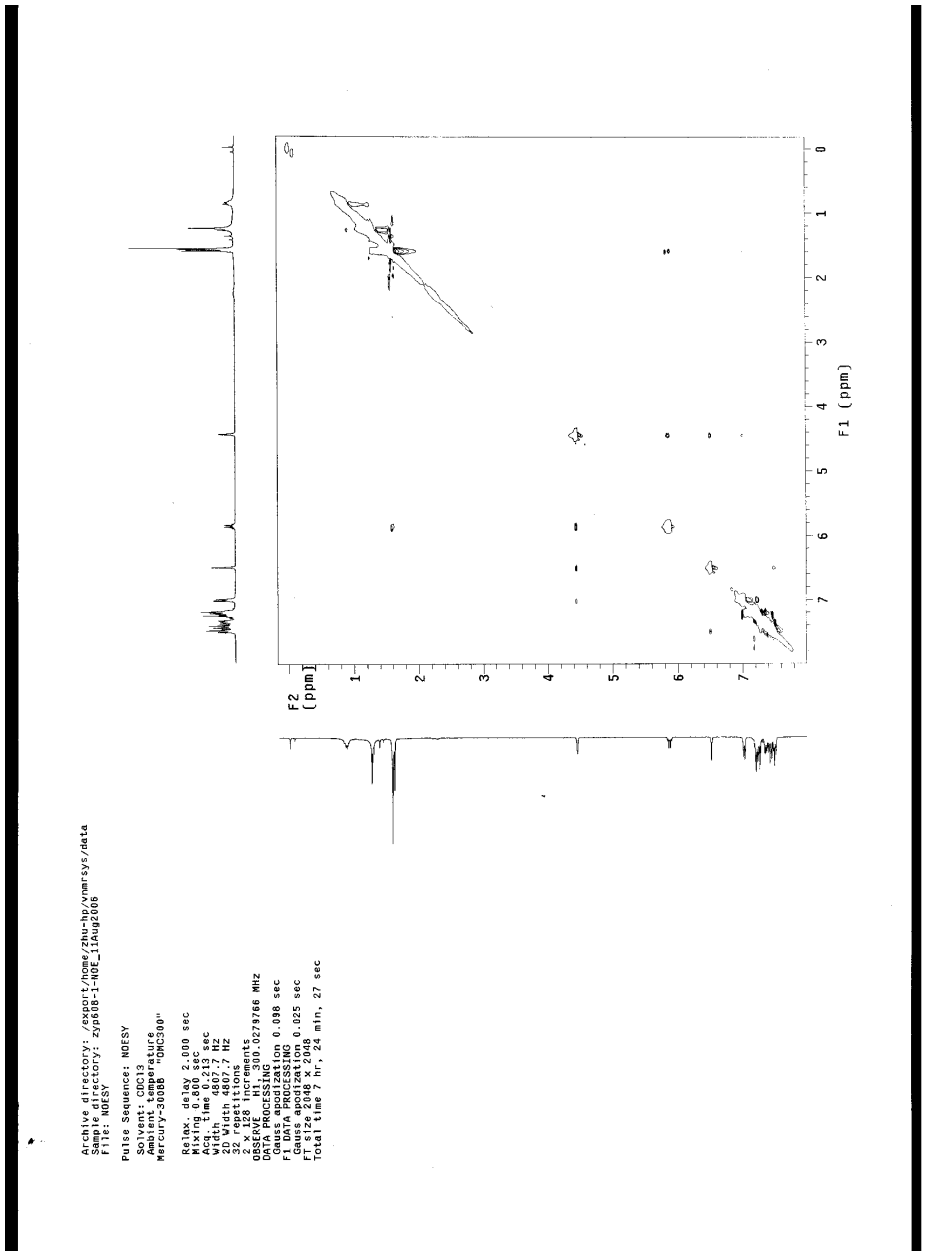




Compound **4h**. A yellow oil. IR (CH_2Cl_2): ν 3056, 3026, 2955, 2922, 2853, 1728, 1655, 1594, 1568, 1493, 1445, 1266, 1182, 1074, 1045, 767 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.59 (3H, d, *J* = 6.9 Hz, CH_3), 4.43 (1H, d, *J* = 1.8 Hz, CH), 5.85 (1H, q, *J* = 6.9 Hz, =CH), 6.49 (1H, d, *J* = 1.8 Hz, =CH), 6.99-7.02 (2H, m, ArH), 7.16-7.50 (12H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.9, 57.9, 120.3, 123.8, 124.2, 125.0, 126.5, 126.6, 127.6, 127.66, 127.68, 128.5, 128.6, 135.1, 135.9, 139.2, 139.5, 143.4, 147.2, 154.0; MS (EI) *m/z* (%): 348 (100) [M^+]; HRMS (EI) Calcd. for $C_{27}H_{24}$ (M^+) requires 348.1878, Found: 348.1881.


12.13
1.90
0.93
1.00
1.00
3.10
10
8
6
4
2
0
PPM


200
150
100
50
0
PPM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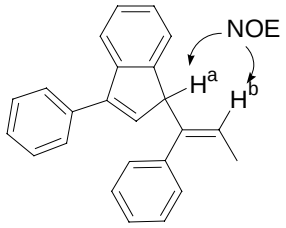
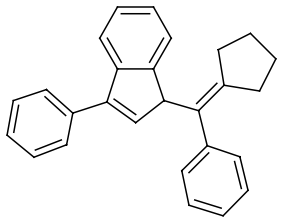
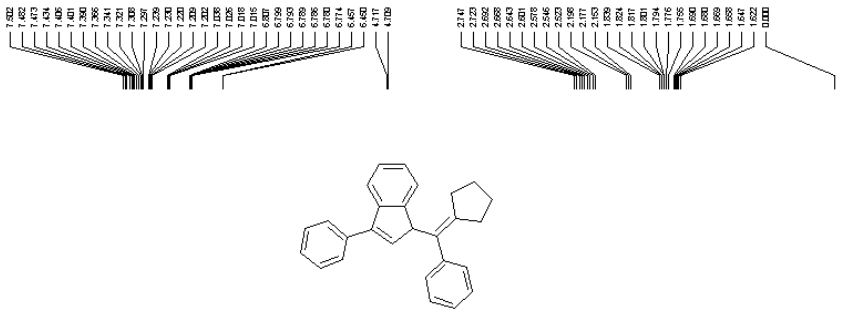
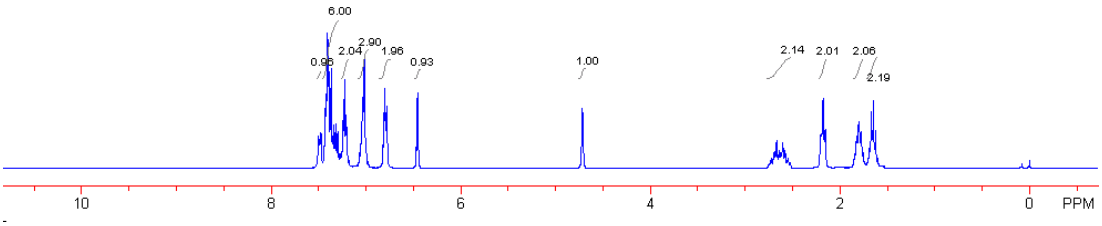
Sample directory: zyp008-1-NOE_1HAd2008
File: NOESY
Pulse Sequence: NOESY
Solvent: CDCl3
Ambient temperature
Mercury-300BB "OMC300"
Relax. delay 2.000 sec
Mixing 0.800 sec
Acq. time 0.213 sec
Width 4807.7 Hz
2D Width 4807.7 Hz
256 t1 increments
32 x 128 increments
OBSERVE H1, 300.0279766 MHz
DATA PROCESSING
Lorentz-Gauss apodization 0.098 sec
F1 DATA PROCESSING
Gauss apodization 0.025 sec
F2 Size 2048 x 2048
Total time 7 hr, 24 min, 27 sec
F2 (ppm)
F1 (ppm)

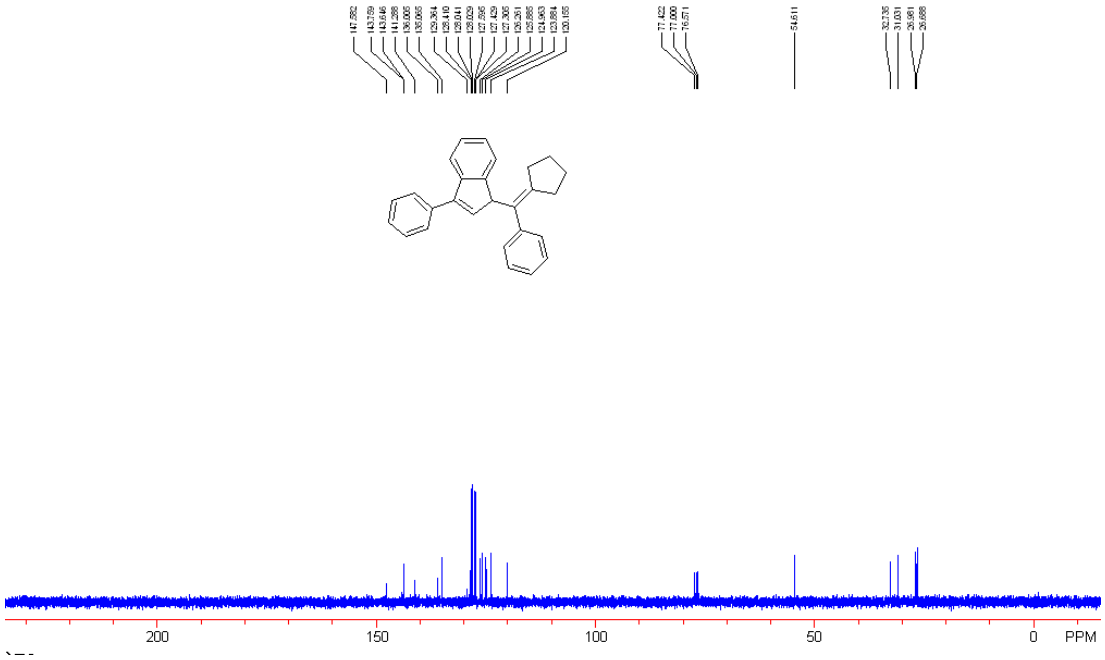

NOE
Ha
Hb

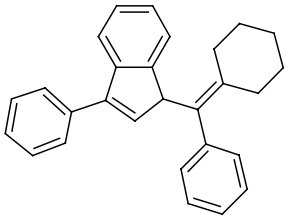


Compound **4j**. A yellow oil. IR (CH_2Cl_2): ν 3051, 3032, 2951, 2923, 2866, 1597, 1491, 1443, 752 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.62-1.69 (2H, m), 1.76-1.84 (2H, m), 2.15-2.20 (2H, m), 2.52-2.75 (2H, m), 4.71 (1H, d, *J* = 2.1 Hz, CH), 6.45 (1H, d, *J* = 2.1 Hz, =CH), 6.77-6.81 (2H, m, ArH), 7.02-7.04 (3H, m, ArH), 7.20-7.24 (2H, m, ArH), 7.30-7.43 (6H, m, ArH), 7.47-7.50 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.7, 27.0, 31.0, 32.7, 54.6, 120.2, 123.9, 125.0, 125.9, 126.3, 127.3, 127.4, 127.6, 128.03, 128.04, 128.4, 129.4, 135.1, 136.0, 141.3, 143.6, 143.8, 147.6; MS (EI) *m/z* (%): 348 (100) [M^+]; HRMS (EI) Calcd. for $C_{27}H_{24}$ (M^+) requires 348.1878, Found: 348.1881.

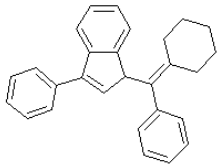


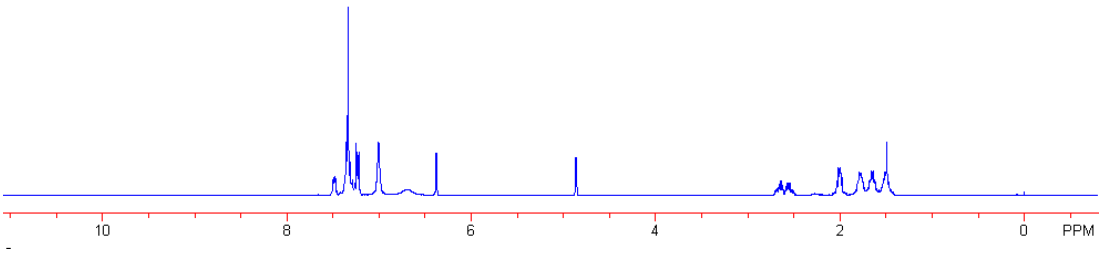


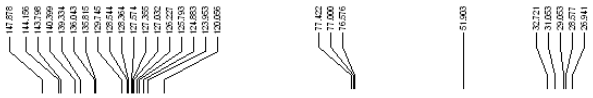


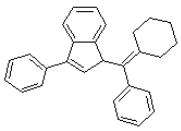


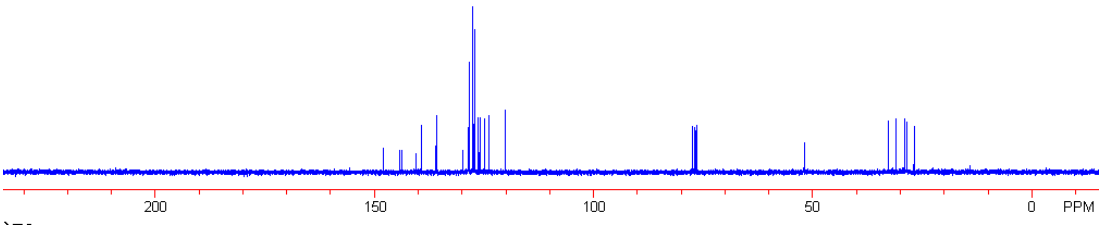
Compound **4k**. A yellow oil. IR (CH_2Cl_2): ν 3058, 3026, 2927, 2854, 1597, 1488, 1463, 1455, 1376, 1232, 1028 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 1.47-1.52 (2H, m), 1.62-1.68 (2H, m), 1.75-1.81 (2H, m), 1.98-2.03 (2H, m), 2.50-2.70 (2H, m), 4.86 (1H, d, *J* = 1.2 Hz, CH), 6.38 (1H, d, *J* = 1.2 Hz, =CH), 6.69 (2H, br, ArH), 7.01 (3H, s, ArH), 7.22-7.25 (2H, m, ArH), 7.29-7.37 (6H, m, ArH), 7.48-7.50 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.9, 28.6, 29.1, 31.1, 32.7, 51.9, 120.1, 124.0, 124.9, 125.8, 126.2, 127.0, 127.4, 127.6, 128.4, 128.5, 129.7, 135.8, 136.0, 139.3, 140.4, 143.8, 144.2, 147.9; MS (EI) *m/z* (%): 362 (100) [M^+]; HRMS (EI) Calcd. for $C_{28}H_{26}$ (M^+) requires 362.2035, Found: 362.2039.

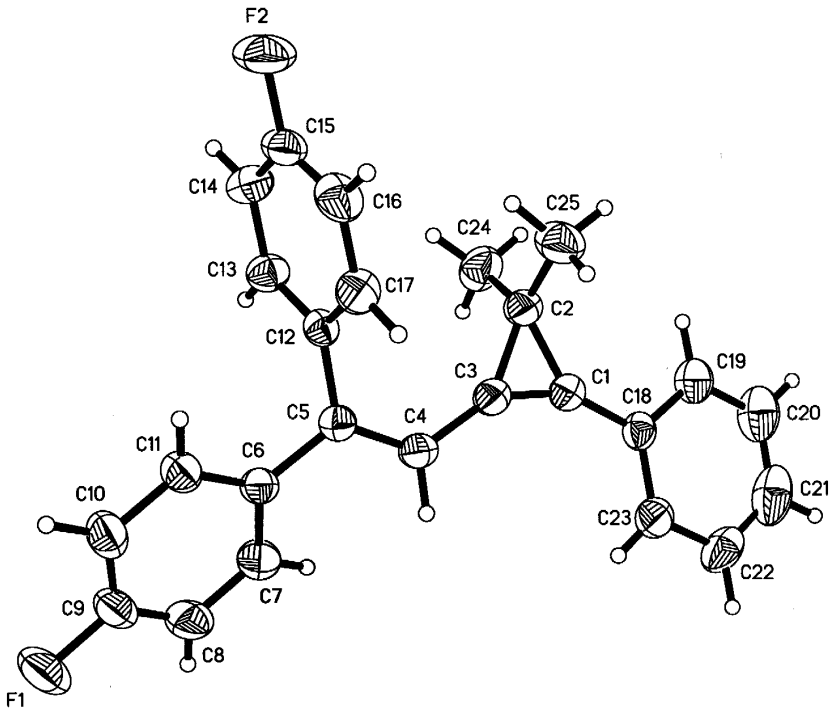



10
8
6
4
2
0
PPM






200
150
100
50
0
PPM



The crystal data of **2b** has been deposited in CCDC with number 602862. Empirical Formula: $C_{25}H_{20}F_2$; Formula Weight: 358.41; Crystal Color, Habit: colorless, prismatic; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 12.5896(12)Å, b = 9.1294(8)Å, c = 17.6833(16)Å, $\alpha = 90^o$, $\beta = 102.192(2)^o$, $\gamma = 90^o$, V = 1986.6(3)Å^3; Space group: P2(1)/n; Z = 4; D_{calc} = 1.198 g/cm^3; F_{000} = 752; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0431, 0.1098.

Table 1. Crystal data and structure refinement for cd26102.

Identification code	cd26102
Empirical formula	C25 H20 F2
Formula weight	358.41
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 12.5896(12) A alpha = 90 deg. b = 9.1294(8) A beta = 102.192(2) deg. c = 17.6833(16) A gamma = 90 deg.
Volume	1986.6(3) A^3
Z, Calculated density	4, 1.198 Mg/m^3
Absorption coefficient	0.081 mm^-1
F(000)	752
Crystal size	0.502 x 0.487 x 0.455 mm
Theta range for data collection	1.82 to 27.49 deg.
Limiting indices	-16<=h<=15, -9<=k<=11, -22<=l<=22
Reflections collected / unique	11560 / 4495 [R(int) = 0.0496]
Completeness to theta = 27.49	98.6 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.55087
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4495 / 0 / 287
Goodness-of-fit on F^2	0.895
Final R indices [I>2sigma(I)]	R1 = 0.0431, wR2 = 0.1098
R indices (all data)	R1 = 0.0733, wR2 = 0.1210
Extinction coefficient	0.0116(16)
Largest diff. peak and hole	0.178 and -0.144 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd26102.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
F(1)	4548(1)	8826(1)	-592(1)	119(1)
F(2)	2320(1)	8094(1)	4212(1)	112(1)
C(1)	5595(1)	2212(2)	3217(1)	60(1)
C(2)	5230(1)	3336(2)	3722(1)	65(1)
C(3)	5135(1)	3371(2)	2858(1)	61(1)
C(4)	4954(1)	4246(2)	2175(1)	61(1)
C(5)	4508(1)	5592(1)	2089(1)	52(1)
C(6)	4543(1)	6478(1)	1393(1)	54(1)
C(7)	5335(2)	6257(2)	965(1)	78(1)
C(8)	5333(2)	7042(2)	298(1)	91(1)
C(9)	4556(2)	8066(2)	72(1)	80(1)
C(10)	3791(2)	8373(2)	482(1)	77(1)
C(11)	3784(1)	7577(2)	1145(1)	64(1)
C(12)	3944(1)	6233(1)	2672(1)	50(1)
C(13)	4353(1)	7436(2)	3108(1)	64(1)
C(14)	3813(2)	8052(2)	3631(1)	75(1)
C(15)	2860(1)	7472(2)	3705(1)	71(1)
C(16)	2412(1)	6310(2)	3289(1)	80(1)
C(17)	2969(1)	5674(2)	2773(1)	69(1)
C(18)	6170(1)	844(1)	3232(1)	57(1)
C(19)	6587(1)	180(2)	3938(1)	74(1)
C(20)	7137(2)	-1115(2)	3979(1)	101(1)
C(21)	7297(2)	-1769(2)	3331(2)	111(1)
C(22)	6908(2)	-1157(2)	2617(1)	96(1)
C(23)	6323(1)	161(2)	2563(1)	75(1)
C(24)	6070(2)	4314(2)	4215(1)	93(1)
C(25)	4248(2)	3009(2)	4059(1)	93(1)

Table 3. Bond lengths [A] and angles [deg] for cd26102.

F(1)-C(9)	1.3613(17)
F(2)-C(15)	1.3587(16)
C(1)-C(3)	1.3039(19)
C(1)-C(18)	1.4407(19)
C(1)-C(2)	1.4954(18)
C(2)-C(3)	1.5077(18)
C(2)-C(25)	1.511(2)
C(2)-C(24)	1.511(2)
C(3)-C(4)	1.4258(19)
C(4)-C(5)	1.3454(19)
C(4)-H(5)	0.970(14)
C(5)-C(6)	1.4811(17)
C(5)-C(12)	1.4897(17)
C(6)-C(7)	1.389(2)
C(6)-C(11)	1.3914(19)
C(7)-C(8)	1.379(2)
C(7)-H(7)	0.947(18)
C(8)-C(9)	1.351(3)
C(8)-H(8)	0.92(2)
C(9)-C(10)	1.351(2)
C(10)-C(11)	1.380(2)
C(10)-H(10)	0.976(17)
C(11)-H(11)	0.949(15)
C(12)-C(17)	1.3757(19)
C(12)-C(13)	1.3766(19)
C(13)-C(14)	1.379(2)
C(13)-H(13)	0.966(18)
C(14)-C(15)	1.343(2)
C(14)-H(14)	0.936(17)
C(15)-C(16)	1.345(2)
C(16)-C(17)	1.390(2)
C(16)-H(16)	0.942(19)
C(17)-H(17)	0.976(16)
C(18)-C(23)	1.3859(19)
C(18)-C(19)	1.388(2)
C(19)-C(20)	1.364(2)
C(19)-H(19)	0.978(16)
C(20)-C(21)	1.345(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.372(3)
C(21)-H(21)	0.9300
C(22)-C(23)	1.403(2)
C(22)-H(22)	0.9300
C(23)-H(23)	0.9300
C(24)-H(24A)	0.9600
C(24)-H(24B)	0.9600
C(24)-H(24C)	0.9600
C(25)-H(25A)	0.9600
C(25)-H(25B)	0.9600
C(25)-H(25C)	0.9600
C(3)-C(1)-C(18)	151.87(14)
C(3)-C(1)-C(2)	64.75(10)
C(18)-C(1)-C(2)	143.10(13)
C(1)-C(2)-C(3)	51.46(8)
C(1)-C(2)-C(25)	118.72(13)
C(3)-C(2)-C(25)	120.45(13)
C(1)-C(2)-C(24)	118.94(13)
C(3)-C(2)-C(24)	117.87(13)
C(25)-C(2)-C(24)	114.89(13)
C(1)-C(3)-C(4)	147.34(14)
C(1)-C(3)-C(2)	63.78(10)
C(4)-C(3)-C(2)	146.53(13)
C(5)-C(4)-C(3)	126.62(13)
C(5)-C(4)-H(5)	118.5(8)
C(3)-C(4)-H(5)	114.8(8)
C(4)-C(5)-C(6)	120.54(12)
C(4)-C(5)-C(12)	121.92(12)

C(6)-C(5)-C(12)	117.53(11)
C(7)-C(6)-C(11)	117.20(14)
C(7)-C(6)-C(5)	121.62(13)
C(11)-C(6)-C(5)	121.18(12)
C(8)-C(7)-C(6)	121.21(18)
C(8)-C(7)-H(7)	119.9(11)
C(6)-C(7)-H(7)	118.9(11)
C(9)-C(8)-C(7)	118.94(18)
C(9)-C(8)-H(8)	118.8(12)
C(7)-C(8)-H(8)	122.1(12)
C(8)-C(9)-C(10)	122.52(16)
C(8)-C(9)-F(1)	118.75(17)
C(10)-C(9)-F(1)	118.73(18)
C(9)-C(10)-C(11)	118.68(17)
C(9)-C(10)-H(10)	119.3(9)
C(11)-C(10)-H(10)	122.0(10)
C(10)-C(11)-C(6)	121.37(16)
C(10)-C(11)-H(11)	120.1(9)
C(6)-C(11)-H(11)	118.4(9)
C(17)-C(12)-C(13)	117.52(13)
C(17)-C(12)-C(5)	120.83(13)
C(13)-C(12)-C(5)	121.60(12)
C(12)-C(13)-C(14)	121.44(15)
C(12)-C(13)-H(13)	115.7(9)
C(14)-C(13)-H(13)	122.8(10)
C(15)-C(14)-C(13)	118.80(17)
C(15)-C(14)-H(14)	118.0(11)
C(13)-C(14)-H(14)	123.2(11)
C(14)-C(15)-C(16)	122.51(15)
C(14)-C(15)-F(2)	118.80(17)
C(16)-C(15)-F(2)	118.69(16)
C(15)-C(16)-C(17)	118.52(16)
C(15)-C(16)-H(16)	124.3(10)
C(17)-C(16)-H(16)	117.2(10)
C(12)-C(17)-C(16)	121.18(17)
C(12)-C(17)-H(17)	116.2(9)
C(16)-C(17)-H(17)	122.6(9)
C(23)-C(18)-C(19)	118.44(14)
C(23)-C(18)-C(1)	122.27(13)
C(19)-C(18)-C(1)	119.29(13)
C(20)-C(19)-C(18)	121.21(18)
C(20)-C(19)-H(19)	121.3(9)
C(18)-C(19)-H(19)	117.4(9)
C(21)-C(20)-C(19)	120.3(2)
C(21)-C(20)-H(20)	119.8
C(19)-C(20)-H(20)	119.8
C(20)-C(21)-C(22)	120.95(19)
C(20)-C(21)-H(21)	119.5
C(22)-C(21)-H(21)	119.5
C(21)-C(22)-C(23)	119.46(17)
C(21)-C(22)-H(22)	120.3
C(23)-C(22)-H(22)	120.3
C(18)-C(23)-C(22)	119.60(16)
C(18)-C(23)-H(23)	120.2
C(22)-C(23)-H(23)	120.2
C(2)-C(24)-H(24A)	109.5
C(2)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(2)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
C(2)-C(25)-H(25A)	109.5
C(2)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(2)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd26102.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
F(1)	187(1)	109(1)	62(1)	17(1)	29(1)	-42(1)
F(2)	114(1)	153(1)	84(1)	7(1)	54(1)	50(1)
C(1)	65(1)	56(1)	59(1)	0(1)	15(1)	4(1)
C(2)	77(1)	62(1)	55(1)	2(1)	14(1)	13(1)
C(3)	70(1)	56(1)	57(1)	-2(1)	14(1)	8(1)
C(4)	71(1)	62(1)	52(1)	-3(1)	17(1)	9(1)
C(5)	51(1)	54(1)	51(1)	-1(1)	10(1)	1(1)
C(6)	58(1)	56(1)	51(1)	-3(1)	14(1)	-3(1)
C(7)	89(1)	78(1)	78(1)	3(1)	40(1)	8(1)
C(8)	120(2)	90(1)	79(1)	-4(1)	57(1)	-15(1)
C(9)	118(2)	72(1)	50(1)	4(1)	18(1)	-28(1)
C(10)	92(1)	71(1)	64(1)	13(1)	5(1)	-3(1)
C(11)	66(1)	68(1)	58(1)	6(1)	13(1)	3(1)
C(12)	47(1)	51(1)	50(1)	6(1)	11(1)	2(1)
C(13)	59(1)	69(1)	66(1)	-8(1)	20(1)	-5(1)
C(14)	80(1)	79(1)	67(1)	-15(1)	21(1)	7(1)
C(15)	75(1)	87(1)	56(1)	13(1)	28(1)	26(1)
C(16)	60(1)	93(1)	94(1)	19(1)	36(1)	5(1)
C(17)	61(1)	68(1)	82(1)	-1(1)	23(1)	-6(1)
C(18)	54(1)	50(1)	70(1)	4(1)	19(1)	-1(1)
C(19)	64(1)	69(1)	86(1)	9(1)	8(1)	7(1)
C(20)	75(1)	85(1)	137(2)	24(1)	10(1)	20(1)
C(21)	76(1)	72(1)	197(3)	12(2)	52(2)	14(1)
C(22)	93(1)	69(1)	146(2)	-30(1)	73(1)	-14(1)
C(23)	84(1)	63(1)	90(1)	-5(1)	43(1)	-11(1)
C(24)	119(2)	76(1)	73(1)	-9(1)	-4(1)	9(1)
C(25)	99(1)	107(1)	83(1)	16(1)	40(1)	28(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd26102.

	x	y	z	U(eq)
H(20)	7403	-1548	4458	121
H(21)	7677	-2648	3366	134
H(22)	7030	-1612	2173	115
H(23)	6041	574	2082	90
H(24A)	6328	3864	4710	139
H(24B)	5748	5245	4283	139
H(24C)	6667	4455	3963	139
H(25A)	3745	2418	3702	140
H(25B)	3903	3911	4149	140
H(25C)	4472	2492	4539	140
H(5)	5222(11)	3828(14)	1747(8)	66(4)
H(7)	5882(15)	5547(19)	1135(10)	101(6)
H(8)	5869(16)	6941(19)	24(10)	109(6)
H(10)	3286(14)	9178(18)	317(9)	91(5)
H(11)	3224(12)	7726(15)	1422(8)	73(4)
H(13)	5035(15)	7812(17)	3021(9)	91(5)
H(14)	4060(14)	8890(19)	3921(10)	99(6)
H(16)	1742(16)	5889(19)	3325(9)	100(6)
H(17)	2690(13)	4828(18)	2455(9)	94(5)
H(19)	6475(13)	677(17)	4405(9)	91(5)

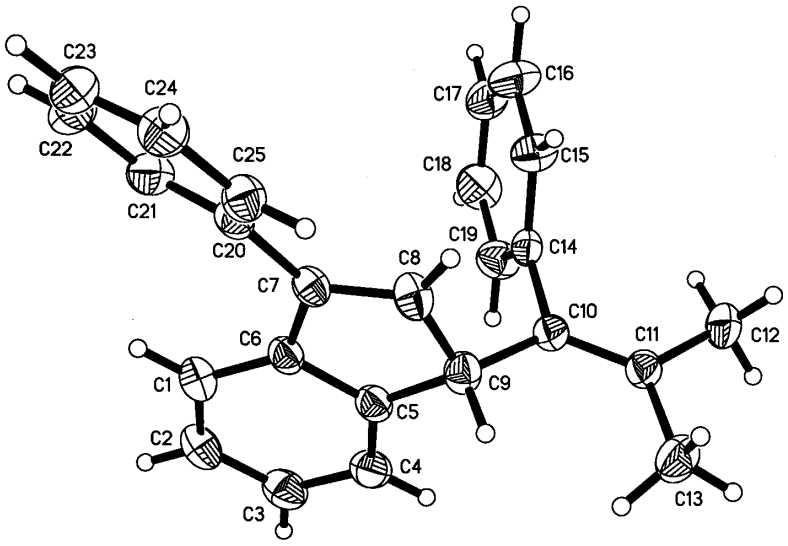
Table 6. Torsion angles [deg] for cd26102.

C(18)-C(1)-C(2)-C(3)	174.8(2)
C(3)-C(1)-C(2)-C(25)	107.61(15)
C(18)-C(1)-C(2)-C(25)	-77.6(2)
C(3)-C(1)-C(2)-C(24)	-104.03(15)
C(18)-C(1)-C(2)-C(24)	70.8(2)
C(18)-C(1)-C(3)-C(4)	-10.7(5)
C(2)-C(1)-C(3)-C(4)	162.7(3)
C(18)-C(1)-C(3)-C(2)	-173.4(3)
C(25)-C(2)-C(3)-C(1)	-104.14(15)
C(24)-C(2)-C(3)-C(1)	106.18(15)
C(1)-C(2)-C(3)-C(4)	-163.1(3)
C(25)-C(2)-C(3)-C(4)	92.8(3)
C(24)-C(2)-C(3)-C(4)	-56.9(3)
C(1)-C(3)-C(4)-C(5)	-168.8(2)
C(2)-C(3)-C(4)-C(5)	-17.7(3)
C(3)-C(4)-C(5)-C(6)	169.30(13)
C(3)-C(4)-C(5)-C(12)	-12.1(2)
C(4)-C(5)-C(6)-C(7)	-24.4(2)
C(12)-C(5)-C(6)-C(7)	156.90(13)
C(4)-C(5)-C(6)-C(11)	155.98(14)
C(12)-C(5)-C(6)-C(11)	-22.70(18)
C(11)-C(6)-C(7)-C(8)	-3.3(2)
C(5)-C(6)-C(7)-C(8)	177.05(15)
C(6)-C(7)-C(8)-C(9)	1.5(3)
C(7)-C(8)-C(9)-C(10)	1.4(3)
C(7)-C(8)-C(9)-F(1)	-179.20(15)
C(8)-C(9)-C(10)-C(11)	-2.2(3)
F(1)-C(9)-C(10)-C(11)	178.42(13)
C(9)-C(10)-C(11)-C(6)	0.1(2)
C(7)-C(6)-C(11)-C(10)	2.5(2)
C(5)-C(6)-C(11)-C(10)	-177.85(13)
C(4)-C(5)-C(12)-C(17)	-68.92(18)
C(6)-C(5)-C(12)-C(17)	109.74(14)
C(4)-C(5)-C(12)-C(13)	113.62(16)
C(6)-C(5)-C(12)-C(13)	-67.72(16)
C(17)-C(12)-C(13)-C(14)	0.6(2)
C(5)-C(12)-C(13)-C(14)	178.11(13)
C(12)-C(13)-C(14)-C(15)	-1.0(2)
C(13)-C(14)-C(15)-C(16)	0.1(3)
C(13)-C(14)-C(15)-F(2)	-179.00(13)
C(14)-C(15)-C(16)-C(17)	1.1(2)
F(2)-C(15)-C(16)-C(17)	-179.80(13)
C(13)-C(12)-C(17)-C(16)	0.7(2)
C(5)-C(12)-C(17)-C(16)	-176.90(13)
C(15)-C(16)-C(17)-C(12)	-1.5(2)
C(3)-C(1)-C(18)-C(23)	-9.7(4)
C(2)-C(1)-C(18)-C(23)	-179.66(17)
C(3)-C(1)-C(18)-C(19)	170.3(3)
C(2)-C(1)-C(18)-C(19)	0.3(3)
C(23)-C(18)-C(19)-C(20)	0.0(2)
C(1)-C(18)-C(19)-C(20)	-179.95(15)
C(18)-C(19)-C(20)-C(21)	0.8(3)
C(19)-C(20)-C(21)-C(22)	-0.4(3)
C(20)-C(21)-C(22)-C(23)	-0.7(3)
C(19)-C(18)-C(23)-C(22)	-1.2(2)
C(1)-C(18)-C(23)-C(22)	178.81(13)
C(21)-C(22)-C(23)-C(18)	1.5(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd26102 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)



The crystal data of **4a** has been deposited in CCDC with number 615026. Empirical Formula: $C_{25}H_{22}$; Formula Weight: 322.43; Crystal Color, Habit: colorless, prismatic; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: a = 8.1168(19)Å, b = 10.068(2)Å, c = 11.493(16)Å, α = 93.001(4)^o, β = 98.563(4)^o, γ = 98.080(4)^o, V = 917.0(4)Å^3; Space group: P-1; Z = 2; D_{calc} = 1.168 g/cm^3; F_{000} = 344; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0655, 0.1722.

Table 1. Crystal data and structure refinement for cd26278.

Identification code	cd26278
Empirical formula	C25 H22
Formula weight	322.43
Temperature	292(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 8.1168(19) A alpha = 93.001(4) deg. b = 10.068(2) A beta = 98.563(4) deg. c = 11.493(3) A gamma = 98.080(4) deg.
Volume	917.0(4) A^3
Z, Calculated density	2, 1.168 Mg/m^3
Absorption coefficient	0.066 mm^-1
F(000)	344
Crystal size	0.358 x 0.321 x 0.222 mm
Theta range for data collection	1.80 to 26.00 deg.
Limiting indices	-8<=h<=10, -12<=k<=12, -14<=l<=10
Reflections collected / unique	4729 / 3485 [R(int) = 0.0710]
Completeness to theta = 26.00	97.2 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.58108
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3485 / 0 / 232
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0655, wR2 = 0.1722
R indices (all data)	R1 = 0.0933, wR2 = 0.1867
Largest diff. peak and hole	0.217 and -0.222 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd26278.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	3427(3)	6623(3)	4395(2)	54(1)
C(2)	1854(3)	6394(3)	4739(2)	62(1)
C(3)	1532(3)	7045(3)	5748(3)	63(1)
C(4)	2770(3)	7952(3)	6436(2)	56(1)
C(5)	4332(3)	8209(2)	6096(2)	45(1)
C(6)	4685(3)	7538(2)	5078(2)	44(1)
C(7)	6423(3)	8070(2)	4928(2)	44(1)
C(8)	7060(3)	9029(2)	5789(2)	52(1)
C(9)	5838(3)	9247(2)	6615(2)	49(1)
C(10)	6514(3)	9170(2)	7911(2)	42(1)
C(11)	6698(3)	10193(2)	8731(2)	46(1)
C(12)	7434(4)	10095(3)	9988(2)	65(1)
C(13)	6242(4)	11561(3)	8475(3)	67(1)
C(14)	7063(3)	7852(2)	8204(2)	43(1)
C(15)	8682(4)	7618(3)	8139(2)	61(1)
C(16)	9211(4)	6422(3)	8423(3)	77(1)
C(17)	8123(4)	5412(3)	8762(3)	72(1)
C(18)	6510(4)	5603(3)	8820(3)	73(1)
C(19)	5976(4)	6811(3)	8551(3)	61(1)
C(20)	7270(3)	7681(2)	3932(2)	45(1)
C(21)	7003(3)	6371(3)	3416(2)	58(1)
C(22)	7758(4)	6057(3)	2453(3)	69(1)
C(23)	8800(4)	7033(3)	2004(3)	69(1)
C(24)	9122(4)	8311(3)	2530(2)	61(1)
C(25)	8361(3)	8639(3)	3481(2)	52(1)

Table 3. Bond lengths [A] and angles [deg] for cd26278.

C(1)-C(2)	1.385(4)
C(1)-C(6)	1.388(3)
C(1)-H(1)	0.9300
C(2)-C(3)	1.378(4)
C(2)-H(2)	0.9300
C(3)-C(4)	1.378(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.376(4)
C(4)-H(4)	0.9300
C(5)-C(6)	1.407(3)
C(5)-C(9)	1.516(3)
C(6)-C(7)	1.476(3)
C(7)-C(8)	1.336(3)
C(7)-C(20)	1.483(3)
C(8)-C(9)	1.502(4)
C(8)-H(8)	0.9300
C(9)-C(10)	1.519(3)
C(9)-H(9)	0.98(3)
C(10)-C(11)	1.335(3)
C(10)-C(14)	1.497(3)
C(11)-C(12)	1.493(3)
C(11)-C(13)	1.508(4)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-C(15)	1.379(4)
C(14)-C(19)	1.388(3)
C(15)-C(16)	1.373(4)
C(15)-H(15)	0.9300
C(16)-C(17)	1.363(4)
C(16)-H(16)	0.9300
C(17)-C(18)	1.360(4)
C(17)-H(17)	0.9300
C(18)-C(19)	1.382(4)
C(18)-H(18)	0.9300
C(19)-H(19)	0.9300
C(20)-C(25)	1.390(3)
C(20)-C(21)	1.391(3)
C(21)-C(22)	1.386(4)
C(21)-H(21)	0.9300
C(22)-C(23)	1.377(4)
C(22)-H(22)	0.9300
C(23)-C(24)	1.366(4)
C(23)-H(23)	0.9300
C(24)-C(25)	1.381(4)
C(24)-H(24)	0.9300
C(25)-H(25)	0.9300

C(2)-C(1)-C(6)	118.9(2)
C(2)-C(1)-H(1)	120.6
C(6)-C(1)-H(1)	120.6
C(3)-C(2)-C(1)	121.1(2)
C(3)-C(2)-H(2)	119.5
C(1)-C(2)-H(2)	119.5
C(4)-C(3)-C(2)	120.7(3)
C(4)-C(3)-H(3)	119.6
C(2)-C(3)-H(3)	119.6
C(5)-C(4)-C(3)	119.0(3)
C(5)-C(4)-H(4)	120.5
C(3)-C(4)-H(4)	120.5
C(4)-C(5)-C(6)	120.9(2)
C(4)-C(5)-C(9)	129.6(2)
C(6)-C(5)-C(9)	109.4(2)
C(1)-C(6)-C(5)	119.4(2)
C(1)-C(6)-C(7)	132.6(2)

C(5)-C(6)-C(7)	107.8(2)
C(8)-C(7)-C(6)	108.7(2)
C(8)-C(7)-C(20)	125.7(2)
C(6)-C(7)-C(20)	125.5(2)
C(7)-C(8)-C(9)	112.6(2)
C(7)-C(8)-H(8)	123.7
C(9)-C(8)-H(8)	123.7
C(8)-C(9)-C(5)	101.5(2)
C(8)-C(9)-C(10)	114.7(2)
C(5)-C(9)-C(10)	116.03(19)
C(8)-C(9)-H(9)	108.9(14)
C(5)-C(9)-H(9)	107.6(15)
C(10)-C(9)-H(9)	107.8(14)
C(11)-C(10)-C(14)	121.4(2)
C(11)-C(10)-C(9)	124.2(2)
C(14)-C(10)-C(9)	114.26(19)
C(10)-C(11)-C(12)	122.6(2)
C(10)-C(11)-C(13)	123.7(2)
C(12)-C(11)-C(13)	113.7(2)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(15)-C(14)-C(19)	116.8(2)
C(15)-C(14)-C(10)	121.3(2)
C(19)-C(14)-C(10)	121.9(2)
C(16)-C(15)-C(14)	121.9(3)
C(16)-C(15)-H(15)	119.0
C(14)-C(15)-H(15)	119.0
C(17)-C(16)-C(15)	120.3(3)
C(17)-C(16)-H(16)	119.9
C(15)-C(16)-H(16)	119.9
C(18)-C(17)-C(16)	119.3(3)
C(18)-C(17)-H(17)	120.3
C(16)-C(17)-H(17)	120.3
C(17)-C(18)-C(19)	120.7(3)
C(17)-C(18)-H(18)	119.7
C(19)-C(18)-H(18)	119.7
C(18)-C(19)-C(14)	120.9(3)
C(18)-C(19)-H(19)	119.5
C(14)-C(19)-H(19)	119.5
C(25)-C(20)-C(21)	118.0(2)
C(25)-C(20)-C(7)	120.0(2)
C(21)-C(20)-C(7)	122.0(2)
C(22)-C(21)-C(20)	120.4(3)
C(22)-C(21)-H(21)	119.8
C(20)-C(21)-H(21)	119.8
C(23)-C(22)-C(21)	120.4(3)
C(23)-C(22)-H(22)	119.8
C(21)-C(22)-H(22)	119.8
C(24)-C(23)-C(22)	119.7(3)
C(24)-C(23)-H(23)	120.2
C(22)-C(23)-H(23)	120.2
C(23)-C(24)-C(25)	120.3(3)
C(23)-C(24)-H(24)	119.8
C(25)-C(24)-H(24)	119.8
C(24)-C(25)-C(20)	121.1(2)
C(24)-C(25)-H(25)	119.5
C(20)-C(25)-H(25)	119.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd26278.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	55(2)	59(2)	44(1)	4(1)	0(1)	2(1)
C(2)	48(2)	72(2)	58(2)	7(1)	-5(1)	-3(1)
C(3)	45(2)	81(2)	65(2)	13(2)	8(1)	10(1)
C(4)	50(2)	69(2)	51(2)	8(1)	8(1)	17(1)
C(5)	46(1)	47(1)	42(1)	6(1)	0(1)	9(1)
C(6)	45(1)	47(1)	38(1)	6(1)	-2(1)	9(1)
C(7)	43(1)	49(1)	36(1)	9(1)	2(1)	4(1)
C(8)	52(2)	60(2)	41(1)	4(1)	5(1)	-4(1)
C(9)	58(2)	45(1)	43(1)	3(1)	4(1)	6(1)
C(10)	40(1)	43(1)	43(1)	0(1)	6(1)	6(1)
C(11)	41(1)	48(1)	46(1)	-2(1)	7(1)	5(1)
C(12)	76(2)	69(2)	47(2)	-14(1)	-1(1)	16(2)
C(13)	81(2)	46(2)	74(2)	-3(1)	10(2)	12(1)
C(14)	48(1)	44(1)	36(1)	-4(1)	2(1)	7(1)
C(15)	55(2)	63(2)	70(2)	7(1)	18(1)	16(1)
C(16)	70(2)	72(2)	99(2)	7(2)	21(2)	33(2)
C(17)	89(2)	52(2)	76(2)	0(1)	1(2)	29(2)
C(18)	77(2)	51(2)	87(2)	15(2)	1(2)	1(1)
C(19)	52(2)	57(2)	73(2)	10(1)	7(1)	7(1)
C(20)	43(1)	54(1)	38(1)	8(1)	0(1)	10(1)
C(21)	53(2)	54(2)	68(2)	6(1)	8(1)	10(1)
C(22)	62(2)	64(2)	80(2)	-16(2)	11(2)	18(1)
C(23)	60(2)	95(2)	57(2)	-4(2)	14(1)	23(2)
C(24)	57(2)	74(2)	54(2)	10(1)	14(1)	9(1)
C(25)	55(2)	56(1)	46(1)	6(1)	7(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd26278.

	x	y	z	U(eq)
H(1)	3637	6172	3718	65
H(2)	1000	5791	4282	74
H(3)	469	6870	5966	76
H(4)	2552	8383	7120	67
H(8)	8142	9508	5868	63
H(12A)	7574	9179	10112	98
H(12B)	6692	10383	10494	98
H(12C)	8510	10659	10167	98
H(13A)	7183	12108	8237	101
H(13B)	5958	11981	9172	101
H(13C)	5293	11464	7852	101
H(15)	9437	8289	7897	73
H(16)	10316	6300	8383	93
H(17)	8479	4601	8952	87
H(18)	5760	4913	9043	88
H(19)	4872	6927	8603	73
H(21)	6313	5703	3720	70
H(22)	7560	5181	2108	82
H(23)	9282	6823	1345	83
H(24)	9858	8963	2246	73
H(25)	8581	9515	3826	63
H(9)	5500(30)	10130(30)	6520(20)	51(7)

Table 6. Torsion angles [deg] for cd26278.

C(6)-C(1)-C(2)-C(3)	-0.8(4)
C(1)-C(2)-C(3)-C(4)	0.5(4)
C(2)-C(3)-C(4)-C(5)	0.6(4)
C(3)-C(4)-C(5)-C(6)	-1.5(4)
C(3)-C(4)-C(5)-C(9)	173.1(2)
C(2)-C(1)-C(6)-C(5)	-0.1(4)
C(2)-C(1)-C(6)-C(7)	-175.5(2)
C(4)-C(5)-C(6)-C(1)	1.3(3)
C(9)-C(5)-C(6)-C(1)	-174.3(2)
C(4)-C(5)-C(6)-C(7)	177.7(2)
C(9)-C(5)-C(6)-C(7)	2.1(2)
C(1)-C(6)-C(7)-C(8)	174.3(2)
C(5)-C(6)-C(7)-C(8)	-1.5(2)
C(1)-C(6)-C(7)-C(20)	-1.3(4)
C(5)-C(6)-C(7)-C(20)	-177.0(2)
C(6)-C(7)-C(8)-C(9)	0.2(3)
C(20)-C(7)-C(8)-C(9)	175.8(2)
C(7)-C(8)-C(9)-C(5)	1.0(3)
C(7)-C(8)-C(9)-C(10)	126.9(2)
C(4)-C(5)-C(9)-C(8)	-177.0(2)
C(6)-C(5)-C(9)-C(8)	-1.9(2)
C(4)-C(5)-C(9)-C(10)	58.0(3)
C(6)-C(5)-C(9)-C(10)	-126.9(2)
C(8)-C(9)-C(10)-C(11)	117.5(3)
C(5)-C(9)-C(10)-C(11)	-124.6(3)
C(8)-C(9)-C(10)-C(14)	-59.3(3)
C(5)-C(9)-C(10)-C(14)	58.7(3)
C(14)-C(10)-C(11)-C(12)	-0.9(4)
C(9)-C(10)-C(11)-C(12)	-177.4(2)
C(14)-C(10)-C(11)-C(13)	177.1(2)
C(9)-C(10)-C(11)-C(13)	0.6(4)
C(11)-C(10)-C(14)-C(15)	-88.7(3)
C(9)-C(10)-C(14)-C(15)	88.1(3)
C(11)-C(10)-C(14)-C(19)	91.4(3)
C(9)-C(10)-C(14)-C(19)	-91.8(3)
C(19)-C(14)-C(15)-C(16)	-1.1(4)
C(10)-C(14)-C(15)-C(16)	179.0(3)
C(14)-C(15)-C(16)-C(17)	1.1(5)
C(15)-C(16)-C(17)-C(18)	-0.2(5)
C(16)-C(17)-C(18)-C(19)	-0.7(5)
C(17)-C(18)-C(19)-C(14)	0.6(5)
C(15)-C(14)-C(19)-C(18)	0.2(4)
C(10)-C(14)-C(19)-C(18)	-179.8(2)
C(8)-C(7)-C(20)-C(25)	-31.7(3)
C(6)-C(7)-C(20)-C(25)	143.2(2)
C(8)-C(7)-C(20)-C(21)	148.8(2)
C(6)-C(7)-C(20)-C(21)	-36.4(3)
C(25)-C(20)-C(21)-C(22)	-2.4(4)
C(7)-C(20)-C(21)-C(22)	177.1(2)
C(20)-C(21)-C(22)-C(23)	0.8(4)
C(21)-C(22)-C(23)-C(24)	1.6(4)
C(22)-C(23)-C(24)-C(25)	-2.4(4)
C(23)-C(24)-C(25)-C(20)	0.7(4)
C(21)-C(20)-C(25)-C(24)	1.7(4)
C(7)-C(20)-C(25)-C(24)	-177.9(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd26278 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)