

Supporting Information

Insights into the General and Efficient Cross McMurry Reactions between Ketones

Xin-Fang Duan*, Jing Zeng, Jia-Wei Lü, Zhan-Bin Zhang

Department of Chemistry, Beijing Normal University, Beijing, 100875, China.

General	S2
Preparation of 2ac via deamination of 2cp (Scheme 1)	S2
Notes for yield calculation through GC Analysis	S3
Characterization data	S3-17
References	S17
^1HNMR and ^{13}CNMR spectra	S18-41
GC spectra for Table 1 Entry 4	S41-44

General

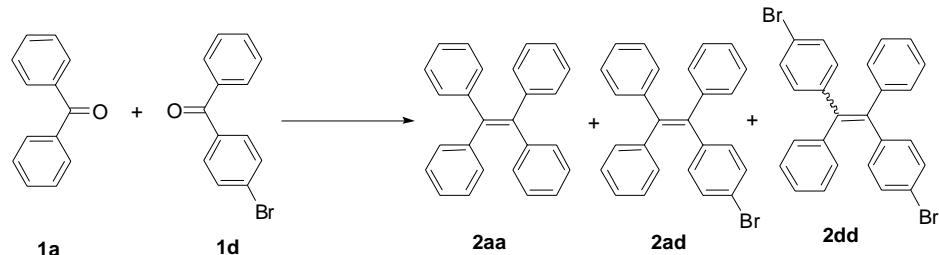
Melting points were obtained on a microscopical instrument and uncorrected. NMR spectra were recorded at 500MHz spectrometer. IR spectra were recorded on a FT-IR spectrometer. All reagents and solvents used for McMurry reactions were freshly dehydrated before use. Zinc powder were freshly activated by 1% HCl aqueous solution, washed by distilled water, absolute alcohol and ether, and then dried under vacuum. All corresponding glassware was oven dried (120 °C) and cooled under a stream of Argon gas.

Preparation of 1, 1, 2-Triphenyl-2-(p-methylphenyl)ethylene (2ac) via deamination of 2cp (Scheme 1):

According to reported procedure:¹ Compound **2cp** (300 mg, 0.8 mmol) was dissolved in ethanol (35ml) and acetic acid (5 mL) at room temperature. Aqueous sodium nitrite (10 mL, 0.8 M) and sodium bisulfite (15 mL, 0.5M) were added sequentially to the stirred solution. The reaction was monitored by TLC. After 17 h, the reaction mixture was extracted with CH₂Cl₂, and organic phase was washed with water and dried over anhy. K₂CO₃. After removal of CH₂Cl₂, the resulting solid was purified by flash chromatography (221 mg, 81.3%).

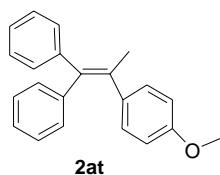
Notes for yield calculation through GC Analysis (e.g. Table 1 Entry 4)

The yields of **2aa**, **2ad** and **2dd** were calculated through GC analyses as following:



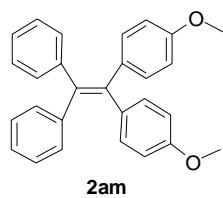
- (1) Standard sample **2aa** and **2dd** were prepared by the homo McMurtry coupling of **1a** and **1d** respectively.
- (2) The amounts of **2aa** and **2dd** in the cross coupling products were determined by external standard method via standard solutions of **2aa** and **2dd**. The amount of **2ad** could be thus calculated.

Characterization data:



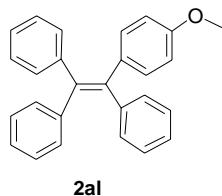
**1,1-Diphenyl-2-(p-methoxyphenyl)propene
(2at)**

White solid, yield: 92%; mp 104.4-105.3 °C (lit.² 103 -104 °C); Anal. Data agrees with its structure.



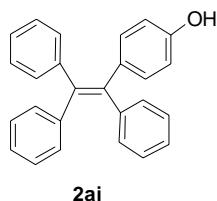
**1,1-Diphenyl-2,2-di(p-methoxyphenyl)
ethylene(2am)**

White solid, yield: 59%; mp 157.8-158.4 °C (lit.³ 157 °C); Anal. Data (IR, ¹HNMR) were identical with the reported .



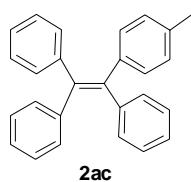
**1,1, 2-Triphenyl-2-(p-methoxyphenyl)
ethylene (2al)**

White solid, yield: 62%; mp 140 -141.5 °C (lit.⁴ 139 -141°C); Anal. Data (IR, ¹HNMR) were identical with the reported.



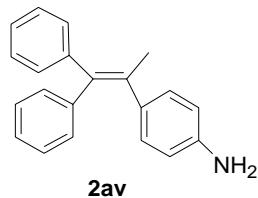
**1,1, 2-Triphenyl-2-(p-hydroxyphenyl)ethylene
(2aj)**

White solid, yield: 66%; mp 215-216 °C (lit.⁵ 215 -217°C); Anal. Data (IR, ¹HNMR) were identical with the reported.



**1,1, 2-Triphenyl-2-(p-methylphenyl)ethylene
(2ac)**

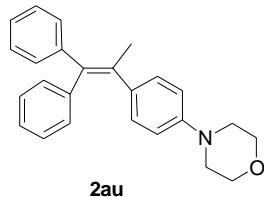
White solid, yield: 81.3 % from **3**, mp 146.3- 147.1°C (lit.⁶ 146-148 °C); Anal. Data (IR, ¹HNMR) were identical with the reported.



**1,1-Diphenyl-2-(p-aminophenyl)propene
(2av)**

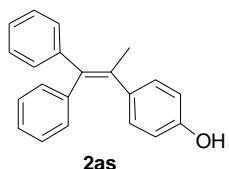
White solid, yield: 80%, mp 130.3-131.5 °C; IR(cm⁻¹, KBr): 3459.5,

3371.0, 1619.3, 1593.5, 1514.0, 1490.5, 1439.9, 826.6, 767.7, 701.2;
¹HNMR(CDCl₃, 500MHz) δ 2.12 (s, 3H), 3.88 (br s, 2H), 6.54 (d, J = 5.80Hz, 2H), 6.94-6.99 (m, 4H), 7.03-7.07 (m, 3H), 7.25- 7.28 (m, 3H), 7.34-7.37 (t, J = 7.25Hz, 2H); ¹³CNMR (CDCl₃, 500MHz) δ 23.2, 114.6, 125.5, 126.3, 127.4, 128.1, 130.1, 130.4, 130.9, 134.1, 135.4, 138.1, 143.7, 144.1, 144.6; Anal. Calc. for C₂₁H₁₉N: C, 88.38; H, 6.71; N, 4.91; Found: C, 88.26, H, 6.94, N, 4.84; MS (EI) m/z: 285 (M⁺, 100), 270 (33); 252 (16); 191 (22); 165 (23); 93 (34).



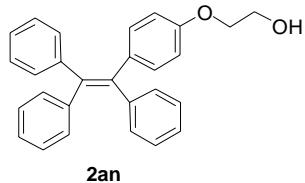
**1,1-Diphenyl-2-(p-morpholinylphenyl)
propene (2au)**

White solid, yield: 94%, mp 162.2 -163.4 °C; IR (cm⁻¹, KBr): 1608.3, 1515.1, 1234.3, 1125.3, 927.9, 765.3, 695.5, 607.1; ¹HNMR (CDCl₃, 500MHz) δ 2.13 (s, 3H), 3.20 (br s, 4H), 3.89 (br s, 4H), 6.92 (d, J = 6.40Hz, 2H), 7.04-7.07 (m, 4H), 7.13 (s, 2H), 7.25-7.26 (m, 3H), 7.35-7.38(m, 3H); ¹³CNMR(CDCl₃, 500MHz) δ 23.1, 49.2, 66.8, 114.8, 125.6, 126.4, 127.5, 128.1, 130.0, 130.3, 130.9, 131.1, 134.9, 138.6, 143.4, 143.9; Anal. Calc. for C₂₅H₂₅NO: C, 84.47; H, 7.09; N, 3.94; Found: C, 84.37; H, 7.33; N, 3.83; MS (EI) m/z 355 (M⁺, 100), 297 (43), 252 (62), 191 (44), 165 (59), 91 (25), 77 (23).



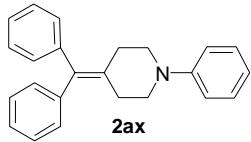
**1,1-Diphenyl-2-(p-hydroxyphenyl)propene
(2as)**

White solid, yield: 71%; mp 130.3 -131 °C ; IR (cm⁻¹, KBr): 3518.5, 1610.1, 1559.4, 1510.6, 1487.8, 1173.4, 846.1, 702.7, 576.6; ¹HNMR (CDCl₃, 500MHz,) δ 2.15 (s, 3H), 4.63 (s, 1H), 6.66 (s, 2H), 6.94 (s, 2H), 7.06 (s, 5H), 7.28 (s, 3H), 7.37(s, 2H); ¹³CNMR (CDCl₃, 500MHz) δ 23.3, 114.7, 125.7, 126.5, 127.5, 128.1, 130.0, 130.6, 130.9, 135.1, 136.5, 138.9, 143.3, 143.7, 153.8; Anal. Calc. for C₂₁H₁₈O: C, 88.08; H, 6.34; Found: C, 87.78; H, 6.64; MS (EI) m/z 286 (M⁺, 100), 271 (19), 252 (19), 191 (29), 165 (33).



1,1,2-Triphenyl-2-[p-(2-hydroxyethoxy)phenyl]ethylene (2an)

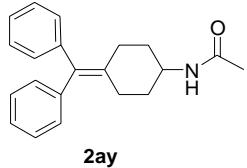
white solid, yield: 60%; mp 82.4-83.6 °C; IR (cm⁻¹, KBr): 3310.9, 3014.8, 1606.2, 1508.1, 1492.4, 1438.3, 1245.5, 1175.0, 1079.9, 917.3, 698.2, 614.7, 577.4; ¹HNMR (CDCl₃, 500MHz) δ 3.96 (t, J = 3.2Hz, 2H), 4.03(d, J = 3.4Hz, 2H), 6.67 (d, J = 8.5Hz, 2H), 6.96 (d, J = 8Hz, 2H), 7.04-7.07 (m, 5H), 7.10-7.18 (m, 9H); ¹³CNMR (CDCl₃, 500MHz) δ 61.5, 68.9, 113.6, 114.2, 125.8, 126.3, 126.4, 127.6, 127.7, 128.1, 128.4, 128.5, 129.5, 129.9, 131.3, 132.6, 136.6, 140.2, 140.4, 143.91, 143.97, 157.0; Anal. Calc. for C₂₈H₂₄O₂: C, 85.68; H, 6.16; Found: C, 85.88; H, 6.44; MS (EI) m/z 392 (M⁺, 100), 348 (30), 253 (20), 241(24), 227 (76), 183 (37), 165 (22).



1-Phenyl-4-benzhydrylideneperididine

(**2ax**)

white solid, yield: 92.5%; mp 79.3-80.5 °C; IR (cm⁻¹, KBr): 2890.0, 2797.8, 1598.3, 1492.3, 1379.3, 1327.1, 1210.1, 921.0, 762.1, 705.1, 696.6, 588.5; ¹H NMR (CDCl₃, 500MHz) δ 2.57 (s, 4H), 3.32 (t, J = 5Hz, 4H), 6.88 (s, 1H), 6.99 (s, 2H), 7.19 (d, J = 7.14, 4H), 7.24-7.35 (m, 8H); ¹³C NMR (CDCl₃, 500MHz) δ 31.4, 51.3, 116.4, 119.4, 126.5, 128.1, 129.1, 129.8, 134.9, 136.6, 142.4, 151.2; Anal. Calc. for C₂₄H₂₃N: C, 88.57; H, 7.12; N, 4.30; Found: C, 88.55; H, 7.07; N, 4.28; MS (EI) m/z Found: 325 (M⁺, 100), 321 (62), 203 (26), 191(44), 165 (47), 104 (72), 91 (93), 77 (99).

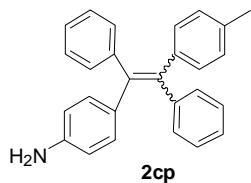


N-(4-Benzhydrylidene)cyclohexylacetamide

(**2ay**)

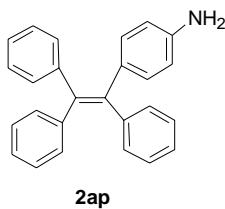
White solid, yield: 90%; mp 173. 9-174.8 °C; IR (cm⁻¹, KBr): 3446.8, 3258.0, 1635.9, 1560.3, 1490.1, 1438.3, 1368.1, 1102.1, 769.5, 699.3, 625.4; ¹H NMR (CDCl₃, 500MHz) δ 1.30-1.36 (m, 2H), 2.00 (s, 3H), 2.03-2.06 (m, 2H), 2.13 (t, J = 11.9Hz, 2H), 2.59 (d, J = 13.6Hz, 2H), 4.04 - 4.06 (m, 1H), 5.44 (s, 1H), 7.13 (d, J = 7.30Hz, 4H), 7.22 - 7.24 (m, 2H), 7.31(m, 4H); ¹³C NMR (CDCl₃, 500MHz) δ 23.6, 29.9, 34.3, 48.1, 126.3, 128.0, 129.6, 136.1, 136.2, 142.6, 169.3; Anal. Calc. for C₂₂H₂₆NO: C, 82.46; H, 8.18; N, 4.37; Found: C, 82.62; H, 7.91; N, 4.54; MS (EI) m/z

305 ([M-CH₃]⁺, 80), 246 (100), 218 (43), 191 (47), 167 (83), 91 (67), 43 (89).



cis+trans-1,2-Diphenyl-1-(p-aminophenyl)-2-(p-methylphenyl) ethylene (2cp)

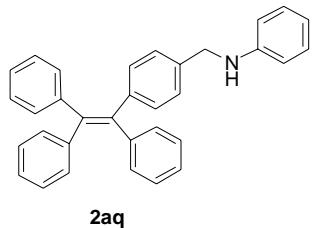
Yellow solid, yield: 76%; mp 170.4 -170.9 °C; IR (cm⁻¹, KBr): 3472.8, 3380.1, 1619.6, 1512.8, 1489.3, 1283.0, 1179.0, 824.9, 802.8, 744.7, 700.4, 629.1, 606.9; ¹HNMR (CDCl₃, 500MHz) δ 2.27 + 2.30 (two sets of singlets, 3H); 3.81(br s, 2H), 6.45-6.82(m, 2H), 6.81-6.86(m, 2H), 6.91(s, 2H), 6.97(s, 2H), 7.02-7.15(m, 10H); ¹³CNMR (CDCl₃, 500MHz) δ 21.1, 21.2, 114.3, 114.4, 126.0, 126.1, 127.50, 127.52, 127.55, 127.6, 128.3, 128.4, 131.2, 131.3, 131.40, 131.45, 131.47, 131.5, 132.4, 132.5, 134.3, 135.7, 140.4, 144.4, 144.5; Anal. Calc. for C₂₇H₂₃N: C, 89.71; H, 6.41; N, 3.87; Found: C, 89.49; H, 6.61; N, 3.79; MS (EI) m/z 361 (M⁺, 100), 268 (18), 252 (17), 180 (26), 165 (27), 93 (67).



**1,1,2-Triphenyl-2-(p-aminophenyl)ethylene
(2ap)**

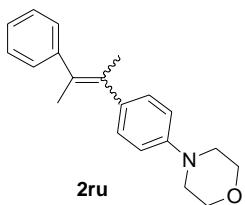
Yellow solid; yield: 80%; mp 202.1-203.4 °C; IR (cm⁻¹,KBr): 3203.3, 1597.3, 1492.0, 1443.3, 1071.6, 694.4; ¹HNMR (CDCl₃, 500 MHz) δ 1.57 (br s, 2H), 6.69 (m, 2H), 6.91 (d, J = 7.8Hz, 2H), 7.04 (m, 7H), 7.16 (m, 8H); ¹³CNMR (CDCl₃, 500MHz) δ 114.4, 126.1, 127.5, 127.6, 127.7, 128.1, 128.2, 131.3, 131.4, 131.5, 132.5, 134.1, 139.3, 140.9,

144.20, 144.24, 144.4, 144.7; Anal. Calc. for C₂₆H₂₁N: C, 89.88; H, 6.09; N, 4.03; Found: C, 89.84; H, 6.06; N, 4.38; MS (EI) m/z 347 (M⁺, 100), 270 (31), 252 (37), 239 (17), 84 (32).



1,1,2-Triphenyl-2-[p-(N-phenyamino)-methyl]ethylene (2aq)

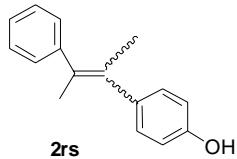
White solid, yield: 68%; mp 148-149 °C ; IR (cm⁻¹, KBr): 3458.2, 3371.9, 2982.1, 1620.7, 1514.5, 1490.9, 1445.0, 1372.5, 829.0, 764.2, 702.1; ¹HNMR (CDCl₃, 500MHz) δ 4.26 (s, 2H), 6.67 (d, J = 7.06Hz, 2H), 6.77 (t, J = 6.98Hz, 1H), 7.01-7.05 (m, 8H), 7.12(s, 11H), 7.20 (t, J = 7.53Hz, 2H); ¹³CNMR (CDCl₃, 500MHz) δ 48.3, 113.1, 117.7, 126.4, 126.9, 127.6, 129.2, 131.3, 131.5, 142.8, 143.7; Anal.Calc.for C₃₃H₂₇N: C, 90.58; H, 6.22; N, 3.20; Found: C, 90.31; H, 6.41; N, 2.97; MS (EI) m/z 437(M⁺, 19), 345 (47), 252 (24), 183 (92), 165 (100).



cis+trans-2-Phenyl-3-(p-morpholinylphenyl)-2-butene (2ru)

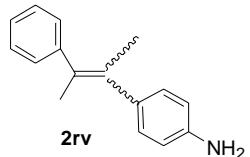
White solid mixture, yield: 76.7%; IR (cm⁻¹, KBr): 2959.7, 2910.5, 1607.8, 1513.4, 1448.6, 1228.5, 1122.7, 1068.8, 926.6, 826.4, 766.3, 701.8; ¹HNMR (CDCl₃, 500MHz) δ 1.89 (s, 3H), 1.94 (s, 3H), 3.23 (br s, 4H), 3.92 (br s, 4H), 6.97 (s, 2H), 7.23 (d, J = 8.12Hz, 2H), 7.30 (s, 3H), 7.39 (t, J = 7.35Hz, 2H); ¹³CNMR (CDCl₃, 500MHz) δ 22.4, 22.5, 49.5, 66.9,

115.3, 126.1, 127.6, 128.1, 128.3, 129.2, 132.4, 132.7, 144.8; Anal. Calc. for C₂₀H₂₃NO: C, 81.87; H, 7.90; N, 4.77; Found: C, 81.82; H, 7.97; N, 4.73; MS (EI) m/z 293 (M⁺, 98), 235 (57), 192 (100), 165 (43), 115 (53).



cis+trans-2-Phenyl-3-(p-hydroxyphenyl)-2-butene (2rs)

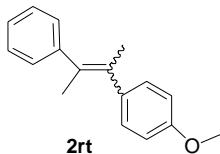
White solid mixture, yield: 58%; IR (cm⁻¹, KBr): 3425.8, 2977.8, 1607.1, 1510.5, 1490.1, 1427.3, 1464.4, 1232.9, 826.8, 700.1, 555.2, 510.9; ¹HNMR (CDCl₃, 500MHz) δ 1.91 (2 sets of singlets, 4H), 2.17 (2 sets of singlets, 2H), 4.67 (2 sets of singlets, 1H), 6.58 (d, J = 8.21Hz, 1H), 6.86 (d, J = 10.64Hz, 2H), 6.98-7.10 (m, 2H), 7.12 (d, J = 7.46Hz, 1H), 6.87 (d, J = 7.87Hz, 2H), 6.98-7.13(m, 2H), 7.18 (d, J = 8.12Hz, 1H), 7.27 (s, 2H), 7.39 (t, J = 7.33Hz, 1H); ¹³CNMR (CDCl₃, 500MHz) δ 21.5, 22.5, 114.4, 114.9, 125.4, 126.2, 127.6, 128.1, 128.2, 128.6, 129.2, 129.5, 129.6, 130.4, 132.4, 132.9, 137.0, 144.6; Anal. Calc. for C₁₆H₁₆O: C, 85.68; H, 7.19; Found: C, 85.47; H, 7.38; MS (EI) m/z 224 (M⁺, 71), 209 (24), 165 (34), 115 (67), 91 (60), 77 (100).



cis + trans-2-Phenyl-3-(p-aminophenyl)-2-butene (2rv)

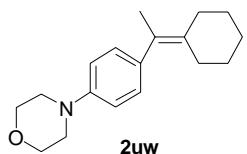
Yellow liquid; yield: 60.2%, IR(cm⁻¹, KBr): 3370.4, 3044.4, 2885.2, 1730.3, 1620.3, 1514.1, 1493.8, 1444.5, 1277.4, 1063.1, 763.9, 701.6; ¹HNMR (CDCl₃, 500MHz) δ 1.97 (2 sets of singlets, 1H), 2.20 (2 sets of singlets, 5H), 3.56 (s, 2H), 6.47 (d, J = 8.21Hz, 2H), 6.74-6.82 (m, 2H),

7.06-7.09 (m, 2H), 7.15 (t, $J = 7.44\text{Hz}$, 2H), 7.29-7.43 (m, 1H); $^{13}\text{CNMR}$ (CDCl_3 , 500MHz) δ 24.9, 25.1, 114.6, 115.0, 125.3, 126.0, 129.9, 127.1, 127.2, 127.4, 127.5, 128.1, 129.3, 130.2, 143.4; Anal. Calc. for $\text{C}_{16}\text{H}_{17}\text{N}$: C, 86.05; H, 7.67; N, 6.27; Found: C, 86.62; H, 7.35; N, 6.45; MS (EI) m/z 223 (M^+ , 53), 207 (33), 130 (57), 97 (49), 77 (100).



cis+trans-2-Phenyl-3-(p-methoxyphenyl)-2-butene (2rt)

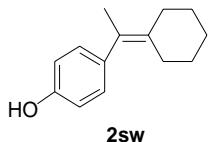
Colorless liquid, yield: 62.2%; IR (cm^{-1} , KBr): 2924.6, 1604.6, 1510.0, 1283.1, 1245.4, 1176.0, 1035.6, 832.3, 765.8, 703.0; $^1\text{HNMR}$ (CDCl_3 , 500MHz) δ 1.91, 1.94 (2 sets of singlets, 2H), 2.20 (s, 4H), 3.75 (s, 2H), 3.87 (2 sets of singlets, 1H), 6.67 (d, $J = 8.49\text{Hz}$, 1H), 6.94 (d, $J = 8.54\text{Hz}$, 1H), 6.97 (d, $J = 8.46\text{Hz}$, 1H), 7.02 (d, $J = 7.08\text{Hz}$, 1H), 7.07 (s, 1H), 7.14 (d, $J = 7.44\text{Hz}$, 1H), 7.26 (d, $J = 8.30\text{Hz}$, 1H), 7.31 (d, $J = 6.82\text{Hz}$, 1H), 7.41 (d, $J = 8.00\text{Hz}$, 1H); $^{13}\text{CNMR}$ (CDCl_3 , 500MHz) δ 21.5, 22.5, 55.0, 55.2, 112.9, 113.5, 125.4, 126.1, 127.6, 128.1, 128.3, 129.2, 130.2, 132.5, 132.8, 136.8, 144.7, 158.0; Anal. Calc. for $\text{C}_{17}\text{H}_{18}\text{O}$: C, 85.67; H, 7.61; Found: C, 85.20; H, 7.83; MS (EI) m/z 238 (M^+ , 95), 223 (40), 178 (51), 135 (84), 95 (98), 81 (100), 77 (96).



N-[4-(1-Cyclohexylideneethyl)phenyl]morpholine (2uw)

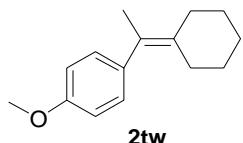
White solid, yield: 60%; mp 70.5-71.6 °C; IR (cm^{-1} , KBr): 2955.6, 2922.0, 2850.9, 1607.9, 1510.9, 1445.6, 1323.8, 1230.2, 1120.6, 922.1, 836.0,

813.9, 688.2, 588.5; ^1H NMR (CDCl_3 , 500MHz) δ 1.15 (s, 2H), 1.60 (s, 4H), 1.96 (s, 3H), 2.05 (s, 2H), 2.31 (d, $J = 5.50\text{Hz}$, 2H), 3.19 (br s, 4H), 3.89 (br s, 4H), 6.89 (d, $J = 6.61\text{Hz}$, 2H), 7.07(d, $J = 7.88\text{Hz}$, 2H); ^{13}C NMR (CDCl_3 , 500MHz) δ 20.2, 26.9, 28.1, 28.5, 30.7, 31.9, 49.5, 67.0, 115.2, 126.5, 129.2, 135.3; Anal. Calc. for $\text{C}_{18}\text{H}_{25}\text{NO}$: C, 79.66; H, 9.28, N, 5.16; Found: C, 79.56; H, 9.16; N, 5.07; MS (EI) m/z 271 (M^+ , 100), 256 (72), 198 (31), 185 (29), 129 (32).



**4-(1-Cyclohexylideneethyl) phenol
(2sw)**

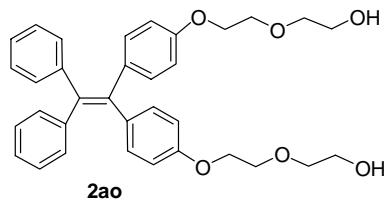
White solid, yield: 70%; mp 92.5- 93.9 °C; IR (cm^{-1} , KBr): 3288.2, 2916.6, 2849.1, 1608.7, 1592.0, 1510.2, 1440.4, 1227.2, 1172.5, 829.7, 780.6, 769.5, 669.7, 643.9, 610.6; ^1H NMR (CDCl_3 , 500MHz) δ 1.47 (t, $J = 6.16$, 2H), 1.62 (t, $J = 9.7\text{Hz}$, 4H), 1.95 (s, 3H), 2.03 (t, $J = 5.69\text{Hz}$, 2H), 2.31 (t, $J=5.05\text{Hz}$, 2H), 6.80 (d, $J = 8.42\text{Hz}$, 2H), 7.02 (d, $J = 8.42\text{Hz}$, 2H); ^{13}C NMR (CDCl_3 , 500MHz) δ 20.3, 26.9, 28.1, 28.5, 30.6, 31.9, 114.7, 129.6, 129.9, 137.9, 153.4; Anal. Calc. for $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12; H, 8.97; Found: C, 83.28; H, 9.25; MS (EI) m/z Found: 202 (M^+ , 35), 187 (30), 145 (39), 115 (42), 91 (48), 77 (45), 41 (100).



**1-(1-Cyclohexylideneethyl)-4-methoxybenzene
(2tw)**

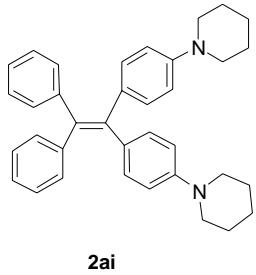
Colorless liquid, yield: 69%; IR (cm^{-1} , KBr): 2923.8, 2852.5, 1604.6, 1509.8, 1464.2, 1368.1, 1242.7, 1176.0, 1043.0, 836.0, 806.5, 773.3;

¹H NMR (CDCl₃, 500MHz) δ 1.38-1.57 (m, 2H), 1.66-1.71 (m, 4H), 2.05 (s, 3H), 2.12 (t, J = 5.73Hz, 2H), 2.41(t, J = 5.22Hz, 2H), 3.88(s, 3H), 6.95 (d, J = 8.58Hz, 2H), 7.15 (d, J = 8.57Hz, 2H); ¹³C NMR (CDCl₃, 500 MHz) δ 20.4, 27.0, 28.2, 28.6, 29.3, 30.0, 30.8, 55.2, 113.4, 126.6, 129.5, 135.4, 137.7, 157.7; Anal. Calc. for C₁₅H₂₀O: C, 83.28; H, 9.32; Found: C, 82.86; H, 9.76; MS (EI) m/z 216 (M⁺, 30), 201 (35), 135 (47), 109 (87), 93 (100), 66 (99).



1,1-Diphenyl-2,2-di{4-[2-(2-hydroxyethoxy)ethoxy]phenyl}ethylene (2ao)

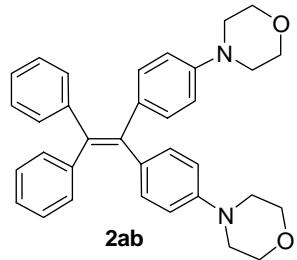
White solid, yield: 89%; mp 112-114 °C; IR (cm⁻¹, KBr): 3431.3, 2937.7, 2891.0, 1601.1, 1508.9, 1449.4, 1248.4, 1070.2, 920.0; ¹H NMR (CDCl₃, 500MHz) δ 3.63 (s, 4H), 3.74 (s, 4H), 3.79 (s, 4H), 4.00 (s, 4H), 6.67 (d, J = 6.2 Hz, 4H), 7.04 (s, 6H), 7.13-7.17 (s, 8H); ¹³C NMR (CDCl₃, 500MHz) δ 54.5, 56.7, 61.7, 67.2, 67.5, 67.6, 72.5, 114.0, 114.3, 125.5, 125.7, 126.0, 126.1, 126.2, 127.7, 128.1, 128.5, 129.3, 129.7, 131.3, 132.6, 132.4, 136.7, 143.6, 148.7, 156.5; Anal. Calc. for C₃₄H₃₆O₆: C, 75.53; H, 6.71; Found: C, 75.91; H, 6.41; MS (EI) m/z 540 (M⁺, 17), 390 (9), 302 (22), 121 (30), 45 (100%).



2ai

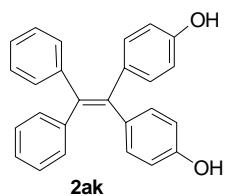
**1,1-Diphenyl-2,2-di(p-piperidinylphenyl)-
ethylene (2ai)**

Yellow solid, yield: 65%; mp 189.2-190.3 °C; IR (cm⁻¹, KBr): 2932.0, 2851.3, 1604.2, 1510.7, 1234.4, 1128.2, 699.2; ¹HNMR (CDCl₃, 500MHz) δ 1.68 (s, 12H), 3.11 (s, 8H), 6.67 (d, J = 6.9Hz, 4H), 6.86 (s, 4H), 7.05-7.11 (m, 10H); ¹³CNMR (CDCl₃, 500MHz) δ 24.3, 25.8, 50.1, 114.9, 125.7, 127.6, 131.5, 132.3, 134.6, 144.9, 150.3; Anal. Calc. for C₃₆H₃₈N₂: C, 86.7; H, 7.68; N, 5.62; Found: C, 86.61; H, 7.59; N, 5.61; MS (EI) m/z 498 (M⁺, 100), 441 (14), 252 (41), 248 (51), 228 (25).

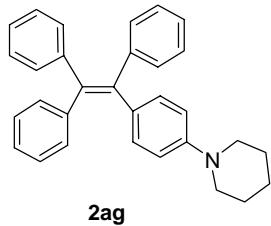


**1,1-Diphenyl-2,2-di(p-morpholinylphenyl)-
ethylene (2ab)**

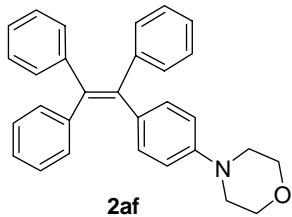
Yellow solid, yield: 73%; mp 205.1-206.2 °C; IR(cm⁻¹, KBr): 3044.5, 2954.4, 1606.5, 1513.3, 1230.6, 1122.9, 925.5, 701.7; ¹HNMR (CDCl₃, 500MHz) δ 3.14 (br s, 8H), 3.86 (br s, 8H), 6.91 (s, 4H), 6.95 (m, 4H), 7.04-7.05 (m, 4H), 7.07-7.14 (m, 6H); ¹³CNMR (CDCl₃, 500MHz) δ 48.9, 66.9, 114.3, 125.9, 127.6, 131.4, 132.4, 135.5, 138.6, 140.3, 144.6, 149.3; Anal. Calc. for C₃₄H₃₄N₂O₂: C, 81.24; H, 6.82; N, 5.57; Found: C, 80.93; H, 7.24; N, 5.58; MS (EI) m/z 503 (M⁺, 100), 444 (36), 386 (26), 252 (30), 165(17).



White solid; yield: 69%, mp 222.9-223.7 °C; IR(cm⁻¹, KBr): 3404.2, 1608.2, 1508.4, 1442.8, 1254.9, 1170.7, 834.6, 765.8, 700.6 □ 592.4, 570.0; ¹HNMR (CDCl₃, 500MHz) δ 4.58 (s, 2H), 6.59 (d, J = 8.44Hz, 4H), 6.92 (d, J = 8.43Hz, 4H), 7.04 (d, J = 7.32Hz, 4H), 7.12 (t, J = 7.03Hz, 6H); ¹³CNMR (CDCl₃, 500MHz) δ 114.4, 125.9, 127.6, 131.1, 132.4, 134.9, 144.6, 156.1; Anal. Calc. for C₂₆H₂₀O₂: C, 85.69; H, 5.53; Found: C, 85.39; H, 5.80; MS (EI) m/z 364 (M⁺, 100), 270 (15), 165(14).

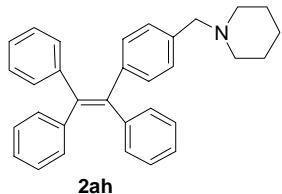


Yellow solid, yield: 53%; mp 156.9-159.3 °C; IR (cm⁻¹, KBr): 2916.6, 2849.1, 1608.7, 1592.0, 1510.2, 1440.4, 1227.2, 1172.5, 829.7, 780.6, 769.5, 669.7, 643.9, 610.6; ¹HNMR (CDCl₃, 500MHz.) δ 1.58 (d, J = 4.59Hz, 2H), 1.69 (s, 4H), 3.14 (d, J = 4.75Hz, 4H), 6.68 (d, J = 7.96Hz, 2H), 6.91 (d, J = 8.11Hz, 2H), 7.03 (d, J = 7.48Hz, 2H), 7.09 (s, 2H), 7.11-7.15 (m, 13H); ¹³CNMR (CDCl₃, 500MHz) δ 24.3, 25.8, 50.1, 115.0, 126.0, 126.1, 126.2, 127.5, 127.6, 127.7, 131.4, 131.5, 131.6, 132.2, 134.1, 139.4, 140.9, 144.3, 150.3; Anal. Calc. for C₃₁H₂₉N: C, 89.60; H, 7.03; N, 3.37; Found: C, 89.33; H, 7.25; N, 3.38; MS (EI) m/z 415 (M⁺, 100), 253 (19), 252 (10), 84 (16).



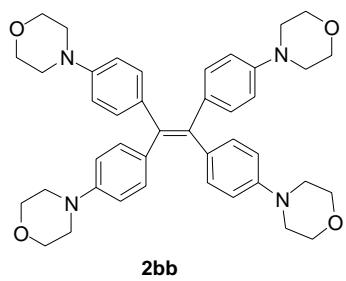
**1,1,2-Triphenyl-2-(p-morpholinophenyl)-
ethylene (2af)**

Yellow solid, yield: 62%; mp 177.4-178.8 °C; IR(cm⁻¹, KBr): 2931.1, 2857.0, 1599.6, 1519.3, 1448.4, 1231.6, 1113.3, 926.5, 771.0; ¹HNMR (CDCl₃, 500MHz) δ 3.24 (br s, 4H), 4.00 (br s, 4H), 6.83-7.49 (m, 19H); ¹³CNMR (CDCl₃, 500MHz) δ 48.8, 66.9, 114.3, 126.2, 126.3, 127.6, 127.7, 131.40, 131.43, 131.5, 132.3, 135.1, 139.7, 140.6, 144.1, 144.17, 144.23, 149.4; Anal. Calc. for C₃₀H₂₇NO: C, 86.30; H, 6.52; N, 3.35; Found: C, 86.59; H, 6.76; N, 3.35; MS (EI) m/z 417 (M⁺, 100), 359 (52), 252 (48).



**N-[4-(Triphenylvinyl)benzyl]
piperidine (2ah)**

White solid; yield: 52%; mp 105.5-106 °C; IR(cm⁻¹, KBr): 3044.4, 2931.7, 1596.9, 1491.1, 1442.3, 865.6, 821.2, 762.4, 699.6, 614.3, 573.7; ¹HNMR (CDCl₃, 500MHz) δ 1.28-1.59 (m, 6H), 2.20-2.35 (m, 4H), 3.41 (s, 2H), 7.06-7.11 (m, 19H); ¹³CNMR (CDCl₃, 500MHz) δ 24.3, 25.8, 54.3, 63.4, 126.4, 127.5, 127.6, 128.7, 131.1, 131.3, 136.1, 140.9, 142.4, 143.6, 143.7, 143.8; Anal. Calc. for C₃₂H₃₁N: C, 89.47; H, 7.27; N, 3.26; Found: C, 89.76; H, 7.51; N, 3.12; MS (EI) m/z 429 (M⁺, 55), 345 (44), 264 (26), 167 (52), 98 (55), 84 (100).



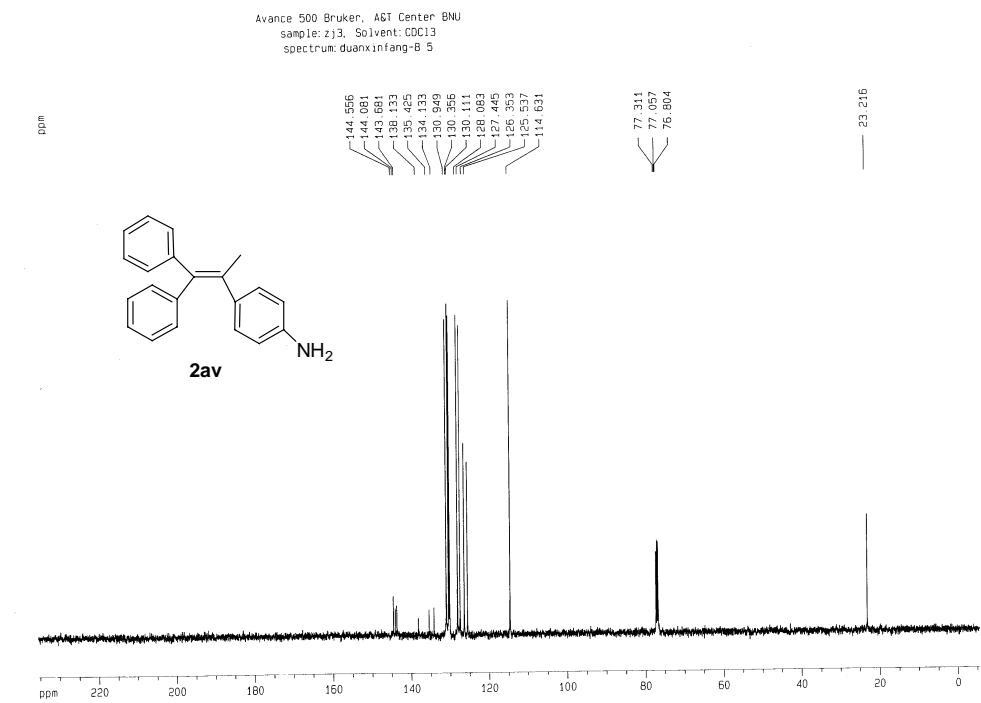
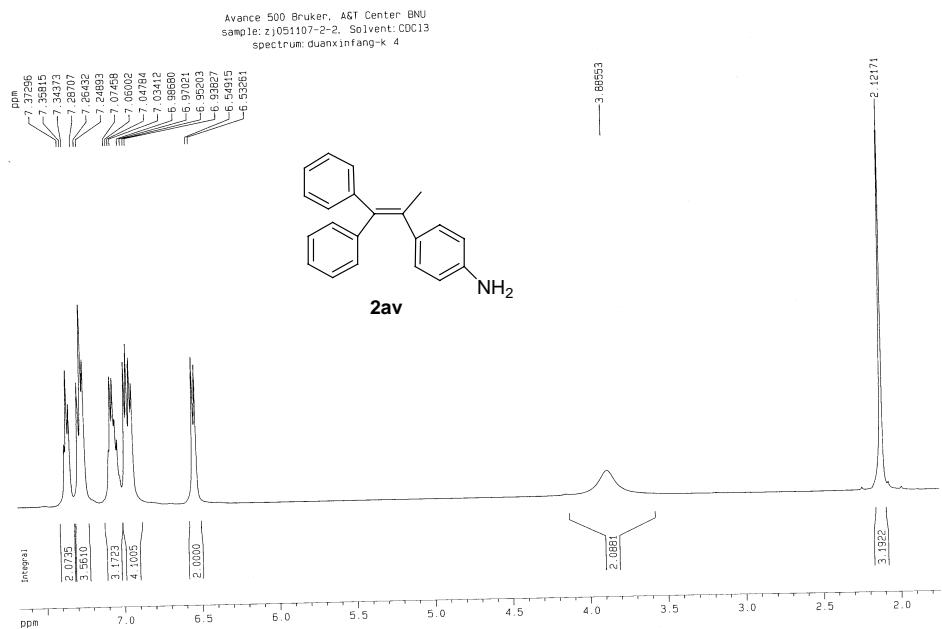
**1,1,2,2-tetra(p-morpholinylphenyl)-
ethylene (2bb)**

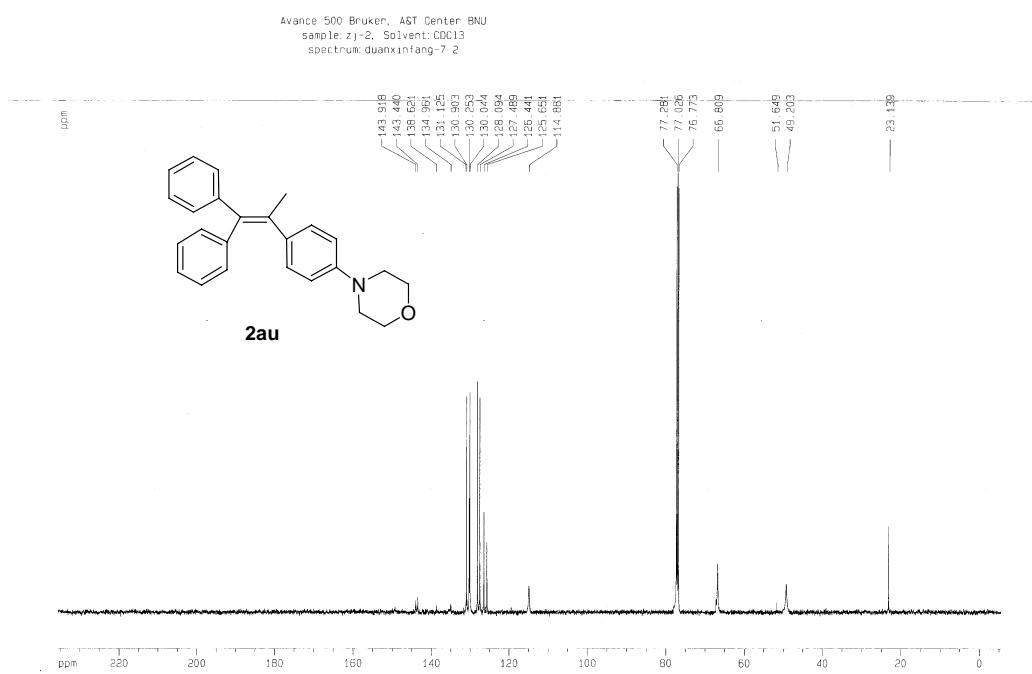
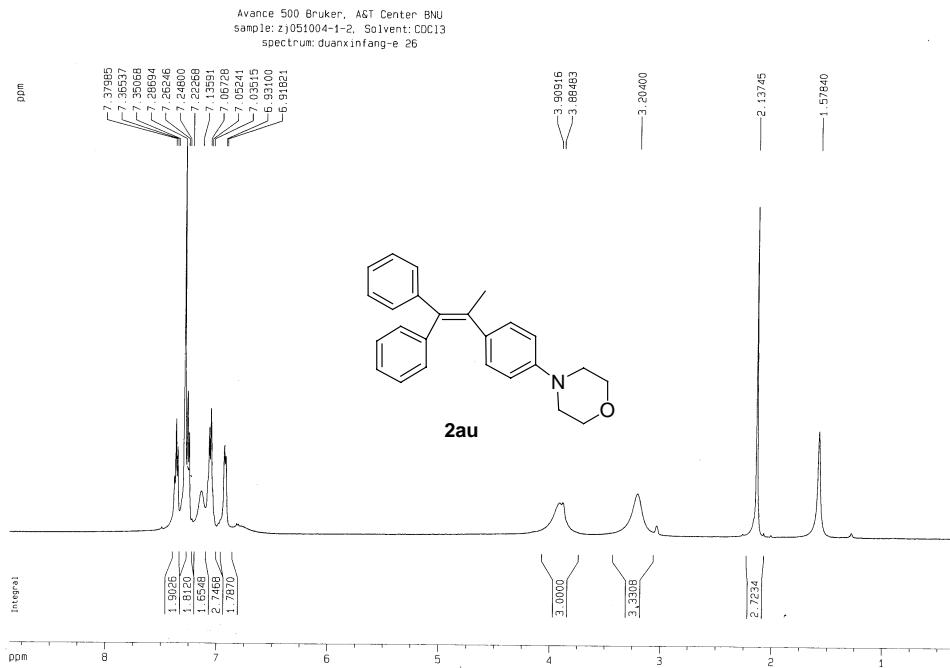
2bb

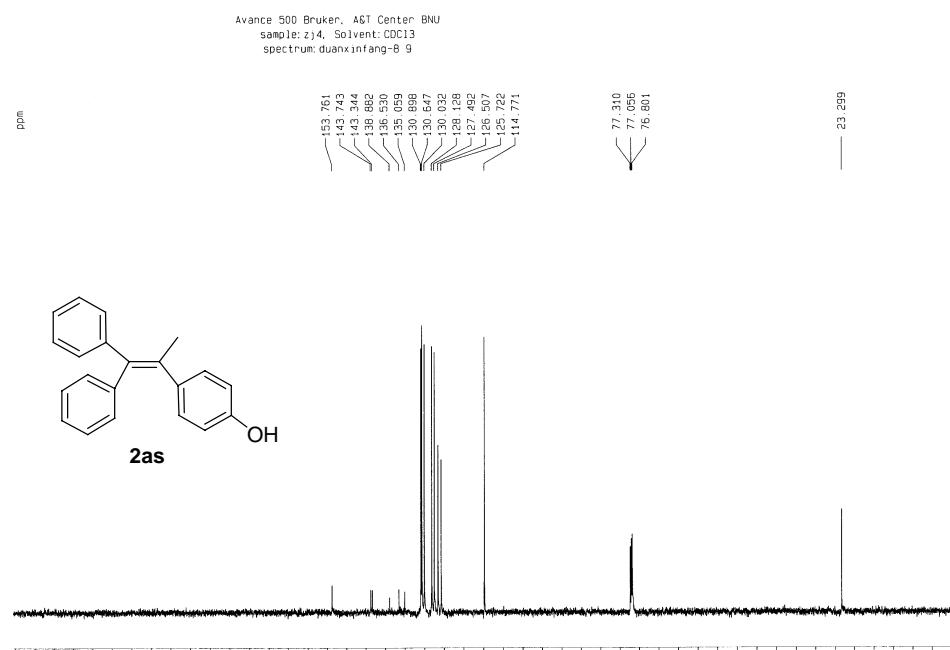
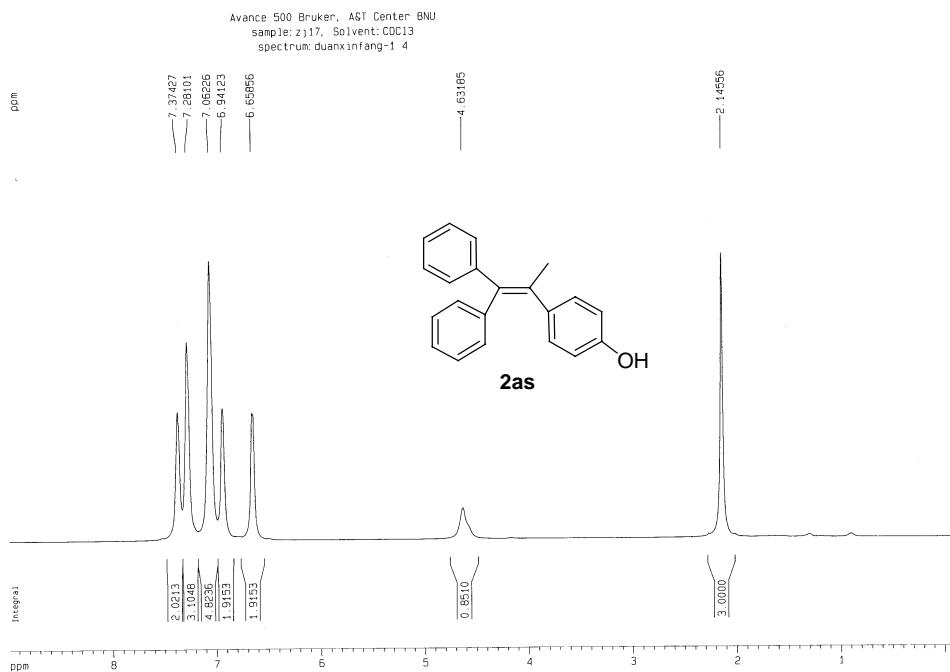
Yellow solid; yield: 90%; mp 238.4-239.1 °C; IR (cm⁻¹, KBr): 2956.4, 2852.2, 1606.4, 1513.4, 1229.8, 1120.5, 926.6; ¹H NMR (CDCl₃, 500MHz) δ 3.15 (br s, 16H), 3.67 (br s, 16H), 6.67 (m, 8H), 6.96 (m, 8H); ¹³C NMR (CDCl₃, 500MHz) δ 49.0, 49.1, 49.2, 49.3, 66.9, 114.4, 115.0, 115.4, 128.1, 130.7, 132.4, 136.4, 138.0, 148.9; Anal. Calc. for C₄₂H₄₈N₄O₄: C, 74.97; H, 7.19; N, 8.33; Found: C, 74.97; H, 7.19; N, 8.33; MS (EI) m/z 672 (M⁺, 100), 252 (31), 77 (9).

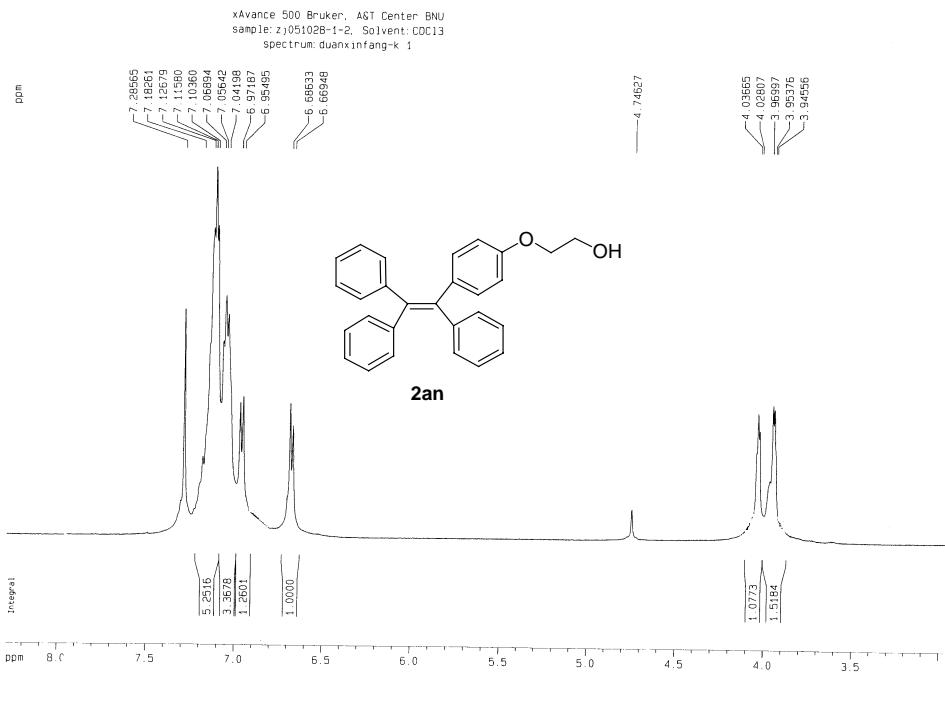
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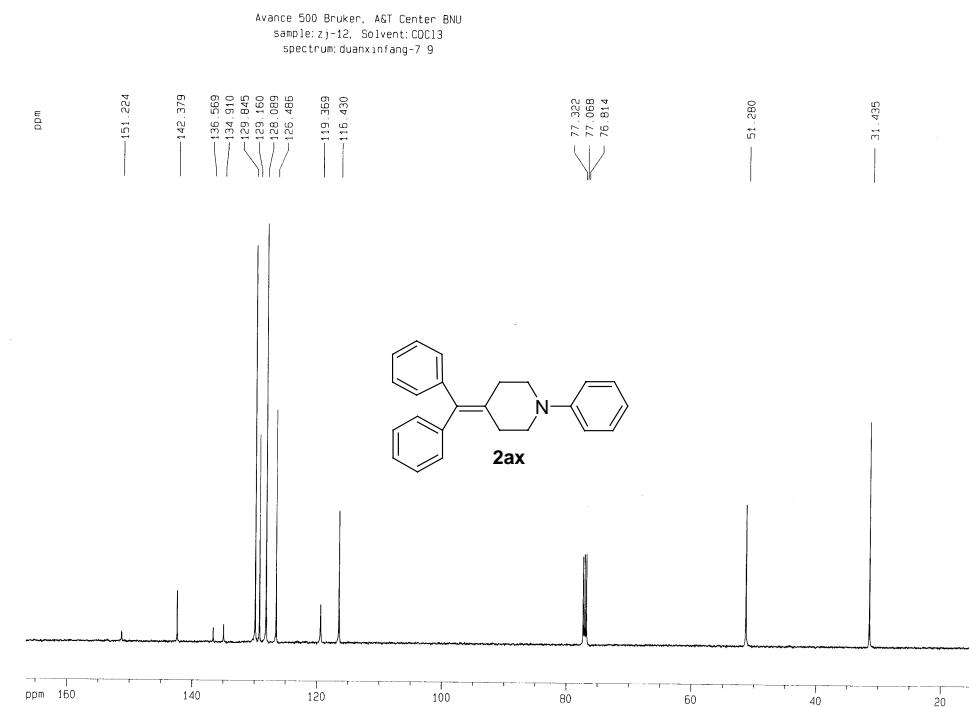
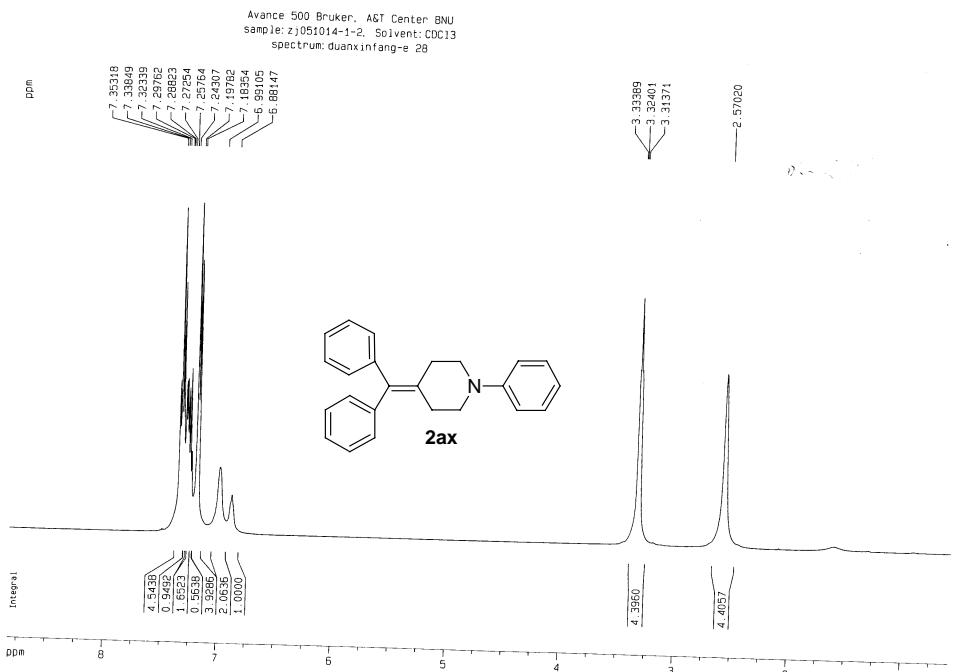
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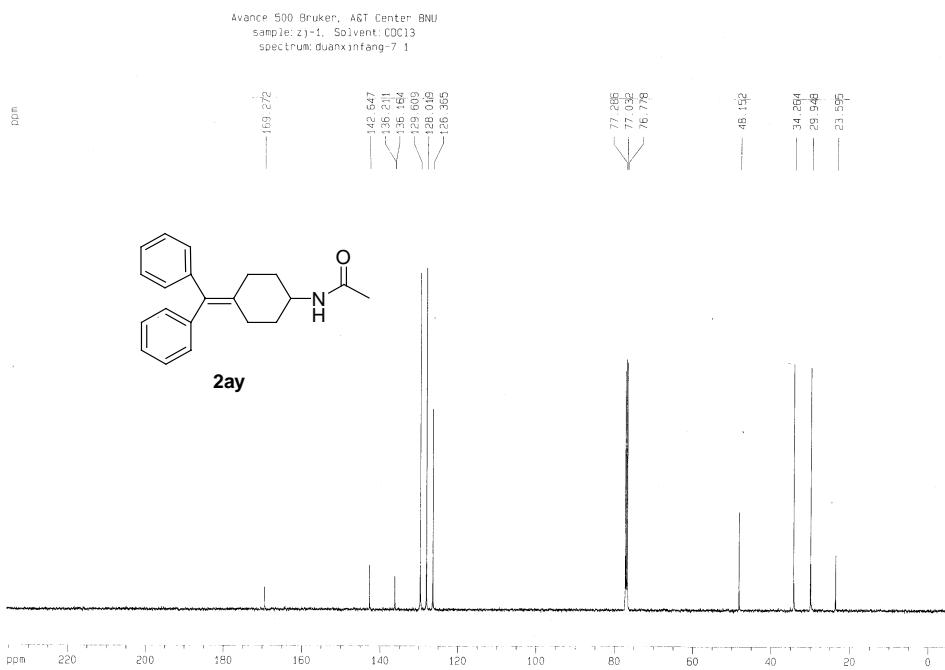
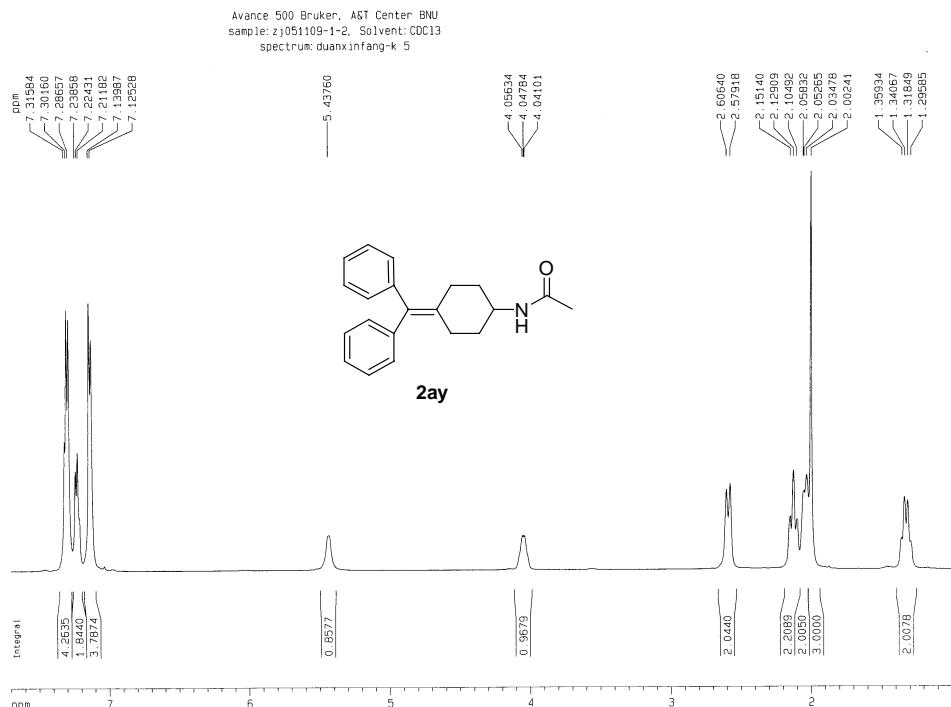


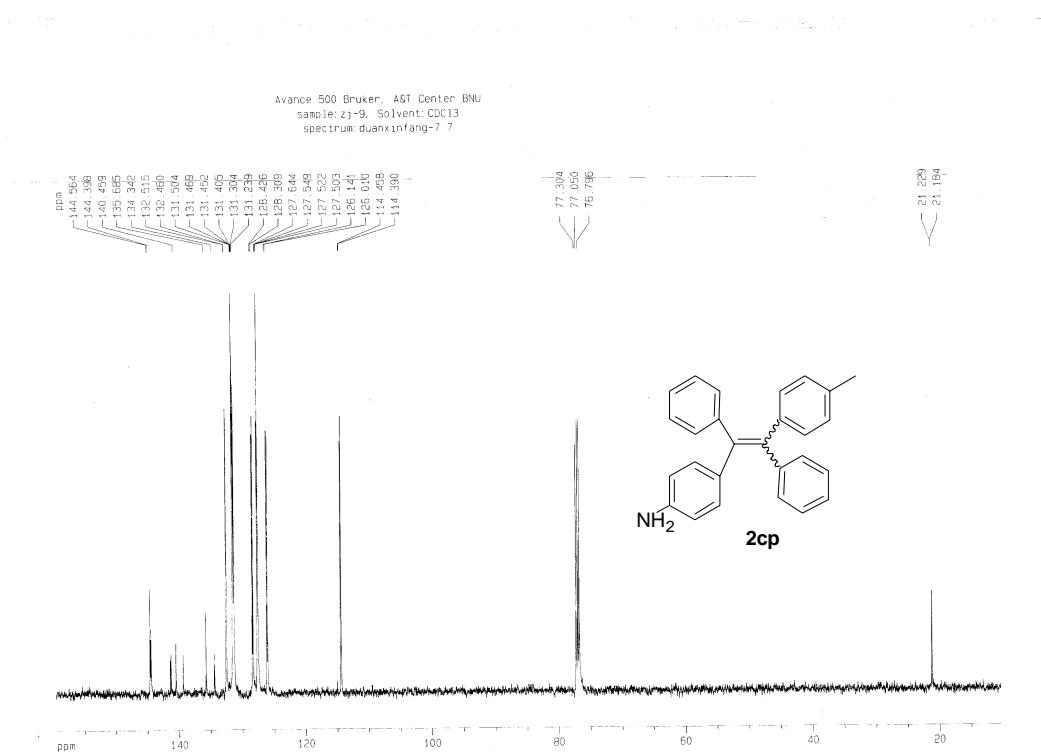
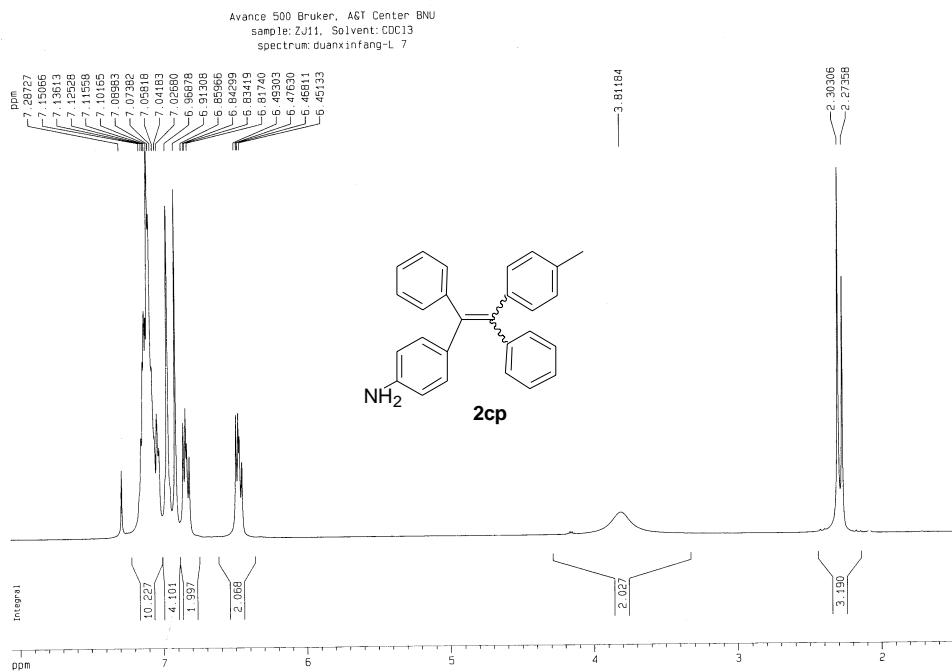


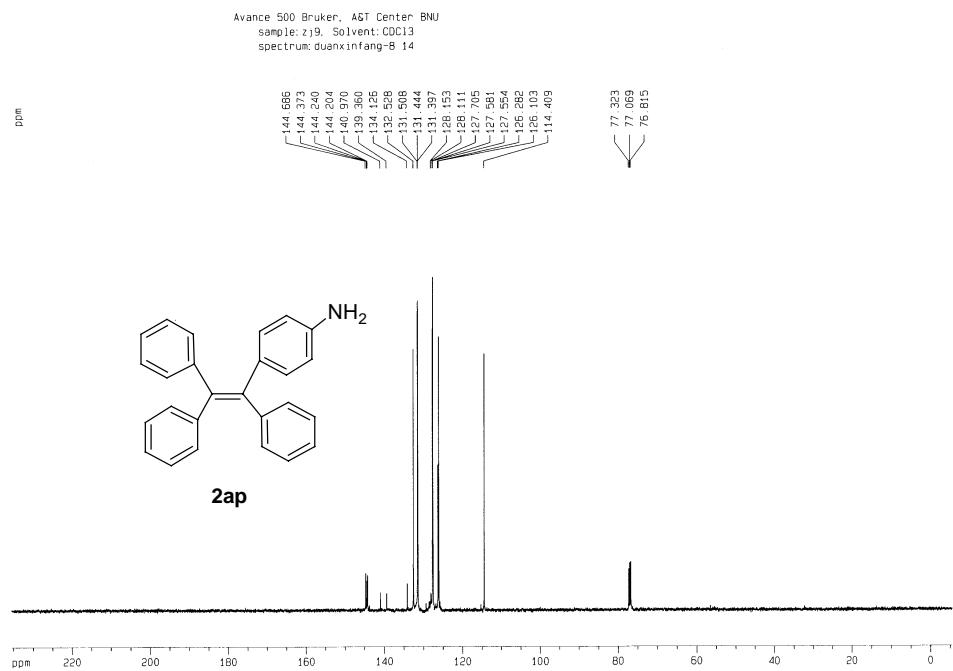
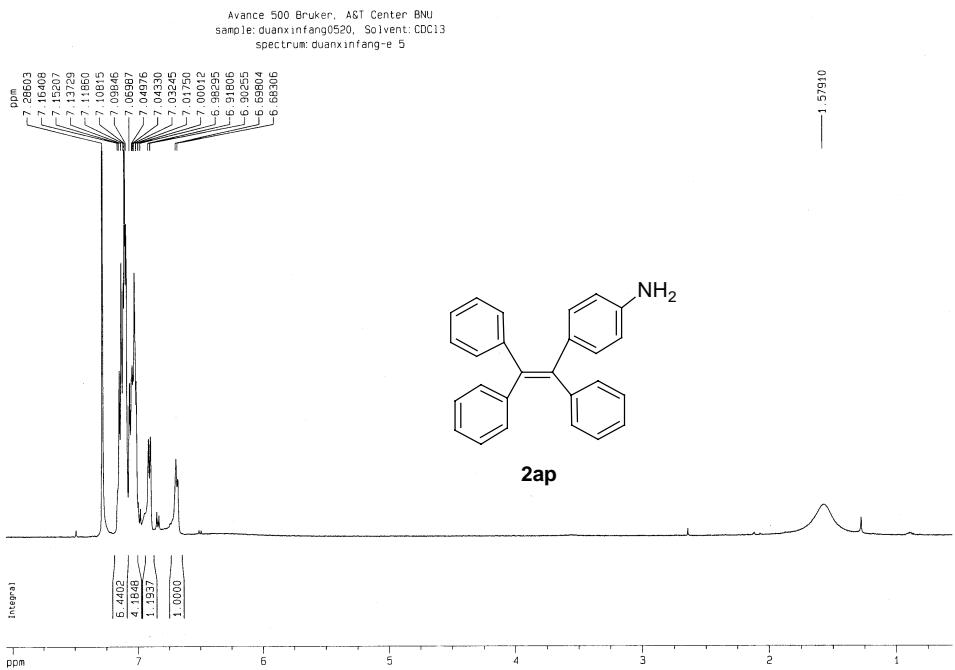


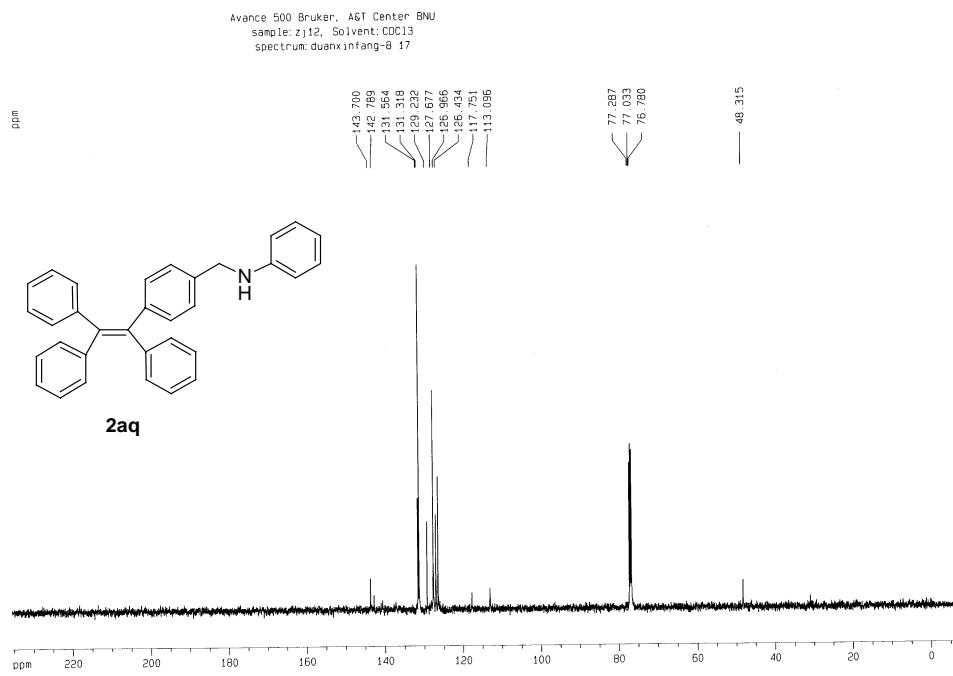
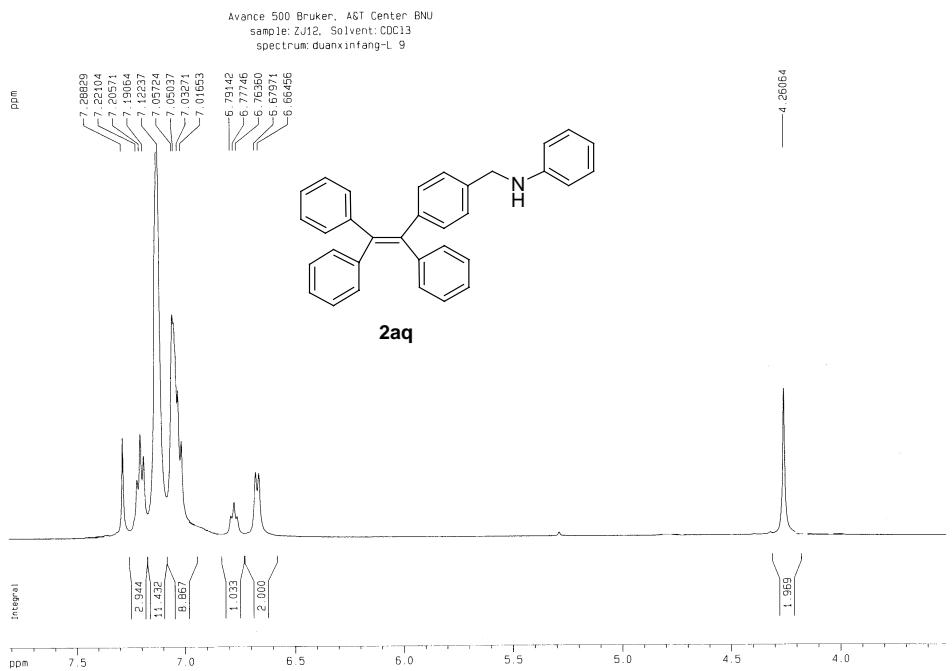


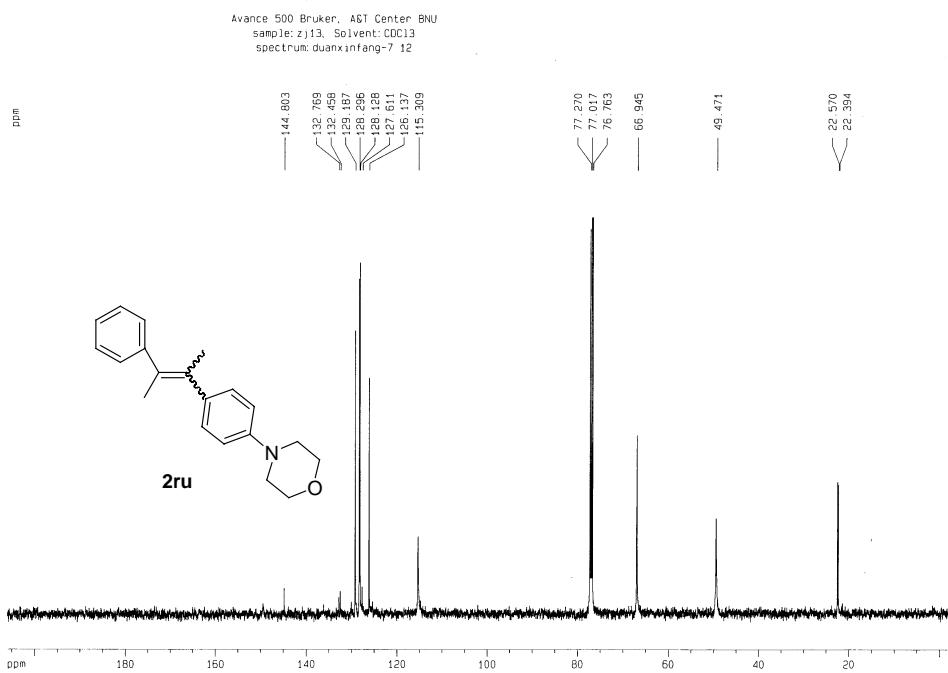
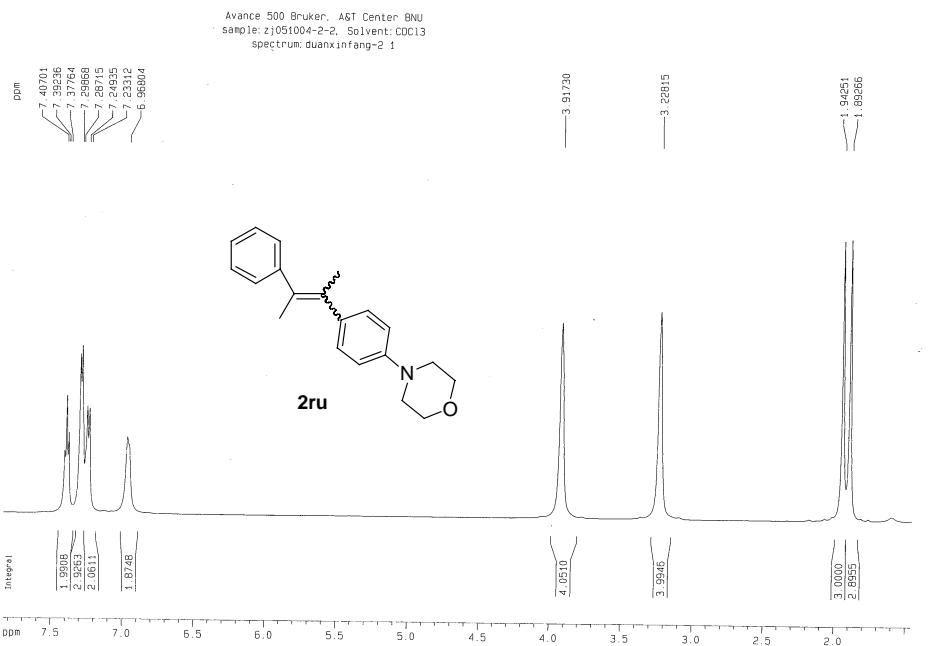


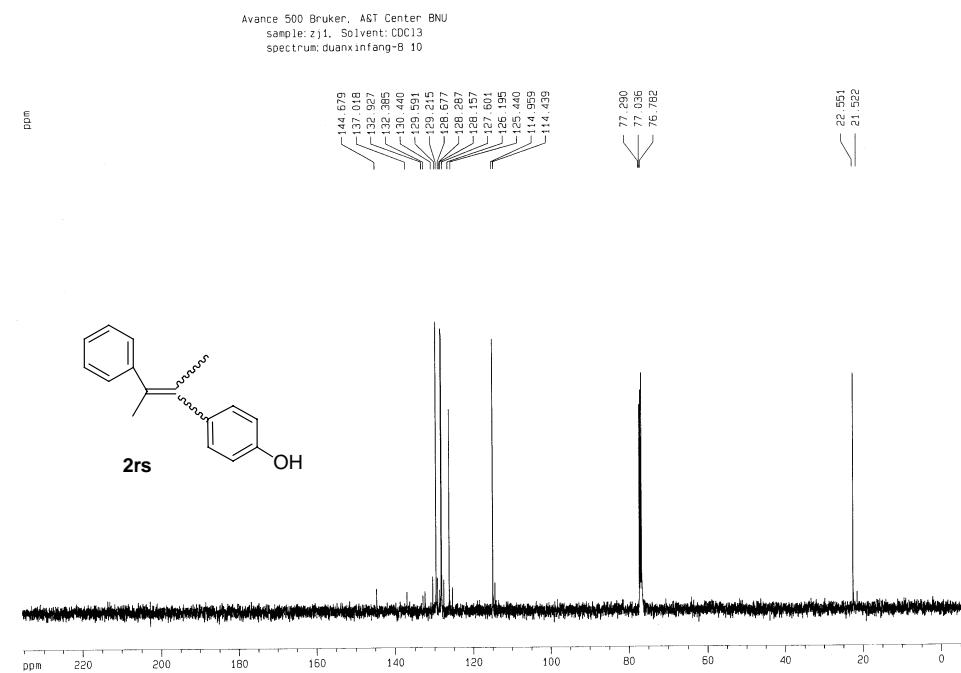
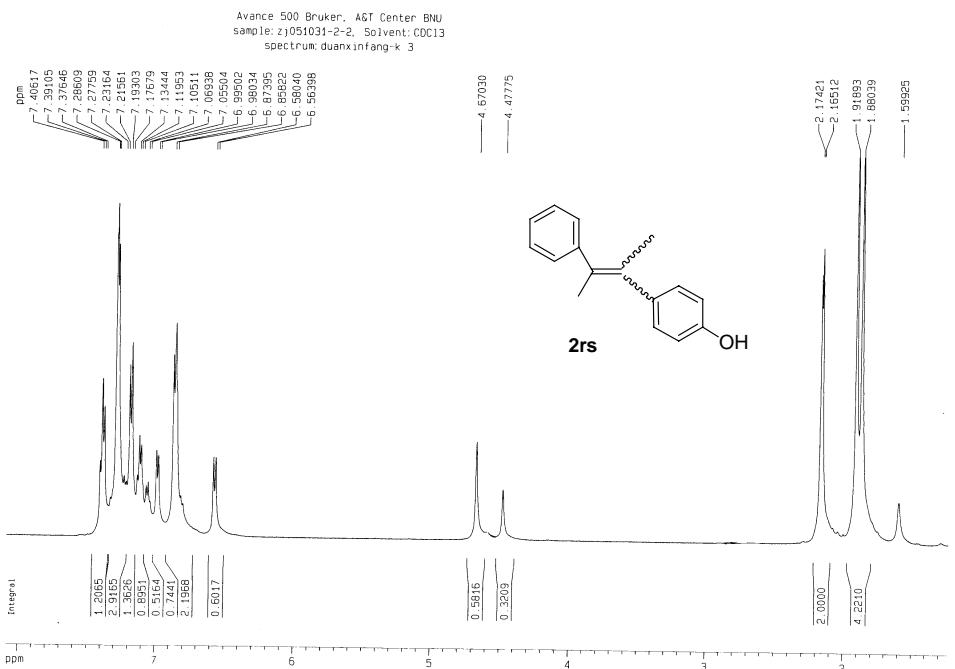


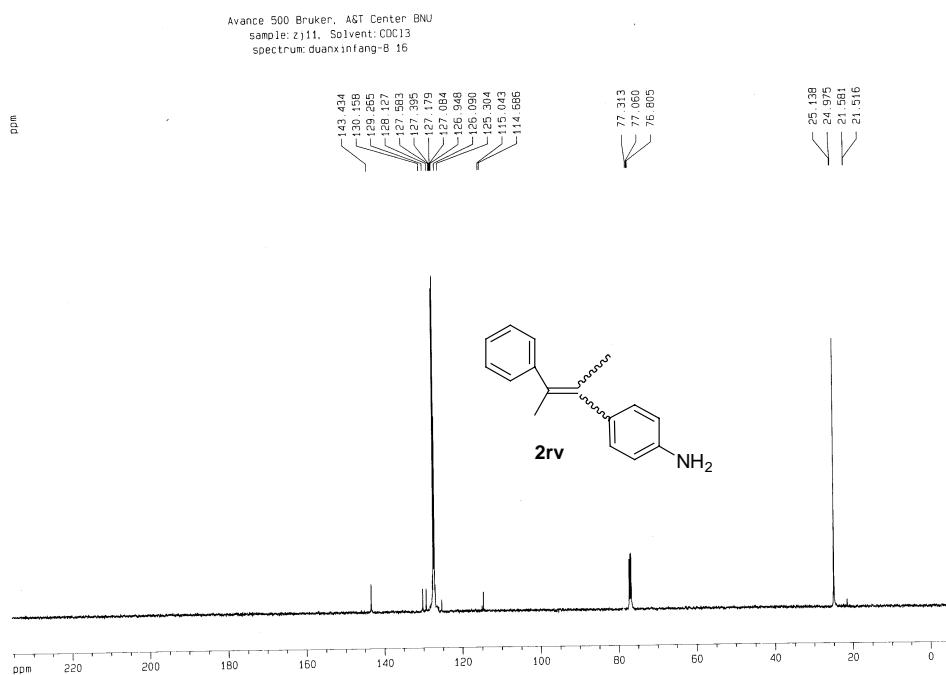
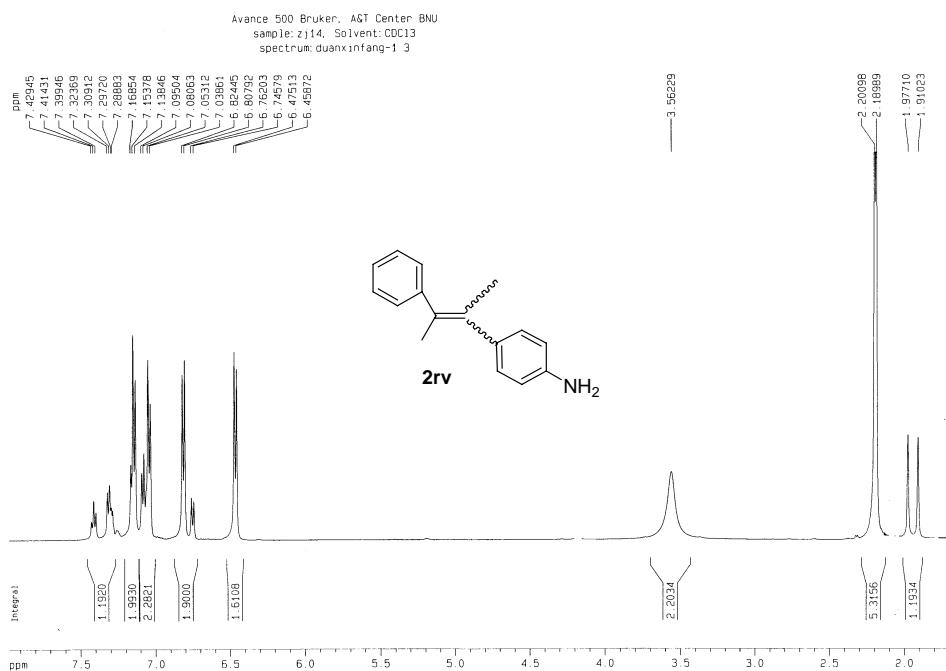


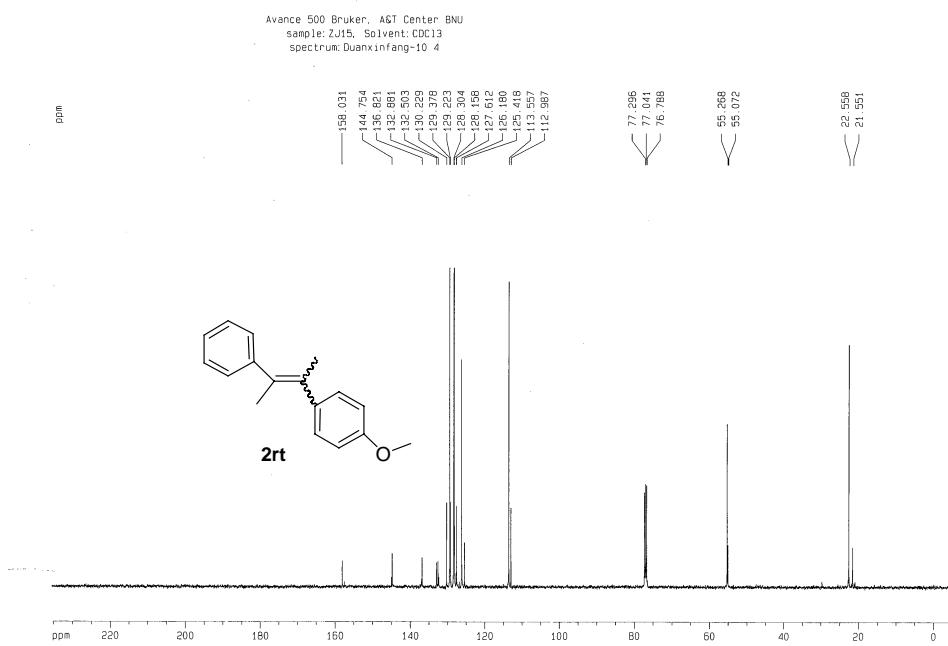
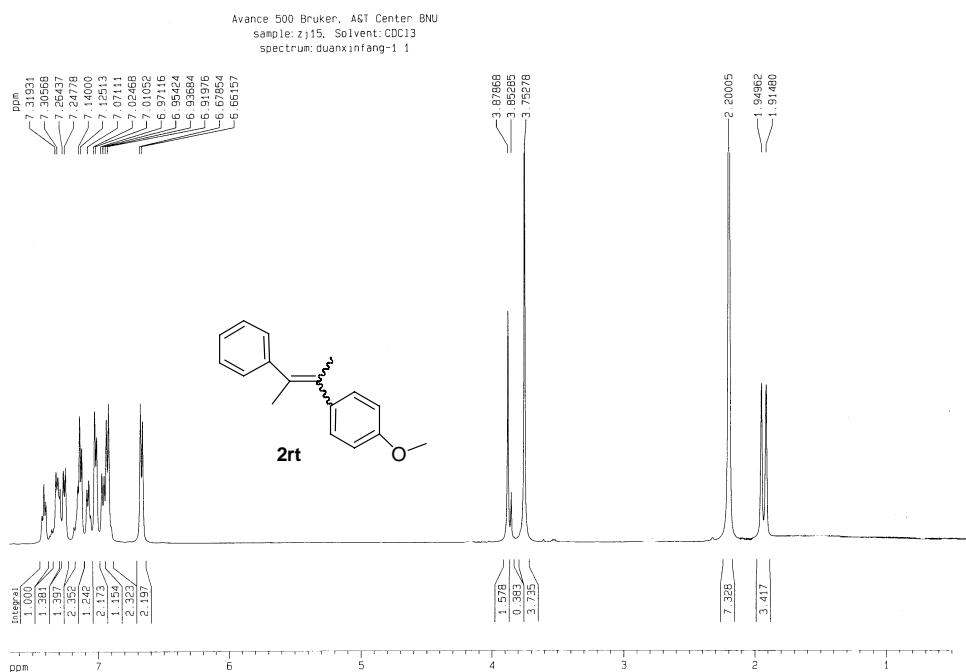


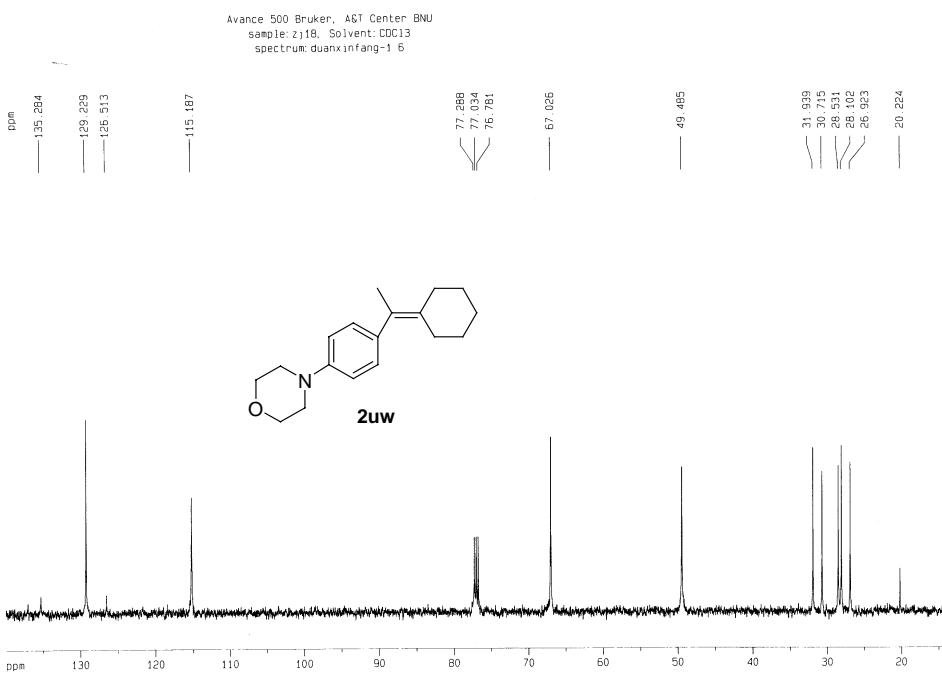
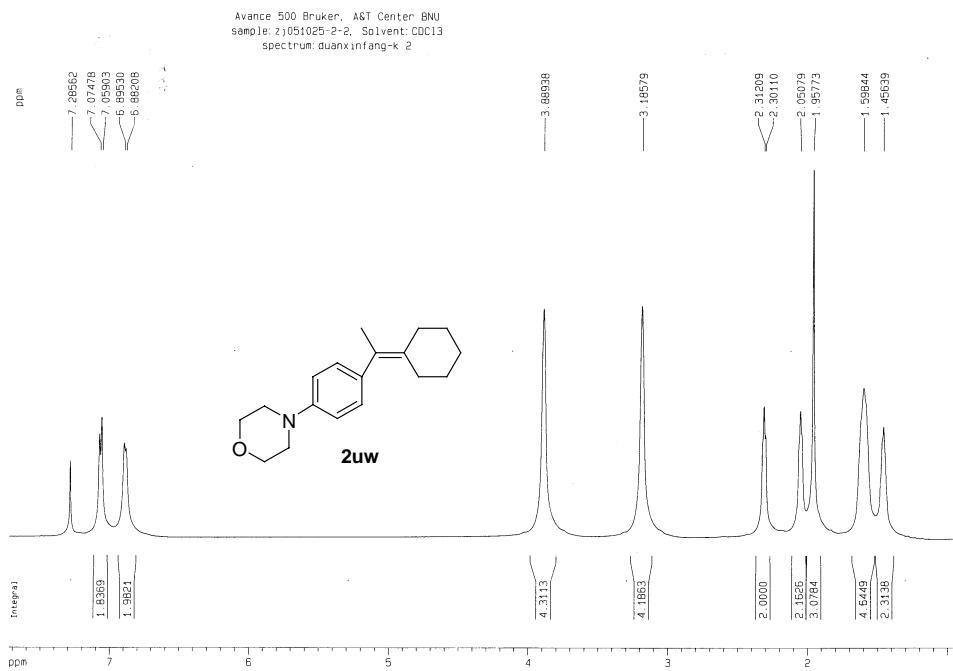


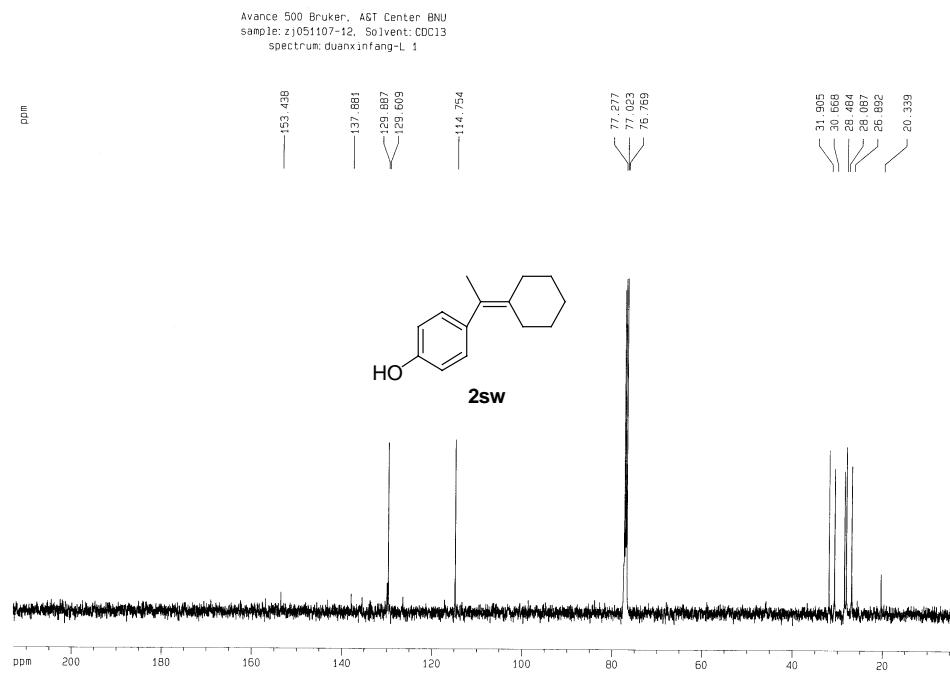
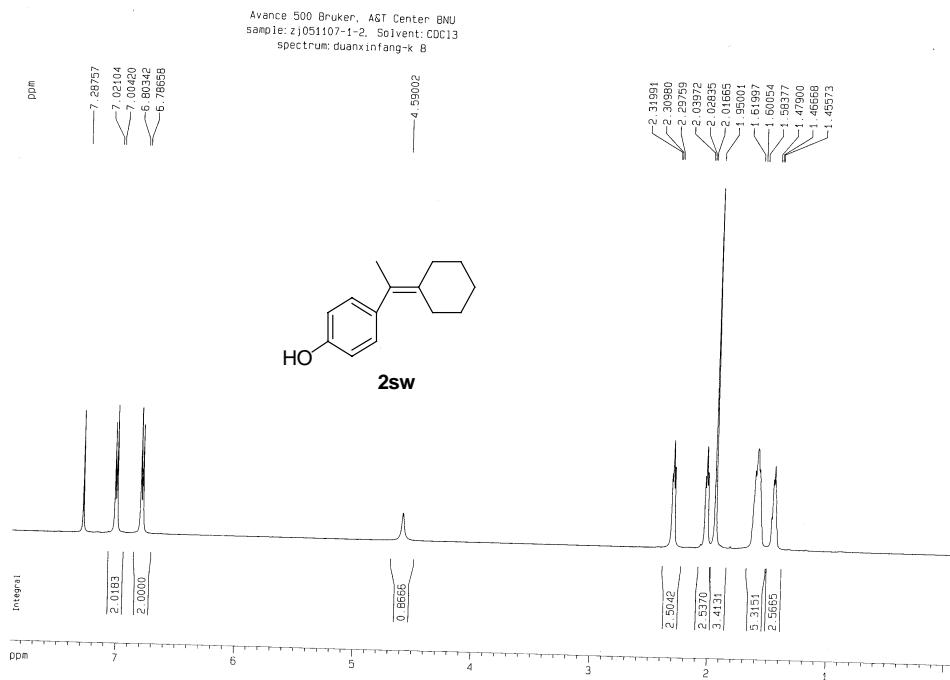


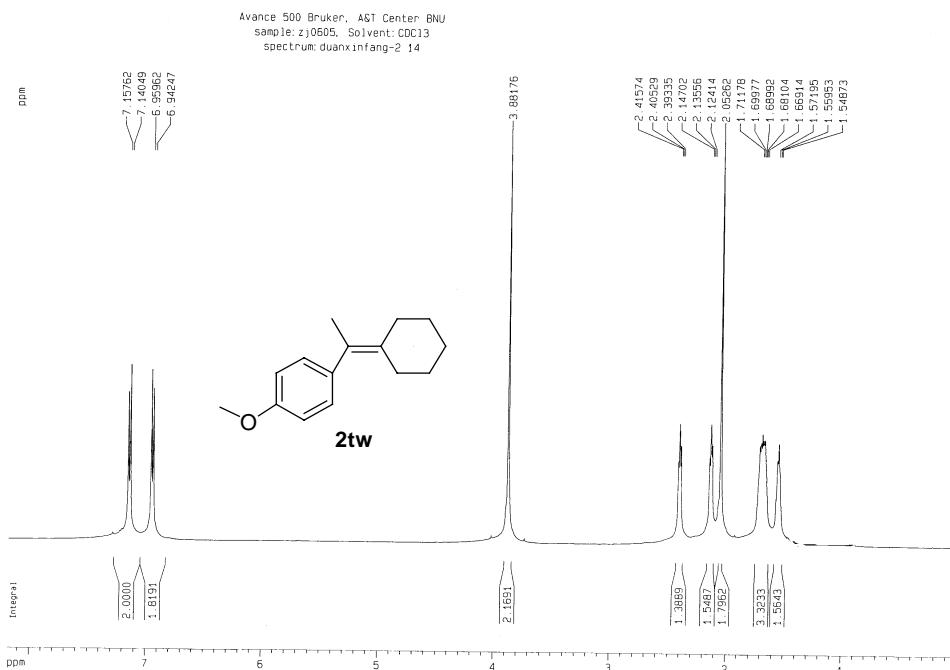


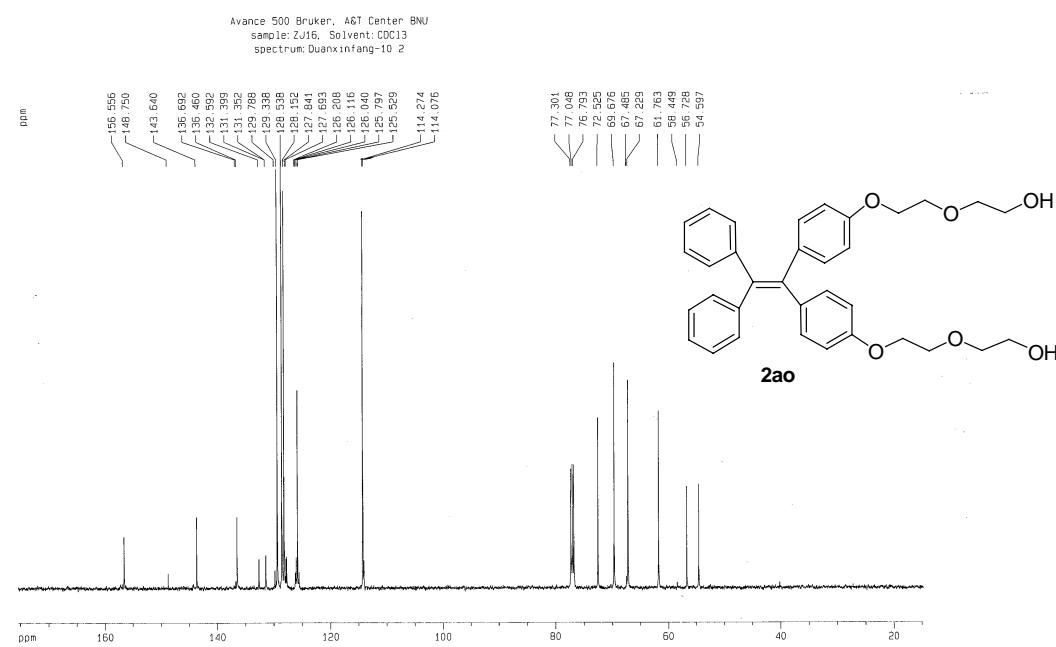
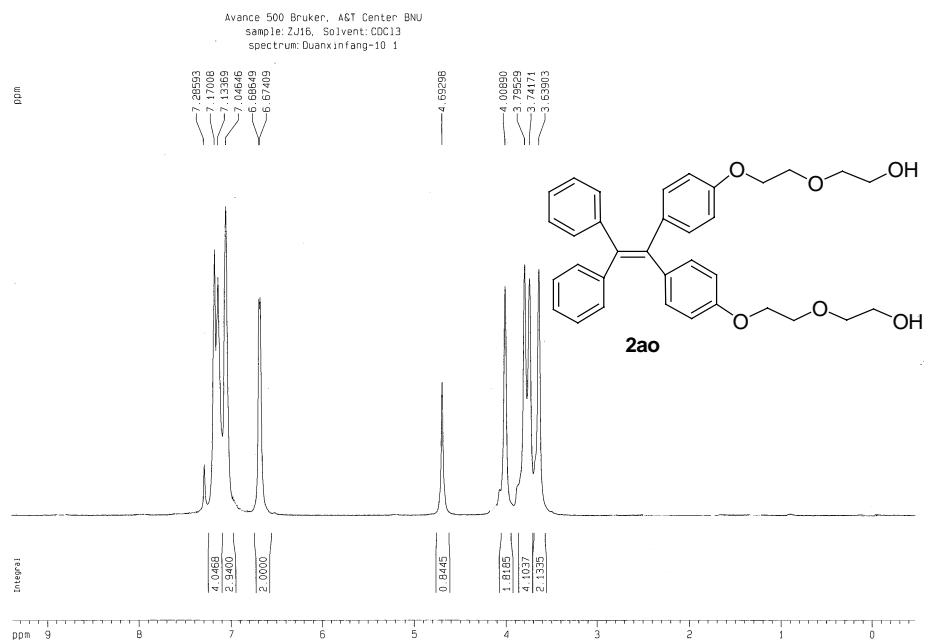


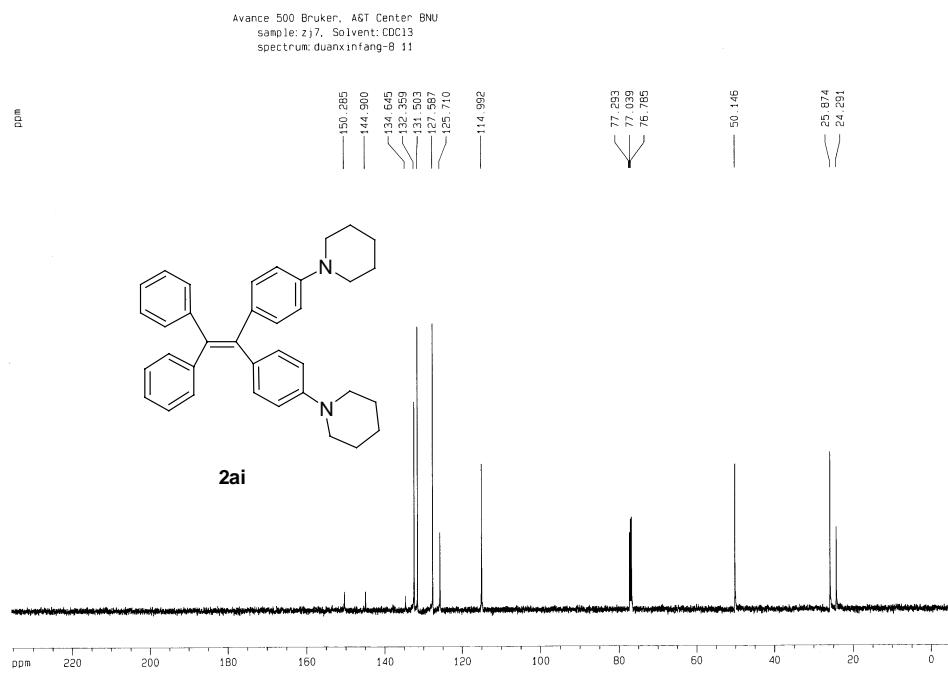
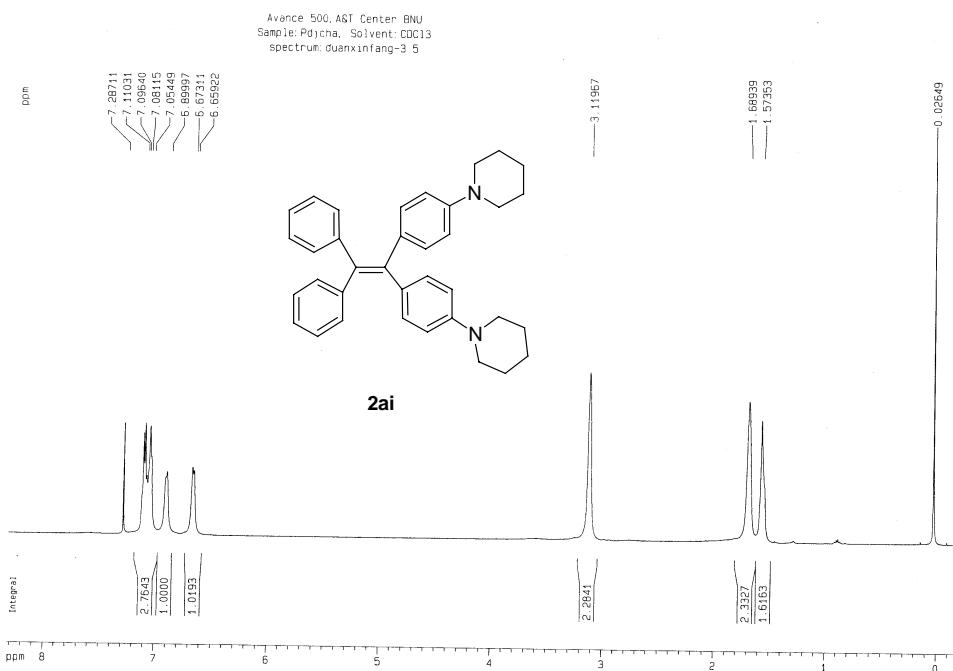


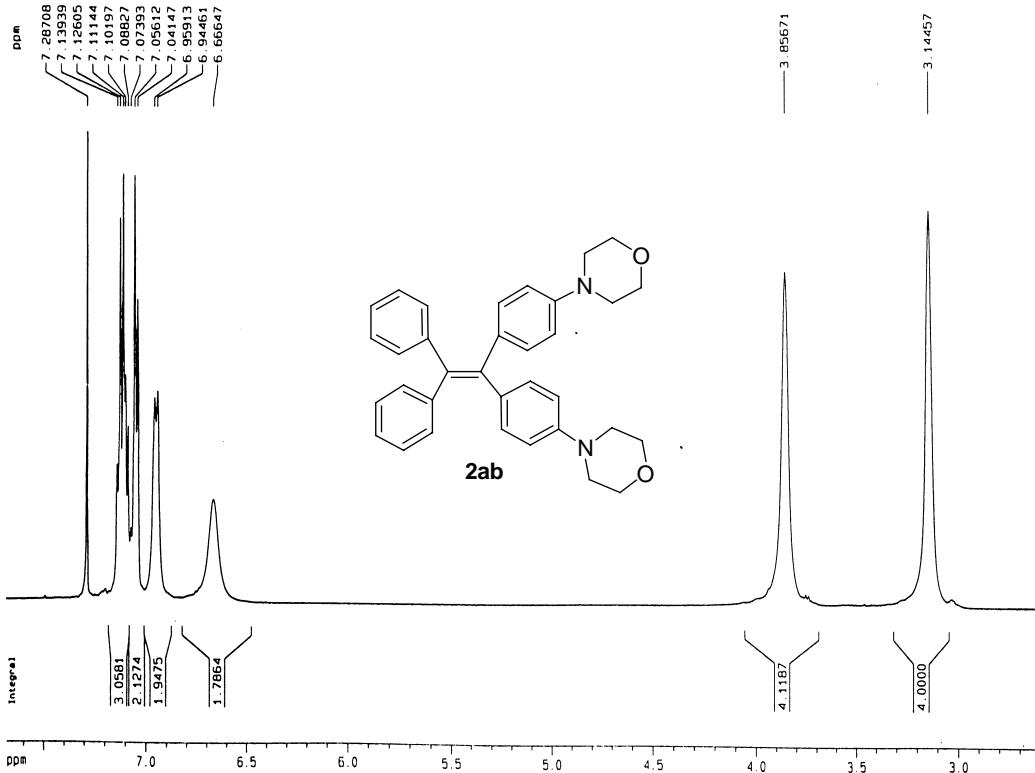




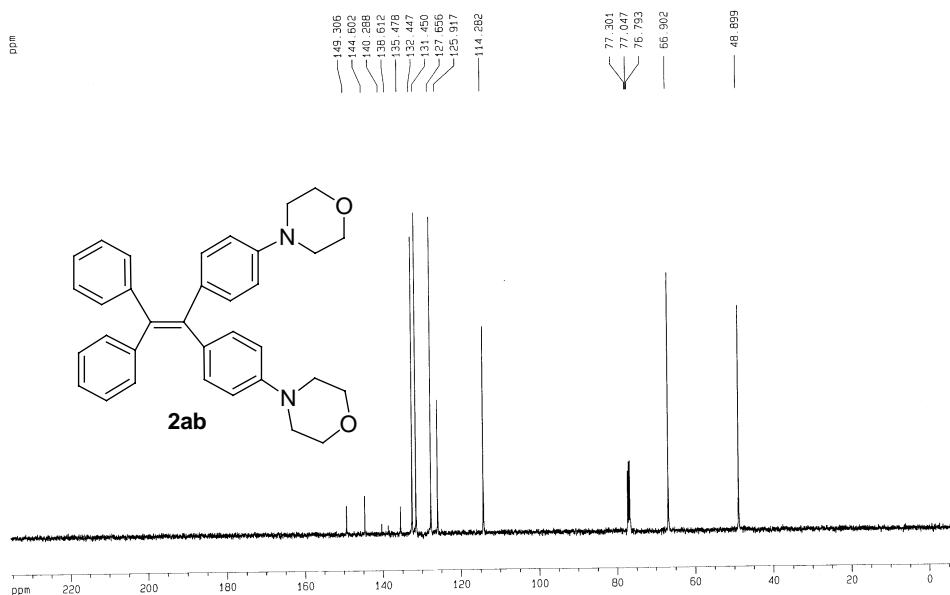


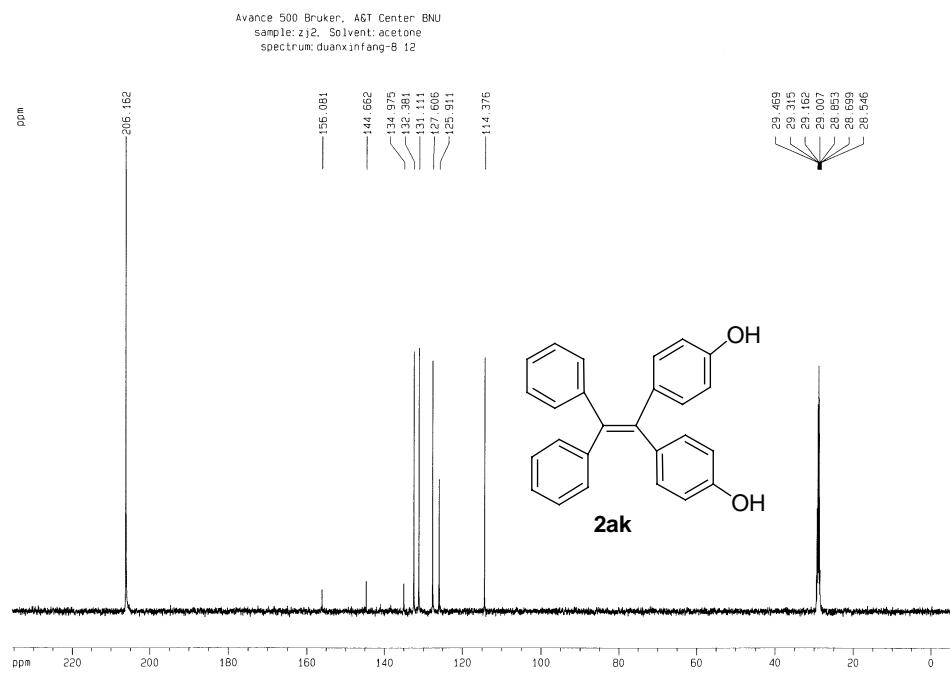
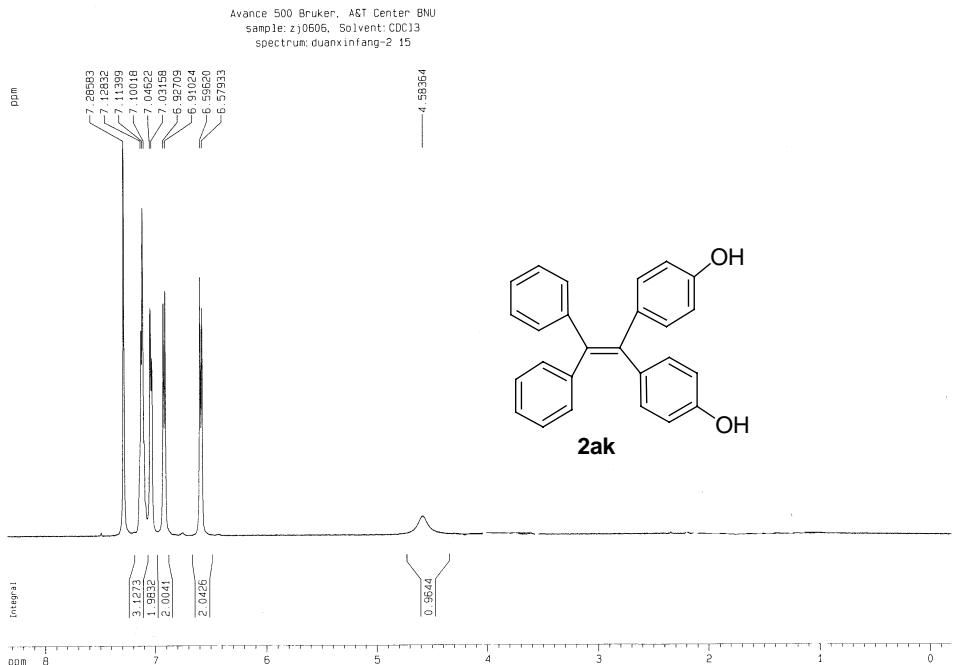


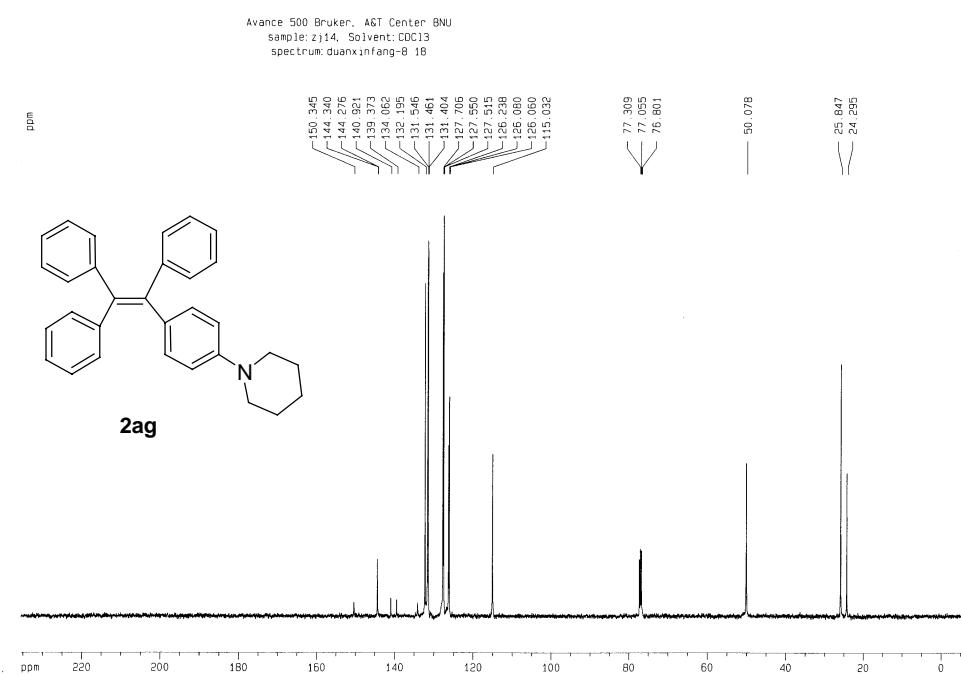
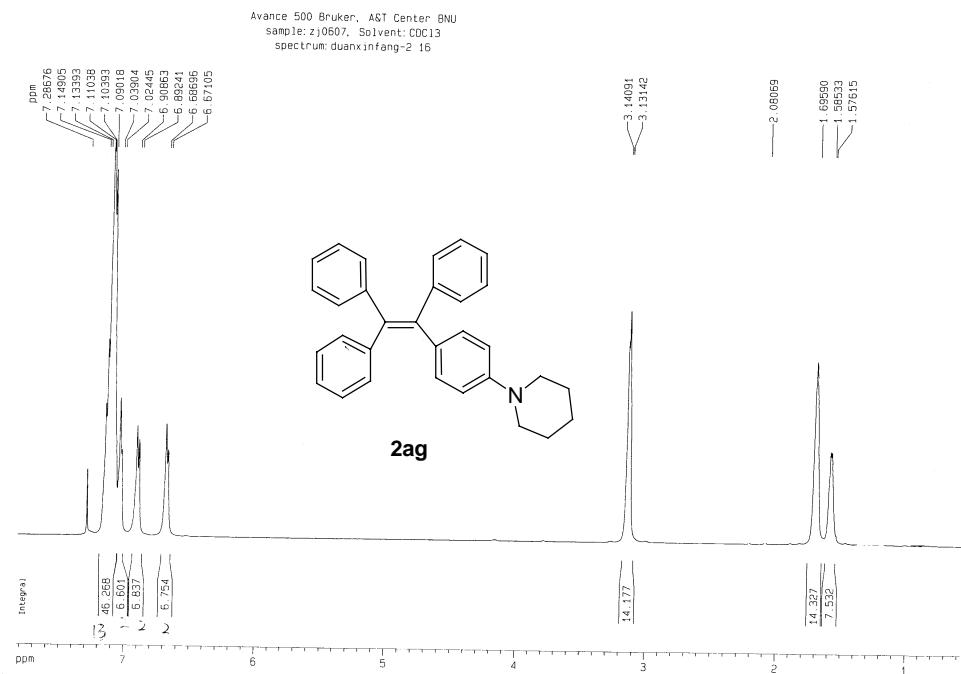


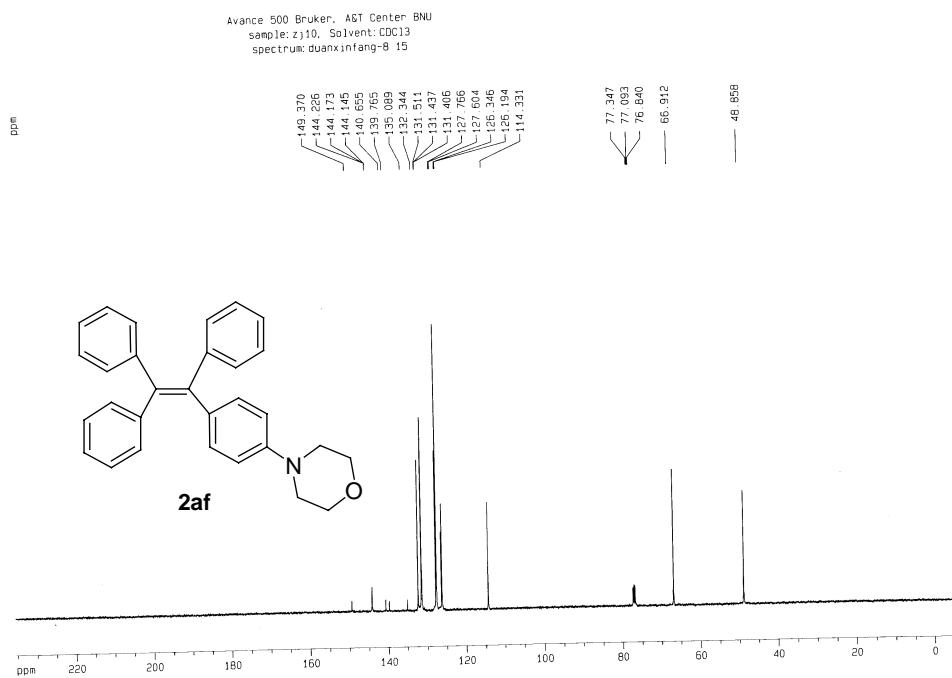
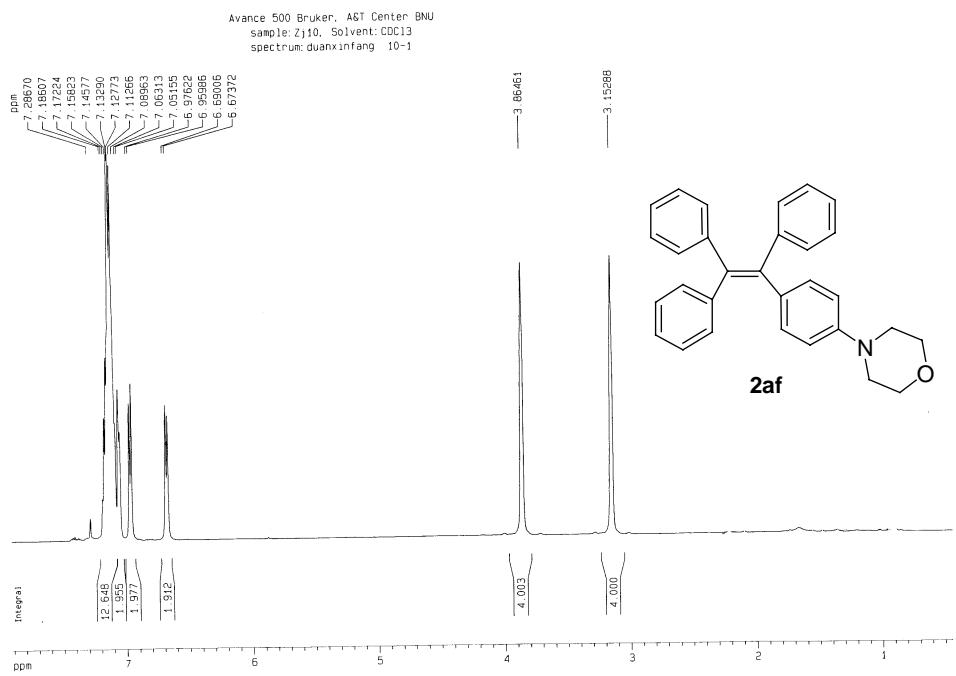


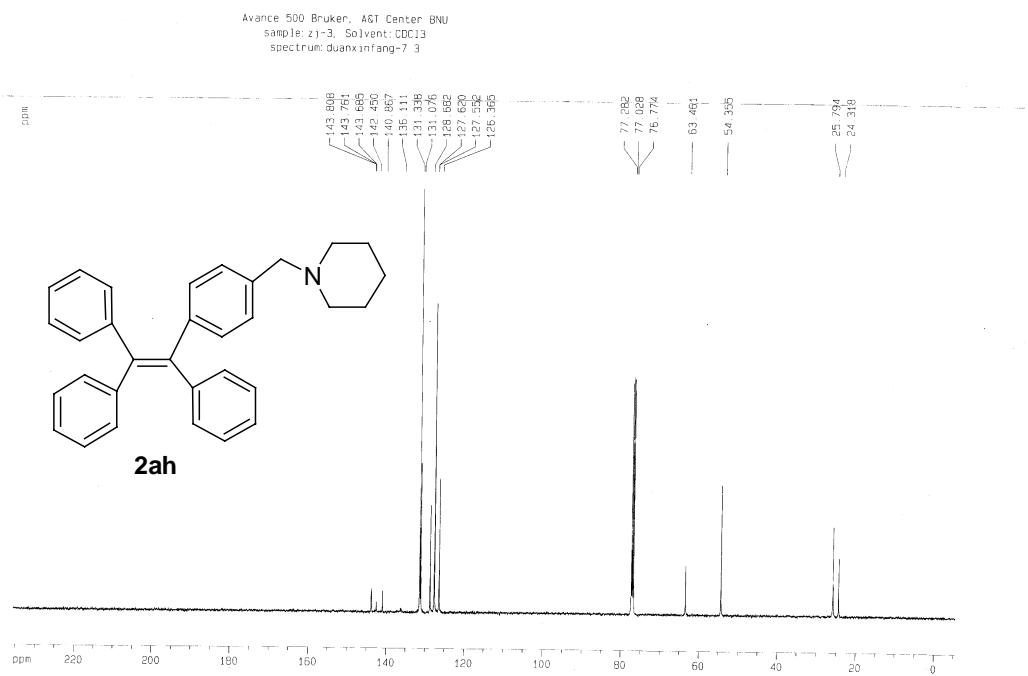
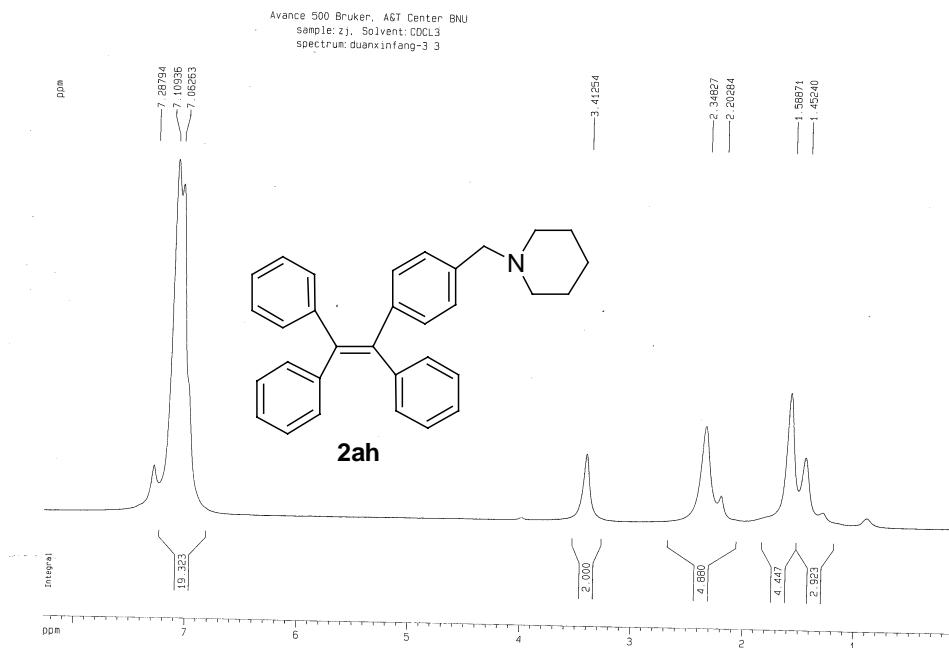
Avance 500 Bruker, A&T Center BNU
sample: zjB, Solvent: CDCl₃
spectrum: duanxinfang-B 6

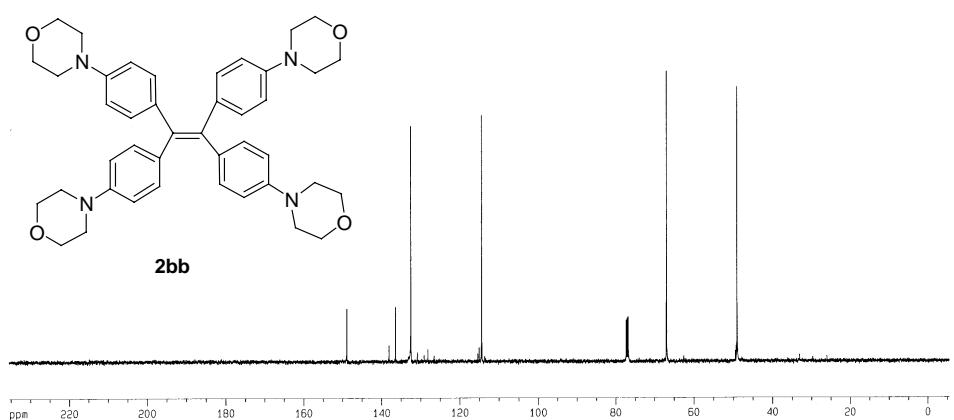
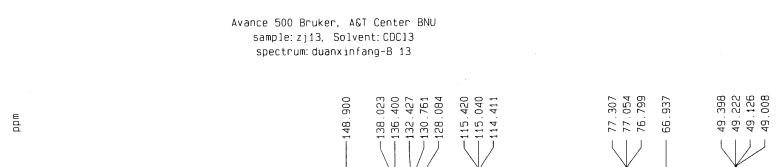
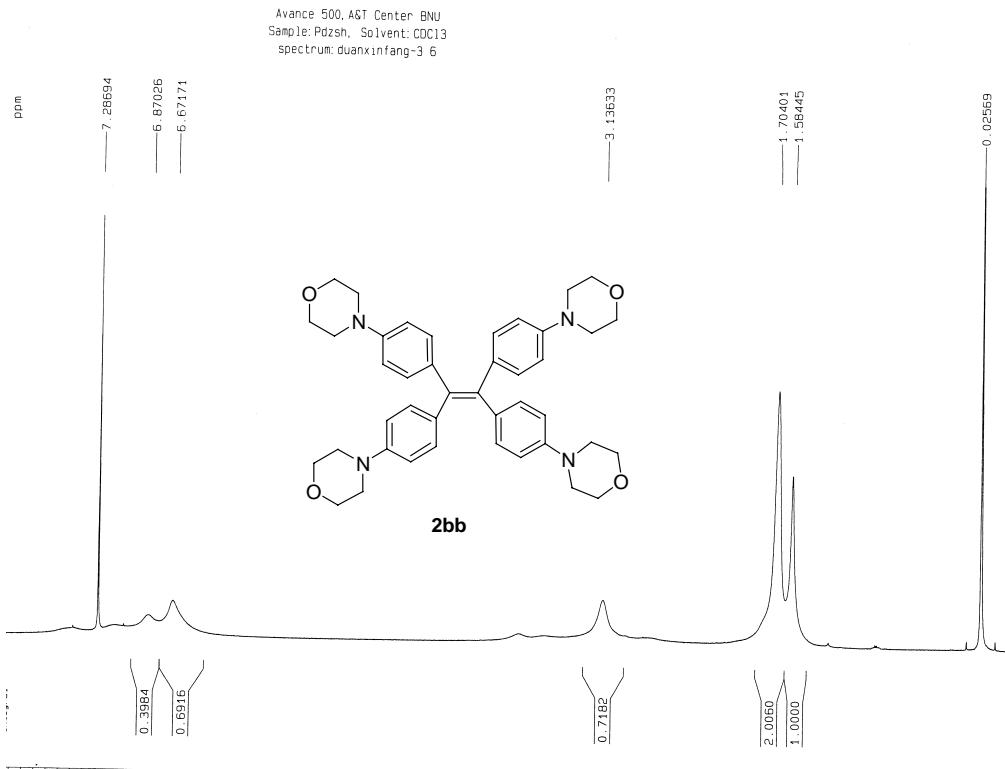


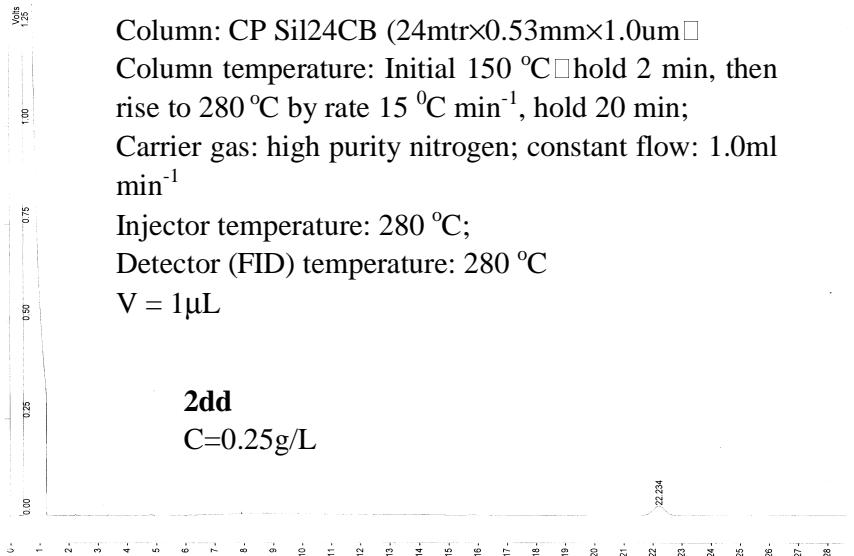












打印日期: Mon Apr 24 15:07:17 2006 第 1 页, 共 1

标题 :
运行文件 : d:\duanxinfang\060420.run
方法文件 : c:\star\zengjing.mth
样品信息 :

进样日期: 06-4-21 10:02 计算日期: 06-4-21 16:32

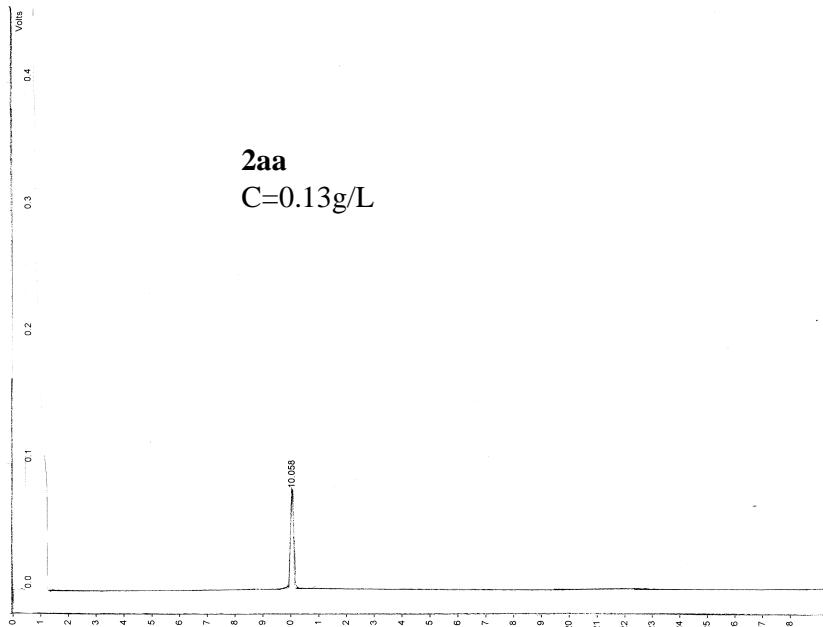
操作者: yang 检测器类型: 3800 (10 Volts)
工作站: 总线地址: 44
仪器 : Varian Star #1 取样速率: 10.00 Hz
通道 : 中间 = FID 运行时间: 28.670 分钟

** Star Chromatography Workstation Version 5.0 ** 00363-7520-4c1-0181 **

运行模式: 分析
峰测试: 峰面积
计算类型: 百分数(归一化结果)

No.	峰 名 称	结 果 O	保 留 时 间 (分)	时 间 偏 置 (分)	面 积 (计数)	Sep. 码	宽 度 1/2 (sec)	组 状态 码
1		100.0000	22.234	0.000	688633	BB	26.8	0
组 0		100.0000		0.000	688633			
	总量:	100.0000		0.000	688633			

未确认总数 : 688633
检测到的峰数: 1 舍弃的峰: 0 已经确认的峰: 0
放大倍数: 1 缩小倍数: 1 未确认的峰系数: 0
基线偏置: 2 microVolts
噪音 (用的): 29 microVolts - 在这个运行前监控的
手动进样



打印日期: Mon Apr 24 15:48:34 2006 第 1 页, 共 1

标题 :
 运行文件 : d:\duanxinfang\060420002.run
 方法文件 : c:\star\zengjing.mth
 样品信息 :

进样日期: 06-4-21 11:07 计算日期: 06-4-24 15:48

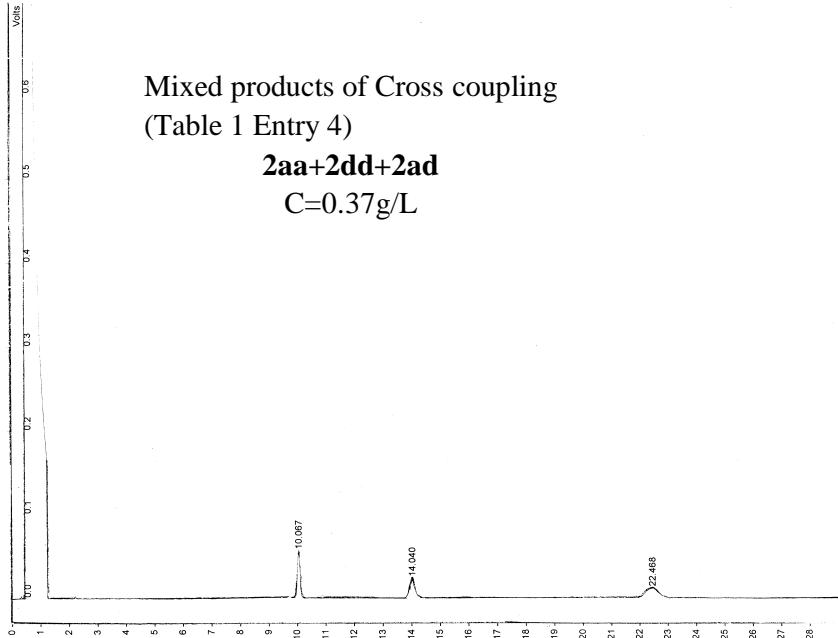
操作者: yang 检测器类型: 3800 (10 Volts)
 工作站: 总线地址: 44
 仪器 : Varian Star #1 取样速率: 10.00 Hz
 通道 : 中间 = FID 运行时间: 28.670 分钟

** Star Chromatography Workstation Version 5.0 ** 00363-7520-4c1-0181 **

运行模式: 分析
 峰测试: 峰面积
 计算类型: 百分数

No.	峰 名 称	结 果	保 留	时 间	偏 置	面 积	Sep.	宽 度	组 状态
		O	时间	(分)	(分)	(计数)	码	1/2 (sec)	
1		100.0000	10.058	0.000		499609	BB	5.8	0
组 0		100.0000		0.000		499609			
总量:		100.0000		0.000		499609			

未确认总数 : 499609
 检测到的峰数: 1 舍弃的峰: 0 已经确认的峰: 0
 放大倍数: 1 缩小倍数: 1 未确认的峰系数: 0
 基线偏置: 49 microVolts
 噪音 (用的): 32 微Volts - 在这个运行前监控的
 手动进样



打印日期: Mon Apr 24 15:04:43 2006 第 1 页, 共 1

标题 :
运行文件 : d:\duanxinfang\060420003.run
方法文件 : c:\star\zengjing.mth
样品信息 :

进样日期: 06-4-21 11:39 计算日期: 06-4-24 14:59

操作者: yang 检测器类型: 3800 (10 Volts)
工作站: 总线地址: 44
仪器 : Varian Star #1 取样速率: 10.00 Hz
通道 : 中间 = FID 运行时间: 28.670 分钟

** Star Chromatography Workstation Version 5.0 ** 00363-7520-4c1-0181 **

运行模式: 分析
峰测试: 峰面积
计算类型: 百分数(归一化结果)

No.	峰 名 称	结 果 (%)	保 留 时 间 (分)	时 间 偏 置 (分)	面 积 (计数)	Sep. 码	宽 度 1/2 (sec)	组 状态 码
1		33.9306	10.067	0.000	358641	BB	5.9	0
2		29.9761	14.040	0.000	316843	BB	12.1	0
3		36.0933	22.468	0.000	381501	BB	28.3	0
	组 0	100.0000		0.000	1056985			
	总量:	100.0000		0.000	1056985			

未确认总数 : 1056985
检测到的峰数: 3 舍弃的峰: 0 已经确认的峰: 0
放大倍数: 1 缩小倍数: 1 未确认的峰系数: 0
基线偏置: -43 microVolts
噪音 (用的): 32 microVolts - 在这个运行前监控的
手动进样
