Supporting Information

A GaCl₃-Catalyzed [4+1] Cycloaddition of α , β -Unsaturated Ketones and Isocyanides Leading to Unsaturated Lactone Derivatives

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General Information. ¹H NMR and ¹³C NMR were recorded on a JEOL JMN-270 spectrometer in CDCl₃ with tetramethylsilane as an internal standard. Data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, sept = septet, m = multiplet and c = complex), coupling constant (Hz), integration, and interpretation. Infrared spectra (IR) were obtained on a Hitachi 270-50 spectrometer; absorptions are reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak). Mass spectra were obtained on a Shimadzu GCMS-QP 5000 instrument with ionization voltages of 70 eV. Elemental analyses were performed by the Elemental Analysis Section of Osaka University. High resolution mass spectra (HRMS) were obtained on a JEOL JMS-DX303. Analytical GC was carried out on a Shimadzu GC-14B gas chromatography, equipped with a flame ionization detector. Column chromatography was performed with SiO₂ (Wakogel).

Materials. Toluene and methylcyclohexane was distilled from CaH₂. GaCl₃ was purchased from Aldrich Chemical Co. and used as a 1.0 M stock solution in methylcyclohexane. 2,6-Dimethylphenylisocyanide, 2,6-bis(1-methylethyl)phenylisocyanide, 2-methylphenylisocyanide and 2-trifluoromethylphenylisocyanide were prepared from the corresponding formamides by a modified method of van Leusen¹. 2,6-Bis(trifluoromethyl)phenylisocyanide was prepared by the procedure of Ito². *tert*-Butyl isocyanide, cyclohexyl isocyanide, enones **1**, **7**, **9**, **15** and aldehyde **13** were purchased from Aldrich Chemical Co. and used after distillation from Ca(SO₄)₂. Enone **3** was prepared from citral by reaction with MeLi³ followed by the oxidation of the resulting alcohol with MnO₂⁴. Enone **11** was prepared by the modified Horner-Wadsworth-Emmons reaction, as reported by Villieras⁵. Enone **5** was prepared from the reaction of 3,3-dimethylacryloyl chloride with 4-(trifluoromethyl)phenylmagnesium bromide in the presence of CdCl₂⁶. Enones **17**, **19** were prepared by the aldol

reaction of the corresponding aldehydes with ketones in the presense of aqueous NaOH⁷ and subsequent acid catalyzed dehydration in benzene.

Typical Procedure . The reaction was carried out in a 50mL screw-cap vial with septum inlet equipped with glass stopcock. To a stirred mixture of mesityl oxide (**1**) (1 mmol, 98 mg), 2,6-xylylisocyanide (1.1 mmol, 144 mg) in toluene (3 mL) under dry nitrogen atmosphere was added GaCl₃ (1.0 M in methylcyclohexane, 0.05 mmol, 50 μ l). The system was immersed in an oil bath at 60 °C. After 18 hours had elapsed, the vessel was removed from the oil bath and allowed to cool to room temperature. The contents were put into the 50-mL round bottom flask and the volatiles were removed in vacuo. The residue was subjected to column chromatography on silica gel (eluent; hexane/Et₂O = 20/1) to give 2,6-Methyl-N-(3,3,5-trimethyl-2-(3*H*)-furanylidene)-benzenamine (**2**) (215 mg, 94% yield) as a white solid.

2,6-Dimethyl-N-(3,3,5-trimethyl-2-(3*H*)-furanylidene)-benzenamine (2**).** White solid; mp 97 °C; *R_f* 0.20 (hexane/ether = 20/1); ¹H NMR (CDCl₃) δ 1.45 (s, 6H, 3'-Me), 1.85 (d, *J* = 1.3 Hz, 3H, 5'-Me), 2.12 (s, 6H, 2-Me, 6-Me), 5.03 (d, *J* = 1.3 Hz, 1H, 4'-H), 6.90 (brt, *J* = 7.2 Hz, 1H, 4-H), 7.02 (brd, *J* = 7.2 Hz, 2H, 3-H, 5-H); ¹³C NMR (CDCl₃) δ 13.9 (5'-Me), 17.9 (2-Me), 27.0 (3'-Me), 45.0 (3'-C), 110.9 (4'-C), 122.7 (4-C), 127.3 (3-C), 127.6 (2-C), 144.5 (1-C), 150.4 (5'-C), 167.0 (2'-C); IR (KBr) 3084 w, 3024w, 2976s, 2932m, 2868m, 1724s, 1682s, 1660m, 1596m, 1530m, 1466s, 1442m, 1382s, 1360w, 1308w, 1250s, 1224s, 1206s, 1146m, 1088s, 1034w, 944s, 886w, 844w, 804m; MS, *m/z* (relative intensity, %) 229 (M⁺, 26), 187 (15), 186 (100), 144 (23), 130 (13), 105 (20), 103 (16), 83 (31), 79 (25), 78 (10), 77 (36), 55 (25), 53 (14), 51 (13); Anal. Calcd for C₁₅H₁₉NO: C, 78.56; H, 8.35; N, 6.11. Found: C, 78.32; H, 8.51; N, 6.18.

2,6-Bis(1-methylethyl)-N-(3,3,5-trimethyl-2-(3*H*)-furanylidene)-benzenamine. Colorless oil; *R_f* 0.24 (hexane/ether = 20/1); ¹H NMR (CDCl₃) δ 1.17 (d, *J* = 6.9 Hz, 12H, CH(CH₃)₂), 1.44 (s, 6H, 3'-Me), 1.81 (d, *J* = 1.6 Hz, 3H, 5'-Me), 2.94 (sept, *J* = 6.9 Hz, 2H, CH(CH₃)₂), 5.02 (d, *J* = 1.3 Hz, 1H, 4'-H), 7.03-7.13 (c, 3H, 3-H, 4-H, 5-H); ¹³C NMR (CDCl₃) δ 13.9 (5'-Me), 23.2 (CH(CH₃)₂), 26.7 (3'-Me), 28.3 (CH(CH₃)₂), 45.0 (3'-C), 110.3 (4'-C), 122.4 (3-C), 123.3 (4-C), 137.9 (2-C), 142.2 (1-C), 150.3 (5'-C), 166.8 (2'-C); IR (neat) 3798 w, 3710 w, 3606 w, 3398 w, 3176 w, 3058 m, 2926 s, 2870 s, 2710 w, 2598 w, 2462 w, 2382 w, 2158 w, 2076 w, 2018 w, 1909 w, 1875 w, 1725 s, 1679 s, 1589 s, 1521 w, 1459 s, 1437 s, 1379 s, 1358 s, 1327 m, 1299 m, 1253 s, 1220 s, 1185 m, 1132 m, 1078

s, 996 w, 941 s, 883 w, 840 w, 805 w, 775 s, 733 s, 684 w, 615 m, 591 w; MS, m/z (relative intensity, %) 285 (M^+ , 25), 270 (2.6), 242 (100), 227 (5.4), 212 (5.4), 200 (12), 186 (7.2), 172 (5.6), 158 (3.6), 144 (4.1), 130 (3.8), 115 (2.5), 91 (4.6), 83 (4.7), 69 (3.6), 55 (2.7); Anal. Calcd for $C_{19}H_{27}NO$: C, 79.95; H, 9.53; N, 4.91. Found: C, 79.68; H, 9.40; N, 4.91.

2-Methyl-*N*-(3,3,5-trimethyl-2-(3*H*)-furanylidene)-benzenamine. White solid; mp 67 °C; R_f 0.21(hexane/ether = 20/1); ^1H NMR ($CDCl_3$) δ 1.40 (s, 6H, 3'-Me), 1.86 (d, J = 1.3 Hz, 3H, 5'-Me), 2.17 (s, 3H, 2-Me), 5.03 (d, J = 1.3 Hz, 1H, 4'-H), 6.90 (d, J = 7.9 Hz, 1H, 6-H), 6.98 (ddd, J = 7.6 Hz, J = 7.3 Hz, J = 1.3 Hz, 1H, 4-H), 7.09-7.19 (c, 2H, 3-H, 5-H); ^{13}C NMR ($CDCl_3$) δ 13.8, 17.8, 27.0, 45.2, 110.4, 120.9, 123.3, 125.9, 129.0, 129.9, 145.6, 150.3, 167.5; IR(KBr) 3912 w, 3690 w, 3396 w, 3344 w, 3190 w, 3082 w, 3010 w, 2966 m, 2926 m, 2862 m, 2738 w, 2672 w, 2564 w, 2524 w, 2442 w, 2318 w, 2174 w, 2080 w, 2030 w, 1991 w, 1965 w, 1940 w, 1901 w, 1725 s, 1679 s, 1599 s, 1577 m, 1485 s, 1460 m, 1440 m, 1376 s, 1355 w, 1303 m, 1287 w, 1256 s, 1217 s, 1187 m, 1139 m, 1110 m, 1088 s, 1033 m, 1009 w, 995 w, 946 s, 931 s, 885 w, 858 w, 850 m, 797 m, 765 m, 736 s, 669 w, 613 w, 524 w; MS, m/z (relative intensity, %) 215 (M^+ , 28), 200 (4.8), 182 (1.0), 172 (100), 144 (1.9), 130(20), 118(9.7), 98(4.0), 91(43), 65(41), 55(27); Anal. Calcd for $C_{14}H_{17}NO$: C, 78.10; H, 7.96; N, 6.51. Found: C, 78.01; H, 7.97; N, 6.58.

2,6-Bis(trifluoromethyl) -*N*-(3,3,5-trimethyl-2-(3*H*)-furanylidene)-benzenamine. Colorless oil; R_f 0.49 (hexane/ether = 20/1); ^1H NMR ($CDCl_3$) δ 1.39 (s, 6H, 3'-Me), 1.83 (d, J = 1.3 Hz, 3H, 5'-Me), 5.05 (d, J = 1.3 Hz, 1H, 4'-H), 7.21 (t, J = 7.9 Hz, 1H, 4-H), 7.78 (d, J = 7.9 Hz, 2H, 3-H, 5-H); ^{13}C NMR ($CDCl_3$) δ 13.4 (5'-Me), 26.0 (3'-Me), 45.6 (3'-C), 110.8 (4'-C), 122.3 (4-C), 122.3 (q, J = 30.5 Hz, 2-C), 123.2 (q, J = 273 Hz, CF_3), 129.7 (q, J = 5.0 Hz, 3-C) 145.6 (1-C), 150.3 (5'-C), 171.1 (2'-C); IR (neat) 3828 w, 3734 w, 3302 w, 3094 w, 2972 s, 2930 s, 2868 m, 2512 w, 2418 w, 2244 w, 2050 w, 1954 w, 1911 w, 1874 w, 1837 w, 1724 s, 1683 s, 1636 w, 1605 s, 1594 s, 1454 s, 1383 s, 1337 s, 1293 s, 1261 s, 1207 s, 1159 s, 1080 s, 1063 s, 998m, 951 s, 884 w, 852 m, 832 s, 786 s, 746 s, 728 s, 673 s, 639 w, 613 w, 576 w, 514 m, 487 m; MS, m/z (relative intensity, %) 337 (M^+ , 19), 332 (10), 294 (100), 240 (9.1), 213 (11), 163 (6.1), 137 (24), 109 (6.5), 95 (11), 84 (90), 67 (14), 55 (46); Exact Mass Calcd for $C_{15}H_{13}F_6NO$: 337.0901, found 337.0895.

2-Trifluoromethyl-*N*-(3,3,5-trimethyl-2-(3*H*)-furanylidene)-benzenamine. Colorless oil; R_f 0.24 (hexane/ether = 20/1); ^1H NMR ($CDCl_3$) δ 1.39 (s, 6H, 3'-Me), 1.85 (d, J = 1.3 Hz, 3H, 5'-Me), 5.05 (d, J = 1.3 Hz, 1H,

4'-H), 7.01 (d, $J = 8.2$ Hz, 1H, 6-H), 7.12 (t, $J = 7.6$ Hz, 2H, 5-H), 7.44 (t, $J = 7.7$ Hz, 2H, 4-H), 7.60 (d, $J = 7.9$ Hz, 2H, 4-H); ^{13}C NMR (CDCl_3) δ 13.6 (5'-Me), 26.5 (3'-Me), 45.4 (3'-C), 110.7 (4'-C), 121.4 (q, $J = 29.8$ Hz, 2-C), 122.8 (6-C), 122.9 (4-C), 123.9 (q, $J = 273$ Hz, CF_3), 126.0 (q, $J = 5.2$ Hz, 3-C), 132.0 (5-C), 145.9 (1-C), 150.3 (5'-C), 169.3 (2'-C); IR (neat) 2974 m, 2932 w, 1729 s, 1683 m, 1607 m, 1581 w, 1491 m, 1457 m, 1383 w, 1260 s, 1221 m,

1156 s, 1129 s, 1110 s, 1054 m, 1033 m, 999 w, 952 s, 872 w, 851 w, 759 s, 732 w, 653 m, 597 w; MS, m/z (relative intensity, %) 269 (M^+ , 17), 254 (6.3), 234 (0.7), 226 (100), 210 (1.0), 186 (3.1), 172 (7.8), 152 (3.3), 145 (16), 125 (4.0), 109 (1.7), 95 (7.2), 83 (63), 74 (7.5), 55(42); Exact Mass Calcd for $\text{C}_{14}\text{H}_{14}\text{F}_3\text{NO}$: 269.1027, found 269.1025.

***N*-(3,3,5-Trimethyl-2-(3*H*)-furanylidene)-1,1-dimethylethylamine (2).** Colorless oil; R_f 0.20 (hexane/ether = 10/1); ^1H NMR (CDCl_3) δ 1.16 (s, 6H, 3-Me), 1.27 (s, 9H, $\text{C}(\text{CH}_3)_3$), 1.90 (d, $J = 1.3$ Hz, 3H, 5-Me), 4.87 (d, $J = 1.3$ Hz, 1H, 4-H); ^{13}C NMR (CDCl_3) δ 14.0 (5-Me), 27.4 (3-Me), 30.0 ($\text{C}(\text{CH}_3)_3$), 45.0 (3-C), 53.3 ($\text{C}(\text{CH}_3)_3$), 109.7 (4-C), 149.5 (5-C), 164.4 (2-C); IR (KBr) 3892 w, 3740 w, 3652 w, 3474 w, 3092 w, 2964 s, 2700 w, 2300 w, 2188 w, 2062 w, 1944 w, 1866 w, 1827 w, 1728 s, 1684 s, 1571 w, 1455 w, 1378 m, 1360 m, 1251 s, 1217 m, 1059 s, 945 s, 914 m, 837 w, 761 w; MS, m/z (relative intensity, %) 181 (M^+ , 6.9), 135 (15), 125 (9.0), 110 (44), 95 (10), 74 (25), 57 (100); Exact Mass Calcd for $\text{C}_{11}\text{H}_{19}\text{NO}$: 181.1467, found 181.1458.

2,6-Dimethyl-*N*-(3-methyl-3-(4-methyl-3-pentenyl)-2(3*H*)-furanylidene)-benzenamine (4). Colorless oil; R_f 0.17 (hexane/ether = 25/1); ^1H NMR (CDCl_3) δ 1.42 (s, 3H, 3'-Me), 1.60-1.73 (c, 7H, $\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 1.80-1.92 (c, 4H, 5'-Me, $\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 1.97-2.10 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 2.13 (s, 6H, 2-Me, 6-Me), 4.98 (d, $J = 1.6$ Hz, 1H, 4'-H), 5.12 (m, 1H, $\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 6.89 (dd, $J = 8.2$ Hz, $J = 6.6$ Hz, 1H, 4-H), 7.00 (d, $J = 7.2$ Hz, 2H, 3-H, 5-H); ^{13}C NMR (CDCl_3) δ 13.9 (5'-Me), 17.8 (Me_{cis}), 18.3 (2-Me), 24.2 ($\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 25.8 (Me_{trans}), 26.3 (3'-Me), 39.8 ($\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 49.2 (3'-C), 108.3 (4'-C), 122.8 (4-C), 123.8 ($\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 127.4 (3-C), 127.7 (2-C), 131.7 ($\text{CH}_2\text{CH}_2\text{CH}=\text{CMe}_2$), 144.6 (1-C), 150.8 (5'-C), 166.3 (2'-C); IR (KBr) 3018 m, 2966 s, 2924 s, 2856 s, 2730 w, 2532 w, 2004 w, 1906 w, 1729 s, 1681 s, 1596 m, 1473 s, 1444 s, 1383 s, 1307 m, 1259 s, 1219 s, 1200 s, 1134 m, 1092 s, 1065 s, 1032 m, 1016 m, 984 w, 947 s, 831 m, 761 s, 729 s, 692 w, 622 w, 527 w; MS, m/z (relative intensity, %) 297 (M^+ , 1.4), 282 (0.3), 254 (4.9), 226 (1.1),

215 (100), 198 (1.9), 184 (2.2), 172 (43), 144 (5.7), 123 (14), 117 (3.0), 105 (13), 77 (25), 55 (13); Anal. Calcd for $C_{20}H_{27}NO$: C, 80.76; H, 9.15; N, 4.71. Found: C, 80.61; H, 8.95; N, 4.66.

2,6-Dimethyl-N-(3,3-dimethyl-5-(4-trifluoromethylphenyl)-2-(3*H*)-furan-2-ylidene)-benzenamine (6). White solid; mp 113 °C (hexane); R_f 0.20 (hexane/ether = 10/1); ^1H NMR ($CDCl_3$) δ 1.57 (s, 6H, 3'-Me), 2.14 (s, 6H, 2-Me, 6-Me), 5.88 (s, 1H, 4'-H), 6.94 (dd, $J = 8.2$ Hz, $J = 6.6$ Hz, 1H, 4-H), 7.05 (d, $J = 7.6$ Hz, 2H, 3-H, 5-H), 7.51 (d, $J = 8.6$ Hz, 2H, 2''-H), 7.56 (d, $J = 8.6$ Hz, 2H, 3''-H); ^{13}C NMR ($CDCl_3$) δ 18.0 (2-Me), 27.0 (3'-Me), 46.2 (3'-C), 111.5 (4'-C), 122.9 (4-C), 123.7 (q, $J = 272$ Hz, CF_3), 125.0 (2''-C), 125.3 (q, $J = 3.9$ Hz, 3''-C), 127.5 (3-C), 127.5 (2-C), 130.6 (q, $J = 32.4$ Hz, 4''-C), 132.1 (1''-C), 144.1 (1-C), 149.8 (5'-C), 164.9 (2'-C); IR (KBr) 3542 w, 3436 w, 3076 m, 3022 w, 2962 m, 2868 w, 2628 w, 2540 w, 2464 w, 2380 w, 2282 w, 2178 w, 2092 w, 2012 w, 1920 w, 1893 w, 1727 s, 1618 w, 1594 m, 1519 w, 1465 m, 1444 m, 1414 m, 1378 m, 1324 s, 1291 s, 1275 m, 1258 m, 1244 m, 1203 m, 1166 s, 1155 s, 1122 s, 1108 s, 1090 s, 1061 s, 1022 m, 1005 s, 956 m, 919 s, 861 w, 842 s, 805 s, 766 s, 722 m, 694 w, 677 m, 627 m, 601 m, 590 m; MS, m/z (relative intensity, %) 359 (M^+ , 14), 340 (1.2), 328 (0.2), 300 (0.2), 276 (0.1), 262 (0.1), 247 (0.1), 227 (0.9), 213 (1.2), 197 (0.4), 186 (100), 173 (5.3), 159 (3.3), 144 (10), 130 (7.8), 105 (11), 77 (17), 55 (7.4); Anal. Calcd for $C_{21}H_{20}F_3NO$: C, 70.18; H, 5.61; N, 3.90. Found: C, 69.98; H, 5.65; N, 3.93.

2,6-Dimethyl-N-(3,3-dimethyl-5-(2-methyl-1-propenyl)-2-(3*H*)-furan-2-ylidene)-benzenamine (8). Colorless oil; R_f 0.19 (hexane/ether = 15/1); ^1H NMR ($CDCl_3$) δ 1.47 (s, 6H, 3'-Me), 1.71 (s, 3H, $CH=CHMe_2$), 1.78 (s, 3H, $CH=CHMe_2$), 2.11 (s, 6H, 2-Me, 6-Me), 5.14 (s, 1H, 4'-H), 5.57 (brs, 1H, $CH=CHMe_2$), 6.86 (dd, $J = 8.2$ Hz, $J = 6.6$ Hz, 1H, 4-H), 6.99 (d, $J = 7.2$ Hz, 2H, 3-H, 5-H); ^{13}C NMR ($CDCl_3$) δ 18.0 (2-Me), 20.1 ($CH=CHMe_2$), 27.1 (3'-Me), 27.2 ($CH=CHMe_2$), 44.6 (3'-C), 111.7 (4'-C), 113.3 ($CH=CHMe_2$), 122.6 (4-C), 127.3 (3-C), 127.5 (2-C), 140.9 ($CH=CHMe_2$), 144.7 (1-C), 150.4 (5'-C), 166.5 (2'-C); IR (KBr) 3434 w, 3018 m, 2968 s, 2930 s, 2862 m, 2734 w, 2034 w, 1910 w, 1845 w, 1728 s, 1670 m, 1596 m, 1466 s, 1377 m, 1357 m, 1309 s, 1271 s, 1246 m, 1222 w, 1202 s, 1184 w, 1146 w, 1093 s, 1051 s, 952 s, 919 m, 846 s, 761 s, 744 s, 722 m, 692 w, 633 w, 604 w; MS, m/z (relative intensity, %) 269 (M^+ , 19), 254 (1.6), 226 (1.2), 212 (0.6), 198 (1.4), 186 (100), 158 (2.1), 144 (13), 123 (23), 105 (14), 77 (19), 55 (28); Anal. Calcd for $C_{18}H_{23}NO$: C, 80.26; H, 8.61; N, 5.20. Found: C, 80.07; H, 8.68; N, 5.26.

2,6-Dimethyl-*N*-(5-methyl-3-butyl-2(5*H*)-furan-2-ylidene)-benzenamine (10). Colorless oil; R_f 0.11

(hexane/ether = 20/1); ^1H NMR (CDCl_3) δ 0.98 (t, $J = 7.2$ Hz, 3H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.32 (d, $J = 6.6$ Hz, 3H, 5'-Me), 1.37-1.52 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.61-1.74 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.12 (s, 6H, 2-Me, 6-Me), 2.44 (t, $J = 7.7$ Hz, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 5.02 (qd, $J = 6.6$ Hz, $J = 1.6$ Hz, 1H, 5'-H), 6.57 (d, $J = 1.3$ Hz, 1H, 4'-H), 6.88 (t, $J = 7.4$ Hz, 1H, 4-H), 7.01 (d, $J = 7.6$ Hz, 2H, 3-H, 5-H); ^{13}C NMR (CDCl_3) δ 14.0 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 18.3 (2-Me), 20.6 (5'-Me), 22.4 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 25.6 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 29.9 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 80.5 (5'-C), 122.6 (4-C), 127.3 (3-C), 127.9 (2-C), 136.1 (3'-C), 141.2 (4'-C), 145.4 (1-C), 161.0 (2'-C); IR (KBr) 3860 w, 3476 w, 3366w, 3022 w, 2958 m, 2932 m, 2862m, 2732w, 2628 w, 1996 w, 1689 s, 1595 m, 1471 m, 1375 w, 1322 m, 1254 w, 1206 m, 1110m, 1081 m, 1023 m, 999 w, 952 w, 918 w, 890 w, 761 m; MS, m/z (relative intensity, %) 257 (M^+ , 37), 242 (9.6), 228 (4.3), 214 (22), 200 (100), 186 (5.0), 172 (32), 148 (17), 147 (58), 132 (12), 105 (22), 79 (46), 67 (91), 53 (23); Exact Mass Calcd for $\text{C}_{17}\text{H}_{23}\text{NO}$: 257.1767, found 257.1767.

2,6-bis(trifluoromethyl)-*N*-(5-methyl-3-butyl-2(5*H*)-furan-2-ylidene)-benzenamine. Colorless oil; R_f 0.40

(hexane/ether = 10/1); ^1H NMR (CDCl_3) δ 0.95 (t, $J = 7.2$ Hz, 3H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.29 (d, $J = 6.6$ Hz, 3H, 5'-Me), 1.33-1.47 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.58-1.70 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.40 (t, $J = 7.7$ Hz, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 5.06 (qd, $J = 6.6$ Hz, $J = 1.6$ Hz, 1H, 5'-H), 6.65 (d, $J = 1.3$ Hz, 1H, 4'-H), 7.19 (t, $J = 7.9$ Hz, 1H, 4-H), 7.77 (d, $J = 7.9$ Hz, 2H, 3-H, 5-H); ^{13}C NMR (CDCl_3) δ 13.9 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 19.4 (5'-Me), 22.3 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 25.3 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 29.5 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 81.8 (5'-C), 121.9 (4-C), 122.7 (q, $J = 90.5$ Hz, 2-C), 123.4 (q, $J = 273$ Hz, CF_3), 129.8 (3-C), 135.9 (3'-C), 143.2 (4'-C), 146.4 (1-C), 164.2 (2'-C); IR (KBr) 3782 w, 3698 w, 3314 w, 2930 s, 2868 s, 2758 w, 2636 w, 2476 w, 2308 w, 2246 w, 2170 w, 2030 w, 1691 s, 1604 s, 1544 w, 1456 s, 1375 w, 1340 s, 1324 s, 1298 s, 1237 m, 1206 s, 1160 s, 1134 s, 1081 m, 1062 m, 1022 m, 953 w, 856 m, 831 m, 787 m, 758 s, 701 m, 674 s, 572 w, 524 w; MS, m/z (relative intensity, %) 365 (M^+ , 16), 336 (4.1), 323 (23), 296 (3.2), 268 (5.4), 254 (85), 236 (17), 213 (8.2), 188 (0.8), 158 (0.5), 121 (4.7), 109 (21), 95 (39), 81 (24), 67 (100), 53 (19); Exact Mass Calcd for $\text{C}_{17}\text{H}_{17}\text{F}_6\text{NO}$: 365.1214, found 365.1209.

2,6-Dimethyl-*N*-(5-methyl-3-(1,1-dimethylethyl)-2(5*H*)-furan-2-ylidene)-benzenamine (12). Colorless oil; bp

120 °C / 1 mmHg; ^1H NMR (CDCl_3) δ 1.28 (d, $J = 6.6$ Hz, 3H, 5'-Me), 1.39 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.11 (s, 6H, 2-Me, 6-Me), 4.92 (qd, $J = 6.6$ Hz, $J = 2.0$ Hz, 1H, 5'-H), 6.52 (d, $J = 2.0$ Hz, 1H, 4'-H), 6.86 (t, $J = 7.5$ Hz, 1H, 4-H), 7.01 (d, $J = 7.5$

Hz, 2H, 3-H, 5-H); ^{13}C NMR (CDCl_3) δ 18.4 (2-Me), 20.7 (5'-Me), 28.6 (CMe_3), 31.9 (CMe_3), 79.2 (5'-C), 122.3 (4-C), 127.3 (3-C), 127.6 (2-C), 140.0 (3'-C), 144.0 (4'-C), 146.0 (1-C), 159.0 (2'-C); IR (KBr) 3070w, 2962s, 2872m, 1681s, 1595m, 1472s, 1442m, 1392w, 1373m, 1364m, 1322m, 1258m, 1209m, 1167m, 1116s, 1088s, 1052m, 1030m, 1005s, 953m, 919w, 893w, 854w, 817w; MS, m/z (relative intensity, %) 257 (M^+ , 29), 242 (10), 201 (12), 200 (72), 172 (19), 171 (10), 158 (13), 148 (23), 147 (100), 132 (12), 121 (11), 120 (15), 109 (17), 107 (18), 105 (25), 103 (15), 95 (29), 93 (12), 91 (21), 81 (13), 79 (39), 78 (13), 77 (55), 67 (71), 65 (20), 55 (41); Anal. Calcd for $\text{C}_{17}\text{H}_{23}\text{NO}$: C, 79.33; H, 9.01; N, 5.44. Found: C, 79.04; H, 9.07; N, 5.55.

2,6-Dimethyl-N-(3,3-dimethyl-2-(3H)-furanylidene)-benzenamine (14). White solid; mp 97 °C; R_f 0.13 (hexane/ether = 20/1); ^1H NMR (CDCl_3) δ 1.48 (s, 6H, 3'-Me), 2.10 (s, 6H, 2-Me, 6-Me), 5.47 (d, $J = 3.3$ Hz, 1H, 4'-H), 6.52 (d, $J = 3.3$ Hz, 1H, 5'-H), 6.90 (dd, $J = 8.2$ Hz, $J = 6.6$ Hz, 1H, 4-H), 7.01 (d, $J = 7.2$ Hz, 2H, 3-H, 5-H); ^{13}C NMR (CDCl_3) δ 17.9 (2-Me), 26.8 (3'-Me), 43.5 (3'-C), 115.9 (4'-C), 123.0 (4-C), 127.43 (3-C), 127.47 (2-C), 140.9 (5'-C), 144.4 (1-C), 166.4 (2'-C); IR (KBr) 3114 m, 2970 m, 1713 s, 1623 w, 1593 w, 1466 m, 1385 w, 1259 m, 1242 m, 1199 s, 1123 s, 1087 s, 1009 s, 952 s, 906 s, 808 m, 788 s, 751 m, 720 s, 599 w; MS, m/z (relative intensity, %) 215 (M^+ , 38), 200 (1.4), 186 (100), 157 (2.6), 144 (25), 130 (22), 116 (12), 103 (20), 79 (23), 77 (37), 55 (15); Anal. Calcd for $\text{C}_{14}\text{H}_{17}\text{NO}$: C, 78.10; H, 7.96; N, 6.51. Found: C, 77.94; H, 7.91; N, 6.42.

(R)-2,6-Dimethyl-N-(4,5,6,7-tetrahydro-3,3,6-trimethyl-2(3H)-benzofuranylidene)-benzenamine (16). Colorless oil; bp 150 °C / 1 mmHg; ^1H NMR (CDCl_3) δ 1.01 (d, $J = 6.3$ Hz, 3H, 6'-Me), 1.29-1.34 (m, 1H, 5'-H), 1.40 (s, 6H, 3'-Me), 1.68-1.79 (c, 3H, 6'-H, 4'-H or 5'-H or 7'-H), 2.06-2.21 (c, 9H, 2-Me, 6-Me, 4'-H, 5'-H, 7'-H), 6.89 (dd, $J = 8.2$ Hz, $J = 6.6$ Hz, 1H, 4-H), 7.01 (d, $J = 7.6$ Hz, 2H, 3-H, 5-H); ^{13}C NMR (CDCl_3) δ 18.0 (2-Me), 18.9 (4'-C), 21.4 (6'-Me), 25.1 (3'-Me), 25.2 (3'-Me), 29.4 (6'-C), 30.4 (7'-C), 30.6 (5'-C), 45.1 (3'-C), 118.2 (3'a-C), 122.7 (4-C), 127.3 (3-C), 127.6 (2-C), 144.6 (1-C), 146.9 (7'a-C), 167.7 (2'-C); IR (KBr) 2968 s, 2924 s, 2856 m, 1738 s, 1708 s, 1598 w, 1466 m, 1444 m, 1370 m, 1314 m, 1272 m, 1194 m, 1152 w, 1134 m, 1084 s, 1048 w, 1010 s, 994 m, 952 m, 934 m, 896 w, 878 w, 816 w; MS, m/z (relative intensity, %) 283 (M^+ , 45), 268 (17), 241 (12), 240 (62), 152 (17), 150 (20), 145 (11), 144 (15), 137 (23), 132 (18), 130 (11), 124 (19), 110 (12), 109 (26), 105 (17), 103 (17), 95 (17), 91 (15), 82 (20), 81 (100), 80 (20), 79 (39), 78 (12), 77 (47), 69 (15), 68 (14), 67 (63), 65

(15), 55 (27), 53 (29), 51 (13); Anal. Calcd for $C_{19}H_{25}NO$: C, 80.52; H, 8.89; N, 4.92. Found: C, 80.52; H, 9.15; N, 4.95.

2,6-Dimethyl-*N*-(5,6,7,7a-tetrahydro-3-methyl-2(4*H*)-benzofuranylidene)-benzenamine (18). White solid; mp 76 °C; R_f 0.21 (hexane/ether = 5/1); ^1H NMR ($CDCl_3$) δ 1.05-1.50 (c, 3H, 4'-H or 5'-H or 6'-H or 7'-H), 1.80-2.00 (c, 5H, 3'-Me, 4'-H or 5'-H or 6'-H or 7'-H), 2.03-2.19 (c, 7H, 2-Me, 6-Me, 4'-H or 5'-H or 6'-H or 7'-H), 2.29-2.42 (m, 1H, 7'-H), 2.82 (dt, $J = 13.8$ Hz, $J = 2.0$ Hz, 1H, 4'-H), 4.60 (dd, $J = 10.9$ Hz, $J = 5.9$ Hz, 1H, 7'-a-H), 6.87 (dd, $J = 8.1$ Hz, $J = 6.8$ Hz, 1H, 4-H), 7.01 (d, $J = 7.2$ Hz, 2H, 3-H); ^{13}C NMR ($CDCl_3$) δ 8.9 (3'-Me), 18.3 (2-Me), 23.1 (6'-C), 25.9 (4'-C), 26.3 (5'-C), 35.0 (7'-C), 83.5 (7'-a-C), 121.1 (3'-C), 122.4 (4-C), 127.3 (3-C), 128.1 (2-C), 145.7 (1-C), 153.7 (3'-C), 162.2 (2'-C); IR (KBr) 2932 m, 2850 w, 1693 s, 1679 s, 1592 m, 1470 m, 1447 m, 1389 w, 1374 w, 1355 w, 1317 w, 1251 w, 1237 w, 1222 w, 1205 w, 1143 m, 1099 m, 1053 w, 1030 s, 941 w, 916 w, 881 w, 844 w, 825 w, 814 w, 773 w, 755 s, 734 w, 719 w, 691 w, 630 w, 597 w, 543 w; MS, m/z (relative intensity, %) 253 (M^+ , 82), 238 (38), 225 (29), 210 (46), 196 (17), 182 (13), 168 (6.1), 158 (18), 144 (12), 131 (25), 107 (12), 103 (25), 91 (33), 79 (73), 77 (100), 53 (32); Anal. Calcd for $C_{17}H_{21}NO$: C, 79.96; H, 8.29; N, 5.49. Found: C, 79.68; H, 8.19; N, 5.38.

2,6-Dimethyl-*N*-(3,3-dimethyl-3,4-dihydro-2*H*-indeno[1,2-*b*]furan-2-ylidene)-benzenamine (20a). Colorless oil; R_f 0.14 (hexane/ether = 20/1); ^1H NMR ($CDCl_3$) δ 1.61 (s, 6H, 3'-Me), 2.15 (s, 6H, 2-Me, 6-Me), 3.31 (s, 2H, 4'-H), 6.95 (ap. dd, $J = 8.4$ Hz, $J = 6.4$ Hz, 1H, 4-H), 7.05 (ap. d, $J = 7.2$ Hz, 2H, 3-H, 5-H), 7.16-7.31 (c, 3H, 6'-H, 7'-H, 8'-H), 7.44 (d, $J = 7.2$ Hz, 1H, 5'-H); ^{13}C NMR ($CDCl_3$) δ 18.0 (2-Me), 26.7 (3'-Me), 29.9 (4'-C), 45.0 (3'-C), 117.8 (8'-C), 123.0 (4-C), 124.8 (5'-C), 125.2 (7'-C), 126.6 (6'-C), 127.5 (2-C), 129.0 (3'-a-C), 133.1 (4'-a-C), 143.8 (1-C), 144.7 (8'-a-C), 155.4 (8'-b-C), 169.5 (2-C); IR (KBr) 3920 w, 3796 w, 3616 w, 3334 w, 3198 w, 3020 w, 2964 m, 2814 w, 2786 w, 2674 w, 2612 w, 2569 w, 2562 w, 2352 w, 2278 w, 2006 w, 1959 w, 1895 w, 1737 s, 1650 w, 1609 w, 1594 w, 1557 w, 1515 w, 1470 m, 1456 m, 1390 s, 1378 m, 1356 w, 1344 w, 1301 w, 1269 w, 1241 w, 1205 w, 1193 w, 1165 w, 1127 m, 1082 w, 1028 s, 1010 m, 974 w, 951 m, 862 m, 813 w, 756 m, 717 w; MS, m/z (relative intensity, %) 303 (M^+ , 77), 288 (48), 172 (86), 157 (52), 144 (14), 129 (100), 115 (25), 103 (42), 79 (26), 77 (67), 51 (37); Exact Mass Calcd for $C_{21}H_{21}NO$ 303.1623, found 303.1628.

2-Trifluoromethyl-*N*-(3,3-dimethyl-3,4-dihydro-2*H*-indeno[1,2-*b*]furan-2-ylidene)-benzenamine (20b).

Colorless oil; R_f 0.18 (hexane/ether = 25/1); ^1H NMR (CDCl_3) δ 1.57 (s, 6H, 3'-Me), 3.32 (s, 2H, 4'-H), 7.13 (d, J = 7.9 Hz, 1H, 6-H), 7.16-7.29 (c, 4H, 5-H, 6'-H, 7'-H, 8'-H), 7.45 (d, J = 6.6 Hz, 1H, 5'-H), 7.52 (t, J = 7.9 Hz, 1H, 4-H), 7.66 (d, J = 7.9 Hz, 1H, 3-H); ^{13}C NMR (CDCl_3) δ 26.3 (3'-Me), 29.8 (4'-C), 45.3 (3'-C), 117.6 (8'-C), 121.4 (q, J = 30.0 Hz, 2-C), 122.6 (6-C), 123.3 (5-C), 123.9 (q, J = 276 Hz, CF_3), 124.9 (5'-C), 125.3 (7'-C), 126.1 (q, J = 5.0 Hz, 3-C), 126.5 (6'-C), 129.2 (3'-a-C), 132.2 (4-C), 132.9 (4'-a-C), 144.7 (8'-a-C, 1-C), 155.3 (8'-b-C), 171.8 (2-C); IR (neat) 3866 w, 3516 w, 3432 w, 3258 w, 3050 m, 2968 s, 2930 s, 2790 w, 2544 w, 2438 w, 2270 w, 2040 w, 1916 w, 1726 s, 1654 m, 1604 s, 1579 s, 1489 s, 1454 s, 1489 s, 1454 s, 1389 s, 1315 s, 1259 s, 1222 m, 1194 m, 1162 s, 1120 s, 1084 m, 1058 s, 1030 s, 1009 s, 955 s, 906 w, 877 s, 863 m, 813 s, 753 s, 710 s, 674 m, 655 m, 640 s, 598 s, 561 w, 551 w, 513 m; MS, m/z (relative intensity, %) 343 (M^+ , 15), 328 (20), 308 (9.0), 234 (3.3), 198 (5.2), 184 (3.3), 172 (100), 157 (67), 145 (22), 129 (91), 102 (16), 77 (20), 51 (30); Exact Mass Calcd for $\text{C}_{20}\text{H}_{16}\text{F}_3\text{NO}$ 343.1184, found 343.1185.

***N,N'*-(4,4-dimethyl-indeno[1,2-*b*]pyran-2,3-diylidene)bis(2,6-dimethylbenzenamine) (21a).**

Yellow solid; mp 148 °C (hexane); R_f 0.22 (hexane/ether = 10/1); ^1H NMR (CDCl_3) δ 1.67 (s, 6H, 4''-Me), 1.73 (s, 6H, 2-Me, 6-H), 2.03 (s, 6H, 2'-Me, 6'-H), 3.45 (s, 2H, 5''-H), 6.77-6.96 (c, 6H, 3-H, 4-H, 5-H, 3'-H, 4'-H, 5'-H), 7.02-7.09 (m, 1H, 9''-H), 7.18-7.26 (c, 2H, 7''-H, 8''-H), 7.40-7.47 (m, 1H, 6''-H); ^{13}C NMR (CDCl_3) δ 18.0 (2-Me), 18.1 (2'-Me), 26.0 (4''-Me), 32.9 (5''-C), 41.9 (4''-C), 117.4 (9''-C), 122.5 (4-C), 122.6 (4''-a-C), 123.2 (4'-C), 124.1 (6''-C), 124.4 (2-C), 125.6 (8''-C), 126.4 (7''-C), 126.8 (2'-C), 127.2 (3-C), 127.4 (3'-C), 136.6 (5''-a-C), 140.9 (9''-a-C), 141.5 (9''-b-C), 143.2 (1-C), 145.6 (2''-C), 148.4 (1'-C), 161.3 (3''-C); IR (KBr) 3916 w, 3640 w, 3320 w, 3022 w, 2962 m, 2920 m, 2858 m, 2732 w, 2324 w, 2194 w, 1947 w, 1905 w, 1868 w, 1842 w, 1797 w, 1773 w, 1665 s, 1612 w, 1593 m, 1534 w, 1468 s, 1440 m, 1393 s, 1384 s, 1376 s, 1326 m, 1302 w, 1251 s, 1211 s, 1170 s, 1122 s, 1080 s, 1025 m, 1013 m, 982 w, 939 w, 918 w, 860 w, 834 m, 820 w, 758 s, 718 s, 661 m; MS, m/z (relative intensity, %) 434 (M^+ , 1.1), 419 (36), 405 (0.3), 303 (100), 288 (45), 286 (4.8), 244 (2.3), 217 (2.1), 198 (16), 184 (1.8), 172 (56), 157 (27), 143 (6.9), 129 (50), 103 (20), 79 (16), 77 (33), 51 (7.5); Anal. Calcd for $\text{C}_{30}\text{H}_{30}\text{N}_2\text{O}$: C, 82.91; H, 6.96; N, 6.45. Found: C, 82.82; H, 7.13; N, 6.31.

***N,N'*-(4,4-dimethyl-indeno[1,2-b]pyran-2,3-diylidene)bis(2,6-bis(1-methylethyl)benzenamine) (21c).**

Yellow solid; mp 158 °C (hexane); R_f 0.47 (hexane/ether = 10/1); ^1H NMR (CDCl_3) δ 0.88 (d, J = 6.6 Hz, 6H, 2-CH(CH_3)₂), 0.98 (d, J = 6.9 Hz, 6H, 2-CH(CH_3)₂), 1.13 (dd, J = 6.6 Hz, J = 1.3 Hz, 12H, 2'-CH(CH_3)₂), 1.66 (s, 6H, 4''-Me), 2.29 (sept, J = 6.9 Hz, 2H, 2-CH(CH_3)₂), 2.77 (sept J = 6.7 Hz, 2H, 2'-CH(CH_3)₂), 3.45 (s, 2H, 5''-H), 6.91-7.07 (c, 7H, 9''-H, 3-H, 4-H, 5-H, 3'-H, 4'-H, 5'-H), 7.15-7.25 (c, 2H, 7''-H, 8''-H), 7.41-7.45 (m, 1H, 6''-H); ^{13}C NMR (CDCl_3) δ 21.9 (2'-CH(CH_3)₂), 23.1 (2'-CH(CH_3)₂), 23.6 (2-CH(CH_3)₂), 24.2 (2-CH(CH_3)₂), 26.0 (4''-C), 28.1 (2-CH(CH_3)₂), 28.7 (2'-CH(CH_3)₂), 33.0 (5''-C), 42.2 (4''-C), 117.4 (9''-C), 121.9 (3'-C), 122.4 (3-C), 122.5 (4''a-C), 123.0 (4-C or 4'-C), 123.7 (4-C or 4'-C), 124.0 (6''-C), 125.4 (8''-C), 126.4 (7''-C), 134.5 (2'-C), 136.6 (5''-C), 136.9 (2-C), 140.2 (1-C), 140.9 (9''a-C), 141.5 (9''b-C), 145.6 (2''-C), 145.7 (1'-C), 159.1 (3''-C); IR (KBr) 3832 w, 3730 w, 3668 w, 3612 w, 3374 w, 3316 w, 3184 w, 3054 w, 2962 s, 2866 m, 2592 w, 2426 w, 2026 w, 1936 w, 1908 w, 1852 w, 1793 w, 1673 s, 1663 s, 1614 w, 1586 w, 1460 s, 1434 m, 1398 m, 1376 s, 1359 m, 1326 m, 1301 w, 1251 s, 1206 s, 1182 w, 1172 s, 1157 s, 1121 s, 1094 m, 1080 m, 1055 w, 1039 w, 1013 w, 953 m, 936 m, 870 w, 859 w, 834 w, 807 w, 788 w, 764 s, 754 s, 746 s, 718 m, 669 w, 634 w; MS, m/z (relative intensity, %) 546 (2.8), 503 (77), 374 (2.2), 359 (39), 344 (14), 316 (100), 288 (4.7), 244 (3.7), 201 (6.3), 172 (23), 157 (8.6), 129 (4.9); Anal. Calcd for $\text{C}_{38}\text{H}_{46}\text{N}_2\text{O}$: C, 83.47; H, 8.48; N, 5.12. Found: C, 83.38; H, 8.46; N, 5.10.

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MENUF
 OBNUC
 OFR
 OBSET
 OBFIN
 PW1
 DEADT
 PREDL
 IMT
 POINT
 SPO
 TIMES
 DUMMY
 FREQU
 FLT
 DELAY
 ACQTM
 PD
 ADBIT
 RGAIN
 BF
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 LKSIG
 CSPED
 FILDC
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1H
 270.05 MHz
 112.00 KHz
 5800.0 Hz
 5.3 us
 108.5 us
 0.2000 ms
 1.0 sec
 16384
 16384
 16384
 8
 1
 5402.4 Hz
 2700 Hz
 74.1 us
 3.033 sec
 3.967 sec
 16.00
 21
 0.12 Hz
 0.00
 0.00
 90.00
 100.00
 NON
 EXMOD
 NON:Single coupled:PW1_ACQTM_PD:1H,1
 1H
 270.05 MHz
 112.00 KHz
 5800.0 Hz
 50 us
 511
 THSAT
 64.60 KHz
 59.0 Hz
 200
 22
 79
 872
 17 Hz

BCM
13C
67.80 MHz
135.00 KHz
5200.0 Hz
3.9 us
30.8 us
0.2000 ms
1.0 sec
32768
32768
10000
1
18315.0 Hz
9150 Hz
21.9 us
1.789 sec
1.211 sec
16.00
27
1.20 Hz
0.00
0.00
90.00
100.00
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDG
FILDG
FILDG

BCM
Bilevel,complete,decoupling:Set_IRRPW
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
THSAT
64.60 KHz
59.0 Hz
200
22
79
863
17 Hz

M:\YG\mgf\N-CF3phenyliminolactone\N-CF3phenyliminolactone H,als
N-CF3phenyliminolactone

MENUF
OBNUC
OFR
OBSET
OBFIN
PW1
DEADT
PREDL
IWT
POINT
SPO
TIMES
DUMMY
FREQU
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC
FILDf

NON
1H
270.05 MHz
112.00 KHz
5800.0 Hz
5.3 us
108.5 us
0.2000 ms
1.0 sec
16384
16384
8
1
5402.4 Hz
2700 Hz
74.1 us
3.033 sec
3.967 sec
16.00
21
0.12 Hz
0.00
0.00
90.00
100.00
NON
NON:Single-coupled:PW1_ACQTM_PD:1H,13C,17O,2H-
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
M:\YG\mgf\N-CF3phenyliminolactone\N-CF3phenyliminolactone
THSAT
64.60 KHz
59.0 Hz
200
22
96
883
13 Hz

M:\Jgm\N-CF3phenyliminolactone\N-CF3phenyliminolactone 13C.als
N-CF3phenyliminolactone

MENUF	BCM
OBNUC	13C
OFR	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.0 Hz
PW1	3.9 us
DEADT	30.8 us
PREDL	0.2000 ms
IWT	1.0 sec
POINT	32768
SPO	32768
TIMES	10000
DUMMY	1
FREQU	18315.0 Hz
FLT	9150 Hz
DELAY	21.9 us
ACQTM	1.789 sec
PD	1.211 sec
ADBIT	16.00
RGAIN	28
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	BiLevel, complete decoupling; Set_IRRPW
IRNUC	1H
IFR	270.05 MHz
IRSET	112.00 KHz
IRFIN	5800.0 Hz
IRRPW	50 us
IRATN	511
DFILE	M:\Jgm\N-CF3phenyliminolactone\N-CF3phenyliminolactone 13C.als
SF	THSAT
LKSET	64.60 KHz
LKFIN	59.0 Hz
LKLEV	200
LGAIN	22
LKPHS	96
LKSIG	877
CSPED	16 Hz
FILDG	
FILDF	

MOR-874

N-Propionylacetone H

MENUF
 OBNUC
 OFR
 OBSET
 OBFIN
 PW1
 DEADT
 PREDL
 IWT
 POINT
 SPO
 TIMES
 DUMMY
 FREQU
 FLT
 DELAY
 ACQTM
 PD
 ADBIT
 RGAIN
 BF
 T1
 T2
 T3
 T4
 EXMOD
 EXPCM
 IRNUC
 IFR
 IRSET
 IRFIN
 IRRPW
 IRATN
 DFILE
 SF
 LKSET
 LKFIN
 LKLEV
 LGAIN
 LKPHS
 LKSIG
 CSPED
 FILDG
 FILDG

NON
 1H
 270.05 MHz
 112.00 KHz
 5800.0 Hz
 5.3 us
 108.5 us
 0.2000 ms
 1.0 sec
 16384
 16384
 8
 1
 5402.4 Hz
 2700 Hz
 74.1 us
 3.033 sec
 3.967 sec
 16.00
 13
 0.12 Hz
 0.00
 0.00
 90.00
 100.00
 NON
 EXMOD
 NON:Single-coupled:PW1_ACQTM_PD:1H,1
 1H
 270.05 MHz
 112.00 KHz
 5800.0 Hz
 50 us
 511
 C:\WINNMR98\COMMON_DEFAULT.ALS
 THSAT
 64.60 KHz
 59.0 Hz
 200
 20
 87
 890
 19 Hz

MENUF BCM
OBNUC 13C
OFR 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.0 Hz
PW1 3.9 us
DEADT 30.8 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 32768
SPO 32768
TIMES 10000
DUMMY 1
FREQU 18315.0 Hz
FLT 9150 Hz
DELAY 21.9 us
ACQTM 1.789 sec
PD 1.211 sec
ADBIT 16.00
RGAIN 27
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel,complete,decoupling,Set_IRAPW
IRNUC 1H
IFR 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRAPW 50 us
IRATN 511
DPLE SF
LKSET 64.60 KHz
LKEIN 59.0 Hz
LKLEY 200
LGAIN 20
LKPHS 87
LKSIG 890
CSPED 16 Hz
FILDC
FILDF

BCM
13C
67.80 MHz
135.00 KHz
5200.0 Hz
3.9 us
30.8 us
0.2000 ms
1.0 sec
32768
32768
10000
1
18315.0 Hz
9150 Hz
21.9 us
1.789 sec
1.211 sec
16.00
27
1.20 Hz
0.00
0.00
90.00
100.00
BCM
Bilevel,complete,decoupling,Set_IRAPW
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
C:\WINNM\F98\COMMON_DEFAULT.ALS
TH5AT
64.60 KHz
59.0 Hz
200
20
87
890
16 Hz

DFILE C:\WINNMR98\COMMON_DEFAULT.ALS
 COMNT mor559ppcf6 H
 DATIM Tue Jun 11 14:22:54 2002
 1H
 OBNUC NON
 EXMOD 270.05 MHz
 OBFRQ 112.00 KHz
 OBSET 5800.0 Hz
 OBFIN 16384
 POINT 5402.4 Hz
 FREQU 8
 SCANS 3.033 sec
 ACQTM 3.967 sec
 PD 5.3 us
 PW1 1H
 IRNUC 24.5 c
 CTEMP CDCL3
 SLVNT 7.26 ppm
 EXREF 0.12 Hz
 BF 17
 RGAIN

M:\YGf\m\butylmonosubimindolactone\butylmonosubimindolactone 13C.als
butylmonosubimindolactone 13C

MENUF
 OBNUC
 OFR
 OBSET
 OBFIN
 PW1
 DEADT
 PREDL
 IWT
 POINT
 SPO
 TIMES
 DUMMY
 FREQU
 FLT
 DELAY
 ACQTM
 PD
 ADBIT
 RGAIN
 BF
 T1
 T2
 T3
 T4
 EXMOD
 EXPCM
 IRNUC
 IFR
 IRSET
 IREFIN
 IRRPW
 IRATN
 DFILE
 SF
 LKSET
 LKFIN
 LKLEV
 LGAIN
 LKPHS
 LKSIG
 CSPED
 FILDC
 FILDF

BCM
 13C
 67.80 MHz
 135.00 KHz
 5200.0 Hz
 3.9 us
 30.8 us
 0.2000 ms
 1.0 sec
 32768
 32768
 10000
 1
 18315.0 Hz
 9150 Hz
 21.9 us
 1.789 sec
 1.211 sec
 16.00
 28
 1.20 Hz
 0.00
 0.00
 90.00
 100.00
 BCM
 Bilvel,complete,decoupling,Set_IRRPW
 1H
 270.05 MHz
 112.00 KHz
 5800.0 Hz
 50 us
 511
 M:\YGf\m\butylmonosubimindolactone\butylmonosubim
 THSAT
 64.60 KHz
 59.0 Hz
 200
 22
 101
 457
 15 Hz

MENUF
OBNUC
OFR
OBSF
OBSF
OBSF
PW1
DEADT
PREDL
WVT
POINT
SPO
TIMES
DUMMY
FREQU
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC

NON
1H
270.05 MHz
112.00 KHz
5800.0 Hz
5.3 us
108.5 us
0.2000 ms
1.0 sec
16384
16384
8
1
5402.4 Hz
2700 Hz
74.1 us
3.033 sec
3.967 sec
16.00
23
0.12 Hz
0.00
0.00
90.00
100.00
NON
NON:Single coupled:PW1_ACQTM_PD:1H:1
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
THSAT
64.60 KHz
59.0 Hz
200
22
91
924
19 Hz

MENUE	BCM
OBNUC	13C
OFI	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.0 Hz
PW1	3.9 us
DEADT	30.8 us
PREDL	0.2000 ms
IWT	1.0 sec
POINT	32768
SPO	32768
TIMES	10000
DUMMY	1
FREQU	18315.0 Hz
FLT	9150 Hz
DELAY	21.9 us
ACQTM	1.789 sec
PD	1.211 sec
ADBIT	16.00
RGAIN	27
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	BiLevel,complete,decoupling;Set_IRRPW
IRNUC	1H
IFR	270.05 MHz
IRSET	112.00 KHz
IRFIN	5800.0 Hz
IRRPW	50 us
IRATN	511
DFILE	THSAT
SF	64.60 KHz
LKSET	59.0 Hz
LKFIN	200
LKLEV	22
LGAIN	91
LKPHS	815
LKSIG	16 Hz
CSPED	
FILDC	
FILDF	

NON
1H
270.05 MHz
112.00 KHz
5800.0 Hz
5.3 us
108.5 us
0.2000 ms
1.0 sec
16384
16384
16384
8
1
5402.4 Hz
2700 Hz
74.1 us
3.033 sec
3.967 sec
16.00
17
0.12 Hz
0.00
0.00
90.00
100.00
NON
EXMOD
NON:Single-coupled:PW1_ACQTM_PD:1H,1
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
C:\WINNMR98\COMMON_DEFAULT.ALS
TH5AT
64.60 KHz
59.0 Hz
200
22
76
923
18 Hz

MENUF
OBNUC
OFR
OBSET
OBFIN
PW1
DEADT
PREDL
IWT
POINT
SPO
TIMES
DUMMY
FREQU
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC
FILDF

BCM
13C
67.80 MHz
135.00 KHz
5200.0 Hz
3.9 us
30.8 us
0.200 ms
1.0 sec
32768
32768
10000
1
18315.0 Hz
9150 Hz
21.9 us
1.789 sec
1.211 sec
16.00
27
1.20 Hz
0.00
0.00
90.00
100.00
BCM
Bilevel complete decoupling: Set_IRRPW
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
THSAT
64.60 KHz
59.0 Hz
180
20
76
865
16 Hz

MENUF
OBNUC
OFR
OBSSET
OBFIN
PW1
DEADT
PREDL
IWT
POINT
SPO
TIMES
DUMMY
FREQU
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC
FILDF

NON
1H
270.05 MHz
112.00 KHz
5800.0 Hz
5.3 us
108.5 us
0.2000 ms
1.0 sec
16384
16384
8
1
5405.4 Hz
2700 Hz
74.1 us
3.031 sec
3.967 sec
16.00
12
0.12 Hz
0.00
0.00
90.00
100.00
NON
NON:Single coupled:PW1_ACQTM_PD:1H:1
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
M:\Gfmf\isopropyliden iminolactone CF3Y
TH5AT
64.60 KHz
59.0 Hz
200
22
79
774
16 Hz

20b

MENUF BCM
OBNUC 13C
OFR 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.0 Hz
PW1 3.9 us
DEADT 30.8 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 32768
SPO 32768
TIMES 10000
DUMMY 1
FREQU 18315.0 Hz
FLT 9150 Hz
DELAY 21.9 us
ACQTM 1.789 sec
PD 1.211 sec
ADBIT 16.00
RGAIN 27
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel,complete decoupling;Set_IRRPW
IRNUC 1H
IFR 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRRPW 50 us
IRATN 511
DFILE THSAT
SF 64.60 KHz
LKSET 59.0 Hz
LKFIN 200
LKLEV 23
LGAIN 79
LKPHS 762
LKSIG 16 Hz
CSPED

C:\WINNMR98\COMMON_DEFAULT.ALS
THSAT
64.60 KHz
59.0 Hz
200
23
79
762
16 Hz