Supporting Information

Ruthenium-Catalyzed Reaction of α,β-Unsaturated Imines with Carbon Monoxide and Alkenes Leading to β,γ-Unsaturated γ-Butyrolactams: Involvement of Direct Carbonylation at Olefinic C-H Bonds as a Key Step

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General Information. ^{1}H NMR and ^{13}C NMR were recorded on a JEOL JMN-270 spectrometer in $CDCl_3$ 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, quint = quintet, and m = multiplet), 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 SiO_2 (Merck Silica Gel 60 (230-400 mesh)).

Materials. Toluene was distilled from CaH_2 prior to use. $Ru_3(CO)_{12}$ was prepared according to the literature procedure[1] and used after recrystallization from hexane. Aldimines **1**, **5**, **7**, **9**, **11**, and **24** were prepared by the treatment of the corresponding aldehydes with *tert*-butylamine in the presence of $MgSO_4$ [2] and used after distillation. Cyclic ketimine **27** was prepared by the reaction of methyl 1-cyclohexene-1-carboxylate with 1-vinyl-2-pyrrolidinone using a modification of the method of Cosford. [3]

2-Methyl-*N*-[3-(4-methoxyphenyl)-2-propenylidene]-2-propanamine (5).[4] yellow oil; ^{1}H NMR ($CDCl_3$) δ 1.25 (s, 9H), 3.79 (s, 3H), 6.79-6.92 (m, 4H), 7.40 (d, *J* = 7.0 Hz, 2H), 8.00 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 29.7, 55.2, 56.9, 114.0, 127.0, 128.3, 128.5, 140.4, 157.2, 160.0; MS, *m*/*z*

(relative intensity, %) 217 (M^+, 20), 216 (28), 202 (48), 161 (32), 160 (100), 146 (33), 145 (87), 103 (18), 77 (22), 57 (73).

2-Methyl-*N*-[3-(4-trifluoromethylphenyl)-2-propenylidene]-2-propanamine (7). Pale yellow oil; bp 120-125 °C/0.1 mmHg; ^{1}H NMR ($CDCl_3$) δ 1.27 (s, 9H), 6.97 (s, 1H), 7.00 (s, 1H), 7.55 (d, *J* = 15.1 Hz, 2H), 7.61 (d, *J* = 15.1 Hz, 2H), 8.05 (dd, *J* = 1.6 Hz, *J* = 6.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 29.6, 57.4, 124.3 (q, *J* = 270.0 Hz, CF_3), 125.5 (complex, Ar), 127.0, 130.2 (q, *J* = 32.2 Hz, Ar), 131.5, 138.8, 139.2, 156.5; IR (KBr) 2968 s, 1922 w, 1690 w, 1640 m, 1622 s, 1478 m, 1418 m, 1326 s, 1212 s, 1166 s, 1126 s, 1066 s, 1014 m, 978 s, 954 w, 904 w, 866 m, 820 m, 766 w, 732 w, 652 w; MS, *m*/*z* (relative intensity, %) 255 (M^+, 3), 240 (35), 198 (16), 138 (17), 57 (100); exact mass calcd for $C_{14}H_{16}F_3N$ 255.1235, found 255.1231.

Typical Procedure. A 50-mL stainless autoclave was charged with *N*-*tert*-butyl-*trans*-cinnamaldimine (**1**) (1 mmol, 187 mg), toluene (2 mL), and $Ru_3(CO)_{12}$ (0.02 mmol, 13 mg). After the system was flushed with 10 atmospheres of ethylene three times, it was pressurized with ethylene to 10 atm and then with carbon monoxide to an additional 10 atm. The autoclave was immersed in an oil bath at 160 °C. After 20 hours had elapsed, the autoclave was removed from the oil bath and allowed to cool for 1 h. The gases were then released. The contents were transferred to a round-bottomed flask with ether and the volatiles removed in vacuo. The residue was subjected to column chromatography on silica gel (eluent; hexane/EtOAc = 4/1) to give 2,3-dihydro-1-(1,1-dimethylethyl)-3-ethyl-3-phenyl-pyrrol-2-one (**2**) (180 mg, 74 % yield) as a clear oil.

2,3-Dihydro-1-(1,1-dimethylethyl)-3-ethyl-3-phenyl-pyrrol-2-one (2).[5] Clear oil; R_f 0.61 (hexane/EtOAc = 2/1); ^{1}H NMR ($CDCl_3$) δ 0.81 (t, *J* = 7.3 Hz, 3H), 1.43 (s, 9H), 1.88-2.09 (m, 2H),

5.50 (d, *J* = 6.1 Hz, 1H), 6.72 (d, *J* = 6.1 Hz, 1H), 7.16-7.31 (m, 3H), 7.42-7.46 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 9.1, 28.1, 31.3, 54.3, 58.9, 111.8, 126.3, 126.5, 128.1, 130.1, 140.3, 179.7; MS, *m*/*z* (relative intensity, %) 243 (M^+, 16), 187 (31), 186 (11), 172 (18), 159 (16), 158 (100), 103 (18), 57 (46)

2,3-Dihydro-1-(1,1-dimethylethyl)-3-phenyl-3-(2-trimethylsilylethyl)-pyrrol-2-one (3). Yellow solid; mp 48-49 °C (hexane); R_f 0.73 (hexane/EtOAc = 3/1); ^{1}H NMR ($CDCl_3$) δ 0.00 (s, 9H), 0.37-0.46 (m, 2H), 1.48 (s, 9H), 1.92-1.98 (m, 2H), 5.57 (d, *J* = 5.4 Hz, 1H), 6.77 (d, *J* = 5.4 Hz, 1H), 7.21-7.36 (m, 3H), 7.48-7.52 (m, 2H); ^{13}C NMR ($CDCl_3$) δ -1.8, 11.1, 28.3, 33.4, 54.3, 60.0, 111.9, 126.5, 126.6, 128.2, 130.2, 140.3, 179.8; IR (KBr) 3124 m, 2960 s, 1690 s, 1612 m, 1498 w, 1450 m, 1386 w, 1368 m, 1308 m, 1284 s, 1250 s, 1214 m, 1178 m, 1088 w, 1034 w, 894 m, 864 s, 834 s, 750 m, 732 w, 696 s; MS, *m*/*z* (relative intensity, %) 315 (M^+, 0.1), 287 (9), 232 (5), 231 (31), 158 (5), 103 (8), 75 (6), 74 (9), 73 (100), 59 (7), 58 (6), 56 (26). Anal. Calcd for $C_{19}H_{29}NOSi$: C, 72.32; H, 9.26; N, 4.44. Found: C, 72.19; H, 9.10; N, 4.45.

2,3-Dihydro-3-(bicyclo[2,2,1]hept-2-yl)- 1-(1,1-dimethylethyl)-3-phenyl-pyrrol-2-one (4). The ^{1}H and ^{13}C NMR spectra and IR data were obtained as a mixture of two isomers. Pale yellow solid; R_f 0.57, 0.49 (hexane/EtOAc 2/1); ^{1}H NMR ($CDCl_3$) δ 0.85-1.29 (m, 8H), [1.32 (major isomer), 1.35 (minor isomer), s, 9H, CCH_3], 1.85 (bs, 1H), 2.04-2.10 (m, 1H), 2.25-2.36 (m, 1H), [5.45 (d, *J* = 5.1 Hz, major isomer), 5.46 (d, *J* = 5.1 Hz, minor isomer), 1H, 4-CH], [6.65 (d, *J* = 5.1 Hz, major isomer), 6.67 (d, *J* = 5.1 Hz, minor isomer), 1H, 5-CH], 7.10-7.24 (m, 3H), 7.41-7.47 (m, 2H); ^{13}C NMR ($CDCl_3$) δ [28.0 (minor isomer), 28.3 (major isomer), C*CH*$_3$], 31.2, 31.7, 33.4, 35.2, 36.2, 36.8, 37.0, 38.0, 38.2, 38.5, 49.6, 50.0, 54.3, 54.4, 61.5, 61.7, [110.0 (major isomer), 110.2 (minor isomer), 4-C], 126.5, 127.0, 127.1, 127.9, 128.1, [129.9 (minor isomer), 130.4 (major isomer), 5-C], 139.6, [179.4 (major isomer), 179.9 (minor isomer), 2-C]; IR (KBr) 3124 w, 2956 s, 2872 s, 2072 w, 2028 w, 1994 w, 1692 s, 1610 m,

1498 m, 1482 m, 1452 m, 1398 m, 1368 m, 1348 m, 1314 m, 1284 s, 1254 s, 1220 s, 1178 s, 1116 w, 1074 w, 1032 w, 970 w, 934 w, 900 w, 842 w, 810 w, 760 m, 708 s, 692 s; MS, *m*/*z* (relative intensity, %) for major isomer: 309 (M^+, 0.56), 216 (7), 215 (48), 160 (11), 159 (100), 158 (26), 130 (12), 115 (7), 103 (19), 95 (37), 91 (5), 77 (6), 67 (27), 65 (5), 57 (27), 55 (9), 53 (5); for minor isomer: 309 (M^+, 0.68), 216 (8), 215 (48), 160 (11), 159 (100), 158 (28), 130 (11), 115 (6), 103 (20), 95 (39), 91 (5), 77 (7), 67 (33), 65 (5), 57 (27), 55 (10), 53 (6); exact mass calcd for $C_{21}H_{27}NO$ 309.2093, found 309.2092 (major isomer), 309.2074 (minor isomer).

2,3-Dihydro-1-(1,1-dimethylethyl)-3-ethyl-3-(4-methoxyphenyl)-pyrrol-2-one (6). Pale yellow solid; mp 72-73 °C (hexane); R_f 0.50 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.72 (t, *J* = 7.3 Hz, 3H), 1.38 (s, 9H), 1.76-1.98 (m, 2H), 3.68 (s, 3H), 5.42 (d, *J* = 5.1 Hz, 1H), 6.65 (d, *J* = 5.1 Hz, 1H), 6.76 (d, *J* = 9.2 Hz, 2H), 7.28 (d, *J* = 9.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 9.2, 28.3, 31.4, 54.4, 55.1, 58.4, 112.1, 113.6, 127.5, 130.0, 132.5, 158.2, 180.2; IR (KBr) 3120 w, 2972 s, 2952 m, 2052 w, 1682 s, 1610 m, 1514 s, 1486 m, 1458 m, 1396 m, 1366 m, 1280 s, 1248 s, 1178 s, 1112 m, 1088 w, 1030 s, 932 w, 924 m, 832 m, 804 m, 754 m, 730 w, 714 m, 692 m, 660 w, 636 w; MS, *m*/*z* (relative intensity, %) 273 (M^+, 18), 217 (36), 202 (21), 189 (20), 188 (100), 158 (14), 133 (27), 115 (11), 57 (44), 53 (11). Anal. Calcd for $C_{17}H_{23}NO_2$: C, 74.69; H, 8.48; N, 5.12. Found: C, 74.57; H, 8.43; N, 5.09.

2,3-Dihydro-1-(1,1-dimethylethyl)-3-ethyl-3-(4-trifluoromethylphenyl)-pyrrol-2-one (8). Clear oil; R_f 0.56 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.71 (t, *J* = 7.3 Hz, 3H), 1.37 (s, 9H), 1.84-1.99 (m, 2H), 5.45 (d, *J* = 5.4 Hz, 1H), 6.71 (d, *J* = 5.4 Hz, 1H), 7.45-7.52 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 9.0, 28.2, 31.5, 54.6, 59.1, 111.3, 124.2 (q, *J* = 271.0 Hz, CF_3), 125.0-125.6 (complex, Ar), 127.1, 129.0 (q, *J* = 32.4 Hz, Ar), 131.0, 144.6, 179.4; IR (neat) 2976 s, 2940 m, 2884 m, 1698 s, 1618

m, 1464 m, 1414 m, 1370 m, 1328 s, 1284 s, 1246 s, 1166 s, 1128 s, 1070 s, 1018 m, 932 w, 900 m, 840 m, 812 w, 746 w, 694 m, 604 m; MS, *m*/*z* (relative intensity, %) 311 (M^+, 4), 255 (14), 240 (6), 227 (8), 226 (44), 58 (5), 57 (100), 53 (5); exact mass calcd for $C_{17}H_{20}F_3NO$ 311.1497, found 311.1486.

2,3-Dihydro-3-butyl-1-(1,1-dimethylethyl)-3-ethyl-pyrrol-2-one (10). Clear oil; R_f 0.60 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.71 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.0 Hz, 3H), 0.99-1.29 (m, 4H), 1.44 (s, 9H), 1.46-1.68 (m, 4H), 5.08 (d, *J* = 2.7 Hz, 1H), 6.61 (d, *J* = 2.7 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 8.7, 14.0, 23.1, 26.5, 28.3, 29.8, 36.5, 54.2, 56.1, 111.8, 129.6, 181.9; IR (neat) 2968 m, 1698 s, 1610 m, 1464 m, 1368 m, 1334 m, 1284 s, 1250 s, 1226 m, 1178 m, 934 w, 812 w, 746 w, 686 m; MS, *m*/*z* (relative intensity, %) 223 (M^+, 5), 208 (18), 138 (45), 124 (23), 111 (63), 110 (100), 96 (36), 95 (13), 83 (16), 82 (27), 67 (21), 57 (51), 55 (25), 53 (16). Anal. Calcd for $C_{14}H_{25}NO$: C, 75.28; H, 11.28; N, 6.27. Found: C, 75.00; H, 11.13; N, 6.14.

2,3-Dihydro-1-(1,1-dimethylethyl)-3-ethyl-4-methyl-3-phenyl-pyrrol-2-one (12). Clear oil; R_f 0.49 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.78 (t, *J* = 7.6 Hz, 3H), 1.47 (s, 9H), 1.56 (d, *J* = 1.6 Hz, 3H), 1.81-1.94 (m, 1H), 2.21-2.34 (m, 1H), 6.46 (d, *J* = 1.6 Hz, 1H), 7.18-7.32 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 8.3, 10.9, 25.5, 28.3, 54.2, 61.3, 120.6, 125.1, 126.2, 126.6, 128.3, 139.7, 180.4; IR (neat) 3384 w, 3092 w, 3060 w, 3028 w, 2972 s, 2932 s, 2880 m, 2744 w, 2456 w, 1944 w, 1872 w, 1698 s, 1660 m, 1604 w, 1500 m, 1460 m, 1396 m, 1384 m, 1360 s, 1328 m, 1282 m, 1264 m, 1210 m, 1184 m, 1170 m, 1134 w, 1118 w, 1066 w, 1034 m, 968 w, 952 w, 906 m, 870 w, 810 w, 790 w, 762 m, 732 m, 716 w, 696 m, 650 w; MS, *m*/*z* (relative intensity, %) 257 (M^+, 13), 201 (12), 186 (25), 173 (15), 172 (100), 117 (15), 115 (19), 57 (32). Anal. Calcd for $C_{17}H_{23}NO$: C, 79.33; H, 9.01; N, 5.44. Found: C, 79.05; H, 8.84; N, 5.30.

2,3,4,5,6,7-Hexahydro-3-(1,1-dimethylethylamino)-2,5,5-trimethyl-1*H*-4,6-methanoinde-1-one (25). The reaction of imine **24** gives two isomers of **25**. For one isomer; White solid; mp 80-82 °C (hexane); R_f 0.24 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.70 (s, 3H), 1.06 (s, 9H), 1.12 (d, *J* = 6.2 Hz, 1H), 1.17 (d, *J* = 7.6 Hz, 3H), 1.34(s, 3H), 2.15 (dq. *J* = 7.6 Hz, *J* = 1.2 Hz, 1H), 2.20-2.24 (m, 1H), 2.29-2.33 (m, 2H), 2.44-2.48 (m, 1H), 2.50-2.58 (m, 1H), 3.36 (bs, 1H); ^{13}C NMR ($CDCl_3$) δ 15.8, 21.6, 25.4, 26.0, 30.1, 31.6, 39.8, 40.2, 43.3, 50.4, 53.1, 60.7, 133.5, 184.3, 208.9; IR (KBr) 3324 m, 2964 s, 1694 s, 1642 s, 1500 m, 1458 m, 1388 s, 1362 m, 1300 m, 1276 w, 1258 m, 1230 m, 1204 m, 1152 w, 1134 w, 1102 w, 1078 w, 1064 w, 1024 w, 992 w, 938 w, 908 w, 884 w, 846 w, 814 w, 786 w, 730 m, 622 w; MS, *m*/*z* (relative intensity, %) 261 (M^+, 21), 246 (20), 190 (44), 136 (27), 117 (22), 105 (27), 91 (39), 79 (21), 77 (26), 58 (100), 57 (67), 56 (24), 55 (31), 53 (22); exact mass calcd. for $C_{17}H_{27}NO$ 261.2092, found 261.2087. For another isomer; White solid; mp 66-68 °C (hexane); R_f 0.07 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.69 (s, 3H), 1.08 (s, 9H), 1.13 (d, *J* = 9.2 Hz, 1H), 1.17 (d, *J* = 7.6 Hz, 3H), 1.34(s, 3H), 2.10 (q. *J* = 7.6 Hz, 1H), 2.19-2.23 (m, 1H), 2.29-2.34 (m, 2H), 2.47-2.60 (m, 2H), 3.26 (bs, 1H); ^{13}C NMR ($CDCl_3$) δ 16.0, 21.0, 25.4, 26.1, 30.1, 31.6, 40.2, 40.4, 42.8, 50.6, 52.6, 62.1, 133.9, 184.4, 208.5; IR (KBr) 3332 m, 2968 s, 1694 s, 1642 s, 1506 m, 1474 m, 1458 m, 1388 s, 1370 m, 1300 m, 1256 m, 1228 m, 1202 m, 1174 w, 1134 w, 1114 m, 1090 m, 1066 w, 1022 w, 992 w, 940 w, 906 w, 882 w, 854 w, 830 w, 782 w, 732 m, 660 w; MS, *m*/*z* (relative intensity, %) 261 (M^+, 19), 246 (19), 190 (37), 136 (22), 105 (16), 91 (30), 79 (17), 77 (18), 58 (100), 57 (57), 56 (20), 55 (22). Anal. Calcd for $C_{17}H_{27}NO$: C, 78.11; H, 10.41; N, 5.36. Found: C, 77.82; H, 10.14; N, 5.31.

6,7,8,9-Tetrahydro-5-ethyl-5*H*-pyrrolo[2,1-a]isoindole (28). Dark brown oil; R_f 0.67 (hexane/EtOAc 3/1); ^{1}H NMR ($CDCl_3$) δ 0.67 (t, *J* = 7.3 Hz, 3H), 1.67-1.80 (m, 5H), 1.82-2.00 (m, 1H), 2.18-2.20 (m, 2H), 2.33-2.39 (m, 2H), 4.33 (bs, 1H), 5.77 (d, *J* = 3.2 Hz, 1H), 6.20 (dd, *J* = 3.2 Hz, *J* =

2.7 Hz, 1H), 6.80 (d, J = 2.7 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 7.6, 21.9, 22.4, 22.8, 23.2, 25.6, 64.8, 94.0, 110.4, 115.1, 120.1, 137.4, 142.2; IR (neat) 3100 w, 2928 s, 2880 m, 1562 m, 1470 m, 1418 m, 1384 m, 1346 m, 1290 m, 1264 m, 1224 m, 1184 w, 1050 m, 1026 m, 904 w, 826 w, 756 m, 694 s, 604 w; MS, m/z (relative intensity, %) 187 (M^+, 72), 172 (100), 159 (31), 158 (88), 144 (19), 143 (16), 131 (23), 130 (70), 117 (15), 91 (15), 80 (16), 77 (35), 65 (18), 63 (16), 51 (28). Anal. Calcd for $C_{13}H_{17}N$: C, 83.37; H, 9.15; N, 7.48. Found: C, 83.17; H, 9.00; N, 7.46.

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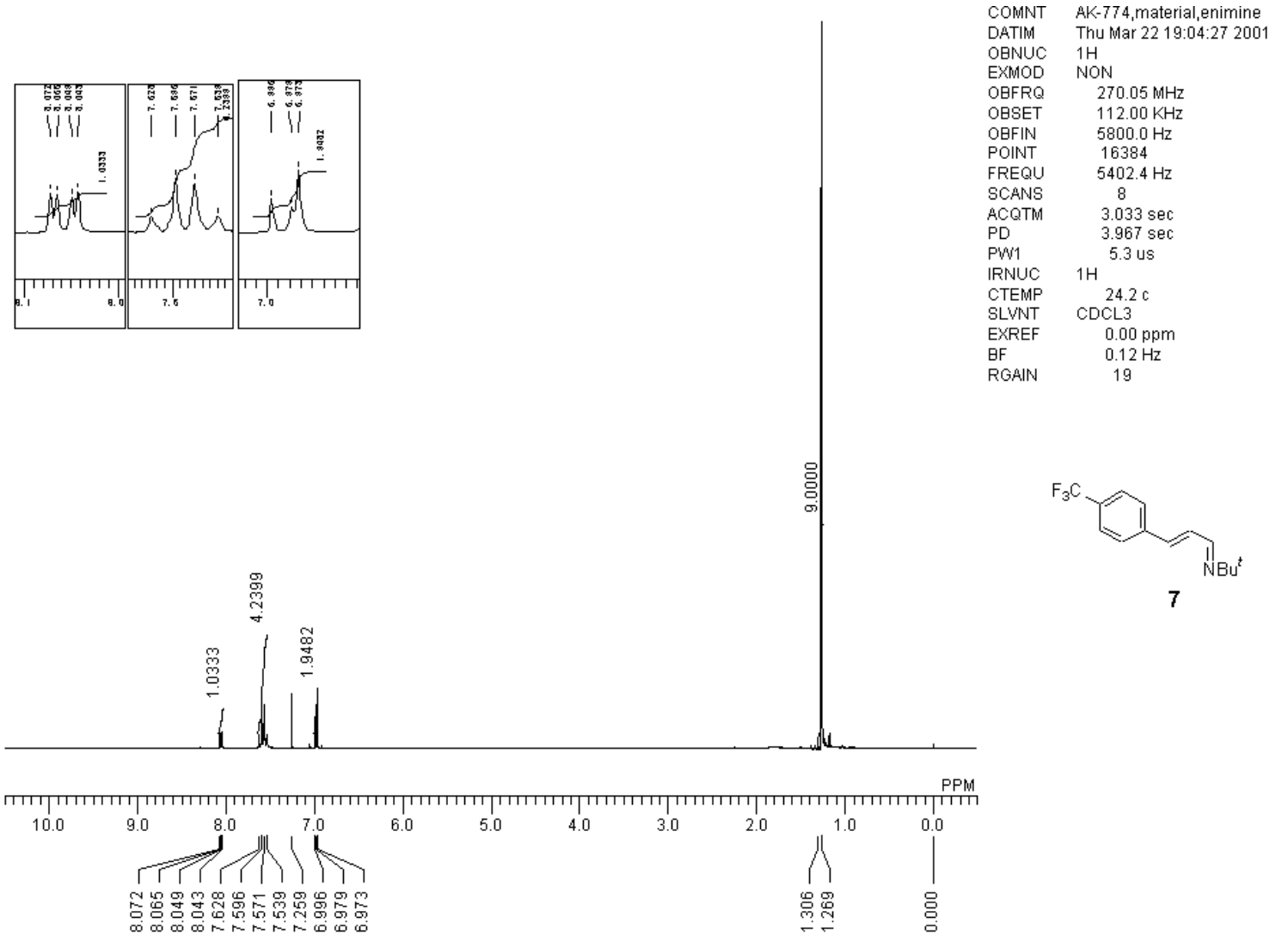
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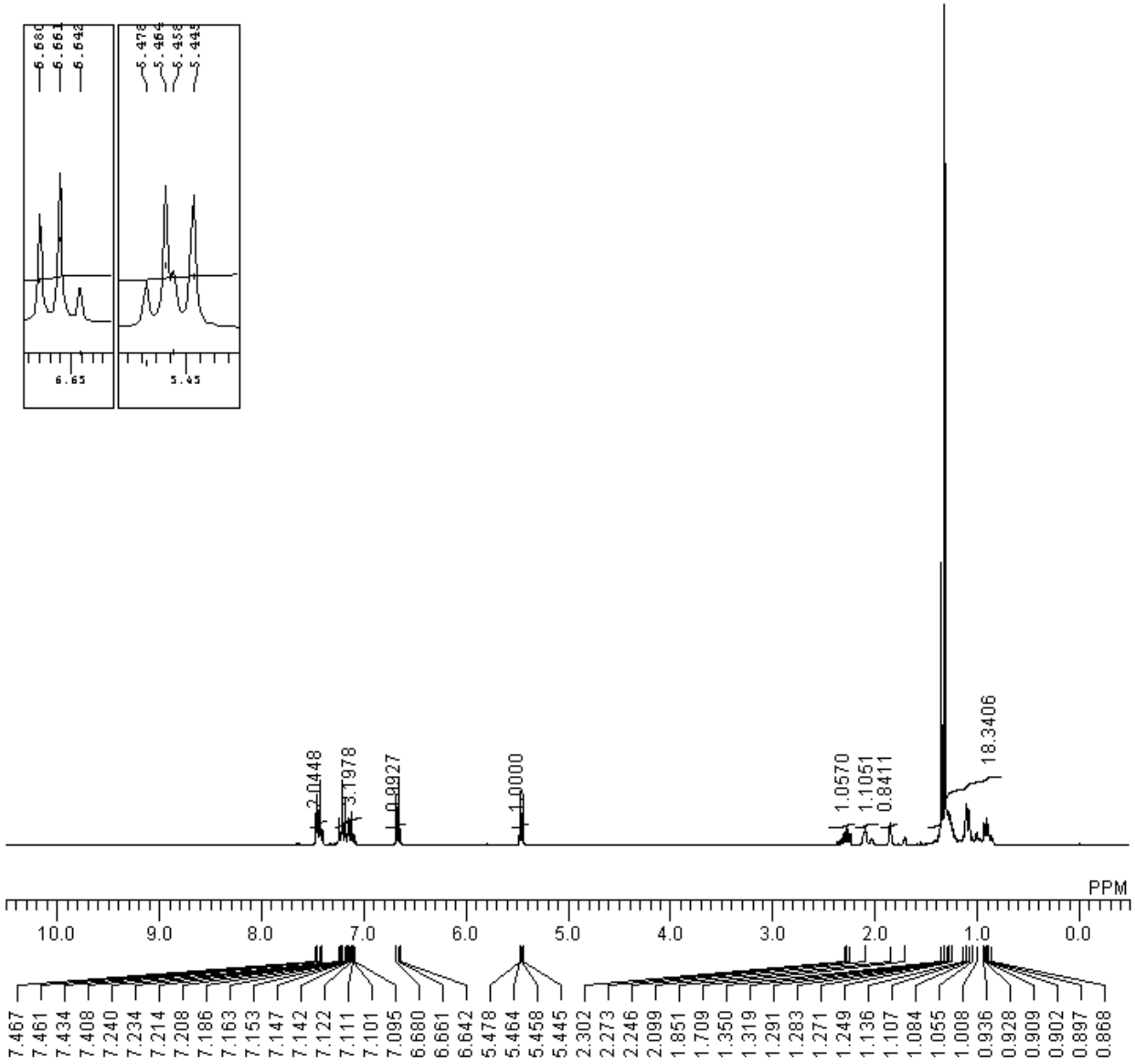

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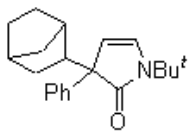

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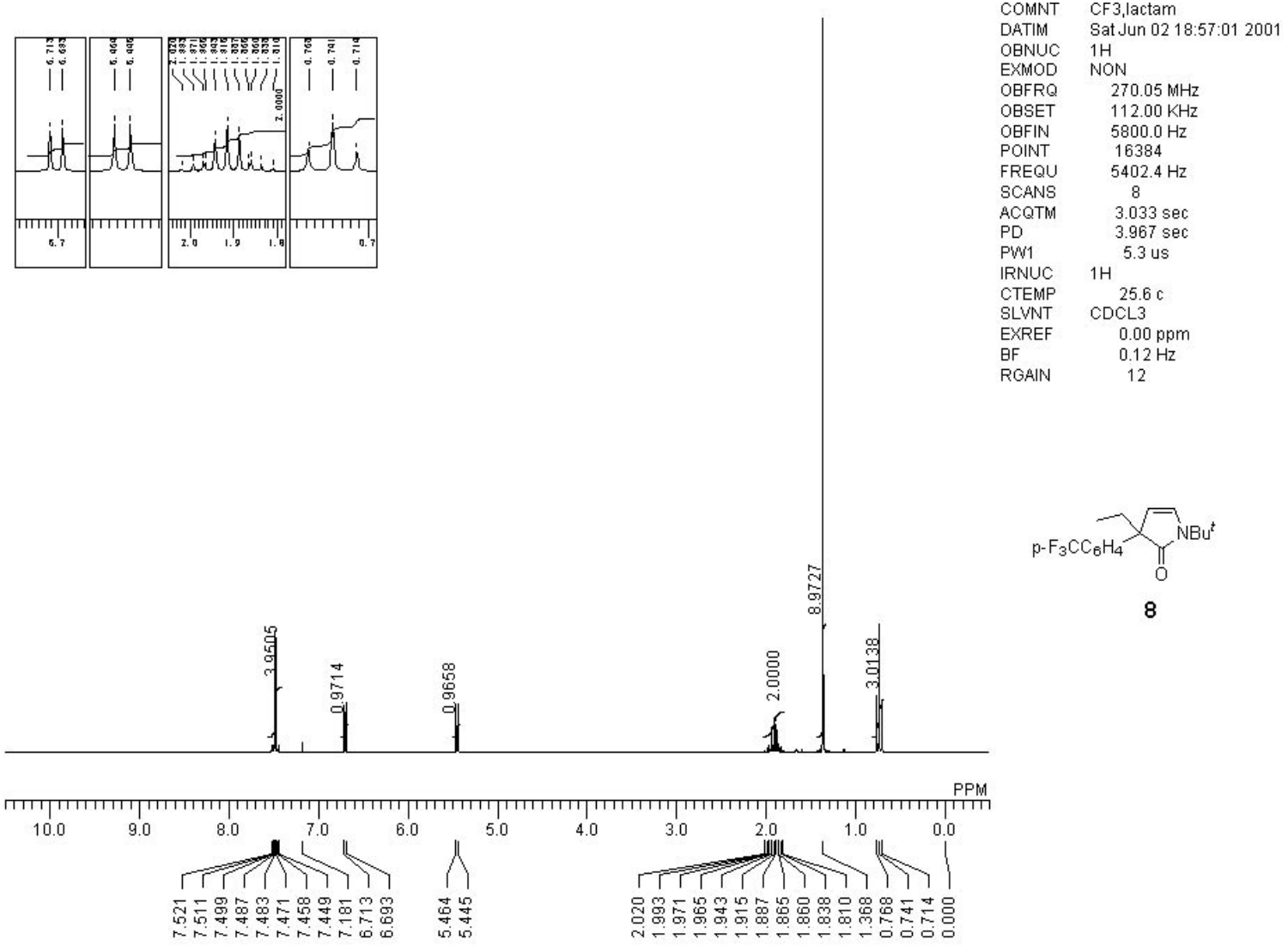
a mixture of two isomers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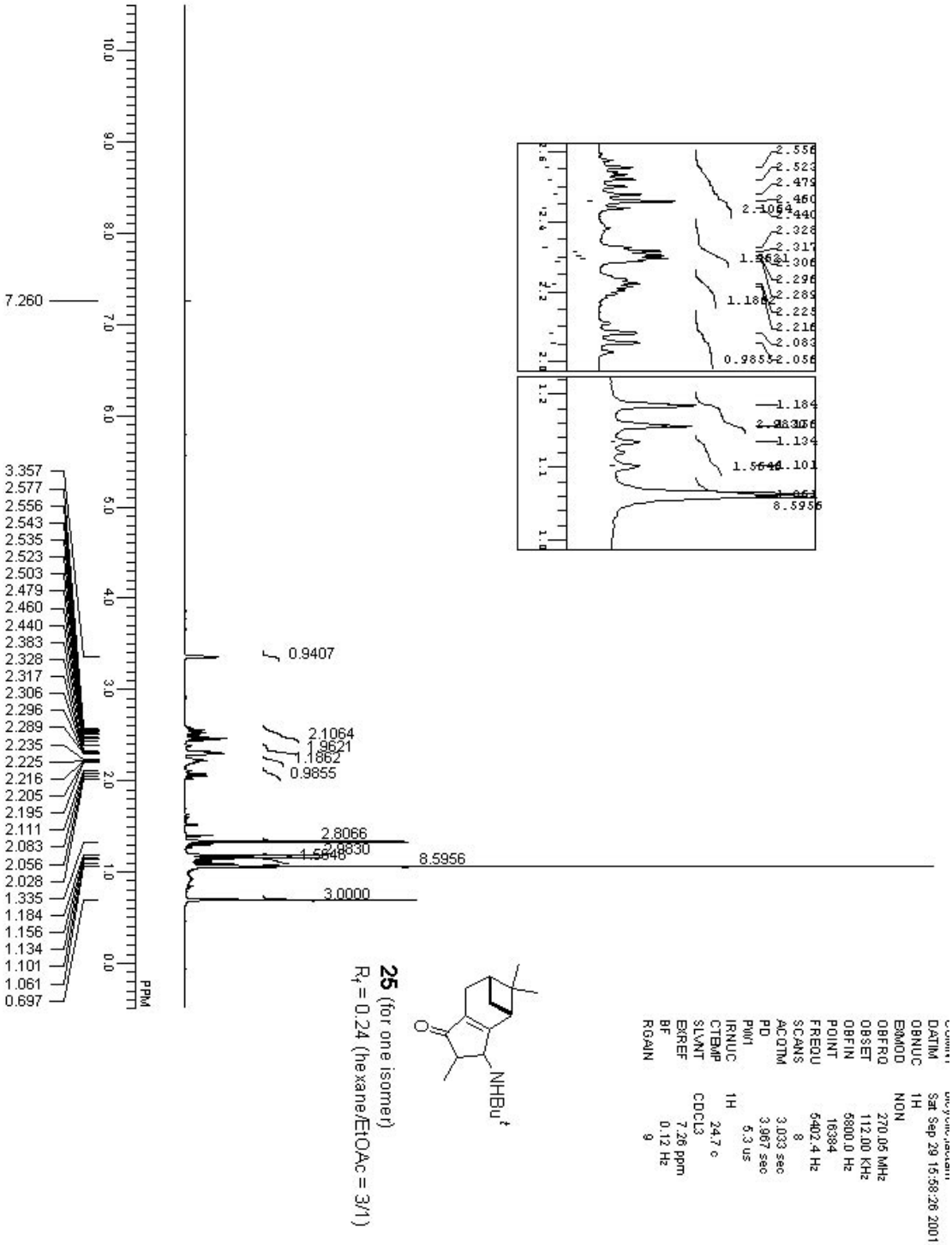
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EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.0 Hz
POINT 16384
FREQU 5402.4 Hz
SCANS 8
ACQTM 3.033 sec
PD 3.967 sec
PW1 5.3 us
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 9
0.9407
2.1064
1.9621
1.1862
0.9855
2.8066
2.9830
1.5848
8.5956
3.0000
NHBu^t
25 (for one isomer)
R_f = 0.24 (hexane/EtOAc = 3/1)
7.260
PPM