Rhodium-Catalyzed Reaction of Terminal Alkynes with Allylamine Leading to (E)-3-Alkylidene-N-Heterocycles

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Supporting Information

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General. ^1H NMR and ^{13}C NMR spectra were recorded on a JEOL EX-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, bs = broad singlet, m = multiplet, c = complex), coupling constant (Hz), integration, and interpretation. Infrared spectra (IR) were obtained on a Horiba FT-720 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. Analytical gas chromatography (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)). Analytically pure samples were obtained by flash column chromatography on SiO_2 (Merck Silica Gel 60 (70-230 mesh)). THF was distilled over Na/benzophenone. Allylamine and alkynes **1a** (TCI), **1e** (Acros), **1g** (Aldrich), **1h** (TCI), and **1i** (TCI) were purchased and purified by distillation before use. $\text{RhCl}(\text{PPh}_3)_3$ and NH_4BF_4 were commercially available from Aldrich and used without further purifications. Alkynes **1b**,¹ **1c**,² **1d**,³ **1f**⁴ and *N*-crotylamine⁵ were prepared according to the literature procedures.

General Procedure for $\text{RhCl}(\text{PPh}_3)_3/\text{NH}_4\text{BF}_4$ -Catalyzed Reaction of Terminal Alkynes with Allylamine. A 5 mL Schlenk tube was flame-dried and then cooled to room temperature. In the tube were placed $\text{RhCl}(\text{PPh}_3)_3$ (46.3 mg, 0.05 mmol), NH_4BF_4 (6.3 mg, 0.06 mmol), THF (2 mL), alkynes (1 mmol), and allylamine (0.075 mL, 1 mmol). The reaction mixture was refluxed for 30 h. After cooling to room temperature, the volatiles were removed *in vacuo*. The product was isolated by column chromatography on silica gel.

(*E*)-3-Methyl-5-pentyl-4-pentylidene-3,4-dihydro-2H-pyrrole (2a). Pale yellow oil, R_f = 0.06 (Hexane:AcOEt = 10:1). ^1H NMR (CDCl_3): δ 0.90 (t, J = 7.5 Hz, 3H, CH_3), 0.93 (t, J = 7.5 Hz, 3H, CH_3), 1.03 (d, J = 6.9 Hz, 3H, CH_3), 1.23-1.49 (c, 8H, $4\times\text{CH}_2$), 1.58-1.69 (c, 2H, $\text{N}=\text{CCH}_2\text{CH}_2$), 2.18 (m, 2H, $\text{C}=\text{CHCH}_2$), 2.38 (t, J = 7.1 Hz, $\text{N}=\text{CCH}_2$), 2.87 (quint, J = 6.9 Hz, 1H, CH_3CH), 3.49 (dd, J = 16.1, 1.5 Hz, 1H, NCH_2), 3.95 (dd, J = 16.1, 6.9 Hz, 1H, NCH_2), 5.78 (td, J = 7.6, 2.1 Hz, 1H, $=\text{CH}$). ^{13}C NMR (CDCl_3): δ 14.04 (CH_3), 14.08 (CH_3), 20.80 (CHCH_3), 22.54 (CH_2), 22.56 (CH_2), 26.60 ($\text{N}=\text{CCH}_2\text{CH}_2$), 29.15 ($\text{C}=\text{CHCH}_2$), 29.53 ($\text{N}=\text{CCH}_2$), 31.84 (CH_2), 31.92 (CH_2), 33.76 (CH_3CH), 66.27 (NCH_2), 126.51 ($=\text{CH}$), 147.31 ($\text{CH}=\text{C}$), 173.91 ($\text{N}=\text{C}$). IR (neat): 2956 s, 2929

s, 2860 s, 1601 m, 1458 m, 1377 m cm^{-1} . MS: m/z (relative intensity, %): 221 (M^+ , 1), 178 (20), 165 (60), 164 (100, $\text{M}^+ - \text{C}_4\text{H}_9$), 150 (61), 136 (33), 123 (18), 122 (29), 108 (39), 106 (10), 95 (13), 94 (18), 93 (10), 82 (47), 81 (18), 80 (18), 79 (18), 77 (13), 68 (11), 67 (20), 55 (29), 54 (12), 53 (21). HRMS. Calcd for $\text{C}_{15}\text{H}_{27}\text{N}$ (M^+): 221.2143., Found: 221.2142.

(*E*)-5-[4-(4-Methoxycarbonyl)butylidene-3-methyl-3,4-dihydro-2*H*-pyrrol-5-yl]pentanoic acid methyl ester (2b). Pale yellow oil, R_f = 0.06 (Hexane:AcOEt = 3:2). ^1H NMR (CDCl_3): δ 1.02 (d, J = 7.3 Hz, 3H, CH_3), 1.69 (bs, 4H, $2 \times \text{CH}_2$), 1.79 (quint, 2H, J = 7.0 Hz, CH_2), 2.18 (m, 2H, $\text{C}=\text{CHCH}_2$), 2.33-2.38 (c, 6H, $\text{N}=\text{CCH}_2$ and $2 \times \text{O}=\text{CCH}_2$), 2.87 (quint, J = 6.9 Hz, 1H, CH_3CH), 3.50 (d, J = 16.2 Hz, 1H, NCH_2), 3.65 (s, 3H, O CH_3), 3.67 (s, 3H, OCH_3), 3.95 (dd, J = 16.2, 6.9 Hz, 1H, NCH_2), 5.74 (t, J = 7.1 Hz, 1H, $=\text{CH}$). ^{13}C NMR (CDCl_3): δ 20.76 (CHCH_3), 24.74 (CH_2), 24.90 (CH_2), 26.19 (CH_2), 28.77 (CH_2), 29.05 (CH_2), 33.53 (CH_3CH), 33.75 ($\text{O}=\text{CCH}_2$), 33.90 ($\text{O}=\text{CCH}_2$), 51.51 (OCH_3), 51.59 (OCH_3), 66.22 (NCH_2), 125.30 ($=\text{CH}$), 148.14 ($\text{CH}=\text{C}$), 173.16 ($\text{N}=\text{C}$), 173.54, ($\text{C}=\text{O}$), 173.81 ($\text{C}=\text{O}$). IR (neat): 2952 m, 2931 m, 1739 s ($\text{C}=\text{O}$), 1456 m, 1437 m, 1246 m, 1203 m, 1173 m cm^{-1} . MS: m/z (relative intensity, %): 309 (M^+ , 8), 278 (11), 236 (30), 222 (10), 209 (29), 208 (64), 194 (22), 162 (10), 148 (22), 139 (10), 136 (66), 135 (11), 134 (31), 132 (15), 123 (81), 122 (74), 121 (23), 120 (47), 118 (15), 109 (20), 108 (100), 107 (16), 106 (35), 105 (13), 95 (12), 94 (41), 93 (25), 91 (33), 82 (12), 81 (17), 80 (29), 79 (47), 78 (10), 77 (36), 68 (13), 67 (37), 66 (11), 65 (23), 59 (55), 56 (11), 55 (77), 54 (23), 53 (34). HRMS. Calcd for $\text{C}_{17}\text{H}_{27}\text{NO}_4$ (M^+): 309.1943., Found: 309.1940.

(*E*)-3-Methyl-5-[4-(tetrahydropyran-2-yloxy)butyl]-4-[4-(tetrahydropyran-2-yloxy)-butylidene]-3,4-dihydro-2*H*-pyrrole (2c, diastereomeric mixtures). Pale yellow oil, R_f = 0.11 (Hexane:AcOEt = 1:1). ^1H NMR (CDCl_3): δ 1.02 (d, J = 6.9 Hz, 3H, CH_3), 1.47-1.50 (c, 18H, $9 \times \text{CH}_2$), 2.23-2.30 (c, 4H, $2 \times \text{CH}_2$), 2.38 (t, J = 7.3 Hz, 2H, $\text{N}=\text{CCH}_2$), 2.87 (bs, 1H, CH_3CH), 3.38-3.51 (c, 5H, NCH_2 and $2 \times \text{CH}_2$), 3.74-3.89 (c, 5H, NCH_2 and $2 \times \text{CH}_2$), 4.55-4.57 (dd, J = 4.1, 2.5 Hz, 2H, OCH), 5.74 (td, J = 7.4, 1.9 Hz, 1H, $=\text{CH}$). ^{13}C NMR (CDCl_3): δ 19.73 (CH_2), 20.72 and 20.75 (CH_3), 23.61 (CH_2), 25.52 and 25.55 (CH_2), 26.27 and 26.29 ($\text{C}=\text{CHCH}_2$), 29.25 ($\text{N}=\text{CCH}_2\text{CH}_2$), 29.68, 29.71, and 29.73 ($\text{C}=\text{CHCH}_2\text{CH}_2$ and $\text{N}=\text{CCH}_2\text{CH}_2\text{CH}_2$), 30.79 and 30.80 (CH_2), 33.76 (CH_3CH), 62.34 62.37 and 62.40 ($2 \times \text{OCH}_2$), 66.18 (NCH_2), 66.78, 66.87, 67.28 and 67.31

(2×OCH₂), 98.79, 98.81, and 98.89 (OCH), 126.16 (=CH), 147.65 and 147.69 (CH=C), 173.63 (N=C). IR (neat): 2941 s, 2868 s, 1600 m, 1452 m, 1442 m, 1352 m, 1201 m, 1138 s, 1120 s, 1076 s, 989 m, 906 m, 870 m cm⁻¹. MS: *m/z* (relative intensity, %): 393 (M⁺, 1), 308 (15), 224 (13), 208 (10), 150 (21), 136 (19), 123 (17), 85 (100), 71 (11), 67 (27), 57 (26), 55 (32). HRMS. Calcd for C₂₃H₃₉NO₄ (M⁺): 393.2879, Found: 393.2881.

(*E*)-5-(4-*tert*-Butyldimethylsiloxy)butyl-4-(4-*tert*-butyldimethylsiloxy)butylidene-3-methyl-3,4-dihydro-2*H*-pyrrole (2d). Pale yellow

oil, *R_f* = 0.06 (Hexane:AcOEt = 10:1). ¹H NMR (CDCl₃): δ 0.02 (s, 6H, SiCH₃), 0.03 (s, 6H, SiCH₃), 0.86 (s, 9H, SiC(CH₃)₃), 0.88 (s, 9H, SiC(CH₃)₃), 1.00 (d, *J* = 7.3 Hz, 3H, CH₃), 1.53-1.70 (c, H, 3×CH₂), 2.23 (qd, *J* = 7.6, 2.6

Hz, 2H, C=CHCH₂), 2.38 (t, *J* = 7.9 Hz, N=CCH₂), 2.86 (broad quint, *J* = 7.3 Hz, 1H, CH₃CH), 3.47 (dd, *J* = 7.3, 2.6 Hz, 1H, NCH₂), 3.59-3.66 (c, 4H, 2×OCH₂), 3.93 (dd *J* = 16.2, 7.3 Hz, 1H, NCH₂), 5.74 (td, *J* = 7.6, 1.9 Hz, 1H, =CH). ¹³C NMR (CDCl₃): δ -5.35 (SiCH₃), -5.31 (SiCH₃), 18.26 (SiC(CH₃)₃), 18.30 (SiC(CH₃)₃), 20.65 (CH₃), 23.18 (CH₂), 25.78 (CH₂), 25.88 (SiC(CH₃)₃), 25.94 (SiC(CH₃)₃), 29.25 (CH₂), 32.64 (CH₂), 32.72 (CH₂), 33.71 (CH₃CH), 62.40 (OCH₂), 62.95 (OCH₂), 66.33 (NCH₂), 125.93 (=CH), 147.80 (CH=C), 173.72 (N=C). IR (neat): 2954 s, 2929 s, 2858 s, 1471 m, 1462 m, 1255 s, 1103 s, 837 s, 775 s cm⁻¹. MS: *m/z* (relative intensity, %): 453 (M⁺, 5), 398 (12), 397 (34), 396 (100, M⁺-*t*Bu), 308 (11), 281 (15), 280 (44), 170 (16), 148 (24), 136 (35), 134 (12), 123 (10), 122 (10), 101 (17), 89 (12), 75 (56), 73 (76), 59 (43), 57 (12), 55 (16). Anal. Calcd for C₂₅H₅₁NO₂Si₂: C, 66.16; H, 11.33; N, 3.09, Found: C, 66.26; H, 11.27; N, 3.13.

(*E*)-5-[4-(4-Cyanobutylidene)-3-methyl-3,4-dihydro-2*H*-pyrrol-2-yl]pentanenitrile (2e). Pale

yellow oil, *R_f* = 0.08 (AcOEt). ¹H NMR (CDCl₃): δ 1.05 (d, *J* = 6.9 Hz, 3H, CH₃), 1.71-1.89 (c, 6H, 3×CH₂), 2.09-2.46 (c, 8H, 4×CH₂), 2.93 (quint, *J* = 6.9 Hz, 1H, CH₃CH), 3.53 (d, *J* = 16.5 Hz, 1H, NCH₂), 3.98 (dd, *J* = 16.5, 6.9 Hz, 1H, NCH₂), 5.71 (t, *J* = 6.9 Hz, 1H, =CH). ¹³C NMR (CDCl₃): δ 16.84

(CH₂), 17.04 (CH₂), 20.76 (CH₃), 25.13 (CH₂), 25.18 (CH₂), 25.46 (CH₂), 28.19 (CH₂), 28.34 (CH₂), 33.83 (CH₃CH), 66.32 (NCH₂), 119.18 (C=N), 119.50 (C=N), 123.49 (=CH), 149.08 (CH=C), 172.15 (N=C). IR (neat): 2956 s, 2931 s, 2865 s, 2245 m (C=N), 1658 m, 1603 s, 1454 s, 1425 s cm⁻¹. MS: *m/z* (relative intensity, %): 243 (M⁺, 1), 203 (20), 176 (24), 175 (100, M⁺-(CH₂)₃C=N), 161 (20), 136 (17), 134 (13), 133 (13), 122 (12), 120 (13), 108 (18), 94 (16), 91 (11), 80 (11), 79 (20), 77 (19), 67 (14), 64 (14), 55 (25), 54 (26), 53 (24). HRMS. Calcd for

$C_{15}H_{21}N_3$ (M^+): 243.1735, Found: 243.1731.

(E)-N-4-(3-methyl-4-phthalimidobutylidene-3,4-dihydro-2H-pyrrol-5-yl)butylphthalimide

(2f). Pale yellow oil, $R_f = 0.08$ (AcOEt). 1H

NMR (acetone- d_6): δ 0.93 (d, $J = 6.9$ Hz, 3H, CH_3), 1.58-1.71 (c, 4H, $2 \times CH_2$), 1.83 (quint, $J = 6.9$ Hz, 2H, CH_2), 2.06-2.33 (c, 4H, $C=CHCH_2$ and $N=CCH_2$), 2.86 (bs, 1H, CH_3CH and H_2O), 3.34 (d, $J = 16.2$ Hz, 1H,

NCH_2), 3.65 (t, $J = 6.9$ Hz, 2H, NCH_2CH_2), 3.68 (t, $J = 6.9$ Hz, 2H, NCH_2CH_2), 3.81 (dd, $J = 16.2, 6.9$ Hz, 1H, NCH_2), 5.76 (t, $J = 6.9$ Hz, 1H, $=CH$), 7.82 (bs, 8H, Ar). ^{13}C NMR ($CDCl_3$): δ 20.53 (CH_3), 23.79 (CH_2), 26.90 (CH_2), 28.09 (CH_2), 28.33 (CH_2), 28.60 (CH_2), 33.63 (CH_3CH), 37.60 ($2 \times NCH_2CH_2$), 66.05 (NCH_2), 123.09 (Ar), 123.16 (Ar), 125.58 ($=CH$), 131.96 (Ar), 132.09 (Ar), 133.78 (Ar), 133.97 (Ar), 148.06 ($CH=C$), 168.30 ($C=O$), 168.35 (Ar), 173.29 ($N=C$). IR (neat): 3059 w, 2933 m, 2864 m, 1770 s, 1709 s, 1601 m, 1437 s, 1396 s, 1371 s, 1188 m, 1119 m, 1039 m, 719 s cm^{-1} . MS: m/z (relative intensity, %): 483 (M^+ , 7), 323 (42), 296 (23), 295 (31), 160 (51), 148 (18), 137 (13), 136 (100), 134 (19), 133 (15), 130 (25), 122 (21), 121 (10), 120 (16), 108 (34), 106 (12), 105 (17), 104 (16), 94 (19), 93 (12), 91 (16), 80 (15), 79 (22), 77 (39), 76 (18), 67 (13), 56 (10), 55 (18), 53 (13). HRMS. Calcd for $C_{29}H_{29}N_3O_4$ (M^+): 483.2158, Found: 483.2161.

(E)-5-Cyclohexylmethyl-4-cyclohexylmethylidene-3-methyl-3,4-dihydro-2H-pyrrole **(2g).**

Pale yellow oil, $R_f = 0.03$ (Hexane:AcOEt = 10:1). 1H NMR

($CDCl_3$): δ 0.78-1.40 (c, 11H, Cy), 1.02 (d, $J = 6.9$ Hz, 3H, CH_3), 1.55-1.76 (c, 10H, Cy), 2.18-2.27 (c, 3H, $N=CCH_2$ and $C=CHCH$), 2.88 (quint, $J = 6.9$ Hz, 1H, CH_3CH), 3.49 (d, $J = 15.7$ Hz, 1H, NCH_2), 3.94 (dd, $J = 15.7, 6.9$ Hz, 1H, NCH_2), 5.59 (d, $J = 10.2$ Hz, 1H, $=CH$). ^{13}C NMR ($CDCl_3$): δ 21.70 (CH_3), 25.73, 25.79, 25.98,

26.27, 26.29, 26.48, 32.98, 33.44, 33.48, 33.65, 33.69, 36.27, 36.95,

38.90 (14C, $2 \times Cy$, CH_3CH and $N=CCH_2$), 66.21 (NCH_2), 132.33 ($=CH$), 145.88 ($CH=C$), 173.29 ($N=C$). IR (neat): 2924 s, 2850 s, 1599 m, 1448 s cm^{-1} . MS: m/z (relative intensity, %): 273 (M^+ , 1), 192 (16), 191 (100), 190 (60), 176 (61), 136 (11), 123 (16), 110 (14), 108 (19), 96 (14), 95 (20), 94 (29), 93 (12), 91 (16), 82 (23), 81 (16), 80 (18), 79 (22), 77 (15), 67 (25), 55 (55), 54 (14), 53 (18). HRMS. Calcd for $C_{19}H_{31}N$ (M^+): 273.2456, Found: 273.2448.

(E)-N-5-(2,2-Dimethylpropyl)-4-(2,2-dimethylpropylidene)-3-methyl-3,4-dihydro-2H-

pyrrole (2h). Pale yellow oil, $R_f = 0.07$ (Hexane:AcOEt = 10:1). ^1H NMR (CDCl_3): δ 0.98 (s, 9H, ^tBu), 1.38 (d, $J = 6.8$ Hz, 3H, CH_3), 1.17 (s, 9H, ^tBu), 2.28 (d, $J = 2.9$ Hz, 1H, $\text{N}=\text{CCH}_2$), 2.43 (d, $J = 2.9$ Hz, 1H, $\text{N}=\text{CCH}_2$), 3.11 (quint, $J = 7.3$ Hz, 1H, CH_3CH), 3.56 (d, $J = 15.9$ Hz, 1H, NCH_2), 3.94 (dd, $J = 15.9, 6.8$ Hz, 1H, NCH_2), 5.77 (s, 1H, $=\text{CH}$). ^{13}C NMR (CDCl_3): δ 22.55 (CHCH_3), 30.26 ($\text{C}(\text{CH}_3)_3$), 30.40 ($\text{C}(\text{CH}_3)_3$), 31.72 ($\text{C}(\text{CH}_3)_3$), 32.84 (CHCH_3), 34.01 ($\text{C}(\text{CH}_3)_3$), 41.34 ($\text{N}=\text{CCH}_2$), 65.74 (NCH_2), 138.43 ($=\text{CH}$), 144.2 ($\text{CH}=\text{C}$), 174.47 ($\text{N}=\text{C}$). IR (neat): 2960 s, 2864 m, 1475 m, 1466 m, 1365 m cm^{-1} . MS: m/z (relative intensity, %): 206 (M^+-CH_3 , 8), 165 (33), 164 (66), 151 (11), 150 ($\text{M}^+-\text{CH}_2^t\text{Bu}$, 100), 108 (12), 94 (12), 77 (11), 67 (11), 57 (19), 55 (20), 53 (12). HRMS. Calcd for $\text{C}_{15}\text{H}_{28}\text{N}$ (CI, M^++H): 222.2222, Found: 222.2221.

(E)-N-5-Benzyl-4-benzylidene-3-methyl-3,4-dihydro-2H-pyrrole (2i). Pale yellow oil, $R_f =$

0.03 (Hexane:AcOEt = 5:1). ^1H NMR (CDCl_3): δ 1.03 (d, $J = 6.9$ Hz, 3H, CH_3), 3.40 (quint, $J = 6.9$ Hz, 1H, CH_3CH), 3.69 (d, $J = 16.2$ Hz, 1H, NCH_2), 3.94 (s, 2H, CH_2Ph), 4.11 (dd, $J = 16.2, 6.9$ Hz, 1H, NCH_2), 6.70 (s 1H, $=\text{CH}$), 7.16-7.41 (c, 10H, Ph). ^{13}C NMR (CDCl_3): δ 19.07 (CH_3), 34.81 (CH_3CH), 36.48 (CH_2Ph), 67.13 (NCH_2), 126.29 (Ph), 126.39 (Ph), 127.86 ($=\text{CH}$), 128.46 (Ph), 128.51 (Ph), 128.65 (Ph), 128.74 (Ph), 135.74 (Ph), 137.07 (Ph), 146.92 ($\text{CH}=\text{C}$), 173.47 ($\text{N}=\text{C}$). IR (neat): 3059 m, 3026 m, 2962 m, 2927 m, 2858 m, 1599 m, 1495 s, 1454 s, 748 m, 727 s, 694 s cm^{-1} . MS: m/z (relative intensity, %): 261 (M^+ , 28), 260 (57), 246 (25), 184 (52), 179 (15), 168 (19), 130 (11), 129 (22), 128 (34), 127 (14), 115 (28), 102 (10), 91 (100), 89 (10), 77 (24), 65 (27), 63 (10), 51 (23). HRMS. Calcd for $\text{C}_{19}\text{H}_{19}\text{N}$ (M^+): 261.1517, Found: 261.1509.

(E)-3-Ethyl-5-pentyl-4-pentylidene-3,4-dihydro-2H-pyrrole (3b). Pale yellow oil, $R_f = 0.06$

(Hexane:AcOEt = 10:1). ^1H NMR (CDCl_3): δ 0.83 (t, $J = 7.4$ Hz, 3H, CH_3), 0.89 (t, $J = 7.5$ Hz, 3H, CH_3), 0.92 (t, $J = 7.5$ Hz, 3H, CH_3), 1.22-1.51 (c, 10H, $5 \times \text{CH}_2$), 1.60 (m, 2H, $\text{N}=\text{CCH}_2\text{CH}_2$), 2.16 (m, 2H, $\text{C}=\text{CHCH}_2$), 2.36 (t, $J = 7.3$ Hz, $\text{N}=\text{CCH}_2$), 2.67 (bs, 1H, $\text{CH}_3\text{CH}_2\text{CH}$), 3.62 (d, $J = 16.2$ Hz, 1H, NCH_2), 3.85 (dd, $J = 16.2, 6.9$ Hz, 1H, NCH_2), 5.78 (t, $J = 6.6$ Hz, 1H, $=\text{CH}$). ^{13}C NMR (CDCl_3): δ 10.75 (CH_3), 14.07 (CH_3), 14.10 (CH_3), 22.56 ($2 \times \text{CH}_2$), 26.66 ($\text{N}=\text{CCH}_2\text{CH}_2$), 27.52 (CH_2), 29.40 ($\text{C}=\text{CHCH}_2$), 29.49 ($\text{N}=\text{CCH}_2$), 31.80 (CH_2), 31.94 (CH_2), 40.49 ($\text{CH}_3\text{CH}_2\text{CH}$),

63.15 (NCH₂), 127.00 (=CH), 145.64 (CH=C), 174.70 (N=C). IR (neat): 2958 s, 2929 s, 2871 m, 2860 m, 1603 m, 1464 m cm⁻¹. MS: *m/z* (relative intensity, %): 220 (M⁺-CH₃, 3), 206 (29), 192 (22), 179 (57), 178 (100), 151 (11), 150 (90), 136 (26), 134 (12), 122 (50), 120 (16), 109 (11), 108 (62), 107 (12), 106 (26), 95 (15), 94 (34), 93 (20), 91 (17), 82 (83), 81 (22), 80 (59), 79 (28), 77 (25), 69 (14), 68 (12), 67 (47), 65 (18), 56 (12), 55 (68), 54 (23), 53 (37). HRMS. Calcd for C₁₆H₂₉N (M⁺): 235.2300., Found: 235.2294.

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YF-2005-5 1HNMR

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OBSET	112.00 KHz
OBFIN	5800.0 Hz
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DEADT	108.5 us
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IWT	1.0 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	5402.4 Hz
FLT	2700 Hz
DELAY	74.1 us
ACQTM	3.033 sec
PD	3.967 sec
ADBIT	16.00
RGAIN	19
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_Ac
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IFR	270.05 MHz
IRSET	112.00 KHz
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IRPW	50 us
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SF	TH5AT
LKSET	64.60 KHz
LKFIN	59.0 Hz
LKLEV	200
LGAIN	20
LKPHS	117
LKSIG	740
CSPED	17 Hz
FILDF

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YF-2005-5' 13CNMR

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

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
300
1
18315.0 Hz
9150 Hz
21.9 us
1.789 sec
1.211 sec
16.00
27
0.12 Hz
0.00
0.00
90.00
100.00
BCM
Blevel, complete decoupling, Se
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
C:\My Documents\O\YF-2005-
THSAT
64.80 KHz
59.0 Hz
200
23
117
753
16 Hz

C:\WINNMR98\TEMPDATA\Auto5DEPT_135_E1_FT.als
YF-2005-5

MENUF	DEPT
OBNUC	13C
OFPR	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.0 Hz
PW1	7.8 us
DEADT	28.9 us
PREDL	0.2000 ms
IWT	1.0 sec
POINT	32768
SPO	32
TIMES	4
DUMMY	
FREQU	16348.6 Hz
FLT	9150 Hz
DELAY	21.9 us
ACQTM	1.786 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	DEPT
EXPCM	DEPT (decoupled); W1 (C90), W2 (H90)
IRNUC	1H
IFR	270.05 MHz
IRSET	112.00 KHz
IRFIN	5800.0 Hz
IRRPW	50 us
IRATN	511
DFILE	C:\WINNMR98\TEMPDATA\Auto5DE
SF	THSAT
LKSET	64.60 KHz
LKFIN	59.0 Hz
LKLEV	200
LGAIN	23
LKPHS	117
LKSIG	640
CSPED	17 Hz
FILDC	
FILDF

DEFILE
COMNT
DATIM
EXMOD
OBNUC
OBFRQ
OBSET
OBFIN
POINT
FREQU
CLPNT
TODAT
CLFRQ
SCANS
ACQTM
PD
PW1
PW2
PW3
P11
P12
P13
IRNUC
CTEMP
SLVNT
EXREF
CLEXR
RGAIN

C:\My Documents\0\VF-2005-05\Auto2\VCOSYNH_E1_FT.als
VF-2005-5
Mon Jul 11 20:53:34 2005
VCOSYNH
1H
270.05 MHz
117.50 KHz
57.5 Hz
512
2871.9 Hz
256
256
2873.5 Hz
8
0.178 sec
1.500 sec
10.6 us
10.6 us
10.0 us
0.195 ms
0.348 ms
1.00 ms
1H
20.7 c
CDCL3
0.00 ppm
0.00 ppm
14

C:\WINNMR98\TEMPDATA\Auto4VCHSHF_E1_FT.als
YF-2005-5

DEFILE
COMNT
DATIM
EXMOD
OBNUC
OBFRQ
OBSET
OBRIN
POINT
FREQU
CLPNT
TODAT
CLFRQ
SCANS
ACQTM
PD
PW1
PW2
PW3
PI1
PI2
PI3
IRNUC
CTEMP
SLVNT
EXREF
CLEXR
RGAIN

C:\WINNMR98\TEMPDATA\Auto4VCHSHF_E1_FT.als
YF-2005-5
Tue Jul 12 02:16:26 2005
VCHSHF
13C
67.80 MHz
140.00 KHz
94.7 Hz
1024
13774.1 Hz
256
256
2873.5 Hz
32
0.074 sec
1.500 sec
7.8 us
15.6 us
21.2 us
3.448 ms
0.174 ms
1.72 ms
1H
20.2 C
CDCL3
77.00 ppm
0.00 ppm
27

Difference NOE Experiment

File Name = difference_noe.20
 Author = K. Ashih
 Sample ID =
 Content = Difference NOE Exper
 Creation Date = 13-DEC-2004 21:16:19
 Revision Date = 15-DEC-2004 08:37:52
 Spec Site = ECP400SL

Spec Type = DELTA NMR
 Data Format = 1D COMPLEX
 Dimensions = 1H
 Dim title =
 Dim Size = 16384
 Dim Units = [ppm]
 Actual_start_time = 13-DEC-2004 21:16:19
 Attenuator = 40[db]
 Delay_of_start = 1[s]
 Digital filter = FALSE
 End time = 13-DEC-2004 21:16:16
 Experiment = difference_noe_1
 Field strength = 9.2981736[T]
 Filter mode = BUTTERWORTH
 Filter width = 2.96912114[kHz]
 Irr code = 210
 Irr domain = 1H
 Irr freq = 395.88252601[MHz]
 Irr noise = WALTZ
 Irr offset = 2.77522326[ppm]
 Irr width = 55[us]
 Iterations = 0
 Local time = 13-DEC-2004 21:16:18
 Obs noise = WAUGH
 Obs width = 1[us]
 On_resonance = 2.77522326[ppm]
 Off_resonance = -20[ppm]
 Probe id = 2692
 Recvr_gain = 14
 Relaxation_delay = 1[s]
 Scans = 256
 Solvent = BENZENE-D6
 Spin_get = 0[Hz]
 Spin_lock_90 = 37.8[us]
 Spin_lock_attn = 11.6[db]
 Temp_get = 24.9[deg]
 X90 = 12.6[us]
 X_acq_duration = 2.7590656[s]
 X_angle = 90[deg]
 X_domain = 1H
 X_freq = 395.88252601[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_pascans = 8
 X_pulse = 12.6[us]
 X_resolution = 5.36244155[Hz]
 X_sweep = 1.93824228[kHz]
 Tri90 = 10[us]
 Tri_noise = WAUGH
 Tri_width = 10[us]
 Qua90 = 10[us]
 Qua_noise = WAUGH
 Qua_width = 1[us]

Difference NOE Experiment

(Millions)

X : parts per Million : 1H

```

---- ACQUISITION PARAMETERS ----
File Name      = difference_noe.20
Author         = JEOL LTD.
Sample ID      = kinashi
Content        = Difference NOE Exper
Creation Date   = 13-DEC-2004 20:46:24
Revision Date  = 15-DEC-2004 08:33:05
Spec Site      = ECP400SL

Spec Type      = DELTA_NMR
Data Format     = 1D COMPLEX
Dimensions     = X
Dim Title      = 1H
Dim Size       = 16384
Dim Units      = [ppm]
Actual_start_time = 13-DEC-2004 20:46:24
Attenuator     = 40[dB]
Delay_of_start = 1[s]
Digital_filter = FALSE
End_time       = 13-DEC-2004 20:46:21
Experiment     = difference_noe_1
Field_strength = 9.2981736[T]
Filter_mode     = BUTTERWORTH
Filter_width    = 2.96912114[kHz]
Irr_code       = 210
Irr_domain     = 1H
Irr_freq       = 395.88252601[MHz]
Irr_noise      = WALZ
Irr_offset     = 5.6609273[Dpm]
Irr_width      = 55[us]
Iterations     = 0
Local_time     = 13-DEC-2004 20:46:22
Obs_noise      = WAUGH
Obs_width      = 1[us]
On_resonance   = 5.6609273[ppm]
Off_resonance  = -20[ppm]
Probe_id       = 2692
Recvr_gain      = 14
Relaxation_delay = 1[s]
Scans          = 256
Solvent        = BENZENE-D6
Spin_get       = 0[kHz]
Spin_lock_90   = 37.8[us]
Spin_lock_attn = 11.8[db]
Temp_get       = 14.5[deg]
X90_duration   = 12.5[us]
X_acq_duration = 2.7590656[s]
X_angle        = 90[deg]
X_domain       = 1H
X_freq         = 395.88252601[MHz]
X_gain         = 5[Dpm]
X_offset       = 16384
X_points       = 8
X_prescans     = 8
X_pulse        = 12.5[us]
X_resolution   = 0.36244155[Hz]
X_sweep        = 5.93824228[kHz]
Tri90          = 10[us]
Tri_noise      = WALZ
Tri_width      = 1[us]
Qua90          = 10[us]
Qua_noise      = WAUGH
Qua_width      = 1[us]
    
```

Difference NOE Experiment

----- ACQUISITION PARAMETERS -----
 File Name = Kinashi-041214-noe-3
 Author = JEOL LTD.
 Sample ID = Kinashi
 Content = Difference NOE Exper
 Creation Date = 13-DEC-2004 21:46:14
 Revision Date = 15-DEC-2004 08:57:57
 Spec Site = ECP400SL
 Spec Type = DELTA_NMR
 Data Format = 1D COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 Actual_start_time = 13-DEC-2004 21:16:23
 Attenuator = 40[dB]
 Delay_of_start = 1[s]
 Digital_filter = FALSE
 End_time = 13-DEC-2004 21:46:11
 Experiment = difference_noe_1
 Field_strength = 9.2981736[T]
 Filter_mode = BUTTERWORTH
 Filter_width = 2.9691214[kHz]
 Irr_code = 210
 Irr_domain = 1H
 Irr_freq = 395.88252601[MHz]
 Irr_offset = WALTZ
 Irr_width = 0.94694132[ppm]
 Iterations = 55[us]
 Local_time = 13-DEC-2004 21:46:12
 Obs_noise = WAUGH
 Obs_width = 1[s]
 Off_resonance = 0.4894132[ppm]
 Probe_idn = 20[ppm]
 Relaxation_delay = 2692
 Scans = 1[s]
 Solvent = BENZENE-D6
 Spin_get = 0[Hz]
 Spin_lock_90 = 37.8[us]
 Spin_lock_attn = 11.6[dB]
 Temp_get = 24.9[deg]
 X90 = 12.6[us]
 X_acq_duration = 2.7590656[s]
 X_angle = 90[deg]
 X_domain = 1H
 X_freq = 395.88252601[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 8
 X_pulse = 12.6[us]
 X_resolution = 0.36244155[Hz]
 X_sweep = 5.93824228[kHz]
 Tri90 = 10[us]
 Tri_noise = WALTZ
 Tri_width = 1[us]
 Qua90 = 10[us]
 Qua_noise = WAUGH
 Qua_width = 1[us]

C:\WINNMR98\COMMON\DEFAULT.ALS
YF-2005-13 1HNMR

MENUF 1H
OBNUC 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.0 Hz
PW1 5.3 us
DEADT 108.5 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 5402.4 Hz
FLT 2700 Hz
DELAY 74.1 us
ACQTM 3.033 sec
PD 3.967 sec
ADBIT 16.00
RGAIN 16
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
NON Single coupled:PW1_ACI
EXMOD 1H
EXPOM 270.05 MHz
IRNUC 112.00 KHz
IRSET 5800.0 Hz
IRPW 50 us
IRATN 511
DFILE C:\WINNMR98\COMMON_DE
SF THEAT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 20
LKPHS 117
LKSIG 675
CSPED 18 Hz
FILDC
FILDF

C:\WINNMR98\COMMON_DEFAULT.ALS
YF-2005-13 13CNMR

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	0.200 ms
IWT	1.0 sec
POINT	32768
SPO	32768
TIMES	500
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	Blvel,complete,decoupling,Se
IRNUC	1H
IFR	270.05 MHz
IRSET	112.00 KHz
IRFIN	5800.0 Hz
IRPW	50 us
IRATN	511
DFILE	C:\WINNMR98\COMMON_DE
SF	TH5AT
LKSET	64.60 KHz
LKFIN	59.0 Hz
LKLEV	200
LGAIN	23
LKPHS	117
LKSIG	564
CSPED	16 Hz
FILDF

CC1=C(C)C(=C(C1)C2CCCC2OCCCCOCCCC3CCCCC3OC4CCCCC4O)C5=CC=CC=C5CC1=C(C(=O)OCC2CCCCC2)C(=O)N1CCCCC2CCCCC2

CC1=C(C)C(=C(C)C1)C(=C(C)C)C(=O)OCC2(C)CCO2

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
IRFET
IRFIN
IRRPW
IRFATN
DFILE
SF
LKSET
LKFIN
LKLEV
LKAIN
LKPHS
LKSGJ
CSPED
FILDG
FILDG

CCCCC1=C(C)C(=N)C(C1)CCCCOSi(C)(C)CCCCOSi(C)(C)CCCC

C:\My Documents\ÓvXfjNfj\TK043\Auto2BCM_E1_FT.als
TK043 13CNMR

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

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
4000
1
18348.6 Hz
9150 Hz
21.9 us
1.786 sec
1.211 sec
16.00
27
1.20 Hz
0.00
0.00
90.00
100.00
BCM
Bilevel, complete decoupling; Se
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
C:\My Documents\ÓvXfjNfj\TH5AT
64.60 KHz
59.0 Hz
200
23
117
463
17 Hz

MENUF
OBNUC
OFR 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.0 Hz
PW1 5.3 us
DEADT 108.5 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 5402.4 Hz
FLT 2700 Hz
DELAY 74.1 us
ACQTM 3.033 sec
PD 3.967 sec
ADBIT 16.00
RGAIN 18
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
NON
EXMOD
EXPCM
IRNUC
IFR 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRRPW 50 us
IRATN 511
DFILE
SF
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 23
LKPHS 117
LKSIG 782
CSPED 13 Hz
FILDC
FILDF

C:\WINNMR98\COMMON_DEFAULT.ALS
YF-2005-11 13CNMR

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
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
500
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: Se
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
C:\WINNMR98\COMMON_DE
TH5AT
64.60 KHz
59.0 Hz
200
23
117
715
16 Hz

C:\WINNMR98\COMMON_DEFAULT.ALS
YF-2005-12 1HNMR

MENUF	NON
OBNUC	1H
OFR	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.0 Hz
PW1	5.3 us
DEADT	108.5 us
PREDL	0.2000 ms
IWT	1.0 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	5402.4 Hz
FLT	2700 Hz
DELAY	74.1 us
ACQTM	3.033 sec
PD	3.967 sec
ADBIT	16.00
RGAIN	19
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single,coupled:PW1_AC(
IRNUC	1H
IFR	270.05 MHz
IRSET	112.00 KHz
IRFIN	5800.0 Hz
IRRPW	50 us
IRATN	511
DFILE	C:\WINNMR98\COMMON_DE
SF	TH5AT
LKSET	64.40 KHz
LKFIN	78.0 Hz
LKLEV	200
LGAIN	20
LKPHS	117
LKSIG	702
CSPED	14 Hz
FILDC

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 5000
DUMMY 1
FREQU 18348.6 Hz
FLT 9150 Hz
DELAY 21.9 us
ACQTM 1.786 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;Se
IRNUC 1H
IRSET 270.05 MHz
IRFIN 112.00 KHz
IRRPW 5800.0 Hz
IRATN 50 us
DFILE 511
SF C:\My Documents\OlyXfyfNfgf
TH5AT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 20
LKPHS 117
LKSIG 710
CSPED 14 Hz
FILDC
FILDF

MENUF
OBNUC
OFR 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.0 Hz
PW1 5.3 us
DEADT 108.5 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 5402.4 Hz
FLT 2700 Hz
DELAY 74.1 us
ACQTM 3.033 sec
PD 3.967 sec
ADB1 16.00
RGAIN 14
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
NON
EXMOD
EXPCM
IRNUC
IFR 270.05 MHz
IRFIN 112.00 KHz
IRRPW 5800.0 Hz
IRATN 50 us
DFILE 511
SF THSAT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 20
LKPHS 117
LKSIG 734
CSPED 16 Hz
FLDC
FLDF

MENUF
OBNUC
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 1000
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
EXPCM
IRNUC
IRSET 270.05 MHz
IRFIN 112.00 KHz
IRPW 5800.0 Hz
IRATN 50 us
DFILE 511
SF
LKSET 64.80 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 20
LKPHS 117
LKSIG 626
CSPED 16 Hz
FLDC
FILDf

BCM
13C
C:\My Documents\O\X\Y\Nfgf

MENUF NON
OBNUC 1H
OFR 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.0 Hz
PW1 5.3 us
DEADT 108.5 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 5402.4 Hz
FLT 2700 Hz
DELAY 74.1 us
ACQTM 3.033 sec
PD 3.967 sec
ADBIT 16.00
RGAIN 17
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACI
IRNUC 1H
IRSET 270.05 MHz
IRFIN 112.00 KHz
IRPW 5800.0 Hz
IRATN 50 us
DFILE 511
SF C:\WINNMR98\COMMON_DE
THSAT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 23
LKPHS 117
LKSIG 637
CSPED 16 Hz
FILDC
FILDF

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 2400
DUMMY 1
FREQU 18348.6 Hz
FLT 9150 Hz
DELAY 21.9 us
ACQTM 1.786 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;Se
IRNUC 1H
IFR 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRPW 50 us
IRATN 511
DFILE C:\My Documents\0\XfyfNf
SF TH5AT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 27
LKPHS 117
LKSIG 1551
CSPED 16 Hz
FILDC
FILDF

MENUF NON
OBNUC 1H
OFR 270.05 MHz
OBSET 112.00 KHz
OBFN 5800.0 Hz
PW1 5.3 us
DEADT 108.5 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 5402.4 Hz
FLT 2700 Hz
DELAY 74.1 us
ACQTM 3.033 sec
PD 3.967 sec
ADBIT 16.00
RGAIN 18
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
NON
EXMOD NON:Single.coupled:PW1_ACI
EXPCM 1H
IRNUC 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRPW 50 us
IRATN 511
DFILE C:\WINNMR98\COMMON_DE
SF TH5AT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 27
LKPHS 117
LKSIG 639
CSPED 16 Hz
FILDC
FILDF

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 1000
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 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel, complete decoupling; Se
IRNUC 1H
IFR 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRPW 50 us
IRATN 511
DFILE C:\WINNMR98\COMMON_DE
SF TH5AT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 22
LKPHS 117
LKSIG 866
CSPED 17 Hz
FILDC
FILDF

MENUF NON
OBNUC 1H
OFR 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.0 Hz
PW1 5.3 us
DEADT 108.5 us
PREDL 0.2000 ms
IWT 1.0 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 5402.4 Hz
FLT 2700 Hz
DELAY 74.1 us
ACQTM 3.033 sec
PD 3.967 sec
ADBIT 16.00
RGAIN 14
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
NON
EXMOD NON:Single coupled:PW1_Ac
EXPCM 1H
IRNUC 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRRPW 50 us
IRATN 511
DFILE C:\WINNMR98\COMMON_DE
SF TH5AT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 23
LKPHS 117
LKSIG 688
CSPED 16 Hz
FILDC
FILDF

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 1500
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 Blevel complete decoupling;Se
IRNUC 1H
IRF 270.05 MHz
IRSET 112.00 KHz
IRFIN 5800.0 Hz
IRRPW 50 us
IRATN 511
DFILE C:\WINNMR98\COMMON_DE
SF TH5AT
LKSET 64.60 KHz
LKFIN 59.0 Hz
LKLEV 200
LGAIN 20
LKPHS 117
LKSIG 717
CSPED 16 Hz
FILDC
FILDF