Supporting Information for
Lithio Siloles: Facile Synthesis and Applications

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Contents

1. Experimental details and characterization data for all new compounds.
2. Copies of ^1H NMR and ^{13}C NMR spectra for all new compounds.
3. Crystallographic data and lists of bond lengths and angles for **5a**.

1. Experimental details and characterization data for all new compounds.

General Methods. All reactions were conducted under a slightly positive pressure of dry, prepurified nitrogen using standard Schlenk line techniques when appropriate. Unless otherwise noted, all starting materials were commercially available and were used without further purification. Diethyl ether was refluxed and distilled from sodium/benzophenone ketyl under a nitrogen atmosphere. 1,4-Dihalo-1,3-butadiene compounds are prepared by the reported ways. ¹ *t*-BuLi was obtained from Acros Organics. ¹H and ¹³C NMR spectra were recorded at 300 and 75.4 MHz, respectively, in CDCl₃ unless stated otherwise.

A typical procedure for synthesis of siloles **4** via reactions of dilithio-diene **1**:

To a 10mL solution of 1,4-diiodo-1,3-butadiene compound **3** (1.0mmol) in Et₂O at -78°C was added *t*-BuLi (4.0mmol, 1.5M in pentane). After this reaction mixture was stirred at -78°C for 1h, HMPA (2.2mmol) was added and kept at -78°C for 0.5h. Then the reaction mixture was heated to reflux and maintained for 1h (when **1b** and **1c** were used, the time for reflux should be 5h). The above reaction mixture was quenched with water and extracted with Et₂O. The extract was washed with brine and dried over MgSO₄. The solvent was then evaporated in vacuo and the residue thereafter was purified by column chromatograph using silica gel and hexane (for **4d**, aluminium oxide basic and hexane was used instead) to afford title products **4**.

4a: Colorless solid, 89% yield (298 mg); m. p. 57.5~58.8 °C; ¹H NMR (300 MHz, CDCl₃, Me₄Si, 25 °C): δ = -0.13 (s, 9H, CH₃), 0.36 (s, 6H, CH₃), 6.19 (s, 1H, CH), 6.90-6.98 (m, 4H, CH), 7.02-7.07 (m, 3H, CH), 7.10-7.14 (m, 3H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.86 (2 CH₃), 0.93 (3 CH₃), 126.36 (1 CH), 126.47 (1 CH), 127.15 (2 CH), 127.21 (2 CH), 128.16 (2 CH), 128.69 (2 CH), 134.61 (1 CH), 141.21 (1 quat. C), 142.73 (1 quat. C), 142.87 (1 quat. C), 162.19 (1 quat. C), 166.86 (1 quat. C). HRMS calcd. for C₂₁H₂₆Si₂: 334.1573, found 334.1575;

4aD: Colorless solid, D > 98%, 88% yield (300 mg); m. p. 56.9~58.4 °C; ¹H NMR (300 MHz, CDCl₃, Me₄Si, 25 °C): δ = -0.13 (s, 9H, CH₃), 0.36 (s, 6H, CH₃), 6.90-6.98 (m, 4H, CH), 7.02-7.07 (m, 3H, CH), 7.10-7.14 (m, 3H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.86 (2 CH₃), 0.92 (3 CH₃), 126.35 (1 CH), 126.47 (1 CH), 127.15 (2 CH), 127.20 (2 CH), 128.16 (2 CH), 128.69 (2 CH), 134.61 (1 CD), 141.18 (1 quat. C), 142.67 (1 quat. C), 142.86 (1 quat. C), 162.14 (1 quat. C), 166.85 (1 quat. C); HRMS calcd. for C₂₁H₂₅DSi₂: 335.1636, found 335.1642;

4b: Colorless oil, 85% yield (249 mg); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = 0.14 (s, 9H, CH₃), 0.15 (s, 6H, CH₃), 0.94 (t, *J* = 7.2 Hz, 6H, CH₃), 1.34-1.54 (m, 8H, CH₂), 2.26 (t, *J* = 7.5 Hz, 2H, CH₂), 2.37 (t, *J* = 7.5 Hz, 2H, CH₂), 5.81 (s, 1H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.79 (2 CH₃), 1.31 (3 CH₃), 14.04 (1 CH₃), 14.15 (1 CH₃), 22.75 (1 CH₂), 23.35 (1 CH₂), 31.03 (1 CH₂), 32.24 (1 CH₂), 33.53 (1 CH₂), 34.00 (1 CH₂), 128.40 (1 CH), 136.32 (1 quat. C), 162.73 (1 quat. C), 169.12 (1 quat. C); HRMS calcd. for C₁₇H₃₄Si₂: 294.2199, found 294.2189;

4c: Colorless oil, 86% yield (301 mg); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = 0.14 (s, 9H, CH₃), 0.15 (s, 6H, CH₃), 0.90 (t, *J* = 6.9 Hz, 6H, CH₃), 1.26-1.54 (m, 16H, CH₂), 2.25 (t, *J* = 7.2 Hz, 2H, CH₂), 2.36 (t, *J* = 7.5 Hz, 2H, CH₂), 5.81 (s, 1H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.79 (2 CH₃), 1.31 (3 CH₃), 14.07 (1 CH₃), 14.11 (1 CH₃), 22.66 (2 CH₂), 28.78 (1 CH₂), 29.36 (1 CH₂), 29.98 (1 CH₂), 31.36 (1 CH₂), 31.74 (1 CH₂), 31.90 (1 CH₂), 32.58 (1 CH₂), 34.34 (1 CH₂), 128.39 (1 CH), 136.29 (1 quat. C), 162.80 (1 quat. C), 169.17 (1 quat. C); HRMS calcd. for C₂₁H₄₂Si₂: 350.2825, found 350.2819;

4d : Colorless oil, 86% yield (204 mg); ¹H NMR (300MHz, C₆D₆, Me₄Si, 25°C): δ = 0.25 (s, 9H, CH₃), 0.26 (s, 6H, CH₃), 1.35-1.51 (m, 4H, CH₂), 2.47 (t, *J* = 6.0 Hz, 2H, CH₂), 2.55 (t, *J* = 6.0 Hz, 2H, CH₂), 5.82 (s, 1H, CH); ¹³C NMR (75 MHz, C₆D₆, Me₄Si, 25 °C): δ = -3.76 (2 CH₃), 0.89 (3 CH₃), 23.79 (1 CH₂), 24.31 (1 CH₂) 32.39

(2 CH₂), 33.10 (1 CH₂), 128.94 (1 CH), 135.35 (1 quat. C), 159.23 (1 quat. C), 165.65 (1 quat. C); HRMS calcd. for C₁₃H₂₄Si₂: 236.1417, found 236.1413;

4e : Colorless oil, 82% yield (208 mg); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = 0.14 (s, 6H, CH₃), 0.92 (t, *J* = 7.2 Hz, 3H, CH₃), 0.925 (t, *J* = 7.2 Hz, 3H, CH₃), 0.93 (t, *J* = 7.2 Hz, 3H, CH₃), 1.25-1.55 (m, 8H, CH₂), 2.19-2.67 (m, 6H, CH₂), 5.51 (s, 1H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -4.00 (2 CH₃), 14.17 (1 CH₃), 14.37 (1 CH₃), 14.63 (1 CH₃), 22.75 (1 CH₂), 23.41 (1 CH₂), 23.74 (1 CH₂), 29.46 (1 CH₂), 30.63 (1 CH₂), 32.00 (1 CH₂), 33.10 (1 CH₂), 120.39 (1 CH), 141.12 (1 quat. C), 151.72 (1 quat. C), 162.60 (1 quat. C); HRMS calcd. for C₁₆H₃₀Si: 250.2117, found 250.2116;

4f: Colorless oil, 55% yield (118 mg); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = 0.28 (s, 6H, CH₃), 0.96 (t, *J* = 7.5 Hz, 3H, CH₃), 1.37-1.51 (m, 2H, CH₂), 1.59-1.69 (m, 2H, CH₂), 2.02 (t, *J* = 7.2 Hz, 2H, CH₂), 5.92 (s, 1H, CH), 7.19-7.24 (m, 1H, CH), 7.34-7.36 (m, 2H, CH), 7.51 (d, *J* = 6.6 Hz, 1H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.95 (2 CH₃), 14.08 (1 CH₃), 22.74 (1 CH₂), 29.95 (1 CH₂), 32.26 (1 CH₂), 121.09 (1 CH), 125.63 (1 CH), 126.49 (1 CH), 129.35 (1 CH), 131.41 (1 CH), 140.18 (1 quat. C), 149.65 (1 quat. C), 160.58 (1 quat. C); HRMS calcd. for C₁₄H₂₀Si: 216.1334, found 216.1332;

A typical procedure for synthesis of functionalized siloles 5 via reactions of dilithio-diene 1a: To a 10mL solution of **3a** (1.0mmol) in Et₂O at -78°C was added *t*-BuLi (4.0mmol, 1.5M in pentane). After this reaction mixture was stirred at -78°C for 1h, HMPA (2.2mmol) was added and kept at -78°C for 0.5h. Then the reaction mixture was heated to reflux and maintained for 1h. The above reaction mixture was cooled to -78°C and electrophiles were added. After reacting at room temperature for 1h the mixture was quenched with water and extracted with Et₂O. The extract was washed with brine and dried over MgSO₄. The solvent was then evaporated in vacuo

and the residue thereafter was purified by column chromatograph using silica gel (for **5a~d**, Hexane was used; for **5e**, [Et₂O : Hexane : Acetic acid = 10 : 100 : 1] was used; for **5f~h**, [Et₂O : Hexane = 1 : 5] was used) to afford title products **5**.

5a: Colorless crystal, 73% yield (336 mg); m. p. 135.6~136.8 °C; ¹H NMR (300 MHz, CDCl₃, Me₄Si, 25 °C): δ = -0.15 (s, 9H, CH₃), 0.37 (s, 6H, CH₃), 6.84-6.89 (m, 4H, CH), 7.05-7.25 (m, 6H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -4.66 (2 CH₃), 0.75 (3 CH₃), 104.94 (1 quat. C), 126.64(1 CH), 126.78 (1 CH), 127.14 (2 CH), 127.44 (2 CH), 128.55 (2 CH), 128.58 (2 CH), 140.82 (1 quat. C), 142.28 (1 quat. C), 143.26 (1 quat. C), 162.84 (1 quat. C), 167.56 (1 quat. C); HRMS calcd. for C₂₁H₂₅Si₂I: 460.0540, found 460.05444; CCDC 631085.

5b: Colorless solid, 90% yield (366 mg); ¹H NMR (300 MHz, CDCl₃, Me₄Si, 25 °C): δ = -0.17 (s, 18H, CH₃), 0.39 (s, 6H, CH₃), 6.81-6.84 (m, 4H, CH), 7.00-7.04 (m, 6H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -2.87 (2 CH₃), 0.64 (6 CH₃), 125.97 (2 CH), 126.90 (4 CH), 128.69 (4 CH), 142.84 (2 quat. C), 144.72 (2 quat. C), 168.63 (2 quat. C); HRMS calcd. for C₂₄H₃₄Si₃: 406.1968, found 406.1973. The NMR data are identical with the literature. ²

5c: Colorless solid, 82% yield (486 mg); m. p. 102.1~103.7 °C; ¹H NMR (300 MHz, CDCl₃, Me₄Si, 25 °C): δ = -0.17 (s, 9H, CH₃), -0.01 (s, 6H, CH₃), 6.41-6.46 (m, 2H, CH), 6.51-6.56 (m, 2H, CH), 6.84-6.86 (m, 2H, CH), 6.97-6.99 (m, 3H, CH), 7.19-7.27 (m, 10H, CH), 7.45-7.47 (m, 6H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.15 (2 CH₃), 0.68 (3 CH₃), 125.65 (1 CH), 126.01 (1 CH), 126.29 (2 CH), 126.85 (2 CH), 127.33 (6 CH), 128.59 (2 CH), 128.69 (3 CH), 128.84 (2 CH), 135.90 (3 quat. C), 136.17 (6 CH), 138.29 (1 quat. C), 140.97 (1 quat. C), 142.81 (1 quat. C), 148.20 (1 quat. C), 168.35 (1 quat. C), 171.99 (1 quat. C); HRMS calcd. for C₃₉H₄₀Si₃: 592.2438, found 592.2421.

5d: Colorless solid, 85% yield (296 mg); m. p. 49.3~50.4 °C; ¹H NMR (300 MHz,

CDCl₃, Me₄Si, 25 °C): δ = -0.16 (s, 9H, CH₃), 0.33 (s, 6H, CH₃), 1.78 (s, 3H, CH₃), 8.65-8.70 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -4.17 (2 CH₃), 0.96 (3 CH₃), 14.65 (1 CH₃), 125.73 (1 CH), 126.09 (1 CH), 126.96 (2 CH), 127.20 (2 CH), 128.56 (2 CH), 129.40 (2 CH), 136.84 (1 quat. C), 138.87 (1 quat. C), 142.75 (1 quat. C), 143.30 (1 quat. C), 154.37 (1 quat. C), 168.70 (1 quat. C); HRMS calcd. for C₂₂H₂₈Si₂: 348.1730, found 348.1721.

5e: Colorless solid, 70% yield (266 mg); m. p. 141.6~143.8 °C (sublimation); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = -0.14 (s, 9H, CH₃), 0.46 (s, 6H, CH₃), 6.80-6.84 (m, 4H, CH), 7.03-7.24(m, 6H, CH), 11.18 (br, 1H, COOH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -4.00 (2 CH₃), 0.54 (3 CH₃), 126.61 (1 CH), 126.92 (1 CH), 127.09 (2 CH), 127.15 (2 CH), 128.05 (2 CH), 128.75 (2 CH), 134.95 (1 quat. C), 138.11 (1 quat. C), 141.45 (1 quat. C), 148.64 (1 quat. C), 166.82 (1 quat. C), 167.13 (1 quat. C), 173.08 (1 COOH). HRMS calcd. for C₂₂H₂₆O₂Si₂: 378.1471, found 378.1476; IR (neat): ν (C=O) = 1673 cm⁻¹.

5f: Colorless solid, 87% yield (376 mg); m. p. 119.7~121.0 °C; ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = -0.20 (s, 9H, CH₃), 0.47 (s, 6H, CH₃), 1.44-1.69 (m, 11H, CH₂ and OH), 6.76-6.79 (m, 2H, CH), 6.88-7.07 (m, 8H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -2.22 (2 CH₃), 0.76 (3 CH₃), 21.56 (2 CH₂), 25.33 (1 CH₂), 38.86 (2 CH₂), 75.44 (1 CH), 125.84 (1 CH), 126.21 (1 CH), 126.78 (2 CH), 127.33 (2 CH), 128.69 (2 CH), 129.51 (2 CH), 137.78 (1 quat. C), 140.38 (1 quat. C), 142.70 (1 quat. C), 151.93 (1 quat. C), 153.75 (1 quat. C), 168.43 (1 quat. C). HRMS calcd. for C₂₇H₃₆OSi₂: 432.2305, found 432.2298.

5g: Colorless solid, 76% yield (343 mg); m. p. 142.9~144.7 °C; ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = -0.08 (s, 9H, CH₃), 0.45 (s, 6H, CH₃), 2.23 (s, 3H, CH₃), 6.77-6.83 (m, 6H, CH), 6.94-6.97 (m, 2H, CH), 6.99-7.02 (m, 2H, CH), 7.08-7.10 (m, 2H, CH), 7.58-7.60 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -3.63 (2 CH₃), 0.79 (3 CH₃), 21.54 (1 CH₃), 126.70 (2 CH), 126.97 (2 CH),

127.24 (2 CH), 128.68 (2 CH), 128.69 (2 CH), 128.72 (2 CH), 129.22 (2 CH), 134.59 (1 quat. C), 137.87 (1 quat. C), 141.70 (1 quat. C), 143.05 (1 quat. C), 143.36 (1 quat. C), 148.47 (1 quat. C), 157.13 (1 quat. C), 166.85 (1 quat. C), 199.43 (1 ketone C=O); HRMS calcd. for C₂₉H₃₂OSi₂: 452.1992, found 452.1985; IR (neat): ν (C=O) = 1642 cm⁻¹.

5h: colorless oil, 81% yield (420 mg); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = -0.18 (s, 9H, CH₃), -0.04 (s, 3H, CH₃), 0.50 (s, 3H, CH₃), 5.47 (s, 1H, CH), 6.87-7.07 (m, 8H, CH), 7.22-7.44 (m, 7H, CH), 7.50 (d, J = 7.2 Hz, 2H, CH), 7.57 (d, J = 7.2 Hz, 2H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = -2.85 (1 CH₃), -2.42 (1 CH₃), 0.81 (3CH₃), 73.48 (1 CH), 126.22 (4 CH), 126.35 (1 CH), 126.74 (2 CH), 126.98 (4 CH), 127.01 (2 CH), 127.13 (1 CH), 127.54 (1 CH), 128.61 (2 CH), 128.72 (2 CH), 138.57 (1 quat. C), 139.65 (1 quat. C), 140.82 (1 quat. C), 140.94 (1 quat. C), 142.61 (1 quat. C), 143.20 (1 quat. C), 147.27 (1 quat. C), 156.15 (1 quat. C), 167.10 (1 quat. C); HRMS calcd. for C₃₄H₃₆OSi₂: 516.2305, found 516.2316.

11: colorless crystal, 83% yield (164 mg); ¹H NMR (300MHz, CDCl₃, Me₄Si, 25°C): δ = 1.52 (d, J = 6.3 Hz, 3H, CH₃), 2.00 (br, 1H, OH), 4.92 (q, J = 6.3 Hz, 1H, CH), 7.22-7.44 (m, 5H, CH), 7.55-7.59 (m, 4H, CH); ¹³C NMR (75 MHz, CDCl₃, Me₄Si, 25 °C): δ = 25.10 (1 CH₃), 70.10 (1 CH), 125.82 (2 CH), 127.03 (1 CH), 127.20 (2 CH), 127.22 (2 CH), 128.72 (2 CH), 140.37 (1 quat. C), 140.80 (1 quat. C), 144.78 (1 quat. C). The NMR data are identical with the literature.³

References

- 1) Representative methods for 1,4-dihalo-1,3-diene derivatives, see: (a) Negishi, E.; Cederbaum, F. E.; Takahashi, T. *Tetrahedron Lett.* **1986**, 27, 2829-2832. (b) Buchwald, S. L.; Nielsen, R. B. *J. Am. Chem. Soc.* **1989**, 111, 2870-2874. (c) Xi, C.; Huo, S.; Afifi, T. H.; Hara, R.; Takahashi, T. *Tetrahedron Lett.* **1997**, 38, 4099-4102. (d) Yamaguchi, S.; Jin, R.; Tamao, K.;

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2) Tamao, K.; Yamaguchi, S.; Shiro, M. *J. Am. Chem. Soc.* **1994**, *116*, 11715-11722

3) Wang, Z.; Fang, H.; Xi, Z. *Tetrahedron Lett.* **2005**, *46*, 499-501.

2. Copies of ^1H NMR and ^{13}C NMR spectra for all new compounds.

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0.62 Hz

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SCANS 32
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 12

4b

D:\Wang chao\tms\4-100\C.a.1

COMNT
DATIM Fri Dec 08 19:53:25 2006
OBNUC 13C
EXMOD BCM
OBFREQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 848
ACQTM 1.606 sec
PD 1.394 sec
FW 5.0 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

4b

D:\Wang chao\tms\4-100\HTMS

DFILE COMNT
 DATIM Fri Dec 08 21:39:13 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 12

D:\Wang chao\tms\4-100\CTMS

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Fri Dec 08 21:36:15 2006
13C
BCM
75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
1256
1.606 sec
1.394 sec
5.0 us
1H
23.9 C
CDCl3
0.00 ppm
0.62 Hz
26

33.997
33.535
32.241
31.030
23.357
22.747
14.151
14.036
1.310
0.000
-3.791

77.430
77.010
76.590

128.405
136.325

162.731
169.118

PPM

0
50
100
150
200

D:\Wang chao\tms\4-HEX\H.al

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Sat Dec 23 20:14:02 2006

1H

NON

300.40 MHz

130.00 KHz

1150.0 Hz

32768

6013.2 Hz

16

5.449 sec

1.551 sec

5.8 us

1H

24.3 c

CDCL3

0.00 ppm

0.62 Hz

13

4c

D:\Wang chao\tms\4-HEX\C-32

COMNT
DATIM Sat Dec 23 20:11:01 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 320
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 24.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

4c

D:\Wang chao\tms\4-HEX\H-TM

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

0.000
0.140
0.148
0.873
0.896
0.919
1.257
1.316
1.400
1.469
1.492
1.519
1.544
2.228
2.253
2.276
2.331
2.355
2.380

5.809

7.251

Sat Dec 23 21:03:09 2006

1H
NON

300.40 MHz
130.00 KHz
1150.0 Hz
32768
6013.2 Hz
32
5.449 sec
1.551 sec
5.8 us
1H
24.1 C
CDCl3
0.00 ppm
0.09 Hz
12

D:\Wang chao\tms\4-HEX\C-tm
 COMNT Sat Dec 23 20:43:46 2006
 DATIM 13C
 OBNUC BCM
 EXMOD
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 440
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 24.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

D:\Wang chao\tms\4-GUO\C6D6

COMNT
 DATIM Fri Dec 22 20:56:56 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 24
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.8 c
 SLVNT C6D6
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 14

4d

D:\Wang chao\tms\4-GUO\C6D6

COMNT
DATIM Fri Dec 22 20:52:57 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 728
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 24.3 C
SLVNT C6D6
EXREF 128.00 ppm
BF 0.62 Hz
RGAIN 24

4d

D:\Wang chao\tms\4-GUO\C6D6
 COMNT Fri Dec 22 21:48:50 2006
 DATIM 1H
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.9 c
 SLVNT C6D6
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 14

D:\Wang chao\tms\4-GUO\C6D6

DFILE COMNT
 DATIM Fri Dec 22 21:36:27 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 624
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 24.3 C
 SLVNT C6D6
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 24

0.890
 0.000
 -3.758

33.098
 32.390
 24.313
 23.794

135.352
 128.940
 128.331
 128.017
 127.696

165.649
 159.228

4d
 (with SiMe₄)

D:\Wang chao\tms\SUNHUI\BAS

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQ
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Mon Dec 11 14:03:06 2006

1H
NON
300.40 MHz
130.00 KHz
1150.0 Hz
32768
6013.2 Hz
16
5.449 sec
1.551 sec
5.8 us
1H
23.8 c
CDCL3
0.00 ppm
0.09 Hz
11

4e

D:\Wang chao\tms\SUNHUI\BAS

CONNT
DATIM Mon Dec 11 13:32:15 2006
OBNUC 13C
EXMOD BCM
OBFFQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQ 20408.1 Hz
SCANS 1488
ACQTM 1.606 sec
PD 1.394 sec
PW 5.0 us
IRNUC 1H
CTEMP 24.3 C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.62 Hz
RGAIN 26

4e

D:\Wang chao\tms\SUNHUI\BAS

Mon Dec 11 15:44:16 2006

DFILE COMNT
 DATIM 1H
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.7 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 11

D:\Wang chao\tms\SUNHUI\BAS

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Mon Dec 11 15:04:07 2006

13C
BCM
75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
1056
1.606 sec
1.394 sec
5.0 us
1H
24.3 C
CDCl3
0.00 ppm
0.62 Hz
26

3.956
0.000
14.167
14.365
14.629
22.747
23.406
23.744
29.456
30.634
32.002
33.107

76.598
77.018
77.438

120.394
141.122
151.720
162.599

4e
(with SiMe₄)

DFILE D:\LUO QIAN\1-112\H.als
 COMNT
 DATIM Fri Dec 29 21:25:15 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 15

D:\LUO QIAN\1-112\C-480.als

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Fri Dec 29 21:22:05 2006
13C
BCM

75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
480
1.606 sec
1.394 sec
5.0 us
1H
23.5 c
CDCL3
77.00 ppm
0.62 Hz
26

D:\LUO QIAN\1-112\HTMS.als
 COMNT Fri Dec 29 22:19:28 2006
 DATIM 1H
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 22.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 14

4f
(with SiMe₄)

D:\LUO QIAN\1-112\C-416-TMS
 COMNT Fri Dec 29 21:58:59 2006
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 416
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 23.5 c
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 0.62 Hz
 BF 26
 RGAIN

4f
(with SiMe₄)

D:\Wang chao\Di Li\E-JOC\4-

DFILE
COMNT
DATIM Sat Dec 30 20:50:08 2006
OBNUC 1H
EXMOD NON
OBFREQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 16
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 17

D:\Wang chao\Di Li\E-JOC\4-
 COMNT
 DATIM Sat Dec 30 20:46:11 2006
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 560
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 23.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 26

5a

D:\Wang chao\Di Li\E-JOC\4-

CONNT
 DATIM Sat Dec 30 21:40:50 2006
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 22.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 15

5a
 (with SiMe₄)

D:\Wang chao\DI Li\E-JOC\4-

COMNT
DATIM Sat Dec 30 21:36:41 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 800
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

0.732
-0.018
-4.683

77.404
76.984
76.563

104.923

126.623
126.763
127.126
127.422
128.535
128.560
140.807
142.265
143.246

162.820
167.543

5a
(with SiMe₄)

D:\LUO QIAN\1-95\H.als
 COMNT
 DATIM Thu Nov 30 14:58:02 2006
 OBNUC 1H
 EXMOD NON

OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 15

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRQ
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

5b

D:\LUO QIAN\1-95\C-888.als

COMNT
DATIM
OBNUC
EXMOD
OBFQ
OBSET
OBFIN
POINT
FREQ
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Thu Nov 30 14:29:00 2006
13C
BCM

75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
888
1.606 sec
1.394 sec
5.0 us
1H
24.5 C
CDCL3
77.00 ppm
0.62 Hz
26

5b

D:\LUO QIAN\1-95\HTMS.als

COMNT
DATIM Thu Nov 30 15:46:01 2006
OBNUC 1H
EXMOD NON
OBFREQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 16
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 24.0 C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 13

5b
(with SiMe₄)

D:\LUO QIAN\1-95\C-660TMS.a

DFILE COMNT
 DATIM Thu Nov 30 15:43:04 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 660
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 24.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

5b
(with SiMe₄)

D:\LUO QIAN\1-96\H-2.als

DFILE

COMNT

Thu Dec 07 15:19:30 2006

DATIM

1H

OBNUC

NON

EXMOD

300.40 MHz

OBFREQ

130.00 KHz

OBFSET

1150.0 Hz

OBFIN

32768

POINT

6013.2 Hz

FREQU

16

SCANS

5.449 sec

ACQTM

1.551 sec

PD

5.8 us

PW1

1H

IRNUC

22.8 c

CTEMP

CDCL3

SLVNT

0.00 ppm

EXREF

0.09 Hz

BF

16

RGAIN

5c

D:\LUO QIAN\1-96\C320.als

DFILE
COMNT
DATIM Wed Dec 06 21:13:22 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 320
ACQTM 1.606 sec
PD 1.394 sec
PWL 5.0 us
IRNUC 1H
CTEMP 24.6 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 27

5c

D:\LUO QIAN\1-96\HTMS.als
 COMNT Wed Dec 06 22:23:33 2006
 DATIM 1H
 EXMOD NON
 OBNUC 300.40 MHz
 EXFRQ 130.00 KHz
 OBSET 1150.0 Hz
 OBFIN 32768
 POINT 6013.2 Hz
 FREQU 16
 SCANS 5.449 sec
 ACQTM 1.551 sec
 PD 5.8 us
 PW1 1H
 IRNUC 24.1 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.09 Hz
 BF 11
 RGAIN

5c
(with SiMe₄)

D:\LUO QIAN\1-96\C384TMS.a1

COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Wed Dec 06 22:02:01 2006

13C
BCM
75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
384
1.606 sec
1.394 sec
5.0 us
1H
24.8 c
CDCL3
0.00 ppm
0.62 Hz
26

0.684
0.000
-3.148

77.405
76.985
76.565

171.995
168.352
148.201
142.811
140.973
138.286
136.168
135.896
128.841
128.693
128.594
127.333
126.847
126.287
126.006
125.652

5c
(with SiMe₄)

D:\LUO QIAN\1-93\AGAIN\C132

DFILE
COMNT

DATIM Mon Nov 27 15:32:52 2006

OBNUC 13C

EXMOD BCM

OBFRQ 75.45 MHz

OBSET 124.00 KHz

OBFIN 1840.0 Hz

POINT 32768

FREQU 20408.1 Hz

SCANS 1332

ACQTM 1.606 sec

PD 1.394 sec

PW1 5.0 us

IRNUC 1H

CTEMP 24.0 C

SLVNT CDCL3

EXREF 77.00 ppm

BF 0.62 Hz

RGAIN 26

5d

D:\LUO QIAN\1-93\AGAIN\HTMS

COMNT
DATIM Mon Nov 27 17:26:44 2006
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 16
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 15

5d
(with SiMe₄)

D:\LUO QIAN\1-93\AGAIN\CL28

COMET
DATIM
OBNUC
EXMOD
OBFRC
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Mon Nov 27 17:23:33 2006
13C
BCM

75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
1280
1.606 sec
1.394 sec
5.0 us
1H
24.0 c
CDCL3
0.00 ppm
0.62 Hz
26

5d
(with SiMe₄)

D:\Wang chao\tms\co2\H.als

COMNT
DATIM Mon Dec 18 21:12:42 2006

OBNUC 1H
EXMOD NON

OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz

POINT 32768
FREQU 6013.2 Hz

SCANS 32
ACQTM 5.449 sec

PD 1.551 sec
PW1 5.8 us

IRNUC 1H
CTEMP 22.6 c

SLVNT CDCL3
EXREF 0.00 ppm

BF 0.09 Hz
RGAIN 15

5e

D:\Wang chao\tms\co2\c-1-92

DFILE
COMNT
DATIM Mon Dec 25 17:21:19 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 920
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

5e

D:\Wang chao\tms\co2\HTMS.a

COMNT
DATIM Mon Dec 18 22:25:52 2006
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 16
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.62 Hz
RGAIN 12

0.148
0.000
0.459

6.796
6.813
6.828
6.840
7.031
7.046
7.049
7.053
7.067
7.236

11.175

5e
(with SiMe₄)

D:\Wang chao\tms\co2\c-864.

DFILE COMNT
 DATIM Mon Dec 18 22:05:10 2006
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 864
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 23.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

5e
 (with SiMe₄)

D:\Wang chao\tms\4-99\H.als

COMNT
 DATIM Sat Dec 02 21:04:11 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.09 Hz
 RGAIN 12

D:\Wang chao\tms\4-99\C560.

COMNT
DATIM Sat Dec 02 20:36:37 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 560
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

D:\Wang chao\tms\4-99\HTMS.

COMNT
DATIM Sat Dec 02 22:06:52 2006
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 16
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 11

D:\Wang chao\tms\4-99\C560T

COMNT
DATIM Sat Dec 02 21:39:14 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 560
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.62 Hz
RGAIN 26

D:\Wang chao\Di Li\E-JOC\4-

COMPT
DATIM Sat Sep 30 14:10:30 2006
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 32
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 15

D:\Wang chao\Di Li\E-JOC\4-

COMNT
DATIM Sat Sep 30 13:41:20 2006
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQ 20408.1 Hz
SCANS 592
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.62 Hz
RGAIN 26

D:\Wang chao\DI Li\E-JOC\4-

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Sat Sep 30 14:36:53 2006
13C
BCM

75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
320
1.606 sec
1.394 sec
5.0 us
1H
23.1 c
CDCL3
0.00 ppm
0.62 Hz
25

0.791
0.000
-3.635

21.535

76.631
77.051
77.471

126.699
126.971
127.243
128.677
128.693
128.726
129.221
129.558
134.594
137.874
141.698
143.050
143.363
148.473
157.135
166.852

199.431

D:\Wang chao\tms\4-92\BU HU

COMNT

DATIM Tue Nov 21 19:44:02 2006

OBNUC 13C

EXMOD BCM

OBFRQ 75.45 MHz

OBSET 124.00 KHz

OBFIN 1840.0 Hz

POINT 32768

FREQU 20408.1 Hz

SCANS 516

ACQTM 1.606 sec

PD 1.394 sec

PW1 5.0 us

IRNUC 1H

CTEMP 25.1 c

SLVNT CDCL3

EXREF 77.00 ppm

BF 0.62 Hz

RGAIN 26

D:\Wang chao\tms\4-92\BU HU

COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Wed Nov 22 20:56:28 2006
1H
NON

300.40 MHz
130.00 KHz
1150.0 Hz
32768
6013.2 Hz
16
5.449 sec
1.551 sec
5.8 us
1H
24.2 c
CDCL3
0.00 ppm
0.09 Hz
12

D:\Wang chao\tms\4-92\BU HU

DFILE COMNT
 DATIM Wed Nov 22 20:21:58 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 600
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 24.8 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

11

D:\Wang chao\tms\4-92\LIAN

COMET
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Tue Nov 21 19:15:20 2006
1H
NON

300.40 MHz
130.00 KHz
1150.0 Hz
32768
6013.2 Hz
16
5.449 sec
1.551 sec
5.8 us
1H
25.0 C
CDCL3
0.00 ppm
0.09 Hz
14

D:\Wang chao\tms\4-92\LIAN

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Tue Nov 21 19:10:11 2006

13C

BCM

75.45 MHz

124.00 KHz

1840.0 Hz

32768

20408.1 Hz

308

1.606 sec

1.394 sec

5.0 us

1H

25.5 c

CDCL3

77.00 ppm

0.62 Hz

26

-0.035

25.094

77.420
77.000
76.571
70.094

144.779
140.807
140.370
128.724
127.216
127.200
127.027
125.815

11

3. Crystallographic data and lists of bond lengths and angles for 5a.

Table 1. Crystal data and structure refinement for 5a.

Identification code	a
Empirical formula	C ₂₁ H ₂₅ I Si ₂
Formula weight	460.49
Temperature	293(2) K
Measurement device	Rigaku RAXIS RAPID IP
Measurement method	\W
Wavelength	0.71073 Å
Program for data collection	Rapid-AUTO (Rigaku, 2000)
Program for cell refinement	Rapid-AUTO
Program for data reduction	CrystalStructure (Rigaku/MSK, 2000)
Program for structure solution	SHELXS-97, (Sheldrick, 1997)
Program for structure refinement	SHELXL-97, (Sheldrick, 1997)
Program for molecular graphics	Siemens SHELXTL V4.2, (Sheldrick, 1990)
Program for publication material	SHELXL-97, (Sheldrick, 1997)
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 27.932(6) Å alpha = 90 deg. b = 11.207(2) Å beta = 111.80(3) deg. c = 15.052(3) Å gamma = 90 deg.
Volume	4374.9(15) Å ³
Refls. No. for cell measurement	11792
Theta range for cell measurement	2.28 to 25.03 deg.
Z, Calculated density	8, 1.398 Mg/m ³
Absorption coefficient	1.574 mm ⁻¹
F(000)	1856
Crystal shape / Crystal colour	block / colourless
Crystal size	0.35 x 0.30 x 0.25 mm
Theta range for data collection	2.28 to 25.03 deg.
Limiting indices	-33<=h<=33, -13<=k<=13, -17<=l<=16
Reflections collected / unique	11792 / 3826 [R(int) = 0.0625]
Reflections with I>2sigma(I)	2894
Completeness to theta = 25.03	99.1 %
Decay correction (%)	0
Absorption correction	Empirical
Max. and min. transmission	0.675 and 0.576
Method for primary solution	direct
Method for secondary solution	difmap
Hydrogen addition / treatment	geom / mixed

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3826 / 0 / 222
Goodness-of-fit on F^2	1.013
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0401$, $wR2 = 0.0935$
R indices (all data)	$R1 = 0.0530$, $wR2 = 0.0968$
Largest diff. peak and hole	0.706 and $-1.142 \text{ e. \AA}^{-3}$
Max. and mean shift/sigma	0.000 and 0.000

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5a.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
I (1)	4488 (1)	4373 (1)	932 (1)	54 (1)
Si (1)	3622 (1)	5395 (1)	1952 (1)	43 (1)
Si (2)	3041 (1)	7703 (1)	2526 (1)	51 (1)
C (1)	3493 (1)	7030 (4)	2010 (3)	42 (1)
C (2)	3780 (1)	7606 (3)	1582 (3)	38 (1)
C (3)	4127 (1)	6857 (3)	1232 (2)	35 (1)
C (4)	4093 (1)	5689 (3)	1373 (3)	40 (1)
C (5)	3777 (1)	8921 (4)	1445 (3)	39 (1)
C (6)	3335 (2)	9500 (4)	844 (3)	52 (1)
C (7)	3327 (2)	10718 (4)	731 (4)	63 (1)
C (8)	3756 (2)	11383 (4)	1227 (4)	63 (1)
C (9)	4191 (2)	10836 (4)	1804 (4)	58 (1)
C (10)	4208 (2)	9601 (4)	1921 (3)	46 (1)
C (11)	4443 (1)	7424 (3)	741 (3)	38 (1)
C (12)	4211 (2)	8044 (4)	-117 (3)	54 (1)
C (13)	4509 (2)	8537 (5)	-576 (3)	69 (1)
C (14)	5033 (2)	8438 (5)	-185 (4)	74 (2)
C (15)	5265 (2)	7841 (4)	652 (4)	63 (1)
C (16)	4977 (2)	7327 (4)	1121 (3)	49 (1)
C (17)	3049 (2)	4595 (4)	1107 (4)	67 (1)
C (18)	3908 (2)	4579 (4)	3091 (3)	63 (1)
C (19)	3008 (2)	6688 (5)	3481 (4)	81 (2)
C (20)	2377 (2)	7798 (6)	1591 (4)	82 (2)
C (21)	3249 (2)	9197 (4)	3087 (4)	69 (1)

Table 3. Bond lengths [Å] and angles [deg] for 5a.

I (1)–C (4)	2.093 (4)
Si (1)–C (18)	1.844 (4)
Si (1)–C (4)	1.855 (4)
Si (1)–C (17)	1.863 (5)
Si (1)–C (1)	1.876 (4)
Si (2)–C (19)	1.860 (5)
Si (2)–C (21)	1.869 (5)
Si (2)–C (20)	1.869 (5)
Si (2)–C (1)	1.871 (4)
C (1)–C (2)	1.362 (5)
C (2)–C (5)	1.489 (5)
C (2)–C (3)	1.518 (5)
C (3)–C (4)	1.336 (5)
C (3)–C (11)	1.489 (5)
C (5)–C (10)	1.379 (5)
C (5)–C (6)	1.389 (6)
C (6)–C (7)	1.375 (6)
C (6)–H (6)	0.9300
C (7)–C (8)	1.372 (7)
C (7)–H (7)	0.9300
C (8)–C (9)	1.349 (6)
C (8)–H (8)	0.9300
C (9)–C (10)	1.394 (6)
C (9)–H (9)	0.9300
C (10)–H (10)	0.9300
C (11)–C (16)	1.389 (5)
C (11)–C (12)	1.396 (5)
C (12)–C (13)	1.380 (6)
C (12)–H (12)	0.9300
C (13)–C (14)	1.364 (7)
C (13)–H (13)	0.9300
C (14)–C (15)	1.359 (7)
C (14)–H (14)	0.9300
C (15)–C (16)	1.378 (6)
C (15)–H (15)	0.9300
C (16)–H (16)	0.9300
C (17)–H (17A)	0.9600
C (17)–H (17B)	0.9600
C (17)–H (17C)	0.9600
C (18)–H (18A)	0.9600

C (18)–H (18B)	0.9600
C (18)–H (18C)	0.9600
C (19)–H (19A)	0.9600
C (19)–H (19B)	0.9600
C (19)–H (19C)	0.9600
C (20)–H (20A)	0.9600
C (20)–H (20B)	0.9600
C (20)–H (20C)	0.9600
C (21)–H (21A)	0.9600
C (21)–H (21B)	0.9600
C (21)–H (21C)	0.9600
C (18)–Si (1)–C (4)	112.75 (19)
C (18)–Si (1)–C (17)	111.2 (2)
C (4)–Si (1)–C (17)	110.2 (2)
C (18)–Si (1)–C (1)	117.7 (2)
C (4)–Si (1)–C (1)	91.78 (17)
C (17)–Si (1)–C (1)	111.70 (19)
C (19)–Si (2)–C (21)	107.2 (2)
C (19)–Si (2)–C (20)	107.3 (3)
C (21)–Si (2)–C (20)	110.1 (3)
C (19)–Si (2)–C (1)	107.9 (2)
C (21)–Si (2)–C (1)	113.84 (19)
C (20)–Si (2)–C (1)	110.1 (2)
C (2)–C (1)–Si (2)	127.7 (3)
C (2)–C (1)–Si (1)	106.4 (3)
Si (2)–C (1)–Si (1)	125.8 (2)
C (1)–C (2)–C (5)	124.1 (3)
C (1)–C (2)–C (3)	117.8 (3)
C (5)–C (2)–C (3)	118.1 (3)
C (4)–C (3)–C (11)	126.0 (3)
C (4)–C (3)–C (2)	113.2 (3)
C (11)–C (3)–C (2)	120.7 (3)
C (3)–C (4)–Si (1)	110.7 (3)
C (3)–C (4)–I (1)	124.3 (3)
Si (1)–C (4)–I (1)	124.9 (2)
C (10)–C (5)–C (6)	118.2 (4)
C (10)–C (5)–C (2)	121.1 (4)
C (6)–C (5)–C (2)	120.7 (4)
C (7)–C (6)–C (5)	120.9 (4)
C (7)–C (6)–H (6)	119.5
C (5)–C (6)–H (6)	119.5
C (8)–C (7)–C (6)	120.1 (4)
C (8)–C (7)–H (7)	120.0

C (6)–C (7)–H (7)	120. 0
C (9)–C (8)–C (7)	119. 9 (4)
C (9)–C (8)–H (8)	120. 0
C (7)–C (8)–H (8)	120. 0
C (8)–C (9)–C (10)	120. 8 (4)
C (8)–C (9)–H (9)	119. 6
C (10)–C (9)–H (9)	119. 6
C (5)–C (10)–C (9)	120. 1 (4)
C (5)–C (10)–H (10)	120. 0
C (9)–C (10)–H (10)	120. 0
C (16)–C (11)–C (12)	118. 7 (4)
C (16)–C (11)–C (3)	120. 4 (3)
C (12)–C (11)–C (3)	120. 9 (3)
C (13)–C (12)–C (11)	120. 2 (4)
C (13)–C (12)–H (12)	119. 9
C (11)–C (12)–H (12)	119. 9
C (14)–C (13)–C (12)	120. 1 (5)
C (14)–C (13)–H (13)	120. 0
C (12)–C (13)–H (13)	120. 0
C (15)–C (14)–C (13)	120. 4 (4)
C (15)–C (14)–H (14)	119. 8
C (13)–C (14)–H (14)	119. 8
C (14)–C (15)–C (16)	120. 9 (5)
C (14)–C (15)–H (15)	119. 6
C (16)–C (15)–H (15)	119. 6
C (15)–C (16)–C (11)	119. 8 (4)
C (15)–C (16)–H (16)	120. 1
C (11)–C (16)–H (16)	120. 1
Si (1)–C (17)–H (17A)	109. 5
Si (1)–C (17)–H (17B)	109. 5
H (17A)–C (17)–H (17B)	109. 5
Si (1)–C (17)–H (17C)	109. 5
H (17A)–C (17)–H (17C)	109. 5
H (17B)–C (17)–H (17C)	109. 5
Si (1)–C (18)–H (18A)	109. 5
Si (1)–C (18)–H (18B)	109. 5
H (18A)–C (18)–H (18B)	109. 5
Si (1)–C (18)–H (18C)	109. 5
H (18A)–C (18)–H (18C)	109. 5
H (18B)–C (18)–H (18C)	109. 5
Si (2)–C (19)–H (19A)	109. 5
Si (2)–C (19)–H (19B)	109. 5
H (19A)–C (19)–H (19B)	109. 5
Si (2)–C (19)–H (19C)	109. 5

H(19A)–C(19)–H(19C)	109.5
H(19B)–C(19)–H(19C)	109.5
Si(2)–C(20)–H(20A)	109.5
Si(2)–C(20)–H(20B)	109.5
H(20A)–C(20)–H(20B)	109.5
Si(2)–C(20)–H(20C)	109.5
H(20A)–C(20)–H(20C)	109.5
H(20B)–C(20)–H(20C)	109.5
Si(2)–C(21)–H(21A)	109.5
Si(2)–C(21)–H(21B)	109.5
H(21A)–C(21)–H(21B)	109.5
Si(2)–C(21)–H(21C)	109.5
H(21A)–C(21)–H(21C)	109.5
H(21B)–C(21)–H(21C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5a.
The anisotropic displacement factor exponent takes the form:
 $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
I (1)	59 (1)	39 (1)	74 (1)	−6 (1)	36 (1)	3 (1)
Si (1)	42 (1)	36 (1)	56 (1)	3 (1)	26 (1)	−2 (1)
Si (2)	47 (1)	51 (1)	66 (1)	−3 (1)	34 (1)	2 (1)
C (1)	41 (2)	39 (2)	49 (2)	−2 (2)	21 (2)	−3 (2)
C (2)	38 (2)	34 (2)	45 (2)	−4 (2)	19 (2)	−2 (2)
C (3)	36 (2)	36 (2)	35 (2)	0 (2)	15 (2)	0 (2)
C (4)	43 (2)	39 (2)	45 (2)	−4 (2)	22 (2)	−1 (2)
C (5)	46 (2)	35 (2)	45 (2)	−1 (2)	25 (2)	2 (2)
C (6)	54 (2)	41 (3)	59 (3)	−1 (2)	19 (2)	0 (2)
C (7)	67 (3)	47 (3)	73 (3)	15 (2)	23 (3)	14 (3)
C (8)	77 (3)	34 (2)	84 (3)	5 (2)	39 (3)	1 (2)
C (9)	68 (3)	44 (3)	73 (3)	−12 (2)	39 (3)	−16 (2)
C (10)	45 (2)	47 (3)	50 (2)	−1 (2)	22 (2)	−4 (2)
C (11)	53 (2)	31 (2)	38 (2)	−6 (2)	26 (2)	−2 (2)
C (12)	64 (3)	57 (3)	50 (3)	4 (2)	31 (2)	6 (2)
C (13)	106 (4)	59 (3)	59 (3)	13 (3)	50 (3)	9 (3)
C (14)	109 (4)	54 (3)	95 (4)	−8 (3)	81 (4)	−15 (3)
C (15)	62 (3)	59 (3)	85 (4)	−17 (3)	48 (3)	−17 (2)
C (16)	44 (2)	49 (3)	57 (3)	−8 (2)	22 (2)	−4 (2)
C (17)	54 (3)	61 (3)	87 (4)	−9 (3)	28 (3)	−5 (2)
C (18)	66 (3)	62 (3)	68 (3)	17 (3)	32 (3)	3 (2)
C (19)	93 (4)	84 (4)	94 (4)	7 (3)	68 (3)	3 (3)
C (20)	48 (3)	97 (5)	101 (4)	−9 (3)	27 (3)	6 (3)
C (21)	78 (3)	64 (3)	82 (4)	−18 (3)	49 (3)	−3 (3)

Table 5. Torsion angles [deg] for 5a.

C(19)–Si(2)–C(1)–C(2)	–153.2(4)
C(21)–Si(2)–C(1)–C(2)	–34.3(4)
C(20)–Si(2)–C(1)–C(2)	89.9(4)
C(19)–Si(2)–C(1)–Si(1)	28.8(3)
C(21)–Si(2)–C(1)–Si(1)	147.7(3)
C(20)–Si(2)–C(1)–Si(1)	–88.0(3)
C(18)–Si(1)–C(1)–C(2)	119.3(3)
C(4)–Si(1)–C(1)–C(2)	2.3(3)
C(17)–Si(1)–C(1)–C(2)	–110.2(3)
C(18)–Si(1)–C(1)–Si(2)	–62.4(3)
C(4)–Si(1)–C(1)–Si(2)	–179.3(3)
C(17)–Si(1)–C(1)–Si(2)	68.1(3)
Si(2)–C(1)–C(2)–C(5)	–0.5(6)
Si(1)–C(1)–C(2)–C(5)	177.8(3)
Si(2)–C(1)–C(2)–C(3)	179.0(3)
Si(1)–C(1)–C(2)–C(3)	–2.7(4)
C(1)–C(2)–C(3)–C(4)	1.8(5)
C(5)–C(2)–C(3)–C(4)	–178.8(4)
C(1)–C(2)–C(3)–C(11)	178.8(3)
C(5)–C(2)–C(3)–C(11)	–1.7(5)
C(11)–C(3)–C(4)–Si(1)	–176.6(3)
C(2)–C(3)–C(4)–Si(1)	0.3(4)
C(11)–C(3)–C(4)–I(1)	0.4(6)
C(2)–C(3)–C(4)–I(1)	177.2(2)
C(18)–Si(1)–C(4)–C(3)	–122.7(3)
C(17)–Si(1)–C(4)–C(3)	112.4(3)
C(1)–Si(1)–C(4)–C(3)	–1.5(3)
C(18)–Si(1)–C(4)–I(1)	60.4(3)
C(17)–Si(1)–C(4)–I(1)	–64.5(3)
C(1)–Si(1)–C(4)–I(1)	–178.4(2)
C(1)–C(2)–C(5)–C(10)	113.2(4)
C(3)–C(2)–C(5)–C(10)	–66.2(5)
C(1)–C(2)–C(5)–C(6)	–65.7(6)
C(3)–C(2)–C(5)–C(6)	114.9(4)
C(10)–C(5)–C(6)–C(7)	–0.4(6)
C(2)–C(5)–C(6)–C(7)	178.5(4)
C(5)–C(6)–C(7)–C(8)	–1.0(8)
C(6)–C(7)–C(8)–C(9)	1.8(8)
C(7)–C(8)–C(9)–C(10)	–1.2(7)
C(6)–C(5)–C(10)–C(9)	1.0(6)

C(2)–C(5)–C(10)–C(9)	–177.9(4)
C(8)–C(9)–C(10)–C(5)	–0.2(7)
C(4)–C(3)–C(11)–C(16)	–63.5(5)
C(2)–C(3)–C(11)–C(16)	119.9(4)
C(4)–C(3)–C(11)–C(12)	115.3(5)
C(2)–C(3)–C(11)–C(12)	–61.3(5)
C(16)–C(11)–C(12)–C(13)	0.6(6)
C(3)–C(11)–C(12)–C(13)	–178.3(4)
C(11)–C(12)–C(13)–C(14)	–1.1(7)
C(12)–C(13)–C(14)–C(15)	0.8(8)
C(13)–C(14)–C(15)–C(16)	0.0(8)
C(14)–C(15)–C(16)–C(11)	–0.5(7)
C(12)–C(11)–C(16)–C(15)	0.2(6)
C(3)–C(11)–C(16)–C(15)	179.1(4)

Symmetry transformations used to generate equivalent atoms: