for 3-methyladeninium ion in aqueous acid.[17] Adenine was deuterated at position 8 by heating in D_2O at 100 °C for 8 h.[13]

Bis[(4-amino-1,2-dimethyl-5-pyrimidinio)methyl]amine Diperchlorate VI. A suspension of 0.950 g (1.98 mmol) of 1′-methylthiaminium diperchlorate[5] 1.4 mL (10 mmol) of 2,4,6-trimethylpyridine, and 0.26 g (2.2 mmol) of ammonium perchlorate in 20 mL of methanol was heated at reflux for 18 h. The filter cake was washed with ethyl acetate to give 0.368 g (0.75 mmol, 76%) of product, mp 259–261 °C dec. Recrystallization from water gives the analytical sample of VI: mp 261–264 °C dec; ^1H NMR (Me_2SO-d_6 Me_4Si) δ 9.0, 8.3 (NH_2), 8.2 (6-H), 3.8 (NCH_3), 3.6 (CH_2), 3.3 (NH and HOD), 2.6 (CCH_3); ^{13}C NMR δ 162.4, 161.4 (C-2 and C-4), 146.1 (C-6), 113.9 (C-5), 44.9 (CH_2), 41.6 (NCH_3), 21.4 (CCH_3). Anal. Calcd for $C_{14}H_{23}N_7Cl_2O_8$: C, 34.44; H, 4.75; N, 20.08. Found: C, 34.30; H, 4.74; N, 19.96.

Nuclear Overhauser Effects. A 50-mg suspension of adenosine derivative III was made to undergo hydrogen–deuterium exchange (OH and some CCH_3) by brief heating in 5 mL of D_2O. Solvent was removed under reduced pressure. The cycle was repeated four times. To a 1% solution of this deuterated III in D_2O was added 0.5 μL of $C_6H_5CH_2OD$ prepared by exchanging the hydroxyl proton in D_2O–DCl, followed by fractional distillation of ether extracts. The solution was filtered through a 1-μm Millipore filter into an NMR tube and degassed by five freeze–thaw–pump cycles. The tube was sealed under vacuum. The NMR tube was soaked in 0.01 M EDTA for 24 h to remove any paramagnetic ions, rinsed with D_2O, and oven dried.

NMR spectra were recorded on a FT JEOL FX-100Q spectrometer. Data were recorded when the CH_2 group was irradiated and also when the sample was irradiated off resonance with a second signal located 500 Hz upfield from the CH_2 signal. The cycle of irradiation on and off resonance was repeated four times; the data were averaged. Signals from the three aromatic protons and the phenyl group of the benzyl alcohol internal standard were processed according to peak heights and areas. The height of the lowest field imidazole-bonded proton essentially was the same during resonance and off resonance. But the height of the signal associated with the proton bonded to the pyrimidine ring of adenosine showed a large NOE enhancement of 33%. The remaining aromatic signal sharpened due to decoupling and possibly increased in intensity due to an NOE, but evaluation is difficult due to the large change in line width associated with decoupling.

The areas of the peaks of interest also were determined by planimetry after the peaks were recorded at a 66.4-Hz sweep width. Areas of the two low-field aromatic protons depend on how a base line is drawn because they do not show base-line separation. The area of the lowest field imidazole signal increased by <3% on irradiation of the CH_2 group, that for the pyrimidine ring of adenine increased by 17–18%, and that for the remaining proton showing decoupling increased by 9–12%. With the exception of the latter signal, line-width changes for the other two aromatic protons were no more than 2%.

Noteworthy is our preliminary experiment which in a few minutes demonstrated an NOE and gave us a proof of structure. A sample of III in D_2O was routinely prepared. Irradiation of the CH_2 signal with a Varian 360L spectrometer showed that the intensity of the proton bonded to the pyrimidine ring of adenosine increased while that of the imidazole did not; the remaining aromatic proton was decoupled. The more elaborate experiment was then performed as a rigorous check.

In order to check assignments for the aromatic protons in III the H-1′ signal of ribose was irradiated. Because the signal due to CH_2 is close, the irradiating power was systematically varied in order to minimize irradiating this neighbor as well. The signal at lowest field showed NOE enhancement first and others as the irradiating power increased. This lowest field aromatic proton signal must be associated with the imidazole proton. The same conclusion was reached from a deuteration experiment.

Fluorescence Measurements. Fluorescence spectra were recorded on a Perkin-Elmer MPF-44A; they are uncorrected. A 2.4×10^{-3} M solution of III in pH 9.2 borate buffer was prepared by weight. Dilutions with buffer and/or water gave other samples. Spectral data are reported in the text. A 5×10^{-10} M sample which stood under air at room temperature for 1 week showed about a 3-fold reduction in intensity.

Several reaction mixtures were checked to determine whether fluorescent materials are formed. (a) 1′-Methylthiaminium diperchlorate (0.2 M) was heated at 65 °C for 30 h in 80% methanol–20% Me_2SO. No fluorescence was detected. (b) Repetition with prior addition of 2,4,6-trimethylpyridine gave a solution which on 10^3-fold dilution with water had excitation and emission bands at 361 and 413 nm, respectively. (c) A third sample containing the thiamine, the pyridine, and adenine was heated under the same conditions. Following 10^5-fold dilution with water, excitation (391 nm) and emission [410, 428 (shoulder)] were observed. Adenine may form a tetracyclic product related to III in a small amount.

Acknowledgment. This work was kindly supported in part by Grant AM 17442 from the National Institute of Arthritis, Metabolism, and Digestive Diseases and by a University of Florida Biomedical Research Grant. Dr. G. Uray and Dr. B. Langhammer kindly provided the systematic name for III.

Registry No. I, 58-61-7; II, 73333-47-8; III, 80584-80-1; V, 80584-82-3; VI, 80584-84-5; adenine, 73-24-5.

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Bis(trialkylsilyl) Chalcogenides. 1. Preparation and Reduction of Group 6A Oxides

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(Alkyl- and arylchalcogeno)silanes (**1**) are compounds of current interest in the literature.[1-3] With the scrutiny that the silicon–chalcogen bond has received with respect to these compounds, it is somewhat surprising that the much older bis(trimethylsilyl) chalcogenides (**2**) have not received more study, particularly of their applications to organic chemistry.[3d,4-6] Herein, we report the facile and high-yield preparation of the compounds **2** and their

$ArYSiR_3$	$Me_3SiYSiMe_3$
1	2a, Y = S
	b, Y = Se
	c, Y = Te

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[1] For $PhSSiR_3$: (a) Hooton, K. A.; Allred, A. L. *Inorg. Chem.* **1965**, *4*, 671–678. (b) Evans, D. A.; Truesdale, L. K.; Grimm, K. G.; Nesbitt, S. L. *J. Am. Chem. Soc.* **1977**, *99*, 5009–5017. (c) Ojima, I.; Nagai, Y. *J. Organomet. Chem.* **1973**, *57*, C42–C44. (d) Evans, D. A.; Grim, K. G.; Truesdale, L. K. *J. Am. Chem. Soc.* **1975**, *97*, 3229–3230.

[2] For $PhSeSiR_3$: (a) Anderson, J. W.; Barker, G. K.; Drake, J. E.; Rodger, M. *J. Chem. Soc., Dalton Trans.* **1973**, 1716. (b) Barker, G. K.; Drake, J. E.; Hemmings, R. T. *Ibid.* **1974**, 450. (c) Drake, J. E.; Hemmings, R. T. *Ibid.* **1976**, 1730. (d) Detty, M. R.; Seidler, M. D. *J. Org. Chem.* **1981**, *46*, 1283–1292 and references therein. (e) Liotta, D.; Paty, P. B.; Johnson, J.; Zima, G. *Tetrahedron Lett.* **1978**, 5091–5094. (f) Miyoshi, N.; Kondo, K.; Murai, S.; Sonoda, N. *Ibid.* **1979**, 909–912. (h) Miyoshi, N.; Ishii, H.; Kondo, K.; Murai, S.; Sonoda, N. *Synthesis* **1979**, 300–301. (i) Derkach, N. Y.; Tishchenko, N. P. *Zh. Org. Khim.* **1977**, *13*, 100–103.

[3] For $ArTeSiR_3$: (a) Praefcke, K.; Weichsel, C. *Synthesis* **1980**, 216. (b) Drake, J. E.; Hemmings, R. T. *Inorg. Chem.* **1980**, *19*, 1879–1883.

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[5] For $R_3SiSeSiR_3$: Schmidt, M.; Ruf, H. *Z. Anorg. Allg. Chem.* **1963**, *321*, 270–273.

[6] For $R_3SiTeSiR_3$: (a) Buerger, H.; Goetze, U. *Inorg. Nucl. Chem. Lett.* **1967**, *3*, 549–552. (b) Bochkarev, M. N.; Sanina, L. P.; Vyazankin, N. S. *Zh. Obshch. Khim.* **1969**, *39*, 135.

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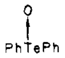
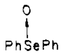
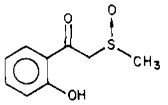
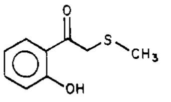
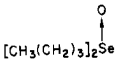
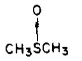
Table I. Reduction of Group 6A Oxides with Bis(trialkylsilyl) Chalcogenides

oxide	reductant	product	solvent	% yield[a]	bp, °C (torr)
 $\mathrm{Ph}\overset{\overset{O}{\uparrow}}{\mathrm{Te}}\mathrm{Ph}$	2a	PhTePh (4)	THF	95	75 (1)[b]
	2b		THF	89	
	2c		THF	85	
 $\mathrm{Ph}\overset{\overset{O}{\uparrow}}{\mathrm{Se}}\mathrm{Ph}$	2a	PhSePh (5)	CH_3CN	100	75 (1)[b]
	2b		THF	96	
	3		THF	100	
	2a	 6	THF	90	103-105 (0.1)
	2b		THF	100	
 $[CH_3(CH_2)_3]_2\overset{\overset{O}{\uparrow}}{Se}$	2b	$[CH_3(CH_2)_3]_2Se$ (7)	THF	62	50 (1.5)[b]
 $CH_3\overset{\overset{O}{\uparrow}}{S}CH_3$	2a	CH_3SCH_3 (8)	CH_3CN	95	38-40 (atm)
	2b		C_6H_5Cl	76	
	2c		THF	80	
	3		THF	80	

[a] Isolated yield. [b] Molecular still.

function as reducing agents for the group 6A oxides.

Although the reaction of dilithium sulfide with chlorotrimethylsilane is reported to occur only at high temperature and pressure to give bis(trimethylsilyl) sulfide (**2a**),[7] both bis(trimethylsilyl) selenide (**2b**) and bis(trimethylsilyl) telluride (**2c**) are formed in modest to good yields from the lithium salts and chlorotrimethylsilane in ether, as indicated in eq 1.[5,6a,8] Even though the preparations of **2b** and

$$Li_2Y + 2Me_3SiCl \rightarrow Me_3SiYSiMe_3 + 2LiCl \quad (1)$$

2c were easily repeated, we were disappointed with the modest yields (in our hands, 30–50%) and the experimental manipulations involved in preparing the dilithium chalcogenide in liquid ammonia and conducting the silylation in a second solvent. We have prepared several grams of compounds **2** in a one-pot, high-yield process by using the Gladysz procedure to prepare dilithiosulfide[9a,c] and dilithioselenide[9b] and extended the procedure to include tellurium.

Compound **2a** was prepared in 83–90% yield by lithium triethylborohydride reduction of elemental sulfur in tetrahydrofuran (THF) followed by the addition of a slight excess of chlorotrimethylsilane. The addition was quite exothermic, with reaction being complete after 1 h at room temperature. This was in marked contrast to the much slower reaction of commercially available dilithium sulfide with chlorotrimethylsilane in refluxing THF, which gave **2a** in 45% yield only after 72 h at reflux. The use of lithium triethylborohydride offers a convenient preparation of small quantitites of **3a** (10 g or less), but larger scale preparations are perhaps best conducted by the procedure of Harpp and Steliou[10a] or the procedure of Kuwajima and Abe.[10b]

Compound **2b** was prepared in 63–95% yields by treating selenium shot (as compared to gray, powdered selenium in the literature procedure)[9a] with lithium triethylborohydride in THF and quenching the dilithium selenide with chlorotrimethylsilane. Bis(trimethylsilyl) selenide (**2b**) was isolated as an air-sensitive, colorless oil. It was conveniently stored at −20 °C in argon-flushed, septum-equipped vials for up to 1 month without serious decomposition.

Similarly, bis(trimethylsilyl) telluride (**2c**) was prepared in 53–75% yield from tellurium shot, lithium triethylborohydride, and chlorotrimethylsilane. Compound **2c** was extremely sensitive to both air and light. Distillation and transfer under red light minimized photochemical decomposition. However, serious decomposition was observed after 24 h in dark storage at −20 °C under argon.

Selenium shot was far more convenient to use than powdered selenium. Weighing and transfer were much easier. Furthermore, the reactivity of the shot form of the metals was more consistent from lot to lot than with the commercial powdered forms. Tellurium shot was somewhat less reactive than selenium shot (the former requiring 4–8 h for consumption, the latter 1–2 h), giving a milky pink or purple mixture on completion of reaction.

A more stable analogue of **2c** was prepared by treating dilithium telluride as prepared above with *tert*-butylchlorodimethylsilane. Bis(*tert*-butyldimethylsilyl) telluride (**3**) was isolated in 79% yield as a low-melting, white, crystalline solid. Compound **3** was stable for several weeks in the dark at −20 °C in argon-flushed vials; when stored at room temperature under red light, it noticeably decomposed after several days.

(Phenylseleno)silanes are useful as one-electron reducing agents for the reduction of group 6A oxides.[11] The solubility and chromatographic and crystalline properties of the diphenyl diselenide produced in these reactions make its removal from the desired reduction products difficult in certain cases. The reduction of sulfoxides with **2a** to give the corresponding sulfide, sulfur, and hexamethyldisiloxane[4c] suggested that the other bis(trialkylsilyl) chalcogenides should function as two-electron reducing agents according to eq 2. The reduction of benzoyl per-

$$RM(\rightarrow O)R' + \underset{2}{Me_3SiYSiMe_3} \rightarrow RMR' + Me_3SiOSiMe_3 + Y \quad (2)$$
$$M = S, Se, Te$$

oxide with **2c** to give tellurium metal and trimethylsilyl

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[9] (a) Gladysz, J. A.; Hornby, J. L.; Garbe, J. E. *J. Org. Chem.* **1978**, *43*, 1204–1208. (b) Gladysz, J. A.; Wong, V. K.; Jick, B. S. *J. Chem. Soc., Chem. Commun.* **1978**, 838–839. (c) Gladysz, J. A.; Wong, V. K.; Jick, B. S. *Tetrahedron* **1979**, 2329.

[10] (a) Harpp, D. M.; Steliou, K. *Synthesis* **1976**, 721–722. (b) Kuwajima, I.; Abe, T. *Bull. Chem. Soc. Jpn.* **1978**, *51*, 2183.

[11] Detty, M. R. *J. Org. Chem.* **1979**, *44*, 4528–4531.

benzoate[12] has also been reported. The elemental chalcogen produced in these reactions would be easily separated from the reduction products.

The reaction occurred as depicted in eq 2 for a variety of sulfoxides, selenoxides, and telluroxides. Table I gives representative examples of the reduction. The elemental chalcogen produced during the reductions was removed by filtration through a Celite pad. Hexamethyldisiloxane was isolated by fractional distillation. Compound **3** gave bis(*tert*-butyldimethylsilyl) ether upon deoxygenation. The reagents **2** failed to reduce other heteroatom oxides such as pyridine *N*-oxide and triphenylphosphine oxide.

The ease with which **2a** reduced the various group 6A oxides was pleasantly surprising in view of the inert nature of (phenylthio)- and (methylthio)trimethylsilane under similar conditions. Although noticeably less vigorous than the reactions of **2b** and **2c**, the reductions with **2a** were mildly exothermic, producing yellow, crystalline, elemental sulfur.

Other chemistry of the bis(trialkylsilyl) chalcogenides may reflect the ease of oxidizing tellurium in the −2 oxidation state to tellurium(0) in these compounds. The reactions of **2c** with halogen to give tellurium metal and silyl halides have been reported.[13] Formally, **2c** is a two-electron reducing agent for halogen molecules. Compound **2b** reacted similarly with iodine, giving iodotrimethylsilane and selenium metal in nearly quantitative yields. Interestingly, silyl halides were not isolated when **2a** was treated with bromine or iodine.[4b] Other reductions incorporating these compounds are being investigated.

Experimental Section

Melting points were determined on a Thomas-Hoover melting point apparatus and are corrected. Boiling points are uncorrected. ^{1}H NMR spectra were recorded on a Varian EM 390 instrument. IR spectra were recorded on a Beckman IR 4250 spectrophotometer. Acetonitrile was distilled from phosphorus pentoxide and stored over 3A molecular sieves. Tetrahydrofuran was distilled from benzophenone ketyl. Chlorotrimethylsilane was distilled from lithium hydride.

Caution: the toxicity of bis(trialkylsilyl) chalcogenides is not known. Care should be exercised in their handling.

Preparation of Bis(trimethylsilyl) Selenide (2b). A 250-mL two-necked, round-bottomed flask was flamed and cooled under an argon atmosphere. The flask was charged with 105 mL (0.105 mol) of 1 M lithium triethylborohydride in THF. The flask was cooled in an ice bath, and selenium shot (3.95 g, 0.0500 mol) was added. The reaction mixture was warmed to ambient temperature and stirred for 2 h. The reaction mixture was cooled to ice-bath temperature, and chlorotrimethylsilane (12.6 g, 0.117 mol) was added in one portion. The resulting mixture was stirred for 2 h at ambient temperature. The low-boiling volatiles were removed by distillation at 20 torr at 30–60 °C directly from the reaction vessel. The product was distilled at higher vacuum, again directly from the reaction vessel, to give 10.6 g (95%) of **2b** as a colorless oil, bp 45–46 °C (5.3 torr). The pot residue was treated with methanol under an inert atmosphere before disposal. For **2b**: ^{1}H NMR (CDCl$_3$) δ 0.50 (s). Anal. Calcd for $C_6H_{18}Si_2Se$: C, 32.0; H, 8.1; Se, 35.0. Found: C, 31.7; H, 8.2; Se, 35.5.

Preparation of Bis(trimethylsilyl) Sulfide (2a). Sulfur (0.64 g, 0.020 mol) was added to 40 mL (0.040 mol) of 1 M lithium triethylborohydride as described. After the reaction mixture had been stirred 0.5 h at ambient temperature, chlorotrimethylsilane (4.32 g, 0.040 mol) was added, giving an exothermic reaction. After 2 h of stirring at ambient temperature, the reaction products were removed by direct distillation under a nitrogen atmosphere. Compound **2a** was isolated as colorless oil: 2.90 g (83%); bp 155–157 °C. Anal. Calcd for $C_6H_{18}SSi_2$: C, 40.4; H, 10.2; S, 18.0. Found: C, 40.4; H, 10.1; S, 18.1.

Preparation of Bis(trimethylsilyl) Telluride (2c). Tellurium shot (3.56 g, 0.0279 mol) and lithium triethylborohydride (58 mL, 0.058 mol) were treated as described. After 8 h at room temperature, the reaction mixture was purple with a chalky white suspension. The reaction vessel was wrapped in foil, chlorotrimethylsilane (7.00 g, 0.0648 mol) was added, and the mixture was stirred at ambient temperature for 6 h. The low-boiling volatiles were removed at 30–60 °C (20 torr). The product **2c** was isolated (4.07 g, 53%) as a clear, colorless oil, bp 49–51 °C (2.5 torr). The receiving flask was foil wrapped to minimize exposure to light. Attempts to obtain ^{1}H NMR spectra and analyses were hindered by rapid decomposition of **2c**.

Preparation of Bis(*tert*-butyldimethylsilyl) Telluride. Tellurium shot (3.56 g, 0.0279 mol), lithium triethylborohydride (58 mL, 0.058 mol), and *tert*-butylchlorodimethylsilane (9.74 g, 0.0580 mol) were treated as described. The product **3** was isolated (7.77 g, 79%) as a white solid (mp 46–49 °C) after initial distillation from the reaction mixture as a colorless oil: bp 90–95 °C (5.5 torr); ^{1}H NMR (CDCl$_3$) δ 1.00 (s, 9 H), 0.57 (s, 6 H). Anal. Calcd for $C_{12}H_{30}Si_2Te$: C, 40.2; H, 8.4. Found: C, 40.3; H, 8.2.

General Procedure for Reduction of Group 6A Oxides. A solution of the oxide in THF, CH_3CN, or chlorobenzene (0.2–1.0 M) was degassed with a slow stream of nitrogen bubbles for 15 min. The bis(trialkylsilyl) chalcogenide was added in one portion under argon. Mildly exothermic reactions occurred. The reaction mixtures were stirred for 1 h at ambient temperature, diluted with ether, and filtered through a Celite pad to remove the chalcogen. Careful distillation of the filtrates gave hexamethyldisiloxane in 80–100% yields. The reduction products gave the following spectral data.

For **4**: ^{1}H NMR (CDCl$_3$) δ 7.67 (m, 4 H), 7.17 (m, 6 H); mass spectrum, *m/e* 284 ($C_{12}H_{10}{}^{130}Te$).

For **5**: ^{1}H NMR (CDCl$_3$) δ 7.43 (m, 4 H), 7.17 (m, 6 H); mass spectrum, *m/e* 234 ($C_{12}H_{10}{}^{80}Se$).

For **6**: ^{1}H NMR (CDCl$_3$) δ 12.10 (s, 1 H), 7.75 (m, 1 H), 7.50 (m, 1 H), 7.00 (m, 2 H), 3.80 (s, 2 H), 2.25 (s, 3 H); mass spectrum, *m/e* 182 ($C_9H_{10}O_2S$).

For **7**: ^{1}H NMR (CDCl$_3$) δ 2.51 (t, 4 H, J = 7.5 Hz), 1.85–1.20 (m, 8 H), 0.90 (t, 6 H, J = 7 Hz); mass spectrum, *m/e* 194 ($C_8H_{18}{}^{80}Se$).

For **8**: ^{1}H NMR (CDCl$_3$) δ 2.00 (s).

Preparation of Iodotrimethylsilane from 2b. A 15-mL two-necked flask was flushed with argon and charged with 1 mL of *m*-xylene. Bis(trimethylsilyl) selenide (1.0 g, 7.4 mmol) was added via syringe, and iodine (1.14 g, 4.50 mmol) was added, to give a mildly exothermic reaction. The iodotrimethylsilane was distilled onto copper wire to give 1.6 g (94%) of a colorless oil, bp 106–107 °C. The pot residue was washed with ether, filtered, and dried to give 0.35 g (100%) of selenium.

Registry No. 2a, 3385-94-2; **2b**, 4099-46-1; **2c**, 4551-16-0; **3**, 80594-86-1; **4**, 1202-36-4; **5**, 1132-39-4; **6**, 56986-82-4; **7**, 14835-66-6; **8**, 75-18-3; phenyl telluroxide, 51786-98-2; phenyl selenoxide, 7304-91-8; 1-(2-hydroxyphenyl)-2-(methylsulfinyl)ethanone, 16697-77-1; butyl selenoxide, 22089-68-5; methyl sulfoxide, 67-68-5.

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(13) Vyazankin, N. S.; Sanina, L. P.; Kalina, G. S.; Bochkarev, M. N. *Zh. Obshch. Khim.* **1968**, *38*, 414.

Constituents of *Eremocarpus setigerus* (Euphorbiaceae). A New Diterpene, Eremone, and Hautriwaic Acid

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Eremocarpus setigerus (Hook) Benth, a member of the spurge family, commonly known as "dove weed" and