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

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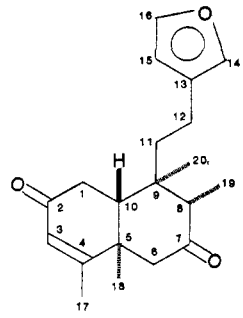
Eremocarpus setigerus (Hook) Benth, a member of the spurge family, commonly known as "dove weed" and

"turkey mullein", was used widely by the California indians as a fish poison, a counter irritant for pain, and a curative for typhoid and other fevers.[1] The hot methanol extract of the ground fresh plant yielded no crystalline substance except hentriacontane, hexacosanol, and β-sitosterol.[2] We report the isolation from the petroleum ether extract of this plant and identification of two related crystalline diterpenes, eremone (1; from roots, stems, leaves, flowers,



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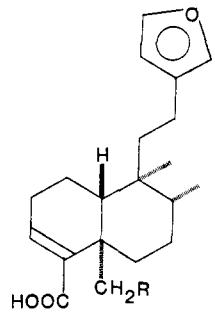

R	
2a	H
2b	OH
2c	OAc
2d	lactone of 2b

and fruits collected in California in Nov 1972) and hautriwaic acid[3-7] (**2b**; from roots, stems, leaves, and flowers collected in Oregon in Aug 1980). Neither compound is responsible for the antitumor activity we have found this extract to possess.

Eremone was formulated as 1 on the basis of spectroscopy. The combination of its elemental analysis and molecular ion peak at m/z 314 led to molecular formula $C_{20}H_{26}O_3$. The IR spectrum showed bands for furan (1500, 1020, and 868 cm^{-1}), saturated (1715 cm^{-1}) and α,β-unsaturated (1672 cm^{-1}) ketones, a trisubstituted double bond (1620 and 835 cm^{-1}), and a *C*-methyl (1378 cm^{-1}). The ^1H NMR spectrum showed the furan to be β-substituted, the α,β-unsaturated ketone to possess an α-hydrogen and a β-methyl group, an unsplit methyl group, and CH_3CH and CH_2CH_2 groupings unsplit by other groupings and with the CH and one of the CH_2's allylic or α to a carbonyl group. This combined data led us to postulate oxidized kolavane-type structure 1 for this substance.

Table I gives ^{13}C NMR shifts and off-resonance multiplicities for 1 and **2b**. ^{13}C NMR assignments for the related diterpenoid hispanolone[8] served as a basis for some of our assignments; others were made from calculated values.[9] The observed values for eremone are completely in accord with structure 1.

The mass spectrum of eremone was strongly confirmatory of structure 1. All the major peaks were readily rationalized (see Scheme I in the supplementary material), and in most cases their compositions were verified by high-resolution exact-mass measurements. Many proposed transformations were substantiated by metastable peak study.

Table I. ^{13}C NMR Chemical Shifts (δ) of Eremone (1) and Hautriwaic Acid (2b)

atom	1 ($CDCl_3$)[c]	2b (C_5D_5N)[c]
1	35.2 t	17.2
2	198.5 s	27.1 t
3	126.1 d	137.3
4	169.2 s	142.5
5	44.4 s	42.2[a]
6	51.2 t	31.2
7	210.9 s	26.8 t
8	50.2 d[a]	36.5
9	44.4 s	42.3[a]
10	45.8 d[a]	46.6
11	38.4 t	39.0
12	19.4 t	18.4
13	124.0 s	125.6
14	143.1 d	142.9
15	110.7 d	111.2
16	138.7 d	138.6
17	18.9 q[b]	171.9 s
18	18.8 q[b]	65.3 t
19	7.8 q	15.9 q
20	18.5 q[b]	18.7 q

[a,b] May be reversed within column. [c] Solvent in parentheses.

An X-ray study confirmed the constitution and relative configurations shown in 1 and showed the conformation. The A and B ring torsion angles are within a few degrees of those of 1,2-diplanar cyclohexene and chair cyclohexane conformations, respectively.[10] The atoms comprising the furan ring and the attached carbon are within experimental error of their least-squares plane. In the side chain, C(12) is within 1° of being anti to C(20), C(13) is 3° from being anti to C(9), and the C(11)–C(12)–C(13)–C(14) torsion angle is 42°.

The absolute configuration of 1 is based on analogy with hautriwaic acid (**2b**),[3-7] found in a later collection of the same species, and is supported by their both possessing large negative optical rotations, as does hardwickiic acid (**2a**).[11] Hautriwaic acid (**2b**), previously isolated from plants of different genera,[3-7] was identified from its properties and those of its acetate (**2c**) and lactone (**2d**).

Eremone (1) and hautriwaic acid (**2b**) certainly appear to be biosynthesized from a common kolavane-type precursor, the former by oxidations at C(2) and C(7), and the latter by oxidations at C(17) and C(18). Whether the seasonal or locational difference in the plant collections was responsible for the difference in the diterpene found is not known.

Experimental Section

Melting points were determined on a Kofler hot-stage apparatus and are uncorrected. Carbon and hydrogen analyses were carried out by the University Analytical Center, Tucson, Az. Optical rotations were measured by using a Perkin-Elmer 241 MC polarimeter. Ultraviolet (UV) and infrared (IR) spectra were run on Cary-15 and Beckman IR-33 spectrometers, respectively. ^1H and ^{13}C NMR spectra were run on a Bruker WM-250 spectrometer. Electron-impact (EI) and chemical-ionization (CI) mass spectra were recorded on a Varian MAT 311 spectrometer with a Varian SS 200 data system, and the intensities of the ions, given in parentheses, are expressed on a scale in which the largest peak in the spectrum is assigned an intensity of 100%. The high-resolution data were obtained at a resolution of 7000 by scanning the mass range from m/z 100 to 500 repetitively at 25 s/deg with PFK as the internal standard. Metastable ion spectra were recorded either by scanning the magnetic (*B*) and electrostatic (*E*)

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fields at constant accelerating voltage with the B/E ratio constant to obtain the daughters of parents or by scanning the accelerating voltage at constant B and E for determination of parents of daughters. Samples were introduced by using a direct probe. The normal ionizing voltage was 70 eV with a source temperature of 250 °C.

Isolation of Eremone (1). The dried plant *Eremocarpus setigerus* (collected in California) was ground in a Wiley mill and stored at −10 °C prior to extraction. The ground material was extracted with petroleum ether in a Lloyd extractor for 72 h. The dried petroleum ether extract residue was separated into acetone-soluble and -insoluble fractions. The acetone-insoluble fraction was further separated into acetonitrile-soluble and -insoluble fractions. The combined acetone- and acetonitrile-soluble fraction, after removal of the solvent, was treated with a small amount of petroleum ether, cooled, and filtered. Evaporation of the solvent from the filtrate yielded an oily residue which was placed on the top of an EM SiO_2-60 column and eluted with hexane followed by ether and methanol. The fraction eluted with ether was subjected to EM SiO_2-60 column chromatography, eluting the column with hexane with an increasing concentration of ether. Fractions eluted with hexane/ether (40:60), hexane/ether (20:80), and ether (100%) were combined and rechromatographed on an EM SiO_2-60 column, eluting the column with various concentrations of hexane/ether as before. Fractions eluted with hexane/ether (1:1) yielded a residue which when subjected to further chromatography (EM SiO_2-60) gave a fraction from which eremone was crystallized out when treated with isopropyl ether: mp 106–107 °C; $[\alpha]^{25}_D$ −93.1° (in pyridine); ^{1}H NMR (pyridine-d_5) δ 7.36 (H-15, 1 H, t, J = 1.7 Hz), 7.22 (H-16, 1 H, m), 6.25 (H-14, 1 H, dd, J = 1.6, 0.8 Hz), 5.82 (H-3, 1 H, q, J = 1.3 Hz), 2.72 (H-8, 1 H, q, J = 6.6 Hz), 2.45–2.63 (H-1α, H1β, H-10, 3 H, m), 2.51 (H-6, 2 H, s), 2.37 (H-12, 2 H, t, J = 8.5 Hz), 1.90 (H-17, 3 H, d, J = 1.3 Hz), 1.74, 1.56 (H-11, 2H, ddd, J = 17.2, 10.5, 6.6 Hz), 1.12 (H-18, 3 H, s), 1.00 (H-19, 3 H, d, J = 6.6 Hz), 0.82 (H-20, 3 H, s); ^{13}C NMR, Table I; mass spectrum, m/z (relative intensity) 314 ($M^{+\cdot}$, 22.6), 299 (4), 286 (2), 243 (12.4), 232 (6.8), 220 (25.4), 219 (69.8), 205 (6.3), 201 (5.4), 191 (4.8), 189 (4.7), 187 (5.8), 178 (10.8), 177 (12.2), 175 (7.3), 173 (10.6), 163 (7.8), 161 (6.3), 149 (26.7), 137 (11.6), 136 (25.6), 135 (48.7), 123 (24.5), 122 (34.1), 121 (25.8), 109 (33.5), 107 (17.1), 105 (8.4), 95 (86.8), 83 (51.7), 81 (100), 79 (28), 77 (15.6), 69 (22.3), 67 (20), 65 (10.5), 55 (22.6), 53 (30.3), 44 (12.8), 43 (16.2), 41 (50.6). The IR, ^{1}H NMR, ^{13}C NMR, and mass spectra were in accord with structure 1.

Anal. Calcd for $C_{20}H_{26}O_3 \cdot 0.5H_2O$: C, 74.35; H, 8.35. Found: C, 74.97; H, 8.32.

Crystallographic Study of Eremone (1). A 0.4 × 0.6 × 0.8 mm crystal was mounted on a Syntex $P2_1$ diffractometer with a graphite monochromator (Mo Kα, λ 0.71069 Å). The cell lengths determined by least-squares treatment of 15 reflections were a = 6.890 (1) Å, b = 12.954 (3) Å, c = 9.984 (2) Å, and β = 102.44 (1)°; the space group was $P2_1$ with Z = 2. The θ–2θ scan technique was used with a 0.8° scan range and a background to scan time ratio of 0.5; 1754 reflections with $2\theta < 50°$ were measured, and 969 with $I > 3\sigma_I$ were considered observed. All nonhydrogen atoms were located on the first E map from MULTAN.[12] Isotropic refinement reduced R to 0.147; anisotropically, it dropped to 0.098. Hydrogen positions were calculated, and when they were included in further nonhydrogen refinements with the isotropic temperature factors of the attached atoms, R dropped to its final value of 0.070.

Isolation of Hautriwaic Acid (2b). The dried plant (collected in Oregon) was milled and extracted with petroleum ether in the same way as for eremone. The dried petroleum ether extract was separated into ether-soluble and -insoluble fractions. The ether-soluble fraction was vacuum dried and subjected to three-funnel partition between 20% aqueous MeOH and petroleum ether. The lower phase was dried under vacuum and separated into ether-soluble and -insoluble fractions. Chromatography of the ether-soluble residue on EM silica gel-60 and elution with hexane/ether (75:25) gave a fraction which on concentration deposited crystals of crude hautriwaic acid (**2b**) which were separated by filtration, washed with ether, and recrystallized from ether/acetone as colorless lustrous rectangular prisms: mp 191–192 °C; $[\alpha]^{25}_D$ −133.5° (in pyridine; lit.[3,4] mp 183–184 °C; $[\alpha]^{25}_D$ −105°). The IR, ^{1}H NMR, and mass spectra were in accord with literature values.[5-7]

Acetylation of Hautriwaic Acid. Acetylation of **2b** with Ac_2O–pyridine at room temperature for 24 h yielded a mixture of **2c** and **2d** whose separation was effected by preparative TLC [EM SiO_2-60 PF-254; methylene chloride/methanol (60:1)] and isolated as TLC-pure samples. No attempt was made to crystallize these samples: mass spectrum, m/z (relative intensity) 374 ($M^{+\cdot}$, 1.5), 357 (2.6), 356 (10.3), 314 (14.8), 301 (12.2), 283 (26), 279 (26.9), 261 (31.1), 255 (7), 220 (22.6), 219 (89.5), 207 (11.2), 203 (10.4), 201 (21.8), 189 (22.5), 173 (20.8), 163 (30.4), 161 (13.7), 159 (11), 151 (22), 149 (39.6), 147 (11.6), 145 (19), 137 (17.3), 135 (16.3), 133 (15), 131 (12.7), 125 (14.3), 123 (12), 121 (17), 119 (16.7), 117 (12.3), 109 (17.6), 107 (21), 105 (29.2), 97 (10.2), 96 (47.6), 95 (82.2), 94 (16.7), 93 (24.7), 91 (43.7), 83 (15.1), 82 (48.2), 81 (100), 79 (30), 77 (26.5), 69 (17.6), 67 (25.3), 65 (14), 60 (15), 55 (30.7), 53 (29.6). The IR and mass spectra of **2c** were in accord with the proposed structure.

The IR, ^{1}H NMR, and mass spectra of **2d** were in accord with literature values.[5-7]

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Registry No. 1, 80594-75-8; **2b**, 18411-75-1; **2c**, 35060-24-3; **2d**, 18411-74-0.

Supplementary Material Available: Mass spectral fragmentation patterns of 1 and **2b** (Schemes I and II) and bond lengths, bond angles, stereoview of a unit cell, fractional coordinates, and temperature factors for 1 (6 pages). Ordering information is given on any current masthead.

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Proton Nuclear Magnetic Resonance Analysis for Meso and *dl* Isomers of Succinic-d_2 Acid

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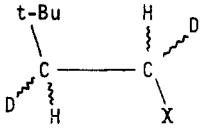
The ability to quantitatively analyze vicinal dideuteriated systems for erythro/threo[2,3] (e.g., **1**) and meso/*dl*[4,5] (e.g., **2**) ratios have been useful in many



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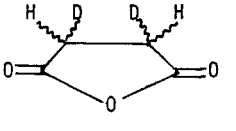

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mechanistic investigations. The analysis of representative examples of compound **1** is relatively straightforward since J_{H_1,H_2} differs substantially and is directly measurable for erythro and threo isomers. Symmetry prevents a simple

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