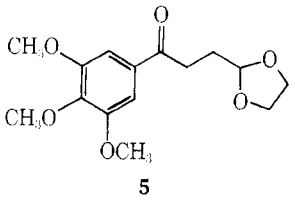
Table I
Naphthalenes

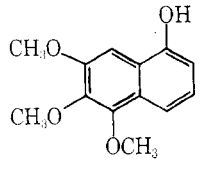
Compd	Yield, %	Mp, °C	Bp, °C	Empirical Formula
4a[a]	95		154–156 (5)	$C_{13}H_{14}O_3$
4b[b]	75	115–118		$C_{12}H_{12}O_2$
4c[c]	84	69–70		$C_{11}H_{10}O$
4d[d]	62		128–132 (1)	$C_{12}H_{12}O_2$
4e[e]	91	95–97		$C_{11}H_8O_2$
4f[f]	84			$C_{12}H_{12}O$
4g[g]	54	48–51		$C_{11}H_{10}O_2$
4h[h]	52	118–120		$C_{10}H_8O$
4i[i]	84		115–120 (1)	$C_{11}H_{10}$
4j[j]	31	78–80		$C_{10}H_8$
6[k]	30	92–95	175–185 (0.05)	$C_{13}H_{14}O_4$

[a] A. Ueno and S. Fukushima, *Chem. Pharm. Bull.*, **14**, 129 (1966). [b] Ng. Buu-Hoi and D. Lavit, *J. Org. Chem.*, **21**, 21 (1956). [c] G. A. Baramki, H. S. Chang, and J. T. Edward, *Can. J. Chem.*, **40**, 441 (1962). [d] R. Heck and C. Ellinger, *J. Amer. Chem. Soc.*, **79**, 3105 (1957). [e] W. Bonthrone and J. W. Conforth, *J. Chem. Soc. C*, 1202 (1969). [f] P. C. Mitter and D. E. Shyamakanta, *J. Indian. Chem. Soc.*, **16**, 35 (1939). [g] H. S. Chang and J. T. Edward, *Can. J. Chem.*, **41**, 1233 (1963). [h] L. Schaeffer, *Ann.*, **152**, 279 (1869). [i] K. E. Schulze, *Ber.*, **17**, 842 (1884). [j] R. Schiff, *Ann.*, **223**, 247 (1884). [k] *Anal.* Calcd for $C_{13}H_{14}O_4$: C, 66.67; H, 5.98. Found: C, 66.82; H, 5.92.

compensated by the methoxy groups, compound **5** in 10% sulfuric acid was converted to naphthol **6** in moderate



5



6

a, $R_1 = R_2 = H$; $R_3 = R_4 = R_5 = OCH_3$
b, $R_1 = R_2 = R_5 = H$; $R_3 = R_4 = OCH_3$
c, $R_1 = R_2 = R_4 = R_5 = H$; $R_3 = OCH_3$
d, $R_1 = R_2 = R_4 = H$; $R_3 = R_5 = OCH_3$
e, $R_1 = R_2 = R_5 = H$; $R_3, R_4 = O\text{-}CH_2\text{-}O$
f, $R_1 = CH_3$; $R_2 = R_4 = R_5 = H$; $R_3 = OCH_3$
g, $R_1 = R_4 = R_5 = H$; $R_2 = OH$; $R_3 = OCH_3$
h, $R_1 = R_2 = R_4 = R_5 = H$; $R_3 = OH$
i, $R_1 = R_2 = R_4 = R_5 = H$; $R_3 = CH_3$
j, $R_1 = R_2 = R_3 = R_4 = R_5 = H$

yield. The results are summarized in Table I. Attempts made in our laboratories to achieve a facile entry into indoles and benzofurans in this particular way failed because of the instability of the pyrrole and furan ring under the cyclization conditions.

Experimental Section

General. Melting points were determined on a Mettler apparatus and are uncorrected. Nmr data were consistent with the assigned structures (Varian T-60, TMS as an internal standard). The intermediates were characterized by means of nmr and converted as is to the products offered in Table I. All starting materials were commercially available. Grignard derivative **2** was prepared according to a known procedure.[3] The preparation of the naphthalenes is illustrated by the synthesis of **4a**.

1-(1,3-Dioxolan-2-yl)-3-hydroxy-3-(3,4,5-trimethoxyphenyl)propane (3a). To a solution of **2**, prepared from 1.6 g (0.065 g-atom) of magnesium and 12.3 g (0.065 mol) of 2-(2-bromoethyl)-1,3-dioxolane in 50 ml of THF, was added dropwise with stirring a solution of 8.5 g (0.045 mol) of 3,4,5-trimethoxybenzaldehyde (**1a**) in 20 ml of THF. After additional stirring for 4 hr the reaction mixture was poured in 500 ml of a 10% NH_4Cl solution and extracted twice with $CHCl_3$. Washing, drying, and evaporation of the organic phase left a viscous oil, which upon treatment with (*i*-Pr)$_2$O afforded 10.6 g of **3a** as a solid; mp [benzene–petroleum ether] 86–87°. *Anal.* Calcd for $C_{15}H_{22}O_6$: C, 60.40; H, 7.38. Found: C, 60.44; H, 7.57. Nmr ($CDCl_3$) δ 1.75 (m, 4, $-CH_2CH_2-$), 3.24 (s, 1, OH), 4.57 (m, 1, ArC*H*(OH)), 4.84 (m, 1, -OCH(R)O-), 6.60 (s, 2, Ar*H*).

1-(1,3-Dioxolan-2-yl)-3-oxo-3-(3,4,5-trimethoxyphenyl)-propane (5). To a solution of 5 g (0.017 mol) of **3a** in 75 ml of benzene was added 20 g of MnO_2. The mixture was refluxed with stirring for 2 hr. Filtration of the reaction mixture and evaporation of the solvent left 4.2 g (87%) of **5** as a white crystalline solid; mp 57–59° [(*i*-Pr$_2$)O–petroleum ether]. *Anal.* Calcd for $C_{15}H_{20}O_6$: C, 60.81; H, 6.76. Found: C, 60,83; H, 6.93. Nmr ($CDCl_3$) δ 2.14 (m, 2, ArCOCH_2), 3.01 (m, 2, ArCOCH$_2$CH_2), 4.49 (t, 1, OC(R)*H*O), 7.22 (s, 2, Ar*H*).

1,2,3-Trimethoxynaphthalene (4a). A solution of 5.96 g (0.02 mol) of **3a** in 10 ml of methanol was added in 5 min to 100 ml of stirred refluxing 10% sulfuric acid. After 1 hr the reaction mixture was cooled and extracted twice with $CHCl_3$. Washing with 5% $NaHCO_3$ solution, drying, and evaporating of the solvent left an oil, which upon distillation yielded 4.0 g (95%) of **4a**; bp 154–156° (5).

Registry No.—**1a**, 86-81-7; **1b**, 120-14-9; **1c**, 591-31-1; **1d**, 7311-34-4; **1e**, 120-57-0; **1f**, 586-37-8; **1g**, 148-53-8; **1h**, 100-83-4; **1i**, 620-23-5; **1j**, 100-52-7; **3a**, 53579-08-3; **3b**, 53597-09-4; **3c**, 53597-10-7; **3d**, 53597-11-8; **3e**, 53597-12-9; **3f**, 53597-13-0; **3g**, 53597-14-1; **3h**, 53597-15-2; **3i**, 53597-16-3; **3j**, 53597-17-4; **4a**, 5892-02-4; **4b**, 10103-06-7; **4c**, 93-04-9; **4d**, 10075-61-3; **4e**, 269-43-2; **4f**, 2825-01-6; **4g**, 1888-41-1; **4h**, 135-19-3; **4i**, 91-57-6; **4j**, 91-20-3; **5**, 53597-18-5; **6**, 53597-19-6; 2-(2-bromoethyl)-1,3-dioxolane, 18742-02-4.

References and Notes

(1) H. J. J. Loozen and E. F. Godefroi, *J. Org. Chem.*, **38**, 1056 (1973).
(2) H. J. J. Loozen and E. F. Godefroi, *J. Org. Chem.*, **38**, 3495 (1973).
(3) G. Büchi and H. Wüest, *J. Org. Chem.*, **34**, 1121 (1949).
(4) For a review concerning naphthalenes, see "Chemistry of Carbon Compounds," Part IIIB, Elsevier, Amsterdam, 1956, p 1273.
(5) M. Harfenist, A. Bavley, and W. A. Lazier, *J. Org. Chem.*, **19**, 1608 (1954).

Synthesis of Prostaglandins Containing the Sulfo Group

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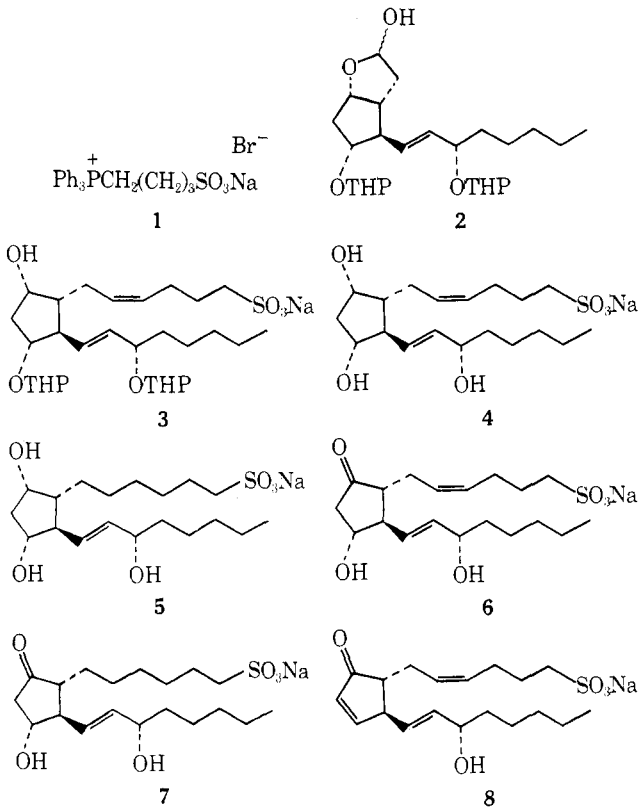
It is a matter of interest to evaluate the biological and pharmacological activities of prostaglandin analogs containing the sulfo group in place of the carboxy function (C-1). We now report the synthesis of prostaglandin sulfonic acids by the Wittig reaction with a phosphorous ylide having the sulfo group.

(4-Sodium sulfonato-*n*-butyl)triphenylphosphonium bromide (**1**) was obtained as colorless crystals, mp 268–270°, from sodium 4-bromo-*n*-butanesulfonate and triphenylphosphine in *N,N*-dimethylformamide on heating. Reaction of the corresponding ylide, prepared from **1** and a solution of sodium methylsulfinyl carbanide in dimethyl sulfoxide, with 2-oxa-3-hydroxy-6-*syn*-(3α-tetrahydropyranyloxy-1-*trans*-octenyl)-7-*anti*-tetrahydropyranyloxy-*cis*-bicyclo[3.3.0]octane[1] (**2**), an intermediate for the Corey synthesis of prostaglandins, in dimethyl sulfoxide at 30° for 3 hr afforded the sulfonic acid **3** as yellow-brown crystals in 48% yield.

According to the procedures of Corey[1,2] and Pike,[3] the sulfonic acid **3** was converted to the corresponding $F_{2\alpha}$ (**4**), $F_{1\alpha}$ (**5**), E_2 (**6**), E_1 (**7**) and A_2 (**8**).



Experimental Section

Preparation of (4-Sodium sulfonato-*n*-butyl)triphenylphosphonium Bromide (1). Sodium 4-bromo-*n*-butanesulfonate (23 g)[4] and triphenylphosphine (85 g) were dissolved in DMF (400 ml) and a solution was heated at 125° with stirring for 10 hr; the solvent was removed by distillation and the residue was washed with ether.

The residue was chromatographed on silica gel using CH_2Cl_2–MeOH (6:1) as eluent to give 1 as colorless crystals (27 g): ir (KBr tablet) ν 1210, 1180, 1038 cm^{-1}; nmr (D_2O) δ 8.02–7.35 (15 H, m), 3.60–3.10 (2 H, m), 3.25–2.80 (2 H, t), 2.42–1.60 (4 H, m).

Preparation of Sodium 6-[2β-{3α-(2-Tetrahydropyranyloxy)-1-*trans*-octenyl}-3α-(2-tetrahydropyranyloxy)-5α-hydroxycyclopent-1α-yl]-4-*cis*-hexenylsulfonate (3). Phosphonium salt (1) (10.8 g), which had been dried under reduced pressure at 100°, was dissolved in dimethyl sulfoxide (50 ml) and the solution was added at ambient temperature to sodiomethyl–sulfinylcarbanide which had been prepared from sodium hydride (1.71 g, content 63.9%) in dimethyl sulfoxide (20 ml) at 70° under nitrogen. After the addition was over, the yellow-red reaction mixture was stirred for 5 min and a solution of 1 (4.00 g) in dimethyl sulfoxide (20 ml) was added and the mixture was stirred at 30° for 3 hr. The bright red reaction mixture was diluted with ice water (500 ml), saturated with sodium chloride, and extracted with ethyl acetate–ether (1:1). The organic layer was washed with brine, dried, and concentrated *in vacuo*. The residue was submitted to column chromatography using methylene chloride–methanol (4:1) as eluent to give 3 (2.44 g, 48% yield) as pale yellow crystals: mp 178–180°; nmr ($CDCl_3$) δ 5.72–5.18 (4 H, m), 4.30–3.70 (4 H, m), 3.70–3.28 (4 H, m), 3.12–2.75 (2 H, m), 1.0–0.73 (3 H, t), 4.82–4.56 (2 H, m); ir (KBr tablet) ν 1200, 1185, 1038, 1023 cm^{-1}; homogeneous by tlc (methylene chloride–methanol 5:1, R_f 0.43).

Preparation of Sodium 6-[2β-{3α-(2-Tetrahydropyranyloxy)-1-*trans*-octenyl}-3α-(2-tetrahydropyranyloxy)-5α-hydroxycyclopent-1α-yl]hexanesulfonate. A solution of 3 (1.08 g) in methanol (60 ml) was reduced under hydrogen atmosphere (1 atm) using 5% palladium on carbon (170 mg) as a catalyst at ambient temperature for 2 hr. Concentration *in vacuo* gave sodium 6-[2β-{3α-(2-tetrahydropyranyloxy)-1-*trans*-octenyl}-3α-(2-tetrahydropyranyloxy)-5α-hydroxycyclopent-1α-yl]hexanesulfonate (913 mg).

Preparation of Sodium 6-{2β-(3α-Hydroxy-1-*trans*-octenyl)-3α,5α-dihydroxycyclopent-1α-yl}-4-*cis*-hexenesulfonate (4). To a solution of 3 (312 mg) in methanol (5 ml) was added several drops trifluoroacetic acid. The mixture was stirred at 19° for 30 min and concentrated *in vacuo*. The residue was subjected to column chromatography on silica gel using methylene chloride–methanol (4:1) as eluent to give 4 (84 mg) as colorless crystals: mp 122–125°; nmr (CD_3OD) δ 5.68–5.28 (4 H, m), 4.31–3.72 (3 H, m), 2.95–2.68 (2 H, m), 1.03–0.77 (3 H, t).

Preparation of Sodium 6-{2β-(3α-Hydroxy-1-*trans*-octenyl)-3α,5α-dihydroxycyclopent-1α-yl}hexanesulfonate (5). Using the experimental conditions described as above, sodium 6-[2β-{3α-(2-tetrahydropyranyloxy)-1-*trans*-octenyl}-3α-(2-tetrahydropyranyloxy)-5α-hydroxycyclopent-1α-yl]hexanesulfonate (283 mg) gave sodium 6-{2β-(3α-hydroxy-1-*trans*-octenyl)-3α,5α-dihydroxycyclopent-1α-yl}hexanesulfonate (59 mg) as a colorless powder: mp 182–183°; ir (KBr tablet) ν 1190, 1060 cm^{-1}; nmr (CD_3OD) δ 5.71–5.39 (2 H, m), 4.28–3.74 (3 H, m), 2.97–2.67 (2 H, m), 1.01–0.75 (3 H, t).

Preparation of Sodium 6-{2β-(3α-Hydroxy-1-*trans*-octenyl)-3α-hydroxy-5-oxocyclopent-1α-yl}-4-*cis*-hexenesulfonate (6). To a solution of sodium 6-[2β-{3α-(2-tetrahydropyranyloxy)-1-*trans*-octenyl}-3α-(2-tetrahydropyranyloxy)-5α-hydroxycyclopent-1α-yl]-4-*cis*-hexenesulfonate (766 mg) in acetone (33 ml) was added Jones reagent (1.8 ml) (prepared by dissolving chromium trioxide (2.67 g) and sulfuric acid (2.3 ml) in water and making up the total volume with water to 10 ml) at −20°, and the solution was stirred at −20 to −15° for 4 hr. 2-Propanol was added to the reaction mixture and the resulting mixture was diluted with brine and the product was extracted with ethyl acetate. The organic layer was washed with brine, dried, and concentrated *in vacuo*. The residue (628 mg) in methanol (5 ml) was treated with 1 *N* hydrochloric acid (0.3 ml) and the mixture was stirred at 27° for 1.5 hr and neutralized with sodium bicarbonate. Concentration *in vacuo* at a low temperature and subjection of the residue to column chromatography on silica gel using methylene chloride–methanol (4:1) as eluent gave sodium 6-{2β-(3α-hydroxy-1-*trans*-octenyl)-3α-hydroxy-5-oxocyclopent-1α-yl}-4-*cis*-hexenesulfonate (204 mg): mp 83–84°; nmr (CD_3OD) δ 5.58–5.27 (4 H, m), 4.13–3.67 (2 H, m), 2.96–2.55 (3 H, m), 1.03–0.67 (3 H, t); homogeneous by tlc (CH_2Cl_2–MeOH 3:1, R_f 0.19).

Preparation of Sodium 6-{2β-(3α-hydroxy-1-*trans*-octenyl)-3α-hydroxy-5-oxocyclopent-1α-yl}hexanesulfonate (7). To a solution of sodium 6-{2β-(3α-hydroxy-1-*trans*-octenyl)-3α-hydroxy-5-oxocyclopent-1α-yl}hexanesulfonate (592 mg) in acetone (81 ml) at −20° was added Jones reagent (prepared as described in above example) (1.5 ml) dropwise, and the mixture was stirred at −20 to −15° for 3.5 hr. 2-Propanol was added to the reaction mixture and the resulting mixture was diluted with brine and the product was extracted with ethyl acetate. The organic layer was washed with brine, dried, and concentrated under reduced pressure. The residue (497 mg) was dissolved in methanol (6 ml), treated with 1 *N* hydrochloric acid (0.36 ml), and stirred at 27° for 1.5 hr and neutralized with sodium bicarbonate. Concentration *in vacuo* at a low temperature and subjection of the residue to column chromatography on silica gel using methylene chloride–methanol (4:1) as eluent gave sodium 6-{2β-(3α-hydroxy-1-*trans*-octenyl)-3α-hydroxy-5-oxocyclopent-1α-yl}hexanesulfonate (173 mg): nmr (CD_3OD) δ 5.69–5.38 (2 H, m), 4.25–3.74 (2 H, m), 2.94–2.56 (3 H, m), 1.03–0.77 (3 H, t).

Preparation of Sodium 6-{2β-(3α-hydroxy-1-trans-octenyl)-5-oxo-3-cyclopenten-1α-yl}-4-*cis*-hexenesulfonate (8). A solution of 5 (64 mg) in 90% aqueous acetic acid (5 ml) was stirred at 55–60° for 16 hr. The reaction mixture was concentrated *in vacuo* and the residue was dissolved in ethyl acetate and washed with brine, and the organic layer was concentrated *in vacuo*. The residue was subjected to column chromatography on silica gel using methylene chloride–methanol (5:1) as eluent to give sodium 6-{2β-(3α-hydroxy-1-*trans*-octenyl)-5-oxo-3-cyclopenten-1α-yl}-4-*cis*-hexenesulfonate (42 mg) as a solid: mp 80–81°; nmr ($CDCl_3$) δ 7.68–7.52 (1 H, q), 6.30–6.12 (1 H, q), 5.75–5.52 (2 H, m), 5.52–5.25 (2 H, m), 4.27–3.96 (1 H, m), 3.43–3.18 (1 H, m), 2.97–2.71 (2 H, m), 1.02–0.75 (3 H, t).

Registry No.—**1**, 53535-01-6; **3**, 53535-02-7; **4**, 53535-03-8; **5**, 53535-04-9; **6**, 53535-05-0; **7**, 53535-06-1; **8**, 53535-07-2; sodium 4-bromo-*n*-butanesulfonate, 53535-08-3; triphenylphosphine, 603-35-0; sodium 6-[2β-{3α-(2-tetrahydropyranyloxy)-1-*trans*-octenyl}-3α-(2-tetrahydropyranyloxy)-5α-hydroxycyclopent-1α-yl]hexanesulfonate, 53535-09-4.

References and Notes

(1) E. J. Corey, N. M. Weinshenker, T. K. Schaaf, and W. Huber, *J. Amer. Chem. Soc.*, **91**, 5675 (1969).

(2) E. J. Corey, T. K. Schaaf, W. Huber, U. Koelliker, and N. M. Weinshenker, *J. Amer. Chem. Soc.*, **92**, 397 (1970).
(3) J. E. Pike, F. H. Lincoln, and W. P. Schneider, *J. Org. Chem.*, **34**, 3552 (1969).
(4) J. H. Helberger and G. Manecke, German Patent 895,559.

Preparation of *cis*-3,4-Ureyleneselenophane[1]

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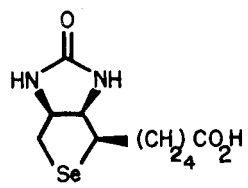
In connection with our continuing study of the synthesis of selenobiotin we have prepared the parent fused biheter-



Selenobiotin

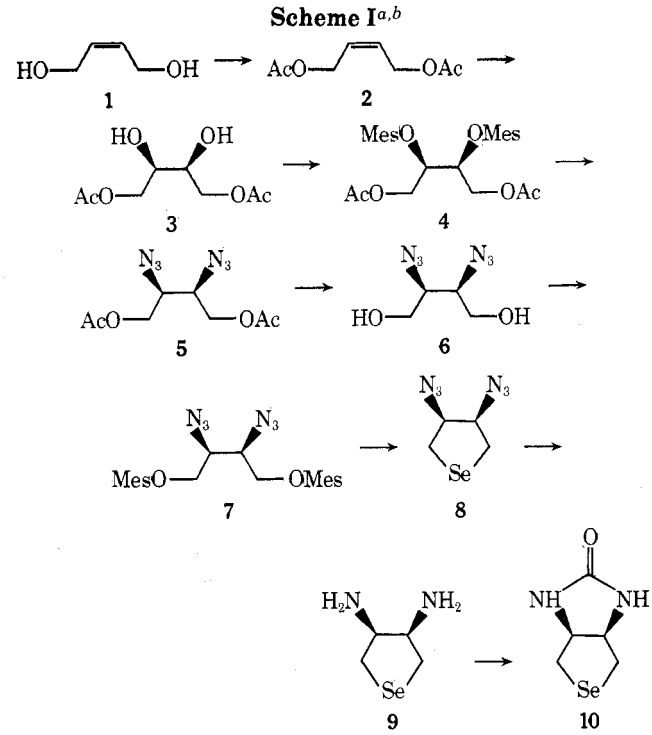
ocyclic system **10**. Effective synthetic entry is based on ring closure with divalent selenium after the cis, vicinal, azide functions, precursors of the needed amine groups, are positioned in a cis relationship on **7**. Approaches utilizing 3,4-disubstituted selenophanes or varied derivatives of *meso*-erythritol other than **4** failed to produce the desired diazidoselenophane (**8**) or diaminoselenophane (**9**), and are the subject of a future communication.

Proof of structure for the diazide **6** was realized by near-quantitative conversion to the known *meso*-2,3-diaminobutane-1,4-diol dihydrobromide[2] and dihydrochloride, by catalytic reduction and treatment with hydrogen halide.

This conversion further establishes the cis stereochemistry of the synthetic intermediates, and of the final product, since this cis arrangement of the nitrogen function is not affected by subsequent reactions. Supporting evidence for this cis, meso sterochemistry is found in the nmr spectra, which present complex multiplets resulting from the six-spin AA'BB'CC' system, the analyses of which are beyond the scope of this report. For instance, the spectra of the noncyclic meso compounds resemble that of meso-1,2,3,4-tetrachlorobutane, rather than *d*-1,2,3,4-tetrachlorobutane,[3] while the nmr spectra of the monocyclic compounds, and *cis*-3,4-ureyleneselenophane, are consistent with that reported for the similar system: *cis*-tetrahydro-2,2-dimethylthieno[3,4-*d*]-1,3-dioxole (the acetone ketal of *cis*-3,4-dihydroxythiophane).[4]

Of the vicinal diazides subsequently described, *meso*-2,3-diazidobutane-1,4-diol (**6**) slowly polymerizes on standing to an unidentified acetone-insoluble substance; in addition, the monomer is initially obtained as a supercooled liquid with such a high heat of fusion that efficient cooling is required at the onset of solidification to avoid detonation. The unexpected stability of some of the diazides was demonstrated by their melting point behavior: the acetate and methanesulfonate diesters, **5** and **7**, give sharp, reproducible melting points, and initially melt in an open flame before deflagrating mildly; under identical conditions, the liquid heterocyclic diazide **8** detonates violently. Most reactions outlined in Scheme I are easily performed within a relatively short period of time under reasonably mild conditions.

Scheme I[a,b]



[a] Compounds **5**–**10** are previously unreported. [b] For all new compounds, except **6** and **8**, analytically pure samples were obtained which gave either satisfactory elemental analyses or mass spectra consistent with the structure indicated.

Evaluation of the biological activity of **10** is underway, and will be separately reported.

Experimental Section

All temperature readings were uncorrected. Ir spectra were determined on a Perkin-Elmer Model 457 spectrophotometer. Nmr spectra were recorded on a Varian A-60 or HA-100D spectrophotometer. Mass spectra were determined at Cornell University, Ithaca, N.Y. Microanalyses were performed by Schwarzkopf Microanalytical Laboratory, Inc., Woodside, N.Y. Sodium selenide was purchased from Alfa Inorganics, Inc., Beverly, Mass.; *cis*-2-butene-1,4-diol was obtained from GAF Corp., Inc., Binghamton, N.Y. Those melting points taken in sealed evacuated capillaries are designated (SEC).

The diacetate **2** and the diol **3** were prepared according to literature procedures.[5]

***meso*-1,4-Di-*O*-acetyl-2,3-di-*O*-(methylsulfonyl)erythritol (4).** A solution of **3** (28.4 g; 0.140 mol) in pyridine (100 ml) is stirred at 0° and treated dropwise with methanesulfonyl chloride (34.4 g, 0.300 mol) over a 0.5-hr period. Stirring is continued 4 hr, and the mixture is then poured into 1 l. of ice water. The crystalline product which separates is collected by suction filtration, washed with several portions of cold water, and air dried to give 50.5 g (99.5%) of **4**: mp 138–140° (SEC) (lit.[7] mp 140–141°).

***meso*-1,4-Diacetyloxy-2,3-diazidobutane (5).** A solution of **4** (36.2 g, 0.100 mol) in DMSO (500 ml) is stirred and warmed to 60° in an oil bath. Finely powdered sodium azide (14.3 g, 0.220 mol) is added portionwise until solution is complete. The solution is then maintained at 90–100° for 24 hr before cooling to room temperature. The solution is poured into 1 l. of ice water. After addition of saturated sodium chloride solution (500 ml), the crystalline product is collected by suction filtration, washed with several portions of cold water, and air dried to give 24.7 g (96%) of **5**: mp (and remelt) 108.5–109.0° (SEC) (CCl_4); ir (KBr) 2180, 2120, 1730, 1320, 1240 cm^{-1}; nmr (pyridine) δ 2.04 (s, 6 H, CH_3), 4.12 (m, 2 H, CH), 4.52 (m, 4 H, CH_2).

Anal. Calcd for $C_8H_{12}N_6O_4$: C, 37.37; H, 5.02; N, 32.70. Found: C, 37.30; H, 4.68; N, 32.90.

***meso*-2,3-Diazidobutane-1,4-diol (6).** A solution of **5** (14.8 g, 0.058 mol) in 0.01% methanolic potassium hydroxide (200 ml) is stirred overnight in an open vessel. Removal of solvent and methyl acetate under reduced pressure and at room temperature produces a pale yellow, slightly opaque oil which is immediately redissolved