

Anal. Calcd for $C_{45}H_{36}O_8 \cdot C_6H_6$: C, 78.24; H, 5.73; benzene, 9.96. Found: C, 78.75; H, 5.73; benzene 7.08.

5-O-Trityl-2-deoxy-D-ribose Ethylene Mercaptal (V).—A solution of 8.8 g (0.042 mole) of 2-deoxy-D-ribose ethylene mercaptal⁴ and 12.3 g (0.043 mole) of trityl chloride in 70 ml of anhydrous pyridine was allowed to stand at room temperature for 3 days. The reaction mixture was poured into a mixture of 350 g of ice and 270 ml of $CHCl_3$ with stirring. After the ice had melted, the $CHCl_3$ layer was removed. The aqueous layer was extracted with 80 ml of $CHCl_3$. The combined $CHCl_3$ solutions were washed as in the previously described acylation but using 120-ml portions. The $CHCl_3$ solution was dried ($MgSO_4$), filtered, and evaporated to dryness under reduced pressure. The residue (23.6 g) was chromatographed on 600 g of alumina as previously described but packing and eluting with C_6H_6 - $CHCl_3$ - CH_3OH (78:20:2) and collecting one hundred 10-ml fractions. Fractions 14–40 were combined and evaporated to dryness under reduced pressure. The residue (19.8 g) was crystallized from 150 ml of C_6H_6 - C_6H_{12} (1:2) to give 6.9 g (36%) of crystalline solid, mp 125–128°.

A portion of the product was recrystallized twice from the same solvent system; mp 130–132°, $[\alpha]_D^{25} -8.8^\circ$ (c 4, $CHCl_3$). The ultraviolet spectrum (C_2H_5OH) had maxima at 230 m μ (ϵ 8150), 253 (896), and 258 (874) with shoulders at 264 and 269 m μ . The infrared spectrum had bands at 3500, 3470, 1590, 1580, 1485, 1065, 775, 760, 740, 705, 700, 690, and 630 cm^{-1} . The nmr spectrum (in $CDCl_3$) had a doublet of doublets centered at δ 1.86 (2 H), a triplet centered at 2.66 (2 H), a singlet at 3.13 (4 H), multiple peaks centered at 3.37 (2 H), multiple peaks centered at 3.89 (2 H), a triplet centered at 4.66 (1 H), and multiple peaks at 7.22–7.58 (15 H).

Anal. Calcd for $C_{28}H_{28}S_3O_3$: C, 68.99; H, 6.23; S, 14.17; O, 10.61. Found: C, 69.17; H, 6.25; S, 14.22; O, 11.08.

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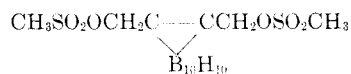
Carboranes. II. An Analog of 1,4-(Dimethanesulfonyl)butane^{1,2}

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The preparation of a boron-containing bismethanesulfonate was undertaken for possible application in neutron-capture therapy.³ An *o*-carborane analog of the alkylating agent, bis-(methanesulfonyl)butane (Myleran),⁴ was synthesized from 1,4-bismethanesulfonylbutyne for evaluation in tumor-bearing animals.



Experimental Section⁵

1,4-Bismethanesulfonylbutyne.—An experimental preparative procedure and the physical characteristics of this compound have not been described, though pharmacological studies are

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(2) Paper I of this series: A. H. Soloway and D. N. Butler, *J. Med. Chem.*, **9**, 411 (1966).

(3) A. H. Soloway in "Progress in Boron Chemistry," Vol. I, A. L. McCloskey and H. Steinberg, Ed., Pergamon Press Inc., New York, N. Y., 1964, pp 203–234.

(4) W. C. J. Ross, "Biological Alkylating Agents," Butterworth and Co. (Publishers) Ltd., London, 1962, pp 37–38.

reported.⁶ A similar procedure to that described for the sulfonation of dihydroxyalkynes⁷ was used.

A solution of 8.6 g (0.1 mole) of 1,4-butyne-1,3-diol in 15.8 g (0.2 mole) of pyridine was added to 100 ml of a 1:1 mixture of THF and ethyl ether. To this stirred anhydrous solution, 22.9 g (0.2 mole) of ethanesulfonyl chloride was added dropwise, maintaining the temperature of the reaction mixture below 5°. Upon completion of the addition, the mixture was stirred for 4 hr at the same temperature. The solution was then concentrated under reduced pressure to one-third of its volume. The pyridine hydrochloride was discarded and the filtrate was treated with an equal volume of water. The mixture became warm and two layers separated. The upper, organic phase was removed and concentrated to an oil which solidified on treatment with ethanol. This product, 5.1 g (21%), was recrystallized from 2 vol of ethanol and yielded white crystals, mp 85.5–86.5°.

Anal. Calcd for $C_6H_{10}O_6S_2$: C, 29.74; H, 4.16; S, 26.47. Found: C, 29.88; H, 4.15; S, 26.23.

1,2-Bis(methanesulfonylmethyl)carborane.—A solution of 4.84 g (0.02 mole) of 1,4-bis(methanesulfonylbutyne) and 2.44 g (0.02 mole) of sublimed decaborane ($B_{10}H_{14}$) in 50 ml of dry acetonitrile was refluxed for 28 hr. The solvent was then removed under reduced pressure and the residue was refluxed in 40 ml of methanol for 4 hr. Removal of the alcohol on a rotatory evaporator left an oil from which 3.4 g (47%) of a crude solid was obtained by use of an ethanol-water mixture. Recrystallization from ethanol yielded 1.5 g of the pure carborane, mp 93–94°.

Anal. Calcd for $C_8H_{12}B_{10}O_8S_2$: C, 19.98; H, 5.59; B, 30.01; S, 17.79. Found: C, 20.23; H, 5.56; B, 29.81; S, 17.54.

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(5) All melting points were taken on a Kofler micro heating stage and are reported as they were observed.

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Some Hydrazine Derivatives of (4-Biphenyl)glyoxal

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4-Biphenylglyoxal and its derivatives have been shown by Cavallini and his co-workers¹ to have both *in vitro* and *in vivo* activity against several viruses. The data presented by these workers indicated that a high degree of antiviral activity was associated with the condensation products of biphenylglyoxals and *p*-aminobenzoic acid, and that these derivatives were better absorbed than the parent glyoxal. In the antibacterial nitrofurans, condensation with a substituted hydrazine confers activity on the weakly active aldehyde.² It was of interest therefore to prepare similar derivatives of (4-biphenyl)glyoxal to compare their activity with that of the parent compound. This communication describes the synthesis of the derivatives listed in Table I.

These compounds, unlike the parent biphenylglyoxal, did not possess *in vivo* activity against herpes simplex or the influenza PR8 virus.³

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(2) "The Nitrofurans," Vol. I, Eaton Laboratories, Norwich, N. Y., 1958. (3) (a) E. L. Anderson, J. E. Casey, Jr., M. Emas, E. E. Force, E. M. Jensen, R. S. Matz, and D. E. Rivard, *J. Med. Chem.*, **6**, 787 (1963). (b) Biological testing of the compounds described in this paper was carried out at Smith Kline and French Laboratories, Philadelphia, Pa.