Recovery of I from Solutions in Concentrated Sulfuric Acid.—2,4-Dinitrobenzenesulfenyl chloride (I), 0.10 g., m.p. 96–97° was finely-ground and added to 20 ml. of 96% sulfuric acid. The mixture was let stand, and stirred occasionally, for one hr. This was filtered through a sintered-glass funnel, the filtrate poured on 50 g. of crushed ice, and the latter mixture was extracted with 50 ml. of carbon tetrachloride. Evaporation of the solvent, from the extract, gave 5 mg. of unchanged I (m.p. and m.m.p. with authentic I, 95–96°).

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NOTES

5-Thiol-7-hydroxy-1-γ-triazolo[d]pyrimidine[1,2]

By Carl Tabb Bahner and Dorothy Ellis Bilancio

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5-Thiol-7-amino-1-γ-triazolo[d]pyrimidine has been reported to be a strong inhibitor of the growth of *Streptococcus faecalis* R.[3] but not of *Adenocarcinoma* 755.[4] The closely related compound having an hydroxy group in place of the 7-amino group has been prepared as described below. It produces a 65% reduction in the growth of *L. casei* at a concentration of 0.005 mg./ml.[5] The effect can be reversed by pteroylglutamic acid or adenine. The results of screening against various tumors are to be published elsewhere.

A solution of 7.0 g. of sodium nitrite in the minimum volume of water was added slowly to a solution of 15.0 g. of 2-thiol-4,5-diamino-6-hydroxypyrimidine[6,7] in one liter of 2 *N* H_2SO_4 at 40°. Stirring was continued 15 minutes after all the sodium nitrite had been added. The solution was chilled for two hours and the solid product was recovered by filtration. It was suspended in boiling water and dissolved by addition of ammonia. The resulting solution was chilled and the crystals which formed were dissolved in boiling water. The hot solution was treated with activated charcoal, filtered hot, acidified with acetic acid and chilled. The crystals which formed were recrystallized from acetone; they then decomposed sharply at 265°. In sodium bicarbonate-buffered solution, the product reacted with iodine in the ratio of 4 atoms of iodine to 1 molecule of product. Grynberg[8] reported similar results with xanthine and guanine. However 8-azaguanine does not behave in the same way. Ultraviolet absorption data for this compound are shown in Table I. The sample for carbon and hydrogen analysis was dried at 110° and 0.1 mm. before analysis, but the analysis indicated the presence of one molecule of water. Roblin,[9] *et al.*, observed that the corresponding dihydroxy compound also was obtained as the monohydrate in spite of careful drying. *Anal.* Calcd. for $C_4H_5N_5O_2S$: C, 25.64; H, 2.69. Found: C, 25.85; H, 2.60.

Table I

Ultraviolet Absorption Spectra

*p*H	λmax.	λmin.
1	242	255
	270	278
	294	
6.5	238	219
	288	253
10	232	252
	276	
11	277	250

We are indebted to Gertrude Elion and George H. Hitchings of the Wellcome Research Laboratories and Lee Bennett, Jr., of the Southern Research Institute for ultraviolet absorption data and to Galbraith Analytical Laboratories for carbon and hydrogen determination.

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[2] Presented in part at the Southeastern Regional Meeting of the American Chemical Society, Auburn, Ala., October 24, 1952.

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On Competition between the Clarke–Eschweiler and Pictet–Spengler Reactions

By Richard Baltzly

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Some years ago in these laboratories Dr. J. S. Buck and the author observed that the attempt to produce dimethylhomoveratrylamine from homoveratrylamine or N-methylhomoveratrylamine by the Clarke–Eschweiler reaction[1] gave mainly 2-methyl-6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline. Similar results were obtained with N-benzylhomoveratrylamine[2] and an attempt to methylate β-(2,5-dimethoxyphenyl)-propylamine by this procedure afforded a compound that was not the corresponding dimethylamine and whose hydrochloride gave analyses consistent with the composition $C_{13}H_{20}ClNO_2$.[3] In this latter case the authors were reluctant to ascribe to this substance the structure of a tetrahydroisoquinoline since there was no activating group in the parent phenethylamine para to the point of prospective ring closure.

More recently Castrillon[4] has reported the cycli-

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