

Pasadena, California) was eluted with  $H_2O$  and crystallized as rosettes from  $H_2O$  by fractional crystallization (final yield, 9.5 mg).

Two types of analyses indicated that the crystalline product consisted of 85% deoxyriboside and 15% free 6-mercaptopurine. Ascending filter paper chromatography with a solvent system consisting of  $(NH_4)_2SO_4$ -isopropyl alcohol- $H_2O$  (5 g of  $(NH_4)_2SO_4$  plus 5 ml of isopropyl alcohol plus  $H_2O$  to a total volume of 100 ml; this mixture was suggested by Dr. GERTRUDE ELION) separated the deoxyriboside,  $R_F$  0.62, and the free base,  $R_F$  0.36. The deoxyriboside upon acid-hydrolysis in 0.12  $N$  HCl for 3 min at 100° was completely split, a fact ascertained by chromatography with the above system. The amount of deoxyribose present was determined by heating with cysteinesulfuric acid<sup>6</sup>; hypoxanthine deoxyriboside was used for a reference standard. The deoxyriboside of 6-mercaptopurine had an absorption spectrum very similar to that of the free base; in 0.12  $N$  HCl the peak absorption was at  $\lambda_{max}$  325 m $\mu$ .

The possible antimetabolic efficacy of 6-mercaptopurine deoxyriboside is being investigated by the group at Wellcome Research Laboratories.

MORRIS FRIEDKIN

Department of Pharmacology, Washington University School of Medicine,  
St. Louis, Mo. (U.S.A.)

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### O-Methylisouronium sulfate, a convenient new guanidinating reagent\*

The conversion of amino groups of peptides and proteins to guanidino groups has received attention in recent years<sup>1,2</sup> and is of interest in the study of chemically modified proteins. HUGHES *et al.* found the common guanidinating reagent, O-methylisouronium chloride to be unstable and they introduced the new reagent O-methylisouronium bisulfate. The preparation of the latter is time consuming, requiring preparation of the chloride from cyanamide, methanol and hydrogen chloride, conversion to the picrate, purification of the picrate and its conversion to the bisulfate and finally purification of the bisulfate.

We have developed a one-step synthesis of analytically pure O-methylisouronium sulfate from cyanamide, methanol and sulfuric acid. This salt appears not to have been described in the literature. This neutral sulfate was as effective as the bisulfate in the guanidination of gelatin at pH 10.5, as indicated by identical reductions of Van Slyke amino nitrogen. The salt appears to be stable to storage at room temperature.

To an ice-cold solution of 10.5 g (0.25 mole) of cyanamide (Eastman Kodak Company, Practical Grade) in 150 ml of methanol (Reagent, anhydrous) was added 12.5 g (0.125 mole) of concentrated sulfuric acid. The solution was filtered from a small amount of insoluble matter and allowed to stand at room temperature for four days. The crystalline precipitate was washed with 50 ml of methanol and dried to yield 13.5 g (44%) of product. An additional 1.5 g (5%) was obtained from the filtrate after two more days. The product had no titratable acidity and melted at 155–156° (uncorr.). The picrate melted at 185–187° (uncorr.), undepressed by admixture of authentic O-methylisouronium picrate prepared according to HUGHES *et al.*<sup>1</sup>.

*Anal. Calcd.* for  $(C_3H_4N_2O)_2SO_4$ :  $SO_4$ , 39.03. *Found*:  $SO_4$ , 39.30.

When excess sulfuric acid was used in an attempt to increase the yield from the filtrate of the first crop, the product was contaminated with the bisulfate.

JAKE BELLO

Gates and Crellin Laboratories of Chemistry, California Institute of Technology,  
Pasadena, Calif. (U.S.A.)

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