Sodium Sulfathiazole Sesquihydrate

When a warm concentrated aqueous solution of sulfathiazole in sodium hydroxide is cooled and the resulting crystalline solid is isolated by filtration, rinsed with a small amount of alcohol to free it of adhering mother liquor and then air dried, sodium sulfathiazole in the form of a hydrate is obtained. Under these conditions our material has regularly been the sesquihydrate. Thus, results found in volatile determinations have regularly been close to 8.7% as compared with 8.9% for the sesquihydrate. This hydrate is a white odorless, crystalline powder, dissolving in water to the extent of 1 g. per 1.9 cc. of water and in ethyl alcohol to the extent of 1 g. per 15 cc. 10% solutions in water are clear and colorless and show *p*H's in the neighborhood of 10. The conclusion that this is a sesquihydrate is further supported by the fact that a nitrite assay shows 83.5–83.9% as compared with a calculated value of 83.9%[1] for the sesquihydrate.

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[1] By calculation each gram of sodium sulfathiazole sesquihydrate contains 0.839 g. of sulfathiazole.

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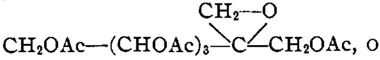
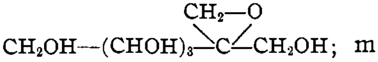
RECEIVED DECEMBER 14, 1940

COMMUNICATIONS TO THE EDITOR

THE ACTION OF DIAZOMETHANE UPON ACYCLIC SUGAR DERIVATIVES

Sir:

In continuation of our studies on the action of diazomethane upon acyclic sugar derivatives,[1] we wish to report the following new results. *keto-d*-Fructose pentaacetate,[2] in absolute chloroform solution containing a trace of methanol, yielded the ethylene oxide derivative (I)  $\mathrm{CH_2OAc—(CHOAc)_3—}\overset{\mathrm{CH_2—O}}{\mathrm{C}}\mathrm{—CH_2OAc}$, on treatment with diazomethane; yield 75%, m. p. 86–87°, spec. rot. +32° (24°, *c* 3, abs. $CHCl_3$, D-line). Saponification of I with barium methylate (0.05 calcd. amount) followed by carbonation yielded II,  $\mathrm{CH_2OH—(CHOH)_3—}\overset{\mathrm{CH_2—O}}{\mathrm{C}}\mathrm{—CH_2OH}$; m. p. 136°. Both I and II showed no coloration on heating with a methanol solution of potassium hydroxide but reduced Tollens reagent (pyridine solution of I).

Treatment of 1-diazo-1-desoxy-*keto-d*-glucoheptulose pentaacetate (III)[1] with an ethereal solution of dry hydrogen chloride produced 1-chloro-*keto-d*-glucoheptulose pentaacetate; m. p. 100–101°, spec. rot. −5.5° (22°, *c* 5, abs. $CHCl_3$, D-line). Similar treatment of III with hydrogen bromide yielded the corresponding 1-bromo-*keto-d*-glucoheptulose pentaacetate; m. p. 86–87°, spec. rot. −4° (24°, *c* 5, abs. $CHCl_3$, D-line). Treatment of a suspension of III in hot water with silver oxide, followed by silver ion removal with hydrogen sulfide and concentration, yielded 2-desoxy-*d*-glucoheptonolactone tetraacetate (IV); m. p. 129–130°, spec. rot. +39.5° (20°, *c* 4, abs. $CHCl_3$, D-line). Saponification of IV with barium hydrate, followed by removal of barium ion with sulfuric acid and concentration, yielded a crystalline product which is under further investigation; m. p. 170°, spec. rot. +20° (26°, *c* 5, H_2O, D-line).

Full details will be communicated in a later publication.

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RECEIVED JANUARY 20 1941

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[1] M. L. Wolfrom, D. I. Weisblat, W. H. Zophy and S. W. Waisbrot, THIS JOURNAL, **63**, 201 (1941).

[2] C. S. Hudson and D. H. Brauns, *ibid.*, **37**, 2736 (1915); E. Pacsu and F. V. Rich, *ibid.*, **55**, 3018 (1933); M. L. Wolfrom and A. Thompson, *ibid.*, **56**, 880 (1934).

APPLICATION OF THE SULFITE CLEAVAGE OF THIAMIN TO THE YEAST FERMENTATION METHOD

Sir:

The problem of specificity in the yeast fermentation method[1,2] for the determination of thiamin has been partially solved by the use of a technique involving oxidation of thiamin to the inactive thiochrome by means of alkaline ferricyanide.[3,4] Application of this method to substances of low potency, *e. g.*, refined white flour, requires extraction and concentration of the thiamin with the attendant dangers of loss or destruction. The work of Williams and co-workers[5] on the sulfite

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[1] Schultz, Atkin and Frey, THIS JOURNAL, **59**, 2457 (1937).

[2] Schultz, Atkin and Frey, *ibid.*, **60**, 1514 (1938).

[3] Schultz, Atkin and Frey, *ibid.*, **60**, 3084 (1938).

[4] Schultz, Atkin and Frey, *J. Biol. Chem.*, **136**, 713 (1940).

[5] Williams and Spies, "Vitamin B_1 and Its Use in Medicine," The Macmillan Co., New York, N. Y., 1938, p. 146.