NOTES

The Reduction of l-Cystine to l-Cysteine.—In a recent paper by Max Bergmann and Giorgios Michalis[1] a method is described for the catalytic hydrogenation of l-cystine to l-cysteine by means of "Palladium Mohr" and hydrogen. The authors also emphasized the possible importance of such a method for certain biological work in which metal-free cysteine preparations are desirable.[2] The use of dissolved metal salts for the reduction can be excluded with this method.

On the other hand Vincent du Vigneaud, L. F. Audrieth and H. S. Loring have presented a paper on "The Reduction of Cystine in Liquid Ammonia by Metallic Sodium" at the meeting of the Federation of the American Societies for Experimental Biology held at Chicago University on March 26, 1930. Without actually having heard this paper it is to be assumed from its title that it has anticipated the results obtained by the present author on the same subject, who also had found the system liquid ammonia–sodium to be a suitable reducing agent for l-cystine. However, some sodium sulfide and alanine also were found to be formed in the course of the reaction.

Another reduction method for l-cystine using aluminum amalgam as a reagent was simultaneously developed by the author and found to have some merits. This amalgam offers the possibility of reducing in aqueous solution, a minimum amount of dissolved metal salts being involved in the reaction due to the insolubility of the aluminum hydroxide formed. This and other advantages of the aluminum amalgam reduction process have been emphasized already several times before. An actual example was given in THIS JOURNAL in the reduction of phenylsulfonylchlorides to the corresponding mercaptans.[3]

A brief outline for the l-cystine reduction, therefore, will be sufficient. l-Cystine is dissolved in carbon dioxide-free water and 1–1.5 times its weight of aluminum amalgam[4] is added. The mixture is heated for approximately one hour. During this time the evolution of some hydrogen sulfide is noticed (hydrogen sulfide is also evolved in the course of the ordinary reduction using tin and hydrochloric acid; see Harrison, Ref. 2). The material is filtered with suction and washed with carbon dioxide-free water until the test with ferric chloride is negative. The filtrate and washings are combined, acidified with hydrochloric acid and evaporated to dryness in a vacuum at as low a temperature as possible. The crystalline

[1] Bergmann and Michalis, *Ber.*, **63**, 987 (1930).

[2] O. Warburg and S. Sakuma, *Arch. ges. Physiol (Pflügers)*, **200**, 203 (1923); S. Sakuma, *Biochem. Z.*, **142**, 68 (1923); D. C. Harrison, *Biochem. J.*, **18**, 1009 (1924).

[3] E. Gebauer-Fuelnegg, THIS JOURNAL, **49**, 1386 (1927).

[4] Prepared according to the procedure given by Wislicenus, *J. prakt. Chem.*, **54**, 18 (1896).

residue consists mainly of l-cysteine hydrochloride, which can be separated readily from very small amounts of foreign matter by recrystallization.

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Chlor Phenol Red.—During the past summer this Laboratory had occasion to examine colorimetrically several commercial samples of this indicator from various sources. The wide variation in the color produced by the indicator in buffer solutions led to a careful examination of the samples.

Anal. Calcd. for dichlorophenolsulfonephthalein, $C_{19}H_{12}O_5SCl_2$: S, 7.58; Cl, 16.76. Found: Sample A, S, 6.76; Cl, 13.5; Sample B, S, 6.54; Cl, 16.00; Sample C, S, 7.14; Cl, 12.10; Sample D, S, 6.48; Cl, 8.46.

This indicator was first prepared by Cohen[1] by condensing *o*-chlorophenol with the crystalline anhydride of *o*-sulfobenzoic acid and subsequently crystallizing the crude product from glacial acetic acid.

The experience of this Laboratory has been that repeated crystallization from acetic acid will not give a pure product. By repeatedly dissolving the crude dye in hot water, acidifying with hydrochloric acid and concentrating on the water-bath to the point of crystallization, a product was obtained that gave the analysis: S, 7.60; Cl, 16.66.

This sample consisted of small, fine, greenish-brown crystals and gave an entirely different color in buffer solutions than samples heretofore examined. The alkaline color, besides being much more intense, contained more blue and less red than the commercial samples.

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[1] *U. S. Public Health Reports*, **41**, No. 53 (1926).