

leaves nothing on the filter. The compound has not been obtained in a crystalline form.

When the various precipitates discarded in the preceding preparation subsequent to the chlorate precipitation are collected and extracted with large volumes of boiling water, a second mercury compound can be salted out of the extracts by sodium chloride. The yield is extremely small, about 0.5% or less, of the weight of methylene blue used. This substance can be washed on the filter free from chlorides and unmercurated methylene blue. The compound gives a strong qualitative test for mercury. Only enough for two analyses has been obtained.

Anal. Subs., 0.3230: CO₂, 0.4069; H₂O, 0.0986. Subs., 0.2325: 16.2 cc. of dry N₂ (25°, 721 mm.). Calcd. for C₁₆H₁₆N₃SClHg: C, 37.06; H, 3.11; N, 8.11. Calcd. for C₁₆H₁₆N₃SClHg.2H₂O: C, 34.63; H, 3.63; N, 7.58. Found: C, 34.64; H, 3.44; N, 7.47.

The combustions were carried out with lead chromate; any oxygen was removed from the nitrogen by means of sodium hydrosulfite. The samples were dried over phosphorus pentoxide at room temperature.

The formulas suggested for these compounds in the introduction are no more than possibilities. The work so far done is inadequate to establish formulas, and the problem is, moreover, complicated by the ease with which methylene blue can be converted into other dyes. In the course of the work small quantities of mercury derivatives of dyes other than methylene blue were, in fact, isolated. One of these had the color and reactions of a methylene-azure derivative.

I wish here to thank Dr. Stieglitz for his kindness to me and for his encouragement during this work.

Summary

1. The work upon the mercuration of methylene blue prior to that described in this paper is reviewed.
2. The mercuration of the benzoyl derivative of leuco methylene blue is described, and it is shown that this reaction, in contrast to most mercurations, does not go to completion except in the presence of a considerable excess of mercuric acetate.
3. An account is given of the preparation of small quantities of two mercury derivatives from methylene blue.

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NOTES

Liberation of Hydrogen from Carbon Compounds.—Under this heading Fry, Schulze and Weitkamp¹ have recently published a paper in which they describe the action of fused alkali upon acetylene, methyl alcohol and formaldehyde, and further propose to investigate the action of alkali on various carbon compounds from the point of view of the more or less general reaction in which, under the influence of fused alkali, hydrogen is liberated and the carbon compound oxidized.

¹ Fry, Schulze and Weitkamp, *THIS JOURNAL*, **46**, 2268 (1924).

In this paper no mention is made of work by Boswell and the writer² in which investigation was made of the action of fused alkali on a long series of compounds, both organic and inorganic. Some of the results obtained may well be of interest to Professor Fry and his collaborators, and in particular their attention is called to the results of the fusion with resorcinol, which are contrary to their description of this reaction. The experimental results showed that, like a number of other compounds, resorcinol does not take part in this general reaction and does not give off hydrogen until it has first absorbed atmospheric oxygen, and has thus presumably been oxidized to some compound which does take part in the general reaction.

The writer will not touch on the theoretical interpretations of this general reaction (to which it is possible that Professor Boswell may later have something to add) but at present merely wishes to call attention to the previous work in this field.

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Preparation of Alkyl Sulfides.—This Laboratory has recently had occasion to use several alkyl sulfides. These sulfides were the purest obtainable. Without exception they all showed the presence of appreciable quantities of mercaptans with the so-called "doctor test" (sodium plumbite solution and free sulfur). It is customary to shake the sulfides repeatedly with sodium hydroxide solution in the final stages of their preparation. This treatment is to remove the impurities including the mercaptans. Due to the ease with which the sodium salts of mercaptans dissociate in aqueous solutions, their removal is never complete with the alkali wash. The complete removal of the mercaptans can be readily accomplished by distilling (preferably under reduced pressure) the alkyl sulfide over finely divided copper. The copper mercaptide is thereby formed and may remain in the residue as such; or if the temperature of distillation is in the neighborhood of 125°, the mercaptide will decompose, in part at least, forming copper sulfide and the corresponding alkyl sulfide. In either case the mercaptans are completely decomposed.

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² Boswell and Dickson, *THIS JOURNAL*, 40, 1773, 1779, 1786 (1918).