

by the addition of sodium carbonate. The experiments on this point have been repeated several times, and in no instance was any important loss of nitrate observed when an excess of sodium carbonate had been added to the solution. Indeed it seems difficult to believe that nitric acid could be volatilized and expelled from a carbonate solution under the conditions usually obtaining in this determination.

It is furthermore of interest that no loss of nitrate was observed from the use of sodium carbonate alone. As was pointed out by Chamot and Pratt¹ carbonates may occasion slight losses of nitrates, due to mechanical causes such as effervescence produced by the reaction of the phenoldisulfonic acid reagent on the carbonates. If sufficient caution be observed, however, such losses may become negligible.

The data submitted in this paper indicate that the making of broad generalizations from specific instances may be entirely unwarranted. The fact that sodium sulfate (supposed to be none other than the normal sulfate) causes a loss of nitrate nitrogen, does not justify the sweeping conclusion that the sulfate radical in any inorganic combination would act in the same way. Although the evidence is of an indirect nature, the above data seem to justify the conclusion that losses of nitrate during evaporation in the presence of sulfates are probably referable to acid sulfates which were either originally present in the solution or perhaps formed by hydrolysis during the evaporation process. The latter view seems the more tenable, since losses took place only in the presence of the sulfates that most easily give rise to the bisulfate.

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NOTE.

Zincoso-zincic Chloride. (Preliminary Paper.)—It has been shown by Borchers and Stocken² that when metallic calcium is melted in presence of calcium chloride, a mass of transparent, red, pleochroitic crystals are formed. This compound was proved by them to be sub-chloride of calcium. This suggested us to investigate the action of metals on fused salts generally. The first case that has been taken for study is the action of metallic zinc on fused zinc chloride. It has been found that zinc dissolves freely in fused zinc chloride to form a bluish white mass and this proved to be a zincoso-zincic salt of the formula, Zn_3Cl_5 .

For a preparation of the compound, zinc chloride is melted in a crucible and then metallic zinc is gradually added, when it begins to dissolve. After some time of addition, the added zinc remains undissolved and forms a globule at the bottom of the crucible. The supernatant fluid is next poured in a basin and allowed to cool when it sets to a homogeneous,

¹ THIS JOURNAL, 33, 25.

² Z. *Electrochem.*, 8, 757 (1902).

bluish white mass. The substance was analyzed with the following results:

0.3196 gave 0.7251 AgCl : Cl = 48.51. 0.2810 gave 0.1824 ZnO : Zn = 52.15. Calculated for Zn_2Cl_3 ; Zn = 52.43; Cl = 47.31.

The substance is extremely hygroscopic and rapidly undergoes hydrolysis when treated with water. The compound is evidently a subchloride of zinc and its constitutional formula could be conveniently represented by the formula $ZnCl \cdot 2ZnCl_2$.

We are at present engaged in studying this compound more fully and attempting to prepare, if possible, the true subchloride of zinc, *viz.*, $ZnCl$, as also similar other sub-salts.

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HYDANTOINS: THE HISTORY OF 2-THIOHYDANTOIN. XXII.

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Received April 11, 1913.

2-Thiohydantoin (I) is not listed in the latest edition of Richter's *Lexikon der Kohlenstoff Verbindungen*, nor is it described in Beilstein's *Handbuch*. Therefore, one who depended upon these works for his literature references, would naturally conclude that such a compound has not been synthesized. This omission is an error. We find that this supposed, unknown compound was described several years ago (22), but apparently has been entirely overlooked by investigators in this field.¹

2-Thiohydantoin (I), $NH.CSNH.CH_2CO$ was apparently first synthesized by Peter Klason and is described in a paper entitled "Ueber Senfölessigsäure und Thiohydantoin," which was published by this investigator in 1890.² Klason prepared the hydantoin by heating the hydrochloride of ethyl aminoacetate with potassium thiocyanate at 140–150°. Regarding the yield obtained by him, we have no knowledge because his paper is not accessible to us. The constitution of the hydantoin was established by its behavior on hydrolysis. Klason evidently obtained thiohydantoic acid (IV) by heating the hydantoin (I) with barium hydroxide. This acid crystallized from water in prismatic crystals and was

¹ In connection with our hydantoin researches we are making a bibliography of papers on hydantoin and thiohydantoin. We intend to publish this later together with a bibliography of papers on the glyoxaline compounds. Since it is our desire to have these as complete as possible, the writer will consider it a favor if informed of any papers, which are not reviewed in *C. A.* or the *Chem. Zentr.*, and therefore likely to be overlooked.

² *Ofv. kongl. Vet.-Ak.*, 87 (1890); *Chem. Ztg.*, 14, Rep., 200; *Chem. Zentr.*, 2, 344 (1890).