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444 Arthur Stahler: On the effect of Calcium Oxide to Hydrazine Hydrate.

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For the preparation of the anhydrous hydrazine there are four known methods: 1) from sodium methylate and hydrazine hydrochloride by evaporation and freezing, 2) by distillation of hydrazine hydrate from barium oxide1, 3) by heating hydrazine borate or 4) by distilling hydrazinecarboxylic acid over calcium

1) Lobry de Bruyn, Rec trav. chim. Pays-Bas 13, 433 [1894], 15, 174 (1896).

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or barium oxides 1). Of these methods, 2) is the most common. In attempting to replace the barium oxide is calcium oxide through, I made ​​the following observation: -

210 g of pure hydrazine hydrate (bp 119°, obtained by distillation of the commercial Raschig preparation) were placed in a copper flask that could be cooled from the outside. Attached to the flask an efficient reflux condenser was fitted using cork. At the same time, 700 g calcium oxide, from marble, which was used partly as powder but partly nut-sized pieces in some cases, was added to the hydrazine hydrate. There was soon a lively heating to 120°, after some time and after some initial vigorous swelling, the calcium oxide disintegrated to a completely dry powder. It has now been heated with a descending condenser in an oil bath. At 145-150 ° almost no liquid went through. Then slowly free hydrazine began to distill, from the first fraction of which was collected 40 g. With increasing temperature, the distillate was visibly ammonia-rich, two other fractions yielded 54 g and 23 g of hydrazine contaminated by some ammonia. The boiling point of all fractions was 113-114°, which was in good agreement with that calculated for N2H4 (113.5 °). Yield 117 g instead of 134 g theoretically

The mechanism of the reaction between calcium oxide and hydrazine hydrate has not yet been elucidated. Either it is the reaction product is a solid solution of hydrazine in calcium oxide - hydroxide or a compound Ca(ON2H5)2, which was based on the following equation:

2CaO + 2N2H5(OH) = Ca(ON2H5)2 + Ca(OH)2.

At moderate temperatures, the compound dissociated into Ca(OH)2 and N2H4, which passes over. At higher temperatures, further decomposition occurs in the form of nitrogen and ammonia, as has been determined by Scott 2 as does hydrazine hydrate. By the above method it is possible to preparation of hydrazine itself, idly from the first fraction while the later (containing some ammonia) should be collected separately.

1) Stolle, diese Berichte 37, 4523 [1904].

2) Proc. Chem. Soc. 20, 84 [1904]; Journ. Chem. Soc. S5, 913 [1904].

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