THE PREPARATION OF CREATININE FROM CREATINE.

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Since pure creatine can be obtained rather easily it would be desirable to be able to use it as a starting point for the preparation of creatinine. Recent investigations from several laboratories have shown that by heating creatine with dilute mineral acids it is possible to convert it quantitatively into creatinine for analytical purposes. No one, however, has yet shown how to apply this method for the preparation of pure creatinine in dry crystallized condition. The method described below does not involve the use of mineral acids or of any solvent. For the transformation of creatine into creatinine we make use only of the water of crystallization of the former, and heat. The dry creatine is converted directly into crystalline creatinine.

The procedure is as follows: Creatine is transferred to a glass stoppered bottle. The closed bottle is placed inside an ordinary preserving jar, the lid of which is held down by a clamp. This jar is then placed in water in an autoclave, and heat is applied until a pressure of 4.5 kilos per sq. cm. is developed. This pressure is maintained for three hours. After being cooled and opened, the contents in the bottle are found to consist entirely of coarsely crystalline creatinine.

The mixture may be a little less white than the original substance, and small amounts of ammonia are apt to form. For purification, the substance is either washed a few times with small quantities of cold alcohol, or it is boiled a few minutes with a very small quantity of absolute alcohol and then washed with a little cold alcohol. About 90 per cent of the theoretical yield of creatinine, consisting of large crystals and assaying 99-100 per cent, is obtained in this way.

Anhydrous creatine does not yield creatinine when subjected to the same treatment. In the presence of a little added moisture, one drop of water for each gram of creatine, the transformation into creatinine proceeds a little more rapidly, but the reaction is then accompanied by the formation of more coloring matter and more ammonia. It is a curious and somewhat unexpected fact that the ammonia generated does not seem to interfere with the reaction.

The creatine used as starting material need not be free from creatinine. Indeed any mixture of creatinine and creatine can by this treatment be converted into creatinine. Other impurities such as mineral acids or inorganic salts must of course be absent as they will otherwise remain in the final product. The method can be applied to large or small quantities of material with equally good results, as the following two experiments show.

Twenty-five grams of crude creatinine zinc chloride were decomposed in the usual manner with lead hydroxide. From the final clear solution was obtained a precipitate weighing 13.5 grams. The colorimetric assay showed this to be a mixture of 40 per cent creatine and 60 per cent creatinine. After heating two and one-half hours in the autoclave, and washing the product with alcohol, 10.5 grams of dry pure creatinine were obtained.

From 100 grams of the same creatinine zinc chloride decomposed in a similar manner we first allowed some of the creatine to separate out by fractional crystallization. After being recrystallized till free from creatinine this fraction weighed 12.4 grams. The mother liquors were then evaporated to a small volume and precipitated by the addition of alcohol, 50.9 grams of a mixture containing 74 per cent creatinine and 26 per cent creatine were obtained. After heating in the autoclave and washing with alcohol, we obtained 46.8 grams of pure creatinine.

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