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AN IMPROVED METHOD FOR THE PREPARATION OF GLYCINE

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The classical method of preparing glycine from monochloroacetic acid and ammonia (1) has been improved by several investigators (2, 3, 4,), but proved generally unsatisfactory. One of the defects is the contamination with large amounts of normal by-product of ammonium chloride, and side reaction products of diglycine and triglycine, which hinder the crystallization and purification of glycine. After much surveying and comparing, we found a simple method for the isolation of pure glycine at a high yield.

The reaction mixture, in which a 60 to 1 molar ratio of ammonia to monochloroacetic acid was employed, was concentrated to crystallize the main portions of glycine and ammonium chloride. After cooling, the precipitate was filtered, and washed with a little amount of cold water. In this way we could separate syrupy diglycine and triglycine almost perfectly from the crystalls. It was proved that very little glycine escaped in the filtrate.

Robertson (2) states "It seems reasonable that either the di-acid or the tri-acid might make a mixture salt or amide with both glycine and ammonia, thus making the impurity persistent in material of low solubility.....Its principal role is that of interfering with the crystallization of glycine and of contaminating the product." On examining our experiments, however, we found that glycine and ammonium chloride in the solution did not combine with diglycine and triglycine to a complex mixture.

For the purpose of isolating glycine from the mixture of glycine and ammonium chloride, magnesium oxide was added to the solution of the mixture.

$$MgO + 2NH_4Cl = 2NH_3 + MgCl_2 + H_2O$$

Since magnesium oxide is insoluble in water, magnesium chloride very soluble in strong alcohol (about 50%), and glycine sparingly soluble in strong alcohol (about 0.2%), we could easily isolate glycine in a pure state using the different solubilities of these substances.

As was suggested by Robertson (2), most investigators have used 60 mols of ammonia and 1 mol of monochloroacetic acid. We compared our method with the methods of several investigators.

Table 1.

Author	Molar ratio of ammonia to monochloroacetic acid	Reagents for separation	Yield of glycine of the theoretical
Robertson	60 : 1	Silver oxide and methanol	50%
Boutwell and Kuick	60 : 1	Pyridine and methanol	54
Orten and Hill	60 : 1	Methanol	60–64
Sasaki and al.	60 : 1	Magnesium oxide and ethanol	76

According to Robertson(2), the molar ratio of ammonia to monochloroacetic acid used and actual amount of glycine formed were as follows.*

Table 2.

Molar ratio of ammonia to monochloroacetic acid	2 : 1	4 : 1	6 : 1	15 : 1	24 : 1	60 : 1	220 : 1
Primary amine produced % of theoretical	29	32	37	58	65	86	95

Experimental

One mol (94.5 g.) of monochloroacetic acid was dissolved in 4 liters of ammonium hydroxide (sp. gr. 0.90) in a stoppered bottle at a room temperature for two days. The excess of ammonia

* The mechanism and kinetics of glycine synthesis are now under investigation.

was distilled off and recovered. The solution was concentrated under reduced pressure until most of glycine and ammonium chloride were crystallized out. After cooling, the precipitate was filtered with Büchner funnel, pressed well, and washed with a small amount of cold water. The filtrate and washing were combined, concentrated and crystallized again.

The combined precipitates were dissolved in water, and the solution boiled by adding about 20 g. of magnesium oxide. After the odor of ammonia has vanished, excess of magnesium oxide, if presented, was filtered off and neutralized with hydrochloric acid. The filtrate was concentrated to almost dryness. The glycine was separated out by gradually adding, with stirring, 500 cc. of 95% alcohol. The mixture was filtered, and washed with 95% alcohol until the filtrate did not contain chlorine ion by testing with silver nitrate. The precipitate, consisting almost pure glycine, was recrystallized with water. The yield was 56.7 g. (theoretical 75.6%). The product was free from chloride by testing with silver nitrate, and ammonium ion by testing with Nessler's solution. Ash content was less than 0.01%. It melted at 235–237°, and showed the theoretical percentage of nitrogen.

Summary

For the preparation of glycine from monochloroacetic acid and ammonia, we have succeeded to obtain pure glycine at a high yield by excluding the by-product of ammonium chloride and the side-reaction products of diglycine and triglycine by magnesia-alcohol method.

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