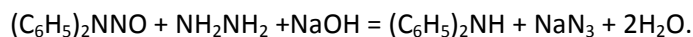


German Patent 273667 A method for producing hydrazoic acid or azide salts
DR. HERMANN STAUDINGER-IN ZURICH Patented in the German Reich of 27th May 1913

In the patent 205683, a method for the preparation of hydrazoic acid is described, by which you can win this acid from the reaction in alkaline alcoholic solution between hydrazine hydrate or hydrazine and nitrous acid.

It has now been found that the hydrazoic acid is also formed in good yield, if one brings together hydrazine or hydrazine hydrate and nitrosamines from a secondary amine such as diphenylnitrosamine in alkaline alcoholic solution. The reaction takes place slowly in the cold, rapidly on heating, after the equation:



The reaction is new and it was not expected that the diphenylnitrosamine would react like amyl nitrite in this reaction. The method differs advantageously from the above, since the hydrazoic acid salt can be prepared in the pure state very easily. Further, the use of the diphenylamine is cheaper and more convenient as that of amyl alcohol.

Example 1:

A solution of 1 mole of soda in 20 parts of ethyl alcohol or methyl alcohol is added to 1 mole of hydrazine hydrate and adding a concentrated solution of diphenylnitrosamine in alcohol to the hot reaction solution slowly. After several hours of heating the sodium azide salt separates in almost pure form from the reaction solution and is filtered off after cooling. Yield 70 percent.

From the mother liquor, a further small quantity of azide salt after evaporation of the alcohol, then treating the residue with water and filtered of the un-dissolved diphenylamine. From the aqueous filtrate hydrazoic acid can be obtained after the addition of small amounts of dilute sulphuric acid. The recovered diphenylamine is almost pure and can be used again for the preparation of diphenylnitrosamine after drying.

The present method can also change in such a way that, instead of adding pure diphenylnitrosamine an alcoholic solution of diphenylamine with the calculated amount of nitrous acid from a calculated amount of sodium nitrite and hydrochloric acid may be used and the resulting solution treated as above.

Example 2:

Instead of the free hydrazine one can also use hydrazine salts and then changes the method as follows. Hydrazine sulphate is poured into ten times the amount of alcohol in which 3 moles of sodium hydroxide have been dissolved. To the hot solution is added the solution of diphenylnitrosamine and again allowed to react as above and refluxed for 7 hours; here a mixture of sodium azide and sodium sulphate result. The alcohol is distilled directly from the solution and the residue extracted by treatment with water; the sodium salts are so separated from the diphenylamine. After filtration, the aqueous solution is mixed with the calculated quantity of sulfuric acid and distilled to recover the hydrazoic acid from the water. Yield about 70 percent.

Instead of sodium hydroxide or potassium hydroxide soda or potash may be used with the same success.

PATENT CLAIM:

A process for the preparation of hydrazoic acid or salts thereof, from hydrazine salts or hydrazine hydrate and nitrosamines of secondary amines, such as diphenylamine, in alcoholic solution with the addition of alkali or alkali metal carbonate is presented.