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PATENT



SPECIFICATION

Application Date, June 17, 1918. No. 9952/18.

Complete Left, Dec. 10, 1918.

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PROVISIONAL SPECIFICATION.

Improvements in and relating to the Production of Azides.

I, WILLIAM RICHARD HODGKINSON, of Ordnance College, Woolwich, London, S.E. 18, Professor of Chemistry and Metallurgy, do hereby declare the nature of this invention to be as follows:—

This invention relates to the production of azides.

- 5 The object of the invention is to provide a process of producing azides from salts of hydrazine without the employment of ethyl benzoate or the like, and the formation of benzoyl hydrazine.

- According to the invention, salts of hydrazine are diazotised by means of nitrous acid or a suitable nitrite under such conditions that the reaction mass
10 is at no time so distinctly acid as to redden litmus paper, but appears on the border line of acidity and alkalinity.

The conditions of acidity under which the reaction is carried out according to the invention, are preferably comparable with the conditions of acidity as indicated by litmus paper, of a solution of boric acid or a like substance.

- 15 Thus, in accordance with the invention, the nitrite solution is brought together with a salt of hydrazine in approximately the proportions required on the basis of theory to diazotise the hydrazine, and an acidified or acid salt of the metal of which the azide is required, is introduced into the mixture under such conditions that the acidity of the reaction mass never exceeds the
20 degree of acidity indicated above, or alternatively, the mixture is added to the solution of the salt of the metal.

- For example, silver azide is formed by adding a neutral solution of hydrazine sulphate and sodium nitrite in molecular proportions to a solution of silver nitrate of a degree of acidity enabling the conditions in regard to acidity set
25 forth above to be realised, while avoiding the precipitation of silver sulphate.

In producing lead azide, a solution of a hydrazine salt of an acid which will not result in the precipitation of an insoluble lead salt of the acid in question, for instance, hydrazine nitrate, is employed.

Dated this 17th day of June, 1918.

COMPLETE SPECIFICATION.

Improvements in and relating to the Production of Azides.

I, WILLIAM RICHARD HODGKINSON, C.B.E., of Ordnance College, Woolwich, London, S.E. 18, Professor of Chemistry and Metallurgy, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the production of azides. 5

The object of the invention is to provide a process of producing azides from salts of hydrazine without the employment of ethyl benzoate or the like, and the formation of benzoyl hydrazine.

According to the invention, salts of hydrazine are diazotised by means of a suitable nitrite under such conditions that the reaction mass is at no time 10 so distinctly acid as to redden litmus paper, but appears on the border line of acidity and alkalinity.

The conditions of acidity under which the reaction is carried out according to the invention, are preferably comparable with the conditions of acidity as indicated by litmus paper, of, for instance a solution of boric acid. 15

Thus in accordance with the invention, the nitrite solution is brought together with a salt of hydrazine in approximately the proportions required on the basis of theory to diazotise the hydrazine, under such conditions that the acidity of the reaction mass never exceeds the degree of acidity indicated above, and an acidified salt of the metal of which the azide is required, is introduced into 20 the mixture or alternatively the mixture is added to the solution of the salt of the metal.

For example, silver azide is formed by adding a neutral solution of hydrazine sulphate and sodium nitrite in molecular proportions to a solution 25 of silver nitrate of a degree of acidity with nitric acid enabling the conditions in regard to acidity set forth above to be realised, while avoiding the precipitation of silver sulphate. Thus, for example, 130 gms. of hydrazine sulphate is converted into the neutral hydrazine ammonium sulphate, which is more soluble in water than is the simple hydrazine sulphate, by the addition of ammonia solution, the mixture being made up to about 500 ccs. To this 30 solution contained in a vessel in which it can be violently agitated, the calculated amount of sodium nitrites dissolved in 500 ccs. of water is added slowly so that the temperature does not rise much above 30° C.

The reaction resulting in the formation of sodium azide may be represented by the following equation:— 35



From this equation it will be seen that the solution would become acid as the result of the reaction were it not for the fact that sodium nitrite is never pure but is alkaline, the alkali in the nitrite ordinarily being sufficient to maintain the solution on the border line of acidity and alkalinity, and addition 40 of a small quantity of alkali being made should such not be the case.

The solution thus produced contains sodium azide, some unchanged reagents and the by-products of the reaction.

The sodium sulphate and sodium azide contained in the solution may be separated by crystallisation but preferably the solution is added gradually to 45 an acidified solution of silver nitrate produced by adding 1.0 cc. of 70% of nitric acid to 1000 ccs. of a normal solution of silver nitrate which is violently

agitated during such adding, the temperature of the reaction mass being prevented from rising much higher than 30—40° C. With adequate cooling and agitation the yield of silver azide may be obtained equivalent to 85% of the calculated possible amount. Instead of adding the diazotized solution to the acidified silver nitrate solution, the latter may be added to the former.

When starting from hydrazine nitrate which is also an acid salt, a solution of ammonia should be added in order to form the neutral hydrazine ammonium salt or, if desired, the neutral hydrazine sodium salt may be produced, the remaining steps of the process being then carried out as above described.

- 10 In producing lead azide, a solution of a hydrazine salt of an acid which will not result in the precipitation of an insoluble lead salt of the acid in question, for instance hydrazine nitrate, is employed, and the reaction mass in this case should be only just acid, as lead azide is particularly easily acted on by acids, while in producing silver azide the acidity of the reaction mass may be rather greater in order to avoid the precipitation of silver sulphate, the reaction being carried out under the conditions in regard to temperature and agitation indicated in the foregoing example.

The azides produced in accordance with the invention are usually amorphous, and in this state they are safer to handle than when crystalline.

- 20 Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. The process of producing azides from salts of hydrazine in which salts of hydrazine are diazotised by means of a suitable nitrite under such conditions that the reaction mass is at no time so distinctly acid as to redden litmus paper, but appears on the border line of acidity and alkalinity.

2. The process as claimed in Claim 1 in which the conditions of acidity under which the reaction is carried out according to the invention are preferably comparable with the conditions of acidity as indicated by litmus paper, of for instance, a solution of boric acid.

- 30 3. The process of producing azides from salts of hydrazine, substantially as hereinbefore described.

Dated this 10th day of December, 1918.

MARKS & CLERK.