

PATENT SPECIFICATION



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COMPLETE SPECIFICATION.

Process for the Manufacture of Salts of 1.3-dinitro-4.5-dinitrosobenzene.

I, Dr. HANS RATHSBURG, a citizen of the German Republic, of 25, Moststrasse, Fürth, Bavaria, Germany, 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:—

The subject matter of the present invention has reference to a simplified process for producing the salts of 1.3-dinitro-4.5-dinitrosobenzene which are soluble with difficulty, that is, the salts which are slightly soluble.

Hitherto in literature as far as I am aware mention has only been made of two processes for the production of salt forming, non-symmetrical 1.3-dinitro-4.5-dinitrosobenzene, namely (1) "Nitrierung von 4.5-dinitrosobenzol (Drost, Annalen der Chemie 307, 55)" and (2) "Abspaltung von 1 Molekul Stickstoff aus Pikrylazid oder Trinitrobenzoldiazoimid, gelöst in Kohlenwasserstoffen wie Benzol oder *p*-Xylol" (Separation of 1 molecule of nitrogen from picrylazide or trinitrobenzoldiazoimide, dissolved in hydro-carbons such as benzol or *p*-xylol), E. Schrader, "Berichte Deutschen, Chem., Ges. 50,777 (1917)".

For technically carrying out the process of manufacture according to the above particulars the 4.5-dinitrosobenzene as a fundamental raw material can only be obtained with difficulty relatively speaking and with an unfavourable yield. According to the latter literary reference the 1.3-dinitro-4.5-dinitrosobenzene is obtained in crystalline form in combination with hydro-carbons, which it does not give up until it has been exposed to the atmosphere for some time. The process for the production of the picrylazide, from which the 1.3-dini-

tro-4.5-dinitrosobenzene is to be obtained, by the interaction of picrychloride dissolved in alcohol with sodiumazide at boiling heat, is tedious owing to the necessity of using a reflux condenser, and furthermore the product according to "Schrader" (loc. cit.) contains a lot of sodium chloride from which it can first be freed by crystallizing it out in alcohol.

The production of salts of 1.3-dinitro-4.5-dinitrosobenzene has as far as I am aware been carried out in the following manner; it was stirred up together with suitable carbonates, for example carbonate of potassium or lead and, if necessary, allowed to stand at a moderate heat. This method, however, has the drawback that the final product either contains an excess of the corresponding carbonate or else of 1.3-dinitro-4.5-dinitrosobenzene whereby the technical blasting properties of the desired salt are greatly detracted from, whilst at the same time it does not turn out in a form suitable for loading.

Now it has been found that the technical production of difficultly soluble salts of 1.3-dinitro-4.5-dinitrosobenzene, such as the potassium or lead salt, can be materially simplified and the products can be obtained in a finely grained and regular crystal form suitable for loading by treating picrylchloride with sodium azide in aqueous suspension and at a suitable temperature, and transforming the 1.3-dinitro-4.5-dinitrosobenzene so obtained by reaction with an alkali (such as an oxide, hydroxide or carbonate) and then by reaction with a suitable salt (such as an acetate, nitrate, sulphate, or chloride of the metal corresponding to the salt desired) to produce the desired salt. It is preferred to heat the suspension of picrylchloride in an aqueous solution of technical sodium azide primarily at a temperature ranging between 80—90° C.

[Price 1/-]

and subsequently at a temperature between 90°—100° C. .

The following is given by way of example;

- 5 25 kilogrammes of picrylchloride are added to a filtered solution of 7 kilogrammes of technical sodium azide in 300 to 400 litres of water and the mixture is heated with steam for one hour at 80° to 10 90° C. whilst being well stirred and for a further 3 hours at 90 to 100° C. The melted picrylchloride of melting point 83° changes into picrylazide of a melting point of 93° which again at this 15 temperature separates off 1 molecule of nitrogen and yields 1.3-dinitro-4.5-dinitrosobenzene in well formed crystals. After removal of the sodium chloride containing mother lye, this yield, is at 20 once converted with soda into a sodium salt, and the sodium salt obtained, after removing the sodium salt mother lye, is converted direct in the moist state (with suitable salt solutions, such as potassium acetate for example), into the desired 25 salt.

- Now it could not be foreseen that picrylchloride could without any difficulty be transformed in aqueous suspension in such a simple manner into 1.3-dinitro-4.5-dinitrosobenzene, which also 30 need not be isolated; that is to say much more cheaply and conveniently than heretofore in an alcoholic solution, passing over picrylazide as a non-isolated 35 bye-product.

- If it be desired to obtain a coarse-grained product then the solid sodium salt is decomposed with the corresponding 40 salt solutions. In this way the salts required attain the approximate crystal size of the solid sodium salts employed.

- The direct conversion of picrylchloride into 1.3-dinitro-4.5-dinitrosobenzene, 45 without the isolation of the picrylazide as intermediate product as heretofore, and also the winning of coarse-grained potassium or lead salt through the sodium salt, is new and can be carried out in a

technically simple way. By doing away 50 with expensive inflammable organic solvent media and reflux condensers formerly requisite for the isolation of picrylazide, the process of manufacture is materially cheapened and thus a 55 technical advancement in the art is realized.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is 60 to be performed, I declare that what I claim is:—

1. A process for the manufacture of 65 difficultly soluble salts of 1.3-dinitro-4.5-dinitrosobenzene such as the potassium or lead salt, consisting in treating picrylchloride with sodium azide in aqueous suspension and at a suitable temperature, and in transforming the 1.3-dinitro-4.5-dinitrosobenzene so obtained by reaction 70 with an alkali and then by reaction with a suitable salt (such as an acetate, nitrate, sulphate, or chloride of the metal corresponding to the salt desired) to produce the desired salt, substantially 75 as described.

2. In the process specified in Claim 1, the step of heating a suspension of picrylchloride in an aqueous solution of 80 technical sodium azide primarily at 80°—90° C. and subsequently 90°—100° C.

3. The improved process for the manufacture of difficultly soluble salts of 1.3-dinitro-4.5-dinitrosobenzene substantially 85 as hereinbefore described.

4. The difficultly soluble salts of 1.3-dinitro-4.5-dinitrosobenzene when produced by the special process herein 90 claimed.

Dated this 20th day of October, 1921.

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