AD-B135 890 PREPARATION OF DINITROBENZOXADIAZOLE OXIDE AND ITS SALTS(U) ARMY FOREIGN SCIENCE AND TECHNOLOGY CENTER CHARLOTTESVILLE VA B CHEN ET AL. 07 JUN 89 1/1 USGO & CONT

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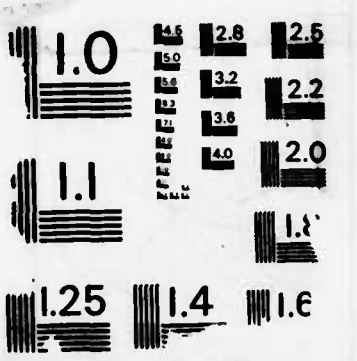
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DATE: 7 Jun 1989

AD-B135 890

ENGLISH TITLE: PRC patent application announcement CN 85 105850: preparation of dinitrobenzoxadiazole oxide and its salts.

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LANGUAGE: Chinese.

COUNTRY: People's Republic of China.

COUNTRY OF ORIGIN: People's Republic of China.

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REQUESTER: *AIFRTC (Sheinfeld).*

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TRANSLATION ACC #

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Agency: FSTC PO No. P333 FSTC-HT-0385-88

Title: Dinitrobenzoxadiazole Oxide

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Patent Office, People's Republic of China

Patent Application Disclosure

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Application Number: 85 1 05850

Application Date: 8/3/85

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Patent Agency: Patent Agency, Beijing Institute of Engineering

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Invention Name: Preparation of Dinitrobenzoxadiazole Oxide and Its Salts

Abstract:

This invention is a method to prepare military chemicals. Dinitrobenzoxadiazole oxide is a high explosive. It is also an intermediate explosive. Its sodium, potassium and barium salts can be used as detonators. Previous methods cannot be used in production on industrial scale.

This invention is new method which uses tetryl as the raw material to make 4,6-dinitrobenzoxadiazole oxide. It also introduces a way to make 4,6-dinitrobenzoxadiazole oxide salts. 4,6-dinitrobenzoxadiazole oxide is prepared by the above method as the intermediate to make its salts. This is a simple, efficient and easily manufacturable method.

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1) The special feature of the method to prepare 4,6-dinitro-benzoxadiazole oxide is to produce 2,4,6-trinitro-triazobenzene by reacting tetryl with sodium azide. 2,4,6-trinitro-triazobenzene is thermally denitrified in acetic acid to form 4,6 dinitrobenzoxadiazole oxide.

2) Based on the method described in Claim 1 of the application, the special feature of the method is the azide reaction. Its solvent is ethanol or another common polar solvent. The temperature of reaction is 15 - 50 °C. The amount of sodium azide required is more than theoretical. The thermal denitrification temperature is approximately 100 °C.

3) The special feature of the method to prepare the intermediate 4,6 dinitrobenzoxadiazole oxide by using tetryl as the raw material and then make sodium, potassium or barium salt of 4,6 dinitrobenzoxadiazole oxide in another reaction.

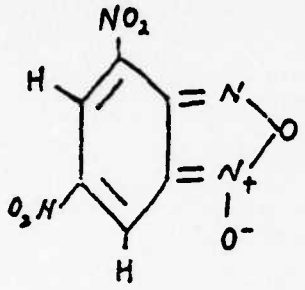
Preparation of Dinitrobenzoxadiazole Oxide and Its Salts

This invention is a method to prepare military chemicals.

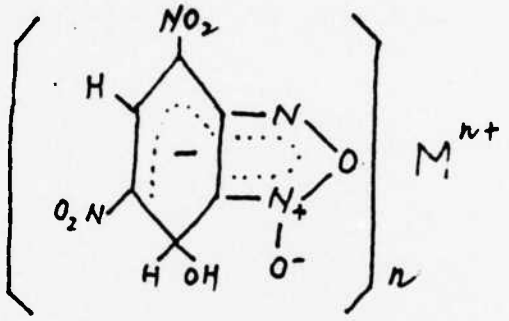
4,6 dinitrobenzoxadiazole oxide is a high explosive and an important intermediate of explosives. Its molecular structure is shown in (I). Its sodium, potassium and barium salts can detonate. Their molecular

→ Keywords: Translations, China, Chinese Language. (AW)

structures are shown in (II). The potassium salt detonates easily and the barium is resistant to high temperature.



(I)



(II) $M^{n+} = Na^{+}, K^{+}, Ba^{2+}$

There are several methods to prepare 4,6 dinitrobenzoxadiazole oxide. Recently, Australian Spear, Robert J., Norris, William P. and Read, Roger W., published a method to prepare 4,6 dinitrobenzoxadiazole oxide in Tech Note - Mater. Res. Lab (Aust) 1983 MRL-TN-470. It starts with picryl chloride and then go through a reaction with azide and followed by denitrification. However, picryl chloride is difficult to make. Another method involves the nitration of benzoxadiazole oxide. The paper published by Chinese scientists Lu Mingjiu [7627 7686 0036] and Liu Hongyu [0491 7703 0060] in the [CHINESE JOURNAL OF ORDINANCE], Volume 3, Chemicals and Explosives, 12 (1982) also employs this approach. Nevertheless, the reaction takes a long time and the yield is

low. Therefore, these two methods cannot be effectively put in production.

The objective of this invention is to use a easily obtainable raw material, tetryl, to prepare 4,6 dinitrobenzoxadiazole oxide and uses it as the intermediate to make its sodium, potassium and barium salts.

The method to prepare 4,6 dinitrobenzoxadiazole oxide is to allow tetryl and sodium azide to react in ethanol or a common polar solvent. The amount of sodium azide required is higher than theoretical. Stir at 15 - 50 °C and then filter it after diluting with water to obtain a light yellowish precipitate, i.e. 2, 4, 6 trinitrotriazobenzene. Put it in acetic acid for thermal denitrification (at approximately 100 °C). Wait until it cools down and then dilute it with water. Wash the yellow solid precipitate with water to get 4,6 dinitrobenzoxadiazole oxide. The yield based on the utilization of tetryl is over 80 percent. It can be further purified in chloroform or acetic acid.

The method to prepare salts of 4,6 dinitrobenzoxadiazole oxide is to make the intermediate, 4,6 dinitrobenzoxadiazole oxide, from tetryl using the above method. It is then reacts with sodium bicarbonate in water at approximately 50 °C to make its sodium salt. The sodium salt solution may react with potassium nitrate or potassium sulfate solution to obtain the potassium salt. The sodium salt solution may react with barium chloride or barium nitrate solution to yield it barium salt.

The raw material used in this invention, tetryl, is a low cost industrial commodity which is easy to get. The process is simple, the condition requirements modest, the reaction time short and the yield high. Similarly, tetryl can be used to prepare the sodium, potassium and barium salts of 4,6 dinitrobenzoxadiazole oxide. It is easily manufacturable at low cost.

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