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TECHNICAL REPORT

WHITE OAK LABORATORY

SYNTHESIS OF MERCURIC 5-NITROTETRAZOLE

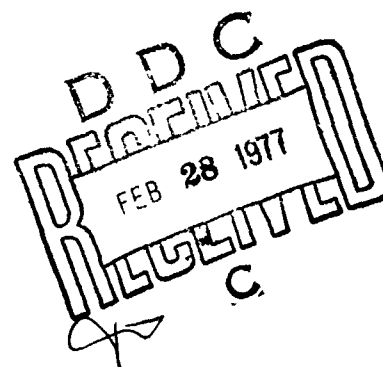
BY

William H. Gilligan
Mortimer J. Kamlet

9 DECEMBER 1976

NAVAL SURFACE WEAPONS CENTER
WHITE OAK LABORATORY
SILVER SPRING, MARYLAND 20910

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1 REPORT NUMBER NSWC/WOL/TR-76-146	2 GOVT ACCESSION NO.	3 RECIPIENT'S CATALOG NUMBER
4 TITLE (and Subtitle) SYNTHESIS OF MERCURIC 5-NITROTETRAZOLE.		5 TYPE OF REPORT & PERIOD COVERED
7 AUTHOR(s) William H. Gilligan and Mortimer J. Kamlet		6 PERFORMING ORG REPORT NUMBER
9 PERFORMING ORGANIZATION NAME AND ADDRESS Naval Surface Weapons Center White Oak Laboratory White Oak, Silver Spring, Maryland 20910		8 CONTRACT OR GRANT NUMBER(s)
11 CONTROLLING OFFICE NAME AND ADDRESS		10 PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 0; 0; OPN; 0;
14 MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12 REPORT DATE 9 Dec 1976
		13 NUMBER OF PAGES 11
		15 SECURITY CLASS (of this report) UNCLASSIFIED
		15a DECLASSIFICATION/DOWNGRADING SCHEDULE
16 DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited		
17 DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18 SUPPLEMENTARY NOTES		
19 KEY WORDS (Continue on reverse side if necessary and identify by block number) Initiators Lead Azide Primary Explosives Detonators Explosives Synthesis		
20 ABSTRACT (Continue on reverse side if necessary and identify by block number) Initial tests indicate Mercury 5-Nitrotetrazole is a promising replacement for lead azide in detonators. During the course of preparation of this material for testing, several improvements in the original synthesis were made. A reliable good yield procedure for mercury 5-nitrotetrazole is now available.		

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NSWC/WOL/TR 76-146

9 December 1976

SYNTHESIS OF MERCURIC 5-NITROTETRAZOLE

An improved synthesis of mercuric 5-nitrotetrazole is described. Mercuric 5-nitrotetrazole is a very promising candidate for the replacement of lead azide in detonators.

This task SF 33-354-316/8460 was funded by the Naval Sea Systems Command, SEA-0332.

Julius W. Enig
JULIUS W. ENIG
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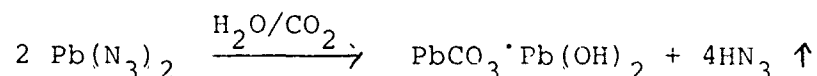
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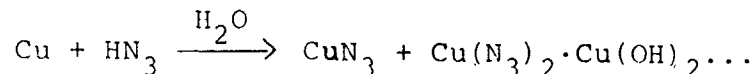
SYNTHESIS OF MERCURIC 5-NITROTETRAZOLE

I. INTRODUCTION

Lead azide is one of the most common primary explosives used in detonators. It has been in widespread use for the past 50 years not only in the United States but throughout the world. Despite its almost universal acceptance, its service life is often shortened because of ready hydrolysis in the presence of moisture and carbon dioxide. One of the by-products of hydrolysis is hydrogen azide which in the presence of copper or copper alloys can form various



copper azides some of which are even more sensitive, particularly to electrostatic discharge than lead azide itself.



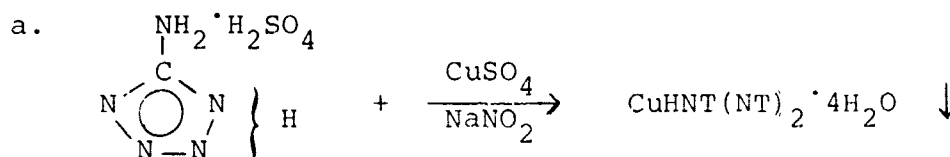
The presence of copper azides is believed to have caused a number of fatal accidents during the handling and storage of munitions, and is also thought to be a factor in premature firings.

For these reasons a program was begun at the Naval Surface Weapons Center, White Oak Laboratory (NSWC/WOL) to find a replacement for lead azide that would be stable to hydrolysis, compatible with detonator construction materials and would at least equal lead azide in performance. Before and during this program, close cooperation was maintained with British personnel at the Explosive Research and Development Establishment (ERDE) at Waltham Abbey, Essex, England, who were conducting a similar investigation.

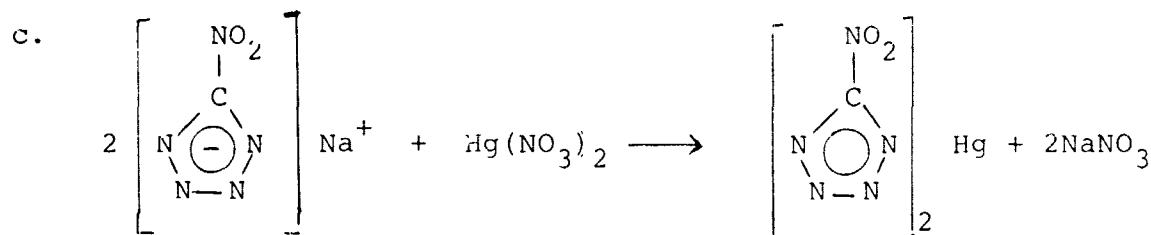
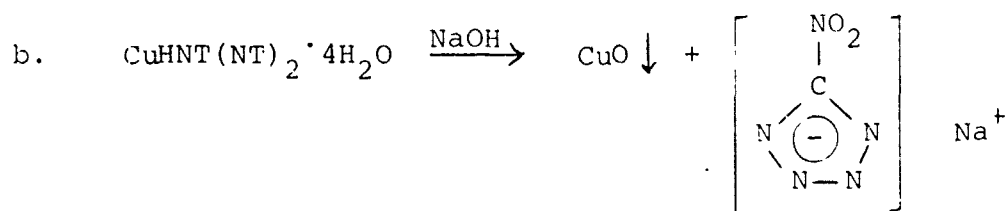
II. VON HERZ PROCEDURE FOR MERCURIC 5-NITROTETRAZOLE

Preliminary screening performed both here and by the British at Waltham Abbey indicated that mercuric 5-nitrotetrazole was a promising lead azide replacement. Our attention then was directed to an examination of the synthesis and properties of this compound.

Mercuric 5-nitrotetrazole was first reported in 1932 by Von Herz¹. He prepared this material via the following three steps:



where NT = 5-nitrotetrazole anion.



He proposed that the heavy metal salts and methyl esters of 5-nitrotetrazole would be useful initiating explosives. Conversely, in 1952 it was reported that mercuric 5-nitrotetrazole was rejected from consideration as a primary explosive because of excessive water solubility², though no data as a basis for this decision were given³.

¹E. Von Herz, U.S. Pat. 2,066,954 (1932).

²F. Taylor, NAVORD 2468, Naval Ordnance Laboratory, White Oak, MD, 3 Jun 1952.

³The solubility of pure mercuric 5-nitrotetrazole in water was determined by D. Glover of this Center, formerly the Naval Ordnance Laboratory, to be 0.09% at 30°C.

During initial experimentation with the Von Herz procedure several problems were encountered. During the diazotization there was a continuous series of minor detonations, which while not harmful in themselves⁴, were psychologically disturbing and did on occasion break glassware. There was the possibility that the potentially dangerous (in the dry state) acid copper nitrotetrazole salt would be spilled over adjacent surfaces.

Upon completion of the diazotization the acid copper salt was present as a voluminous gel-like precipitate which required long periods (six hours or longer) to separate by filtration and to wash free of impurities. This would seriously hamper scale-up operations where large quantities would have to be processed. In addition, although Von Herz claimed yields of around 90% for this step, we were only able to obtain yields of 60 to 65%.

The purity of the sodium 5-nitrotetrazole used for the reaction with mercuric nitrate had a marked effect on both the yield and the properties of the resultant mercuric 5-nitrotetrazole. When raw solutions of sodium 5-nitrotetrazole obtained by treating the acid copper salt with sodium hydroxide and filtering off the cupric oxide were used the mercury salt dead pressed at moderate pressures (20 kpsi) and so was unsuitable for detonator use.

It was necessary then to further purify the sodium 5-nitrotetrazole. This was done by recrystallizing the sodium salt twice from water. This proved to be a time consuming procedure which required the evaporation of large volumes of water. Also, because of the solubility of the sodium salt, yield losses were high. The mercuric 5-nitrotetrazole prepared by the reaction of twice recrystallized sodium 5-nitrotetrazole with mercuric nitrate did not dead press up to 80 kpsi, and had good detonant properties, but even here there were batch to batch variations in crystal form and size⁵.

III. ELIMINATION OF MINOR DETONATIONS

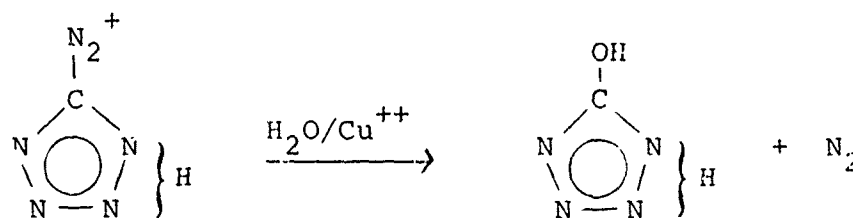
5-Diazotetrazole will spontaneously detonate in aqueous solution when the concentration exceeds 1%⁶. It was reasoned then

⁴Private communication from Dr. J. Jenkins, ERDE, Waltham Abbey, Essex, England. An aqueous slurry of the copper acid salt of 5-nitrotetrazole could not be exploded with a detonator cap.

⁵A more consistent product could be obtained by recrystallizing the sodium salt two additional times from acetone but this, of course, further increased both the yield losses and the time required for purification.

⁶F. R. Benson, "Heterocyclic Compounds," 8, p. 1 (1967).

that the detonations occurring during the diazotization were caused by nitrogen oxide fumes arising from the reaction solution and reacting with droplets of 5-aminotetrazole solution on various surfaces of the apparatus. Since copper salts catalyze the reaction of 5-diazotetrazole with nucleophiles, it was felt that the addition of small amounts of copper sulfate to the 5-aminotetrazole solution would eliminate any build-up of the diazotetrazole by catalyzing its conversion to 5-hydroxytetrazole.



This proved to be the case since the addition of small amounts ($\sim 2\text{g}$) of copper sulphate to the 5-aminotetrazole solution completely eliminated the detonations previously experienced.

IV. SEPARATION OF THE COPPER ACID SALT OF 5-NITROTETRAZOLE

Table 1 summarizes the results of a number of experiments in which the sodium nitrite concentration was gradually increased by the addition of excess sodium nitrite and by decreasing the reaction volume. There was a concomitant increase in the yield of the acid copper salt from 62 to 85%. In addition, the physical nature of the precipitate was markedly altered from gel-like to a more compact easily filtered material. The filtration and washing which were previously a major headache could now be quickly accomplished in about ten minutes. The substitution of nitric acid for sulfuric acid under Vor Herz conditions did appear to be somewhat advantageous (expt 3), however at higher nitrite concentrations there was no discernible difference.

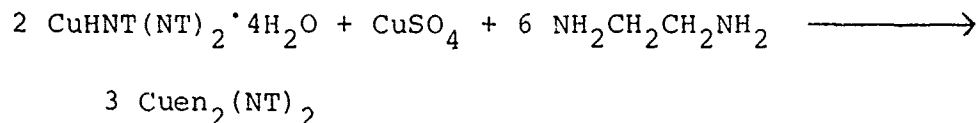
Because of the increased yield and the importance of ease of filtration the more favorable modifications were incorporated into a revised procedure for the preparation of the acid copper salt (see Experimental).

V. PURIFICATION OF 5-NITROTETRAZOLE VIA BIS(ETHYLENEDIAMINE) COPPER (II) BIS(5-NITROTETRAZOLE)

As a result of investigations carried out at ERDE, Waltham Abbey, Essex, England⁷, it was found that the copper acid salt

⁷Private communication from Drs. J. Jenkins and R. McGuchan. English patent pending.

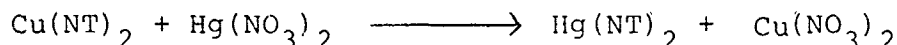
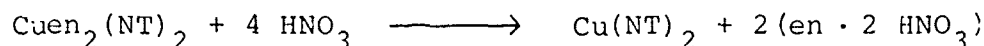
of 5-nitrotetrazole could be reacted with ethylenediamine to give bis(ethylenediamine)copper(II)bis(5-nitrotetrazole.) This



where en = $\text{NH}_2\text{CH}_2\text{CH}_2\text{NH}_2$;

NT = anion of 5-nitrotetrazole

coordination compound could be readily purified by recrystallization from water, was relatively non-sensitive⁸ and so could be handled safely in the dry state. Mercuric 5-nitrotetrazole could then be prepared by dissolving the $\text{Cuen}_2(\text{NT})_2$ in hot water adding nitric acid to tie up the ethylenediamine followed by the addition of mercuric nitrate solution.



Mercuric 5-nitrotetrazole prepared in this manner was in the form of discrete well shaped crystals which showed little variance from run to run. The product showed excellent detonant properties as an initiator explosive. This procedure eliminated the necessity for the time-consuming purification of sodium 5-nitrotetrazole and was easily adaptable to scale-up operations.

With the adoption of the bis(ethylenediamine) Cu(II) bis(5-nitrotetrazole) purification method, all of the problems associated with the original Von Herz procedure were eliminated. A reliable good yield procedure for mercuric 5-nitrotetrazole is now available.

During the course of this work, approximately 5 pounds of sodium 5-nitrotetrazole and about 2.5 pounds of mercuric 5-nitrotetrazole were prepared in the laboratory for investigative purposes without incident.

VI. EXPERIMENTAL

MERCURIC 5-NITROTETRAZOLE FROM SODIUM 5-NITROTETRAZOLE

I. Sodium 5-Nitrotetrazole

⁸ $\text{Cuen}_2(\text{NT})_2$ gave evidence of burning at 50 cm (2.5 kg wt) but did not detonate up to 300 cm on the NSWC impact machine.

TABLE 1
EFFECT OF SODIUM NITRITE CONCENTRATION ON ACID COPPER SALT PREPARATION

A Solution*	B Solution	Time for Filtration and Washing	% Yield Cu(NT) ₂ HNT·4H ₂ O
1. 51.5 g 5-AT·H ₂ O + 18 cc conc H ₂ SO ₄ in 1500 ml H ₂ O	77 g NaNO ₂ + 75 g CuSO ₄ ·5H ₂ O in 750 ml H ₂ O	6 hours	62%
2. 51.5 g 5-AT·H ₂ O + 18 cc conc H ₂ SO ₄ + 2 g CuSO ₄ ·5H ₂ O in 1500 ml H ₂ O	77 g NaNO ₂ + 55 g CuSO ₄ ·5H ₂ O in 750 ml H ₂ O	6 hours	65%
3. 51.5 g 5-AT·H ₂ O + 43 ml 70% HNO ₃ + 2 g CuSO ₄ ·5H ₂ O in 1500 ml H ₂ O	77 g NaNO ₂ + 55 g CuSO ₄ ·5H ₂ O in 750 ml H ₂ O	2 hours, 25 minutes	63%
4. 51.5 g 5-AT·H ₂ O + 53 ml 70% HNO ₃ + 2 g CuSO ₄ ·5H ₂ O in 1000 ml H ₂ O	87 g NaNO ₂ + 55 g CuSO ₄ ·5H ₂ O in 500 ml H ₂ O	45 minutes	74%
5. 51.5 g 5-AT·H ₂ O + 64 ml 70% HNO ₃ in 1600 ml H ₂ O + 2 g CuSO ₄ ·5H ₂ O	104 g NaNO ₂ + 55 g CuSO ₄ ·5H ₂ O in 500 ml H ₂ O	15 minutes	82%
6. 51.5 g 5-AT·H ₂ O + 2 g CuSO ₄ ·5H ₂ O + 64 ml 70% HNO ₃ in 600 ml H ₂ O	104 g NaNO ₂ + 55 g CuSO ₄ ·5H ₂ O in 300 ml H ₂ O	12 minutes	85%
7. 51.5 g 5-AT·H ₂ O + 28 ml conc H ₂ SO ₄ + 2 g CuSO ₄ in 700 ml H ₂ O	104 g NaNO ₂ + 55 g CuSO ₄ ·5H ₂ O in 300 ml H ₂ O	10 minutes	--

* Solution A dropped into Solution B at 15-18° over a period of 90 minutes.
5-AT = 5-aminotetrazole.

Reagents:

- A. 104g (1.5 mol) NaNO_2 and 55g (0.22 mol) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 300 ml water.
- B. 51.5g (0.5 mol) 5-aminotetrazole monohydrate, 2g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 64 ml 70% nitric acid in 700 ml H_2O .
- C. 70 ml 70% HNO_3 in 30 ml H_2O .
- D. 50% NaOH solution (ca 30 ml).

Caution:

The copper acid salt of 5-nitrotetrazole, $\text{CuHNT}(\text{NT})_2$, can be handled safely in the wet state; however in the dry state, it is very sensitive to shock and electrostatic discharge. Air dried sodium 5-nitrotetrazole containing two or more mole-equivalents of water of crystallization is relatively insensitive to shock; it cannot be detonated with a hammer blow. However when completely dry, it is also a sensitive explosive. Both compounds will detonate violently if dropped on a hot plate. All precautions consistent with the handling of potentially dangerous explosive materials should be observed throughout this operation.

Procedure:

Place solution A in a 2L beaker and cool to 5°C . Add solution B to A dropwise with efficient stirring over a period of about 90 minutes holding the temperature at 15° to 18°C (Note 1). Stir for 15 minutes, add solution C dropwise and then stir for an additional 30 minutes. Filter with suction and wash the copper acid salt with 250 ml 1.8 N HNO_3 and three times with 250 ml of water. Do not allow the cake to dry during the filtration and washing. Transfer the wet cake to a 1500 ml beaker and adjust the volume to about 600 ml with water.

Adjust the pH of the slurry to ca 9 with 50% sodium hydroxide solution to precipitate copper hydroxide and then heat the efficiently stirred slurry to 70°C and digest for 30 minutes (Note 2). Allow the precipitate to partially settle and filter with suction through a packed layer of "celite" (Note 3). Wash the precipitate twice with 100 ml of water.

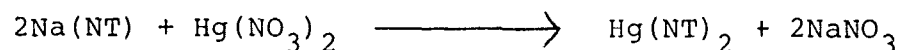
Adjust the pH of the combined filtrate and washes to 4, with conc. HNO_3 . Reduce the volume to ca 350 ml using a rotovac at a bath temperature of 60°C . Cool to 2°C and filter the sodium 5-nitrotetrazole (Note 4). Reduce the filtrate to 200 ml and take a second crop...etc. Combine the crops, redissolve in water and recrystallize a second time. Air dry the product.

Dissolve the sodium 5-nitrotetrazole in acetone on a steam bath and filter to remove inorganic salts. Cool the filtrate in an ice bath and remove the sodium 5-nitrotetrazole by filtration. Recrystallize a second time from acetone and air dry (Note 5). The yield is about 45-55% of theory based on 5-aminotetrazole.

Notes:

1. During the diazotization of 5-aminotetrazole nitrogen oxide fumes are given off from the reaction solution. This step should be carried out in an efficient hood.
2. The blue hydrated cupric hydroxide is converted to the brownish-black cupric oxide at 70°C.
3. Without "celite" the filtration and washing requires several hours since the finely divided cupric oxide clogs the filter.
4. Sodium 5-nitrotetrazole crystallizes from water as a voluminous hydrated mass. After air drying, the salt contains two to five moles of water of crystallization depending on ambient humidity.
5. Sodium 5-nitrotetrazole crystallizes from acetone as a dihydrate. This appears to be stable at ambient conditions.

II. Mercuric 5-Nitrotetrazole from Sodium 5-Nitrotetrazole



where NT = anion of 5-nitrotetrazole

Reagents:

- A. $\text{Hg}(\text{NO}_3)_2$ solution. Dissolve 54.0 g of red mercuric oxide in 200 ml of 35% HNO_3 . Filter and dilute the filtrate to 250 ml with 35% HNO_3 .
- B. 70% HNO_3 .
- C. Sodium 5-nitrotetrazole dihydrate.

Caution:

Sodium 5-nitrotetrazole containing two or more mole-equivalents of water of hydration is relatively insensitive to shock; it cannot be detonated with a hammer blow. However when completely dry, it is a sensitive explosive.

Mercuric 5-nitrotetrazole is a powerful primary explosive. It detonates at 7 cm (2.5 kg wt) on the NSWC impact machine. It is also sensitive to friction. All operations should be carried out behind a safety screen with minimum exposure, by experienced personnel.

Procedure:

Dissolve 16.7 g of sodium 5-nitrotetrazole in 188 ml water. Add 12 ml 70% HNO_3 and place a magnetic stirring bar in the solution. In a separate container, add 60 ml of the $\text{Hg}(\text{NO}_3)_2$ solution to 140 ml of water. Heat both the tetrazole solution and the $\text{Hg}(\text{NO}_3)_2$ solution to 75°C in a water bath.

Remove the solutions, quickly add the $\text{Hg}(\text{NO}_3)_2$ solution to the tetrazole solution and begin stirring at slowest speed possible. Allow to cool gradually to ca 30°C with continuous stirring while the mercuric salt crystallizes. Stop the stirring, allow the mercury salt to settle and then decant the supernatant liquid. Transfer the solids to a "Nalgene" beaker by means of a water wash bottle. Add 200 ml of water, swirl, and again decant.

Transfer about 1/3 of the solids to a small "Nalgene" Buchner funnel (#2 Whatman paper) by means of a water wash bottle and wash consecutively with 50 ml water, twice with 50 ml of methanol, and finally with 50 ml of methylene chloride. Air dry on the funnel. Carefully transfer the dried salt by gently pouring the contents into a weighed wide-mouth conductive plastic container. After weighing place mercuric salt into a blast-proof container. Repeat filtration and washing for remainder of product. Yield is 75 to 80% of theory based on sodium 5-nitrotetrazole.

MERCURIC 5-NITROTETRAZOLE FROM BIS(ETHYLENEDIAMINE)COPPER(II) BIS(5-NITROTETRAZOLE)

I. Bis(Ethylenediamine)Copper(II)Bis(5-Nitrotetrazole)

Reagents:

- A. 104 g (1.5 mol) NaNO_2 and 55 g (0.22 mol) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 300 ml H_2O .
- B. 51.5 g (0.5 mol) 5-aminotetrazole monohydrate, 2 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 64 ml 70% nitric acid in 700 ml H_2O .
- C. 70 ml 70% HNO_3 in 30 ml H_2O .
- D. Ethylenediamine.

Caution:

The copper acid salt of 5-nitrotetrazole [$\text{CuHNT}(\text{NT})_2$] can be handled safely in the wet state; however when dried, it is very sensitive to shock and electrostatic discharge. Bis(ethylene-diamine)copper(II)bis(5-nitrotetrazole) has been routinely handled in the dry state without incident. On the NSWC impact machine, it burns at 50 cm (2.5 kg wt) but does not detonate. When heated in a capillary, in an aluminum block, it begins melting at 210°C and completely decomposes with a burst of gas at 225°C leaving a brownish-black residue. Under these conditions, it does not detonate. Nevertheless, all precautions consistent with the handling of potentially dangerous materials should be observed throughout these operations.

Procedure:

Place solution A in a 2L beaker and cool to 5°C . Add solution B to A dropwise, with efficient stirring over a period of about 90 minutes holding the temperature at $15 - 18^\circ\text{C}$ (Note 1). Stir for 15 minutes, add solution C dropwise and then stir for an additional 30 minutes. Filter the copper acid salt with suction and wash with 250 ml 1.8N HNO_3 and three times with 250 ml water. Do not allow the cake to dry during the filtration and washing. Transfer the wet cake to a 1500 ml Erlenmeyer and make up the volume to about 600 ml with water.

Heat the slurry (water bath) to 75°C with efficient stirring and add a solution of 21 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 45 ml of ethylenediamine in 80 ml of H_2O . Stir until the slurry has completely dissolved and then cool quickly with swirling in an ice bath. After cooling allow to remain in the ice bath for 45 minutes to complete the crystallization. Filter, wash twice with 200 ml cold water and air dry. The yield of $\text{Cuen}_2(\text{NT})_2$ is about 77 g; 75% based on 5-aminotetrazole.

Note 1. During the diazotization of 5-aminotetrazole, nitrogen oxide fumes are given off from the reaction solution. This step should be carried out in an efficient hood.

II. Mercuric 5-Nitrotetrazole from Bis(Ethylenediamine)Copper(II) Bis(5-Nitrotetrazole)

Reagents:

- A. 100 g $\text{Cuen}_2(\text{NT})_2$ in 1100 ml H_2O at 75°C .
- B. 65 ml 70% HNO_3 in 160 ml H_2O .
- C. 4 ml 70% HNO_3 in 255 ml of 1.0M $\text{Hg}(\text{NO}_3)_2$.

Caution:

Mercuric 5-nitrotetrazole is a powerful primary explosive. It detonates at 7 cm (2.5 kg wt) on the NSWC impact machine and is also sensitive to friction. All operations should be carried out behind a safety screen with minimum exposure, by experienced personnel.

Procedure:

Add solution B to A, dropwise, with stirring at 75° to 80°C over a period of 15 minutes. Then add solution C in the same manner over a period of 55 minutes. Stir for 15 minutes then allow to cool to 55°C. Decant the supernate from the solid product and wash by decantation 5 times with 500 ml H₂O. Transfer the product to a plastic bottle with a water wash² bottle and store under water. Yield is about 92 g (89%).

Analysis of Mercuric 5-Nitrotetrazole

The 5-nitrotetrazole ring has a maximum in the ultraviolet at 257 mμ, which can be used for analysis⁹. The molecular extinction coefficient is 5.4×10^3 .

About 60 mg of mercuric 5-nitrotetrazole is dissolved in 20 ml of 20% ammonium acetate solution and then diluted to 250 ml with water. A second dilution of about one to ten is then made with 2% ammonium acetate solution and the absorption is measured at 257 mμ against a reagent blank.

Sodium 5-nitrotetrazole dihydrate which was recrystallized twice from water and three times from acetone and then air dried was used to prepare a standard curve.

⁹N. J. Blay, D. G. Davies, D. C. Mullinger and R. J. Rapley, ERDE, TR 163, Explosives Research and Development Establishment, Waltham Abbey, Essex, England, Jan 1974.

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