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Technical Report No. 172

The Salts of 5-Substituted Tetrazoles:
Part 3: Further Investigations into
the Preparation and Properties of
Silver 5-Nitrotetrazole and
Mercuric 5-Nitrotetrazole (U)

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J. M. Jenkins
J. R. White

February 1974

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EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

Technical Report No 172

February 1974

The Salts of 5-Substituted Tetrazoles: Part 3:
Further Investigations into the Preparation and
Properties of Silver 5-Nitrotetrazole and Mercuric
5-Nitrotetrazole (U)

by

J M Jenkins

J R White

SUMMARY

A form of silver 5-nitrotetrazole has been developed which can be filled into small devices. It has been tested, together with mercuric 5-nitrotetrazole, for detonant and stab-sensitive properties. Both compositions have been shown to be stable and will efficiently detonate RDX in large detonators but neither, in their current physical form, are technically suitable for current UK design of small detonators. The silver salt dead presses at loads too low for adequate consolidation; the mercuric salt detonates the RDX but not as efficiently as lead azide. Neither salt is stab-sensitive. Recommendations are made concerning the future synthetic and crystal modification programmes, methods of assessment, and potential application of these compounds.

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CONTENTS

	<u>Page No</u>
1 INTRODUCTION	5
2 OBJECT	5
3 EXPERIMENTAL	6
3.1 Preparation of Intermediates on the 600 ml Scale	6
3.2 The Preparation of the Intermediates on the 10-litre Scale	6
3.3 Preparation of Silver 5-Nitrotetrazole	8
3.4 Preparation of Mercuric 5-Nitrotetrazole	9
3.5 Destruction Procedure for 5-Nitrotetrazole Salts	9
4 PROPERTIES OF SILVER AND MERCURIC 5-NITROTETRAZOLES	10
4.1 Chemical Analysis	10
4.2 Sensitiveness	11
4.3 Thermal Stability/Compatibility	11
4.4 Stab-Sensitivity	12
4.5 Detonant Tests	12
5 CONCLUSIONS	13
6 RECOMMENDATIONS	13
7 REFERENCES	14
Figures 1 to 5	

1 INTRODUCTION

The primary explosives compositions that are now in service are by no means totally satisfactory for the current and future demands of long life and high thermal stability. Lead azide, the main detonant composition, although thermally stable, deteriorates on storage as it is readily hydrolysed by water vapour; an effect which is accelerated by carbon dioxide and gaseous emanations from plastics and rubbers. Silver azide, as RD1336, has recently entered service as a replacement for lead azide in some detonators and has proved to be a substantial improvement in that its rate of hydrolysis is very low. However, silver azide is not the complete solution as it suffers from incompatibility with certain materials especially sulphur containing compounds. Thus there is a need for a new stable detonant composition that will function under the same conditions as lead and silver azide.

The key composition in the current stab-sensitive compositions is the sensitizer, tetrazene. This composition although very efficient as a sensitizer is limited by its low thermal stability; onset of decomposition can begin as low as 60°C. Thus there is a need for either a new sensitizer or a new single component stab-sensitive compound.

The literature^{1,2} and our own laboratory preliminary study³ suggested that it would be worthwhile to carry out a more detailed study on silver 5-nitrotetrazole and mercuric 5-nitrotetrazole with a view to obtaining a stable detonant and/or possibly a new stab sensitive composition.

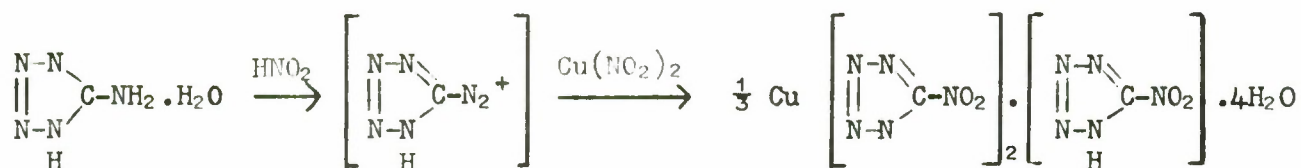
2 OBJECT

The objects of the work described were (a) to prepare the silver and mercuric salts of 5-nitrotetrazole as pure single phases in a form suitable for assessment in small detonators and stab igniters, (b) to assess their service applicability. This latter object would involve the collation of data on preliminary compatibility, sensitiveness, preparative feasibility in addition to functioning tests. In view of the priority of the work it was decided that the compatibility tests would start at the same time as the physical form modifications were begun.

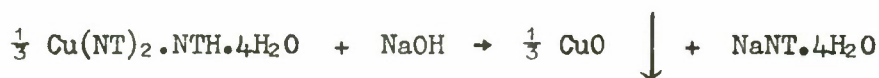
3 EXPERIMENTAL3.1 Preparation of Intermediates on the 600 ml Scale

The preparation is carried out in two stages viz

- (1) The conversion of 5-aminotetrazole monohydrate into copper 5-nitrotetrazole tetrahydrate.



- (2) The conversion of the acid copper salt to the sodium salt solution.
(5-nitrotetrazole anion = $\text{NT}^\ominus$.)



The preparation of the acid copper salt of 5-nitrotetrazole and the subsequent preparation of the sodium salt on this scale are described in our previous note.³ Further work was carried out on this scale to evaluate the effect of temperature on the reaction. It was shown that 9 - 10°C was the optimum temperature of reaction; below that, the reaction continues for a long while after the addition of the 5-aminotetrazole is completed, at about 16 - 18°C there are considerable nitrous fumes evolved. If the temperature is allowed to rise above 20°C during the addition of the 5-aminotetrazole then there are minor explosions in the reaction vessel.

3.2 The Preparation of the Intermediates on the 10-litre Scale

Plant The 10-litre stainless steel initiatory explosive plant was used for both stages 1 and 2. The stirrer speed was kept at 200 r/min during the reactions. The reaction vessel is cooled and heated through a jacket. It is NOT advisable to use steam heating in stage 1. In stage 2 steam heating can be used.

Solutions All solutions were made up with distilled water and were filtered before use.

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3.2.1 The Preparation of the Copper Salt

The stirred base solution consisting of 1.0 litre of sodium nitrite solution (154 g/l) and 0.5 litre of copper sulphate pentahydrate solution (192 g/l) was cooled to 6°C. To this base solution was added in 40 min a solution of 5-aminotetrazole. This solution was made up by dissolving 103 g of solid 5-aminotetrazole monohydrate in a solution of 3 litres water to which 35 ml of concentrated sulphuric acid had been added. The base solution temperature was not allowed to go above 9°C during the addition. There was slight fuming (nitrous fumes) during the reaction. On completion of the addition the suspension was stirred for a further 10 min; then it was warmed. When the suspension temperature reached 20°C 180 ml of a 1:1 v/v water/sulphuric acid solution was added over 5 min. At the end of this addition the temperature was 24°C. The suspension was then stirred for one hour then cooled to 10°C. The blue gelatinous precipitate was filtered off on a Buchner funnel (27 cm; Whatman 541 paper) under vacuum. The product was washed three times with water and was finally sucked damp dry to give a cake weighing 390 g. This cake was wrapped in a cloth and kept under a little water.

3.2.2 Preparation of the Sodium Salt Solution

Two cakes of the copper salt, prepared as above, were washed into the 10 litre pan with a total of 2.5 litre of water. The temperature of the suspension was taken up to and maintained at 80°C. A normal sodium hydroxide solution was run in until the solution proved to be alkaline (pH paper). This required 1030 ml of the sodium hydroxide solution. The resultant black suspension was cooled and filtered by gravity through Whatman 541 papers to remove the cupric oxide precipitate. 5.2 litres of combined filtrate and washings were obtained which when analysed contained 24.0 g/l NT^θ. This meant that an addition of further 65 ml of normal sodium hydroxide was required to convert all the NT^θ to sodium salt. (1.095 moles of NT^θ formed but only 1.030 moles of NaOH added previously). The resultant solution was evaporated down on a steam bath to 1.5 litres, filtered and diluted to 2.0 litres. This solution to be diluted as required for the preparation of the silver salt. Analysis of this solution showed it contained 59.5 g/l NT. Overall yield from 5-aminotetrazole = 52%.

Notes

(a) The sodium salt is used as a solution because it crystallizes as a tetrahydrate which readily loses water and rehydrates according to the ambient humidity and temperature.

(b) The reaction of sodium hydroxide with $\text{Cu}(\text{NT})_2 \cdot \text{NTH} \cdot 4\text{H}_2\text{O}$ can be followed by sampling the reaction mixture and measuring the pH of the solution. The end point occurs at pH 9. The use of pH paper is not satisfactory because of the cupric oxide suspension which tends to confuse the colours towards the end point.

3.3 Preparation of Silver 5-Nitrotetrazole

3.3.1 Pure A Type

This was prepared from sodium 5-nitrotetrazole and silver nitrate at high temperature as described previously.³ It was obtained as colourless acicular crystals which did not flow freely and had a low bulk density (Fig 1).

3.3.2 Pure F Type

To a base solution containing 67 ml of sodium 5-nitrotetrazole solution (59.5 g/l NT^θ), 90 ml of water, 2 ml of 0.880 ammonium hydroxide solution and 15 ml of a 1% solution of ORATAN SN at 20°C was added in 60 min a solution containing 5.9 g of silver nitrate in 60 ml of water. The resultant suspension was then treated with 25 ml of nitric acid (110 g/l) to give 7.6 g (98.4%) of silver 5-nitrotetrazole (F type). (Fig 2.)

3.3.3 F Type with Silver Carboxy Methyl Cellulose

To a base solution containing 67 ml of sodium 5-nitrotetrazole solution (59.5 g/l NT^θ), 100 ml of water, 2 ml of 0.880 ammonium hydroxide solution and 7.5 ml of 1% sodium carboxymethyl cellulose solution at 25°C was added over 45 min a solution containing 5.9 g of silver nitrate in 60 ml of water. The resultant suspension was treated with 20 ml of nitric acid (110 g/l) to give 7.3 g (94.5%) of silver 5-nitrotetrazole (F type). (Fig 3.)

3.3.4 Other Crystal and Polymorph Modification Experiments

Although some six types of silver 5-nitrotetrazole were found by X-ray powder photography patterns only three could be isolated pure - those designated ADF. In all cases the reactions were carried out with silver nitrate except for the production of the pure D form.

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The precipitation conditions and the products obtained are summarised below:

(a) From nitric acid solutions the A form was the main product. It could be obtained pure as in 3.3.1 but with varying conditions and additives it was obtained in a mixture with the B, C, D and E Forms.

(b) From neutral solution the main product was the A form but usually in admixture with the D and E forms.

(c) From ammoniacal solutions the main product was F which was obtained pure as in 3.3.3. With varying conditions and additives mixtures of the F, D or E form were obtained.

The pure D form (Fig 4) was obtained by simultaneous addition of sodium nitrotetrazole and silver nitrate to dilute ammonium hydroxide followed by neutralization with dilute nitric acid.

3.4 Preparation of Mercuric 5-Nitrotetrazole

This was carried out in the 600 ml mixer in a similar manner to that outlined previously³ except that the concentrations were increased as below.

To a stirred solution of 195 ml of sodium nitrotetrazole (59.5 g/l NT⁰) and 2 ml conc nitric acid at 80°C was added a solution of mercuric nitrate monohydrate (17.1 g) in dilute nitric acid (water 100 ml, nitric acid 2 ml) over a period of 40 min. The product was isolated as small aggregates in 80% yield (17.2 g). (Fig 5.)

Only one form of mercuric salt was ever isolated (A type).

3.5 Destruction Procedure for 5-Nitrotetrazole Salts

3.5.1 Copper Salt

This was destroyed by treating with an excess of 5 N hydrochloric acid and zinc metal for 24 hours. A precipitate of copper metal is formed and is filtered out together with the excess zinc metal. The resultant solution was shown by UV-visible spectroscopy to contain no nitrotetrazole anion. The solid metal residues were non-explosive as shown by dropping them on a hot plate.

3.5.2 Sodium Salt

It was shown that this could be reduced by ammoniacal ferrous sulphate, titanous chloride as well as zinc and hydrochloric acid. The preferred method

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is to reduce with zinc metal and 5 N hydrochloric acid for 24 hours.

3.5.3 Silver Salt

This was destroyed by wetting the salt with water then adding an excess of 5 N hydrochloric acid. This caused silver chloride to precipitate out. Zinc metal was then added to the resultant suspension and left to reduce the nitro-tetrazole anion for 24 hours.

3.5.4 Mercuric Salt

Again this was destroyed with zinc and hydrochloric acid in a manner similar to that for the copper salt.

All filling residues of the salts were destroyed by burning after soaking in paraffin/diesel oil mixtures overnight.

4 PROPERTIES OF SILVER AND MERCURIC 5-NITROTETRAZOLES

4.1 Chemical Analysis

TABLE 1

Compound	Lot No	C, H, N % Analysis	X-Ray Analysis
Silver 5-nitrotetrazole	BW6	Calc C, 5.45; H, 0.0; N, 31.8 Found C, 5.8; H, <0.1; N, 31.8	A
"	BW123	Calc C, 5.45; H, 0.0; N, 31.8 Found C, 5.8; H, 0.3; N, 32.1	F
"	BW125	Calc C, 5.45; H, 0.0; N, 31.8 Found C, 5.7; H, 0.4; N, 31.7	F + little E (Ag CMC)
"	BW96	Calc C, 5.45; H, 0.0; N, 31.8 Found C, 5.67; H, 0.2; N, 31.7	D
Mercuric 5-nitrotetrazole	BW128	Calc C, 5.6; H, 0.0; N, 32.6 Found C, 5.67; H, 0.2; N, 32.4	A

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4.2 SensitivenessTABLE 2

Composition	Silver 5-nitrotetrazole A Polymorph	Silver 5-nitrotetrazole F + Ag CMC	Mercuric 5-nitrotetrazole A
Batch	BW6	BW125	BW128
Minimum electrostatic energy to fire metal/metal electrodes (μ J) (RD1346 = 4 μ J)	12	12	270 (partials)
Ball and disc RD1651 4 cm 34/50 fires 3 1/2 " 20/50 " 3 " 1/50	4 cm 6/10 fires 2 1/2 " 4/50 " 2 " 2/50 "	5 cm 6/10 fires 4 " 10/50 " 3 " 6/50 " 2 " 0/50 "	6 cm 3/50 fires 5 cm 0/50 "
Emery friction RD1651 5 ft/s 16/20 fires 4 " 9/20 " 3 " 1/20 " 2 1/2 " 0/20 "	4 ft/s 5/5 fires 3 " 2/20 " 2 " 1/20 " 1 1/2 " 0/20 "	3 ft/s 3/10 fires 2 " 1/20 " 1 1/2 " 0/10 "	5 ft/s 4/10 fires 4 " 2/10 " 3 " 0/10 "

4.3 Thermal Stability/Compatibility4.3.1 Thermal StabilityTABLE 3

Compound	Silver 5-nitrotetrazole A Polymorph BW6	Silver 5-nitrotetrazole F + Ag CMC BW125	Mercuric 5-nitrotetrazole BW128
Temperature of ignition, °C 5 °C/min	265	250	190
% Mass loss at 100 °C 0.2 mmHg vac for 1 week	0.3	Not done	0.06
% Mass loss at 230 °C* 1 mm pressure 1 hour	4.5	Not done	Not done

*Ref 4

COMPATIBILITY (4)

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4.3.2 Compatibility of Mercuric 5-Nitrotetrazole

Preliminary tests indicated that the mercuric salt is less compatible with sulphur-containing rubbers and aluminium metal than the silver salt.⁴ Further work was concentrated on the silver salt.⁴

4.3.3 Compatibility of Silver 5-Nitrotetrazole

The conclusions reached by the Compatibility Section of ERDE are as follows:⁵

- 1 It is not unduly susceptible to hydrolysis.
- 2 It does not readily evolve corrosive acid vapours on hydrolysis.
- 3 It is likely to be less susceptible to attack by vapours from plastics and rubbers than are lead and silver azides.
- 4 It reacts rapidly with tetrazene at high humidities and high temperatures.
- 5 Its incompatibility with aluminium, under humid conditions casts doubt upon the advisability of filling it into shells of this metal.
- 6 It corrodes aluminium worse than it does copper; stainless steel is unaffected.

4.4 Stab-Sensitivity

Both the silver and mercuric salts were tested by EM1 RARDE for stab-sensitivity and were found to be insensitive.

4.5 Detonant Tests

4.5.1 ERDET Test

TABLE 4

Compositions	Silver 5-nitrotetrazole A BW6	Silver 5-nitrotetrazole F + Ag CMC BW125	Mercuric 5-nitrotetrazole A
ERDET Test (wt found to detonate RDX in an ERDET 75 mg RD1343 required)	30 mg	50 mg	30 mg
Pressing load	Dead pressed at 46 MN/m ²	Dead pressed at 62 MN/m ²	Performance improves over 77 MN/m ²

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4.5.2 2.3 gr LZY Test (Conducted by EM1 RARDE)

(a) Silver 5-Nitrotetrazole BW125 did not detonate CE most probably as 120 - 150 MN/m² pressing load was necessary to get a reasonable quantity into the shell.

(b) Mercuric 5-Nitrotetrazole did detonate CE but its performance was well down compared with service lead azide. It was possible to get 55 mg of the salt into the detonator compared with over 90 mg of service lead azide.

5 CONCLUSIONS

5.1 Silver 5-nitrotetrazole is an efficient detonant under low pressing loads but dead presses at 62 MN/m² which renders it unserviceable as a lead azide replacement for small detonators.

5.2 Silver 5-nitrotetrazole is a stable, compatible priming explosive and should be considered in any requirement in which the dead pressing level can be tolerated or avoided.

5.3 Mercuric 5-nitrotetrazole is slightly less stable and compatible than the silver salt. However, it does not dead press and could be considered as a detonant if stainless steel detonator shells could be used. Work would be required to increase the bulk density of the material to bring it closer to that of service lead azide.

5.4 Neither the silver nor mercuric salt of 5-nitrotetrazole show any promise as stab-sensitive compositions.

6 RECOMMENDATIONS

6.1 It has been shown that the 5-nitrotetrazole anion can be produced on a technically useful scale and that it can give rise to stable salts which are capable of acting as detonants. It is recommended that as part of the programme to search for new detonants other salts of 5-nitrotetrazole be investigated.

6.2 This work has demonstrated that the only meaningful test for assessment of a replacement of lead and silver azide as detonants in small detonators is to actually use small detonators themselves. It is therefore recommended that ERDE set up its own facility for testing compositions in small detonators.

6.3 Although unsuitable for current UK design requirements the results show gains in several respects over existing detonants in service and the explosives may have potential application to other designs, eg to US Navy

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detonators. It is recommended that technical interchange on their properties and assessment should be maintained.

6.4 It is recommended that further crystal modification work be carried out on the silver and mercuric salts, to give materials suitable for use in UK small detonators.

7 REFERENCES

- | | | |
|---|-----------------------------------|---|
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| 2 | Morisson H and references therein | "Chimie des derives explosives des tetrazoles". Collogue International sur l'utilisation des Elements Pyrotechnics et Explosives, July 1968, p 111. |
| 3 | Jenkins J M, White J R | ERDE Technical Note No 21. |
| 4 | Blay N J, Rapley R J | Private communication. |
| 5 | Blay N J et al | ERDE Technical Report No 163. |



FIG.1 A TYPE. BW6
SILVER 5-NITROTETRAZOLE

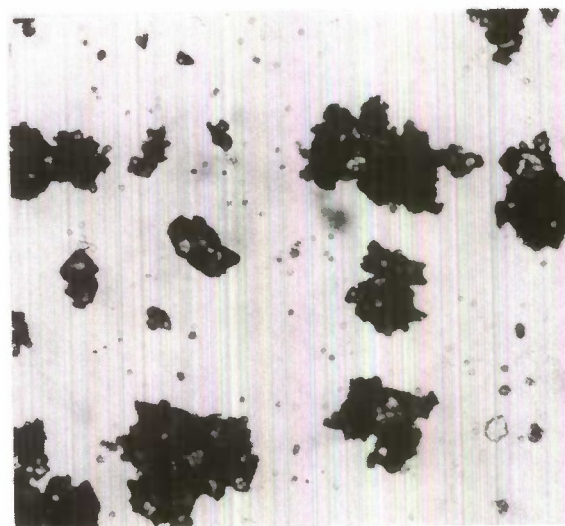


FIG.2 F TYPE. BW123
SILVER 5-NITROTETRAZOLE



FIG.3 F TYPE + Ag CMC. BW125
SILVER 5-NITROTETRAZOLE

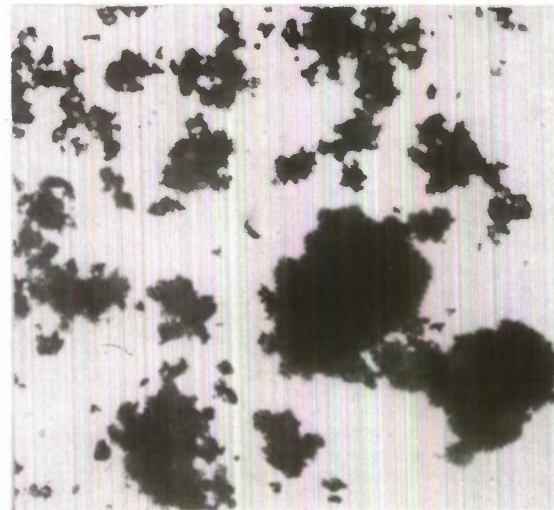


FIG.4 D TYPE. BW96
SILVER 5-NITROTETRAZOLE

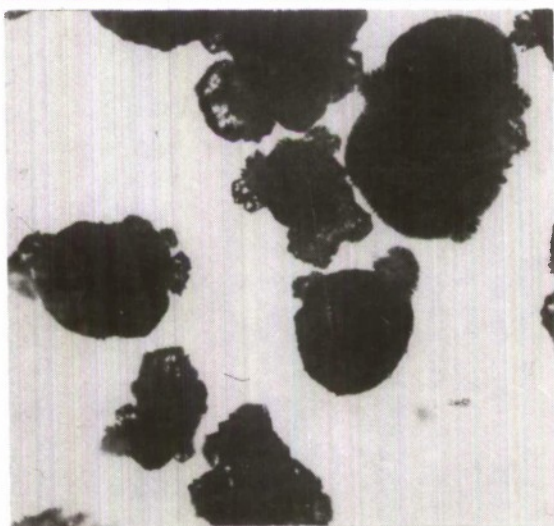


FIG.5 BW128
MERCURIC 5-NITROTETRAZOLE

SILVER AND MERCURIC SALTS OF
5-NITROTETRAZOLE. X 100

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Abstract A form of silver 5-nitrotetrazole has been developed which can be filled into small devices. It has been tested, together with mercuric 5-nitrotetrazole, for detonant and stab-sensitive properties. Both compositions have been shown to be stable and will efficiently detonate RDX in large detonators but neither, in their current physical form, are technically suitable for current UK design of small detonators. Recommendations are made concerning the future synthetic and crystal modification programmes, methods of assessment, and potential application of these compounds.			

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