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NAVORD REPORT 2307

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PRIMARY EXPLOSIVES RESEARCH

III. Synthesis and Examination of 5-Amino-1,2,3,4-Thiatriazole and the Metal Salts of 3,5-Dinitro-1,2,4-Triazole

20 December 1951

U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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PRIMARY EXPLOSIVES RESEARCH

III. Synthesis and Examination of 5-Amino-1,2,3,4-Thiatriazole and the Metal Salts of 3,5-Dinitro-1,2,4-Triazole

Prepared by:

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ABSTRACT: The following metal salts of 3,5-dinitro-1,2,4-triazole were prepared and examined as possible new primary explosives.

Cupric	Lead	Silver
Ferric	Mercuric	Tin
Ferrous	Mercurous	Zinc

All but the silver and mercurous salts were rejected because of excessive water solubility. These compounds had satisfactory thermal stability, but their impact sensitivities were in the range of that of RDX. Further tests are being made on the silver salt and the results will be reported elsewhere.

5-Amino-1,2,3,4-thiatriazole was prepared and examined as a possible primary explosive. Its impact sensitivity was in the range of that of lead azide, but the compound was thermally unstable. No further work on it is contemplated.

Explosives Research Department
U. S. NAVAL ORDNANCE LABORATORY
White Oak, Maryland

NAVORD Report 2307 20 December 1951

This report, for information only, is a description of the preparation of the heavy metal salts, particularly the silver and mercurous, of 3,5-dinitro-1,2,4-triazole, and of 5-amino-1,2,3,4-thiatriazole, performed under Task NOL-Re2b-41-1-52. The reliability of the work and the validity of the conclusions are the responsibility of the author and of the Chemistry Division, Explosives Research Department, of the U. S. Naval Ordnance Laboratory.

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By direction

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PRIMARY EXPLOSIVES RESEARCH

III. Synthesis and Examination of 5-Amino-1,2,3,4-Thiatriazole and the Metal Salts of 3,5-Dinitro-1,2,4-Triazole

INTRODUCTION

1. Working on a Bureau of Ordnance Contract for high explosives research, the Naugatuck Chemical Division of the United States Rubber Company discovered a method of preparing 3,5-dinitro-1,2,4-triazole and prepared several of its salts. They suggested that the silver and lead salts might find some use as primary explosives, since they were too sensitive for consideration as high explosives.

2. Of the following metal salts, the only ones insoluble in water were the silver and mercurous compounds.

Cupric	Lead	Silver
Ferric	Mercuric	Stannous
Ferrous	Mercurous	Zinc

It is believed that water insolubility is a desirable physical property for a compound being considered as a possible new primary explosive. Therefore all of the water soluble salts were dropped from consideration.

3. Freund and Schander reported the preparation of 5-amino-1,2,3,4-thiatriazole in the early literature (2). The Naugatuck Chemical Division repeated the work and suggested the compound as a possible primary explosive (3).

EXPERIMENTAL

Silver Salt of 3,5-Dinitro-1,2,4-Triazole

4. The Naugatuck Chemical Division supplied a sample of the sodium salt of 3,5-dinitro-1,2,4-triazole, the starting material for the preparation of the silver and mercurous salts, prepared as described in their report (1). The sodium salt (2 grams, 0.01 mole) was dissolved in 20 cc of distilled water and stirred mechanically. To the aqueous sodium salt solution was added dropwise an aqueous solution of silver

nitrate (1.8 grams, 0.01 moles in 15 cc of distilled water). The silver salt precipitated immediately. The reaction mixture was stirred for an additional ten minutes and then filtered on a Buchner funnel. The yellow solid was washed with liberal quantities of distilled water and finally dried under vacuum at 50°C. The salt was analyzed for silver by the method described by Pregl (4).

Calculated for $C_2O_4N_5Ag$: Ag, 40.6

Found: Ag, 39.3, 41.27

The physical properties observed for the silver salt are tabulated in Table I.

Mercurous Salt of 3,5-Dinitro-1,2,4-Triazole

5. The mercurous salt of 3,5-dinitro-1,2,4-triazole was also prepared using the sodium salt as the starting material. An aqueous solution of mercurous nitrate was prepared by dissolving 15 grams (0.055 moles) in 500 cc of distilled water containing 5 cc of concentrated nitric acid. The aqueous mercurous nitrate was stirred mechanically while an aqueous solution of the sodium salt (4.5 grams, 0.025 moles, in 15 cc of distilled water) was added dropwise. On addition of the sodium salt a yellow solid precipitated immediately. The solid was filtered, washed with distilled water, and dried under vacuum at 50°C. The solid was analyzed for mercury by the Oakwold Laboratory, Alexandria, Virginia.

Calculated for $C_2O_4N_5Hg$: Hg, 56.0

Found: Hg, 53.64, 53.82

This compound was reported by the Oakwold Laboratory to be very volatile and subject to violent decomposition, so that the accuracy of the analysis is open to some question. The physical properties observed for the mercurous salt are listed in Table I.

5-Amino-1,2,3,4-Thiatriazole

6. The method described by Freund and Schander for the preparation of 5-amino-1,2,3,4-thiatriazole was repeated (2). The crude material isolated from the reaction melted at 120°C. One recrystallization from ether raised the melting point 128-129°C. It was observed that sometimes the sample would melt in a capillary but that occasionally it would detonate. The literature reported the melting point as a detonation temperature. The physical properties observed are tabulated in Table I.

TABLE I

Compound	Impact Sensitivity 2.5 kg weight	Vacuum Stability cc gas/48 hrs/100°C	Density	Ignition Temperature
Silver-3,5-Dinitro 1,2,4-Triazole	26 cm	2.09 cc	2.781	286°C /1
Mercurous-3,5-Dinitro-1,2,4-Triazole	34 cm	0.12 cc	------	------
5-Amino-1,2,3,4-Thia-Triazole	8.6 cm	EC /2	------	128°C

/1 Found to detonate in two out of two trials with a hot wire energized by 1000,000 ergs of electrical energy (one mil diameter tungsten).

/2 Exceeded the capacity of the thermal stability equipment (30 cc) both at 100°C and 90°C.

CONCLUSIONS

7. The silver and mercurous salts of 3,5-dinitro-1,2,4-triazole exhibit satisfactory thermal stability but neither appeared to possess the necessary sensitivity for a primary explosive. The 5-amino-1,2,3,4-thiatriazole possessed the necessary sensitivity for a primary explosive, but had very poor thermal stability. A sample of the silver salt was prepared for further evaluation by the Explosives Properties Division, Explosives Research Department, and the Chemistry Division, Engineering Department, of the U. S. Naval Ordnance Laboratory. These results will be reported elsewhere. No further work is planned on the 3,5-dinitro-1,2,4-triazole molecule.

ACKNOWLEDGEMENTS

8. The author wishes to acknowledge the assistance of J. M. Rosen for the thermal stability measurements and of B. O. Wilkerson for the metal analyses, both of the Chemistry Division, and of G. Svadeba, Explosives Properties Division, who made the impact sensitivity measurements.

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