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NAVORD REPORT

4442

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PRIMARY EXPLOSIVES RESEARCH

VII FREE FLOWING BASIC LEAD STYPHNATE

19 DECEMBER 1956



U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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NAVORD Report 4442

PRIMARY EXPLOSIVES RESEARCH
VII FREE FLOWING BASIC LEAD STYPHNATE

Prepared by:

FRANCIS TAYLOR, JR.

Approved by: DARRELL V. SICKMAN, Chief
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ABSTRACT: Following suggestions of Dr. G. W. C. Taylor, Explosives Research Development Establishment, Waltham Abbey, three modifications of the beta polymorph of lead styphnate have been made by altering the crystal habit by addition of 2-nitroresorcinol. The three products were extremely free flowing, of larger particle size than the commercially available alpha polymorph, and they compared favorably with the alpha polymorph on impact sensitivity and thermal stability. The particle size and crystal shape was dependent on the method of preparation, purity of reactants, and the proportion of the additive, 2-nitroresorcinol. Photomicrographs show the shape of the crystal aggregates and the particle size. X-Ray diffraction data is given for the alpha and beta polymorphs. The material is at present being evaluated for use in Primer mixes NOL 60 and NOL 130.

EXPLOSIVES RESEARCH DEPARTMENT
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This report is a description of the preparation of three modifications of the β -polymorph of basic lead styphnate performed under Task NO 800-667/76004/01040.

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. This report is for information only.

W. W. WILBOURNE
Captain, USN
Commander


J. E. ABLARD
By direction

TABLE OF CONTENTS

	Page No.
INTRODUCTION.....	1
RESULTS AND DISCUSSIONS.....	2
EXPERIMENTAL.....	3
Preparation of Tan-Yellow Form of β -Basic Lead Styphnate (RD1346-A).....	3
Preparation of the Red Form of β -Basic Lead Styphnate (RD1346).....	4
Preparation of the Tan Form of β -Basic Lead Styphnate (RD1349).....	4
CONCLUSIONS AND RECOMMENDATIONS.....	5
ACKNOWLEDGEMENTS.....	5
TABLE I Interplanor "d" Spacing for Basic Lead Styphnates.....	6
FIGURE I Basic Lead Styphnate.....	7
APPENDIX I Styphnic Acid.....	8
BIBLIOGRAPHY.....	11

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NAVORD Report 4442

PRIMARY EXPLOSIVES RESEARCH
VII. FREE FLOWING BASIC LEAD STYPHNATE

INTRODUCTION

Previous research on primary explosives in our laboratory has covered a broad class of compounds. We have investigated the salts of ethylenedinitramine, the polymorphs of HMX, and derivatives of 5 substituted tetrazoles. This work has been reported in previous NAVORD Reports (1)(2)(3)(4)(5)(6).

The Navy at one time relied heavily on mercury fulminate priming mixtures for initiator material in many percussion and stab primers and detonators. The fulminate priming mixtures showed unsatisfactory surveillance characteristics and their use was curtailed around 1930 (7). Attempts to replace mercury fulminate with lead azide were not completely satisfactory particularly for electric initiation (7). In an attempt to obtain priming mixtures which would better fulfill the requirements of the Navy, the U. S. Naval Ordnance Laboratory initiated a task to formulate priming mixtures which would best meet specific requirements.

Previous work at Western Cartridge and Remington Arms Company showed that both the normal and basic lead salts of styphnic acid (2,4,6-trinitroresorcinol) were satisfactory components of the initiating mixture for a delay detonator (8). The lead salts of styphnic acid are described in the early literature. Griess reported the discovery of the basic salt in 1874 (9) and Herz claimed the first preparation of the normal salt in 1919 (10).

Two primer mixes were developed at the U. S. Naval Ordnance Laboratory which utilized basic lead styphnate, Primer mix NOL 60 and NOL 130. The NOL 60 is used in percussion primers and the NOL 130 in all of the Navy's stab primers and detonators (7).

The commercially available basic lead styphnate used in Primer mixes NOL 60 and NOL 130 is the alpha polymorph. The salt is tan-brown in color and all of the particles are less than 40 microns in the longest dimension. This is shown by the photomicrograph in Figure I. The material, which is sensitive to static electricity, dusts badly during handling, and clings to weighing scoops and bridges funnel openings. These primer mixes are thus not very adaptable to automatic loading devices.

Dr. G. W. C. Taylor (11) of the Explosives Research Development Establishment, Waltham Abbey, has been studying normal and basic lead styphnates, paying particular attention to their crystal habit and size. Taylor has had considerable success in isolating three modifications of the beta polymorph

of basic lead styphnate (12). One modification, reddish brown in color, designated by Taylor as RD1346 will be referred to as such throughout the report. Another modification, designated RD1346A, consisted of tan oval shaped crystal aggregates. The third modification was also tan colored crystals and identified as RD1349. The British feel that RD1349 is probably the best form of basic lead styphnate to combine good flowing and mixing properties with safe handling.

RESULTS AND DISCUSSION

The reddish brown modification, RD1346, appeared as rounded crystal aggregates with a particle size range of 0.05 - 0.10 mm. The size of these crystal aggregates is considerably larger than that of commercial basic lead styphnate available in the United States. RD1346 compared favorably on impact sensitivity and thermal stability with the alpha polymorph.

Our first attempt to prepare RD1346A resulted in the alpha polymorph. Taylor attributed this to the purity of our styphnic acid and the use of too small a quantity of additive. On subsequent preparations we were able to isolate the beta polymorph, but the particle size was smaller and it was not as free flowing as RD1346. The RD1346 A modification was the least desirable of the three from our limited experience.

RD1349 was readily prepared and its physical properties compared favorably with those of RD1346. Figure I shows photomicrographs of RD1346, RD1349 and the alpha polymorph.

The British found that the crystal shape and size of the modified beta polymorph are very dependent on the method of preparation employed, purity of the reactants, and the amount of additive used to modify the crystal growth. The additive used was 2-nitroresorcinol. If the same percentage of 2-nitroresorcinol was used in both a 100 g. and 300 g. preparation, the resulting products differed in shape and size. The amount of additive needed must be determined experimentally, and may vary from 0.5 to 4.0 mole percent based on styphnic acid. The purity of the styphnic acid also has a marked effect on crystal size. The British recommendations for the purity of the styphnic acid used in the preparation of the beta polymorph of the basic lead salts are given in Appendix I.

X-Ray diffraction measurements were relied upon to determine the polymorphic forms isolated from the various preparations. The characteristic interplanar spacings are

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NAVORD Report 4442

listed in Table I for both the alpha and beta polymorphs.

The red polymorph, RD1346, was selected as the material for evaluation in the JAN Cycle Test MIL.-STD.-304. The test vehicle was the Primer Mk 102 Mod 1 loaded with primer mix NOL 130. There was no significant difference in sensitivity between the test samples and samples made using the alpha polymorph both at the beginning and after twenty-eight days of cycling. The results of the complete surveillance evaluation will be reported elsewhere by the Fuze Department. Static sensitivity measurements already have been reported for RD1346 and RD1349 (13).

EXPERIMENTAL

PREPARATION OF TAN-YELLOW FORM OF β -BASIC LEAD STYPHNATE
(RD1346-A)

Reagents:

Styphnic acid, m. 175°C

2-nitroresorcinol

Ammonium hydroxide solution, 56 g/l. NH_3

Lead acetate solution, 315 g/l. $\text{Pb}(\text{AC})_2 \cdot 3\text{H}_2\text{O}$

Procedure:

Styphnic acid (200 g, 0.8 mole) and 4.0 g. of 2-nitroresorcinol were suspended in two liters of distilled water and stirred mechanically. The mixture was heated to 50°C and then one liter of an ammonium hydroxide solution (56 g/l. NH_3) added and the temperature of the clear solution raised to 68°-70°C. A solution of 1.94 liters of lead acetate (315 g/l. $\text{Pb}(\text{AC})_2 \cdot 3\text{H}_2\text{O}$, 1.6 mole) was added dropwise over a period of forty minutes to the vigorously stirred ammonium styphnate solution maintained at 68°-70°C with external heating. After the addition of the lead acetate solution was complete, the mixture was warmed and stirred an additional fifteen minutes. The stirrer was stopped and the precipitate allowed to settle for five minutes. The supernatant liquid was decanted while hot. The product was washed three times with three liters of distilled water by stirring for three minutes and then allowing the solid to settle for five minutes before decanting. After the third wash the product was washed into a wide mouth bottle and stored water wet.

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NAVORD Report 4442

PREPARATION OF THE RED FORM OF β -BASIC LEAD STYPHNATE

(RD1346)

Reagents:

Styphnic acid, m. 175°C
2-Nitroresorcinol
Sodium hydroxide solution, 84.2 g/l. NaOH
Lead nitrate solution 275 g/l. $\text{Pb}(\text{NO}_3)_2$

Procedure:

To a stirred suspension of 103 g (0.4 mole) of styphnic acid in 1.5 liters of distilled water was added a solution of 760 ml of sodium hydroxide (84.2 g/l. NaOH, 1.6 mole) diluted with 740 ml of distilled water, and 1.0 g of 2-nitroresorcinol. The clear red solution was filtered and added dropwise over a period of ninety minutes to a stirred solution of one liter of lead nitrate (275 g/l. $\text{Pb}(\text{NO}_3)_2$ 0.8 mole) diluted with 1.65 liters of distilled water and heated at 68°-70°C. The temperature was maintained at 68°-70°C and the reaction mixture stirred an additional ten minutes after the addition of the sodium styphnate was completed. The stirrer was stopped and the precipitate allowed to settle for two minutes. The supernatant liquid was decanted hot and the product washed three times with three liters of distilled water, allowing three minutes stirring and two minutes settling time per wash. The product was washed into a wide mouth bottle and stored under water.

PREPARATION OF THE TAN FORM OF β -BASIC LEAD STYPHNATE

(RD1349)

Reagents:

Styphnic acid, m. 175°C
2-Nitroresorcinol
Ammonium hydroxide solution 56 g/l. NH_3
Lead acetate solution, 315 g/l. $\text{Pb}(\text{AC})_2 \cdot 3\text{H}_2\text{O}$

Procedure:

Styphnic acid (153 g, 0.6 mole) and 3.0 g of 2-nitroresorcinol were suspended in 2.25 liters of distilled water and stirred vigorously. The mixture was warmed to 50°C and then 750 ml of ammonium hydroxide solution (56 g/l. NH_3 ,

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NAVORD Report 4442

2.45 mole) added and the temperature of the clear solution raised to 68°-70°C and stirred for five minutes. A solution of 1.45 liters of lead acetate (315 g/l. $\text{Pb}(\text{AC})_2 \cdot 3\text{H}_2\text{O}$, 1.2 mole) to which was added 300 ml of ammonium hydroxide (56 g/l. NH_3) was added dropwise over a period of thirty-five minutes to the stirred ammonium styphnate solution heated at 68°-70°C. The temperature was maintained and stirring continued an additional fifteen minutes, then the solid was allowed to settle for five minutes. The supernatant liquid was decanted while hot and the precipitate washed three times with three liters of distilled water, stirred three minutes, and a five minute settling period allowed per wash. The product was washed into a wide mouth bottle and stored under water.

CONCLUSIONS AND RECOMMENDATIONS

Three modifications of the beta polymorph of basic lead styphnate can be prepared on a 100 and 300 gram scale, and the quality of the products duplicated in repeated lots. We are not equipped to make the material on a larger scale at the present. Preparation on a ten pound scale, as normally done by the British, should offer no difficulty, although some experimentation may be needed to establish the operating details.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the assistance of Dr. G. W. C. Taylor who kindly supplied us with complete laboratory directions for the preparation of the three modifications of the beta polymorph. He also wishes to acknowledge the assistance of Dr. J. M. Rosen for the photomicrographs and Dr. J. R. Holden for the X-ray crystallographic data.

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NAVORD Report 4442

TABLE I
INTERPLANAR "d" SPACING FOR BASIC LEAD STYPHNATES*

Alpha Polymorph		Beta Polymorph	
Intensity	A° "d"	Intensity	A° "d"
S ¹	9.00	S	8.07
W ²	8.12	W	6.62
W	7.33	M	5.76
W	5.96	W	5.12
W	4.83	M	4.51
M ³	4.48	W	4.00
W	4.05	W	3.86
W	3.91	M	3.64
M	3.71	M	3.23
S	3.40	M	3.05
S	3.00	W	2.87
W	2.72	W	2.75
WD ⁴	2.93	S	2.56
W	2.60	M	2.44
WD	2.50	W	2.34
M	2.37	W	2.28
W	2.20	W	2.24
W	2.08	M	2.18
W	1.94	W	2.12
M	1.79	M	2.07
W	1.73	W	2.01
WD	1.62	W	1.94
		S	1.88
		W	1.84
		M	1.80

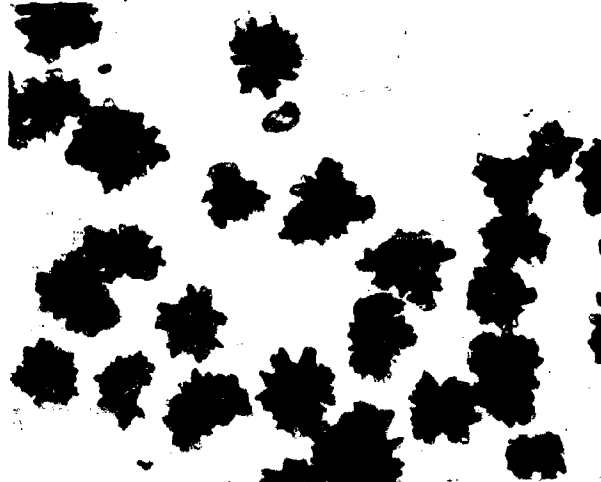
1. S - Strong Intensity
2. W - Weak Intensity
3. M - Medium Intensity
4. WD - Weak Diffuse Intensity

* Data obtained by powder film technique using a camera of 114.6 mm in diameter. Angles of diffraction, 2Θ , were measured from the film and converted to the interplanar "d" spacings, but uncorrected for film shrinkage.

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FIG. 1 BASIC LEAD STYPHATE

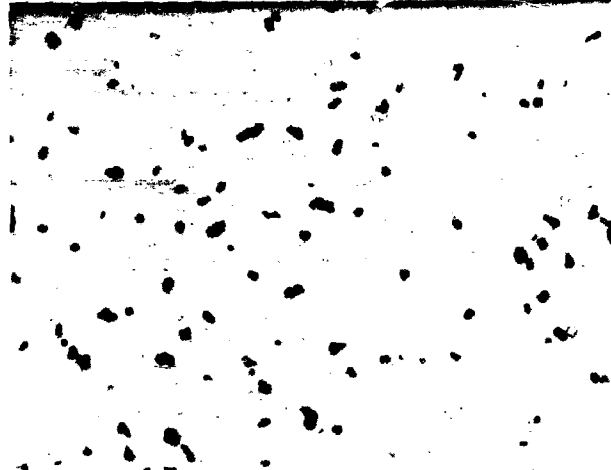
MODIFIED β POLYMORPH X200 RD 1346



MODIFIED β POLYMORPH X400 RD 1349



ALPHA POLYMORPH X200 COMMERCIAL GRADE



7
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APPENDIX I

STYPHNIC ACID

For use in the manufacture of Explosive Compositions

Specification to govern supply and inspection

270
Specs.
1209

Approved 15.12.1944

1. GENERAL. The Styphnic Acid must comply in every respect with terms of this specification.
2. DESCRIPTION. The material must consist essentially of the 2:4:6 trinitro derivative of resorcinol $C_6H_1:3(OH)_2$. 2:4:6(NO_2)₃. It must be in the form of a uniform yellow powder, free from gritty particles, visible impurities and foreign matter. For the purpose of storage and for transport in bulk, the material must be uniformly wetted to contain not less than 20 per cent and not more than 25 per cent of water, calculated on the wet material.
3. EXAMINATION. Samples taken from any portion of the supply must comply with the following requirements:-
 - (a) General. The material must be in accordance with the requirements of para. 2 above.
 - (b) Water Content
 - (i) of the wetted material. The water content of the wetted material, determined as loss in weight when dried in the boiling water oven, must comply with the requirements at 2 above.
 - (ii) of the material without added water. The water content as determined by the procedure of entrainment distillation, Dean and Stark method, using dry benzene as solvent, must not exceed 5.0 per cent.
 - (c) Styphnic Acid Content. The Styphnic Acid content calculated on the material free from moisture must be not less than 98.0 per cent.
 - (d) Melting Point. The melting point of the material, ground to a fine powder and thoroughly dried in vacuo, must be not lower than 175°C and not higher than 178°C.
 - (e) Matter Insoluble in Benzene.
 - (1) The total matter insoluble in dry benzene

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NAVORD Report 4442

determined as described in the Appendix must not exceed 0.25 per cent.

- (ii) The residue on ignition of the insoluble matter must not exceed 0.1 per cent.
- (iii) The ignited residue must be wholly capable of passing a No. 240 British Standard Sieve*.

- (f) Chlorides. The material free from added water must not contain chlorides, calculated as sodium chloride (NaCl) in excess of 0.05 per cent.
- (g) Sulphates. The material free from added water, must not contain sulphates, calculated as sodium sulphate (Na₂SO₄) in excess of 0.1 per cent.
- (h) Nitrates. The material must be free from nitrates.

4. PACKING AND MARKING OF PACKAGES. The Styphnic Acid is to be supplied in sound, clean, approved, lead free packages containing an approved quantity.

In no part of the package shall the amount of lead compounds calculated as metallic lead exceed 0.5%.

The package constituting a consignment must each be marked with:- a description of the contents, the appropriate contract number, a distinctive lot number, a consecutive package number, the tare weight of the package, the net weight of Styphnic Acid (Wet), the net weight of Styphnic Acid (Dry), the date of supply, the contractor's name or recognized trade mark. The paint or other material used for marking and also the paint for the packages, when required, must be of good quality to the satisfaction of the inspecting officer, and must not contain lead or lead compounds calculated as metallic lead together exceeding 0.2 per cent. The inclusion of any foreign matter or impurities in any of the packages will render the whole consignment liable to rejection.

5. INSPECTION. The Styphnic Acid and the packages in which it is contained will be subject to inspection by, and final approval of, the Inspecting Officer.

Samples of the material and of the packages may be taken from any portion of a consignment.

* The British Standard (B.S.) Sieve (s) mentioned above is/are the Fine Mesh Normal Test Sieve (s) governed by British Standard Specification No. 410-1943 (obtainable from the British Standards Institution, 28, Victoria Street, London, S.W.1).

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NAVORD Report 4442

The weight of dry material in a consignment will be determined on the basis of the average moisture content of the samples taken from the cases comprising the consignment.

If, on examination, any sample be found not to conform to this specification, the whole consignment may be rejected. All samples are to be supplied free of charge.

The foregoing provisions shall apply equally to prime contractors and to any sub-contractors.

Grind a portion of the thoroughly mixed sample and pass the ground material three times through a No. 36 B.S. sieve to ensure homogeneity of the resulting product. Dry the ground material, spread in a layer 0.5 to 1 centimetre in depth over freshly dried silica gel to constant weight.

Dissolve 5 grams of the dried and blended sample in 200 ml. of dry benzene on a boiling water bath, and stand the beaker, not exposed to steam, on the bath for half-an-hour to allow the insoluble matter to settle. Decant the supernatant liquid through a tared asbestos-padded gooch crucible and wash the insoluble matter in the beaker by decantation with hot benzene. Wash the crucible with hot benzene, dry and weight. Let the difference in weight be 'a' grams.

Dissolve the residue in the beaker in a minimum quantity of hot water, and wash completely into a tared light glass dish. Evaporate to dryness on a water-bath. Dry for one hour at 100°C and reweigh. Let the difference in weight be 'b' grams.

The percentage insoluble matter = $20(a + b)$.

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NAVORD Report 4442

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