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HEAT RESISTANT EXPLOSIVES XII
QUANTITATIVE ANALYSIS OF 1,3,5-
TRIAMINO-2,4,6-TRINITROBENZENE (C)

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HEAT RESISTANT EXPLOSIVES XII
QUANTITATIVE ANALYSIS OF 1,3,5-TRIAMINO-2,4,6-TRINITROBENZENE (C)

By

Donald J. Glover

Approved by: D. V. SICKMAN, Chief
Organic Chemistry Division

ABSTRACT: A spectrophotometric assay method using concentrated sulfuric acid as the solvent is presented for 1,3,5-triamino-2,4,6-trinitrobenzene. When the purity is 90 percent or better there is no interference from any of the precursors.

In addition to the assay method, a procedure is given for determining water soluble impurities. Qualitative identification of the precursors is afforded by a spectral determination in gamma-butyrolactone or dimethylformamide.

The quality of 1,3,5-triamino-2,4,6-trinitrobenzene, prepared by amination in benzene and dioxane, is discussed. (C)

CHEMISTRY RESEARCH DEPARTMENT
U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, SILVER SPRING, MARYLAND

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This report describes analytical methods for evaluating the quality of 1,3,5-triamino-2,4,6-trinitrobenzene, TATB, an excellent heat resistant explosive. The spectrophotometric procedures give an accuracy and precision each of $\pm 1\%$. The chlorine analysis is accurate to about 0.05 mg and the water soluble analysis to about 0.1 mg. The precision of both of these methods will depend on the sample size taken for analysis. As additional interest has been generated in TATB, the methods described should be useful in research and engineering studies planned for the near future.

Funds allocated under Tasks FR-44 and RUME 3E012 were used for this project.

W. D. COLEMAN
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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HEAT RESISTANT EXPLOSIVES XII
QUANTITATIVE ANALYSIS OF 1,3,5-TRIAMINO-2,4,6-TRINITROBENZENE

INTRODUCTION

In view of its good high temperature stability, 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) is being evaluated as an explosive for use in high velocity missiles. Its method of preparation and properties are described elsewhere (1) (2). This report is concerned with its analysis.

TATB melts between 440-450°C with decomposition, and consequently, the melting point cannot be used to judge its quality. The criteria of its purity has been its vacuum stability and total chlorine content, and for special cases, its elemental analysis. It can be analysed by determining the evolved ammonia from the reaction with aqueous sodium hydroxide (8). The spectrophotometric procedures herein described allow an estimation of the impurities also, even though the assay can only be accurate to ±1%.

RESULTS

The best solvents for TATB are concentrated sulfuric acid (96% by weight) in which the solubility is approximately 0.1g/ml, nitrobenzene (0.9g/liter), dimethylformamide, aniline, phenylhydrazine, ethylenediamine, and gamma-butyrolactone. There is a reaction of TATB with the latter three solvents, at least to some degree, and also with hot concentrated sulfuric acid. From concentrated sulfuric acid at room temperature, and the other solvents, TATB may be recovered unreacted. The purest samples have been obtained by recrystallization from nitrobenzene and dimethylformamide (3). The analytical methods described in this report are based on TATB recrystallized from nitrobenzene.

Spectra of TATB and its Precursors

In either dimethylformamide or gamma-butyrolactone, not only TATB, but its precursors also (when prepared from purified 1,3,5-trichloro-2,4,6-trinitrobenzene), have well defined maxima. The basic solvent ethylenediamine gives essentially the same maxima location for TATB and its precursors, but the

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acidic solvent sulfuric acid represses the maxima of the precursors and does not interfere with that of TATB. Thus one can get a reasonably good assay in concentrated sulfuric acid, and at least a qualitative identification of the organic impurities in dimethylformamide or gamma-butyrolactone.

When benzene is used as the solvent for the amination of 1,3,5-trichloro-2,4,6-trinitrobenzene (I), the impurities are (I), ammonium chloride (II), 1-amino-3,5-dichloro-2,4,6-trinitrobenzene (III), and 1-chloro-3,5-diamino-2,4,6-trinitrobenzene (IV)(2). The spectral data of TATB, (I), (III), and (IV) in various solvents are given in Figures 1-3. (II), which reacts with concentrated sulfuric acid to give ammonium ion and hydrogen chloride gas, of course, shows no absorption in the region of TATB absorption.

The spectrum of TATB in concentrated sulfuric acid does not change in three hours, but does change either on heating or standing overnight. Occasionally the spectrum of a solution was found to be decreasing with time, but this was found to be due to faulty cleaning of a flask from a previous determination. The presence of as much as 10 volume per cent of water did not affect the spectrum of TATB in concentrated sulfuric acid in the region of its absorption. There was some increase in absorption at higher wavelengths.

The results of several analyses using concentrated sulfuric acid as solvent are given in Table I.

Water Soluble Impurities

(II) and any other water soluble impurities may be readily determined by dissolving the sample of TATB in concentrated sulfuric acid, then precipitating by adding water. When (II) is present, gaseous hydrogen chloride is evolved when the sample is dissolved in the sulfuric acid, as indicated by its odor, and may be further identified by absorption in water, followed by either titration of the acidity or chloride ion, or both. The ammonium ion in the filtrate may also be determined. Some results using this procedure are given in Table I.

A spectral analysis of the recovered material showed that the TATB was unchanged.

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Chlorine Determination and Vacuum Stability

The total chlorine content of the TATB samples was determined by a Parr Bomb fusion (6) and Volhard titration (7). Analytical results are shown in Table I for samples both before and after removal of water soluble impurities.

Also in Table I are the standard vacuum stability results obtained at 260°C (9). A pure sample of (I) by this test gives gas off at less than 0.1 cc/g/hr and (II) gives essentially no gas. However, (III) and (IV) decompose, as evidenced by the fact that they give off greater than 30 cc/g in 8 minutes and 15 minutes, respectively.

DISCUSSION AND CONCLUSIONS

From Figure (1), for TATB, the value of the ratio of the 325 mμ peak to the 272 mμ peak in sulfuric acid, R, is 1.95 ± 0.02 , which can be used as a guide for estimating the amount of absorbing impurities. As little as 5% of (III) or (IV) give a ratio of 1.91 and 1.89, respectively, whereas a value greater than 1.97 indicates that an impurity other than the predecessors is present. The preparations in Table I had R values near 1.95, except sample 4970-80, crude, which was 1.91 ± 0.04 .

The R value for sample 4970-80 indicates that (III) or (IV), or both, may be present in this sample. The low chlorine value rules out the presence of much (II), while the vacuum stability result tends to bear out the presence of (III) or (IV), as also does the slight improvement in purity by treatment with organic solvents. In fact, (III) and (IV), and probably (I) was shown to be present by analysing the gamma-butyrolactone filtrate (See experimental).

The difficulty in removing water soluble impurities from TATB is shown by the data for sample 4969-145. That the major impurity is (II) was shown by analysing the filtrate from the water soluble determination for ammonium ion. There was found an amount calculated as (II) equivalent to 9.47% of the TATB sample. Likewise, with sample 4993-91 there was found ammonium ion calculated as (II) equivalent to 3.5% of the TATB sample.

By extracting with acetonitrile, there was removed from sample 4993-91 about 0.3-4% of material, which when analyzed in sulfuric acid was shown to be 29% TATB and 22% (IV). The remainder of this material was either (I) or (II), or both, as

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TABLE I
ANALYSIS OF TATB IN CONCENTRATED SULFURIC ACID

Sample	Comments	% TATB	% Water Soluble	% Cl	Vacuum Stability cc/g/hr at 260°
PREPARED IN BENZENE					
4969-106	2nd crop, crude	91.6	----	2.6	4.73
	2nd crop, benzene washed	93.6	----	2.4	4.30
4969-145	crude	----	9.36	6.8	----
	portion of crude washed with water containing Tide	----	----	6.3	----
	portion of crude washed with water containing Tween-60	----	----	6.4	----
	portion of crude washed with 6 N sulfuric acid	----	----	6.5	----
	water soluble removed /a	----	----	0.44	----
4970-143	crude	87.9	----	3.4	----
4993-91	crude washed with ethyl acetate	89.8	4.31	3.81	6.98
	water soluble removed /a	93.9	----	0.86	----
PREPARED IN DIOXANE					
4969-159A	crude, commercial dioxane	----	----	0.18	9.2
4969-159B	crude from re-used dioxane	----	----	0.12	7.8
4970-80	crude, commercial dioxane	93.6	----	0.28	5.2
	recrystallized from gamma-butyrolactone	96.9	----	----	----
	washed with benzene	98.0	----	----	----
	recrystallized twice from gamma-butyrolactone	97.4	----	----	----
4970-82	crude, commercial dioxane	98.2	----	----	3.6
	washed with benzene	98.4	----	----	----
	recrystallized from gamma-butyrolactone after benzene wash	98.0	-----	----	----
	refluxed with acetonitrile	96.7	----	----	----
	residue dimethylformamide extr.	99.8	----	----	/b
4970-115	crude from re-used dioxane	97.1	----	----	4.5
	washed with benzene	97.2	----	----	----
	residue from dimethylformamide	99.2	----	----	/b
4970-137	crude from purified dioxane	96.3	----	----	2.0
	water soluble removed /a	96.6			

/a Sample dissolved in concentrated (96%) sulfuric acid and precipitated by adding water.

/b After washing the residue five times with dimethylformamide, the product had a vacuum stability of 0.7 cc/g/hr at 260°C. Such a material had the following elemental analysis(5): % calcd., C = 27.92, H = 2.34; % found, C = 27.97, H = 2.32

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it was non-absorbing. From the R value, there would not be much of (IV) in 4993-91.

Generally, TATB prepared in dioxane had a better purity than that prepared in benzene. The assay after the water soluble determination (Table 1) for sample 4970-137 shows that there is little (II) present, while the vacuum stability shows that there is little (III) or (IV). Therefore, the probable impurity here is (I).

Pure TATB may be prepared by dimethylformamide extraction of the impurities as shown by the data for samples 4970-82 and 4970-115. A direct spectral comparison of sulfuric acid solutions of this material with TATB recrystallized from nitrobenzene showed them to be identical.

The data show that in both benzene and dioxane there may be incomplete amination and, particularly in benzene, there is considerable occlusion of ammonium chloride in the product.

The presence of (III) or (IV) is definitely indicated when the vacuum stability value is greater than 5 cc/g/hr. at 260°C. These impurities may be determined by a spectral analysis of the residue from a dimethylformamide extraction.

Ammonium chloride as an impurity may be determined by the water soluble procedure described here, provided it is present to at least 1%. Estimates of amounts less than this may be made by determining the ammonium ion in the filtrate from this procedure. The amount of hydrochloric acid evolved when the sample is dissolved in concentrated sulfuric acid may also be determined by bubbling it through a basic solution and titrating the excess base or the chloride ion, or both. Simmons (10) has been successful in recovering about 90% of the chloride ion from known ammonium chloride samples by this procedure.

EXPERIMENTAL

Equipment

All volumetric glassware was calibrated.

Spectrophotometric measurements were made using 1 cm cells in both the Cary Model 14 and Beckman Model DU spectrophotometers.

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Reagents

CP or Reagent grade sulfuric acid (96%, sp.gr. 1.84) was used as received. The organic solvents were re-distilled before use.

Molar Absorbancy Index

During the course of this investigation, two DU spectrophotometers were used in addition to the Cary. The molar absorbancy index was determined on all of the machines by the analytical procedure below, using TATB recrystallized from nitrobenzene and the data are summarized in Table II.

TABLE II
MOLAR ABSORBANCY OF TATB IN SULFURIC ACID

Spectrophotometer	$\epsilon \times 10^4 \text{ } \underline{a}$				
	325 m μ	No. of Detns.	272 m μ	No. of Detns.	Ratio
DU(I)	2.56 $\pm$ 0.04	5	1.32 $\pm$ 0.03	4	1.94
DU(II)	2.43 $\pm$ 0.03	5	1.25 $\pm$ 0.02	5	1.94
Cary	2.48 $\pm$ 0.03	2	1.26 $\pm$ 0.02	2	1.97

a Arithmetical average and average deviation

Analytical Procedures

Assay

Weigh accurately 2-7 mg (2-20 mg for the Cary) of TATB on a microbalance and dissolve in concentrated sulfuric acid in a 100 ml volumetric flask. After diluting to volume and mixing, pipette a 5 ml sample of this solution into a 50 ml volumetric flask, dilute to volume with concentrated sulfuric acid and mix thoroughly. The solution should be allowed to stand 5-10 minutes to remove any trapped air. This solution is $(0.8-2.7) \times 10^{-5}$ M $(0.8-7.8) \times 10^{-5}$ M for the Cary) and is used for the

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spectral absorption determination. Read the absorbancy at 272 mμ and 325 mμ. Calculate the percent TATB as follows:

$$\% \text{ TATB} = \frac{A (258) (100)}{\epsilon (\text{g sample})}$$

where A is the absorbancy and ϵ is the molar absorbancy index, both at 325 mμ.

Calculate the ratio, A_{325}/A_{272} , and note whether it is less than 1.93. The "% TATB" calculated as above is correct to +1% if the ratio is not less than 1.93. If the ratio is too small, the "% TATB" is in error, but represents a maximum value. If the ratio is greater than 1.97, the presence of an impurity other than the predecessors is indicated.

Total Chlorine - Determine by a Parr bomb fusion (6) and Volhard titration (7).

Water Soluble Impurities

If the total chlorine content is 0.3% , or less, this determination is unnecessary in most instances, as the content of (II) can not be greater than 0.45% and is usually less.

Weigh accurately a 0.2 g sample into a 250 ml beaker and dissolve in 2 ml (Note 1) of concentrated sulfuric acid with swirling. Do not heat. After 5 minutes, add 200 ml of water all at once with stirring. Allow the precipitate to settle for 10 minutes or longer, and filter onto a previously weighed, sintered-glass crucible (medium porosity). Wash thoroughly with 10 ml portions of water (about 100 ml total), dry at 100°C for 2 hours, and weigh. The loss in weight is (II).

$$\% (II) = \frac{\text{loss in weight} (100)}{\text{weight of sample}}$$

Note 1: The solubility of TATB is approximately 0.1 g/ml.

Measure to the nearest 0.1 ml. This procedure was checked on pure TATB, giving a recovery of 99.0% and 100.2% on a 10 mg sample and a 40 mg sample, respectively.

Ammonium Ion

To the combined filtrate and washings from the above determination, add 2.8 g (97% of calculated) of solid sodium hydroxide and dissolve. Add a few drops of 0.1% Methyl Red

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indicator (in ethanol) and neutralize with 0.1 N sodium hydroxide.

To 50 ml of 37% formalin, add 50 ml of water and a few drops of 1% phenolphthalein indicator (in ethanol), and neutralize with 0.1 N sodium hydroxide. Add this solution to the above neutralized solution and allow to stand at least one minute. Titrate with 0.1 N sodium hydroxide to the pink of phenolphthalein. Calculate the percent (II) as follows:

$$\% (II) = \frac{(\text{ml NaOH})(N \text{ NaOH})(53.5)(100)}{\text{mg sample}}$$

where 53.5 is the meq weight of (II).

Absorption Spectra

Generally, absorption spectra were measured on solutions which gave absorbancy readings near 0.5 at the maxima. Heat was necessary for either solution of the samples or stability of the absorbancy readings in all solvents except concentrated sulfuric acid.

Stability of TATB Spectrum in Sulfuric Acid

Occasionally, the spectrum of a solution of TATB in concentrated sulfuric acid was found to be decreasing with time. It was thought that perhaps water was causing this, so the effect of added water was determined. A solution of TATB in concentrated sulfuric acid was prepared as usual and 5 ml aliquots were diluted to 50 ml with (1) concentrated sulfuric acid, (2) concentrated sulfuric acid plus 1 ml water and (3) concentrated sulfuric acid plus 5 ml water. In (1) and (2) the total spectrum was the same. In (3) the spectrum below 340 mμ was the same, but there was an increase in absorbancy at wavelengths greater than 340 mμ. This increase was greatest around 380 mμ where it amounted to 70%. Since the assay was based on the absorbancy at 325 mμ, there was no interference from water in the amounts given above.

As stated previously, the spectrum of TATB in concentrated sulfuric acid does not change in three hours, but does change either on heating or standing overnight. After two days at room temperature, the absorption at 325 mμ decreased 15% and the

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maximum shifted to 320 mμ. This particular solution was heated to 130°C, cooled, and the spectrum redetermined. The maximum was then at 315 mμ and the absorption at 325 mμ had decreased by 36%. There was no maximum at 272 mμ, and there was a minimum at 255 mμ.

Analysis of Sample 4993-91

That (IV) was present in this sample was shown as follows. A five gram sample was extracted in a Soxhlet extractor with acetonitrile for several days. The solvent was evaporated, giving 15-20 mg of a yellow-orange solid. The solid was taken up in concentrated sulfuric acid and the spectrum determined. The experimentally determined curve was well reproduced from 230 mμ to 400 mμ by a calculated curve assuming 29% TATB and 22% (IV). The other 49% of the extract was either (II) or (I) or both, as it was non-absorbing. This does not mean that (III) was absent in sample 4993-91.

Two independent check analyses were run on this sample. The spectrophotometric analysis in gamma-butyrolactone gave 90.3% as the purity. The other check consisted of dissolving a sample in concentrated sulfuric acid, precipitating by adding water, washing with water, and drying without removing the sample from the original container. Nitrobenzene was then added and the mixture alternately boiled and cooled until only crystalline material remained. Acetone was then added and the solution was filtered. After washing with three 5 ml portions of acetone, the precipitate was dried at 100°C for 3 hours and weighed. On two such determinations, there was recovered 89.9±1.2% of the original sample by weight. Insufficient nitrobenzene was added to dissolve all of the sample at the boiling point in order that as little TATB be dissolved as possible. As (I), (III), and (IV) are soluble in acetone and TATB is not, acetone was added to the nitrobenzene before filtering.

Analysis of Samples 4970-80 and 4970-137

The impurities present in Sample 4970-80 were identified by a spectral analysis of the gamma-butyrolactone filtrate from the recrystallization. Using Figure (2), (IV) and TATB plus (III) were determined using 410 mμ and 440 mμ absorbancies. After subtracting the absorbancy due to (IV), TATB and (III) were determined using the 353 mμ and 410 mμ absorbancies. Finally, (I) was estimated at the wavelengths below 280 mμ after subtracting for the other three. These results are given

in Table III.

TABLE III
COMPOSITION OF ~~gamma~~-BUTYROLACTONE FILTRATE FROM
RECRYSTALLIZATION OF TATB (4970-80)

<u>Compound</u>	<u>Molarity</u>	<u>Mole %</u>
TATB	6.6×10^{-4}	30
IV	8.8×10^{-4}	40
III	5.4×10^{-4}	25
I	1×10^{-4}	5

From the dioxane from the preparation of Sample 4970-137, Table I, was obtained a white solid, which was essentially insoluble in concentrated sulfuric acid and water. This solid did dissolve in gamma-butyrolactone and gave a spectrum which showed it to be mostly (I).

Acknowledgement

The vacuum stability data were obtained by Mr. Herbert T. Simmons of this Laboratory.

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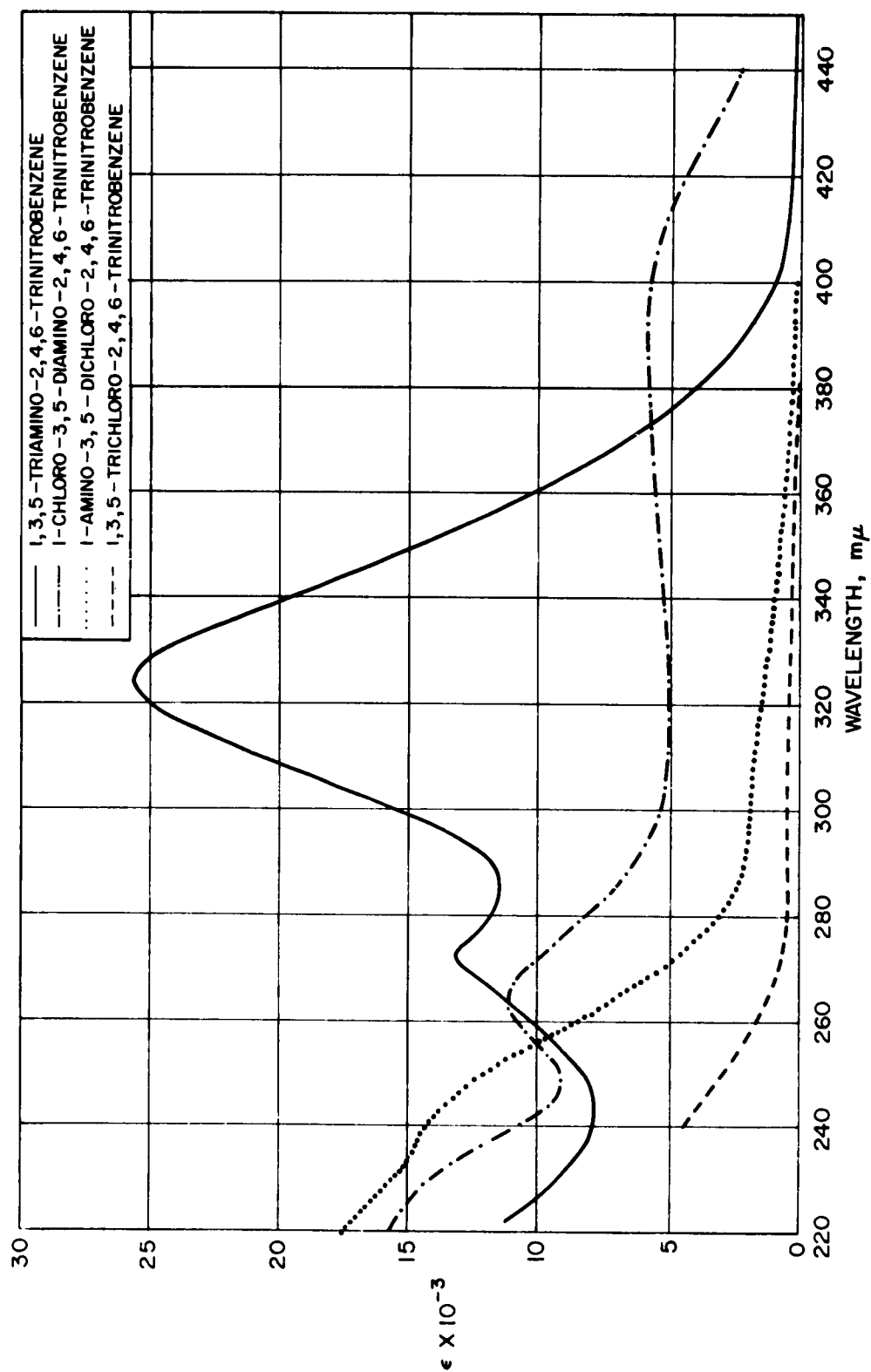


FIG. 1 SPECTRA IN CONCENTRATED SULFURIC ACID

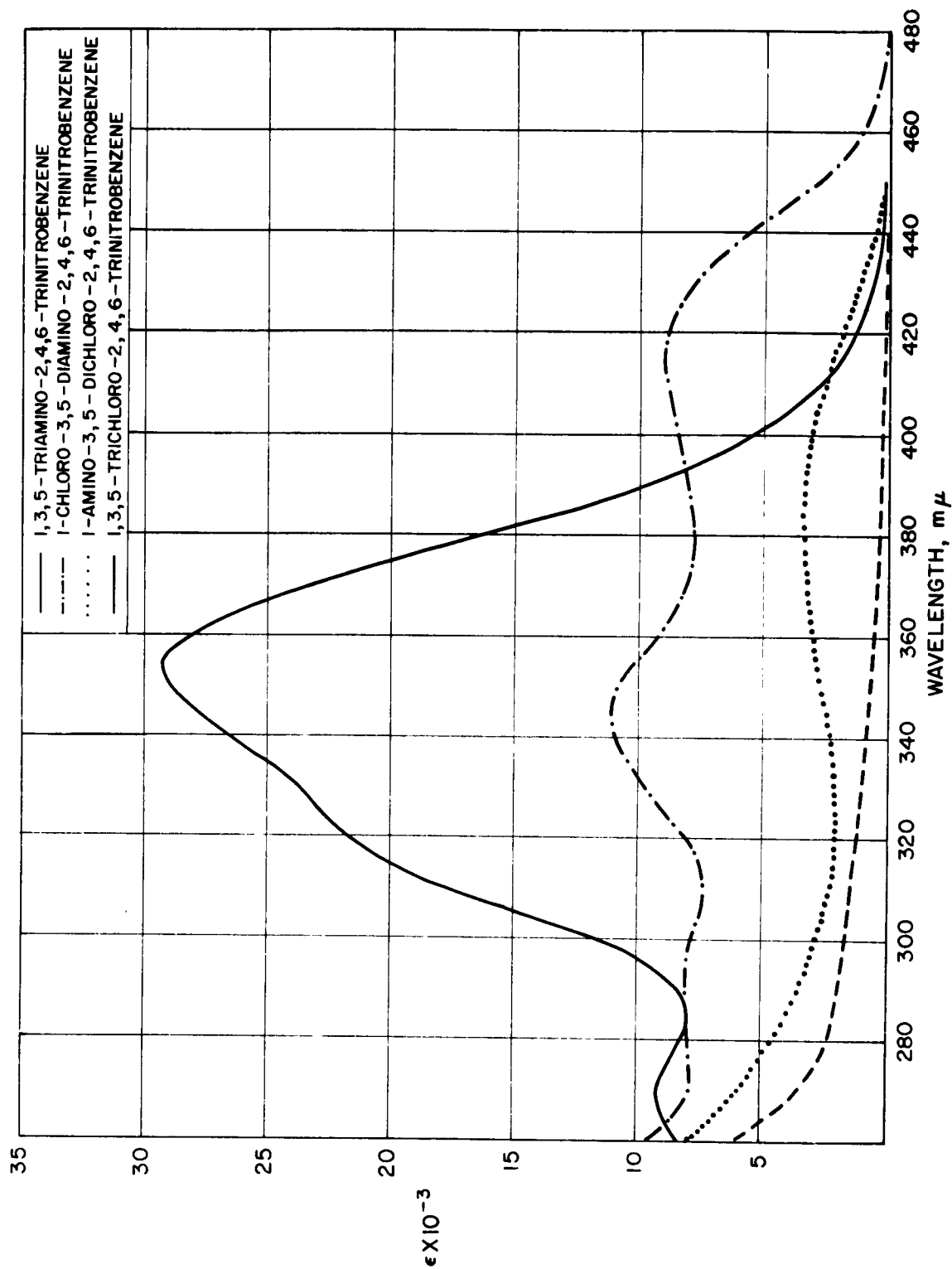


FIG. 2 SPECTRA IN γ -BUTYROLACTONE (AFTER HEATING)

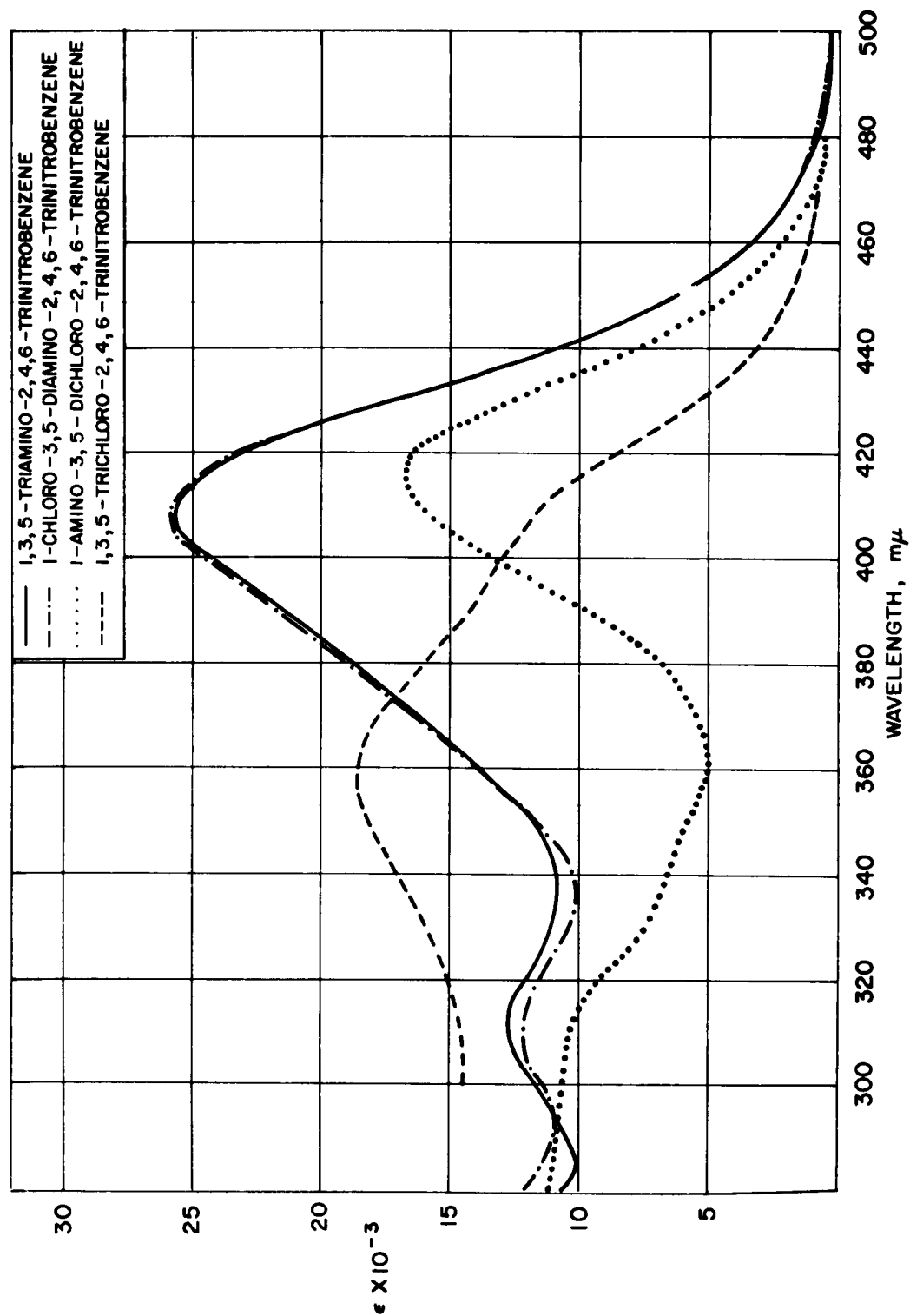


FIG. 3 SPECTRA IN ETHYLENEDIAMINE (AFTER HEATING)

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Attn: Dr. J. R. Fischer (Contract Nonr-2655(00)).....	1
Aerojet-General Corporation, P. O. Box 1947, Sacramento, California	
Attn: Dr. Marvin H. Gold	1
American Cyanamid Company, P. O. Box 270, New Castle, Pa.	
Attn: L. V. Clark, Director (Contract NOW-60-0745-c) Explosives Res. and Development	1
Minnesota Mining and Manufacturing Company 2301 Hudson Rd., St. Paul 19, Minn. (ARPA Proj. 201-2)	
Attn: Dr. Morton H. Friedman, Central Res. Dept.	1
Thiokol Chemical Corporation, Reaction Motors Division Denville, New Jersey (Contract Nonr 3664(00))	
Attn: Dr. D. D. Perry	1
Commander, Naval Ordnance Test Station, China Lake, Calif.	
Attn: Chemistry Research Section	1
Dr. Martin H. Kaufman (Code 4544)	1
Director, Naval Research Laboratory, Washington 25, D.C.	
Attn: Chemistry Division	1

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Descriptors	Codes	Security Classification and Code Count	Descriptors	Codes
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		<u>Bibliographic (Suppl., Vol., etc.)</u>		

SUBJECT ANALYSIS OF REPORT

Descriptors	Codes	Descriptors	Codes
<u>Explosives</u>	<u>EXPL</u>	<u>Acid</u>	<u>ACID</u>
<u>Heat-resistant</u>	<u>HEAA</u>	<u>Impurities</u>	<u>IMPU</u>
<u>Spectrophotometry</u>	<u>SPEO</u>	<u>Preursors</u>	<u>PCRR</u>
<u>Quantitative</u>	<u>QUIT</u>	<u>Qualitative</u>	<u>QUAL</u>
<u>Analysis</u>	<u>ANAL</u>	<u>Gamma</u>	<u>GAMM</u>
<u>Tri</u>	<u>TRIY</u>	<u>Butyro</u>	<u>BUYR</u>
<u>Amino</u>	<u>AMIN</u>	<u>Lactone</u>	<u>LACO</u>
<u>Trinitro</u>	<u>TRIT</u>	<u>Dimethyl</u>	<u>DMT</u>
<u>Benzene</u>	<u>BENZ</u>	<u>Formic</u>	<u>FORI</u>
<u>Assay</u>	<u>ASAY</u>	<u>Amide</u>	<u>AMID</u>
<u>Method</u>	<u>METD</u>	<u>Amination</u>	<u>AMNA</u>
<u>Sulfuric</u>	<u>SULF</u>	<u>Dioxane</u>	<u>DION</u>

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Naval Ordnance Laboratory, White Oak, Md.
(NOL technical report 62-76)
HEAT RESISTANT EXPLOSIVES XII, QUANTITATIVE
ANALYSIS OF 1,3,5-TRIAMINO-2,4,6-TRINITRO-
BENZENE (C), by Donald J. Glover. 15 May
1962. 10p. charts, tables. Tasks FR-44 and
RUME 3E012.
CONFIDENTIAL

A spectrophotometric assay method using
concentrated sulfuric acid as the solvent
is presented for 1,3,5-triamino-2,4,6-trini-
trobenzene. When purity is 90 percent or
better there is no interference from any
precursors.

In addition, a procedure is given for de-
termining water soluble impurities. Qualita-
tive identification of precursors is afford-
ed by spectral determination in gamma-buty-
rolactone or dimethylformamide.
Quality of 1,3,5-triamino-2,4,6-trinitro-
benzene, prepared by amination in benzene
and dioxane, is discussed.

1. Explosives, Heat resis-
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