Test Report T70-10-1

SPECTROPHOTOMETRIC DETERMINATION OF SULFATE IN PROPELLANTS AND NITROCELLULOSE

by

George Norwitz

August 1970

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DEPARTMENT OF THE ARMY
FRANKFORD ARSENAL
Philadelphia, Pa. 19137

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Quality Assurance Directorate
FRANKFORD ARSENAL
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I. SUMMARY

An accurate method is proposed for the determination of sulfate in propellants and nitrocellulose that requires only 45 minutes. The sample is treated with a mixture of nitric acid, perchloric acid and magnesium nitrate, and the solution is evaporated to dryness over a Bunsen burner. The salts are heated for 5 minutes with a mixture of hydriodic, hypophosphorous and hydrochloric acids in a simplified distillation apparatus while passing nitrogen through the system, the hydrogen sulfide which results from the reduction of sulfate is absorbed into ammonium hydroxide, lead citrate is added, and the brownish-yellow lead sulfide color is measured.

II. RECOMMENDATIONS

It is recommended that the method described in this report be inserted into the military specification for propellants (MIL-STD-286B).

III. INTRODUCTION

Sodium sulfate and potassium sulfate are frequently added to propellants in amounts up to 2%. Sulfate is found in nitrocellulose as adsorbed sulfuric acid or cellulose sulfate ester; unstabilized nitrocellulose contains up to 1% sulfate (calculated as H_2SO_4) while stabilized (purified) nitrocellulose usually contains less than 0.05%.

Sulfate in propellants has been determined by precipitation as barium sulfate after oxidizing the organic matter by treatment with either a mixture of nitric acid and perchloric acid (11) or a mixture of nitric acid and sodium chlorate (2). Frequently, the cation of the sulfate salt is determined and calculation made to the sulfate salt (13).

The above methods for the determination of sulfate in propellants have proved to be troublesome; therefore, this laboratory undertook the development of a spectrophotometric method depending on the reduction of sulfate to hydrogen sulfide by a mixture of hydriodic and hypophosphorous acids.

IV. STUDY

A. DEVELOPMENT OF SPECTROPHOTOMETRIC METHOD

Principle of Method and Prior Work

St. Lorant (16, 17, 18) showed that sulfate could be determined by reduction to hydrogen sulfide by use of a mixture of hydriodic acid and red phosphorus. He collected the hydrogen sulfide in zinc acetate and determined the sulfide by methylene blue. Roth (15) modified St. Lorant's method somewhat. Johnson and Nishita (3) determined sulfate in plant material by use of the hydriodic acid-red phosphorus mixture but finished the determination iodometrically.

Luke (8, 9) proposed the use of a mixture of hydriodic and hypophosphorous acids in place of the hydriodic acid and red phosphorus. He collected the hydrogen sulfide in cadmium solution and titrated with iodate. In applying the method to the determination of sulfate in rubber he conducted a preliminary evaporation to fumes of perchloric acid to destroy organic matter. Luke (6) determined sulfur in metals by dissolving with hydrochloric and nitric acids, destroying the nitrate with formic acid, adding the hydriodic-hypophosphorous acid reagent, collecting the hydrogen sulfide in ammonium hydroxide, adding lead citrate, and measuring the brownish-yellow color. Luke (7) used the hydriodic-hypophosphorous acid reagent and a titrimetric finish for the determination of sulfur in steels. Bethge (1) in determining sulfate in organic compounds, evaporated to fumes of perchloric acid,added the hydriodic-hypophosphorous acid reagent, collected the hydrogen sulfide in sodium hydroxide solution, and titrated with iodate. Pepkowitz (14) used a somewhat similar method but collected the hydrogen sulfide in ammoniacal cadmium solution. Pepkowitz and Shirley (12) developed a method for the detection of sulfur by combustion with copper oxide, treatment with the hydriodic-hypophosphorous acid mixture, and reduction of a molybdenum-thiocyanate reagent. Keattch (4) and McKinley and Jones (10) used the hydriodic-hypophosphorous acid reagent and an iodometric titration for the determination of sulfate in chrome plating baths. Keattch (5) applied the same technique to the determination of sulfate in soils. Steinbergs, Iismaa, Freney, and Barrow (19) determined sulfur in soil and plant material by igniting with a mixture of sodium bicarbonate and silver oxide for 3 hours, adding a hydriodic-hypophosphorous-formic acid reagent, and using the methylene blue spectrophotometric method. Vandael (20) determined sulfate in barium sulfate by refluxing for 1 hour with the hydriodic-hypophosphorous acid reagent. The same investigator considered the chemistry of the reaction and concluded that hydriodic acid and hypophosphorous acid both participate in the reduction of the sulfate. Previously it had apparently been believed that hydriodic acid was the active reducing agent and that the hypophosphorous acid merely served the purpose of reducing the iodine formed by the reaction between the sulfate and hydriodic acid.

The present investigator used the hydriodic-hypophosphorous acid reagent since that reagent is more easily prepared than the hydriodic-red phosphorous reagent. The spectrophotometric technique employing the lead sulfide color was used.

The various factors affecting the reduction and the spectrophotometric procedure were investigated, using samples prepared by adding 5 drops of approximately 0.1N sodium hydroxide solution to portions of standard potassium sulfate solution and evaporating to dryness.

Preparation of Reagent

Some hydrochloric acid was mixed with the hydriodic and hypophosphorous acids as has been suggested by several investigators. The purpose of the hydrochloric acid is to provide high acidity and permit the use of less hydriodic acid, a chemical that is somewhat expensive. In the preparation of the hydriodic-hypophosphorous-hydrochloric acid reagent, the mixture is boiled for 5 minutes with glass beads in an Erlenmeyer flask (there is no significant loss of volume during the boiling). The reagent is stable for at least 6 months and need not be stored in a dark bottle or refrigerator. Fifteen milliliters of the reagent, containing 8.6 ml of hydriodic acid, 2.1 ml of hypophosphorous acid, and 4.3 ml of hydrochloric acid, were used per determination.

Apparatus

The apparatus is shown in Figure 1. This simple apparatus is somewhat similar to that used by Luke (6) except that a 50-ml round bottom flask is used (in place of an Erlenmeyer flask) to obtain more rapid flushing and better heating control (using a flame). A 100-ml volumetric flask (instead of a 25-ml volumetric flask suggested by Luke) is used as the receiving flask, since a larger volume of ammonium hydroxide was employed to absorb the larger amount of hydrogen sulfide distilled. The volumetric flask is cooled in ice contained in a beaker. The beaker is covered with asbestos cloth to insulate it from the heat of the Bunsen burner.

Manner of Heating

The manner of heating the solution during the distillation that was most effective was to heat with a Bunsen burner to an incipient boil and then maintain at or just below an incipient boil by brushing the flame of the Bunsen burner back and forth across the bottom of the flask. The temperature of the solution remains at approximately 120°C. Vigorous boiling is unsatisfactory because it drives over too much hydriodic and hydrochloric acids, causing heating of the ammonium hydroxide by the neutralization reaction. Luke (6) conducted the distillation by use of an electric hot plate which was regulated so that the the temperature of silicone oil placed in a container alongside of the distillation flask was 185° ±3°C. It is difficult to control heating conditions using an electric hot plate.

Heating Time

The total heating time in the method was only 5 minutes; longer heating than this did not give a higher result. Luke (6) heated the solution for 5 minutes after the appearance of white fumes in the neck of the volumetric flask. Other investigators heated the solution from 30 to 60 minutes. These investigators, however, frequently worked with fairly large amounts of sulfate. The present author distilled up to 0.25 mg of sulfur (Luke distilled up to 0.05 mg (6)).

Flow of Inert Gas

The optimum flow of inert gas was found to be 55 to 65 bubbles per 15 seconds as determined by inserting the exit tube of the adapter into a volumetric flask containing 90 ml of water at room temperature. For the control of the flow rate,it is essential that a needle valve be used. If the regulator used with the tank does not include a satisfactory needle valve, it is necessary to insert a needle valve into the line. Luke (6) regulated the flow so that 100 ml of water was displaced from an inverted graduated cylinder in 30 ±1 seconds. This manner of controlling the flow rate is troublesome. The flow rate as determined in the method of the present author (on the basis of ml per minute) was approximately half of that used by Luke.

It is not necessary to flush out the flask before heating with the hydriodic-hypophosphorous acid reagent, since the small amount of air that remains when the hydrogen sulfide starts to distill does not affect the result (by oxidation). When a flow of air was substituted for the nitrogen, a surprising 80% recovery of the sulfur was obtained. It is not necessary to purify the nitrogen.

Measurement of the Color

The spectrophotometric curve for the color from 370 to 600 millimicrons is shown in Figure 2. Maximum absorbance in this range occurs at 370 millimicrons. It is recommended, however, that the color be measured at 400 millimicrons since better reproducibility was obtained at this wave length. A study made of the stability of the color showed that it developed immediately and was stable for 15 minutes. It then slowly decreased in intensity. It is recommended that the color be read within 5 minutes.

Blank

As recommended by Luke (6) the color is measured against distilled water and a correction made for the blank (which is also measured against distilled water). The blank carried through the entire procedure (including the evaporation to dryness with the nitric acid-perchloric acid-magnesium nitrate reagent) as measured against distilled water was about 95% and it did not vary significantly.

The blank is caused by the absorbance of the ammonium hydroxide-lead citrate solution and traces of sulfur in the reagents. The hydriodic-hypophosphorous-hydrochloric acid reagent is purified in its preparation. The nitric acid, perchloric acid, and magnesium nitrate used in the method are ordinary reagent grade materials. The fact that the blank was low and reproducible would indicate that no significant amount of sulfur was picked up from the Bunsen burner.

Effect of Water

To test the effect of water on the distillation, various amounts of water were added to evaporated portions of the potassium sulfate solution (0.20 mg of sulfur) and the distillations conducted using 15 ml of the hydriodic-hypophosphorous acid reagent. The results (Table I) show that up to 5 ml of water can be present.

Preparation of the Sample

Having established optimum conditions for the reduction of sulfate, attention was then turned to means for preparing the sample. This was accomplished by treating with a reagent containing nitric acid, perchloric acid, and magnesium nitrate, evaporating to dryness over a Bunsen burner and then heating for 2 more minutes.

The nitric and perchloric acids destroy the organic matter and the fuming to dryness with the perchloric acid eliminates the nitrate. The purpose of the 2 minute heating period after the evaporation to dryness is to drive off the free perchloric acid; it was found that better results were obtained when no free perchloric acid was present.

The magnesium nitrate in the nitric acid-perchloric acid-magnesium nitrate reagent ties up the sulfate as magnesium sulfate so that there are no losses of sulfate during the heating to dryness. When the addition of the magnesium nitrate was omitted, low results were obtained. When sodium nitrate was substituted for the magnesium nitrate, the results were not as satisfactory.

Up to a 0.4-gram sample of propellant or nitrocellulose can be handled in the method.

Speed of the Method

The method is rapid. A sample can be completely analyzed in 45 minutes. This is in contrast with the time-consuming gravimetric method which requires two filtrations, an overnight standing, and weighing of crucibles.

Calibration Curves

Three calibration curves were prepared.

The first curve was made by treating aliquots of standard potassium sulfate solution with 5 drops of approximately 0.1N sodium hydroxide solution, evaporating to dryness, and proceeding with the distillation.

The second curve was prepared by treating aliquots of standard potassium sulfate solution with the nitric acid-perchloric acid-magnesium nitrate reagent, evaporating to dryness, and proceeding with the distillation.

The third curve was prepared by treating aliquots of standard sodium sulfide solution with ammonium hydroxide and lead citrate. The difficulty in this method was the preparation of the standard sodium sulfide solution. The common method of preparing standard sodium sulfide solution is to dissolve hydrated sodium sulfide (which has the approximate formula $Na_2S \cdot 9H_2O$) in water, standardize a portion with iodine, and use the remainder of the solution immediately for the purpose at hand. The present author proceeded by first analyzing a sample of fused, flake sodium sulfide (a technical material, Fisher Scientific Co., containing approximately 60% sodium sulfide, the balance being mainly water). The analysis was conducted by dissolving 0.20 to 0.25-gram portions in 30 ml of oxygen-free water in glass stoppered flasks, immediately adding 50.00 ml of 0.1N iodine solution and 2 ml of hydrochloric acid, and titrating with 0.1 N sodium thiosulfate solution, using starch indicator. The assay was found to be 24.47% $S^=$ (59.55% Na_2S). A catch weight (0.0487 gram) of the material was dissolved in water and the solution was diluted to 2 liters in a volumetric flask (the solution contained 0.00596 mg of sulfur per ml). Then, 10, 20, 30, and 40 ml of this solution were pipetted into 100-ml volumetric flasks and the solutions diluted to approximately 60 ml with water. Thirty milliliters of ammonium hydroxide and 5 ml of lead citrate solution were added, the volume was brought up to 100 ml, and the transmittance was measured.

The three curves were straight lines and were identical, a fact that helped to establish the validity of the method. The first method of preparing the curve is recommended because it is the simplest.

Interferences

No constituent found in ordinary propellants or in nitrocellulose interferes with the method. Cellulose esters in nitrocellulose would be readily hydrolyzed to inorganic sulfate by nitric acid. As would be expected from the work of Luke (6), metals such as copper, iron, lead, and tin do not interfere. The tin partially distills but does not interfere because tin sulfide is soluble in strong ammonium hydroxide solution (see Table III). Ammonium salts found in some propellants (ammonium nitrate and ammonium perchlorate) are destroyed during the evaporation and heating.

Barium sulfate does not dissolve readily in the hydriodic-hypophosphorous acid reagent and cannot be analyzed by the method. Fortunately, barium sulfate is not found in propellants or nitrocellulose.

Sulfides, sulfites, thiosulfates, elemental sulfur, and organic sulfur compounds would interfere; however, these compounds are not found in nitrocellulose or ordinary propellants.

Absorption of Sulfide into Sodium Hydroxide Solution Instead of Ammonium Hydroxide

Some experiments were conducted using sodium hydroxide solution (25 ml of 10% solution diluted to 90 ml with water in the volumetric flask) in place of the ammonium hydroxide solution. The advantage of the sodium hydroxide solution is that it will hold the sulfide quantitatively even when warm, consequently the cooling is unnecessary. The results obtained using sodium hydroxide were not as reproducible as those obtained with ammonium hydroxide. Also, the color was not as stable and did not quite follow Beer's law. In view of this, the use of ammonium hydroxide is recommended.

The author also investigated the use of the methylene blue method after absorbing the hydrogen sulfide into zinc acetate solution. The results were not as satisfactory as with the lead sulfide spectrophotometric method.

B. RECOMMENDED SPECTROPHOTOMETRIC METHOD

Apparatus

The apparatus (Figure 1) consists of a 50-ml round bottom flask with a 19/38 ground glass joint, an adapter, a 100-ml volumetric flask, and a 1-liter beaker surrounded by asbestos cloth. All glass is Pyrex. The diameter of the tubing is 0.6 mm (internal). The 50-mm round bottom flask is held by a clamp about 1 foot above the table. The inlet tube of the adapter is connected to a tank of nitrogen by Tygon tubing. The nitrogen must be controlled by a needle valve. If the regulator does not have a satisfactory needle valve, insert a needle valve in the system (Lab-Crest Needle Valve, 1 1/4 mm, Fischer and Porter, Warminster, Pa.).

Reagents

Standard potassium sulfate solution (1 ml = 0.02 mg of S). Dissolve 0.1088 gram of potassium sulfate (previously dried at 120°C) in water and dilute to 1 liter in a volumetric flask.

Hydriodic-hypophosphorous-hydrochloric acid reagent. Transfer 200 ml of hydriodic acid (57%), 50 ml of hypophosphorous acid (50%), and 100 ml of hydrochloric acid to a 500-ml Erlenmeyer flask (ground glass stopper). Add several glass beads, heat to boiling without a cover, boil 5 minutes, insert the stopper, and allow to cool to room temperature.

Lead citrate reagent. Dissolve 10 grams of lead nitrate in 200 ml of water. Add 40 grams of citric acid monohydrate and stir with a stirring rod to dissolve. If salts settle out on standing overnight, use the supernatant liquid.

Nitric acid-perchloric acid-magnesium nitrate reagent. Mix 300 ml of nitric acid, 100 ml of perchloric acid (70%), and 50 ml of water. Add 20 grams of $Mg(NO_3)_2 \cdot 6H_2O$ and stir to dissolve. The small amount of water is necessary because magnesium nitrate is not too soluble in concentrated acids.

Preparation of Calibration Curve

Pipet 0.2, 2, 5, 10, and 12.5-ml aliquots of standard sulfate solution (1 ml = 0.02 mg of S) into 50-ml round bottom flasks (with 19/38 ground joints). Carry along a blank. Add 5 drops of approximately 0.1N sodium hydroxide solution and evaporate just to dryness by some suitable means. Allow to cool to room temperature.

Attach the adapter of the apparatus to an empty 50-ml round bottom flask and insert the exit tube into a 100-ml volumetric flask containing approximately 90 ml of water. Connect up the nitrogen and regulate the flow to 55 to 65 bubbles per 15 seconds.

Add 30 ml of ammonium hydroxide to six 100-ml volumetric flasks and dilute to approximately 90 ml with water. Place the flasks into cracked ice contained in a tray. After a few minutes place one of these flasks into the asbestos-covered beaker and fill the beaker to the top with cracked ice.

With the nitrogen flowing, insert the adapter into the flask containing the sample and insert the exit tube into the volumetric flask contained in the beaker. Take care that the apparatus fits together properly and that the exit tube reaches to the bottom of the volumetric flask. Raise the adapter, add 15 ml of the hydriodic-hypophosphorous-hydrochloric acid reagent, and immediately reconnect the apparatus. Start a timer, heat the round-bottom flask with a

Bunsen burner until the solution comes to an incipient boil, and then move the burner back and forth so as to maintain the solution at or just below an incipient boil (this will require about a 2-second interval after each pass). Incipient boiling is indicated by bubbles at the interface of the solution and the sides of the flask. The total heating time is 5 minutes. At the end of this 5-minute period lower the beaker and remove the volumetric flask. Immediately add 5 ml of lead citrate solution, swirl, and dilute to the mark. Within 5 minutes measure the transmittance at 400 millimicrons with a spectrophotometer that has been set to 100% transmittance with distilled water.

Calculate the actual percent transmittance as follows:

$$\text{Actual \% transmittance} = \frac{\text{\% transmittance of sample vs. water}}{\text{\% transmittance of blank vs. water}} \times 100$$

Plot milligrams of sulfur against percent transmittance.

Procedure

If the material is nitrocellulose, dry it by heating at 40° - 45°C for 2 hours and then at 98° - 120°C for 2 hours.

Weigh the sample of propellant or nitrocellulose into a 50-ml round bottom flask with a 19/38 ground glass joint. Use a sample containing preferably 0.1 to 0.2 mg of sulfur but do not use a sample greater than 0.4-gram. The proper size sample for a propellant can usually be ascertained from the specification requirements. Secure the flask with a clamp attached to a ring stand. Add 7 ml of nitric acid-perchloric acid-magnesium nitrate reagent. Brush the flame of a Bunsen burner back and forth across the bottom of the flask in such a manner that the heat is not sufficient to cause the solution to foam out of the flask. When the excessive foaming has ceased, place the Bunsen burner under the flask, heat until the salts at the bottom of the flask are completely dry, and then heat for approximately 2 more minutes. Allow to cool and proceed with the addition of the hydriodic-hypophosphorous-hydrochloric acid reagent and distillation as in the preparation of the calibration curve. Carry a blank through the entire procedure.

Calculate as follows:

$$\text{\% sulfur} = \frac{\text{mg of sulfur as read from curve}}{\text{grams of sample} \times 10}$$

% Na_2SO_4 = 4.43 x % sulfur

% K_2SO_4 = 5.43 x % sulfur

% H_2SO_4 = 3.06 x % sulfur

% $SO_4^{=}$ = 3.00 x % sulfur.

Note:

Rinse the volumetric flasks with nitric acid (1 to 1) before using them for the next determination in order to remove lead salts that adhere to the sides of the flasks. Rinse the spectrophotometer cells with concentrated nitric acid daily.

C. RESULTS FOR SULFATE IN PROPELLANTS AND NITROCELLULOSE

The results obtained for sulfate in four nitrocellulose-base propellants, one composite propellant, and a sample of nitrocellulose by the spectrophotometric method are shown in Table II. Also shown are the results obtained for sulfate in the nitrocellulose-base propellants and the nitrocellulose by the gravimetric nitric acid-perchloric acid method (11). The results by the proposed spectrophotometric method check the results for the gravimetric method for the samples of nitrocellulose-base propellants reasonably well when significant amounts of sulfur are present. The gravimetric method gives low and inaccurate results for samples of nitrocellulose or propellants containing less than 0.01% sulfur because of incomplete precipitation of the sulfur (this was checked by use of synthetic samples).

Experiments on recoveries by the spectrophotometric method were conducted by evaporating aliquots of potassium sulfate solution to dryness in 50-ml flasks, adding 0.4 gram of nitrocellulose or composite propellant and carrying the samples through the procedures. The recoveries were good (Table III).

D. APPLICATION OF THE METHOD TO OTHER MATERIALS

Obviously, the method can be used for many materials besides propellants and nitrocellulose. This laboratory is investigating the applicability of the method to the determination of sulfate in chrome plating baths.

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VI. TABLES

Table I. Effect of Water

ml of Water	% Transmittance
0	36
3	36
5	36
7	42
10	67

Table II. Results for Sulfate in Propellants and Nitrocellulose

Sample	Found Gravimetrically (%)	Sample Size Used for Spectrophotometric Method (gram)		Found Spectrophotometrically (%)
140[a]	0.02 S; 0.09 Na_2SO_4	0.4		0.027 S; 0.120 Na_2SO_4
		0.4		0.026 S; 0.115 Na_2SO_4
		0.4		0.028 S; 0.124 Na_2SO_4
			Avg.	0.027 S; 0.120 Na_2SO_4
297[b]	0.02 S; 0.09 Na_2SO_4	0.4		0.020 S; 0.089 Na_2SO_4
		0.4		0.020 S; 0.089 Na_2SO_4
		0.4		0.022 S; 0.097 Na_2SO_4
			Avg.	0.021 S; 0.092 Na_2SO_4
487[c]	0.08 S: 0.43 K_2SO_4	0.2		0.084 S; 0.46 K_2SO_4
		0.2		0.089 S; 0.48 K_2SO_4
		0.2		0.082 S; 0.45 K_2SO_4
		0.2		0.084 S; 0.46 K_2SO_4
			Avg.	0.085 S; 0.46 K_2SO_4

Table II. Results for Sulfate in Propellants and Nitrocellulose (cont'd)

Sample	Found Gravimetrically (%)	Sample Size Used for Spectrophotometric Method (gram)		Found Spectrophotometrically (%)
446^d	0.18 S; 0.98 K_2SO_4	0.1		0.170 S; 0.92 K_2SO_4
		0.1		0.184 S; 1.00 K_2SO_4
		0.1		0.184 S; 1.00 K_2SO_4
			Avg.	0.179 S; 0.97 K_2SO_4
COM^e		0.4		0.000 S
		0.4		0.000 S
		0.4		0.000 S
			Avg.	0.000 S
NC	0.00 S	0.4		0.003 S; 0.009 H_2SO_4
				0.004 S; 0.012 H_2SO_4
				0.005 S; 0.015 H_2SO_4
			Avg.	0.004 S; 0.012 H_2SO_4

Table II. Results for Sulfate in Propellants and Nitrocellulose (cont'd)

[a] Contains (%): 97.0 nitrocellulose, 0.70 nitroglycerin, 0.70 dinitrotoluene, 0.80 diphenylamine, 0.50 calcium carbonate, 0.16 graphite.

[b] Contains (%): 82.4 nitrocellulose, 10.07 nitroglycerin, 0.36 dinitrotoluene, 0.92 diphenylamine, 5.17 dibutylphthalate, 0.59 calcium carbonate, 0.34 graphite.

[c] Special nitrocellulose-base propellant coated with potassium sulfate and 0.44% potassium nitrate.

[d] Contains (%): 93.0 nitrocellulose, 5.45 dinitrotoluene, 0.68 diphenylamine, and 0.14 graphite.

[e] Contains (%): 70 ammonium perchlorate, 10 aluminum, and 20 organic binder.

Table III. Recovery of Sulfate from Synthetic Samples

Sample	mg of S Found	mg of S Recovered
0.4 gram COM[a] + 0.020 mg S	0.021	0.021
0.4 gram COM + 0.100 mg S	0.105	0.105
0.4 gram COM + 0.200 mg S	0.196	0.196
0.4 gram COM + 0.250 mg S	0.253	0.253
0.4 gram NC[b] + 0.020 mg S	0.038	0.022
0.4 gram NC + 0.050 mg S + 10 mg Sn	0.064	0.048
0.4 gram NC + 0.100 mg S	0.120	0.104
0.4 gram NC + 0.200 mg S + 10 mg Sn	0.212	0.196

[a] Contains 0.000% S (Table I).

[b] Contains 0.004% S (Table II).

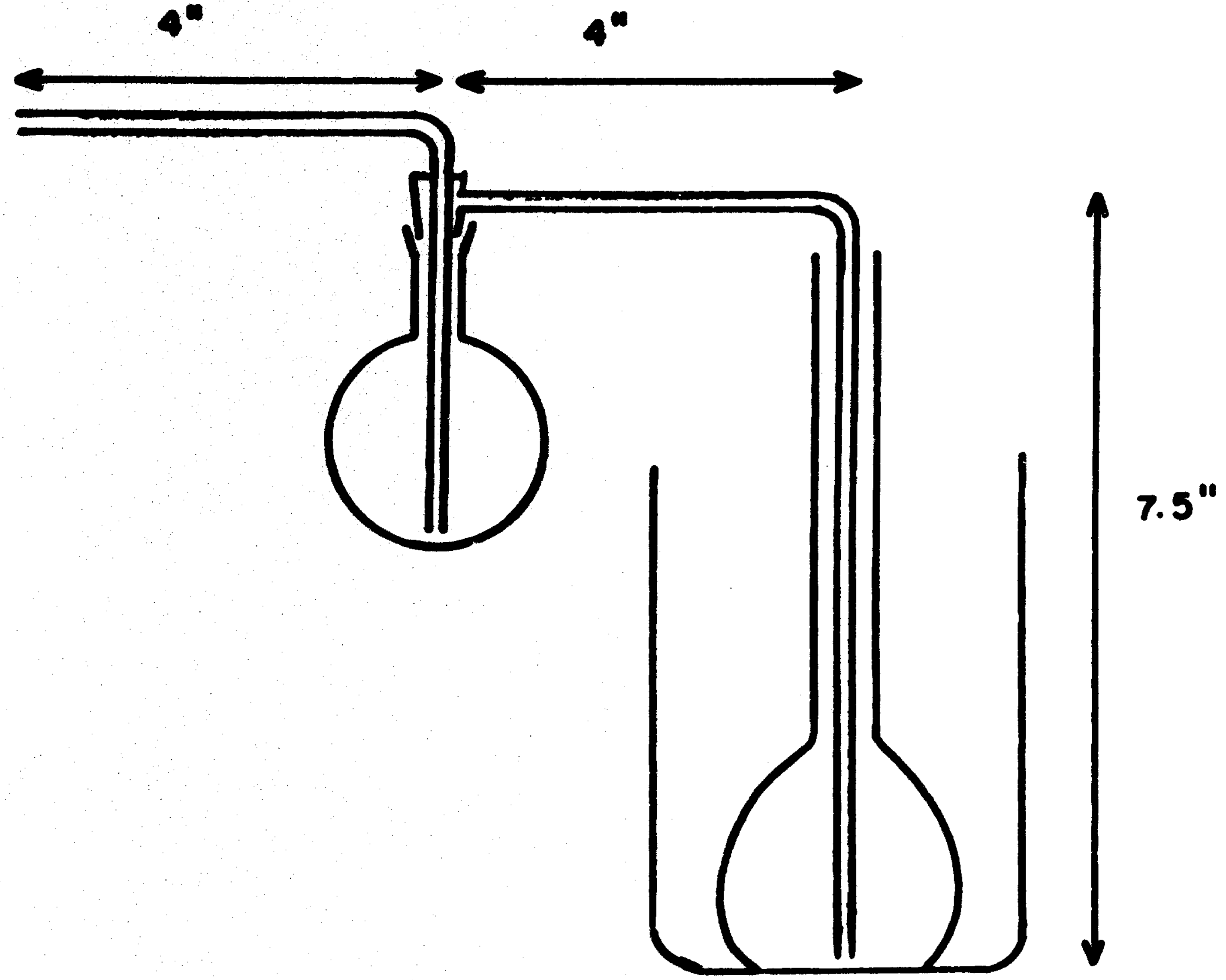

FIGURE 1. DISTILLATION APPARATUS

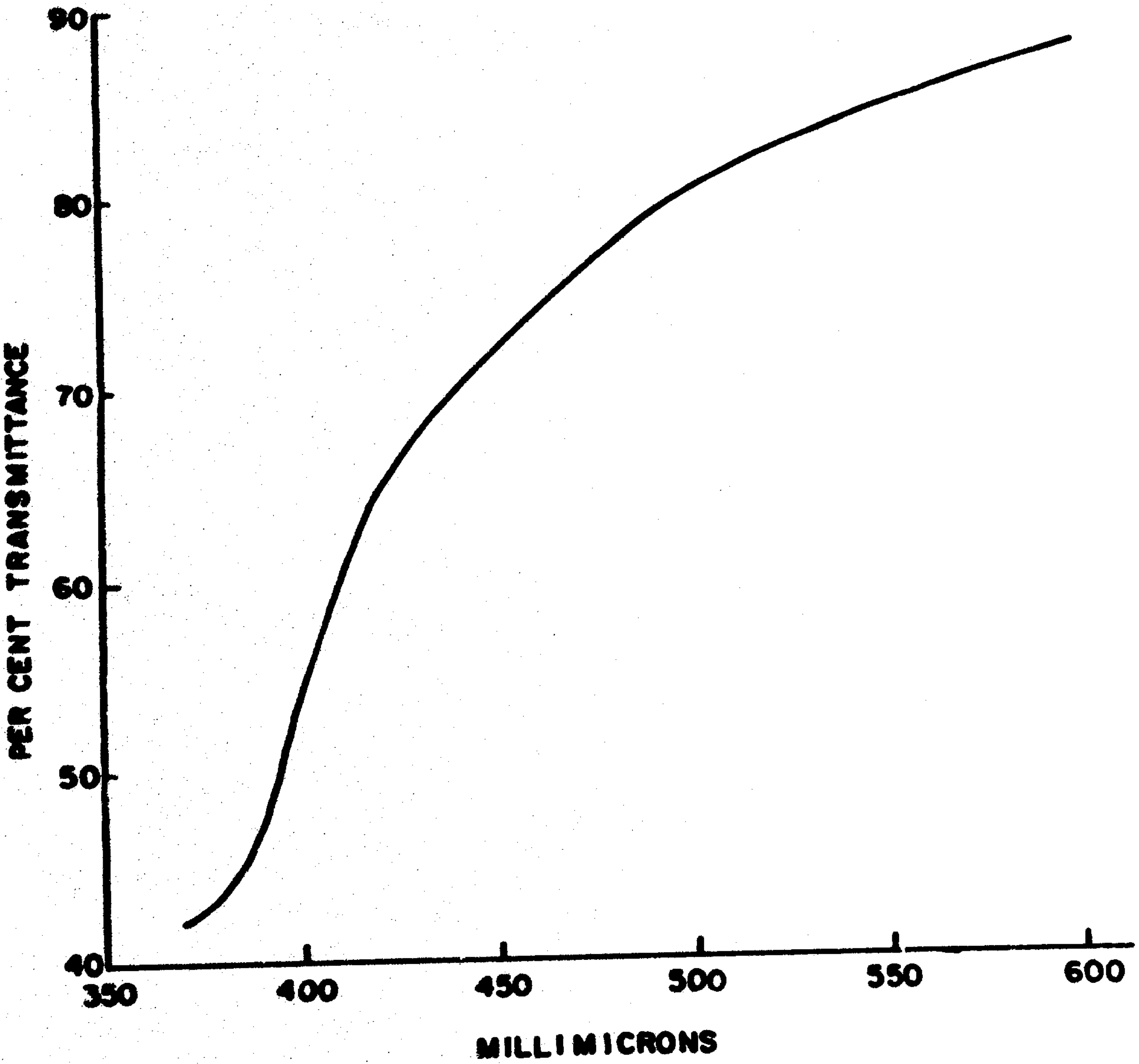

FIGURE 2. SPECTROPHOTOMETRIC CURVE FOR COLLOIDAL LEAD SULFIDE SOL

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13. ABSTRACT

An accurate method is proposed for the determination of sulfate in propellants and nitrocellulose that requires only 45 minutes. The sample is treated with a mixture of nitric acid, perchloric acid and magnesium nitrate, and the solution is evaporated to dryness over a Bunsen burner. The salts are heated for 5 minutes with a mixture of hydriodic, hypophosphorous and hydrochloric acids in a simplified distillation apparatus while passing nitrogen through the system, the hydrogen sulfide which results from the reduction of sulfate is absorbed into ammonium hydroxide, lead citrate is added, and the brownish-yellow lead sulfide color is measured.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Sulfate						
Propellants						
Nitrocellulose						