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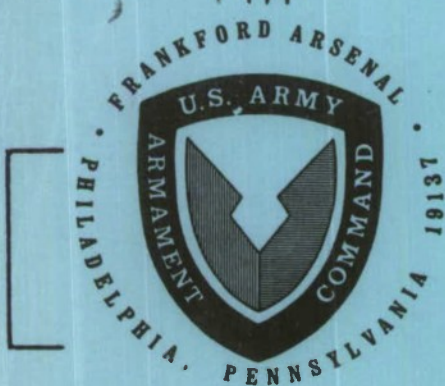
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SPECTROPHOTOMETRIC DETERMINATION OF TRACE AMOUNTS OF SILICON, IRON, AND ALUMINUM IN NITROCELLULOSE-BASE PROPELLANTS

August 1974

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Technical Support Directorate

**U.S. ARMY ARMAMENT COMMAND
FRANKFORD ARSENAL
PHILADELPHIA, PENNSYLVANIA 19137**

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER FA-TT-74006		2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) SPECTROPHOTOMETRIC DETERMINATION OF TRACE AMOUNTS OF SILICON, IRON, AND ALUMINUM IN NITROCELLULOSE-BASE PROPELLANTS		5. TYPE OF REPORT & PERIOD COVERED Technical Test - Final	
7. AUTHOR(s) George Norwitz Herman Gordon		6. PERFORMING ORG. REPORT NUMBER	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Commander FRANKFORD ARSENAL, ATTN: SARFA-TSE-M-64-1 Philadelphia, PA 19137		8. CONTRACT OR GRANT NUMBER(s) AMS Code 685801.49.11100 PRON A1-4-R0020-AW-F6	
11. CONTROLLING OFFICE NAME AND ADDRESS Commander FRANKFORD ARSENAL, ATTN: SARFA-TSE-519-2 Philadelphia, PA 19137		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Materials Testing Technology	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE August 1974	
		13. NUMBER OF PAGES 21	
		15. SECURITY CLASS. (of this report) UNCLASSIFIED	
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.			
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)			
18. SUPPLEMENTARY NOTES			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Silicon Propellant Iron Nitrocellulose Aluminum			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Methods are proposed for the spectrophotometric determination of trace amounts of silicon, iron, and aluminum in nitrocellulose-base propellants. The silicon is determined by the molybdenum blue method after treatment of the propellant with nitric acid, evaporation to dryness, ignition, fusion with sodium carbonate, and adjustment to pH 1.2-1.3. The iron is determined with ortho-phenanthroline after treatment with nitric and sulfuric (Continued on reverse)			

acids, evaporation to fumes of sulfuric acid, adjustment to pH 5.5-5.6, and reduction with hydroxylamine hydrochloride. The aluminum is determined using a composite aluminon reagent after treatment with nitric and perchloric acids, evaporation to fumes of perchloric acid, removal of the iron by a cupferron precipitation, and destruction of the cupferron by evaporation to fumes of perchloric acid. Results are given for silicon, iron, and aluminum in twenty propellant samples. The silicon ranged from 0.001 to 0.022%, the iron from 0.004 to 0.032%, and the aluminum from 0.0001 to 0.0040%.

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I. SUMMARY

Methods are proposed for the spectrophotometric determination of trace amounts of silicon, iron, and aluminum in nitrocellulose-base propellants. The silicon is determined by the molybdenum blue method after treatment of the propellant with nitric acid, evaporation to dryness, ignition, fusion with sodium carbonate, and adjustment to pH 1.2-1.3. The iron is determined with ortho-phenanthroline after treatment with nitric and sulfuric acids, evaporation to fumes of sulfuric acid, adjustment to pH 5.5-5.6, and reduction with hydroxylamine hydrochloride. The aluminum is determined using a composite aluminum reagent after treatment with nitric and perchloric acids, evaporation to fumes of perchloric acid, removal of the iron by a cupferron precipitation, and destruction of the cupferron by evaporation to fumes of perchloric acid. Results are given for silicon, iron, and aluminum in twenty propellant samples. The silicon ranged from 0.001 to 0.022%, the iron from 0.004 to 0.032%, and the aluminum from 0.0001 to 0.0040%.

II. RECOMMENDATIONS

It is recommended that the methods in this report be included in MIL-STD-286 (Military Standard, Propellants, Solid: Sampling, Examination and Testing).

III. INTRODUCTION

It is the purpose of the present investigation to develop methods for the determination of silicon, iron, and aluminum in nitrocellulose-base propellants and to ascertain how much silicon, iron, and aluminum is present in typical nitrocellulose-base propellants. It is known that small amounts of these elements are present in nitrocellulose-base propellants but no exact figures are available because of the lack of adequate analytical methods.

IV. STUDY

A. DEVELOPMENT OF METHODS FOR THE DETERMINATION OF SILICON, IRON, AND ALUMINUM

1. Determination of Silicon

In the determination of silicon, the sample was dissolved in nitric acid in a platinum crucible, the solution evaporated to dryness, the organic matter burned off, the residue fused with sodium carbonate, and the silicon determined spectrophotometrically by the molybdenum blue method. The present authors modified the molybdenum blue method of Carlson and Banks (1, 3) (previously used for the determination of silicon in zirconium). A sodium carbonate fusion for bringing silicon into solution prior to the spectrophotometric determination has been used previously for organic materials (2). It is recommended that the calibration curve be prepared by evaporating aliquots of standard silicon solution to dryness and fusing with sodium carbonate as in the method.

2. Determination of Iron

The iron was determined spectrophotometrically with ortho-phenanthroline after treating the sample with nitric and sulfuric acids and evaporating to fumes of sulfuric acid. The ortho-phenanthroline color was developed after neutralizing to pH 5.5-6.0 and reducing the iron with hydroxylamine hydrochloride, as has been done previously in the determination of iron in zirconium (1).

3. Determination of Aluminum

The aluminum was determined spectrophotometrically with aluminon after treating the sample with nitric and perchloric acids, evaporation to fumes of perchloric acid, elimination of the iron by a cupferron precipitation, and destruction of organic matter by evaporation to fumes of perchloric acid. The composite aluminon reagent of Craft and Makepeace (4), slightly modified for convenience, was used. This composite reagent contains aluminon, ammonium acetate (prepared by mixing ammonium hydroxide and acetic acid and adjusting the pH), gelatin, and a preservative (benzoic acid). The present authors developed the color by adding 15.0 ml of the composite reagent to 50 ml of solution

and heating in a boiling water bath for about 13-15 minutes. This produced a clear lake and very reproduceable results. Craft and Makepeace developed the color by adding 15.0 ml of the composite reagent to 5 ml of solution, heating at 90^o-100^oC for 10 minutes, and allowing to stand at room temperature for 10 minutes. In the present method, the pH of the solution after the addition of the composite aluminon reagent was 5.35. In the method of Craft and Makepeace (4) the pH of the solution after the addition of the composite aluminon reagent was 5.4 (however, in formulating the composite aluminon reagent, Craft and Makepeace established a recommended pH of 5.3 as determined by diluting to 100 ml after adding the composite aluminon reagent).

A study of the effect of time of heating in the boiling water showed that the color developed fully in 12 minutes and did not change on continued heating for 1 hour. The composite reagent, after it has been aged for 3 days, is stable for a month. As has been stated by previous investigators (4, 12, 13), the intensity of the color varies with different lots of aluminon, hence a new calibration curve must be prepared with each lot of aluminon. Craft and Makepeace (4) established that perchlorate does not interfere with the determination of aluminum by aluminon.

The elimination of the iron by a cupferron precipitation in a 10% perchloric acid solution offered no difficulties. Previous investigators (5) have used a sulfuric or hydrochloric acid solution for this precipitation. It was found that elimination of the iron by precipitation with cupferron gave better results for aluminum than were obtained by complexing the iron with mercaptoacetic acid (the latter reagent decreases the intensity of the aluminon color (12)).

B. RECOMMENDED METHOD FOR THE DETERMINATION OF SILICON

1. Reagents

Silicon dioxide, prepared by treating about 3 grams of sodium metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$) with water, adding 25 ml of perchloric acid, evaporating to fumes of perchloric acid, fuming for 10-15 minutes, adding water, filtering, washing with water, burning off the filter paper, and igniting at red heat.

NOTE: Store all of the following reagents in polyethylene bottles.

Standard silicon solution No. 1 (1 ml = 0.05 mg Si). Fuse 0.1070 gram of silicon dioxide with 1 gram of sodium carbonate, cool, dissolve the melt in water, and dilute to 1 liter in a volumetric flask.

Standard silicon solution No. 2 (1 ml = 0.01 mg Si). Dilute 100 ml of standard silicon solution No. 1 to 500 ml in a volumetric flask.

Molybdic acid solution. Dissolve 25 grams of ammonium molybdate tetrahydrate ($(\text{NH}_4)_6\text{MoO}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) in 200 ml of water, add 20 ml of sulfuric acid, cool, and dilute to 250 ml.

Tartaric acid solution (20%).

Reducing solution. Dissolve 27 grams of sodium hydrogen sulfite (NaHSO_3), 2 grams of sodium hydroxide, and 0.5 gram of 1-amino-2-naphthol-4-sulfonic acid in water, and dilute to 250 ml. Prepare fresh every 2 weeks.

Ammonium hydroxide (silica-free). Bubble ammonia gas into 250 ml of water (10°C) in a polyethylene bottle until appearance of excess gas bubbles indicates that the solution is saturated.

2. Preparation of Calibration Curve

Transfer 0.0, 2.0, 4.0, 7.0, 10.0, and 12.5 ml of standard silicon solution No. 2 (1 ml = 0.01 mg Si) to platinum crucibles. Evaporate to dryness and fuse with 1 gram of sodium carbonate over a Meker burner. Add 25 ml of water and warm on the hot plate until the melt is dissolved. Wash into polyethylene beakers, dilute to about 80 ml, and cool to $23^\circ\pm 2^\circ\text{C}$ (maintain this temperature to the end of the determination). Add 4 ml of molybdic acid solution from a pipet and swirl to eliminate excess carbon dioxide. Using hydrochloric acid (1 to 3) or ammonium hydroxide (silica-free), adjust the pH to 1.2-1.3 by means of a pH meter, swirling to eliminate carbon dioxide. Allow the solution to stand 8-12 minutes. Working with only one solution at a time, add 4 ml of tartaric acid solution (20%) from a graduate, swirl, and immediately follow with 4 ml of reducing solution from a graduate (have the reducing solution ready in the graduate so that little time will elapse between the addition of the tartaric acid and reducing solutions). Wash into 100-ml volumetric flasks and dilute to the mark. Read the absorbance in 30 to 45 minutes at 815 nm with a spectrophotometer that has been set to 100% transmittance with the reagent blank. Plot absorbance against mg of silicon (per 100 ml).

3. Procedure

For up to 0.012% silicon use a 1-gram sample; for 0.012 to 0.024% silicon use a 0.5-gram sample. Transfer the sample to a platinum crucible and add 5 ml of nitric acid. Warm until the vigorous reaction

has ceased and then evaporate to dryness. Ignite at red heat until the residue is white. Fuse with 1 gram of sodium carbonate, dissolve the melt in water, and develop the color as described under the preparation of the calibration curve. Convert the reading to mg of silicon by referring to the calibration curve and calculate the percent silicon as follows:

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

C. RECOMMENDED METHOD FOR THE DETERMINATION OF IRON

1. Reagents

Standard iron solution No. 1 (1 ml = 1.0 mg Fe). Dissolve 1.0000 gram of high-purity iron in 50 ml of hydrochloric acid (1 to 1) by heating on the hot plate, cool, and dilute to 1 liter.

Standard iron solution No. 2 (1 ml = 0.1 mg Fe). Pipet 25 ml of standard iron solution No. 1 into a 250-ml volumetric flask and dilute to the mark.

Tartaric acid solution (20%).

Hydroxylamine hydrochloride (10%). Store in a refrigerator.

Ortho-phenanthroline solution (0.2%). Dissolve 0.20 gram of ortho-phenanthroline in hot water, cool, and dilute to 100 ml. Store in a refrigerator.

2. Preparation of Calibration Curve

Transfer 0.0, 0.5, 1.0, 2.0, 3.0, and 4.0 ml of standard iron solution No. 2 (1 ml = 0.1 mg Fe) to 150-ml beakers and dilute to about 25 ml. Add 5 ml of tartaric acid solution (20%), 1 ml of hydroxylamine hydrochloride solution (25%), and 5 ml of ortho-phenanthroline solution (0.2%), swirling after the addition of each reagent. Add ammonium hydroxide (1 to 2) until the pH is 5.5-6.0 (using pH paper). Wash the solution into 100-ml volumetric flasks, dilute to about 90 ml, and heat in a water bath at about 60^o-70^oC for about 30 minutes. Cool to room temperature and dilute to the mark. Read the absorbance at 510 nm with a spectrophotometer that has been set to 100% transmittance with the reagent blank. Plot absorbance against mg of iron (per 100 ml).

3. Procedure

Transfer a 5-gram sample to a 400-ml beaker, add 20 ml of nitric acid, and warm gently until the vigorous reaction has ceased. Add 5 ml of sulfuric acid, evaporate to fumes of sulfuric acid, and add nitric acid dropwise until the organic matter is destroyed. Evaporate to a volume of about 2 ml, adding more nitric acid dropwise if necessary. Allow to cool, add about 50 ml of water, and boil for a few minutes. Allow to cool, filter through a Whatman No. 40 filter paper into a 100-ml volumetric flask, and wash with water. Discard the filter paper and dilute the filtrate to the mark. Pipet a 25-ml aliquot (equivalent to a 1.25-gram sample) into a 150-ml beaker and develop the color as described under preparation of the calibration curve. Convert the reading to mg of iron by referring to the calibration curve and calculate the percent iron as follows:

$$\text{Percent iron} = \frac{\text{mg of iron as read from curve}}{\text{grams of sample in aliquot} \times 10}$$

D. RECOMMENDED METHOD FOR THE DETERMINATION OF ALUMINUM

1. Reagents

Standard aluminum solution No. 1 (1 ml = 0.25 mg Al). Dissolve 0.2500 gram of high-purity aluminum in 25 ml of hydrochloric acid (1 to 1) by heating on the hot plate, cool, and dilute to 1 liter in a volumetric flask.

Standard aluminum solution No. 2 (1 ml = 0.01 mg Al). Pipet 10 ml of standard aluminum solution No. 1 into a 250-ml volumetric flask and dilute to the mark. Prepare fresh daily.

Benzoic acid solution (10%). Dissolve 50 grams of benzoic acid in 500 ml of methanol.

Acetate buffer for aluminum reagent. Cool 470 ml of ammonium hydroxide in an ice bath and add 430 ml of glacial acetic acid slowly with stirring. Cool to room temperature and add more acid or base to bring the pH to 5.25-5.35 when 2.5 ml of the solution is diluted to 50 ml with water. After the proper pH has been obtained, dilute to 1 liter with water.

Gelatin solution (1%). Add 3.5 grams of gelatin to about 250 ml of hot water while stirring, warm until clear, cool, and dilute to 350 ml.

Composite aluminon reagent. Dissolve 0.35 gram of aluminon in about 200 ml of water, add 70 ml of benzoic acid solution (10%), and dilute to 350 ml. Add 350 ml of buffer solution and 350 ml of gelatin solution (1%). Allow to stand for 3 days or more before using. Store in a dark bottle.

Cupferron solution (5%), cold, freshly prepared, filtered.

Cupferron wash solution. Add 30 ml of cupferron solution (5%) and 3 ml of perchloric acid to 200 ml of ice water.

2. Preparation of Calibration Curve

Transfer 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of aluminum solution No. 2 (1 ml = 0.01 mg Al) to 100-ml of volumetric flasks and dilute to about 50 ml. Add 15.0 ml of composite aluminon reagent with a pipet and place the flasks in boiling tap water (about 500 ml) contained in 600-ml beakers on the hot plate for 13-15 minutes. Cool and dilute to the mark. Read the absorbance within 2 hours at 540 nm with a spectrophotometer that has been set to 100% transmittance with the reagent blank. Plot absorbance against mg of aluminum (per 100 ml).

3. Procedure

Transfer a 4-gram sample to a 400-ml beaker, add 20 ml of nitric acid, and warm gently until the vigorous reaction has ceased. Boil down to about 10 ml and allow to cool somewhat. Add 10 ml of perchloric acid (70%), evaporate to fumes of perchloric acid, and add nitric acid dropwise until the solution is colorless. Adjust the volume of perchloric acid to about 5 ml, either by further heating or by adding more perchloric acid. Allow to cool somewhat, add 45 ml of water, and cool in an ice bath. Add about 2 ml of cupferron solution (5%) dropwise from a pipet while stirring. Filter through a Whatman No. 42 filter paper into a 150-ml beaker. Wash with cupferron wash solution, add 20 ml of nitric acid to the filtrate, evaporate to fumes of perchloric acid, and continue fuming to a volume of about 2 ml. Allow to cool and add 25 ml of water. Add ammonium hydroxide (1 to 1) until the solution is just alkaline to litmus paper and then add hydrochloric acid (1 to 1) until the solution is just acid to the litmus paper. Wash into a 100-ml volumetric flask and dilute to the mark. For up to 0.0025% aluminum, pipet a 50-ml aliquot (equivalent to a 2-gram sample) into a 100-ml volumetric flask; for 0.0025 to 0.0050% aluminum, use a 25-ml aliquot and add 25 ml of water. Add 15.0 ml of composite aluminon reagent and develop the color as described under preparation of calibration curve. Convert the reading to mg of aluminum by referring to the calibration curve and calculate the percent

aluminum as follows:

$$\text{Percent aluminum} = \frac{\text{mg of aluminum as read from curve}}{\text{grams of sample in aliquot} \times 10}$$

NOTE: Clean the spectrophotometer cells and volumetric flasks with hydrochloric acid (1 to 1) after each determination.

E. RESULTS FOR SILICON, IRON, AND ALUMINUM

The results obtained for silicon, iron, and aluminum in twenty propellant samples are shown in Table I. It is seen that the precision is satisfactory. The silicon ranged from 0.001 to 0.022%, the iron from 0.004 to 0.032%, and the aluminum from 0.0001 to 0.0040%.

Synthetic samples were prepared by adding portions of standard silicon, iron, and aluminum solutions to a sample that contained only slight amounts of silicon, iron, and aluminum (sample 18, Table I) and proceeding as in the methods, using the sample as a blank. The recoveries (Table II) were satisfactory.

The sources of the silicon, iron, and aluminum are the raw materials and the equipment used in the manufacturing process. Insofar as the raw materials are concerned, it is believed that most of the silicon, iron, and aluminum originate from nitrocellulose, graphite, calcium carbonate, potassium nitrate, potassium sulfate, and sodium sulfate. The last five substances are minor ingredients of nitrocellulose-base propellants (the total for all the ingredients will rarely exceed 1.5%). The military specification for nitrocellulose (8) has no requirement for metallic impurities but has a maximum ash requirement of 0.4%. The military specification for graphite (7) likewise has no requirement for metallic impurities but has a maximum ash requirement of 0.6%. The military specification for calcium carbonate (6) has a requirement of iron plus aluminum, calculated as the oxides, of 0.7% maximum. The military specification for potassium nitrate (9) has a requirement of iron plus aluminum, calculated as the oxides, as 0.5% maximum. The military specifications for potassium sulfate (10) and sodium sulfate (11) have no requirement for metallic impurities; however, they have minimum assay requirements of 99% and 98%, respectively.

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VI. TABLES

Table I. Results for Silicon, Iron, and Aluminum in Nitrocellulose-Base Propellants

<u>Sample</u>	<u>Type</u>	<u>Silicon (%)</u>	<u>Iron (%)</u>	<u>Aluminum (%)</u>
1	WC846	0.004, 0.005	0.016, 0.014	0.0015, 0.0016
2	WC852	0.004	0.016, 0.014	0.0025, 0.0026
3	WC852	0.004	0.016	0.0021
4	CAN141	0.006	0.014	0.0022
5	IMR5010	0.001, 0.001	0.005, 0.006	0.0011, 0.0013
6	CMR100-126	0.001, 0.002	0.006	0.00072
7	Ball carb.	0.009	0.030, 0.032	0.0021
8	WC846	0.022 ^a , 0.022 ^a	0.030, 0.028	0.0038 ^b , 0.0040 ^b
9	IMR8208	0.002	0.007	0.00083
10	WC844	0.010, 0.009	0.014	0.0014
11	WC870	0.004	0.012	0.0001, 0.0001
12	IMR4895	0.001	0.007	0.0001
13	IMR4475	0.001	0.004	0.0001
14	WC846	0.001	0.013	0.0015
15	WC844	0.007	0.005	0.0021
16	IMR5010	0.001	0.005	0.00070
17	SR7641	0.002	0.004	0.00051
18	IMR5010	0.001	0.004	0.00051
19	Foreign	0.003	0.004	0.00092
20	SR8231	0.001	0.004	0.00083

^a0.5-gram sample.

^b25-ml aliquot.

Table II. Results for Synthetic Samples

<u>Synthetic sample</u>	<u>Found</u>
1 gram sample 18 ^a + 0.010 mg Si	0.012 mg Si
1 gram sample 18 + 0.030 mg Si	0.032 mg Si
1 gram sample 18 + 0.100 mg Si	0.097 mg Si
5 grams sample 18 + 0.20 mg Fe ^b	0.20 mg Fe
5 grams sample 18 + 0.80 mg Fe ^b	0.84 mg Fe
5 grams sample 18 + 1.20 mg Fe ^b	1.28 mg Fe
4 grams sample 18 + 0.020 mg Al ^c	0.018 mg Al
4 grams sample 18 + 0.040 mg Al ^c	0.044 mg Al
4 grams sample 18 + 0.080 mg Al ^c	0.086 mg Al

^aThis sample was used as the blank (see Table I).

^b25-ml aliquot.

^c50-ml aliquot.

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Date Printed: 9 September 1974