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DETERMINATION OF POTASSIUM, SODIUM, CALCIUM, AND BARIUM IN NITROCELLULOSE-BASE PROPELLANTS BY ATOMIC ABSORPTION

by

Julius B. Apatoff
George Norwitz

February 1969

AMS Code 4931.0M.5042

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DEPARTMENT OF THE ARMY
FRANKFORD ARSENAL
Philadelphia, Pa. 19137

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Test Report T-69-1-1

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Quality Assurance Directorate
FRANKFORD ARSENAL
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I. SUMMARY

Atomic absorption methods are proposed for the determination of potassium, sodium, calcium, and barium in nitrocellulose-base propellants. A 1-gram sample is treated with a mixture of 10 ml of nitric acid and 3 ml of perchloric acid and the solution is evaporated to a volume of approximately 1 ml. The solution is then diluted to an appropriate volume (usually 500 ml for potassium or 100 ml for sodium, calcium, and barium) and the absorption is determined with an atomic absorption spectrophotometer. There are no significant interferences from the amounts and ratios of salts found in nitrocellulose-base propellants. The accuracy of the methods was checked with actual and synthetic propellants. The proposed methods are more reliable and considerably more rapid than the wet chemical methods presently used for the determination of inorganic salts in nitrocellulose-base propellants.

II. RECOMMENDATIONS

It is recommended that the methods in this report be included in the military standard for propellants (MIL-STD-286B) and other appropriate military documents.

III. INTRODUCTION

Potassium salts (potassium sulfate and potassium nitrate), sodium sulfate, calcium carbonate, and barium nitrate are found in many nitrocellulose-base propellants. The following are the amounts of these salts that are present in the ordinary types of propellants:

0.5 to 2%	potassium sulfate.
0.5 to 2%	potassium nitrate.
0 to 0.2%	sodium sulfate plus 0.5 to 1% calcium carbonate.
0.5 to 4%	barium nitrate plus 0.5 to 2% potassium nitrate.

The methods presently used for the determination of the above salts in nitrocellulose-base propellants are time-consuming and leave much to be desired in the way of reliability. The potassium sulfate and potassium nitrate are ordinarily determined gravimetrically by precipitation with tetraphenylboron (6). Sodium sulfate and calcium carbonate are usually determined together by dissolution of the propellant in morpholine, filtration of the sodium sulfate and calcium carbonate by means of a Gooch, weighing the combined salts, washing with water to dissolve the sodium sulfate, weighing again, dissolution of the calcium carbonate with dilute acid, and weighing once more (4). Barium nitrate is ordinarily determined by precipitation as barium sulfate (3). Methods exist for the determination of potassium (5) (and other metals) in nitrocellulose-base propellants by use of flame photometry but the results over a period of years have not been gratifying.

In view of the need for more accurate and more rapid methods for the determination of potassium, sodium, calcium, and barium salts in propellants, this laboratory undertook an investigation to develop atomic absorption procedures.

IV. STUDY

A. DEVELOPMENT OF METHODS

It was decided that the best means for preparing the sample was to treat the propellant with a mixture of nitric and perchloric acids and evaporate to fumes of perchloric acid to destroy the organic matter. This treatment is more rapid than treatment with nitric and sulfuric acids, evaporation to fumes of sulfuric acid, and further treatment with nitric acid. The latter treatment is not applicable when barium is to be determined because of the precipitation of barium sulfate. A third method of preparing the sample is to treat with nitric acid, evaporate to dryness, ignite, and dissolve the ash in dilute hydrochloric acid. There is considerable likelihood of losses and contamination using this ignition technique, therefore, it is not recommended.

The solutions obtained after evaporating with perchloric acid and adding water were clear, except for a slight amount of graphite that sometimes remained. This graphite settled out quickly after dilution to volume and caused no difficulty.

An investigation was made of the effect of the volume of perchloric acid in the determination of potassium, sodium, calcium, and barium (Table I). It is seen that more than 2 ml of perchloric acid causes a decrease in absorption for potassium and sodium, while more than 1 ml of perchloric acid causes a decrease in absorption for calcium and barium. It is recommended, therefore, that the solution be evaporated to a volume of about 1 ml to eliminate any possible effect from perchloric acid on the absorption.

The perchloric-nitric acid treatment is completely safe and the authors never encountered any instance of violent reaction. The fact that only a 1-gram sample is used is probably helpful in the quick and quiet destruction of the organic matter.

The instrument settings chosen for the atomic absorption measurements for potassium, sodium, calcium, and barium were essentially those recommended by Perkin-Elmer Corp. (1).

Since barium has a lower sensitivity than potassium, sodium, or calcium, it is necessary to use a more concentrated solution of barium and an expanded absorption scale in order to obtain a satisfactory absorption range.

The question of interferences was considered. This essentially means the possible mutual interference of potassium, sodium, calcium, and barium. Judging by previous work (1) it would not be expected that there would be significant interference from the amounts and ratios of the salts found in propellants. This is borne out by the results obtained with actual and synthetic propellants. These results will be discussed later.

The proposed methods would not be applicable, to mixtures containing barium nitrate and a sulfate salt, because of the precipitation of barium sulfate. Fortunately this combination of salts was not found in propellants.

B. RECOMMENDED METHODS

1. Apparatus

Atomic absorption spectrophotometer. This laboratory used a Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer.

2. Reagents

Potassium stock solution (0.1 mg of potassium per ml). Dissolve 0.1910 gram of potassium chloride in water and dilute to 1 liter in a volumetric flask.

Sodium stock solution (0.05 mg of sodium per ml). Dissolve 0.1271 gram of sodium chloride in water and dilute to 1 liter in a volumetric flask.

Calcium stock solution (0.5 mg of calcium per ml). Transfer 1.2490 grams of calcium carbonate to a 250-ml beaker, add 150 ml of water and 5 ml of hydrochloric acid slowly, warm until dissolved, cool, and dilute to 1 liter in a volumetric flask.

Barium stock solution (2.5 mg of barium per ml). Dissolve 4.7575 grams of barium nitrate in water and dilute to 1 liter in a volumetric flask.

Perchloric acid, 60-percent.

Nitric acid, 70-percent.

3. Method for Potassium

The operating conditions and settings for potassium using the Perkin-Elmer Model 303 Spectrophotometer are as follows:

Wavelength - 383.6

Scale - 1

Meter Response - 1

Range - Visual

Slit - 4

Gain - 4

Filter - IN

Source - Hollow cathode potassium lamp (26 ma)

Flow (Acetylene) - 9.0

Flow (Air) - 9.0 [The pressure at the burner regulator should be 30 pounds per square inch as read on the gauge (psig)].

Prepare a calibration curve for potassium, using solutions prepared by transferring 2.50, 5.00, 7.50, and 10.00 ml of potassium stock solution to 100-ml volumetric flasks and diluting to volume with water. This gives a range of 0.25, 0.50, 0.75 and 1.00 mg of potassium per 100 ml. Perform the atomic absorption measurements on the solutions and plot mg of potassium (per 100 ml) vs. absorbance. The calibration curve should be checked daily.

The directions for operating the Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer (2) are given in the Appendix of this report.

Perform the determination of potassium in propellants as follows:

Transfer a 1.00-gram sample to a 150-ml beaker and add 10 ml of nitric acid and 3 ml of perchloric acid. Warm on a hot plate until the propellant is dissolved and then evaporate to fumes of perchloric acid. Continue the fuming with occasional swirling until not more than 1 ml of liquid remains. Carry along a blank determination through the entire procedure. Cool, transfer to a 500-ml volumetric flask, and dilute to the mark. This dilution is satisfactory for samples containing up to 1.0% potassium salt. Perform the atomic absorption measurements and convert the reading to mg of potassium (per 100 ml) by referring to the calibration curve. Deduct the blank.

Calculate as follows:

$$\text{Percent } K_2SO_4 = \frac{\text{mg of K (per 100 ml)} \times \frac{500}{100}}{\text{grams of sample} \times 10 \times 0.4488}$$

$$= \frac{1.114 \times \text{mg of K (per 100 ml)}}{\text{grams of sample}}$$

$$\text{Percent } KNO_3 = \frac{\text{mg of K (per 100 ml)} \times \frac{500}{100}}{\text{grams of sample} \times 10 \times 0.3867}$$

$$= \frac{1.293 \times \text{mg of K (per 100 ml)}}{\text{grams of sample}}$$

4. Method for Sodium

The operating conditions and settings for sodium using the Perkin-Elmer Model 303 Spectrophotometer are as follows:

Wavelength - 295

Scale - 1

Meter Response - 1

Range - Visual

Slit - 3

Gain - 2

Filter - IN

Source - Hollow cathode sodium lamp (15 ma)

Flow (Acetylene) - 9.0

Flow (Air) - 9.0 (the pressure at the burner regulator should be 30 psig).

Prepare a calibration curve for sodium, using solutions prepared by transferring 1.00, 2.00, 4.00, and 6.00 ml of sodium stock solution to 100-ml volumetric flasks and diluting to volume with water. This gives a range of 0.05, 0.10, 0.20, and 0.30 mg of sodium per 100 ml. Perform the atomic absorption measurements on the solutions and plot mg of sodium (per 100 ml) vs. absorbance.

Perform the determination of sodium in propellants as follows:

Treat a 1.00-gram sample the same way as described in method for potassium. Transfer to a 100-ml volumetric flask and dilute to the mark. This dilution is satisfactory for samples containing up to 0.10% sodium sulfate. Perform the atomic absorption measurements and convert the reading to mg of sodium by referring to the calibration curve. Deduct the blank.

Calculate as follows:

$$\text{Percent } Na_2SO_4 = \frac{\text{mg of Na (per 100 ml)}}{\text{grams of sample} \times 10 \times 0.3237}$$

$$= \frac{0.3089 \times \text{mg of Na (per 100 ml)}}{\text{grams of sample}}$$

5. Method for Calcium

The operating conditions and settings for calcium using the Perkin-Elmer Model 303 Spectrophotometer are as follows:

Wavelength - 212

Scale - 1

Meter Response - 1

Range - Visual

Slit - 4

Gain - 1

Filter - OUT

Source - Hollow cathode calcium lamp (15 ma)

Flow (Acetylene) - 9.0

Flow (Air) - 9.0 (the burner regulator should be set to 30 psig).

Prepare a calibration curve for calcium using solutions prepared by transferring 2.00, 5.00, 8.00, and 10.00 ml of calcium stock solution to 100-ml volumetric flasks and diluting to volume with water. This gives a range of 1.00, 2.50, 4.00, and 5.00 mg of calcium per 100 ml. Perform the atomic absorption measurements on the solutions and plot mg of calcium (per 100 ml) vs. absorbance.

Perform the determination of calcium in propellants as follows:

Treat a 1.00-gram sample in the same way as described in method for potassium. Transfer to a 100-ml volumetric flask and dilute to the mark. This dilution is satisfactory for samples containing up to 1.0% calcium carbonate. Perform the atomic absorption measurements and convert the reading to mg of calcium by referring to the calibration curve. Deduct the blank.

Calculate as follows:

$$\text{Percent } CaCO_3 = \frac{\text{mg of Ca (per 100 ml)}}{\text{grams of sample} \times 10 \times 0.4004}$$

$$= \frac{0.2498 \times \text{mg of Ca (per 100 ml)}}{\text{grams of sample}}$$

6. Method for Barium

The operating conditions and settings for barium using the Perkin-Elmer Model 303 Spectrophotometer are as follows:

Wavelength - 277

Scale - 5

Meter Response - 1

Range - Visual

Slit - 3

Gain - 3

Filter - OUT

Source - Hollow cathode barium lamp (20 ma)

Flow (Acetylene) - 9.0

Flow (Air) - 9.0 (the burner regulator should be set at 30 psig).

Prepare a calibration curve for barium using solutions prepared by transferring 2.00, 5.00, 8.00, and 10.00 ml of barium stock solution to 100-ml volumetric flasks and diluting to volume with water. This gives a range of 5.0, 12.5, 20.0, and 25.0 mg of barium per 100 ml. Perform the atomic absorption measurements on the solutions and plot mg of barium (per 100 ml) vs. absorbance.

Perform the determination of barium in propellants as follows:

Treat a 1.00-gram sample in the same way as described in method for potassium. Transfer to a 100-ml volumetric flask and dilute to the mark. This dilution is satisfactory for samples containing up to 4.5 percent barium nitrate. Perform the atomic absorption measurements and convert the reading to mg of barium by referring to the calibration curve. Deduct the blank.

Calculate the percent barium nitrate as follows:

$$\text{Percent } Ba(NO_3)_2 = \frac{\text{mg of Ba (per 100 ml)}}{\text{grams of sample} \times 10 \times 0.5255}$$

$$= \frac{0.1903 \times \text{mg of Ba (per 100 ml)}}{\text{grams of sample}}$$

7. Analysis of Propellants Containing Two Salts

Propellants containing sodium and calcium salts, or barium and potassium salts need but one sample preparation. Dilute the sample for the salt requiring the smaller dilution first and take a proper aliquot for the salt requiring a higher dilution.

C. RESULTS

The results obtained for the determination of potassium sulfate, potassium nitrate, sodium sulfate, calcium carbonate, and barium nitrate by atomic absorption are shown in Tables II to VI. The results show satisfactory agreement with the results obtained by the wet methods except in the case of sodium sulfate. The latter finding was not unexpected since the gravimetric sodium sulfate method has proved troublesome.

The results obtained for the recovery of potassium sulfate, sodium sulfate, calcium carbonate, and barium nitrate in synthetic samples prepared by adding these salts to known propellants are shown in Tables VII, VIII, IX, and X, respectively. The results bear out the conclusion that there are no significant interferences with the determination of inorganic salts in nitrocellulose-base propellants by atomic absorption.

Acknowledgment

The authors are indebted to Samuel Sitelman and Michael Galan for their suggestions.

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(2) "Instruction Manual for Perkin-Elmer Atomic Absorption Spectrophotometer, Model 303", Norwalk, Conn., June 1967.

(3) Military Standard, Propellants, Solid: Sampling, Examination and Testing, MIL-STD-286B, December 1967, Method 304.1.2.

(4) Ibid., Method 305.1.2.

(5) Ibid., Method 310.3.1.

(6) Ibid., Method 310.4.2.

VI. TABLES

Table I. Effect of Perchloric Acid

	Absorption			
Perchloric Acid, ml	Potassium (5 ppm)	Sodium (2 ppm)	Calcium (5 ppm)	Barium (150 ppm)
0	26.2	41.2	18.5	27.1
1	26.5	41.1	18.5	27.0
2	26.2	41.0	17.4	25.6
3	25.5	40.6	17.2	23.0
5	25.5	40.0	17.2	23.0

Table II. Results for K_2SO_4 in Propellants

	K_2SO_4 Found (%)	
Propellant	Tetraphenylboron Method (6)	Atomic Absorption
No. 1 (IMR 5010, Lot 26226)	0.66	0.70
		0.72
		0.70
		0.69
		0.70
		Avg. 0.70
		Std. Dev. 0.011
No. 2 (HPC-13, Lot 6)	0.36	0.39
		0.40
		0.41
		0.41
		0.40
		Avg. 0.40
		Std. Dev. 0.009
No. 3 (SR 7641)	0.10	0.12
		0.11
		0.11
		0.11
		0.11
		Avg. 0.11
		Std. Dev. 0.005
No. 4 (DA 67, Lot 548)	0.91	0.90
		0.92
		0.93
		0.91
		0.93
		Avg. 0.92
		Std. Dev. 0.013

Table III. Results for KNO_3 in Propellants

	KNO_3 Found (%)		
Propellant	Tetraphenylboron Method (6)		Atomic Absorption
No. 5 (NNP-160, NL5089)	1.60		1.81
			1.78
			1.81
			1.77
			1.78
		Avg.	1.79
		Std. Dev.	0.018
No. 6[a] (HES 5250.70)	0.78		0.79
			0.79
			0.77
			0.78
			0.80
		Avg.	0.79
		Std. Dev.	0.012
No. 7[b] (M-2)	0.82		0.83
			0.81
			0.83
			0.82
			0.81
		Avg.	0.82
		Std. Dev.	0.010

[a] Contains 1.43% $Ba(NO_3)_2$ (See Table VI).

[b] Contains 1.46% $Ba(NO_3)_2$ (See Table VI).

Table IV. Results for Na_2SO_4 in Propellants

	Na_2SO_4 Found (%)	
Propellant [a]	Morpholine Method (4)	Atomic Absorption
No. 8 (WC 818, AL 45362)	0.11	0.051
		0.051
		0.052
		0.051
		0.051
		Avg. 0.051
		Std. Dev. 0.001
No. 9 (WC 818, LC 12641)	0.12	0.059
		0.060
		0.059
		0.059
		0.059
		Avg. 0.059
		Std. Dev. 0.001
No. 10 (WC 846, FA 133)	0.11	0.069
		0.069
		0.068
		0.067
		0.069
		Avg. 0.068
		Std. Dev. 0.010
No. 11 (WC 846, FA 172)		0.048
		0.047
		0.049
		0.048
		0.047
		Avg. 0.048
		Std. Dev. 0.009

[a] All samples contain approx. 0.6% $CaCO_3$ (See Table V).

Table V. Results for $CaCO_3$ in Propellants

Propellant [a]	Morpholine Method (4)	Atomic Absorption
No. 8 (WC 818, AL 45362)	0.63	0.64
		0.64
		0.64
		0.64
		0.65
		Avg. 0.64
		Std. Dev. 0.001
No. 9 (WC 818, AL 12641)	0.62	0.65
		0.65
		0.66
		0.65
		0.65
		Avg. 0.65
		Std. Dev. 0.001
No. 10 (WC 846, FA 133	0.62	0.65
		0.64
		0.64
		0.64
		0.65
		Avg. 0.64
		Std. Dev. 0.007
No. 11 (WC 846, FA 172		0.59
		0.59
		0.58
		0.58
		0.57
		Avg. 0.58
		Std. Dev. 0.009

[a] All samples contain approx. 0.10% Na_2SO_4 (See Table IV).

Table VI. Results for $Ba(NO_3)_2$ in Propellants

	$Ba(NO_3)_2$ Found (%)		
Propellant	Gravimetric Method (3)		Atomic Absorption
No. 12[a] (SR 4990, Lot 79)	3.58		3,43
			3.43
			3.38
			3.38
			3.43
		Avg.	3.41
		Std. Dev.	0.027
No. 6[b] (HES 5250.70)	1.32		1.45
			1.41
			1.45
			1.41
			1.43
		Avg.	1.43
		Std. Dev.	0.020
No. 13[c] (HES 5250.73)	1.38		1.47
			1.41
			1.43
			1.45
			1.47
		Avg.	1.45
		Std. Dev.	0.027
No. 7[d] (M-2)	1.38		1.45
			1.45
			1.47
			1.45
			1.47
		Avg.	1.46
		Std. Dev.	0.011

[a] Contains 0.76% KNO_3.

[b] Contains 0.78% KNO_3 (See Table III).

[c] Contains 0.85% KNO_3.

[d] Contains 0.82% KNO_3 (See Table III).

Table VII. Results for Recovery of Added K_2SO_4[a]

Added (gram)	K_2SO_4 Found (gram)	Recovery (%)
0.0030	0.0031	103.3
0.0050	0.0052	104.0
0.0060	0.0057	95.0
0.0080	0.0082	102.5
0.0100	0.0105	105.0
		Avg. 102.0

[a] The K_2SO_4 was added to propellant No. 8 which contains 0.051% Na_2SO_4 (see Table IV) and 0.64% $CaCO_3$ (see Table V).

Table VIII. Results for Recovery of Added Na_2SO_4[a]

Added (gram)	Na_2SO_4 Found (gram)	Recovery (%)
0.00050	0.00052	104.0
0.00100	0.00105	105.0
0.00150	0.00151	100.7
0.00200	0.00208	104.0
		Avg. 103.4

[a] The sodium sulfate was added to sample No. 1 which contains 0.70 K_2SO_4 (see Table II).

Table IX. Results for Recovery of Added $CaCO_3$[a]

Added (gram)	$CaCO_3$ Found (gram)	Recovery (%)
0.00100	0.00103	103.0
0.00300	0.00303	101.0
0.00400	0.00406	101.5
0.00600	0.00619	103.2
0.00700	0.00713	101.9
		Avg. 102.1

[a] The $CaCO_3$ was added to sample No. 1 which contains 0.70% K_2SO_4 (see Table II).

Table X. Results for Recovery of Added $Ba(NO_3)_2$[a]

Added (gram)	$Ba(NO_3)_2$ Found (gram)	Recovery (%)
0.0100	0.0100	100.0
0.0200	0.0202	101.0
0.0300	0.0297	99.0
0.0400	0.0400	100.0
0.0500	0.0500	100.0
		Avg. 100.0

[a] The $Ba(NO_3)_2$ was added to sample No. 5 which contains 1.79% KNO_3 (see Table III).

VII. APPENDIX

DIRECTIONS FOR OPERATING PERKIN-ELMER MODEL 303 ATOMIC ABSORPTION SPECTROPHOTOMETER

Make certain that the burner body shield is fastened securely to the mounting bracket and that the hold-down cable assembly is connected to the burner body shield. The safety glass shield should be in place. Determine that there is water in the drain tube trap and that the end of the drain tube is in water.

Set the SLIT Control for the recommended slit width.

Make sure the SOURCE Control is at zero.

Install the recommended lamp source.

Set the POWER Switch to ON.

Set the SOURCE Control to obtain the recommended current.

Allow the instrument to warm up for 15 to 20 minutes.

Set the RANGE Switch to Visual.

Set the FILTER to IN.

Adjust the coarse WAVELENGTH Control to obtain the correct indication on the WAVELENGTH Counter.

Turn the fine WAVELENGTH Control very slowly to obtain the maximum needle deflection to the right on the ENERGY Meter. While tuning with the WAVELENGTH Control, adjust the GAIN Control to center the ENERGY Meter needle in the black region on the scale.

Set the Burner Regulator Air Control to obtain an air pressure gauge indication of 30 psig. Adjust the Auxiliary Air Control to provide an Air Flow Meter reading of 9.0.

Set the fuel supply (Acetylene) pressure regulator to obtain a gauge reading of 8 psig. Use the quick shut-off switch to turn fuel on and off.

Light the burner after full fuel flow is established.

Set the Burner Regulator Fuel Control to obtain the Fuel Flow Meter reading of 9.0.

Allow five minutes warmup of the burner system before the first aspiration.

Set the SCALE Switch to obtain the desired expansion of the Absorption Scale.

Set the METER RESPONSE Switch as required.

Insert the burner-nebulizer sample tubing into water. Set the ABSORPTION Counter at 000 and while aspirating, turn the ZERO Control until the NULL Meter needle indicates null (mid-scale). If it is not convenient to set the ABSORPTION Counter at 000, press the zero check button while setting null.

Remove the water and insert the sample tubing into the sample solution or a standard solution. While aspirating, set the NULL Meter needle at null (mid-scale) by turning the ABSORPTION Control. Read the ABSORPTION Counter to determine percent absorption of the solution. Take an average of 3 readings. Obtain the percent absorption of all the standard solutions in similar manner. Keep sample tubing in water between all determinations.

Refer to the table (2) to convert percent absorption to absorbance.

To extinguish the flame, shut off the fuel supply to the burner-nebulizer, by closing main valve of fuel tank first, then the valve to the gauge on the tank and finally the quick shut-off switch. This allows complete bleeding of the line. Shut off the air supply last.

Set the POWER Switch to OFF. Set SOURCE at zero.

Notes on the operation:

1. Always turn Air on first when starting. To shut down turn Fuel off first and Air last.
2. Wear safety glasses when working in the neighborhood of the flame.
3. Never leave the flame unattended.
4. Never aspirate a solution with the flame off.

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13. ABSTRACT

>Atomic absorption methods are proposed for the determination of potassium, sodium, calcium, and barium in nitrocellulose-base propellants. A 1-gram sample is treated with a mixture of 10 ml of nitric acid and 3 ml of perchloric acid and the solution is evaporated to a volume of approximately 1 ml. The solution is then diluted to an appropriate volume (usually 500 ml for potassium or 100 ml for sodium, calcium, and barium) and the absorption is determined with an atomic absorption spectrophotometer. There are no significant interferences from the amounts and ratios of salts found in nitrocellulose-base propellants. The accuracy of the methods was checked with actual and synthetic propellants. The proposed methods are more reliable and considerably more rapid than the wet chemical methods presently used for the determination of inorganic salts in nitrocellulose-base propellants.

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