

NATO UNCLASSIFIED
NORTH ATLANTIC TREATY ORGANIZATION
ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

MILITARY AGENCY FOR STANDARDIZATION (MAS)
BUREAU MILITAIRE DE STANDARDISATION (BMS)
1110 BRUSSELS

Tel : 707.55.76
Fax : 707.57.18

MAS/148-PCS/4299
23 May 1996

To : See MAS Distribution List No. 2

Subject : STANAG 4299 PCS (EDITION 1) - SPECIFICATION AMMONIUM PERCHLORATE (NH₄ClO₄) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

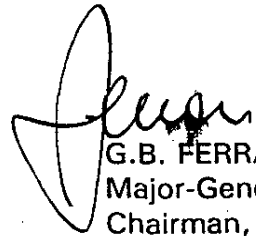
Reference : AC/310-D/107 dated 27 January 1993

Enclosure : STANAG 4299 (Edition 1)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.
2. The reference listed above is to be destroyed in accordance with local document destruction procedures.
3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

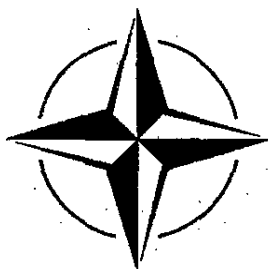
4. National staffs are requested to examine page iii of the STANAG and if they have not already done so, to advise the Defence Support Division, IS, through their national delegation as appropriate of their intention regarding its ratification and implementation.


G.B. FERRARI
Major-General, ITAF
Chairman, MAS

NATO UNCLASSIFIED

STANAG 4299
(Edition 1)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)



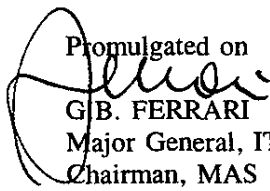
MILITARY AGENCY FOR STANDARDIZATION

(M A S)

STANDARDIZATION AGREEMENT

SUBJECT: SPECIFICATION AMMONIUM PERCHLORATE (NH_4ClO_4) FOR DELIVERIES
FROM ONE NATO NATION TO ANOTHER

Promulgated on 23 May 1996


G.B. FERRARI
Major General, ITAF
Chairman, MAS

NATO UNCLASSIFIED

STANAG 4299

(Edition 1)

RECORD OF AMENDMENTS

N o	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTESAGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.
2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.
3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "The declaration by which a nation formally accepts the content of this Standardization Agreement".
5. Implementation is "The fulfilment by a nation of its obligations under this Standardization Agreement".
6. Reservation is "The stated qualification by a nation which describes that part of this Standardization Agreement which it cannot implement or can implement only with limitations".

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page (iii) gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page (iv) (and subsequent) gives details of reservations and proprietary rights that have been stated.

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)

SPECIFICATION AMMONIUM PERCHLORATE (NH_4ClO_4)
FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

- Annexes: A. Physical and Chemical Requirements for Type 1 NH_4ClO_4 for use in Composite Rocket Propellants (Table I)
- B. Physical and Chemical Requirements for Type 2 NH_4ClO_4 for use in High Explosives
- C. Physical and Chemical Requirements for Type 3 NH_4ClO_4 for use in Pyrotechnics
- D. Tests Procedures

Related Documents: None

AIM

1. The aim of this agreement is to ensure that ammonium perchlorate shall possess properties which make it suitable for military use and to provide, within NATO, an acceptable basis for the production and certification of ammonium perchlorate for general use.

AGREEMENT

2. Participating nations agree that ammonium perchlorate Types 1, 2 and 3, proposed for military use, shall meet the physical and chemical requirements of Table I (Annex A), Table II (Annex B) or Table III (Annex C) respectively. The test procedures used to verify the requirements of Tables I, II, and III are described in Annex D, paragraphs 1 through 21.
3. The material shall be in the form of a white, free flowing powder complying with the particle size and/or shape specified in the contract or order. Unless the contract or order state otherwise the material shall be free from anti-caking agents.

NATO UNCLASSIFIED

GENERAL

4. This STANAG gives the detailed requirements for ammonium perchlorate, as described, for use in composite rocket propellants, high explosives, and pyrotechnics.
5. The purchaser shall specify the inclusion of an anti-caking agent and the shape and particle size requirements for the ammonium perchlorate in his contract or order. For ammonium perchlorate containing anti-caking agent, the specification values (i.e., sulfated ash, pH, sodium, potassium, calcium, and water insoluble, if the anti-caking agent is TCP) must be adjusted upward consistent with the percent of anti-caking agent permitted by the contract or order.
6. A lot shall consist of the total quantity of cross-blended material. For noncross-blended material a lot shall consist of the quantity produced in a single batch, or when manufactured by a continuous process a lot shall consist of the total quantity offered for acceptance at one time.
7. A representative sample of each lot shall be selected for testing. A minimum of two 150 gram random samples will be used.
8. In paragraph 21, Annex D, a method is described for the determination of tricalcium phosphate (TCP) which is used by some suppliers as an anti-caking agent.
9. The requirements for lead and lead compounds, nitrate, chromate, ether solubles, loss in weight, color, stability, and grit may be waived at the discretion of the purchaser.
10. Alternate analytical methods consistent with the advances in technology and subject to acceptance by the purchaser may also be used provided the results can be shown to be equivalent to the specification method.

IMPLEMENTATION OF THE AGREEMENT

11. This STANAG is implemented when a nation has issued the necessary orders/ instructions putting the contents of this agreement into effect.

Table 1
Physical and Chemical Requirements for Type 1
 NH_4ClO_4 for Use in Composite Rocket Propellants

Requirements	Values (weight percent)
Purity	≥ 99.0
Chloride (as NH_4Cl)	≤ 0.1
Chlorate (as NH_4ClO_3)	≤ 0.02
Bromate (as NH_4BrO_3)	≤ 0.004
Sulfated Ash	≤ 0.15
H_2O -Insolubles	≤ 0.03
Moisture; total	≤ 0.05
Moisture; surface	≤ 0.02
pH	$\geq 4.3 \leq 5.8$
Sulfate (as $(\text{NH}_4)_2\text{SO}_4$)	≤ 0.20
Iron (as Fe_2O_3)	≤ 0.0035
Total Amount of Sodium, Potassium, and Calcium	≤ 0.08
* Chromate (as K_2CrO_4)	≤ 0.015
* Nitrate (as NH_4NO_3)	≤ 0.001
* Total Amount of Lead and Lead Compounds	≤ 0.005
* Ether solubles	≤ 0.01
* Loss in weight at 177°C	≤ 0.04
* Color	not darker than color of standard
* Grit (retained on a $63\text{ }\mu\text{m}$ sieve)	≤ 0.01
* Grit (retained on a $250\text{ }\mu\text{m}$ sieve)	nil
* Stability	solution colorless after 3 hours

* may be waived at the discretion of the purchaser

Table II

Physical and Chemical Requirements for Type 2 NH_4ClO_4
For Use in High Explosives

Requirements	Values (weight percent)
Purity	≥ 99.5
Chloride (as NH_4Cl)	≤ 0.01
Chlorate (as NH_4ClO_3)	≤ 0.01
Bromate (as NH_4BrO_3)	≤ 0.005
Sulfated Ash	≤ 0.25
H_2O insoluble	≤ 0.05
Moisture; total	≤ 0.05
pH	$\geq 4.5 \leq 6.5$
Sulfate (as $(\text{NH}_4)_2\text{SO}_4$)	≤ 0.12
Iron (as Fe_2O_3)	≤ 0.007
*Nitrate (as NH_4NO_3)	≤ 0.001
*Total Amount of Lead and Lead Compounds	≤ 0.03
*Grit (retained on a 63 μm sieve)	≤ 0.01
*Grit (retained on a 250 μm sieve)	nil
*Stability	Solution colorless after 3 hours

* may be waived at the discretion of the purchaser

Table III

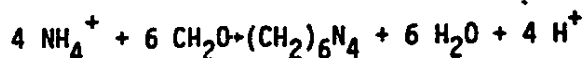
Physical and Chemical Requirements for Type 3 NH_4ClO_4
for Use in Pyrotechnics

Requirements	Values (weight percent)
Purity	≥ 99.5
Chloride (as NH_4Cl)	≤ 0.2
Chlorate (as NH_4ClO_3)	≤ 0.01
Bromate (as NH_4BrO_3)	≤ 0.005
Sulfated Ash	≤ 0.25
H_2O insoluble	≤ 0.05
Moisture; total	≤ 0.2
pH	$\geq 4.5 \leq 6.5$
Sulfate (as $(\text{NH}_4)_2\text{SO}_4$)	≤ 0.12
Iron (as Fe_2O_3)	≤ 0.007
* Nitrate (as NH_4NO_3)	≤ 0.001
* Total Amount of Lead and Lead Compounds	≤ 0.03
* Grit (retained on a 63 μm sieve)	≤ 0.01
* Grit (retained on a 250 μm sieve)	nil
* Stability	Solution colorless after 3 hours

* may be waived at the discretion of the purchaser

TEST PROCEDURES1. Determination of PurityPrinciple of Determination

Formaldehyde polymerizes in the presence of NH_4^+ ion to urotropine according to the equation:



The hydrogen ions are titrated with alkali.

Weight of Sample

$\text{NH}_4\text{ClO}_4 = 0.5 \text{ g}$

Reagents

Formaldehyde solution GR (min. 37 %) stabilized with 10 % CH_3OH (p.a. Merck)

1 : 1 in H_2O

Adjust the pH of the formaldehyde solution to a value of pH = 8.5 using 0.1 N sodium hydroxide.

Test Apparatus

200 ml stoppered Erlenmeyer flask

50 ml burette

pH-Meter

Procedure

Weigh a sample of 0.5 g NH_4ClO_4 (in dried condition) in an Erlenmeyer flask. Add 40 ml H_2O and prepare a water solution of the sample. Add 40 ml formaldehyde solution. Stopper the flask, shake vigorously, and allow to stand for 30 minutes. The solution is then titrated with 0.1 N sodium hydroxide to a pH of 8.5 with the help of a pH-meter. A double determination is necessary and the average is calculated and reported.

Calculation

$$\text{Percentage } \text{NH}_4\text{ClO}_4 = \frac{F \times A \times N}{E}$$

F = factor for NH_4ClO_4 = 11.75

A = ml 0.1 N NaOH

E = weight of sample (g)

N = normality of NaOH

2. Determination of Chloride (as NH_4Cl)

Principle of Determination

The determination is done by the Volhard Method by precipitation with an excess of silver nitrate (AgNO_3) and retitration of unreacted AgNO_3 .

Reagents

0.1 N Silver nitrate

Iron (III) ammonium sulfate indicator.

(The indicator solution consists of a cold saturated solution of iron (III) ammonium sulfate; for example: 200 ml H_2O , 110 g salt, 10 ml HNO_3).

Nitrobenzene p.A (or reagent grade)

0.1 N Ammonium thiocyanate (NH_4SCN)

Nitric acid 65 % diluted 1:1 with water.

Test Apparatus

Beaker 250 ml

Magnetic stirrer

50 ml Burette

Procedure

In a 250 ml beaker, dissolve 15 g of the sample in 150 ml distilled water. Under stirring add exactly 20 ml of 0.1 N AgNO_3 solution. Thereafter, add 5 ml of the indicator solution, 2 ml nitrobenzene, and 5 ml HNO_3 .

The mixture is titrated to the endpoint (light brown) with ammonium thiocyanate solution. A blank determination should be carried out on the equivalent amounts of reagents used in the test.

Calculation

$$\text{Percentage Chloride as } \text{NH}_4\text{Cl} = \frac{F (Aa - Bb)}{E}$$

A = total amount of AgNO_3 solution (ml)

B = amount of 0.1 N NH_4SCN used (ml)

a = normality of AgNO_3

b = normality of NH_4SCN

E = weight of sample (g)

F = factor for $\text{NH}_4\text{Cl} = 5.35$

3. Determination of Chlorate (as NH_4ClO_3)

Principle of Determination

Reduction of NH_4ClO_3 and precipitation as AgCl .

Reagents

a) Iron (II) sulfate solution:

Dissolve 176 g Fe (II) sulfate in 400 ml of distilled water in a 1000 ml stoppered volumetric flask. Add 500 ml of sulfuric acid (1:1) cautiously. Allow to cool and fill up to the mark with distilled water.

250 ml are necessary for three determinations.

Filter the solution before use.

b) 10 % Silver nitrate solution

c) 65 % Nitric acid solution

Procedure

Weigh accurately 20 g of the sample into a 400 ml beaker.

Dissolve in 100 ml distilled water and add 10 ml of a 10 % silver nitrate solution. Filter through a filter of medium porosity into a 600 ml beaker.

Wash with water very carefully. After the addition of 50 ml of iron (II) sulfate solution to the filtrate cover the beaker and allow to boil on a hot plate for 15 minutes. Remove the beaker from the hot plate and allow to cool for 15 minutes. Add 10 ml of nitric acid while stirring. Allow to boil an additional 5 minutes and cool to room temperature.

The solution is filtered through a glass filter crucible (Grade No. 4).

The precipitate is washed with dilute nitric acid and then with distilled water. Dry to constant weight in an oven at 135°C.

Cool in a desiccator and weigh.

Calculation

$$\text{Percentage Chlorate as } \text{NH}_4\text{ClO}_3 = \frac{F \times B}{E}$$

B = weight of the precipitate (g)

E = weight of sample (g)

F = factor for NH_4ClO_3 = 70.8

4.

Determination of Nitrate (as NH_4NO_3)**Principle of Determination**

Zinc metal and acetic acid are added to a solution of the sample.

Any nitrate present is reduced to nitrous acid, which is used to diazotize sulphanilic acid. The diazo compound is then reacted with 1-naphthylamine-7-sulphonic acid (Cleve's acid) to form an azo compound, which is measured colorimetrically.

Reagents

- a) Sulphanilic acid solution: Add 30 ml of glacial (17M) acetic acid to 70 ml of water and dissolve 1 g of sulphanilic acid in the mixture, warmed as necessary.

- b) Cleve's acid solution: Dissolve 0.5 g of Cleve's acid (1-naphthylamine-7-sulphonic acid) in 120 ml of water warming in a water bath. Filter and cool the solution. Then add 30 ml of glacial (17M) acetic acid. Store in a brown bottle.
- c) Standard nitrate solution: Dissolve 1.00 g of ammonium nitrate in distilled water in a 100 ml graduated flask and fill up to the mark with distilled water.

Transfer 1.0 ml of the solution to a 1 liter graduated flask and fill up to the mark with water. This solution must be freshly prepared.

$$\begin{aligned} 1 \text{ ml} &= 0.01 \text{ mg of } \text{NH}_4\text{NO}_3 \\ &= 0.001 \% \text{ } \text{NH}_4\text{NO}_3 \text{ for 1 g of sample.} \end{aligned}$$

- d) "Nitrate free" ammonium perchlorate: "Laboratory reagent" grade ammonium perchlorate, available from BDH Limited, or equivalent, has been found suitable for this purpose.

Procedure

Dissolve 1 ± 0.1 g of sample in 30 ml of water in a 50 ml Nessler tube. Prepare working standards by dissolving 1 ± 0.1 g of "nitrate-free" ammonium perchlorate in 30 ml of water in each of five similar Nessler tubes. Add, each to a separate cylinder, 1, 2, 3, 4, and 5 ml of the standard nitrate solution, then treat these and the sample solution in the same manner.

Ensure that the temperature of the solutions is below 15°C during the remainder of the procedure. Add to each solution about 0.1 g of zinc dust and 2 ml of glacial acetic acid, and mix thoroughly. Add 2.0 ml of the sulphanilic acid solution and 2.0 ml of the Cleve's acid solution. Fill up to the mark with water, mix thoroughly, and allow the solutions to stand for 15 minutes.

Compare the intensities of the color, if any, of the sample solution with those of the standards.

Calculation

$$\text{Percentage Nitrate as } \text{NH}_4\text{NO}_3 = n \times 0.001$$

where n = volume in ml of the standard nitrate solution in the working standard solution which matches the sample solution.

5. Determination of Bromate (as NH_4BrO_3)Principle of Determination

NH_4ClO_4 is dissolved in distilled water. Potassium iodide, diluted hydrochloric acid, and starch are added. An equivalent of iodine is liberated and titrated with sodium thiosulfate solution.

Reagents

Hydrochloric acid 1:4

Sodium thiosulfate solution 0.01 N

Potassium iodide 10 g/100 ml

Starch solution 1 g/100 ml H_2O

Test Apparatus

Erlenmeyer flask 250 ml

Procedure

Weigh 20 g of the sample accurately to the nearest 0.01 g and place it in a 250 ml Erlenmeyer flask. Add 150 ml of distilled water and dissolve by shaking. Add 0.5 g of potassium iodide, 5 ml of hydrochloric acid 1:4, and 5 ml of the starch solution. Shake the stoppered flask and allowed to stand in darkness for one hour. Open the flask and wash the walls with distilled water.

The liberated iodine is titrated with 0.01 N sodium thiosulfate to the point where the blue color disappears (± 0.01 ml burette reading).

A blank determination should be carried out on the equivalent amounts of reagents used in the test.

Calculation

$$\text{Percentage Bromate as } \text{NH}_4\text{BrO}_3 \text{ (incl. } \text{NH}_4\text{IO}_3) = \frac{(V_1 - V_2) \times N \times 2.43}{E}$$

N = normality of sodium thiosulfate solution

V_1 = used sodium thiosulfate solution (ml)

V_2 = used sodium thiosulfate solution for blank (ml)

E = weight of sample (g)

6. Determination of Sulfated Ash

Sulfated ash shall be determined by the following method. Transfer 5.0 g of dried, finely ground sample to a previously dried and tared silica crucible. Cover. Heat carefully with a Bunsen flame until decomposition occurs. Continue the ignition of small increments until the entire sample has decomposed and the volatile salts have been driven off. Allow the crucible to cool. Add 3 ml of concentrated sulfuric acid, heat again, and complete the volatilization of sulfuric acid at a dull red heat. Cool in a desiccator and weigh. Calculate the percentage of sulfated ash in the specimen as sodium sulfate by the following formula:

$$\text{Percentage ash} = \frac{W \times 100}{E}$$

W = weight of residue (g)

E = weight of sample (g)

(The material from the sulfated ash determination should be retained for the determination of iron)

7. Determination of Water Insolubles

Dissolve 25 g of the sample in 175 ml of distilled water and filter the solution through a tared crucible of fine porosity (Grade No. 4). Wash with distilled water, dry for 1.5 hours at 105 °C, cool, and weigh. Calculate the percentage of water insolubles as follows:

$$\text{Percentage water insolubles} = \frac{W \times 100}{E}$$

W = weight of residue (g)

E = weight of sample (g)

8. Determination of Total Moisture

Total moisture shall be determined in accordance with ASTM E 203-75, or equivalent, using 1:3 methanol/pyridine as the carrier.

9. Determination of Surface Moisture

Surface moisture shall be determined in accordance with ASTM E 203-75, or equivalent, if a direct titration is used, or ASTM D 1348-61, or equivalent, if a back titration method is used. In either case a 1:1 methanol/toluene mixture shall be used as the carrier solution.

10. Determination of pH

Add 50 ml of hot, freshly-boiled distilled water to 20.0 g of sample. Stir and cool to room temperature (20 to 25°C) and allow the precipitate to settle. During the cooling period cover the beaker and introduce dry CO₂-free nitrogen gas. Measure the pH on a suitable potentiometer having glass calomel electrodes.

11. Determination of Chromate (as K₂CrO₄)

Dissolve 1 g of sample weighed to the nearest 0.1 mg in approximately 95 ml of water, filter, and dilute filtrate in a Nessler tube to 100.0 ml. Add 3.0 ml of a solution containing one part concentrated sulfuric acid and five parts water. Mix. Add 1.0 ml of 0.25 % diphenyl carbazide solution in alcohol and mix. Prepare a blank in the same manner and add standard (0.0004 g/ml) potassium chromate solution until the color matches that of the sample solution. Calculate the percentage of chromates in the specimen as potassium chromate by the following formula:

$$\text{Percentage Chromate as K}_2\text{CrO}_4 = \frac{0.04 A}{W}$$

where

- A = volume of standard potassium chromate solution added to blank (ml)
W = weight of sample (g)

12. Determination of Sulfate (as $(\text{NH}_4)_2\text{SO}_4$)Procedure

Dissolve $10 \text{ g} \pm 0.1 \text{ g}$ of sample in distilled water in a 400 ml beaker. Add 2 ml of concentrated hydrochloric acid, and heat to boiling. Add slowly 10 ml of barium chloride solution (100 g/liter), stirring continuously during the addition. Place the beaker and contents in a boiling water bath to digest for about 30 minutes, then allow to stand overnight. Filter off any precipitate on a sintered glass crucible (Grade No. 4) and wash thoroughly with water. Dry in an oven at $120 \pm 2^\circ\text{C}$ for one hour. Cool in a desiccator and weigh.

Calculation

$$\text{Percentage Sulfate as } (\text{NH}_4)_2\text{SO}_4 = (W_2 - W_1) \times 5.66$$

W_1 = sintered glass crucible

W_2 = sintered glass crucible and precipitate

13. Determination of Iron (as Fe_2O_3)Reagents

Hydrochloric acid, concentrated, American Chemical Society (ACS) reagent or equivalent
Nitric acid, concentrated, ACS reagent or equivalent
Sulfuric acid, concentrated, ACS reagent or equivalent
Ammonium thiocyanate, 1 % solution of ACS reagent or equivalent
Iron wire, ACS reagent or equivalent
Potassium permanganate (KMnO_4) 1 % solution of ACS reagent or equivalent

Apparatus

50 ml Nessler tubes

1. Weigh accurately 0.07 g of bright iron wire. (An equivalent weight of any other recognized primary standard, with appropriately modified preparation procedure, may be substituted for the iron wire standard).

Dissolve the iron wire in 5 ml of concentrated hydrochloric acid. Transfer the solution to a 1 liter volumetric flask and dilute to volume. This is the standard solution. To the ash from the sulfated ash procedure, as specified in 6., add 1 ml of concentrated hydrochloric acid and 1 ml of concentrated nitric acid. Transfer to a 50 ml beaker with a minimum of distilled water and boil to dryness on a hot plate. Moisten the residue with 2 drops of concentrated hydrochloric acid, and wash with distilled water into a 100 ml glass-stoppered volumetric flask. For Type 1 NH_4ClO_4 (Table I) accurately transfer a 1.75 ml portion of the standard solution to a second 100 ml glass-stoppered volumetric flask. For Type 2 and 3 NH_4ClO_4 (Tables II and III) a 3.5 ml portion shall be transferred. Add 2.0 ml of concentrated sulfuric acid, 2 drops of potassium permanganate solution, and 10 ml of 1 % ammonium thiocyanate solution to each flask. Dilute to volume, mix well, and transfer to 50 ml Nessler tubes. The sample shall be no darker than the standard.

14. Determination of Sodium, Potassium, and Calcium by Flame Spectrophotometry

Preparation of reagents

Quantitatively transfer 1.9066 g of potassium chloride, 2.5416 g of sodium chloride and 2.7692 g of calcium chloride into separate 1 liter volumetric flasks containing approximately 250 ml of water. Swirl to dissolve the salt and dilute to volume with water. Each solution contains 1 mg/ml of cation. From these stock solutions transfer aliquots to other volumetric flasks and dilute with water so that a least four standard solutions are prepared in the range 0.005 to 0.03 mg/ml for the potassium ion, 0.001 to 0.005 mg/ml for the sodium ion, and 0.008 to 0.04 mg/ml for the calcium ion.

Preparation of standard curves

Determine the net emission in terms of percentage of transmittance of the standard solutions by flame photometry using a wavelength of 766.5 nanometers (nm) for the potassium ion, 589 nm for the sodium ion and 422.7 nm for the calcium ion. The minimum practical slit-width should be used. Prepare a graph either by plotting

absorbance versus concentration on linear coordinates or percentage of transmittance versus concentration on semilog coordinates. Fit a straight line to the points.

Analysis of sample

Quantitatively transfer 4.0 g of sample, weighed to the nearest mg, into a 100 ml volumetric flask. Dissolve in distilled water and dilute to volume. Determine by plotting the percentage of transmittance of each standard solution and the sample solution. Read the concentration of the sample directly from the standard curve.

Calculation

Calculate the potassium, sodium, and calcium content as follows:

$$\text{Percentage of potassium, sodium, and calcium} = 10 \times \frac{R_K + R_{Na} + R_{Ca}}{W}$$

where

- R_K = potassium concentration as read from standard curve (mg/ml)
- R_{Na} = sodium concentration as read from standard curve (mg/ml)
- R_{Ca} = calcium concentration as read from standard curve (mg/ml)
- W = weight of sample (g)

15.

Determination of Ether Solubles as Organic Material

Transfer 25 ± 0.1 g of the sample into an extraction thimble of a Soxhlet extraction apparatus. Extract with 100 ml of anhydrous ethyl ether for 2 hours. Transfer the ether extract to a tared Vycor dish and evaporate to dryness on a water bath. Run a blank determination on 100 ml of ether. The increase in weight minus the weight of the blank is calculated as organic material as follows:

$$\text{Percentage of ether solubles} = \frac{(W-B) \times 100}{E}$$

W = increase in weight (g)

B = weight of blank (g)

E = weight of sample (g)

16.

Determination of Loss in Weight at 177°C

Transfer a 10 g portion of the sample, weighed to the nearest 0.1 mg, to a tared wide-form moisture dish and weigh. Place in a gravity convection oven and allow to remain for 2 hours at $177^\circ \pm 2^\circ\text{C}$. Cool in a desiccator and weigh. Calculate the percentage of volatile matter as follows:

ANNEX D to
STANAG 4299
(Edition 1)

$$\text{Percentage of volatile matter} : \frac{(W_1 - W_2) 100}{S}$$

where

W_1 = weight of dish plus sample (g)
 W_2 = weight of dish plus sample after heating (g)
 S = weight of sample (g)

17.

Determination of Color

Dissolve a 10 g portion of sample in 100 ml of water and transfer 100 ml of solution to a Nessler tube. Compare with a standard containing 0.005 g of potassium chromate per liter. The sample shall be no darker than the standard.

18.

Determination of Grit

Definition

Grit is defined as inorganic matter insoluble in aqua regia and retained on the 63 micron and 250 micron sieves as described below.

Apparatus

Sieves 50 mm in diameter, aperture sizes 63 micron and 250 micron.

Procedure

Transfer 50 ± 0.5 g of the sample to a 600 ml beaker and add 200 ml of hot water. Stir for 2-3 minutes, allow undissolved solids to settle, and decant the solution carefully through a No. 42 Whatman filter paper. Repeat the operation with further 200 ml portions of hot water until no further solids dissolve.

Transfer the undissolved solids to the filter paper, wash thoroughly with hot water, and transfer the paper and contents to a 30 ml porcelain crucible. Dry the crucible and its contents in an oven at $103 \pm 2^\circ\text{C}$, then ignite at about 700°C for 30 minutes.

After cooling add 10 ml of aqua regia and boil gently in a fume cupboard for 10 minutes. Cool. Add 20 ml of water, and filter through a 7 cm No. 42 Whatman

filter paper, transferring the undissolved solids to the paper. Wash thoroughly with water, and dry. Ignite in the same crucible as previously described. Allow to cool, initially in air, finally in a desiccator, then transfer the residue to the 63 micron sieve, and brush lightly. Weigh the material retained (W).

Transfer the retained material to the 250 micron sieve, brush lightly, and record the number and nature of any particles retained.

Calculation and reporting

Grit retained on a 63 micron sieve, percentage = 2 W.

Report the number and nature of particles retained on the 250 micron sieve.

19.

Determination of Stability

For the determination of thermal stability transfer four 10 g portions of the sample to aluminium weighing dishes of 60 mm diameter which have previously been heated to approximately 200 °C and cooled. Place on shelf located 10 cm from top of gravity-convection oven. Maintain oven temperature at $177^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Remove one sample from the oven after 2 hours and an additional sample every hour thereafter. Cool the sample in a desiccator for 20 to 30 minutes. Add approximately 5 g of cooled sample to a mixture of 100 ml distilled water, 0.5 g granular potassium iodide, and 5 ml of 0.2 % starch solution. Stir to dissolve the ammonium perchlorate. The solution shall remain almost colorless. Immediate appearance of violet or blue color indicates decomposition. Repeat the procedure for each sample removed from the oven.

20.

Determination of Lead

Principle of Determination

The sample is treated with 2N hydrochloric acid to convert the lead and lead compounds to the chloride. The pH of the solution is adjusted to 5-7, and the lead is estimated turbidimetrically as the sulfide by comparison with known standards.

Reagents

- a. Standard lead solution: Dissolve 1.598 g of lead nitrate in water to make 1 liter of solution. Transfer 5.0 ml of this solution to a 250 ml graduated flask and make up to the mark with water.

$$\begin{aligned} 1 \text{ ml} &= 0.00002 \text{ g of lead} \\ &= 0.01 \% \text{ lead on 0.2 g of sample.} \end{aligned}$$

- b. Ammonium hydroxide solution: Dilute one volume of concentrated ammonia solution (18N) with three volumes of water.
- c. Ammonium acetate solution: Dissolve 10 g of ammonium acetate in water to make 100 ml of solution.
- d. Ammonium citrate solution: Dissolve 10 g of ammonium citrate in water to make 100 ml of solution.
- e. Potassium cyanide solution: Dissolve 1 g of potassium cyanide in water to make 10 ml of solution. CAUTION toxic material
- f. Sodium sulfide solution: Dissolve 1 g of sodium sulfide in water to make 10 ml of solution. This solution should be freshly prepared for each series of determinations.

Procedure

Transfer 10 ± 0.1 g of the sample to a 400 ml beaker, add 100 ml of approximately 2N hydrochloric acid, and warm on a water bath until solution of the sample is complete. Slowly add anhydrous sodium carbonate, while stirring, until a pH of 5-7 is attained (measured by test papers). If excess carbonate is added, neutralize with hydrochloric acid. Transfer the solution to a 250 ml graduated flask and make up to the mark with water.

Transfer 5.0 ml of the solution to a 50 ml Nessler tube.

Carry out the following procedure in a fume cupboard. Using a measuring cylinder, add the following reagents in the order given. Mix thoroughly after each addition.

- a. 10 ml of the ammonium acetate solution,
- b. 5 ml of the ammonium citrate solution,
- c. 2 ml of the ammonium hydroxide solution,
- d. 1 ml of the potassium cyanide solution, CAUTION toxic material
- e. Water to make the volume of the solution up to the 50 ml mark,
- f. 2 drops (approximately 0.1 ml) of the sodium sulfide solution.

Mix thoroughly, and compare the resultant color with that produced in Nessler tubes containing 0.5, 1.0, 2.0 and 3.0 ml respectively of standard lead solution, treated in the same way and at the same time. These solutions are equivalent to 0.005, 0.01, 0.02, 0.03 % lead respectively in the sample.

Report the percentage lead in the sample as indicated by comparison with the standards.

21.

Determination of Tricalcium Phosphate (TCP)

Principle of Determination

Determination of absorbance of the sample solution and reference from the calibration curve (Atomic Absorption Spectroscopy (ASS)).

Reagents

Potassium nitrate p.a.
Water soluble calcium salt p.a. (CaCO_3)
Hydrochloric acid (conc.)
Nitrous oxide gas
Acetylene gas

Apparatus

Atomic absorption spectrophotometer

Wavelength (nm): 422.7

Preparation of calibration curve

Calcium 500 micrograms/ml stock solution: To 1.249 grams (weigh to four significant figures) of primary calcium carbonate (CaCO_3) add 50 ml of distilled water. Add drop-wise a minimum volume of HCl (approximately 10 ml) to effect complete solution of the CaCO_3 . Dilute to 1 liter with distilled water. Dilute this stock solution to obtain 1, 2, and 4 mg/liter solutions.

Procedure

Dissolve 7 to 8 g of sample in distilled water using a 1 liter volumetric flask. Add 1 ml of HCl (1:2) and 5.12 g of KNO_3 and fill up to the mark. Measurements are carried out at the wavelength given under section Apparatus.

Calculation

$$\text{Percentage TCP} = \frac{\text{mg Ca} \times 2.58 \times 100}{E}$$

mg Ca = measured Ca concentration

2.58 = factor for calculation of Ca to TCP

E = sample weight (mg)