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DRAFT EAST AFRICAN STANDARD

Sodium sulfate, anhydrous — Specification

EAST AFRICAN COMMUNITY

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Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 069, *Organic and Inorganic chemicals.*

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

Sodium Sulphate, Anhydrous, for industrial use — Specification

1 Scope

This Draft East African Standard specifies requirements, sampling and test methods for sodium sulphate, anhydrous. for industrial use

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696 *Water for analytical laboratory use — Specification and test method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
high purity grade sodium sulphate
highly refined inorganic salt containing ≥99.0% active sodium sulphate and chemically defined by two sodium ions (Na^+) ionically bonded to one sulphate ion (SO_4^{2-}).

3.2
Technical grade sodium sulphate
industrial inorganic chemical compound defined by the chemical formula Na_2SO_4 (sodium salt of sulphuric acid) with the purity ranging from 85 % – 99%

4. Grade

Sodium sulphate shall be of the two grades:

i. High purity

ii. Technical

5 Requirements

5.1 General requirements

Sodium sulphate, anhydrous shall be in the form of white, free-flowing powder, free from visible impurities and shall be completely soluble in water.

5.2 Specific requirements

Sodium sulphate shall comply with the specific requirements given in Table 1 when tested in accordance with the test methods specified therein.

Table 1 — Specific requirements of sodium sulphate, anhydrous. for industrial use

S. N	Characteristics	Requirement		Test method.
		High purity	Technical	
i.	Sodium sulphate [a] (as Na2SO4), %m/m,	≥99	85-99	Annex A
ii.	Matter insoluble in water, [a] %m/m, max.	0.25	1	Annex B
iii.	Chlorides [a] (as NaCl), %m/m. max	0.3	2	Annex C
iv.	Iron, Aluminium and Chromium [a] (as M_2O_3), %m/m, max.	0.02	0.02	Annex D
v.	Iron [a] (as Fe), %m/m, max	0.002	0.002	Annex E
vi.	Loss on drying, %m/m, max	0.5	1	Annex F
vii.	pH (of 10 % solution)	7.0-7.5	7.5-8.0	Annex G

6 Packaging

Sodium sulphate, anhydrous shall be packaged in suitable well-sealed containers that shall protect the contents and shall not cause any contamination or react with the product

.7. Labelling

7.1 Each package shall be legibly and indelibly labelled in English and/or any other official language (French, Kiswahili, etc) used in the importing East African Partner State with the following information:

a) product name as “sodium sulphate, anhydrous”

b) name and physical address of the manufacturer;

c) country of origin;

d) recognized trade mark, if any

e) batch/lot number; and

f) manufacturing date.

g) net weight

h) expiry date

i) intended purpose

7.2 The product shall also be labelled with appropriate safety symbols as specified in Globally Harmonized System of Classification and Labelling of Chemicals

8. Sampling

Sampling shall be done in accordance with Annex H

Annex A
(normative)

Determination of sodium sulfate

A.1 Quality of reagents

Unless specified otherwise, analytical/reagent grade chemicals and water for analytical laboratory use ISO 3696 shall be used in tests.

A.2 Preparation of sample

Procedure – Crush 50 g of sample to pass through a 1.00 mm sieve. Dry to constant mass at 105 ± 5 °C to obtain the prepared sample and keep in a clean, glass-stoppered bottle in a desiccator for test purposes.

A.3 Determination of sodium sulfate (as Na2SO4)

A.3.1 Principle

Sodium sulfate is determined by precipitation with barium chloride solution and weighed as barium sulfate.

A.3.2 Reagents

A.3.2.1 Barium chloride solution – approximately 10 percent.

A.3.2.2 Dilute hydrochloric acid – approximately 4 N.

A.3.3 Procedure

A.3.3.1 Weigh about 0.2 g of the prepared sample and dissolve 100 mL of water. Filter to remove undissolved matters and wash the filter paper thoroughly. To the filtrate and washings, add 10 mL of dilute hydrochloric acid and boil. Add slowly to the hot solution a slight excess of hot barium chloride solution and continue boiling for 2 min to obtain a granular precipitate of barium sulfate. Allow to stand for 4 hours and filter through a tared sintered glass crucible (G No.4). Wash the precipitate thoroughly and dry at 105 to 110°C to constant mass.

A.3.3.2 An excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by the addition of barium chloride slowly with constant stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

A.3.3.4 Calculation

$$= \frac{60.86\,M}{M1}$$

where

M = mass in g of the dried precipitate, and

M_1 = mass in g of the material taken for the test.

Annex B
(informative)

Determination of matter insoluble in water

B.4 Determination of matter insoluble in water

B.4.1 Procedure

Weigh about 10 g of the prepared sample. Dissolve in 100 mL of water by warming, if necessary, and by stirring the solution. Filter through a tared filter paper or sintered crucible (G No.4). Wash thoroughly the residue till it is free from all soluble compounds and dry at 105 to 110 °C to constant mass.

B.4.2 Calculation

Matter insoluble in water, percent by mass = $\frac{100M}{M1}$

where,

M = mass in g of the residue, and

M1 = mass in g of the prepared sample taken for the test

Annex C
(normative)

Determination of chlorides (as NaCl)

C.5 Determination of chlorides (as NaCl)

C.5.1 Principle

Chlorides are determined by precipitating with silver nitrate solution and titrating the excess of silver nitrate solution with, standard ammonium thiocyanate solution using ferric alum as indicator

C.5.2 Reagents

C.5.2.1 Standard silver nitrate – 0.1 N

C.5.2.2 Concentrated nitric acid

C.5.2.3 Ferric alum indicator solution - saturated,

C.5.2.4 Standard ammonium thiocyanate solution – 0.1 N

C.5.2.5 Nitrobenzene

C.5.3 Procedure

Weigh about 5 g of the prepared sample and dissolve in 80 mL of water. Filter the residue, if any, through a folded filter paper and wash thoroughly with water, collect the filtrate and washings in a 250 mL conical flask. Add with a pipette 25 mL of standard silver nitrate solution, 2 mL of concentrated nitric acid and 10 mL, of nitrobenzene. Shake vigorously and add 2 mL of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

C.5.4 Calculation

Chlorides (as NaCl), percent by mass = $\frac{0.5846\,(25-V)}{M}$

where,

V = volume in mL of standard ammonium thiocyanate solution used in the titration, and

M = mass in g of the prepared sample taken for the test.

Annex D
(normative)

Determination of Iron, Aluminium and Chromium (as M_2O_3)

D.6 Determination of Iron, Aluminium and Chromium (as R2O3)

D.6.1 Principle - Iron, aluminium and chromium compounds are determined by precipitation with ammonium hydroxide.

D.6.2 Reagents

D.6.2.1 Ammonium chloride – Solid

D.6.2.2 Dilute Ammonium Hydroxide - approximately 4 N.

D.6.2.3 Ammonium Nitrate Solution - approximately 2 percent.

D.6.2.4 Dilute Hydrochloric Acid - approximately 4 N.

D.6.2.5 Conc. Nitric acid

D.6.3 Procedure

Weigh about 10 g of the prepared sample and dissolve in 150 mL of water. Acidify with dilute hydrochloric acid, add 2 to 3 drops of concentrated nitric acid, boil for 1 to 2 min, and then filter to remove undissolved matter. Wash the filter paper thoroughly and, to the hot solution add, first 3 to 5 g of ammonium chloride and then ammonium hydroxide solution till there is a faint permanent smell of ammonia (in order to precipitate completely the iron, aluminium and chromium as hydroxides). Boil the contents for 2 min and filter through Whatman filter paper No. 40 or equivalent. Wash with hot ammonium nitrate solution till free from chlorides. Dry the precipitate at 100 ± 2°C and ignite at 1100 °C in a tared crucible to constant mass.

D.6.3 Calculation

Iron, Aluminium and Chromium (M2O3), percent by mass = $\frac{100M1}{M2}$

where,

M1 = mass in g of the ignited residue, and

M2 = mass in g of the prepared sample taken for the test.

Annex E
(normative)

Analysis of sodium sulphate, anhydrous

E.7 Determination of iron (as Fe) by Colorimetric method

E.7.1 Principle – Iron is determined colorimetrically by visual comparison of the colour in Nessler cylinders, using-potassium thiocyanate.

E.7.2 Apparatus

Nessler cylinders – 50 mL capacity.

E.7.3 Reagents

E.7.3.1 Concentrated nitric acid

E.7.3.2 Ammonium persulfate – solid.

E.7.3.3 Butanolic potassium thiocyanate – Dissolve 10 g of potassium thiocyanate in 10 mL of water. Add sufficient n-butanol to make up to 100 mL and shake vigorously until the solution is clear.

E.7.3.4 Dilute sulfuric acid – approximately 10 percent (v/v).

Weigh 0.702 g of ferrous ammonium sulfate [FeSO4 (NH4) SO4.6H2O] and dissolve in 10 mL of dilute sulfuric acid. Dilute with water to make up the volume to 1000 mL. Transfer 10 mL of this solution and again dilute with water to make up the volume to 100 mL. 1 mL of this solution is equivalent to 0.01 mg of iron (as Fe).

E.7.4 Procedure

Weigh about 1 g of the prepared sample, dissolve it in water and make up the volume to 100 mL. Pipette out exactly 10 mL of this solution into a beaker, add 1 mL of nitric acid and boil. Cool, transfer the solution to a Nessler cylinder and add 30 mg of ammonium persulfate and 15 mL of butanolic potassium thiocyanate solution. Shake the contents vigorously for 30 s and then allow the liquid to separate. Carry out a control test in the other Nessler cylinder adding slowly from a burette a quantity of the standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture until the colours of butanol layer in the two cylinders are exactly matched.

E.7.5 Calculation

Iron (as Fe), percent by mass = $\frac{0.01V}{M}$

where,

V = volume in mL of standard iron solution required in the control test, and

M = mass in g of the material taken for the test

Annex F
(normative)

Determination of loss on drying

F.8 Determination of loss on drying

F.8.1 Procedure

Weigh about 5 g of the crushed material into a tared dish and dry at 105 ± 5°C. Cool and weigh repeatedly till a constant mass is, obtained.

F.8.2 Calculation

Loss on drying, percent by mass = $\frac{M1-M2}{M1}100$

where,

M1= mass in g of the material taken for the test, and

M2 = mass in g of the residue obtained after drying.

Annex G
(normative)

Determination of pH

G.1 Principle

pH is measured using a pH-meter, which is sensitive to the hydrogen ions that define the pH value and a sensor that supplies the reference signal or potential for the pH sensor. These sensors can be separate or in combination. The difference in potential of both these electrodes is used to determine the pH value of the measured solution.

G.2 Materials and apparatus

G.2.1 pH meter, preferably equipped with a combined pH glass electrode

G.2.1 Analytical balance, well calibrated and maintained

G.2.1 Beaker, 250-mL

G.2.1 Glass rod

G.3 Reagents

G.3.1 Buffer solutions, buffer 4, buffer 7 and buffer 10

G.3.2 Distilled water

G.4 pH meter electrode calibration

A pH electrode shall be calibrated regularly with a recommended frequency of at least once daily before use.

G.5 Procedure

Weigh 5 g of the prepared sample and dissolve in 50 mL of distilled water. Determine the pH using the pH meter

Annex H
(informative)

Sampling of sodium sulphate, anhydrous

H.1 General requirements of sampling

In drawing, preparing, storing and handling test samples the following precautions and directions shall be observed.

H.1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

H.1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

H.1.3 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

H.1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

H.2 Scale of sampling

H.2.1 Lot – All the containers in a single consignment of the material of one grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

H.2.2 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

H.2.3 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

Table 2 — Number of containers to be selected

Lot Size	Number of containers to be selected
N	n
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

H-2.4 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used. In case such tables are not available, the following procedure may be

adopted: Starting from any container, count them as 1, 2, 3 .... up to r and so on in a systematic manner, where *r* is the integral part of *N/n.* Every *r*th container thus counted shall be taken out for drawing samples.

H-3 Test samples and referee sample

H-3.1 Preparation of Test Samples

H-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given in 4 and shall not exceed 1 kg.

H-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as reference sample.

H-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given in B-1.4. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as reference sample.

H-3.2 Reference Sample

The reference sample shall consist of the composite sample (*see* B-3.1.2) and a set of individual samples *(see* B-3.1.3) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place' agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

H-4. Number of tests

Tests for the determination of sodium sulphate shall be conducted on each of the individual samples for all the grades.

H-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

H-5 Criteria for Conformity

H-5.1 For individual samples

For sodium sulfate - The test results for sodium sulfate shall be recorded; the mean and the range for these test results calculated as follows:

Mean *(x)* = Sum of the test results divided by the number of test results, and Range *(R)* = Difference between the maximum and minimum values of test results. The value of expression *X* - 0.6 *R* shall be calculated. If the value of this expression is more than or equal to the limit specified for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirements for this characteristic.

H-5.2 For composite sample

The test results on the composite sample shall meet the corresponding requirements.

H-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics.

Bibliography

[1] TZS 2939: 2022, *Sodium sulfate, anhydrous — Specification*

[2] IS 255:2023, *Sodium Sulphate, Anhydrous (Technical Grade) — Specification*

