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DRAFT EAST AFRICAN STANDARD

Sulfuric acid — Specification

EAST AFRICAN COMMUNITY

Second Edition 2021

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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) and other deliverables. 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 and other deliverables 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.

This second edition cancels and replaces the first edition (EAS 122:1999), which has been technically revised.

Sulfuric acid — Specification

1 Scope

This Draft East African Standard specifies requirements, sampling and test methods for sulfuric acid. This standard covers four grades of sulfuric acid namely, technical, battery, pure and analytical reagents.

2 Normative references

The following referenced documents are indispensable for the application 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.

EAS 123, *Distilled water*

EAS 101, *Foodstuffs — Method of determination of arsenic*

3 Terms and definitions

No terms and definitions are listed in this document. 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**”**

4 Requirements

4.1 General requirements

4.1.1 Grades of sulfuric acid

4.1.1.1 There shall be four grades of the material, namely;

a) technical,

b) battery

c) pure and

d) analytical reagents

4.1.1.2 The battery grade acid shall have two types, namely, concentrated and dilute depending on the use.

4.1.2 Description

4.1.2.1 Technical grade
Sulfuric acid of technical grade shall be a liquid not darker than light brown in colour.

4.1.2.2 Battery grade concentrated and dilute
Sulfuric acid for battery grade shall be a colourless liquid. The concentrated acid on dilution with an equal volume of distilled water, and the dilute acid as received shall be free from suspended matter and other visible impurities.

4.1.2.3 Pure and analytical reagent grades

Sulfuric acid of pure and analytical reagent grades shall be a clear and colourless liquid, free from suspended matter and other visible impurities.

4.2 Specific requirements

The sulfuric acid shall comply with specific requirements given in Table 2 when tested according to the methods prescribed.

Table - 1: **Specific requirements for sulphuric acid**

S/No.	Characteristic	Requirements					Test method
		Technical	Battery		Pure	Analytical	
			Concentrated	Dilute			
i)	Specific gravity at 27 °C, min.	1.825	1.825	1.235	1.825	1.825	B.2
ii)	Sulfuric acid (as H_2SO_4), % by mass, min	95.0	95.0	29.7	95.0	96.0	B.3
iii)	Residue on ignition, % by mass, max.	0.2	0.06	0.02	0.01	0.002 5	B.4
iv)	Iron (as Fe), mg/kg, max.	500	20	6	1	1	B.5
v)	Chlorides (as Cl) mg/kg, max	-	10	3	35	3	B.6
vi)	Lead mg/kg, max.	50	50	50	20	2	B.7
vii)	Arsenic (as As_2O_3), mg/kg, max.	100	3	1	5	0.1	B.8
viii)	Oxidizable impurities (as SO_2)	-	To pass test	To pass test	40 mg/kg, max.	5 mg/kg, max	B.9
ix)	Organic matter	-	To pass test	0.003		30	B.10
x)	Nitrates (as NO_3) mg/kg, max.	-	-	-	-	0.2	B.11
xi)	Ammonia (as NH_3), mg/kg, max.	-	-	-	-	5	B.12
xii)	Selenium (as Se), mg/kg, max.		20	6	-	-	B.13
xiii)	Manganese (as Mn), mg/kg, max.		1	0.3	-	-	B.14
xiv)	Copper (as Cu), mg/kg, max.		30	10	-	-	B.15
xv)	Zinc (as Zn), mg/kg, max.		30	10	-	-	B.16
xvi)	Nitrates, nitrites and ammonia (as N), mg/kg, max.		30	10	30	-	B.17

5 Packaging

5.1 The sulfuric acid shall be packaged in suitable containers that protect the product's integrity.

5.2 When sulfuric acid is supplied in screw-stoppered earthenware or glass carboys, the containers shall be fitted with leak-tight stoppers. and, if required by the purchaser, provided with rubber or polyethylene washers. The stoppers shall further be sealed by a putty made of china clay, or a mixture of sodium silicate and asbestos flour, or a mixture of sulfur and send.

5.3 Sulfuric acid of pure and analytical reagent grades shall be supplied in glass bottles or glass carboys fitted with tight-fitting ground glass stoppers and no washers shall be used in closing them. The use of suitable caps over the stoppers is recommended.

5.4 The bottles or jars shall be packed in suitable pent top packing cases. These shall be placed in an upright position on one layer of sand, or ashes free from cinders, or chalk, or dry earth and the empty surrounding space shall also be filled with the same material to prevent movement. Carboys shall be packed in suitable iron hampers or wooden crates stuffed with straw or wood shavings.

5.5 In case of dilute sulfuric acid of specific gravity other than 1.235 certified in writing by the consignor, the inside packing of the cases may be straw or glass or wood shavings and the mass of each case shall not exceed 130 kg.

6 Labelling

6.1 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) name of product as "sulfuric acid"

b) grade;

c) purity

d) "specific gravity

e) manufacturer's name and physical address;

f) net content

g) country of origin;

h) batch number or lot number.

i) date of manufacture and best before date

j) cautions for safety such as signal words, hazard pictogram, precaution statement, and hazard statement

6.2 Sulfuric acid shall be supplied with the safety data sheet which is written in language as depicted in 6.1.

7 Sampling

Sampling shall be done in accordance with Annex A.

Annex A

(normative)

Sampling of sulfuric acid

A.1 General requirements of sampling

In drawing samples, the following precautions and directions shall be observed.

A.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.

A.1.2 To draw a representative sample, the contents of each container shall be mixed thoroughly by shaking or stirring by suitable means and with necessary caution.

A.1.3 The samples shall be placed in suitable, clean, dry and airtight glass containers.

A.1.4 Each sample container shall be sealed airtight after filling and shall be marked with full details of sampling, the date of sampling and the year of manufacture of the material.

A.2 Scale of sampling

A.2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

A.2.2 Sample shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. The number of containers to be selected from lots of different sizes shall be in accordance with Table 2.

A.2.3 The containers shall be drawn at random from the lot, and to ensure randomness, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and, starting from anyone, count them as 1, 2, 3, ... *r,* where *r* is the integral part of *N/n (N* and *n* being the lot size and the sample size respectively). Every *r*th container thus counted shall be included in the sample till the required number of containers specified in Column 2 of Table 2 is taken out.

Table 2 – Number of containers to be selected for sampling (See A.2.2)

Lot size *N*	**Number of containers to be selected** *n*
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1 000	10

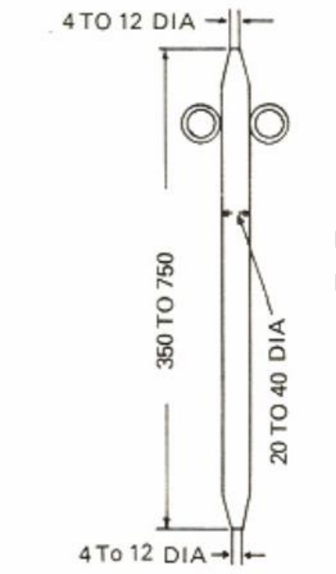
A.3 Procedure

A.3.1 Carboys, drums, jars and bottles

For drawing representative samples from these containers, agitate the material well by stirring with a glass rod. Draw the required quantity of the material with the glass-sampling instrument shown in Figure 1 and transfer it to a clean receptacle.

A.3.2 Tank wagons

While loading tank cars, samples shall be drawn at the discharge pipe where the material enters the tank car. The composite sample of not less than 25 L drawn from each wagon shall consist of small portions of not more than 500 mL each, taken at regular intervals during the period of filling. When samples are drawn at the purchaser's end, equal size samples of not less than one litre each shall be taken at 10 cm depth intervals and a composite sample shall be prepared by mixing these in a receptacle.



All dimensions in millilitres

Figure 1 – Sampling instrument

A.4 Test samples and reference sample

A.4.1 In case of sulfuric acid of battery grade, three sets of test samples of 600 mL each shall be obtained from the composite sample of each selected container after thorough agitation of the acid. These shall be placed in clean, dry, glass-stoppered bottles, closed tightly, sealed and labelled with all the particulars of sampling.

A.4.2 In case of other grades of sulfuric acid, three sets of test samples of 250 mL each shall be obtained from the composite sample of each selected container after thorough agitation of the acid. These shall be placed in clean, dry, glass-stoppered bottles and closed tightly, sealed and labelled with all the particulars of sampling.

A.4.3 The vendor shall retain one set of sealed samples and deliver one set to the purchaser or his agent, if so required.

A.4.4 The third set of test samples, bearing the seals of the purchaser and the vendor, shall constitute the reference sample to be used in case of dispute between the purchaser and the vendor.

A.5 Tests for acceptance

A.5.1 Examination and tests

The purchaser may examine and test each of the reduced samples constituting the test sample separately for compliance with the requirements of this standard, or he may prepare, for the purpose of such examination and at any stage of the progress of the examination, a composite sample representative of the whole lot, by mixing all the reduced samples constituting the test sample.

A.5.2 Criterion for conformity

When the individual reduced samples in a test sample are separately examined and the results vary from one reduced sample to another, the criterion for confirming the quality of the lot for the purpose of acceptance on the basis of the results obtained shall be at the discretion of the purchaser, unless otherwise previously agreed to between the purchaser and supplier.

Annex B

(Normative)

Analysis of sulfuric acid

B.1 Quality of reagents

Unless otherwise specified, analytical, grade chemicals and distilled water conforming to EAS 123, *Distilled water — Specification* shall be used in tests.

B.2 Determination of specific gravity

B.2.1 Apparatus

Hydrometer, Twaddell or Baume hydrometer

B.2.2 Procedure

Pour the material to be tested into a clean hydrometer jar the diameter of which shall be at least 25 mm greater than the diameter of the hydrometer used. Care should be taken to remove all the air bubbles in the liquid. Keep the jar in a vertical position in a bath maintained at 27.0 °C ± 0.5 °C. When the sample in the jar attains the temperature of the bath, lower the hydrometer gently into the material. When it has settled, depress it about two scale divisions into the liquid, the un-immersed portions of the stem being kept dry, as any unnecessary liquid on the stem changes the effective mass of the instrument and affects the reading obtained. Allow 5 min for the hydrometer to become stationary and for all air bubbles to come to the surface. Read the point on the hydrometer scale to which the sample rises with the eye placed at the level of the principle surface of the material.

B.2.3 Calculation

i. With Twaddell hydrometer

sp.gr.at 27°/ 27°C = 0.005 × Deg.Twadell+1 ……………………………..…(1)

ii. With Baume hydrometer

$$\text{Specific gravity at 27 / 27 °C} = \frac{45}{145-\text{Deg.Baume}} \quad \text{……………………..(2)}$$

B.3 Determination of sulfuric acid

Method A

B.3.1 Apparatus

Pipette, of suitable type, such as Lunge-Ray pipette of the shape end dimensions shown in Figure 2.

B.3.2 Reagents

B.3.2.1 Standard sodium hydroxide solution, 1.0 M .

B.3.2.2 Methyl red indicator solution, dissolve 0.1 g of methyl red in 100 ml of rectified spirit.

B.3.3 Procedure

Pipette about 100 ml of distilled water in 250-ml flask, add accurately 2 g of sulfuric acid to dilute and cool.

Titrate against standard sodium hydroxide solution using methyl red as indicator.

B.3.4 Calculation

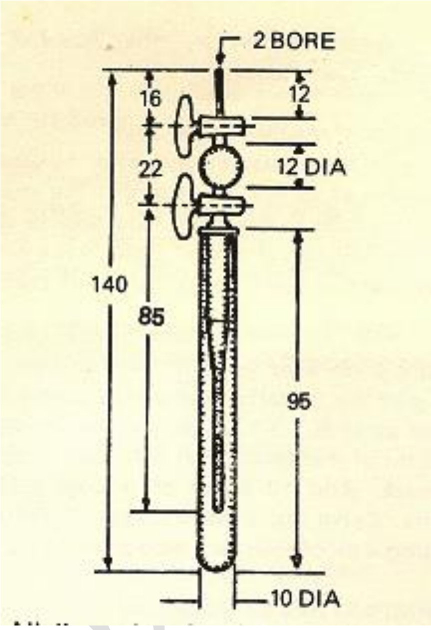
Sulfuric acid as H_2SO_4, % by mass = $\frac{9.8\,VM}{W}$ ……………………………………………..…(3)

where

V is the volume in ml of standard sodium hydroxide solution used;

M is the molarity of standard sodium hydroxide solution; and

W is the mass in g of the material taken for the test.



All dimensions in millimetres

Figure 2 - Lunge-Ray pipette

Method B

Alternative method for determination of sulfuric acid

Tare accurately a 125-ml glass-stoppered conical flask containing 50 ml of 1N sodium hydroxide. Partially fill, without the use of vacuum, a 10-ml serological pipet from near the bottom of a representative sample, remove any acid adhering to the outside and discard the first ml flowing from the pipet. Hold the tip of the pipet just above the surface of the sodium hydroxide solution, and transfer between 2.5 and 3 ml of the sample into the flask, mix the contents, and weigh accurately to obtain the weight of the sample. Add methyl orange and titrate the excess of sodium hydroxide with 1N hydrochloric acid.

Calculation:

Sulphuric acid, expressed as % m/m, is calculated as

$$\% \; H_2SO_4 = \frac{(50 \text{ x Normality of sodium hydroxide – titre value x normality of hydrochloric acid}) \text{ x } 9.8}{\text{weight of sample}}$$

B.4 Determination of residue on ignition

B.4.1 Procedure

Weigh exactly 25 g of the material into a tared round bottom silica or porcelain dish. If the technical grade acid contains suspended impurities, allow it to settle for 24 h before transferring it to the porcelain dish and weighing. Heat the basin on a sand-bath until the acid is practically driven off. Ignite on a good Bunsen flame or transfer to a suitable muffle furnace and heat for 15 min or until no further fumes of sulfur trioxide are evolved. Cool the basin in a desiccator and weigh. Preserve the residue obtained from the battery grade concentrated and dilute acids for the test prescribed in B.15.

B.4.2 Calculation

Residue on ignition, % by mass = $\frac{100\,W_1}{W_2}$ …………………………………….. …(4)

where

$W1$ is the mass in g of the residue, and

$W2$ is the mass in g of the material taken for the test.

B.5 Test for iron

B.5.1 Apparatus

Nessler tubes, 50 ml capacity.

B.5.2 Reagents

B.5.2.1 Ammonium persulfate

B.5.2.2 Concentrated hydrochloric acid

B.5.2.3 Butanolic potassium thiocyanate solution — 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.

B.5.2.4 Standard iron solution — Dissolve 7.022 g of Iron (III ammonium sulfate ($FeSO4.(NH4)2SO4.6H2O$) in water in a one litre flask, add 4 mL of concentrated sulfuric acid and make up with water to the mark. One millilitre of this solution contains one milligram of iron (as Fe). Dilute this iron solution suitably according to the requirements of the test. The diluted solution shall not be used for more than three days after preparation.

B.5.3 Procedure

B.5.3.1 For battery grade and pure grade acid

Dilute exactly one gram of the material to 10 ml with water in a Nessler tube. Add about 30 mg of ammonium persulfate, one millilitre of concentrated hydrochloric acid and 15 mL of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for 30 s. and allow the layers to separate. Carry out a control test in the other Nessler tube in the same manner using standard iron solution containing the following amounts of iron:

a) 0.02 mg of iron in the case of battery grade concentrated acid;

b) 0.006 mg of iron in the case of battery grade dilute acid; and

c) 0.01 mg of iron in the case of pure grade acid.

B.5.3.2 For technical grade acid

Dilute exactly one gram of the material to 100 ml. Take 10 ml of this solution and carry out the test as prescribed under B.5.3.1, but using 0.05 mg of iron in the control test.

B.5.3.3 For analytical reagent grade acid

Take exactly 10 g of the material and carry out the test as prescribed under B.5.3.1, but using 0.01 mg of iron in the control test.

B.5.3.4 Limits

The limit prescribed for the appropriate grade in Table 1 shall be taken as not having been exceeded if the intensity of the red colour produced in the butanolic layer with the material is not greater than that produced in the respective control tests.

B.6 Test for chlorides

B.6.1 Apparatus

Nessler tubes 50 ml capacity

B.6.2 Reagents

B.6.2.1 Dilute nitric acid, approximately 4 M

B.6.2.2 Silver nitrate solution % (w/v), approximately five

B.6.2.3 Standard chloride solution — dissolve 0.164 9 g of ignited sodium chloride in 1 000 ml of water. Dilute 100 ml of this solution again to one litre. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl). Dilute this solution suitably according to the requirements of the test.

B.6.3 Procedure

Dilute exactly 2 g of the material to 50 ml with water in a Nessler tube. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution and mix. Carry out a control test in the other Nessler tube in the same manner using standard chloride solution containing the following amounts of chloride.

a) 0.02 mg in the case of battery grade concentrated acid;

b) 0.006 mg in the case of battery grade dilute acid, and analytical reagent grade acid; and

c) 0.07 mg in the case of pure grade acid.

The limit prescribed for the appropriate grade in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test solution is not greater than that produced in the respective control test.

B.7 Test for heavy metals

B.7.1 Apparatus

Nessler tubes, 50 ml

B.7.2 Reagents

B.7.2.1 Ammonium hydroxide, approximately 6 M .

B.7.2.2 Acetic acid, 33 % by mass that complies with the following test for freedom from lead:

Take 20 ml of acetic acid and make it alkaline with ammonium hydroxide, add one millilitre of potassium cyanide solution, dilute to 50 ml with water and add two drops of sodium sulphide solution. No dark colour shall appear.

B.7.2.3 Standard lead solution — dissolve 1.6000 g of lead nitrate in 100 mL of water and a few drops of nitric acid. Dilute the solution to 1 000 mL. Take 10 ml of the solution and make up to 1 000 mL. 1 mL of this solution contains 0.01 mg of lead (as Pb).

B.7.2.4 Sodium sulfide solution — Dissolve 10 g of sodium sulfide in sufficient quantity of water to produce 100 mL of solution. Filter and preserve in a dark bottle.

B.7.3 Procedure

B.7.3.1 For technical grade acid

Transfer exactly 4 g of the material to a beaker containing about 15 mL of water and dilute to about 25 mL with water. Neutralize the acid with measured quantity of ammonium hydroxide. Acidify with 1 ml of acetic acid and make up the solution to 50 mL. Take 10 mL of the solution in one Nessler tube and dilute to the mark. Add 10 drops of sodium sulphide solution and mix. Carry out a control test in the other Nessler tube using 4 mL of standard lead solution.

B.7.3.2 For analytical reagent grade acid

Take exactly 4 g of the material and after neutralization and acidification as in B.7.3.1, transfer the solution to a Nessler tube. Dilute to the mark and add 10 drops of sodium sulfide solution and mix. Carry out a control test in the other Nessler tube using 0,8 mL of standard lead solution.

B.7.3.3 For pure grade acid

Take exactly 4 g of the material and after neutralization and acidification as in B.7.3.1, make up the solution to 50 mL. Take 25 mL of the solution in a Nessler tube and dilute to the mark. Add 10 drops of sodium sulphide solution and mix. Carry out a control test in the other Nessler tube using 4 mL of standard lead solution.

B.8 Determination of arsenic

Arsenic shall be determined according to EAS 101, *Foodstuffs — Method of determination of arsenic*.

B.9 Test for oxidizable impurities

B.9.1 Reagent

Potassium permanganate solution, exactly 0.002 M .

B.9.2 Procedure

B.9.2.1 For battery grade (concentrated and dilute) acids

Dilute one millilitre of the material to 10 mL with water, cool and add one millilitre of potassium permanganate solution. The material shall be taken to have passed the test if the pink colour of the solution is not discharged at once.

B.9.2.2 For pure grade acid

Add 20 mL of the material, slowly and with stirring, to 60 mL of water, cool and add 4 mL of potassium permanganate solution.

The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the mixture remains pink for 5 min.

B.10 Test for organic matter

Heat the acid in a clean beaker until the acid begins to fume strongly. The material shall be taken to have passed the test if it shows no perceptible charring.

B.11 Test for nitrates

B.11.1 Reagent

B.11.1.1 Concentrated hydrochloric acid

B.11.1.2 Diphenylamine solution — Prepare by mixing 90 mg of diphenylamine with 60 ml of nitrogenfree sulfuric acid and adding the mixture to 20 ml of water.

B.11.2 Procedure

Dilute 6 ml of the material with 2 ml of water, cool to 60 °C and add one drop of concentrated hydrochloric acid and one millilitre of diphenylamine solution.

The limit prescribed in Table 1 shall be taken as not having been exceeded if no blue colour is produced.

B.12 Test for ammonia

B.12.1 Reagents

B.12.1.1 Sodium hydroxide solution, approximately 30 % (w/v).

B.12.1.2 Nessler solution — Dissolve 10 g of potassium iodide in 10 mL of water and add to it slowly, with stirring, a saturated aqueous solution of mercury (II) chloride until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 mL more of mercury (II) chloride solution and dilute to 200 mL with water. Allow settling overnight and then decanting the clear solution. Keep the solution in a bottle closed with a well-fitting rubber stopper.

B.12.1.3 Standard ammonium chloride solution — Dissolve 0.314 g of ammonium chloride in one litre of water. Further dilute 100 ml of the solution to one litre. One millilitre of the diluted solution contains 0.01 mg of ammonia (as NH3).

B.12.2 Procedure

Add exactly 20 g of the material to 40 mL of water, cool in ice and make alkaline with sodium hydroxide solution. Add to the mixture one millilitre of Nessler solution. Carry out a control test using 10 mL of standard ammonium chloride solution in place of the material, finally diluting the contents to the same volume as in the test with the material.

The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced is not more than that produced in the control tests.

B.13 Test for selenium

B.13.1 Reagents

B.13.1.1 Concentrated hydrochloric acid

B.13.1.2 Sodium sulfide

B.13.1.3 Standard selenium solution — Dissolve 0.100 g of selenium in 5 ml of concentrated nitric acid and 10 ml of concentrated hydrochloric acid. Evaporate to dryness and then take up the residue with water and a little dilute sulfuric acid (3 M). Make up the volume to 1 000 ml. 1 ml of this solution contains 0.1 mg of selenium (as Se).

B.13.2 Procedure

B.13.2.1 For battery grade concentrated acid

Cool exactly 5 g of the material contained in a test-tube by surrounding the tube with crushed ice. Carefully add to the acid 10 mL of concentrated hydrochloric acid containing about 10 mg of sodium sulfite. When the effervescence ceases, no red colour shall appear at the zone of contact of the two liquids at the end of 3 min. from the time the acid is completely covered by the first addition. The reddish brown ring caused by selenium, if present, forms slightly above, and separates from, a yellow ring, which may result from the presence of copper and iron. Carry out a control test using the millilitre of standard selenium solution in the same total volume and compare the selenium rings viewing them transversely against a white background.

B.13.2.2 For battery grade dilute acid

Carry out the test as described under B.13.2.1 using exactly 5 g of the material for the test and 0.3 ml of standard selenium solution for the control test.

B.14 Test for manganese

B.14.1 Reagents

B.14.1.1 Concentrated nitric acid

B.14.1.2 Potassium periodate

B.14.1.3 Standard potassium permanganate solution, exactly 0.000 2 M .

B.14.2 Procedure

B.14.2.1 For battery grade concentrated acid

Dilute exactly 25 g of the material to 95 mL and add 5 mL of concentrated nitric acid. Add 0.5 g of potassium periodate and boil the solution vigorously in a covered beaker for 5 min. Cool and compare the colour with that of an equal volume of a solution containing 2.3 mL of standard potassium permanganate solution, 5 ml of nitric acid and 0.5 g of potassium periodate in water, this solution having been treated in the same manner. The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced by the material is not greater than that produced in the control test.

B.14.2.2 For battery grade dilute acid

Carry out the test as described under B.14.2.1 using exactly 25 g of the material for the test and 0.7 mL of standard potassium permanganate solution for the control test.

B.15 Test for copper

If the residue on ignition as obtained in B.4.1 exceeds 0.003 %, use it for the test for copper. If the residue on ignition does not exceed 0.003 %, the test for copper shall not be carried out.

B.15.1 Apparatus

Nessler tubes, 25 ml capacity.

B.15.2 Reagents

B.15.2.1 Concentrated hydrochloric acid

B.15.2.2 Ammonium hydroxide, sp. gr. 0.90.

B.15.2.3 Standard copper solution — Dissolve 0.393 g of cupric sulfate ($CuSO4.5H_2O$) in 1 000 ml of water. 1 ml of the solution contains 0.1 mg of copper (Cu).

B.15.3 Procedure

Add to the residue obtained in B.4.1, 1 mL of concentrated hydrochloric acid, warm on a steam bath to dissolve copper and iron oxides, and dilute to 10 mL. Neutralize the solution with ammonium hydroxide and add 4 mL of ammonium hydroxide in excess. Heat sufficiently to coagulate any precipitate, filter into a Nessler tube, and dilute to 25 mL. The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour in the solution is not greater than that of a control standard prepared from the following amounts of standard copper solution treated with 4 mL of ammonium hydroxide and diluted to 25 mL in another Nessler tube:

a) 7.5 mL in the case of battery grade concentrated acid, and

b) 2.5 mL in the case of battery grade dilute acid.

B.16 Test for zinc

Test for zinc shall be carried out only if the residue on ignition as determined in B.4.1 exceeds 0.003 %.

B.16.1 Reagents

B.16.1.1 Ammonium hydroxide, sp. gr. 0.90.

B.16.1.2 Dilute sulfuric acid, 3 M and 0.5 M .

B.16.1.3 Hydrogen sulfide gas

B.16.1.4 Methyl red indicator solution, same as in B.3.2.2

B.16.2 Procedure

Neutralize 100 mL of the material with ammonium hydroxide. Dilute the solution to 200 mL, add 10 mL of sulfuric acid (3 M) and saturated with hydrogen sulphide. Filter, boil to remove all hydrogen sulphide, and neutralize exactly the cooled solution with ammonium hydroxide, using methyl red as indicator. Add exactly one millilitre of dilute sulfuric acid 0.5 M for each 100 mL of solution and pass a rapid stream of hydrogen sulfide through the solution for 20 min. If a precipitate forms, filter, wash, ignite and weigh the precipitate. The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the mass of the ignited precipitate does not exceed 5.5 mg in the case of battery grade concentrated acid and 1.2 mg in the case of battery grade dilute acid.

B.17 Test for nitrates, nitrites and ammonia

B.17.1 Apparatus

Nessler tubes, 50 ml capacity.

B.17.2 Reagents

B.17.2.1 Sodium hydroxide solution, approximately 40 %.

B.17.2.2 Devarda's alloy — containing 45 parts aluminium, 50 parts copper and 5 parts zinc. Heat the aluminium in a Hessian crucible in a furnace until the aluminium begins to melt. Add copper in small portions and heat until liquefied. Now plunge zinc into the molten mass. Cover the crucible and heat the mixture for a few moments and stir with an iron rod. Allow it to cool slowly with the cover on and then pulverize the crystallized mass.

B.17.2.3 Dilute hydrochloric acid, approximately 6 M.

B.17.2.4 Nessler solution, same as in B.12.1.2.

B.17.2.5 Standard ammonium chloride solution — prepare a solution containing 0.381 9 g of ammonium chloride per litre. 1 mL of this solution contains 0.1 mg of nitrogen (N).

B.17.3 Procedure

Weigh exactly 20 g of the material and add cautiously to 100 mL of water in a 500- mL flask. After cooling, carefully neutralize with sodium hydroxide solution till the liquid is distinctly alkaline. Add 2 g of Devarda's alloy and fit a distillation head and condenser. After 1 h, distil off 50 mL into a conical flask containing 1 ml of dilute hydrochloric acid. Transfer the distillate to a Nessler tube; add 1 mL of sodium hydroxide solution and 2 mL of Nessler solution. Carry out a control test following the same procedure using the following quantities of standard ammonium chloride solution.

a) 6 ml in the case of pure grade acid and battery grade concentrated acid; and

b) 2 ml in the case of battery grade dilute acid.

The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the respective control test.

Bibliography

EAS 122: 1999 Sulfuric acid - Specification

TZS 74:2017 Sulfuric acid - Specification