

**DRAFT TANZANIA STANDARD**

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**Sodium Sulfate, Anhydrous – Specification**

*Draft Standard - For Comment Only*

**TANZANIA BUREAU OF STANDARDS**

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## CDC 7 (13) DTZS

### Introduction

Sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) is the sodium salt of sulfuric acid. It is a white crystalline solid also known as the *mineral thenardite*, while the decahydrate  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  has been known as *Glauber's salt or mirabilis*.  $\text{Na}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$  is transformed to mirabilite when it is cooled. Mirabilite is the natural mineral form of the decahydrate. It is also produced from by-products of chemical processes such as hydrochloric acid production.

There are two types of sodium sulfate natural and by product, also known as synthetic.

- i. Natural sodium sulfate is produced from naturally occurring brines and crystalline deposits. It is also found as a constituent of saline lakes
- ii. Synthetic sodium sulfate is recovered as a by-product of various manufacturing processes.

Both types of sodium sulfate have several important and useful applications in various consumer products.

Some of the industrial application of Sodium sulfate are:

- Used to dry organic liquids.
- As a filler in powdered home laundry detergents.
- As a fining agent which removes small air bubbles from molten glass.
- For defrosting windows, in carpet fresheners, starch manufacture, as an additive to animal feed.
- In the manufacture of detergents and in the Kraft process of paper pulping.

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## Foreword

This Draft Tanzania Standard is being developed by the Industrial and Laboratory Chemicals Technical Committee under supervision of the Chemical Division Standards Committee and it is in accordance with the procedures of the Bureau.

In the preparation of this Tanzania Standard assistance has been drawn from:

IS 255:1982 (Reaffirmed 2006) *Specification for Sodium sulphate, anhydrous*, published by Bureau of Indian Standards.

Acknowledgement is hereby made for the assistance derived from this source.

In reporting the result of a test or analysis made in accordance with this Tanzania Standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with TZS 4 *rounding off numerical values*.



## Sodium Sulphate, anhydrous – Specification

### 1 Scope

This Draft Tanzania standard prescribes the requirements, sampling and test methods for sodium sulfate, anhydrous.

### 2 Normative references

The following referenced documents are indispensable for the application of this document. The latest edition of the referenced document (including any amendments) applies;

TZS 59 *Water for analytical laboratory use – Specification and test method*

### 3 Terms and definitions

<<<<<Not applicable>>>>>

### 4 Requirements

#### 4.1 General requirements

The material shall consist essentially of sodium sulfate, anhydrous. It shall be in the form of white, free-flowing powder, free from visible impurities and shall be completely soluble in water.

#### 4.2 Specific requirements

The material, dried in accordance with the method given in A-2.1, shall comply with the requirements given in Table 1. when tested according to the methods prescribed in Annex A.

**Table 1 Specific requirements for Sodium Sulfate, Anhydrous.**

S/N	Characteristic	Requirement (on dry basis)	Method of test as referred to clause No. in annex A
i.	Sodium sulphate (as Na <sub>2</sub> SO <sub>4</sub> ), percent by mass, min	98.0	A-3
ii.	Matter insoluble in water, percent by mass, max	0.5	A-4
iii.	Chlorides (as NaCl), percent by mass, max	2.0	A-5
iv.	Iron, Aluminium and Chromium (as R <sub>2</sub> O <sub>3</sub> ), percent by mass, max	0.02	A-6
v.	Iron (as Fe), percent by mass, max	0.002	A-7
vi.	Loss on ignition, percent by mass, max	1.0	A-8
vii.	pH (of 10 percent solution)	7.0 – 8.0	A-9
	NOTE: from (i) to (v) carried out on dry basis.		

### 5 Packing and Marking

#### 5.1 Packaging

The material shall be supplied in clean, dry and air tight containers, without faults, made of materials which do not affect the contents. The method of closing the containers shall prevent the contents from contamination and evaporation.

## **5.2 Marking**

Each container shall bear the following information given in visible, legible and durable marking in English and/or Kiswahili:

- a) Name of the material
- b) Manufacturer's name and address
- c) Country of origin
- d) Recognized trade mark, if any
- e) Batch/Lot number
- f) Manufacturing date

## **6. Sampling**

The procedure for drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex B.

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**Annex A**  
(normative)

**Analysis of sodium sulphate, anhydrous**

**A-1. Quality of reagents**

Unless specified otherwise, analytical/reagent grade chemicals and *water for analytical laboratory use* (TZS 59) shall be used in tests.

**A-2. Preparation of sample**

**Procedure** - Crush 50 g of sample to pass through a 1.00mm sieve. Dry to constant mass at  $105 \pm 5^\circ\text{C}$  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  $\text{Na}_2\text{SO}_4$ )**

**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  $110^\circ\text{C}$  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**

Sodium sulphate (as  $\text{Na}_2\text{SO}_4$ ), percent by mass =  $\frac{100 M}{M_1}$

where

$M$  = mass in g of the dried precipitate, and

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

**A-4. Determination of matter insoluble in water**

**A-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  $110^\circ\text{C}$  to constant mass.

**A-4.2 Calculation**

Matter insoluble in water, percent by mass =  $\frac{100 M}{M_1}$

Where,

$M$  = mass in g of the residue, and

$M_1$  = mass in g of the prepared sample taken for the test

## A-5. Determination of chlorides (as NaCl)

**A-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

### A-5.2 Reagents

**A-5.2.1** Standard silver nitrate – 0.1 N

**A-5.2.2** Concentrated nitric acid

**A-5.2.3** Ferric alum indicator solution - saturated,

**A-5.2.4** Standard ammonium thiocyanate solution – 0.1 N

**A-5.2.5** Nitrobenzene

### A-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.

### A-5.4 Calculation

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

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.

## A-6. Determination of Iron, Aluminium and Chromium (as $R_2O_3$ )

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

### A-6.2 Reagents

**A-6.2.1** Ammonium chloride – Solid

**A-6.2.2** Dilute Ammonium Hydroxide - approximately 4 N.

**A-6.2.3** Ammonium Nitrate Solution - approximately 2 percent.

**A-6.2.4** Dilute Hydrochloric Acid - approximately 4 N.

**A-6.2.5** Conc. Nitric acid

### A-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  $100 \pm 2^\circ\text{C}$  and ignite at  $1100^\circ\text{C}$  in a tared crucible to constant mass.

### A-6.3 Calculation

$$\text{Iron, Aluminium and Chromium (R}_2\text{O}_3\text{), percent by mass} = \frac{100 M_1}{M_2}$$



where

$M_1$  = mass in g of the ignited residue, and

$M_2$  = mass in g of the prepared sample taken for the test.

## A-7. Determination of iron (as Fe) by Colorimetric Method

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

### A-7.2 Apparatus

Nessler cylinders - 50 mL capacity.

### A-7.3 Reagents

**A-7.3.1 Concentrated nitric acid**

**A-7.3.2 Ammonium persulfate** - solid.

**A-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.

**A-7.3.4 Dilute sulfuric acid** - approximately 10 percent (v/v).

Weigh 0.702 g of ferrous ammonium sulfate [  $\text{FeSO}_4(\text{NH}_4)\text{SO}_4 \cdot 6\text{H}_2\text{O}$  ] 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).

### A-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.

### A-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.

## A-8. Determination of loss on drying

**A-8.1 Procedure** - Weigh about 5 g of the crushed material into a tared dish and dry at  $105 \pm 5^\circ\text{C}$ . Cool and weigh repeatedly till a constant mass is obtained.

### A-8.2 Calculation

Loss on drying, percent by mass =  $100 \frac{M_1 - M_2}{M_1}$

where

$M_1$  = mass in g of the material taken for the test, and

$M_2$  = mass in g of the residue obtained after drying.

## A-9. Determination of pH

**Procedure** - Weigh 5 g of the prepared sample and dissolve in 50 mL of water. Determine the pH of the solution with the suitable calibrated pH meter, using glass and calomel electrode.

**Annex B**  
(normative)

**Sampling of sodium sulphate, anhydrous**

**B-1. General requirements of sampling**

**B-1.0** In drawing, preparing, storing and handling test samples the following precautions and directions shall be observed.

**B-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.

**B-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.

**B-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.

**B-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.

**B-2. Scale of sampling**

**B-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.

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

**B-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

**B-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.

**B-3. Test samples and referee sample**

**B-3.1 Preparation of Test Samples**

**B-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.

**B-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.

**B-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.

#### **B-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.

#### **B-4. Number of tests**

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

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

#### **B-5. Criteria for Conformity**

##### **B-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 ( $\bar{x}$ ) = 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  $\bar{X} - 0.6 R$  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.

##### **B-5.2 For composite sample**

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

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