

**KENYA STANDARD**

**DKS 2927:2020**

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**First Edition**

# **Industrial Solvents and Chemicals — Sodium bicarbonate — Specification**



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**DKS [2927:2020]**

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# **Industrial Solvents and Chemicals — Sodium bicarbonate — Specification**

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## Foreword

This Kenya Standard was prepared by the Industrial solvents and Chemicals Technical Committee under the guidance of the Standards Projects Committee, and it is in accordance with the procedures of the Kenya Bureau of Standards

During the preparation of this standard, reference was made to the following document (s):

IS 2124:2000- Sodium bicarbonate - specification

ISO 740- Sodium carbonate for industrial use - Determination of total soluble alkalinity - Titrimetric method

ISO 746- Sodium carbonate for industrial use - Determination of matter insoluble in water at 50°C

Acknowledgement is hereby made for the assistance derived from this (these) source (s)



# Industrial Solvents and Chemicals —Sodium bicarbonate — Specification

## 1 Scope

This standard prescribes the requirements, methods of test and sampling for sodium bicarbonate. ]

## 2 Normative references

There are no normative references in this document. ]

## 3 Terms and definitions

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

### 3.1

Sodium bicarbonate

Is a white solid that is crystalline but often appear as a fine powder with a chemical formula  $\text{NaHCO}_3$ . ]

## 4 Grades

4.1 There shall be three grades of the material, namely, Pure, Analytical Reagent and Technical

4.1.2 Pure Grade — Suitable for the fine chemical, food application and pharmaceutical industry

4.1.3 Analytical Reagent Grade —Suitable as a reagent.

4.1.4 Technical Grade —Suitable for animal feed and detergent application

## 5 Requirements

5.1 Physical Requirement — The material of all the three grades shall be in the form of small, opaque, monoclinic crystals or as a white powder, free from dirt and other foreign matter.

5.1.1The pure grade material suitable for the manufacture of effervescent powders shall have the particle size distribution as agreed to between the purchaser and the supplier.

5.1.2The material, when tested according to the methods prescribed in Annexes A to P, shall also comply with the requirements specified in Table 1.

5.4 Chemical Requirement — The material shall comply with the chemical properties specified in Table 1 when tested according to the methods prescribed in Annexures of this specification.

## 6 Packaging and Marking

6.1 Packaging — the material shall be packed in air-tight and moisture proof containers.

6.2 Marking — When supplied in packages, each package shall be securely closed and marked indelibly with the following information:

- (i) Name and grade of the material;
- (ii) Net weight of the content of the package or container;
- (iii) Manufacturer's name and/or recognized trade mark if any;
- iv) Batch/lot number;
- (iv) Date of manufacture/ expiry; and
- v) Country of origin.

When supplied in bulk, a good sized metallic or cardboard label bearing the above information printed with suitable paint or ink shall be conspicuously displayed on the carrier and also placed inside.

## 7 Sampling and size sample

### 7.1 Sampling

To test for the compliance of sodium bicarbonate to the requirements of this standard, a sample of about 500g shall be taken and stored in an air-tight container.

The method of drawing representative samples of the material from the containers shall be as prescribed in Annex P

**Caution:** in taking samples from a container or package, care shall be taken to exclude portions where caking is noticeable (due to absorption of moisture and carbon dioxide). This may be done by removing from the top about 20cm of the material in the container and then taking out sample from the center of the remaining portion.

**TABLE 1. Requirements for Sodium Bicarbonate**

SL NO.	CHARACTERISTICS	Requirements			METHOD OF TEST
		Pure Grade	Analytical Grade	Technical Grade	
1	2	3		4	Annex
i)	Total alkalinity (as NaHCO <sub>3</sub> ) percent by mass, Min	99.0	99.8	98.0	A
ii)	Insoluble matter, percent by mass, Max	0.05	0.010	0.100	B
iii)	pH (1 percent aqueous solution), Max	8.6	8.4	8.8	pH meter
iv)	Sulphates (as SO <sub>4</sub> ), percent by mass, Max.	0.05	0.003	0.07	C
v)	Iron (as Fe), percent by mass, Max.	0.0015	0.0005	0.004	D
vi)	Heavy metals (as Pb), parts per million, Max		0.001		E
vii)	Ammonium compounds (as NH <sub>4</sub> ), parts per million, Max.	To pass test	5	A13	F
viii)	Chlorides (as Cl <sup>-</sup> ), percent by mass, Max	0.01	0.001	0.06	G
ix)	Substances reducing iodine, percent by mass, Max	-	Pass	-	H
x)	Phosphates (as PO <sub>4</sub> ), percent by mass, Max	-	0.0005	-	J
xi)	Calcium and magnesium (as Ca), percent by mass, Max		0.015		K
xii)	Nitrates (as NO <sub>3</sub> ), percent by mass, Max		0.001		L
xiii)	Potassium (as K), percent by mass, Max		0.01		M
xv)	Copper (as Cu), parts per million, Max	30	-	30	N



**Annex A**  
(normative)**Determination of total alkalinity****A.1 General**

The total alkalinity of a processing solution is defined as the millilitres of standardized 0.1 N sulfuric acid required to adjust a specified volume of processing solution to pH 4.3

**A1.1 Reagents**

**A1.1.1 Standard Hydrochloric Acid 1.0 N.**

**A1.1.2 Standard Hydrochloric Acid 0.1 N**

**A1.1.3 Methyl Orange or Bromothymol Blue Indicator**

**A.2. Procedure**

Weigh accurately 4.4 g of the material and dissolve in 100 ml of freshly boiled and cooled water in a 250ml conical flask. Add 5 drops of the methyl orange or bromothymol blue indicator and 50 ml of standard hydrochloric acid (1 N). Stir the contents thoroughly and further titrate with standard hydrochloric acid (0.1 N) until the colour of the indicator solution changes from orange to red (blue to green in case of bromothymol blue). Calculate the total volume in terms of hydrochloric acid (1 N).

**A.3 Calculation**

Total alkalinity (as NaHCO<sub>3</sub>), percent by mass=

$$\frac{8.4VN}{M}$$

Where,

v = volume in ml of standard hydrochloric acid (1N) required for titration

where,  $V = 50\text{ml } 1\text{N HCL} + (\text{Titer Value of } 0.1\text{N HCL} \times 0.1)$

$N$  = normality of standard hydrochloric acid used, and

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

**Annex B**  
(normative)**Determination of matter insoluble in water at 50°C****B.1 Principle**

Dissolution of a test portion and filtration of the solution through a tared filter. Drying of the residue and weighing.

**B.2 Procedure**

Weigh accurately about 10 g of the material, transfer it to a 400 ml beaker, add about 200 ml of freshly boiled water and boil the resulting solution for 15 minutes. Filter any undissolved residue through a tared Gooch or a sintered glass crucible (G No.4) and wash the residue free from soluble salts with water. Collect the filtrate and washings in a one-litre graduated flask and dilute to mark. Preserve the solution so obtained for the test under Annex K. Dry the crucible along with the residue to constant mass at 105 to 110°C.

**B.3 Calculation**

The matter insoluble in water at 50 °C, expressed as a percentage by mass, is given by the formula

$$\frac{M_1}{M_0} \times 100$$

Where;

M<sub>0</sub>=the mass, in grams, of the test portion;

M<sub>1</sub>=the mass, in grams, of the filtered and dried insoluble matter.

## Alternate method: Determination of matter insoluble in water using filter membrane under Vacuum

### B.1a. Apparatus

0.45micron cellulose filter membrane

Membrane filter assembly

Vaccum pump

Oven

Dessicator

### B.2a. Procedure

Weigh accurately 5g of the sample. Transfer it into a 400ml beaker, add about 200ml of distilled water and boil the solution of about 10min and filter the solution through a weighed 0.45 microns cellulose filter membrane, using a vacuum pump. Transfer any insoluble matter into the filter assembly with a jet of distilled water. Wash the residue five times with 5ml portions of distilled water at room temperature. Dry the filter membrane in an oven at 50oC for 1hr, cool in desiccator and weigh.

### B.3a. Calculation

Matter insoluble in water, %by mass =

$$\frac{M1 \times 100}{M2}$$

Where;

M1 = mass in grams of the residue; and

M2= mass in grams of the sample taken for the test.

**Annex C**  
(normative)  
**Determination of Sulphates**

**C.1.0 For technical grade****C.1.0.1 Sodium bicarbonate, analytical reagent grade.****C.1.0.2 Barium chloride solution, approximately 10 % (m/v).****C.1.0.3 Filter paper****C.1.2. Procedure**

Weigh to the nearest 0.01 g about 10 g of the material ( $m_1$ ) in a porcelain dish, add 0.5 g of sodium bicarbonate and evaporate to dryness. Moisten the residue with about 1000 g of the material accurately weighed ( $m_2$ ), add 200 mL of boiling water and filter if necessary. Bring the contents to boil over a low flame and add slowly, with stirring, 5 mL of hot barium chloride solution. Boil the contents for 2 min and allow the precipitate to settle for 4 h. Filter the supernatant liquid through a tared sintered glass crucible (G No. 4) or a tared Gooch crucible and transfer the precipitate carefully into the crucible. Wash thoroughly with hot water till the washings are free from chlorides. Heat the crucible at 1100 C to constant mass.

**C.1.3. Calculation**Sulphates (as H<sub>2</sub>SO<sub>4</sub>), % by mass =

$$\frac{42.02}{m_1 + m_2}$$

Where;

m = mass in g of the precipitate.

m<sub>1</sub> = mass in g of the sample taken initially; andm<sub>2</sub> = mass in g of the sample added subsequently

## **C.2 For analytical reagent grade**

### **C.2.1 Reagents**

**C.2.1.1 Sodium carbonate, analytical reagent grade.**

**C.2.1.2 Hydrochloric acid, approximately 1N, sulphate-free.**

**C.2.1.3 Barium chloride solution, approximately 10 % (m/v)**

**C.2.1.4 Standard sulphate solution, dissolve 0.178 g of potassium sulphate in water and make up the volume to 1000 ml. Dilute 100 mL of the solution in a graduated flask to 1000 ml. One milliliter of the diluted solution contains 0.01 mg of sulphate (as H<sub>2</sub>SO<sub>4</sub>).**

### **C.3 Procedure**

To 20.00 g of the material add 20 mg of sodium carbonate and evaporate to dryness. Take up the residue with 5 ml of water and 0.5 mL of hydrochloric acid. Filter, wash with water to make up to 10 ml and add 1 ml of barium chloride solution. Carry out a control test in the same manner using 4 mL of standard sulphate solution in place of the material. Stir the two solutions and compare the turbidity produced.

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

**Annex D**  
(normative)  
**Determination of Iron Content**

**D.1 Principle**

The iron present in the sample is reduced to the ferrous state and determined using 1,10-phenanthroline spectrophotometrically; or by Atomic Absorption Spectroscopy (AAS) method.

**D.2 Spectrophotometric method****D.2.1 Apparatus**

D.2.1.1 Spectrophotometer with 1 cm cells, or alternatively Nessler cylinders.

D.2.2.2 Twelve 100 ml volumetric flasks.

D.2.2.3 250 ml volumetric flask.

D.2.2 Reagents

D.2.2.1 Hydrochloric acid, concentrated,  $d=1.18$ . free from iron.

D.2.2.2 Hydrochloric acid, approximately, N solution free from iron.

D.2.2.3 Hydroxyl ammonium chloride, 10 % (w/v) solution.

D.2.2.4 Ammonium acetate, 20 % (w/v) solution.

D.2.2.5 Bromine water, saturated.

D.2.2.6 1,10-phenanthroline, 0.1 per cent (w/v) solution, dissolve 0.1 g of the reagent in 50 mL of water containing 2 ml of N hydrochloric acid and dilute to 100 ml.

D.2.2.7 Standard iron solution, dissolve 7.022 g of ammonium ferrous sulphate in a mixture of 600 ml of water and 350 ml of concentrated sulphuric acid,  $d = 1.84$ . Dilute to 1000 ml with deionized water and further dilute 10 ml of the solution so obtained to 1 000 ml with water. 1 ml of the solution contains 10 micrograms of iron (10  $\mu\text{g}$  of iron).

D.2.2.8 Methyl orange indicator, 0.05 % (w/v) solution.

**D.2.3 Procedure**

Preparation of standard iron solutions (color standards) Into eleven of the 100-mL volumetric flasks, each containing 50 ml of water, and 2 ml of N hydrochloric acid, transfer amounts of the standard iron solution, containing from 0 to 100 micrograms of iron (0 g to 100 g of iron) increasing by stages 10 micrograms (10  $\mu\text{g}$ ) and treat each solution as follows:

Add 2 ml of the hydroxyl ammonium chloride solution and allow to stand for one minute, add 10 ml of the ammonium acetate solution and 3 mL of the 1,10-phenanthroline solution. Dilute the contents of the flask to 100 mL and mix thoroughly. These standard solutions are used directly for visual comparison of the colour with that of the sample.

If an instrument is to be used, measure the optical density of each solution at a wavelength of 515 nm and prepare a calibration chart.

### D.2.3.2 Determination

Weigh accurately to the nearest 0.01 g, about 25 g of the sample and transfer to a 400-ml beaker. Add 100 ml of water one drop of methyl orange indicator and acidify with concentrated hydrochloric acid adding 2 ml in excess. Boil the solution for a few minutes. Add a few drops of bromine water to destroy the colour of the indicator and boil the solution to expel excess bromine. Cool the solution transfer to the 250-ml one-mark volumetric flask, add 2 ml of the hydroxyl-ammonium chloride solution and allow to stand for 1 min. Add 10 ml of the ammonium acetate solution mix and add 3 ml of the 1,10-phenanthroline solution, dilute to 100 ml with water and thoroughly mix. At the same time carry out a blank test on the reagents alone. Measure the optical density of the solution at wavelength of 515 nm and read the amount of iron present from the calibration chart prepared in F.2.3.1. Alternatively compare the colour of the sample solution in matched Nessler cylinders with the series of the prepared standard iron solutions

### D.2.4 Calculation

$$\text{Iron content, as Fe parts per million by mass} = \frac{25 m_2}{m_1}$$

where,

$m_2$  = mass in micrograms, of iron found; and

$m_1$  = mass in grams of sample taken.

## D.3 Atomic absorption spectroscopy method

### D.3.1 Apparatus

D.3.1.1 Atomic Absorption Spectrometer

D.3.1.2 100 ml beaker

D.3.1.3 Filter paper No. 1

D.3.1.4 Analytical balance

D.3.1.5 100 ml volumetric flask

### D.3.2 Reagents



# KENYA STANDARD

DKS [2927:2020]

ICS [##.###]

First Edition

D.3.2.1 Conc. Nitric acid  $d= 1.42$ .

## D.3.3 Procedure

D.3.3.1 Take 2 g of the sample and add 6 ml of conc. Nitric acid and dissolve. Filter the solution through filter paper No 1, into 100 ml beaker. Wash the filter paper using distilled water. Transfer the filtrate to the 100-ml volumetric flask. Top up to the mark with distilled water. Use this solution to determine the iron content in sodium bicarbonate using AAS.

## D.3.4. Calculations

Fe content, mg/L = 
$$\frac{\text{Reading} \times \text{volume} \times \text{dilution factor (if any)}}{\text{Weight of sample taken}}$$

# **Annex E**

## **(normative)**

### **Test for Heavy Metal (Lead)**

#### **E.1 Outline of the Method (spectrophotometric method)**

Lead reacts with dithizone (diphenylthio Carbazone) to form a pink coloured complex in chloroform solution. The complex is separated by extraction with chloroform from an aqueous Ammonia-Cyanide Sulphite solution. The absorbance of the extracted complex is measured using a spectrophotometer at 510 nm wavelength.

#### **E.2 Apparatus**

E.3.2.1 Standard Laboratory Apparatus

E.3.2.2 Separating Funnels 250 ml capacity.

E.3.2.3 Spectrophotometer or Photocolorimeter

#### **E.2.1 Reagents**

E.2.1.1 Standard Lead Solution

Dissolve 0.799 2 g of Analytical Reagent lead nitrate [Pb(NO<sub>3</sub>)<sub>2</sub>] in distilled water in a beaker, transfer quantitatively into a 500-ml volumetric flask, dilute to the mark and mix well. One ml of this solution contains 1 mg lead.

E.2.1.2 Dilute Standard Lead Solution Transfer 10 ml of the above solution (G.3.3.1) into a 1000ml volumetric flask, dilute to mark and mix well. 1 ml of this solution contains 10 µg of lead.

E.2.1.3 Chloroform Reagent grade chloroform.

E.2.1.4 Dithizone Reagent 50 mg/l solution in chloroform. Dissolve 5 mg of the reagent in 100 ml of chloroform.

E.2.1.5 Ammonia-Cyanide-Sulphite Reagent

To 3 ml of 10 percent potassium cyanide solution (CAUTION- Potassium cyanide is highly poisonous) add 35 ml of concentrated ammonia and dilute to 100 ml. Add 0.15 g of sodium sulphite and mix well.

E.2.1.6 Dilute Hydrochloric Acid Solution Approximately 1 mol/l.

E.2.1.7 Hydrochloric Acid Solution Approximately 6 mol/l.

#### **E.3 Procedure**

E.3.1 Calibration Transfer dilute standard lead solution to a series of six 250-ml separating funnels as given below:

Standard Lead solution, ml	Corresponding lead content $\mu\text{g}$ ,
0 (Compensation)	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

Add to each funnel 75 ml of ammonia-cyanide-sulphite reagent solution and cautiously adjust pH to 9.5 using a pH meter (equipped with a combined electrode). This operation must be carried out slowly because, if the pH of the solution falls below 9.5 even temporarily, HCN gas may be liberated. Use of fume cupboard during this operation is necessary. Then add 7.5 ml of dithizone reagent to each separating funnel. Maintain time difference among each such that the time after addition of dithizone reagent and final absorbance measurement is same for all. Then add 17.5 ml of chloroform and shake for one minute to extract the lead complex into the chloroform layer, allow the phases to separate. Transfer the chloroform layer (lower one) through a cotton plug (placed in the stem of the separating funnel) into a 1 cm cell of the spectrophotometer. Immediately replace the lid of the cell. Measure absorbance (using chloroform extract of compensation solution, in reference cell) at 510 nm wavelength. Plot a graph using 20-25 ml of lead (in 25 ml of chloroform) as abscissae and corresponding absorbance values as ordinates.

**E.3.2 Determination** Weigh accurately correct to 1 mg about 10 g of Sodium bicarbonate sample (the quantity to be weighed, should be such that it contains 20 to 100  $\mu\text{g}$  of lead) and transfer to 50 ml beaker dissolve in about 20-25 ml distilled water and transfer quantitatively into a 250 ml separating funnel and rinse the beaker twice with 5 ml portions of distilled water. 75 ml of Ammonia-Cyanide-Sulphite reagent and then by the corresponding lead content in micrograms from the cautious addition of dilute hydrochloric acid solution calibration graph. Adjust pH to 9.5. Use a pH meter with combined electrode to adjust pH. This operation must be carried out slowly because if the pH of the solution falls below 9.5 even temporarily, HCN gas may be liberated. Use of fume cupboard during this operation is necessary. Now add 7.5 ml of dithizone reagent to the separating funnel followed by 17.5 ml of chloroform. Shake for one minute to extract the lead into the chloroform layer. Allow the phase separate. Transfer the chloroform layer through a cotton plug (placed in the separating funnel stem) into a 1 cm cell of the spectrophotometer and measure the absorbance at 510 nm with chloroform in the reference cell. Read the corresponding lead content in micrograms from the calibration graph.

### **E.3.3 Blank Test**

Carry out a blank test exactly as described in G-3.4.2 but excluding the test solution, use distilled water instead. Read the corresponding lead content in the blank from the calibration graph.

## E.4 Calculations

Calculate the lead content in the material from the relation given below:

Heavy metals (as Pb), ppm =

$$\frac{M1 - M2}{M0}$$

Where;

M1 = mass of lead in the test solution as obtained from graph in  $\mu\text{g}$ ,

M2= mass of lead blank as obtained from graph in  $\mu\text{g}$ , and

M0 = mass of sample taken for analysis in grams

**Annex F**  
(normative)  
**Determination of Ammonium Compounds**

**F.1 Reagents**

F.1.1 Sodium hydroxide solution, 5N, ammonia-free.

F.1.2 Standard ammonium solution, dissolve 2.97 g of ammonium chloride in water and dilute to 1000 ml. 1 ml of this solution contains 1 mg of ammonia. Dilute 1 ml of this solution to 100 ml with water immediately before use to get 0.01 mg of ammonia per ml.

F.1.3 Nessler's reagent, dissolve 35 g of potassium iodide and 12.5 g of mercuric chloride in 800 ml of water and add a saturated solution of mercuric chloride until a slight permanent precipitate is produced. Then add 120 g of sodium hydroxide solution and enough water up to 1000 ml. Shake the solution occasionally for 4 days. Allow to settle and decant the clear liquid.

**F.2 Procedure**

Dilute 2.8 mL (3.3 g) of the material with water to about 20 mL and add sodium hydroxide solution, until alkaline to litmus paper (about 7 mL is required). Dilute to 50 mL with water. Prepare standard by diluting 1 mL of standard ammonium solution to 50 mL with water. To both solutions add 2 mL of Nessler's reagent and compare after 1 min.

F.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the yellow colour produced, if any, in the test with the material is not deeper than that of the standard.

**Alternatively, use High Performance Liquid Chromatography (HPLC) method.**

# Annex G (normative) Determination of Chlorides ( method A- volumetric method)

## G.1 Principle

Chlorides are determined by neutralizing the sample with excess silver nitrate with ferric ammonium sulphate.

### G.1.1 For Pure and Technical Grades

#### G.1.1.1 Reagents

- G.1.1.1.1 Concentrated nitric acid
- G.1.1.1.2 Standard silver nitrate solution, 0.1 N.
- G.1.1.1.3 Nitrobenzene, analytical reagent.
- G.1.1.1.4 Standard ammonium thiocyanate solution, 0.1 N.
- G.1.1.1.5 Ferric ammonium sulphate indicator, 25 % m/v.

#### G.1.1.2 Procedure

Weigh accurately about 20g of the material, dissolve in water and neutralize with concentrated nitric acid and then add about 5 ml in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene, shake vigorously and titrate

with standard ammonium thiocyanate solution using ferric ammonium sulphate indicator. At the end point a permanent faint reddish-brown colouration appears.

#### G.1.1.3 Calculation

$$\text{Chloride (as Cl) \% by mass} = \frac{3.547(10N_1 - VN_2)}{M}$$

Where;

N1 = normality of standard silver nitrate solution

v = volume in ml of standard ammonium thiocyanate solution consumed in the titration,

N2 = normality of standard ammonium thiocyanate solution, and

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

### G.1.2 For Analytical Reagent Grade

#### G.1.2.1 Apparatus and reagents

- G.1.2.1.1 Nessler cylinders 100 ml capacity.
- G.1.2.1.2 Concentrated nitric acid
- G.1.2.1.3 Silver nitrate solution Approximately 4 percent.

G.1.2.1.4 Standard chloride solution; Dry sodium chloride at 250°C before making the solution. Dissolve 1.649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml from this solution and dilute with water to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

### **G.1.2.2 Procedure**

Dissolve 1.000g of the material in 30 ml of water and add slight excess of concentrated nitric acid. Boil the solution, cool and add 1 ml of silver nitrate solution. Transfer into a Nessler cylinder and make up the volume to 100 ml mark with water. Carry out a control test using 1 ml of standard chloride solution and the same quantities of other reagent in the same total volume of the reaction mixture. Compare the turbidity in both the cylinders after 5 minutes. The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

## **G.2 METHOD B (SPECTROPHOTOMETRIC (METHOD))**

### **G.2.1 Outline of the Method**

To a neutralized solution of sodium bicarbonate, addition of mercuric thiocyanate and ferric ammonium sulphate produces a highly coloured ferric thiocyanate complex, the intensity of which is proportional to the chloride content. The absorbance of this colour is measured at 450 nm wavelength.

### **G.2.2 Apparatus**

G.2.2.1 Standard Laboratory Apparatus 4

G.2.2.2 Spectrophotometer or Photocolorimeter

G.2.2.3 Platinum Evaporating Dish 60 ml capacity.

G.2.3 Reagents

G.2.3.1 Ferric Ammonium Sulphate Solution (0.25mol/l). Dissolve 49.02 g of Ferric Ammonium Sulphate Dodecahydrate  $[\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$  in 203 ml of dilute nitric acid (about 1.5 mol/l) and dilute to 500 ml with distilled water.

G.2.3.2 Mercury (II) Thiocyanate Solution Saturated solution in absolute alcohol.

G.2.3.3 Standard Sodium Chloride Solution Weigh accurately 0.165 0 g of previously dried (at 500°C) and cooled A.R. sodium chloride, dissolve in a beaker in distilled water and quantitatively transfer into a one litre volumetric flask, dilute to mark and mix well 1 ml of this solution = 100 µg of Cl.

G.2.3.3.1 Dilute Standard Sodium Chloride Solution Transfer 50 ml of standard sodium chloride solution into a 500 ml volumetric flask, dilute with distilled water to the mark and mix well. 1 ml of this solution = 10 µg of Cl.

G.2.3.4 Sulphuric Acid Solution Approximately 1 mol/l - Pour 56 ml of concentrated sulphuric acid (AR quality) into 944 ml of distilled water slowly, mix and cool.

## G.2.4 Procedure

### G.2.4.1 Calibration

Into a series of six 50-ml volumetric flasks, transfer standard sodium standard sodium chloride solution as given below:

Standard Sodium Chloride Solution ml	Corresponding Mass of Chloride (as Cl) µg
2	20
4	40
6	60
8	80
10	100

Add to each, 2 ml of ferric ammonium sulphate solution and 2 ml of mercury thiocyanate solution in that order. Dilute to mark and mix well. Allow to stand for 15 minutes, transfer to the cell of spectrophotometer and measure absorbance of 450 nm wavelength with compensation solution in reference cell. Plot chloride content in micrograms as abscissa vs. corresponding absorbance values as ordinates.

### G.2.4.2 Determination

Weigh suitable mass of sodium bicarbonate sample containing 50 to 150 µg of chloride (as Cl) correct to 1 mg and transfer into the platinum evaporating dish, wet this with 15 to 20 ml of distilled water and slowly neutralize with sulphuric acid solution add a few drops in excess. Evaporate to nearly dryness on hot water bath. Add 20 to 30 ml of distilled water and quantitatively transfer into a 100 ml volumetric flask, cool and make up to the mark with distilled water and mix well. Filter through Whatman No. 41 filter paper and discard a small volume of initial filtrate. Transfer suitable volume (25 to 40 ml) into 50 ml volumetric flask add 2 ml of ferric ammonium sulphate solution and 2 ml of mercury thiocyanate solution in that order, dilute to mark with distilled water and mix well. Allow to stand for 15 minutes, transfer to the cell of spectrophotometer and measure absorbance at 450 nm wavelength with distilled water in the reference cell.

### G.2.4.3 Blank Test

Carry out a blank test using the same procedure as used for determination using same quantities of all reagents but emitting the test solution. Instead of test solution use distilled water.

### G.2.4.4 Calculations

The chloride content is calculated from the formula:

$$\text{Chloride (as Cl) \% by mass} = \frac{M1 - M2}{M0 \times 10,000}$$

Where;

M1 = mass in micrograms of chloride corresponding to absorbance of test solution,



# KENYA STANDARD

DKS [2927:2020]

ICS [##.###]

**First Edition**

M2, = mass in micrograms of chloride corresponding to absorbance of blank solution,

M0, = mass in grams of sodium bicarbonate in the test portion of the aliquot used for color development.

**Annex H**  
(normative)  
**Test for substance reducing Iodine**

**H.1 REAGENTS**

H.1.1 Starch Solution

Titrate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water and boil for 3 minutes. Allow the solution to cool and decant off the clear liquid.

H.1.2 Standard Iodine Solution 0.01 N.

**H.2 PROCEDURE**

Dissolve 10 g of the material in 200 ml of freshly boiled and cooled water. Add 2 ml of starch solution and titrate with standard iodine solution. The material shall be taken to have passed the test if not more than 0.5 ml of iodine solution is required to produce a permanent blue colour.

**Annex J**  
(normative)  
**Test for Phosphates**

(SPECTROPHOTOMETRIC METHOD)

**J.1 Outline of the Method**

Silica is eliminated by precipitation and filtration. Formation of phosphomolybdic acid complex in acidic solution and reduction of yellow complex to molybdenum blue complex. Measurement of the intensity of the blue colour (which is proportional to phosphate concentration) using a spectrophotometer or photocolourimeter at 580 nm wavelength.

**J.2 Apparatus and reagents****J.2.1 Apparatus**

J.2.1.1 Standard Laboratory Apparatus

J.2.1.2 Spectrophotometer or Photoelectric Colorimeter

J.2.1.3 Platinum Evaporating Dish Approximately 60 ml capacity.

**J.2.2 Reagents**

J.2.2.1 Perchloric Acid

60 to 70% solution (m/m).

J.2.2.2 Ammonium Molybdate Solution

Dissolve 10 g of ammonium molybdate tetra hydrate  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  in 100 ml of water. Add slowly 220 ml of sulphuric acid (1:1 solution) and 70 ml distilled water. Mix and allow to cool.

J.2.2.3 Reducing Solution

Dissolve 0.25 g of 1-amino 2-naphthol 4-sulphonic acid and 0.5 g of anhydrous, sodium sulphite ( $\text{Na}_2\text{SO}_4$ ) in about 70 ml distilled water. Dissolve separately 24 g of anhydrous sodium bisulphite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) in about 100 ml distilled water. Mix both solutions and dilute to 200 ml with distilled water, filter, if necessary and store in a cool dark place.

J.2.2.4 Standard Phosphate Solution

Weigh accurately 1.433 g of finely powdered and dried A.R. potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ) dissolve in 100 to 150 ml distilled water in a 250 ml beaker, transfer quantitatively to 1 000 ml volumetric flask, dilute to mark and mix well. Transfer 50 ml of this solution to a 500 ml volumetric flask, dilute to mark and mix well. 1ml of this solution contains 100  $\mu\text{g}$  of phosphate (as  $\text{PO}_4$ ).

#### J.2.2.4.1 Dilute Standard Phosphate Solution

10  $\mu\text{g}$   $\text{PO}_4$  per litre - Transfer 50 ml of standard phosphate solution into a 500 ml volumetric flask, dilute to mark and mix well.

#### J.2.2.5 Hydrochloric Acid Solution

Approximately 6 mol/l - Add slowly 535 ml of concentrated hydrochloric acid (specific gravity 1.18) into 465 ml of distilled water and mix.

### J.3 Procedure

#### J.3.1 Calibration

Into a series of six 100ml volumetric flasks, transfer dilute standard phosphate solution as given below:

Standard Phosphate Solution, ml	Corresponding Mass of Phosphate as $\text{PO}_4$ , $\mu\text{g}$
0 (Compensation)	0
2	20
4	40
6	60
8	80
10	100

Add to each flask 2 ml of perchloric acid, 20 ml of distilled water, 10 ml of ammonium molybdate solution and finally 5 ml of reducing solution. Make upto the mark and mix well. Place flasks in boiling water-bath for 15 minutes, cool in running water and measure absorbance using 2 cm cell in a spectrophotometer (or photolorimeter) at 620 nm wavelength using compensation solution in the reference cell. Plot a graph with  $\mu\text{g}$  of  $\text{PO}_4$  as abscissae and corresponding absorbance values as ordinates.

#### J.3.2 Determination

Weigh about 1 g of sodium bicarbonate accurately to 1mg (the quantity to be weighed should be such that it contains 20 to 100  $\mu\text{g}$  of phosphate) and transfer into a 60 ml platinum evaporating dish, wet it with some distilled

water and neutralize with hydrochloric acid solution and add a few drops in excess. Evaporate to nearly dryness and add 2 ml of perchloric acid and again evaporate to nearly dryness. Add a few drops of hydrochloric acid solution and about 20 ml of distilled water and transfer quantitatively into a 100 ml volumetric flask. Cool and dilute to mark and mix well. Filter through a Whatman No. 41 filter paper, discard a small volume of initial filtrate and then transfer 50 ml of filtrate into 100 ml volumetric flask and develop colour as described under calibration

(J.3.1).

Measure absorbance at 620 nm wavelength using same size cell as used for calibration, with distilled water in the reference cell.

### J.3.3 Blank Test

Carry out a blank test using the same procedure and same quantities of all reagents as in the case of determination (K-3.4.2) except omitting the sample solution. Use distilled water instead of sample solution.

### J.4 Calculations

Phosphate (as PO<sub>4</sub>), percent by mass =  $\frac{M1-M2}{M0 \times 10,000}$

Where;

M1= mass in micrograms of phosphate corresponding to the absorbance of the test portion,

M2, = mass in micrograms of phosphate corresponding to the absorbance of the blank, and

M<sub>0</sub> = mass in grams of the sodium bicarbonate content corresponding to the test portion of the aliquot solution used for colour development.

# **Annex K**

## **(normative)**

### **Determination of Calcium and Magnesium**

#### **K.1 REAGENTS**

##### **K.1.1 Standard Calcium Solution**

Weigh 1.000 g of calcium carbonate dried at 120°C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to one litre in a graduated flask. One ml of this solution is equivalent to 0.400 08 mg of calcium (as Ca).

##### **K.1.2 Standard EDTA Solution**

Dissolve 3.72 g of disodium ethylene diamine tetra-acetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized frequently against the standard calcium solution, following the procedure given in K.2.1.

##### **K.1.3 Eriochrome Black-T Indicator Solution**

Dissolve 0.1 g of the dye in 20 ml of rectified spirit. This solution shall be prepared fresh every week.

##### **K.1.4 Ammonium Chloride-Ammonium Hydroxide Buffer Solution**

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp gr 0.90) and 2.50 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetra-acetate dihydrate and 0.616 g of magnesium sulphate ( $MgSO_4 \cdot 7H_2O$ ) in about 50 ml of water. Mix the two solutions and dilute to one litre.

NOTE-Five millilitres of the buffer solution added to 50 ml of distilled water should not consume more than a drop of EDTA solution to change to distinct blue with Eriochrome black-T indicator.

#### **K.2 PROCEDURE**

K.2.1 Standardization of EDTA Solution ;Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the Eriochrome black-T indicator solution and titrate against the EDTA solution to a pure blue end point.

K.2.2 Titrate 25 ml of the buffer solution with EDTA solution using Eriochrome black-T indicator. Subtract

the buffer correction for 10 ml (usually, it will be 0.1 ml) from the reading obtained in K.2.1 and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say A).

K.2.3 Transfer exactly 100 ml of solution preserved in B.2 into a 250 ml conical flask. Add 20 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of Eriochrome black-T indicator solution and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue endpoint. Note the volume of EDTA solution used in the titration.

#### **K.3 CALCULATION**

# KENYA STANDARD

DKS [2927:2020]

ICS [##.###]

**First Edition**

Calcium and magnesium (as Ca), percent by mass  $= \frac{AV_1}{M} \times 100$

where A = calcium equivalent calculated in g of 1 ml of EDTA solution determined in K.2.2,

V1 = volume in ml of standard EDTA solution used in K.2.3, and

M = mass in g of the material taken for the test in B.2.

# **Annex L**

## **(normative)**

### **Test for Nitrates**

#### **L.1 REAGENTS**

L.1.1 Dilute Sulphuric Acid

Approximately 5 N.

L.1.2 Concentrated Hydrochloric Acid

L.1.3 Potassium Nitrate Solution 0.001 N.

L.1.4 Concentrated Sulphuric Acid

L.1.5 Standard Indigo Carmine Solution

Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid and add 20 ml of concentrated hydrochloric acid and sufficiently dilute sulphuric acid to produce 1 000 ml. Standardize the solution so that 10 ml added to 3.3 ml of potassium nitrate solution is just decolourized on adding 13 ml of concentrated sulphuric acid and heating to boiling. One millilitre of this solution is equivalent to 0.02 mg of nitrate (as NO<sub>3</sub>).

#### **L.2 PROCEDURE**

Dissolve 2.000 g of the material in 10 ml of dilute sulphuric acid, and 10 ml of standard indigo carmine solution followed by 10 ml of concentrated sulphuric acid and heat to boiling. The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced in the test with the material does not entirely disappear.



**Annex M**  
(normative)  
**Determination of Potassium**

**M.1 APPARATUS**

M.1.1 Flame Photometer Equipped with interference filter.

**M.2 REAGENTS**

M.2.1 Standard Potassium Solution

Weigh exactly 1 g of potassium chloride, dissolve in distilled water and dilute to one liter with distilled water in a measuring flask. This solution contains 0.1 g KCl per 100 ml.

**M.3 PROCEDURE****M.3.1 Calibration Graph**

Take 10, 20, 40, 60 and 80 ml of standard potassium solution and dilute to 100 ml with distilled water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g KCl per 100 ml. Use these dilute solutions to obtain a corresponding galvanometer reading as given in M.3.2.1 and plot the concentration against galvanometer reading in a rectangular co-ordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.01 to 0.1 percent KCl. M.3.2 Sample Solution Dissolve approximately 10 g of the sample in minimum quantity of distilled water and dilute to 100 ml in measuring flask with distilled water.

**M.3.2.1 Determination**

Use a flame photometer equipped with atomizer, burner, optical selective device consisting of the reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element. Insert the potassium filter corresponding to wavelength 767 nm, light the burner fed by illuminating gas (laboratory gas), adjust the specified air pressure between 0.5 to 0.6 kg/cm<sup>2</sup> and maintain the above air pressure constant, such that flame is non-luminous by turning the control knob. First spray distilled water and adjust the pointer to zero in galvanometer scale by zero adjustment knob. Then spray the KCl standard solution and adjust the deflection to maximum 100 by using sensitivity control knob. Again, spray water to see that the pointer comes to zero; then spray a standard solution reads 100 with same adjustment during both the operations. A reading zero by water and with the same adjustment 100 by standard solution indicate that the instrument has been made ready for measurement.

Without altering the earlier adjustment of the instrument spray various diluted solutions prepared in M.3.1 and obtain a calibration graph in the range 0.0 1 to 0.1 percent KCl. After washing with distilled water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of KCl in the solution (say A).

#### **M.4 CALCULATION**

Potassium (as K), percent by mass =  $52.35 \frac{A}{M}$

Where;

A = concentration of KCl in the sample solution, And

M=mass of dried sample taken for the test.

**Annex N**  
(normative)  
**Test for copper**

**N.1 Outline of the Test Method**

The material is neutralized with hydrochloric acid. The copper in the soluble form is treated with diethyldithiocarbamate solution to produce a colored copper complex. This is extracted with carbon tetrachloride. The absorbance of the CCl<sub>4</sub> extracts measured at 440 nm using a spectrophotometer or a photocolourimeter.

**N.2.0 Apparatus**

N.2.0.1 Spectrophotometer or a Photocolourimeter Suitable for measuring absorbance at 440 nm.

N.2.0.2 Standard Laboratory Glass Apparatus

N.2.0.3 Platinum Dish, of about 60 ml capacity.

**N.2.1 Reagents**

N.2.1.1 Distilled Water

N.2.2.2 Hydrochloric Acid Approximately 6 mol/l - Dilute 500 ml conc HCl (sp gr 1.19) with 500 ml distilled water.

N.2.2.3 EDTA Solution 5 percent solution - Dissolve 50 g Ethylene diamine tetra-acetic acid, disodium salt (C<sub>10</sub>H<sub>14</sub> O<sub>8</sub>Na<sub>2</sub> · 2H<sub>2</sub>O) in distilled water, dilute to 1 000 ml and keep the solution in a polyethylene bottle.

N.2.2.4 Sodium Diethyldithiocarbamate Solution (DEDTC) Dissolve 0.1 g of DEDTC

[(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>N.CS<sub>2</sub>Na.3H<sub>2</sub>O] in 100 ml of distilled water and keep the solution in a dark bottle.

N.2.2.5 Standard Copper Solution Dissolve 0.100 g pure copper metal in the smallest possible quantity nitric acid in a 1 000 ml volumetric flask, cool and dilute to mark. One millilitre of this solution contains 100 µg copper. Transfer 10 ml aliquot of this solution (M-3.3.5) in to 100 ml volumetric flask, dilute to mark and mix. One millilitre of this solution is equivalent to 10 µg of copper.

N.2.2.6 Ammonium Hydroxide Solution Concentrated.

N.2.2.7 Carbon Tetrachloride Reagent grade CCl<sub>4</sub>.

N.2.2.8 Phenolphthalein Solution Dissolve 0.5 g phenolphthalein powder in 500 ml of 50-70 percent ethanol or methanol. Neutralize the solution by adding 0.1 mol/l NaOH until a faint pink color appears and then remove this color with a drop or two of 0.1 mol/l hydrochloric acid.

N.2.2.9 Hydrochloric Acid Solution Approximately 1 mol/l.

### N.3 Procedure

N.3.1 Calibration Into a series of six 250-ml separatory funnels, transfer standard dilute copper solution as given below:

Standard Copper Solution, ml	Corresponding Mass of Copper, $\mu\text{g}$
0 (Compensation)	0.0
2.0	20.0
4.0	40.0
6.0	60.0
8.0	80.0
10.0	100.0

Add to each 20 ml hydrochloric acid solution, 10 ml EDTA solution and then 5 drops of phenolphthalein solution and mix well. Neutralize each with concentrated ammonium hydroxide solution to a faint pink color and cool to room temperature. Add distilled water to each to a final volume of 45 ml. Then add to each 5 ml DEDTC solution mix well and add 20 ml carbon tetrachloride. Shake the funnel vigorously for at least 3 minutes and allow the phases to separate. Filter  $\text{CCl}_4$  phase (the lower phase) through a cotton plug inserted in the outlet stem of the separatory funnel into photometer cells. Apply the lid of the cell and measure absorbance at 440 nm with extract of copper free solution (compensation solution) as reference. Plot copper content ( $\mu\text{g}/20$  ml) as abscissae 'vs corresponding absorbance values as ordinates.

N.3.2 Determination Weigh accurately correct to 1 mg an amount of sodium bicarbonate sample containing 200 to 1 000  $\mu\text{g}$  of copper and transfer into a platinum dish. Wet it with minimum amount of distilled water and neutralize with 6 mol/l hydrochloric acid solution and add a few drops in excess. Evaporate to dryness on a water-bath. Wet it with a few drops of dilute hydrochloric acid solution and then dissolve in 20 to 30 ml of distilled water and quantitatively transfer into a 100 ml one mark volumetric flask, cool, make up to the mark and mix well. If the solution is turbid, filter through a Whatman No. 541 filter paper discarding 10 to 15 ml of initial filtrate. Transfer 10.0 ml of the aliquot volume (or the filtrate if filtered) into a 250-ml separatory funnel and proceed further exactly as described under calibration.

### N.3.3 Blank Test

Carry out a blank test exactly as described in M.3.4.2 but excluding the test solution, use distilled water instead. Read the corresponding lead content in the blank from the calibration graph.

### N.4 Calculations

By means of the calibration graph determine the mass of copper in micrograms corresponding to the absorbance value of the test solution.

# KENYA STANDARD

DKS [2927:2020]

ICS [##.###]

**First Edition**

Copper (as Cu), parts per million =  $\frac{M1-M2}{M0}$

Where;

M1 = mass of copper in micrograms in the test portion,

M2=mass of copper in micrograms in the blank, and

M0= mass of the sodium bicarbonate content in grams in the test portion used.

## Annex P (normative) Sampling

**Table E1 — Number of containers to be selected for sampling**

SI No	Lot size	No. of containers to be Selected
	N	11
(1)	(2)	(3)
i)	Up to 15	2
ii)	16 to 25	3
iii)	26 to 50	4
iv)	51 to 100	5
v)	101 to 300	6
vi)	301 to 500	7
vii)	501 to 800	8
viii)	801 to 1 000	9
ix)	1 001 and above	10

The containers shall be selected at random from a lot. In order to ensure randomness of selection random number tables shall be used.

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**DKS [2927:2020]**

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