

KENYA STANDARD

DKS 2436:2012

**Formaldehyde Solution for Industrial
Use—Specification**



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KENYA STANDARD

Formaldehyde Solution for Industrial Use —Specification

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Preface

This Kenya Standard was prepared by the Technical Committee on Industrial solvents and chemicals under the authority of Kenya Bureau of Standards.

Formaldehyde solution, also known as formalin is a colourless liquid and has a characteristic pungent, irritating odor. It is an important precursor to many other chemical compounds, especially for polymers. It is used in the manufacture of synthetic resins, dyes, explosives and other chemicals. It is also used in embalming fluids, and as a hardening and preservative agent, among other uses.

This Kenya Standard addresses important characteristics such as the pH, acidity, methanol and iron content

During the preparation of this standard, reference was made to the following publications and acknowledgement is made for its assistance with thanks:

- IS 3321:1973 Specification for formaldehyde solution
- D2087 – 06 Standard Test Method for Iron in Formaldehyde Solutions

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Formaldehyde solution for Industrial Use — Specification

1. SCOPE

This Kenya Standard specifies the requirements and methods of test and sampling for formaldehyde solution for industrial use. It does not, however, apply to the material intended for medicinal purposes.

2. REQUIREMENTS

2.1 Description — The material shall be clear and nearly colourless (see Note), having a characteristic pungent and irritating odour. It shall consist essentially of a solution of formaldehyde (HCHO) in water, with methanol as a stabilizing agent. It shall be free from foreign and precipitated matter.

Note – On long standing the solution may become cloudy due to separation of paraformaldehyde, which will clear when the solution is warmed.

2.2 The material shall comply with the requirements prescribed in table 1

Table 1 Requirements for formaldehyde solution			
	Characteristic	Requirement	Method of Test
1	Formaldehyde content	25% to 45%	KS ISO 2227:1972
(i)	Acidity (as HCOOH), percent by mass, Max	0.05	Annex A
(ii)	Ash, percent by mass, Max	0.01	Annex B
iii)	Aldehyde content (as HCHO), percent by mass (m/m),	37.0 ±0.5	Annex C
iv)	Iron (as Fe), parts per million, Max:		Annex D
	a) In non-iron containers	2	
	b) In iron containers	10	
v)	Methanol content, percent by mass	4 to 6	Annex E

2.3 The requirements for heavy metals (other than iron) and pH at 27 ± 1) shall be subject to agreement between the purchaser and the supplier. The tests prescribed under Annex F and G shall be followed.

3. PACKING AND MARKING

3.1 Packing — unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in air-tight containers made of stainless steel, aluminium or glass or in steel drums suitably lined with acid-resistant material. The material may also be packed in HDPE barrels. It is desirable to store it in a place not below 17°C.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information.

- a) Name of the material;
- b) Name of the manufacturer and recognized trade mark, if any;
- c) Net mass of the material; and
- d) Lot or batch number, in code or otherwise.
- e) Standardization mark

4 SAMPLING

4.1 The method of drawing representative samples of the test sample and the criteria for conformity shall be as prescribed in Annex H

Analysis of formaldehyde solution

Quality of reagents

Unless specified otherwise, pure chemicals and distilled water shall be used in test.

NOTE – ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

Annex A

Determination of Acidity

A-1.1 Reagents

A1.1.1 Standard Sodium Hydroxide Solution – 0.1 N.

A.1.1.2 Bromothymol Blue Indicator Solutions – Grind 0.4 g of powdered bromothymol blue in an agate mortar with 6.4 ml of the sodium hydroxide solution and dilute to 100 ml with freshly boiled and cooled distilled water.

A-1.2 Procedure – Mix 100 ml of the test sample with 100 ml of freshly boiled and cooled distilled water in a 500ml conical flask and titrate the mixture with standard sodium hydroxide solution, using four drops of bromothymol blue indicator.

A-1.3 Calculation

$$\text{Acidity (as HCOOH), percent by mass} = \frac{0.046VN}{S}$$

Where

V = Volume in ml of standard sodium hydroxide solution used,
N = normality of standard sodium hydroxide solution, and
S = relative density of the test sample at room temperature.

Annex B

Determination of ash

B.1 Procedure - Place 100 ml of the test sample in a 500-ml beaker on a hot plate and evaporate until the volume is reduced to 50 ml. Transfer the liquid quantitatively to a platinum or silica dish previously heated to $600 \pm 30^{\circ}\text{C}$ and cooled in a desiccator and weighed and evaporate to dryness on a boiling water-bath. Heat the dish and its contents gently at first to volatilize the paraformaldehyde and then at $600 \pm 30^{\circ}\text{C}$ in an electric furnace until the mass is constant. Cool in a desiccator and weigh.

B.2 Calculation

$$\text{Ash, percent by mass} = \frac{M}{S}$$

Where

M = mass in g of the residue, and
S = relative density of the test sample at room temperature.

Annex C

Determination of Iron

D.1 Method A (Thioglycollic Acid Method)

D.1.1 Reagents

D.1.1.1 Dilute sulphuric acid – 20 percent (v/v).

D.1.1.2 Thioglycollic acid – 10 percent solution (v/v) in water.

D.1.1.3 Hydrogen peroxide – 15 percent (m/m).

D.1.1.4 Standard iron solution – dissolve 7.022 g of ferrous ammonium sulphate in a mixture of 600 ml of water and 350 ml of dilute sulphuric acid, and dilute to 1 000 ml with water. Further dilute 10 ml of the solution so obtained to 1 000 ml with water. One millilitre of the dilute solution contains 0.1. mg of iron (as Fe).

D.1.2. Procedure

D.1.2.1 Place 50 ml of the test sample in the case of bulk consignment or 10 ml of the test sample in the case of a small container, in a 400-ml beaker and add to it 10 ml of dilute sulphuric acid. Evaporate the mixture on a hot-plate until acid fumes are evolved. Allow to cool to room temperature. Add to the cold solution, in successive small portions, 10 ml of hydrogen peroxide and heat on a hot-plate until acid fumes are evolved. Allow to cool again to room temperature and transfer the solution to a 50-ml Nessler cylinder. Wash the beaker with a small quantity of water and transfer the washings to the cylinder., Add 1 ml of thioglycollic acid solution to the mixture and make slightly alkaline with ammonia solution. Mix thoroughly and dilute with water to the graduation mark.

D.1.2.2 To 40 ml of water in another 50-ml Nessler cylinder, add 1 ml of thioglycollic acid and after making the mixture slightly alkaline with ammonia solution run in standard iron solution from a burette, shaking with each addition until the intensity of the colour in the two cylinders is identical when they are viewed along their axis. Record the volume of standard iron solution added.

D.1.2.3 At the same time carry out a blank test on the reagents alone, and determine, in the same manner as for the test, the amount of iron present.

D.1.3 Calculation

$$\text{Iron (as Fe), parts per million} = \frac{10(V_1 - V_2)}{VS}$$

Where

V_1 = volume in ml of standard iron solution added to Nessler cylinder in the case of the sample,

V_2 = volume in ml of standard iron solution added to another Nessler cylinder in the case of the blank test,

V = volume in ml of formaldehyde solution taken for the test, and

S = relative density of the formaldehyde solution at room temperature.

D.2 Method B (1,10-phenanthroline Method)

D.2.1 General – Ferrous iron gives with 1,10-phenanthroline a red coloured complex having maximum absorption at 550nm. The molecular absorption coefficient of the complex is 8 000. The iron is first reduced into ferrous state for complex formation.

D.2.2. Reagents

D.2.2.1 Dilute sulphuric acid – approximately 0.1 N solution.

D.2.2.2 Hydrogen peroxide – 15 percent (m/m).

D.2.2.3 Hydrogen ammonium chloride – 10 percent solution in water

D.2.2.4 Ammonium acetate – Dissolve 50 g of ammonium acetate in water and dilute to 100 ml

D.2.2.5 Standard iron solution – Dissolve 7.02 g ammonium ferrous sulphate $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$ in water containing 10 ml of concentrated sulphuric and dilute with water to 1 000 ml. One millilitre of this solution contains 1,0 mg of iron (as Fe). It may be diluted suitably to contain 10,25 μg of iron per millilitre

D.2.3 Procedure

D.2.3.1 To several aliquots of the standard iron solution containing 20, 40, 70, 100, 150 and 200 μg of iron, add 10 ml of sulphuric acid and evaporate on a sand-bath until white fumes are fast evolved. Allow to cool to room temperature and add in successive small portions 10 ml of hydrogen peroxide solution and heat on the sand-bath again to evolve out white fumes of sulphuric acid. Again cool and transfer the contents quantitatively to 100 ml one-mark flask. To each flask, add 2 ml of hydroxylammonium chloride solution, mix and allow to stand for 2 minute. Then add 30 ml of ammonium acetate solution and 5 ml of 1,10-phenanthroline - bipyridyl solution dilute to the mark and mix thoroughly.

Determine the absorbance against a reagent blank at 550 nm using a 1-cm cell. Plot absorbance against concentration of iron (in $\mu\text{g}/\text{ml}$).

D.2.3.2 Take an aliquot of clear solution of the material containing 0.01 to 0.02 mg of iron. Determine the absorbance of the test solution by treating an aliquot of the sample solution in the same manner as described above against a reagent blank using 1-cm cell. Read the concentration of iron (in $\mu\text{g}/\text{ml}$) from the calibration curve.

D.2.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{M_1 \times 100}{M_2}$$

Where

M_1 = mass in g of the iron found in the sample solution, and

M_2 = mass in g of the material taken for the test

Note: For automated spectrophotometer, a direct reading of concentration will be obtained

D.3

D.3.1 Summary of Test Method

The specimen is evaporated and ashed; the iron, reduced to the divalent state by the addition of hydroxylamine hydrochloride, is reacted with *o*-phenanthroline to develop a color that is measured at 510 nm.

D.3.2 Apparatus

Spectrophotometer, capable of measuring light absorption at 510 nm.

Absorption Cells, minimum light path, 10 mm.

Evaporating Dishes, 90-mm diameter, high-silica glass.

D.3.3 Reagents and Materials

Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water.

Ammonium Acetate Solution (100 g/L)—Dissolve 100 g of ammonium acetate ($\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$) in 100 mL of water. Add 200 mL of acetic acid (CH_3COOH) dilute to 1 L with water, and mix.

Ammonium Hydroxide (1 + 1)—Mix equal volumes of concentrated ammonium hydroxide and water.

Congo Red Paper.

Hydrochloric Acid (1 + 1)—Mix equal volumes of concentrated hydrochloric acid and water.

Hydroxylamine Hydrochloride Solution (100 g/L)—Dissolve 10 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in water and dilute to 100 mL.

Iron, Standard Solution (1 mL = 0.05 mg Fe)—Dissolve 0.3510 g of ferrous ammonium sulfate ($\text{FeSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$) in 50 mL of water and 20 mL of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84). Dilute with water to 1 L in a volumetric flask and mix.

**o*-Phenanthroline Solution (1 g/L)*—Dissolve 0.1 g of *o*-phenanthroline in 10 mL of iron-free ethyl alcohol and dilute to 100 mL with water.

D.3.4 Calibration

Prepare a series of standards by adding the reagents described above to 0.2, 0.5, 1.0, 2.0, 3.0, 4.0, and 5.0-mL of standard iron solution in 100-mL volumetric flasks, and diluting to volume. Make spectrophotometer comparisons in the absorption cells and prepare a calibration curve by plotting the absorbances of the standard iron solutions against the milligrams of iron per 100 mL of solution. This curve must be determined for each instrument and should be checked periodically.

D.3.5 Procedure

Clean a high-silica glass evaporating dish as follows: Add 10 mL of HCl (1 + 1), cover with a watch glass, and digest on a steam bath for about 20 min. Then discard the HCl solution, rinse the dish with water, and dry.

Weigh 50 g of sample into the cleaned dish and evaporate to dryness on an electric hot plate in a hood. If any organic matter remains, ignite for 5 min over a high temperature gas burner.

Add 10 mL of HCl (1 + 1), cover with a watch glass, and digest on the steam bath for 15 min. Transfer quantitatively to a 100-mL volumetric flask. Add the following reagents in order, mixing after the addition of each: 1 mL of NH₂OH·HCl solution, 5 mL of o-phenanthroline solution, enough NH₄OH (1 + 1) as required to make the solution just alkaline to Congo red paper (approximate pH range 3.0 to 5.0), and 5 mL of NH₄C₂H₃O₂ solution. Dilute to the mark with water and mix thoroughly.

Allow to stand for 5 min, fill an absorption cell, and measure the absorbance at 510 nm with the spectrophotometer.

From the calibration curve, read the milligrams of iron present.

Carry a blank containing no added iron through all steps of the procedure in order to correct for any iron contamination in the reagents. Correct the results obtained above accordingly.

D.3.6 Calculation

Calculate the parts per million of iron *I* as follows:

$$I = (W/S) \times 1000$$

where:

W = weight, of iron found mg, and

S = weight of sample used, g.

Annex D Determination of Methanol Content

Methods – Two methods, namely, Method A and Method B have been prescribed. In case of dispute Method B shall be used as a reference method.

E.1 Method A (Graphical Method)

E.1.1 procedure

E.1.1.1 Determine the relative density of the material at 25/25^oC by any suitable method capable of giving a result accurate to 0.001.

E.1.1.2 Read the value of methanol content percentage by mass from the graph in Fig. 1 in which the relationship between the formaldehyde content (as determined in annex C), relative density at 25/25^oC and methanol content is plotted.

E.2 Method B (Chemical Method)

E.2.1 Reagents

E.2.1.1 Concentrated sulphuric acid

E.2.1.2 Chromic oxide solution – Mix 83.3 g of pure oxide of chromium (Cr₂O₃) with 688 ml of concentrated sulphuric acid to get a clear homogeneous solution. Filter through a Gooch crucible if necessary

E.2.1.3 Sodium thiosulphate solution – 0.1N.

E.2.1.4 Potassium iodide solution – 40 percent

E.2.1.5 Starch indicator – Mix 1 g of starch with 10 ml of cold water and pour with constant stirring into 200 ml of boiling water. Allow to settle and use the clear supernatant liquid.

E.2.2. Procedure – Take 10 ml of the material into 1000-ml measuring flask and fill up to the mark with distilled water. Measure 20 ml of this solution and add to it about 10 ml of chromic oxide solution contained in a round-bottom flask fitted with ground-glass stopper. Heat the above mixture under reflux on a boiling water-bath for half-an-hour. Cool the contents and transfer quantitatively to a 100-ml volumetric flask and fill up to the mark with distilled water. Take 10 ml of this solution and mix with 5 ml of potassium iodide solution and titrate with sodium thiosulphate solution using starch as an indicator.

E.2.3 Calculation

$$\text{Methanol, percent by mass} = [10xf - \left(\frac{v}{2} + \%CH_2O \times 0.133 d \right)] \times 5$$

Where

f= factor of chromic acid solution,

v= volume in ml of 0.1 N sodium thiosulphate solution used in the titration, and

d= density in g/ml of formaldehyde solution.

E.2.3.1 Determination of factor– Pipette 10ml of chromic acid solution into 20ml of distilled water contained in a 100-ml round-bottom flask fitted with ground-glass stopper and reflux the contents on a boiling water water-bath for about 30 minutes. Cool and transfer the contents into a 100-ml volumetric flask and fill up to the mark with distilled water. Mix 10ml of this solution with 5 ml of potassium iodide solution and titrate against sodium thiosulphate solution using starch as an indicator.

$$\text{Then, factor } (f) = \frac{V}{20}$$

Where

V=volume in ml of sodium thiosulphate solution used in the titration.

Annex E Test for heavy metals as lead

F.1 Reagents

F.1.1 Hydrochloric Acid-approximately 1 N

F.1.2 Sodium Sulphide Solution –. This solution shall be prepared fresh.

F.1.3 Standard Lead Solution – Weigh to the nearest 0.2 mg, 0.016 g of lead nitrate. Place in a 1 000-ml one-mark volumetric flask and dissolve in water and fill up to the mark. One millilitre of this solution contains 0.01 mg of lead (as Pb).

F.2 Procedure – Take 20 ± 0.2 g of the material in a Nessler cylinder and add 2.5 ml of hydrochloric acid solution. Dilute the contents to 100 ml with water and add Sodium Sulphide. To another Nessler cylinder containing 80 ml of water, add an agreed volume of standard lead solution followed by 2.5 ml of hydrochloric acid solution and dilute to 100 ml.

F.2.1 The material shall be taken to have passed the test if the colour produced with the material is not darker than that produced with the standard lead solution.

Annex F Determination of pH

G.1 Apparatus

G.1.1 pH Meter – Use a standard laboratory pH meter

G.2 Procedure – Take about 5 g of the material and dissolve in 100ml water. Allow 5 minutes time and measure the pH of the solution at $27 \pm 1^{\circ}\text{C}$ using a standard pH meter.

Annex G Sampling of Formaldehyde Solution

H.1 General requirements for sampling

H.1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

H.1.2 The sampling instrument shall be clean and dry.

H.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

H.1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

H.1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers with which the material does not react.

H.1.6 The sample containers shall be of such a size that an ullage of about 5 percent is left after pouring in the sample.

H.1.7 Each sample container shall be sealed air-tight after filling, and marked with the full details of sampling, the date of sampling and details given under 3.2.

H.2 Scale of sampling

H.2.1 Lot – All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the 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.

H.2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Lot Size	Sample Size
N	n
(1)	(2)
Up to 25	3
26 to 50	4
51 " 100	5
101 to 200	6
201 to 300	7
301 to 500	8
501 to 800	9
801 and above	10

H.2.3 In order to ensure the randomness of selection, random sampling procedure shall be followed

H.3 Individual samples and composite sample

H.3.1 From each of the containers selected according to H-2.3, a representative portion of the material about 300 ml in volume, shall be drawn. These samples shall constitute individual samples.

H.3.2 From each of these individual portions (H-3.1) an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 600ml in volume. The composite shall be divided into three equal parts, one for the purchaser, one for the supplier and the third for the reference.

H.3.3. Referee Sample – The referee sample consists of the composite sample marked for this purpose, and shall bear the seals of purchaser and the supplier. It shall be kept at a place agreed to between the two and shall be use in case of any dispute between the two.

H.4 Number of tests

H.4.1 Tests for the determination of all the characteristics given in Table 1 shall be carried out on the composite sample.

H.5 Criteria for conformity

H.5.1 For declaring the conformity of the lot to the requirements of all the characteristics, the test results on the composite sample shall meet the corresponding requirement specified.