KENYA STANDARD **DKS 2472:2013**

FOOD GRADE HEXANE - SPECIFICATION

TECHNICAL COMMITTEE REPRESENTATION

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Agrochemical and Food Company
Kenyatta University
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Government Chemist
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REVISION OF KENYA STANDARDS

In order to keep abreast of progress in industry, Kenya Standards shall be regularly reviewed. Suggestions for improvements to published standards, addressed to the Managing Director, Kenya Bureau of Standards, are welcome.

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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.

Hexane is a hydrocarbon which is a colourless liquid at room temperature.

Food grade Hexane is mainly used as a solvent in various kinds of extraction operations. The formula for n-Hexane is $CH_3(CH_2)_4CH_3$.

This Kenya Standard addresses the requirements of food grade hexane, test methods and packaging and marking requirements

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

- IS 3470:2002: - Hexane, Food Grade - Specification

KENYA STANDARD

FOOD GRADE HEXANE - SPECIFICATION

1. SCOPE

This Kenya Standard specifies requirements and methods of test for hexane, food grade, used as a solvent for extraction of oily /oleaginous materials.

2. REQUIREMENTS

Food Grade Hexane shall be a petroleum distillate fraction containing a high proportion of n-hexane and shall be clear, colourless liquid with characteristic petroleum like odour, free from sediment, suspended matter and moisture. Food grade hexane shall also satisfy the requirements specified in the Table 1 below:

Table 1: Requirements for Food Grade Hexane

SL.NO	Characteristics	Requirements	Method of test
1	Appearance	Clear and free from suspended matter	Visual
2	Colour, Lovibond scale, Hazen Units	10 maximum	Annex A
3	Density,g/ml at 20°C	0.660-0.687	KS 1180
4	Distillation range at 1013 mbar, °C IBP D.P	 63 95	KS 1180
5	Refractive Index at 20°C	1.375-1.384	KS 1180
6	Residue on evaporation g/100ml,	0.0005 max	KS 1180
7	Odor	Characteristic petroleum like odour	KS 914
8	Reaction of non-volatile residue	Neutral to methyl orange	Annex B
	Benzene content, % v/v Max	0.05	KS 2470 Annex C
	Sulfur, mg/kg, Max	5	KS 2470 Annex F
9	Lead, as Pb, mg/kg, Max	1	Annex D
10	Polycyclic aromatic hydrocarbon	To pass the test	Annex C

4. MARKING AND PACKING

Food Grade Hexane shall be packaged in suitable and serviceable containers and marked indelibly with the following information:

(a) Name of the product i.e. Food Grade Hexane.
(b) Manufacturer's name and/or registered trade mark; and address
(c) Country of origin
(d) Net weight;
(e) Batch Number
(f) The words 'inflammable' and its Standard Symbol;
(g) Date of Manufacture
(h) Precautionary measures i.e. may cause irritation to the eyes, respiratory system and skin.

ANNEX A

DETERMINATION OF COLOUR IN HAZEN UNITS (LOVIBOND SCALE)

A.1 PRINCIPLE

Visual comparison of the colour of a sample with that colour of standards, and expression of the result in terms of hazen

Note:For routine control purposes an instrument such as a comparator, colorimeter or spectrophotometer may be used, provided that it has first been established that the results so obtained are identical with those obtained by visual comparison.

A.2 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

A.3 APPARATUS

Ordinary laboratory apparatus and the Lovibond comparator

A.3.1 Two colorimetric tubes, flat based if possible, with a graduation mark at least 100 mm above the base and matched especially with respect to colour of glass and height of graduation mark above the base. Suitable tubes are available commercially as 50 mL or 100 mL Nessler Cylinders.

For the measurement of low colorations (less than 50 Hazen Units), the height of the graduation mark above the base must be greater than for the measurement of deeper colours and must be sufficient that, on looking through this greater depth of liquid, clear distinction between the standard Hazen matching solutions can be observed.

NOTE: 1. For routine control purposes, a colorimetric or spectrophotometer may be used, the instrument being standardized by means of the standard colorimetric solutions provided that it has been confirmed that the use of that instrument gives the same results as does visual comparison.

A.4 Expression of Results

Express the colour of the sample as the number of Hazen colour units

A.5 TEST REPORT

The test report shall include the following particulars:

(a) the reference of the method used

(b) the result, expressed in Hazen colour units

(c) any unusual features noted during the determination

(d) any operation not included in this standard or regarded as optional.

Annex B
Neutrality Test (Reaction of Non-Volatile Residue)

G.1 Reagent
Methyl orange shall be used as an indicator.

G.2 Procedure
Dissolve 0.01g of methyl orange in 100ml of water.
To a conical flask containing the non-volatile residue as in KS 914, add 100ml of distilled water and shake the contents thoroughly. Transfer the clean aqueous layer to a clean test tube by means of a pipette. Add one drop of methyl orange solution.
The residue shall be taken to have passed the test if no pink or red colour is formed.

Annex C
Determination of Polycyclic Aromatic Hydrocarbons

C.1 All glassware should be scrupulously cleaned to remove all organic matter such as oil, grease, detergent residue and other related materials. Examine all glassware including stopper and stopcocks under ultraviolet light to detect any residual fluorescent contamination. Rinse all glassware with iso-octane immediately before use because some polynuclear hydrocarbons sought in this test are susceptible to photo-oxidation. This procedure shall be carried out under subdued light.

C.2 Apparatus
C.2.1 Separatory funnel-125ml capacity equipped with tetraflouroethylene (PTFE) polymer stopcocks
C.2.2 Spectrophotometric cells – fused quartz cells, optical path length in the range of 5.0 ± 0.005 cm.
C.2.3 Spectrophotometer spectral range of 200-450 with spectral slit width of 2nm or less and with Wavelength accuracy of ±1.0 and absorbance accuracy of ±0.05 at 0.4 absorbance.

C.3 Reagents

C.3.1 iso-octane (2,2,4-trimethyl Pentane)- UV spectroscopic grade
C.3.2 Dimethyl sulphoxide (DMSO) –UV spectroscopic grade
C.3.3 n-Hexane - UV spectroscopic grade
C.3.4 Naphthalene Analytical grade

C.4 Procedure
C.4.1 Prepare a standard solution of naphthalene in iso-octane containing 7.0mg of naphthalene per litre. Measure absorbance of the standard solution at 275nm using iso-octane as blank.

C.4.2 Add 25 ml of n-Hexane to a 125ml separating funnel. Add 5ml of dimethyl sulphoxide. Shake vigorously for 1 minute and allow to stand until two clear layers are formed. Collect the clear lower layer and use as a blank to measure the absorbance of test sample

C.4.3 Take two 125ml separating funnels. Transfer 25ml of sample to the first 125ml separating funnel and add 25ml of n-Hexane. Mix and add 5ml of DMSO. Shake vigorously for 1 minute and allow to stand until two clear layers are formed.

C.4.4 Transfer the lower layer to a second separating funnel, add 2ml of n-Hexane and shake the mixture vigorously. Allow to stand until two clear layers are formed. Separate the lower layer and measure the absorbance over the range 260nm to 420nm against the blank.

C.5 At any wavelength in the range 260nm to 420nm, the absorbance of the sample shall not exceed one third that of the standard solution at 275nm

Annex D
Determination of Lead

D.1 Introduction

The sample is treated with bromine, heated on a steam bath to decompose the alkyl lead and alkyl lead salts, and then extracted with dilute nitric acid. The pH of the aqueous extract is adjusted by means of a buffer and the lead is extracted with a chloroform solution of dithizone. The absorbance of the chloroform extract is measured and the lead content is determined from a previously prepared calibration curve.

D.2 Apparatus
D.2.1 The glassware should be borosilicate and confirmed to be lead-free.
D.2.2 Spectrophotometer –Fitted with covered absorption cells having a 1 cm light path.
D.2.3 Shaking machine (optional) capable of approximately 250 rpm
D.2.4 Separatory Funnel -125 ml volume

D.3 Reagents

D.3.1 Analytical grade reagents and distilled water shall be used.

D.3.2 Bromine Solution- Dilute300ml of Bromine to 1000ml with chloroform. Filter through a sintered glass filter before using.

D.3.3 Buffer Solution –Dissolve 20g of Potassium Cyanide (KCN), 6g of ammonium citrate, and 6g of sodium sulphite (Na_2SO_3) separately in water. Mix the solutions, add 150ml of concentrated ammonium hydroxide, (specific gravity 0.90) and dilute to 1000ml with water. This solution is stable for 3 months (in a refrigerator).

D.3.4 Dithizone solution – Dissolve 30mg of Dithizone in 1000ml of chloroform. This solution is stable for only 4 weeks.

D.3.5 Lead standard solution – (1ml = 0.005 mg Pb). Dissolve 1.5985 of lead nitrate [$Pb(NO_3)_2$] in 250ml of water contained in a litre volumetric flask. Add 8 ml of concentrated HNO_3 dilute to volume with water..., and mix thoroughly. For calibration purposes, pipette 5.0 ml of this solution into a 1 litre volumetric flask, add 8ml of concentrated HNO_3 and dilute to volume with water.

D.3.6 Nitric acid-Concentrated nitric acid

D.3.7 Nitric Acid – Mix 8ml of concentrated nitric acid with 992 ml of water.

D.4 Calibration

D.4.1 Prepare a calibration curve using the lead solution (1 ml = 0.005 mg Pb) as follows:
Pipette 0.0, 2.0, 5.0, 10.0 and 15.0 ml of the solution respectively into each of the five separatory funnels and dilute each solution to 50 ml with nitric acid (D.3.7). Treat these solutions as described in D.5.3 and D.5.4 using one not containing lead as a blank.

D.4.2 Construct a calibration curve by plotting the absorbance of the solution against the mg of lead per 25 ml dithizone solution.

D.5 Procedure

D.5.1 Pipette 50g of the sample into a 250ml beaker. Add bromine solution until the bromine colour persists for at least 2 minutes, then allow the beaker to stand for an additional 5 minutes. At the same time prepare a blank by adding the same amount of bromine solution to 25.o ml of nitric acid (D.3.7) in a 250ml beaker. Place the beaker on a steam bath and heat until the bromine colour disappears. Place the beakers on a hot plate and bring the solutions to a vigorous boil. Cool to room temperature and transfer each solution quantitatively, into separating funnels using 25.0 ml of nitric acid (D.3.7). Shake the test sample for 2 minutes.

D.5.2 Drain the aqueous extract of the sample into another separatory funnel. Repeat the extraction of the sample using 25.0 ml of nitric acid (D.3.7). Drain the aqueous layer into the separatory funnel containing the initial extract. Transfer the blank solution quantitatively to a 50 ml volumetric flask using 25 ml of nitric acid (D.3.7). Dilute to the mark with water and drain the contents of the flask into a separatory funnel.

D.5.3 Add 120 ml of the buffer solution to both separatory funnels to adjust the Ph to a point between9.5 and 11.0. Pipette 25 ml of dithizone solution and shake for 2 minutes.

D.5.4 Drain and discard a small portion of the chloroform layer from the funnel to remove any water or lead that may have accumulated in the stem. Transfer a portion of each chloroform layer into separate absorption cells. Adjust the spectrophotometer to read zero absorbance for the blank and then measure the absorbance of the sample with respect to the blank at a wavelength of 510nm.

D.5.5 Convert the absorbance measurement to concentration of lead in milligrams of lead per 25 ml of Dithizone solution by means of the previously prepared calibration curve.

D.6 Calculation

Calculate the lead content in mg per Kg as follows:

Lead, mg/Kg $= \frac{100A}{50} = 20A$

Where A =Lead concentration, in mg/25ml of dithizone solution corresponding to the measured absorbance.