# DRAFT TANZANIA STANDARD

## TBS/CDC 2 (5794) P3 - Linear alkylbenzene - Specification



**TANZANIA BUREAU OF STANDARDS**



First Edition 2018

## Foreword

This Draft Tanzania Standard is being developed by the Soap and Detergents Technical Committee under supervision of the Chemicals Divisional Standards Committee and it is in accordance with the procedures of the Bureau.

In the preparation of this Draft Tanzania Standard assistance was drawn from IS 12795: 1989 (Reaffirmed 2016) Linear alkyl benzene - specification; Prepared by the Indian Standards Institutes

In reporting the results of analysis of a test if the final value is to be rounded off, it shall be done in accordance with TZS 4 *Rounding off numerical values*



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# Linear alkylbenzene - Specification

## 1 Scope

This Draft Tanzania Standard prescribes the requirements and the methods of sampling and test for linear alkylbenzene. The standard is intended for Linear alkylbenzene suitable for detergents preparation.

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 35, *Soaps – Sampling and test methods*

TZS 1396-2/ISO 672*Soaps — Determination of moisture and volatile matter content — Oven method*

TZS 59/ISO 3696 *Water for analytical laboratory use – Specification and test method*

ISO 3839 *Petroleum products -- Determination of bromine number of distillates and aliphatic olefins -- Electrometric method*

ISO 1523 *Determination of flash point -- Closed cup equilibrium method*

ASTM D1298 *Standard Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method*

ASTM D4952 *Standard Test Method for Qualitative Analysis for Active Sulfur Species in Fuels and Solvents (Doctor Test)*

ASTM D7344 *Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Mini Method)*

ASTM D156 *Standard Test Method for Saybolt Colour of Petroleum Products (Saybolt Chromometer Method)*

## 3 Requirements

### 3.1 General requirements

Linear alkylbenzene shall be a clear colourless liquid with a desirable odour.

### 3.2 Specific requirements

The material shall also comply with requirements specified in Table 1.



 

**Table 1 – Requirements for Linear alkylbenzene**

| S/N | Characteristic | Requirement | Method of test |
|---|---|---|---|
| **i.** | Colour,<br><br>Saybolt, *Min* | <br><br>30 | <br><br>ASTM D156 |
| **ii.** | Specific gravity at 15°C/15°C | 0.858 – 0.868 | ASTM D1298 |
| **iii.** | Moisture, mg/kg, *Max* | 100 | TZS 1396-2/ISO 672 |
| **iv.** | Doctor test | Negative | ASTM D4952 |
| **v.** | Flash point (closed cup) °C, *Min* | 130 | ISO 1523 |
| **vi.** | Bromine number., *Max* | 0.05 | ISO 3839 |
| **vii.** | Ready biodegradability of sulphonate* | 92 | Annex A |
| **viii.** | Distillation, 95 percent recovery at °C, *Max* | 320 | ASTM D7344 |
| **ix.** | Molecular weight | 236 - 246 | Annex B |
| **x.** | Paraffins, percent by mass, *Max* | 0.2 | Annex C |
| **xi.** | Sulphonability, percent by mass, *Min* | 98 | Annex D |
| **xii.** | Chain-length distribution, percent<br>a) Less than $C_{10}$, *Max*<br>b) $C_{10}$, *Max*<br>c) $C_{10} + C_{11}$<br>d) $C_{12}$<br>e) $C_{13}$, *Max*<br>f) $C_{14}$, *Max* | <br>0.5<br>15.0<br>30.0 – 55.0<br>25.0 – 50.0<br>30.0<br>5.0 | Annex E |
| *Linear alkyl benzene is to be sulphonated before carrying out ready biodegradability test, as given in Annex D | | | |



## 4 Packaging and labelling

### 4.1 Packaging

The material shall be packed in moist-free container as agreed to between the purchaser and the supplier.

### 4.2 Labelling

The container shall be securely closed and labelled with the following particulars:

a) Name of the material;
b) Name and address of the manufacturer;
c) Recognized trade mark (if any)
d) Country of origin;
e) Net volume of the product;

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f) Batch number or lot number; and
g) Month and year of manufacture (refer on MSD)

**5 Sampling**

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in TZS 35.

**6 Quality of Reagents**

Unless specified otherwise analytical chemicals and distilled water (*see* TZS 59) shall be used in tests.



 

## Annex A

(Normative)

## Determination of biodegradability of sulphonates ( shake culture method)

### A.1 Outline of the method

In this test, micro-organisms are inoculated into a flask that contains a chemically defined microbial growth medium (basal medium) and the surfactant to be tested. Aeration is accomplished by continuous shaking of the flask. Following two adoptive transfers, biodegradation is determined by measuring the reduction in surfactant content during the test period.

### A.2 Apparatus

Shaking Machine

A reciprocating shaker operating at about 128 strokes of 51 to 101.6 mm/mm or a gyratory shaker operating at 225 to 250 rev/min with an amplitude of 25 to 51 mm should be used. Other shakers may be used if equivalent aeration can be demonstrated.

### A.3 Reagents

**A.3.1** *Purity of Water*

Either distilled or deionized water may be used; must be free of bacteriostatic materials. Water derived from steam condensate in many cases may contain amines which are inhibitory to microbial growth.

**A.3.2** *Basal Medium*

**A.3.2.1** The composition of the basal medium shall be as described in Table 2 below:

Table 2: Composition of the basal medium

| **Material** | **Quantity (g)** |
|---|---|
| Ammonium chloride ($NH_4Cl$) | 3.0 |
| Potassium hydrogen phosphate ($K_2HPO_4$) | 1.0 |
| Magnesium sulphate heptahydrate($MgSO_4.7H_2O$) | 0.25 |
| Potassium chloride (KCl) | 0 25 |
| Ferrous sulphate ($FeSO_4.7H_2O$) | 0.002 |
| Yeast extract | 0.30 |
| Water (see A.3.1) | 1.01 |



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**A.3.2.2** The basal medium may be prepared by sequentially dissolving the dry ingredients in water, or by adding stock solutions of the salts. The yeast extract shall be added in dry form immediately before use, or alternatively, solutions containing yeast shall be sterilized and maintained sterile if held for more than 8 hours before beginning the test. The basal medium shall be dispensed into one of the following flasks: 500 mL in a 1 L flask, 1 000 mL in a 2 L flask and 1 500 mL in a 4 L flask. 1 L and 2 L flasks are best suited for a gyratory shake and the 4 L flask for a reciprocating shaker.

**A.3.3** *Microbial Culture*

**A.3.3.1** *Source*

The microbial inoculum may be obtained from any of the following sources.

**A.3.3.1.1** Natural sources (soil, water, sewage, activated sludge, etc).

**A.3.3.1.2** Laboratory cultures (activated sludge, river die-away, etc).

**A.3.3.2** *Maintenance of Culture*

If desired, the culture may be maintained as a shake flask culture by weekly transfers in the basal medium. For each weekly transfer use mL of 7-day culture for each 100 mL of fresh medium.

**A.4 Procedure**

Add 30 mg/L surfactant to the flasks containing basal medium. If surfactant stock solutions are used,stability during storage shall be confirmed, use one flask for each surfactant being tested and one blank flask containing all basal medium components but with no surfactant. Using the culture described in **A.3.3** inoculate the flasks. Use the same culture for all flasks including blank. Use 1 mL of inoculum for each 100 mL of basal medium in the flask. Place the flasks containing basal medium, surfactant and inoculum on a shaking machine that will produce acceptable aeration and mixing for biodegradation. Maintain the temperature of the flask contents at 25°C ± 3°C.

Prior to beginning the biodegradation test, make two 72 h acclimation transfers from the flasks. To follow the course of biodegradation, removesamples from the shake flasks for analysis. Samples shall be taken during the 8-day test at zero time (immediately after inoculation and mixing of the flask contents) and on the seventh and eighth days. Samples at zero time of the two adaptive transfers are desirable to ensure proper initial concentration. Unless analyses are run immediately, the addition of 1 mL of formaldehyde/100 mL of sample shall beused for preservation of any sample (0 time or 7 or 8 days). When preservative is used, add to all samples including blank. Since the analytical result from the blank sample is used to correct the results from other flasks, use the same sample size (or dilution factor) for the blank as used for the other samples

**A.5 Calculation**

Calculate net surfactant concentration by subtracting the analyzed blank values from the analyzed values for the other flasks. Calculate the percentage removal from the reduction in surfactant concentration as follows:

Percentage removal (day *x)*

$$= \left[\frac{(S_0 - B_0) - (S_r - B_z)}{(S_0 - B_0)}\right] \times 100$$

Where;

$S_0$ and $S_r$= analysis of test surfactant cultures, and

$B_0$and $B_z$= analysis of blank cultures, on days 0 and x, respectively.



The result shall be calculated as the average of the seventh and eighth day percentage removals.

## Annex B

(Normative)

## Determination of molecular weight

## (Gas liquid chromatographic method)

### B.1 Outline of the method

The gas liquid chromatogram of the sample is recorded as described in **C.1** and assignments made as in **B.2.**

### B.2 Apparatus

Gas liquid chromatography

### B.3 Procedure

**B.3.1** The linear alkylbenzene (LAB) sample is injected and the chromatogram is obtained as per the method described in **C.3**. Calculate the weight percent of all the linear alkylbenzene isomers; for example, $C_{10}$, $C_{11}$, $C_{I2}$ ... etc; after adjusting its area percent with response factors obtained by injecting the synthetic blend under identical conditions. Add up all the $C_{10}$ isomers which will be the total $C_{10}$-LAB mass percent in the sample. Likewise calculate for all the carbon number LAB's such as $C_{11}$-LAB, $C_{12}$-LAB, etc. Calculate the total linear alkyl benzene content in the sample by adding up all weight percentages of $C_{10}$-LAB, $C_{11}$-LAB, etc.

### B.4 Calculation

**B.4.1** The percentage of different homolog LAB with respect to total linear alkyl benzene in the sample is calculated as follows:

$$X = \frac{Y}{Z} \text{ X } 100$$

where

*x* = weight percent of LAB-$C_{10}$, LAB-$C_{11}$….etc, with respect to LAB in the sample;

*Y* = weight percent of LAB-$C_{10}$, LAB-$C_{11}$….etc, in the sample; and

Z = total linear alkyl benzene in the sample.

**B.4.2***Average molecular weight*

The average molecular weight of linear alkylbenzene is obtained by multiplying eachpercentage homolog LAB, as obtained from **B.4.1**, With its molecular weight and adding up all thevalues.

$$M = \frac{(A \times C_{10} + B \times C_{11} + C \times C_{12} + D \times C_{13} + E \times C_{14} + \cdots)}{100}$$

where

*M* = average molecular weight of linear alkyl benzene;

$C_{10}$, $C_{11}$ ... etc = weight percent of LAB-$C_{10}$, LAB-$C_{11}$, etc; and

*A, B,* C. ... etc = molecular weight of LAB-$C_{10}$, LAB-$C_{11}$, etc

## Annex C

(Normative)

## Determination of Paraffins

### C.1 Outline of the method

The sample to be analyzed is injected into a gas chromatograph that is equipped with fused silica capillary column. A quantitative blend, containing known amount of normal paraffins in linear alkyl benzene (LAB) is chromatographed under identical conditions. The *calibration chromatogram* thus developed is used to ascertain the normal paraffins in the sample by comparing the retention of the normal paraffins to the observed retention times of the components obtained in the synthetic mixture. Quantitative results are obtained from the measured areas of the recorded peaks, adjusted for differences in response by the use of factors determined from the analysis of synthetic blend.

### C.2 Apparatus

Gas liquid chromatography

### C.3 Procedure

**C.3.1**Inject $C_{10}$ to $C_{14}$ individual normal paraffins namely decane, undecane dodecane, tridecane and tetradecane into GC and find out the retention times of each paraffin. Prepare a synthetic blend containingclose to 0.2 by mass of total normal paraffin in linear alkyl benzene. Now inject the synthetic blend and run the chromatogram. Identify the normal paraffin peaks from $C_{10}$ to $C_{14}$. Calculate the area per-
cent of individual normal paraffin and sum it up to get total area percent of the total normal paraffin in the blend. Calculate the area response factor as given in **C.4.1**.

**C.3.2** Inject linear alkylbenzene sample and run the chromatogram. From the area percent of total normal paraffin calculates the weight percent as given in **C.4.2**.

### C.4 Calculation

**C.4.1** The area response factor is calculated as follows:

$$F = \frac{W}{A}$$

Where;

*F* = response factor;

*W* = weight percent of total normal paraffin in the blend; and

*A* = area percent of total normal paraffin in the blend;

**C.4.2** The weight percent of tot at normal paraffin in linear alkyl benzene as follows:
Percent total normal paraffin in linear alkyl benzene = *B x F*



where

*B* = area percent of total normal paraffin obtained from the chromatogram; and

*F* = response factor of total normal paraffin.

## Annex D

(Normative)

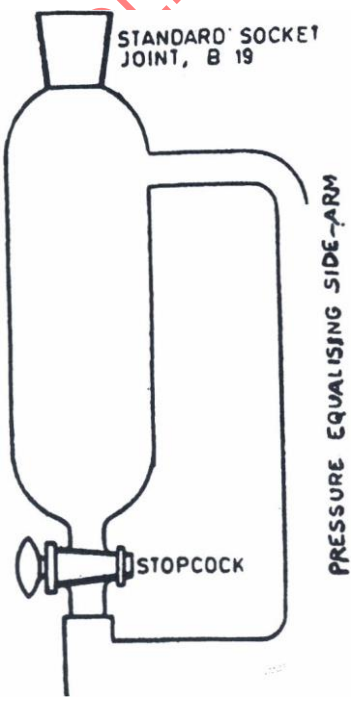
## Determination of Sulphonability

### D.1 Outline of the method

A known mass of linear alkyl benzene of known molecular mass is sulphonated with 20% oleum. Knowing the mass of the product after sulphonation and its active matter content, the percent sulphonability is calculated.

### D.2 Apparatus

A 500-ml capacity 4-necked round bottom flask, with standard joints, is fitted with a mercury-sealed stirrer, thermometer (0 °C– 110 °C) and a 100ml, capacity addition funnel with pressure equalizing side-arm *(see* Fig. 1). A guard-tube filled with granular, fused calcium chloride is inserted into the fourth neck of the flask.




....Standard socket joint, B 24 (check correctness of drawings)

Fig. 1 Apparatus for determination of Sulphonability



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**D.3 Reagents**

**D.3.1***20% Oleum*

**D.3.2***Purified Hyamine* 1622

Commercially-available, Hyamine 1622 is crystallized 6 to 7 times from chloroform-petroleum ether (60°C-80°). In the last crystallization, it is dissolved in chloroform and filtered through filter paper to removetraces of dust and dirt. Then, the chloroform solution is concentrated on a hot-water bath and petroleum ether is added to the hot solution until it is slightly hazy. This solution is then cooled, scratching the sides with a clean glass rod. The separated crystals are filtered in a Buchner funnel under vacuum and the cake of crystals pressed dry with an inverted bottle stopper. The crystals are then scraped gently from the filter-paper into a glass dish and dried subsequently on a steam-bath. The melting point of the dried crystals is 163.5°C.

**D.4 Procedure**

**D.4.1**The *empty* 500-ml round bottomed flask,fitted with stirrer and thermometer (partly coming in contact with the reaction mixture) is weighed, to the second decimal 100.00 g of the linear alkyl benzene sample is now weighed into it. The mercury seal and calcium chloride guard tube are fitted. 120g of 20% oleum is weighed into the addition funnel, which is stoppered at once and fitted to the 4-necked flask. The linear alkyl benzene is then cooled with stirring in a bath containing ice and water to 20°C. Dropwise addition of the 20% oleum is started. The reaction is exothermic, but, by adding more ice to the bath, reaction temperature is held below 20°C and a fairly fast rate of addition of oleum is maintained. After all the oleum is added, reaction mixture temperature is allowed to come up to 40°C, by replacing ice and water in the bath with warm water. The reaction mature is maintained at 40°C with stirring for 1 h. The calcium chloride guard tube, additional funnel and mercury seal are removed. The outside of the flask is wiped dry and weighed, as before, with stirrer and thermometer, to the second decimal place. The net weight of the contents after sulphonation is thus found out. Its anionic active matter content is then found out as given in D.4.2 below.

**D.4.2***Determination of Anionic Active Matter*

**D.4.2.1** *Sample solution preparation*

Weigh a suitable quantity of the sample containing100 mg to 160 mg of anionic active matter per 100 mL of solution. For this take an appropriate quantity of sample and neutralize with required quantity of sodium hydroxide solution to get sodium alkyl benzene sulphonate. About1 g of sodium alkyl benzene sulphonate per500 mL of the solution is suitable.

**D.4.2.2** *Procedure*

Pipette 10 mL sample solution into a 100 mL graduated cylinder provided with a glass stopper. Add 15 mL of chloroform and 25 mL of methylene blue reagent to the cylinder then Shake well. The chloroform layer (lower)shall be coloured blue or greenish blue.Add from the burette hyamine 1622 solution(see D.3.2) slowly, initially in portions of0.2 mL. After each addition, stopper the cylinder,shake well and allow the phases to separate. Initially the chloroform phase will be colouredblue or greenish blue. Towards the end the colour would start migrating to the aqueouslayer. Note the reading at which the colour intensity in both the phases is the same when viewed under standard conditions of light, for example, against a white porcelain tile under normal daylight.

Calculate the molarity of hyamine 1622 solution (i.e. benzethonium chloride solution) as follows:

Molarity of benzethonium chloride solution, $T_2 = \frac{10\ T_1}{V_1}$



 

where

$T_1$ = molarity of sodium lauryl sulphate solution, and

$V_1$ = Volume in ml of benzethonium chloride solution added.

**D.5 Calculation**

Percent sulphonability = $\frac{M_1 \times A \times M_2}{100(M_2+80)}$

Where

$M_1$ = netmass of reaction mixture after sulphonation;

$A$ = percent active matter of the sulphonated mixture; and

$M_2$ = average molecular mass of the sample.



**Annex E**

(Normative)

**Determination of chain length distribution**

The gas liquid chromatogram of the sample is taken and recorded as described in **C.3** and assignments made as in **B.3**. Thereafter, the chain length distribution of the sample is calculated as given in **B.4.1**.