DRAFT TANZANIA STANDARD

g and Ar as Cc Stakeholders Denatured Ethanol for Use as Cooking and Appliance Fuel -

TANZANIA BUREAU OF STANDARDS

Foreword

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

This Tanzania Standard has been prepared with assistance drawn from:

ASTM E3050 – 16 Standard Specification for Denatured Ethanol for Use as Cooking and Appliance Fuel Standards, published by ASTM International.

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DRAFT TANZANIA STANDARD

CDC 19 (720) DTZS

Denatured Ethanol for Use as Cooking and Appliance Fuel — Specification

1 Scope

This Draft Tanzania Standard specifies requirements, sampling and tests method for denatured ethanol for use as cooking or heating appliance fuel, or both.

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 644 / ISO 3170 Petroleum liquids - Manual sampling.

TZS 1445/ISO 3165 Sampling of chemical product for industrial use - Safety in sampling.

TZS 2273-2/ISO 10101-2 Natural gas - Determination of water by the Karl Fischer method - Part 2: Titration procedure.

TZS 2273-3 / ISO 10101-3: Natural gas — Determination of water by the Karl Fischer method — Part 3: Coulometric procedure.

FTZS 2522/ISO 1388-2 Ethanol for industrial use Methods of test – Part 2: Detection of alkalinity or acidity to phenolphthalein.

ASTM D4815 Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography published by ASTM International.

ASTM D6045 – 20 Standard Test Method for Colour of Petroleum Products by the Automatic Tristimulus Method, published by ASTM International.

ISO 22854 Liquid petroleum products — Determination of hydrocarbon types and oxygenates in automotive, motor gasoline and in ethanol (E85) automotive fuel — Multidimensional gas chromatography method, published by International Organization for Standardization (ISO).

3 Terms and definitions

For the purpose of this standard, the following terms and definition shall apply;

3.1 denatured ethanol,

n - ethanol made unfit for beverage use by the addition of toxic or noxious materials

3.2 higher alcohols,

n - aliphatic alcohols of general formula $C_nH_{2n}+1OH$ with n being 3 to 8

3.3 hydrocarbon,

n - components in an ethanol-hydrocarbon blend containing only hydrogen and carbon

4 Requirements

4.1 General requirements

- **4.1.1** The product shall be visually free of sediment and suspended matter.
- **4.1.2** The product shall be free of any adulterant or contaminant that can render the material unacceptable for its commonly used applications.
- **4.1.3** The product shall be colored to visually indicate that it is not potable.

4.2 Specific requirements

The product, when tested according to the methods prescribed, shall comply with the specific requirements given in Table 1.

Table 1 Specific requirements for Denatured Ethanol.

S/N	Characteristic	Requirement	Method of test
i.	Ethanolic strength , % volume, Min	90	Annex A
ii.	Water, volume %,Max	10	TZS 2273-2 or
			TZS 2273-3
iii.	Higher Alcohols (C ₃ –C ₈), volume %, Max	2	ASTM D4815
iv.	Hydrocarbon ^A , % volume, Max		ISO 22854
V.	Acidity (as acetic acid), mg/kg, Max	40	FTZS 2522/ISO 1388-2
vi.	Denatonium Benzoate, mg/Kg, (Min–Max)	10-20	Annex B
vii.	Colored Dye, mg/Kg, Max	10	ASTM D6045

A The hydrocarbons approved for use under this specification are as follows: gasoline, unleaded gasoline, natural gasoline, heptane, or rubber hydrocarbon solvent.

5 Packing and labelling

5.1 Packing

The product shall be supplied and stored in clean, dry and tight containers, without faults, made of material which does not react with alcohol. The method of closing the containers shall prevent the contents from contamination and evaporation.

5.2 labelling

Each container shall bear the following information given in prominent, legible and durable labelling:

- a) manufacturer's name.
- b) recognized trade mark, if any.
- c) name of the product as "Denatured ethanol"
- d) intended use.
- e) name of denaturant
- f) nominal content
- g) nominal strength.
- h) batch number.
- i) manufacturing date
- j) Pictorial symbols to show flammability and toxicity nature.
- k) Instruction of use
- I) storage instructions

Sampling

The product shall be sampled according to the method specified in TZS 1445 / ISO 3165 (see clause 2).

7 Testing

Tests shall be carried out according to the methods prescribed in **Table 1** of this standard (see clause 4.2).

For Stakeholder's comments only

Annex A

(normative)

Relationship between density in air and alcoholic strength

Table 2 gives the relation between density in air and alcoholic strength, expressed in either percentage by volume, or as a percentage by mass, at a 20 °C.

Table 2: Relationship between density in air and alcoholic strength

Density in air (g/mL)	Alcoholic strength at	t 20 °C
20 °C	% (<i>m/m</i>)	% (v/v)
0.8281	85.7	90.0
0.8264	86.3	90.5
0.8248	87.0	91.0
0.8231	87.6	91.5
0.8213	88.3	92.0
0.8196	89.0	92.5
0.8178	89.6	93.0
0.8160	90.3	93.5
0.8141	91.0	94.0
0.8122	91.7	94.5
0.8103	92.4	95.0
0.8083	93.1	95.5
0.8063	93.8	96.0
0.8043	94.6	96.5
0.8022	95.3	97.0
0.8000	96.1	97.5
0.7978	96.8	98.0
0.7955	97.6	98.5
0.7932	98.4	99.0
0.7907	99.2	99.5
0.7882	100.0	100.0

Note: For comprehensive ethanolic strength table refer Annex C

Annex B

(normative)

Determination of Denatonium Benzoate in Alcoholic Products by HPLC-UV

B.1 Principles

This document describes a standard method for the determination of denatonium benzoate (DB) in CDA (completely denatured alcohol) formulations using HPLC with UV detection at 210 nm. The samples are directly injected into the HPLC system after membrane filtration. The working range for quantitative determination of DB is 0.5 to 20.0 mg / L.

B.2 Instrumentation and materials

- **B.2.1.** HPLC system equipped with.
 - Pumping system suitable for isocratic elution.
 - Solvent degassing system (on-line/off-line).
 - Injection system with 20 µl loop.
 - Analytical column, for example: LiChrospher 100 CN (5 μm) in LiChroCART 250-4 guard column
 - Thermostated column compartment (oven).
 - Diode array detector (DAD) or UV detector.
 - Integrator or computer with data acquisition software and printer.
- **B.2.2.** Analytical balance with a precision of 0.1 mg.
- **B.2.3.** 0.5, 2, 5, 10 and 20 ml pipettes.
- B.2.4. 100 and 1000 ml volumetric flasks.
- **B.2.5.** Weighing bottle.
- B.2.6. Syringes.
- B.2.7. 0.45 µm cellulose membrane filters
- B.2.8 250 ml beaker.

B.2.12 1000 ml graduated cylinder.

With regard to the HPLC column, the recommended analytical column in this method is LiChrospher 100 CN (5 μ m) as described above. However, alternative HPLC columns (C₁₈ / C₈), buffers and chromatographic parameters may be used provided that good peak shape is obtained for denatonium benzoate and good separation of denatonium benzoate from potential interferences can be achieved.

B.3 Reagents and solutions.

- **B.3.1** Denatonium benzoate, purity ≥ 99 %. Handle it with gloves.
- **B.3.2** Ethanol 96 % vol.
- **B.3.3** Sodium chloride, extrapure.

B.3.4 Acetonitrile, HPLC grade.

B.3.5 Water, HPLC grade.

B.3.6 0.2 % sodium chloride solution.

Weigh 0.4 g of sodium chloride in a weighing bottle and dissolve it in a beaker with 200 ml of water HPLC grade.

B.3.7 Mobile phase

Add in a 1000 ml volumetric flask, 200 ml of 0.2 % sodium chloride solution and 800 ml of acetonitrile HPLC grade.

B.4 Standard solutions

B.4.1 Preparation of the stock solution (100 mg DB / L).

Weigh, recording the exact weight, 0.1 g of denatonium benzoate in a weighing bottle and dissolve it in a 1000 ml volumetric flask with ethanol 96 % vol. Mix gently.

Measure the mass of this solution with a top loading balance and the density at 20°C with an electronic densimeter.

B.4.2 Preparation of the working calibration solutions.

Add, in 100 ml volumetric flasks, 20 ml of ethanol 96 % vol. (to minimize weighing errors), then 0.5, 2, 5, 10 or 20 ml (weighing) of the stock solution and top up to the filling mark with ethanol 96 % vol. Mix gently.

B.5 Chromatographic and calibration parameters.

When using LiChrospher 100 CN (5 µm) column, chromatographic and calibration parameters recommended are:

- Column flow: 1.2 ml / min.
- Stoptime: 14 min.
- Detector: signal 210 nm (bandwidth 8 nm), reference 360 nm (bandwidth 100 nm).
- Mobile phase: acetonitrile 80:20 0.2 % sodium chloride solution.
- Injection volume: 20 µl.
- Column oven temperature: 27°C.
- Calibration: external standard.
- Signal: peak area.
- Curve type: linear.
- Origin: included.
- Weight: equal.

B.6 Calibration.

Working solutions containing the following concentrations of denatonium benzoate 0.5, 2, 5, 10 and 20 mg / I are analysed by injecting one replicate of each working solution. Peak areas corresponding to denatonium benzoate are plotted according to the respective concentrations in order to obtain a linear regression line expressed by the formula y = ax + b. The correlation coefficient must be > 0.99. Otherwise, the system must be checked to improve the linear regression if possible, or the working solutions must be discarded and a new set of calibration solutions should be prepared.

B.7 Analysis of samples

No specific sample preparation is required. The samples are directly injected into the HPLC system after 0.45 µm cellulose membrane filtration.

B.8 Instrument Results:

ine s. is calibrated the contribution of the c The instrument denatonium benzoate results are calculated by comparing the sample denatonium peak area response to the calibration curve for the denatonium benzoate. This calibration curve is part of the

Annex C

(Informative)

Comprehensive alcoholimetric table

For Stakeholder's comments only

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IX b q	20.5	899.33 39.33 39.33	89.0 88.9 88.8 88.8 88.7 88.6 88.5	88.4 88.3 88.3 88.2 88.1 88.1 88.1 87.9 87.9	87.8 87.6 87.6 87.4 87.4 87.2 87.2	87.1 87.0 87.0 86.9 86.9 86.8 86.7 86.7 86.7 86.7	86.5
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790.0	788.0 788.2 788.4 788.6 788.8 789.2 789.2 789.4 789.8	786.0 786.2 786.4 786.6 786.8 787.0 787.2 787.4 787.4 787.5	784.0 784.2 784.4 784.6 784.8 785.0 785.2 785.2 785.6	782.0 782.2 782.4 782.6 782.8 783.0 783.2 783.4 783.6 783.8	780.0 780.2 780.4 780.6 780.8 781.0 781.2 781.4 781.6 781.8	6	<u>.</u>
						~	1
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99.4	99.8 99.7 99.6 99.6 99.5 99.5 99.4	100.0 99.9 99.9 99.8 99.8				23.0	IX b q
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99.2	99.5 99.5 99.5 99.4 99.3 99.3	100.0 99.9 99.9 99.8 99.8 99.8 99.8 99.7 99.7				24.0	
99.1	99999999999999999999999999999999999999	99.9 99.8 99.8 99.8 99.7 99.7 99.6 99.6 99.6	100.0			24.5	
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