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| **ECOWAS** |  | **DHS ECOSTAND XX: 2022** |
| **STANDARD** |  |  |

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|  | | **Cassava Products - Ethanol for Industrial use - Specification** | |
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**Foreword**

The Economic Community of West African States (ECOWAS) was established on 28th May 1975 by Heads of States and Governments of fifteen (15) Member States as an Economic Community of the Region. The Treaty was reaffirmed in 1993.

One of the important mandates of ECOWAS is to promote the establishment of Common Market, the development and harmonization of Standards and conformity assessment procedures and Measures in order to reduce Technical Barriers to Trade, encourage intra and international Trade as well as enhance the industrialization of the region.

ECOWAS Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The work of preparing ECOWAS Standards is normally carried out through ECOWAS Technical Committees. Each member body interested in a subject for which a Technical Committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ECOWAS, also take part in the work.

The main task of the Technical Committees is to prepare ECOWAS Standards. Draft ECOWAS harmonized Standards adopted by the technical Committees are circulated to the member states for voting. Publication as an ECOWAS Standard requires approval by at least 75% of the member states casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ECOWAS shall not be held responsible for identifying any or all such patent rights.

The work of preparing this standard was carried out by the ECOWAS Technical Harmonization Committee 3 (THC3) *Chemistry.*

**ECOWAS REGIONAL STANDARD DHS ECOSTAND XX: 2022(E)**

**Cassava Products - Ethanol for Industrial use – Specification**

1. **Scope**

This standard specifies requirements and methods of sampling and test for ethanol for industrial use.

1. **Normative references**

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

1. *ECOSTAND XX, Cassava Products – Good manufacturing practices (GMP)*
2. *ECOSTAND XX, Cassava Products — Packaging and Labelling*
3. *ISO 759, Volatile organic liquids for industrial use — Determination of dry residue after evaporation on water bath — General method*
4. *ISO 1388-2, Ethanol for industrial use — Methods of test — Part 2: Detection of alkalinity or determination of acidity to phenolphthalein*
5. *ISO 1388-3, Ethanol for industrial use — Methods of test — Part 3: Estimation of content of carbonyl compounds present in small amounts — Photometric method*
6. *ISO 1388-5, Ethanol for industrial use — Methods of test — Part 5: Determination of aldehydes content — Visual colorimetric method*
7. *ISO 1388-6, Ethanol for industrial use — Methods of test — Part 6: Test for miscibility with water*
8. *ISO 1388-7, Ethanol for industrial use — Methods of test — Part 7: Determination of methanol content (methanol contents between 0,01 and 0,20 % (V/V)) — Photometric method*
9. *ISO 1388-8, Ethanol for industrial use — Methods of test — Part 8: Determination of methanol content (methanol contents between 0,10 and 1,50 % (V/V)) — Visual colorimetric method*
10. *ISO 1388-12, Ethanol for industrial use — Methods of test — Part 12: Determination of permanganate time*
11. *ISO 2096, Glycerols for industrial use — Methods of sampling*

**3. Terms and definitions**

For the purpose of this standard, the following definitions and terms shall apply:

**3.1**

**ethanol**

colourless volatile flammable liquid which is produced by the natural fermentation of sugars

**3.2**

**foreign matter**

* inorganic matter such as sand, glass, metal, stones, clay and mud and
* organic matter such as chaff, straw, weed, seeds and insects or insect fragments, rodent hairs

**4. Quality Requirements**

**4.1 General quality requirements**

**4.1.1**Ethanol shall be produced in accordance with ECOSTAND XXX on Good manufacturing practices for cassava products.

**4.1.2** Ethanol shall be:

a) clear, colourless homogenous liquid, free from foreign matter in suspension when assessed visually; and

d) free of any foreign smell, odour and/or taste uncharacteristic of the product.

* 1. **.3 Raw Material**

Ethanol shall be prepared from Cassava roots complying with ECOSTAND XXX free from any fungal and bacterial contamination.

* 1. **Physicochemical requirements**

Ethanol shall conform to the requirements in Table 1, when tested in accordance with the appropriate test method as stated in Table 1.

**Table 1 – Physicochemical requirements of ethanol**

|  |  |  |
| --- | --- | --- |
| **Parameters** | **Limits** | **Test Methods** |
| Ethanol content at 200C, % (v/v), min | 95 | Annex A |
| Miscibility with water (ethanol:water), 1:19 (v/v) | No Opalescence shall be observed | ISO 1388-6 |
| Acidity (as CH3COOH), % (m/m), *max* | 0.005 | ISO 1388-2 |
| Residue on evaporation, % (m/m), *max* | 0.005 | ISO 759 |
| Carbonyl compounds content, (as acetaldehydes), % (m/m), *max* | 0.01 | ISO 1388-5 |
| Alkalinity, % (m/m), *max* | 0.005 | ISO 1388-2 |
| Permanganate time, minutes, min | 30 | ISO 1388-12 |
| Methanol content (CH3OH), % (v/v), *max* | 0.01 | ISO 1388-7 or  ISO 1388-8 |

1. **Packaging and labelling**
   1. **Packaging**

**5.1.1** Packaging shall be done in accordance with requirements in ECOSTAND XXX standard on packaging and labelling for cassava products.

* + 1. Type of packaging materials shall be as agreed on between manufacturer and customer in accordance with regional regulations.
    2. All containers used shall be dry, clean, and free from substances soluble in ethanol.
    3. Ethanol shall be packaged in a well sealed container to prevent evaporation and contamination

**5.2 Labelling**

In addition to the requirements of ECOSTAND XX (standard of packaging and labelling for cassava products), the following specific provisions apply:

* + 1. **Labelling of retail containers**
* The name of the product to be shown on the label shall be “ethanol”;
* The percentage purity; and
* Safety precaution with the words “highly inflammable”.
  + 1. **Labelling of non-retail containers**

Information for non-retail containers shall either be given on the container or in accompanying documents, except that the name of the product, lot identification and the name and address of the manufacturer or packer shall appear on the container. However, lot identification and the name and address of the manufacturer or packer may be replaced by an identification mark, provided that such a mark is clearly identifiable with the accompanying documents.

1. **Methods of analysis and sampling**

Sampling shall be done in accordance with ISO 2096(to change).Testing shall be done in accordance with the methods indicated against each requirement or other equivalent validated methods.

**ANNEX A**

**(Normative)**

**DETERMINATION OF ETHANOL CONENT**

**A-1 GENERAL**

Two methods have been prescribed for determination of ethanol content or ethanolic strength. Both the methods can be used for determination of ethanol content on routine basis. However, in the event of any dispute, Method 2 should be treated as a referee.

**A-2 METHOD 1- ALCOHOLOMETRIC METHOD USING HYDROMETER**

Determine the ethanol content of anhydrous ethanol using method below:

The table 3 below gives the relation between density in vacuo, density in air and ethanolic strength, expressed either as a percentage by volume, or as a percentage by mass, at 20°C. The values for density in vacuo are taken in from 'Practical alcohol tables', volume 2, issued by the Commission of the European Communities. The values for density in air are calculated from the formula relating the temperatures (in °C), the percentage by mass of ethanol and the densities in vacuo (in kg/m3) contained in 'Recommendation No. 22' of the International Organisation of Legal Metrology. Densities in vacuo have been converted into density air and that of the masses adopted in 'Recommendation No. 33' of that Organization.

**Table A.1: Relation between density in vacuo, density in air and ethanolic strength**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Ethanolic strength  at 20 °C | | Density in vacuo (kg/m3) as indicated  by EEC pattern glass hydrometer at | | | | Density in air  (kg/m3)\* |
| %(*V/V*) | % (*mlm*) | 10 °C | 15 °C | 20 °C | 25 °C | 20 °C |
| 90.00 | 85.66 | 837.6 | 833.4 | 829.2 | 825.0 | 828.10 |
| 90.50 | 86.31 | 836.0 | 831.8 | 827.6 | 823.4 | 826.44 |
| 91.00 | 86.97 | 834.2 | 830.2 | 826.0 | 821.6 | 824.75 |
| 91.50 | 87.63 | 832.6 | 828.4 | 824.2 | 820.0 | 823.05 |
| 92.00 | 88.29 | 830.8 | 826.6 | 822.4 | 818.2 | 821.31 |
| 92.50 | 88.96 | 829.2 | 825.0 | 820.8 | 816.4 | 819.55 |
| 93.00 | 89.64 | 827.4 | 823.2 | 819.0 | 814.6 | 817.77 |
| 93.50 | 90.32 | 825.6 | 821.4 | 817.2 | 812.8 | 815.95 |
| 94.00 | 91.01 | 823.6 | 819.4 | 815.2 | 811.0 | 814.10 |
| 94.50 | 91.70 | 821.8 | 817.6 | 813.4 | 809.2 | 812.22 |
| 95.00 | 92.41 | 819.8 | 815.6 | 811.4 | 807.2 | 810.30 |
| 95.50 | 93.12 | 818.0 | 813.8 | 809.6 | 805.2 | 808.34 |
| 96.00 | 93.84 | 816.0 | 811.8 | 807.6 | 803.2 | 806.34 |
| 96.50 | 94.57 | 813.8 | 809.6 | 805.4 | 801.2 | 804.29 |
| 97.00 | 95.31 | 811.8 | 807.6 | 803.4 | 799.2 | 802.19 |
| 97.50 | 96.05 | 809.6 | 805.4 | 801.2 | 797.0 | 800.04 |
| 98.00 | 96.81 | 807.4 | 803.2 | 799.0 | 794.8 | 797.82 |
| 98.50 | 97.59 | 805.2 | 801.0 | 796.8 | 792.6 | 795.54 |
| 99.00 | 98.38 | 802.8 | 798.6 | 794.4 | 790.2 | 793.17 |
| 99.50 | 99.18 | 800.2 | 796.2 | 792.0 | 787.8 | 790.72 |
| 100.00 | 100.00 | 797.8 | 793.6 | 789.4 | 785.2 | 788.16 |
| **\*Values for density in air are quoted to five significant figures because it is possible to determine density in air to a greater accuracy than the density in vacuo using a hydrometer.** | | | | | | |

**A-3 METHOD 2- GAS CHROMATOGRAPHIC METHOD**

**A-3.1 General**

This test method covers the determination of the ethanol content and other components such as aldehyde, ester (as ethyl acetate), methyl alcohol, n-propanol and iso-amyl alcohol of denatured rectified spirit by gas chromatography.

**A-3.1.1** The chromatographic analysis given here is for information and guidance only

**A-3.1.2** Water cannot be determined by this test method

**A-3.1.3** This test method is inappropriate for impurities that boil at temperatures higher than 225°C or for impurities that cause poor or no response in a flame ionization detector, such as water.

**A-3.2** Summary of Test Method

**A-3.2.1** A representative aliquot of the ethanol sample is introduced into a gas chromatograph equipped with a methyl silicon bonded phase fused silica capillary column. Suitable carrier gas transports the vapourized aliquot through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The ethanol, methanol and other components are identified by comparing their retention times to the ones identified by analyzing standards under identical conditions. The concentration of all components are determined in mass percent area by normalization of the peak areas.

**A-3.3** **Apparatus**

**A-3.3.1** Gas Chromatograph, capable of operating at the conditions listed below. A heated flash vaporizing injector designed to provide a linear sample spilt injection (for example, 2;1) is required for proper sample introduction. Carrier gas controls shall be of adequate precision to provide reproducible column flows and split ratios in order to maintain analytical integrity. Pressure control devices and gauges shall be designed to attain the linear velocity required in the column used. A hydrogen flame ionization detector with associated gas controls and electronics designed for optimum response with open tabular columns is required.

**A-3.3.2** Sample introduction - Manual or automatic liquid syringe sample injection to the splitting injector is employed. Devices capable of 0.1 to 0.5 ul injections are suitable. It should be noted that inadequate splitter design, poor injection technique and overloading the column can result in poor resolution. Avoid overloading, particularly of the ethanol peak and eliminate this condition during analysis.

**A-3.3.3** Column – This test method utilizes a fused silica open tabular column with non-polar methyl silicon bonded (cross-linked) phase internal coating. Any column with equivalent or better chromatographic efficiency and selectivity to those described in A-3.3.3.1 can be used.

**Table A-2 - Column temperature programme**

|  |  |
| --- | --- |
| Column | Capillary column coated with 6 percent cyanopropylphenyl, 94 percent dimethylpoly- siloxane |
| Column length | 30 m |
| Internal diameter | 0.53 mm |
| Film thickness | 3 µm |
| Initial temperature | 40°C |
| Initial hold time | 5 min |
| Programme rate | 30°C/ min |
| Final temperature | 230 ̊C |
| Final hold time | 2 min |

|  |  |
| --- | --- |
| **Injector** Temperature | 140°C |
| Split ratio | 2;1 |
| Sample size | 0.1 to 0.5 µl |

**Detector**

|  |  |
| --- | --- |
| Type | Flame ionization |
| Temperature | 240°C |
| Carrier gas | Nitrogen (3 ml/min) |

**Table A-3 Approximate Retention Time**

|  |  |  |
| --- | --- | --- |
| SI No | Name of the component | Retention times (min) |
| i) | Acetaldehyde | 3.96 |
| ii) | Methyl alcohol | 4.20 |
| iii) | Ethyl alcohol | 5.65 |
| iv) | n- Propanol | 9.12 |
| v) | Ethyl acetate | 10.53 |
| vi) | Iso-Butanol | 12.75 |
| vii) | Benzene | 13.46 |
| viii) | Crotonaldehyde | 14.83 |
| ix) | n-Butanol | 15.50 |
| x) | Acetal | 17.78 |
| xi) | Iso- amyl alcohol | 20.88 |

**A-3.4 Procedure**

Separately inject 0.5 µl each of standard and sample and record the chromatogram. Calculate the acetaldehyde, methanol, ethyl acetate, n-n- propanol and iso-amyl alcohol in the sample by area normalisation method. A typical gas chromatogram using FID showing retention time is given in Fig . A.1

NOTE- Retention time may change slightly as per actual GC conditions.

