



DRAFT TANZANIA STANDARD

Methods of sampling and test for crude and refined glycerine
Part 7: Determination of sulphated ash — Gravimetric method

Draft Standard for Comment Only

TANZANIA BUREAU OF STANDARDS

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Foreword

This Draft Tanzania Standard was prepared under the direction of Cosmetics and Cremeries Technical Committee, which is under the supervision of Chemicals Divisional Standards Committee.

For the purpose of deciding whether a particular requirement of this Tanzania Standard is complied with, the final value, observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with TZS 4 (see clause 2).

This second edition cancels and replaces the first edition (TZS 1199-7:2015), which has been technically revised.

Methods of sampling and test for crude and refined glycerine

Part 7: Determination of sulphated ash — Gravimetric method

1 Scope

This Draft Tanzania Standard specifies a gravimetric method for determining sulphated ash deriving from glycerine for industrial use.

2 Normative references

The following referenced documents are indispensable for the application of this Tanzania Standard:

TZS 1199 – 1 *Crude and refined glycerine — Methods of sampling and test Part 1: Samples and test methods — General*

3 Principle

Combustion of a test portion and calcinations of the residue in the presence of sulphuric acid, between 800 °C and 850 °C. Weighing of the sulphated ash thus obtained.

4 Reagents

During the analysis, use only reagents of recognized analytical grade.

4.1 Sulphuric acid, density, approximately 1.84 g/mL, 98 % (m/m) solution, or approximately 18 M.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum or porcelain dish, diameter 70 mm to 90 mm and height 25 mm to 50 mm.

5.2 Electric furnace, capable of being controlled at 800 °C to 850 °C.

6 Procedure

6.1 Test portion

Heat the dish (see 5.1) for several minutes in the furnace (see 5.2) controlled between 800 °C and 850°C, allow it to cool to ambient temperature in a desiccator and weigh to the nearest 0.001 g. Weigh 50 g ± 0.1 g of the test sample to the nearest 0.01 g, in the previously weighed dish (see TZS 1199–1).

6.2 Determination

Gently heat the dish containing the test portion (see 6.1) over a small flame avoiding sputtering, and ignite the vapour. Stop the heating and allow to burn until a carbonaceous mass is obtained.

After cooling, moisten the residue with a few drops of the sulphuric acid solution (see 4.1) and remove the excess acid by heating until the white fumes disappear and combustible material is burned away.

Repeat this operation and then place the dish for 5 min in the furnace (see 5.2) controlled between 800 °C and 850 °C.

Allow to cool in a desiccator to ambient temperature and weigh to the nearest 0.001 g.

7 Expression of results

The sulphated ash is given, as a percentage by mass, by the formula:

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (see 6.1);

m_1 is the mass, in grams, of the empty dish (see 5.1);

m_2 is the mass, in grams, of the dish containing the sulphated ash.

8 Test report

The test report shall include the following particulars;

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination.
- d) all operations not included in this draft Tanzania Standard or regarded as optional.

Bibliography

TZS 1199-7:2015 *Methods of sampling and test for crude and refined glycerine Part 7: Determination of sulphated ash — Gravimetric method*

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