



DRAFT TANZANIA STANDARD

Determination of total and acid insoluble ash in cocoa products

TANZANIA BUREAU OF STANDARDS

0. Foreword

This draft Tanzania standard prescribes the method for determination of ash in cocoa products.

In the preparation of this draft Tanzania standard, assistance was derived from AOAC Official Method 920.72.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with TZS 4

1.0. Scope

This method specifies determination of ash in cocoa products.

2.0. Method of Application

It is applicable to all cocoa and cocoa based products.

3.0 Principle

3.1. Total Ash is obtained by complete ignition of sample and keeping in a muffle furnace at 600°C until ash.

3.2 Water insoluble Ash is obtained by extracting total ash with hot water and filtered. The residue is dried and ignited to give the water-insoluble ash. The water-soluble ash is obtained by difference from the total ash.

3.3. Acid insoluble ash is obtained by extracting either the total ash or the water-insoluble ash with diluted hydrochloric acid and filtered. The residue is dried and ignited

4.0 Reagents

4.1. Hydrochloric acid, 3 – 4 N.

4.2. Silver nitrate solution, 0.1N.

5.0 Apparatus

5.1. Crucible (porcelain, platinum or silica)

- 5.2. Desiccator Containing silica gel or anhydrous calcium chloride.
- 5.3. Muffle furnace 600 °C
- 5.4. Filter paper, ashless.
- 5.5. Analytical balance, capable of weighing to the nearest 0,0001 g.
- 5.6. Hot plate

6.0. Procedure

6.1. Total Ash

Weigh accurately 2 g of the dried material in a tared porcelain, silica or platinum dish. Ignite with a Meker burner/ Hot plate for about one hour. Complete the ignition by keeping in a muffle furnace at 600°C until grey ash and cool into a desiccator.

6.2 Water-insoluble ash

Ash sample as in Clause 6.1 Add 10 mL water to ash, heat nearly to boiling, filter into 300 mL Erlenmeyer, and wash with hot water to total volume of 60 mL. Cool and reverse for alkalinity of soluble ash. Return paper and content to ashing dish, burn off paper, and reignite 30 min at 600°C. Cover with watch glass, cool in desiccator, and weigh as soon as room temperature is attained. Reserve for acid-insoluble ash (6.3).

6.3 Acid-insoluble ash

Boil water-insoluble residue (6.2) or total ash (Clause 6.1) with 25 mL 3 - 4N hydrochloric acid 5 min (or heat on steam bath 15 min), covering dish with watch glass to prevent spattering; filter through paper or previously ignited and weighed crucible and wash with hot water until a negative chloride test is obtained with silver nitrate solution. Return paper and content to crucible, dry at 100 °C -102 °C, and ignite 30 min at 600 °C. Cover with watch glass, cool in desiccator, and weigh as soon as room temperature is attained.

7.0 Calculations and expression of results

7.1. Total Ash =
$$\frac{A \times 100}{W}$$

Where

A = is weight of ash in g (weight dish after ashing – weight empty dish).
W = is weight of sample in g.

7.2 Water insoluble ash

$$\% \text{water insoluble ash} = \frac{B \times 100}{W}$$

where

B = is weight of water-insoluble ash in g (weight dish after ashing – weight empty dish).

W = is weight of sample in g.

7.3 Water soluble ash

% water soluble ash = % total ash – % water insoluble ash.

7.4. Acid-insoluble ash

$$\% \text{ acid insoluble ash} = \frac{C \times 100}{W}$$

where

C = is Weight acid-insoluble ash in g (weight dish after ashing – weight empty dish).

W = is weight of sample in g.

8.0 Test report

Report results to nearest 0.01 %.