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Methods of analysis Part 1:
Determination of ash and alkilinity
EAST AFRICAN STANDARD

Milk powders — Methods for analysis — Part 1: Determination of ash and alkalinity

EAST AFRICAN COMMUNITY

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Second Edition 2006
EAS 81-1:2006

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in East Africa. It is envisaged that through harmonized standardization, trade barriers which are encountered when goods and services are exchanged within the Community will be removed.

In order to achieve this objective, the Partner States in the Community through their National Bureaux of Standards, have established an East African Standards Committee.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

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Milk powders — Methods for the analysis — Part 1: Determination of ash and alkalinity

1 Scope

This East African Standard gives methods for the determination of ash and alkalinity together with guidance for sample preparation.

2 Preparation of sample

Transfer the dried milk to clean, dry container provided with an airtight lid of a capacity about twice the volume of the powder. Close the container immediately and thoroughly mix the dried milk by repeatedly shaking and inverting the container. During the preparation of the sample, exposure of the dried milk to the atmosphere shall be avoided as far as possible to minimize absorption of moisture.

3 Determination of ash

3.1 Apparatus

The following apparatus is required.

3.1.1 Muffle furnace to operate at 525 °C to 550 °C.

3.1.2 Silica or platinum dishes 60 mm to 80 mm.

3.2 Procedure

Heat a silica or platinum dish for 60 min in the muffle furnace at 525 °C to 550 °C. Cool in an efficient desiccator for 60 min. Accurately weigh about 2g of dried milk into the dish spreading the powder uniformly in the dish before weighing. Heat either at the mouth of the muffle or over a low Bunsen flame until the powder is completely charred and smoke is no longer given off. Place the dish in the muffle cool in the desiccator and moisten the ash with water. Break up the ash with a flat-topped glass rod, wash any particles from the rod back into the dish and evaporate to dryness. The ash shall be further dried at 102 °C to 103 °C for a further hour. Cool in a desiccator for 60 minutes and weigh. Express the result as mass of the ash per 100g of dried milk as received.

4 Determination of alkalinity

4.1 Reagents

All reagents used shall be of a recognized reagent quality. The following reagents shall be used:

4.1.1 0.2 N hydrochloric acid solution.

4.1.2 Calcium chloride neutral 40 % w/v solution

4.1.3 0.2 sodium hydroxide solution.

4.1.4 5 % phenolphthalein in 50 % ethanol.

4.2 Procedure

Ash about 2g accurately weighed of dried milk as described in clause 6 and using a fine bore pipette or burette add 10 ml of 0.2 N hydrochloric acid drop wise to the dish containing the ash allowing the acid to run from the rim downwards. Grind the ash to a fine powder with a small flattened glass rod and wash the sides of the basin and the rod with a few drops of distilled water or water of at least equivalent purity. To obtain a reproducible end-point the volume not exceeding 15 ml. Warm the basin
carefully stirring until the ash is completely dissolved. Cool to air temperature and run in 2 ml of 40 percent neutral calcium chloride solution. Stir and add 0.5 of the phenolphthalein indicator solution.

Titrate with 0.2N sodium hydroxide solution until a faint pink colouration remains visible for 30 s on addition of one drop of titrant. Express the result as the volume of 0.2 N hydrochloric acid solution in millilitres required per 100 g of powder as received to the nearest whole number.

NOTE Slow efficient carbonisation at this stage facilities ashing and is essential if the ash is to be used subsequently for the determination of ash alkalinity.