DRAFT EAST AFRICAN STANDARD

Paper and board intended to come into contact with foodstuffs — Determination of formaldehyde in an aqueous extract

EAST AFRICAN COMMUNITY

Foreword

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

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS).The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. 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 Principles and procedures for development of East African Standards.

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.

The committee responsible for this document is Technical Committee EASC/TC 066, *Packaging*.

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

During the preparation of this Standard, reference was made to the following document:

EN 1541, *Paper and board intended to come into contact with foodstuffs — Determination of formaldehyde in an aqueous extract*

Acknowledgment is hereby made for the assistance derived from this source.

Paper and board intended to come into contact with foodstuffs — Determination of formaldehyde in an aqueous extract

1 Scope

This Draft East African Standard specifies the determination of formaldehyde in aqueous extracts prepared from paper and board intended to come in contact with foodstuffs. The limit of determination is 1 mg/kg.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 6588-1, *Paper, board and pulps — Determination of pH of aqueous extracts — Part 1: Cold extraction*

ISO 6588-2, *Paper, board and pulps — Determination of pH of aqueous extracts — Part 2: Hot extraction*

3 Terms and definitions

For the purposes of this standard, the following terms and definitions apply.

3.1
cold water extract
water solution obtained as a result of cold extraction ISO 6588-1

3.2
hot water extract
water solution obtained as a result of hot extraction ISO 6588-1

4 Principle

Formaldehyde reacts with pentane-2, 4-dione (acetyl acetone) in the presence of ammonium acetate to form 3, 5-diacetyl-1, 4-dihydrolutidine. The absorbance of the extract is measured at a wavelength of 410 nm.

NOTE   The extract should also be scanned by ultraviolet (UV) spectroscopy for confirmation where the level of formaldehyde exceeds specified limits (see clause 10).

5 Reagents

All reagents shall be of analytical grade and the water shall be distilled or of equivalent purity.

5.1   Anhydrous ammonium acetate

5.2   Acetic acid 99 % (d = 1.05)

5.3   Pentane-2.4-dione

5.4   Hydrochloric acid, 1 mol/l

5.5   Sodium hydroxide solution, 1 mol/l

5.6   Starch solution freshly prepared, 2 g/l

5.7   Formaldehyde solution, 370 g/l to 400 g/l

5.8   Standard iodine solution, 0.05 mol/l

5.9   Standard sodium thiosulphate solution, 0.1 mol/l

5.10   Pentane-2, 4-dione reagent

In a 100.0-ml volumetric flask dissolve:

    a)   15.0 g anhydrous ammonium acetate (see 5.1);

    b)   0.2 ml pentane-2, 4-dione (see 5.3);

    c)   0.3 ml acetic acid (see 5.2);

    d)   25 ml of water.

Make up to 100.0 ml with water. This reagent shall be freshly prepared.

5.11   Reagent (see 5.10) without pentane-2, 4-dione

5.12   Formaldehyde-standard: Stock solution

5.12.1   Measure 5.0 ml formaldehyde solution (see 5.7) into a 1000 ml volumetric flask and make up to 1000 ml with water.

5.12.2   Just before use determine the concentration of this solution as follows.

Transfer 10.0 ml of the stock solution into a conical flask, add 25.0 ml of a standard iodine solution (see 5.8) and 10.0 ml of sodium hydroxide solution (see 5.5). Allow to stand for 5 min. Acidify with 11.0 ml of hydrochloric acid (see 5.4) and determine the excess iodine by titration with a standard sodium thiosulphate solution (see 5.9), using 0.1 ml of the starch solution (see 5.6) as indicator.

NOTE   Add the starch solution when the solution being titrated has become a pale straw colour. Theoretically, 1.0 ml of 0.05 mol/l iodine consumed is equivalent to 1.5 mg formaldehyde.

5.13 Formaldehyde-standard: Dilute solution

Dilute an aliquot of the formaldehyde stock solution (see 5.12) to 20 times its volume with water, and then furtherdilute an aliquot of this second solution to 100 times its volume so that 1.0 ml of the final solution contains about 0.001 mg of formaldehyde. Use pipettes and volumetric flasks. Calculate the actual formaldehyde content. This solution shall be freshly prepared.

6   Apparatus

6.1   Ordinary laboratory apparatus

6.2   Spectrophotometer for use at the wavelength of 410 nm, with cells of an optical path length of 10 mm.

6.3   Scanning ultraviolet (UV) spectrophotometer in the range of 300 nm to 500 nm (Only for the confirmation steps).

6.4   Thermostatic water-bath capable of maintaining a temperature of (60 °C ± 2 °C).

7 Preparation of sample

Sampling, sample preparation and extraction shall be carried out according to the methods for the preparation of cold water (ISO 6588-1) or hot water extracts (ISO 6588-2).Two parallel extractions shall be carried out.

The test shall be performed not longer than 24 h after extraction.

8 Procedure

8.1 For each extract at least two parallel determinations shall be carried out

Att room temperature the cold water extract is applied while for paper and board materials intended for boiling and hot filtering purposes the hot water extract is applied

8.2 Sample solution

Into a 50-ml conical flask add:

- 25.0 ml extract (V1) (see 8.1);

- 5.0 ml pentane-2.4-dione reagent (see 5.10).

8.3 Reference solution

Possible interference due to coloured substances in the aqueous extract is eliminated by the use of this reference solution.

Into a 50-ml conical flask add

- 25.0 ml extract (see 8.1);

- 5.0 ml reagent without pentane - 2.4-dione (see 5.11).

8.4 Blank test

This is performed in order to construct the calibration curve.

Into a 50-ml conical flask add

- 25.0 ml water;

- 5.0 ml pentane - 2.4-dione reagent (see 5.10).

8.5 Determination

8.5.1 Shake the solutions 8.2, 8.3 and 8.4 for about 15 s. Immerse the conical flasks in a thermostatic waterbath (see 6.4) at (60 °C ± 2°C) for 10 min ± 10 s. Allow to cool for at least 2 min in a bath of iced water.

8.5.2 Bring the solutions into the measuring cells (see 6.2). Measure the absorbance at 410 nm of the sample solution (see 8.2) with the reference solution (see 8.3) in the reference cell (A1).

8.5.3 Measure the absorbance of the blank test (see 8.4) with water in the reference cell (A2).

8.5.4 The absorbance measurements shall be made between 35 min and 60 min from the time when the conical flasks were placed in the water bath at 60 °C.

8.5.5 If the value of formaldehyde obtained exceeds the range covered by the calibration solutions, the measurement shall be repeated with a more dilute sample solution and using an equally diluted reference solution.

8.6 Calibration curve

8.6.1 Into a 50-ml conical flask add:

- 1.0 ml of the formaldehyde diluted standard solution (see 5.13);

- 5.0 ml of the pentane – 2.4-dione reagent (see 5.10);

- make up with water to 30.0 ml.

8.6.2 Continue as described in 8.5 and measure the absorbance with water (see 8.5) in the reference cell.

8.6.3 Repeat the procedure with 5.0 ml; 10.0 ml; 15.0 ml; 20.0 ml and 25.0 ml of the formaldehyde diluted standard solution (see 5.13).Construct the calibration curve after subtraction of the blank test value (see 8.5) from each of the absorbance obtained.

9 Calculation and expression of results

9.1 Subtract A2 from A1 and read off from the calibration curve (see 8.6) the amount C in mg of formaldehyde in the sample solution (see 8.2).

9.2 Calculate the formaldehyde content of the sample (Cs) or (Cm) as follows:

$$C_2\text{-C x}\frac{V0}{V1} \text{ x } \frac{b}{100} \text{ x } \frac{1}{G} \hspace{6cm} 1$$

$$C_m\text{-C x}\frac{V0}{V1}\text{x } \frac{1}{G} \text{ x } \frac{100}{100-f} \text{ x } 100 \hspace{4.5cm} 2$$

where,

    C_s amount of formaldehyde soluble of the sample in mg/dm²;

    C_m amount of formaldehyde soluble of the sample in mg/kg;

    C   amount of formaldehyde read from the calibration graph, in mg;

    V_0 otal volume of extract (250 ml), in ml;

    V_1 volume taken for the test (25.0 ml), in ml;

    B   grammage, in g/m²;

    G   mass, in grams, of the sample taken under the same condition as grammage;

    F   moisture content of the sample, in %.

Report the result with two significant figures.

10 Confirmation

10.1 Requirement for confirmation

Where the level of formaldehyde in the water extract under test (see clause 7) exceeds any specified limit, the determination shall be confirmed by scanning ultraviolet (UV) spectroscopy (see 6.3).

10.2 Standard spectrum

Whilst preparing the formaldehyde derivative (see 8.5), scan the intermediate standard (10.0 ml standard from 8.6) from 300 nm to 500 nm. Record the position and absorbance value at the peak maximum and calculate the ratio of the measurements of the absorbance measured at 20 nm increments either side of the maximum.

The spectrum shall satisfy the following conditions:

a) the maximum shall be in the range from 408 nm to 411 nm;

b) the spectrum shall tend to zero absorbance, that is less than 0.02 absorbance units, below 320 nm.

Examples of the absorbance ratios to be expected are listed in Table 1.

Maximum absorbance: 410 nm

Table 1 — Examples of absorbance ratios at corresponding wavelengths

Wavelength pair in nm	Ratio
370 / 410	0.520 ± 0.02
390 / 410	0.843 ± 0.01
430 / 410	0.802 ± 0.01
450 / 410	0.386 ± 0.02

12 Test report

The test report shall refer to this East African Standard and state:

a) extraction method;

b) nature, origin and designation of the sample;

c) date of sampling;

d) date of test;

e) mean result;

f) whether confirmation test has been carried out and if so, its result;

g) any deviations from this East African Standard.

Bibliography

<mark>Kenyan std</mark>