DEAS 886: 2025

ICS 55.120

Second Edition

DRAFT EAST AFRICAN STANDARD

Packaging — Flexible packaging material — Determination of residual solvents by headspace gas chromatography — Test method

EAST AFRICAN COMMUNITY

Second Edition 2025

Copyright notice

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.

With regard to the first edition (EAS 886: 2018), the following changes have been made in this Second

edition:

- A list of solvents were added to clause 4.2
- The note on clause 4.3 was incorporated into the opening sentence of this clause.
- Deletion of an example under clause 5.7
- Clauses 15,16,17 were rearranged in the standard with the calculation clause positioned last.
- Update of the bibliography.

Packaging — Flexible packaging material — Determination of residual solvents by headspace gas chromatography — Test method

1 Scope

This Draft East African Standard prescribes a method for the quantitative determination of residual solvents in flexible packaging materials by headspace gas chromatography.

Residues from thermal decomposition products are not within the scope of this standard.

The method is applicable to flexible packaging materials that may consist of mono- or multilayer plastic films, paper or board, foil or combinations thereof.

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 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Principle

The sample is heated for a specific period, the volatiles are passed into the chromatograph for final quantitative determination of the residual solvents.

4 Reagents

4.1 General

All reagents shall be of recognized analytical reagent grade.

NOTE Grades stated as being suitable for chromatography are commercially available and are recommended for use as reference for standard calibration solutions. Appropriate safety precautions should be used when handling toxic and/or flammable solvents.

4.2 Reference solvents

A solution containing a mixture of the following solvents shall be used for the preparation of standard calibration solutions.

Methanol (MeOH), Acetonitrile (ACN), Acetone, Isopropanol/2-Propanol, Ethanol , Hexane, Toluene, Dichloromethane , Dimethyl Sulfoxide (DMSO) Dimethylformamide (DMF), Cyclohexane, Ethyl acetate, Methyl Ethyl Ketone (MEK), Propyl acetate, Methyl Isobutyl Ketone (MiBK), N-Propanol, Butyl acetate,

Isobutanol (i-Butanol), Ethyl benzene, p-Xylene, m-Xylene, o-Xylene, 2-Methoxy ethanol, Styrene, and Cyclohexanone

NOTE Commercially available standard mixture solutions may be used.

4.3 Dilution solvent

A solvent with a retention time different from those of residual solvents in the sample shall be used for dilution.Solvents like hexane, cyclohexanone, acid amides and glycerol triacetate (triacetin) are appropriate.

5 Apparatus

5.1 Suitable headspace accessory compatible with the gas chromatograph (5.6)

5.2 Analytical balance, capable of weighing to the nearest 0.1 mg

5.3 Template, for cutting samples

5.4 Scalpel or sharp knife

5.5 Syringes (for example 1-µl, 10-µl)

5.6 Gas chromatograph, having a flame ionization detector or a mass-spectrometer or an FTIR spectrometer or another detector suitable for the solvents to be determined

5.7 Gas chromatographic column, either packed or capillary, that will give good resolution of the solvents to be determined from any other components that might eventually be present in the headspace.

Examples for suitable columns and operation conditions are:

Packed column:

Length: 3 m;

Internal diameter: 3.2 mm;

Column filling: 80/120 mesh graphitized carbon, deactivated with polyethylene glycol;

Carrier gas: N_2, 20 ml/min;

Injector temperature: 220 °C;

Temperature programme 80 °C; raised to 160 °C at 6 °C/min; raised to 225 °C at 1.5 °C/min; held for 16 min.

6 Sampling

6.1 Samples of packaging materials that are to be analyzed shall be handled and stored so as to prevent either loss of volatile solvents or contamination by absorption of volatile solvents that may be present in the surrounding atmosphere.

6.2 Sampling and analysis shall be done in a place where the air is solvent-free in order to reduce the problem of contamination of the samples from their surroundings due to the low concentrations of residual solvents in the samples.

NOTE Samples should be in tightly packed roll form if possible. Sheet samples can be prepared from the roll by cutting out a square window (several layers of sheets) with a knife. At least the first five layers should be discarded. When in the form of sheets they should be stacked tightly together to form a compact "block" and wrapped tightly in a barrier

material, preferably aluminium foil with a thickness of 30 µm to 40 µm. For storage periods of more than 1 h, the wrapped samples should be stored at temperatures below 5 °C and in an atmosphere free of volatile contaminants.

7 Test specimens

7.1 Specimen area

The specimen area to be cut out shall depend on the equipment and the level of residual solvents to be determined in the material.

NOTE In order to optimize the solvents quantification and avoid pollution of the equipment, it is recommended to make preliminary trials with increasing area of the specimens.

7.2 Test specimens from sheets

From the top of the sample, take a block of about 15 sheets to 20 sheets out without separating them. Immediately put back the remaining part of the sample in its packaging under the conditions specified in Clause 6. After withdrawal of at least one layer (or more depending on the number of specimens to be prepared from the block) from the top of the block take the following sheet and rapidly cut the first specimen using a template (5.3). The specimen should be immediately put into the desorption device. Further specimens are prepared accordingly.

The following precautions shall be taken:

a) the different steps of the preparation shall be done very rapidly to avoid evaporation of the solvents.

b) the sample sheet from which the specimen is cut shall be taken out of the block immediately before preparation.

c) the specimens shall always be cut at a defined place for example on the same drawing printed on the same place in the width and the template for cutting is placed always in the same manner.

8 Analysis

8.1 Conditioning

As an initial guidance, the sample conditioning can be carried out at a temperature between 80 °C and 150 °C for a period of time necessary to guarantee that in a further analysis on the same specimen, the total solvent extract should be less than 5 % of the first one. If this percentage cannot be reached, revise the parameters of the method (temperature, conditioning time, stripping flow, amount of specimen etc.) and repeat the extractions.

NOTE In each laboratory the conditioning time can be ascertained by repeating the analysis several times with different samples (packaging materials with different structure) and choosing the longest.

8.2 Purging

The purging time and the stripping flow shall be chosen according to the different available head space equipment and the type of the sample in order to optimize the extraction. However the gas extracted shall be at least three times the volume of the desorption device. A periodic control of the stripping flow is necessary in order to maintain the analytical efficiency.

8.3 Concentration

The choice of the concentration method depends on the available system. The methods most widely in use are based on the use of cryogenic systems and entrapment adsorbent materials. The temperature of the adsorbent material during sampling shall be controlled.

Regardless of the trapping system, the amount of each solvent shall be in the calibration range.

9 Procedure

9.1 Incubate the sample at time and temperature defined in 8.1.

9.2 Carry the desorbed solvents in an inert gas stream and send them in a gas trap containing one or more solid absorbers or equipped with a cryogenic system.

9.3 Thermally desorb the trap to transfer the solvents into gas chromatographic column.

9.4 For the second extraction: incubate the sample at time and temperature defined in 8.1.

9.5 Repeat at least two more times the operations from 9.2 to 9.4 on the same vial, unless with the second injection the areas of all solvents are not increased by more than 5 % with respect to the areas obtained with the first injection. If so, it is not necessary to go any further and one can take the sum of the areas of the first two injections as a reference value for the calculations.

9.6 Third extraction in accordance with the procedure as in 9.1.

10 Preparation of standard solutions

A mix of standard solvents (4.2) of same quantities by weight is prepared. If necessary it can be diluted. Care should be taken when weighing to avoid loss by evaporation.

11 Chromatographic parameters

These depend on the type of column and the specific equipment used for the determination.

12 Number of test specimens

From each sample, at least three test specimens for analysis shall be prepared as in Clause 7.

13 Incubation of the test specimens

Incubation time and temperature have to be optimized accordingly for each individual system (see 8.1).

14 Determination with an external standard

Analysis of the standard solution shall be as follows:

a) incubate a vial containing 1.0 µl of standard solution at time and temperature defined in 8.1.

b) follow procedure as with the samples (9.2 to 9.6).

15 Precision data

Repeatability and reproducibility should be determined in accordance with ISO 5725-2 within each laboratory.

16 Detection limits

For each solvent, the detection limit has to be determined according to the equipment in use.

17 Calculation

Calculation of amount of one residual solvent in the sample

Q =

where

Q is the quantity in milligrams per square metre of packaging material of one residual solvent;

a is the area of peak on chromatogram for this solvent;

p is the mass, in milligrams, of the same solvent in standard solution;

e is the area of peak of the same solvent in standard solution; and

s is the area, in square metres, of the specimen.

Bibliography

EAS 886:2018 Packaging — Flexible packaging material — Determination of residual solvents by headspace gas chromatography — Test method.