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Foodstuffs — Determination of water activity

Produits agricoles et alimentaires — Détermination de l'activité de l'eau

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Foodstuffs — Determination of water activity

WARNING — The use of this document may involve the use of hazardous materials, operations and equipment. This document does not purport to address all the safety risks associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices.

1 Scope

This document establishes basic principles and specifies requirements for the methods of determining water activity (a_W) of food products for human consumption and animal feed within a measurement range of 0 to 1.

The measurement principles are based on the dew-point measurement or on the determination of the change in electrical conductivity of an electrolyte or in the permittivity of a polymer.

The method does not apply to products stored below their freezing point (equivalent to the temperature at which ice crystals appear in the product), neither to products corresponding to a water-in-fat emulsion, nor to crystal products such as sugars, salt or minerals.

For products containing volatile compounds, such as alcohols, specific equipment adaptations may be necessary to apply the method.

The results of the interlaboratory studies that were carried out are given in Annex B.

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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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
water activity
a_W
ratio of the partial water-vapour pressure in equilibrium with the product analysed to the water-vapour saturation pressure in equilibrium with pure water at the same temperature

$$a_W = \frac{pF(T)}{P_S(T)}$$

where

$pF(T)$ is the partial water-vapour pressure in equilibrium with the product analysed at the temperature T (kept constant during measurement);

$P_S(T)$ is the water-vapour saturation pressure in equilibrium with pure water at the same temperature T.

Note 1 to entry: Water activity is therefore a dimensionless quantity between 0, which corresponds to a completely anhydrous sample, and 1, which corresponds to pure salt-free water.

Note 2 to entry: The relative humidity at the equilibrium point is 100 times the water activity.

3.2

saturated solution

solution in which further amounts of the relevant solute added to the solution are no longer dissolved

4 Measurement principles

4.1 The two measurement principles that shall be used to determine the water activity are described in 4.2 and 4.3.

4.2 Dew-point measurement. The sample is placed in a hermetic measurement chamber that contains a mirror of which the temperature can be made to vary (using a Peltier thermo-electric module). The mirror is cooled until condensation appears on its surface. This water activity measurement technique is based on the fact that air can be cooled down to the saturation point without modifying the water content. At the equilibrium point, the relative humidity within the chamber is equal to the water activity of the sample. The exact temperature is determined (the dew-point temperature or dew-point) at which water-vapour condensation occurs. The surface temperature of the sample is also recorded. With these two temperatures the water activity can be determined.

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4.3 Determination of the change in electrical conductivity of an electrolyte (ion displacement) or in the permittivity of a polymer (charge displacement by the dipoles). These sensors measure the relative humidity of the air surrounding the sample. The relative humidity is equal to the water activity only if the sensor temperature is identical to the sample temperature. The electric resistance of a hygroscopic salt is measured (it depends on the water content).

NOTE Other principles for measuring water activity can be used, for example

- measurement of the change in length of a thread, or
- determination of the freezing point in an open system without establishing equilibrium.

These principles are considered alternative methods that still need to be validated.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Water activity measurement apparatus.

The instrument shall have the following characteristics:

- response linearity in the calibration range (Some instruments can present several ranges of linearity from 0 to 1. In this case, the laboratory shall take this information into account in order to choose standard solutions);
- a measurement cell suitable for the measurement principles described in Clause 4;

- a temperature regulation system for the measurement cell or that can be set up in a thermostatic enclosure so as to guarantee a temperature of 25 °C ± 1 °C;
- an internal resolution of at least 0,000 1 a_{w} units;
- a display of at least 0,001 a_{w} units;
- determination of the final measurement point by reaching a plateau defined with a maximum amplitude of 0,000 3, either by three consecutive measurements, or by stability over 1 min;
- where applicable, a system that makes it possible to eliminate interference due to the volatile compounds contained in the sample (e.g. specific filters).

The equipment shall operate under conditions according to the manufacturer’s instructions.

Refer to the manufacturer’s instructions on device maintenance.

5.2 One-mark volumetric flask, class A.

5.3 Balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.

5.4 Homogenization instruments, suitable for the sample, if necessary, e.g. plungers, knife mills, choppers.

5.5 Measurement cups, suitable for the apparatus and equipped with lids if necessary. Use a new cup preferably for each sample. If the cups are reused, care shall be taken with the cleaning (regarding fats especially), drying and integrity (free of warping, cracks, scratches, etc.).

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6 Reagents

All reagents shall be of recognized analytical grade. The water used shall comply with ISO 3696.

6.1 Salts.

More than 99 % pure, enabling production of the solutions in accordance with Annex A.

6.2 Standard solutions.

Saturated solutions (see Annex A) prepared by the laboratory using salts (6.1) or commercially-available solutions. The uncertainty of these solutions shall be known.

For shelf-life, refer to information provided by the producers for commercially-available solutions. Saturated solutions can be kept in fully tight jars at ambient temperature as long as the saturation and purity are kept.

6.3 Activated carbon (optional).

7 Sampling

Sampling of the various products shall be carried out in accordance with sector-specific standards, where available (see the Bibliography). In any event, it shall be suited to the measurement objective and representative of the product or product fraction to be measured for water activity.

8 Procedure

8.1 Sample preservation

The means of preservation selected (e.g. temperature, container, hygrometry, duration) shall not cause any chemical and physical modifications to the a_w value of the product. The laboratory may refer to the standards or sector-specific documents on the subject, where available, or as agreed with the customer.

Any form of chemical modification, such as moisture content change, and physical modification, such as freezing, forming salt or sugars crystals, can lead to a modification of the product a_w.

Some products can be preserved at negative temperatures and are not considered frozen as no ice crystals form.

Air-tight containers (e.g. sealed aluminium bags, cans, jars with appropriate caps) shall be used.

8.2 Sample preparation

If the sample's water activity is not uniform, the product to analyse shall undergo a suitable preparation procedure to make it uniform for a global measurement of water activity. The laboratory may make use of sector-specific standards where applicable. A preparation is not useful, if the sample's water activity is uniform. The test portion can be taken from the product as is.

If a preparation step is needed, the laboratory shall make sure that the preparation and ambient conditions do not cause an increase (e.g. in the case of products that have an ability to exchange moisture with the environment) or decrease in humidity (e.g. excessive milling, which could lead to heating of the sample).

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The preparation of the sample is under the responsibility of the laboratory.

Depending on its objectives, the laboratory may need to perform analyses:

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- in pre-defined areas of the product;

  EXAMPLE In the case of dried meat-based products (cured hams) characterized by a water-activity gradient between the inside and outside areas because of the drying process, the water-activity conditions can be determined in the inside and outside areas, or even at points distributed over the cross-section, so as to cover all the constituents by selecting the measurement points systematically.

- for compound products (e.g. sandwiches) on the individual components or contact surfaces;
- for dried products that need time to reach to a water activity equilibrium; this shall be considered before analysis.

In such cases, these provisions shall be indicated in the test report.

8.3 Test portion

The test portion is taken from the prepared or unprepared sample (8.2), according to the manufacturer's instructions (e.g. for filling the cups). The test portion shall be taken as quickly as possible to minimize exposure to the humidity in the laboratory, especially for the products that have an ability to exchange moisture with the environment.

8.4 Storage before measurement (optional)

After the preparation step, the measurement shall be taken within a maximum of 4 h.

The test portion shall be stored in air-tight packaging (air-tight lid or stretch-film over the seals) to avoid exposure to the ambient humidity.

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Storage shall be at ambient or measurement temperature so as to prevent any modification of the water activity in the sample. The samples should be stored directly in the measurement cup.

To lower the measurement time, it is advisable to ensure the test portion is at a temperature close to 25 °C.

8.5 Measurement

To take the measurement the manufacturer's recommendations shall be followed, taking into account that the measurement shall be taken at 25 °C using the water activity measurement apparatus (5.1).

NOTE In some cases, the measurement can be taken at a temperature other than 25 °C with a specific technological monitoring objective. The test assembly (cell and sensor, standard solution with the corresponding reference value, etc.) and the sample are then subjected to the selected temperature.

Before each analytical series, the instrument shall be checked with a standard that is preferably close to the values of the samples to be analysed. If there is a mean deviation from the target value that is greater than 0,005, carry out an adjustment (see 8.6).

During each analytical series, the measurement stability shall be verified using standard solutions (6.2).

Some types of instruments can present a significant carry-over effect when consecutively analysing samples with different water activity. In order to minimize this effect, operating conditions shall be adapted, including

- — waiting around 15 min between the two measurements after opening the lid of the instrument,
- — analysing granular activated carbon between the two samples (this solution is also useful in cases of potential interference due to volatile substances), and
- — analysing samples from the lowest to the highest water activity level.

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If the method using the principle of determination of the change in electrical connectivity of an electrolyte or permittivity of a polymer (4.3) is used, the water activity may be displaced by a hysteresis effect. For this purpose, the measurement cell shall be ventilated long enough for the next measurement to be started at as low a reading as possible (e.g. ambient humidity).

For the analysis of pulverulent products, refer to the manufacturer's instructions (use of a filter, ventilation speed). The cleanliness of the sensor (for instruments that use the dew-point measurement principle) shall be checked.

8.6 Calibration, adjustment and verification

Calibration shall be performed with at least two standards that frame the a_w values of the samples to be measured.

On the basis of the calibration results, a verification at one point is performed that takes into account a maximum tolerated error of ±0,005.

If this value is exceeded, an adjustment is performed in accordance with the manufacturer's instructions and a verification is performed after the adjustment at an intermediate point with a maximum tolerated error of ±0,005.

The laboratory shall ensure the linearity within the range of calibration.

Out of the range of calibration, the laboratory is allowed to make an external calibration of the device.

9 Calculation and expression of results

Results shall be expressed to two decimal places.

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