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Microbiology of food and animal feeding stuffs — Determination of water activity

Microbiologie des aliments — Détermination de l'activité de l'eau



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Foreword

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Introduction

Microorganisms require water for their metabolic activities, but only a certain fraction, the so-called “free water”, of the total water present in any foodstuff is available for this purpose. The amount of “free water”, termed the water activity, depends upon the nature and quantity of the components dissolved in the aqueous phase of the product (see reference [1]). Various species of microorganisms tolerate only water activities that are within certain levels. Water activity can therefore be used to predict microbial growth and determine the microbiological stability of a food product.

Microbiology of food and animal feeding stuffs — Determination of water activity

1 Scope

This International Standard gives basic principles and requirements for physical methods of determining the water activity of products intended for human consumption and the feeding of animals.

Water activity can be used to predict microbial growth and determine the microbiological stability of a food product, and it also provides an important, quantitatively determinable criterion for estimating the times for which a foodstuff can be kept.

2 Normative references

The following referenced documents are indispensable for the application 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 7218, *Microbiology of food and animal feeding stuffs — General rules for microbiological examinations*

3 Terms and definitions

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

3.1 water activity

a_w
ratio of the water-vapour pressure in the foodstuff to the vapour pressure of pure water at the same temperature

$$a_w = \frac{c_{EM}}{100} = \frac{p_F(T)}{p_S(T)}$$

where

- c_{EM} is the relative equilibrium moisture content of the atmosphere in contact with the foodstuff;
- $p_F(T)$ is the partial water-vapour pressure in equilibrium with the foodstuff at the temperature T (kept constant during measurement);
- $p_S(T)$ is the saturated partial pressure of pure water at the same temperature (T); this can be obtained from reference tables of water vapour pressure.

NOTE The water activity is therefore a dimensionless quantity, with a completely anhydrous test specimen having a water activity of 0,0 and pure salt-free water having one of 1,0. The water activities of most foodstuffs are at the upper end of this scale, and range from approximately 0,992 for untreated, raw meat down to about 0,800 for salted and dried products.

4 Measurement principles and apparatus

A wide variety of measurement principles can be used to determine the water activity of foodstuffs (for details see references [2] and [3]), including the direct or indirect determination of the equilibrium water-vapour pressure in closed systems. Examples of such methods are as follows:

- a) direct manometric pressure measurement;
- b) dew-point measurement
- c) determination of the change in the capacitance of a capacitor;
- d) determination of the change in the electrical conductivity of an electrolyte;
- e) measurement of the change in length of a thread;
- f) determination of the increase in mass of a sorbent;
- g) determination of changes in temperature (micropsychrometry) when equilibrium is established in closed systems;
- h) determination of the freezing point in an open system without establishing equilibrium.

5 Requirements for the measurement of water activity

The general rules for microbiological examinations given in ISO 7218 shall be followed.

Data on water activity reported in the literature are predominantly based on a measurement temperature of 25 °C and this often applies also to most tables containing calibration standards for testing the measuring instruments.

For methods a) to g) in Clause 4, it is therefore advisable to measure a_w at 25 °C. Variations of ± 1 °C in the actual measurement temperature will have no appreciable effect on the water activity.

Methods for determining the a_w value of foodstuffs shall comply with the following requirements.

- a) The method shall be accurate and reproducible and with a clear endpoint. The speed of measurement, ease of use and durability are also other important attributes relevant to the selection of a method.
- b) The method shall be capable of operating from the upper range of a_w from 0,999 to 0,600.
- c) Calibration of a method shall be carried out, and the accuracy measured, using reference standards of either saturated salt solutions (see Annex A) or solutions of sodium chloride (see Annex B).
- d) The repeatability limit shall correspond to a standard deviation of 0,002 s_{n-1} for the a_w range from 0,999 down to 0,600.
- e) The method shall be capable of measuring a sufficiently large and, consequently, representative sample.

6 Handling of instruments

6.1 The user shall always comply with the instructions given by the manufacturer of the measuring instrument concerned and shall check that the requirements specified in Clause 5 are fulfilled.

Points 6.2 to 6.8 apply to methods carried out in closed systems [i.e. methods a) to g) in Clause 4], while points 6.9 to 6.11 apply to method h).

6.2 Before carrying out a single or series of measurements, the equipment shall be recalibrated (at least daily) using the salt standards given in Annex A or B. If the device used cannot be calibrated internally, this may be done by plotting the experimental a_w value determined using a particular salt solution on the x -axis and the associated theoretical a_w value on the y -axis. (See Annex B for an example of such a curve.)

At least three measurement points shall be used for calibration, chosen in such a way that the measured sample value is within this range. Measured values for the salts/solutions may also be checked against existing calibrations.

6.3 Steps shall be taken to ensure that the temperature is constant while equilibrium is being established in the specimen chamber (measurement cell). The temperature shall not exceed 1 °C.

6.4 The test specimen shall be conditioned at the temperature of the specimen chamber (measurement cell) beforehand. During this period, the test specimen shall be kept in an hermetically sealed container to prevent water vapour movement. This container shall only be opened shortly before the specimen is placed in the specimen chamber, which shall immediately be closed again.

6.5 To prevent any soiling of the sensor by the test specimen, it shall be checked before every measurement and cleaned if necessary, following exactly the recommendations of manufacturer.

6.6 Gases emitted by the specimen material [e.g. ethanol, ammonia (in the case of fermented products)] shall be prevented from affecting the measurement by selecting a suitable measurement method or by protective devices (e.g. active carbon filters).

6.7 The measurement time will depend both on the test specimen and also on the measurement method used. For methods a) to g) in Clause 4, the average measurement time can range from several minutes to several hours because of the need to establish equilibrium.

6.8 In the case of methods in which the a_w value is determined by using sorption processes [methods c) to g) in Clause 4], the measurement shall always be carried out adsorptively because the measurement characteristic may be displaced by a hysteresis effect in the case of desorptive measurement. 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. room humidity).

6.9 In all cases, salt solutions of fairly high concentration are inherently unsuitable for calibrating systems employing method h) since the salt may be precipitated when the calibrating specimen is cooled as a result of the solution becoming more concentrated. Distilled water ($a_w = 1,000$) and NaCl solutions with a concentration of up to about 8 % may be used for calibration.

6.10 Depending on the test specimen, the measurement time of method h) is roughly 6 min to 20 min, but this can be cut down further by cooling the specimen beforehand in a refrigerator (set to 2 °C or above).

6.11 Method h) is not susceptible to interference effects due to the specimen and external factors.

7 Obtaining a representative specimen

The distribution of water activity may be presumed to be largely homogeneous in virtually all foodstuffs. Homogenization using a mincer is therefore unnecessary. Such treatment is also inadvisable since the sample material may become hot during mincing and give off water, with the result that it will no longer be representative of the foodstuff under examination.

Fermented meat products (e.g. sausage and ham) in which a water activity gradient is formed between the inside and outside regions because of drying are an exception. If necessary, the water activity conditions may be determined in the inside and outside regions, or even at points distributed over the cross-section, so as to cover all the constituents by systematically selecting the measurement points.

Another exception is water-in-oil emulsions (e.g. margarines) which have heterogeneous water activity even after homogenization.

Annex A (normative)

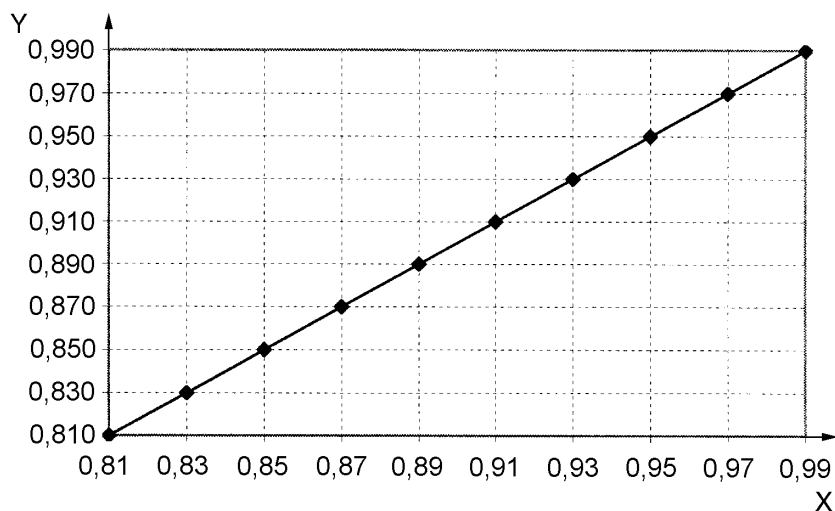
Water activity of saturated salt solutions at 25 °C

| Salt | a_w | Salt | a_w |
|-----------------------------------|-------|---|-------|
| MgCl ₂ | 0,328 | KBr | 0,809 |
| K ₂ CO ₃ | 0,432 | (NH ₄) ₂ SO ₄ | 0,810 |
| Mg(NO ₃) ₂ | 0,529 | KCl | 0,843 |
| NaBr | 0,576 | Sr(NO ₃) ₂ | 0,851 |
| CoCl ₂ | 0,649 | BaCl ₂ | 0,902 |
| SrCl ₂ | 0,709 | KNO ₃ | 0,936 |
| NaNO ₃ | 0,743 | K ₂ SO ₄ | 0,973 |
| NaCl | 0,753 | | |
| NOTE Taken from Reference [4]. | | | |

Annex B (normative)

Water activity of aqueous NaCl solutions of various concentrations at 25 °C

| Molarity | NaCl content (% mass fraction) | a_w |
|----------|-----------------------------------|---------|
| 0,1 | 0,58 | 0,996 6 |
| 0,2 | 1,15 | 0,993 4 |
| 0,3 | 1,72 | 0,990 0 |
| 0,4 | 2,28 | 0,986 8 |
| 0,5 | 2,84 | 0,983 5 |
| 0,6 | 3,39 | 0,980 2 |
| 0,7 | 3,93 | 0,976 9 |
| 0,8 | 4,47 | 0,973 6 |
| 0,9 | 5,00 | 0,970 2 |
| 1,0 | 5,52 | 0,966 9 |
| 1,2 | 6,55 | 0,960 1 |
| 1,4 | 7,56 | 0,953 2 |
| 1,6 | 8,55 | 0,946 1 |
| 1,8 | 9,52 | 0,938 9 |
| 2,0 | 10,46 | 0,931 6 |
| 2,2 | 11,39 | 0,924 2 |
| 2,4 | 12,30 | 0,916 6 |
| 2,6 | 13,19 | 0,908 9 |
| 2,8 | 14,20 | 0,901 1 |
| 3,0 | 14,92 | 0,893 2 |
| 3,2 | 15,75 | 0,885 1 |
| 3,4 | 16,58 | 0,876 9 |
| 3,6 | 17,38 | 0,868 6 |
| 3,8 | 18,17 | 0,860 0 |
| 4,0 | 18,95 | 0,851 5 |
| 5,0 | 22,65 | 0,806 8 |
| 6,0 | 25,97 | 0,759 8 |



Key

- X experimental value of a_w
- Y theoretical value of a_w

Figure B.1 — Example of a theoretical/experimental curve for calibrating a_w measuring instruments using unsaturated NaCl solutions

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