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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 4:

Test method for estimation of the moisture content as weight loss upon heating

Aides pour absorption d'urine — Méthodes d'essai pour caractériser les matériaux absorbants à base de polymères —

Partie 4: Détermination de la teneur en humidité au moyen de la perte de masse par chauffage

ICS: 11.180.20

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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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This document was prepared by Technical Committee [or Project Committee] ISO/TC [or ISO/PC] ###, [name of committee], Subcommittee SC ##, [name of subcommittee].

This second/third/... edition cancels and replaces the first/second/... edition (ISO #####:####), which has been technically revised.

The main changes compared to the previous edition are as follows:

— xxx xxxxxxxx xxx xxxxx

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Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 4:

Test method for estimation of the moisture content as weight loss upon heating

1 Scope

This test method covers the evaluation of mass loss upon heating for cross-linked polyacrylate superabsorbent powders.

In general, this method is expected to be applicable to powdered polymeric superabsorbent materials that are free-flowing under the specified test conditions. Substances other than water that are volatile in this temperature range will interfere.

NOTE - This standard does not claim to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. It is expected that the person performing this test has been fully trained in all aspects of this procedure.

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 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

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:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

sample

product or portion of a product taken from a production lot for testing purposes and identifiable and traceable back to its origin

3.2

specimen

specific portion of the identified sample upon which a test is performed

4 Principle

This procedure determines the mass loss upon dehydration of the test portion in an electrically heated drying oven at $105 \pm 2^\circ\text{C}$ at atmospheric pressure.

5 Reagents and Materials

None

6 Apparatus

6.1 Analytical balance capable of weighing a sample amount of 4 grams plus the weight of the drying container with an accuracy of $\pm 0.001\text{ g}$

6.2 Drying container (glass or aluminium dish), with corresponding removable lid, and with a bottom surface area of about 50 cm^2

6.3 Air-Circulating thermostated oven, capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$

6.4 Desiccator with indicating silica gel (or equivalent) as an active drying agent.

6.5 Spatula, capable of holding about 1 g of polyacrylate superabsorbent powder

7 Conditioning

Samples shall be delivered in a closed container, to prevent absorption of atmospheric moisture. Allow the closed container to equilibrate to the laboratory conditions. The preferred test conditions are $(23 \pm 2)^\circ\text{C}$ and $(45 \pm 15)\%$ relative humidity. If these conditions are not available, test at ambient conditions and report the temperature and relative humidity. Measure these laboratory conditions in accordance with ISO 187.

8 Sampling

SAFETY WARNING Power Handling – The German MAK has provided a guideline value for long-term exposure to the respirable portion of superabsorbent polyacrylate dust of $0.05\text{ mg}\cdot\text{m}^{-3}$. The respirable portion is defined as those particles of less than $10\text{-}\mu\text{m}$ diameter. Commercial superabsorbent polymers typically contain less than 0.1% of such particles. Precautions should be taken to avoid routine exposure to atmospheric respirable particles above this guideline MAK value.

8.1 Before taking a test portion out of the container to run the test, move the container five to ten times in a figure of eight motion, so as to obtain a homogeneous product. For that matter, sample bottles should not be filled more than 80% of their nominal capacity.



Figure 1 — Sense of motion of the container

8.2 Make sure the test portion is substantially free of lumps of size greater than 1 mm in diameter before proceeding with testing.

9 Procedure

9.1 Dry each dish and lid in the oven at 105 °C to constant weight to remove any moisture. During heating, the lid must be separated from the dish.

9.2 At the end of this heating period, transfer the dish and lid to a desiccator and allow to cool to room temperature.

9.3 Do not combine the dish and lid during cooling as a partial vacuum may form making it difficult to separate them.

9.4 Weigh to the nearest 0.001 g, the combined dish and lid and record this mass as m_1

9.5 Remove the cover and add approximately 4.0 grams of a well-mixed test portion of polyacrylate superabsorbent powder test sample that is free from lumps.

NOTE Transfer the sample portion from the sample bottle to the weighing vessel or laboratory paper in one spatula portion. Discard any excess material on the spatula. Do not return it to the sample bottle.. Keep the sample container closed as much as possible during this process.

9.6 Distribute the test portion in a uniform particulate layer over the bottom of the dish, e.g. by gentle tapping or shaking

9.7 Replace the lid and weigh the closed dish containing the sample immediately to the nearest 0.001 g. Record this mass as m_2

9.8 Place the open dish, containing the sample, and the corresponding lid, together in the oven at 105°C for 3 hours.

9.9 After drying, remove each sample dish with its corresponding lid and transfer to a desiccator, close the desiccator and allow to cool for 30 minutes.

9.10 Do not combine the dish and lid as a partial vacuum may form making it difficult to separate them.

9.11 When the dish containing the test portion has cooled to room temperature, combine the dish and lid and remove from the desiccator.

9.12 Weigh immediately to the nearest 0.001 g. Record this mass as m_3 .

9.13 Carry out at least two determinations on the same well-mixed laboratory sample, simultaneously or successively, by the same analyst.

10 Calculation

Calculate the moisture, w_m , expressed as a percentage of the weight of the original test sample, using equation (1):

$$w_m = \frac{\text{Mass of water lost during drying}}{\text{Mass of the polymer before drying}} = \frac{m_2 - m_3}{m_2 - m_1} 100$$

where

m_1 is the mass, expressed in grams, of the dried empty, complete (top and bottom) dish

m_2 is the mass, expressed in grams, of the complete dish with the test portion before drying

m_3 is the mass, expressed in grams, of the complete dish with test portion after drying

Calculate the average of the 2 replicate determinations

11 Report

In addition to the precise test results, the report shall include the following information:

- a) Reference the test method used
- b) Complete identification of all materials tested and method of sampling
- c) Name and address of testing institution
- d) The type of polymer-based absorbent materials, including all technical details and source information required for complete identification of the sample
- e) The results of moisture content by mass loss for each test portion expressed as mass fraction in percent to the nearest 0.1% and average for duplicate determinations
- f) Make and model of testing equipment
- g) Laboratory testing conditions
- h) Number of specimens tested
- i) For computer processed data, identify the software used and the version
- j) Deviation from the standard test procedure, if any
- k) Whether or not samples were conditioned prior to testing and, if so, for how long
- l) Anything unusual noted during the testing
- m) Any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met

SI values are regarded as the official standard system of measurement for this standard test method. If other systems of measurement are used in place of SI units (including inch-pound) their values must be reported independently. Systems of measurement must not be combined in any way, but shall be regarded and reported separately.

12 Precision

Laboratory data was returned to EDANA and compiled and anonymised before analysis. A statistical summary was prepared and presented to the (former) SPACE Analytical & Industrial Hygiene Committee. The general form of the data was checked by the members and its validity confirmed. At the same time, it was agreed that only one round of outliers would be removed from the analyses.

Data distributions were evaluated and extreme outliers were removed before analysis of variance was performed. The data from the analysis of variance was used to calculate repeatability and reproducibility statistics for each test and for each of the samples tested.

The method has been validated over the range of 0.4% to 1.82%. In the opinion of EDANA, the method can be used for values beyond this range, but such values should be validated by the interested parties.

Table 1 — Repeatability (r) and Reproducibility (R) of the method

Test	Sample	N	Min	Max	Mean	r	R
LOD	AJ224	127	0.70	1.82	1.20	0.47	0.54
	WR384	119	0.45	1.42	0.88	0.39	0.45
	XZ329	144	0.40	1.60	0.85	0.65	0.74

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