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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 7: Test method for gravimetric determination of absorption against pressure

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

Partie 7: Détermination gravimétrique du pouvoir d'absorption sous pression

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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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 xxxxxxx xxx xxxx

A list of all parts in the ISO ##### series can be found on the ISO website.

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 7:
Test method for gravimetric determination of absorption against pressure

1 Scope

This test method determines the capacity of polyacrylate superabsorbent powders to absorb saline solution under a specified enclosing pressure.

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*

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

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

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

The test portion is weighed and spread evenly on the bottom filter screen closing a specified cylinder. A uniform pressure is applied on the test portion. The cylinder is then placed on a filter plate, which is placed in a Petri dish filled with saline solution. After an absorption time of 1 hour, the cylinder is removed from the filter plate and weighed to determine the amount of fluid absorbed.

5 Reagents and Materials

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Water

Grade 1 water according to ISO 3696

5.2 Sodium chloride solution

5.2.1 0.9% by mass (m/m) Weigh 9.00 ± 0.01 g of sodium chloride into a 1 l beaker and add 991.0 ± 0.1 g of deionised water (grade 3). Stir until dissolved.

5.2.2 The conductivity of the solution should be checked prior to each use using properly calibrated measuring equipment. The expected conductivity of a 0,9% saline solution is of the order of 16mS/cm at 25 °C. Each testing lab must determine the correct conductivity for the conditions obtaining in the lab. It is also recommended that the temperature of the solution be maintained at (23 +/- 2) °C for the duration of the test.

6 Apparatus

Apparatus for measuring absorbency under pressure, shown in Figure 1 and consisting of the following elements

6.1 Petri dish or tray large enough to accommodate the apparatus and supply sufficient saline solution to meet the absorption capacity of the sample for the duration of the test.

— A circular Petri dish of 20-cm diameter gives an area of about 314-cm^2, whereas a square dish of 20 cm per side gives an area of about 400-cm^2.

— A balance must be struck between providing sufficient saline for absorption and evaporation of water leading to increasing salt concentration during the 60 minutes of the test.

6.2 Ceramic filter plate at least 80 mm in diameter and at least 5 mm in thickness/height, centred and bi-plane ground, with the outside edge not fused. The porosity should be 0 (nominal pore size 160-250 µm), ISO 4793. E.g. VitraPOR® filter discs (ROBU®).

NOTE A filter paper with a diameter of at least 70 mm, but not larger than the ceramic filter plate may be employed to reduce contamination of the filter plate by water soluble extracts from the polymer.

6.3 Polymethylmethacrylate (PMMA, or equivalent) cylinder with an internal diameter of d1 = (60.0 ± 0.2) mm, a height equal to (50.0 ± 0.5) mm with a nylon cloth filter screen or stainless steel screen in the bottom (400 mesh = 36 µm). For different diameters or materials see Annex A. It is highly recommended this cylinder is machined from a solid block rather than cut from a tube.

6.4 Polytetrafluoroethylene (PTFE, or equivalent) piston with a height greater than 60 mm and a diameter that is between 0.5 and 1.0 mm less than the internal diameter of the PMMA cylinder and designed to accommodate a cylindrical weight.

6.5 Piston weight designed to fit the PTFE piston, which in combination with the PTFE piston will provide a pressure of 49 g.cm-2 (0.7 psi) to a tolerance of 1 %.

The combined mass of the piston and the piston weight, M is calculated as follows:

$$M = P\pi r^2$$

Where P is the required pressure (e.g. 49 g.cm^{-2}) and r is half the diameter of the piston (6.5)

Table 1 — Example where the piston diameter is 59 mm, radius 29.5 mm or 2.95 cm

M	=	P	x	π	x	r^2
M	=	49 g.cm^{-2}	x	3.142	x	$(2.95)^2$ cm^2
M	=	49 g.cm^{-2}	x	3.142	x	8.7025 cm^2
M	=	1340 g				

Table 2 — Example for 21 g.cm-2: where the piston diameter is 59 mm, radius 29.5 mm or 2.95 cm

M	=	P	x	π	x	r^2
M	=	21 g.cm^{-2}	x	3.142	x	$(2.95)^2$ cm^2
M	=	21 g.cm^{-2}	x	3.142	x	8.7025 cm^2
M	=	574 g				

6.6 Analytical balance capable of weighing a mass of 0.9 ± 0.001 g of polymer powder in combination with the mass of the weighing vessel or laboratory paper employed

6.7 Analytical balance capable of weighing a mass of 30.000 ± 0.001 g of polymer gel in combination with the mass of the Plexiglas cylinder employed. E.g.: 500 g with a precision of 0.0001 g

6.8 Analytical balance, capable of weighing a mass of 9.00 ± 0.01 g of sodium chloride in combination with the mass of the weighing vessel or laboratory paper employed

6.9 Analytical balance, capable of weighing a mass of 1000.00 ±1.00 g of sodium chloride solution in combination with the mass of the vessel employed

6.10 Weighing vessel or laboratory paper

6.11 Metal spatula to accommodate 5 g of superabsorbent powder

6.12 Timer accurate to 1 second per 1 hour

6.13 Grade "A" 1 l volumetric flask

6.14 Filter paper with pore size < 25µ and diameter greater than that of the PMMA cylinder and less than that of the ceramic filter plate

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 ± 2) °C and (45 ± 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 mg.m-3. The respirable portion is defined as those particles of less than 10-µm 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.

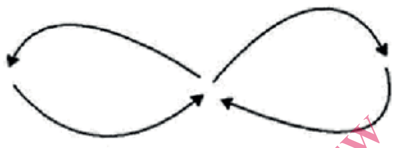

Figure 1 — Sense of motion of the container

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

NOTE 1 Ensure the integrity (holes or gel blocking) of the screen of each Plexiglas cylinder (see 6.4) prior to beginning the test.

9.1 Place a clean, dry weighing vessel onto a balance and tare the balance.

9.2 Add 0.89 to 0.91 g of a test portion of polyacrylate superabsorbent powder test sample to the weighing vessel and tare the balance once more.

NOTE 2 Transfer the sample portion from the sample bottle to the weighing vessel 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.

9.3 Carefully distribute the test portion onto the screen of the clean and dry Plexiglas cylinder to provide an even bed.

9.4 Place the weighing vessel back on the balance. The negative weight displayed is the mass of the sample transferred. Record this as ms.

9.5 Carefully holding the cylinder 1-2 centimetres above the bench, slowly insert the piston into the cylinder. This allows air to flow freely through the open mesh and avoids forcing the powder to the edges of the cylinder, creating an uneven bed.

9.6 Weigh the completed cylinder apparatus plus sample and record the mass as mA.

NOTE 3 In this form, without the weight, the apparatus can stand on the bench, e.g. on a clean dry paper towel whilst several samples are prepared.

9.7 When all the samples in a run have been prepared, the filter plates may be placed in their Petri dishes or trays.

9.8 Add the sodium chloride solution until the surface of the liquid reaches the same level as the surface of the filter plate. The saline must not overflow onto the filter plate.

9.9 At this stage a round filter paper may be placed on each filter plate allowing it to thoroughly wet with the sodium chloride solution. Avoid any surface liquid on the filter paper. Make sure the filter plate is fully saturated with saline.

9.10 Place the completed apparatus on the damp filter paper carefully and simultaneously adding the weight.

9.11 Allow the test portion to absorb the saline solution for a period of absorption of 60 minutes. Refill if necessary to keep sufficient saline solution in the Petri dish or tray.

9.12 Lift the complete apparatus and then remove the weight. This avoids sucking saline into the apparatus and giving false high results.

9.13 Reweigh the cylinder apparatus and record the mass as mB.

9.14 Clean the cylinder and piston thoroughly. Clean with deionised water then dry carefully. Do not use a drying temperature greater than 50° C, to prevent damage.

9.15 Repeat the above steps to obtain duplicate measurements.

9.16 Wash the filter plate with copious amounts of deionised water to remove any remaining saline.

9.17 Depending on required turnaround time the filter plates can be dried overnight at 40°C or more quickly (2-3 hours) at 105°C.

10 Calculation

For each test portion, calculate the absorption against pressure (AAP) expressed as a mass fraction in g/g:

$$AAP = \frac{(m_B - m_A)}{m_S}$$

where

m_S is the mass, expressed in grams, of dry test portion;

m_A is the mass, expressed in grams, of assembled dry cylinder group before absorption;

m_B is the mass, expressed in grams, of the assembled wet cylinder group after absorption.

Report the average for each of the 2 calculated values.

Take the average of the 2 calculated values and round it to the nearest 0.1 units.

11 Report

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

a) Reference the test method used