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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 8: Test method for determination of the permeability dependent absorption under pressure of saline solution by gravimetric measurement

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

Partie 8: Détermination gravimétrique du débit

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 ISO/TC 173, *Assistive products*, Subcommittee SC 3, *Aids for ostomy and incontinence*.

This second edition cancels and replaces the first edition (ISO 17190-8:2001), which has been technically revised. The main changes compared to the previous edition are as follows:

— full text review and new laboratory analysis with statistical evaluation;

— descriptions of the equipment required and the handling procedure improved;

— request for duplication removed.

A list of all parts in the ISO 17190 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 8:
Test method for determination of the permeability dependent absorption under pressure of saline solution by gravimetric measurement

WARNING — This document 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 document 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.

1 Scope

This document provides a test method for measuring the permeability-dependent absorption under pressure (PDAUP) of polyacrylate superabsorbent powder, where permeability is a significant controlling factor under the conditions of the test.

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*

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* (3.1) upon which a test is performed

4 Principle

This method is a modified version of NWSP 242.0[5] and ISO 17190-7.

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.

During the test, a swollen gel layer is formed at the bottom of the cell through which liquid shall be actively drawn in order for further absorption to occur. Under the conditions of the test, the thickness of the swollen gel layer, its permeability and the absorption capacity of the polymer are significant factors. The results thus provide information that may be used to interpret the polymers absorption capacity under conditions where the permeability of the swollen gel is a controlling factor.

5 Reagents and materials

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

5.1 Water.

Grade 3 water in accordance with ISO 3696, with the exception that the conductivity can be as high as 30 µS/cm.

5.2 Sodium chloride solution.

5.2.1 0,9 % mass fraction of sodium chloride solution in water. 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 1 600 S/m at 25 °C. Each testing lab shall 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. As this matches the required laboratory temperature it is not necessary to record the solution temperature.

6 Apparatus

The apparatus for measuring absorbency under pressure is illustrated on Figures A.1 and A.2. It comprises 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.

It is necessary to minimise evaporation of water, as this leads to increasing saline concentration during the test, without compromising the availability of sufficient saline to be absorbed by the polymer.

A practical solution is to use circular Petri dish of 20 cm diameter, which gives an area of about 314 cm^2, or a square dish of 20 cm per side, which gives an area of about 400 cm^2.

— The PDAUP requires much more saline than the equivalent superabsorbent polymer (AAP) and the dish or tray shall be refilled to the level of the ceramic filter plate on a regular basis.

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), in accordance with ISO 4793.

EXAMPLE VitraPOR® filter discs (ROBU®)[1)].

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 d_1 = (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 other diameters and 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 4 805 pascals, which for practical convenience can be converted to 49 g/cm^2 or 0,7 psi, to a tolerance of 1 %. The SI units are unwieldy, and the calculations exemplified in this standard use the cgi unit g/cm^2.

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. Table 1 provides and example of the calculation.

Table 1 — Example: where the piston diameter is 59 mm, radius 29,5 mm

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	8,702 5 cm^2
M	=	1 340 g				

6.6 Analytical balance, capable of weighing a mass of (5,00 ± 0,01) 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 (9,00 ± 0,01) g of sodium chloride in combination with the mass of the weighing vessel or laboratory paper employed.

6.8 Analytical balance, capable of weighing a mass of (30,00 ± 0,01) g of polymer gel in combination with the mass of the PMMA cylinder employed. E.g.: 500 g with a precision of 0,001 g.

6.9 Analytical balance, capable of weighing a mass of (1 000,00 ± 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.

1) VitraPOR® and ROBU are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

6.14 Filter paper, with pore size <25 µ and diameter greater than that of the plastic 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

WARNING — Powder Handling – The German Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area (MAK Commission) 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 value.

8.1 Before taking a test portion out of the container to run the test, rotate the container five to ten times in a three-dimensional figure of eight motion (see Figure 1), so as to obtain a homogeneous product. For that matter, sample bottles should not be filled more than 80 % of their nominal capacity.

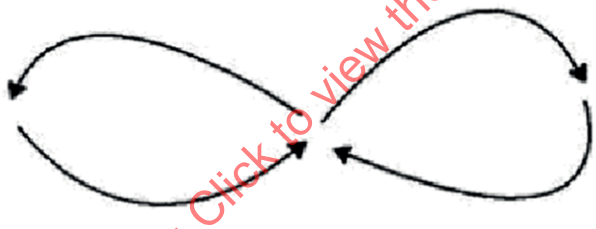


Figure 1 — Sense of motion of the container

8.2 Make sure the test portion is substantially free of lumps of size greater than 1 mm in diameter before proceeding with testing. Lumps can pierce the screen and disqualify the equipment and the test.

9 Procedure

Ensure the integrity (check for holes or gel blocking) of the screen of each PMMA cylinder (see 6.3) prior to beginning the test.

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

9.2 Add 4,9 g to 5,1 g of a test portion of polyacrylate superabsorbent powder test sample to the weighing vessel or laboratory paper and tare the balance once more.

Transfer the sample portion from the sample bottle to the weighing vessel or 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.

9.3 Distribute the test portion onto the screen of the clean and dry the PMMA cylinder to provide an even bed.

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

9.5 Hold the cylinder a few 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 (record the mass as m_A)

NOTE 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 shall 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.

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

9.11 Allow the test sample to absorb the saline solution for a period of equilibrium, which should be at least 60 minutes. Refill, if necessary, to keep sufficient saline solution in the Petri dish or tray.

9.12 Lift the complete apparatus before immediately removing 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 m_B.

9.14 Clean the cylinder and piston thoroughly. Clean with deionized water then dry carefully. Do not use a drying temperature greater than 40 °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 b, the PDAUP expressed as a mass fraction in g/g.

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

where

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

m_A is the mass, expressed in grams, of dry cylinder apparatus;

m_B is the mass, expressed in grams, of the cylinder group after suction.

An alternative calculation is the ratio of the absorption of a high weight of superabsorbent polymer (PDAUP, 5 g sample) against the absorption of a low weight of superabsorbent polymer (abbreviated to AAP, 0,9 g sample), formerly termed the permeability potential under pressure (abbreviated to PPUP). For this a separate the AAP test, (ISO 17190-7) shall be performed.

Calculate *p,* the PPUP, as the ratio of the PDAUP to the AAP as follows:

$$p(\%)=\frac{b}{a}100$$

where

b is the permeability-dependent absorption under pressure;

a is the absorption against pressure.

This allows easy interpretation as a higher percentage indicates higher permeability of the gel formed during testing.

11 Report

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

a) Reference to this document, i.e. ISO 17190-8:2020;

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 permeability-dependent absorption under pressure, expressed as a mass fraction in grams per gram (g/g) to the nearest 0,1 g/g and the average for duplicate determinations;

f) Any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met;

g) Make and model of testing equipment;

h) Laboratory testing conditions;

i) Number of specimens tested;

j) For computer processed data, identify the software used and the version;

k) Deviation from the standard test procedure, if any;

l) When calculated, the standard deviation or the coefficient of variation;

m) Whether or not samples were conditioned prior to testing and, if so, for how long.;

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 shall be reported independently. Systems of measurement shall not be combined in any way, but shall be regarded and reported separately.

12 Precision

Laboratory data was returned to EDANA and compiled and anonymized 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. Table 2 provides the results of that evaluation.

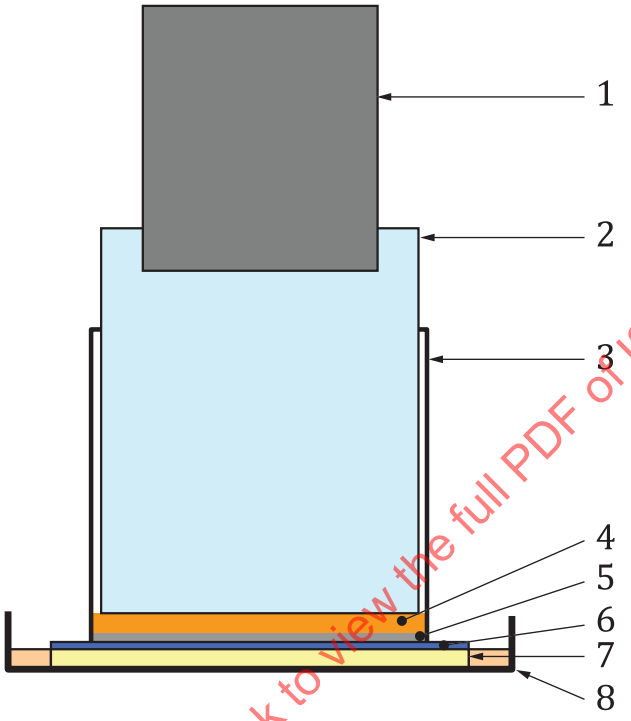
The method has been validated over the range of 8,80 to 13,23 g/g. In the opinion of EDANA, the method can be used for values beyond this range, but such values should be validated by the interested parties.

Table 2 — Repeatability (*r*) and reproducibility (*R*) of the method

Test	Sample	*N*	Min	Max	Mean	*r*	*R*
PDAUP	AJ224	143	8,80	11,28	10,00	0,92	1,08
	WR384	144	9,05	11,09	9,96	1,16	1,24
	XZ329	140	10,17	13,23	11,60	2,06	2,11

Annex A
(informative)

Apparatus for measuring absorbency under pressure



Key

1 metal weight
2 polytetrafluoroethylene (PTFE) piston
3 PMMA cylinder
4 sample
5 metal mesh
6 filter paper
7 ceramic filter plate
8 petri dish or tray

Figure A.1 — Schematic of the apparatus for measuring dependent absorption under pressure