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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 1: Test method for determination of pH

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

Partie 1: Détermination du pH

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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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 xxxxxxxx xxx xxxxx

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 1: Test method for determination of pH

1 Scope

This test method is for the determination of the pH of granular superabsorbent polyacrylates.

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*

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 pH of the saline solution to which polyacrylate superabsorbent powders are added is determined using a pH meter and a standardized glass pH-responsive electrode.

5 Reagents and Materials

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

5.1 Water

Grade 3 water according to ISO 3696, with the exception that the conductivity can be as high as 30 $\mu\text{S}/\text{cm}$

5.2 Sodium hydroxide solution

$c(\text{NaOH}) = 0.1 \text{ mol/l}$. Obtained as ready-made analytical grade solution.

5.3 Hydrochloric acid solution

$c(\text{HCl}) = 0.1 \text{ mol/l}$. Obtained as ready-made analytical grade solution.

5.4 Sodium chloride solution

5.4.1 0.9% by mass (m/m) Weigh $9.00 \pm 0.01 \text{ g}$ of sodium chloride into a 1 l beaker and add $991.0 \pm 0.1 \text{ g}$ of deionised water (grade 3). Stir until dissolved.

5.4.2 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 16 mS/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 $(23 \pm 2) ^\circ\text{C}$ for the duration of the test.

5.4.3 Adjust the pH of the saline solution to 6.0 ± 0.05 using 0.1 mol/l sodium hydroxide or hydrochloric acid solution.

5.5 Standard buffer solutions

Prepare buffer solutions according to ISO 10523 (or equivalent national standards) with for example the following pH values:

- a) 4.0 ± 0.02
- b) 7.0 ± 0.02

6 Apparatus

6.1 Analytical balance, capable of weighing a mass of $0.500 \pm 0.001 \text{ g}$ of polymer powder in combination with the mass of the weighing vessel or laboratory paper employed

6.2 Analytical balance, capable of weighing a mass of $9.00 \pm 0.01 \text{ g}$ of sodium chloride in combination with the mass of the weighing vessel or laboratory paper employed

6.3 Analytical balance, capable of weighing a mass of $1000.00 \pm 1.00 \text{ g}$ of sodium chloride solution in combination with the mass of the vessel employed.

6.4 pH meter

Glass pH-responsive electrode (suitable for polymer solutions)

6.5 Magnetic stirrer and stirring bar

Cylindrical stirring bars can be unstable when used on a multipoint magnetic stirring block. The mixture of magnetic fluxes can cause the bar to freeze periodically. Both cylindrical and star-shaped stirrers can tear the gel. It is recommended to use a cross-centered circular bar, which provides more stable stirring and minimum tearing of the gel. It is also important to make sure that the cross is properly centred in the circle. Cheaply made versions can be off-centre and this increases variability in the test.



Figure 1 — Cowie double cross-head stir bar (typical dimensions: $20 < d < 25$ mm, $12 < h < 18$ mm)

6.6 Graduated 250 ml beaker (a tall form will give better separation of gel and saline)

6.7 Grade A 100 ml graduated measuring cylinder

6.8 Grade A 1 l volumetric flask

6.9 Weighing vessel or laboratory weighing paper

6.10 Metal spatula to accommodate 0.5 g of 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

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 $10\text{-}\mu\text{m}$ 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.

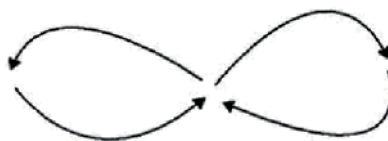


Figure 2 — Sense of motion of the container

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 Electrode calibration: Calibrate the pH electrode using at least two standard buffer solutions (for example, pH 4.00 and pH 7.00) in accordance with the operating instructions provided by the manufacturer.

9.2 Add 100 ml of 0.9% saline solution to the 250 ml beaker and place the beaker on the magnetic stirrer. Stir at a speed that avoids air from being drawn into the solution.

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

9.4 Add 0.49 to 0.51 g of a test portion of polyacrylate superabsorbent powder test sample to the weighing vessel or laboratory paper and tare the balance once more.

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.

9.5 Disperse the test portion of PA superabsorbent powder into the saline solution and slowly mix the suspension for 10 minutes. There should be no visible vortex on the surface during stirring, so a rotation speed of between 50 and 150 r.min⁻¹ is recommended.

9.6 Return the weighing vessel or laboratory paper to the balance. The negative weight displayed is the mass of the sample transferred.

9.7 Switch off the stirring and allow the gel to completely settle to the bottom of the beaker; 10 minutes is recommended.

9.8 Rinse the pH electrode with deionised water gently blot and immerse in the supernatant layer of the test solution avoiding any direct contact between the electrode and the polymer gel.

9.9 At 30 second intervals observe the pH. When two successive readings are identical (+/- 10%) record the value.

9.10 Between successive measurements rinse the electrode with deionised water and gently blot before immersing in the next sample solution.

9.11 Once all measurements are completed, rinse the electrode with deionised water and store according to the manufacturer's instructions.

10 Calculation

Not applicable

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) Laboratory testing conditions
- f) The presence or absence of lumps in the sample
- g) The results of pH for each test, expressed in pH units to the nearest 0.1 unit, and average value in case of duplicate determinations

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 5.84 to 6.22. 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
pH	AJ224	175	5.90	6.20	6.06	0.18	0.19
	WR384	170	5.84	6.15	6.02	0.19	0.20
	XZ329	176	5.95	6.22	6.07	0.19	0.19