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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 10: Test method for determination of extractable polymer content by potentiometric titration

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

Partie 10: Détermination de la teneur en polymère extractible par titrage potentiométrique

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Contents

Page

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 17190-10:2001), 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.

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 10: Test method for determination of extractable polymer content by potentiometric titration

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 to determine the mass fraction of soluble polymers present in crosslinked polyacrylate superabsorbent powders that can be extracted into saline solution.

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
extractables
sum of the soluble acid and salt groups of monomeric, oligomeric and polymeric carboxylates extracted from the superabsorbent polymer

3.2
sample
product or portion of a product taken from a production lot for testing purposes and identifiable and traceable back to its origin

4 Principle

The amount of extractable polymer in polyacrylate superabsorbent powders is determined by mixing the polymer in saline solution for 1 hour. The resulting saline suspension is filtered in its entirety through a paper filter of <12 µ pore size. An aliquot of the filtrate is titrated against a standard base solution (NaOH) to pH 10,0 to determine the concentration of free carboxylic acid groups. The resulting solution is then back-titrated against a standard acid solution (HCl) to pH 2,7 to determine the concentration of neutralized carboxylate groups. The titration data are used to calculate the total amount of extractables present in the superabsorbent powder.

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 hydroxide solution.

c(NaOH) = 0,1 mol/l. Obtained as ready-made analytical grade solution.

5.3 Hydrochloric acid solution.

c(HCl) = 0,1 mol/l. Obtained as ready-made analytical grade solution.

5.4 Sodium chloride solution.

5.4.1 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 deionized 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 16mS/cm 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.

5.5 Standard buffer solutions.

Prepare 3 buffer solutions in accordance with ISO 10523 with pH values suitable to work in a range from pH 2,7 till pH 10. For instance

— 3,0 ± 0,02

— 7,0 ± 0,02

— 10,0 ± 0,02

6 Apparatus

6.1 Analytical balance, capable of weighing a mass of (1,000 ± 0,001) 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 ± 0,01) 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 (1 000,00 ± 1,00) g of sodium chloride solution in combination with the mass of the vessel employed

6.4 pH meter with a combined glass pH-responsive electrode, referred to in the text as the pH electrode, which is suitable for titrating polymer solutions and capable of accurately measuring pH in the range 2,7 to 10,0. For example, a large membrane surface increases the electrode sensitivity for high pH measurement and a leaky sleeve electrode is less likely to suffer contamination from the polymer in the titration solution.

6.5 Volumetric flask, grade "A" of 1 l capacity.

6.6 Weighing vessel or laboratory paper.

6.7 Titration vessels - glass beakers or conical flasks.

6.8 Conical flask, of 250 ml capacity.

6.9 Beaker, of 250 ml capacity. A tall form is recommended to allow better gel separation.

6.10 Metal spatula, to accommodate 1,0 g of superabsorbent powder.

6.11 Paraffin film or suitable cover for glass beakers.

6.12 Filter papers, to accommodate 100 ml supernatant, with a pore size <12 µm.

6.13 Measuring cylinder, of 200 ml capacity and accurate to ±0,5 %.

6.14 Magnetic stirrer, having the capability of stirring at a rate of 250 ± 50 $r.min^{-1}$.

6.15 Magnetic stirrer and stirring bar.

6.16 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-centred circular bar (see Figure 1), 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)

6.17 Analytical burette, of 10 ml to 20 ml volume, with an accuracy to ±0,01 ml or automated titration equipment of similar or better accuracy.

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 2), so as to obtain a homogeneous product. For that matter, sample bottles should not be filled more than 80 % of their nominal capacity.

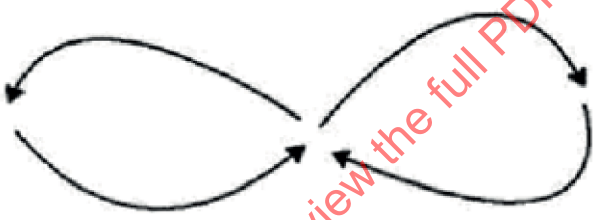

Figure 2 — Sense of motion of the container

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

9 Procedure

9.1 Electrode Calibration

Calibrate the pH electrode using three buffer solutions with pH values between pH 2,00 and pH 10,00. Monitor the accuracy and precision of the electrode according to the manufacturers' instructions.

9.2 Accurately add (200,0 ± 0,1) ml of saline solution to a 250 ml beaker or conical flask, using the measuring cylinder.

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

9.4 Add (0,95 to 1,05) g of a test portion of superabsorbent powder test sample to the weighing vessel or laboratory paper and tare the balance again.

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 Carefully distribute the test portion into the flask containing the (200,0 ± 0,1) ml of saline.

9.6 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_{sam}.

9.7 Stopper/Cover/Seal the beaker or conical flask, and stir the solution at a rate of (250 ± 50) $r.min^{-1}$ for 1 hour.

9.8 Prepare a titration blank by treating (200,0 ± 0,1) ml of the same batch of saline solution as used for the sample preparation in the same way.

9.9 Stop stirring the solutions, and allow the gel to completely settle to the bottom of the beaker; 10 minutes is recommended.

9.10 Filter 100 ml of the solution supernatant through a filter paper.

Blank Filtration**:** It is possible that filter papers change the pH of the blank saline solution. If filtration is used it shall be shown that the pH of the blank is not changed by filtration. Or simply do not filter the blank saline solution.

9.11 Take a volume of this filtrate appropriate for the titration system used. In this method, 50-ml is used as an example in the procedure and the calculations.

9.12 Using a burette or automated titration system, titrate 50 ml of the blank saline to pH 10,0 against the standard sodium hydroxide solution, and then to pH 2,7 against the standard hydrochloric acid solution. Record each titrant volume used to reach the end point.

9.13 Using a burette or automated titration system, titrate 50 ml of the sample filtrate to pH 10,0 against the standard sodium hydroxide solution, and then to pH 2,7 against the standard hydrochloric acid solution. Record each titrant volume used to reach the end point.

10 Calculation

The amount of carboxylic acid (e.g. polycarboxylic acid), expressed in moles, in the supernatant aliquot, n_{COOH}, is given by Formula (1):

$$n_{COOH} = \left(V_{NaOH,s} - V_{NaOH,b}\right) C_{NaOH} \quad (1)$$

where

$V_{NaOH,s}$ is the volume, expressed in millilitres, of the NaOH titrant necessary to titrate the sample filtrate to pH 10,0

$V_{NaOH,b}$ is the volume, expressed in millilitres, of the NaOH titrant necessary to titrate the blank to pH 10,0

c_{NaOH} is the concentration, expressed in moles per litre, of the NaOH titrant used for the titration to pH 10,0

The total amount of carboxylate, n_{tot}, expressed in moles, in the supernatant aliquot is given by Formula (2):

$$n_{tot} = \left(V_{HCl,s} - V_{HCl,b}\right) C_{HCl} \quad (2)$$

where

$V_{HCl,s}$ is the volume, expressed in millilitres, of the HCl titrant necessary to titrate the sample filtrate from pH 10,0 to pH 2,7

$V_{HCl,b}$ is the volume, expressed in millilitres, of the HCl titrant necessary to titrate the blank from 10,0 to pH 2,7

C_{HCl} is the concentration, expressed in moles per litre, of the HCl titrant used for the titration from 10,0 to pH 2,7

The amount of carboxylate, n_{COONa}, expressed in moles, in the supernatant aliquot is given by Formula (3):

$$n_{COONa} = n_{tot} - n_{COOH} \quad (3)$$

The relative masses, expressed in grams, of carboxylic acid groups, m_{COOH}, and sodium carboxylate groups, m_{COONa}, are given by Formulae (4) and (5) respectively:

$$m_{COOH} = n_{COOH} M_{COOH} F_{dil} \quad (4)$$

$$m_{COONa} = n_{COONa} M_{COONa} F_{dil} \quad (5)$$

where

M_{COOH} is the molar mass of acrylic acid, equal to 72 g.mol^{-1}

M_{COONa} is the molar mass of sodium acrylate, equal to 94 g.mol^{-1}

F_{dil} is the dilution factor, equal to 200/50 = 4

The extractable content, w, expressed as a mass fraction in percent, of the superabsorbent polymer is given by Formula (6)

$$w = \frac{\left(m_{COOH} + m_{COONa}\right)}{m_s} 100 \quad (6)$$

Where m_s is the mass, in grams, of the test portion

Calculate the average extractable content for the sample from the results obtained for the two test portions.

11 Report

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

a) Reference to this document, i.e. ISO 17190-10:2020;

b) Complete identification of all materials tested and method of sampling;

c) Name and address of testing institution;

d) Make and model of testing equipment;

e) The type of polymer-based absorbent materials, including all technical details and source information required for the complete identification of the sample;

f) Whether or not lumps were present in the sample;

g) The results of the extractables content for each test, expressed as a mass fraction in percent to the nearest 0,1 %, and the average for duplicate determinations;

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

i) Laboratory testing conditions;

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

k) Any deviations from this procedure or any operations regarded as optional (e.g. different stirring time);

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;

n) Anything unusual noted during the testing.

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 1 provides the results of that evaluation.

The method has been validated over the range of 2,29 to 8,9 % (as a percentage of the superabsorbent polymer). 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*
Ext	AJ224	169	5,87	8,90	7,35	1,64	1,73
	WR384	176	2,29	7,90	5,54	3,49	3,88
	XZ329	157	4,30	6,18	5,24	1,32	1,34