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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 10: 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

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 17190 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 17190-10 was prepared by Technical Committee ISO/TC 173, *Technical systems and aids for disabled or handicapped persons*, Subcommittee SC 3, *Aids for ostomy and incontinence*.

ISO 17190 consists of the following parts, under the general title *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials*:

— *Part 1: Determination of pH*

— *Part 2: Determination of amount of residual monomers*

— *Part 3: Determination of particle size distribution by sieve fractionation*

— *Part 4: Determination of moisture content by mass loss upon heating*

— *Part 5: Gravimetric determination of free swell capacity in saline solution*

— *Part 6: Gravimetric determination of fluid retention capacity in saline solution after centrifugation*

— *Part 7: Gravimetric determination of absorption under pressure*

— *Part 8: Gravimetric determination of flowrate*

— *Part 9: Gravimetric determination of density*

— *Part 10: Determination of extractable polymer content by potentiometric titration*

— *Part 11: Determination of content of respirable particles*

ISO 17190 is intended to be used in conjunction with ISO 17191, *Urine-absorbing aids for incontinence — Airborne polyacrylate superabsorbent material in the workplace — Determination of the content in respirable dust by sodium atomic absorption spectrometry*.

Annex A of this part of ISO 17190 is given for information only.

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Introduction

ISO 17190 consists of a series of test methods originally developed by *European Disposables and Nonwovens Association (EDANA)*. These test methods have been incorporated without technical changes into one International Standard consisting of eleven parts.

These test methods have been in practical use for several years, and have proven to be reliable with respect to common criteria of quality of test methods (validity, repeatability, etc.). They are applicable to polyacrylate superabsorbent materials, which occur in hygiene products, including urine-absorbing aids for incontinent persons. The test methods are addressed to the *material* exclusively. They are not intended to be used, and are not applicable for use with finished manufactured urine-absorbing aids.

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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 10: Determination of extractable polymer content by potentiometric titration

1 Scope

This part of ISO 17190 specifies a method for determining the mass fraction of carboxylate groups of varying chain length present in polyacrylate (PA) superabsorbent powders which are extractable in saline solution.

This method has been tested in the extractable polymer mass fraction range from 5,29 % to 9,00 % (see annex A), but it is expected to be applicable to a wider range.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 17190. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 17190 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this part of ISO 17190, the following terms and definitions apply.

3.1
extractables
sum of the soluble acid and salt groups of monomeric, oligomeric and polymeric carboxylates extracted from the superabsorbent polymer

3.2
salt group
alkali metal salt of carboxylic acid group, also referred to as neutralised carboxylate

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3.3
acid group
carboxylic acid group

4 Principle

The content of extractables in PA superabsorbent powders is determined by first mixing the PA superabsorbent powder with saline solution for a period of time sufficient to substantially approach equilibrium. The saline solution is allowed to settle and a portion of the supernatant liquid filtered. 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 subsequently titrated against a standard acid solution (HCl) to pH 2,7 to determine the concentration of neutralised carboxylates. The titration data are used to calculate total amount of extractables present in the PA superabsorbent powders.

5 Reagents

WARNING — Concentrated acids and bases shall be handled with care. Safety protection, including gloves and face shields, shall be worn. Concentrated hydrochloric acid shall be handled under a fume hood.

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

5.1 Water, complying with ISO 3696.

5.2 Sodium hydroxide solution, c(NaOH) = 0,1 mol/l.

Weigh, to the nearest 0,1 g, 4 g sodium hydroxide into a 1 l volumetric flask (6.3) and make up to the mark with deionized water (grade 1, see 5.1). Stir until dissolved.

5.3 Hydrochloric acid solution, c(HCl) = 0,1 mol/l.

Add, to the nearest 0,1 ml, 8,9 ml concentrated hydrochloric acid to a 1 l volumetric flask (6.3) and make up to the mark with deionized water (grade 1, see 5.1). Stir until dissolved.

5.4 Sodium chloride solution, c(NaCl) = 0,9 % by mass.

Weigh, to the nearest 0,1 g, 9 g of sodium chloride into a 1 l volumetric flask (6.3) and make up to the mark with deionized water (grade 3, see 5.1). Stir until dissolved.

5.5 Standard buffer solutions, having the following pH:

a) 4,0 ± 0,02

b) 7,0 ± 0,02

c) 10,0 ± 0,02

6 Apparatus

6.1 Analytical balance, capable of weighing, to the nearest 0,001 g, masses up to 1 g.

6.2 pH meter and **combined glass pH-responsive electrode** (referred to in the text as pH electrode).

6.3 Volumetric flask, Grade A of 1 l capacity.

6.4 Weighing dish, made of aluminium.

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6.5 Titration vessels, glass beakers or conical flasks.

6.6 Conical flask, made of glass, of 250 ml capacity, equipped with NS 29/32 standard joints and stoppers.

6.7 Beaker, made of glass, of 250 ml capacity.

6.8 Paraffin film.

6.9 Vacuum filter bottles.

6.10 Buchner funnels, for holding 70 mm diameter filters.

6.11 Filter papers, 70 mm in diameter with a pore size < 20 µm.

6.12 Filter pump system, capable of achieving filtration of the sample within 5 min (e.g. water jet pump).

6.13 Measuring cylinder, of 200 ml capacity and accurate to $\pm$ 0,5 %.

6.14 Magnetic stirrer, capable of stirring at a rate of (500 $\pm$ 50) r/min.

6.15 Magnetic stirrer bars, having a cylindrical form, 30 mm $\times$ 6 mm or equivalent.

6.16 Burette, made of glass, 10 ml to 20 ml capable of being read to the nearest 0,01 ml.

7 Sampling

CAUTION — Use respiratory protection, dust mask or fume hood, when handling sample amounts greater than 10 g.

In order to guarantee that a representative sample is taken from the bulk material contained in a large bag or a silo truck, remove the top layer (approximately 20 cm). Take the test sample with a scoop. Place it in an airtight container of adequate size within 3 min after sampling.

Keep the test samples in a closed container and allow them to equilibrate to the ambient laboratory temperature before removing a test portion to run the test. The preferred test conditions are (23 $\pm$ 2) °C and (50 $\pm$ 10) % 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.

Before taking a test portion out of the container to run the test, rotate the container three to five times so as to obtain a homogeneous product. Allow the container to sit 5 min before opening the lid and removing the test portion.

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

8 Procedure

8.1 Prepare each test portion in duplicate.

8.2 Accurately add 200 ml of saline solution (5.4) using the measuring cylinder (6.13) to a 250 ml conical flask (6.6) or 250 ml beaker (6.7) containing a magnetic stirrer bar (6.15).

8.3 Weigh, to the nearest 0,001 g, a 1 g test portion from the PA superabsorbent powder test sample into a weighing dish (6.4), record the mass, and disperse it into the saline solution. Ensure that all the sample has been transferred.

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8.4 Stopper the flask, or seal the beaker with paraffin film (6.8), and stir (see 6.14) the solution at a rate of (500 ± 50) r/min for 16 h.

8.5 Prepare a titration blank by stirring 200 ml of saline solution alongside the sample solutions.

8.6 After 16 h stop stirring the solutions and allow the gel to settle.

8.7 Filter the supernatant using the Buchner apparatus (6.10 and 6.12) with filter paper (6.11) and collect more than 50 ml.

8.8 Calibrate the pH electrode (6.2) using pH 4,0, pH 7,0, and pH 10,0 buffer solutions (5.5).

8.9 Using a burette (6.16), titrate 50 ml of the blank saline filtrate to pH 10,0 against the standard sodium hydroxide solution (5.2), and then to pH 2,7 against the standard hydrochloric acid solution (5.3). Record each titrant volume used to reach the end point.

8.10 Using a burette (6.16), titrate 50 ml of the sample filtrate to pH 10,0 against the standard sodium hydroxide solution (5.2), and then to pH 2,7 against the standard hydrochloric acid solution (5.3). Record each titrant volume used to reach the end point.

8.11 Each titration should take a few minutes to complete.

9 Calculation

The amount of carboxylic acid (e.g. polycarboxylic acid), expressed in moles, in the supernatant aliquot, n_{COOH}, is given by equation (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 saline filtrate 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 equation (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 to pH 2,7;

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

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

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The amount of neutralised carboxylate, n_{COONa}, expressed in moles, in the supernatant aliquot is given by equation (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 equations (4) and (5) respectively:

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

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

where

M_{COOH} is the molar mass of acrylic acid, equal to 72 g/mol;

M_{COONa} is the molar mass of sodium acrylate, equal to 94 g/mol;

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 equation (6):

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

where m_s is the mass, in grams, of the test portion.

If the degree of neutralisation is known, equation (7) can be used to determine the extractable content, w, expressed as a mass fraction in percent, of the superabsorbent polymer:

$$w = \frac{\left(V_{HCl,s} - V_{HCl,b}\right) c_{HCl} \times M_{acr} \times F_{dil}}{m_s \times 1\,000} \times 100 \quad (7)$$

where

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

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

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

M_{acr} is the molar mass of the monomer unit of acrylate, equal to 87,46 g/mol for a 70 % neutralised salt;

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.

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10 Precision

The data for the repeatability and reproducibility limits of this method are the result of interlaboratory tests carried out in 1997 by EDANA and are given in annex A.

The absolute difference between two single test results obtained under repeatability test conditions in accordance with ISO 5725-2 shall not exceed the repeatability limit r in more than 5 % of cases:

r = 1,94 %

The absolute difference between two single test results obtained under reproducibility test conditions in accordance with ISO 5725-2 shall not exceed the reproducibility limit R in more than 5 % of cases:

R = 10,47 %

If the repeatability and reproducibility test criteria are not met, the test shall be repeated twice, each in duplicate, after ensuring that the original sample is thoroughly mixed. If these criteria are still not met, report the results as unusual, then diagnose the source of error for example by verifying correct operation of the instruments and testing a portion of a material with a known value.

11 Test report

The test report shall include the following information:

a) the name and address of the testing institution;

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

c) a reference to this part of ISO 17190, i.e. ISO 17190-10;

d) whether or not lumps were present in the sample;

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

f) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 10);

g) any deviations from the procedure, or any operations regarded as optional.

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Annex A
(informative)

Statistical results of interlaboratory tests

Figures for the repeatability and reproducibility of this method are the result of collaborative studies carried out in 1997 by EDANA. The evaluation of the interlaboratory test was carried out in accordance with ISO 5725-2 and results as follows:

Sample identification	A	B	C
Number of participating laboratories	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	10	10	10
Number of accepted test results	40	40	40
Mean value (%)	5,29	9,00	8,67
Repeatability standard deviation (s_r)	0,41	0,57	0,69
Repeatability coefficient of variation	7,82 %	6,32 %	7,99 %
Repeatability limit (r) ($2{,}8 \times s_r$)	1,16	1,59	1,94
Reproducibility standard deviation of reproducibility (s_R)	1,29	3,74	1,28
Reproducibility coefficient of variation	24,41 %	41,51 %	14,79 %
Reproducibility limit (R) ($2{,}8 \times s_R$)	3,62	10,47	3,59

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