INTERNATIONAL STANDARD

ISO 17190-2

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

Part 2: Determination of amount of residual monomers

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

Partie 2: Détermination de la quantité de monomères résiduels

Reference number
ISO 17190-2:2001(E)

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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 17190-2 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*.

Annexes A and B of this part of ISO 17190 are given for information only.

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.

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

Part 2: Determination of amount of residual monomers

1 Scope

This part of ISO 17190 specifies a method for determining the sum of residual monomeric sodium acrylate and acrylic acid present in polyacrylate (PA) superabsorbent powders as acrylic acid.

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 Term and definition

For the purposes of this part of ISO 17190, the following term and definition applies.

3.1
amount of residual monomers
sum of residual monomeric sodium acrylate and acrylic acid

4 Principle

Residual acrylic acid is extracted from the PA superabsorbent powders and amount of residual acrylic acid is determined by HPLC.

5 Reagents

5.1 General

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

5.1.1 Water, complying with ISO 3696.

5.1.2 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 (6.5) and make up to the mark with deionized water (grade 3, see 5.1.1). Stir until dissolved.

5.1.3 Phosphoric acid, concentrated $c(H_3PO_4)$ = 85 % by mass, of HPLC grade or better.

5.1.4 Phosphoric acid solution, $c(H_3PO_4)$ = 0,1 % by mass (1 g/l or 0,008 7 mol/l). Dilute concentrated H_3PO_4 (5.1.3) to volume with deionized water (grade 3, see 5.1.1). Stir until mixed.

5.1.5 Acetonitrile, of HPLC grade or better.

5.1.6 Acrylic acid, of > 99,5 % purity.

It is well known that acrylic acid degrades over time. It is therefore important to measure the purity of acrylic acid used to calibrate the HPLC.

5.2 Standard solutions for calibration

5.2.1 Solution S1 [ρ_{cal}(S1) = 1000 mg/l].

Weigh, to the nearest 0,000 5 g, 0,100 0 g acrylic acid (5.1.6) into the 100 ml volumetric flask (6.5) labelled S1. Make up to the mark with ultrapure water (5.1.1). Using this solution, prepare the dilutions given in 5.2.2 to 5.2.6.

5.2.2 Solution S2 [ρ_{cal}(S2) = 100 mg/l].

Pipette 10 ml from S1 into the 100 ml volumetric flask (6.5) labelled S2 and make up to the mark with ultrapure water (5.1.1)

5.2.3 Solution S3 [ρ_{cal}(S3) = 1 mg/l].

Pipette 1 ml from S1 into the 100 ml volumetric flask (6.5) labelled S3 and make up to the mark with ultrapure water (5.1.1)

5.2.4 Solution S4 [ρ_{cal}(S4) = 2 mg/l].

Pipette 2 ml from S1 into the 100 ml volumetric flask (6.5) labelled S4 and make up to the mark with ultrapure water (5.1.1).

5.2.5 Solution S5 [ρ_{cal}(S5) = 3 mg/l].

Pipette 3 ml from S1 into the 100 ml volumetric flask (6.5) labelled S5 and make up to the mark with ultrapure water (5.1.1).

5.2.6 Solution S6 [ρ_{cal}(S6) = 4 mg/l].

Pipette 4 ml from S1 into the 100 ml volumetric flask (6.5) labelled S6 and make up to the mark with ultrapure water (5.1.1).

6 Apparatus

6.1 Analytical balance, capable of weighing, to the nearest 0,000 1 g, masses up to 0,1 g.

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

6.3 Glass beaker or **conical flasks**, of 250 ml capacity.

6.4 Graduated cylinder, of 200 ml capacity and accurate to ± 0,5 %.

6.5 Volumetric flasks, Grade A, of 100 ml and 1 l capacities.

6.6 Volumetric pipettes, Grade A, of 1 ml, 2 ml, 3 ml, 4 ml and 10 ml capacities.

6.7 Magnetic stirrer, capable of stirring at a rate of (500 ± 50) r/min**,** with **stirring bars**.

6.8 Filters, 0,45 µm.

6.9 HPLC injection system, for injection volumes ranging from 20 µl to 100 µl, of analyte solution and accurate to within ± 1 %.

6.10 HPLC pump, capable of delivering flows with a the theoretical back-pressure to within ± 10 %.

6.11 C18 column, with 5-µm particle-size packing material, 250 mm length × 4,6 mm internal diameter (ID), fitted with a guard column (6.12).

6.12 Guard column, C18, with 5-µm particle-size packing material, 50 mm length × 4,6 mm internal diameter (ID).

6.13 UV detector for HPLC, capable of making measurements at a wavelength of 210 nm.

7 Sampling

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

7.1 Test sample

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 ± 2) °C and (50 ± 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.

7.2 Sample preparation

7.2.1 Weigh, to the nearest 0,005 g, a 1,000 g test portion of PA superabsorbent powder test sample into a clean weighing boat and record the mass (m_{sam}) as the test portion.

7.2.2 Add the test portion to a clean beaker (6.3) or conical flask (6.3) and make sure that all of the sample has been transferred.

7.2.3 Measure 200 ml of 0,9 % saline solution (5.1.2) using the measuring cylinder and add it to the beaker or flask.

7.2.4 Add a magnetic stirrer bar then seal the beaker with paraffin film or stopper the flask.

7.2.5 Stir the solution at (500 ± 50) r/min for 60 min.

7.2.6 After 60 min, stop the stirring.

7.2.7 Allow the polymer to settle for 5 min.

7.2.8 Filter the supernatant through a 0,45 µm filter and keep this for HPLC analysis.

8 Procedure

8.1 HPLC chromatographic conditions

Use the following HPLC chromatographic conditions:

Injection: 20 µl to 100 µl

Mobile phase (by volume): [10 % acetonitrile (5.1.5)]:[90 % phosphoric acid solution (5.1.4)]

Flow rate: 1 ml/min

Analytical column: C18 column (6.11)

Guard column: C18 (6.12)

Detection: UV at 210 nm (6.13)

The peak areas are obtained using an integrator, data station, or computer. Under these conditions, acrylic acid usually elutes within 5 min to 6 min.

Alternative methods and chromatographic conditions are listed in annex A.

8.2 Calibration

Analyse the standard solutions S3 (5.2.3) to S6 (5.2.6) in duplicate. Average the peak area obtained for each level (A_i).

If the difference between the duplicate peak areas of duplicate analyses exceeds 5 % of the average, repeat the determination in duplicate. If this difference is less than 5 %, use the average value of the duplicate determinations for the calibration curve.

8.3 Determination

Analyse the test portion obtained in 7.2.8 in duplicate. Average the peak area obtained.

9 Calculation

9.1 Calibration curve

Plot the acrylic acid mass concentration, $\rho_{cal,i}$, against the average peak area A_i to obtain the calibration curve. Assuming this relationship is linear, determine the equation of the line of regression of mass concentration ρ on the average peak area A as follows:

$$\rho_{cal} = aA + b \quad (1)$$

where

ρ_{cal} is the mass concentration, expressed in milligrams per litre, of analyte;

A is the peak area of analyte;

a is the slope of the line;

b is the intercept on the y-axis (ρ_{cal}).

Determine the correlation coefficient for the line of regression obtained for equation (1). This correlation coefficient should be greater than 0,99.

9.2 Amount of residual monomers

Calculate the mass concentration of the extraction solution ρ_{sam} taken from the test portion (8.3) using equation (1) obtained for the calibration curve in 9.1, where A_{sam} is the peak area obtained for the sample.

The amount of residual monomers in PA superabsorbent powders is the mass fraction of residual acrylic acid determined (w_{acr}), expressed in milligrams per kilogram, and is calculated as follows:

$$w_{acr} = \rho_{sam} \frac{200}{m_{sam}} \quad (2)$$

where

ρ_{sam} is the mass concentration, expressed in milligrams per litre, of the extraction solution;

m_{sam} is the mass, expressed in grams, of polymer taken for the test portion;

200 is the volume, expressed in millilitres, of extraction solution.

Report the results to the nearest 1 mg/kg.

10 Precision

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

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 = 55 mg/kg

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 = 416 mg/kg

The high reproducibility limit stems probably from calibration problems due to ageing of acrylic acid (see 5.1.6).

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 it fails to satisfy this criterion, report 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.

Deviations from the given chromatographic procedure are acceptable if the level of precision and accuracy in both peak separation and quantitation are equal to, or better than those listed above. Some examples of deviations from the chromatographic procedure are listed in annex A.

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 17190-2;

d) the results of the amount of residual monomers for each test (9.2), expressed as mass fraction of residual acrylate monomer in PA (mg/kg), and the average for duplicate determinations;

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

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

Annex A
(informative)

Alternative methods and chromatographic conditions

A.1 Neyer chromatographic method

The procedure specified in this part of ISO 17190 is a modified version of that published by J.M. Neyer *et al* (see reference [1] in the Bibliography).

The linear range of the Neyer method is 0,01 mg/l to 10 mg/l acrylic acid, equivalent to 2 mg/kg to 2 000 mg/kg of residual monomers in PA superabsorbent powders. The limit of detection of this method is 0,01 mg/l of acrylic acid.

A.2 Alternative chromatographic conditions

A.2.1 Acceptable deviations from the chromatographic method

Deviations from the given chromatographic procedure are acceptable if the level of precision and accuracy in both peak separation and quantitation are equal to, or better than those given in clause 10. For example:

— Any equivalent reversed-phase chromatography column may be used.

— The mobile phase organic modifier can be omitted if high purity, pH-stable reversed-phase columns are used. See, for example, Figure A.1 where separation was obtained without an organic modifier.

Note that C18 phases are subject to “hydrophilic collapse” in the absence of an organic modifier in the mobile phase. This can result in shifts in retention time. Column performance should be verified under such circumstances.

— Ion exclusion chromatography columns may be used in place of reversed-phase chromatography columns with 0,005 mol/l sulfuric acid recommended as the mobile phase.

— Chromatography columns of different dimensions may be used as long as analyte separation is achieved. See Figure A.1 where the chromatographic column is specified in A.2.2.

— Filtration aids may be added at the end of the 1 h stirring time, for example 1 ml of H_3PO_4.

A.2.2 Alternative HPLC system

The following system is an alternative HPLC system which gives the separation shown in Figure A.1.

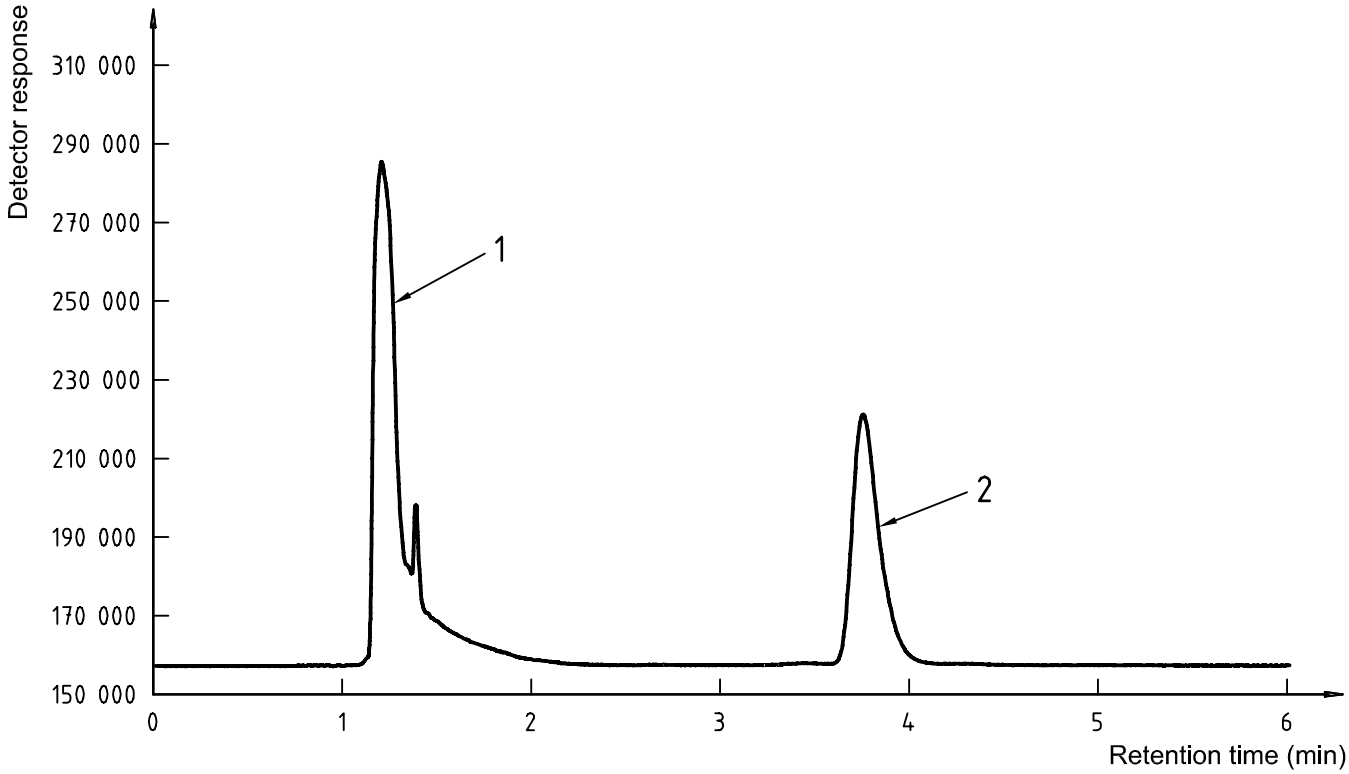
Mobile phase (by volume): [10 % acetonitrile (5.1.5)]:[90 % phosphoric acid solution (5.1.4)]

Flow rate: 1 ml/min

Analytical column: C18, 5-µm particle-size packing material, 100 mm length × 4,6 mm ID

Guard column: C18 (6.12)

Detection: UV at 210 nm (6.13)



Key

1 Hold-up volume of column

2 Acrylic acid

Figure A.1 — Chromatogram of residual acrylate using an alternative HPLC system

Annex B
(informative)

Statistical results of interlaboratory tests

Samples for the round robin test have been mainly selected for testing the accuracy of the method. They are not representative of the products on the market (for example sample A came from a batch produced in the past).

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 are 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)	9	9	9
Number of accepted test results	36	36	36
Mean value (mg/kg):	716	356	400
Repeatability standard deviation (s_r)	18,58	13,95	19,52
Repeatability coefficient of variation	2,60 %	3,92 %	4,88 %
Repeatability limit (r) ($2{,}8 \times s_r$)	52,03	39,07	54,66
Reproducibility standard deviation of reproducibility (s_R)	148,51	83,98	84,76
Reproducibility coefficient of variation	20,77 %	23,58 %	21,19 %
Reproducibility limit (R) ($2{,}8 \times s_R$)	415,83	235,15	237,32

Bibliography

[1] NEYER J.M., VIGOUROUX A., VAMVAKARIS C. and MANDERY H. High-Performance Liquid Chromatographic Determination of Monomeric Sodium Acrylate and Acrylic Acid in Polyacrylic Gelling Agents. *Chromatographia*, **25** (10) 1988

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