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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 2:

Test method for determination of the amount of residual acrylate 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

ICS: 11.180.20

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Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Reagents and Materials	2
5.1 Water	2
5.2 Sodium chloride solution	2
5.3 Phosphoric acid	2
5.4 Phosphoric acid solution	2
5.5 Acetonitrile	2
5.6 Acrylic acid	2
6 Apparatus	2
7 Calibration	4
8 Conditioning	4
9 Sampling	4
10 Procedure	5
11 Calculation	6
11.1 Calibration curve	6
11.2 Amount of residual monomers	6
12 Report	6
13 Precision	7
Annex A (informative) Notes on chromatographical testing	8
Annex B (informative) Modification Track Sheet	10
Bibliography	11

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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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

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Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 2:

Test method for determination of the amount of residual acrylate monomers

1 Scope

This test method determines the sum of residual monomeric sodium acrylate and acrylic acid present in polyacrylate (PA) superabsorbent powders as acrylic acid.

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

amount of residual monomers

sum of residual monomeric sodium acrylate and acrylic acid

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

3.3

specimen

specific portion of the identified sample upon which a test is performed

4 Principle

Residual monomeric sodium acrylate and acrylic acid are extracted from the polyacrylate superabsorbent powders and the amount determined as residual acrylate monomer by HPLC.

The method described here uses a simple 1-g in 200-ml dilution factor. However, it is equally valid to use different masses and volumes as long as the mass: volume dilution ratio is the same and suitable experimental validation is performed.

5 Reagents and Materials

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

5.1 Water

Grade 1 water according to ISO 3696

5.2 Sodium chloride solution

5.2.1 0.9% by mass (m/m) 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 16mS/cm (depending on temperature). 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 ± 2) °C for the duration of the test.

5.3 Phosphoric acid

concentrated $c(\text{H}_3\text{PO}_4) = 85\%$ by mass, of HPLC grade or better

5.4 Phosphoric acid solution

$c(\text{H}_3\text{PO}_4) = 0.1\%$ by mass (1 g/l or 0.0087 mol/l). Dilute concentrated H_3PO_4 to volume with deionised water (grade 1). Stir until dissolved.

5.5 Acetonitrile

HPLC grade or better

5.6 Acrylic acid

Greater than 99.5% purity

Acrylic acid degrades over time owing to slow polymerisation, particularly dimerization, and water uptake. It is important to obtain acrylic acid that meets the purity required for this test method. It is not recommended to obtain the reference substance from a laboratory chemicals distributor as the purity of this material from such a source is likely to be inaccurately defined.

6 Apparatus

6.1 Analytical balance, capable of weighing a mass of 100.000 ± 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 1000.00 ± 1.00 g of sodium chloride solution in combination with the mass of the vessel employed

6.4 Graduated 250 ml beaker (tall form to give better separation of gel and saline)

6.5 Grade A 100 ml graduated measuring cylinder, accurate to 0.5%

6.6 Grade A volumetric flasks 100 ml and 1 l

6.7 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)

6.8 Metal spatula to accommodate 1.0 g of superabsorbent powder

6.9 Filters - 0.45 μ m pore size PTFE filters

6.10 HPLC injection system allowing injection volumes between 10 μ l and 100 μ l of analyte solution, accurate to $\pm 1\%$

6.11 HPLC pump capable of delivering flows with a maximum back-pressure variability of $\pm 10\%$

6.12 C18 column with suitable packing material. For example, 5 μ m particle size, 100 mm length x 4.6 mm internal diameter (ID), fitted with a guard column.

6.13 Guard column with suitable packing material. For example, 5 μ m particle size, 10 mm length x 4.6 mm internal diameter (ID).

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

6.15 Standard Solutions for Calibration

Use grade 1 ultra pure water

6.15.1 Solution S1 [$\text{pcal}(S1) = 1000$ mg/l]

Weigh 0.1000 g, ± 0.0005 g, of acrylic acid into the 100 ml volumetric flask labelled S1. Make up to the mark with ultra pure water. This contains 1000 mg/l acrylic acid. Using this solution, prepare the dilutions as follows:

6.15.2 Solution S2 [pcal(S2) = 100 mg/l]

Pipette 10 ml from S1 into the 100 ml volumetric flask labelled S2 and make up to the mark with ultra pure water. This contains 100 mg/l acrylic acid.

6.15.3 Solution S3 [pcal (S3) = 1 mg/l]

Pipette 1 ml from S2 into the 100 ml volumetric flask labelled S3 and make up to the mark with ultra pure water.

6.15.4 Solution S4 [pcal (S4) = 2 mg/l]

Pipette 2 ml from S2 into the 100 ml volumetric flask labelled S4 and make up to the mark with ultra pure water.

6.15.5 Solution S5 [pcal (S5) = 3 mg/l]

Pipette 3 ml from S2 into the 100 ml volumetric flask labelled S5 and make up to the mark with ultra pure water.

6.15.6 Solution S6 [pcal (S6) = 4 mg/l]

Pipette 4 ml from S2 into the 100 ml volumetric flask labelled S6 and make up to the mark with ultra pure water.

7 Calibration

7.1 Analyze the standard solutions S3 to S6 in duplicate. Average the peak area obtained for each level (Ai).

7.2 If the difference between the duplicate peak areas for S3 to S6 is less than 5%, use the average value for the calibration curve. If the difference of any of the duplicate analyses exceeds 5% of the average, repeat the calibration in duplicate. If the differences continue to exceed 5%, identify and eliminate the source of error.

8 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.

9 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-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 MAK value.

9.1 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.

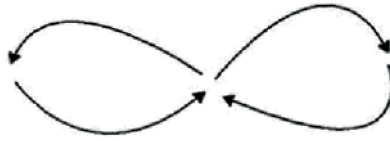


Figure 2 — Sense of motion of the container

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

10 Procedure

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.

10.1 Accurately add 200.0 ± 0.1 ml of saline solution to a 250 ml glass beaker using the measuring cylinder.

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

10.3 Add 1.000 ± 0.005 g of a test portion of PA superabsorbent powder test sample to the weighing vessel or laboratory paper and tare the balance again.

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.

10.4 Carefully distribute the test portion into the beaker containing the 200.0 ± 0.1 ml of saline.

10.5 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} .

10.6 Cover the beaker with a Petri dish lid or seal with paraffin film, and stir the solution at a rate of 250 ± 50 r.min⁻¹ for 1 hour.

10.7 After 1 hour, stop stirring the solutions and allow the polymer to settle for 5 minutes.

10.8 Filter a portion of the supernatant through a $0.45 \mu\text{m}$ PTFE filter and use this for the HPLC analysis.

Table 1 — Example HPLC chromatographic conditions:

Injection:	10 μl to 100 μl
Mobile phase (by volume):	[10% acetonitrile: 90% phosphoric acid solution]
Flow rate:	1 ml.min ⁻¹
Analytical column:	C18 column (e.g. 100 x 4.6 mm)
Guard column:	C18 column (e.g. 10 x 4.6 mm)
Detection:	UV at 210 nm