
**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



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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.

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 17190-2:2001), 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 2:

Test method for determination of the amount of residual acrylate monomers

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 for determining the sum of residual monomeric sodium acrylate and acrylic acid present in polyacrylate superabsorbent powders as acrylic acid.

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* (3.2) 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, for the standard solutions for the calibration and Grade 3 water according to ISO 3696, with the exception that the conductivity can be as high as 30 $\mu\text{S}/\text{cm}$, for the saline solution.

5.2 Sodium chloride solution.

5.2.1 0,9 % mass fraction of sodium chloride solution in water. Weigh $(9,00 \pm 0,01)$ g of sodium chloride into a 1 l beaker and add $(991,0 \pm 0,1)$ g of deionized 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 1600 S/m (depending on temperature). 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.

5.3 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 deionized 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 polymerization, 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 defined inaccurately.

6 Apparatus

6.1 Analytical balance, capable of weighing a mass of $(100,000 \pm 0,001)$ 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 $(9,00 \pm 0,01)$ 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 $(1000,00 \pm 1,00)$ g of sodium chloride solution in combination with the mass of the vessel employed.

6.4 Graduated 250 ml tall form beaker, 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-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 crosshead 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 µm pore size PTFE filters.

6.10 HPLC injection system, allowing injection volumes between 10 µl and 100 µl of analyte solution, accurate to ± 1 %.

6.11 HPLC pump, capable of delivering flows with a maximum back-pressure variability of ± 10 %.

6.12 C18 column, with suitable packing material. For example, 5 µm 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 µm 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 [pcal(S1) = 1 000 mg/l].

Weigh (0,1000 g $\pm$ 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 1 000 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 (A_i).

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 $\pm$ 2) °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.

9 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 μ 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.

9.1 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.

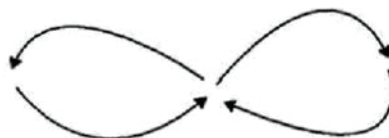


Figure 2 — Sense of motion of the container

9.2 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.

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 $(200,0 \pm 0,1)$ 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 $(1,000 \pm 0,005)$ g of a test portion of polyacrylate 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.

10.4 Evenly distribute the test portion into the beaker containing the $(200,0 \pm 0,1)$ 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, using the chromatographic conditions as indicated in [Table 1](#).

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

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

11 Calculation

11.1 Calibration curve

Plot the acrylic acid mass concentrations, p_{cal} , against the average peak areas A for calibration solutions S3 to S6 to obtain the calibration curve. Determine the equation of the line of linear regression of mass concentration p vs. the average peak area A by Formula (1):

Formula 1:

$$p_{cal} = aA + b$$

where

p_{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.

Determine the correlation coefficient for the line of regression obtained for Formula (1). This correlation coefficient should be greater than 0,99. An example of such a curve is given in [Annex A](#) (see [Figure A.2](#)).

11.2 Amount of residual monomers

Obtain the mass concentration of the extraction solution, p_{sam} , taken from the test portion by calculation using Formula (1) or by interpolation from the calibration curve, where A_{sam} is the peak area obtained for the sample.

The amount of residual monomers in the test portion is the mass fraction of residual acrylate monomer determined, W_{acr} , expressed in milligrams per kilogram, and calculated as follows:

$$W_{acr} = p_{sam} \frac{200}{M_{sam}}$$

where

- p_{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

12 Report

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

- Reference to this document, i.e. ISO 17190-2:2021;
- Complete identification of all materials tested and method of sampling;
- Name and address of testing institution;
- The type of polymer-based absorbent materials, including all technical details and source information required for complete identification of the sample;
- The results, to the nearest 1 mg.kg⁻¹, of the amount of residual monomers for each test, expressed as mass fraction of residual acrylate monomer in polyacrylate (mg.kg⁻¹), and the average for duplicate determinations;
- Laboratory testing conditions;
- Number of specimens tested;
- For computer processed data, identify the software used and the version;
- Deviation from the standard test procedure, if any.

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.

13 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 2](#) provides the result of that evaluation.

This method has been validated over the range of 236 mg/kg to 457 mg/kg. 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 2 — Repeatability (r) and Reproducibility (R) of the method

Test	Sample	N	Min	Max	Mean	r	R
RM	AJ224	173	236,00	367,00	300,47	44,69	53,78
	WR384	172	240,00	367,50	307,83	38,02	48,52
	XZ329	173	330,00	457,00	392,86	38,45	47,80

Annex A (informative)

Notes on chromatographical testing

Chromatographic conditions

Deviations from the given chromatographic procedures are acceptable provided the level of precision and accuracy in both peak separation and quantification are equal to, or better than those listed above. Some examples of deviations from the chromatographic procedure are listed here.

Neyer chromatographic method

This procedure is a modified version of the Neyer method published in Reference [3].

The linear range of the Neyer method is 0,01 mg/l to 10 mg/l acrylic acid, equivalent to 2 mg.kg⁻¹ to 2000 mg.kg⁻¹ of residual monomers in polyacrylate superabsorbent powders. The limit of detection of this method is 0,01 mg/l of acrylic acid.

Alternative chromatographic conditions

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.

CAUTION — The 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.

In exclusion chromatography columns may be used in place of reversed-phase chromatography columns with 0,005 mol/l sulphuric 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 [Figure A.2](#), showing an example of a calibration curve.

Filtration aids may be added at the end of the 1-hour stirring time, for example 1 ml of H₃PO₄.