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

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

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

Partie 11: Détermination de la teneur en particules respirables



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 17190-11 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.

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 11: Determination of content of respirable particles

1 Scope

This part of ISO 17190 specifies a method for determining the content of respirable particles in polyacrylate (PA) superabsorbent powders.

NOTE Commercial polyacrylate superabsorbent powders consist of particles averaging 400 μm to 800 μm in diameter, which are much larger than the maximum size for respirable particles of approximately 10 μm . Due to the production process, very low amounts of respirable particles may be present in commercial superabsorbent powders.

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*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

3 Term and definition

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

3.1

respirable particle

particle having a size smaller than 10 μm

4 Principle

The particle size fraction < 106 µm of the superabsorbent powders is separated from the bulk by sieving the bulk with a set of sieves with 500 µm, 250 µm and 106 µm pore sizes. The fraction of respirable particles (< 10 µm) is determined by analysing the fraction of particle sizes < 106 µm by a laser-light scattering particle-size analyser using light diffraction. A wet dispersion in dry petroleum ether of the particle size fraction < 106 µm is used for the determination of respirable particles.

5 Reagents

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

5.1 Petroleum ether, dried, with a boiling range from 60 °C to 80 °C.

6 Apparatus

6.1 Analytical balance, capable of weighing, to the nearest 0,01 g, masses up to 800 g.

6.2 Beaker, made of glass, of 150 ml capacity.

6.3 Sieve shaker, type Retsch 3 D¹⁾, RO-TAP Model B¹⁾, or equivalent, designed to hold at least three stainless steel screens (6.4) and equipped with a bottom receiving pan and a lid, and grounded for avoiding static electricity.

6.4 Stainless steel screens, with pore sizes of 500 µm, 250 µm and 106 µm. Use 200-mm diameter screens for the RO-TAP Model B sieve shaker and 100-mm or 200-mm diameter screens for the Retsch 3 D (or equivalent) sieve shaker.

6.5 Brush, for example made of camel's hair, for cleaning of standard sieves.

6.6 Dust mask.

6.7 Laser-light scattering particle analyser, using the diffraction theory, e.g. Coulter LS 100²⁾, Sympatec²⁾, Malvern²⁾, Cilas²⁾ or equivalent devices.

6.8 Sample divider, for example a Retsch type PT³⁾.

1) Retsch 3 D and RO-TAP Model B are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 17190 and does not constitute an endorsement by ISO of this product.

2) Coulter LS 100, Sympatec, Malvern and Cilas are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 17190 and does not constitute an endorsement by ISO of this product.

3) Retsch type PT is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 17190 and does not constitute an endorsement by ISO of this product.

7 Sampling

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

7.1 Preparation of 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 $(23 \pm 2) ^\circ\text{C}$ 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.

7.2 Test portion

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.

Using a sampling technique specified in ISO 8213, prepare, to the nearest 1 g, 100 g test portions.

If a Retsch sample divider PT (6.8) is to be used, prepare the test portions as follows:

- a) connect eight glasses (250 ml) to the eight tubes of the divider;
- b) weigh, to the nearest 1 g, 800 g of the representative homogeneous test sample in a beaker;
- c) transfer the test sample into the funnel of the sample divider;
- d) start the divider and set the timer to 15 min.

8 Procedure

8.1 Check screens (6.4) for damage and cleanliness and weigh, to the nearest 0,01 g, and record the mass of the bottom pan.

Place the screens in the right order on the shaker, i.e. the finest at the bottom and the coarsest at the top.

8.2 Transfer the contents from one divider tube or equivalent 100 g from another type of sample divider into a beaker, and weigh this test portion to the nearest 0,01 g (m_s).

8.3 Transfer the entire test portion to the sieve tester and pour it on the top of the screens. Place the lid on the sieves and secure it in accordance with the manufacturer's instructions.

8.4 Set the sieve shaker as follows:

- RO-TAP: (150 ± 5) beats/min and (285 ± 10) r/min
- Retsch: $(70 \pm 2) \%$ intensity

Earth the equipment to avoid static electricity. Set the shaker time to 10 min and start the shaker.

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- 8.5** Carefully remove the screens. Weigh, to the nearest 0,01g, the bottom pan with its contents and calculate the mass of the particles passing through the 106 µm sieve and collected in the bottom pan (m_{106}).
- 8.6** Repeat 8.1 to 8.5 for each divider tube or equivalent test portion.
- 8.7** Collect and pool the samples below 106 µm particle size for measurement by laser scattering.
- 8.8** Calibrate the laser particle analyser (6.7) using an appropriate standard in accordance with the manufacturer's instructions (e.g. silicon carbide or equivalent).
- 8.9** Divide the superabsorbent powders collected in the bottom pan into equal aliquots. The size of one aliquot is determined by the amount which is necessary to run an analysis in the laser-light particle size analyser.
- 8.10** Transfer one aliquot to the cuvette with dry petroleum ether (5.1) as described in the manufacturers' operating manual of the laser-light particle size analyser. While measuring this aliquot, keep the remaining aliquots away from humidity (close the bottom pan or use individual closed containers for the aliquots).
- 8.11** Place the cuvette in the sample holder of the laser-light particle size analyser and run the determination according to the manufacturers' operating manual. Stir the solution during the measurement to avoid sedimentation.
- 8.12** Record the percentage of respirable particles in the aliquot, p_i .
- 8.13** Repeat 8.10 and 8.12 with all aliquots prepared in 8.9.
- 8.14** Repeat 8.1 to 8.13 at least three times.

9 Calculation

Calculate the average percentage, p , of respirable particles from all aliquots, p_i .

Calculate the amount of respirable particles based on initial test portion mass, w_{106} , expressed as a mass fraction in percent according to equation (1).

$$w_{106} = p \frac{m_{106}}{m_s} \quad (1)$$

where

m_{106} is the mass, expressed in grams, of the particles passing through the 106 µm sieve and collected in the bottom pan;

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

Calculate the average amount of respirable particles on the (at least) three test portions.

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:

$$r = 0,011 \%$$

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:

$$R = 0,050 \%$$

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 17190-11;
- d) the sampling technique used;
- e) the number of tested samples;
- f) the type of sieve shaker;
- g) the type of laser analyser;
- h) the mass of the < 106 μm fraction, m_{106} , for each test portion;
- i) the results of the percentage of respirable particles based on initial sample mass, w , expressed as a mass fraction in percent, for each test portion and the average percentage of respirable particles;
- j) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 10);
- k) any deviations from the procedure, or any operations regarded as optional.

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, with the following data. 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	7	7	7
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	5	6	4
Number of accepted test results	15	18	12
Mean value (%)	0,007	0,040	0,011
Repeatability standard deviation (s_r)	0,001	0,004	0,001
Repeatability coefficient of variation	13,2 %	11,8 %	9,6 %
Repeatability limit (r) ($2,8 \times s_r$)	0,003	0,011	0,003
Reproducibility standard deviation of reproducibility (s_R)	0,005	0,018	0,005
Reproducibility coefficient of variation	64,3 %	44,9 %	47,4 %
Reproducibility limit (R) ($2,8 \times s_R$)	0,014	0,050	0,014

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