

# DRAFT INTERNATIONAL STANDARD

## ISO/DIS 17190-9

ISO/TC 173/SC 3

Secretariat: SIS

Voting begins on:  
2019-08-15

Voting terminates on:  
2019-11-07

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## Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

Part 9:

### Test method for gravimetric determination of flow rate and bulk density

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

*Partie 9: Détermination gravimétrique de la masse volumique*

ICS: 11.180.20

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Reference number  
ISO/DIS 17190-9:2019(E)

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Published in Switzerland

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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](http://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](http://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](http://www.iso.org/iso/foreword.html).

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

A list of all parts in the ISO ##### 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](http://www.iso.org/members.html).

# Urine-absorbing aids for incontinence — Polyacrylate superabsorbent powders —

## Part 9:

# Test method for gravimetric determination of flow rate and bulk density

## 1 Scope

This test method is used to determine the mass flow rate and bulk density (or apparent density) of polyacrylate superabsorbent powders.

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*

## 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

#### **Bulk density**

mass of unit volume of the powder after free fall, expressed in grams per milliliter

### 3.2

#### **flowability**

time for a mass of the powder to pass through a specified funnel, with the mass of the powder expressed in grams and the flowability in seconds

### 3.3

#### **flow rate**

mass of the powder flowing through a specified funnel per unit time, expressed in grams per second

### 3.4

#### **sample**

product or portion of a product taken from a production lot for testing purposes and identifiable and traceable back to its origin

### 3.5 specimen

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

## 4 Principle

The flowability of polyacrylate superabsorbent powders is determined by pouring a representative sample through a specified funnel and recording the time required for the entire sample to flow out of the funnel. The mass of the sample is divided by the time recorded to calculate the flow rate.

The bulk density of polyacrylate superabsorbent powders is determined by pouring a representative sample through a specified funnel into a density cup. The mass, expressed in grams, of the sample in the cup is divided by the volume, expressed in milliliters, of the density cup to calculate the bulk density of the polymer.

NOTE For the purpose of simplification of this test method, “flow rate” is used to mean “mass flow rate”.

## 5 Apparatus

**5.1 Density cup** (see [Figure A.1](#)), consisting of a stainless steel cylinder, smoothly finished inside (steel designation X5CrNiMo 17-12-3 specified in ISO/TR 15510), having the following characteristics:

- a) Capacity (100.0 ± 0.5) ml
- b) Internal diameter (45.0 ± 0.1) mm
- c) Internal height (h) (63.1 ± 0.1) mm

**5.2 Funnel**, with/without orifice damper, made of polished stainless steel (steel designation X5CrNiMo 17-12-3 specified in ISO/TR 15510, with an Rz value of preferably 1.6), having the following characteristics:

- a) Orifice internal diameter (10.00 ± 0.01) mm
- b) Inclination angle of cone 20°
- c) Height (145.0 ± 0.5) mm

**5.3 Camel hair brush or vacuum cleaner** for cleaning the funnel

**5.4 Flat metal blade**, e.g. spatula, paint or palette knife

**5.5 Analytical balance** capable of weighing, to the nearest 0.01 g, for test specimens with a mass of 100.00 g plus the mass of the density cup

**5.6 Utility tray**, 40 cm x 25 cm x 6 cm

**5.7 Beaker** with a capacity of 250 ml.

**5.8 Ring stand** capable of holding the funnel in the ring

**5.9 Timer or stopwatch**

## 6 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.

## 7 Sampling

**SAFETY WARNING** Power Handling – The German MAK has provided a guideline value for long-term exposure to the respirable portion of superabsorbent polyacrylate dust of  $0.05 \text{ mg}\cdot\text{m}^{-3}$ . The respirable portion is defined as those particles of less than  $10\text{-}\mu\text{m}$  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 MAK value.

**7.1** 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 1**— Sense of motion of the container

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

## 8 Procedure

**NOTE** The test must be performed on a level surface and the level of the apparatus must be checked with a bubble level device or equivalent.

**8.1** Place the funnel into the ring of the ring stand. Then place the ring stand holding the funnel into the utility tray with the density cup directly beneath the orifice of the funnel. Adjust the height of the funnel outlet to  $(40.0 \pm 2)$  mm above the top of the density cup.

**8.2** Weigh the density cup to the nearest 0.01 g and record the mass as  $W_1$ .

**8.3** Weigh  $100.00 \pm 0.01$  g test portion from the PA superabsorbent powder test sample into a 250 ml beaker.

**8.4** Close the orifice using either a flat blade or the damper located at the bottom of the funnel and pour the test portion into the funnel along the wall to avoid settling.

**8.5** Open the orifice by either removing the flat blade or by opening the damper and immediately start the timer.

**8.6** Stop the timer when the last portion of the test portion empties out of the funnel, and note the time ( $t$ ) in seconds.

**8.7** Gently move the density cup away from beneath the funnel and allow the remainder of the sample to spill into the utility tray.

**8.8** Using the flat blade, held perpendicular to the top of the cup, level off the sample flush with the top of the cup to remove excess polyacrylate superabsorbent powder. Tap the cup gently to settle the sample to avoid spilling during transfer to the balance.

Weigh the density cup containing the sample to the nearest 0.01 g and record the mass as W<sub>2</sub>.

**8.9** Carry out at least two determinations on the same well-mixed laboratory sample, in rapid succession by the same analyst.

**8.10** Clean the density cup and the funnel using the camel hair brush.

DO NOT tap the funnel during the course of the test.

If the polymer stops flowing, record the result as not flowing/flowable and repeat the test.

DO NOT use a spatula to clean the inside of the funnel.

Inspect the funnel and orifice on a regular basis for scratches. If scratched it will be necessary to replace the orifice and/or funnel.

## 9 Calculation

The flowability is the time (t) in seconds for the 100 g test portion to pass through the funnel. Calculate the flow rate (q), expressed in grams per second for each 100g test portion:

$$q = \frac{m_s}{t}$$

The bulk density,  $\rho$ , expressed in grams per milliliter, of the polyacrylate superabsorbent powder is calculated as follows:

$$\rho = \frac{W_2 - W_1 (g)}{100(ml)}$$

where

$m_s$  is the mass, expressed in grams, of the test portion;

$t$  is the time, expressed in seconds, for the test portion to pass through the funnel;

$W_1$  is the mass, expressed in grams, of the density cup;

$W_2$  is the mass, expressed in grams, of the density cup containing the test portion;

100 is the volume, expressed in milliliters, of the density cup.

Report results to the nearest 0.01 significant figure.

Calculate the average flow rate for the sample from the two test results obtained.

Calculate the average bulk density for the sample from the results obtained for the two test runs.



## 10 Report

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

- a) Reference the test method used
- b) Complete identification of all materials tested and method of sampling
- c) Name and address of testing institution
- d) The type of polymer-based absorbent materials, including all technical details and source information required for complete identification of the sample
- e) Whether or not lumps were present in the sample
- f) The results of the apparent density for each test portion, expressed in grams per milliliter (g.ml<sup>-1</sup>) to the nearest 0.01 g.ml<sup>-1</sup>, and the average for duplicate determinations
- g) The results of the flowrate for each test portion, expressed in grams per second (g.s<sup>-1</sup>) to the nearest 0.1 g.s<sup>-1</sup>, and the average for duplicate determinations
- h) Make and model of testing equipment
- i) Laboratory testing conditions
- j) Number of specimens tested
- k) For computer processed data, identify the software used and the version
- l) Deviation from the standard test procedure, if any
- m) When calculated, the standard deviation or the coefficient of variation
- n) Whether or not samples were conditioned prior to testing and, if so, for how long
- o) Any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met.

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 must be reported independently. Systems of measurement must not be combined in any way, but shall be regarded and reported separately.

## 11 Precision

Laboratory data was returned to EDANA and compiled and anonymised 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.

The method has been validated for flowrate over the range of 9.5 to 13.46 g/s and for density over the range of 0.66 to 0.72 g/ml. 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.