INTERNATIONAL STANDARD

ISO 19699-1

First edition
2017-08

Superabsorbent polymer — Sodium polyacrylate resin for absorbing blood —

Part 1: Test methods

Résines super-absorbantes — Polyacrylate de sodium pour l'absorption du sang —

Partie 1: Méthodes d'essai

Reference number
ISO 19699-1:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products.*

A list of all parts in the ISO 19699 series can be found on the ISO website.

Introduction

The test methods described in this document have been practically used by relevant production enterprises 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 testing superabsorbent polymer of sodium polyacrylate used in hygiene products (such as sanitary towels and pads) and medical products (such as tourniquets and surgery coats).

The International Organization for Standardization (ISO) draws attention to the fact that it is claimed that compliance with this document may involve the use of a patent concerning test methods of simulated blood absorption capacity and simulated blood absorption rate given in 4.8 and 4.9.

ISO takes no position concerning the evidence, validity and scope of this patent right.

The holder of this patent right has assured ISO that he/she is willing to negotiate licences under reasonable and non-discriminatory terms and conditions with applicants throughout the world. In this respect, the statement of the holder of this patent right is registered with ISO. Information may be obtained from:

Jinan Haoyue Resin Co., Ltd

Bucun District, Zhangqiu City, Shandong Province

China

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights other than those identified above. ISO shall not be held responsible for identifying any or all such patent rights.

Superabsorbent polymer — Sodium polyacrylate resin for absorbing blood —

Part 1: Test methods

1 Scope

This document specifies the testing methods for the properties of superabsorbent polymer (SAP) of sodium polyacrylate used in physical hygiene and medical products for absorbing blood. It also gives a formulation for simulated blood, a kind of viscous liquid, for replacing blood when testing the properties of the superabsorbent polymer.

The test methods and simulated blood in this document apply to sodium polyacrylate resin, as raw material, and apply to SAP for the final products used for absorbing blood.

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 758, *Liquid chemical products for industrial use — Determination of density at 20 °C*

ISO 2470-2, *Paper, board and pulps — Measurement of diffuse blue reflectance factor — Part 2: Outdoor daylight conditions (D65 brightness)*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6388, *Surface active agents — Determination of flow properties using a rotational viscometer*

ISO 17190-1, *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials — Part 1: Determination of pH*

ISO 17190-2, *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials — Part 2: Determination of amount of residual monomers*

ISO 17190-3, *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials — Part 3: Determination of particle size distribution by sieve fractionation*

ISO 17190-4, *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials — Part 4: Determination of moisture content by mass loss upon heating*

ISO 17190-9, *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials — Part 9: Gravimetric determination of density*

EN 14370, *Surface active agents — Determination of surface tension*

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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
superabsorbent polymer
SAP
polymer that can absorb and retain extremely large amount of liquid relative to its mass

Note 1 to entry: Absorption is dependent on liquid type.

Note 2 to entry: It is insoluble in water or organic solvents. However, after it has contacted with water, in a short time it swells into gel. Even an external pressure cannot force out the liquid.

3.2
amount of residual monomers
quantity of remaining sodium acrylate and acrylic acid in the sodium polyacrylate resin

Note 1 to entry: It is expressed in milligrams per kilogram (mg/kg).

3.3
volatile content
quantity of water and other small molecules in the sodium polyacrylate resin

Note 1 to entry: It is expressed as a percentage by mass (%).

3.4
particle size distribution
percentage of each of fraction of the particles based on mass

3.5
bulk density
mass of sodium polyacrylate resin per unit volume after free fall including voids

3.6
simulated blood
solution having a similar absorption trend as in human blood

3.7
simulated blood absorption capacity
amount of *simulated blood* (3.6) absorbed by 1 g of sodium polyacrylate resin after a set amount of time

3.8
simulated blood absorption rate
time taken for 1 g of sodium polyacrylate resin to absorb 5,0 ml of *simulated blood* (3.6)

4 Test methods

4.1 General

Use only reagents of recognized analytical grade and grade 3 water as specified in ISO 3696, unless otherwise specified.

4.2 Determination of amount of residual monomers

The amount of residual monomers shall be determined according to ISO 17190-2.

4.3 Determination of volatile content

The volatile content shall be determined according to ISO 17190-4.

4.4 Determination of pH

The pH shall be determined according to ISO 17190-1.

4.5 Determination of particle size distribution

The particle size distribution shall be determined according to ISO 17190-3.

4.6 Determination of bulk density

The bulk density shall be determined according to ISO 17190-9.

4.7 Determination of whiteness

In terms of physical appearance, sodium polyacrylate resin is white solid.

Whiteness shall be measured with a whiteness meter by using the test conditions specified in ISO 2470-2. Pour the specimen into a cylinder-shaped container made of clear glass and measure the tristimulus values using the reflection method. The specimen container shall be covered with a light trap.

4.8 Determination of simulated blood absorption capacity

4.8.1 General principle

The quantity of the simulated blood absorbed in a defined time is weighted.

4.8.2 Reagents

4.8.2.1 Simulated blood.

The solution of simulated blood shall be prepared in accordance with Annex A.

4.8.3 Apparatus

4.8.3.1 Analytical balance, capable of weighing up to 100 g, to the nearest 0,000 1 g.

4.8.3.2 Polyamide "tea bag", of 300 mesh with dimensions 100 mm × 150 mm and a basis weight of (58 ± 2) g/m^2.

4.8.3.3 Glass beaker, of 2 000 ml capacity.

4.8.3.4 Timer, accurate to 0,1 s over 60 min.

4.8.3.5 Drying rack or **line with clips**.

4.8.3.6 Thermometer, of 100 °C measuring range.

4.8.4 Sampling

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

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 thick). Take a sample of 500 g or more with a scoop and 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 taking a test portion to carry out the test. The preferred test conditions are specified in ISO 291[1]. If these conditions are not available, carry out the test at ambient conditions and report the temperature and relative humidity.

4.8.5 Procedure

4.8.5.1 Weigh (1,000 ± 0,005) g of sample to be tested using a spoon which accommodates to take 1,0 g sample by a weighing paper. Record the mass of sample as m_0. Place all sample in the bottom of the tea bag (4.8.3.2) and make sure that any sample attached to the inner sides of the tea bag is also put in the bottom of the tea bag. Heat seal about 3 mm to 5 mm from the open edges.

4.8.5.2 Fill the beaker (4.8.3.3) with 1 800 ml simulated blood (4.8.2.1), and put two sets of two tea bags (4.8.5.1) into the beaker, keeping the simulated blood surface 2 cm higher than the tea bags, and simultaneously start the timer.

4.8.5.3 After 30 min, remove the tea bags from the simulated blood in order, fold the tea bags about half from the left top corner, and hang them on the drying rack with clip according to the serial number, keeping about 45°.

4.8.5.4 Let the liquid drip for 10 min while the tea bags are maintained motionless. When multiple tea bags that contain sample are hung, they should not come into contact with each other. Weigh the tea bag containing the sample and record the mass as m_1.

4.8.5.5 Use the same procedure to prepare the tea bags that have been used above and do not contain the sample to test the blank value. Weigh the empty tea bag and record the mass as m_2.

4.8.6 Calculation and expression of results

The simulated blood absorption capacity is calculated according to Formula (1):

$$m = \frac{m_1 - m_2 - m_0}{m_0} \quad (1)$$

where

m is the capacity of absorbing simulated blood, expressed in grams per gram;

m_1 is the mass of the tea bag containing sample after absorbing simulated blood, expressed in grams;

m_2 is the mass of the empty tea bag, expressed in grams;

m_0 is the mass of sample, expressed in grams.

Two tests shall be conducted simultaneously and the arithmetic mean of these two measurements accurate to one decimal place shall be used as the result.

Annex B provides a comparison of human blood and simulated blood absorption capacity of SAP.

4.9 Determination of simulated blood absorption rate

4.9.1 General principle

The simulated blood absorption rate measurement is performed until the pre-set amount of simulated blood is absorbed. The time taken for 1 g of sodium polyacrylate resin to absorb 5,0 ml of simulated blood is measured.

4.9.2 Reagents

4.9.2.1 Simulated blood.

The solution of simulated blood shall be prepared according to Annex A.

4.9.3 Apparatus

4.9.3.1 Analytical balance, capable of weighing to the nearest 0,000 1 g.

4.9.3.2 Glass beaker, of 100 ml capacity.

4.9.3.3 Glass graduated cylinder, type A or B (laboratory glass ware) of 5 ml capacity (accurate to 0,1 ml).

4.9.3.4 Timer, accurate to 0,1 s over 60 min.

4.9.4 Sampling

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

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 thick). Take a sample of 500 g or more 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 taking a test portion to carry out the test. The preferred test conditions are (23 ± 1) °C and (50 ± 10) % relative humidity (ISO 291:2008, class II)[1].

4.9.5 Procedure

A minimum of two tests shall be carried out as follows.

4.9.5.1 Using the analytical balance (4.9.3.1), weigh (1,000 ± 0,005) g of sample to be tested to the nearest 0,001 g. Record the mass and place the sample into the beaker (4.9.3.2).

4.9.5.2 Shake or tap the beaker by hand so that the sample distributes in the bottom of the beaker evenly.

4.9.5.3 Use a glass graduated cylinder (4.9.3.3) to measure 5,0 ml of simulated blood (prepared according to 4.9.2.1) at (23 ± 1) °C. Pour the test solution into the centre portion of the glass beaker (4.9.5.2) and simultaneously start the timer.

4.9.5.4 Stop the timer and record the time, *t*, when the simulated blood solution is fully absorbed into the sample.

NOTE One way to check the full absorption is to allow the beaker to tilt slightly and observe if any solution remains or not.

4.9.6 Calculation

Calculate the arithmetic mean value from the simulated blood absorption rate measurements made, rounded to an integer, expressed in seconds.

The absorption rate is calculated as the time taken for 1,0 g of SAP to absorb 5,0 ml of simulated blood solution.

Annex A
(normative)

Preparation of simulated blood

A.1 Principle

Simulated blood is prepared to mimic the major physical properties of human blood. The solution prepared shall have similar liquidity and viscosity properties as well as the characteristics that of the human blood.

A.2 Formulation

Use only regents of recognized analytical grade, unless otherwise specified. The chemical formulation of simulated blood is as follows:

— deionized water (complying with ISO 3696, grade 3): 860,000 g ± 1,000 g

— sodium chloride: 10,000 g ± 0,010 g

— sodium carbonate: 40,000 g ± 0,040 g

— glycerin: 140,0 ml

— sodium benzoate: 1,000 g ± 0,001 g

— blue colouring: 0,050 ml

— sodium carboxymethylcellulose (molecular weight is 25 000): 5,000 g ± 0,005 g

— surface tension modifier (containing fluorine surfactant of non-ionic polymerization): 10,0 ml

A.3 Physical properties of simulated blood

The properties of simulated blood at (23 ± 1) °C shall satisfy the requirements given in Table A.1.

Table A.1 — Property requirements for simulated blood at (23 ± 1) °C

Property	Value	Relevant standard
Density	(1,05 ± 0,05) g/ml	ISO 758
Viscosity	(7,3 ± 1,1) mPa·s	ISO 6388
Surface tension	(40 ± 4) mN/m	EN 14370
pH	11,0 ± 0,1	ISO 6353-1

NOTE Information on properties of the human blood can be found in Reference [2].

A.4 Procedure

A.4.1 Sequentially weigh 10,00 g of sodium chloride, 40,00 g of sodium carbonate, 1,00 g of sodium benzoate and 5,00 g of sodium carboxymethylcellulose with an analytical balance, and place them together into a beaker of 2 000 ml.

A.4.2 Measure 140,0 ml of glycerin using a 250 ml graduated cylinder and pour them into the mixture in A.4.1. Measure 860 g of distilled water with the above analytical balance and pour them into the mixture in A.4.1, mixing well.

A.4.3 Take 300 ml using a 500 ml graduated cylinder of A.4.2 mixed liquid into the mixer, turn on the switcher mixer and simultaneously start the timer. After 7 min, stop stirring and pour out the mixture. Mix the remaining liquid using the above method.

A.4.4 Mix the mixture obtained in A.4.3, repeating the method described in A.4.3 again. Add 10,0 ml surface tension modifier and 0,05 ml of blue colouring to the mixture and stir until evenly coloured. Leave for 24 h before using.