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Urine-absorbing aids for incontinence — Measurement of airborne respirable polyacrylate superabsorbent materials — Determination of dust in collection cassettes by sodium atomic absorption spectrometry

Aides pour absorption d'urine — Dosage des particules respirables de matériaux superabsorbants de polyacrylate en suspension dans l'air — Dosage de la poussière dans des cassettes par spectrométrie à absorption atomique du sodium

Reference number
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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ISO 17191 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*.

Introduction

This International Standard was originally developed by European Disposables and Nonwovens Association (EDANA) as one of a twelve part series of test methods. It specifies a test method for determining the amount of airborne dust emanating from polyacrylate superabsorbent material. The other eleven parts differ from ISO 17191 in that they cover test methods for the characterization of polymer-based absorbent materials. They have been transformed into International Standards as a series of eleven parts of ISO 17190.

This test method has been in practical use for several years, and has proven to be reliable with respect to common criteria of quality of test methods (validity, repeatability, etc.). It is 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 — Measurement of airborne respirable polyacrylate superabsorbent materials — Determination of dust in collection cassettes by sodium atomic absorption spectrometry

1 Scope

This International Standard specifies a method for the determination of polyacrylate (PA) superabsorbent powders in airborne dust by measurement of sodium (Na) by atomic absorption spectrometry (AAS). PA dust samples are collected in polystyrene acrylonitrile air-monitoring cassettes with polytetrafluoroethylene filters and porous plastic backing pads.

This method is applicable to the determination of collected superabsorbent powder in the range between 0,2 µg and 60 µg (limit of detection near 0,2 µg) of superabsorbent powders.

NOTE This test is designed to determine low levels of sodium and requires that very clean handling conditions be observed. The use of deionized/distilled water containing very low sodium content is critical for successful analysis.

2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

3 Principle

Atomic absorption analysis is used to determine the sodium content in PA superabsorbent powders collected in air-monitoring cassettes. The sodium is determined after being released from the PA superabsorbent powder by exchange with potassium in a potassium chloride solution. The amount of dissolved sodium is then determined using AAS, and the mass of the PA superabsorbent powder collected is calculated after subtracting the background sodium levels present in blank cassettes.

To minimize sodium contamination, cassettes are prewashed before collecting the airborne superabsorbent powder. For the analysis, an aliquot of the potassium chloride solution is drawn from the cassette directly into the atomic absorption spray chamber.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified. Store all reagents and solutions in plastic containers with the exception of KCl (4.5) and IPA (4.3). Store these in the glass containers in which they are shipped.

Pre-analyse reagents and solutions as specified in 4.1, 4.2 and 4.3 for sodium (Na) using AAS, before using to clean cassettes or for Na analysis.

4.1 Potassium chloride (KCl), KCl content 99,999 % minimum; Na concentration less than 10 µg/g.

4.2 Water, deionized or distilled, in accordance with ISO 3696:1987, Grade 1, containing < 0,1 mg/l of Na.

4.3 **Isopropanol** (IPA), analytical reagent grade.

4.4 **Sodium standard reference solution**, $\rho(\text{Na})$ = 1 000 mg/l [1 000 ppm[1]].

4.5 **Potassium chloride solution**, $\rho(\text{KCl})$ = 2 000 mg/l.

Dissolve 2,0 g of potassium chloride (4.1) in 1 l of deionized water (4.2) in a polypropylene flask. The solution shall contain < 0,1 mg/l of Na.

4.6 **Sodium standard solutions,** for AAS as follows: $\rho(\text{Na})$ = 0,1 mg/l, 0,5 mg/l, 1,0 mg/l, 2,0 mg/l.

Using polypropylene volumetric flasks, dilute the 1 000 mg/l sodium standard reference solution (4.4) with the 2 000 mg/l potassium chloride solution (4.5) as required, and add isopropanol (4.3) to obtain a solution of 10 % by volume.

4.7 **Atomic absorption zeroing solution,**

Add isopropanol (4.3) to 2 000 mg/l potassium chloride solution (4.5) to obtain a solution of exactly 10,0 % isopropanol by volume.

5 Apparatus

5.1 **Atomic absorption spectrometer** (AAS).

Use a single-beam spectrometer with air-acetylene flame, or equivalent, capable of photometric accuracy of ± 0,0005 AU.

5.2 **Spraychamber** and **burner**: use a unit that is dedicated for trace-level sodium determination.

5.3 **Hollow cathode lamp for sodium.**

5.4 **Pipette**, capable of measuring 0,5 ml.

5.5 **Pipette,** capable of measuring 4,5 ml.

5.6 **Plastic gloves**, non-powdered.

5.7 **Volumetric flasks,** polypropylene, 1 000 ml and 100 ml capacity.

5.8 **Air-monitoring cassettes**[2], see Figure 1.

5.9 **Flatbed laboratory shaker**, capable of holding and shaking air-monitoring cassettes (5.8)

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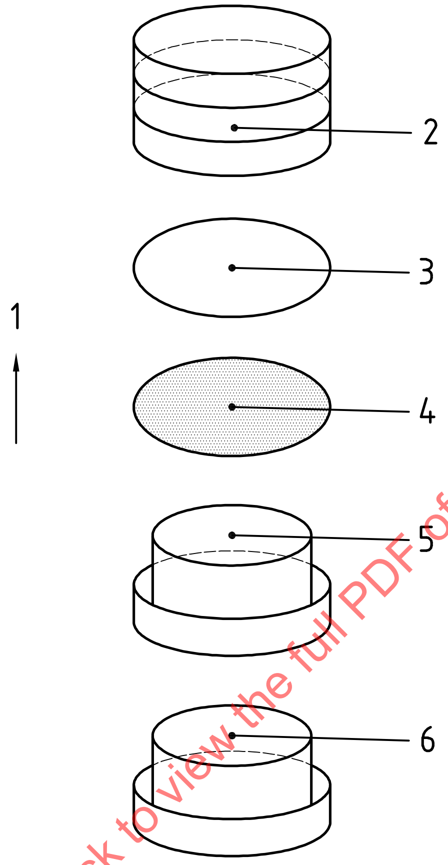
1) 1 mg/l = 1 ppm. The use of ppm is deprecated.

2) Commercially available air-monitoring cassettes with the following specifications have been shown to be suitable: three-piece clear polystyrene cassette for respirable dust cyclone sampling, as specified in NIOSH methods 7500 & 0600, 37 mm diameter, with porous high density polyethylene or PTFE support pad [e.g. cassette SKC catalogue part No. 225-3; support pad = SKC catalogue part No. 225-2902; filter = PTFE filter with PTFE support, 37 mm diameter, 2 µm thickness, typical air flow 25,3 l/min/cm^2 at 0,7 bar pressure drop, typically 99,99 % aerosol retention (e.g. Pall Gelman catalogue part No. P5pj037, Zefluor filter 37 mm, 2 µm)]. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of these products.

5.10 Air filter[3], for drying assembly manifold (see Figure 2), capable of retaining airborne particles and aerosols of > 0,3 µm diameter at a minimum flowrate of 5 l/min.



Key

1	air flow	4	filter
2	cassette outlet	5	cassette ring (middle)
3	support pad	6	cassette ring (bottom)

Figure 1 — Air-monitoring cassette

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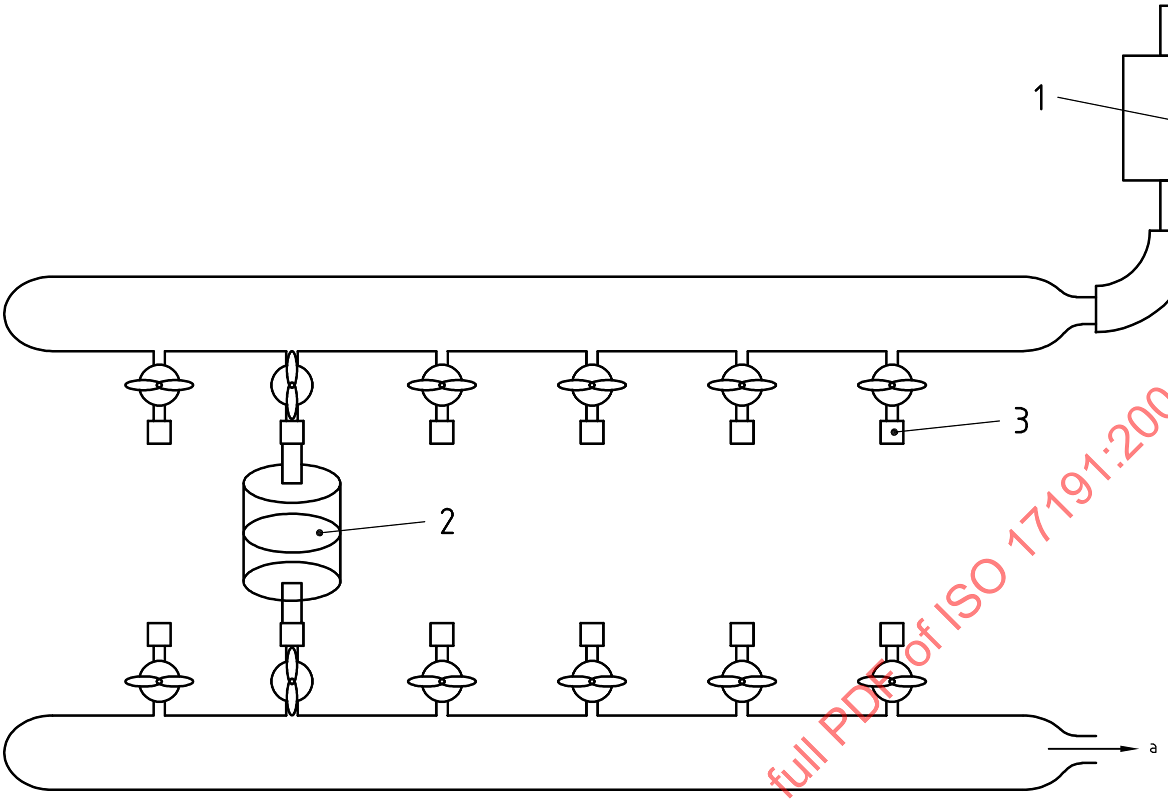
[3] Commercially available High Efficiency Particulate-free Air (HEPA) capsule with the following specification has been shown to be suitable: Pall Corporation, catalogue No. 12144. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.



Key

1 HEPA filter

2 cassette

3 Tygon tubing

[a] To vacuum.

Figure 2 — Cassette-drying unit

6 Procedure

6.1 Sample preparation and precautionary measures

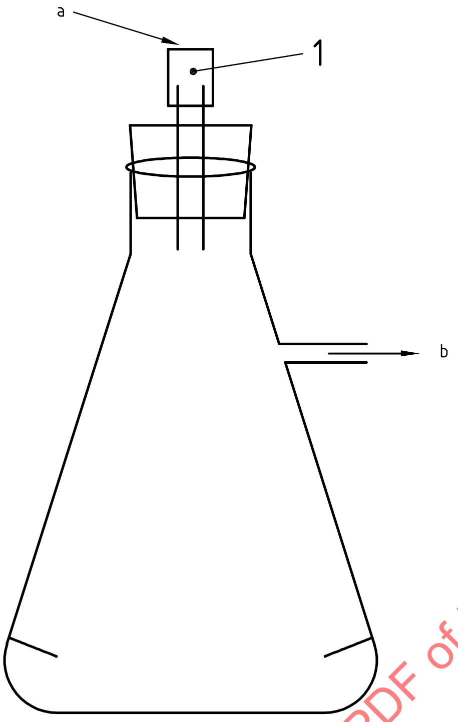
6.1.1 Preparation and cleaning of cassettes prior to air monitoring

6.1.1.1 In summary, cassettes are assembled, filters are wet with 0,5 ml IPA, washed with 0,2 % KCl solution, then rinsed with de-ionized water and dried before use as dust collection devices or blanks.

6.1.1.2 Assemble the desired number of cassettes without touching with the fingers any inside surface of the cassette. Analysts may wear plastic gloves to reduce contamination. Use forceps to handle filters and backing pads. If filters or backing pads are dropped, they should be discarded. Wash any hardware before re-use if dropped. Put only the bottom-end plug in the cassette, keeping the other plugs in the plastic bag. Tape the cassette over the circumference at the joint to assure a good seal.

6.1.1.3 Add 0,5 ml of IPA to the cassette to wet the filter and fill the cassette with 0,2 % KCl. Allow the KCl solution to stand in the cassette for at least 15 min, but not longer than 2 h.

6.1.1.4 Using vacuum suction (Figure 3), draw the solution through the cassette. Turn-off the vacuum just as the cassette empties. Do not pull air through the cassette after the solution has been drawn out. Fill the cassette with deionized water and pull through to rinse the cassette. Repeat this three more times.



Key

1 Tygon tubing

[a] Put cassette here.

[b] To vacuum.

Figure 3 — Vacuum washing stand

6.1.1.5 After the last rinse, connect the inlet of the cassette to an HEPA (High Efficiency Particulate-free Air) filter with retains particles and aerosols with diameters in excess of 0,3 µm. For efficiency, up to six cassettes may be fitted in parallel to the same HEPA filter (see Figure 2). Using vacuum suction, pull air through the cassette for drying. The required time will vary for each cassette, but experience has shown that all visible water is removed after 2 h if the air flow rate is 0,85 l/min for each cassette. Excess drying is not recommended in order to minimize contamination risk. The filter appearance will change to a bright white when dry, and all water droplets will be evaporated from the interior of the cassettes.

6.1.1.6 Fit the inlet and outlet plugs in the cassette with forceps; do not touch the plugs with the fingers. Tighten the plugs by turning and pressing by hand. Re-inspect the cassettes after 12 h for signs of moisture. If moisture is seen, dry for as short time as possible (as in 6.1.1.5) to remove the moisture. If a cleaned cassette is accidentally opened, it should be recleaned before use.

6.1.1.7 Before releasing cleaned cassettes for air sampling, each shall be clearly labelled with a unique identification so that its preparation date can be traced. In addition, 10 % of the cassettes shall be tested for sodium contamination. These cassettes shall be selected from the first, middle and last part of the batch. Cassettes are tested for contamination as if they were samples from air monitoring. If the test cassettes contain over 0,17 µg of sodium, then all cassettes shall be re-cleaned and again 10 % shall be tested before release.

Keep records of the absorbance values of the water used for rinsing, and the values from the sodium determination for the 10 % blank cassettes that were tested.

6.1.1.8 In addition to the cassettes that are tested for release, five of the remaining clean cassettes should be retained for blank determinations. These are for determining the limit of detection for the test at the time a series of cassettes from air monitoring are tested for PA dust level. If cassettes from a batch of clean cassettes are sent to more than one location, then five blanks shall be kept for each location involved. If any of these blank cassettes are not used during testing, they may be made part of another batch of cassettes and carried along through the cleaning procedure.

6.1.2 Preparation of cassettes for sodium analysis after air monitoring

6.1.2.1 Note that it is very important to use the same chemicals and solutions for preparing the calibration curve, and analysis of corresponding samples and blanks. If all the test samples cannot be processed with the same batch of solutions, run a new calibration curve with newly prepared solutions for the remaining samples and treat the results of these samples as if they are an independent series.

6.1.2.2 Take appropriate care to avoid contaminating the cassettes. Assume that fingers, work bench surfaces, the laboratory floor, etc. are contaminated with sodium. As little as 0,02 µg of sodium in the cassette will interfere with the analysis.

Rinse plastic gloves with deionized water after donning, and air dry. Wear plastic gloves as needed to make sure that inner cassette parts are not touched.

6.1.2.3 Remove the inlet plug from the cassette. Pipette 0,5 ml of IPA (4.3) onto the filter. Add 4,5 ml of 2 000 mg/l KCl (4.5) and reinstall the plug (using forceps).

6.1.2.4 Shake the cassette for 1 h on a flatbed shaker (5.9). Cassettes should be placed with the inlet side facing upwards. Keep the cassette plugged until ready for analysis.

6.2 Sodium determination

6.2.1 After ensuring that the set-up of the AAS instrument (5.1) is complete and flame stability is reached, using the zeroing solution (4.7), run the sodium standard solutions (4.6) to establish the calibration curve. Make sure each standard, as well as the zeroing solution, contains 2 000 mg/l KCl (4.5) and 10 % IPA (4.3) by volume.

6.2.2 Remove the cassette inlet plug and aspirate the sample and blank solutions directly from the cassette through the inlet hole. Record the absorbance values to an accuracy of 0,001 AU.

6.2.3 Occasionally, solid particles from the material that was collected on the filter may obstruct the aspiration tube. Therefore, check the absorbance reading of the zeroing solution each time after running five samples. At that time, also check one standard solution (1,0 mg/l Na). If the absorbance value for the standard solution has changed by more than 5 % relative to the initial value, this indicates that the tube is plugged and should be cleaned.

After cleaning, recheck the absorbance reading of the standard solution. A complete recalibration may be needed if the absorbance reading for the standard has changed significantly (more than 5 % relative) versus the initial value, after cleaning the tube. Then, remeasure the last five sample solutions.

6.2.4 Run a set of five blank cassettes (6.1.1.1) retained from the same batch of cassettes for every set of samples.

7 Calculation

7.1 Plot a calibration curve using a second-order regression fit between the absorbance values (ordinate) units and the sodium mass concentration (abscissa). This may be done by the instrument software (e.g. Perkin Elmer Atomic Absorption Benchtop[4] software). The response of the AAS to sodium is non-linear with respect to the mass concentration. Therefore, do not use a linear regression fit.

7.2 For each of the test samples, determine the mass concentration of sodium from the calibration curve (7.1). Convert these values to the apparent mass of PA superabsorbent powder, m_a, expressed in micrograms (µg), according to Equation (1) as follows:

$$m_a = \rho_a \text{ x } V_s \times F_{Na} \tag{1}$$

where

ρ_a is the apparent mass concentration, expressed as milligrams per litre (mg/l), of sodium extracted in the solution aspirated from the cassette as determined from the calibration curve;

V_s is the total volume of extraction solvents added to the cassette (= 5 ml);

F_{Na} is the conversion factor for Na to PA superabsorbent powder. It depends on the degree of neutralization of the superabsorbent and should be determined for the superabsorbent to be measured. It is usually 5,88.

7.3 From the blank values (obtained from the blank cassettes, see 6.1.1) that were calculated from the calibration curve for each set of samples, calculate the value of the mean blank, m_b. Use this value in the calculations to correct for background.

Calculate the standard deviation for the blank (σ).

Calculate the limit of detection (L_d) for the superabsorbent powder, according to Equation (2):

$$L_d = m_b + 3\sigma \tag{2}$$

7.4 Subtract the mean blank value m_b from each sample result to obtain the corrected level of superabsorbent powders in each sample. This value, m_s, expressed in micrograms (µg), is the corrected mass of superabsorbent powder found in each cassette and is calculated according to Equation (3) as follows:

$$m_s = m_a - m_b \tag{3}$$

where

m_a is the apparent mass, expressed in milligrams, of PA superabsorbent powder determined in the cassette using Equation (1);

m_b is the mean blank determined in 7.3.

7.5 Compare all corrected values (from 7.4), m_s, with the limit of detection for the test (7.3). Report the corrected value if greater than the limit of detection, but not those that are equal to or less than the limit of detection. Report this latter as ND (none detected) and give the limit of detection for the test. See examples.

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[4] Perkin Elmer Atomic Absorption Benchtop software is an example of a suitable product for performing this regression fit and is available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.