
Plastics — Ion exchange resin —

Part 2:

**Determination of water content of
anion exchange resins in hydroxide
form by centrifugation**

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Contents

Page

Foreword.....	iv
Introduction.....	v
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
4 Principle.....	1
5 Reagents.....	2
6 Apparatus.....	2
7 Samples.....	3
8 Procedure.....	3
8.1 Removal of the external water.....	3
8.2 Determination of water content.....	3
9 Calculation.....	4
10 Test Report.....	4
Annex A (normative) Sampling.....	5
Annex B (informative) Formulae and derivations.....	7

Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

A list of all parts in the ISO 4907 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.

Introduction

Anion exchange resins in hydroxide form have poor thermal stability, so direct drying at high temperatures should be avoided. This document solves this problem by form transition process and conversion.

Formulae and derivations are given in [Annex B](#).

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Plastics — Ion exchange resin —

Part 2:

Determination of water content of anion exchange resins in hydroxide form by centrifugation

1 Scope

This document specifies test methods by centrifugation of water content of styrene anion exchange resins in hydroxide form.

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

ISO 4907-3:2023, *Plastics — Ion exchange resin — Part 3: Determination of exchange capacity of anion exchange resins in hydroxide form*

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 <https://www.electropedia.org/>

3.1

hydroxide form styrene anion exchange resin

ionic type of styrene anion exchange resins regenerated by sodium hydroxide solution under the conditions specified in this document

Note 1 to entry: It is a general term that includes the strong-base groups existing in hydroxide form and the weak-base groups existing in free amine form.

3.2

water content

equilibrium water content in *hydroxide form styrene anion exchange resin* (3.1)

4 Principle

Remove the external water of the styrene anion exchange resin in hydroxide form absorbed enough water by centrifugation and convert it to chloride form by hydrochloric acid. Wash away the excess acid with anhydrous ethanol. The water content of the sample can be tested by the loss of mass on drying at 105 °C, as well as the increment of mass on the form conversion.

5 Reagents

WARNING — Reagents used in this document may can potential hazards to human health and the environment. Ensure that the instructions for the use of reagents are strictly followed.

Unless otherwise indicated, the reagents specified in this document should be analytical grade.

Commercially available, ready-made solutions may be used.

5.1 Anhydrous ethanol

5.2 Water, grade 2 in accordance with ISO 3696.

5.3 Hydrochloric acid solution, $c(\text{HCl}) = 1 \text{ mol/l}$.

Dilute 90 ml of hydrochloric acid (1,19 g/ml) to 1 000 ml with water.

5.4 Silver nitrate solution indicator.

Dissolve 5 g of silver nitrate in water and dilute to 100 ml. Store in an amber glass bottle.

5.5 High temperature resistant grease.

Dropping point shall be more than 200 °C.

6 Apparatus

WARNING — Apparatus used in this document may have potential hazards to human health and the environment. Ensure that the instructions for the use of apparatus are strictly followed.

Usual laboratory equipment and, in particular, the following should be used.

6.1 Water content analyser (see [Figure 1](#)), standard grinding mouth BM 19/26, No.1 micro porous (80 µm to 120 µm) sand core, $\phi = 3 \text{ mm}$.

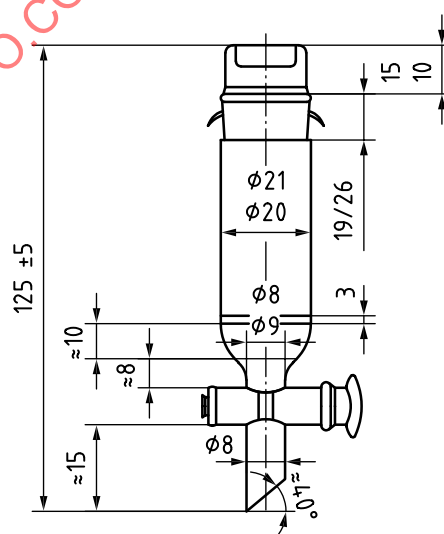


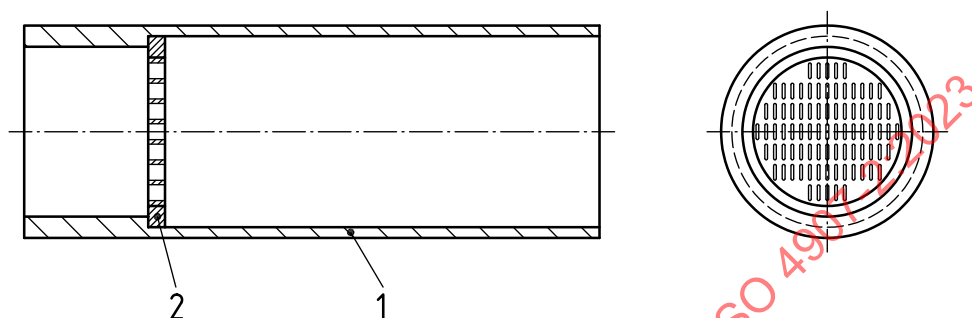
Figure 1 — Water content analyser

6.2 Separating funnel, standard grinding mouth BM 19/26.

6.3 Centrifuge, maximum adjustable speed: 5 000 r/min (523 rad/s), the accuracy is below ± 200 r/min (21 rad/s); maximum RCF: about $4\,400 \times g$; time adjustable range: higher than 5 min, the accuracy is ± 2 s; capacity: 4×100 ml.

6.4 Centrifugal filter tube (see [Figure 2](#)), internal diameter $\phi \geq 30$ mm.

6.5 Oven, maximum temperature is higher than $110\text{ }^{\circ}\text{C}$, and the accuracy of temperature control is better than $3\text{ }^{\circ}\text{C}$.



Key

- 1 centrifugal filter tube
- 2 filter plate

Figure 2 — Structure of centrifugal filter tube

7 Samples

Obtain a representative sample of the ion exchange resins in accordance with [Annex A](#).

Sample preparation shall be in accordance with ISO 4907-3:2023, 7.2.

8 Procedure

8.1 Removal of the external water

Transfer the pre-treated samples (see [Clause 7](#)) to the centrifugal filter tubes with water, and let them stand until no water runs out, and then place them into the centrifuge. Set the centrifuge speed of 2 500 r/min (262 rad/s) for 5 min, and start the centrifuge to remove external water.

After the centrifuge completely stops, take out the centrifugal filter tubes, wipe the bottom and external moisture of the filter tubes with filter paper, and then pour the sample into a pre-dried weighing bottle, cover tightly for later use.

NOTE Take care of the balance of centrifuge during working.

8.2 Determination of water content

Clean and dry water content analyser, take off the cock and coat with high temperature grease, preventing blocking the core hole. Open the cock and remove the plug, and put into the oven at $(105 \pm 3)\text{ }^{\circ}\text{C}$ until constant weight together. Transfer it to the desiccator and cool to room temperature, remaining the plug covered and the cock closed. Weigh the mass of the water content analyser (m_1) to the nearest 0,000 1 g.

Weigh 1 g of samples ([8.1](#)) into the water content analyser, and record the mass of the water content analyser and samples (m_2) to the nearest 0,000 1 g.

At the same time, weigh the samples for determining the total exchange capacity (E_{OH}) and the strong-base group capacity (E_A) in accordance with ISO 4907-3.

Add some water to the water content analyser containing samples to remove bubbles. Add 250 ml of hydrochloric acid solution to flow through the resins layer at a flow rate of 5 ml/min to 8 ml/min. Avoid hydrochloric acid solution contact the outer wall of water content analyser in this process. Wash the sample by anhydrous ethanol in the separating funnel at a flow rate of 3 ml/min to 5 ml/min, until the effluent is free of chloride ion as indicated by silver nitrate solution indicator. Extract the remaining ethanol after washing.

Dry the water content analyser containing the plug and cock open in the oven for 3 h at $(105 \pm 3) ^\circ\text{C}$. Cover the plug and close the lower cock in the oven, then take out to the desiccator and cool to room temperature. Record the mass of the water content analyser and samples (m_3) to the nearest 0,000 1 g.

9 Calculation

Calculate the water content according to [Formula \(1\)](#):

$$X = 100 \times \left\{ 1 - \left[\frac{m_3 - m_1}{m_2 - m_1} - \left(36,5 - 18 \times \frac{E_A}{E_{OH}} \right) \times E_{OH} \times 10^{-3} \right] \right\} \quad (1)$$

where

- X is the water content, expressed in percent (%), of anion exchange resins in hydroxide form;
- m_1 is the mass, expressed in grams (g), of the empty water content analyser;
- m_2 is the mass, expressed in grams (g), of the water content analyser and sample before dried;
- m_3 is the mass, expressed in grams (g), of the water content analyser and sample after dried;
- E_{OH} is the total exchange capacity, expressed in millimoles per gram (mmol/g) (wet), of anion exchange resin in hydroxide form in wet state;
- E_A is the strong-base group capacity, expressed in millimoles per gram (mmol/g) (wet), of anion exchange resins in hydroxide form in wet state.

10 Test Report

The test report shall include the following particulars:

- a) all information necessary to identify the test material;
- b) a reference to this document; i.e. ISO 4907-2:2023;
- c) the sampling method used;
- d) the test method used;
- e) the test results obtained or the final reference results obtained through repeatability test;
- f) all operating details not specified in this document, or regarded as optional, and details of any incidents which may influence the test results;
- g) any unusual features (anomalies) observed during the test;
- h) the date of the test.

Annex A (normative)

Sampling

A.1 Overview

This annex specifies sampling methods of the ion exchange resins. This annex applies to sample the ion exchange resins.

A.2 Apparatus

A.2.1 Sampler

A.2.1.1 Material, stainless steel or plastic.

A.2.1.2 Structure

Internal diameter 25 mm to 30 mm, width 18 mm to 20 mm. The opening length shall not be less than 80 % of the height of the ion exchange resins layer, as shown in [Figure A.1](#).

The total length of the sampler can be adjusted appropriately according to the height of the ion exchange resins layer in the container. Ensure that the opening section can reach the bottom of the container and the height is not less than 200 mm from the surface of the ion exchange resins layer to the rear end section.

NOTE The sampler can be segmented, which is convenient to sample different height of ion exchange resins layer.

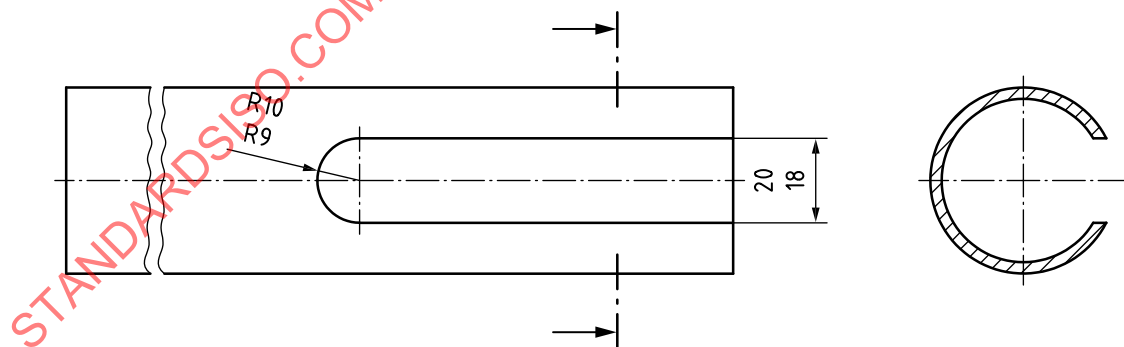


Figure A.1 — Sampler

A.2.2 Separator, clean and dry.

A.2.3 Container, clean and sealed.

A.3 Sampling rules

A.3.1 Sampling

When sampling from the production line, the unit of sampling shall be one kettle.

When samples are from the same batch and there are n packages, each package shall be sampled no less than 3 times when $n \leq 3$. When $n > 3$, random sampling method shall be adopted. The total number of samples shall be calculated according to the formula $\sqrt{n} + 1$ (in the case of decimals, integer shall be taken by rounding method), and each shall be sampled no less than twice.

Samples are taken from the top, middle and bottom for an individual upright packages.

A.3.2 Volume

More than 1 l.

A.4 Procedure

A.4.1 Sampling

Open the package and insert the sampler vertically down into the bottom of the ion exchange resins layer at different parts.

To prevent the ion exchange resins from sliding down, the sampler shall be facing upwards and tilting at an angle of 15° to 20° from the perpendicular before pulling out. Then pour the sample into the storage container.

A.4.2 Division

Divide the sample by quartering until the required volume is obtained.

A.5 Storage

Seal in the sample storage container and store in a cool place.

A.6 Identification

The sample container shall include the following particulars:

- a) name;
- b) batch No.;
- c) sampling spot;
- d) sampling date;
- e) signature of sampler.