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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

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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.

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(HCl) = 1 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, ϕ =3 mm.

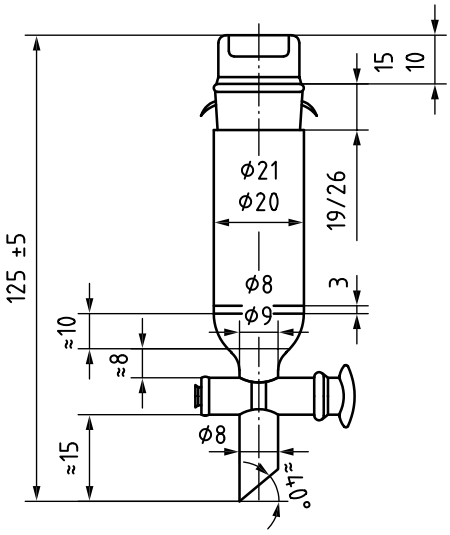


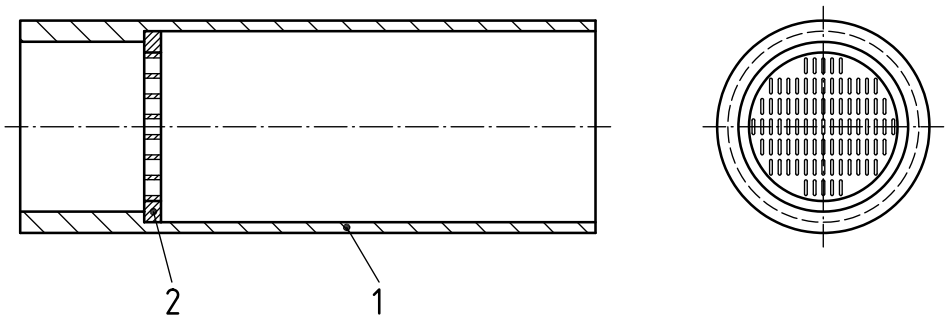
Figure 1 — Water content analyser

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 × 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 °C, and the accuracy of temperature control is better than 3 °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 ± 3) °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 ± 3) °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.