
Plastics — Ion exchange resin —

Part 3:

**Determination of exchange capacity
of anion exchange resins in hydroxide
form**

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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 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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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.

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

Exchange capacity often determines the performance. In practical use, the anion exchange resin is usually converted to hydroxide (or amine) form. What is more, from the change of strong-base and weak-base groups, the degree of pollution and degradation can be judged. This document specifies how to determine the exchange capacity of anion exchange resins in hydroxide (or amine) form.

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Plastics — Ion exchange resin —

Part 3:

Determination of exchange capacity of anion exchange resins in hydroxide form

1 Scope

This document specifies test methods of the total exchange capacity, the strong-base group capacity and the weak-base group capacity of the 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 4907-2:2023, *Plastics — Ion exchange resin — Part 2: Determination of water content 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

total exchange capacity

quantity of active groups in *hydroxide form styrene anion exchange resin* (3.1) that can exchange with strong acid under the conditions specified in this document

Note 1 to entry: It is expressed in millimoles per gram or in moles per litre of anion exchange resins.

3.3

strong-base group capacity

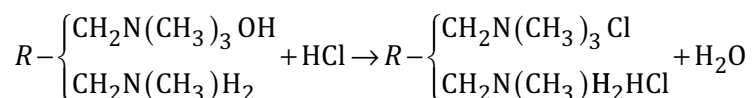
quantity of active groups in *hydroxide form styrene anion exchange resin* (3.1) that can exchange with neutral salts under the conditions specified in this document

Note 1 to entry: It is expressed in millimoles per gram or in moles per litre of anion exchange resins.

4 Principle

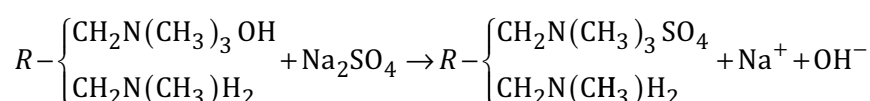
When styrene anion exchange resins in hydroxide form reacts with strong acid solution (such as hydrochloric acid solution), it can exchange active hydroxyls with other anions, and the substituted hydroxyls will neutralize with hydriions.

The reaction formula is:



When styrene anion exchange resins in hydroxide form reacts with neutral salt solution (such as sodium sulphate solution), it can exchange strong hydroxyls with other anions.

The reaction formula is:



5 Reagents

WARNING — Reagents used in this document may have potential hazards to human health and the environment. Ensure that the 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 Water, grade 2 in accordance with ISO 3696.

5.2 Sodium hydroxide, standard solution, $c(\text{NaOH}) \approx 0,10 \text{ mol/l}$.

Dissolve 4 g of sodium hydroxide to 1 000 ml with water. Standardize this solution at least weekly as follows.

5.2.1 Calibration

Dry 10 g of potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$, Guaranteed Reagent) at 105 °C to 110 °C for 4 h. And then cool to room temperature in a desiccator.

Weigh 0,75 g of potassium hydrogen phthalate (m_1) to the nearest 0,00 1 g, and dissolve with 100 ml water in a flask. Add 0,1 ml of phenolphthalein indicator solution. Titrate with 0,10 mol/l sodium hydroxide solution (5.2) until the pink colour appears and persists for 15 s. Record the consumption volume of alkali (V_1).

5.2.2 Blank determination

Pipet 100 ml of water. Carry out a blank determination according to the appropriate procedure 5.2.1. Record the consumption volume of alkali (V_2).

5.2.3 Calculation

See [Formula \(1\)](#):

$$c(\text{NaOH}) = \frac{1\,000 \times m_1}{204,220 \times (V_1 - V_2)} \quad (1)$$

where

- $c(\text{NaOH})$ is the actual concentration, expressed in moles per litre (mol/l), of the sodium hydroxid solution;
- m_1 is the mass, expressed in grams (g), of potassium hydrogen phthalate;
- V_1 is the titration consumption volume, expressed in millilitres (ml), of the sodium hydroxide solution;
- V_2 is the blank consumption volume, expressed in millilitres (ml), of the sodium hydroxide solution.

5.3 Hydrochloric acid, standard solution, $c(\text{HCl}) \approx 0,10$ mol/l.

Dilute 9 ml of hydrochloric acid (1,19 g/ml) to 1 000 ml with water. Calibrate this solution at least weekly as follows.

5.3.1 Calibration

Dry 5 g of sodium carbonate (Na_2CO_3 , Guaranteed Reagent) at 270 °C to 300 °C for 4 h. And then cool to room temperature in a desiccator.

Weigh 0,2 g of sodium carbonate (m_2) to the nearest 0,000 1 g, and dissolve with 100 ml water in a flask. Add 0,1 ml of bromocresol green-methyl red indicator solution. Titrate with 0,1 mol/l hydrochloric acid solution (5.3) until the greenish-blue colour disappears. Record the consumption volume of acid (V_3).

5.3.2 Blank determination

Pipet 100 ml of water, carry out a blank determination according to the appropriate procedure 5.3.1. Record the consumption volume of acid (V_4).

5.3.3 Calculation

See Formula (2):

$$c(\text{HCl}) = \frac{1\,000 \times m_2}{52,994 \times (V_3 - V_4)} \quad (2)$$

where

- $c(\text{HCl})$ is the actual concentration, expressed in moles per litre (mol/l), of the hydrochloric acid solution;
- m_2 is the mass, expressed in grams (g), of sodium carbonate;
- V_3 is the titration consumption volume, expressed in millilitres (ml), of hydrochloric acid solution;
- V_4 is the blank consumption volume, expressed in millilitres (ml), of hydrochloric acid solution.

5.4 Sodium hydroxide solution, $c(\text{NaOH}) = 2$ mol/l.

Dissolve 80 g of sodium hydroxide to 1 000 ml with water.

5.5 Sodium sulfate solution, $c(\text{Na}_2\text{SO}_4) = 0,5$ mol/l.

Dissolve 71 g of sodium sulfate to 1 000 ml with water.

5.6 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.

5.7 Bromocresol green-methyl red indicator solution.

Dissolve 0,2 g of bromocresol green and 0,015 g of methyl red in 100 ml of ethanol [$\geq 95 \%$ (V/V) ethanol]. Store in an amber glass bottle.

5.8 Phenolphthalein indicator solution.

Dissolve 1,0 g of phenolphthalein in ethanol [$\geq 90 \%$ (V/V) ethanol] and dilute to 100 ml.

5.9 Methyl red-methylene blue mixed indicator solution.

Dissolve 0,1 g of methyl red and 0,1 g of methylene blue in 100 ml of ethanol [$\geq 90 \%$ (V/V) ethanol], respectively. Mix the above two solutions in equal volumes. Store in an amber glass bottle.

5.10 Sodium chloride solution, $c(\text{NaCl}) = 10 \%$.

Dissolve 100 g of sodium chloride to 1 000 ml with water.

NOTE According to titration (GM 31.2) in ISO 6353-1, automatic potentiometric apparatus can also be used as an alternative. Select equivalence point of the acid-base titration by pH electrode as the endpoints, or equivalence point of the chloridion content titration by silver electrode as the endpoints.

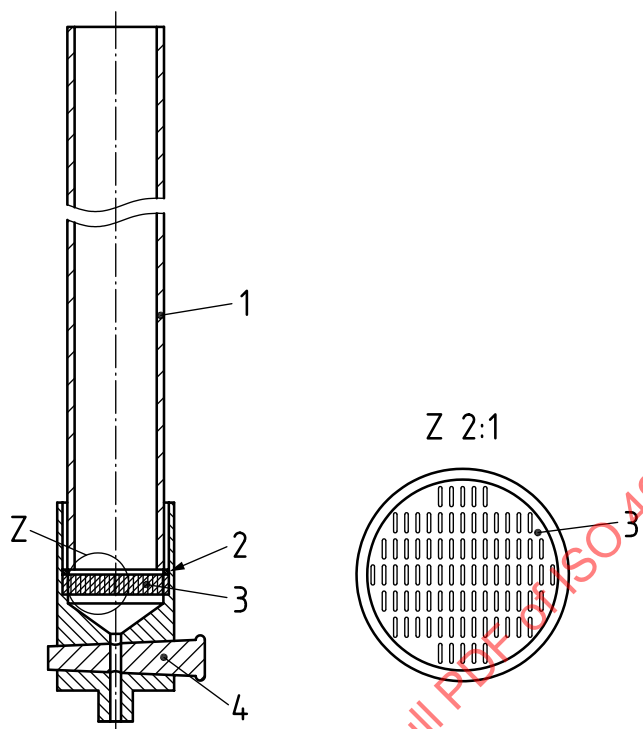
6 Apparatus

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 Exchange column (see [Figure 1](#)), internal diameter $\geq 20 \text{ mm}$.

6.2 Water bath, the accuracy of temperature control is better than 2 °C.



Key

1 exchange column

2 rubber washer

Z partial zoom, the ratio is 2:1

3 filter plate

4 cock plug

Figure 1 — Structure of exchange column

7 Samples

7.1 Sampling

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

7.2 Sample preparation

Transfer 50 ml of samples to the plexiglass exchange column ([6.1](#)), then backwash with water, where the expansion rate of the sample is 50 % to 100 %, until the effluent is clear, and no impurities are visible. The resins are allowed to settle freely and drain until the liquid level is about 20 mm higher than the resin.

Treat the resin according to the requirements in [Table 1](#).

Contain 20 mm layer of liquid above the resins bed and ensure no bubbles throughout the process. Maintain the temperature of the water and all pretreatment solutions at $(25 \pm 10) ^\circ\text{C}$.

Transfer and store the treated sample into a clean wide-mouth bottle for use.

Table 1 — Treatment of anion exchange resins in hydroxide form

Solution	Volume ml	Time min
HCl (5.6) or NaCl (5.10)	400	up to 30
Water	400	20 to 30
NaOH (5.4)	750	up to 60
Water	Maintain a flow rate of 7 ml/min and wash the resins until 5 ml of the effluent remains colourless with the addition of the phenolphthalein indicator.	

8 Procedure

8.1 General

Remove the external water of the samples in accordance ISO 4907-2:2023, 8.1.

The water content of the samples is determined according to ISO 4907-2.

8.2 Total exchange capacity

Weigh 2,0 g of the anion exchange resins samples with external water removed (8.1) into a dry plugged bottle. Record the mass of sample (m_3) to the nearest 0,000 1 g.

Pipet 100 ml of 0,1 mol/l hydrochloric acid solution (5.3) into the bottle, stopper tightly and shake well, put it into 40 °C water bath (6.2) for no less than 2 h, then take out and cool to room temperature.

Pipet 25 ml of soaking solution into a flask, then add 50 ml of water. Add 0,1 ml of phenolphthalein indicator solution. Titrate with 0,1 mol/l sodium hydroxide solution (5.2) until the pink colour appears and persists for 15 s. Record the volume of alkali consumed (V_5).

8.3 Strong-base group capacity

Weigh 2,0 g of the strong-base anion exchange resins samples with external water removed (8.1) or 10,0 g of the weak-base anion exchange resins samples with external water removed (8.1) into a dry plugged bottle. Record the mass of sample (m_4) to the nearest 0,000 1 g.

Pipet 100 ml of sodium sulfate solution (5.5) into the dry plugged bottle, stopper tightly and shake well. Set at room temperature over 1 h.

Pipet 25 ml of soaking solution into a flask, and then add 50 ml of water. Add 0,1 ml of methyl red-methylene blue mixed indicator solution. Titrate with 0,1 mol/l hydrochloric acid solution (5.3) until the purple colour appears and persists for 15 s. Record the volume of hydrochloric acid consumed (V_6).

NOTE 1 Avoid resins particles being sucked out when pipetting soaking solution.

NOTE 2 According to titration (GM 31.2) in ISO 6353-1, automatic potentiometric apparatus can also be used as an alternative. Select equivalence point of the acid-base titration by pH electrode as the endpoints, or equivalence point of the chloridion content titration by silver electrode as the endpoints.

9 Calculation

9.1 Total exchange capacity

Calculate the total exchange capacity according to [Formulae \(3\), \(4\) and \(5\)](#):

$$E_{OH} = \frac{100 \times c(\text{HCl}) - 4 \times c(\text{NaOH}) \times V_5}{m_3} \quad (3)$$

$$Q_1 = \frac{E_{OH}}{1-X} \quad (4)$$

$$Q_{V1} = E_{OH} \times d_b \quad (5)$$

where

E_{OH}	is the total exchange capacity, expressed in millimoles per gram (mmol/g) (wet), of anion exchange resins in hydroxide form in wet state;
Q_1	is the total mass exchange capacity, expressed in millimoles per gram (mmol/g) (dry), of anion exchange resins in hydroxide form;
$c(\text{HCl})$	is the actual concentration, expressed in moles per litre (mol/l), of the nominally 0,1 mol/l hydrochloric acid;
$c(\text{NaOH})$	is the actual concentration, expressed in moles per litre (mol/l), of the nominally 0,1 mol/l sodium hydroxide solution;
V_5	is the volume, expressed in millilitres (ml), of sodium hydroxide solution consumed in procedure 8.1 ;
m_3	is the mass, expressed in grams (g), of the sample in procedure 8.1 ;
X	is the water content, expressed in percent (%), of the sample in hydroxide form;
Q_{V1}	is the total volume exchange capacity, expressed in moles per liter (mol/l), of resins sample;
d_b	is the wet apparent density, expressed in gram per millilitres (g/ml), of resins sample in compacted and wet state.

9.2 Strong-base group capacity

Calculate the strong-base group capacity according to [Formulae \(6\), \(7\) and \(8\)](#):

$$E_A = \frac{4 \times c(\text{HCl}) \times V_6}{m_4} \quad (6)$$

$$Q_2 = \frac{E_A}{1-X} \quad (7)$$

$$Q_{V2} = E_A \times d_b \quad (8)$$

where

- 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;
- Q_2 is the strong-base group mass capacity, expressed in millimoles per gram (mmol/g) (dry), of anion exchange resins in hydroxide form;
- $c(\text{HCl})$ is the actual concentration, expressed in moles per litre (mol/l), of the nominally 0,1 mol/l hydrochloric acid;
- V_6 is the volume, expressed in millilitres (ml), of hydrochloric acid consumed in procedure [8.2](#);
- m_4 is the mass, expressed in grams (g), of the sample in procedure [8.2](#);
- X is the water content, expressed in percent (%), of the sample in hydroxide form;
- Q_{V2} is the strong-base group volume exchange capacity, expressed in moles per liter (mol/l), of resins sample;
- d_b is the wet apparent density, expressed in gram per millilitres (g/ml), of resins sample in compacted and wet state.

9.3 Weak-base group capacity

Calculate the weak-base group capacity according to [Formula \(9\)](#):

$$Q_3 = Q_1 - Q_2 \quad (9)$$

where Q_3 is the weak-base group capacity, expressed in millimoles per gram (mmol/g) (dry), of anion exchange resins in hydroxide form.

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 4907-3:2023;
- 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.