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Plastics — Ion exchange resin — Part 1: Determination of exchange capacity of acrylic anion exchange resins

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Foreword

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A list of all parts in the ISO 4907 series can be found on the ISO website.

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Introduction

Following the traditional chemical processes such as distillation and extraction, ion exchange and adsorption technology has also become a typical chemical separation technology, which plays an important role in efficient extraction, concentration and refining. Since the realization of organic synthesis, ion exchange resin has become one of the key materials for exchange and adsorption. At present, they have been widely used in water treatment, environmental protection, petrochemical industry, food and medicine, hydrometallurgy and energy industry, almost involving the core content of the United Nations Sustainable Development Goals (SDGs).

Ion exchange resin is a kind of high polymer organic copolymer, which is composed of insoluble threedimensional space network framework, functional groups connected to the framework and exchangeable ions with opposite charges. The main features determined by the structure are exchangeable, selective, adsorbable and catalytic. However, even the same resin has different properties in different forms, such as exchange capacity and water content, so a unified method is needed to provide basis for manufacturing, quality supervision, technical exchange, factory inspection and arbitration.

Because of the special structure, acrylic anion exchange resins contain not only strong-base and weakbase groups, but also weak-acid groups, and the content of weak-acid groups directly affects the using effect. This document specifies how to determine the exchange capacity of acrylic anion exchange resins.

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Plastics — Ion exchange resin — Part 1: Determination of exchange capacity of acrylic anion exchange resins

1 Scope

This document specifies test methods of the strong-base group capacity, the weak-base group capacity and the weak-acid group capacity of acrylic anion exchange resins.

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

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

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3.1

standard form acrylic anion exchange resin

ionic type of the samples treated under the pretreatment and transformation conditions specified in this document

3.2

strong-base group capacity

quantity of active groups in *standard form acrylic 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 of per gram or moles per literlitre (mol/l) of acrylic anion exchange resins.

3.3

weak-base group capacity

quantity of active groups in *standard form acrylic anion exchange resin* (3.1) that can exchange with acids under the conditions specified in this document

Note 1 to entry: It is expressed in millimoles of per gram or moles per literlitre (mol/l) of acrylic anion exchange resins.

3.4

weak-acid group capacity

quantity of active groups in *standard form acrylic anion exchange resins* (3.1) that can exchange with alkali under the conditions specified in this document

Note 1 to entry: It is expressed in millimoles of per gram or in moles per literlitre (mol/l) of acrylic anion exchange resins.

4 Principle

According to the definitions, strong-base group in acrylic anion exchange resins has the ability to split neutral salts, while weak-base groups and weak-acid groups have not.

Calculate the strong-base group capacity by the exchanged chloride ion contents in titration, when the standard form acrylic anion exchange resins react with neutral salts (such as sodium nitrate solution).

The reaction formula is:

 $R = \begin{cases} CH_2N(CH_3)_3 Cl \\ CH_2N(CH_3)H_2 + NaNO_3 \rightarrow R \\ CH_2COOH \end{cases} \begin{cases} CH_2N(CH_3)H_2 + NaCl \\ CH_2COOH \\ cH_2C$

Calculate the weak-base groups capacity by the unreacted acid contents in titration, when the standard form acrylic anion exchange resins react with excessive monobasic acid solution (such as hydrochloric acid).

The reaction formula is:

$$R = \begin{cases} CH_2N(CH_3)_3 Cl & CH_2N(CH_3)_3 Cl & CH_2N(CH_3)_3 Cl & 4907-1 \\ CH_2N(CH_3)H_2 + HCl \rightarrow R - \begin{cases} CH_2N(CH_3)H_2 + HCl \rightarrow R - \\ CH_2COOH & CH_2COOH \end{cases} CH_2N(CH_3)_3 Cl & CH_2N(CH_3)_3 Cl \\ CH_2N(CH_3)H_2 + HCl \rightarrow R - \begin{cases} CH_2N(CH_3)_3 Cl & CH_2N(CH_3)_3 Cl \\ CH_2N(CH_3)H_2 + HCl \rightarrow R - \\ CH_2COOH & CH_2COOH & CH_2N(CH_3)H_2HCl \\ CH_2COOH & CH_2COOH & CH_2COOH & CH_2N(CH_3)H_2HCl \\ CH_2COOH & CH_2N(CH_3)H_2 + HCl \rightarrow R - \begin{cases} CH_2N(CH_3)H_2 + HCl \rightarrow R - \\ CH_2N(CH_3)H_2 + HCl \rightarrow R - \\ CH_2COOH & CH_2COOH & CH_2N(CH_3)H_2HCL \\ CH_2COOH & CH_2COOH & CH_2COOH & CH_2N(CH_3)H_2HCL \\ CH_2COOH & CH_2COOH &$$

Calculate the weak-acid group capacity by the unreacted alkali and the exchanged chloride ion contents in titration, when the standard form acrylic anion exchange resins react with excessive monobasic solution (such as sodium hydroxide).

The reaction formula is:

 $\begin{array}{c} \mathcal{R} = \begin{cases} \mathsf{CH}_2\mathsf{N}(\mathsf{CH}_3)_3 \, \mathsf{Cl} \\ \mathsf{CH}_2\mathsf{N}(\mathsf{CH}_3)\mathsf{H}_2 + \mathsf{NaOH} \rightarrow \mathcal{R} = \begin{cases} \mathsf{CH}_2\mathsf{N}(\mathsf{CH}_3)_3 \, \mathsf{OH} \\ \mathsf{CH}_2\mathsf{N}(\mathsf{CH}_3)\mathsf{H}_2 &+ \mathsf{H}_2\mathsf{O} + \mathsf{NaCH} \\ \mathsf{CH}_2\mathsf{COOH} \\ \end{cases} \end{cases}$

$$R - \begin{cases} CH_2N(CH_3)_3 Cl \\ CH_2N(CH_3)H_2 + NaOH \rightarrow R - \\ CH_2COOH \end{cases} \begin{pmatrix} CH_2N(CH_3)_3 OH \\ CH_2N(CH_3)H_2 + H_2O + NaCl \\ CH_2COONa \end{pmatrix}$$

5 Reagents

WARNING — Reagents used in this document <u>maycan</u> have potential hazards to human health and the environment. <u>Please strictly followEnsure that</u> the instructions for the use of reagents <u>are strictly followed</u>.

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, $c(NaOH) \approx 0,10 \text{ mol/l}$.

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

5.2.1 Calibration

Dry 10 g of potassium hydrogen phthalate ($KHC_8H_4O_4$, 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 0,001 g, and dissolve with 100 ml of 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 procedure 5.2.1. Record the consumption volume of alkali (V_2).

5.2.3 Calculation

See Formula (1):

$$\frac{c(\text{NaOH}) = \frac{1000 \times m_1}{204,220 \times (V_1 - V_2)} c(\text{NaOH}) = \frac{1000 \times m_1}{204,220 \times (V_1 - V_2)}$$
(1)

where

- c(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, $c(HCl) \approx 0,10 \text{ mol/l}$.

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 (Na₂CO₃, 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 0,001 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):

$$\frac{c(\text{HCl}) = \frac{1000 \times m_2}{52,994 \times (V_3 - V_4)} c(\text{HCl}) = \frac{1000 \times m_2}{52,994 \times (V_3 - V_4)}$$
(2)

where

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- c(HCl) is the actual concentration, expressed in moles per litre (mol/l), of the hydrochloric acid solution; $\underline{\text{ISO/FDIS } 4907-1}$
- m_2 is the mass, expressed in grams (g), expressed of sodium carbonate; 8d-b714
- 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 Silver nitrate, standard solution, $c(\text{AgNO}_3) \approx 0,10 \text{ mol/l}$.

Dissolve 17,5 g of silver nitrate to 1 000 ml with water. Store in an amber glass bottle. Calibrate this solution at least weekly as follows.

5.4.1 Calibration

Dry 5 g of sodium chloride (NaCl, Guaranteed Reagent) at 500 $^\circ$ C for 10 min. And then cool to room temperature in a desiccator.

Weigh 1,649 g of sodium chloride (m_3) to the nearest 0,001 g, and dissolve to 1 000 ml with water.

Pipet 10 ml of sodium chloride solution in a flask, and add 90 ml water and 1 ml of 10 % potassium chromate indicator. Titrate with 0,1 mol/l silver nitrate standard solution (5.4) until the colorcolour changes to brick-red and persists for 15 s. Record the consumption volume of the silver nitrate standard solution (V_5).

5.4.2 Blank determination

Pipet 100 ml of water. Carry out a blank determination according to the appropriate procedure 5.4.1 and record the consumption volume of the silver nitrate standard solution (V_6).

5.4.3 Calculation

See Formula (3):

$$\frac{c(\text{AgNO}_3) = \frac{1000 \times m_3}{58,442 \times (V_5 - V_6)} c(\text{AgNO}_3) = \frac{1000 \times m_3}{58,442 \times (V_5 - V_6)}$$
(3)

where

$c(AgNO_3)$	is the actual concentration, expressed in moles per litre (mol/l), of the silver nitrate standard solution;
m_3	is the mass, expressed in grams (g), of sodium chloride;
V_5	is the titration consumption volume, expressed in millilitres (ml), of the silver nitrate standard solution;
V_6	is the blank consumption volume, expressed in millilitres (ml), of the silver nitrate standard solution.

5.5 Sodium nitrate solution, *c*(NaNO₃) = 1 mol/l.

Dissolve 85 g of sodium nitrate to 1 000 ml with water.

5.6 Sodium hydroxide solution, *c*(NaOH) = 1 mol/l.

Dissolve 40 g of sodium hydroxide to 1 000 ml with water.

5.7 Hydrochloric acid solution, *c*(HCl) = 1 mol/l.4007-1

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

5.8 Ammonia-ammonium chloride solution ($pH = 9,25 \pm 0,15$).

Dissolve 53 g of ammonium chloride and 67 ml of ammonia to 1 000 ml with water, adjust the solution to $pH = 9,25 \pm 0,15$.

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 [a volume fraction of \geq 90 % ethanol], respectively. Mix the above solutions in equal volumes. Store in an amber glass bottle.

5.10 Potassium chromate indicator.

Dissolve 10 g of potassium chromate to 100 ml with water. Store in an amber glass bottle.

5.11 Nitric acid (1 + 9, volume fraction).

Mix 100 ml of nitric acid (1,42 g/ml, Guaranteed Reagent) and 900 ml of water together.

5.12 Silver nitrate solution indicator.

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

5.13 Phenolphthalein indicator.

Dissolve 1,0 g of phenolphthalein in ethanol [a volume fraction of ≥90 % ethanol] and dilute to 100 ml.