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Diffusion dialysis anion exchange membrane

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Foreword

SAC/TC382 (National Technical Committee on Separation Membrane of SAC) is in charge of this English translation. In case of any doubt about the contents of English translation, the Chinese original shall be considered authoritative.

This document is drafted in accordance with the rules given in the GB/T 1.1-2009 *Directives for standardization - Part 1: Structure and drafting of standards*.

This document was proposed by China Petroleum and Chemical Industry Federation.

This document was prepared by SAC/TC382.

Diffusion dialysis anion exchange membrane

1 Scope

This document specifies the terms and definitions, model and naming, requirements, test methods, inspection rules, sign, packaging, transport and storage of anion exchange membrane for diffusion dialysis.

This document is applicable to anion exchange membrane for diffusion dialysis.

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.

GB/T 191 *Packaging - Pictorial marking for handling of goods*

GB/T 601-2002 *Chemical reagent - Preparations of standard volumetric solutions*

GB/T 603-2002 *Chemical reagent - Preparations of reagent solutions for use in test methods*

GB/T 4456 *Polyethylene blown film for packaging*

GB/T 6682 *Water for analytical laboratory use - Specification and test methods*

GB/T 9174 *General specification for transport packages of general cargo*

GB/T 9969 *General principles for preparation of instructions for use of industrial products*

GB/T 12464 *Wooden boxes*

GB/T 14436 *General principles of industrial product guarantee documents*

GB/T 20103-2006 *Technical terms for membrane separation*

HY/T 166.1-2013 *Ion-exchange membrane - Part 1: Electro-driven membrane*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in GB/T 20103-2006 and the following apply. For ease of use, the related terms and definitions of GB/T 20103-2006 are listed below.

3.1

membrane

thin barrier with certain physical or chemical properties on the surface, which forms a discontinuous interval between two adjacent fluid phases and affects the permeation rate of components in the fluid

[Source: GB/T 20103-2006, 2.1.1]

3.2

ion exchange membrane

polymeric membrane selectively permeable to ions

[Source: rewrite GB/T 20103-2006, 3.1.1]

3.3

anion exchange membrane

ion exchange membrane with positively charged fixed groups, which is selectively permeable to anions, referred to as “anion membrane”

[Source: rewrite GB/T 20103-2006, 3.1.3]

3.4

co-ion

ion with like charge as the fixed groups in ion exchange membrane

3.5

counter-ion

ion with opposite charge to the fixed groups in ion exchange membrane

3.6

diffusion dialysis process

process of selective dialysis driven by concentration gradient, in which counter-ions in solution pass through ion exchange membrane and drive co-ions with smaller size and lower valence to pass preferentially

3.7

anion exchange membrane diffusion dialysis process

process of selective dialysis driven by concentration gradient, in which anions in solution pass through anion exchange membrane and drive cations with smaller size and lower valence to pass preferentially

3.8

diffusion dialysis anion exchange membrane, DDAM (abbreviation)

anion exchange membrane which can be used for diffusion dialysis process

3.9

dialysate

feed liquid to be separated in a diffusion dialysis process

3.10

diffusate

liquid receiving diffused components in a diffusion dialysis process

3.11

proton dialysis coefficient

amount of substance of proton passing through a membrane per unit area per unit time per unit concentration gradient, unit: m/s

3.12

metal ion leakage coefficient

amount of substance of the metal ion passing through a member per unit area per unit time per unit concentration gradient, unit: m/s

3.13

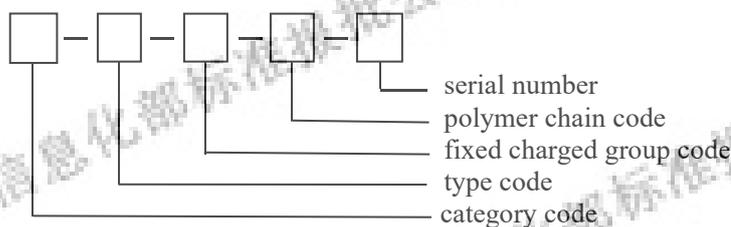
acid/salt separation factor

ratio of proton dialysis coefficient to metal ion leakage coefficient

4 Model and naming

4.1 Model composition

The model of diffusion dialysis anion exchange membrane consists of five parts: category code, type code, fixed charged group code, polymer chain code and serial number. All parts are connected with a hyphen "-" as shown below:



4.2 Category code

The category code is represented by DDAM (the abbreviation of diffusion dialysis anion exchange membrane).

4.3 Type code

The type code indicates the sequence of functionalization reaction and membrane forming. The one with

subsequent functionalization reaction is type "I", and the other with antecedent functionalization reaction is type "II".

4.4 Fixed charged group code

The fixed charged group code indicated by Arabic numerals is shown in Table 1.

Table 1 The fixed charged group code

Code	Fixed charged group
1	Strongly alkaline fixed group with positive charge
2	Weakly alkaline fixed group with positive charge
3	Strongly alkaline fixed group with positive charge and auxiliary hydroxyl group or other groups
4	Weakly alkaline fixed group with positive charge and auxiliary hydroxyl group or other groups
5	Alternate positively charged fixed group and negatively charged fixed group
6	Others

4.5 Polymer chain code

The polymer chain code indicated by Arabic numerals is shown in Table 2.

Table 2 The polymer chain code

Code	Polymer chain
1	Polyphenylene ether
2	Polyvinyl alcohol
3	Organic-inorganic hybrid
4	Others

4.6 Serial number

The serial number indicates the serial products of the same polymer chain and fixed charged group, expressed in Arabic numerals.

4.7 Example of membrane model and naming

The model and naming example of diffusion dialysis anion exchange membrane is as follows:

Example: DDAM-I-1-1-3

DDAM means diffusion dialysis anion exchange membrane; I means subsequent functionalization reaction; the first 1 means that the membrane contains strongly alkaline fixed groups with positive charge; the latter 1 means that the polymer chain of the membrane is polyphenylene ether; 3 means the third product in the same series.

5 Requirements

5.1 Appearance

The appearance of a diffusion dialysis anion exchange membrane shall be smooth and clean, no mechanical damage, no pinholes, no wrinkles, no oil stains and no debris.

5.2 Technical indicators

5.2.1 Thickness deviation

The absolute value of the thickness deviation of each measurement point of a single membrane product shall not exceed 10%.

5.2.2 Proton dialysis coefficient

The proton dialysis coefficient shall not be less than 5×10^{-7} m/s.

5.2.3 Acid/salt separation factor

The acid/salt separation factor shall not be less than 15.

6 Test methods

6.1 Appearance

The appearance shall be inspected by visual method.

6.2 Thickness deviation

6.2.1 The average thickness

The average thickness of the wet membrane shall be measured in accordance with the provisions given in HY/T 166.1-2013, 6.2.

6.2.2 Calculation of thickness deviation

The difference between the thickness of each measurement point and the average thickness of the wet membrane shall be divided by the average thickness, and the percentage of the obtained ratio is the thickness deviation.

The formula of T_d is as (1):

$$T_d = \frac{T - \bar{T}}{\bar{T}} \times 100\% \dots\dots\dots(1)$$

where

T_d is the thickness deviation of membrane;

T is the thickness of each measuring point of the wet membrane, mm;

\bar{T} is the average thickness of the wet membrane, mm.

6.3 Proton dialysis coefficient, metal ion leakage coefficient and acid/salt separation factor

6.3.1 Test conditions

The pure water used in the test shall comply with the third-grade water standard specified in GB/T 6682, and proton dialysis coefficient, metal ion leakage coefficient and acid/salt separation factor shall be measured with a water temperature of $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ and a room temperature of $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

6.3.2 Test principle

The proton dialysis coefficient and metal ion leakage coefficient are tested in a diffusion dialysis device. The diffusion dialysis device consists of a dialysate chamber and a diffusate chamber with a diffusion dialysis anion exchange membrane placed between the two chambers (see Annex A for the diffusion dialysis device). The dialysate is a mixed solution of acid and salt, and the diffusate is pure water initially. Due to the concentration gradient, the acid and salt will diffuse through the membrane and enter the diffusate chamber. By measuring the concentrations of acid and salt in the dialysate and diffusate, the proton dialysis coefficient and the metal ion leakage coefficient are calculated.

6.3.3 Apparatuses

The apparatuses required for the test are as follows:

- a) Brown acid burette: specification 50 mL, precision 0.1 mL;
- b) Alkaline burette: specification 50 mL, precision 0.1 mL;
- c) Conical flask with stopper: specification 250 mL;
- d) Pipette: specification 10 mL, precision 10 μL ;
- e) Balance: specification 200 g, precision 0.1 mg.
- f) Stopwatch.

6.3.4 Chemical reagents

The chemical reagents are as follows:

- ferrous chloride tetrahydrate, analytical grade;
- potassium permanganate, analytical grade;
- sodium carbonate, benchmark grade;
- sodium oxalate, benchmark grade;
- hydrochloric acid, analytical grade;
- sulfuric acid, analytical grade;
- phosphoric acid, analytical grade.

6.3.5 Solutions and indicators

6.3.5.1 Mixed solution of hydrochloric acid and ferrous chloride [$c(\text{HCl})=3 \text{ mol/L}$ and $c(\text{FeCl}_2)=0.3 \text{ mol/L}$]

Measure 500 mL of pure water in a dry beaker, and add 250 mL of hydrochloric acid and stir well. 59.64 g of ferrous chloride tetrahydrate is added into the same beaker and stirred well. The solution is diluted to 1000 mL. As ferrous ion is extremely unstable under acidic conditions, the mixed solution of hydrochloric acid and ferrous chloride needs to be prepared for immediate use.

6.3.5.2 Phosphoric acid solution

It shall be prepared from one volume of phosphoric acid and six volumes of pure water.

6.3.5.3 Potassium permanganate standard solution [$c(\text{KMnO}_4)=0.002 \text{ mol/L}$]

It shall be prepared in accordance with GB/T 601-2002, 4.12.1. It shall be calibrated in accordance with GB/T 601-2002, 4.12.2.

6.3.5.4 Sodium carbonate standard solution [$c(\text{Na}_2\text{CO}_3)=0.05 \text{ mol/L}$]

It shall be prepared in accordance with GB/T 601-2002, 4.4.1.

6.3.5.5 Methyl orange indicator

It shall be prepared in accordance with GB/T 603-2002, 4.1.4.8.

6.3.6 Membrane pretreatment

Three pieces of 3 cm×3 cm membrane samples are soaked in a mixed solution of hydrochloric acid and ferrous chloride for at least 24 h.

6.3.7 Testing procedures

The test procedures for proton dialysis coefficient and metal ion leakage coefficient are as follows:

- Take out the pretreated membrane, rinse the membrane with pure water three times, then wipe off the surface droplets with filter paper, clamp it between the dialysate and the diffusate chambers, and seal it with rubber rings (see Annex A);
- Accurately measure 100 mL of the mixed solution of hydrochloric acid and ferrous chloride and 100 mL of pure water, and pour them into the dialysate and the diffusate chambers respectively;
- Stir for 1 h. The proton and ferrous ion concentrations in the dialysate and the diffusate chambers are measured respectively. See Annex B for the analysis method of proton and ferrous ion concentrations.

6.3.8 Data processing

The formula of U_H is as (2):

$$U_H = \frac{M_H}{A \cdot t \cdot \Delta C_H} \dots\dots\dots(2)$$

where

U_H is the proton dialysis coefficient (m/s);

M_H is the amount of substance of proton diffusing into the diffusate chamber (mol);

A is the effective area of the membrane (see Figure A.1) (m²);

t is the test time of diffusion dialysis, s (usually t is 3600 s);

ΔC_H is the logarithmic average concentration of proton in the two solutions (mol/m³).

The formula of ΔC_H is as (3):

$$\Delta C_H = \frac{C_{D,H}^0 - (C_{D,H}^t - C_{d,H}^t)}{\ln \left[\frac{C_{D,H}^0}{(C_{D,H}^t - C_{d,H}^t)} \right]} \dots\dots\dots(3)$$

where

$C_{D,H}^0$ is the concentration of proton in the dialysate chamber at the beginning (mol/m³);

$C_{D,H}^t$ is the concentration of proton in the dialysate chamber after test for 1 h (mol/m³);

$C_{d,H}^t$ is the concentration of proton in the diffusate chamber after test for 1 h (mol/m³).

The formula of U_{Fe} is as (4):

$$U_{Fe} = \frac{M_{Fe}}{A \cdot t \cdot \Delta C_{Fe}} \dots\dots\dots(4)$$

where

U_{Fe} is the metal ion leakage coefficient (m/s);

M_{Fe} is the amount of substance of ferrous ion diffusing into the diffusate chamber (mol);

ΔC_{Fe} is the logarithmic average concentration of ferrous ion in the two solutions (mol/m³).

The formula of ΔC_{Fe} is as (5):

$$\Delta C_{Fe} = \frac{C_{D,Fe}^0 - (C_{D,Fe}^t - C_{d,Fe}^t)}{\ln \left[\frac{C_{D,Fe}^0}{C_{D,Fe}^t - C_{d,Fe}^t} \right]} \dots\dots\dots(5)$$

where

$C_{D,Fe}^0$ is the concentration of ferrous ion in the dialysate chamber at the beginning (mol/m³);

$C_{D,Fe}^t$ is the concentration of ferrous ion in the dialysate chamber after test for 1 h (mol/m³);

$C_{d,Fe}^t$ is the concentration of ferrous ion in the diffusate chamber after test for 1 h (mol/m³).

The formula of S is as (6):

$$S = \frac{U_H}{U_{Fe}} \dots\dots\dots(6)$$

where

S is the acid/salt separation factor.

7 Inspection rules

7.1 Factory inspection

7.1.1 Inspection items

Each batch of diffusion dialysis anion exchange membranes shall be inspected before delivery. The inspection items shall be carried out in accordance with Table 3.

Table 3 Factory inspection

No.	Inspection items	Clause of requirements	Clause of test methods
1	appearance	5.1	6.1

2	thickness deviation	5.2.1	6.2
3	proton dialysis coefficient	5.2.2	6.3
4	acid/salt separation factor	5.2.3	6.3

7.1.2 Batch rule

Membrane rolls produced from the same raw materials are a batch.

7.1.3 Sampling method

Two for every ten membrane rolls of the same batch are sampled for inspection.

7.1.4 Judgement method

When the results of all inspection items comply with requirements of this document, the batch of products is judged to be qualified. If any there are unqualified items in the results of the inspection items, double samples shall be taken from the original batch of products and the unqualified items will be re-tested. If any there are still unqualified items, the batch of products shall be judged to be unqualified.

7.2 Type inspection

7.2.1 Type inspection conditions

Type inspection shall be carried out in one of the following situations:

- a) Production for one year;
- b) Major variation in materials or processes;
- c) Appraisal of new product type or appraisal of old product produced by switching to other production line;
- d) Production resuming after suspended for more than half a year;
- e) Requested by the State Quality Supervision Agency.

7.2.2 Inspection items

Inspection items shall be all items specified in Clause 5.

7.2.3 Sampling method

Five for every ten membrane rolls of the same batch are sampled for inspection.

7.2.4 Judgement method

When the results of all inspection items comply with requirements of this document, the batch of products is judged as qualified. If any there are unqualified items in the results of the inspection items, the batch of products shall be judged to be unqualified.

8 Sign, packaging, transport and storage

8.1 Sign

Diffusion dialysis anion exchange membrane shall be delivered with sign on the inner packaging. The sign includes:

- a) Product name and model;
- b) Trademark, product series number;
- c) Production date;
- d) Name and address of the manufacturer;
- e) The implemented production standards.

8.2 Packaging

The packaging of the diffusion dialysis anion exchange membrane shall be as specified in GB/T 191.

Polyethylene blown film as the inner packaging shall be as specified in GB/T 4456. Wooden box as the outer packaging shall be as specified in GB/T 12464.

The packing box shall be accompanied by packing list, inspection certificates, instruction manuals and other documents, and the inspection certificates shall be as specified in GB/T 14436 and the instruction manual shall be as specified in GB/T 9969.

8.3 Transport

The transport of diffusion dialysis anion exchange membrane shall be as specified in GB/T 9174. The transport, loading and unloading processes shall not be subjected to violent impacts, bumps, throws and great pressure. Contacting with corrosive chemical reagents shall be avoided.

8.4 Storage

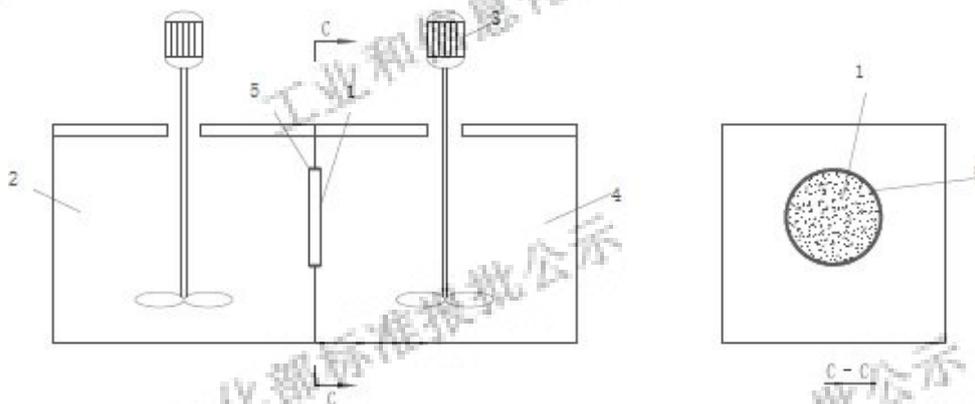
The product shall be stored in a clean, ventilated and dry space, with a temperature between 5 °C and 40 °C, and shall be kept away from heat, fire and chemicals, and shall not be stored together with solvents, oxidizing or reducing chemicals.

Annex A

(informative)

Schematic diagram of diffusion dialysis device

The schematic diagram of the diffusion dialysis device is shown in Figure A.1.



where

- 1 diffusion dialysis anion exchange membrane;
- 2 dialysate chamber;
- 3 stirrer;
- 4 diffusate chamber,
- 5 rubber ring.

NOTE 1 At the beginning the dialysate chamber is filled with 100 mL of feed liquid and the diffusate chamber is filled with 100 mL of pure water. The membrane sample shall be fully immersed.

NOTE 2 The effective membrane area is the area of a circle with a diameter of 2.5 cm.

NOTE 3 The figure on the right is a cross-sectional side view of position C in the figure on the left.

Figure A.1 Schematic diagram of the diffusion dialysis device

Annex B

(informative)

Analysis method of proton and ferrous ion concentrations

B.1 Analysis method of proton concentration

The proton is titrated with sodium carbonate standard solution. When the dialysate is analyzed, 1.00 mL of the solution is taken; when the diffusate is analyzed, 10.00 mL of the solution is taken. 50 mL of pure water and two or three drops of methyl orange indicator are added, and the solution is titrated with 0.05 mol/L sodium carbonate standard solution. When the solution changes color from red to orange and keeps orange for 30 seconds, it is regarded as the titration end point. A blank experiment shall be carried out with pure water at the same time.

The formula of C_H is as (B.1):

$$C_H = \frac{2 \cdot C_{Na_2CO_3} \cdot (V_1 - V_2)}{V_0} \quad \dots\dots\dots(B.1)$$

where

C_H is the proton concentration (mol/m³);

$C_{Na_2CO_3}$ is the concentration of sodium carbonate standard solution (mol/m³);

V_1 is the volume of sodium carbonate standard solution consumed by titration (mL);

V_2 is the volume of sodium carbonate standard solution consumed by titration during the blank experiment (mL);

V_0 is the sampling volume of the titrated solution (mL).

B.2 Analysis method of ferrous ion concentrations

The ferrous ion is titrated with potassium permanganate standard solution. When the dialysate is analyzed, 1.00 mL of the solution is taken; when the diffusate is analyzed, 10.00 mL of the solution is taken. 50 mL of pure water and 10 mL of phosphoric acid solution are added. The dialysate is titrated with 0.002 mol/L potassium permanganate standard solution; the diffusate is titrated with 0.0004 mol/L potassium permanganate standard solution (diluted 5 times from 0.002 mol/L potassium permanganate standard solution). When the solution turns pink and keeps pink for 30 seconds, it is regarded as the titration end point. A blank experiment shall be carried out with pure water at the same time.

The formula of C_{Fe} is as (B.2):

$$C_{Fe} = \frac{5 \cdot C_{Mn} \cdot (V_1 - V_2)}{V_0} \dots\dots\dots(B.2)$$

where

C_{Fe} is the ferrous ion concentration (mol/m³);

C_{Mn} is the concentration of potassium permanganate standard solution (mol/m³);

V_1 is the volume of potassium permanganate standard solution consumed by titration (mL);

V_2 is the volume of potassium permanganate standard solution consumed by titration during the blank experiment (mL);

V_0 is the sampling volume of the titrated solution (mL).