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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 20042.4-2009

**Proton exchange membrane fuel cell - Part 4: Test method for
electrocatalysts**

(English Translation)

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1 Scope

This part of GB/T 20042 specifies the terminology and definition, platinum content test, electrochemical active area test, specific surface area, pore volume, pore size distribution test, morphology and particle size distribution test, crystal structure test, catalyst bulk density test, and single cell polarization curve test of the electrocatalyst test method for proton exchange membrane fuel cells.

This part applies to various types of platinum-based (Pt-based) electrocatalyst for proton exchange membrane fuel cell.

2 Normative references

The provisions in following documents become the provisions of this part through reference in GB/T 20042. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard;

however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable.

For undated references, the latest edition of the referenced document applies.

GB/T 5816-1995 Catalyst and adsorbents - Determination of surface area

GB/T 13566-1992 Determination of bulk density for fertilizers (ISO 3944.1980, EQV)

GB/T 15072.7-2008 Test methods of precious metal alloys - Determination of chromium and iron contents for gold alloys - Inductively coupled plasma atomic emission spectrometry

GB/T 20042.1 Proton exchange membrane fuel cell - Terminology

3 Terms and definitions

The terms and definitions defined in GB/T 20042.1 and the following terms and definitions apply to this part.

Electrochemical active area

L - Pt loading capacity, in unit of %;

W1 - The mass of the sample at endpoint temperature, in milligrams (mg);

W0 - The original mass of the sample, in milligrams (mg).

TAKE 3 samples as a group, CALCULATE the average value as the test result.

4.2

Pt content test by ICP (inductively coupled plasma spectroscopy) method

The test is performed in accordance with the method as specified in GB/T 15072.7-2008.

4.2.1 Scope of application

This method is suitable for testing Pt content in Pt/C catalysts and Pt alloy catalysts.

4.2.2.1 Ion-coupled emission spectrometry (ICP)

The minimum detection limit is $\leq 1 \mu\text{g/L}$.

4.2.2.2 Analytical balance

The precision is 0.1 mg.

4.2.2.3 Caliper

The measurement precision is 0.01 mm.

4.2.3 Sample preparation

The sample mass is not less than 2 g.

The sample is placed in a vacuum oven and dried at 80 °C for 12 h.

4.2.4.1

Concentrated sulfuric acid (98%), guaranteed reagent.

4.2.4.2

Concentrated hydrochloric acid (37%), guaranteed reagent.

4.2.4.3

Concentrated nitric acid (68%), guaranteed reagent.

4.2.4.4

Secondary distilled water, resistivity $\geq 18.2 \text{ Mohm} \cdot \text{cm}$.

4.2.4.6

Corundum crucible with cover.

4.2.5 Test method

C - Smooth Pt surface adsorption hydrogen peroxide adsorption constant, in 0.21 millicoulombs per square centimeter (0.21 mC/cm^2);

v - Scanning speed, in millivolts per second (mV/s);

M - The mass of Pt on the electrode, in grams (g).

TAKE 3 samples as a group, CALCULATE the average value as the test result.

6 Specific surface area, pore volume, pore size distribution test

The test is performed in accordance with the method as specified in GB/T 5816-1995.

6.1.1

Fully automatic physical adsorption instrument.

6.1.2 Analytical balance

the precision is 0.01 mg.

6.1.3 Test gas

The oil-free high-purity nitrogen gas and helium gas after drying, the purity is not less than 99.999%.

6.2 Test methods

The static nitrogen adsorption capacity method is used to measure the volume of nitrogen adsorbed by the catalyst at different low pressures. At least four test points satisfying the BET linear relationship are measured, and the surface area is calculated using the BET two-parameter equation.

6.2.1 Sample pretreatment and degassing

a) In accordance with the degassing requirements, PLUG the empty sample tube after degassing treatment to weigh it, accurate to 0.01 mg. At this point the mass is recorded as m1;

b) TAKE the appropriate amount of sample and ADD it into the sample tube.

SET the heating temperature (generally less than 200 °C) to heat the sample and EMPTY it. When the heating temperature reaches the set temperature and the system vacuum reaches 1.3 Pa, CONTINUE degassing for at least 4 hours. It is allowed for the sample to be degassed overnight;

c) COOL the degassed sample tube to room temperature, ADD plug to weigh it, accurate to 0.01 mg. This mass is recorded as m2, and the difference to 0.1 mg.

9.3.2

USE a test funnel, POUR a certain amount of sample into the measuring cylinder within 20 s ~ 25 s. The sample volume must exceed the amount required to fill the measurement cylinder full. During the pour-in process, USE a rod to gently knock the measuring cylinder wall at the frequency of 2 ~ 3 times per second, to make the sample compact. If the sample flow is not smooth, it may use a glass rod with a diameter of about 4 mm to clean the funnel discharge port to make it unblocked.

9.3.3

CLOSE the funnel, then RAISE the measuring cylinder by 2 mm ~ 3 mm,

LET it fall down to further compress the sample, REPEAT this operation for 20 times, READ out the sample volume (mL).

9.3.4

WEIGH the total mass of the measuring cylinder and the sample,

RECORD it as M2, accurate to 0.1 mg.