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Test method of activity for rhodium carbon catalysts

铑炭催化剂活性试验方法

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1 Scope

This Standard specifies the test method of activity for rhodium carbon catalysts.

This Standard applies to rhodium carbon catalysts for hydrogenation reduction of aromatic rings, heterocyclic rings, nitro, nitriles, fatty carboxylic acids and other fine chemicals.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 6679, General rules for sampling solid chemical products

GB/T 6682, Water for analytical laboratory use. Specification and test methods

3 Principle

O-chloronitrobenzene in the raw material reacts with hydrogen to form o-chloroaniline and water under the action of rhodium carbon catalyst. Its chemical reaction formula is as follows:

$$C_6H_4ClNO_2 + 3H_2 = C_6H_6ClN + 2H_2O$$

Use a gas chromatograph to determine the mass fraction of o-chloronitrobenzene before and after the reaction; calculate the conversion rate of o-chloronitrobenzene, to characterize the activity of the catalyst.

4 Reagents or materials

4.1 O-chloronitrobenzene: purity greater than 99.0%.

4.2 Methanol: analytical reagent.

4.3 Deionized water: grade-II water which is specified in GB/T 6682.

forbidden on the site; preventive facilities such as necessary fire extinguishing equipment and exhaust equipment shall be equipped; it is strictly forbidden to carry out disassembly operation of reactor under pressure. This Standard does not reveal all possible safety issues; users shall use it strictly in accordance with relevant regulations, and have the responsibility to take appropriate safety and health measures.

7.1 Filling of the reactor

Add 30.0 g of o-chloronitrobenzene, 150 mL of methanol, and catalyst samples (see 6.3) to the clean and dry reactor in order; cover the kettle lid; tighten the nuts evenly; then, connect it to the system.

7.2 Leakage test

Open the nitrogen valve; introduce nitrogen into the system; boost the system to 1.00 MPa; close the inlet and outlet valves of the system. If the pressure drop is less than 0.05 MPa within 30 min, the system is considered to be sealed. Open the system outlet valve to reduce the pressure of the system to normal pressure; insert the temperature measuring thermocouple into the thermowell.

7.3 Gas replacement and activity determination

Use nitrogen to replace the system for 3 times; then, exhaust and reduce the pressure to normal pressure. Switch to hydrogen and replace for 3 times; then, exhaust and reduce the pressure to normal pressure. Warm and heat the reactor. When the temperature of the reactor is raised to 75°C, increase the system pressure to 1.00 MPa. Control and maintain the reactor pressure at 1.00 MPa, the temperature at 75°C, and the stirring speed at 1 000 r/min. After 1 hour, end the test.

7.4 Power-off

Turn off the hydrogen and turn on the cooling water; after the temperature of the reactor drops to below 40°C, release the pressure. Use nitrogen to replace the system for 3 times; perform sampling and analysis; use deionized water to clean the reactor body.

7.5 Determination of o-chloronitrobenzene mass fraction

7.5.1 Chromatograph operating conditions

Chromatographic column: DB-1 (30 m × 0.25 mm × 0.25 μm).

Nitrogen flow: 30.0 mL/min.

Hydrogen flow: 40.0 mL/min.

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