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**Analytical method of chemical composition for
activated carbon mercury removal catalyst**

活性炭脱汞催化剂化学成分分析方法

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Analytical method of chemical composition for activated carbon mercury removal catalyst

WARNING - Some reagents used in this Standard are toxic or corrosive, and some operations are dangerous. This Standard does not reveal all possible safety issues. Users shall exercise caution and have the responsibility to take appropriate safety and health measures when operating.

1 Scope

This Standard specifies the analytical method of chemical composition for activated carbon mercury removal catalyst.

This Standard is applicable to the determinations of mass fractions of sulfur (S), calcium (Ca), magnesium (Mg), iron (Fe), aluminum (Al) in activated carbon mercury removal catalyst.

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 602, *Chemical reagent - Preparations of stock standard solutions*

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

GB/T 6003.1, *Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth*

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

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

3 General provisions

All reagents and water used in this Standard, when no other requirements are

Mix the sample and the mixed flux well and burn, to make all sulfur into soluble sulfate. Add barium chloride solution to make sulfate to generate barium sulfate precipitation. According to the mass of barium sulfate, calculate the content of sulfur in the sample.

5.2 Reagents

5.2.1 Mixed flux: Take 2-portion masses of light magnesium oxide and 1-portion mass of anhydrous sodium carbonate. Mix well and grind well. Store in a sealed container.

5.2.2 Hydrochloric acid solution: 1+1.

5.2.3 Barium chloride solution: 100g/L.

5.2.4 Silver nitrate solution: 10g/L. 1g of silver nitrate is dissolved in 100mL of water. Add 2~3 drops of nitric acid. Store in a brown bottle.

5.2.5 Methyl orange indicator liquid: 2g/L.

5.3 Test steps

5.3.1 Weigh about 0.2g of specimen (see 4.2), to the nearest of 0.0001g. Place in a 50mL porcelain crucible. Add 2g of mixed flux. Mix well. Add 1g of mixed flux on top. Place the crucible in a muffle furnace. Within 1h~2h, gradually heat it from room temperature to 800°C~850°C. And remain 1h at this temperature. Take it out to cool to room temperature. Use a glass rod to carefully stir the burning matter in the crucible to loosen. Then move the burning matter into a 400mL beaker. Use hot water to rinse the inner wall of the crucible. Combine the washing liquid into the beaker. Then add 100mL~150mL of distilled water that has just boiled. Stir completely.

5.3.2 Use medium-speed qualitative filter paper to filter by the pouring method. Use hot water to rinse 3 times. Then move the residues into the filter paper. Use hot water to carefully wash at least 10 times. The total volume of the washing liquid is about 250mL~300mL. Add 2~3 drops of methyl orange indicator into the filtrate. Add hydrochloric acid solution to make the filtrate turn from yellow to red and exceed by 2mL. Heat the solution till it is boiling. Under continuous stirring, slowly add 15mL of barium chloride solution. And keep it 2h under the slight boiling condition. The final solution volume is about 200mL. After the solution is cooled or left overnight, use a slow-speed quantitative filter paper to filter. Then use hot water to wash till it is chloride-free (when silver nitrate solution is tested without turbidity). Move the filter paper that has precipitation into a porcelain crucible with a known mass. After ash the filter paper at a low temperature, burn in an 850°C~900°C muffle furnace for 40min. Take out the porcelain crucible. Put into a dryer to cool to room temperature then weigh.

Atomic absorption spectrophotometer: with hollow calcium cathode lamp.

6.4 Test steps

6.4.1 Drawing of working curve

6.4.1.1 Take 5 pieces of 100mL volumetric flasks. Respectively add 0mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL of calcium standard solution. In each volumetric flask, respectively add 2mL of strontium chloride solution and 2mL of hydrochloric acid solution. Use water to dilute to the scale. Shake well.

6.4.1.2 According to the operating conditions of the instrument, use air-acetylene flame, use the blank solution that does not have calcium standard solution to perform zero adjustment. At a wavelength of 422.7nm, determine the absorbance of solution.

6.4.1.3 Take the mass concentration of calcium in the above solution (in micrograms per milliliter) as the abscissa and the corresponding absorbance value as the ordinate, to draw the working curve or calculate the linear regression equation.

6.4.2 Determination

Weigh 10.00mL of test material solution. Place in a 50mL volumetric flask. Add 1mL of strontium chloride solution. Use water to dilute to the scale. Shake well. According to the provisions of 6.4.1.2, determine the absorbance of the solution. Find the mass concentration of calcium in the test solution from the working curve or calculate it by the linear regression equation.

6.5 Test data processing

Calcium (Ca) mass fraction w_2 is calculated according to formula (2):

$$w_2 = \frac{\rho_1 V_1 \times 10^{-6}}{m_4} \times 100\% \quad \dots\dots\dots (2)$$

Where,

ρ_1 - Numerical value of the mass concentration of calcium in the test solution that is found from the working curve or calculated by the linear regression equation, in micrograms per milliliter ($\mu\text{g/mL}$);

V_1 - Numerical volume value of test material solution, in milliliters (mL);

m_4 - Numerical value of distributed test material's mass, in grams (g).

Take the arithmetic mean of two parallel determination results as the

micrograms per milliliter) as the abscissa and the corresponding absorbance value as the ordinate, to draw the working curve or calculate the linear regression equation.

7.4.2 Determination

Weigh 10.00mL of test material solution in a 100mL volumetric flask. Add 2mL of strontium chloride solution. Use water to dilute to the scale. Shake well. According to the provisions of 7.4.1.2, determine the absorbance of solution. Find the mass concentration of magnesium in the test solution from the working curve or calculate it from the linear regression equation.

7.5 Test data processing

Calculate magnesium (Mg) mass fraction w_3 according to formula (3):

$$w_3 = \frac{\rho_2 V_2 \times 10^{-6}}{m_5} \times 100\% \quad \dots\dots\dots (3)$$

Where,

ρ_2 - Numerical value of the mass concentration of magnesium in the test solution that is found from the working curve or calculated by the linear regression equation, in micrograms per milliliter ($\mu\text{g/mL}$);

V_2 - Numerical volume value of test material solution, in milliliters (mL);

m_5 - Numerical value of distributed test material's mass, in grams (g).

Take the arithmetic mean of two parallel determination results as the measurement result. The relative deviation of the parallel determination results is not more than 5%.

8 Determination of iron (Fe) mass fraction - Atomic absorption spectrophotometry

8.1 Principle

Under acidic conditions, use atomic absorption spectrophotometer, use air-acetylene flame, at a wavelength of 248.3nm, to determine the absorbance of iron in the test material solution. Use the working curve method or the linear regression equation to quantify.

8.2 Reagents

m_6 - Numerical value of test material's mass, in grams (g).

Take the arithmetic mean of two parallel determination results as the measurement result. The relative deviation of the parallel determination results is not more than 5%.

9 Determinations of mass fractions of calcium (Ca), magnesium (Mg), iron (Fe), aluminum (Al) - Inductively coupled plasma emission spectroscopy

9.1 Principle

Under acidic conditions, use high purity argon flame to introduce the solution atomization into inductively coupled plasma. Determine the signal intensity of the analytical lines of calcium, magnesium, iron, and aluminum in the test material solution. Use the working curve method to quantify.

9.2 Reagents

9.2.1 Hydrochloric acid solution: 1+1.

9.2.2 Calcium standard solution: 0.1mg/mL.

9.2.3 Magnesium standard solution: 0.1mg/mL.

9.2.4 Iron standard solution: 0.1mg/mL.

9.2.5 Aluminum standard solution: 0.1mg/mL.

9.3 Instruments

Inductively coupled plasma emission spectrometer.

9.4 Test steps

9.4.1 Drawing of working curve

9.4.1.1 Take 5 pieces of 100mL volumetric flasks. Respectively pipette corresponding standard solution. Prepare standard solution series according to Table 1. In each volumetric flask, respectively add 2mL of hydrochloric acid solution. Use water to dilute to the scale. Shake well.