SiO₂-content relating to the substance ignited to constant weight at 1000 °C of AEROSIL® - and AEROPERL® - products

PA 0501

1. Background / Reason
As the silicon dioxide content of AEROSIL® is very high, the impurities need not be removed beforehand. To determine the SiO₂ content, the sample is treated with a sulphuric acid/hydrofluoric acid mixture. Where samples are older than 10 days or for hydrophobic AEROSIL® in general, the samples are ignited and weighed beforehand and then treated with the sulphuric acid/hydrofluoric acid mixture. As the generated silicon tetrafluoride is volatile, the SiO₂ content can be determined by the loss in weight.

2. Apparatus and Reagents

Apparatus
- Analytical balance (accuracy of reading 0.1 mg)
- Platinum evaporating dish
- Muffle furnace
- Desiccator

Reagents
- Dry Agent, e.g. Orange gel
- Sulphuric acid,
- Hydrofluoric acid

3. Sampling
Before the sample is taken out of the sample box provided, a good mixing of the sample should be ensured.

4. Description
4.1. Determination

4.1.1. Determination of SiO₂ content in samples which are younger than 10 days

First 5 ml sulphuric acid and then 50 ml hydrofluoric acid are poured into a large platinum dish. Approx. 10 g of the substance to be tested is weighed to a precision of 0.01 g (m₁). The sample is added in portions and fumed off. The residue is ignited until no more white fog appears. Then it is ignited at 1000 °C in the muffle furnace for 30 minutes. The residue's mass (m₃) is determined after cooling down in the desiccator (over orange gel).

4.1.2. Determination of SiO₂ content of hydrophobic AEROSIL®-products or in samples which are older than 10 days

Approx. 1 g of the substance to be tested is weighed to a precision of 0.1 mg into an ignited platinum evaporating dish (m₀). Then the sample is ignited at 1000 °C ± 20 °C to constant weight (for approx. 2 h) in a muffle furnace. After cooling down in the desiccator (over orange gel), the mass of the ignited substance (m₂) is determined. The sample (m₂) is wetted in the platinum evaporating dish with 2-3 ml ultrapure water after which 1 ml sulphuric acid and 15 ml hydrofluoric acid are added. The residue is ignited until no more white fog appears. Then it is ignited at 1000 °C in the muffle furnace for 30 minutes and the mass (m₃) is determined after cooling down in the desiccator.
4.2. Calculation

4.2.1. Calculation of SiO$_2$ content in samples which are younger than 10 days

The following equation is used to calculate the percentage by mass of the SiO$_2$:

$$\text{SiO}_2\text{Content} = \left(\frac{m_1 - m_3}{m_1}\right) \times 100$$

Where:
- $m_1$: mass in gram of substance after ignition at 1000 °C.
- $m_2$: mass in gram after the treatment with hydrofluoric acid and ignition to constant weight.

4.2.2. Calculation of SiO$_2$ content of hydrophobic AEROSIL®-products or in samples which are older than 10 days

$$\text{SiO}_2\text{Content} = \left(\frac{m_2 - m_3}{m_2}\right) \times 100$$

Where
- $m_2$: mass in gram of substance after ignition at 1000 °C.
- $m_3$: mass in gram after the treatment with hydrofluoric acid and ignition to constant weight.

Report the result to the nearest 0.1 %

5. Reference

This method is in accordance with ISO 3262-20.