

SiO₂-Content relating to the substance ignited to constant weight at 1000°C of AEROSIL® and AEROPERL® products PA 0501 or ACM-118

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

Analytical balance (accuracy of reading 0.1 mg)
Platinum evaporating dish
Muffle furnace
desiccator
Dry Agent, e.g. Orange gel
Sulphuric acid,
Hydrofluoric acid

Notes: The analysis is executed using only reagents with a certain degree of purity and ultrapure water (degree of purity of at least 3 in accordance with ISO 3696).

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 (m_1). The sample is added in portions and fumed off. Avoiding any splashes, the mixture is concentrated to a sirupy consistency. Then the mixture is evaporated to dryness. If the silicon tetrafluoride has not yet been fully volatilised, the treatment must be continued with a further 10 ml of hydrofluoric acid.

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 (m_3) 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

For older samples, the weighed in primary mass must be corrected by the total loss on ignition. Approx. 1 g of the substance to be tested is weighed to a precision of 0.1 mg into an ignited platinum evaporating dish (m_0). Then the sample is ignited at 1000°C ± 20°C to constant weight (for approx. 2 hrs) in a muffle furnace. After cooling down in the desiccator (over orange gel), the mass of the ignited substance (m_2) is determined.

The sample (m_2) 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. Avoiding any splashes, the mixture is concentrated to a sirupy consistency.

After the platinum dish has cooled down, the internal sidewalls are washed with distilled water and a further 10 ml hydrofluoric acid is added and evaporated to dryness. If the silicon tetrafluoride has not yet been fully volatilised, the treatment must be continued with a further 10 ml of hydrofluoric acid.

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 (m_3) is determined after cooling down in the desiccator.

4.2. Calculation

4.2.1. Calculation of SiO₂ content in samples which are younger than 10 days

The following equation is used to calculate the percentage by mass of the SiO₂:

$$\text{SiO}_2 \text{ Content [\%]} = \frac{(m_1 - m_3)}{m_1} * 100$$

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

$$\text{SiO}_2 \text{ Content [\%]} = \frac{(m_2 - m_3)}{m_2} * 100$$

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 DIN EN ISO 3262-20.