Determination of fluorine and sulfur in phosphate rock certified reference material

Sit No.: AQF_MR_019E
Category: J

Instruments: AQF-2100H System
HF-210, GA-210, ABC-210/A
SC-240S

Method: Combustion-ion chromatography

Related standard
It is critically important to know the Fluorine and Sulfur content in the sample which is a resource of fluoride products. Concentrations of fluorine, chlorine, bromine, iodine, and sulfur can be determined and accurately by using a combustion ion chromatography (CIC) system combining an Automatic Quick Furnace Model AQF-2100H which safely combusts samples with an ion chromatograph.

Sample name | CERTIFIED REFERENCE MATERIAL BCR032 Moroccan phosphate rock
Sample status | Measuring items: Fluorine (F), Sulfur (S)
Measurement principle | Sample is thermally decomposed in argon (Ar) atmosphere, then combusted in oxygen (O₂) atmosphere. Halogens in the sample are converted to hydrogen halide and halogen gas and sulfur turns into sulfur oxide. These components are collected into absorbing solution and converted to halide ion and sulfate ion. The resulting solution is analyzed by injecting into an ion chromatograph (IC).

Analyzing flow
[Sample weighing] ⇒ [Combustion] ⇒ [Collection of combustion gas] ⇒ [IC analysis]

Parameters

1. AQF-2100H

Sample size: 5mg
Sample boat: Ceramic sample boat, SXSMBS
Additive: WO₃ 100mg
Pyrolysis tube: Quartz tube filled with quartz wool
Absorbent: Hydrogen peroxide / water
Mode: Constant volume mode

HF-210
Heater Temp.
Inlet: 1000degC
Outlet: 1100degC
Gas flow Ar: 200 ml/min
O₂: 400 ml/min

GA-210
Absorbent volume: 10ml
Sampling loop: 20 ul
Absorption tube: For 10 ml
Water supply: 4
Ar flow for water supply: 100 ml/min
2. Ion chromatograph

Ion chromatograph: DIONEX ICS-1500
Column: DIONEX Ion Pack AG12A / Ion Pack AS12A
Eluent: 2.7mM Na₂CO₃ / 0.3mM NaHCO₃
Eluent flow: 1.50mL / min
Detector: Conductivity
Suppressor: ASRS-300 4-mm
Measuring time: 30min
Sampling loop: 20 μL using GA-210 sampling loop
Calibration: F Cl Br S: 5ppm to 40ppm

Chromatogram

Result

<table>
<thead>
<tr>
<th>Measurement</th>
<th>F (mg/kg)</th>
<th>Cl (mg/kg)</th>
<th>Br (mg/kg)</th>
<th>S (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CERTIFY</td>
<td>40400</td>
<td>-</td>
<td>-</td>
<td>7360</td>
</tr>
<tr>
<td>± 600</td>
<td>-</td>
<td>-</td>
<td></td>
<td>± 320</td>
</tr>
<tr>
<td>1</td>
<td>38984</td>
<td>-</td>
<td>-</td>
<td>6614</td>
</tr>
<tr>
<td>2</td>
<td>39072</td>
<td>-</td>
<td>-</td>
<td>6569</td>
</tr>
<tr>
<td>3</td>
<td>38944</td>
<td>-</td>
<td>-</td>
<td>6486</td>
</tr>
<tr>
<td>Avg.</td>
<td>39000</td>
<td>-</td>
<td>-</td>
<td>6556</td>
</tr>
<tr>
<td>RSD(%)</td>
<td>0.17</td>
<td>-</td>
<td>-</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Recovery: F=97%  S=89%

Remarks

- Handling of reagents: Confirm labels and safety data sheets of reagents and handle them with enough care.
- Automation is possible by using an Automatic Sample Changer, ASC-240S. When ASC-240S is used, the boat to be used will be a ceramic boat, TX3SCX.
- Use an internal standard material other than phosphate ion (PO₄³⁻) when analysis is performed by the internal standard method.

This application sheet is provided as reference, and does not assure the measurement results. Please consider analysis environment, external factors and sample nature for optimal conditions before the measurement.