

Sheet No.

**AQF MR 019E** Reference Materials

# Determination of fluorine and sulfur in phosphate rock certified reference material \_\_\_\_\_ 1/2

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 (O <sub>2</sub> ) 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). <b>Analyzing flow</b> [Sample weighing]→[Combustion]→[Collection of combustion gas]→[IC analysis]
Parameters	<p><b>1.AQF-2100H</b></p> <p>Sample size : 5mg  Sample boat : Ceramic sample boat, SXSMBS  Additive : WO<sub>3</sub> 100mg  Pyrolysis tube : Quartz tube filled with quartz wool  Absorbent : Hydrogen peroxide / water  Mode : Constant volume mode</p> <p>HF-210 Heater Temp. Inlet : 1000degC  Outlet : 1100degC  Gas flow Ar : 200 ml/min  O<sub>2</sub> : 400 ml/min</p> <p>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</p>

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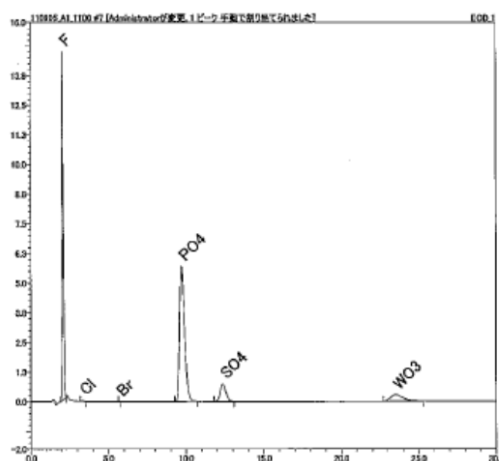
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**2. Ion chromatograph**

Ion chromatograph : DIONEX ICS-1500  
 Column : DIONEX Ion Pack AG12A / Ion Pack AS12A  
 Eluent : 2.7mM Na<sub>2</sub>CO<sub>3</sub> / 0.3mM NaHCO<sub>3</sub>  
 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

Results

**Chromatogram**



**Results**

	Measurement			
	F(mg/kg)	Cl(mg/kg)	Br(mg/kg)	S(mg/kg)
CERTIFY	40400 ±600			7360 ±320
1	38984			6614
2	39072			6569
3	38944			6486
Avg.	39000			6556
RSD(%)	0.17			0.99

**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 (PO<sub>4</sub><sup>3-</sup>) 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.

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