

Sheet No.

**AQF MR 009E** Reference Materials

# Determination of fluorine and sulfur in fluorite certified reference material

1/2

Instruments : AQF-100

Method : Combustion-ion chromatography

Related standard :

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-100 which safely combusts samples with an ion chromatograph.

Sample name	Fluorite (Reference material certified by the Iron and Steel Institute of Japan)																																				
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-100</b></p> <p>Sample size : 10mg  Sample boat : Ceramic sample boat, SXSMBS  Additive : WO<sub>3</sub> 50mg  Pyrolysis tube : Quartz tube filled with quartz wool  Absorbent : 1000ppm Hydrogen peroxide / water  Mode:</p> <p>Heater Temp. Inlet : 1100degC  Outlet : 1100degC  Gas flow Ar : 200 ml/min  O<sub>2</sub> : 400 ml/min</p> <p>GA-100 Absorbent volume : 20 ml  Sampling loop : 20 ul  Absorption tube : For 20 ml  Water supply : 4  Ar flow for water supply : 150 ml/min</p> <p>ABC-100/ASC120S</p> <table border="1"> <thead> <tr> <th></th> <th></th> <th>1st</th> <th>2nd</th> <th>3rd</th> <th>4th</th> <th>5th</th> <th>End</th> <th>Cool</th> </tr> </thead> <tbody> <tr> <td>Position</td> <td>(mm)</td> <td>0</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> <tr> <td>Time</td> <td>(sec)</td> <td>0</td> <td></td> <td></td> <td></td> <td></td> <td>900</td> <td>30</td> </tr> <tr> <td>Speed</td> <td>(mm/sec)</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> </tbody> </table> <p style="text-align: right;">Ar Time 0 (sec) O<sub>2</sub> Time 900(sec)</p>			1st	2nd	3rd	4th	5th	End	Cool	Position	(mm)	0							Time	(sec)	0					900	30	Speed	(mm/sec)							
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	<p><b>2. Ion chromatograph</b></p> <p>Ion chromatograph : DIONEX DX-320          Column : DIONEX Ion Pack AG12A / Ion Pack AS22          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-4-mm          Measuring time : 30min          Sampling loop : 20 ul using GA-100 sampling loop          Calibration : F Cl Br S :5ppm to 40ppm</p>												
<p>Results</p>	<p><b>Results</b></p> <table border="1" data-bbox="392 920 1431 1039"> <thead> <tr> <th>Sample</th> <th>Component</th> <th>Indicated value (%)</th> <th>Results (%)</th> </tr> </thead> <tbody> <tr> <td>Fluorite (CaF<sub>2</sub>)</td> <td>F</td> <td>36.7</td> <td>35.5</td> </tr> <tr> <td>NTST Standard</td> <td>S</td> <td>0.39</td> <td>0.4</td> </tr> </tbody> </table>	Sample	Component	Indicated value (%)	Results (%)	Fluorite (CaF <sub>2</sub> )	F	36.7	35.5	NTST Standard	S	0.39	0.4
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<p>Remarks</p>	<p>*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-120S.          *When ASC-120S is used, the boat to be used will be a ceramic boat, TX3SCX.</p>												

\*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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