

Sheet No.

AQF CH 010E Materials

Determination of chlorine and sulfur in 3,5-dichloro-2-hydroxybenzenesulfonic acid sodium salt

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	Sodium 3,5-dichloro-2-hydroxy benzene sulfonate																																				
Sample status																																					
Measuring items	Chlorine(Cl), Sulfur (S)																																				
Measurement principle	Sample is thermally decomposed in argon (Ar) atmosphere, then combusted in oxygen (O ₂) 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	<div>1. AQF-100<div>Sample size : mg</div><div>Sample boat : Quartz sample boat, TX2SBT</div><div>Additive : WO₃ 100mg</div><div>Pyrolysis tube : Quartz tube filled with quartz wool</div><div>Absorbent : 90ppm Hydrogen peroxide / water</div><div>Heater Temp. Inlet : 900degC</div><div>Outlet : 1000 to 1100degC</div><div>Gas flow Ar : 200 ml/min</div><div>O₂ : 400 ml/min</div></div> <div>GA-100<div>Absorbent volume : 10 ml</div><div>Sampling loop : 100 ul</div><div>Absorption tube : For 10 ml</div><div>Water supply : 4</div><div>Ar flow for water supply : 150 ml/min</div></div> <div>ABC-100/ASC-120S<table><tr><td></td><td></td><td>1st</td><td>2nd</td><td>3rd</td><td>4th</td><td>5th</td><td>End</td><td>Cool</td></tr><tr><td>Position</td><td>(mm)</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr><tr><td>Time</td><td>(sec)</td><td></td><td></td><td></td><td></td><td></td><td>90</td><td>60</td></tr><tr><td>Speed</td><td>(mm/sec)</td><td></td><td></td><td></td><td></td><td></td><td>20</td><td>40</td></tr></table></div> <div>Ar Time 0 (sec) O₂ Time 300(sec)</div>			1st	2nd	3rd	4th	5th	End	Cool	Position	(mm)								Time	(sec)						90	60	Speed	(mm/sec)						20	40
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	2. Ion chromatograph Ion chromatograph : DIONEX ICS-1500 Column : DIONEX Ion Pack AG12A / Ion Pack AS12A Eluent : 2.7mM Na ₂ CO ₃ / 0.3mM NaHCO ₃ Eluent flow : 1.50ml / min Detector : Conductivity Suppressor : ASRS-4-mm Measuring time : 15min Sampling loop : 100 ul using GA-100 sampling loop Calibration : F Cl Br S : 5ppm ~ 40ppm														
Results	<p>Theoretical value: Cl=26.75% S=12.10%</p> <table><tr><th rowspan="2">Conditions</th><th colspan="2">Results</th></tr><tr><th>Cl,%</th><th>S,%</th></tr><tr><td>900/1000℃</td><td>26.51</td><td>5.86</td></tr><tr><td>900/1100℃</td><td>26.41</td><td>5.95</td></tr><tr><td>900/1100℃+WO₃</td><td>26.75</td><td>11.92</td></tr></table> <p>*Result of Sulfur measurement became better by mixing WO₃ with sample. We suppose sodium sulfate is converted to sulfur oxide by WO₃. Na₂SO₄ + WO₃ . Na₂WO₄ + SO₃</p>	Conditions	Results		Cl,%	S,%	900/1000℃	26.51	5.86	900/1100℃	26.41	5.95	900/1100℃+WO ₃	26.75	11.92
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Remarks	<p>*Handling of reagents: Confirm labels and safety data sheets of reagents and handle them with enough care.</p> <p>*Automation is possible by using an Automatic Sample Changer ASC-120S.</p> <p>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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