

Sheet No.

AQF PT 008E Pharmaceuticals &amp; Cosmetics

**Determination of bromine in polyvinyl chloride resin** 1/2

Instruments : AQF-100

Method : Combustion-ion chromatography

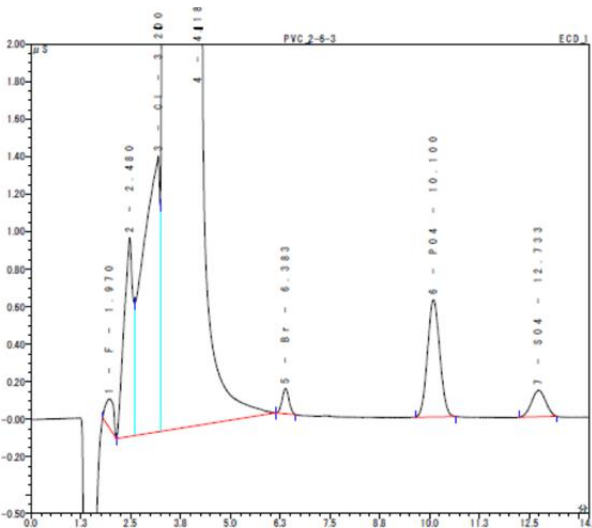
Related standard :

For plastics which contain flame retardant, it is important to know the Bromine content as a main component. 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           | Polyvinyl chloride resin  |     |     |     |     |     |     |      |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
|-----------------------|---|-----|-----|-----|-----|-----|-----|------|-----|------|----------|------|-----|-----|-----|--|--|--|--|------|-------|-----|-----|-----|--|--|-----|----|-------|----------|--|--|--|--|--|--|--|
| Sample status         |   |     |     |     |     |     |     |      |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
| Measuring items       | Bromine (Br)  |     |     |     |     |     |     |      |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
| 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).<br><b>Analyzing flow</b><br>[Sample weighing]→[Combustion]→[Collection of combustion gas]→[IC analysis]   |     |     |     |     |     |     |      |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
| Parameters            | <p><b>1. AQF-100</b></p> <p>Sample size : 20mg<br/> Sample boat : Ceramic sample boat, SXSMBS<br/> Additive : Not used<br/> Pyrolysis tube : Quartz tube filled with quartz wool<br/> Absorbent : Hydrogen peroxide / water<br/> Mode :</p> <p>Heater Temp. Inlet : 900degC<br/> Outlet : 1000degC<br/> Gas flow Ar : 200 ml/min<br/> O<sub>2</sub> : 400 ml/min</p> <p>GA-100 Absorbent volume : 10ml<br/> Sampling loop : 100 ul<br/> Absorption tube : For 10 ml<br/> Water supply : 1<br/> Ar flow for water supply : 150 ml/min</p> <p>ABC-100/ASC-120S</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>140</td> <td>150</td> <td>160</td> <td></td> <td></td> <td></td> <td></td> </tr> <tr> <td>Time</td> <td>(sec)</td> <td>120</td> <td>120</td> <td>120</td> <td></td> <td></td> <td>300</td> <td>60</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 600(sec)</p> |     |     | 1st | 2nd | 3rd | 4th | 5th  | End | Cool | Position | (mm) | 140 | 150 | 160 |  |  |  |  | Time | (sec) | 120 | 120 | 120 |  |  | 300 | 60 | Speed | (mm/sec) |  |  |  |  |  |  |  |
|                       |   | 1st | 2nd | 3rd | 4th | 5th | End | Cool |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
| Position              | (mm)  | 140 | 150 | 160 |     |     |     |      |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
| Time                  | (sec)   | 120 | 120 | 120 |     |     | 300 | 60   |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |
| Speed                 | (mm/sec)  |     |     |     |     |     |     |      |     |      |          |      |     |     |     |  |  |  |  |      |       |     |     |     |  |  |     |    |       |          |  |  |  |  |  |  |  |

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**AQF PT 008E** Determination of bromine in polyvinyl chloride resin \_\_\_\_\_ 2/2

|                |  |
|----------------|--|
|                | <p><b>2. Ion chromatograph</b></p> <p>Ion chromatograph : DX 320<br/>         Column : DIONEX Ion Pack AG12A / Ion Pack AS12A<br/>         Eluent : 2.7mM Na<sub>2</sub>CO<sub>3</sub> / 0.3mM NaHCO<sub>3</sub><br/>         Eluent flow : 1.50ml / min<br/>         Detector : Conductivity<br/>         Suppressor : ASRS-4-mm(Ultra II)<br/>         Measuring time : 15min<br/>         Sampling loop : 50 ul using GA-100 sampling loop<br/>         Calibration : F Cl Br S :0.1ppm to 5.0ppm</p> |
| <p>Results</p> | <p><b>Chromatogram</b></p>    |
|                | <p><b>Results</b></p>  |
| <p>Remarks</p> | <p>*Handling of reagents: Confirm labels and safety data sheets of reagents and handle them with enough care.<br/>         *Automation is possible by using an Automatic Sample Changer, ASC-120S.<br/>         *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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