Sheet No.
AQF PT 016E Reference Materials

Determination of chlorine, bromine and sulfur in polyethylene certified reference material (2)

Instruments : AQF-2100H System,HF-210,GA-210,ABC-210/ASC-240S
Method : Combustion-ion chromatography
Related standard :

Polyethylene is widely used for containers and packaging materials. As halogen compounds may be added to polyethylene materials for various purposes, it is critically important to know the content of the halogen compounds when polyethylene materials are disposed or recycled.
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	ERM-EC680k (Reference sample certified by IRMM)
Sample status	Resin pellet
Measuring items	Chlorine (Cl), Bromine (Br), 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	**1. AQF-2100H** Sample size : 15 to 30mg (2 to 3 pellets) Sample boat : Ceramic sample boat, SXSMBS Additive : None Pyrolysis tube : Quartz tube filled with quartz wool Absorbent : 300ppm Hydrogen peroxide / water Mode : Constant volume mode Heater Temp. Inlet : 900degC Outlet : 1000degC Gas flow Ar : 200 ml/min O_2 400 ml/min GA-210 Absorbent volume : 10 ml Sampling loop : 100 ul Absorption tube : For 10 ml Water supply : 2 Ar flow for water supply : 100 ml/min ABC-210/ASC-240S

		1st	2nd	3rd	4th	5th	End	Cool
Position	(mm)	100	160					
Time	(sec)	90	90				300	60
Speed	(mm/sec)	10	0.12				10	40

Ar Time 0 (sec) O_2 Time 300(sec)

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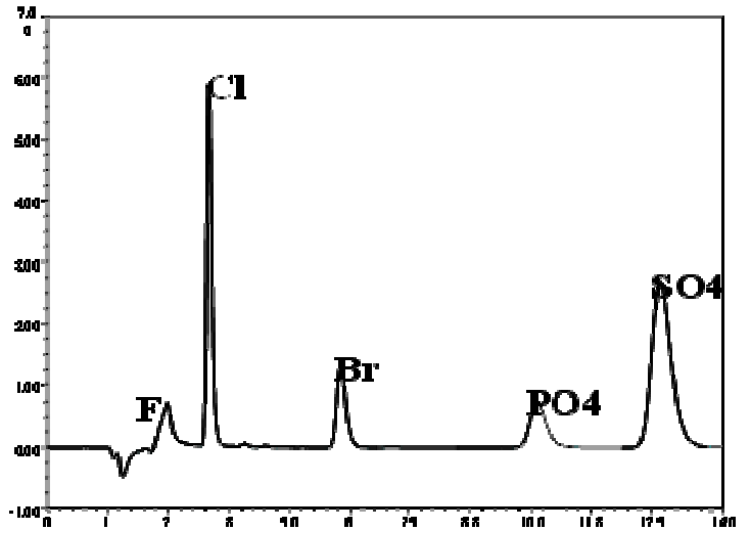
2. Ion chromatograph

Ion chromatograph	: DIONEX ICS-1500
Column	: DIONEX Ion Pack AG12A / Ion Pack AS12A
Eluent	: 2.7mM Na_2CO_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-210 sampling loop
Calibration	: F Cl Br S :0.1ppm to 5.0ppm

Results

Chromatogram



Results

	1	2	3	Average (ppm)	RSD (%)	Certified value (ppm)
Cl	104	105	106	105	0.95	102.3+/-3
Br	96.9	95.5	97.4	96.6	1.0	96+/-4
S	73.8	72.8	75.5	74.0	1.8	76+/-4

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, TX2SCX.
*Use an internal standard material other than phosphate ion (PO_4^{3-}) when analysis is performed by the internal standard method.