SHIMADZU

Application News

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Plastic

■ Description

An ICP emission spectrometer can be used to accurately measure low concentrations of harmful metals like lead and cadmium in plastics. Decomposition of plastic samples for analysis is conducted by the dry-ashing, wet decomposition or microwave digestion method. This application introduces the analysis of plastic using the multi-type ICPE-9000 ICP emission spectrometer.

■ Sample

BCR680, 681 (Polyethylene standard)

■ Pretreatment: Dry-ashing

(1) Shave off a very small piece of the sample, and weigh out 0.2 g into a quartz crucible.

(2) Add sulfuric acid to soak the sample, and heat on a hot plate until no SO_3 (white) fumes are produced.

(3) Place the residue in an electric furnace, and ash it at 450°C.

(4) After ashing, add 5 mL hydrochloric acid (1:2) to the residue, and evaporate it on a water bath until it is dry.

(5) Add 10 mL of 1 mol/L nitric acid, and dissolve it on a hot plate.

(6) After cooling, measure out 20 mL of the solution.

(Reference: Food Hygiene Test Guidelines, 2005 Physics and Chemistry Vol., Ministry of Health, Labor and Welfare)

■ Pretreatment: Wet Decomposition

(Decomposition using Kjeldahl Flask)

(1) Shave off a very small piece of the sample, and weigh out 0.2 g into a Kjeldahl flask.

(2) Add sulfuric acid, nitric acid and hydrogen peroxide, and decompose the sample by heating on a mantle heater (about 300°C) to carbonize (turn black) the contents and drive off the SO_3 fumes (white).

(3) After the solution turns black, add nitric acid and hydrogen peroxide, and continue heating (about 350°C). Repeat this procedure until the contents turns light yellow in color.

(4) Cool the flask, and measure out 20 mL of the solution.

(Reference: BS EN1122 Method A: 2001)

■ Pretreatment: Microwave Decomposition

(1) Shave off a very small piece of the sample, and weigh out 0.2 g into a PTFE decomposition vessel.

(2) Add nitric acid and hydrogen peroxide, seal the vessel, and perform microwave heating to achieve pressurized digestion.

(3) After cooling the decomposition vessel, measure out 20 mL of digested solution.

*For samples containing large amounts of additives or coexisting constituents, use hydrofluoric acid.

(Reference: US EPA SW-846 Method3052)

■ Analytical Conditions

Instrument	**:**	**ICPE-9000**
Radio Frequency Power	:	1.2 (kW)
Plasma Gas	:	10 (L/min)
Auxiliary Gas	:	0.6 (L/min)
Carrier Gas	:	0.8 (L/min)
Sample Introduction	:	Nebulizer 10
Sample Aspiration	:	1.0 (mL/min)
Misting Chamber	:	Cyclone Chamber
Attached Instruments:	:	Mini Torch
View Direction	:	Axial

■ Measurement Results

Table 1 shows the quantitation results and detection limits of the elements in the plastic sample. Good results were obtained for lead and cadmium in the dry-ashing method, cadmium, total chromium and mercury in the Kjeldahl method, and all elements in the microwave method, with the quantitation values matching the certified standard values. Fig. 1 shows the spectral profiles, and Fig. 2 the calibration curves.

Table 1: Polyethylene Resin Quantitation Results (Unit: mg/kg) (Concentration in Solid Sample)

Sample	BCR680				BCR681				*
Pretreatment / Element	Dry Method	Wet Method	MW Method	Certified Value	Dry Method	Wet Method	MW Method	Certified Value	Detection Limit (3σ)
Cd	141	140	140	140.8	21.0	21.4	21.7	21.7	0.02
Pb	105	< 0.2	108	107.6	13.1	< 0.2	13.5	13.8	0.2
Cr	105	112	112	114.6	16.2	17.2	17.5	17.7	0.03
Hg	< 0.2	24.0	25.6	25.3	< 0.2	4.3	4.6	4.5	0.2
As	28	31	30	30.9	4	4	5	3.93	0.5

* Detection limit when conducting pretreatment using 0.2g/20mL dilution

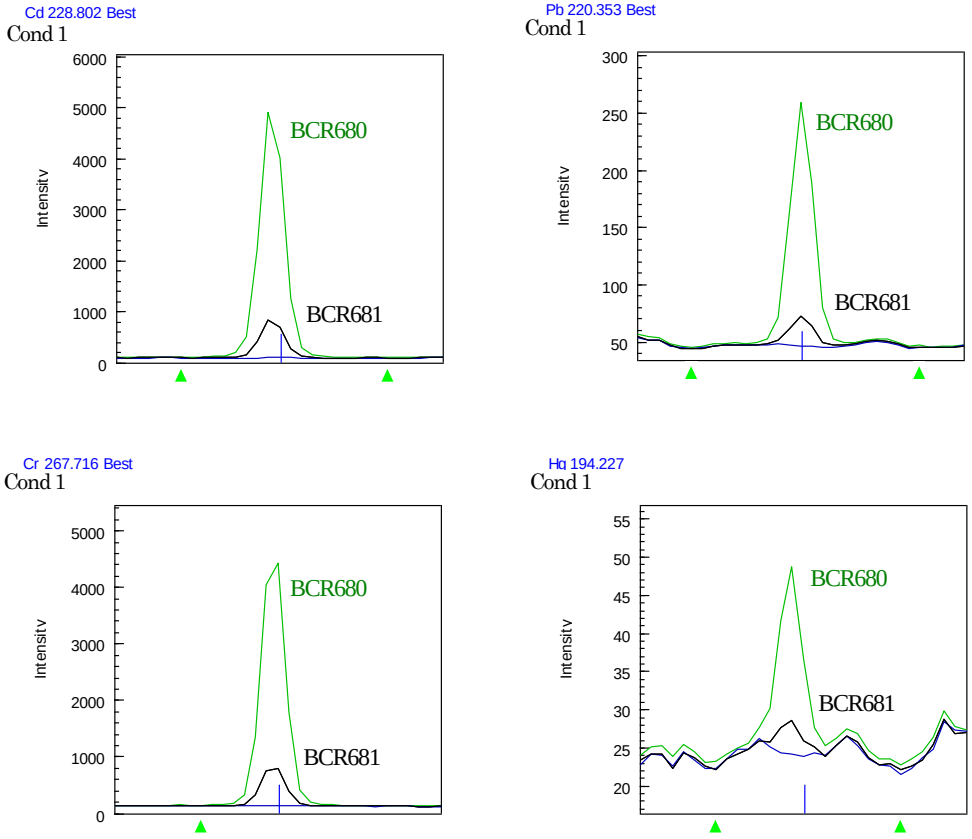

Figure 1: Spectral Profiles

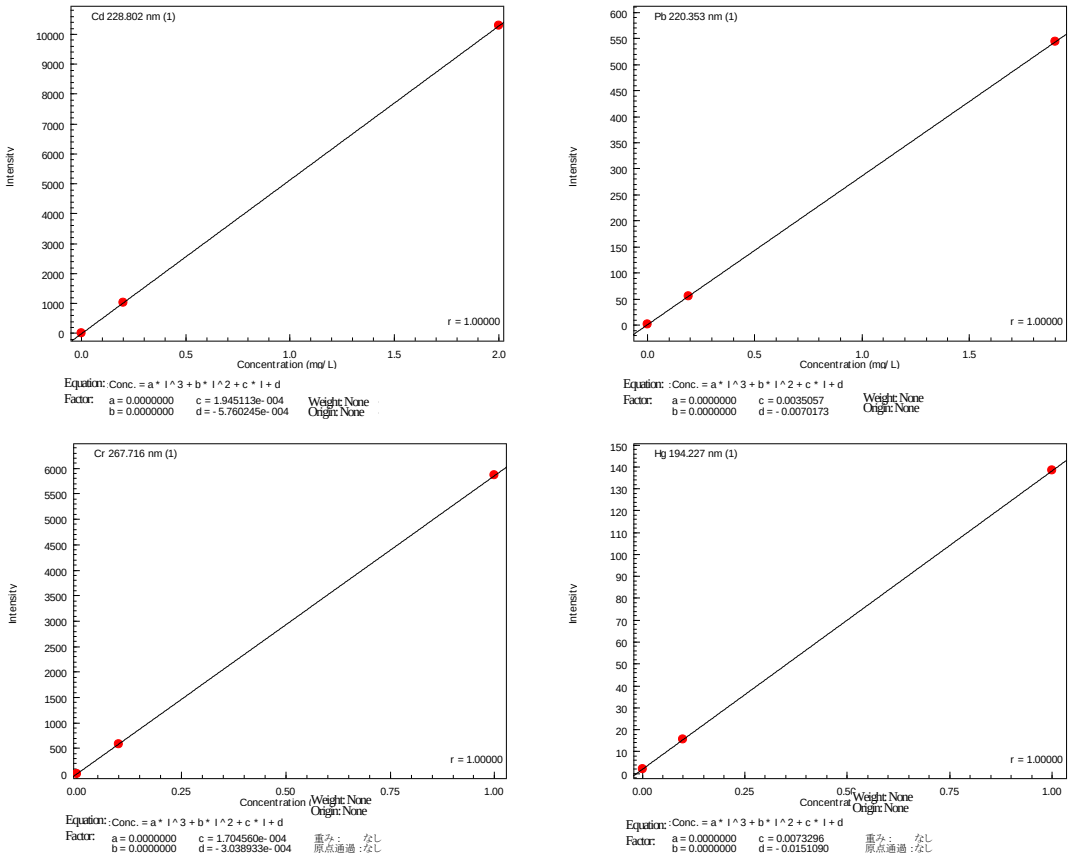

Figure 2: Calibration Curves

Shimadzu Scientific Instruments
7102 Riverwood Drive, Columbia, MD 21046
Phone: 800-477-1227, Fax: 410-381-1222
www.ssi.shimadzu.com
webmaster@shimadzu.com