

# Application News

Inductively Coupled Plasma Atomic Emission Spectrometry

No. J93

## Analysis of Br in Plastic by ICP-AES

In modern society where the recycling of energy resources has become a priority, it is needless to say that recycling of the huge quantities of generated plastics waste or recycling it as an energy resource is an important matter. However, some portion of plastic waste contains brominated flame retardants, and understanding of the quantity of bromine contained in these plastics from the standpoint of environmental control is important for the promotion of appropriate recycling processing.

In 2009, the Japan Industrial Standard (JIS) K7392 "Waste Plastics-Test Method for Total Bromine Contents", regarding the analysis of bromine in

plastics, was established. According to this standard, the bromine contained in plastic is gasified through combustion, absorbed into an absorption solution, and that solution is submitted to measurement by ion chromatography to determine the bromine content.

However, bromine in a liquid sample can also be measured using ICP emission by analysis with an instrument capable of short-wavelength measurement in the vacuum ultraviolet region. Here, we introduce an example of measurement of such an absorption solution, obtained by the sample combustion preparation method, using an ICP emission spectrometer.

### ■ Sample Preparation

In the above-mentioned JIS K7392 (2009), a plastic sample is combustion-oxidized using an oxidation/combustion test instrument, and the generated hydrogen bromide (HBr) is collected in an absorption solution.

The absorption solution is prepared by dissolving 0.0252 g sodium hydrogen carbonate and 0.2862 g sodium carbonate (anhydrous) in 3.33 g of 30 % hydrogen peroxide solution, and then bringing the final volume to 1000 mL using pure water.

The sample preparation described here was for an approximate bromine concentration of 0.01 to 1 %, and we used 50 mg of sample and 50 mL of absorption solution.

The bromine collection solution obtained using this combustion method can be used as the ICP-AES sample solution as is.

### ■ Standard Solutions for Calibration Curve

The calibration curve standard solutions were prepared by adding an aqueous solution of potassium bromide (KBr) as the bromine standard to the same composition absorption solution described for sample preparation. The solutions were prepared with bromine concentrations in the range of 0 to 4 mg/L. This corresponds to a 0 to 0.4 % concentration range in plastic.

### ■ Equipment

Bromine has a high-sensitivity measurement wavelength at 154.065 nm. Although the Shimadzu ICPS-8000 series is equipped with a vacuum monochromator which supports vacuum ultraviolet wavelengths below vacuum ultraviolet 190 nm, the typical measurement wavelength is from 160 nm. The improved optical system of the ICPS-8100<sub>CL</sub> achieves measurement from 134 nm, permitting measurement of such halogen elements as bromine and chlorine (134.724 nm).

Here, we used this ICPS-8100<sub>CL</sub> as the analytical instrument.

Table 1 Specifications of Spectrometer (ICPS-8100<sub>CL</sub>)

	First Monochromator	Second Monochromator
Focal Distance	1 m	1 m
Diffraction Grating	4960 lines/mm	4320/1800 lines/mm
Wavelength Range	134 nm – 372 nm	250 nm – 850 nm

### ■ Analytical Conditions

Instrument	: ICPS-8100 <sub>CL</sub>
RF output	: 1.2 kW
Plasma gas flowrate	: 14 L/min
Auxiliary gas flowrate	: 1.2 L/min
Carrier gas flowrate	: 0.7 L/min
Sample introduction	: Coaxial nebulizer
Spray chamber	: Cyclone chamber
Plasma torch	: Torch
Observation method	: Radial
Observation height	: 11 mm

## ■ Analysis

As plastic samples, we used the reference materials BCR680 and BCR681 certified by IRMM (Institute for Reference Materials and Measurements) of the EU. BCR680 and BCR681 are polyethylene resin standard materials used for trace element analysis. In addition to metals, they contain bromine (Br), and are provided

with certified values.

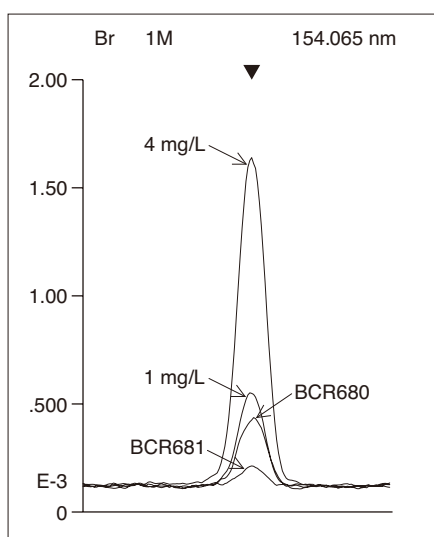
The BCR sample analytical results are shown in Table 2. The results corresponded quite well with the certified values.

The Br 154.065 nm spectral line profile is shown in Fig. 1, and the calibration curve is shown in Fig. 2.

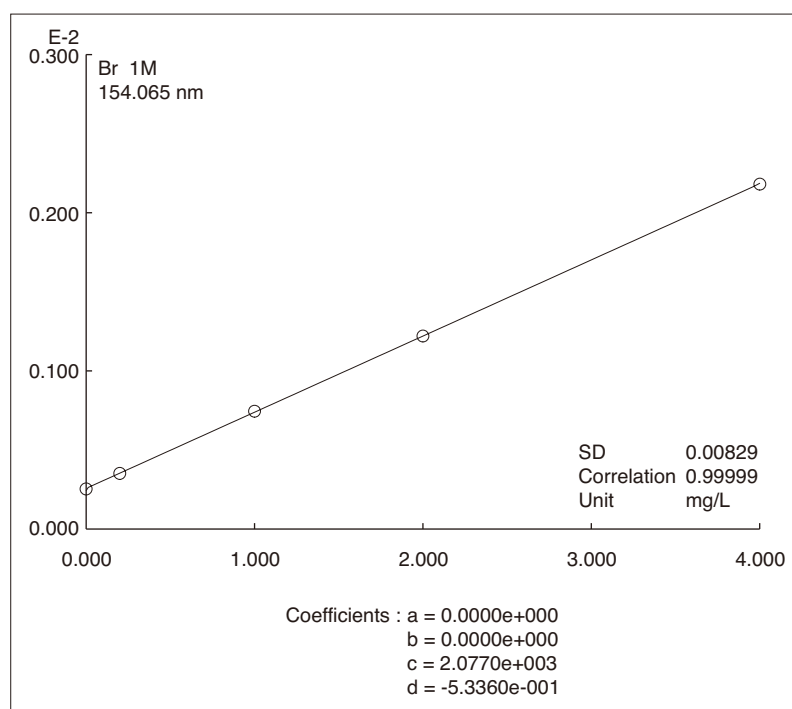
**Table 2 Results of BCR Samples**

	BCR680		BCR681	
	Analysis Value	Certified Value	Analysis Value	Certified Value
Br	810	808 ± 19	99	98 ± 5

Unit: mg/kg



**Fig. 1 Spectral Profile of Br (154.065 nm)**



**Fig. 2 Calibration Curve of Br**

[Reference]

JIS K7392: 2009 "Waste Plastics - Test Method for Total Bromine Contents"