

Application News

High Performance Liquid Chromatography

No.L418

Analysis of Fluorine in Iron and Steel Slag by Combustion – Ion Chromatography

Iron and steel slags contain blast furnace slag produced in making pig iron from iron ore, and steel slag formed from the decarburization of pig iron and the removal of impurities for imparting stickiness in the converter furnace. The iron and steel slags produced as byproducts of the steelmaking process, amounting to several tens of million tons per year, contain fluorine due the addition of fluorite (CaF_2) as a flux for the melting process. Compliance with various standards

and criteria^{1), 2)} is required when iron and steel slags are to be used in road building materials, ground improvement materials, concrete mixture materials, etc., and management of ingredient concentrations is important.

Here, we introduce an example of analysis of fluorine in iron and steel slag using the Shimadzu combustion – ion chromatography system.

■ Combustion – Ion Chromatography System

Sample combustion / decomposition and analysis were conducted using a combustion – ion chromatography system comprised of a combustion device with dual-layer reaction tube (with inner ceramic tube), within which the sample is combusted in a wet atmosphere (Fig. 1), and the effluent connected directly to an ion chromatograph.

The sample was placed in the inner tube of the dual-layer reaction tube and heated to 1100 °C while an Ar/O_2 gas mixture flowed continuously through the inner tube. The evolved gas was collected using absorption liquid which was automatically analyzed by the ion chromatograph, thereby reducing the risk of contamination during the analysis process.

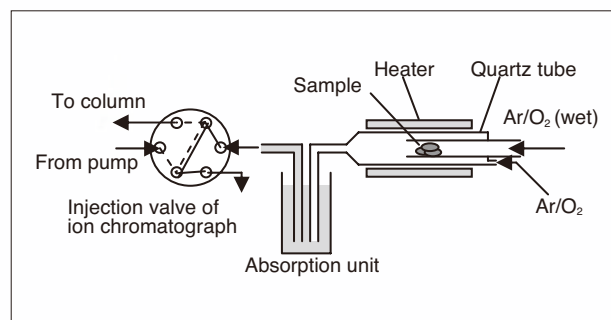


Fig. 1 Combustion Device

■ Analytical Conditions for Ion Chromatography

The fluoride ion is typically eluted quickly and is easily influenced by the water dip generated during sample injection, so method development is often a challenge for optimum fluoride quantitation.

Slag analysis can be conducted effectively using the analytical conditions developed here, because the fluoride ion is sufficiently retained, and the sulfate ion generated from the sulfur present in the slag is quickly eluted. (The phosphate acid ion is the internal standard.)

Table 1 Analytical Conditions

Column	: Shim-pack IC-SA2 (250 mm L.×4.0 mm I.D.)
Mobile Phase	: 12 mmol/L sodium bicarbonate 0.6 mmol/L sodium carbonate
Flow Rate	: 1.0 mL/min
Column Temp.	: 30 °C
Detection	: CDD-10A _{SP} (suppressor)
Injection Volume	: 50 µL

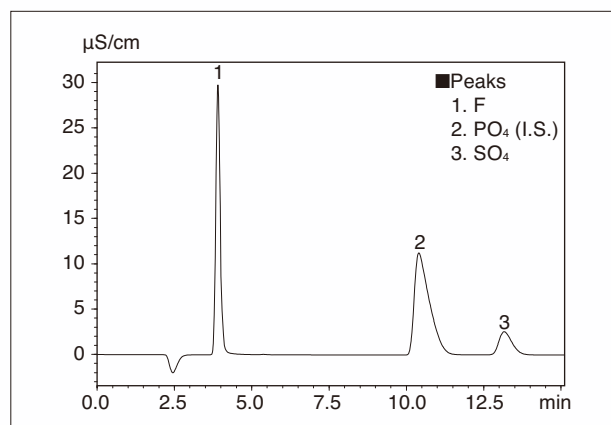


Fig. 2 Separation of 3 Anions

■ Calibration Curve of Standard Solution

Fig. 3 shows the overlaid chromatograms of fluoride standard solutions with fluoride concentrations from 0.1 to 10 mg/L, and Fig. 4 shows the calibration curve.

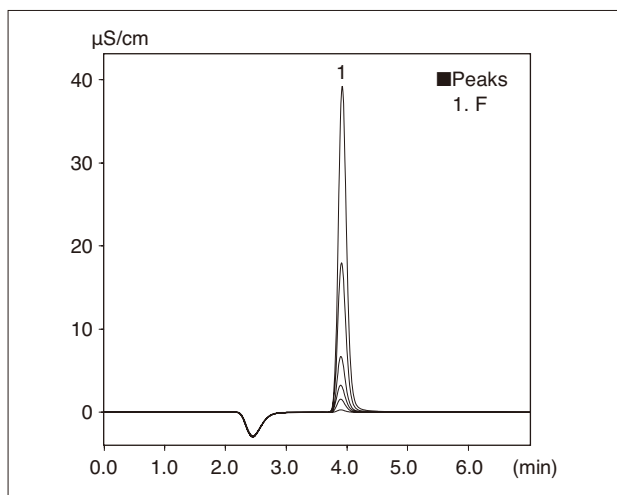


Fig. 3 Chromatograms of Fluoride Standard Solutions(0.1 – 10 mg/L)

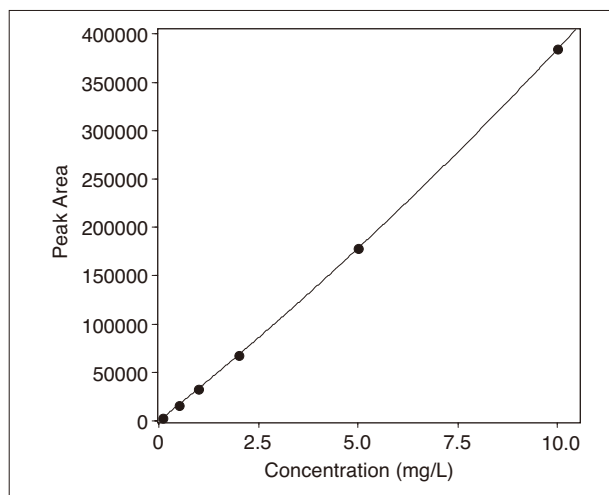


Fig. 4 Calibration Curve of Fluoride (0.1 – 10 mg/L)

■ Analysis of Slag Samples

Since the slag samples have a melting point higher than 1000 °C, quantities up to 100 mg of tungsten oxide (WO₃) were mixed in with the finely-ground samples, and combustion / decomposition with O₂ was conducted while wet Ar gas flowed continuously over the samples.

The combustion conditions are shown in Table 3, and Figures 5 and 6 show the chromatograms obtained from analysis of the samples. This method permits analysis of fluorine in iron and steel slags at concentrations ranging in levels from ppm to %.

Table 3 Combustion Conditions

Combustion Furnace	: 1100 °C
Flow Gas	: Ar/O ₂
Additive	: WO ₃ (100 mg)
Absorption Liquid	: Pure H ₂ O (I.S. : Internal Standard: PO ₄)
Equipment (Mitsubishi Chemical Analytech)	
Combustion	: AQF-2100H
Absorption	: GA-210

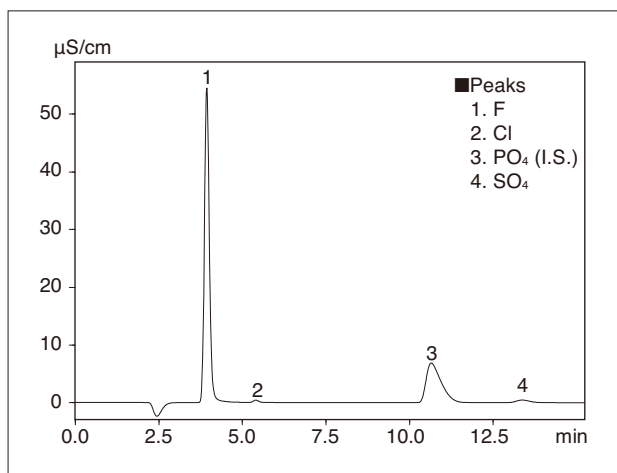


Fig. 5 Chromatogram of Slag Sample A

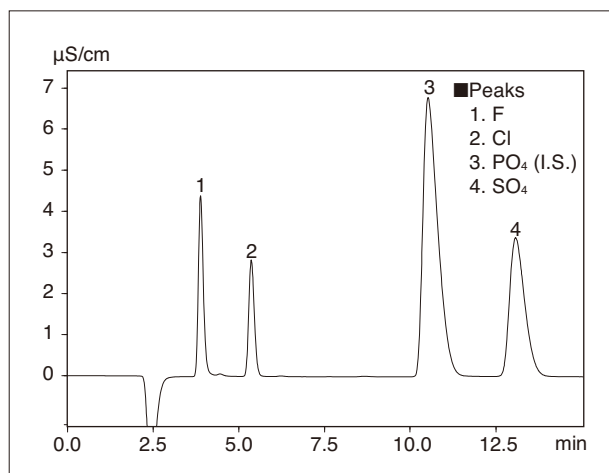


Fig. 6 Chromatogram of Slag Sample B

[References]

- 1) "Notification of Amendments Relating to the Promotion of the Implementation of the Recycling of Solidified Molten Materials in Municipal Waste" (October 2, 2009, Japan's Ministry of Environment, Notice about Environmental Waste No. 091002001)
- 2) Japan Testing Center for Construction Materials Standard JSTM H8001:2008, "Steel Slag Crushed Stone for Earthworks" (Japan Testing Center for Construction Materials)



SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710

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