JFE TECHNICAL REPORT
No. 9 (Mar. 2007)

Determination of Trace Amounts of Mercury, Lead, and Cadmium in Steel and Iron Ore[†]

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Abstract:

Trace amounts of mercury in steel and iron ore were determined by direct vaporization from solid sample and reduction vaporization from digested sample solution with cold-vapor atomic absorption spectrometry. The direct method has been applied to the determination of mercury in iron ore in the level of ng/g with satisfactory precision equal to the reduction method. However, mercury in steel could not be detected by the both methods. Trace amounts of mercury in steel, lead, and cadmium in the level of ng/g in steel have been determined by inductively coupled plasma-mass spectrometry (ICP-MS) after cation exchange chromatographic separation. The sample was decomposed with nitric acid and separated from iron matrix in 1 M hydrofluoric acid solution, using the cation exchange resin that were packed in a mini-column. The elements adsorbed on the resin were eluted by 6 M hydrochloric acid solution and determined by ICP-MS simultaneously. The lower limits of detection based on 3σ of blank values were 2 ng/g for mercury, 9 ng/g for lead, and 0.5 ng/g for cadmium in steel.

1. Introduction

Environmental controls focused on home electric appliances and automobiles are now advancing in Europe. In accordance with the WEEE (Waste Electrical and Electronic Equipment) directive and RoHS (Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment) (Dec. 2002), the manufacturers of home electric appliances are now prohibited from using four heavy metals (mercury, lead, cadmium, and hexavalent chromium) and two bromine-base fire retardants in their products. In principle, the EUELV (EU End-of-Life Vehicle) directive established in Oct. 2000 prohibits automakers from using lead, mercury, cadmium, and hexavalent chromium in newly sold vehicles. In Japan, Law for the Recycling of End-of-Life Vehicles established in July 2003 calls for enhanced monitoring of automotive components with environmental loads.

In addition to the restrictions on the products themselves, there have also been enhancements in the regulation of environmental loads in manufacturing processes. The discharge of trace elements is now being closely monitored. Annual mercury emissions, for example, recently reached 48 t or more in the U.S. This compelled U.S. regulatory authorities to call for a 90% reduction in mercury emissions from coal-fired power plants by 2008 through strict enforcement of the Clean Air Act. Japan is still working out the levels to which different industries will be required to abate their emissions under the revised Air Pollution Control Law. Nevertheless, the Japanese restrictions on mercury emissions from mainly coal-fired power plants are very likely to be tightened in the future. Japanese steel companies, meanwhile, are now required “to clearly disclose data on the concentrations of substances with environmental loads” and “to pledge and ensure chemical substance control” in the production of steel products.

In accordance with recent laws on the reporting, etc. of the release of specific chemical substances and on management improvements (the law for PRTR (pollutant release and transfer register) and the promotion of chemical management) issued in July 1999, Japanese steel companies will probably be required to clearly disclose the transition and balance of environmentally harmful substances in steel manufacturing.

The Japanese Industrial Standard (JIS) for the analysis of environmentally harmful elements in steel speci-

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[†] Originally published in *JFE GIHO* No. 13 (Aug. 2006), p. 35–41

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fies methods for the analysis of lead, an element with a great effect on the properties of steel. Spectrophotometry after diphenylcarbazone extraction1) can be used to analyze trace amounts of lead of up to 2 μg/g in steel. Similarly, atomic absorption spectrometry after iodide extraction2) can be used to analyze trace amounts of lead of up to 5 μg/g in steel. Trace amounts of lead in the range from 10 to 100 ng/g in steel can be determined by combining methods of separation and concentration from iron matrix such as solvent extraction3,4) and ion exchange chromatography5,6), and also by highly sensitive detection methods such as atomic absorption spectrometry, inductively coupled plasma atomic emission spectrometry (ICP-AES), and inductively coupled plasma mass spectrometry (ICP-MS). For the analysis of cadmium in steel, trace amounts up to the nearest sub μg/g can be analyzed by the same methods used for lead. For the analysis of mercury, analytical methods based on the reduction evaporation/cold vapor atomic absorption spectrometry, thermal vaporization/cold vapor atomic absorption spectrometry, etc. have yielded few promising results when applied to steel. All, however, have been applied with some degree of success to environmental samples7), biological samples8), etc. As of this writing, there have been no close examinations or detailed reports on the mercury analysis of steel samples or related problems such as the volatilization during sample preparation.

In the present study the authors investigated a method to determine trace levels of mercury, a critical target of environmental controls, in steel and iron ore. The authors also determined the levels (ng/g) of mercury, lead, and cadmium in steel simultaneously by ICP-MS after ion exchange chromatography of each element in the specimens tested. The results of our investigations9) are reported below.

2. Experiment

2.1 Apparatus

(1) Mercury Analyzers

A thermal vaporization mercury measuring device was used to directly analyze solid samples and a reduction evaporation mercury analyzer was used to determine the mercury levels in digested sample solutions. The thermal vaporization analyzer works by vaporizing mercury in the sample with heat in a tubular furnace and then amalgamating the mercury with gold and collecting it as amalgam. After reheating the amalgam to separate the mercury, the absorbance of the mercury is measured by an atomic absorption spectrometer with a quartz cell. The reduction evaporation method works by adding a reducing agent (tin(II)-chloride) to a sample solution to induce a reduction evaporation, then measuring the absorbance of the mercury in the evaporated vapor as it passes through a quartz cell in the high pass of a mercury hollow cathode lamp. Both devices measured the absorbance at a wavelength of 253.7 mm.

Table 1 Operating conditions for ICP-MS

ICP conditions	RF power Plasma gas flow rate Auxiliary gas flow rate Nebulizer gas flow rate	1.4 kW 15.5 *l*/min 1.0 *l*/min 1.15 *l*/min
Mass spectrometric conditions	Mode Integration time	Peak jump mode 0.3 s/mass × 3 times

(2) ICP Mass Spectrometer

The ICP mass spectrometry was performed with a quadrupole type ICP-MS, with correction of the drifts of signal intensity using ^{115}In as an internal standard. The ICP conditions and mass spectrometric conditions are shown in **Table 1**.

2.2 Reagents

(1) Standard Solution and Reagents

The investigation of recovery from ion-exchange and the preparation of calibration curves were performed using mercury, lead, and cadmium standard solutions procured from Kanto Chemical Co., Inc. A reagent for semiconductors made by Hirota Chemical Industry Co., Ltd. was used for hydrofluoric acid. Reagents for electronic engineering from Kanto Chemical Co., Inc. were used for hydrochloric acid, nitric acid, and the hydrogen peroxide solution.

(2) Ion-Exchange Resins

A cation-exchange resin DOWEX® 50W-X8 (100–200 mesh) and anion-exchange resin DOWEX® 1-X8 (100–200 mesh) were packed in a polypropylene column (capacity: 2.5 m*l*) and used in that condition.

2.3 Experimental

(1) Mercury Analysis by Thermal Vaporization

A steel sample of about 400 mg cut in chip form and a sample of iron ore of several tens of milligrams were weighed directly into a ceramic boat, respectively heated at 600°C for 4 min and 970°C for 6 min using the apparatus described in paragraph 2.1 (1) above, and tested to determine the quantity of vaporized mercury. The calibration curves were prepared by placing a quartz boat in the ceramic boat, adding a standard mercury solution dropwise to the quartz boat, and vaporizing the mercury with heat under the same conditions used for the preparation of analytical samples.

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* DOWEX® is a registered trademark of Dow Chemical Company.

(2) Mercury Analysis by Reduction Evaporization

Mercury analyses were performed based on nitric acid-potassium permanganate reflux decomposition, a method for total mercury determination described in section 5.14.1.1 of “Bottom Sediment Investigation Methods,”[10)] a report issued by the Ministry of the Environment in 2001. After the addition of 50 m*l* of nitric acid (1 + 1) to 2 g of each sample, the samples were heated and dissolved in a constant temperature bath at 95°C for 2 h, cooled to room temperature, and then modified by adding 20 m*l* of a 3% potassium permanganate solution, heating in a constant temperature bath at 95°C for 1 h, adding 10 m*l* of a 10% urea solution, and finally adding a 20% hydroxylammonium chloride solution dropwise until the purplish red color disappeared. Next, the residues were separated by filtration with a glass filter (pore diameter: 1 μm), the solution was diluted with pure water to 250 m*l*, a 20 m*l* portion was taken, and 1 m*l* of sulfuric acid (1 + 1) was added. The mercury was determined by the mercury analyzer described in paragraph 2.1 (1) above.

(3) Analysis of Mercury, Lead, and Cadmium by Ion-Exchange Separation/ICP-MS

A steel sample of 0.1 g was dissolved in 0.5 m*l* of nitric acid (1 + 1) at room temperature. After adding 1.5 m*l* of hydrofluoric acid (1 + 1) and 0.5 m*l* of a hydrogen peroxide solution, the sample was completely decomposed by heat at 120°C. The sample solution was then diluted with 20 m*l* of 1M hydrofluoric acid and treated in the column with 1 m*l* of (DOWEX® 50W-X8 (100–200 mesh) 1 m*l*) until the analytes were adsorbed on the resin. After washing with 1 M hydrofluoric acid, the analytes were eluted with 5 m*l* (2.5 m*l* × 2) of 8 M hydrochloric acid and 50 ng of indium with 5 m*l* was added. The ion intensities of ^{114}Cd, ^{205}Hg, and ^{208}Pb were measured by ICP-MS using ^{115}In as an internal standard, then converted to contents using calibration curves prepared with standard solutions.

3. Results and Discussion

3.1 Direct Determination of Mercury by Thermal Vaporization/Atomic Absorption Spectrometry

The authors used the thermal vaporization analyzer described in paragraph 2.1 (1) above to test the utility and feasibility of the analyzer as a simplified method for the high-accuracy analysis of mercury in steel and raw materials. The linearity of the calibration curves from less than 1 ng to about 200 ng was good, and the lower limit of detection, defined as the concentration equal to 3 times the standard deviation of blank values, was 0.08 ng.

Table 2 Analytical results of mercury in commercial steel and iron ore (CRM) by thermal vaporization and reduction evaporation/cold-vapour atomic absorption methods (CV-AAS)

(n = 3)

Sample	Thermal vaporization/CV-AAS			Reduction evaporation/CV-AAS	
	Sample weight (mg)	Mean (μg/g)	σ (μg/g)	Mean (μg/g)	σ (μg/g)
Commercial steel	400	Not detected	–	Not detected	–
Iron ore (JSS 820-2)	50	0.012	0.000 9	0.009	0.001 0

Table 2 shows the analytical results of this method. The authors verified the accuracy of this method by preparing the samples for analysis of the bottom sediments (described in paragraph 2.3 (2) above) and then analyzing the samples by the reduction evaporization method. The analytical results are also shown in the table. Mercury at the 10 ng/g level was detected in iron ore (JSS 820-2: Robe River Ore) by both methods. The mercury in steel was below the lower limit of determination. The analytical values from both the reduction evaporation method and vaporization method were precise and in good agreement, but the former were slightly lower. The authors note also that it was difficult to completely decompose these samples when applying the bottom sediment preparation to the reduction evaporation method. Thus, a part of the mercury may have remained in the residues and reduced the analytical value. A direct thermal vaporization method without sample preparation might yield results closer to the true values.

On the basis of the above results, we surmise that the direct thermal vaporization method for solid samples can be applied to analyses of mercury in ore at levels of ng/g or higher. The method is easy to perform and requires no pretreatments such as sample dissolution. The authors thus propose that this method may be useful for the determination of mercury.

However, neither of the methods was applicable to the analysis of mercury in steel samples. We therefore decided to investigate a technique for determining ultra-trace amounts of mercury by ICP-MS after ion-exchange separation, in the hopes of finding a more sensitive method for analyzing steel samples.

3.2 Determination of Trace Mercury in Steel by Ion- Exchange Separation/ICP-MS

Inductively coupled plasma-MS is widely used as a highly sensitive element analytical technique. The drawback of ICP-MS is interference from the co-existing matrices (physical, chemical, and spectral interferences). This interference is so marked, the matrices must be removed before the trace elements in iron and steel can be determined. JFE steel has enabled the analysis of trace elements in steel using solvent extraction[11,12)], ion-exchange chromatographic separation[13,14)], gel chromatographic separation[15,16)], and similar methods. Mercury, however, is not included among them. Difficulties caused by the high vapor pressure in sample preparation have even disallowed investigations of analytical methods for mercury. In the present study we established a method for pre-concentrating ultra-trace mercury in steel by ion-exchange chromatographic separation. This method can be applied to analyses of the other environmentally hazardous elements, as well as simultaneous determinations of mercury, lead, and cadmium in steel in concentrations as low as 1 ng/g.

3.2.1 Selection of mass number measured by ICP-MS

Mercury, lead, and cadmium all contain multiple isotopes. For the isotopes present at abundant levels of 10% or more, the authors prepared calibration curves (1% nitric acid solution) for each element in a range from 0.01 to 1 ng/m*l* and compared the isotopes with each other to determine their respective correlation coefficients and sensitivities (background equivalent concentration (BEC) and signal intensity). The results are shown in **Table 3**. For some mass numbers, the authors obtained a good correlation coefficient when no internal standard is used. During the measurement by ICP-MS, however, the drifts of the signal and background intensity where relatively large and co-existing substances (acids etc.) in the sample solutions caused fluctuations in signal intensity. Thus, the authors selected ^{115}In, an element with a good correlation coefficient for each element tested, as the internal standard. And based on their correlation factors, BECs, and signal intensities, they selected ^{202}Hg, ^{208}Pb, and ^{111}Cd for testing in the following analyses.

3.2.2 Separation from iron matrix by ion-exchange chromatography

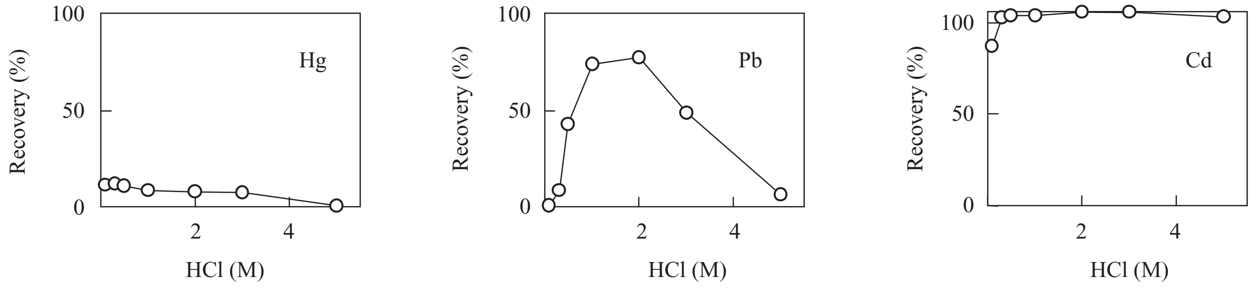
Iron (III) is adsorbed on both cation-exchange resins[17)] and anion-exchange resins[18)] in hydrochloric acid solutions of high concentrations, and not on cation-exchange resins[19)] and anion-exchange resins[20)] in hydrofluoric acid solutions of low molarity. Mercury, on the other hand, is adsorbed on both anion-exchange resins and cation-exchange resins in hydrofluoric acid solutions. Accordingly, the authors decided to investigate the distribution behavior of these elements on cation and anion exchanger in hydrochloric acid solutions and hydrofluoric acid solutions.

Each element was added in a trace amount of 0.2 μm to 10 m*l* of a solution with the hydrochloric acid concentration adjusted in a range from 0.1 to 5 M, then the solutions were passed through a column packed with 1 m*l* of an anion-exchange resin. After washing with about 20 m*l* of hydrochloric acid solution adjusted to the same concentration, the elements were eluted twice each with 2.5 m*l* of nitric acid (1 + 3)-1.5% hydrogen peroxide. Indium was added as an internal standard and

Table 3 Correlation coefficient of calibration curve and sensitivity (BEC* and ion counts) in each isotope of Hg, Pb, and Cd

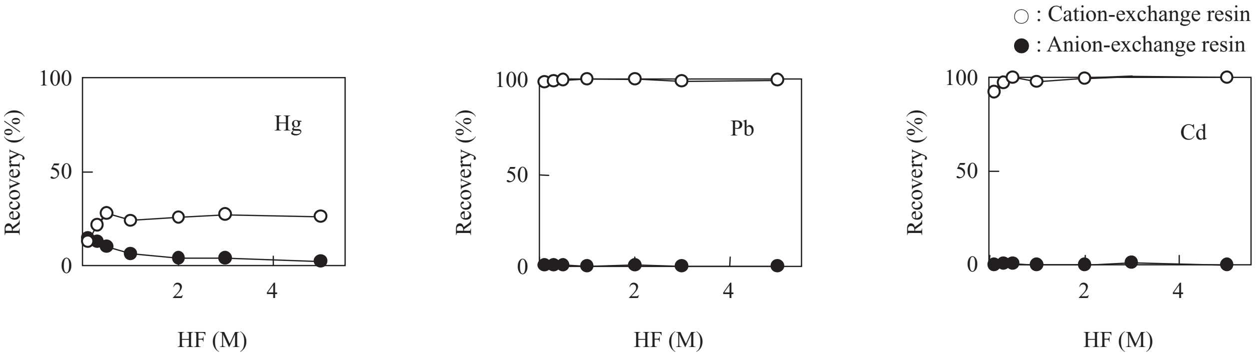
Element		Correlation coefficient			BEC (ng/m*l*)	Ion counts (cps/ppb)	Interference
Mass	Abundance	Without correction	^{103}Rh correction	^{115}In correction			
^{198}Hg	10.02	0.977 9	0.969 0	0.972 1	0.346	73	
^{199}Hg	16.83	0.990 6	0.994 2	0.990 6	0.080	94	
^{200}Hg	23.13	0.999 2	0.994 5	0.997 5	0.089	140	
^{201}Hg	13.22	0.997 6	0.994 5	0.998 7	0.078	73	
^{202}Hg	29.80	0.998 7	0.998 7	0.999 7	0.088	164	
^{204}Hg	29.52	0.931 2	0.901 9	0.937 8	0.101	234	
^{206}Pb	24.14	0.988 1	0.969 4	0.994 1	0.587	949	
^{207}Pb	22.08	0.986 2	0.966 9	0.992 7	0.584	800	
^{208}Pb	52.35	0.987 2	0.968 1	0.993 5	0.574	1 931	
^{110}Cd	12.49	0.999 1	0.998 6	0.998 1	0.002 7	246	
^{111}Cd	12.80	0.999 4	0.999 5	0.999 6	0.001 4	238	^{112}Sn
^{112}Cd	24.13	0.999 9	0.999 9	0.999 9	0.039 8	466	
^{113}Cd	12.22	0.396 5	0.011 9	0.880 7	6.88	1 180	
^{114}Cd	28.73	0.999 0	0.998 7	0.997 9	0.025 3	555	^{114}Sn
^{116}Cd	7.49	0.554 2	0.457 5	0.607 1	3.83	327	

* Background equivalent concentration



Hg, Pb, Cd: 0.2 μg in 10 m*l* of HCl solution, Amount of resin: 1 m*l*,
Eluent: 3.5 M HNO_3 (1.5%H_2O_2), 2.5 m*l*-2 times elution

Fig. 1 Recovery of elements by DOWEX® 1-X8 anion-exchange resin from HCl solutions



Hg, Pb, Cd: 0.2 μg in 10 m*l* of HF solution, Amount of resin: 1 m*l*,
Eluent: 3.5 M HNO_3 (1.5 % H_2O_2), 2.5 m*l*-2 times elution

Fig. 2 Recovery of elements by DOWEX® 50W-X8 cation-exchange resin and DOWEX® 1-X8 anion-exchange resin from HF solutions

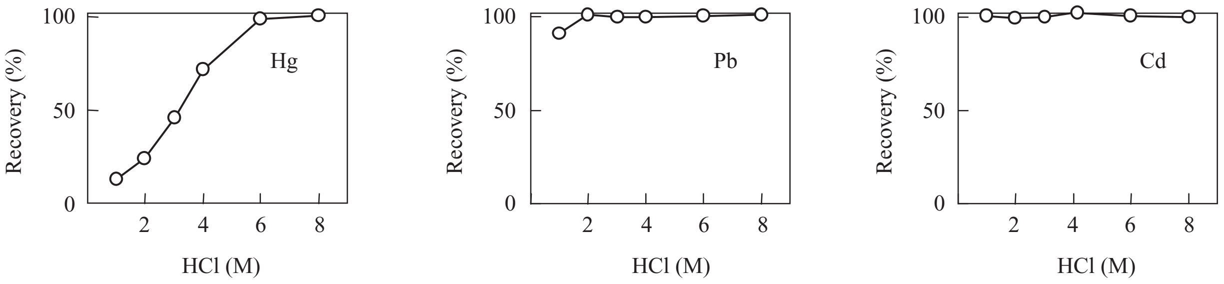
recoveries were determined by measuring the concentrations of each element by ICP-MS. The results are shown in **Fig. 1**. Whereas the cadmium could be almost completely recovered when the hydrochloric acid concentration was 0.3 M or more, the quantitative recoveries of mercury and lead were difficult to determine at the hydrochloric acid concentrations tested.

Accordingly, we investigated the distribution behavior of each element in hydrofluoric acid solutions. Solutions with hydrofluoric acid concentrations adjusted in a range from 0.1 to 5 M (the same concentrations used for the hydrochloric acid) were passed through a column packed with 1 m*l* of a cation-exchange or an anion-exchange resin, then the elements were eluted twice each with 2.5 m*l* of nitric acid (1 + 3)-1.5% hydrogen peroxide. The results are shown in **Fig. 2**.

With the samples using the cation-exchange resin, lead and cadmium could be completely recovered from all of the hydrofluoric acid solutions (0.1 to 5 M), whereas the mercury was only recovered at a rate of 20 to 30%. The authors speculated that the considerable fluctuation in the mercury recovery from experiment to experiment might have resulted not from the adsorption on the ion-exchange resins, but from insufficient elution. They thus re-examined the elution conditions using hydrochloric acid, a compound with a reportedly small distribution coefficient of mercury with cation exchange resin. **Figure 3** shows recoveries observed when the elements were adsorbed on a cation-exchange resin from a 1 M hydrofluoric acid solution and eluted with hydrochloric acids of different concentrations. Total amounts of lead were recovered from hydrochloric acid of about 2 M, and cadmium was recovered from above 1 M. Mercury was completely recovered from the hydrochloric acid above 6 M.

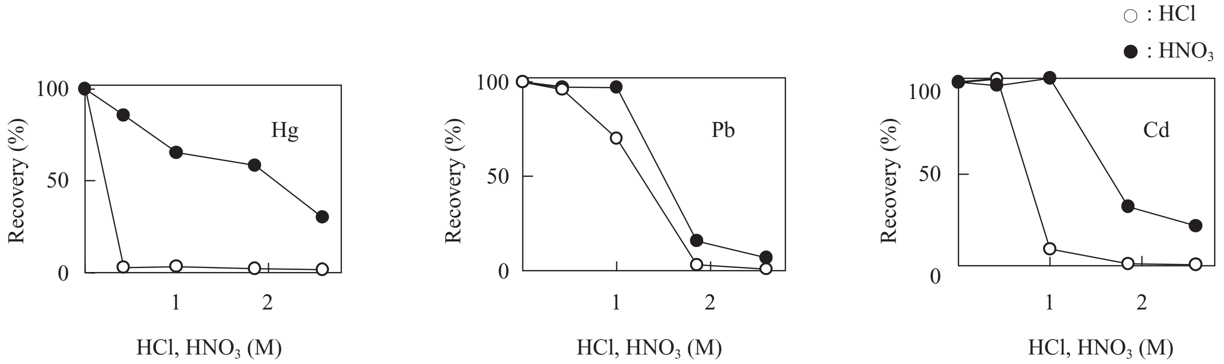
Noting that mercury is apt to vaporize when no oxidizing acids coexist during decomposition (described in paragraph 3.2.3 above), the authors also investigated the effect of the coexistence of these acids during ion exchange separation. The results are shown in **Fig. 4**. When hydrochloric acid and nitric acid coexisted in the 1 M hydrofluoric acid, the authors observed a sharp decrease in the recovery of each element, particularly with the hydrochloric acid.

Therefore, we investigated the extent to which the addition of hydrofluoric acid reduces the effect of the coexistence of a certain amount of nitric acid (0.25 m*l*). The results are shown in **Fig. 5**. For each element, the effect was nullified by adding 20 m*l* of 1M hydrofluoric acid, that is, by setting the nitric acid concentration at



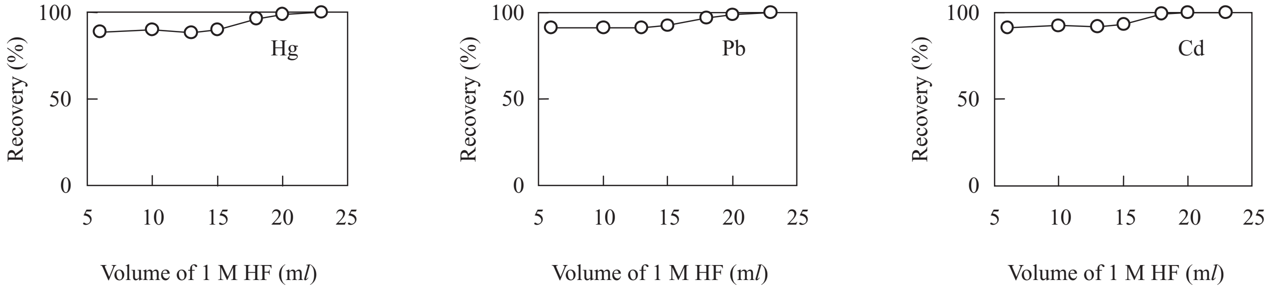
Hg, Pb, Cd: 0.2 μg in 10 m*l* of 1 M HF,
Amount of resin: DOWEX® 50W-X8 (100-200 mesh), 1 m*l*
Volume of Eluent: 2.5 m*l*-2 times elution

Fig. 3 Effect of concentration of HCl as eluent on recovery of elements



Hg, Pb, Cd: 0.2 μg in 10 m*l* of 1 M HF,
Amount of resin: DOWEX® 50W-X8 (100-200 mesh), 1 m*l*,
Eluent: 8 M HCl, 2.5 m*l*-2 times elution

Fig. 4 Effect of concentration of co-existing HCl and HNO_3 in 1 M HF at cation exchange treatment



Hg, Pb, Cd: 0.2 μg in 0.25 m*l* of HNO_3,
Amount of resin: DOWEX® 50W-X8 (100-200 mesh), 1 m*l*,
Eluent: 8 M HCl, 2.5 m*l*-2 times elution

Fig. 5 Effect of valume of 1 M HF on recovery of each elements

0.2 M or less.

3.2.3 Optimization of sample preparation methods

Mercury has a high vapor pressure and is apt to vaporize during decomposition. To avoid this, we performed our experiment by adding standard solutions of mercury, lead, and cadmium to the respective acid solutions, concentrating them by heating, and determining the concentration of each by ICP-MS. The solutions were heated at 130°C for 1 h in a hydrofluoric acid plus hydrogen peroxide solution (2 + 1 + 2) (used in the above-described ion exchange separation), hydrochloric acid plus nitric acid (1 + 12) (used most frequently in the analysis of steel samples), and nitric acid (1 + 1) (as an oxidizing acid). None of the elements were volatilized and the total amounts could be recovered. In a parallel experiment the authors added the same amount of standard solution to 0.1 g of iron and a steel sample, then decomposed them by a similar method of heating, at 130°C for 1 h. Almost all of the mercury vaporized, as shown in **Table 4**. Thus, the total amounts of mercury, lead, and cadmium could be recovered when a similar treatment was performed using nitric acid in decomposi-

Table 4 Recovery of Hg, Pb, and Cd after decomposition of steel samples

(%)

Element	Sample A		Sample B	
	$HF + H_2O_2$ (2 + 1 + 2)	7 M HNO_3	$HF + H_2O_2$ (2 + 1 + 2)	7 M HNO_3
Cd	100 , 100	99, 99	98 , 101	99, 100
Hg	1.5, 0.7	98, 100	1.5, 9.5	97, 101
Pb	97 , 100	99, 102	97 , 100	96, 103

Table 5 Analytical results of Cd, Hg, and Pb in commercial steels and CRM (JSS 002-4: High-purity iron)

(μg/g)

Sample	JSS 002-4 (Pb < 0.3 μg/g)			Steel A			Steel B		
Added (μg)	0	0.2	0.5	0	0.2	0.5	0	0.2	0.5
Cd	0.009	0.215	0.527	0.015	0.220	0.534	0.008	0.208	0.498
Hg	0.007	0.189	0.496	0.009	0.199	0.502	0.005	0.202	0.500
Pb	0.102	0.296	0.588	0.493	0.687	0.997	0.206	0.415	0.738

tion. On the basis of these results and the results shown in Fig. 5, the authors decided to adopt a new method. Specifically, the authors diluted the sample solution with 1 M hydrofluoric acid in order to bring down the nitric acid concentration to less than 0.2 M after the decomposition of steel sample with nitric acid, and then separated the mercury, lead and cadmium from the iron matrix by cation exchange chromatography.

3.2.4 Analysis of high-purity iron and steel

The mercury, lead, and cadmium in commercial cold-rolled steel sheets and a certified reference material (high-purity iron) were determined by the method described in paragraph 2.3 (3) above. The results are shown in **Table 5**. To verify accuracy, the authors conducted analyses by performing similar treatments after adding standard solutions of each element in amounts equivalent to 0.2 μg/g and 0.5 μg/g in steel to each sample. These results are also shown in Table 5. The recovery of each element from each standard solution was favorable, and the mercury was determined without volatilization. The lower limits of detection (3σ of blank values) of this method were 2 ng/g for mercury, 0.5 ng/g for cadmium, and 9 ng/g for lead.

4. Conclusions

The authors compared the sensitivities and accuracies of two methods used for the determination of trace amounts of mercury in steel and iron ore: a direct determination from solid samples by the direct thermal vaporization, and a determination from digested sample solutions by the reduction evaporation method. In tests conducted with powder samples of ore, the direct thermal vaporization method could be reliably applied to the analysis of mercury at levels of ng/g or higher. Direct thermal vaporization, a method which can be easily performed without pretreatments such as sample dissolutions, thus proved to be useful for analyzing mercury.

Both methods, however, were too insensitive to be reliably applied to the analysis of steel samples. Accordingly, the authors investigated the determination of ultra-trace amounts of mercury by ICP-MS after ion-exchange chromatographic separation. After the decomposition of a steel sample with nitric acid, mercury is adsorbed on a cation exchange resin in 1 M hydrofluoric acid and separated from iron. The mercury is eluted with a 6 M hydrochloric acid solution and determined by ICP-MS. Ultra-trace amounts of mercury can now be determined at levels of ng/g in steel. This method can also determine cadmium and lead at levels of ng/g or lower during the determination of mercury. The authors thus conclude that it can be effectively applied for evaluations of harmful elements in steel.

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