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Coprecipitation with yttrium phosphate as a separation technique for iron(III), lead, and bismuth from cobalt, nickel, and copper matrices

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Abstract

The coprecipitation behavior of 44 elements (47 ions because of chromium(III,VI), arsenic(III,V), and antimony(III,V)) with yttrium phosphate was investigated at various pHs. Yttrium phosphate could quantitatively coprecipitate iron(III), lead, bismuth, and indium over a wide pH range; however, 18 ions, including alkali metals and oxo anions, such as vanadium(V), chromium(VI), molybdenum(VI), tungsten(VI), germanium(IV), arsenic(III,V), selenium(IV), and tellurium(VI), were scarcely collected. In addition, 19 ions, including cobalt, nickel, and copper(II), were hardly coprecipitated at pHs below about 3. Based on these results, the separation of iron(III), lead, and bismuth from cobalt, nickel, and copper(II) matrices was investigated. Iron(III), lead, and bismuth ranging from 0.5 to 25 μg could be separated effectively from a solution containing 0.5 g of cobalt, nickel, or copper at pH 3.0. The separated iron(III), lead, and bismuth could be determined by inductively coupled plasma atomic emission spectrometry using internal standardization. The detection limits (3σ, $n = 7$) of iron(III), lead, and bismuth were 0.008, 0.137, and 0.073 μg, respectively. The proposed method was applied to the analyses of metals and chlorides of cobalt, nickel, and copper.

Keywords: Iron(III); Lead; Bismuth; Coprecipitation; Yttrium phosphate; ICP-AES; Metals and salts of Co, Ni, and Cu

1. Introduction

For the determination of trace elements in various samples, the separation technique is sometimes required to prevent or reduce the interference from the matrices in the samples. Among various techniques for the separation of trace elements proposed until now, coprecipitation is one of the most useful ones [1–4]. Metal hydroxides are widely used as coprecipitants because they have the ability to coprecipitate various elements quantitatively [1–4]; the hydroxides of iron(III) [5–9], magnesium [10,11], beryllium [12], nickel [13], gallium [14], bismuth [15], scandium [16], lanthanum [17], cerium(IV) [18], samarium [19], and titanium [7] have been recently applied to the separation of some trace elements from the matrices of samples, such as sea water [5,8,10,11,14,16,18], wastewater [9,17], high-purity metals [6,12,15], and sediments [7,18,19]. On the other hand, we have sought a coprecipitant that is capable of selectively separating some elements and reported in a previous communication that lanthanum phosphate was useful for the separation of iron(III) and lead [20]. However, lanthanum phosphate was slightly too fine when the precipitate was formed at room temperature, and as a result, the filtration for the quantitative collection of the precipitate was sometime troublesome and time-consuming.

We also previously found that yttrium phosphate was useful for the selective coprecipitation of iron(III) and lead [21], and the quantitative collection of the precipitate could be smoothly achieved because yttrium phosphate was readily filtrable. In this work, we examined the coprecipitation behavior of 44 elements (47 ions because of chromium(III,VI), arsenic(III,V), and antimony(III,V)) in detail to evaluate the ability of yttrium phosphate as a coprecipitant. Our results indicated that yttrium phosphate was effective for the coprecipitation of bismuth and indium as well as iron(III) and lead,

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as these four elements could be coprecipitated quantitatively over a wide pH range. In addition, yttrium phosphate scarcely coprecipitated 18 ions, including alkali metals and oxo anions, over a wide pH range and 19 ions, including cobalt, nickel, and copper(II), at pHs below about 3. These results indicate that the effective separation of iron(III), lead, bismuth, and indium from some matrices can be attained when the coprecipitation is carried out at pHs below about 3. To evaluate the availability of the coprecipitation technique using yttrium phosphate, this coprecipitation technique was applied to the separation of iron(III), lead, and bismuth in metals and chlorides of cobalt, nickel, and copper prior to their inductively coupled plasma atomic emission spectrometric (ICP-AES) determination.

For the separation of some impurities in the metals, alloys, and compounds of cobalt [22], nickel [23–27], and copper [24,27–36], the coprecipitation using the hydroxides of lanthanum [22–24,28–33], zirconium [34], and indium [35] and manganese dioxide [25–27,36] has often been utilized. The coprecipitation using hydroxides [22–24,28–35] is available for the separation of many kinds of elements, including iron [22,23,28,33,34], lead [28,31,33–35], and bismuth [24,28,31,33–35]. In these techniques using hydroxides, however, the coprecipitation had to be carried out at pHs 8.5–10.5 to form the precipitate; therefore, the addition of large amount of an alkaline solution, such as an ammonia solution, was required. The coprecipitation using manganese dioxide [25–27,36] is also applicable to the separation of lead [25,26] and bismuth [25,26,36]. In this technique, the precipitation could be formed even under the acidic conditions, and the elements were collected at pHs 1–4.5; however, the formation of the precipitate, in which a solution containing both manganese(II) and permanganate ions was boiled for a while, was a tedious operation. The coprecipitation using yttrium phosphate overcomes these weak points, although this technique can be used for the determination of only iron(III), lead, and bismuth.

2. Experimental

2.1. Apparatus

A Perkin-Elmer Optima 3000 XL inductively coupled plasma atomic emission spectrometer and a Hitachi 170-50 atomic absorption spectrometer (flame type) were used for the measurements of elements. The pH measurements were carried out with a Horiba model N-8F ion meter equipped with a 6328-10C combination-pH-electrode.

2.2. Reagents

An yttrium solution ($5\,\mathrm{g\,L^{-1}}$) was prepared by dissolving 99.99% of yttrium oxide (Nacalai Tesque) in 50 mL of concentrated hydrochloric acid and diluting to 1000 mL with distilled–deionized water. Standard solutions of arsenic(V) and antimony(V) were prepared by dissolving disodium hydrogen arsenate heptahydrate (guaranteed reagent grade, Kanto Kagaku) and potassium hexahydroxoantimonate(V) (extra-pure grade, Kanto Kagaku) in distilled–deionized water, respectively. A standard solution of chromium(III) was also prepared by dissolving chromium(III) nitrate nonahydrate (99.9%, Wako Pure Chemical Industries) in a small amount of concentrated nitric acid and diluting with distilled–deionized water; the solution was standardized by complexometric back titration. The standard solutions of ruthenium and iridium ($1000\,\mathrm{mg\,L^{-1}}$) were purchased from Acros Organics. For the standard solutions of the other elements, including iron, lead, and bismuth, commercially available $1000\,\mathrm{mg\,L^{-1}}$ solutions (Kanto Kagaku) were used. The phosphoric acid (guaranteed reagent grade, Wako Pure Chemical Industries) was used without further purification. All other reagents used were of guaranteed or analytical reagent grade.

2.3. Coprecipitation behavior of some elements

To 100 mL of a solution containing 10 μg of each ion, except for cadmium (0.5 μg), manganese(II) (5 μg), and copper(II) (5 μg), 1 mL of the yttrium solution and 3 mL of a $0.5\,\mathrm{mol\,L^{-1}}$ phosphoric acid solution were added. The pH of the solution was adjusted with ammonia solutions of approximately 7 and $1\,\mathrm{mol\,L^{-1}}$ by using the pH meter. After the formed precipitate had settled, the precipitate was filtered by suction using a membrane filter (Nihon Millipore, Omnipore membrane, pore size 1.0 μm). The precipitate was dissolved with 2 mL of approximately $6\,\mathrm{mol\,L^{-1}}$ nitric acid and diluted to 10 or 5 mL with distilled–deionized water. The amount of ion in the solution was then measured by ICP-AES or flame atomic absorption spectrometry (FAAS) under the operating conditions shown in Table 1.

2.4. Determination of iron, lead, and bismuth in metals and chlorides of cobalt, nickel, and copper

For the analyses of chlorides, cobalt chloride hexahydrate, nickel chloride hexahydrate, or copper(II) chloride dihydrate (guaranteed reagent grade) was dissolved in distilled–deionized water. For the analyses of metals, powdery nickel (99%) or copper (99.85%) was dissolved with approximately $8\,\mathrm{mol\,L^{-1}}$ nitric acid and diluted with distilled–deionized water. To 100 mL of a sample solution, in which approximately 0.5 g of cobalt, nickel, or copper was contained, 2 mL of the yttrium solution and 3 mL of a $0.5\,\mathrm{mol\,L^{-1}}$ phosphoric acid solution were added. The pH of the solution was adjusted to 3.0 with an ammonia solution of approximately $7\,\mathrm{mol\,L^{-1}}$ by using the pH meter. After the formed precipitate had settled, the precipitate was filtered by suction using a membrane filter. The precipitate collected on the filter was washed with about 40 mL of a $0.5\,\mathrm{mol\,L^{-1}}$ phosphoric acid solution adjusted at pH 3.0 by using an ammonia solution and about 20 mL of distilled–deionized water and

Table 1
Operating conditions for ICP-AES and FAAS

ICP-AES			
Radio frequency output (kW)	1.3		
Argon gas flow rate (L min^{-1})			
Plasma gas	15		
Auxiliary gas	0.5		
Neblizing gas	0.8		
Pump parameter			
Sample flush time (s)	10		
Sample flush flow rate (mL min^{-1})	4.0		
Sample flow rate (mL min^{-1})	1.2		
Read delay time (s)	30		
Reading time	Auto (minimum 0.2 s–maximum 20 s)		
Analytical wavelength (nm)	Li, 610.362	Na, 589.592	K, 766.490
	Rb, 780.023	Be, 234.861	Mg, 279.553
	Ca, 396.847	Sr, 421.552	Ba, 233.527
	Sc, 361.384	Y, 324.228	Ti, 334.941
	Zr, 339.198	V, 310.230	Mo, 202.030
	W, 207.911	Ru, 240.272	Os, 225.585
	Rh, 343.489	Ir, 224.268	Pd, 340.458
	Pt, 265.945	Ag, 338.289	Au, 242.795
	B, 249.773	Al, 396.152	Ga, 417.206
	In, 230.606	Tl, 351.924	Ge, 265.118
	Sn, 283.999	As, 193.696	Sb, 252.852
	Bi, 223.061	Se, 196.026	Te, 214.281
	La, 408.672 (internal standard)		
Flame AAS			
Lamp current (mA)	7.5		
Flame	Air–acetylene		
Air pressure (kPa)	160		
Acetylene pressure (kPa)	20		
Background correction	D_2 method		
Analytical wavelength (nm)	Mn, 279.5	Fe, 248.3	Co, 240.7
	Ni, 232.0	Cd, 228.8	Pb, 283.3

then dissolved with 1 mL of approximately 6 mol L^{-1} nitric acid. Lanthanum (10 μg) was added to the solution as an internal standard element and the solution was diluted to 5 mL with distilled–deionized water. The amounts of iron, lead, and bismuth in the solution were measured by ICP-AES using internal standardization at 259.939 nm for iron, 283.306 nm for lead, 190.171 nm for bismuth, and 408.672 nm for lanthanum (internal standard element).

3. Results and discussion

3.1. Precipitation of yttrium phosphate

When an ammonia solution was added to 100 mL of a solution containing 5 mg of yttrium and 3 mL of 0.5 mol L^{-1} phosphoric acid, the precipitate was formed at pH around 1.8, and the quantitative precipitation of yttrium was observed over a wide pH range from 2.3 to at least 10.5. The ratios of yttrium to phosphorous in every precipitate formed at pH 3.0, 5.0, and 7.0 were 1.1 after standing for 1 h; the ratios did not show any significant change even after standing for 5 days. The formed precipitate was readily filterable by suction using a membrane filter with a pore size of 1.0 μm, although a membrane filter with a pore size of 0.2 μm was required for the quantitative recovery of slightly too fine lanthanum phosphate [20]. The yttrium phosphate collected on the filter could be dissolved with more than 0.5 mol L^{-1} of some mineral acids, such as hydrochloric, nitric, sulfuric, and perchloric acids.

3.2. Coprecipitation behavior of some elements with yttrium phosphate

The recoveries of 44 elements (47 ions because of chromium(III,VI), arsenic(III,V), and antimony(III,V)) were investigated at various pHs. The obtained results are shown in Fig. 1. The investigated ions could be classified into five types by their coprecipitation behavior: (A) the ions that were quantitatively coprecipitated over a wide pH range; (B) the ions whose recoveries increased with increasing the pH in the solution; (C) the ions whose recoveries decreased with increasing the pH; (D) the ions that were scarcely recovered over a wide pH range; (E) the ions that had different coprecipitation behavior. The type for each ion is also appended in Fig. 1. Since iron(III), lead, bismuth, and indium belong to type A, yttrium phosphate seems to be useful for the coprecipitation of these ions; this coprecipitation technique would

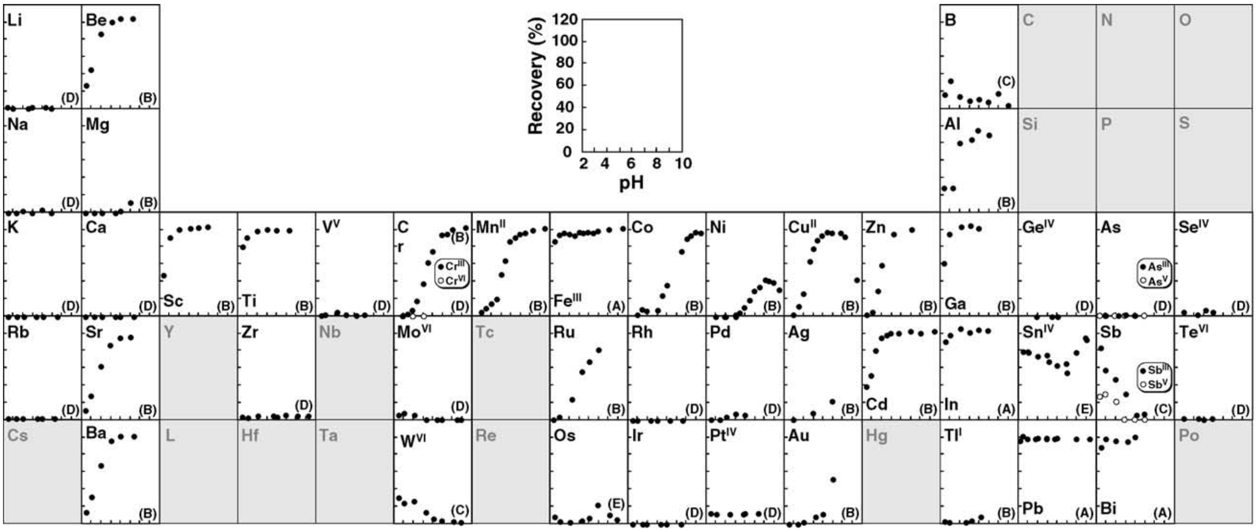

Fig. 1. Effect of pH on the recoveries of some ions by coprecipitation with yttrium phosphate. Sample volume, 100 mL; each ion, 10 μg (except for Cd (0.5 μg), Mn(II), and Cu(II) (5 μg)); yttrium, 5 mg; 0.5 mol L^{-1} H_3PO_4, 3 mL.

be applicable to the separation of type A ions from a solution also containing types C and D ions. This technique would also be available for the separation of type A ions from a solution also containing magnesium, chromium(III), manganese(II), cobalt, nickel, copper(II), and zinc, which were categorized as type B ions, at pHs below about 3.

The coprecipitation behavior of each ion with yttrium phosphate may be closely related to the coprecipitation process of each ion. From this point of view, the relationship between the pH coprecipitated and the distribution of the ion, D, which is defined as the ratio of the amount of the ion coprecipitated to that remaining in the solution, was investigated for some ions according to the procedure for the coprecipitation behavior of some elements. As shown in Fig. 2, straight lines were obtained for many ions classified as type B; the slopes for some divalent ions are summarized in Table 2. A linear relationship between $\log D$ and the pH has also been obtained in the coprecipitation of some elements with hydrous ferric oxide [4,37] and manganese dioxide [4,38]. In those cases, it was suggested that the ions were adsorbed on the surface of the precipitate according to the simple ion exchange [4,37,38]. On the other hand, it is well known that the relationship between the amount of ion coprecipitated and that remaining in the solution sometimes obeys the Freundlich adsorption isotherm when the ion is adsorbed on the precipitate as a molecular form or compound [4]. In our investigation, iron(III) (100–900 μg), lead (300–1500 μg), cobalt (50–500 μg), nickel (25–175 μg), or copper(II) (50–500 μg) was coprecipitated at pH 3.0 from 100 mL of an aqueous solution using 5 mg of yttrium and 3 mL of 0.5 mol L^{-1} phosphoric acid, and the solution was then allowed to stand for 5 h at 25.0 ± 0.1 °C. As shown in Fig. 3, the relationship for these ions followed the Freundlich adsorption isotherm:

$$\log\left(\frac{x}{m}\right) = \left(\frac{1}{n}\right)\log C + \log k, \qquad (1)$$

where x is the coprecipitated amount of each ion (mol), m the amount of yttrium used for the coprecipitation (mol), C the concentration of the remaining ion in the solution (mol L^{-1}), and n and k are constants. In these experiments, n and $\log k$

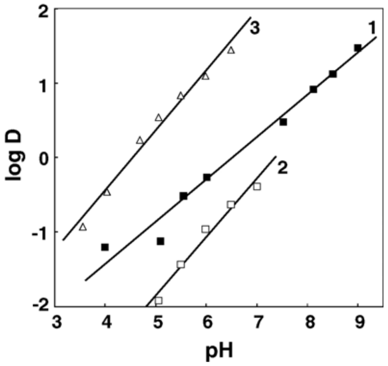

Fig. 2. Relation between the pH coprecipitated and the distribution of some ions. Sample volume, 100 mL; yttrium, 5 mg; 0.5 mol L^{-1} H_3PO_4, 3 mL. (1) Co, 10 μg; (2) Ni, 10 μg; (3) Cu(II), 5 μg. D: the ratio of the coprecipitated amount of ion to that remaining in the solution.

Table 2
Slopes for some divalent ions

Ion	Slope
Be^{2+}	0.82
Sr^{2+}	0.65
Ba^{2+}	0.86
Mn^{2+}	0.77
Co^{2+}	0.56
Ni^{2+}	0.78
Cu^{2+}	0.81
Zn^{2+}	1.19
Cd^{2+}	0.93

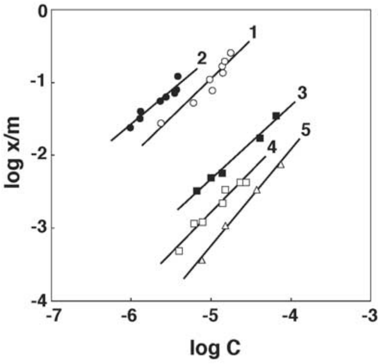

Fig. 3. Relation between the coprecipitated amounts of iron(III) (1); lead (2); cobalt (3); nickel (4); copper(II) (5), and the remained amounts of them in the solution after the coprecipitation. Sample volume, 100 mL; yttrium, 5 mg; 0.5 mol L^{-1} H_3PO_4, 3 mL; pH, 3.0; temperature, 25.0 ± 0.1 °C. *x*: the coprecipitated amount of ion (mol); *m*: the amount of yttrium used for the coprecipitation (mol); *C*: the concentration of ion remaining in the solution after the coprecipitation (mol L^{-1}).

were 0.94 and 4.37 for iron(III), 1.09 and 3.93 for lead, 0.99 and 2.71 for cobalt, 0.89 and 2.81 for nickel, and 0.76 and 3.36 for copper(II), respectively. From these results, the adsorption involving the ion exchange and the formation of phosphate on the surface of the precipitate may occur in the coprecipitation of ions with yttrium phosphate. If the formation of phosphate occurs, the coprecipitation of the ion seems to be controlled over the solubility of the phosphate (the Paneth–Fajans–Harn rule) [2–4]. In Table 3, the solubility product constants and the solubilities calculated on the basis of solubility product constants are summarized [39–41]. The solubility of phosphate roughly decreased in the following order: type A > type B > type D. Although lead was effectively coprecipitated with yttrium phosphate, the solubility of lead phosphate was not very small (Table 3); this seems to indicate that not a simple phosphate but, rather, a more insoluble compound, such as chloropyromorphite, $Pb_5(PO_4)_3Cl$ [42–44], would form during the coprecipitation or that another mechanism, such as solid solution formation [1–4], would dominate the coprecipitation of lead. It is well known that the phosphate of zirconium is extremely insoluble [45]. In our investigation, however, zirconium was scarcely recovered over a wide pH range, as shown in Fig. 1; the coprecipitated zirconium might remain on the filter because zirconium phosphate cannot be dissolved even in concentrated nitric acid [45]. For nickel and copper(II), the recoveries decreased with increasing the pH in the alkaline region. The behavior would be due to the formation of their ammine complexes [22,23]. Many of the ions including vanadium(V), molybdenum(VI), tungsten(VI), chromium(VI), boron, germanium(IV), arsenic(III,V), antimony(V), selenium(IV), and tellurium(VI), which belonged to types C or D and were present as oxo anions in the aqueous solution, were also hardly coprecipitated with yttrium phosphate. The adsorption of these ions appears to be inhibited by the large amount of phosphate ions existing in the solution. These results seem to suggest that the adsorption mechanism plays an important role in the coprecipitation of the ion with yttrium phosphate, although more detailed studies will be required to clarify the coprecipitation process of each ion, including magnesium or calcium, with yttrium phosphate.

Table 3
Solubility product constants and/or solubilities of some phosphates [39]

Phosphate	Solubility product constant	Solubility (mol L^{-1})	Temperature (°C)
YPO_4[a]	1.0×10^{-25}	3.2×10^{-13}	25
$BiPO_4$	1.3×10^{-23}	3.6×10^{-12}	20
$CrPO_4$ (green)	2.4×10^{-235}	4.9×10^{-12}	20
$FePO_4$	1.3×10^{-22}	1.1×10^{-11}	20
$InPO_4 \cdot 2H_2O$	2.2×10^{-22}	1.5×10^{-11}	25
$GaPO_4$[b]	1.0×10^{-21}	3.2×10^{-11}	25
$AlPO_4$	1.3×10^{-20}	1.1×10^{-10}	20
$Pb_3(PO_4)_2$	8×10^{-43}	2×10^{-9}	20
$CrPO_4$ (violet)	1.0×10^{-17}	3.2×10^{-9}	20
$Be_3(PO_4)_2$	1.9×10^{-38}	1.1×10^{-8}	20
$Cu_3(PO_4)_2$	1.3×10^{-37}	1.6×10^{-8}	20
$Co_3(PO_4)_2$	2×10^{-35}	5×10^{-8}	20
$Cd_3(PO_4)_2$	2.5×10^{-33}	1.2×10^{-7}	20
$Zn_3(PO_4)_2$	9.1×10^{-33}	1.5×10^{-7}	20
$Ni_3(PO_4)_2$	5×10^{-31}	3×10^{-7}	20
$Sr_3(PO_4)_2$	1.6×10^{-28}	1.1×10^{-6}	35
$Mg_3(PO_4)_2$	2×10^{-27}	2×10^{-6}	38
$Ba_3(PO_4)_2$	3.4×10^{-23}	1.3×10^{-5}	20
Ag_3PO_4	–	1.5×10^{-5}	20
$Ca_3(PO_4)_2$	–	4.8×10^{-7}–9.7×10^{-7}	–
Tl_3PO_4	–	7.0×10^{-3}	15

[a] Ref. No. [40].
[b] Ref. No. [41].

Table 4
Recoveries of iron(III), lead, and bismuth from the spiked cobalt, nickel, or copper chloride solution at pH 3.0

Sample solution	Y added (mg)	Recovery (%)		
		Fe(III)	Pb	Bi
$CoCl_2$	5	79.4	106.5	102.5
	10	101.2	104.1	101.9
$NiCl_2$	5	92.9	96.9	96.9
	10	97.5	105.6	103.6
$CuCl_2$	5	88.0	96.5	91.1
	10	98.0	104.0	101.8

3.3. Determination of iron, lead, and bismuth in metals and chlorides of cobalt, nickel, and copper

As described above, yttrium phosphate was effective for the collection of iron(III), lead, bismuth, and indium, whereas the recoveries of many ions including cobalt, nickel, and copper(II) were considerably low at low pHs. Since iron, lead, and bismuth as impurities have been determined in metals and some compounds of cobalt, nickel, and copper [22–26,28,31,33–36], the coprecipitation technique using yttrium phosphate was then applied to the pre-separation technique for their ICP-AES determination.

3.3.1. Optimum conditions for coprecipitation of iron, lead, and bismuth

The conditions for the coprecipitation of iron(III), lead, and bismuth were optimized using 100 mL of a solution containing approximately 0.5 g of cobalt, nickel, or copper(II), which were added as a chloride. The amounts of yttrium and phosphoric acid required for the quantitative recoveries of iron(III), lead, and bismuth were investigated. When 5 mg of yttrium was used for the coprecipitation, the recovery of iron(III) was relatively low, as shown in Table 4. However, the quantitative recoveries of all the elements were obtained using 10 mg of yttrium. The presence of yttrium ranging from 5 mg to at least 15 mg did not influence their determination by ICP-AES. More than 1 mL of 0.5 mol L^{-1} phosphoric acid was also required for the quantitative coprecipitation. The coprecipitated amounts of cobalt, nickel, and copper were also investigated using 5 or 10 mg of yttrium. The results, which were determined by electrothermal atomic absorption spectrometry using the standard addition method, are summarized in Table 5. The coprecipitated amounts of nickel and copper increased with increasing the added amount of yttrium, although the coprecipitated amount of cobalt was not significantly different in the coprecipitation using 5 and 10 mg of yttrium. The coprecipitated amounts of nickel and copper did not show a significant increase with increasing the amounts of their metals in the solution when a constant amount of yttrium was used for the coprecipitation (Table 5). Although a lower pH in the coprecipitation was preferable for the effective separation of iron(III), lead, and bismuth from the matrices, as shown in Fig. 1, the recoveries of these elements were often insufficient at pH 2.5 from a solution also containing large amounts of cobalt, nickel, or copper. The quantitative recoveries were obtained at pH 3.0; however, the increase of the pH above 4.0 resulted in an increase of the cobalt, nickel, and copper recoveries. In view of these results, the coprecipitation was carried out using 10 mg of yttrium and 3 mL of 0.5 mol L^{-1} phosphoric acid at pH 3.0 in the proposed method. For the dissolution of the precipitate, nitric acid was chosen because this acid had only a slight influence on the measurements of iron(III), lead, and bismuth by ICP-AES. Although the precipitation could be dissolved even with 0.5 mol L^{-1} nitric acid, as mentioned above, a higher concentration was required to dissolve the precipitate collected on the filter rapidly by using a small volume of the acid. In the proposed method, approximately 6 mol L^{-1} nitric acid was used for the dissolution of the precipitate. Additionally, lanthanum was added to the solution as an internal standard element after the coprecipitation in order to prevent a reduction in accuracy in the measurement by ICP-AES.

3.3.2. Calibration curves

The relationship among the emission intensities of iron, lead, and bismuth and the amounts of iron(III), lead, and bismuth in the solution after the coprecipitation was investigated. Straight lines were obtained over the ranges of at least 0.5–25 μg of iron(III), lead, and bismuth. The detection limits, defined as three times the standard deviations obtained from seven replicate determinations of the blank, were 0.008 μg for iron(III), 0.137 μg for lead, and 0.073 μg for bismuth.

3.3.3. Recoveries of iron, lead, and bismuth from the spiked cobalt, nickel, and copper solutions

Using the procedure recommended, the recoveries of iron(III), lead, and bismuth were investigated from a solution containing approximately 0.5 g of cobalt, nickel, or copper and 10 μg of iron(III), lead, and bismuth. The results obtained are shown in Table 6. The iron(III), lead, and bismuth spiked

Table 5
Results for the amounts of cobalt, nickel, and copper coprecipitated with yttrium phosphate at pH 3.0

Metal	Added (mg)	Y added (mg)	Found (μg)
Co	500	5	3.52
	500	10	4.14
Ni	500	5	1.15
	1000	5	1.53
	1500	5	1.51
	1000	10	78.4
	1000	15	470
Cu	250	5	11.5
	500	5	27.1
	500	10	110

The results were determined by electrothermal atomic absorption spectrometry using standard addition method.

Table 6
Recoveries of 10 μg of iron(III), lead, and bismuth from some metal chloride solutions

Sample solution	Found (μg)[a]		
	Fe(III)	Pb	Bi
$CoCl_2$	10.12 ± 0.16	10.41 ± 0.36	10.19 ± 0.13
$NiCl_2$	9.75 ± 0.18	10.56 ± 0.43	10.36 ± 0.12
$CuCl_2$	9.80 ± 0.21	10.56 ± 0.40	10.36 ± 0.18

[a] Mean ± standard deviation ($n = 4$).

Table 7
Determination of iron, lead, and bismuth in metals and chlorides of cobalt, nickel, and copper

Sample (grade or purity)	Found ($\mu g\, g^{-1}$)[a]		
	Fe	Pb	Bi
$CoCl_2 \cdot 6H_2O$ (G.R.[b])			
A	0.52 ± 0.02	4.40 ± 0.15	N.D.
B	0.50 ± 0.002	4.20 ± 0.12	N.D.
$NiCl_2 \cdot 6H_2O$ (G.R.[b])			
A	1.74 ± 0.03	0.20 ± 0.02[c]	N.D.
B	1.73 ± 0.05	0.16 ± 0.02	N.D.
$CuCl_2 \cdot 6H_2O$ (G.R.[b])			
A	9.16 ± 0.08	0.84 ± 0.04	N.D.
B	9.21 ± 0.41	0.75 ± 0.03	N.D.
Ni powder (99%)			
A	2.52 ± 0.36	2.06 ± 0.15	N.D.
B	2.36 ± 0.32	2.07 ± 0.17	N.D.
Cu powder (99.85%)			
A	7.95 ± 0.31	7.34 ± 0.24	0.28 ± 0.04
B	7.93 ± 0.52	7.53 ± 0.50	0.30 ± 0.03

A: calibration curve method; B: standard addition method; N.D.: not detected.

[a] Mean ± standard deviation ($n = 4$).

[b] Guaranteed reagent grade.

[c] The result was estimated by the extrapolation of the calibration curve.

in the sample solution could be recovered at 97.5–105.6% within relative standard deviation ranges of 1.2–4.1%. These results indicate that the coprecipitation using yttrium phosphate is effective for the separation of iron(III), lead, and bismuth from a solution also containing large amounts of cobalt, nickel, and copper.

3.3.4. Determination of iron, lead, and bismuth

Based on the results mentioned above, the determination of iron, lead, and bismuth in chlorides of cobalt, nickel, and copper and powdery metals of nickel and copper was attempted. As shown in Table 7, the results obtained by the calibration curve and the standard addition methods were in good agreement.

4. Conclusion

Yttrium phosphate is an attractive coprecipitant for iron(III), lead, bismuth, and indium. The coprecipitation with yttrium phosphate could be applied to the separation of iron(III), lead, and bismuth in metals of nickel and copper, and chlorides of cobalt, nickel, and copper(II) prior to their determination by ICP-AES. The coprecipitation would be available for the analyses of not only other salts of cobalt, nickel, and copper(II) but also the metals and salts of other elements. Yttrium phosphate could also be used as a coprecipitant in other determination techniques, including atomic absorption spectrometry.

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