Separation and Concentration of Trace Lead by Coprecipitation with Yttrium Phosphate

Shigehiro Kagaya,* Yasuko Araki, and Kiyoshi Hasegawa
Department of Chemical and Biochemical Engineering, Faculty of Engineering, Toyama University, Gofuku, Toyama 930-8555

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Yttrium phosphate coprecipitates quantitatively lead and iron(III) and hardly does chromium(III), manganese(II), cobalt, nickel, copper, and zinc and alkali and alkaline earth metals at pH about 3. The determination of lead separated and concentrated by the coprecipitation was demonstrated using flame atomic absorption spectrometry.

On the determination of trace metals, a selective separation and concentration of the desired metals from matrixes is frequently required. Coprecipitation is one of the most useful techniques for the separation and concentration of trace metals, and a variety of coprecipitants have been proposed.[1-4] Among them, hydroxides of metals such as iron(III) and aluminum are widely utilized as coprecipitants, however, these collectors generally have poor selectivity for the coprecipitation of metals.[4]

We investigated a new coprecipitant for the separation and concentration of trace metals, and found that yttrium phosphate is a selective collector for lead and iron(III); this collector quantitatively coprecipitates these metals and hardly does chromium(III), manganese(II), cobalt, nickel, copper, and zinc and alkali and alkaline earth metals at pH about 3. In this letter, we report the potential of yttrium phosphate as a coprecipitant for trace metals and the usefulness of the coprecipitation with yttrium phosphate for the preconcentration of lead in water on the flame atomic absorption spectrometric determination.

Measurements were carried out using Hitachi 170-50 atomic absorption spectrometer with Hitachi or Hamamatsu photonics hollow cathode lamp and Hitachi D_2 lamp; air-acetylene flame was used for all measurements. An yttrium solution (5 mg cm^{-3}) was prepared by dissolving yttrium oxide (99.99%) in concentrated hydrochloric acid and diluting with distilled water. All the other reagents used were of guaranteed reagent grade.

Yttrium phosphate precipitated quantitatively over the wide pH range from about 2.5 to at least 10.5 by the addition of ammonia solution. The recoveries of trace metals by coprecipitation with yttrium phosphate from 100 cm^3 of sample solutions spiked with each metal ion were investigated using 5 mg of yttrium and 1.5×10^{-3} mol of phosphoric acid at pH about 3. The precipitate was filtered by suction using a membrane filter (Nihon Millipore Omnipore membrane, pore size 1.0 μm) and then dissolved with nitric acid. After the adequate dilution of the obtained solution with distilled water, the recovery of each metal was determined using flame atomic absorption spectrometry. The results are shown in Table 1. Yttrium phosphate hardly coprecipitated chromium(III), manganese(II), cobalt, nickel, copper, and zinc and alkali and alkaline earth metals. In contrast, lead and iron(III) were able to quantitatively collected with yttrium phosphate.

Table 1. Recoveries of some metal ions by coprecipitation with yttrium phosphate

Ion	Added/μg	Recovery/%	Ion	Added/μg	Recovery/%
Pb^{2+}	10	97.8	Zn^{2+}	10	4.8
Fe^{3+}	10	96.2	Cd^{2+}	5	32.7
Cr^{3+}	10	0.4			
Mn^{2+}	5	5.2	Na^{+}	5000	6.7
Co^{2+}	10	1.2	K^{+}	5000	0.9
Ni^{2+}	10	0.0	Mg^{2+}	5000	0.2
Cu^{2+}	5	2.0	Ca^{2+}	5000	2.7

Coprecipitation was carried out at pH about 3 using 5 mg of yttrium an
1.5×10^{-3} mol of phosphoric acid from 100 cm^3 of aqueous solution.

Table 2. Recoveries of matrix ions and lead by coprecipitation with yttrium phosphate

Matrix			Lead	
Ion	Added/μg	Found/μg	Added/μg	Found/μg
Fe^{3+}	1000	896.2	10	10.5
Cr^{3+}	1000	31.6	10	9.9
Mn^{2+}	1000	14.6	10	9.9
Co^{2+}	1000	1.0	10	10.1
Ni^{2+}	1000	0.8	10	10.1
	10000	1.6	10	9.9
Cu^{2+}	1000	8.7	10	10.2
Zn^{2+}	1000	13.1	10	10.3

Coprecipitation was carried out at pH about 3 using 5 mg of yttrium an
1.5×10^{-3} mol of phosphoric acid from 100 cm^3 of aqueous solution.

Then, we investigated the recovery of lead from the solution coexisting large amount of each heavy metal as a matrix ion. As shown in Table 2, yttrium phosphate was able to collect lead satisfactorily from the solution containing chromium(III), manganese(II), cobalt, nickel, copper, or zinc, which was little recovered by yttrium phosphate. Although a high recovery of iron(III) was obtained in this investigation, lead was quantitatively recovered. These results indicate that yttrium phosphate has an excellent collecting ability for lead.

To evaluate the usefulness of yttrium phosphate as the coprecipitant, we investigated the application of this collector for preconcentration of lead in water on the flame atomic absorption spectrometric determination. Lead was recovered almost completely over the pH range from 2.5 to 10.5. The recovery of lead reached its maximum value at a few minutes after the formation of the precipitate and this value remained almost constant for at least 24 h. The necessary amounts of yttrium and phosphate for the quantitative coprecipitation of lead were more than 1 mg and 0.25×10^{-3} mol, respectively. Yttrium phosphate readily dissolved in common mineral acids. When nitric acid was used for the dissolution of the precipitate, almost constant atomic absorbances of lead were obtained over the concentration range of about 0.3-3.5 mol dm^{-3} in the final sample solution.

From the above, the following recommended procedure for the lead determination can be proposed. To a sample solution (100 cm^3) containing 1.0-30.0 µg of lead, 5 mg of yttrium and 3 cm^3 of 0.5 mol dm^{-3} phosphoric acid solution are added. Yttrium phosphate is then precipitated at pH about 3 using about 7 mol dm^{-3} ammonia solution. After the precipitate is allowed to settle, the solution is filtered by suction using a membrane filter. The collected precipitate is dissolved in 1 cm^3 of about 6.4 mol dm^{-3} nitric acid solution and the solution is diluted to 5 cm^3 with distilled water. The atomic absorbance of lead is then measured at 283.3 nm.

As the calibration curve, a straight line passing through the point of origin was obtained over the concentration range of, at least, 0.2-6.0 µg cm^{-3} of lead in the final sample solution. The relative standard deviation which was obtained from four replicate determinations was 0.8% for 10 µg of lead in 100 cm^3 of the sample solution. Twenty-eight diverse ions tested gave no significant interferences for the determination of lead in 100 cm^3 of the sample solution spiked with 10 µg of lead; lead was determined within ±5% error in the presence of 2000 mg of Cl^-, 1000 mg each of Na^+ and NO_3^-, 560 mg of K^+, 100 mg each of Ca^{2+}, Br^-, I^-, and SO_4^{2-}, 10 mg of Ni^{2+}, 5 mg of Mg^{2+}, 1 mg each of Li^+, Be^{2+}, Sr^{2+}, Ba^{2+}, Al^{3+}, Tl^+, Sn^{4+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, V(V), Cr^{3+}, Mo(VI), W(VI), Mn^{2+}, Fe^{3+} and Co^{2+}, and 0.1 mg of Bi^{3+}. The recoveries of lead from some water samples spiked with 10 µg of lead were examined, and the good results were obtained from tap, river, spring, and sea water samples (Table 3).

Table 3. Recovery of lead from 100 cm^3 of some spiked water samples

Sample	Added/µg	Found/µg	RSD/%
Tap water	10	9.9	2.2
River water	10	10.0	0.1
Spring water	10	9.8	1.1
Sea water	10	9.5	3.2

The recoveries obtained are the average of four replicate determinations.
RSD: Relative standard deviation.

From these results, it can be seen that yttrium phosphate has the excellent ability to collect lead. The coprecipitation method using yttrium phosphate is applicable to the preconcentration of lead in water on the flame atomic absorption spectrometry and is also efficient for the separation of lead from the sample solutions coexisting large amounts of some heavy metals. Furthermore, yttrium phosphate would be the effective collector for iron(III). It seems that the coprecipitation method using yttrium phosphate may be available for the separation and concentration of these metals on not only flame atomic absorption spectrometry but also various determination techniques.

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