

## On-line Preconcentration and Determination Of Lead in Iron and Steel by Flow Injection-Flame Atomic Absorption Spectrometry

**Abstract:** A flow injection method has been developed for the determination of lead in steel samples by coupling on-line preconcentration using a lead selective resin, Pb Resin, with flame atomic absorption spectrometry. The sample prepared as a nitric acid solution is passed through a column packed with Pb Resin. The lead retained on the column is then eluted with ammonium oxalate solution and introduced directly into the nebulizer of a flame atomic absorption spectrometer. The proposed method was successfully applied to the determination of lead in a pure iron certified reference material [ $0.19 \pm 0.01$  ppm ( $n=6$ ); certified value: 0.2 ppm] and a lead free cutting steel certified reference material [ $0.0952 \pm 0.0008\%$  ( $n=3$ ); certified value: 0.097%]. The detection limit ( $3\sigma$ ) was  $0.05 \mu\text{g}$  of lead.

### Introduction:

This work was undertaken to examine a lead-selective chromatographic resin, Pb Resin (Eichrom Technologies, Inc.) to preconcentrate and separate lead from other sample matrix components by a flow injection (FI) technique. Eichrom's Pb Resin is an extraction chromatographic resin which has a high affinity for lead and very low affinity for most other metal cations (including iron.) Figure 1 shows the uptake of Pb as function of nitric acid concentration. Table 1 shows the elution behavior of selected metal ions. It can be seen that iron has no affinity for the Pb Resin column.

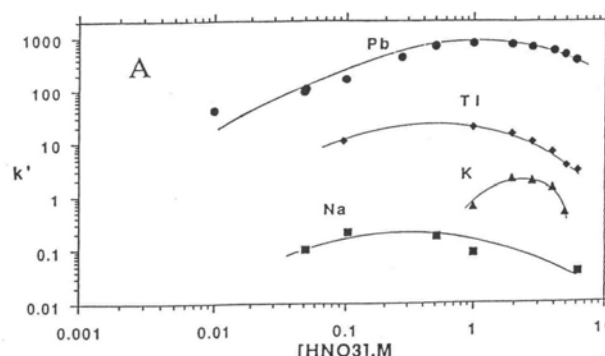


Figure 1. Uptake of various metals on Pb Resin

Element	Portion eluting by column volume (%)				
	0.1 M nitric acid			0.1 M AOX	
	1-5	11-30	31-50	51-60	61-70
Li	100	-	-	-	-
K	100	-	-	-	-
Mg	100	-	-	-	-
Sr	100	-	-	-	-
Cr	100	-	-	-	-
Mn	100	-	-	-	-
Fe	100	-	-	-	-
Ni	100	-	-	-	-
Cu	100	-	-	-	-

Table 1. Elution Behavior of Selected Elements on Pb Resin. AOX = ammonium oxalate

**Experimental:** About 1 gram of high purity iron sample was digested with 3 ml of nitric acid and 1 ml of water with gentle heating. After evaporation nearly to dryness, the residue was dissolved in a mixture of 3 ml of hydrochloric acid and 3 ml of nitric acid, and evaporated nearly to dryness. The residue was then taken up with 3 ml of nitric acid and again evaporated nearly to dryness. The sample treated thus was finally dissolved in 30 ml of 1 M nitric acid with heating.

A flow injection system was constructed with a column of Pb Resin (7.5mm i.d. x 100 mm). The column was conditioned with 1M nitric acid at 3 ml/min for 3 min. The sample solution (as prepared above) was introduced at a flow rate of 3 ml/min. After washing the column with 1 M nitric acid for 2 minutes at the same flow rate, the lead was eluted with approximately 12 mL of 0.1M ammonium oxalate solution at a flow rate of 4.0 ml/min, in a reverse flow direction. The lead was eluted directly into the nebulizer of the atomic absorption spectrometer (AAS) and peak height was used for quantitation of the lead.

**Results:** The effect of the presence of iron(III) on the determination of lead(II) was examined by injecting 30 ml of 1M nitric acid solution containing 10, 100, or 1000 mg of iron(III) and 1 µg lead(II) into the FI system. The results are shown in Table 2. **Lead was quantitatively recovered even from admixtures with iron in excess of lead by a factor of even  $10^4 - 10^6$ .**

<i>Fe(III) added/mg</i>	<i>Pb(II) added/µg</i>	<i>Pb(II) found/µg</i>
10, 100, or 1000	0	<0.02
10	1.00	1.01
10	1.00	1.01
100	1.00	1.04
100	1.00	1.01
1000	1.00	1.01
1000	1.00	1.01

**Table 2. Determination of lead in synthetic mixtures of iron and lead.**

To examine the accuracy and precision of the method, it was applied to the analysis of high purity iron and lead-free cutting steel for lead. The analytical results obtained for certified reference materials provided by the Japan Iron and Steel Federation are summarized in Table 3. The data obtained for both certified reference materials are in good agreement with the reference values and precision is excellent.

Sample size/g	Pb found/µg	Pb content	Ref. Value
<i>Pure iron (certified reference material JSS001-4)</i>			
0.7137	0.142	0.20ppm	
1.039	0.20	0.19	
0.8839	0.174	0.20	
0.7991	0.142	0.18	
0.7006	0.136	0.19	
0.9673	0.182	0.19	
	Mean: <b>0.19 ± 0.008</b>	<b>0.2 ppm</b>	
<i>Lead-free cutting steel (certified reference material JSS519)</i>			
0.1363	130	0.0954%	
0.1303	125	0.0959	
0.1727	163	0.0944	
	Mean: <b>0.0952 ± 0.0008</b>	<b>0.097%</b>	

**Table 3. Determination of lead in certified reference materials supplied by the Japanese Iron and Steel Federation.**

## References:

- Seki, T., et al., *Analytical Sciences*, **2000**, *16*, 513.  
 Horwitz, E. P., et al., *Anal. Chim. Acta*, **1994**, *292*, 263.

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