Liquid - liquid extraction and spectrophotometric determination of lead

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Lead(II) forms a pale yellow water insoluble 1:2 (metal:reagent) complex with potassium propyl xanthate (KPX) in the pH range 4.0 to 6.0. The complex is extracted into carbon tetrachloride and measured at 320 nm. Beer's law is obeyed over the concentration range of 2.5 to 30 ppm of lead. The molar absorptivity and Sandell's sensitivity are $0.75 \times 10^4$ lit. mol$^{-1}$ cm$^{-1}$ and 0.0277 $\mu$g cm$^{-2}$ respectively. The interferences of various ions are studied and the method is applied for the determination of lead in industrial effluents.

Xanthates have been employed as reagents for separation and quantitative determination of transition metals$^{1-3}$. In this note, a simple, sensitive and rapid liquid - liquid extraction and spectrophotometric method has been proposed for the determination of lead with potassium propyl xanthate (KPX). The method is applied for the determination of lead in industrial effluents.

Experimental

A Shimadzu PR-1, UV visible recording spectrophotometer, UV-240 and an Elico digital pH meter (LI-120, Elico, India) were used in the present work.

All the chemicals used were of AR quality. Standard lead (II) solution (1 mg/ml) was prepared by dissolving appropriate quantity of lead nitrate in deionised doubly distilled water. Potassium propyl xanthate was prepared$^4$ and purified$^5$.

Buffer solutions of different pH values were prepared by mixing 0.2 M solutions of hydrochloric acid-potassium chloride (below pH 4.0), sodium acetate-acetic acid (pH 4.0-6.0) and ammonium chloride-ammonium hydroxide (above pH 7.0).

General procedure

A portion of lead solution (25 to 300 $\mu$g) was taken and to this was added the reagent (1 ml, 0.38%) and sodium acetate-acetic acid buffer (2 ml, 0.2 M). The aqueous phase was diluted to 10 ml with deionised doubly distilled water. The light yellow coloured lead - KPX complex formed was extracted with 10 ml of carbon tetrachloride after shaking for 2 min. The absorbance of the organic layer was measured at 320 nm against the reagent blank. The reagent has less absorbance at this wavelength.

Results and discussion

On the basis of preliminary studies, the extraction was found to be complete in the pH range 4.0 to 6.0. Further studies were carried out at pH 5.0 using 2 ml of 0.2 M solutions of sodium acetate-acetic acid buffer. The experiments carried out by varying the amount of the reagent showed that 1 ml of 0.38% solution of the reagent was sufficient for quantitative complexation.

Of the several non-aqueous solvents tried for the extraction of lead (II) from aqueous phase, carbon tetrachloride was found to be the most suitable solvent for the present investigation. Further the extraction was found to be quantitative after 2 min of equilibration.

Under the optimum conditions described above, a calibration curve was plotted. It was linear over the concentration range 2.5 to 30 ppm of lead. The molar absorptivity of lead (II) complex calculated from Beer's law was found to be $0.75 \times 10^4$ lit. mol$^{-1}$ cm$^{-1}$ at 320 nm and Sandell's sensitivity was 0.0277 $\mu$g cm$^{-2}$. The complex was stable practically for 12 h.

The composition of the lead (II) complex was determined by Job's method of continuous variation and mole ratio methods. The equimolar solutions ($1.206 \times 10^{-3} M$) of lead and the reagent were used for these studies. The ratio of metal to reagent was found to be 1:2.

Effect of diverse ions

The following foreign ions (in ppm) did not interfere in the determination of 15 ppm of lead by the above procedure : chloride (500), acetate (400), phosphate (100), bicarbonate (75), tartrate (300), iodide (300), sulphate (100), thiocyanate (60) and bromide (80). But fluoride was found to interfere. Of the metal ions tested, Ba (II) (500), Th (IV) (460), Ce (III) (150), Zn (II) and Sn (II) (400), Sr (II) and U (VI) (450), Mg (II) (300) and Mn (II) (320) were tolerated. Although the metal ions Hg (II) and U (VI) form complexes with KPX, they are not extractable into carbon tetrachloride. The metal ions Fe (III) (400)
and Cu (II) (350) were masked using 1% ascorbic acid and 2% thiourea respectively.

Determination of lead in industrial effluents

The proposed method was applied for the determination of lead in industrial effluents. The results of these investigations are presented in Table 1 along with those obtained by dithizone method.

The present method is rapid and sensitive compared to the methods developed by Deshmukh and Saraswathi\(^6\) and Rao and Chandrasekhar\(^7\) for the determination of lead using alkyl xanthates. The procedure given by Puri \(^8\) et al. for the estimation of lead using butyl xanthate is slightly more sensitive than the present method, but it is time consuming. The molar absorptivity and Sandell’s sensitivity of both the methods are comparable. Naidu and Naidu\(^9\) developed a method for the estimation of lead using n-butyryl xanthate by AAS. The procedure is sensitive, tedious and the recovery is 80% compared to the present method.

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Amount of lead added (μg)</th>
<th>Lead found</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Dithizone method ppm</td>
<td>KPX method ppm</td>
</tr>
<tr>
<td>1</td>
<td>—</td>
<td>4.04</td>
<td>3.86* (0.042)**</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>5.08</td>
<td>4.92 (0.038)</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>7.10</td>
<td>6.89 (0.045)</td>
</tr>
</tbody>
</table>

* Average of three determinations
** Standard deviation

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References


The advantage of the present method over other spectrophotometric methods are (a) wide pH range (4-6) unlike Buhl and Mikula\(^10\) and Alonso \(^11\). (b) greater stability of the complex (12h) compared to Jaiswal and Rao\(^12\) and Zhan\(^13\) methods where the stabilities are 10-20 min and 1h respectively and (c) applicable in the concentration range 2.5 to 30 ppm which is less for the procedures developed by Kish \(^14\) and Naidu and Naidu\(^15\). The developed method is simple, sensitive and rapid and applicable for the determination of lead at trace levels.