Solvent Extraction & Spectrophotometric Determination of Scandium(III) with N-Phenylbenzohydroxamic Acid

Y K AGRAWAL & A K NAGAR
Pharmacy Department, Faculty of Technology and Engineering, M.S. University of Baroda, Kalabhavan, Baroda 390001

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Scandium(III) is extracted with N-phenylbenzohydroxamic acid (PBHA) into isoamyl alcohol at pH 4.5 and then the mixed complexes are formed with xylene orange (XO) or morin. The Sc-PBHA-XO complex is red in colour with a molar absorptivity of $1.7 \times 10^4$ dm$^3$ mol$^{-1}$ cm$^{-1}$ at 540 nm. The Beer’s law is obeyed in the Sc(III) concentration range of 0.05-3.0 ppm. The Sc-PBHA-morin complex gives a greenish yellow fluorescence and the scandium is determined fluorimetrically in the range of 0-1.4 ppm.

N-Phenylbenzohydroxamic acid (PBHA) has earlier been used for the gravimetric estimation of Sc(III). Presently we have shown that PBHA can be successfully used for the extractive separation and spectrophotometric determination of Sc(III).

Procedure

To an aliquot of Sc(III) solution (1.25-75.0 ppm Sc) were added the buffer (10 ml) and water to make up the volume 25 ml. To this was added a solution (10 ml) of PBHA in isoamyl alcohol and content shaken for 5 min. The organic layer was collected and dried over anhydrous Na$_2$SO$_4$. The extraction was repeated with 5 ml of PBHA solution to ensure complete recovery of scandium. To the dried organic extract was added xylene orange solution (1 ml) and the volume made up to 25 ml with isoamyl alcohol. The absorbance of the Sc-PBHA-XO red coloured complex was measured at 540 nm against the reagent blank.

Similarly Sc-PBHA-morin complex was obtained by the addition of a solution (1 ml) of morin in isoamyl alcohol to the Sc-PBHA extract and the final solution made up to 25 ml by adding isoamyl alcohol. The fluorescence intensity of greenish yellow complex was measured after 5 min against the reagent blank.

The extractions were carried out between pH 3.5 and 5.5. Quantitative extraction of Sc(III) was achieved in the narrow pH range of 4.3 to 4.7. For maintaining the desired pH of 5, buffer solution of pH 4.5 (10 ml) was adequate. A 0.2% PBHA solution (10 ml) was found sufficient for complete extraction of scandium.

However, a large excess of reagent could also be used with no adverse effect on the extraction of scandium. Shaking time of 10 min was enough for the complete extraction of scandium. Results of studies using varying concentrations of xylene orange (XO), keeping PBHA concentration constant revealed that 1.0 ml of 0.25% of XO gave the most satisfactory results. Higher XO concentrations increased the intensity of the blank and hence 10 ml of XO solutions were used throughout this study. Similarly 1 ml of 0.5% morin was found adequate for fluorimetric determination of Sc(III). Increase in the concentration of morin increased the interference of the blank.

Amongst the solvents, viz. chloroform, benzene, methyl isobutyl ketone (MIBK) and isoamyl alcohol, the best solvent for extraction, spectrophotometric and fluorimetric determination of scandium was isoamyl alcohol. The colour of the complex was stable for more than 48 hr and the absorbance and fluorescent intensities were constant.

The Sc-PBHA-XO complex obeys Beer’s law in the Sc(III) concentration 0.05-3.0 ppm, giving a molar absorptivity of $1.7 \times 10^4$ dm$^3$ mol$^{-1}$ cm$^{-1}$ at 540 nm. In the fluorimetric determination, Beer’s law is obeyed in the Sc(III) concentration range of 0-1.4 ppm.

Scandium(III) at 2 ppm level was determined in the presence of several closely associated metal ions by the recommended procedure. Most of the common ions associated with scandium, except Bi$^{3+}$, Hg$^{2+}$, Ga$^{3+}$, Ti$^{4+}$, Th$^{4+}$ and large amounts of Ce$^{4+}$, U$^{6+}$ and V$^{5+}$, were tolerated. However, Ta$^{5+}$ and Nb$^{5+}$ interfered in the fluorimetric determination of Sc(III).

Table I—Estimation of Scandium in Standard Geological (USGS) and IAEA Soil-5 Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Sc(III) (ppm)</th>
<th>Standard value</th>
<th>Found*</th>
<th>% Error</th>
<th>Standard deviation</th>
<th>Relative standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>BCR-1</td>
<td>32.0</td>
<td>32.03</td>
<td>0.09</td>
<td>±0.05</td>
<td>0.15</td>
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</tr>
<tr>
<td>(52/19)</td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>W1</td>
<td>35.9</td>
<td>35.88</td>
<td>0.06</td>
<td>±0.03</td>
<td>0.08</td>
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<tr>
<td>G2</td>
<td>3.2</td>
<td>3.23</td>
<td>0.03</td>
<td>±0.05</td>
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<tr>
<td>GSP-I</td>
<td>6.1</td>
<td>6.10</td>
<td>0.00</td>
<td>±0.01</td>
<td>0.16</td>
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<td>(17/22)</td>
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<tr>
<td>Soil-5</td>
<td>15.5</td>
<td>15.51</td>
<td>0.06</td>
<td>±0.02</td>
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<td>Soil†</td>
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<td></td>
<td>±0.01</td>
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<td>Plant†</td>
<td>1.10</td>
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<td></td>
<td>±0.02</td>
<td>1.81</td>
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<td>Water†</td>
<td>0.30</td>
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<td></td>
<td>±0.01</td>
<td>3.33</td>
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</table>

*Average of 6 determinations
†Samples from Nandesari area of Baroda
was found to be from 0.08 to 0.33 ppm. For comparison the nitrate contents of the same samples have been determined after hydrazine reduction which gives lower concentration values as expected.

The interference studies have been performed with the ions commonly found in ground water. High excess of chloride, sulphate, carbonate and bicarbonate do not interfere. Nitrite in small amounts can be removed by sulphamic acid treatment. Calcium and magnesium can be tolerated in 100-fold excess over that of nitrate. The interference of Fe$^{3+}$, Fe$^{2+}$, Ni$^{2+}$, Al$^{3+}$, Zn$^{2+}$ and Mn$^{2+}$ can be removed by prior separation on cation exchange column. Oxidising anions like persulphate, chlorate and perchlorate interfere with the determination.

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References