Voltammetric determination of trace level nickel in presence of cadmium, lead and zinc

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The polarographic reduction of nickel has been studied employing DC polarography and differential pulse polarography in ammonium tartrate buffer. Optimum conditions have been found for devising a simple and routine method of determination of microamounts of nickel in presence of cadmium, lead and zinc.

Results and discussion

In the present report a differential pulse polarographic (DPP) method has been proposed for the determination of nickel alone and in presence of cadmium, lead and zinc. Ammonium tartrate buffer was found to be a suitable medium for this purpose. The method has been applied to determine the nickel contents of the industrial waste water samples of Jodhpur.

Experimental

A PAR 174-A polarographic analyzer alongwith a drop timer (Model 174/70) and x-y recorder was used for DC and DPP experiments. The instrumental settings for DPP were as follows: pulse amplitude 50 mV, clock time of pulse 1 sec and scan rate 5 mV/sec. Potentials were measured against a saturated calomel electrode (SCE).

Cyclic voltammetry was performed on a BAS cyclic voltammograph (Model CV-27) using a hanging mercury drop electrode (HMDE) as working electrode and Ag/AgCl as reference electrode. Initial and final potentials were −0.5 V and −1.3 V, respectively. The scan rate was varied from 20 to 100 mV/sec.

A platinum wire was used as the auxiliary electrode in all the observations.

Sample treatment

Samples were collected from different selected points of Marudhar and Basni Industrial Areas of Jodhpur in cleaned polyethylene sample containers. All glasswares and sample containers were soaked in 2M nitric acid for at least one week and washed several times with doubly distilled water. Sample containers were kept filled with 0.1 M hydrochloric acid until the time of sample collection. The water samples were filtered to separate the suspended particulate matter. To 50 ml of this aliquot, 1 ml of an oxidising mixture (10 ml H₂SO₄ + 1.5 g K₂S₂O₈/100 ml) was added and contents were heated till the solution fumed to remove organic matrices. Subsequently, the contents were transferred to a volumetric flask and made upto the requisite volume.

All the experiments were carried out in an air-conditioned laboratory where the temperature was maintained at 26 ± 1°C. The solutions were deaerated by bubbling pure nitrogen prior to experiments.

Results and discussion

The nickel(II) ion in presence of ammonium tartrate buffer (pH 9.0) displayed a single reduction wave whose half-wave potential was observed at −0.94 V. The $E_{1/2}$ of the wave was found to be pH-dependent. At higher pH (10.5 or more) the polarogram of nickel consisted of two waves at 0.95 and 1.27 V. The limiting current of the first wave did not appear to be linear with respect to the nickel concentration. The second wave also could not be used for nickel estimation in presence of zinc because of similar potential range of their reduction.

To check the reversibility of electrode reaction, cyclic voltammetry of nickel at pH 9.0 was carried out at different scan rates. Cyclic voltammograms showed a single cathodic peak ($E_{pc} = −1.0$ V at scan rate 60 mV/sec). No corresponding peak was obtained in the reverse scan suggesting an irreversible electrode process. Thus DPP was carried out for nickel determination where a sharp

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ni added (ppb)</th>
<th>Ni found (ppb)</th>
<th>Standard deviation (%)</th>
<th>% Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>120</td>
<td>122.98*</td>
<td>6.4</td>
<td>2.5</td>
</tr>
<tr>
<td>Industrial waste water</td>
<td>—</td>
<td>380.29†</td>
<td>8.3</td>
<td>—</td>
</tr>
</tbody>
</table>

*Average of four determinations
†Average of six determinations
Table 2—Concentration of Ni in waste waters determined by DPP and AAS

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Conc. of Ni (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>DPP</td>
</tr>
<tr>
<td>1</td>
<td>0.38</td>
</tr>
<tr>
<td>2</td>
<td>0.34</td>
</tr>
<tr>
<td>3</td>
<td>0.40</td>
</tr>
<tr>
<td>4</td>
<td>0.42</td>
</tr>
<tr>
<td>Average</td>
<td>0.38</td>
</tr>
</tbody>
</table>

DP peak was observed at $-0.94 \text{ V}$. The $E_p$ appeared to be dependent on the pH of the test solution. The peak height was found to be proportional to the concentration of nickel in the range 0.05-11.4 ppm. DP polarograms were also recorded for a mixture containing lead(II), cadmium(II), nickel(II) and zinc(II) metal ions. The DP peaks for these metal ions were distinguishable from each other and had the following peak potentials (vs SCE): Lead, $-0.51 \text{ V}$; cadmium, $-0.66 \text{ V}$; nickel, $-0.94 \text{ V}$; and zinc, $-1.16 \text{ V}$.

Thus in ammonium tartrate buffer (pH 9.0) nickel can be simultaneously determined along with lead, cadmium and zinc without interference as the peak potential of nickel did not overlap with peak of any other ion. The method was checked for precision and accuracy and the observations are listed in Table 1. The detection limit was found to be 50 ppb.

Estimation of nickel in water samples

The DPP reduction of Ni(II) to metallic state has been made the basis of determination of nickel in aqueous matrices at micro levels. The digested water samples were taken in the polarographic cell. The pH was adjusted to 9.0 by adding ammonium tartrate buffer and the DP polarograms were recorded in the potential range of $-0.8$ to $-1.1 \text{ V}$. The peak currents were measured at $-0.94 \text{ V}$ after making blank correction. The concentration of nickel was determined by standard addition method. The total number of samples analysed was 37. The minimum amount of Ni found was 0.248 ppm and maximum amount of Ni found was 1.090 ppm. Atomic absorption spectroscopic method was used to compare the values obtained by DPP. These observations are listed in Table 2.

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References