Turbidimetric estimation of cesium in the typical leachants of nuclear waste

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Received 29 April 1991; revised 29 July 1991; accepted 27 August 1991

A simple, fast and reliable turbidimetric method has been developed for the estimation of cesium using sodium tetraphenylborate as a precipitating reagent in the leachants of nuclear waste. The reliability and the reproducibility of the method is found to be excellent in demineralised water, ground water and synthetic samples of sea water.

The solid and liquid wastes generated in the nuclear industry need immobilization in a matrix, viz., glass, cement etc. The integrity of such a matrix is generally ascertained by carrying out leaching studies. Cesium is one of the major radionuclides of importance in waste which has higher diffusivity than some of the important radionuclides such as strontium, cobalt etc. In the present study demineralised water, ground water, and synthetic samples of sea water as well as actual sea water were used as leachants. Inactive cesium is normally used in the simulation studies considering the ease of handling. Estimation of cesium in the leachant at a concentration below 1 μg/ml is generally carried out using atomic absorption spectrophotometer1. An alternate simple method based on turbidimetry using UV/visible spectrophotometer has been developed which is equally reliable in the estimation of cesium at a concentration range of 1 to 10 μg/ml. Sodium tetraphenylborate (STB)2-3 which is normally used in the estimation of potassium, radioactive cesium4 and also as a precipitant for the removal of cesium5, has been employed in this study as the precipitating reagent. The precipitate of cesium tetraphenylborate remains in suspension for a fairly long time allowing accurate measurement of the absorbance. The present paper summarises the results obtained in the estimation of cesium in different media as well as in aqueous solutions having different pH values.

Experimental
All the chemicals used in the experiments were of GR/AR grade. A 1% solution of sodium tetraphenylborate (GR/Romali) was prepared in demineralised water. The stock solution was prepared according to the method given in the literature6. A known amount of cesium chloride (1.267 g) was dissolved in demineralised water and diluted to 250 ml. A sample (25 ml) of this solution was placed in a platinum dish and a drop of conc. H2SO4 was added. The contents were evaporated to dryness and heated to constant weight at ~800°C. The concentration of cesium was calculated using the following equation:

\[ \text{Cs(μg/ml)} = (40)(0.734) (\text{Wt of residue}) \]

In order to produce a uniform suspension before the measurement of absorbance, the conditions such as sequence and rate of addition of reagents, temperature etc., were strictly adhered to, in accordance with the standard procedure7. Standard solutions of cesium were prepared in the range of 1-10 μg/ml in 100 ml flask each containing sodium tetraphenylborate (1 ml, 1%). The absorbance of cesium tetraphenylborate suspension was measured with a Hitachi model spectrophotometer at a scan speed of 200 nm/minute. From the absorption curve it was found that the absorbance was almost constant in the range of 250-600 nm. The absorbance was measured at 325 nm as it falls in the plateau region. The calibration curve was found to be linear in the range of 1-10 μg/ml.

Results and discussion

Determination of cesium in spiked water samples

250 μg of cesium was dissolved in 100 ml each of ground water8, synthetic samples of sea water9 and actual sea water. Cesium was estimated spectrophotometrically at 325 nm. Similarly, cesium was estimated in demineralised water at different pH values. The experiments were repeated six times. The relative error was evaluated with reference to demineralised water; the relative standard deviation (RSD) in each case is given in Table 1. The results indicate that there was a negative interference in the lower pH region and a positive interference at a higher pH. Furthermore, the reproducibility of absorbance values in the synthetic samples of sea water was better than that in actual sea water. The low accuracy of cesium with increase in salt concentration is due to increase in the solubility of cesium tetraphenylborate. This method is observed to be
ideal in the range of 1-2.5 µg/ml which can be extended up to 10 µg/ml with lower accuracy. This method, therefore, can be adopted for the estimation of cesium in demineralised water, ground water and synthetic samples of sea water as it is a simple and rapid method with excellent reproducibility.

Table 1—Estimation of cesium in different media

<table>
<thead>
<tr>
<th>Sample</th>
<th>Relative error</th>
<th>Relative std. dev.</th>
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<tbody>
<tr>
<td>Ground water</td>
<td>2</td>
<td>5</td>
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<tr>
<td>Actual sea water</td>
<td>16</td>
<td>34</td>
</tr>
<tr>
<td>Synthetic sea water</td>
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<td>9</td>
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<td>pH, 2</td>
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<td>5</td>
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<td>5.5</td>
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<tr>
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<tr>
<td></td>
<td>4</td>
<td>8.3</td>
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<td></td>
<td>13</td>
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References