Detection of Hexacyanoferrate(II) and Hexacyanoferrate(III) Using Setoglaucine 0 and Setocyanine Supra

By
V. V. S. Eswara Dutt and N. Venkateswara Rao

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Recently we have reported the use of the triphenylmethane dyes Setoglaucine 0 and Setocyanine Supra (Colour Index Nos. 42025 and 42140 respectively) for the detection of cerium(IV)¹ and palladium². We have now observed that these two dyes can be used for the detection of hexacyanoferrate(II) and hexacyanoferrate(III).

Experimental

Dye solutions. 0.3% solutions of the dyes, Setoglaucine 0 and Setocyanine Supra (from Edward Gurr Ltd., London), are prepared in distilled water.

Hexacyanoferrate(II) and hexacyanoferrate(III) solutions. 0.05 N solutions are prepared from reagent grade samples of potassium salts. These solutions are suitably diluted with distilled water.

The chemicals used in the interference studies are of reagent grade quality.

Procedure

0.1 ml of 0.3% solution of Setoglaucine 0 or Setocyanine Supra is placed on a spot plate, treated with one drop (0.05 ml) of the test

Table I. Observations and limits of identification and dilution

<table>
<thead>
<tr>
<th>Ion detected</th>
<th>Observation</th>
<th>Identification limit, µg in 0.5 ml</th>
<th>Dilution limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexacyanoferrate(II)</td>
<td>Blue precipitate</td>
<td>0.25</td>
<td>1 : 2·10⁶</td>
</tr>
<tr>
<td>Hexacyanoferrate(III)</td>
<td>Green precipitate</td>
<td>6.5</td>
<td>1 : 7.6·10⁴</td>
</tr>
</tbody>
</table>
solution, the mixture is diluted to 0.5 ml with distilled water and thoroughly mixed. The observations and limits of identification and dilution are recorded in Table I.

Results and Discussion

We believe that the precipitates obtained are due to the formation of addition compounds between the dyes and hexacyanoferrate(II) or hexacyanoferrate(III). Further work is in progress to elucidate this point.

We have found that the tests can be carried out in slightly acidic medium also. If the acidity is increased beyond 0.1 N, the sensitivity of the test is decreased and at 0.5 N and above, the reaction is almost completely inhibited. The optimum pH range for a satisfactory test has been found to be 2 to 8. Above pH 10, the carbinol bases of the dyes slowly separate out.

Interferences

Chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, thiocyanate, acetate, nitrate, phosphate, and tartrate do not interfere in the tests even if they are present in 100-fold excess concentration. Although vanadate, dichromate, permanganate, perchlorate, periodate, persulphate, molybdate, tungstate, and thiosulphate also form coloured precipitates with the two dyes, the following amounts of these ions do not interfere in the tests for hexacyanoferrate(II) and hexacyanoferrate(III).

Detection of hexacyanoferrate(II). Vanadate: 20-fold excess; perchlorate and thiosulphate: 100-fold excess; periodate: 200-fold excess; persulphate: 120-fold excess; molybdate and tungstate: 10-fold excess. The sensitivity of the test is reduced in presence of dichromate and permanganate. This is perhaps due to the oxidation of the hexacyanoferrate(II) by these two oxidants.

Detection of hexacyanoferrate(III). Vanadate: equal concentration; dichromate: 2-fold excess; permanganate: 3-fold excess; perchlorate and persulphate: 5-fold excess; molybdate, tungstate, and thiosulphate: 6-fold excess; and periodate: 10-fold excess.

Heavy metal ions, which form precipitates with hexacyanoferrate(II) and hexacyanoferrate(III), interfere in the tests.

In this connection, it may be pointed out that the test now reported for hexacyanoferrate(II) is more sensitive than the tests using uranyl nitrate (Feigl\textsuperscript{3}, identification limit: 1.0 μg) or ferric chloride (Davidson\textsuperscript{4}, identification limit: 1.3 μg).