DIAMMONIUM-5, 5'-INDIGO DISULPHONATE
AS A REAGENT FOR THE GRAVIMETRIC
DETERMINATION OF LANTHANUM,
GADOLINIUM AND YTTRIUM

BY B. D. JAIN AND J. J. SINGH
(Department of Chemistry, University of Delhi, Delhi-6)
Received October 25, 1962
(Communicated by Prof. T. R. Seshadri, F.R.S., F.A.Sc.)

INTRODUCTION

DIAMMONIUM-5, 5'-INDIGO DISULPHONATE forms insoluble complexes with a
large number of cations and has earlier been used by the present authors
for the gravimetric determination of thorium, cerium (III), zirconium as
well as their separation from other elements.1-2 It has now been found
that when an aqueous solution of the reagent is added to trivalent rare-earth
solutions, it precipitates quantitatively these metals and can be used for their
estimation.

Lanthanum, gadolinium and yttrium complexes are quantitatively
precipitated between pH 3·0-7·5 and after ignition are weighed as oxides.
Quantities as low as 4·75 mg. of La₂O₃, 3·3 mg. of Gd₂O₃ and 4·2 mg. of
Y₂O₃ have been thus quantitatively determined.

EXPERIMENTAL

Reagents and equipment.—Diammonium-5, 5'-indigo disulphonate was
prepared as reported earlier¹ and an aqueous solution (1%) of it was used
as the precipitant while its 0·05% aqueous solution was used as wash liquid.

Gadolinium nitrate and yttrium nitrate were made from Gd₂O₃ (Johnson
Mathey R.E. 454) and Y₂O₃ (Johnson Mathey R.E. 112) respectively.
Lanthanum nitrate (E. Merck\textsuperscript{a} Pro Analysis) was used for making its solution. All other salts used were of A.R. quality.

Dilute hydrochloric acid and ammonium hydroxide were used for pH
adjustment.

Beckman pH Meter Model H 2, with a suitable glass electrode, was
used for pH measurements.
**General method for the estimation of lanthanons with diammonium-5, 5'-indigo disulphonate.**—On the addition of about 10 ml. of 1·0% aqueous solution of diammonium-5, 5'-indigo disulphonate to approximately 200 ml. of lanthanon solution (nitrate or chloride) containing about 20 mg. lanthanon oxide at pH 3·0-7·5, a dark blue precipitate is immediately formed. This precipitate is digested on a steam-bath for about 20 minutes, cooled to room temperature and filtered through a Whatman No. 42 filter-paper and washed with a dilute solution of the reagent. The precipitate, after drying and ignition, is weighed as oxide. Tables I, II and III give the results obtained in case of lanthanum, gadolinium and yttrium using this reagent.

**TABLE I**

**Determination of lanthanum**

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Weight of La₂O₃ present (with oxine) (mg.)</th>
<th>Weight of La₂O₃ found (with present reagent) (mg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>23·8</td>
<td>23·8</td>
</tr>
<tr>
<td>2</td>
<td>23·8</td>
<td>23·8</td>
</tr>
<tr>
<td>3</td>
<td>11·9</td>
<td>11·9</td>
</tr>
<tr>
<td>4</td>
<td>11·9</td>
<td>11·9</td>
</tr>
<tr>
<td>5</td>
<td>4·75</td>
<td>4·75</td>
</tr>
<tr>
<td>6</td>
<td>4·75</td>
<td>4·75</td>
</tr>
</tbody>
</table>

**Effect of pH on the formation of the lanthanum, gadolinium and yttrium complexes.**—The effect of pH on the formation of the lanthanum, gadolinium and yttrium complexes with diammonium-5, 5'-indigo disulphonate was studied over a wide range of pH (0·5-8·0) using dilute ammonium hydroxide solution for higher pH and dilute hydrochloric acid for lower pH. It was found that though the precipitation of the complexes started in all cases at low pH, it was quantitative only above pH 3·0. Table IV gives the results of the determination of lanthanum, gadolinium and yttrium at different pH. Since the rare-earth solutions get hydrolysed at about pH 8·0 the determinations were restricted up to pH 7·5 only.