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Gravimetric Estimation of Yttrium (III) Using Hydroxy Lawsone

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ABSTRACT

Isonaphthazarin (2-hydroxy Lawsone i.e. 2,3-dihydroxy-1,4-naphthalenedione) is well known spectrophotometric reagent for thorium determination[1]. Yttrium is found to be an important element in analytical, biological Chemistry as well as in industrial purposes. Because of its novel coordination geometry, it is used for the kinetic studies. It is also used in industrial glass devices, electronic devices, graded index lenses and semiconductors. Yttrium can be gravimetrically estimated by using an analytical reagent Isonaphthazarin.

Keywords: Lawsone, Isonaphthazarin, Yttrium metal, Gravimetric Estimation.

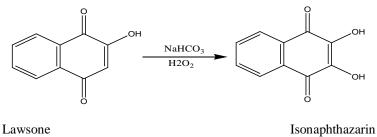
INTRODUCTION

Lawsone (2-hydroxy-1,4-naphthalenedione), a naturally occurring pigment has been used as a analytical reagent [2-6]. Its oxime derivatives are shown analytical applications as a gravimetric reagent [7,8] for Nickel (II) and Palladium (II). Aizenberg [9] has reported the gravimetric determination of nickel (II) using Juglone (5-hydroxy-1,4-naphthalenedione). The syntheses, characterization and analytical studies on metal complexes of monohydroxy-1,4-naphthalenediones [10-17], dihydroxy-1,4-naphthalenediones [1,18-22] has been reported. Here we are reporting 2,3-dihydroxy-1,4-naphthalenedione is gravimetric reagent for estimation of Yttrium.

MATERIALS AND METHODS

All chemicals used were of A. R. or equivalent Grade. Lawsone were purchased from Fluka A. G.The elemental analysis for the percentage of carbon, hydrogen and other elements were performed in the micro analyzer. The electronic spectra were recorded in ethyl alcohol on using Shimadzu UV-300 Spectrophotometer model using 1 cm matched quartz cell. The Infra Red spectra were recorded in KBr on FTIR Shimadzu – 8400. The ¹H NMR spectra of ligand were recorded on VARIAN MERCURY YH – 300 MHz IN CDCl₃ in the Department of Chemistry, Pune. The structures of Isonaphthazarin is simulated by using Cambridge software (Chemoffice 2008 – Chem3D ultra 8.0 followed by MM2) for to measure bond length and bond angle.

Synthesis of reagent (Isonaphthazarin)



Scheme 1. Synthesis of Isonaphthazarin

Isonaphthazarin was synthesized from Lawsone[19]. 5.115 mmol (8.9 g) of Lawsone was added to about 500 ml of water containing 5.119 mmol (4.3 g) of sodium bicarbonate. This was treated with 17.647 mmol (20 ml) of 30% H₂O₂ at room temperature and kept for 24 hours without disturbing in a dark cupboard. The resultant dark red needles were filtered off, dried in *vacuo* over fused calcium chloride at ambient temperature and re-crystallize from acetone. The MM2 structure of isonaphthazarin is given in figure 1, while bond length and bond angles were given in table 1.



Figure 1. MM2 structure of Isonaphthazarin

Bond Lengths in Å		Bond Angles			
C(1)-C(2) 1.422,	C(1)-C(6) 1.699,	C(2)-C(1)-C(6) 119.124,	C(2)-C(1)-C(7) 119.929,	C(6)-C(1)-C(7) 120.947	
C(1)-C(7) 1.418	C(2)-C(3) 1.699,	C(1)-C(2)-C(3) 119.124,	C(1)-C(2)-C(10), 119.93,	C(3)-C(2)-C(10) 120.946,	
C(2)-C(10) 1.418,	C(3)-C(4) 1.701	C(2)-C(3)-C(4) 119.918,	C(2)-C(3)-O(12) 120.548,	C(4)-C(3)-O(12)119.532,	
C(3)-O(12) 1.213,	C(4)-C(5) 1.326,	C(3)-C(4)-C(5) 120.955,	C(3)-C(4)-O(15) 119.675,	C(5)-C(4)-O(15) 119.369,	
C(4)-O(15) 1.411	C(5)-C(6) 1.701,	C(4)-C(5)-C(6) 120.956,	C(4)-C(5)-O(13)119.368,	C(6)-C(5)-O(13) 119.677	
C(5)-O(13) 1.411,	C(6)-O(11) 1.213	C(1)-C(6)-C(5) 119.917,	C(1)-C(6)-O(11) 120.546,	C(5)-C(6)-O(11) 119.530	
C(7)-C(8) 1.419,	C(7)-H(20) 1.12,	C(1)-C(7)-C(8) 120.059,	C(1)-C(7)-H(20)120.099,	C(8)-C(7)-H(20) 119.842	
C(8)-C(9) 1.417	C(8)-H(18) 1.123,	C(7)-C(8)-C(9)120.011,	C(7)-C(8)-H(18) 119.986,	C(9)-C(8)-H(18)120.003	
C(9)-C(10) 1.419,	C(9)-H(17) 1.123	C(8)-C(9)-C(10) 120.011,	C(8)-C(9)-H(17)120.003,	С(10)-С(9)-Н(17) 119.986	
C(10)-H(19) 1.123,	O(13)-H(14) 0.991,	C(2)-C(10)-C(9)120.059,	C(2)-C(10)-H(19) 120.099,	C(9)-C(10)-H(19)119.842	
O(15)-H(16) 0.991		C(5)-O(13)-H(14) 108.807,	C(4)-O(15)-H(16)108.807		

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Characterization: Yield, 3.2 g, 32.9% (1.683 mmol), m. p. 277 $^{\circ}$ C (m. p. 267 $^{\circ}$ C). Analysis: Calculated for C₁₀H₆O₄: C, 63.16; H, 3.17. Found: C, 63.14; H, 3.18. IR: 3310, 1630, 1585, 1272, 1250 cm⁻¹.UV: 284 (Benzeniod),334 (Quinonoied) and 454 ($n \rightarrow \pi^*$) nm. 1 H NMR: (dd, δ 7.73, C-6-H and C-7-H), (dd, δ 7.89, C-5-H and C-8-H) ppm.

Preparation of Yttrium (II) solution: The known amount of pure Y_2O_3 was heated in a silica crucible to dryness in the presence of sufficient amount of concentrated HCl. It was then dissolved in distilled water and diluted to volume ~ 5.0 mg ml⁻¹.

Preparation solution: The solution (1% w/v) of isonaphthazarin was prepared in ethyl alcohol.

Preparation of precipitate: The warm solution of known amount and volume of Yttrium was taken in a beaker. The 1% ethanolic solution of isonaphthazarin reagent was then added in excess with continuous stirring. After addition of reagent, pH of the solution was adjusted to 4.5 to 5.0 by adding sodium acetate solution, when yttrium isonaphthazarinate was precipitated. The precipitate was digested on a sand bath for an hour and then filtered through Gooch crucible (G-4) and washed with distilled water till colorless filtrate was obtained. The precipitate is directly weighted after drying at 110°C. The amount of Yttrium was calculated from the weight as Yttrium complex. In other way the precipitate was filtered through Whatman filter paper and ignite, then weighed as a Y_2O_3 .

RESULTS AND DISCUSSION

The blue colored precipitate of Yttrium complex with reagent isonaphthazarin was characterized on the basis of elemental analysis, spectroscopy (Infrared and electronic) and Thermogravimetry. The data is consistent with $C_{30}H_{16}O_{16}Y_2$. Results of the gravimetric estimation for Yttrium complex are compiled in table 2. The results indicate that the isonaphthazarin can serve as a satisfactory reagent for the gravimetric estimation of Yttrium (III) ions. Although, the isonaphthazarin is, however, not selective and many common metal ions interfere in the estimation. But this reagent is highly sensitive to form a complex with Yttrium. Therefore, isonaphthazarin is very important for gravimetric estimation of Yttrium.

mg of Y(III)	Wt. of Y(III) Observed and error					
taken in	By complex	% error	By oxide	% error		
solution	formation		formation			
	method		method			
5	4.7	-6.00	4.9	-2.0		
10	9.6	-4.00	9.7	-3.00		
15	14.7	-2.00	15.1	+0.6		
20	19.8	-1.00	20.1	+0.5		

Table 2. Results of the Gravimetric Estimation of Yttrium in mg

It is observed that, as we go for the higher concentration from 5 mg, the error minimizes. For the concentration 20 mg of Yttrium shows very less error in estimation.

APPLICATIONS

Reagent isonaphthazarin can be successfully applied for the estimation of Yttrium metal ions in trace amount in ores, alloys, synthetic mixtures and unknown samples. Both complex formation and oxide formation method are good for the estimation of Yttrium metal by gravimetric method.

CONCLUSIONS

Isonaphthazarin is a sensitive reagent as a gravimetric reagent for Yttrium estimation. It is useful for above than the 5 mg Yttrium present in the solution. Yttrium can be determined quantitatively by gravimetric method using Isonaphthazarin reagent.

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