

# Solvent Extraction and Determination of Yttrium in Geological Samples by Atomic Absorption Spectrophotometry

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A simple and rapid method for the concentration of yttrium by solvent extraction with 100% tri-n-butyl phosphate (T.B.P.) in 12N HNO<sub>3</sub> and stripping by water followed by its determination in an emission mode by Atomic Absorption Spectrophotometer (A.A.S.) in 50% acetone medium has been developed.

## INTRODUCTION

Literature survey for the estimation of yttrium<sup>1-4</sup> by Atomic Absorption Spectrophotometry indicated that most of the methods make use of classical techniques i.e. fluoride-oxalate-hydroxide precipitation for concentrating yttrium. This method was found to be time consuming, cumbersome and there is possibility of its loss during various stages. Moreover, this method of concentration becomes unmanageable when one is compelled to take higher weight of samples containing low amount of yttrium.

It was thought worthwhile to try an alternative procedure for the concentration of yttrium. A solvent extraction procedure for concentrating yttrium has been tried. Yttrium was subsequently determined by A.A.S. in an emission mode in 50% acetone or aqueous medium depending upon its concentration.

## EXPERIMENTAL

### Materials and Methods

Stock solution of yttrium was prepared from standard salt yttrium oxide (Koch Light 99.99%) in dil. HCl and diluted to the required concentration (1 ml = 100 µg). Solutions of foreign elements (niobium, tantalum, titanium, cerium, europium, lanthanum, phosphorus, etc.) studied were prepared by standard methods using respective reagent grade salts. Tri-n-butyl phosphate (L.R., B.D.H.) was used. A Varian model AA-475 Atomic Absorption Spectrophotometer with a single slot, nitrous oxide-acetylene burner was used for all the experiments. Instrumental conditions are given in Table 1.

Calibration curves were made with low or high range standards as required. The standards were prepared in 50% acetone, methanol, ethanol and in aqueous solution containing different spectroscopic buffers (La, Na, K) and 2% HCl in order to choose the best solvent and spectroscopic buffer for obtaining optimum sensitivity and stability for measurement with A.A.S. at the above instrumental conditions.

TABLE 1

INSTRUMENTAL CONDITIONS FOR DETERMINATION OF YTTRIUM	
Mode	= Emission
Wavelength	= 407.7 nm
Slit width	= 0.2 nm
Fuel	= C <sub>2</sub> H <sub>2</sub> (3.7 L/min)
Oxidant	= N <sub>2</sub> O (8.3 L/min)
Flame condition	= Reducing red cone (1.5-2 cm)
Optimum burner height	= 13.2 mm

### Procedure for Solvent Extraction

An aliquot of standard yttrium solution (100 µg) in 12N HNO<sub>3</sub> medium was extracted with 10 ml 100% Tri-n-butyl phosphate (T.B.P.)<sup>5</sup> twice for 5 minutes. The organic phase was stripped twice with 10 ml water for five minutes. Recovery of yttrium was checked in stripped solution spectrophotometrically by Alizarin-Red-S' method<sup>6</sup>. Besides, the aqueous phase after T.B.P. extraction was tested for the presence of yttrium. This method of concentration was followed for standard samples as well as for geological samples.

### Study of Interference

In order to check the scope of the method for natural samples, a study on recovery of yttrium in presence of various associated elements like cerium, niobium, tantalum, titanium, europium, thorium, lanthanum and phosphate, oxalate anions were made using the above method. Recovery of yttrium (200 µg) was checked in the presence of above mentioned elements in the range (1 mg to 8 mg).

## RESULTS AND DISCUSSION

It is observed that yttrium is quantitatively extracted with 100% T.B.P. in 12N HNO<sub>3</sub> medium when extracted twice for five minutes each and is completely stripped from T.B.P. phase when shaken twice with water for five minutes each. Extraction of yttrium decreases below this concentration of HNO<sub>3</sub>. This was confirmed after testing the stripped solution and the aqueous solution left after extraction spectrophotometrically with Alizarin-red-S' reagent<sup>6</sup> (Table 2). It is also observed by using this method that 200 µg of yttrium in presence of niobium (8 mg), tantalum (8 mg), cerium (8 mg), thorium (5 mg), titanium (2 mg), europium (1 mg) and lanthanum (8 mg) is quantitatively recovered. However, oxalates prevent the extraction of yttrium to some extent. Phosphorus (5 mg) does not interfere in the extraction process (Table 3).

It was observed that absorption methods whether in aqueous or containing organic miscible solvents (acetone, methanol, ethanol) are very less sensitive compared to emission mode for determination of yttrium. So

TABLE 2  
CHECKING THE RECOVERY OF YTTRIUM SPECTROPHOTOMETRICALLY<sup>6</sup>

Y taken ( $\mu\text{g}$ )	Y found in the aqueous phase after extraction ( $\mu\text{g}$ )	Y found in the stripped solution ( $\mu\text{g}$ )	% Extraction
500	nil	491.5	98
1,000	nil	990.9	99

TABLE 3  
STUDY OF RECOVERY OF YTTRIUM IN PRESENCE OF FOREIGN ELEMENTS

Yttrium taken = 200  $\mu\text{g}$

S.No.	Element added (mg)	Recovery of Y%
1	Nb (8)	99
2	Ta (8)	93
3	P (5)	99
4	Ce (8)	99
5	Th (5)	99
6	Ti (2)	98
7	Eu (1)	99
8	La (8)	97

studies were conducted in the emission mode only. It was also observed that in an emission mode 50% acetone was found to be the best medium for obtaining optimum flame stability and sensitivity. Although methanol (80%) and ethanol (80%) were found to be of comparable sensitivity, they were abandoned due to frequent clogging of the burner and fluctuating readings (Table 4). Among the three spectroscopic buffers (Na, K, La) tried, La gave the highest blank and was thus discarded. Na or K (4 mg/ml) can be used with equal preference. Optimum sensitivity of the method was found to be 5  $\mu\text{g}/\text{ml}$  in 50% acetone medium.

TABLE 4  
COMPARISON OF SENSITIVITY IN ABSORPTION AND EMISSION MODE IN DIFFERENT MEDIA

Yttrium Taken = 28  $\mu\text{g}/\text{ml}$   
Spectroscopic buffer = K, 4 mg/ml

Mode	Medium			
	Water	50% Acetone	80% Methanol	80% Ethanol
Absorption	nil	nil	0.002	Fluctuating readings
Emission	0.02	0.169	0.189	do

Two international standard samples (SY-2 and SY-3) were processed and analysed by this method. Results obtained are in good agreement with the reported value (Table 5). Recovery of yttrium was also checked

by standard addition method in some samples containing phosphate (monazite) and found to be > 95%. Following the above procedure many rock samples of localities Umra (Rajasthan) and Siri river (M.P.) were analysed. The method was also applied to leach liquors and beneficiation products of Siri river. The method is specially useful for silicate and phosphate types of rocks.

TABLE 5  
ANALYSIS OF INTERNATIONAL STANDARD SAMPLES

S.No.	Sample Nos.	Certified Value (Y ppm)	Found* (Y-ppM) (by this method)
1.	SY-2	130	130.7
2.	SY-3	740	753.6

\* Mean of two values

### Recommended Procedure

0.5 gm of the geological sample is treated with HF-H<sub>2</sub>SO<sub>4</sub> (10 ml each) mixture in a platinum dish, fumed on sand bath, cooled in ice, diluted with ice-cold water and filtered. The residue is fused with sodium peroxide in nickel crucible and mixed with the filtrate. Yttrium is precipitated with ammonium hydroxide, precipitates dissolved in 12N HNO<sub>3</sub> and extraction done two times with 10 ml 100% T.B.P. each for 5 min. Yttrium is stripped twice with 10 ml water each for 5 min. The stripped solution is evaporated on water bath and the residue leached with 2 ml 10% HCl and made up to 10 ml containing 5 ml acetone and 4 mg/ml K for final determination with A.A.S. in emission mode. Blank and a series of standards are also processed in the same way and calibration curve obtained at the instrumental conditions as mentioned above (Table 1).

### ACKNOWLEDGEMENTS

Authors are thankful to Shri B. N. Tikoo and Shri K. P. Cheria for constant encouragement for completing the work.

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[Received : 15 April 1988; Accepted : 12 October 1988]