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### USE OF ERIOCHROME CYANINE R FOR SEPARATION-FLOTATION AND MICRODETERMINATION OF HAFNIUM AND ZIRCONIUM IN REAL SAMPLES

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## USE OF ERIOCHROME CYANINE R FOR SEPARATION-FLOTATION AND MICRODETERMINATION OF HAFNIUM AND ZIRCONIUM IN REAL SAMPLES

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### ABSTRACT

Eriochrome cyanine R (ECR) is investigated as a collector for separation and flotation of hafnium from zirconium through the use of an oleic acid surfactant. Selective separation of Zr(IV) is achieved at pH 2 with maximum efficiency, whereas Hf(IV) is completely separated at pH 7.0–7.5. Excess ECR is used to avoid interferences from foreign ions without any effect on the separation efficiency. The procedure is successfully applied to the determination of Zr(IV) and Hf(IV) in real samples. A flotation mechanism is proposed.

*Key Words:* Zirconium (IV); Hafnium (IV); Eriochrome cyanine R (ECR); Flotation

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## INTRODUCTION

Zirconium and hafnium, which are gaining great attention, are used in the production of jewels, jet-engine parts, steel, ceramics, refractory materials, and optical glass (1). All naturally occurring zirconium (zircon) contains a fractional percentage of Hf(IV). Also, the chemical similarity of Hf and Zr is evident in their geochemistries. This resulted in deficiency in the chemistry studies of Zr(IV) and Hf(IV) (2). The rarity of Hf and the difficulty in separating it from zirconium encouraged many workers to separate and determine trace amounts of these elements in different samples by various techniques (3–7). Ion flotation (8) and froth flotation (9) have been used in the separation of Zr(IV) and Hf(IV) from aqueous solutions using different reagents. Nuclear reactors require hafnium-free zirconium for controlling and protecting the devices, so both pure zirconium and hafnium must be available. However, most of the methods for separating Hf(IV) and Zr(IV) from the same samples are not selective (3). Moreover, the majority of spectrophotometric methods reported in the literature for Zr(IV) and Hf(IV) determination give the total content of the 2 elements simultaneously (10–13). In 2000, the organophosphorous extractant Cyanex 925 was used for hafnium/zirconium separation (14) from hydrochloric acid solutions of the metals. In addition, Zr(IV) and Hf(IV) amounts have been determined by energy dispersive X-ray fluorescence with solid-phase preconcentration (15). However, these methods are tedious, time-consuming, and expensive. According to the aforementioned, development of simple procedures for separation and determination of these elements is of utmost importance. As a consequence, we aimed to find a simple, rapid, inexpensive, and accurate procedure for selective separation via flotation, through adjusted pH and spectrophotometric determination, from the natural forms of Hf and Zr.

## EXPERIMENTAL

### Stock Solutions

A stock solution (10 mmol/L) of eriochrome cyanine R (ECR) was prepared by dissolving the requisite amount in double-distilled water. The oleic acid (HOL) stock solution (63.6 mmol/L) was prepared by dispersing 20 mL HOL (food grade specific gravity 0.895, provided from J. T. Baker Chemical Co) in 1 L kerosene. A zirconyl chloride stock solution (1 mg/mL) was prepared by dissolving 3.531 g of zirconyl chloride octahydrate in 2 mol/L HCl and diluted with doubly distilled water to 1 L with the acid. A hafnium stock solution (1 mg/mL) was prepared by fusion of 1.179 g HfO<sub>2</sub> with Na<sub>2</sub>CO<sub>3</sub>, and the melt was dissolved in double-distilled water to 1 L. All other chemicals used were of reagent-grade quality. An ar-



sinazo III stock solution was prepared by dissolving 0.5 g of arsinazo reagent in 1 L with double-distilled water. The real metal samples were obtained from the National Institute of Metallurgy, South Africa.

### Apparatus

A cylindrical, graduated, glass tube of 16-mm i.d. and 290-mm length, with a stopper at the top and a stopcock at the bottom, was used as a flotation cell. The spectral data were recorded on Unicam UV 2100 UV/vis and MATTSON 5000 Fourier Transform Infrared Spectrometers. The pH values of all solutions were measured with a Hanna Instruments 8519 digital pH meter.

### Procedure

Twenty milliliters of double-distilled water, acidified with 0.01 mol/L HCl (to pH = 2), containing  $5 \times 10^{-5}$  mol/L of Zr(IV) and Hf(IV) and  $5 \times 10^{-4}$  mol/L ECR were introduced into the flotation cell. To this solution, 3 mL of  $2 \times 10^{-4}$  mol/L HOL were added. The flotation cell was turned upside-down 20 times by hand. After 5 minutes, to complete the flotation, the liquor containing Hf(IV) was completely rinsed into another flotation cell. The scum layer in the first flotation cell containing Zr(IV) was dissolved in 3 mL HNO<sub>3</sub>. The solution was quantitatively transferred into a 10-mL measuring flask that contained 1 mL of 0.05% aqueous solution of Arsinazo III. After the addition of 10 mol/L HCl, the solution was measured spectrophotometrically at 665 nm (16). Two drops of NaOH (approximately 0.1 mol/L) was added to the liquor in the second flotation cell to raise the pH to 7, and the previous steps for flotation, metal elution, and spectrophotometric determination were carried to completion. The separation rate ( $R$ ) of each metal was calculated from its concentration in the scum using the relation

$$R = c_s/c_i,$$

where  $c_i$  and  $c_s$  denote the initial and scum concentration of each metal.

### RESULTS AND DISCUSSION

The oleic acid surfactant, as previously reported (17–19), was an excellent surfactant for precipitate (20,21) and ion (22,23) flotation. Therefore, HOL was the first choice for the flotation processes undertaken in this study.

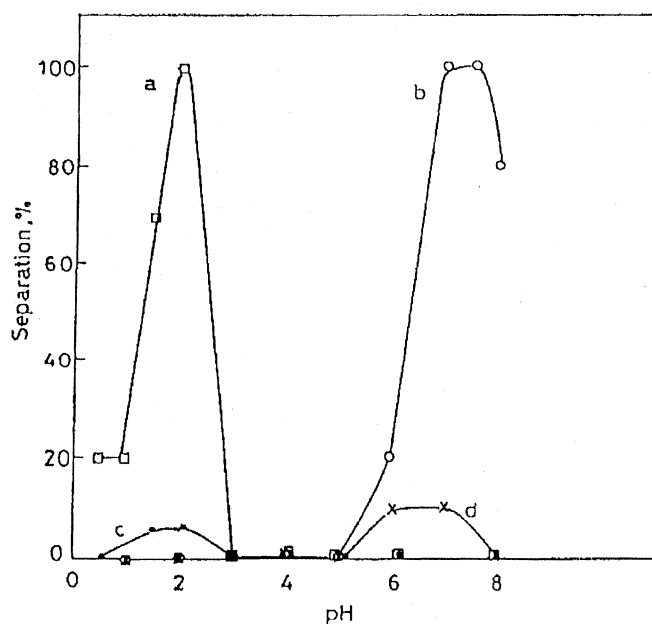


### Selective Separation of Zr(IV) and Hf(IV) by Flotation

Different and diverse factors affecting the separation process have been studied and reported here.

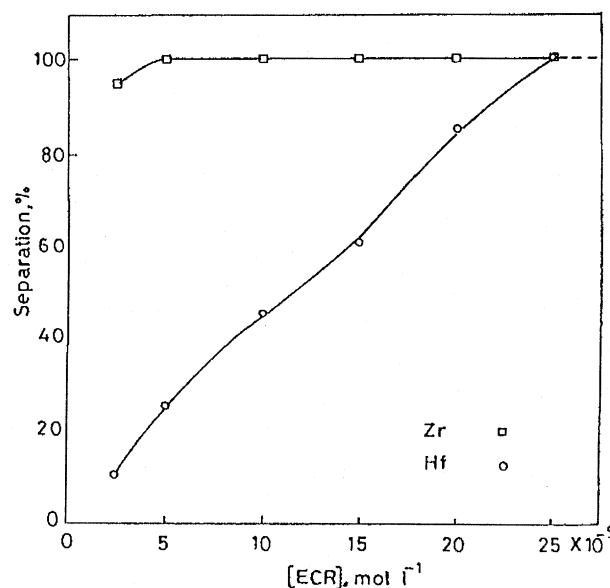
#### Effect of pH

A series of experiments was conducted to float the appropriate concentration of each metal using suitable concentrations of HOL. The unsatisfactory results obtained resulted in the use of an additional collector. ECR was found to be suitable collector to enhance separation. Figure 1 shows that the maximum separation rates of Zr(IV) and Hf(IV) in the absence of ECR were 0.05 and 0.10 (curves c and d, respectively). In the presence of ECR (curves a and b), the separation rates reached approximately 1.0 for both Zr(IV) and Hf(IV). These results



**Figure 1.** The role of hydrogen ion concentration on the selective separation-flotation of  $5 \times 10^{-5}$  mol/L of Zr(IV) and Hf(IV) in an admixture with  $2 \times 10^{-4}$  mol/L HOL in the presence and absence of  $5 \times 10^{-4}$  mol/L ECR. (a) Zr(IV) + ECR + HOL, (b) Hf(IV) + ECR + HOL, (c) Zr(IV) + HOL, (d) Hf(IV) + HOL.





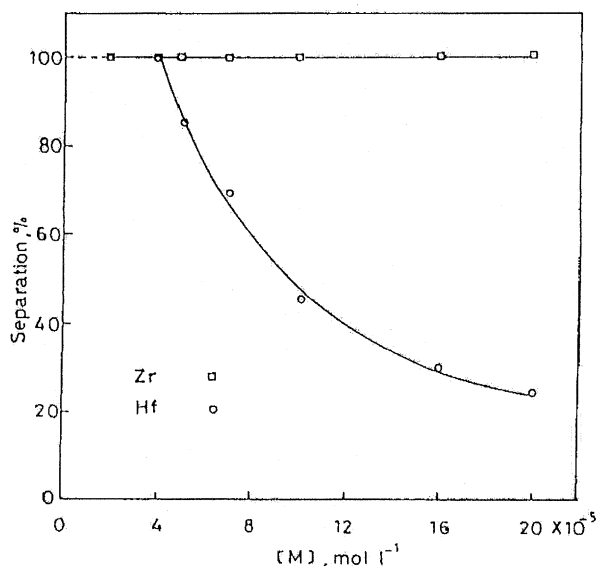
**Figure 2.** Influence of ECR concentration on the floatability of  $5 \times 10^{-5}$  mol/L Zr(IV) at pH 2 and Hf(IV) at pH 7 with  $2 \times 10^{-4}$  mol/L HOL.

show that complete and selective separation takes place at pH 2 for Zr(IV) and pH 7.0–7.5 for Hf(IV). Accordingly, all subsequent experiments were carried out at the pH that was experimentally determined as optimal for each element.

#### Effect of Eriochrome Cyanine R

ECR forms complexes with Zr(IV) and Hf(IV) (24). Experimental data showed that the zirconium complex is first formed at pH 2. Therefore the first added quantity of ECR was used for zirconium complexation and complete separation at a 1:1 (Zr/ECR) molar ratio. Increasing ECR above this ratio has no apparent effect on the complexation or on the separation percentage. However, excess ECR reacts with hafnium at pH 7 with a molar ratio of 1:5 (Hf/ECR), and the addition of more ECR does not affect the complexation and separation of Hf(IV). All these results are graphically illustrated in Fig. 2. From these data, one can see that in the presence of excess ECR in the Zr(IV) and Hf(IV) mixture, Zr(IV) is quantitatively separated at approximately pH 2, and at pH 7, Hf(IV) is completely separated. Therefore, excess ECR (approximately  $5 \times 10^{-4}$  mol/L) was selected for further experiments.





**Figure 3.** Floatability of different concentrations of Zr(IV) at pH 2 and Hf(IV) at pH 7 in presence of  $2 \times 10^{-4}$  mol/L ECR and  $2 \times 10^{-4}$  mol/L HOL.

#### Effect of Metal Ion Concentration

To confirm the previously obtained data,  $2 \times 10^{-4}$  mol/L ECR was used at suitable concentrations (Fig. 2) for a series of experiments designed to float different concentrations of the Zr(IV) and Hf(IV) ( $0$ – $20 \times 10^{-5}$  mol/L). At  $2 \times 10^{-4}$  mol/L ECR, Zr(IV) was completely separated out of the sample, but a 5-fold amount of ECR was required to separate Hf(IV) from the sample at pH 7. These data are graphically illustrated in Fig. 3.

#### Effect of Oleic Acid Surfactant Concentration

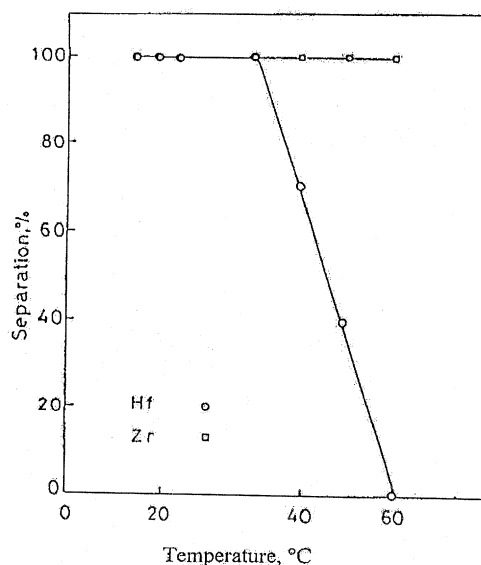
To determine the appropriate concentration of the surfactant required for complete separation of Zr(IV) and Hf(IV), several experiments were conducted. Results showed that  $5 \times 10^{-5}$  mol/L of Zr(IV) and Hf(IV) in the presence of  $5 \times 10^{-5}$  mol/L of ECR was completely separated selectively at pH 2.0 and 7.0 at a wide range of HOL concentrations ( $10$ – $40 \times 10^{-3}$  mol/L). Outside of this HOL concentration range, the separation percentages were lower than they were within the range. At lower-than-range HOL concentrations, the degradation in the separation efficiency may be due to an insufficient quantity of surfactant, whereas at



higher concentrations the decrease may have been due to redispersion of the ECR-metal-HOL complex in the original liquor. The range of HOL concentrations that is most effective for Hf and Zr ion separation is less than the critical micelle concentration (25). Therefore, concentration of HOL was kept constant at  $2 \times 10^{-4}$  mol/L throughout the present study.

### Effect of Temperature

Solutions of Zr(IV) or Hf(IV) were either cooled or heated to the same temperature as that of the ECR and HOL solutions. The ECR and HOL solutions were quickly and simultaneously poured into each metal ion solution. The solutions were introduced into 2 flotation cells that were jacketed with 1-cm thick fiberglass insulation. The flotation steps were conducted, as previously described, at pH 2 and 7 for Zr(IV) and Hf(IV), respectively. The data in Fig. 4 clearly indicate that the floatability of the Zr-ECR complex was not affected by raising the temperature up to 60°C. These data agree well with those found in the literature (26); Zr(IV) forms a colored, stable complex at low pH in hot aqueous solutions. However, the separation of the Hf-ECR complex was not affected up to 35°C. Increasing temperature over 35°C led to a decrease in the floatability such that it was



**Figure 4.** Effect of temperature on floatability of  $5 \times 10^{-5}$  mol/L Zr(IV) at pH 2 and Hf(IV) at pH 7 in presence of  $5 \times 10^{-4}$  mol/L ECR and  $2 \times 10^{-4}$  mol/L HOL.





zero at 60°C. This result of temperature on floatability reflects a weak interaction between the complex and surfactant. Also, these data demonstrate that complete elution of Hf(IV) can be achieved at 60°C. All measurements for both Zr(IV) and Hf(IV) were carried out at an approximate environment of 25°C.

#### Effect of Volume

A series of experiments was conducted to float different quantities of Zr(IV) or Hf(IV) introduced at fixed aqueous volumes (10 mL) under the recommended conditions for each element. The smallest amounts of Zr(IV) and Hf(IV) that can be safely and quantitatively separated from 10 mL are 0.09 and 0.9 ppm, respectively.

However, another series of experiments was conducted to float a fixed quantity of each metal (400 µg) from different aqueous volumes using suitably large flotation cells under the recommended conditions. The results showed that 400 µg of each element can be quantitatively separated from different volumes up to 700 mL. According to the experiments, the detection limit of such separation procedure is 0.57 ppm.

#### Interferences

The application of this flotation procedure in selectively separating traces of Zr(IV) and Hf(IV) depends on the effect of foreign ions. Possible interferences of foreign ions on the effectiveness of separation were determined at  $5 \times 10^{-5}$  mol/L of each metal ion and ECR by the recommended procedure in the presence of various ions. The pH of the sample solutions was first adjusted to 2 to separate zirconium then raised to 7 to separate hafnium. The data are presented in Table 1. The investigated interfering ions had no effect on the separation and determination of zirconium even at  $5 \times 10^{-4}$  mol/L ECR. With respect to hafnium, Ca, Mg, Sr, Ba, Pb, Zn, UO<sub>2</sub>(II), Hg, Cd, Si, and Li had no effect at  $5 \times 10^{-4}$  mol/L ECR, whereas the rest of the investigated ions showed marked effects on Hf recovery. However, these effects can be completely eliminated by increasing the amount of ECR ( $2 \times 10^{-3}$  mol/L) to promote complexation of the foreign ions. The recovery problem is still present from Pt, Au, Pd, and V on hafnium even with the use of excess of ECR. However, such elements are not found in real samples of zirconium and hafnium.

Sodium, potassium, calcium, and magnesium as chloride, nitrate, sulfate, perchlorate, and iodide salts were added during the flotation separation of zirconium and hafnium under the recommended conditions to study the effect of ionic strength. Most of these salts, even up to the 0.1 mol/L concentration level, had no effect on the flotation efficiency of either hafnium or zirconium.



**Table 1.** Effect of Select Foreign Ions on the Separation of Zr(IV) at pH 2 and Hf(IV) at pH 7 in Presence of ECR and HOL

Foreign Ions	Recovery (%)				
	a	b	c		
	ppm	Zr	Hf	Zr	Hf
Co, Cu, Ni, Mn, Cr, Sc	100	100	70	100	95
Al, Fe	500	100	50	100	100
Mo, W, As	100	100	90	100	100
Pt, Pd, Au, V	100	100	60	100	90
Y, La, Th	200	100	55	100	100
Ca, Sr, Mg, Ba, Pb, Zn, UO <sub>2</sub> , Hg, Cd, Si, Li	1000	100	100	100	100

Samples:  $5 \times 10^{-5}$  mol/L Hf(IV) and Zr(IV)

ECR:  $2 \times 10^{-3}$  mol/L

HOL:  $2 \times 10^{-4}$  mol/L

a tolerance limit (maximum amount added)

b with  $5 \times 10^{-4}$  mol/L ECR

c using  $2 \times 10^{-3}$  mol/L ECR

## APPLICATION

### Determination of Zr(IV) and Hf(IV) in Water and Simulated and Real Samples

To investigate the applicability of this procedure to natural water samples, the recoveries of  $5.0 \times 10^{-1}$  and  $1 \times 10^{-4}$  mol/L of Zr(IV) at pH 2 and Hf(IV) at pH 7 that were added to distilled, domestic tank, and Nile water samples were examined by the above procedure. The results obtained demonstrate that the technique is applicable to separate Hf(IV) and Zr(IV) in different natural water samples and had a satisfactory recovery of metal ions ( $100 \pm 0.1$ – $0.5\%$ ).

Table 2 summarizes the effects of numerous ions, which are naturally allied with Zr(IV) and Hf(IV) in real samples, on the efficiency of separation and determination of the metals in synthetic mixtures.

The aforementioned discussion introduced a good procedure for successful selective separation and determination of Zr(IV) and Hf(IV) in real samples. Exactly 0.25 g of the solid real sample was completely dissolved in a Teflon beaker with a mixture of acids (45 mL HF, 15 mL H<sub>2</sub>SO<sub>4</sub>, and 5 mL HNO<sub>3</sub>). After complete dissolution, the solution was evaporated to dryness. The residue was dissolved in 20 mL HCl 1:1 HCl/H<sub>2</sub>O and completed to 25 mL with double-distilled water. Into a flotation cell, 0.5 mL of the sample and 3 mL of ECR ( $10^{-2}$  mol/L) were introduced, and the solution volume was raised to 10 mL with double-dis-



**Table 2.** Recovery of 5 ppm Zr(IV) at pH 2 and Hf(IV) at pH 7 from Different Synthetic Mixtures in Presence of  $2 \times 10^{-3}$  mol/L ECR and  $2 \times 10^{-4}$  mol/L HOL

Synthetic Mixture 50 ppm of each element	Found		Abs. Error		R. Error		SD		RSD	
	Zr	Hf	Zr	Hf	Zr	Hf	Zr	Hf	Zr	Hf
Th + Y + Co + Ca	5.024	5.03	0.024	0.03	+0.48	+0.6	0.02	0.014	0.40	0.28
Y + La + Cu + Cd	5.027	5.042	0.027	0.042	+0.54	+0.84	0.022	0.012	0.44	0.24
Th + La + Cr + Si	4.99	5.035	0.01	0.035	-0.2	+0.7	0.01	0.021	0.20	0.42
Mg + Ba + Co + Zn	5.03	5.03	0.03	0.03	+0.6	+0.6	0.028	0.014	0.56	0.28
Al + Fe + Zn + Cd	5.033	5.037	0.033	0.037	+0.66	+0.74	0.033	0.024	0.66	0.48

Abs. error, absolute error; RSD, relative standard deviation; *R* error, relative error.

tilled water. The cell was shaken vigorously for complete complexation, which is reached at pH 2. After 5 minutes, 3 mL HOL ( $2 \times 10^{-4}$  mol/L) were added to the cell. The same steps for flotation, metal elution, and spectrophotometric determination as those previously described were conducted in the presence of aqueous solution containing 10% tartaric acid for spectrophotometric determination of hafnium. The results are listed in Table 3.

### SEPARATION MECHANISM

In the studies concerning the separation via flotation, the role of surfactant is very important. The nature of the interaction between the HOL and the complex formed (Zr-ECR or Hf-ECR) must be studied to understand the mechanism of flotation. The proposed mechanism may proceed through: 1) formation of a physical force, such as through van der Waals force, 2) formation of a hydrogen bond between the hydrophilic part of HOL and the active sites in the ligand complex, or 3) by interactions between oleic acid and the complex, formed in solution through

**Table 3.** Comparative Results for Analysis of Zr(IV) at pH 2 and Hf(IV) at pH 7 in Some Ore Samples in Presence of  $3 \times 10^{-3}$  mol/L ECR with  $2 \times 10^{-4}$  mol/L HOL

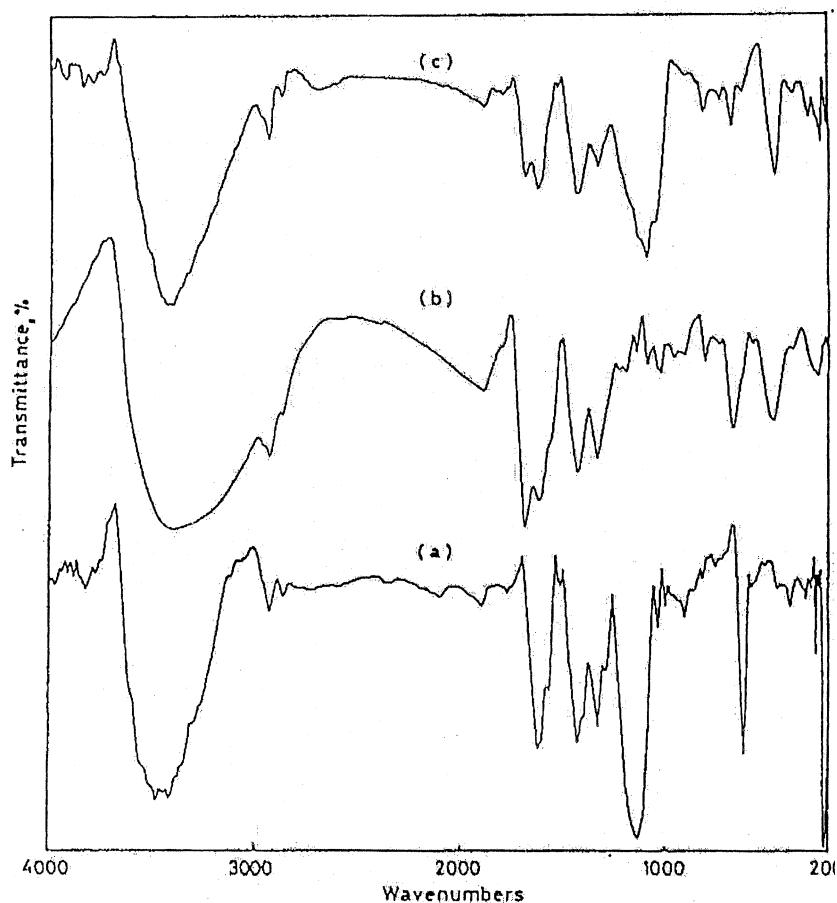
Ore Name	Zr, ppm			Hf, ppm		
	Certified	Present Method	R%	Certified	Present Method	R%
202 83 MALNiM-G Granite	300	299.14	99.7	12	11.8	98.3
203 83 MAL, NiM-L Lujavrite	$11 \times 10^3$ (1.100%)	$11.043 \times 10^3$ (1.104%)	100.4	190	191.15	100.6

R%, mean recovery rate.



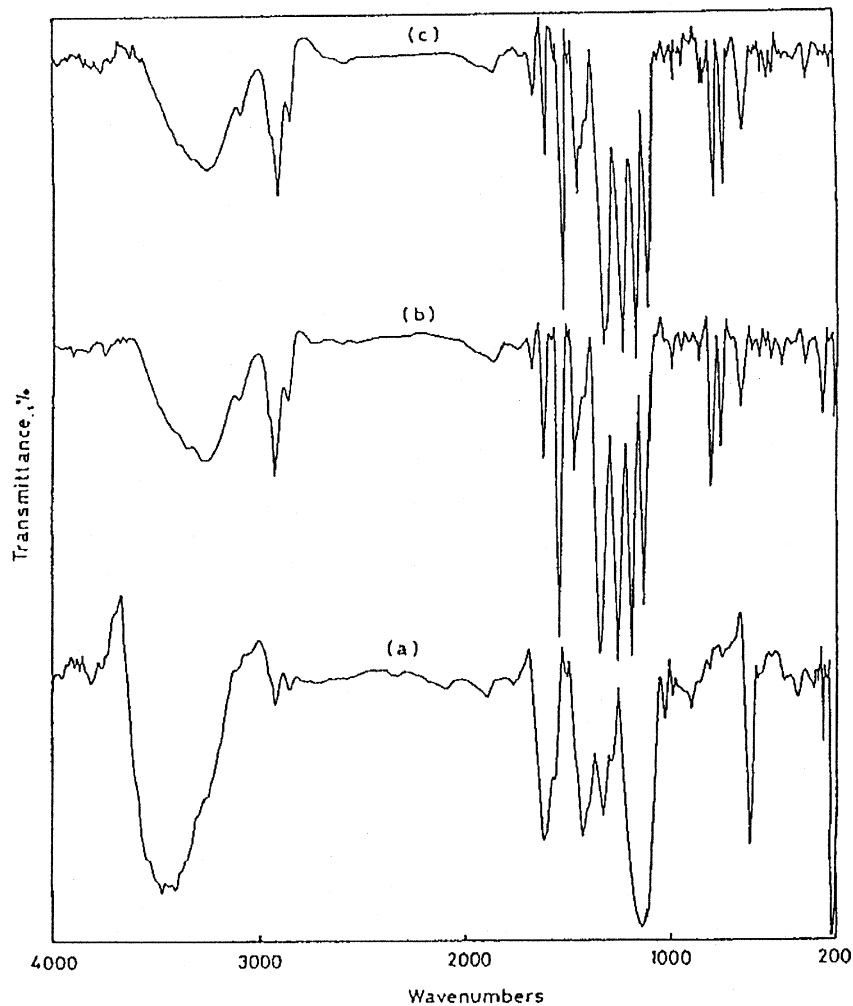
a coordinate bond that forms a self-floatable species (metal- ECR- HOL). In this investigation, Van der Waal's physical force was observed in zirconium and hafnium separation. To determine the mechanism of separation, some observations and experiments were considered:

1. Complexes in which zirconium and hafnium were isolated in the absence and presence of HOL were washed with water, ethanol, and ether several times before elemental analyses showed that the complexes are free from HOL.
2. The floated complex of hafnium and zirconium has the same color as that obtained in the aqueous solution.

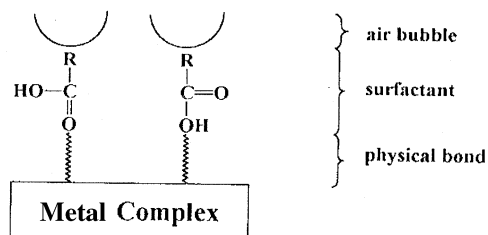


**Figure 5.** Infrared spectra of (a) ECR in KBr, (b) Zr-ECR isolated in aqueous solution in KBr, and (c) Zr-ARS-HOL isolated in oleic acid in KBr.





**Figure 6.** Infrared spectra of (a) ECR in KBr, (b) Hf-ECR isolated in aqueous solution in KBr, and (c) Hf-ECR-HOL isolated in oleic acid in KBr.



**Structure I.** Schematic diagram of the surfactant and metal complex.



3. Figures 5 and 6 do not show obvious differences between the infrared spectrum of Zr(IV) or Hf(IV) complexes isolated in aqueous or in HOL solutions.

All the mentioned studies confirm that separation of hafnium and zirconium is most probably due to physical interactions between the HOL surfactant and the complexes of Hf; Zr (structure I). This work provides a simple and economic procedure for quantitative separation and determination of Zr(IV) and Hf(IV) and also opens the way for studying the chemistry of the isolated Hf(IV) in some detail.

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