



# Spectrophotometric determination of iridium after complexation and membrane filtration



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## ABSTRACT

A simple, ultra sensitive and selective method for the spectrophotometric determination of Ir<sup>3+</sup> in various synthetic and environmental samples was reported. The analyte ions were collected on a membrane filter in the form of their red complex with 5-(2-benzothiazolyl-azo)8-hydroxy-quinoline (BTAHQ), and the absorption spectra of the colored membrane filters were acquired. Effects of pH value, sample volume, and amount of BTAHQ were examined in order to optimize sensitivity. The interference by common other ions was eliminated using appropriate masking agents. The absorbance is linearly related to the concentration of Ir<sup>3+</sup> in the ranges from 0.1 to 0.8 µg/L, and from 1.0 to 7.0 µg/L, respectively, the correlation coefficients (R<sup>2</sup>) being 0.9996 and 0.9994. The molar absorptivities were calculated to be  $4.81 \times 10^5$  and  $4.26 \times 10^7$  L mol<sup>-1</sup> cm<sup>-1</sup> at 563 nm, whereas Sandell sensitivity was found as 0.391 and 0.004 ng cm<sup>-2</sup>, respectively. The novelty and advantages of the proposed method is the highly sensitivity of the proposed method compared with all previously cited methods for iridium determination. Under the optimal conditions, the detection limit is 0.03 µg/L. The recoveries in case of spiked samples are between 98.7% and 101.5%, and the relative standard deviations range from 1.21% to 1.75%.

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## 1. Introduction

Spectrophotometry is the most common technique used for metal determination using various chromogenic reagents [1–10], owing to its simplicity and low cost. However, the spectrophotometric determination of metals in ultra trace level in solution is difficult due to various factors, particularly their low concentrations and matrix effects.

Iridium occurs as osmiridium alloy and one of the minor constituents in most platinum metal deposits in North and South America and the Urals. It is commonly used in various jewellery, dental alloys, electrical equipments, corrosion-resistant chemical wares, crucibles for high temperature reactions and extrusion dies for high melting-point glasses. Therefore, highly selective, sensitive, rapid and economical methods are needed for its trace and ultra trace determination. Neutron activation analysis (NAA) [11], atomic absorption spectrometer (AAS) [12], graphite furnace AAS [13], individual catalytic [14], imprinted polymer-based [15], sequential voltammetric [16] and flow injection analysis [17] may be used for the trace determination of iridium in complex materials, however, these instruments are highly expensive, day

to day maintenance is high and not free from various types of interferences [18–20]. A survey of the literature reveals that iridium may be determined by zero order spectrophotometry using phenanthrenequinone monoxime [21] ( $\epsilon = 2.3 \times 10^4$  L mol<sup>-1</sup> cm<sup>-1</sup>) perazine dimalonate [22] ( $\epsilon = 9.93 \times 10^3$  L mol<sup>-1</sup> cm<sup>-1</sup>), tetrahydrofurfuryl xanthate [23] ( $\epsilon = 5.02 \times 10^3$  L mol<sup>-1</sup> cm<sup>-1</sup>), 1-phenyl-4,4-6-trimethyl-(1H,4H)-pyrimidine-2-thiol [24].

Several publications describe the determination of metals ions with improved sensitivities using azo dyes and polymeric supports [25–27], but these methods involves complicated procedures, use of more reagents and time-consuming elution steps. There are few papers [28,29] reported the direct trace determination of heavy metals on membranes followed by metal complexation without using complicated procedures and elution steps. Takahashi et al. [28] used the complexation and membrane filtration method for Zn<sup>2+</sup>. The concentration of Zn<sup>2+</sup> used was up to 65 µg/L with maximum sample volume of 100 mL. The method resulted in improvement of sensitivity. But the sensitivity is still insufficient and the method lacks adequate selectivity and application to real samples. Dolgin et al. [29] tested the application of complexation reactions followed by pre-concentration of the products on various filters and also performed the complexation kinetic investigation. The detection limit of Ni<sup>2+</sup> obtained was 1.2 µg/L using 1000 mL sample volume. However, they have not sufficiently

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studied the effects of potentially interfering ions and soluble ligands present in the sample solution. Therefore, the reported method also suffers from poor selectivity and there is further possibility to enhance the sensitivity and detection limits.

In this work, we reported on the application of membrane filtration procedure for the enrichment of  $\text{Ir}^{3+}$  as its 5-(2-benzothiazolyl-azo)8-hydroxy-quinoline BTAHQ complex in alloys, synthetic and environmental samples. Large volume sample solution (1000 mL) was used to improve the sensitivity. Quantification was achieved based on the measurement of absorbance at a defined wavelength. Some parameters such as pH, the amount of BTAHQ, the sample volume, and effect of potentially interfering ions were investigated in detail. Using appropriate masking agents, most of the routine interfering ions did not interfere with the determination so the method has good selectivity. The presented method has been satisfactorily applied to the determination of ultra trace amounts of  $\text{Ir}^{3+}$  in various synthetic standard alloys and environmental samples.

## 2. Experimental

### 2.1. Apparatus

A Perkin-Elmer Lambda 12 UV-vis spectrophotometer equipped with the integrating sphere accessory was used for absorbance measurements. The laboratory glassware was kept overnight in a 6.0% nitric acid solution and rinsed with ultra-pure water three times. Ultra-pure water was obtained from an ultra-pure water purification system (SARTORIUS arium 611DI, Germany, 18.2 M). A Perkin Elmer atomic absorption spectrometer model AAnalyst 300 was used for all GF-AAS measurements. An Orion research model 601 A/digital ionalyzer pH meter was used for checking the pH of solutions.

### 2.2. Reagents

All chemicals used were of analytical reagent grades. The solution of iridium chloride, 0.1550 g was dissolved in 100 mL of  $6.0 \text{ mol L}^{-1}$  HCl and then diluted it to 1000 mL in a standard flask. This solution was standardized by known methods [30,31] and made  $1000 \mu\text{g/mL}$  by appropriate dilution. A more dilute solution of iridium can be prepared by diluting this solution with ultra-pure water.

BTAHQ was synthesized according to the method described previously [32]. Stock solution of  $2 \times 10^{-3} \text{ mol L}^{-1}$  was prepared by dissolving an appropriate weight of the pure reagent in least amount of ethanol (10 mL) and then diluted to the mark in a 100 mL measuring flask with ethanol. The solutions of pH 2.5–12 thiel buffer were prepared as described earlier [33]. Solutions of alkali metal salts and various metal salts were used to study the interference of anions and cations, respectively. NaF, sodium sulphide, thiourea and ethanolamine were purchased from Aladdin (Shanghai, China, [www.aladdinreagent.com](http://www.aladdinreagent.com)). Cellulose acetate membrane filters used in the present study were purchased from Dalian Elite Analytical Instruments Co., Ltd. (Dalian, China, [www.elitehplc.com](http://www.elitehplc.com)) with the pore size of  $0.22 \mu\text{m}$  and 50 mm diameter.

### 2.3. Preparation of membrane for spectral measurements

In the experiment, the concentration ranges of  $\text{Ir}^{3+}$  were  $0.1\text{--}0.8 \mu\text{g/L}$  and  $1.0\text{--}7.0 \mu\text{g/L}$ . The pH of 1000 mL of each sample solution was adjusted to 6.5 with 25 mL thiel buffer. Then 1.0 mL of  $2 \times 10^{-3} \text{ mol L}^{-1}$  of BTAHQ was added into each sample solution under vigorous stirring. This started the formation of red-color Ir-BTAHQ complex. After 5.0 min standing for completion of

complexation reaction, the Ir-BTAHQ complex was collected by filtering the complex with a cellulose acetate membrane filter. The collection was performed very quickly by filtration under suction with an aspirator and almost all the complex was retained on the membrane filter surface uniformly. Similarly the reagent blank was also obtained. The membrane filters were air dried and the absorbance spectra were measured. Fig. 1 is showing the big visual color difference between  $0.0 \mu\text{g/L}$  (reagent blank) and  $10 \mu\text{g/L}$  of  $\text{Ir}^{3+}$ .

### 2.4. Iridium determination in alloys

0.10–0.05 g of alloy is dissolved in 10 mL of HCl +  $\text{HNO}_3$  (1:1) mixture. The obtained solution is evaporated to wet salts and again dissolved in 50 mL of HCl ( $3.0 \text{ mol L}^{-1}$ ). If the alloy dissolves incompletely sintering of the new sample with NaOH +  $\text{NaNO}_3$  (1:3) oxidative mixture is carried out. The obtained alloy is dissolved in HCl of  $3.0 \text{ mol L}^{-1}$  concentration. Then it is placed in 250 mL volumetric flask and distilled water is added to the mark. If necessary, the solution with iridium lower concentration is prepared. The aliquot of concentrated solution is placed in 100 mL flask and HCl solution (1.0 M) is added to the mark. The samples were filtered through a  $0.45 \mu\text{m}$  membrane filter. The pH of the samples was adjusted to the optimum value and the proposed procedure was applied for the determination of  $\text{Ir}^{3+}$ .

## 3. Results and discussion

### 3.1. Absorption spectra

Upon the addition of ethanolic solution of BTAHQ to an aqueous solution containing  $\text{Ir}^{3+}$  and a buffer solution at pH 6.5, a marked change in color from orange to red is observed. The change in the color intensity is proportional to the increasing concentration of  $\text{Ir}^{3+}$ . Thus, the 'naked-eye' detection of  $\text{Ir}^{3+}$  was easily observable.

The absorption spectra of orange color BTAHQ and Ir-BTAHQ red-colored complex on membrane filters were recorded in the range of 360–600 nm as shown in the Fig. 2. One can observe two traditionally spectral bands at 489 and 563 nm which is corresponding to BTAHQ and Ir-BTAHQ complex. The band at 563 nm related to the complex clearly increase with  $\text{Ir}^{3+}$  concentration enabling quantitative determination. One can observed that the color intensity is proportional to the concentration of  $\text{Ir}^{3+}$  in two ranges, thus, the 'naked-eye' detection of iridium is possible. The color development and the absorbance increase firstly regularly up to certain limit, then the increase in absorbance and color intensity is steady decrease with increasing  $\text{Ir}^{3+}$  concentration. So, the calibration plots based on the absorbance at 563 nm were linear in both concentration ranges.

### 3.2. Optimization of variables

A pH dependence was studied by applying the 1000 mL procedure. The optimum buffer solution was investigated by examining different types of buffer (acetate, borate, phosphate, thiel, and universal) solutions. Thiel buffer gave the best results. The effect of pH on the Ir-BTAHQ complex was tested in the pH range 2.5–12 using  $4.0 \mu\text{g/L}$  of  $\text{Ir}^{3+}$  solutions. The test results showed that the most intense red color and maximum band height could be obtained when pH of  $\text{Ir}^{3+}$  solutions was 6.5. Thus, pH 6.5 was selected in this study using thiel buffer.

The influence of BTAHQ concentration on the determination of  $4.0 \mu\text{g/L}$  of  $\text{Ir}^{3+}$  was investigated in the concentration range of  $5 \times 10^{-7}$  to  $5 \times 10^{-6} \text{ mol L}^{-1}$ . The results showed that the absorbance height rose when concentration of BTAHQ changed from

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