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**RESEARCH ARTICLE**

## Extractive Spectrophotometric Determination of Ruthenium (III) Using Novel Salen Ligand

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**ABSTRACT:**

2-[(E)-N-(2-{{2-[(E)-[(2-hydroxyphenyl) methylidene] amino] phenyl} (methyl) amino} phenyl) carboximidoyl] phenol (HHMCP) was synthesized and employed to develop an extractive spectrophotometric method for the determination of Ru (III). The reagent forms a complex with Ru (III) and can be quantitatively extracted in Chloroform at pH = 4.0. The extracted species showed an absorption maximum at 505 nm. A systematic study of the extraction was carried out by varying the parameters like pH, reagent concentration and equilibration time.

**KEYWORDS:** Ruthenium (III), Extractive Spectrophotometry, solvent extraction.

**INTRODUCTION:**

Ruthenium is a rare transition metal belonging to the platinum group of the periodic table. Like the other metals of the platinum group, ruthenium is inert to most chemicals. The Russian scientist Karl Ernst Claus discovered the element in 1844 and named it after Ruthenia, the Latin word for Rus'. Ruthenium usually occurs as a minor component of platinum ores and its annual production is only about 12 tonnes worldwide. Most ruthenium is used for wear-resistant electrical contacts and the production of thick-film resistors. A minor application of ruthenium is its use in some platinum alloys. Ruthenium is a scarce element that is found in about 10<sup>-8</sup> % of the earth crust.

Ruthenium is widely used in electronic industry, as one of the most effective hardeners in high density alloys. Ruthenium alloyed with other platinum group metals is used to make electrical contacts for heavy wear resistance materials. Recently, platinum metals especially ruthenium and its chloro complexes have been much used in the catalytic oxidation of some organic compounds. Ruthenium shows resistance to common acids including aqua-regia. It may be electrodeposited from molten salt for electroplating of brass metals. There has been considerable current interest in the chemistry of ruthenium primarily because of the fascinating electron transfer, photochemical and catalytic properties obtained by the complexes of this metal.

Beamish<sup>1</sup> has reviewed the reagents used for the spectrophotometric determination of ruthenium. One of widely used photometric method for Ru (III) is formation of blue complexes with thiourea<sup>2</sup> in an alcoholic acid medium. Several other methods for the spectrophotometric determination of Ru (III) mainly include the use of ligands such as a dithio-oximide<sup>3</sup>, p-nitrosodium methylaniline<sup>4</sup> and 1, 10- phenanthroline<sup>5</sup>.

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Other reagents used for spectrophotometric determination of Ru (III) are 1-naphthyl amino-3,5,7-trisulphonic acid<sup>6</sup> and 1-nitroso-2-naphthol<sup>7</sup> and 2-nitroso-1-naphthol<sup>8</sup>. These reagents are used for spectrophotometric determination of Ru (III) in hydrochloric or sulphuric acid medium. Pyridine-2-aldoxime<sup>9</sup> is also used for the determination of Ru (III) but it suffers interference from many metal ions and low tolerance for platinum metals.

Solvent extraction processes are well established and efficient techniques for the separation of ruthenium (III) from platinum and palladium<sup>10</sup>, Osmium and indium<sup>11</sup>. Extraction of ruthenium and its separation from Rhodium and Palladium with 4-pyridone derivatives was reported<sup>12</sup>. Spectrophotometric determination of Ru (III) using sodium iso-amylxanthate in presence of surfactants was reported by Malik<sup>13</sup>. Catalytic activity of polymer bound Ru (III)- EDTA complex was reported<sup>14</sup>. Reactions of Ru (II) complexes containing bidentate Schiff bases and triphenylphosphine or triphenylarsine have been reported by Viswanathamurthi<sup>15</sup> et al.

## EXPERIMENTAL:

All absorbance measurements were made on Systronics Digital Double Beam spectrophotometer model-2101 with 1 cm quartz cell. Standard volumetric flasks, 125ml separatory funnels, beakers were used for volumetric measurements. All dilutions were made using double distilled water. Solvents like chloroform, ethanol were used after double distillation. All interfering ion solutions were prepared in double distilled water

A stock solution of Ru (III) was prepared by dissolving 1 g Ruthenium chloride in 1M hydrochloric acid and diluted the solution up to the mark in 250 ml with double distilled water and standardized. A working solution of 300µg/ml was prepared by dilution of the stock solution with double distilled water in a standard volumetric flask.

2-[(E)-N-(2-{[2-(E)-(2-hydroxyphenyl) methylidene] amino} phenyl) (methyl amino) phenyl) carboximidoyl] phenol (HHMCP),<sup>16</sup> ( $10^{-2}$  M) was always prepared by dissolving 0.478 g of HHMCP in 100 ml chloroform and used. The reagent was successfully used in spectrophotometric determination of Ni (II)<sup>17</sup> and Pd (II).<sup>18</sup>

## EXTRACTION PROCEDURE:

To an aliquot of solution containing 300 µg of Ru (III) in a separatory funnel, 10 ml of buffer solution of pH 4 and 8 ml  $10^{-2}$  M HHMCP in chloroform were added. After shaking for 3 minutes, separatory funnel was kept for equilibrium and allowed to separate into two layers. The organic layer containing Ru-HHMCP complex was

collected in a 50 ml beaker containing a pinch of anhydrous sodium sulphate to remove traces of water. The absorbance of the extracted yellow complex was recorded at 505 nm against chloroform blank. A calibration graph was prepared and unknown amount of Ru (III) was determined from the calibration curve. Raffinates were analyzed for determination of Ru (III).

## RESULTS AND DISCUSSION:

### a) Absorption Spectrum:

After extraction, Ru (III): HHMCP complex present in organic phase was scanned from 200 nm to 1000 nm against reagent blank. The absorbance spectra of the extracted complex in chloroform were compared with chloroform. It was found that the Ru (III) complex has  $\lambda_{max}$  at 505 nm.

### b) Effect of pH:

The complex formation between ruthenium(III) and HHMCP depends upon pH of the solution. The optimum pH was determined by varying the pH from 1-12. The absorbance of the organic phase was measured as a function of pH of the aqueous phase. The complexation of Ru (II) was carried out at pH range from 1-12. The data obtained shows (Table 5.1) maximum absorbance at pH 4. In more acidic or basic solutions, it was found that absorbance decreases (Figure 1)

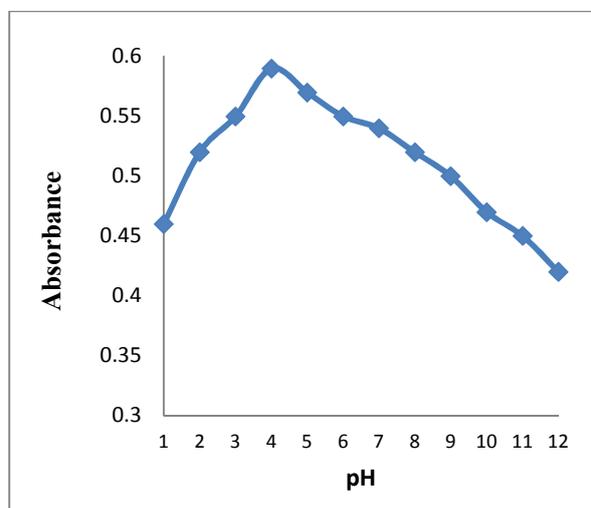


Figure 1: Effect of pH on Ru-HHMCP Complexation

### c) Effect of HHMCP reagent concentration

The minimum amount of reagent required for complete complexation of 300µg of Ru (III) was studied by varying the concentration of HHMCP (Table 5.2). The results obtained from the plot of absorbance versus concentration of HHMCP indicate that 7 ml of  $10^{-2}$  M reagent solution was sufficient for the quantitative extraction and spectrophotometric determination of 300µg Ru (III) (Figure 2) Addition of more reagent did not interfere with complexation and extraction of the

complex. Further study of complexation was carried out by using 7 ml of  $10^{-2}$  M HHMCP solution in chloroform to ensure the complete complexation.

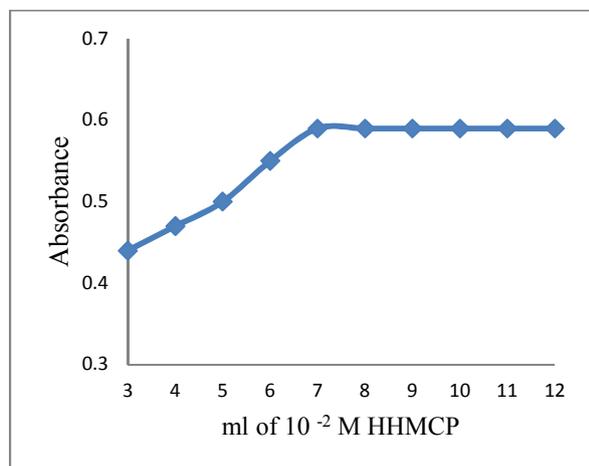


Fig.2 Effect of HHMCP reagent concentration

#### d) Effect of equilibrium time

The minimum equilibrium time for complete complexation of  $300\mu\text{g}$  Ru (III) was studied by varying the equilibrium period from 5 seconds to 10 minutes. (Table 5.3) The results obtained from the plot of absorbance versus equilibrium time indicated that minimum 1.0-minute equilibrium time was required for the quantitative extraction and spectrophotometric determination of  $300\mu\text{g}$  of Ru (III) (Figure 3). It was also observed that equilibrium time above 1.5 minute did not affect the complexation and extraction of the complex. Thus, further study of complexation was carried out by using 3 minutes as an equilibrium period.

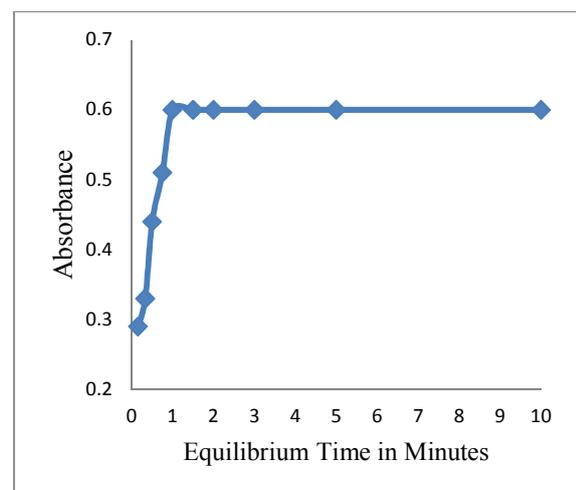


Figure 3: Effect of Equilibrium Time on Ru (III)-HHMCP Complexation

#### e) Calibration curve:

A calibration graph of Ru (III) was prepared by complexing varying amount of Ru (III) in the range  $0\mu\text{g}$  to  $80\mu\text{g}$  with 7 ml  $10^{-2}$  M HHMCP in chloroform. (Figure 4)

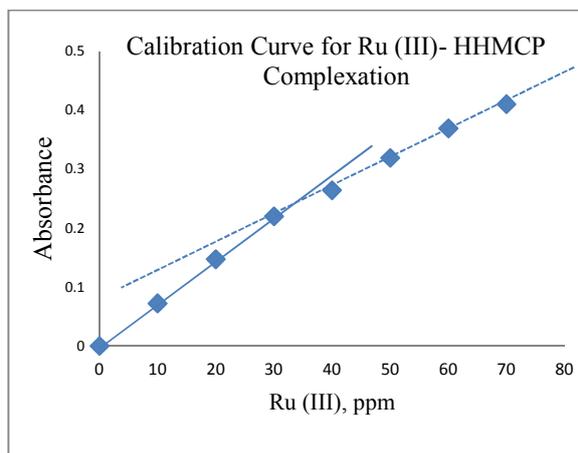


Figure 4: Calibration Curve for Ru(III)-HHMCP Complex

#### CONCLUSIONS:

An extractive spectrophotometric method was developed for estimation of Ruthenium (III). 2-[(E)-N-(2-{[2-(E)-[(2-hydroxyphenyl) methylidene] amino] phenyl} (methyl) amino} phenyl) carboximidoyl] phenol (HHMCP) was synthesized<sup>18</sup> and successfully used for quantitative extraction of Ruthenium (III) at pH 4.0. Since the equilibration time is very less, the method is very quick. The method is applicable for determination of Ruthenium (III) from alloys and mixtures.

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