



## Extractive Recovery of Ruthenium (III) from Organic Acid Solutions with 2-Dodecylaminopyridine

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### ABSTRACT

A novel approach was designed to extract ruthenium (III) from malonic acid solution through solvent extraction method. In this work, solvent extraction of ruthenium (III) from platinum group metals and base metals using high molecular weight amine: 2-dodecylaminopyridine as the extractant was investigated. The extraction behavior of ruthenium (III) was investigated as function of various parameters: pH and organic acid concentration of aqueous phase, concentration of extractant, phase ratio, phase contact time, diluents, and stripping from organic phase. Under the optimal conditions, extraction efficiency of ruthenium (III) was 99.50%, which indicate that it is an efficient extraction system for recovery of ruthenium.

**Key words:** Solvent extraction, Ruthenium (III), 2-dodecylaminopyridine, Malonic acid, Base metals.

### 1. INTRODUCTION

A significant portion of the platinum group metals (PGMs) resources has been moved during the past century from underground to above ground [1]. This movement has important economic, health, environmental, ecological, and sustainability implications for the earth and its population. Solvent extraction is generally used in hydrometallurgy for the separation of PGMs from laboratory scale to large scale operations. Separation of PGMs is extremely difficult task on account of their occurrence in the nature as well as due to their similar structure and chemical behavior [2]. These elements have paramount significance in technology, mainly these are used as metallic catalysts in automobiles to reduce air pollution [3] and applicable for surgical appliances, electronic devices, and hydrogen storage materials [4,5]. It is very difficult to fulfill the present demand of these elements just based on natural resources, hence, secondary resources should be tackled to meet the present requirement. The existing forms of PGMs and availability of metal species are highly depending on the acidic, basic, and chloride concentration in aqueous solution [6]. As the aqueous chemistry of one metal may resemble that of some others in mixture, selective recovery of any desired metal from such complex mixture is difficult [7]. The growing use of ruthenium metal in widely number of fields has made it necessary to develop simple,

inexpensive, and sensitive separation methods. When ruthenium is present in various matrices in extremely low concentrations, direct determination is not successful without previous preconcentration, and separation. Hence, it is inevitable to improve the separation factors and extraction efficiency by way of introducing newer extractants and by changing the solvent systems.

Several analytical methods such as ion exchange [8,9], sorption [10], precipitation [11], extraction chromatography [12], and solvent extraction has been reported for the separation of PGMs and separation from chloride solution [13,14]. Among them, solvent extraction has been widely used studied as a hot technology for recovering ruthenium from natural and secondary resources. Among the most recent solvent extraction of ruthenium (III) approaches, the systematic uses of extractants such as tertiary and quaternary amines [15-17] from concentrated acid solutions but difficult to strip from organic phase to aqueous phase [17,18], N-octylaniline used for selective extraction of ruthenium (IV) from hydrochloric acid medium [19], Ruthenium (III) has extracted from succinate media by N-n-decylaminopyridine as extractant [20]. However, all of traditional solvent extraction system used a large amount of extractant in different organic solvents, which may cause some of the environmental and workers health problems.

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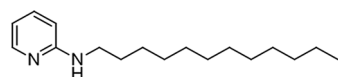
Furthermore, most of the systems were conducted under strong acidic condition [21,22] and resulted in severe corrosion to the equipment used for extraction purpose. For sustainability of the solvent extraction, it is necessary to find efficient, safer, and greener solvent extraction system.

In the present work, studies on the ruthenium (III) extraction from the oxalic acid, malonic acid, succinic acid, and citric acid solution were carried out using 2-dodecylaminopyridine (2-DDAP) as new extractant. The effects of several experimental parameters such as extractant concentration, aqueous phase pH, organic acid concentration, phase volume ratio (A/O), phase contact time, and diluents for organic phase on extraction behavior of ruthenium (III) have investigated and the results illustrated that the 2-DDAP as extractant and organic acid in aqueous phase was promising extraction method for ruthenium (III). The stripping of ruthenium (III) from organic phase has been performed. The proposed method was extended for the recovery of ruthenium (III) from synthetic mixture of other PGMs and base metals.

## 2. EXPERIMENTAL

### 2.1. Materials and Apparatus

A Shimadzu ultraviolet (UV)-Visible Spectrophotometer (UV 1800) with 1 cm<sup>2</sup> quartz cell was used for absorbance measurements, and pH optimization was carried out by Elico digital pH meter Model LI -120 ( $\pm 0.01$ ). All the chemicals used were of AnalaR grade and purchased from commercial suppliers Alfa Aesar (Thermo Fisher Scientific, Hyderabad, India) and Spectrochem (Spectrochem Pvt., Ltd., Mumbai, India). These are used as such without further purification. The stock solutions were prepared by dissolving appropriate amount of RuCl<sub>3</sub>·3H<sub>2</sub>O and other metal ions in deionized distilled water. A series of working solutions were prepared by diluting the stock solution. An extractant, 2-DDAP was synthesized by reported method [23] and characterized by spectral techniques. Doubly distilled water was invariably used throughout the experiments. The structure of the extractant 2-DDAP is shown as;



### 2.2. Solvent Extraction Procedure

To evaluate the extraction efficiency of ruthenium (III) from the aqueous phase, xylene was used as the organic phase owing to its hydrophobic property and good solubility for 2-DDAP as extractant. The extraction procedure is as follows: 100  $\mu\text{g mL}^{-1}$  of ruthenium (III) in organic acid solution as aqueous phase (25 mL) and 2-DDAP in xylene as organic phase (10 mL) were mixed in separatory funnel and vigorously shaken at ambient temperature within appropriate time. After

phase separation, ruthenium (III) from organic phase was stripped with two 10 mL portions of 2% sodium chloride solution. The extract was evaporated to moist dryness and leached with 1 mL of dil. hydrochloric acid solution, and ruthenium (III) was estimated spectrophotometrically with pyrimidine-2-thiol [20]. Distribution ratio (D) was calculated as the ratio of ruthenium (III) concentration in organic phase  $[\text{Ru}]_o$  to aqueous phase  $[\text{Ru}]_{aq}$  after extraction using equation:

$$D = \frac{[\text{Ru}]_o}{[\text{Ru}]_{aq}} \quad (1)$$

The percentage extraction of ruthenium (III) was calculated by equation, where  $V_{aq}$  is volume of aqueous phase and  $V_o$  is volume of organic phase,

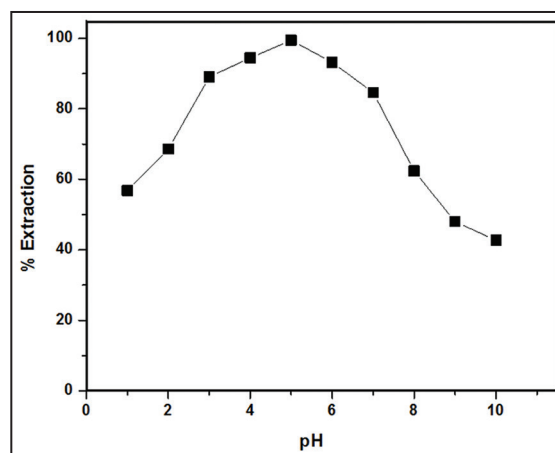
$$\% E = \frac{100 D}{D + \frac{V_{aq}}{V_o}} \quad (2)$$

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of pH of Aqueous Phase on Extraction

Aqueous phase pH is one of the determining factors for the ruthenium (III) extraction by 2-DDAP [24]. To examine the behavior of the extractant in different pH values and to determine the optimum pH, we have carried out a competitive extraction of ruthenium (III) at different pH. The extraction properties of ruthenium by the extractant 2-DDAP were investigated over the pH range 1-10.

Figure 1 shows the effect of pH on the selective extraction of ruthenium (III) which clearly indicate that the extraction efficiency decreased slowly with increasing pH of aqueous phase, which can be explained by stable ion pair formation [19]. Under the pH 8-10 efficiency decreases seriously and neglected when the pH attained a value of pH 10, this can be explained by rapid and total hydroxyl ions availability



**Figure 1:** Effect of pH on extraction of ruthenium (III) with  $5 \times 10^{-4}$  M 2-dodecylaminopyridine.

in the aqueous phase which impairs the ion pair formation of ruthenium (III) with 2-DDAP. It seems that the basic medium is not favorable for solvent extraction of ruthenium (III) with extractant 2-DDAP. Therefore, the aqueous phase pH 5 was used for further extraction experiments.

### 3.2. Effect of 2-DDAP Concentration

The extraction behavior of ruthenium (III) with various concentrations of 2-DDAP in organic phase was studied. The 2-DDAP concentration was varied over the range  $1 \times 10^{-4}$  M to  $1 \times 10^{-3}$  M. As shown in Figure 2, the increasing 2-DDAP concentration showed positive effect on ruthenium (III) extraction performance. To optimize the concentration of extractant required for maximum extraction, we carried out the extractions at different concentrations of 2-DDAP and the values of percentage extractions were plotted against concentrations of 2-DDAP then by extrapolating the straight line portion to get maximum concentration of extractant corresponding to the point of intersection of extrapolated lines. At  $5 \times 10^{-4}$  M concentration extraction is 99.50%. Whereas below or above, the extraction efficiency was less than maximum. Therefore,  $5 \times 10^{-4}$  M concentration of 2-DDAP was kept for further extraction experiments.

### 3.3. Effect of Diluents

In general, solvent extraction of PGMs is dependent on the nature of diluents (organic solvent) used during the experiments; therefore, selection of proper diluent is important for efficient extraction. A number of diluents were tested for efficient extraction of ruthenium (III) to assess a suitable solvent for the extraction of ion pair complex of ruthenium (III) with 2-DDAP. The results obtained by the use of different diluents are presented in Table 1, it is found that efficient extraction 99.5%, 92.48%, and 89.88% of ruthenium (III) was achieved if 2-DDAP solution was prepared in xylene, toluene, and ethylbenzene respectively while extraction was found incomplete in chloroform, n-hexane, and methyl isobutyl ketone. Xylene is recommended for further experiments because it offers efficient extraction and better phase separation.

### 3.4. Effect of Organic Acid Concentration

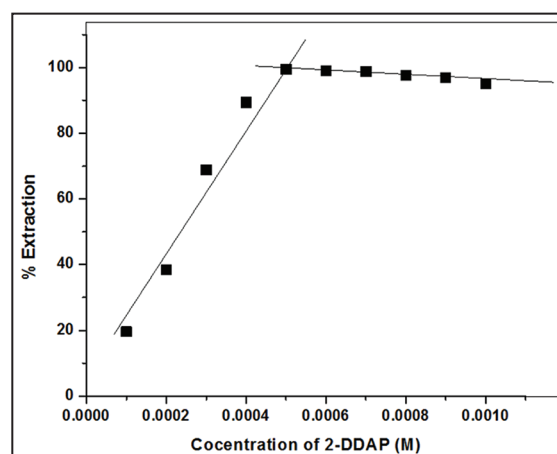
It is well known that mineral acids present in aqueous phase played a key role in the solvent extraction process of ruthenium [25]. Some researchers have developed the extraction of ruthenium (III) from hydrochloric acid solutions [26,27]. Now, we studied the extraction of ruthenium (III) by 2-DDAP in the presence of sodium salts of malonic acid, oxalic acid, succinic acid, and citric acid in aqueous phase, varied in the concentration range 0.01-0.1 M. The results obtained are presented in Figure 3. Obviously, the extraction efficiency increased rapidly with increasing organic acids concentration in the range 0.01-0.04 M, then followed a gradual decline. The maximum extraction

was found to be 99.50% in the presence of malonic acid and it decreased from 99.50% to 61.80% by increasing concentration up to 0.1 M in aqueous phase. This suggested that the addition of too much organic acid in aqueous phase reduces the probability of ion pair formation of ruthenium (III) with 2-DDAP at interfacial region [28]. Maximum percentage extraction showed by oxalic acid, succinic acid, and citric acid is 85.2%,

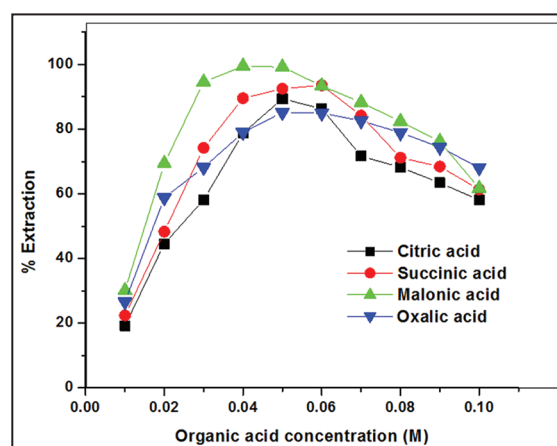
**Table 1:** Percentage extraction of ruthenium (III) in different diluents.

Entry	Solvent	% E	D
1	Xylene	99.50	497.50
2	Toluene	92.48	30.80
3	Ethylbenzene	89.88	22.45
4	n-Hexane	64.10	4.48
5	Chloroform	58.40	3.51
6	Isobutylmethylketone	61.76	4.05

D: Distribution ratio, %E: Percentage extraction



**Figure 2:** Effect of 2-dodecylaminopyridine concentration on extraction of ruthenium (III).



**Figure 3:** Effect of organic acids concentration in aqueous phase on extraction of ruthenium (III) with  $2 \times 10^{-4}$  M 2-dodecylaminopyridine.

83.6%, and 79.4%, respectively. In consideration of extraction efficiency, malonic acid exhibits as most excellent aqueous phase media for ruthenium (III) extraction with other used acids [18,22,26,27]. It is evidence to that the ion pair exchange mechanism leads to high extraction efficiency by the use of malonic acid in aqueous phase for extraction of ruthenium (III) by proposed method. Finally, neither third face formation nor stable emulsion was observed in this series of solvent extraction experiments. Therefore, for further studies of ruthenium (III) extraction, an aqueous phase containing 0.04 M malonic acid was chosen.

### 3.5. Effect of Aqueous Organic Phase Volume Ratio

The phase ratio also has considerable influence on the solvent extraction process of PGMs. Several workers have reported A/O phase volume ratio was 1 for the extraction of ruthenium (III). To evaluate the extraction potential of 2-DDAP toward ruthenium (III) under proposed conditions, the effect of phase ratio on the ruthenium (III) extraction was investigated covering different volume ratios of aqueous phase to organic phase from 1:1 to 10:1. The extraction of ruthenium (III) was efficient when volume ratios of aqueous phase to organic phase from 1:1 to 5:1, when the ratios were higher than 5:1, the extraction efficiency had decreased. Notably, 2-DDAP is a best extractant, and shows manifold distribution ratio for ruthenium (III) ( $D_{Ru} = 497.50$ ) than reported extractants. Considering the higher distribution ratio and extraction efficiency, the A/O phase ratio of 2.5 was used for further studies.

### 3.6. Effect of Phase Contact Time

The extraction behavior of ruthenium (III) prepared in malonate (0.04 M) solution with 2-DDAP in xylene was investigated at varied contact times of aqueous and organic phase from 20 to 180 s. Results obtained shows that the extraction efficiency reached 99.50% within 60 s, and after that the extraction remained almost constant without significant changes. The results indicate that 2-DDAP enables fast extraction of ruthenium (III) compared to previously used extractant. In consideration of sufficient extraction time, 60 s was chosen as contact time for the selective extraction experiments of ruthenium (III).

## 4. SELECTIVE EXTRACTION OF RUTHENIUM (III)

### 4.1. Separation of Ruthenium (III) from Base Metals

The proposed method allowed for the selective separation and determination of ruthenium (III) from binary mixture containing iron (III), cobalt (II), nickel (II), and copper (II). This method permits the separation of ruthenium (III) from commonly associated metals due to their differences in extraction conditions. Ruthenium (III) was successively extracted from base metals into organic phase by 2-DDAP at proposed conditions. Iron (III), cobalt (II),

nickel (II) and copper (II) remain quantitatively in aqueous phase and determined spectrophotometrically with thiocyanate [29], 1-nitroso-2-naphthol [29], DMG [29], and pyrimidine-2-thiol [30], respectively. Ruthenium (III) was stripped from organic phase with two 10 mL portion of 2% sodium chloride solution. The extract was evaporated to moist dryness and leached with 1 M hydrochloric acid solution and determined by spectrophotometrically with pyrimidine-2-thiol method. Recovery of ruthenium (III) from base metals was found to be efficient and results of experiments presented in Table 2.

### 4.2 Separation of Ruthenium (III) from Multicomponent Synthetic Mixture PGMs and Base Metals

Natural occurrence of ruthenium is always associated with PGMs and base metals; hence, its selective and efficient separation from these metals is of great importance. The selectivity shown for ruthenium (III) by 2-DDAP is obviously determinant key point that requires evaluation. Therefore, and again focusing the research on the use of malonic acid in aqueous phase solution with more practical interest, aqueous phase of ruthenium (III) with PGMs and base metals, the extraction experiments were carried out under proposed conditions. The overall results obtained are illustrated in Table 3. The displayed percentage extraction values are depicted significant digits, to allow the correct quantification. As expected, there are no significant differences between feed amount and extracted amount of ruthenium (III). It can be assumed 2-DDAP is selective for ruthenium (III) in presence of PGMs and base metals.

## 5. CONCLUSIONS

This systematic experimental study of the extraction of ruthenium (III) by 2-DDAP from malonate media showed that it is an efficient extraction system for the recovery of ruthenium (III) from other PGMs and base metals. The present investigation highlights that 2-DDAP is very effective ion-pair forming reagent which is highly basic than reported amines as

**Table 2:** Binary separation of ruthenium (III) from iron (III), cobalt (II), nickel (II) and copper (II).

Composition of metal ions ( $\mu\text{g}$ )	Ru (III) found* ( $\mu\text{g}$ )	Recovery $\pm$ S (%)
Ru (III), 100; Fe (III), 1000	99.5	99.5 $\pm$ 0.5
Ru (III), 100; Co (II), 2000	99.4	99.3 $\pm$ 0.3
Ru (III), 100; Ni (II), 1500	99.7	99.5 $\pm$ 0.4
Ru (III), 100; Cu (II), 1500	99.6	99.4 $\pm$ 0.6

\*Average six determination. S: Standard deviation

**Table 3:** Analysis of synthetic mixture of PGMs and base metals.

Composition ( $\mu\text{g}$ )	Ru (III) found* ( $\mu\text{g}$ )	Recovery $\pm$ S (%)
Ru, 100; Pt, 500	99.6	99.5 $\pm$ 0.2
Ru, 100; Pd, 500	99.4	99.4 $\pm$ 0.3
Ru, 100; Rh, 500	99.5	99.5 $\pm$ 0.2
Ru, 100; Au, 500	99.7	99.5 $\pm$ 0.4
Ru, 100; Pt, 500; Pd, 500	99.3	99.3 $\pm$ 0.1
Ru, 100; Pt, 500; Rh, 500	99.4	99.5 $\pm$ 0.4
Ru, 100; Pt, 500; Au, 500	99.5	99.4 $\pm$ 0.6
Ru, 100; Pt, 500; Pd, 500; Au, 500	99.6	99.7 $\pm$ 0.3
Ru, 100; Pt, 500; Rh, 500; Au 500 Fe, 1000; Co, 2000; Ni, 1500; Cu, 1500	99.5	99.4 $\pm$ 0.5

\*Average six determination. PGM: Platinum group metals, S: Standard deviation

extractant. Extraction of ruthenium (III) does not need the addition of any modifier and extracted in single step from organic acid solution at pH 5. The stripping reagents used simple and convenient for selectivity and separation of ruthenium (III). The important feature of this method are that low extractant concentration is required for complete extraction of ruthenium (III) within 60 s. The proposed method showed a remarkable high affinity and selectivity toward ruthenium (III) under the optimized conditions and expected to be the new benchmark for the separation of ruthenium (III) from PGMs.

## 6. ACKNOWLEDGMENTS

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