Abstract

In the current study, a novel analytical reagent called 2,4-dimethyl-3H-1,5-benzodiazepine (DBA) is developed for extractive spectrophotometric analysis to determine the amount of rhodium (III) in an alloy mixture.

In this study, a novel analytical reagent was investigated, and its characterisation using NMR, IR, and mass spectrometer techniques was also described.

When subjected to an extraction using n-butanol as the selected solvent at a constant pH of 8.9, the red-colored product produced by the reaction of Rhodium (III) and analytical reagent (DBA) provides exceptional results. Rhodium (III) ion concentrations between 1 mg L-1 and 10 mg L-1 completely satisfy the Beers law. Maximum and average molar absorption coefficient values for the coloured compound were noted to be 510 nm, 4863 L mol-1 cm-2. Rhodium (III) metal traces in alloy mixtures can be determined utilising DBA analytical reagent in a more efficient, cost-effective, and superior manner than with prior spectrophotometric extraction techniques.

1. Introduction

Rhodium is a precious element belonging to the platinum group that is frequently found in minerals. It is a member of the metals group, which includes metals like Ruthenium, Platinum, Palladium, Rhodium, Osmium, and Iridium. Due to their remarkable physical and chemical qualities, as well as new applications outside the jewellery and ornamental sectors, these metals are in greater demand. Currently, it has several applications in the realm of catalysis and cutting-edge electrical equipment. In addition, it is in high demand for use as a catalyst in modern electrical appliances and petroleum refinery activities.

There are other methods of extraction utilised for this purpose, but the majority of them have certain drawbacks like taking more time and costing more money.

But among all separation techniques, solvent extraction is one of the most popular methods for identifying rhodium metal ions at both the micro and macro levels in supplied samples since it is quick, precise, and affordable.

Experiments were conducted utilising various analytical reagents for the extraction of platinum group elements, such as tributyl phosphate [2], Alamine 336 [3], BMMT [4], HMAINH [5], and bis-(2-ethylhexyl) phosphoric acid [6]. In comparison to the examples given above, Schiff bases (which contain nitrogen and sulphur) showed promising outcomes when it came to the identification and separation of a group of platinum metals [7].

The extractive determination of rhodium (III) has also been studied using a number of reagents, including diocetyl sulphides [8], Kelex 100 [9], N,N-Dialkyl-N'-benzoyltioureas [10], and trialkylphosphinesulphide [11]. It has been reported that rhodium (III) was extracted in the presence of tin using the analytical reagent N, N-dimethyl N, N' diphenyltetradecylmalonamide (DMDPHTDMA) [12]. Sodium malonate was used to extract rhodium (III) using N-n-octylaniline as the analytical reagent [13].

Additionally, it was found that the analytical reagents cyanex 923 and cyanex 471X could be used to recover rhodium (III) ions from bromide medium when stannous chloride was present [14]. For the purpose of determining rhodium (III), an experimental research utilising the reagents water-soluble porpyrin 5, 10, 15, and 20 - tetrakis(4-N- methylpyridyl) and porphine (TMPYM) was described [15].

The current experimental investigation shows the creation of a precise and dependable approach for extractive spectrophotometric measurement of rhodium (III) metal traces employing fresh analytical reagent 2, 4-dimethyl-3H-1, 5 benzodiazepine (DBA).

For the determination of rhodium (III) traces, the devised method is found to be superior to the other published extraction procedures.
2.2 Analytical reagent development (DBA)
To make the DBA reagent, as described in, 1 mole of o-phenylenediamine and 1 mole of acetyl acetone are well combined in 1 mole of ethanol (solvent) (Figure 1). After that, the prepared mixture is heated in a water bath to a constant temperature of 100 o C in a round-bottomed glass flask and kept it completely refluxing for two hours. Following an elaborate process, the solution is obtained. It is then put in an ice bath to form crystals, which are subsequently recrystallized using ethanol as a solvent. After being produced, the novel reagent (M.P. 274°C) is identified by its NMR and IR spectra. Rhodium (III) metal traces can be determined spectrophotometrically using the developed reagent. Additionally, a 0.05% concentration stock solution of the DBA reagent in methanol was made in preparation for additional experimental work.

2.3 Stock Solution
By dissolving 0.255 mg of rhodium chloride (RhCl3(H 2 O)3) (Merck-AR Grade) in distilled water, a stock solution containing 100 ml (100 ppm concentration) of rhodium(III) metal ions was created. As and when needed, more diluted solutions were created by diluting the necessary volume of aliquots from the stock solution with distilled water.

2.4. pH Extraction Selection
Rhodium (III) extraction was carried out in this investigation using a variety of buffer solutions with pH ranges from 1 to 11, while keeping a constant 1:1 molar ratio between the organic phase and aqueous phase. After that, when the pH rises, the absorbance falls. Therefore, it was suggested that future research use a buffer with a pH of 8.9.

2.5. Extraction Solvent Selection
Various organic solvents were examined in this part to determine their appropriateness for extracting rhodium(III) metal ions, and the results are shown in Figure 3. The n-butanol was shown to be the best solvent compared to all others tested for extractive rhodium determination (III).

2.6. Wavelength selection
Picture 4 depicts the relationship between wavelength and absorbance. It is clear from this figure that as wavelength increases, absorbance similarly rises, with maximum absorbance observed at 510 nm. Further wavelength increases beyond 510 nm cause a sharp decline in absorbance values. We used this wavelength's maximum absorption value for our further research.

3. Experimental Method for Rhodium Determination(III)
1 ml of rhodium chloride stock solution and 0.05% DBA reagent in methanol should be well combined in a beaker. The pH was then corrected to 8.9 by adding buffer solution. This mixture was then combined with 10 ml of n-butanol and put through a glass funnel for separation. The organic and aqueous phases are separated using this glass funnel. The spectrophotometer operating at 510 nm wavelength is used to measure the concentration of rhodium (III) in the 688 phase.

3.1 Calibration Curve Formation, Section
In addition to the experimental approach, the absorbance of known concentrations of rhodium (III) ions were measured using a spectrophotometer. Rhodium (III) concentration samples in a range of concentrations (1–10 ppm) were generated, combined with DBA reagent, and then subjected to extraction using n-butanol as solvent.

3.2. Choice of Rhodium (III) to DBA Molar Ratio
Illustrates the use of various techniques for determining the ideal molar ratio of rhodium (III) ions to DBA reagents. The Job's continuous variation method, one of the frequently used recommended techniques, was used to confirm the extracted species' composition ratio.

Additionally, for the aim of validation, the results of Job's continuous variation approach were compared to those of the mole ratio method and the slope ratio method. From the aforementioned validation, it is advised to maintain an ideal molar ratio of 1:1 between the analytical reagent (DBA) and the rhodium (III) in subsequent experimental studies.

3.3 Impact of Different Ions
By employing the suggested analytical process to determine 100 g/mL of rhodium (III) with a known quantity of various ion solutions, the effects of many different ions were experimentally tested in order to validate the present method. The tolerance criteria for a certain ion were taken into account when the absorbance value deviated by more than 2% from the value of rhodium (III) ions as expected. When determining the acceptable limit for rhodium (III) ions, the tolerated concentration of various metal ions is presented in Table 3. For the real determination of rhodium (III), the interference effects of these ions are inhibited by a number of agents, as shown in Table 3.

3.4. Different Analytical Reagents for Rhodium (III) Determination and Their Shortcomings
Compares the use of DBA reagent and a variety of analytical reagents by earlier studies in order to determine the presence of rhodium (III).
Comparing the results of DBA reagents to certain previously published reagents, it is discovered that they are in good accord.

4. Use of the Work at Hand
With the help of the current research, the determination of rhodium (III) ions has been successfully made. This newly discovered technique has numerous uses in the glass,
pharmaceutical, chemical, and petroleum processing sectors. Additionally, it may be effectively used to measure rhodium ions in synthetic and alloy sample materials. It is determined that the newly created extractive spectrophotometric method is superior to the older approaches.

5. Finalization

The current experimental investigations show that a newly created analytical reagent, 2, 4-dimethyl-3H-1, 5-benzodiazepine (DBA), is excellent for accurate rhodium prediction (III).

The following are some advantages of the created method:

i. Synthesis of analytical reagents is simple.

ii. The extraction process is straightforward, single-stage, and quick.

iii. The technique for quantifying rhodium calls for a very low reagent concentration (III).

iv. The developed approach has exceptional selectivity and virtually no anions or most cations interfere when used. Using a proper masking agent, the area and suitability of applications can be improved.

v. The new method’s key advantages are accuracy and precision in addition to simplicity, sensitivity, and speed. It may be successfully used to estimate the presence of rhodium (III) metal ions in a synthetic mixture.

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References


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