



CHROMATOGRAPHIC EXTRACTION OF PALLADIUM(II) IONS IN MULTI-COMPONENT MIXTURE USING POLY[DIBENZO-18-CROWN-6] AS SORBENT

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Abstract

The extractive and separation properties of poly[dibenzo-18-crown-6] are studied for palladium (II). In present study, poly [dibenzo-18-crown-6] was used as a sorbent for chromatographic extraction and separation of palladium (II) in glycine medium. At optimized conditions the sorbent shows greater than 98% recovery for palladium(II) ion. The separations are carried out using glycine medium. palladium(II) uptake was quantitative from 1×10^{-7} to 5×10^{-1} M glycine. Palladium(II) was quantitatively eluted with 3.0–8.0 M HCl, 2.0-5.0 M HClO₄, 4.0-8.0 M HBr and 4.0-8.0 M H₂SO₄, while CH₃COOH was not an effective eluent for palladium(II). The sorption capacity of poly [dibenzo-18-crown-6] was found to be 0.845 ± 0.1 mmol /g. The sorption study of Palladium(II) as function of glycine, capacity of poly[dibenzo-18-crown-6], effect of interfering ion and elution profile has been studied. palladium(II) was separated from multi-component mixtures. The developed techniques of palladium(II) ion is simple, effective with good stability and reproducibility (approximately $\pm 2\%$).

Keywords: Palladium(II), Poly[dibenzo-18-crown-6], Extraction Chromatography, Glycine.

I. Introduction

In separation Science today much attention is paid to the analytical chemistry of platinum group metals. Interest in the developing analytical techniques for determination of the noble metals is growing as a result of their applications in medicine micromechanics and chemical engineering. The increasing demand of the industry for precious metals with the ever-increasing demand for catalysts in the chemical industries and automotive manufacture, in the production of ultrapure hydrogen, in electric and electronic devices etc., the global demand for palladium metal has increased manifold. Elements of the platinum group are present in the earth's crust in low concentrations and the requirement for precious metals is increasing¹⁻³. Palladium has no biological role, but all palladium compounds should be regarded as highly toxic and carcinogenic, causing asthma, allergy, rhino conjunctivitis⁴. The platinum group elements for multicomponent mixtures the chromatographic techniques have been applied in the search for better and faster separation methods⁵. Photoconductive, Electrical and optical properties of Poly[dibenzo-18-crown-6] have been investigated⁶. In field of chemistry, various crown ethers are used. Polymerization of crown ethers resulted in a new type of stationary phases in extraction chromatography. polymeric of crown ether shows high resistance to chemicals, radiolysis and temperature as well as to polar solvents shown by poly[dibenzo-18-crown-6] could be exploited for the sorption studies of various cations using column chromatography⁷⁻¹².

Several sorbents have been reported for palladium (II)¹³⁻²⁸. A comparative study of present technique with other reported technique for palladium(II) as shown Table 1.

According to literature review, it is concluded that there are no reports on the use of poly[dibenzo-18-crown-6] for the chromatographic separation of palladium(II) in glycine medium. The advantages of present method over reported method is that, the

glycine is not hazardous, the concentration of glycine required for quantitative sorption of palladium(II) is very low. Sorption capacity is relatively high as compared to other sorbent activated carbon modified with ethyl-3-(2-aminoethylamino)-2-chlorobut-2-enoate. The aim of the present study is to optimize the laboratory scale conditions for the chromatographic separation and recovery of palladium (II) from other associated metal ions employing poly[dibenzo-18-crown-6] as the sorbent in glycine medium. In our study we have reported the optimum sorption conditions were determined by investigating effective parameters such effect of concentration of glycine, various eluting agents, sorption capacity, effect of diverse ion and stability and reusability of the poly[dibenzo-18-crown-6]. In addition selective extraction and separation of palladium(II) from platinum(IV), zinc(II), cobalt(II), copper(II), strontium(II), cadmium(II), nickel(II), manganese(II), vanadium(III), beryllium(II), thallium(III).

II. Material and methods

2.1 Apparatus and reagents

A Visible spectrophotometer (Chemito 215D), a digital pH meter (Model LI-120, Elico, India) with glass and calomel electrodes and a digital flame photometer (PI, Model no. 041, and India) were used.

All chemicals used were of analytical reagent grade and distilled deionized water was used in the preparation of all solutions in the experiment. A palladium(II) stock solution was prepared by dissolving 1.0023 g of palladium chloride (S. d fine chemical) in 100 mL distilled deionized water and calibrated gravimetrically²⁹. A solution containing 50 µg/mL of palladium(II) was prepared by appropriate dilution of the standard stock solution. Glycine solution (1×10^{-1} M) was prepared by dissolving 0.705 g of glycine (Merck Limited Mumbai) in distilled deionized water and diluted to 100 mL.

A crown ether polymer, poly[dibenzo-18-crown-6] from Merck (Darmstadt, Germany) was used after screening to 100–200 mesh. A total of 0.5 g of polymer was slurred with distilled deionized water and poured in to a Pyrex glass chromatographic column (20x0.8 cm I.D.). The column was used after preconditioning with a glycine solution.

2.2 General procedure

To an aliquot of total volume 10 ml solution containing 50 µg of palladium(II) was added glycine solution in the concentration range of 1×10^{-1} to 1×10^{-8} M. The resulting solution was then passed through a poly[dibenzo-18-crown-6] column at flow-rate 0.5 mL/min. The column was preconditioned with the same concentration of glycine as that of sample solution. The column was then washed with the same concentration of glycine. The sorbed palladium(II) ion was eluted with different eluting agents at a flow rate of 0.5 mL/min. A total of 5.0 ml fractions were collected and the palladium(II) content was determined by spectrophotometrically with PAR at 520 nm³⁰⁻³¹. The concentration of palladium(II) was evaluated from a calibration graph.

III. Results and discussion

3.1 Sorption of palladium(II) as function of glycine

palladium(II) sorption studies were performed from glycine medium. The concentration of glycine was varied from 1×10^{-8} to 1×10^{-1} M. After sorption, palladium(II) was eluted with 4.0 M HCl. It was found that there was quantitative sorption of palladium(II) from 5×10^{-2} to 1×10^{-4} M as shown in Table 3.1.1. The percent sorption of palladium(II) increased with decreasing glycine concentrations up to 1×10^{-4} M. Glycine acts as a counter anion and forms an ion pair formation that sorbed on poly[dibenzo-18-crown-6] for palladium(II). For further study sorption of palladium(II) were carried out with 1×10^{-3} M glycine.

3.2 Elution Profile of palladium(II)

50 µg of palladium(II) was sorbed on the poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration. After sorption, the elution of palladium(II) was eluted from column with different strength of acids such as HBr, HClO₄, HCl, CH₃COOH and H₂SO₄. The concentration of eluting agent varied from 0.1 to 8.0 M. palladium(II) was quantitatively eluted with 3.0–8.0 M HCl, 4.0–8.0 M HBr, 2.0–5.0 M HClO₄ and 4.0–8.0 M H₂SO₄, with CH₃COOH was being an ineffective eluent for palladium(II). For elution study, the total volume of eluent was 25 mL and maximum volume of the collected eluent fraction was 5.0 mL. The obtained results showed that 15 mL of 4.0 M HCl solution was sufficient for the complete elution of palladium(II) and therefore in this work 15 mL of 4.0 M HCl solution was chosen. Fraction study of palladium(II) with various eluting agent is shown in Figure.3.2.1-3.2.5.

3.3 Capacity of poly [Dibenzo-18-crown-6]

4.0 M HCl as eluent to find out the sorption studies of palladium(II) were performed on 1.0 g poly[dibenzo-18-crown-6] column with 1×10^{-3} M glycine. The palladium(II) concentration was varied from 50 to 600 µg/10mL solution. The results indicate that the quantitative sorption was up to 450 µg/10mL solution for palladium(II). As the palladium(II) concentration increased, the percent sorption was decreased as shown in Figure.3.3.1. The polymer capacity of poly[dibenzo-18-crown-6], determined at 1×10^{-3} M glycine was found to be 0.845 ± 0.01 mmol/g for palladium(II).

3.4 Effect of interfering ion

Palladium(II) was extracted in the presence of other cations and anions as shown in Table 3.4.1. The different concentration of some cations and anions as their nitrate, chloride and sulphate salt were added individually to total volume 10 ml of solution containing 50 µg of palladium(II) and 1×10^{-3} M glycine. Under the optimized condition, multiple foreign ions were not sorbed and therefore passed through the column. The tolerance limit was set as the amount of foreign ions required to cause to $\pm 2\%$ error in the recovery of palladium(II). The alkali and alkaline earth metal ion indicate high tolerance limit except Ba(II), Mg(II) and Ca(II). Some of the p-block and d-block cation indicate low tolerance limit except Sn(II), Hg(II). Among other things, the inner transition elements indicate low tolerance limit. The anions of inorganic and organic acids indicate high tolerance limit. Co-extraction of different cations was demonstrated by performing separate experiments spectrophotometrically using various chromogenic reagents.

3.5 Application for Separation of palladium(II) from multi-component mixture

In present study, it was shown that the proposed poly[dibenzo-18-crown-6] can be successfully used for the separation of Palladium(II) and Platinum(IV) ion with other ions. A mixture containing platinum(IV), zinc(II) /cobalt(II)/copper(II)/ strontium(II)/ cadmium(II)/ nickel(II)/manganese(II)/ vanadium(III)/ beryllium(II)/ thallium(III) and palladium(II) was passed through poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration. Platinum(IV) was not sorbed on column and therefore passed through column, whereas zinc(II) /cobalt(II)/copper(II)/ strontium(II)/ cadmium(II)/ nickel(II)/manganese(II)/ vanadium(III)/ beryllium(II)/ thallium(III) and palladium(II) were sorbed on column. The sorbed zinc(II)/cobalt(II)/copper(II)/ strontium(II)/ cadmium(II)/ nickel(II)/manganese(II)/ vanadium(III)/ beryllium(II)/ thallium(III) and palladium(II) was first eluted with 15 mL of 0.5 M CH₃COOH under this condition palladium(II) was not eluted and finally palladium(II) was eluted with 4.0 M HCl. The zinc(II)/cobalt(II)/copper(II)/

strontium(II)/ cadmium(II)/ nickel(II)/manganese(II)/ vanadium(III)/ beryllium(II)/ thallium(III) / platinum(IV) and palladium(II) in the respective effluents are determined Spectrophotometrically. The data show that excellent separation and recovery is above 98 % as shown in Table 3.5.1 and all chromatograms indicate in figure 3 5.1- 3 5.10.

3.6 Stability and reusability of Crown polymer

The reusability of the poly[dibenzo-18-crown-6] was tested by passing 50 µg of palladium(II) solution in 1×10^{-3} M glycine medium. The sorbed metal ions were eluted with 4.0 M HCl and analyzed spectrophotometrically with PAR at 520 nm³⁰⁻³¹. Similar operations were performed on the same column and it was found that the sorption capacity of poly[dibenzo-18-crown-6] remains constant with standard deviation 0.06 even up to 29 cycles repeatedly. It was also noted that the poly[dibenzo-18-crown-6] shows good chemical stability which is of great significant. The sorbent Poly[dibenzo-18-crown-6] is highly efficient, reusable and chemically stable for number of extractions.

3.7 Comparative study of sorption capacities of some sorbent used for separation of Palladium(II)

The sorption capacity is an important factor. for palladium(II) sorption capacity of poly[dibenzo-18-crown-6] was also compared with other reported sorbents as shown in Table 1. As seen from data; sorption capacity of poly[dibenzo-18-crown-6] has relatively high compared to reported sorbents except activated carbon modified with ethyl-3-(2-aminoethylamino)-2-chlorobut-2-enoate. Use of this sorbent palladium(II) can be separated from associated metal ions. The Poly[dibenzo-18-crown-6] is reusable and chemically stable for number of extractions as compared with reported sorbents.

IV.CONCLUSIONS

In present research, the recovery and separation behavior of palladium (II) were investigated by using poly[dibenzo-18-crown-6] loaded column and It is concluded that the poly[dibenzo-18-crown-6] is an efficient sorbent for chromatographic extraction and separation of palladium(II) from multicomponent mixture. Quantitative sorption of palladium(II) was required for very low glycine concentrations. The capacity of the Poly[dibenzo-18-crown-6] sorbent in the glycine medium is quite more than other reported sorbents. In this study, we report an strategy for the selective and sequential separation of palladium(II) from platinum(IV)/ copper(II)/ zinc(II)/cobalt(II)/ strontium(II)/ cadmium(II)/ manganese(II)/ nickel(II)/ vanadium(III)/ beryllium(II)/ thallium(III). The sorption capacity of crown polymer determined at 1×10^{-3} M glycine was found to be 0.8450 ± 0.01 mmol g⁻¹. Poly[dibenzo-18-crown-6] is used as a good solid support because it has higher stability as a stationary phase. The developed techniques of palladium(II) is simple, effective and good reproducibility that's accounts for an recovery greater than 98% (approx. $\pm 2\%$).

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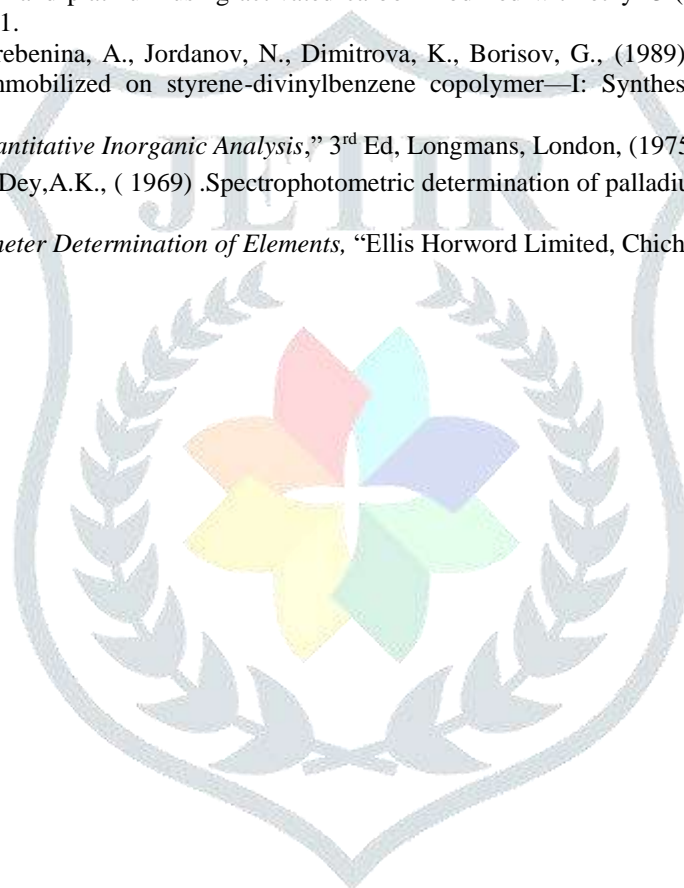


Table 1 Comparative study of the present technique with other techniques for palladium(II)

Sr. No	Sorbent	Capacity mmol / g	Condition	Ref. No
1	Bis(3aminopropyl)amine bonded silica gel (BAPA-SG)	0.299	pH=1	13
2	Melamine-formaldehyde-thiourea (MFT) resin	0.143	pH =4	14
3	PA-lignin EN-lignin	0.094 0.213	0.5M HCl 0.5M HCl	15
4	Fe ₃ O ₄ nanoparticle	0.103	pH =2.5	16
5	Alumina modified polyethylene imines (PEI)	0.122	pH = 6–8	17
6	Thiol cotton fiber	0.300–0.394	0.5 mol/L HCl (10% NH ₄ Cl)	18
7	Bayberry tannin immobilized collagen fiber membrane	0.313	1mol/L HCl	19
8	Amerlite XAD-16	0.33	pH =4	20
9	Silica gel immobilized thiourea	0.284	0.5 M HCl	21
10	9,10-phenanthraquinone ethylthiosemicarbazone (PET)	0.31	pH =8, 2 mol/dm ³ H ₂ SO ₄	22
11	macroporous poly(vinyl-aminoacetone)	0.630	Cl ⁻ , pH=5-9	23
12	Dimethyl glyoxime on Silica gel	0.038	pH = 4	24
13	Amerlite IRC 718	0.489–0.549	pH = 1–6	25
14	Ion-exchange resins containing s-bonded i)Dithizone ii)Dehydrodithizone	0.680 0.490	1 M HCl 1 M HCl	26
15	Activated carbon modified with ethyl-3-(2-aminoethylamino)-2-chlorobut-2-enoate	0.864	pH=1-6	27
16	pyrazolone amines immobilized on styrene-divinylbenzene copolymer	0.560	HCl, pH=1-6	28
17	Poly(dibenzo-18-crown-6)	0.845	0.001 M glycine	P.M*

* = Present method

Table 3.1.1. Sorption of palladium(II) as function of glycine
Pd(II)-50µg/mL, Eluent-4.0 MHCl

Concentration of glycine (M)	Percentage sorption of Pd(II)
1x10 ⁻¹	86
1x10 ⁻²	95
5x10 ⁻³	100
1x10 ⁻³	100
5x10 ⁻⁴	100
1x10 ⁻⁴	100
1x10 ⁻⁵	97
1x10 ⁻⁶	91
1x10 ⁻⁷	79.30
1x10 ⁻⁸	66.57

Table 3.4.1. Effect of diverse ion

Pd(II)-50µg/ mL, Eluent-4.0 MHCl, Condition-1x10⁻³ M glycine

Ion	Added as	Tol.Limit(mg)	Ion	Added as	Tol.Limit(mg)
Li ⁺	LiCl	8	Al ³⁺	Al ₂ (SO ₄) ₃ .16H ₂ O	0.4
Na ⁺	NaCl	14	Cr ³⁺	Cr(NO ₃) ₃ .9H ₂ O	0.5
K ⁺	KCl	4	Cu ²⁺	CuCl ₂	0.1
Rb ⁺	RbCl	9	Ce ³⁺	CeCl ₃ .6H ₂ O	0.2
Cs ⁺	CsCl	14	Zr ⁴⁺	Zr(NO ₃) ₄ .4H ₂ O	0.2
NH ₄ ⁺	NH ₄ Cl	3	Th ⁴⁺	Th (NO ₃) ₄ .6H ₂ O	Co-extract
Be ²⁺	BeSO ₄ .4H ₂ O	Co-extract	V ⁴⁺	VO ₂ .4H ₂ O	0.3
Mg ²⁺	MgCl ₂ .6H ₂ O	2	U ⁶⁺	UO ₂ (NO ₃) ₂ .6H ₂ O	Co-extract
Ca ²⁺	CaCl ₂	1	W ⁶⁺	Na ₂ WO ₄ .4H ₂ O	1.5
Sr ²⁺	Sr(NO ₃) ₂	6	Cl ⁻	HCl	4
Co ²⁺	CoCl ₂ .6H ₂ O	Co-extract	Br ⁻	HBr	2
Ba ²⁺	Ba(NO ₃) ₂	0.9	SCN ⁻	NaSCN	2
Ni ²⁺	NiCl ₂ .6H ₂ O	Co-extract	CH ₃ COO ⁻	CH ₃ COOH	3
Mn ²⁺	MnCl ₂ .4H ₂ O	0.2	ClO ₄ ⁻	HClO ₄	4
Zn ²⁺	ZnCl ₂	0.3	SO ₄ ²⁻	H ₂ SO ₄	2
Cd ²⁺	(CH ₃ COO) ₂ .H ₂ O	6	EDTA	EDTA	1
Pb ²⁺	Pb(NO ₃) ₂	0.1	Ascorbate	Ascorbic acid	2
Hg ²⁺	HgCl ₂	3	Tartrate	Tartaric acid	3
Sn ²⁺	SnCl ₂ .2H ₂ O	2	Oxalate	Oxalic acid	2
Tl ³⁺	Tl(NO ₃) ₃ .3H ₂ O	0.1	Citrate	Citric acid	7
Fe ³⁺	FeCl ₃ .6H ₂ O	0.1			

Table 3.5.1. Application for separation of palladium(II) from multicomponent mixture

Sorption condition=1x10⁻³M

*Average of analysis
 **NSPC=No
 Passing
 Column

Sr. No	Mixture	Taken (µg)	Recovery (%)*	Eluent	Standard Deviation
1	Pt(IV)	100	99.03	NSPC**	0.48
	Ni(II)	25	99.68	0.5 M CH ₃ COOH	0.15
	Pd(II)	50	99.26	4.0 M HCl	0.56
2	Pt(IV)	100	98.71	NSPC**	0.36
	Zn(II)	25	99.27	0.5 M CH ₃ COOH	0.24
	Pd(II)	50	99.17	4.0 M HCl	0.72
3	Pt(IV)	100	98.87	NSPC**	0.36
	Cu(II)	25	99.28	0.5 M CH ₃ COOH	0.23
	Pd(II)	50	99.17	4.0 M HCl	0.41
4	Pt(IV)	100	98.0	NSPC**	0.30
	Co(II)	25	97.0	0.5 M CH ₃ COOH	0.38
	Pd(II)	50	99.01	4.0 M HCl	0.56
5	Pt(IV)	100	98.61	NSPC**	0.37
	Cd(II)	25	99.08	0.5 M CH ₃ COOH	0.40
	Pd(II)	50	99.10	4.0 M HCl	0.67
6	Pt(IV)	100	99.43	NSPC**	0.38
	Mn(II)	25	98.65	0.5 M CH ₃ COOH	0.59
	Pd(II)	50	99.09	4.0 M HCl**	0.69
7	Pt(IV)	100	98.62	NSPC**	0.37
	Sr(II)	25	99.36	0.5 M CH ₃ COOH	0.36
	Pd(II)	50	99.17	4.0 M HCl	0.72
8	Pt(IV)	100	98.51	NSPC**	0.36
	Be(II)	10	98.95	0.5 M CH ₃ COOH	0.43
	Pd(II)	50	99.45	4.0 M HCl	0.27
9	Pt(IV)	100	98.90	NSPC**	0.48
	Tl(III)	25	98.77	0.5 M CH ₃ COOH	0.35
	Pd(II)	50	99.28	4.0 M HCl	0.56
10	Pt(IV)	100	99.02	NSPC**	0.73
	V(III)	25	99.01	0.5 M CH ₃ COOH	0.49
	Pd(II)	50	99.52	4.0 M HCl	0.71

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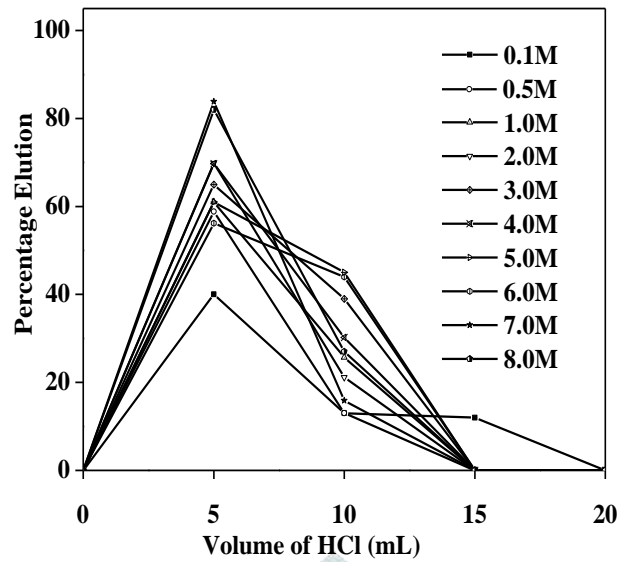


Figure. 3.2.1: Elution profile of palladium(II) with HCl

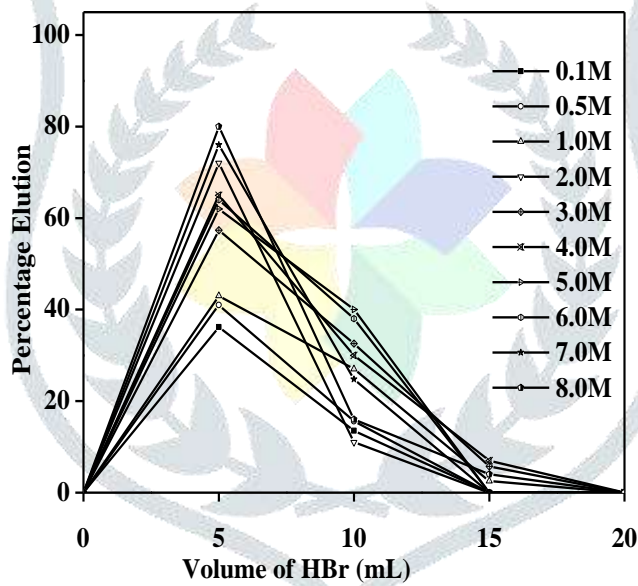


Figure. 3.2.2: Elution profile of palladium(II) with HBr

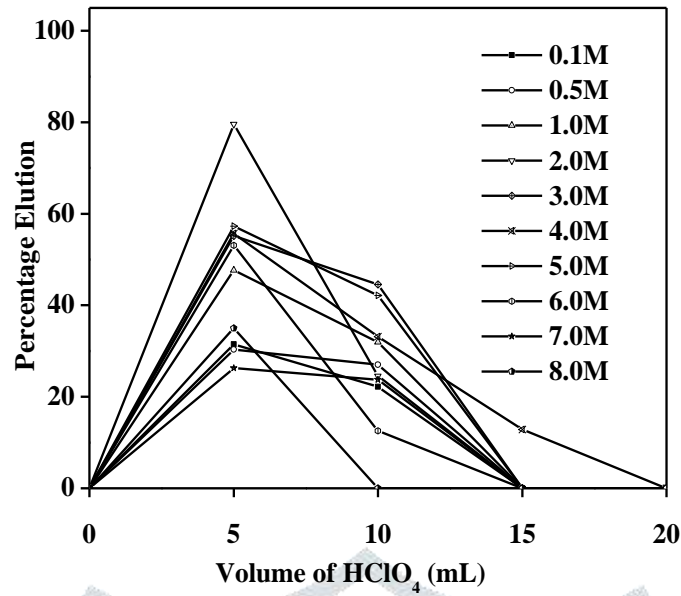


Figure3.2.3: Elution profile of palladium(II) with HClO₄

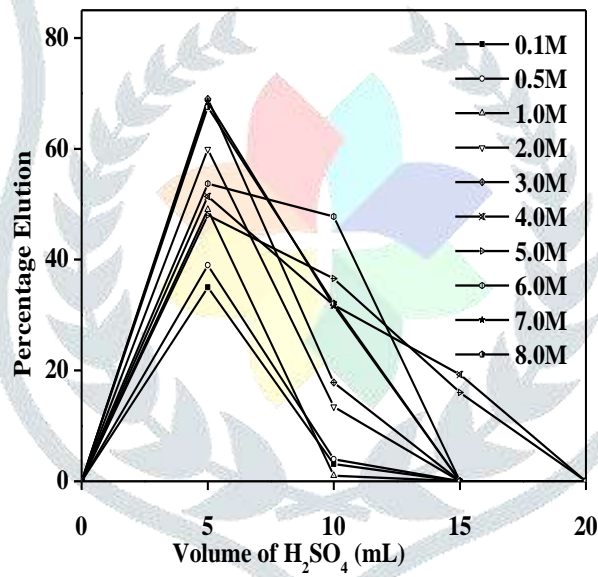


Figure. 3.2.4: Elution profile of palladium(II) with H₂SO₄

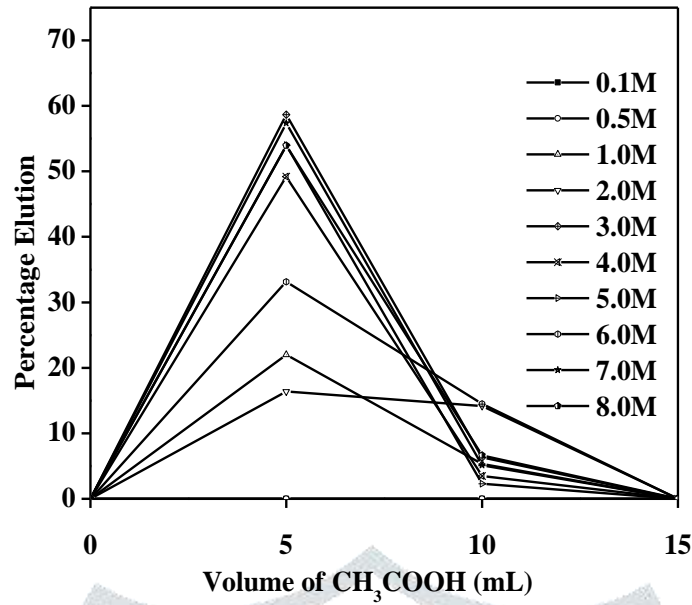


Figure. 3.2.5: Elution profile of palladium(II) with CH₃COOH

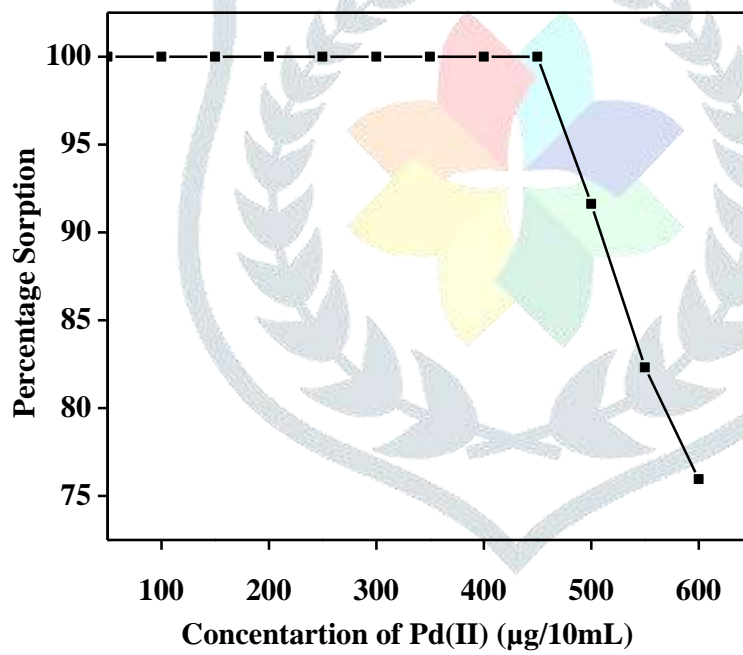


Figure. 3.3.1 Effect of varying concentration of palladium(II)

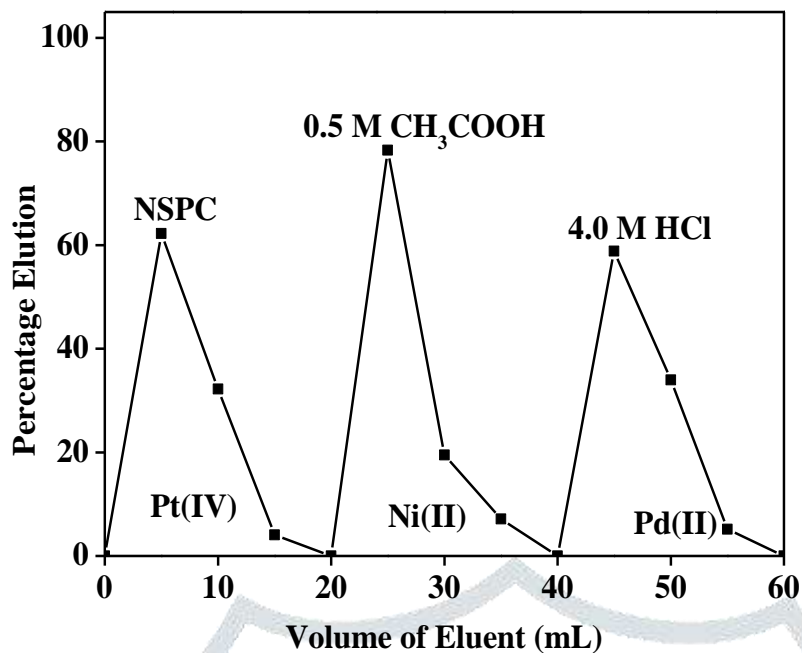


Figure. 3.5.1- Separation of Pt(IV), Ni(II), Pd(II)

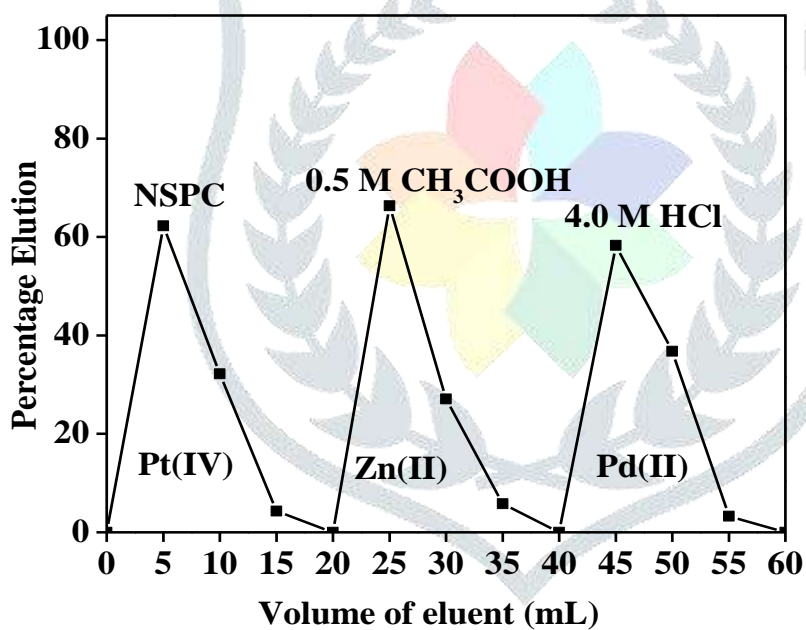


Figure. 3.5.2 Separation of Pt(IV), Zn(II), Pd(II)

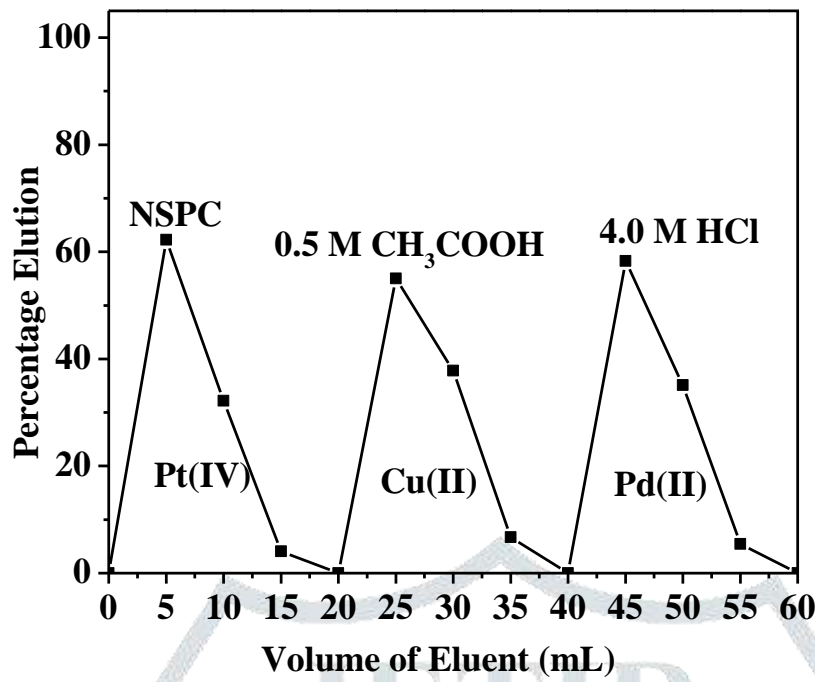


Figure. 3.5.3 Separation of Pt(IV), Cu(II), Pd(II)

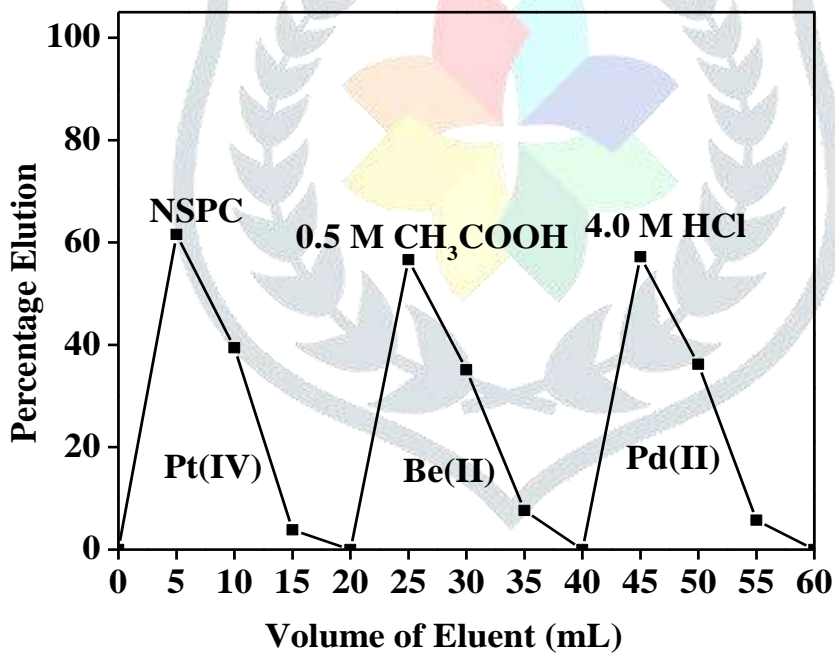


Figure3.5.4 Separation of Pt(IV), Be(II), Pd(II)

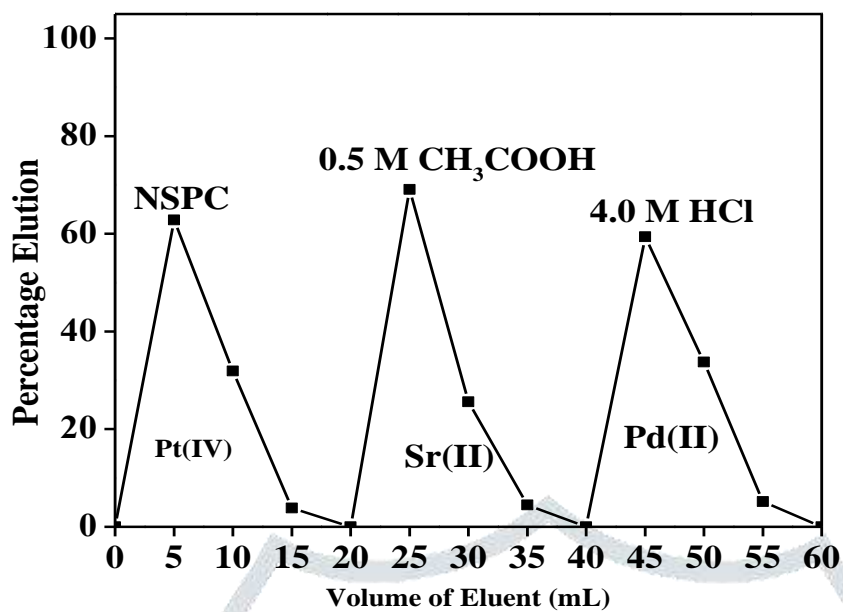


Figure 3.5.5 Separation of Pt(IV), Sr(II), Pd(II)

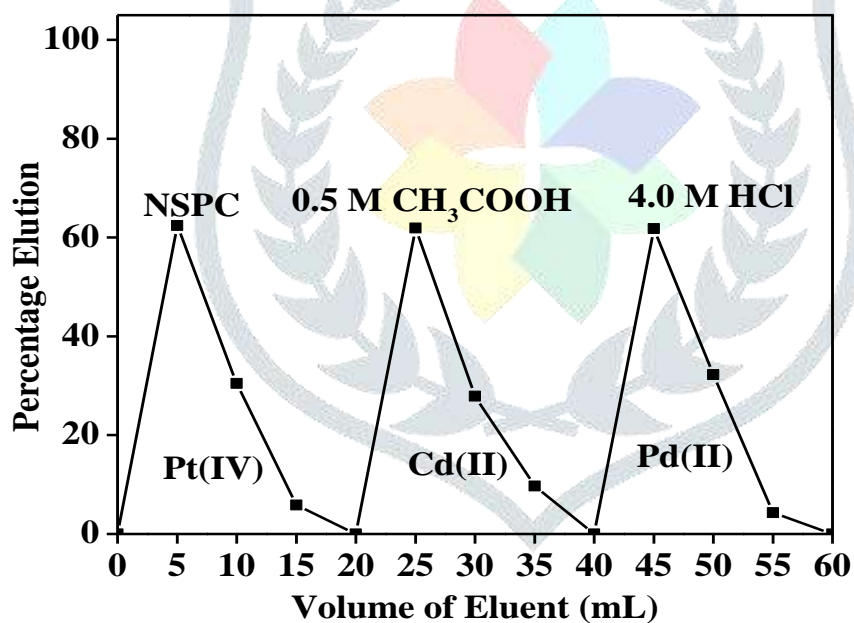


Figure. 3.5.6 Separation of Pt(IV), Cd(II), Pd(II)

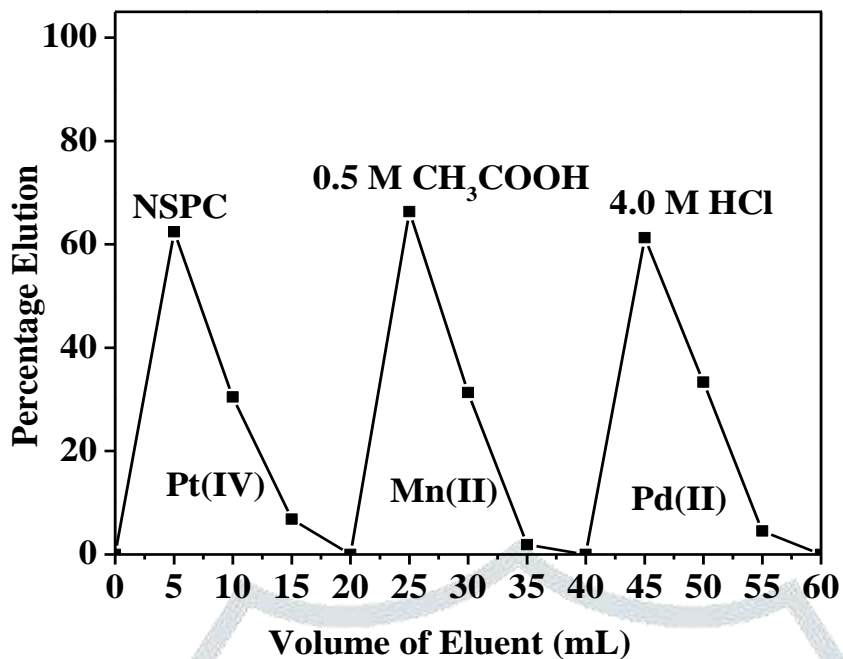


Figure 3.5.7 Separation of Pt(IV), Mn(II), Pd(II)

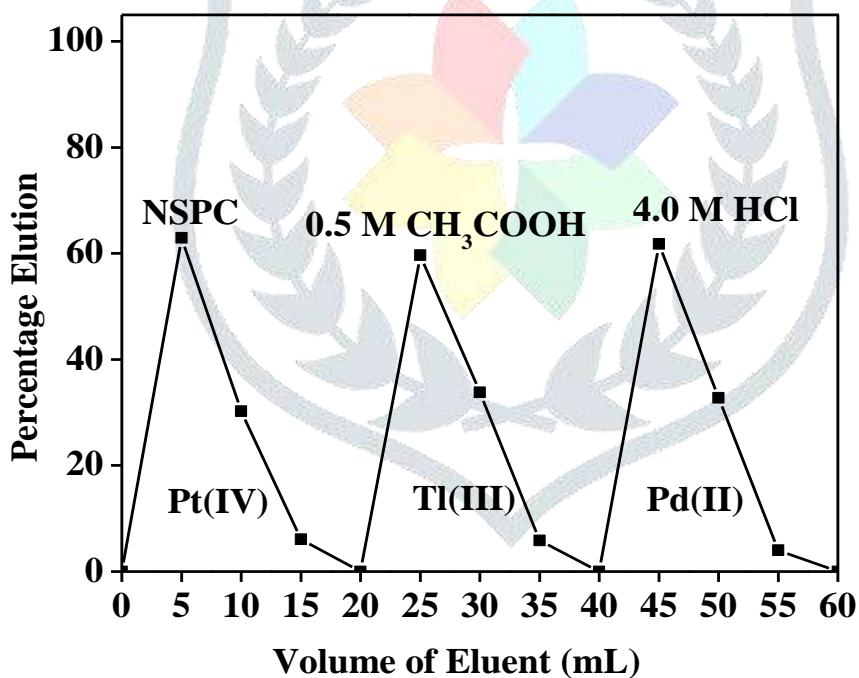


Figure. 3.5.8 Separation of Pt(IV), Tl(III), Pd(II)

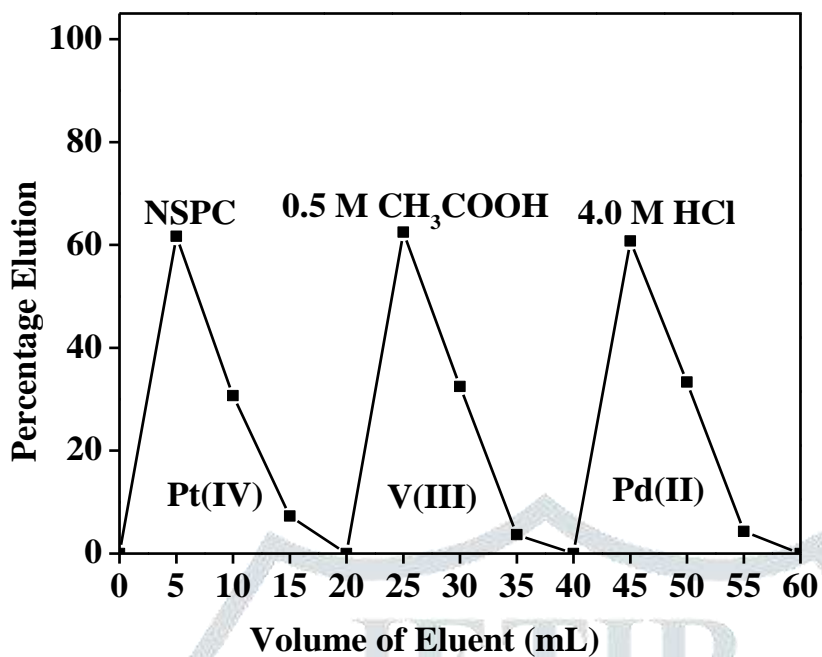


Figure 3.5.9 Separation of Pt(IV), V(III), Pd(II)

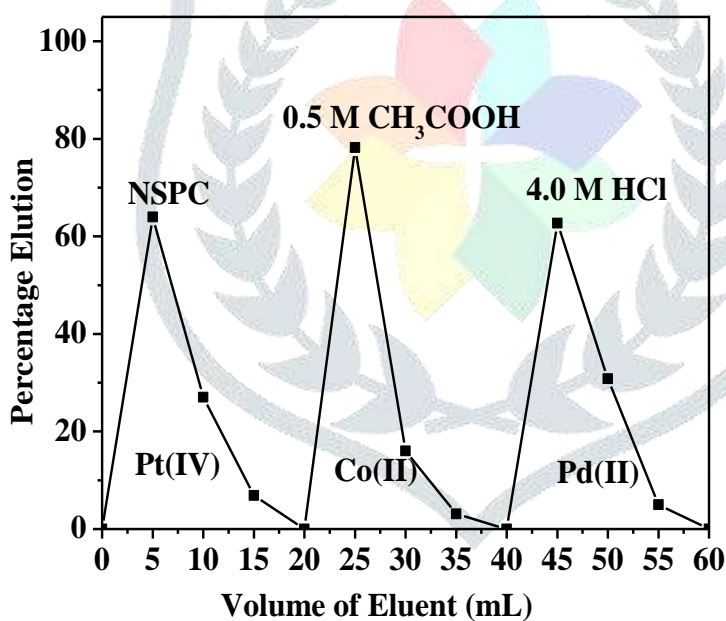


Figure. 3.5.10 Separation of Pt(IV), Co(II), Pd(II)