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Title Key: Extraction of Pd(II) and its ...

Extraction of Pd(II) and its spectrophotometric determination

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Abstract:

The extraction of Pd(II) metal from hydrochloric acid media using 4-(4-dimethylaminobenzylideneimino)-5-methyl-4*H*-1, 2, 4-triazole-3-thiol(DMABIMTT) in chloroform was studied. The optimum conditions have been determined by making a critical study of acid, metal ion and extractant concentration, period of equilibration, pH, various diluents and stripping agents. The effect of diverse ions on the quantitative extraction of Pd(II) has also been studied. The proposed method was further applied for the separation of Pd(II) from binary mixtures, synthetic mixtures, alloys and commercially available samples.Extraction of Pd(II) with DMABIMTTfrom hydrochloric acid media.

Introduction:

The recovery of precious metals is a challenging task for chemists.The name precious metals include the six elements of the platinum group: Ru, Rh, Pt, Pd, Os, and Ir, together with Au and Ag. In the past few decades precious metalshave found new applications outside the jewellery and decorative industries due to its excellent physical and chemical properties. Due to their wide applications and scarcity different separation techniques have been developed to separate and recover PGM's from primary and secondary sources.

Solvent extraction is one of the most efficient, and widely used separation method than other because ofspeed, ease/simple to operate, ability to combine with different detection techniques, selectivity towards specified metal[1-4].

Thus, the objective of the present article is to extract the trace amount of Pd(II) with a newly synthesized sulphur containing ligand. Efforts have been made to recover Pd(II) from real samples, synthetic mixtures and alloys. Moreover, the effect of acid concentration, reagent concentration, effect of time (shaking period), diluents, stripping agent and divers ions on Pd(II) extraction has also been studied.

1. Experimental

1.1 General Procedure:

An aqueous solution containing 100µg Pd(II) and enough hydrochloric acid

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and water were added to give final concentration of 1*M* with respect to hydrochloric acid in 25 ml. The resulting solution was transferred to 125 ml separating funnel. The aqueous phase was equilibrated once with 10 ml of 0.1M DMABIMTT solution in chloroform for 30 seconds. It was allowed to separate and the metal from the organic phase was back stripped with two 5 ml portions of 50% ammonia solution. The extract was evaporated to moist dryness. The residue was dissolved in minimum amount of dilute hydrochloric acid to form the solution. Pd(II) was estimated spectrophotometrically with pyrimidine -2-thiol [5].

2. Results and discussion:

2.1 Effect of acidity:

The extraction of Pd(II) was carried out from different acid media with 0.1M DMABIMTTin chloroform keeping the aq: org ratio 2.5:1. The extraction was quantitative from 1-8M hydrochloric acid. The extraction was found to be quantitative in very high concentration of nitric acid but was incomplete in sulphuric acid. Hence the use of 1 M hydrochloric acid is used for further studies.

2.2 Effect of concentration of extractant:

In order to optimize the conditions for extraction of Pd(II), chloroform solutions of extractant with varying concentration (0.01- 0.20 M) were employed. It was found that 10 ml of 0.05 M extractant is sufficient for quantitative extraction of 100µg Pd(II) from hydrochloric acid media, but in recommended procedure 0.1 M extractant in chloroform was used to ensure the complete extraction of metal ion. There was no adverse effect if one can use excess of extractant. However, a decrease in concentration of extractant resulted in lower distribution ratio, D, values for Pd(II)(Figure:1).

2.3 Effect of Equilibration time on Extraction:

The effect of time was observed on the system for a period of 5s to 30min (hand shaking) the extraction was found quantitative over the periods longer than 10 seconds. But to ensure the complete extraction of Pd(II) 1 min equilibration time was recommended. However, a prolonged shaking period doesn't have any adverse effect on the extraction.

2.4 Effect of Stripping Agents:

Pd(II) loaded in the organic phase was stripped with various stripping agents such as 1M HCl, 1M HNO_3, 1M NaOH, 50% NH_3, 1M Na_2CO_3. The stripping was observed only with NaOH, 50% NH_3, 1M Na_2CO_3. It was found that stripping was less than 50% with NaOH solution and 40% when Na_2CO_3 was used as stripping agents. It was quantitative when 50% NH_3 solution was used. Hence, 50% NH_3 (1:1 ammonia) solution is suitable strippent for palladium (Table 1).

2.5 Effect of diverse ions:

Pd(II) was extracted in the presence of different diverse ions (Table.2). The tolerance limit was set as the amount of foreign ions cause ±2% error in the recovery of palladium. The results showed that in the extraction and determination 100µg of the Pd(II), these ions did not interfered at the level tested. The reproducibility of palladium extraction investigated from six replicate measurements was 99.00 ± 0.95%.

3. Application:

3.1 Analysis of synthetic mixtures:

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The separation of Pd(II) from the platinum group metals and other metals is very difficult. A solution containing 100µg of Pd(II) was taken and known amount of other metals were added. Under the optimum extraction conditions of Pd(II), there is a quantitative extraction of Au(III), Pt(IV), Ru(III) and Rh(II). But the co-extracted metal ions cannot be back-stripped by 1:1 ammonia solution. Thus, the reagent (DMABIMTT) is made selective towards Pd(II) by taking an advantage of the stripant used. (Table.3)

3.2 Binary separation of Pd(II)from Fe(III), Co(II), Ni(II) and Cu(II)

The method allowed separation and determination ofPd(II) from a binary mixture containing either Fe(III), Co(II), Ni(II) and Cu(II). In a typical experiment, solution containing 100µg of Pd(II) was taken and known amounts of other metals were added. The separation of Pd(II) from Fe(III), Co(II), Ni(II) and Cu(II)was accomplished with 0.1M DMABIMTT in chloroform at 1M hydrochloric acid. Pd(II) was estimated spectrophotometrically with pyrimidine-2-thiol. The recovery of Pd(II) and that added ions was 99.5% and results are reported in (Table 4).

3.3 Analysis of alloys:

Toascertain the selectivity of the reagent, the proposed method was successfully used in the determination of Pd(II) from alloys. The synthetic mixture was prepared corresponding to the composition of alloy. The results of the analysis are reported in (Table 5). The average recovery of Pd(II) was 99.5%.

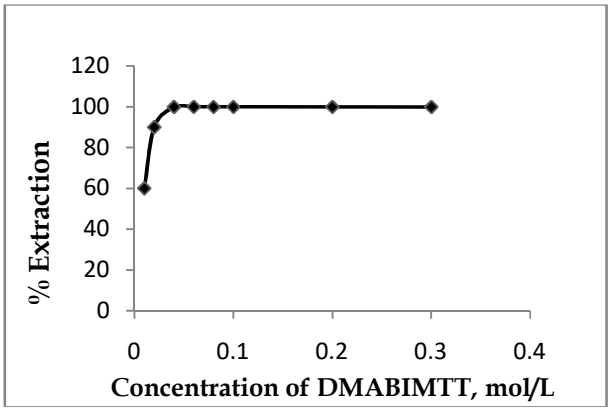

Fig.1 Extraction of Pd(II) as a function of DMABIMTTconcentration

Table.1Effect of different stripping agents

Stripping agent	Concentration	Extraction(%)
HCl	1M-6M(2×10 ml)	-
HNO_3	1M-6M(2×10 ml)	-
NH_3	1:1 (2×10 ml)	99.9
NaOH	1 $mol.dm^{-3}$ (2×5 ml)	50
Na_2CO_3	(2×10 ml)	40

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Table .2Effect of foreign ions on the extraction of Pd (II) with DMABIMTT

Foreign ions	Amount tolerated (mg)	Foreign ions	Amount tolerated (mg)
Mn(II)	10	Pb(II)	5
Fe(II)	15	Hg(II)	5
Fe(III)	20	Ni(II)	5
Bi(III)	20	Co(II)	15
Zn(II)	5	Sn(II)	5
Ca(II)	20	Be(II)	20
Mo(VI)	5	Ba(II)	20
Pt(II)	0.5	Cu(II	5
Au(III)	1	Pt(IV)	1
Sb(III)	5	Cd(II)	5
Fluorid	200	Sr(II)	10
EDTA	100	Oxalate	200
Iodide	100	Acetate	100
Mg(II)	20	Bromide	100

Table.3 Analysis of synthetic mixtures

Amount of metal ions (µg)	Palladium found (µg)	Recovery[a] (%)	R.S.D (%)
Pd(II)100+Au(III)500	99.2	99.7	0.04
Pd(II)100+Pt(IV)500	99.1	99.2	0.06
Pd(II)100+Rh(III)200	99.1	99.6	0.08
Pd(II)100+Pt(IV)200+Rh(III)200	98.7	99.7	0.07
Pd(II)100+Au(III)500+Rh(III)200	99.8	99.9	0.07
Pd(II)100+Pt(IV)200+Au(III)500	99.5	99.8	0.05
Pd(II)100+Pt(IV)200+Au(III)500+ Fe(III)2000+Co(III)2000	99.2	99.4	0.04
Pd(II)100+Pt(IV)500+Au(III)500+ Rh(III)200+Fe(III)200+Co(III)2000	98.8	99.6	0.08

a=Average of six determination

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Table.4 Binary separation of Pd(II)from Fe(III), Co(II), Ni(II) and Cu(II)

Composition of metal ions, (µg)	Recovery Pd(II) (%)	R.S.D (%)
Pd(II)100; Fe(III)15000	99.6	0.13
Pd(II)100; Co(II)10000	99.7	0.07
Pd(II)100; Ni(II)5000	99.6	0.11
Pd(II)100; Cu(II) 5000	99.8	0.13

Table.5 Analysis of alloys

Alloy	Composition	Palladium taken, (µg)	Palladium found[a](µg)	Recovery (%)	R.S.D (%)
Pd-Cu alloy	Pd,60; Cu,40	100	99.5	99.58	0.10
Pd-Ag alloy	Pd,60; Ag,40	100	99.6	99.9	0.06

a Average of six determinations

Conclusion:

The present work points out that the synthesized extractant shows a good potential for the extraction of Pd(II) from hydrocloric acid media. 4-(4-dimethylaminobenzylideneimino)-5-methyl-4*H*-1, 2, 4-triazole-3-thiol(DMABIMTT) is able to effectively extract Pd(II). The extraction time is short and the extractant presents a good loading capacity and reusable. The proposed method is used for rapid and selective separation of Pd(II) from associated ions in their binary mixtures, synthetic mixtures and alloys.

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