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– *Reviews* –

Liquid-liquid extraction of platinum from acidic solutions - A review

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Liquid-liquid extraction (LLE) of platinum is a most important topic in separation science. The platinum separation process is a challenging task to researchers**.** The present review article is the first discussion on LLE of platinum and the review pertains to the literature review on the LLE of platinum from acidic solutions over more than three decades.

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1. Review of literature on platinum extraction/classification of extractants

In this review paper we discus the LLE methods for platinum by the nature of the extractant systems: 1.1. Nitrogen based extractants, 1.2. Phosphorous based extractants, 1.3. Sulfur containing extractants, 1.4. Other extractants:

1.1. Nitrogen based extractants

The extraction of noble metals with *p*-octylaniline at varying normalities of different mineral acids has been studied and optimum conditions were established for their separation from the base metals commonly present in Pt-bearing materials [2]. Pd(II), Rh(III), Ir(III), Au(III), and Pt(IV) were extracted from HCl and HBr with 5% triisooctylamine solution in CCl_4 and methods were devised for separating Au from Pt and its determination and also for the simultaneous determination of Pd and Pt [3]. A review on the extraction of Pt metals with ethers, ketones, esters, organophosphorus compounds has been [9]**.** The fundamental aspects of the extraction and stripping of Pt(II) from its chloride solution by Aliquat 336 diluted with toluene were studied [10].2-Nonylpyridine-1-oxide (L) is a versatile LLE reagent when dissolved in $CHCl_3$. A range of separations is achieved by adjustment of the acid strength (HCl) and reagent concentration. Thus $[AuCl_4]^-$ may be separated from chloro anions of the Pt-group metals, and $[FeCl_4]^-$ may be co-extracted with $[AuCl_4]^-$ at high acid concentration (6 $mol.dm^{-3}$ HCl) [12]. Pt(II) was selectively extracted (84%) at pH 3 from an aqueous mixture of Pt(II), Cu(II), and Ni(II) by complexation with the macrocyclic dioxotetraamine in the presence of $Na_2S_2O_3$ [21]. The extraction of Pt, Pd, and Ir by TOA-kerosene was studied under different conditions and extraction of Au, Pd, and Pt by N530 was also studied [26]. Studies of the extraction equilibrium of Pt by $CHCl_3$ solutions of 7-substituted-8-hydroxyquinoline derivatives (HQ) from aqueous chloride media demonstrated that Pt(IV) is extracted as the ion-pair $PtCl_6^{2-}(H_2Q^+)_2$ [35]. 2-Ethylhexylaminomethylpyridine can extract Pd(II), Rh(III) and Pt(IV) with excellent selectivity over Cu(II), Ni(II), Co(II), Cd(II), Zn(II), and Ir(III) from aqueous hydrochloric acid solutions. It forms 1:1 complexes with palladium(II), rhodium(III) and platinum(IV) ions accompanied by chloride ions [39]. Extraction and back-extraction of Pt in aqueous HCl with tri-n-octylamine (TOA) toluene solutions were studied to clarify the complexes formed and the equilibrium constant NH_4Cl in toluene reacted with Pt in aq HCl solution to form an amine-metal complex (TOA-Pt) [40]. Solvent extraction of platinum(IV) and palladium(II) with tri-n-octylamine(TOA) in o-xylene from 4.0 mol dm^{-3} (Na, H)(Cl, SO_4) was investigated at 298 K for comparison with a similar extraction by trioctylphosphine oxide (TOPO) [44]. The selective separation by solvent extraction of Au(III), Pt(IV), Pd(II) and Rh(III) from hydrochloric acid with *N*-benzyl aniline (NBA) was investigated [47]. The extraction and stripping equilibria of platinum(IV) between acidic chloride media and tri-*n*-octylamine (TOA) in toluene were investigated at 303 K [48]. The extraction of Pt(IV) by Alamine 304 has been studied and the percentage of metal extraction determined as a function of different amine and metal concentrations and aqueous ionic strengths. Results indicated the formation of the species $(R_3NH^+)2PtCl_6^{2-}$ in the organic phase and that the extraction reaction is exothermic ($DH° = -1.9$ $kJ.mol^{-1}$) [50]. P-50 oxime dissolved in Escaid 100 extracted platinum(II), prepared in situ, from aqueous chloride solutions, but rates of extraction were low [52]. Bidentate ligands of *N*-*N*, 2-ethylhexylaminomethylpyridine (=EHAP) and N-S, t-dodecylthiomethylpyridine (=DTMP) were synthesized to investigate their extraction behaviors for precious metals [55]. *N*-*n*-octylaniline in xylene can be used for the extractive separation of platinum(IV) from acidic media [56]. Extraction equilibria of precious metals (Au(III), Pd(II) and Pt(IV)) with various ion exchange extractants were measured [59].

The distribution equilibrium of platinum(IV) species between aqueous ascorbate media and 0.1 mol.dm^{-3} of *N*-*n*-octylaniline in xylene was studied as a function of different parameters, such as the concentration of the extractant in the organic phase, weak organic acid concentration, different diluents and the effect of the shaking time on extraction [68]. The selective extraction of Pd(II) over Au(III) and Pt(IV) as well as subsequently selective extraction of Au(III) over Pt(IV) from hydrochloric acid (HCl) solution was achieved by using 2-hydroxy-4-sec-octanoyl diphenyl-ketoxime diluted in kerosene as extractant [72]. The extraction behaviour of Pd(II) and Pt(IV) was studied with LIX84 I (2-hydroxy-5-nonylacetophenone oxime) in HCl medium [74].

1.2. Phosphorous based extractants

The Pt group metals were extracted with Ph_3P in $(ClH_2C)_2$ from HCl medium [1]. The separation of compounds of Rh, Pd, Ir and Pt by multistage batch LLE with TBP from HCl solutions was studied [6]. The effect of $SnCl_2$ on the extraction of tetrachloroplatinate(II) in 1.0-1.5 M HCl into a dichloromethane solution of triphenylphosphine (TPP) has been described [16]. A synergistic effect was observed for the extraction of Pd when a solution containing trace amounts of Pt(IV) and Pd(II) was extracted with chloroform containing 8-hydroxyquinoline (HQ) and tributylphosphite (TBPI) [22]. The extraction of Pt(II) and/or Pd(II) with bis(2,4,4-trimethylpentyl)phosphinodithioic acid from chloride solution was studied. Extraction into heptane is more rapid than that for chloroform [23]. In order to compare the extraction of Pt(IV) and Pd(II) complex acids with trioctylphosphine oxide (TOPO) to those of other inorganic acids, the distribution ratio of these metals between o-xylene containing TOPO and 4.0 mol dm^{-3} (Na, H) (HCl, H_2SO_4) was measured at 298 K [25]. The equilibrium distribution of Pt(IV) between HCl and trioctylphosphine oxide in toluene at 303 K was examined from the concentration dependencies of the distribution ratio and an extraction mechanism was postulated [29]. Distribution equilibria were investigated in the LLE of Pd(II) and Pt(IV) from chloride media with *N*,*N*-dioctylglycine in toluene at 303 K [34]. LLE of Pt(IV) and Rh(III) with TOPO in toluene from HCl was investigated to elucidate the extraction reaction at 303 K [31]. Using a ^{191}Pt radiotracer, the LLE behavior of Pt(IV) from HCl medium with rubeanic acid in tributylphosphate (TBP), with TBP and with thenoyltrifluoroacetone (TTA) in n-butyl alcohol with acetophenone was examined [34]. Di-alkylphosphonic acid containing a nitrogen atom, di(2-ethylhexyl)aminophosphonic acid (=HNR), was synthesized in order to investigate its extraction properties in toluene solutions for palladium(II) and platinum(IV) from acidic chloride media [36].

1.3. Sulfur containing extractants

The extraction of Pt from aq HX (X = Cl, Br, I) by di-n-octyl sulfide (L) in cyclohexane was examined [7]. Diphenylthiourea was used in conjunction with KI or $SnCl_2$ in a systematic scheme for the extraction/separation of Pt, Pd, Rh, Ir, and Au in HCl solutions [8]. A method was developed for the rapid and selective extraction of Pt(II) with 2-mercaptobenzothiazole (2-HMBT) into chloroform from 1M HCl in the presence of $SnCl_2$ [11]. The Pt-group metals can be extracted selectively by adjusting the pH. The extraction of milligram quantities of Pt by 2-mercaptobenzothiazole in a number of solvents was studied [15]. The separation of Au, Pt, Pd, Ru, Os, Rh, and Ir in chloride solutions by the use of heterocyclic *N*-oxides is discussed [17]. Benzoylthioureas are excellent reagents for LLE and have a high selectivity for Pt-group metals [24]. A novel sulfur-containing solvent extraction reagent, 3,3-diethylthietane (DETE) was synthesized and the mechanism of platinum(IV) extraction from aqueous hydrochloric acid solutions

was investigated at 30°C [42]. 3,3-Diethylthietane was synthesized to investigate its extraction behavior for Pt(IV) from acidic aqueous chloride media [43]. The LLE of platinum(II) with 12-, 14- and 16-membered cyclic tetrathioethers from chloride solutions was studied [57]. The LLE of Pt, Pd, Ru, Rh, and Ir with pure synthesized *N*,*N*-diethyl-*N*'-benzoylthiourea (DEBT) was carried out by optimizing the concentration of acid, mole ratio of metal to chelating agent, temperature and extraction time [58]. The LLE of palladium(II), platinum(II), platinum(IV), rhodium(III), iridium(III) and iridium(IV) from hydrochloric acid solutions by some dialkyl sulfoxides of the types R_2SO, RR'SO and R'_2SO, where R = alkyl and R' = cycloalkyl, was investigated [63]. Extraction of palladium(II) and platinum(IV) from acidic chloride solutions with 3,7-dimethyl-5-thianonane-2,8-dione in toluene and chloroform and the complexation of this reagent with platinum metals in aqueous acetone were studied by ^{1}H and ^{13}C NMR and IR spectroscopy [65]. The extraction of Pt and Pd from chloride solutions by binary extractants, salts of tetraoctyl-hexylenediamine with organic acids (di(2-ethylhexyl)phosphoric and di(2-ethylhexyl)dithio-phosphoric acids), has been studied [66]. An investigation into the nature of the complex species formed by the extraction of Pt(II) and Pt (IV) from HCl by using *N*-benzoyl-*N*',*N*'-diethylthiourea and *N*-benzoyl-*N*',*N*'-di(n-butyl)thiourea in chloroform and toluene has been carried out [67]. The extractant nonylthiourea (NTH) in chloroform has been investigated for the extraction of Pt(IV) from chloride solutions at an ionic strength of I = 4.0 $mol.dm^{-3}$ and at 22 °C [70]. The extraction of gold(III), palladium(II) and platinum(IV) from acidic media with the cyclic sulfoxide derivative of dodecyl-tetrahydrothiophene 1-oxide (dtmso) was investigated [73].

1.4. Other extractants

Various other extractants such as Me_2CHCH_2COMe [4], *N*,*N*,-di-n-hexyl-N'-benzoylthiourea [18], *N*,*N*-bis(2-ethylhexyl)glycine [19], Isoamyl alcohol - methyl isobutyl ketone mixture [20], *N*,*N*-Dioctylglycine [27], *N*,*N*-Dioctylsuccinamic acid [28], *N*,*N*-Dioctylglycine/*N*,*N*-dioctylsuccinamic acids [38], 5,11,17,23-tetra-tert-butyl-25,26,27,28-tetra-(2-hydroxyethoxy)calix[4]ar ene [41], *p*-Tert-butylcalix[4]arenes [46], hostarex [54], dioxodialkyl tetraaza crowns [61] and DC18C6 [64] were used for platinum extraction from different mineral acids.

The literature review reveals that a limited number of extractants are used for LLE of platinum from acidic solutions (Table 1).

Table 1 Summary of platinum liquid-liquid extraction systems

Name of the extractant	Other metals	Aqueous medium and range	Diluent	Ref.
Triphenylphosphine	Pd, Pt, Os	HCl (>6 $mol.dm^{-3}$)	$(ClH_2C)_2$	1
n-Octylaniline	Ir, Ru, Pd, Rh, Au	Mineral acids	--	2
Tri-iso-octylamine	Pd, Rh, Ir, Au	HCl and HBr	CCl_4	3
Me_2CHCH_2COMe	Pd, Au	--	--	4
Trioctylphosphine oxide (TOPO)	Pd, Au	HCl and HBr	MIBK and 2,2'-	5

			dichlorodiethyl ether	
TBP	Rh, Pd, Ir	HCl	--	6
di-*n*-Octyl sulfide (DOS)	Au, Pd	HBr	--	7
Diphenylthiourea	Pd, Rh, Ir, Au	HCl	KI or $SnCl_2$	8
Aliquat 336	--	HCl ($0.1\ mol.dm^{-3}$)	Toluene	10
2-Mercaptobenzothiazole	--	HCl ($1.0\ mol.dm^{-3}$)	Chloroform	11
2-Nonylpyridine 1-oxide (L)	Ir, Rh, Au	HCl ($6\ mol.dm^{-3}$ HCl)	$CHCl_3$	12
R1R2NCSNHR3 where R1 and R2 = Me, Bu, or hexyl group and R3 = Bz, COOMe, or COOEt.	Pd, Rh, Ir, Ru, Os, Cu, Fe, Ni, Zn, Co, Mn	HCl	Xylene	13
4-Methyl-2-pentanone	Co, Ni, Fe, Cu	HCl	--	14
2-Mercaptobenzothiazole	--	HCl	$CHCl_3$	15
Triphenylphosphine	--	HCl ($1.0\text{-}1.5\ mol.dm^{-3}$)	Dichloromethane	16
1,3,4-Thiadiazole-2-nonylmercapto-5-thiol	Au, Pd, Rh	HCl	--	17
N,*N*,-di-n-hexyl-N'-benzoylthiourea	Ru, Rh, Ir	$SnCl_2$	Toluene	18
N,*N*-bis(2-ethylhexyl)glycine	Pd	HCl	Toluene	19
Isoamyl alcohol - methyl isobutyl ketone mixture	Al, Ca, Mg, Mn, Ni, Cr	HCl	--	20
Macrocyclic dioxotetraamine	Cu, Ni	pH 3	--	21
8-Hydroxyquinoline and tributylphosphite	Pd	--	Chloroform	22
bis(2,4,4-Trimethylpentyl)phosphinodithioic acid	Pd	HCl	Chloroform	23
N-Mono- and *N*,*N*-disubstituted benzoylthioureas	Pt-group metals	--	--	24
Trioctylphosphine oxide	Pd	HCl	*o*-Xylene	25
2-Hydroxy-4-sec-octyloxylbenzophenoxime	Au, Pd	--	--	26
N,*N*-Dioctylglycine	Pd	HCl	Toluene	27
N,*N*-Dioctylsuccinamic acid	Pd, Hg, Cu, Ni, Co, Zn, Fe, Al, Ga	HCl	Toluene	28
Trioctylphosphine oxide	--	HCl	Toluene	29

N,*N*-Dioctylglycine	Pd	HCl	Toluene	30
Trioctylphosphine oxide	Rh	HCl	Toluene	31
S-containing methylphosphonates	Au, Pd	HCl	--	32
Tris(2,6-dimethoxyphenyl)phosphine	Au, Pd, Fe, Cd, Mo	HCl	$CHCl_3$ or 1,2-dichloroethane	33
Tri-Bu phosphate (TBP) and thenoyltrifluoroacetone (TTA)	--	HCl (3.0 or 4.0 $mol.dm^{-3}$)	*n*-Bu alc-acetophenone	34
7-Substituted-8-hydroxyquinoline derivatives	--	HCl	$CHCl_3$	35
Di(2-ethylhexyl)aminophosphonic acid	Pd	HCl	Toluene	36
Diamine and monoamine extractants	Pd	HCl	Isodecanol-benzene	37
Amino- and amido-carboxylic acids	Pd	HCl	Toluene	38
2-Ethylhexylaminomethylpyridine	Pd, Rh, Cu, Ni, Co, Cd, Zn, Ir	HCl	--	39
Tri-n-octylamine	--	HCl	Toluene	40
N,*N*-Dimethyldithiocarbamoylethoxy substituted calix[4]arene	Pd	HCl	--	41
3,3-Diethylthietane	--	HCl	Toluene	42
3,3-Diethylthietane	Pd	HCl	--	43
Tri-n-octylamine and trioctylphosphine oxide	Pd	HCl, H_2SO_4 (4 $mol.dm^{-3}$)	*o*-Xylene	44
p-(1,1,3,3-Tetramethylbutyl)-phenylhydrogen[*N*,*N*-di(2-ethylhexyl)amino methyl]phosphonate	Pd	HCl	--	45
p-Tert-butylcalix[4]arenes	Pd, Au	HCl	--	46
N-Benzyl aniline	Au, Pd, Rh	HCl (1.0 – 4.0 $mol.dm^{-3}$)	--	47
Tri-n-octylamine (TOA)	--	HCl	Toluene	48
Bis(2-ethylhexyl) hydrogen phosphate	Pd, Au, Os	HCl	Toluene	49
Amine alamine 304	--	HCl	Xylene	50
P-(1,1,3,3-Tetramethylbutyl)phenyl *H N*,*N*-di(2-ethylhexyl)amino-methylphosphonate	Pd, Fe	HCl	Toluene	51
P-50 oxime	--	HCl	Escaid 100	52
2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester (PC-88A)	Pd	HCl (4.0 $mol.dm^{-3}$)	Toluene	53
Hostarex	Pd, Rh, Fe,	HCl	Decanol and	54

	Ni	(6.0 mol.dm^{-3})	Exxsol	
N-*N*, 2-ethylhexylaminomethylpyridine and *N*-*S*, t-dodecylthiomethylpyridine	Pd, Rh, Cu, Ni, Co, Zn, Cd, Fe	HCl	--	55
N-*n*-octylaniline	Fe, Co, Ni, Cu	HCl (0.5 to 10 mol.dm^{-3}) and H_2SO_4 (2.5 to 10 mol.dm^{-3})	Xylene	56
Cyclic tetrathioethers	--	HCl	1,2-Dichloroethane	57
N,*N*-diethyl-*N*'-benzoylthiourea	Pd, Ru, Rh, Ir	HCl (1.0 to 2.0 mol.dm^{-3})	Toluene	58
Tetraamyl ammonium chloride	Au, Pd	HCl	--	59
Cyanex 921 and Cyanex 925	--	HCl	Toluene	60
Dioxodialkyl tetraaza crowns	--	HCl	--	61
Cyanex 921	Rh, Pd	HCl (6.0 mol.dm^{-3})	--	62
Dialkyl sulfoxides	Pd, Rh, Ir	HCl (1.0 to 6.0 mol.dm^{-3})	--	63
Dicyclohexyl-18-crown-6 (DC18C6)	--	HCl (0.1 mol.dm^{-3})	Chloroform	64
3,7-Dimethyl-5-thianonane-2,8-dione	Pd, Cu, Ni, Co, Mn, Fe	HCl	Toluene and chloroform	65
Tetraoctyl-hexylenediamine with organic acids (di(2-ethylhexyl)phosphoric and di(2-ethylhexyl)dithio-phosphoric acids)	Pd	HCl	--	66
N-Acyl(aroyl)-*N*', *N*'-dialkylthioureas	Pd	HCl	Toluene and chloroform	67
N-n-octylaniline	Pd, Au, Rh, Ir, Os, Ru, Fe, Co, Ni, Cu	HCl	Xylene	68
Cyanex 923		HCl (3.0 to 8.0 mol.dm^{-3})	Toluene	69
Nonylthiourea (NTH)	--	HCl	--	70
Bis(2-ethylhexyl) *N*-butyl-*N*-octylaminomethylphosphonate	Au, Pd, Fe, Cu, Ni, Co	HCl	Chloroform and xylene	71
2-Hydroxy-4-sec-octanoyl diphenyl-ketoxime	Pd, Au	HCl	Kerosene	72
Cyclic sulfoxide derivative	Pd, Au	HCl	--	73
2-Hydroxy-5-nonylacetophenone	Pd, Fe, Al,	HCl	Dodecane	74

oxime (LIX 84 I)	Zn, Cu, Ni	(0.1 mol.dm^{-3})		
Bis(2,4,4-trimethylpentyl) monothiophosphinic acid (Cyanex 302)	Pd, Fe, Cu, Ag, Al, Mg, Ca	HCl, H_2SO_4 and HNO_3 (0.01 to 5.0 mol.dm^{-3})	Kerosene	75

2. Conclusions

The present review summarizes the liquid-liquid extraction (LLE) of platinum from acidic solutions using various organic complexing extractants and more than three decades of published papers on the LLE methods for the separation and extraction studies of platinum from various mineral acids is discussed.

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