THE DEVELOPMENT OF POLAROGRAPHIC ANALYSIS OF INORGANIC SUBSTANCES (REVIEW)

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I. V. Pyatnitskii

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In recent years, many reviews reflecting the development of various trends in polarographic analysis have been published. We shall cite reviews on polarographic analysis of organic compounds [1-3] and the study of polymers [4], on the use of polarographic analysis for automatic control of industrial processes [5], the use of solid electrodes for the study of fused systems [6], and on vector polarographic analysis [7] and pulse polarographic analysis [8, 9]. There are also several reviews of a general nature [10-15]. Works in the area of determining trace amounts of elements by anode polarographic analysis with preconcentration on a stationary mercury drop are summarized in [16].

The theory and practice of oscillographic [17-35] and square-wave [36-45] polarographic analysis are illuminated in a number of works. Determinations by a-c polarographs have gained wide use [46-53]. A theory of polarographic currents in a moving electrolyte [54], and a theory and technology of differential polarographic analysis [55-56] have been described. The characteristics of polarograph circuits have been given in [57], and more refined operating methods have been given in [58]. The designs of cells for micropolarographic analysis have been described in [59-61]. Theoretical problems of amalgam polarographic analysis are examined in [62-64].

In the present review, which has been compiled according to works from the last two years, the principal attention is given to the chemical side of the question, and particularly to the effect of complexing base electrolytes on the polarographic properties of the ions to be determined. The characteristics of the conditions for determining individual elements by polarographic analysis using various base electrolytes are given in the table.

The Relationship Between the State of Ions in a Solution and Their Polarographic Characteristics

An important problem is that of establishing a relationship between the state of ions in a solution, the composition and stability of complex compounds, on the one hand, and the values of the half-wave potentials and their distribution in the polarographic spectrum, on the other.

It is of interest to investigate polarographic reduction of ions of pentavalent vanadium under various pH conditions of the solution [119]. The change in the form of polarograms with acidity is explained by the presence of various forms of vanadium ions in the solution. For example, at Ph= 3, one wave is due to the reduction of isopolyvanadates, and the other is due to reduction of the uncondensed form of vanadium ions. At pH= 5.5, the compound $H_XV_{10}O_{28}^{6-X}$ is reduced on the mercury-drop electrode, and at higher pH, metavanadates, etc.

In [123-126] the nature of the polarographic waves of selenium and tellurium is investigated, and it is shown that one wave is due to reduction of the selenite ion to elemental selenium, and that the second is due to six-electron reduction with the formation of hydrogen selenide. A relationship between the half-wave potential of a cupric ion in a solution of diethylenetriaminetetraacetic acid with a complexing composition and the change in the nature of electrochemical reduction under the influence of the pH of the solution is shown in [68]. In the pH range from 7 to 10, the electrode process corresponds to the equation

$$CuA^{3-} + H^+ + 2e = Cu_{(Hg)} + HA^{4-},$$

and the half-wave potential of the copper can be calculated from the stability constant of the complex and the pH

of the solution. In [154] it is shown that of all of the possible complexes of bivalent cobalt with ethylenediamine, only the complex of composition CoA_3 is polarographically active.

Analogous data on the relationship between the composition of complex compounds and their polarographic characteristics are contained in the following works on: complexes of tin with pirogallic acid [103]; lactate, sulfosalicylate, and glycolate complexes of uranium [136-139]; complexes of boric acid with fructose [164]; complexes of iron with nitrilotriacetic acid [152]; cyanide complexes of mono-, bi-, and trivalent manganese [148]; and complexes of niobium with ethylenediaminetetraacetic acid sodium salt [120].

Indirect Methods of Determining Polarographically Inactive Elements

Ions of aluminum, berillium, zirconium, thorium, and certain other elements are not reduced on a mercury-drop cathode, or else they give waves that have little suitability for polarographic determination. Indirect methods are suggested for such ions.

One group of methods is based on displacement of polarographically active ions from their complex compounds or weakly soluble precipitates by polarographically inactive ions. An indirect method of determination, based on measuring the limiting current of cadmium that has been displaced from a cadmium complexonate by zirconium ions, is given in [165]. In order to determine calcium in the presence of magnesium, the height of the waves of cadmium ions displaced from the cadmium complexonate by calcium ions in an ammoniacal medium is measured [79]. Under these conditions, calcium does not react with the cadmium complexonate. A similar method is described in [91] for determining gallium; gallium displaces cadmium ions from a complex compound of the latter with diaminocyclohexanetetraacetic acid $C_6H_8[N(CH_2COOH)_2]_2$ (SDTA):

$$2Ga^{3+} + CdJ^{2-} + CdHJ^{-} = GaJ^{-} + GaHJ + 2Cd^{2+},$$

where J^{4-} is a quadruply-charged anion of SDTA.

The determination can also be performed in the presence of aluminum by masking it with a fluoride.

Indium gives a more stable complex with SDTA than does gallium. The sum of gallium and indium is determined, and then in another batch of solution the reaction of gallium with Cd-SDTA is masked by oxalic acid and indium is determined. The amount of gallium is calculated according to the difference. Here we see an example of the use the instability constants of complex compounds for predicting the polarographic behavior of individual ions in a solution and for developing a method for determining them. The example described is used in [84] for separate determination of ions with similar half-wave potentials. In order to determine cadmium in metallic indium, the wave of cadmium ions displaced from its complex with 1,2-diaminocyclohexanetetraacetic acid by thorium ions was measured. Under these conditions, the indium remained bound to the complex and did not hinder the determination of cadmium.

To determine the amount of rare-earth elements and cerium, it was proposed in [163] to treat a precipitate of lead oxalate with a solution of salts of these metals and to polarographically analyze the lead ions that have passed into solution; their concentration is equivalent to the concentration of the amount of rare-earth elements in the solution.

The second group of indirect methods is based on the interaction of polarographically inactive ions with various dyes. Complexes of zirconium and thorium with Solochrome violet dye at pH= 2 to 3 give sharp waves in the region of 0.3 to 0.5 V. These waves are situated after the wave for reduction of the free dye [166]. A thorium wave is not formed in the presence of acetate ions. On the basis of this a method has been developed for determining $3 \cdot 10^{-5}$-M zirconium and thorium in joint presence; thorium is determined according to the difference. Other dyes, for example, mordant blue 2R, have been proposed for determining zirconium. The height of the second wave of the zirconium complex with the dye is proportional to the zirconium content. This same method has been used to determine aluminum in the presence of thorium (the latter masked by an acetate) with Superchrome Garnet Y in [33], gallium with Epichrome violet in [90], aluminum with Acid-chrome blue K in [87], and aluminum with Solochrome violet in [167-169]. In all cases when a polarographically inactive ion is added to the dye solution, the wave of the latter is split in two. The second wave, which is located at a more negative potential, serves for quantitative determination.

Polarographic Determination and Study of Elements (Abbreviations: ap) amalgam polarographic analysis with preaccumulation on mercury drop; cl) classical; dr) derivative; sw) square-wave; ip) indirect polarography; op) oscillograph polarographic analysis; ac) alternating-current polarographic analysis; PI) ions preventing determination; AI) accompanying ions in whose presence determination is made; S) sensitivity; CM) concentration method; MI) method of isolating interfering ions.

Element	Base electrolyte	Half-wave potential and type of polarographic analysis	Characteristics of method	Object of determination	Source
Lithium	Tetramethyl ammonium iodide, tetraethyl ammonium bromide	−2.3 cl	Aqueous and aqueous-standard solutions	–	65
Sodium Potassium	Lithium hydroxide, potassium chloride	dr	Simultaneous determination	Water, soil	66
Sodium	Choline	cl	–	–	67
Copper	Diethylenetriaminepentaacetic acid	−0.22 cl	Study of complexing	–	68
	Mannitol	ac	Study of complexing	–	69
	Phosphoric acid	−0.14 cl	Study of complexing, pH= 2.6 to 10.0	–	70
	Phosphoric and sulfuric acids	sw	PI: Molybdenum, bismuth	Indium	44
	Sodium arsenate	0.03 ap	S: $8 \cdot 10^{-6}$%	Arsenic	71
	Tartrate	−0.55 cl	AI: lead	Tellurium	72
		ac	S: 10^{-4}%	Products of Nonferrous metallurgy	53
	Phosphoric acid	−0.35 sw	S: 5γ. MI: uranium−ion exchange	Uranium	41
	Oxalic acid	0.25 op	MI: selenium−distillation from HBr solution	Selenium	19
	Hydroxylamine	−0.05 op	PI: titanium, antimony, arsenic	Ferromanganese	20
		sw	AI: lead, molybdenum, tungsten, zinc	Zirconium, hafnium	38
	Sodium hydroxide	ap	MI: indium−extraction	Indium	73
	Ethylenediaminetetraacetic acid sodium salt, pH= 11	−0.46 ip	AI: arsenic	Paris green	74
		ap	S: 10^{-5}% AI: Pb, Cd, Zn	Aluminum, zirconium	32
Silver	Ethelenediaminetetraacetic acid sodium salt	ip	Displacement reaction	Alloys	75
	Potassium cyanide	−0.5 ip	Surplus is bonded	–	76
		ap	S: $5 \cdot 10^{-6}$% AI: Fe, Hg, Cu	Lead salts	77
Gold	Hydrochloric, sulfuric, and phosphoric acids	0.50−0.88 ip	Platinum electrode	–	78
Potassium	Ethylene glycol-bis-aminoethyl ether tetraacetic acid	ip	$CdJ + Ca^{2} + 4NH_3 = Cd(NH_3)_4^{2+} + CaJ$	–	79
Barium		−1.65 op	S: $1.2 \cdot 10^{-5}$ g/ml	Potassium iodide	31
Zinc	Sodium gluconate	−1.50 ip	In alkaline medium	–	80
	Nitric acid	ip	CM: extraction of zinc ditysonate	Blood	81
	Citrate, oxalate	−1.00 ap	AI: Cd, Pb, Cu	Sea water	82
		sw	AI: Cu, Pb, Mo, W	Zirconium	38
	Sodium hydroxide	ap	AI: Cd, Pb, Cu MI: indium−extraction of $InBr_3$, S: 10^{-6}%	Indium	73
		ap	AI: Cu, Pb, Cd; S: 10^{-5}%	Aluminum, zirconium	32
	Phosphoric acid	−1.36 sw	S: 5γ: MI: uranium−ion exchange	Uranium	41
	Chlorides, pH= 3	−1.00 ap	S: $6 \cdot 10^{-5}$%; AI: Ga, Cd	Aluminum	83
	Perchloric and phosphoric acids	ac	S: 10^{-4}%	Products of nonferrous metallurgy	53
Cadmium	Potassium chloride	sw	AI: Pb; S: 0.01γ MI: uranium−ion exchange	Uranium	39
	Diaminocyclohexanetetraacetate	ip	$CdJ + Th^{4+} \rightarrow ThJ + Cd^{2+}$ AI: In	Indium	84
	Bis(2-hydroxybutyl)-2-hydroxyethylamine	−1.26 ip	AI: Tl, Pb. Without isolation of indium	"	85
	Oxalic acid	0.75 op	MI: selenium−distillation	Selenium	19
	Citrate, oxalate	−0.70 ap	AI: Zn, Pb, Cd	Sea water	82
	Sodium hydroxide	ap	AI: Zn, Pb, Cu; MI: extraction of $InBr_3$;	Indium	73

(continued)

Element	Base electrolyte	Half-wave potential and type of polarographic analysis	Characteristics of method	Object of determination	Source
			S: 10^{-6}%		
	Phosphoric acid	−0.94 sw	S: 5γ; MI: uranium−ion exchange	Uranium	41
	Chlorides, pH= 2 to 3	ap	S: $2 \cdot 10^{-6}$%; AI: Ga, Zn	Aluminum	83
	Perchloric and phosphoric acids	ac	S: 10^{-4}%	Products of nonferrous metallurgy	53
Aluminum	Tetrabutyl ammonium chloride	ip	pH= 3.7	Zinc alloys	86
	Sulfotrihydroxyazobenzol	−0.62 op	Thorium masked by acetate. PI: Fe, Ni, Pb, V	Thorium compounds	33
	Acid chrome blue	dr		Siloxanes	87
Gallium	Silicylates	−0.75 ap	AI: Bi, In, Cd, Pb; PI: Cu	−	88
	Pyrocatechol	−1.10 ip	AI: Fe, Al, Co PI: Zn, Ni, Cu, Pb, Cd, Tl, In	−	89
	Eriochromoviolet	ip	MI: from Al−extraction of gallium chloride	−	90
	Diaminocyclohexanetetraacetate	ip	$CdJ + Ga^{3+} \rightarrow GaJ + Cd^{2+}$		91
	Salicylate, pH= 3	−0.9 ap	S: 10^{-5}%; AI: Cd, Zn	Aluminum	83
Indium	Iodides, chlorides	−0.65 ac	S: 10^{-2}%	Cd, Pb, Sn	92−93
	Tartrate, pH= 2	−0.75 ip	AI: Zn; MI: $Pb \rightarrow PbSO_4$	Lead, zinc	94
	Ammonium thiocyanate	ip	Constants determined	−	95
	Citrate, pH= 1 to 6	ip	Mechanism of electrode reaction studied	−	96−97
	Hydrochloric acid	−0.64 ac	AI: Sb, Te, S: 10^{-6} moles/liter	Alloys	47
	Pyrogallol and thiocyanate	ap	AI: Sn, S: 10γ	Tin	98
Thallium	Tripolyphosphate and camphor	−0.5 ip	Masking of Pb, Cu, Fe, Bi by camphor	−	99
	Sodium formate	−0.46 ip −0.55 ip	Study of reduction conditions	−	100
	Diethylenetriaminepentaacetate and Triton	−0.46 op	AI: Mn, Co, Ni, Zn, Mo, V, As Triton masks Pb, Bi, Cd, Cr	−	101
	Sulfuric and phosphoric acids	−0.60 ac	AI: Cu, Cd	−	48
			AI: Fe, Cu, Cd, Pb	Selenium	19
	Bis-(2-Hydroxybutyl)-2-hydroxyethylamine	ip	AI: Cd, Pb, In	Indium	85
Germanium	o-Diphenols	−0.6 −0.9 ip	Study of reduction conditions	−	102
Tin	Pyrogallol	0.22 0.40 ip	Study of reduction conditions	−	103
		ap	"	Hydrochloric acid	104
	Oxalate, pH= 1	−0.51 ip	AI: As, Sb	−	105
Tin	Citrate	ip	Indirect determination	Steel	106
	Hydrochloric acid	ap	S: $3 \cdot 10^{-5}$%; AI: Pb	Aluminum	107
Lead	Phosphoric acid	ap	S: 1γ Pb	−	108
	Hydrochloric acid	ip	Improvement of procedure	Steel	109
		ip	CM: ion exchange	Steel, alloys	110
	" "	ip	CM: extraction of Pb ditysonate	Antimony, concentrates	111
	Sodium hydroxide	−0.76 ip	AI: Cu, Te	Tellurium	72
	Hydroxylamine	−0.20 op	PI: Ti, Sb, As	Ferromanganese	20
	Sodium arsenate	−0.39 ap	S: $2 \cdot 10^{-5}$%; AI: Cu	Arsenic	71
	Potassium cyanide	−0.76 sw	MI: uranium−ion exchange AI: Ni; S: 1γ	Uranium	40
		sw	AI: Cu, Zn, Mo, W	Zirconium	38
	Tartrate	ac	AI: Cu, Cd, Zn	Products of nonferrous metallurgy	53
	Hydrochloric acid	ap	S: 10^{-6}%; AI: Cu, Cd, Zn MI: indium−extraction	Indium	73
	Phosphoric acid	−0.76 sw	MI: uranium−ion exchange	Uranium	41

(continued)

Element	Base electrolyte	Half-wave potential and type of polarographic analysis	Characteristics of method	Object of determination	Source
Titanium	Phosphoric and sulfuric acids	ip	AI: Fe, Zr, Nb, Ta PI: Cr, Sn, Cu, As, Sb, Bi, U, Mo, V, Cd	Titanium-zirconium ores	112
	Pyrophosphoric acid	−0.6 ip	AI: Fe, W, Nb	–	113
Nitrogen		ip	Determination of nitrates and nitrites after conversion to nitroxylenol	Air, soil, water	114, 115 116
Arsenic	Citrate, pH= 2.5	−0.87 ip	AI: Sb; PI: Pb, Cd Masking of Mn, Cu, Fe, Cr	–	117
	Oxalate	−0.73 ip	AI: Sb, Sn	–	105
Antimony	Hydrochloric acid	−0.18 ac	AI: In, Te; S: 10^{-6} moles/liter	Alloys	47
	Citrate, pH= 2.5	−0.43 ip	See arsenic	–	117
	Oxalate	−0.27 ip	AI: As, Sn	–	105
Bismuth	Perchloric acid	op	Bismuth reduction in the presence of chlorine ions	–	29
	Tartrate	op	S: 10^{-4}%; CM: extraction of bismuth ditysonate	Ores	118
	Hydrochloric acid	ap	S: $2 \cdot 10^{-6}$%; AI: Sn, Sb	Aluminum	107
Antimony	Hydrochloric acid	ap	S: $2 \cdot 10^{-5}$%; AI: Sn, Bi	Aluminum	107
Vanadium	Chlorides, pH= 1 to 10	ip	Relationship between ion state and $E_{1/2}$	–	119
Niobium	Ethylenediaminetetraacetic acid sodium salt, pH= 0.3 to 6.0	−0.70 ip	AI: Ta. Study of reduction conditions	–	120
	Phosphoric acid	ip	MI: from Fe−hydrolysis	Steel, ferroniobium	121
	Pyrophosphoric acid	−0.85 ip	AI: W, Ti, Fe	–	113
Sulfur	Hydrochloric acid	0.45 ac	Indirect sulfate-ion determination	–	34
	Hydrochloric and sulfuric acids	ip	Mechanism of oxidation of sulfide, thiosulfate, and sulfite ions	–	24, 122 123−124
Selenium	Hydrochloric, nitric, sulfuric, and perchloric acids	ip	Nature of tellurium wave under various conditions	–	125, 126
	Ammonium hydroxide	−0.90 ac	S: $5 \cdot 10^{-5}$%; determination in form of Na_2SeSO_3	–	49
	Tartrate, citrate	−0.6 ip	pH= 0.8−4.5	–	127
Tellurium	Hydrochloric and sulfuric acids	ip	Nature of tellurium wave under various conditions	–	125, 126
	Hydrochloric acid	−0.8 ac	S: 10^{-6} moles/liter; AI: In, Sb	Alloys	47
	Iodide, chloride, bromide, phosphate	ac	AI: Se. Tables of $E_{1/2}$ given	Products of nonferrous metallurgy	36
Molybdenum	Fluoride	ip	AI: tungsten	–	128
	Sulfuric acid	−0.55 ip	Study of nature of polarographic waves	–	129
Molybdenum	Heteropoly acids	ip	Study of polarographic behavior of Mo	Steel	130, 131
		sw	AI: Cu, Pb, W, Zn	Zirconium	38
	Ethylenediaminetetraacetic acid sodium salt	ip	MI: uranium−extraction Masking of V by citrate	Uranomolybdenum alloys	132
	Citrate	ip	CM: extraction of complex with benzoyloxime	Steel	133
Tungsten	Hydrochloric acid	ip	Reduction mechanism	–	134
	Pyrophosphoric acid	−0.45 ip	AI: Nb, Ti, Fe	–	113
	Hydrochloric acid	sw	S: 10^{-4}%	Perrenate	45
Uranium	Tripolyphosphate	−0.19	Electrode-reaction mechanism, pH= 7 to 10	–	135

(continued)

Element	Base electrolyte	Half-wave potential and type of polarographic analysis	Characteristics of method	Object of determination	Source
	Lactate	−1.1 −0,3 ip −0.6	Study of complexing process	–	136, 137
	Sulfosalicylate	ip	Composition and stability of complexes	–	138
	Glycolic acid	ip	Composition and stability of complexes	–	139, 140
	Ethylenediaminetetraacetic acid sodium salt	−0.3 dr	S: $2 \cdot 10^{-6}$ M; Al: Bi, Mo	Bismuth	141
Chlorine	pH= 1−10	ap ip	S: $5 \cdot 10^{-6}$ M $HgCl + e = Cl^- + Hg$ $HClO_2 + 4e + 3H^+ \rightarrow Cl^- + 4H_2O$	– –	142−144 145
Halogens	Sulfuric acid	op and ac	Nature of polarographic waves	–	22, 50
Iodine		ip	$KClO_3 + I^- \rightarrow KIO_3$	–	146
Manganese	Tartrate	−1.55 ip	Effect of pH on reaction $Mn^{3+} \rightarrow Mn^{2+}$	–	147
	Potassium cyanide	−1.36 −1.72 ip	Relationship between polarographic waves and complexing	–	148
	Bis-(2-Hydroxypropyl)-2-hydroxyethylamine	−0.25 −0.53 ip	Study of polarographic conditions	Manganese ores	149
	Sodium formate	−1.6 ip	Composition of complexes	–	150
Rhenium	Ammonium phosphate, et al.	−1.6 ip	Tables of $E_{1/2}$	–	151
Iron	Nitrilotriacetic acid	−0.25 −1.4 ip	Complexing and nature of waves	–	152
Cobalt	Sulfate, bromide, iodide	−1.5 ip	Nature of waves as a function of conditions	–	153
	Ethylenediamine	ip	Complexing and nature of waves	–	154
Nickel	Sodium formate and salts of inorganic acids	−1.09 −1.5 ip	Study of nature of polarographic waves	–	155 156
	Thiocyanate, urotropin, salicylate	−0.7 op	PI: Cu, Pb, Tl, Cd, Cr, Ti, V, Mo; methods of eliminating PI's given	Ores, steels, alloys	23
Ruthenium	Hydroxy acids, inorganic acids	op	Tables of potentials	–	17
	Sodium gluconate	−0.67 ip	PI: Pt; Al: Ir, Rh, Pd, Os	–	157
	Oxalates	ip	Study of complexing	–	158
Rhodium	Pyridine, picoline	−0.5 ip	Al: Ir; Reduction mechanism	–	159
	Ethylenediaminetetraacetic acid sodium salt	−0.9 ip	Al: Ir; optimum pH= 6 to 7; S: $3 \cdot 10^{-2}$M	–	160
	Thiocyanate	−0.6 op	Al: palladium	Rhodium-palladium alloys	30
Palladium	Pyridine	op	Al: rhodium	Rhodium-palladium alloys	30
	Ethanolamine	−0.78 op	Al: silver, gold	Alloys	27
Iridium	Hydrochloric acid	ip	Study of analytic application	–	161
Cerium	Acetate buffer, pH= 4.5 to 5.6	−0.9 ip	Reaction mechanism and optimum determination conditions	–	162
Cerium, rare earth elements	Ammonium chloride	−1.95 ip	Displacement reaction: $PbCrO_4$ + rare earth elements $\rightarrow Pb^{2+}$	Alloys	163

The third group of methods is connected with the study of the polarographic behavior of silico-, phosphoro-germanomolybdic, and other heteropoly acids [130, 131, 170-174]. In polarographic analysis of these acids, several degrees of reduction are observed, depending upon the nature of the extraneous electrolytes. Silicomolybdic acid in a base of potassium chloride and oxalic and tartaric acids gives two waves; the first corresponds to reduction to pentavalent molybdenum, and the second to reduction to trivalent molybdenum. Three waves are observed in a solution of citric acid; the first and third correspond to molybdenum reduction, and the second to reduction of the heteropoly acid:

$$Si(Mo_{12}O_{40})^{4-} + 8H^+ + 4e = H_4Si[Mo_{12}O_{36}(OH)_4].$$

On the basis of this, methods have been developed for determining silicon, phosphorous, molybdenum and certain other elements in silicates, aluminum alloys, uranium salts, steels, etc.

Indirect determinations according to the catalytic waves of hydrogen reduction are known. Beryllium can be determined [175] according to the catalytic current of hydrogen caused by the introduction of cobalt hydroxyquinolinate. Ions of beryllium, aluminum, and certain other elements reduce the height of this catalytic wave, and on this is based the determination of trace amounts of these elements. The determination of zirconium according to the nitrate ion reduction wave, which is catalyzed by zirconium and thorium ions [177], has also been described in [176].

Direct methods of determination are less widely used. A direct determination of aluminum in zinc alloys with a base of tetrabutyl ammonium chloride at pH= 3.7 is described in [86].

The Use of Complexing Agents as Ions for Determining Several Ions

The reduction of many ions in a hydrochloric acid solution with the aim of developing a procedure for determining traces of bismuth, copper, indium, and cadmium in gallium arsenide has been studied in [37]. The peak potentials of the elements studied are given in this work. The reduction potentials of ruthenium and osmium for their oscillographic determination in solutions of mineral and organic acids and their salts have been determined in [17]. In order to determine indium in the presence of large amounts of cadmium, lead, and tin, [92] recommends polarographic analysis of indium in a solution of potassium iodide, sodium chloride, or hydrochloric acid. Hydriodie acid was found to be a good base for polarographic analysis of tellurium in the presence of selenium in [36].

A study of the polarographic behavior of a number of elements in an orthophosphoric acid solution in [178] has shown that this base can be easily used for determining lead in tin. It is also suitable for the joint determination of niobium and tantalum, which are reduced at potentials of -1.05 and -1.20 V. The behavior of niobium, titanium, tungsten, and iron in a pyrophosphoric acid solution has been studied in [113, 121, 179]. By varying the acid concentration, it is possible to create conditions for the joint determination of four elements. In an 18 N $H_4P_2O_7$ solution, first of all iron is reduced, and then titanium and tungsten give a joint wave; niobium reduction occurs at a more negative potential. But in a 10 N solution of this acid, titanium and tungsten give separate waves; the niobium and hydrogen waves are merged into one. Thus, the amount of each component can be determined by polarographic analysis in 10 N and 18 N $H_4P_2O_7$.

A determination of thallium in a solution of tripolyphosphate $Na_5P_3O_{10}$ containing 0.1% camphor has been described in [99]. The reduction potentials of lead, copper, iron, bismuth, and cadmium ions are shifted to the more negative region in this case, and it becomes possible to determine thallium in the presence of the ions enumerated above. Tripolyphosphate has also been suggested as a base for determining uranium in [135].

Copper, cadmium, thallium, lead, and iron in selenium can be polarographically analyzed in an oxalic acid solution [19]. Here, separate derivative peaks of these elements were obtained. Trivalent antimony, arsenic, and tetravalent tin have been determined in an oxalate buffer [105].

A solution of diethylenetriaminepentaacetic acid containing Triton X 100 was used in [101] for determining thallium in the presence of Mn, Co, Ni, Zn, Cu, Pb, Bi, Cd, Cr, Mo, V, and As. The half-wave potentials of 25 different ions in solution of triethylenetetraaminehexaacetic acid at several pH values were determined in [180]. It was found, however, that there was no essential difference in the polarographic behavior of metal complexes with the indicated complexing agent and ethylenediaminetetraacetic acid sodium salt.

The use of various amino compounds as polarographic bases has been studied in a number of works [27, 85, 149, 181]. Some of them are suitable for determining thallium and cadmium, thallium and lead with indium, and also manganese and iron in joint presence. Complexes of ethanolamine have been used for polarographic analysis of copper, gold, and palladium.

A determination of silver in a cyanide solution has been described in [76]. If the excess of the potassium cyanide introduced is bound by nickel ions, then at a potential of -0.5 V a well expressed silver wave is obtained. The inhibiting effect of the lead ions was eliminated by adding an excess of ethylenediaminetetraacetic acid sodium salt.

Polarographic analysis of germanium in the form of its complexes with diphenols—pyrocatechol, pyrogallol—is described in [102]; and analyses of tin and lead [106], and also antimony and bismuth in citrate solutions [117], and selenium in the form of the compound Na_2SeSO_3 [49] have been described.

Polarographic Analysis in Nonaqueous Solutions

Anhydrous ethylenediamine [182-187], formamide and dimethylformamide [188, 189], dimethylsulfoxide [187, 190], acetic anhydride [187], and acetonitrile [191] have been investigated.

Clear irreversible waves of the cations of the alkali and alkali earth metals, Zn, Pb, Cd, Tl, Nb, and Ta have been obtained in anhydrous ethylenediamine in a base of tetraethylammonium nitrate. The absence of catalytic waves of hydrogen reduction and the receipt of good waves of the alkali metals, calcium, and niobium are advantages. Ethylenediamine has also been used as a base for polarographic analysis of lanthanum, praseodymium, and ytterbium in [186]. Compounds of thallium, lead, tin, and titanium in dimethylsulfoxide—compounds of thallium, antimony, zirconium, and hafnium, and also $SiCl_4$ and SiF_4—also give good waves in acetic anhydride. The waves of Cr, Fe, Co, Ni, Zn, Cd, and Mn are clearly expressed in anhydrous formamide with a base of 0.2 M $NaClO_4$. Polarographic analysis of nickel in acetonitrile has been studied in [191].

Methods of Concentration and Isolation of Inhibiting Elements

Various methods of removing the principal component of the material to be analyzed determining admixtures have been described. Before polarographic analysis of copper, cadmium, thallium, lead, and iron in selenium, the latter was removed by distillation of selenium bromide in [19]. In analysis of molybdenum alloys, uranium was removed by extraction with tributylphosphate in [132]; when determining nickel and lead in [40] and cadmium in [39], uranium was removed with an ion exchanger.

Various chemical methods have been proposed for concentrating trace amounts of elements before their polarographic analysis. Bismuth has been concentrated by extraction of diethylammonium—bismuth diethyldithiocarbaminate in [118]; copper, lead, cadmium, and zinc were concentrated when determining them in metallic uranium and other materials in [110], and by absorption with ion exchangers in [41]. Zinc and lead were extracted in the form of ditysonates in [81, 111]; molybdenum in the form of a complex with benzoyl-α-oxime in [133]; and gallium in the form of a chloride in [90].

Electrochemical concentration, i.e., polarographic analysis with accumulation on a stationary mercury drop, has gained the widest use. Its basic merit is an increase in determination sensitivity of one to two orders of magnitude [192-198]. Methods of determining thallium and copper at a concentration of up to 10^{-8} moles/liter [192]; Cu, Bi, Sb, Pb, Sn, and Cd in high-purity zinc and in reagents at a concentration of up to 10^{-5}% [193, 194] have been described. Lead, copper, and cadmium can be determined in uranium compounds even when the concentration of these ions in the initial solution reaches 10^{-7} to 10^{-9} moles/liter. The lower limit of determinable admixtures is a function of the duration of the electrolysis, the volume of solution, the sensitivity of the galvanometer, and a number of other conditions. Optimum conditions for reaching high sensitivity have been described in [198], and optimum conditions for achieving reproducibility of the method have been described in [200]. Techniques of determining metals in extremely low concentrations have been described in [201, 202].

In recent years, a method has begun to be used for determining elements that have been precipitated under various conditions on an electrode in the form of some insoluble compound. The determination of chlorides in [142-144] was based on electrochemical concentration of them on the surface of a mercury electrode in the form of a weakly soluble film.

The use of the following reaction was described in [203] for electrochemical concentration of manganese:

$$Mn^{2+} + 4OH^- = MnO_2 + 2H_2O + 2e.$$

The precipitated manganese dioxide was dissolved on the cathode: $MnO_2 + 2H_2O + 2e = Mn^{2+} + 4OH^-$, by setting the current which under definite conditions is proportional to the concentration of bivalent manganese in the initial solution. In electrolysis of ions of ferrous iron in a rich buffer solution at pH = 8, a film of trivalent iron hydroxide (ferric hydroxide) was formed on the mercury drop in [204]. The film was then subjected to cathodic polarographic analysis.

The Use of Catalytic Waves

In [176] zirconium was determined according to the nitrate ion wave, which was catalyzed by zirconium ions, and thorium was determined according to the catalytic wave of hydrogen in [177]. A number of works [205-208] have been devoted to studying catalytic waves with the participation of molybdenum, tungsten, and vanadium. For example, molybdenum catalyzed the reduction of chlorates in an acid medium: the Mo (V) ions formed on the mercury cathode served as the catalyst. The catalytic chlorate wave is suitable for determining molybdenum within the limits of from 10^{-4} to 10^{-5} moles/liter. Cysteine catalyzed hydrogen reduction in [209, 210]; the catalytic effect was increased in the presence of nickel ions. On the basis of this, it was possible to determine nickel and cobalt at a concentration of $5 \cdot 10^{-6}$ g-ion/liter. In the presence of Hg, Cu, Cd, Fe, Bi, Sn, Sb, and Tl ions, which form more stable compounds with cysteine than nickel, the catalytic wave is reduced in proportion to the concentration of the elements.

Other Works

Work on the polarographic analysis of fusions has continued. The behavior of oxides of various metals with a base of fused carbonates in [55, 211], borax in [212], sodium metaphosphate in [213-215], and various slags in [219] has been studied.

The errors of polarographic determinations by the addition method have been evaluated in [220]. The possibility of determining radioactive substances by precipitating them on a drop electrode at a certain determined potential in the form of an amalgam or an insoluble film, and the possibility of later measurement of the radiation intensity were studied in [221]. Acids could be determined in [222] by measuring the diffusion current of the reduction of quinone to quinol, the intensity of which is a function of the hydrogen ion concentration in the solution. The sensitivity method of polarographic analysis in [223] consisted of measuring the difference between the total current in the analyzed solution and the residual current in the base electrolyte. This difference, and, therefore, the determination sensitivity, is maximal at a definite moment in the life of the mercury drop. It has been shown that with this method it is possible to determine ions of lead, copper, and zinc at concentrations of up to 10^{-6} moles/liter. Polarographic analysis of trace amounts of europium has been described in [224].

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