METHODS OF CHEMICAL ANALYSIS

COULOMETRIC ANALYSIS

INDIRECT COULOMETRY (REVIEW)

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The wide use of semiconductor technology, heat-resisting alloys, and construction materials and the requirement for continuous control of technological processes and the determination of impurities in natural media have predetermined the development of the coulometric method in the last ten years. In this period coulometry was promoted to one of the leading places among physicochemical analytical methods and the volume of information on this method has increased significantly. New variations of the coulometric method, different methods, instruments, data units, cells, and electrodes have appeared.

We recognized the expediency of examining direct and indirect coulometry [1]. Acid–base reactions, oxidation–reduction reactions, precipitation, and complex formation are used in indirect coulometry (IC); the content of material is established from the amount of electricity (Q) lost during electrotransformation of both the auxiliary reagent, from which the titrant is generated, and from the determined material, if it is electroactive. In IC the process of determination consists of electrochemical and chemical reactions, without consideration of remaining compulsory conditions. The determination can be carried out at a constant value of the electrolysis current ($i = \text{const}$) and at a controlled potential of the working electrode ($E = \text{const}$).

Papers which appeared in the period 1966-1974 on the determination of inorganic materials by the IC method are examined in this review. The last review in the USSR [2] on coulometry included papers published in 1964-1966. Data on earlier papers and theoretical fundamentals of the method are presented in reviews [3-14], monographs [15-18], and books [1, 19-21].

Literature data on IC at $i = \text{const}$ and $E = \text{const}$ are presented in Tables 1 and 2.

It follows from the tables that the IC method is used for checking the content of materials after transforming them into solution. More than 65 elements and their compounds are determined in natural and commercial materials. The use of IC in nonaqueous and mixed solvents [87, 60-66, 114-116, 120, 123-130, 171] and the use of titrants, unstable under normal conditions and generated from amalgams [177, 232, 240-242, 252, 288], active electrodes [114-116, 120, 131-132, 155-162, 184-191, 194-196, 212-213], and ion-selective membrane electrodes (ISME) [163, 280, 287] have broadened significantly the range of problems solvable by the given method.

At the present time IC has found use in the analysis of semiconductor materials, meteorites [240-242, 247, 248, 252, 266, 284-286], various reagents [121, 122, 277, 278], natural water and sewage [96, 100, 107], steels, alloys [87, 108-113, 179-183, 192, 193, 303], standard and raw materials for content of main material [117-119, 208, 257-261], and products of the chemical industry. Analogous instruments and electronic computers [309] are used for the study of kinetics of homogeneous [306, 307] and stepwise [308] reactions by the IC method. The necessary conditions (differences in reaction rate constants $\geq$ three orders of magnitude) of two materials, interacting with the electrically generated titrant, at which they can be titrated with sufficient accuracy and reproducibility, are examined.

Investigations on the determination of the basic and stoichiometric composition of certain semiconductor compounds and commercial materials, the preparation of oxidizing agents such as In (I), unusual from the point of view of titrimetry, by electrosolution of an indium grid in a $HClO_4$ medium [310], and the preparation of titrants in a eutectic melt [131-135, 246] are of interest. It is particularly necessary to note the use of this method for the determination of ultramicro amounts of nickel [240-242], zinc [247-248], chromium [200, 201, 209], and

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TABLE 1. Indirect Coulometry, i-const. (Assumed abbreviations: A is amperometry, BA is biamperometry, P is potentiometry with nonpolarized electrodes i-0, P≠0 is potentiometry with polarized electrodes, Ph is photocolorimetry, SPh is spectrophotometry, V is the visual methods using an indicator, I* is the indicator electrode, G* is the generator electrode, → is transformation of material from one form into another, i is the size of the electrolysis generator current)

Determined component	Object of analysis	Determinable amounts	Titrant	Experimental conditions. Interfering ions	Method of establishing end point	Literature
1	2	3	4	5	6	7
H_2	Sn, Ti, Nb, Be, stainless steel	$6.5 \cdot 10^{-2}$-$1 \cdot 10^{-5}$%	BrO^-	$H_2 \rightarrow NH_3$, 5 M NaBr in borate buffer pH~ 8.6	A	22, 23, 24, 25
H_2O	Minerals, silicon-containing materials	0.4-4.5%	I_2	Fischer reagent, i = 2 mA, I is graphite	BA	26, 27, 28
	Adsorbed on the surface of steel, glass	> 0.20 μg/cm²	BrO^-	$H_2O \rightarrow NH_3$, 5 M NaBr in borate buffer, pH~ 8.6	A	24, 25, 29
	Gases and polymeric materials	$1 \cdot 10^{-3}$-$3 \cdot 10^{-4}$%	I_2	Fischer reagent, i 25-100 mA	BA	30, 31, 32, 33
	Peroxides, silanols	0.4-0.9 μg	I_2	Fischer reagent, I is graphite	BA	34, 35
	Gases, liquids	5-500 μg/ml	I_2	Fischer reagent	BA, A	36, 37
	Gases, organic materials	> 1 μg	I_2	Fischer reagent	A	38-41
H_2O_2	H_2O_2 solutions	1-100 mg	BrO^-	1 M NaBr in borate buffer, pH~ 8.2, i 4-19 mA	A	42
Strong and weak acids (HCL, H_2SO_4, H_3BO_3)	Aqueous and nonaqueous acid solutions	> 0.001 m/liter	OH^-	1 M K_2SO_4, Na_2SO_4, 0.03-0.2 M KBr, i ≥ 1 mA/cm², I is glass	Ph, P	23, 42-57
H_2S, H_2SO_4	Gases, oils, sulfonic acids	> 5 mg/liter	I_2	$H_2SO_4 \rightarrow SO_2$, 1 N KI. S interferes	BA	58, 59
Strong and weak bases	Aqueous and nonaqueous solutions	> 0.001 m/liter	H^+	An AcOH–Ac_2O (95:5) mixture in 0.1 M $NaClO_4$, 6-8 M $NaClO_4$ and $LiClO_4$ in acetone i 1.5 mA, I is Bi or glass	P, BA	60-66
Be	Beryllium samples	8-10 mg	Br_2	0.1 M KBr + 0.14 M HNO_3 + 2-methyl-8-quindine		67
C	Ti, Ni, Pu, Mo, alloys, steels, pig irons, rocks, uranium and plutonium alloys	> 0.5 μg	OH^-	$C \rightarrow CO_2$, $Ba(OH)_2$ (pH~ 9.9), $BaCl_2 \cdot 2H_2O$ or $Ba(ClO_4)_2$ (pH~ 9.5), I is glass, i ≥ 1 mA, acidic and basic gases interfere	P, Ph	68-86
	Technical samples, steels	> 0.5 μg	CH_3OK	$C \rightarrow CO_2$, acetone (anh.) + 0.5% CH_3OH + KI, dimethylformamide + 2 ml of 0.1% thymolphthalein + KI + 3 ml of monoethanolamine	V	87

CN^-	Gas mixtures, electrolytes	> 94 μg	Br_2	Borate buffer solution	P ≠ 0	88, 89
CO_2	Atmospheric air	> 0.001%	OH^-	$Ba(ClO_4)_2$, pH > 9.0, i ≥ 2 mA, I is glass, bismuth. Acidic and basic gases interfere	P	90-93
N_2	Titanium, steels, sewage, petroleum ether, sea water	$< 3 \cdot 10^{-3}$%	BrO^-, H^+ or OH^-	$N_2 \rightarrow NH_3$. Acidic and basic gases interfere	A, Ph	94-103
NO_2^-	Aqueous solutions	$5 \cdot 10^{-5}$ g-eq/liter	Br_2, Ti (III)	1.6 M KCl + 0.1 M KBr + $2.5 \cdot 10^{-2}$ M $CuSO_4$, pH ~ 3.5, 0.6 M $TiO(SO_4)_2$ + 8 M H_2SO_4	BA, SPh	104, 105, 304
NO_3^-	Electrolytes	0.8-10 mmole	Fe (II), Ti (III) Viologen	80% H_2SO_4 containing H_3PO_4 and Fe (III), 0.6 M $TiOSO_4$ + 8 M H_2SO_4. Mn (II), Sb (III), SCN^- interfere	BA	106, 153, 304
O_2	Natural waters, gases			Acetate buffer solution	BA	107
	Titanium and its alloys, pig irons, pyrolysis products	> $5.5 \cdot 10^{-4}$%	OH^-	$O_2 \rightarrow CO \rightarrow CO_2$, 5 M $Ba(ClO_4)_2$, pH ~ 9.5. Acidic and basic gases interfere	P	108-113
F^-	Electrolytes	> $4 \cdot 10^{-4}$%	Al (III), La (III)	70% C_2H_5OH + 0.3 M KCl + 0.3 M CH_3CO_2H, G is Al, LaB_6. I is an ion-selective membrane electrode, beryllium	P	114-116
Na_2CO_3, $Na_2B_4O_7$	Salts, standard samples	Macroamounts	OH^-	1 M Na_2SO_4, pH < 7, i ≥ 1 mA, I is glass. CO_2 interferes	P	117-119
NaCl	Monocrystal	Macroamounts	Ag^+	1 M $NaNO_3$ + 1 M CH_3CO_2H + 50% CH_3OH, G is Ag, I is Ag	P	120
$NaNO_3$, Na_2SO_4, $NaCH_3CO_2$	Salt solutions	> 1 mg	OH^-	$NaNO_3 \rightarrow HNO_3$, $Na_2SO_4 \rightarrow H_2SO_4$, 1 M Na_2SO_4 + 10^{-3} M H_2CO_4, I is glass	P	121, 122
S	Iron, steel, petroleum, sulfide lye, petroleum ether	> 0.01 mg, > 8 μg/liter	I_2	$S \rightarrow SO_2$, 0.1% KI + 1% CH_3CO_2H, 0.05 M KI + 0.05 M HCl. Unsaturated hydrocarbons interfere	P, A	123-130
S^{2-}	Electrolytes	> 1 μg/ml	Ag (I)	3-10 M NaCN + 0.1 M NaOH, LiCl–KCl melt, G is Ag, I is Ag/Ag_2S	P, BA	131-135
SO_2	Air, gas mixtures, exhaust gases	> 0.5 > 0.7	I_2 Br_2	≥ 0.1 M KBr + 7.5 M H_2SO_4, 1.5% KI. In phosphate buffer, pH ~ 7.4, i 10^{-7} A/cm^2. NO_2, olefins interfere	P, BA	29, 122, 134-146
SO_3^{2-}, $S_2O_3^{2-}$	H_3PO_4 solutions	> 5 ppm	Br_2, I_2Cr (VI), Cu (II)	I_2 is generated from KI by reaction with Cr(VI), pH ≤ 7, K_2SO_4, $(NH_4)_2SO_4$, G is Cu. NO_2, olefins interfere	P	147-149
H_2S	Natural gases, gas mixtures, hydrocarbons	> 10^{-6} vol. %	Br_2, I_2	≥ 0.1 M KBr, containing 32.4 g of K citrate, 15 g of KI, and 10 g of dimethyl sulfoxide. NO_2, olefins interfere	P	140-142, 147, 150-152

*Electrode materials are indicated if they are prepared from a material other than platinum.

TABLE 1 (continued)

Phosphites	Hypophosphites	> 0.4 μg/ml	Br_2	0.5 M KBr, pH ~ 6.6, i = 0.4-4 mA/cm², t = 50°C	P	154
Cl^-	Natural waters, 35% H_2SO_4, 53% H_3PO_4, 37% $HClO_4$	> 0.1 μg/liter	Ag (I)	80% CH_3OH + 0.05 HNO_3, CH_3CO_2H : H_2O = 3 : 1, G is Ag; I is Ag/AgCl. Br^-, I^-, SCN^-, CN^-, SO_3^{2-} interfere	P	155-162
ClO^-	1 M HNO_3	> 0.2 mmole	Ag (I)	0.1 M $AgNO_3$, i 10 mA, G is Ag_2S, I is an ion-selective membrane electrode. Br^-, I^-, SCN^-, CN^-, NO_2^- interfere	P	163
	Aqueous solutions	> 1 μg	Hg (I)	0.2 M KNO_3, water–methanol medium, pH ~ 2-3, G is Hg, I is $Hg/HgCl_2$. Br^-, I^-, $C_2O_4^{2-}$ interfere	P, A	161, 164-167
ClO^-	Technical $Ca(ClO_2)$	Microamounts	I_2	Excess of As (III) standard solution, pH > 5	BA	168
ClO_3^-, ClO_4^-	Electrolytes	> 2 mmole/liter	Ti (III)	0.08 M KSCN + 2 M HCl or NaCl + 0.1 M Ti (IV), pH < 7, G is Hg	A, P	169
			Fe (II)	Acetate buffer containing Fe (III)	BA, A	170
K^+	Solutions	> 1 mg-eq	Ag (I)	Excess tetraphenyl borate (I), 40% acetone + 42.5 g of $NaNO_3$, G is Ag	BA, P	171, 172
Ti (III)	Alloys, solutions	> 0.5 mg	Cr (II) Tl (III)	2-3 M HBr + 0.1 M CrB_3, 2 M H_2SO_4, containing Tl (I), I is Hg	A	173, 175
Ti (IV)	Alloys, solutions	> 0.39 μg/ml	Fe (III) Ti (III)	Ti (IV) → Ti (III), 1.5 M H_2SO_4 + 0.37 M HCl or 0.74 M $H_2C_2O_4$ + 0.12 M Mohr's salt. Excess $Ce(SO_4)_2$ + 0.5 N $Ti(SO_4)_2$	BA, V, BA	176, 177
V (II)	Model solutions, synthetic mixtures	> 0.02 mg/liter	Fe (III)	0.1 M Fe (II) + H_2SO_4, pH > 1, G is Hg, I is Hg	BA	178
V (IV) V (V)	Three-components Mn–Ce–V systems, alloys, steels	0.025-0.0034%	Fe (II)	0.03 N EDTA, Fe (III) + 2 M H_2SO_4, 12 M H_3PO_2 containing Fe (III), pH ~ 4-5. NO_3^- interferes	BA, A P ≠ 0	179-183
		4-10 mg-eq	Mo (V)	4.5 M H_2SO_4 + 0.1 M $(NH_4)_2MoO_4$, i 0.05-10 mA/cm²	A, P, BA	148, 167, 184
	Alloying additions, instrumental steels, alloys, chromite ores	Microamounts	Sn (II) Fe (II)	1 M H_2SO_4 or HCl, i 0.1-30 mA/cm², G is Sn, 1.0-7.0 M H_3PO_4, HCl, H_2SO_4. H_3PO_4 + KOH, pH 2-3, G is Fe, i 8-100 mA/cm²	P, BA, BA	184-191
V (IV)	Brasses, bronzes, steels	0.04-0.08 mg	Cr (VI) Sn (II)	HCl, H_2SO_4, H_3PO_4 solutions, G is Sn, Cr, i 5-100 mA/cm²	A	184, 192, 193, 303
	Metallic zinc	Microamounts	Cu (I)	1 M HCl, G is Cu	A, P ≠ 0	194-196

Cr (III)	Bichromate lye, Cr (IV) compounds	$> 2 \cdot 10^{-3}$%	$[Fe(CN)_6]^{3-}$	0.1 M $K_4[Fe(CN)_6]$, pH > 7, i 1 mA. Mg (II) interferes	BA	197, 198
$Cr_2O_7^{2-}$	Alloying additions, binary mixtures	Microamounts	Sn (II) Cu (I)	1 M H_2SO_4, HCl or H_3PO_4, i 0.1-30.0 mA/cm^2, G is Cu, Sn	P, BA	185, 195, 303
Cr (VI)	Model solutions, chromium compounds, $K_2Cr_2O_7$ primary standards, aluminum, steels, alloys	≥ 0.015 μg	Fe (II)	0.1 M $Fe(NH_4)_2(SO)_4 + 2$ M H_2SO_4, pH < 7, i 3.2 mA/cm^2. 1.0-7.0 M H_3PO_4, i 8-10 mA/cm^2, G is Fe, H_3PO_4 + KOH, pH ~ 2-3, G is Fe	A, BA, V	178, 182, 189, 190, 199-210
	Model solutions	> 0.03 mg/ml	Ti (III)	6 M $H_2SO_4 + 0.5$ M $Ti(SO_4)_2$	V	211
	Model solutions, brasses, Armko iron, steels	> 0.005 mg/ml	V (III) VO_2^+	0.05-3 M H_2SO_4, 1 M H_3PO_4. G is V	P, BA	212, 213
Cr (VI)	Model solutions	$> 1 \cdot 10^{-4}$ mole/liter	Pb (II)	0.2-0.5 M KNO_3, i 15-40 mA, G is Pb, I is Hg	A	214
			Mn (III)	0.5 M $MnSO_4$ + 1 M H_2SO_4. 0.5 M $MnSO_4$ + 1 M H_2SO_4 + 0.1-0.5 M KF, 0.1 M $Mn(ClO_4)_2 + H_2O + H_2SO_4 + H_3PO_4$	P	215-218
			OH^-			219
Mn (II)	Brasses, steels, bronzes,	0.04 mg	Cr (VI)	0.2 M H_3PO_4, HCl or H_2SO_4, i 5-100 mA/cm^2, G is Cr	P, A	151, 192, 193
	Manganese salts, alloyed steels, ferromanganese	Milligram	Fe (II)	1 M $Fe_2(SO_4)_2$	P, BA	220
Mn (III) MnO_4^-	Iron	≥ 1 μg	$Ag_7O_8NO_3$	$AgNO_3$ solution, i 0.1-0.3 mA, 70°C	P	221
	Ferrites	0.05-30%	Fe (II)	1-7.0 M H_3PO_4, i 8-100 mA/cm^2, G is Fe	P	222
	Steels	≥ 0.5%	Co (VII)	12 M H_3PO_4, G is Cr	P	223
	Model solution, alloys, chromium compounds, ferrites	0.1-2.3 mg	Fe (II)	2 M $H_2SO_4 + Fe_2(SO_4)_3$, i 9.0 mA, 1.0-7.0 M H_3PO_4, G is Fe i 8-100 mA/cm^2	P, A, V	179, 189, 206, 222, 224
	Model solutions, steels, alloys, chromite ores	> 0.005 mg/ml	V (III) VO_2^+, Cu (I), Fe (II), Mo (V)	0.05-3 M H_2SO_4 + 1 M H_3PO_4, G is V. 1 M HCl or H_3PO_4, G is Cu, H_3PO_4 + KOH pH ~ 2-3, G is Fe 4 M H_2SO_4 + 0.1 M $(NH_4)_2MoO_4$ i 0.05 mA/cm^2. NO_3^- interferes	P, BA	148, 179, 189, 206, 222, 224

TABLE 1 (continued)

Fe (II)	Galvanic vats	Milligram	Mn (III) Br_2	190 ml of H_2SO_4 + 120 g of $MnSO_4$ in 1 liter of 0.1 M KBr + $2.5 \cdot 10^{-2}$ M $CuSO_4$, pH ~ 3.5	P	105, 225
	Model solutions	> $5 \cdot 10^{-4}$ mole/liter	OH^- MnO_4^-	Fe (II) → $Fe(OH)_3$, 0.5 M Na_2SO_4 I is glass	P	226
	U–Fe mixtures, magnesiowüstite	≥ 5 μg	Ce (IV)	0.01 M H_2SO_4 + 0.03 M $Ce_2(CO_4)_3$	P	227, 228
	Salt solutions	≥ 0.5 mg	Br_2	0.1 M KBr	BA	229
	Model solutions, synthetic mixtures	Milligram	$Cr_2O_7^{2-}$	2.0 M H_2SO_4 or 1.0 M HCl, i 0.3–150 mA/cm², G is Cr	P, A	230, 231
Fe (III)	Model solutions, synthetic mixtures, brasses, steels	≥ 0.5 mg	Ti (III)	Fe (III) → Fe (II), excess $Ce(SO_4)_2$, SCN^- interferes, G is Cu, amalgamated copper	BA	177, 232
			Cu (II)	1 M HCl or H_3PO_4. G is Cu	Ba, A, P	195
			Mn (III)	0.2 M $MnSO_4$ + 3 M H_2SO_4	V	211, 233, 234
	Ferrites	Milligram	Cu (I)	0.1 M HCl, 0.36 M H_2SO_4 + 0.01 M $CuSO_4$	P	235, 236
	Model solutions, synthetic mixtures	Milligram	V (IV)	1–7 M NaOH, G is V	P	237
			Cr (III)			238
			MnO_4^-	6 M H_2SO_4 + $MnSO_3$	V	206
$Fe(CN)_6^{3-}$	Model solutions	4–10 mg-eq	Mo (V)	4 M H_2SO_4 + 0.1 M $(NH_4)_2MoO_4$, i 0.5 mA/cm² ClO_4^-, PO_4^{3-}, NO_3^- interferes.	A, BA	148
	Microimpurities, alloying additions, Dural	Microamounts	Sn (II)	1 M H_2SO_4 or HCl, i 0.1–30.0 mA/cm², G is Sn	P, BA	185, 303
Co (II)	Synthetic samples	$2 \cdot 10^{-2}$–$8 \cdot 10^{-3}$ mmole	La (III)	EDTA (excess) + H_3BO_3, G is LaB_6	V	239
Ni (II)	Semiconductor compounds, alloys, meteorites	≥ $1 \cdot 10^{-3}$ μg	EDTA	EDTA Hg solution, G is amalgamated, Ag	P	240–242
	Synthetic samples	$2 \cdot 10^{-2}$–$8 \cdot 10^{-3}$ mmole	La (III)	EDTA (excess) + H_3BO_3, G is LaB_6	V	239
	Model solutions	0.70–8.8 mg	OH^-	0.1–0.5 M Na_2SO_4, I is glass	P	243

Cu (II)	Model solutions, organic materials	6-100 μg	I_2	0.1 M KI + 0.5 NH_4SCN + 0.1 H_2SO_4 + I_2 (excess)	BA	244
		> 1 μg	OH^-	$3 \cdot 10^{-4}$ M K_2SO_4, G is Hg	V	245
		Microamounts	I^-	(Li, K)NO_3 melt, 430 K, i 1-25 mA/cm², G, I are Pt coated with AgI	P, BA	246
Cu (II)	Alloying additions, Dural 287	Microamounts	Sn (II)	1 M H_2SO_4 or HCl, i 0.1-30.0 mA/cm², G is Sn	P, BA	186, 303
Zn (II)	Semiconductor compounds, alloys, Zn (II) salt solutions	> $1 \cdot 10^{-3}$ μg	$Fe(CN)_6^{4-}$	$7 \cdot 10^{-3}$ M $K_3[Fe(CN)_6]$, pH ~ 1.5-2.0	P ≠ 0, BA	247, 248
				0.1 M $Na_4[Fe(CN)_6]$ + 0.1 M $HClO_4$, pH ~ 1.0, 0.1 M KCl, G is glassy carbon, I is Ag/Ag$[Fe(CN)_6]$	A	249-251
	Synthetic samples	$2 \cdot 10^{-2}$-$8 \cdot 10^{-3}$ mmole	La (III)	EDTA (excess) + H_3BO_3, G is LaB_6	V	239
	Semiconductor compounds, alloys	0.27-0.062 μg in 3-4 μl	EDTA	Hg complexonate in acetate buffer, pH ~ 5-6, G is Ag (amalgamated)	A, BA	242, 252
	Model solutions	10^{-4}-10^{-3} mole/liter	OH^-	Zn (II) → $Zn(OH)_2$, indirect determination from H_3O^+	V	253
Ge (II)	Model solutions	Microamounts	I_2	0.1 M KI + 5 M H_3PO_4, pH ~ 1	P, A	254
As (III)	Model solutions, nonferrous metals	> 0.2 mg/ml	Br_2	1 M KBr + 3M H_2SO_4, G is glassy carbon	P, BA	255, 256
As (III)	Arsenous oxide, monocrystal	Main component	I_2	0.1 M KI in phosphate buffer, pH ~ 7, i 1.5-3.0 mA/cm²	BA	257-261
		4.7-9.5 μg	$Cr_2O_7^{2-}$	2.5% KI in acetate buffer + Ca^{2+} (catalyst) pH ~ 6, G is Cr, i 3-100 mA/cm²	BA, A	262
Se (IV)	Model solutions	> 0.4 μg/ml	I^-	$4 \cdot 10^{-3}$ M I_2 + 6 M HCl, i 0.5-5.0 mA/cm², G is graphite	BA, A P, P ≠ 0	263-265
Se (IV) Te (IV)	Model solutions	> 0.4 μg/ml	BrO^-	0.1 M $NaHCO_3$ + 1 M KBr, pH ~ 6.3, 70°C, i 0.5-5.0 mA/cm²	P	264
	Semiconductor compounds, residues from Pb manufacture	> 3 μg/ml	Fe (II)	0.5 M H_2SO_4, 4 N H_2SO_4 + 1 M iron–ammonia alums and excess $KMnO_4$, 0.5-5.0 mA/cm²	P, BA	266
			Tl (III)	1.0 M $Ti(SO_4)_2$ in 6 M H_2SO_4, i 0.5-5.0 mA/cm², 20-70°C, G is Ag, W, C	P, BA	267
		> 0.8 μg/ml (Se) > 1.3 μg/ml (Te)	Sn (II)	0.8 M $SnCl_4$ in 6 M HCl, i 1.5 mA/cm²	A, Ph	268

TABLE 1 (continued)

Te (IV) Te (VI)	Model solutions	≥1.5 μg/ml	Ti (III)	0.6 M $TiOSO_4$ in 4-6 M H_2SO_4, i mA/cm^2	P	269
Br^-	Salt solutions	5-100 μg/ml	Ag (I)	1.3 M in NH_3 and 0.1 M in NaOH, G is Ag, I is Ag. Cl^- interferes	BA	270
			Hg (I)	0.2 M KNO_3, pH ~ 2-3, G is Hg, I is $Hg/HgCl_2$. Cl^- interferes	P	165, 166
Rb^+	Solutions	> 1 mg/eq	Ag (I)	Excess tetraphenyl borate (i), 40% acetone + 42.5 g of $NaNO_3$, G is Ag	BA, P	171
Mo (III)	Model solutions	> 0.02 mg/ml	Fe (III)	0.1 M $FeSO_4$+H_2SO_4, pH ~ 1.1, G, I are Hg	BA	178
Mo (VI)	Model solutions, alloys	Microamounts	Ti (III)	0.6TMiOSO$_4$ in 8 M H_2SO_4, i 1-5 mA, G is Hg, I is W, Pt. Cl^- interferes	P, A, BA, Ph, P ≠ 0	271, 272
			Fe (II)	0.1 M $Fe_2(TO_4)_3$ + 11 M H_3PO_4 + 2 M H_2SO_4, i mA/cm^2, G is W	A, BA	178, 273
	MoO_3-γ phase			$-Pt/MoO_3-\gamma/O_2/O_2$ (g) Pt^+		274
	Alloying additions	Microamounts	Sn (II)	1 M H_2SO_4 or HCl, i 0.1-12.0 mA/cm^2, G is Sn	P, BA	188
MoO_4^{2-}	Solutions	1 μg	Hg (I)	0.2 M KNO_3 in water–methanol medium, pH 2-3, G is Hg, I is $Hg/HgCl_2$, Br^-, I^-, CrO_4^{2-}, Cl^- interferes	P, BA	165
Ru (IV)			Ti (III)	0.2 M $TiCl_4$ + 4-6 M HCl or H_2SO_4, G is W or C	P, A	275
Ir (IV)	Model solutions, synthetic mixtures	≥10^{-5} mole/liter	Cu (I)	≥0.02 M $CuSO_4$ + 4 M HCl	P, BA, Ph	276
Ir (IV) Ru (IV)	Solutions $(NH_4)_2[IrCl_6]$ $(NH_4)_2[RuOHCl_5]$, $Na_2[RuCl_6]$	Microgram amounts	Sn (II)	0.16-1.6 $SnCl_4$, 20-50°C, i 1 mA/cm^2, G is W	P, BA, Ph	277
		≥1·10^{-6} mole/liter	Ti (III)	0.1 M $TiCl_4$ in 0.1 M HCl or H_2SO_4, i 1 mA/cm^2, G is W, C	P, BA	278
Ag (I)	Model solutions, metallic films	> 12 μg/ml	Cr (II)	1 M H_2SO_4, G is Cr	BA	279
Ag (I)	Model solutions	≥10^{-4} mole/liter	SCN^-, S^{2-}, I^-	0.1 M $NaNO_3$, pH ~ 1-9, i 125 mA/cm^2. (Li, K)NO_3 melt, 430°C, i 10 mA, G is Ag_2S, AgSCN, U is Ag, Pt/AgI	P, BA	164, 246, 280, 181
Cd (II)	Model solutions	Microamounts	Cu (I)	Reinecke salt, 1 M HCl, G is Cu, Al, Sn, Zn interfere	BA, Ph	282
		2.795 mg	OH^-	0.1-0.5 N Na_2SO_4	P	243

In (III)	Alloys, semiconductor compounds	> $1 \cdot 10^{-3}$ μg	$Fe(CN_6)^{4-}$	$7 \cdot 10^{-3}$ M $K_3[Fe(CN)_6]$, pH ~ 1.5-2.0, i 4-7 mA	V	247
Sn (II)	Model solutions	≥ 1 mg/ml	Hg (II)	1 g of tartaric acid + 1 ml of H_2SO_4 + 1 ml of phenolsulfonic acid in 10 ml of H_2O, i H_2O, G is Hg, I is Ag	P	283
		Milligram	$Cr_2O_7^{2-}$	2 M H_2SO_4 or 1.0 M HCl, i 0.3-150 mA/cm^2, G is Cr		231
	Monocrystals, Pb–Sn alloys	5-96%	I_2 + F (III)	0.018 M in H_2CO_4, $Fe(NH_4)_2(SO_4)_2$ and KI, pH ~ 1.1	BA	284-286
I^-, IO_3^-	Salt solutions	Microamounts	Ag (I), Hg_2^{2+}	0.2 M KNO_3, pH ~ 2-3, G is Hg, I is Hg/Cl_2	P, A	166
		4-10 mg-eq	Mo (V)	4.5 M H_2SO_4 + 0.1 M $(NH_4)_2MoO_4$, i 0.05-10 mA/cm^2, NO_3^- interferes	BA	148
Cs^+	Solutions	> 1 mg-eq	Ag (I)	Excess of tetraphenyl borate (I), 40% acetone + 42.5 g of $NaNO_3$, G is Ag	BA	171
La (III)	Model solutions	0.4-14.0 mg	F^-	0.3 M KNO_3 in 50% ethanol, i 15 mA/cm^2, G is LaF_3, I is an ion-selective membrane electrode	P	287
Nd (III)	Model solutions	1.2-170 μg	EDTA	Hg (II) complexonate, pH ~ 4.8, G is Hg, I is amalgamated Pt	P	288
Eu (III)	Misch metals	1-100 μg	Cr (VI)	Eu (III) → Eu (IV), 0.1-2.0 M HCl, G is Cr	P	289
			Br_2			290
Yb (II)			W (II)	Melt of KCl + LiCl		291
Ce (III)	Model solutions	6-8 mmole	$Mo(CN)_8^{3-}$	$4 \cdot 10^{-3}$ M $Mo(CN)_8^{4-}$ + 0.4 N K_2CO_3 + 1.5 N $KHCO_3$, 20-40°C. Mn (II), V (IV), U (IV) interferes		292
Ce (IV)	Model solutions, binary salts	> 0.3 μg	Fe (II) Ti (III)	0.5 M $(NH_4)_2SO_4 \cdot Fe_2(SO_4)_3$ + 2 M H_2SO_4 + 0.5 M $Ti(SO_4)_2$	P, BA	232, 233
			Cu (I)	1 M HCl or H_3PO_4, G is Cu	P	195
		4-10 mg-eq	Mo (V)	4 M H_2SO_4 + 0.1 M $(NH_4)_2MoO_4$, i 0.05 mA/cm^2. NO_3^- interferes	BA	148
		0.005 μg/ml	VO_2^+, V (III)	0.05-3 M H_2SO_4 + 1 M H_3PO_4, G is V	P	212, 213
	Steels	0.04-0.08 mg	Cr (VI)	Solutions of HCl, H_2SO_4, H_3PO_4, G is Cr	A, P	193

TABLE 1 (continued)

Re (VII)	Model solutions	0.4-4.0 mg	Ti (III)	0.6 M $TiSO_4$ + 8 M H_2SO_4 + 0.3 M H_3PO_4, 85°C	P	293
Pt (II) Pt (IV)	Model solutions	Microamounts	Br_2 · Sn (II)	Pt (II) → Pt (IV), 4 M NaBr, 0.2 M $SnCl_4$ + 0.3 M HCl, i 5-20 mA/cm²		294
	Binary salts		Cu (I)	1 M HCl or H_3PO_4, G is Cr	P, A	195
Au (III)	Synthetic mixtures	Microamounts	Cu (I)	1 M HCl or H_3PO_4, G is Cu	P, A	195
Hg (I)	Salt solutions	≥ $5 \cdot 10^{-5}$ g-eq/liter	I^-	0.1 M KNO_3, 0.1 M and 0.2 M $HClO_4$, G is Ag/AgI	P, BA	105, 246, 295
Hg (II)	Inorganic compounds, containing Hg (II)	0.1-0.5 μg	I_2	$K_4[Fe(CN)_6]$ + KI. Ag (I) interferes.	P, A	296
Tl (I)	Model solutions	Microgram amounts	$Mo(CN)_8^{3-}$	$4 \cdot 10^{-3}$ M $Mo(CN)_8^{4-}$ + 0.4 M Na_2CO_3 + 0.8 M $KHCO_3$	P	297
Pb (II)	Solutions, metallic films	≥ $1 \cdot 10^{-4}$ mole/liter	S^{2-}	50% acetone, G is Ag_2S, I is Ag	P, BA	280
Th (IV)	Salt solutions	Micro- and milligram	EDTA	0.016-0.006 M Hg (II), complexonate, G is Hg	P	298
U (III) U (IV)	U–Fe mixtures	≤ 5 mg	Ce (IV)	0.01 N H_2SO_4 + 0.03 N $Ce_2(SO_4)_3$	P	227
	Salt solutions		Fe (II)	4.0 M H_3PO_4 + 4 M H_2SO_4 + 5 g-ion/liter of Fe (III) + $K_2Cr_2O_7$ (standard solution)	A	300
U (VI)	Salt solutions	≥ 1 mg/ml	U (III)	0.1 HCl + 0.04 M UCl_4, i 2 mA/cm², G is Hg	P	299
U (VI)	Solutions of salts of U (VI) compounds	Microgram	Ce (IV)	U (VI) → U (IV), 0.5 g of sulfanilic acid in 2 M H_2SO_4 + 3 M HNO_3 + Fe (III) + $Ce_2(SO_4)_3$	P	301
	Th oxide	0.8-5.0 mg	Cr (II)	0.1 N $[CrBr_2(H_2O)_4]Br$ in 1-3 N HBr or $HClO_4$, G is amalgamated Pt or W, I is Hg	P	302
Np (IV)	Model solutions	Microamounts	Fe (II)	4 M H_3PO_4 + 4 M H_2SO_4 + 5 g-ion/liter of Fe (III)	A	300
Pu (IV)	Model solutions	Microamounts	Fe (II) Ce (IV)	4.5 M H_2SO_4 + 0.5 M H_3PO_4 + 5 g-ion/liter of Fe (III)	A	300, 302, 305

TABLE 2. Indirect Coulometry, E = const. (Assumed abbreviations: A is amperometry, P is potentiometry, BA is biamperometry, G is the generator electrode, I is the indicator electrode, V is the auxiliary electrode, I is generation of titrant with controlled potential from an external current source, a potentiostat, II is generation of titrant with controlled potential from a galvanic element, E is potential of titrant generation

Determined component	Object of analysis	Determined amounts	Titrant	Experimental conditions	Method of establishing end point	Literature
1	2	3	4	5	6	7
H_2O	Natural objects	$5 \cdot 10^{-4}$–5 mg	I_2	Fisher reagent, I		312
$N_2H_4 \cdot H_2SO_4$		≥6 μg/ml	Br_2	0.1 M HCl + 0.01 · 1 N KBr, G is C, V is platinum placed in 8 M H_2SO_4 saturated $KMnO_4$. II	P, BA	313
PO_4^{3-}	Salt solutions	≥0.162 mg	Pb (II)	0.1-0.2 M CH_3CO_2Na, G is lead amalgam, V is platinized platinum. II, E = 1.07 V	A	314
	Salt solutions	$1.66 \cdot 10^{-4}$–$1.33 \cdot 10^{-3}$ M	Bi (III)	Saturated NaCl solution, I is Hg, G is Bi, I, E = 0.050 V	A	315
S^{2-}	Sulfite lye	Milligram	Pb (II)	0.1-2 M CH_3CO_2Na, G is lead amalgam, V is platinized platinum, II, E = 1.07 V	A	314
S^{2-}	Salt solutions	0.01-0.2 μg/ml	Ag (I)	0.1 M NaOH, G is Ag. I, E = −0.360 V	P	316
$S_2O_3^{2-}$, SO_3^{2-}	Salt solutions	≥$6 \cdot 10^{-3}$ mg/ml	I_2	0.1 M HCl + 0.01-1 M KI, G is C, V is platinum immersed in 8 M H_2SO_4, saturated $KMnO_4$. II	P, BA	313
SO_4^{2-}	Sulfite lye	≥0.480 mg	Pb (II)	0.1-2 M CH_3SO_2Na, G is lead amalgam, V is platinized platinum. II, E = 1.07 V	A	314
SO_2	Digestion acid, sulfite lye	≥$4 \cdot 10^{-3}$ mg/ml	I_2	0.01 M HCl + 0.01-1 M KI, G is C, V is platinum immersed in a 8 M H_2SO_4 solution, saturated $KMnO_4$. II	P, BA	313
Ca (II)	Natural objects	Microamounts	Diethylenetriamine-pentaacetic acid	Complex of Hg (II) with diethylenetriaminepentaacetic acid, pH ~ 10, G is amalgamated Pt, I, E = −0.05 V	P	317
$CaSO_3$	Digestion acid, sulfite lye	≥$2.4 \cdot 10^{-3}$ mg/ml	I_2	0.01 M HCl + 0.01-1 M KI, G is C, V is platinum immersed in an 8 M H_2SO_4 solution, saturated $KMnO_4$. II	P, BA	313
V (V)	Salt solutions	≥0.017 mg/ml	Sn (II)	4 M HCl, G is tin amalgam, V is platinum immersed in a saturated solution of KCl and 0.5 M HNO_3 solution, II	P	318, 319
		Microgram	Cu (I)	4–M6, HCl, G is Cu, I, E = −0.05 V	P	320

TABLE 2 (continued)

$Cr_2O_7^{2-}$	Salt solutions	≥0.017 mg/ml	Sn (II)	4 M HCl, G is tin amalgam, V is platinum immersed in a saturated solution of KCl and a 0.5 M HNO_3 solution, II	P	318
	01 Bronze	Microamount	Cu (I)	4–M6 HCl, G is Cu, I, E=–0.05 V		320
MnO_4^-	Kaolin, gypsum	≥0.01 mg/ml	Sn (II)	0.01-4 M H_2SO_4, G is tin amalgam, II	P, A	321
	Brass, No. 65	Microamount	Cu (I)	4–6 M HCl, G is Cu, I, E=–0.05 V	P	320
Fe (III)	Kaolin, gypsum	≥0.01 mg/ml	Sn (II)	0.01-4 M H_2SO_4, G is tin amalgam, II	P, A	321, 318
Fe (III), Fe (II)	Slags	Milligram		II		322
$Fe(CN)_6^{3-}$	Salt solutions	≥0.017 mg/ml	Sn (II)	4 M HCl, G is tin amalgam, V is platinum immersed in a saturated solution of KCl and 0.5 M HNO_3 solution, II	P	318
Co (II)	Metallic nickel, steels	~3 mg	$Fe(CN)_6^{3-}$	0.5 M $K_4[Fe(CN)_6]$, pH~9.5-11.5, containing 80-100-fold excess of glycerin. I, E=–0.3 V	P	323
Cu (II)	Model solutions	27-109 μg-eq	Sulfidryl	0.01 M NaOH and 0.1 M NH_4OH+0.1 M NaOH, I, G is Hg, E=–0.90 V	A	324
	Natural objects	Microamounts	Diethylenetriamine-pentaacetic acid	Complex of Hg (II) with diethylenetriaminepentaacetic acid, pH~10, G is amalgamated Pt, I, E=–0.05 V or E=0.015 V at pH~5.0	P	317
Zn (II)	Natural objects	Microamounts	Diethylenetriamine-pentaacetic acid	Complex of Hg (II) with diethylenetriaminepentaacetic acid, pH~10, G is amalgamated Pt, I, E=–0.05 V	P	317
MoO_4^{2-}	Salt solutions	Microgram	Cu (I)	4–6 M HCl, G is I,* E=–0.05 V	P	320
	Salt solutions	≥0.398 mg	Pb (II)	0.1-2 M CH_3CO_2Na, G is lead amalgam, V is platinized platinum, II, E=1.07 V	A	314
Pt (II) Pt (IV)	Model solutions	0.2-7.7 mmoles	Br_2 Sn (II)	4 M NaBr+0.2 M $SnCl_4$+0.3 M HCl, I, E=0.14–0.5 V	P	325
U (VI)	Salt solutions	15-60 mg	U (III)	U (VI)→U (IV), 0.2 M HCl, 0.03 M HCl+0.3 M KCl, G is Hg, I, E=0.68 V	A, P	326
		2-20 mg	–	U (VI)→U (IV), 1 M H_2SO_4, G is Hg, I, E=0.20 V. Hg (II) interferes	P	327
Am (II)	Salt solutions. Standard solutions	7-60 mg	–	0.1 M $LiClO_4$+0.05 M Na_2CO_3, G is SnO_2 activated with antimony (conducting glass). I, E=0.30 V, Pu, Np, Fe, F, and radiolysis products interfere.	P	328, 329

*A word appears to be omitted in the Russian – Publisher.

cerium [212-213] in semiconductor compounds, alloys, and meteorites. The combination of IC with other physicochemical methods, for example, chromatography (chromatocoulometry), makes it possible to increase the sensitivity and accuracy of the determination of the latter and reveals great promise [311] for the detection of separation products on the chromatographic column.

Optical methods have obtained wide use in recent years, together with classical methods (amperometry, potentiometry), to establish end point: photocolorimetry and spectrophotometry [304, 268, 277], radiometry [312], ISME with solid and liquid membranes [114-116, 131-135, 163, 287], polarized electrodes having a reverse system of connections [90-93].

Of the methods used for indication of end point from the point of view of continuity and automation of the process of product determination in a liquid or gas flow preference should be given to potentiometry with polarized electrodes having a reversed system of connections. In the last case the size of the generator current is a direct measure of the concentration of the analyzed material.

It seems promising to us to use IC as formerly because of the relative simplicity of the experimental technique both in aqueous and nonaqueous media for standardization of solutions, preparation of titrants from active electrodes and gas mixtures having a known content of the dosed component, control of impurity content and distribution in natural objects, determination of the stoichiometric composition of semiconductor compounds and the ionic state of an element, products of chemical reactions and radioactive transformations and control of technological processes. The further development of an ultramicro variation of IC, described by I. P. Alimarin and M. N. Petrikova, is of definite interest.

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