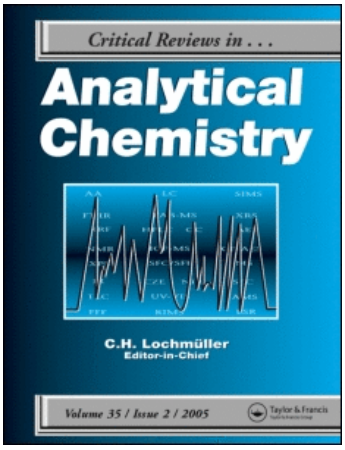

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Analytical Potentialities of Thiosemicarbazones and Semicarbazones

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Analytical Potentialities of Thiosemicarbazones and Semicarbazones

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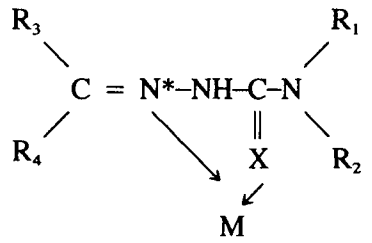
KEY WORDS: thiosemicarbazone, semicarbazone, spectrophotometry, fluorimetry, indicators, gravimetric reagents, potentiometric studies.

I. INTRODUCTION

Thiosemicarbazones (TSC) and semicarbazones (SC) are a class of compounds obtained by condensing thiosemicarbazide or semicarbazide with suitable aldehydes or ketones. The active grouping for chelation is shown in Structure I.



(I)

when X = O, semicarbazone;
X = S, thiosemicarbazone

Domagk and co-workers[1,2] reported for the first time the antitubercular activities of metal TSCs and SCs. Since then, a number of papers have appeared on the pharmacology[3,4] of these compounds. The metal-TSC complexes were found to be active against influenza,[5] protozoa,[6] smallpox,[7,8] and certain kinds of tumors,[9,10] and active as pesticides,[11] and fungicides.[12] The biological activity of TSCs is thought to be due to their power of chelation with traces of metal ions present in biological systems. The antitubercular activity of *p*-acetamidobenzaldehyde TSC is found to be enhanced by the presence of small amounts of copper ions.[13] In cancer treatment it has been shown that the active species is not the TSC itself, but a metal chelate of TSC.[14,15]

Depending upon the type of aldehyde or ketone used for condensation, TSC and SC can act as uni-, bi-, or multidentate chelating agents for metal ions, producing highly colored complexes. These colored complexes are useful in the selective and sensitive determination of metal ions. The aim of this review is to summarize these analytical applications.

II. PREPARATION OF THE REAGENTS

SCs are obtained by condensing semicarbazide hydrochloride with suitable aldehydes or ketones in the presence of sodium acetate. Sometimes hydrochloric acid (HCl) must be present in the preparation of SCs.[16]

TSCs are prepared by condensing thiosemicarbazide with an aldehyde or ketone in the presence of a few drops of glacial acetic acid. Preparation of the monoderivatives is simple, but the diderivatives are a little more difficult and require

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special treatment. To illustrate dipyridylglyoxal dithiosemicarbazone[17] was prepared by cyclizing the monoderivative with 6 *M* HCl, while 1,4-dihydroxyphthalimide dithiosemicarbazone was prepared from the dioxime derivative with thiosemicarbazide in excess.[18]

III. CHEMICAL PROPERTIES

Just as hydrazones are weaker bases than hydrazines, SCs and TSCs are weaker bases than semi-carbazides and thiosemicarbazides, respectively. Hydrolysis of these compounds yields the respective hydrazones, hence these compounds resemble hydrazones in many of their reactions.

Mild reductions of SCs and TSCs yield l-substituted semicarbazide and thiosemicarbazide, respectively. Catalytic reduction of these compounds yields hydrazides which are further hydrolyzed to hydrazines. Reaction with alkoxides such as sodium ethoxide converts semicarbazones into hydrazones, and with a strong base, hydrocarbons are obtained. This reaction may be used for the replacement of the carbonyl group by a CH_2-group.

The reagents can be readily hydrolyzed to give the original carbonyl compound, and hence are often useful for identification and isolation of carbonyl compounds. One method of obtaining the equivalent weight of the parent carbonyl compound is to hydrolyze the semicarbazone with aqueous HCl and titrate with standard iodate solution.[19]

IV. CLASSIFICATION

On the basis of reactants employed in the synthesis of SCs and TSCs, the reactions may be classified into four types:

1. Aldehyde or substituted aldehyde with semicarbazide or thiosemicarbazide.
2. Aldehyde or substituted aldehyde with substituted semicarbazide or substituted thiosemicarbazide.
3. Ketone or substituted ketone with semicarbazide or thiosemicarbazide.
4. Ketone or substituted ketone with substituted semicarbazide or substituted thiosemicarbazide.

The various SCs and TSCs that have been used as analytical reagents are summarized in Table 1.

V. USES AS SPECTROPHOTOMETRIC REAGENTS

Due to their ability to form intense colored complexes with various metal ions, SCs and TSCs have been employed in the spectrophotometric analysis of such metals.

Several papers have reported on the synthesis, properties, and analytical application of BABPT, DPGPT,[49] BAHPT,[60] 1,3-CDDT,[68] DBMT,[93] DPGBPT[129] ITMS,[158] HCT,[146] HMCOT, HMCODT,[150] NDIDDT,[187] ODDT,[194] SBPT, and SBPPT[246] with several metal ions (see Table 1 for an explanation of all abbreviations used in this article).

Ueda et al.[178] synthesized, characterized, and tested the color reactions of SCs and TSCs and their derivatives having sulfonic acid at 4-position, derived from 1,2-naphthoquinone (1,2-NQT), and observed that TSCs with potassium sulfonate at 4-position gave a specific color reaction with copper, which was investigated spectrophotometrically. 1,2-NQT was also explored for its analytical potentialities by Luque de Castro et al.[180] They reported optimum pH ranges, λ_{max}, and ϵ values for reactions with several metal ions. The pK value for 1,2-NQT was reported by Pilipenko and Tulyupa.[181] They determined pK values in 24 to 50% ethanol medium and carried out color reactions in 24% DMF medium.

The effect of cationic, anionic, and nonionic surfactants on the spectra of 1,2-NQPT-4S and their Zn and Cd complexes was studied.[182] It was also observed that both selectivity and sensitivity were enhanced by the addition of the surfactants.

Cano Pavon and Pino[220] studied the analytical properties of picolinaldehyde selenosemicarbazone in examining the spectral changes produced when Se is substituted for the S atom in PAT. They found the complexes to be less stable and that the absorption spectra showed batho- and hypsochromic shifts in comparison to PAT. Thus,

TABLE 1
List of TSCs and SCs Used As Analytical Reagents

Systematic name	Abbreviation	Ref.
4-Acetamido-benzaldehyde thiosemi-carbazone	ABAT	20
Acenapthene-quinone monothiosemi-carbazone	AQMT	21–23
Acetone thiosemi-carbazone	AT	24
Acetophenone thiosemi-carbazone	APT	25
Acetylacetone dithiosemi-carbazone	AADT	26, 27
2-Acetyl-pyridine thiosemi-carbazone	APyT	28–31
2-Acetyl-pyridine-4-phenyl-3-thiosemicarbazone	APPT	32
Anisaldehyde thiosemi-carbazone	AST	33, 34, 332
p-Anisaldehyde-4-phenyl-3-thiosemi-carbazone	ASPT	35–37
Benzal-dehyde thiosemi-carbazone	BAT	38, 289, 303, 304
Benzoin thiosemi-carbazone	BT	39
2-Benzoylpyridine thiosemi-carbazone	BPyT	317
2-Benzoylpyridine-4-phenyl-3-thiosemi-carbazone	BPyPT	40
Benzophenone thiosemi-carbazone	BPT	316
Benzothiazolyl phenyl ketone thiosemi-carbazone	BTPKT	41
1-Benzylidene-4-(2-pyridyl) thiosemi-carbazone	1-B-2PT	313
Biacetyl-monothiosemi-carbazone	BAMT	42
Biacetyl bis(4-phenyl-3-thiosemi-carbazone)	BABPT	43–49, 307, 309

TABLE 1 (continued)
List of TSCs and SCs Used As Analytical Reagents

Systematic name	Abbreviation	Ref.
Biacetyl-monoxime semi-carbazone	BAMOS	50, 297
Biacetyl-monoxime thiosemi-carbazone	BAMOT	51–57, 312, 318
Biacetyl-monoxime-4-phenyl-3-thiosemi-carbazone	BAMOPT	58, 59
Biacetyl (2-pyridyl) hydrazone thiosemi-carbazone	BAPHT	60, 61
β-Benzyl-mercapto-pyruvic acid thiosemi-carbazone	BMAPAT	62
5-Chloro-salicyl-aldehyde semi-carbazone	CSAS	322
5-Chloro-salicyl-aldehyde thiosemi-carbazone	CSAT	322
Cyclohexanone thiosemi-carbazone	CT	62
1,2-Cyclohexanedione dithiosemi-carbazone	1,2-CDDT	64–67, 208
1,3-Cyclohexanedione dithiosemi-carbazone hydrochloride	1,3-CDDT·HCl	68–81
1,3-Cyclo-hexanedione bis(4-methyl-3-thiosemicarbazone hydrochloride	1,3-CDBMT·HCl	82–84
1,3-Cyclohexanedione bis(4-phenyl-3-thiosemicarbazone)	1,3-CDBPT	85, 86
1,4-Cyclo-hexanedione dithiosemi-carbazone	1,4-CDDT	87
1,3-Cyclo-pentanedione bis(4-methyl-3-thiosemi-carbazone) hydro-chloride	1,3-CPDBMT·HCl	88–92
Dibenzoyl-methane thiosemi-carbazone	DBMT	93
3,5-Dibromosalicyl-aldehyde-4-phenyl-3-thiosemi-carbazone	DBSAPT	94, 95
3,5-Dichlorosalicyl-aldehyde-4-phenyl-3-thiosemi-carbazone	DCSAPT	96, 97
Diethyl-3,4-dioxadipate bis thiosemi-carbazone	DDBT	98
2,4-Dihydroxy-acetophenone semicarbazone	DAPS	100

TABLE 1 (continued)
List of TSCs and SCs Used As Analytical Reagents

Systematic name	Abbreviation	Ref.
2,4-Dihydroxy-acetophenone thiosemi-carbazone	DAPT	100–104
2,4-Dihydroxy-benzaldehyde semi-carbazone	DBAS	251–253
2,4-Dihydroxy-benzaldehyde thiosemi-carbazone	DBAT	105, 291, 292, 331
2,4-Dihydroxybenzophenone semicarbazone	DBPS	106
2,4-Dihydroxybenzophenone thiosemicarbazone	DBPT	106
2,2′-Dihydroxybenzophenone thiosemicarbazone	2,2′-DBPT	107, 108
4,4′-Dihydroxybenzophenone thiosemicarbazone	4,4′-DBPT	109, 110, 112
1,4-Dihydroxyphthalimide dithiosemicarbazone	DPIDT	18, 113, 115
4-Dimethylaminobenzaldehyde thiosemicarbazone	DABAT	116
5,5′-Dimethyl-1,3-cyclohexanedione bis(thiosemicarbazone) hydrochloride	D-1,3-CDBT·HCl	117–119, 299, 314
5,5′-Dimethyl-1,3-cyclohexanedione bis(4-phenyl-3-thiosemicarbazone)	D-1,3-CDBPT	85, 120
5,5′-Dimethyl-1,2,3-cyclohexanetrione-1,2-dioxime-3-thiosemicarbazone	D-1,2,3-CTDT	121–123
Di-2-pyridyl ketone thiosemicarbazone	DPKT	124, 125, 316
Di-2-pyridyl ketone-4-phenyl-3-thiosemicarbazone	DPKPT	126
Di-2-pyridylglyoxal dithiosemicarbazone	DPGDT	17, 125, 127, 128
Di-2-pyridylglyoxal bis(4-phenyl-3-thiosemicarbazone)	DPGBPT	49, 129, 130
Di-2-pyridylglyoxal bis(4,4-diphenylsemicarbazone)	DPGBDPS	131
Diethyldiketosuccinate dithiosemicarbazone	DSDT	99
Ethylpyruvate thiosemicarbazone	EPT	62
p-Ethylsulfonyl benzaldehyde thiosemicarbazone	ESBAT	132, 293, 294
2-Furaldehyde thiosemicarbazone	FAT	133, 134, 274, 295
2-Furaldehyde-4-phenyl-3-thiosemicarbazone	FAPT	296
Furylacrolein thiosemicarbazone	FACT	135, 136
Furylpentadienyl thiosemicarbazone	FPT	137
Furoin thiosemicarbazone	FT	138, 139
Glyoxal dithiosemicarbazone	GDT	140, 141
Glyoxal bis (4-phenyl-3-thiosemicarbazone)	GBPT	142, 143, 308, 320
o-Hydroxyacetophenone thiosemicarbazone	HAPT	144, 145
p-Hydroxybenzaldehyde thiosemicarbazone	HBAT	303, 304, 319
2-Hydroxycyclohexanone thiosemicarbazone	HCT	146
2-Hydroxyiminodimedone dithiosemicarbazone	HIDDT	147
2-Hydroxy-5-methylacetophenone semicarbazone	HMAPS	283
2-Hydroxy-5-methylacetophenone thiosemicarbazone	HMAPT	148, 149, 283, 302
2-Hydroxy-4-methylbenzaldehyde semicarbazone	HMBAS	275, 276
2-Hydroxy-3-methyl-2-cyclopenten-1-one thiosemicarbazone	HMCOT	150, 151
2-Hydroxy-3-methyl-2-cyclopenten-1-one dithiosemicarbazone	HMCODT	150
2-Hydroxy-4-methoxy-5-sulfobenzophenone thiosemicarbazone	HMSBPT	152
2-Hydroxy-1-naphthaldehyde semicarbazone	HNAS	277, 278, 254
2-Hydroxy-1-naphthaldehyde thiosemicarbazone	HNAT	153, 255
2-Hydroxy-1-naphthaldehyde-4-phenyl-3-thiosemicarbazone	HNAPT	154, 155, 300
3-Hydroxypicolinaldehyde thiosemicarbazone	HPAT	156, 271
1,3-Indanedione bis thiosemicarbazone	IDBT	157
1,2,3-Indanetrione monosemicarbazone	ITMS	158

TABLE 1 (continued)
List of TSCs and SCs Used As Analytical Reagents

Systematic name	Abbreviation	Ref.
1,2,3-Indanetrione monothiosemicarbazone	ITMT	159, 160
β-Ionone thiosemicarbazone	IT	161
Lawsone semicarbazone	LS	162
3-Methoxysalicylaldehyde semicarbazone	MSAS	176
3-Methoxysalicylaldehyde thiosemicarbazone	MSAT	176
3-Methyl-1,2-cyclopentanedione dithiosemicarbazone	MCPDDT	163–166
2-Methyl-1,3-cyclohexanedione bis(4-phenyl-3-thiosemicarbazone)	MCHDBPT	85–167
Methylglyoxal bis(4-phenyl-3-thiosemicarbazone)	MGBPT	168–170
2-Methyl-1,4-naphthoquinone thiosemicarbazone	MNQT	171, 172
6-Methylpicolinaldehyde thiosemicarbazone	MPAT	125, 173, 174
Methyl-2-pyridylketone thiosemicarbazone	MPKT	175, 281
Methyl thienylketone thiosemicarbazone	MTKT	177
1,2-Naphthoquinone-2-semicarbazone	1,2-NQS	178
1,2-Naphthoquinone-2-thiosemicarbazone	1,2-NQT	178–181, 326, 327, 335
1,2-Naphthoquinone-2-semicarbazone-4-sulfonic acid	1,2-NQS-4S	178, 179
1,2-Naphthoquinone-2-thiosemicarbazone-4-sulfonic acid	1,2-NQT-4S	166, 183–186, 286–288
1,2-Naphthoquinone-2-phenylthiosemicarbazone-4-sulfonic acid	1,2-NQPT-4S	182, 280
4-Nitrobenzaldehyde thiosemicarbazone	NBAT	20
2-Nitro-5,6-dimethyl-1,3-indanedione dithiosemicarbazone	NDIDDT	187
5-Nitro-2-furaldehyde semicarbazone	NFAS	188, 189, 282
5-Nitrosalicylaldehyde-4-phenyl-3-thiosemicarbazone	NSAPT	190
Oximinocyclohexanone thiosemicarbazone	OCT	191–193
Oximinodimedone dithiosemicarbazone	ODDT	194–198
Phenanthroquinone monosemicarbazone	PQMS	199
Phenanthroquinone monothiosemicarbazone	PQMT	200–206, 284
Phthaldehyde dithiosemicarbazone	PhADT	207, 108
Phthalimide dithiosemicarbazone	PIDT	157, 209–213
Picolinaldehyde semicarbazone	PAS	50
Picolinaldehyde thiosemicarbazone	PAT	294, 214–220, 316
Picolinaldehyde-4-phenyl-3-thiosemicarbazone	PAPT	221–223
Picolinaldehyde-4,4-diphenyl semicarbazone	PADS	224
Pyridoxal thiosemicarbazone	PT	225, 226
Pyruvic acid thiosemicarbazone	PyAT	62
Quinoline-2-aldehyde thiosemicarbazone	QAT	227, 228, 310
Resorcyl-4-aldehyde thiosemicarbazone	RAT	319
Salicylaldehyde semicarbazone	SAS	229–232, 256–264, 273, 274, 259, 321–325
Salicylaldehyde thiosemicarbazone	SAT	225, 233–239, 265–270, 272, 290, 301–304, 298, 240–243, 333

TABLE 1 (continued)
List of TSCs and SCs Used As Analytical Reagents

Systematic name	Abbreviation	Ref.
Salicylaldehyde-4-phenyl-3-thiosemicarbazone	SAPT	244, 301
4-Salicylamido-1-diacetyl monoxime-3-thiosemicarbazone	SDMOT	245
2-(3′-Sulfobenzoyl) pyridine thiosemicarbazone	SBPT	246, 247
2-(3′-Sulfobenzoyl) pyridine-4-phenyl-3-thiosemicarbazone	SBPPT	246
Thiophen-2-aldehyde thiosemicarbazone	TAT	38, 248, 289
Tripyridylglyoxal bis (4-phenyl-3-thiosemicarbazone)	TPGBPT	249
o-Thymolaldosemicarbazone	TyAS	285
Vanillin thiosemicarbazone	VT	250

the substitution of Se for the S atom appears to offer no advantage in this type of analytical application.

The introduction of a phenyl radical at the end of the thiosemicarbazide molecule is an excellent example of how group action in organic compounds can be modified to provide increased sensitivity; the molar absorptivities of metal-TSC complexes are favorably influenced by this substitution, which also causes a shift of absorption peaks toward longer wavelength. Some phenyl-TSCs have been studied as reagents;[32,43,142] however, compounds with the bis(phenyl-TSCs) grouping have received very little attention, probably due to their sparse solubilities in water. Ballschmiter[309] suggested the use of these reagents for extractive spectrophotometric procedures in metal trace analysis.

HPAT was used to determine Co(II) in highly acidic medium.[156] Salicylaldehyde thiosemicarbazone (SAT) was used to determine Mo(VI) in the presence of iron in highly acidic medium.[233] Extraction of the complexes not only increases the sensitivity but is also helpful in the simultaneous determination of the metal ion. To illustrate, DPGDT[128] reacts with Ni(II) and Co(II) at pH 5.2, but only the Ni(II) complex is extractable into $CHCl_3$, thus allowing the determination of both metals when present together. PAT was used for the determination of Fe, Co, Ni, and Cu in admixtures.[217] The different complexation rates of pyridoxal thiosemicarbazone (PT) with Fe(III), Ni(II), and Cu(II) are used in a single-point method for the simultaneous determination of Fe and Ni and of Fe and Cu.[226] Procedures also are described for the accurate analysis of binary and ternary mixtures of Zn, Cd, and Hg[163] and of Bi, Cu, and Co,[164] using MCPDDT.

Torres et al.[170] used MGBPT for the determination of Cu(II) and Pd(II) in a variety of catalyst preparations and in animal feed.

In recent years several papers reported on kinetic studies for the determination of trace amounts of metal ions. Kinetic methods are described for the determination of trace amounts of Co(II),[91] Cu[92] concerning its catalytic effect on the oxidation of 1,3-CPDBMT·HCl, Mn(II) with respect to DPIDT,[18,115] and In(III) with respect to 4,4′-DBPT.[110] In addition the oxidation of the sodium salt of 1,2-NQT-4S by H_2O_2 is catalyzed by I′ or Cu(II) in the presence of ascorbic acid or Co(II). A kinetic method based on this reaction was developed for the determination of I′,[184] Cu(II),[185] and Co(II).[186] The reaction rate was followed spectrophotometrically in each case.

A kinetic method has been described for the determination of nanogram amounts of Mn(II),[153] based on its catalytic effect on H_2O_2 oxidation of 2-hydroxy-1-naphthaldehyde TSC. Several wine and brandy samples have been analyzed by this method without the need for previous ashing of the samples.

Leggett and McBryde[219] determined stability constants of several PAT-metal ion complexes spectrophotometrically.

TSCs and SCs that are employed in the spectrophotometric determination of metal ions and anions are summarized in Table 2.

TABLE 2
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
AQMT	Co(II)	410	0.48	8.4–9.8	1–10.5		1:2		21
	Os(VIII)	525	1.53	7.6	2.1–11.1		1:3	Os determined in osmiridium	22
	Ir(III)	520	0.49	8.2	6.6–26.4		1:3		22
	Ru(III)	655	0.98	5.3–6.8	2.02–7.09		1:2	Complex soluble in 70% DMF	23
	Pt(IV)	515	1.2	1.9–4.1	2.83–11.6		1:2	Complex soluble in 70% DMF	23
ABAT	Os(VIII)	436		Acidic	1.2–14.7		1:3		20
AT	Mo(VI)	472	1.9	HCl medium	0.1–9.5	$CHCl_3$	1:4:2 (Mo: SCNd: AT)	Mo determined in Mo steels	24
APT	Cu(I)	590	0.37	Alkaline medium			1:1		25
	Cu(II)	490	0.37	Alkaline medium					25
	Cu(II)	418	0.83	Alkaline medium	Up to 5 μM	$C_2H_4Cl_2$			25
AADT	Cu(II)				2–24		1:1	80% DMF medium	26, 27
	Ni(II)				0.5–6		1:1	80% DMF medium	26, 27
	Co(II)				0.5–3.5		1:2	80% DMF medium	26, 27
APyT	Au(III)	460	1.5	11	4–9	$CHCl_3$	1:3	Au determined in synthetic Au-Pd, Au-V, and Au-Cu mixtures; instability const., 4.7×10^{-17}	28
	V(V)	400	0.56	3.5	2.6–7.4		1:1		29
	Fe(II)	550	0.53	2–3	3–6		1:2	Fe determined in ferrites and blood	30
		610	0.7	7–10	2–6	$CHCl_3$	1:1		30
	Mo(VI)	470	1.7	1.5 *M* HCl	0.1–6.5	$CHCl_3$	1:4:2	Mo determined in Mo steel	31
APPT	Fe(II)	650	0.68	4.9–11	3–7	C_6H_6			32
	Fe(III)	395	2.4	3.4–7.8	0.6–1.8				32
AST	Ni(II)	402	0.63	Alkaline medium	0.08–4.9		1:1	Ni determined in alloys and minerals	33
	Pd(II)	366–375	2.16	2.9–4.3	0.06–3.3		1:1	Method applied to analysis of alloys and minerals	34
	Cu(II)	398–406	0.61	0.1–0.6 *M* NaOH	0.1–10		1:2		34
ASPT	Cu(II)	365	4.57	5	0.35–1.3	IBMK		Cu determined in steel	35
	Au(III)	365	2.12	4–7	0.1–12.3	Ethyl-acetate	1:1	Au determined in synthetic samples	36
	Pt(IV)	360	1.58	1.7–3	0.1–20			Pt determined in synthetic samples corresponding to alloys	37
BPyPT	Fe(II)	670	1.14	4.7	1–4	C_6H_6			40
	Fe(III)	395	2.77	4.7	0.6–1.4				40
BTPKT	Cu(II)	446		6.5	0.2–15			By measuring at pH 2.5, 4, and 6 and solving equations	41

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Co(II)	466		8	0.2–15			Simultaneous determination of Ni, Cd, and Zn is possible	41
	Ni(II)	446		8	0.2–7.5				41
	Cd(II)	418		8	0.2–7.5				41
	Zn(II)	420		8	0.2–7.5				41
BAMT	Cu(II)	410		Acetate buffer	2–9		1:1	At pH 3:3; 1 part of Cu determined in presence 30 parts of Ni	42
	NI(II)	400		Weakly acidic	1–3.5		1:2	In PO_4 buffer 1 part of Ni determined in presence of 8 parts Cu using $S_2O_3^{2-}$ as masking agent	42
BABPT	Cu(II)	485	1.27	1.8–11.9	0.2–4		1:1	Cu determined in white metal, blends, and in wastewater	43
		530	0.82		0.5–6				43
	Hg(II)	420	1.45	4.2–9.7	2–12		1:1	EDTA masks several interferences	44
	Pd(II)	428	2.53	1.5–10	10–100		1:1	In 70% DMF	45
		425		4.7	35–275	$CHCl_3$	1:1	In presence of $NaClO_4$; for 0.3–2.3 mg, extinction is measured at 600 nm	45
	Zn(II)	440	2.15	5.5–9.9	0.15–2.5		1:1	Zn determined in wastewater from pyrite process	46
	Pb(II)	440	1.65	6.5–10	1.5–9 mg		2:3		47
	Ni(II)	460	2.28	2.5	0.5–2		1:1	In 60% DMF	48
BAMOS	Cu(II)	335		10	1–5				50
	Ni(II)	340		11.4	0.5–2.5				50
	Co(II)	330		8.6	1–8				50
BAMOT	Mn(II)	540			Up to 0.2 mg		1:2	Mn(II) is oxidized by air to Mn(III)	51
	Mn(III)	545	0.27	9.2–10.3	Up to 0.2 mg		1:2		52
	Co(II)	325	0.49	5					53
	Ni(II)	356	0.88	9.5			1:2		54
	Fe(II)	335	1.45	5.3	1–4.5		1:2		55
		410	1.45	5.3	1–8		1:2		55
	Cu(II)	345	1.06	8.5–9.5	0.5–5		1:1		56
	Bi(III)	335		2.5–3.5	8–22	IBMK	1:2	Extraction in presence of $NaClO_4$	56
	Fe(II)	507			1–20				57
BAMOPT	Mn(III)	550	0.36	10	2–12	Amyl alcohol	1:2		58
	Co(II)	345	1.77	3.8–8			1:2		58
	Ni(II)	375	1.71	5.2–10			Variable		58

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Fe(II)	350	1.76	4–9.3			1:2		58
	Cu(II)	360	1.27	8.5–9.7			1:1		58
	Bi(III)	385	1.70	3.2–5	10–100	Amyl alcohol	1:2	Co, Fe, Cu, Ni, Cd interference	59
	Cu(II)	395		4.8	10–60	Amyl alcohol			59
BAPHT	Fe(II)			Weakly acidic	0.8–11.2			Fe and Cu determined in ores, alloys, and pharmaceuticals	61
	Cu(II)			Acidic-alkaline	1–10				61
BMAPAT	Os	430			Up to 1				62
CT	Cu(II)	405	0.24	8.6	5–26	Ethyl-acetate or $CHCl_3$			63
		600	0.12		6–42				63
		400	0.20		5–31				63
1,2-CDDT	Zn(II)	415	0.73	4.3–5.2	1.1–6.6		1:1		64
	Cu(II)	467	0.57	3.8–10	2–8		1:1	Cu determined in alkaline medium, alkaline tartrate, or EDTA solution and saturated brine	65
	Co(II)	450	0.64	5.2–6.8	2.2–5.5		1:1		66
		500	0.43		2.2–8.7				
	Ni(II)	400	0.81	2.3–3.4	2.2–5.6		1:2		66
	Fe(II)	640	0.47	4.8–7	1.5–9		1:3		66
	Fe(III)	400	1.49	2.5–4.2	1–3.2		1:2		66
		480	0.53	2.5–4.2	3.2–8		1:2		66
	Pd(II)	470	1.4	6.2–8	50–290	$CHCl_3$	1:2		67
		590	0.53	1.8–3.7	185–675	$CHCl_3$	1:2		67
1,3-CDDT·HCl	Bi(III)	540	3.13	2.7–3.9	0.7–7.4		3:1		69
		550	3.30	2.1–3	0.2–1.2	IBMK	2:1		
	IO_3^-	415	1.6		1–9			Heating reaction at 70°C for 5–20 min	70
	IO_4^-	415	1.56	0.9–1.2	2–9		1:3	Method used for indirect determination of glycerol, diacetyl, or glucose	71
	BrO_3^-	415			1–8		1:2	Reaction const. is 8.4×10^{15}	72
	ClO_4^-	402	1.70	5.1–5.8N	1–4		1:3	$ClO_4^- + 5Cl^- + 6H^+ \rightleftharpoons 3Cl_2 + 3H_2O$ $3Cl_2 + 3$ Red. reagent $\rightleftharpoons 6 Cl^- +$ 3Oxd. reagent	73
	Cd(II)	510–520	1.21	Neutral	1–4		1:1	Heating at 50-60°; stability const., 1.21×10^4	74
	Cu(II)	390	5.0	6.5	0.31–0.28		1:2	Cu and Zn determined in milk, vegetable oil, and sheep liver samples	75

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Zn(II)	510	1.4	9.25	1.3–4.5		1:1		75
	Pd(II)	360	1.1	1.5–3.5	1–4		1:1		75
	Ni(II)	430	2.5	8.5–10	0.23–1.88		1:3	Ni determined in alloys; Fe, Mn are masked with triethanolamine	77
	Os(VIII)	375	1.87	3–6	0.5–9.1		1:2	Os determined in synthetic samples	78
		510	0.92	3–6	0.5–9.1		1:2	Corresponding to Russian and Colombian Pt ores	78
	Cr(V)	370	1.2	1.5–4	0.8–3.3		1:1	Cr determined in Cr alloys	79
	Pt(IV)	375	1.0	2.5–5	0.8–18.7		1:1	Pt determined in dental alloy samples	80
	Co(II)	390	1.25	6–8.5	0.3–4.4		1:3	Co determined in alloys and synthetic samples	81
1,3-CDBMT·HCl	NO_2^-	365	1.10		0.5–5		1:3		82
	Pb(II)	490	3.85		0.1–1	Isoamyl alcohol	1:1	Apparent extraction const., 6.2×10^5	83
	Zn(II)	375	3.3		0.1–2.5			Apparent stability const., 6.1×10^4	84
		493	4.8		0.02–0.30	Ethyl-acetate		Apparent extraction const., 1.4×10^4	
	U(VI)	640	1.82	5.8–8.3	1–12		1:1		86
1,4-CDDT	Cu(II)	410	0.25	8.8	8–22				87
		600	0.41	5.5	4–8	$C_6H_5NO_2$			
1,3-CPDBMT · HCl	ClO_4^-	397	1.26	$HClO_4$ medium	0.5-6			Cl^- needed for color reaction $ClO_3^- + 5Cl^- + 6H^+ \rightleftharpoons 3Cl_2 + 3H_2O$; $3Cl_2 + 3$ (red. agent) $\rightleftharpoons 6\ Cl^- + 3$ (oxidation agent)	88
	IO_3^-	415	1.08	Acetic acid medium	1-11			$IO_3^- + 2$ (red. agent) $\rightleftharpoons I^+ + 2$ (oxidation agent)	89
	BrO_3^-	400	1.8	$HClO_4$ medium					90
DBSAPT	Cu(II)	400	2.06	2.8–7.4	0.2–5	$CHCl_3$	1:1		94
	Pd(II)	399	1.47	0.74–4	0.5-6	$CHCl_3$	1:2	Pd determined in brazing filter metals	95
DCSAPT	Pd(II)	410	1.43	pH 0	Up to 8	$CHCl_3$	1:2	Pd determined in Pd catalyst (Pd-C powder)	96
	Cu(II)	410		1.5	Up to 4			Cu determined in Al alloys	97
DDBT	Co(II)	386		6.5–8.3	0.16–0.96				98
DSDT	Cu(II)	490			0.75–9		1:1		99
DAPS	Fe(III)	355	1.0	4–6	0.3–5.6			Cr(III) interferes	100
	Cu(II)	345	1.1	4-7	0.3–10.1				100
DAPT	Fe(III)	360	0.5	4–6	1.1–7.8			Stability const., 2.21×10^9	100
	Cu(II)	390	1.5	5		n-Butanol	1:1	Cupronickel analyzed	101
	Ni(II)	385	0.82	7.5		n-Butanol	1:1		101
	Co(II)	390	1.4	7.9	0.024–2.4 mg		1:1	$pK_1 = 9.6$, $pK_2 = 11.2$, and $pK_3 = 12.4$	102
	Cu(II)	400	2.0	3.6	0.025–2.5 mg		1:2		102
DBAT	Cu(II)	300		2.5-3.8			1:1		102
DBPS	Cu(II)	355	1.14	2–6	0.51–4.06				105
DBPT	Cu(II)	370	1.06	2.5–4.5	0.5–5			Stability const., 4.5×10^9	106

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
2,2'-DBPT	Mo(VI)	500	0.33	Acidic	1–20		1:1	$SnCl_2$ is necessary; many interferences removed by prior extraction with dithiazone	107
	Cu(II)	385	0.86					Binary, ternary, and quartenary mixtures analyzed	108
	Ni(II)	380	1.54						108
	Fe(III)	365	0.79						108
4,4'-DBPT	Rh(III)	430				Benzyl alcohol	1:2	Pd interferes at equimolar level	109
DPIDT	Mn(II)	594			10–90 ng			Method based on the catalytic effect of Mn(II) on the aerial oxidation of DPIDT; kinetic parameters are described	
	Mo(VI)	425	0.90	2.5–3.5	1.6–9		1:1		113
		435	0.94	2–4	5–100	Isopentyl alcohol	1:1	Extraction method is selective and sensitive	113
	Hg(II)	430	2.56	4.7	Up to 9				114
D-1,3-CDBT · HCl	IO_4^-	415	1.72	0.5–1.2	1–10				117
	Glycerol	415	0.33	5–9	2–10			5 ppm of iodate present; this is the Malaprade reaction	117
	ClO_3^-	417	1.86	$HClO_4$ medium	0.5–5				118
	BrO_3^-	415			25–100		1:2	Heating reaction mixture at 70°C for 5–20 min	119
	IO_3^-	415			25–100		1:2	Heating reaction mixture at 70°C for 5–20 min	119
DABAT	Os(VIII)	430			0.6–12				116
D-1,3-CDBPT	Cu(II)	430		2.5–3M-HCl	0.8–8			Kinetic method is based on slow rate of complexation between Cu(II) and reagent	120
D-1,2,3-CTDT	Fe(II)	550	0.89	Highly acidic medium	Up to 0.05			Fe determined in wine, food, and minerals	121
		550		Highly acidic medium	Up to 0.015	Amyl alcohol		Fe determined in water, wine, beer, and fruit juice	122
	Ni(II)	383	0.34		Up to 9.2		1:3	Stability const., 3.64×10^{12}	123
	Cu(II)	390	0.46		Up to 11.2		1:3	Stability const., 1.09×10^{12}	123
DPKT	Fe(II)	620	0.93	2.5-7.5	1–4			Fe(III) gives max at 410 nm	124
	Ni(II)	395	1.96	6–6.9 or 7.5–9.5	0.5–2.5			Interference level not listed	124
	Co(II)	415	1.0	3.8–6	1–5			Interference level not listed	124
	Cu(I)	395	1.13	3.9–4.2 or 5.5–9.2	1–5			Interference level not listed	124
	Hg(II)	380	2.08	4.7	Up to 9				125
DPKPT	Fe(II)	665	1.30	4.7	1–3	C_6H_6		Fe determined in Al and cement	126
DPGDT	Fe(II)	550	0.56	<2.5	2–9		1:2	Ethanol used to stabilize the complex; both complexes occur in intermediate pH range	127
		610	0.71	>6	2-7.5		1:1		127
		630	0.66	5–10	2–8	$CHCl_3$	1:1		127
	Hg(II)	363	2.37	4.7	Up to 9				125

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Co(II)	410	0.91	5.2	1–7		1:2	At pH 5.2 Co is not extractable while Ni is, hence 1 ppm of either is determined in presence of 5 ppm of the other	128
	Ni(II)	410	0.17	5.2	1–5	$CHCl_3$	1:1	Method applicable to determination of Ni and Co in industrial catalyst	128
DPGBPT	Fe(II)	640	0.79	4.7	1–7		1:2	In presence of ascorbic acid; Fe determined in minerals, Al, and cement	130
DPGBDPS	Ni(II)	420	5.42	4.5–10.6	0.1–1.2		1:2		131
DSDT	Cu(II)	490			0.75–9		1:1		99
EPT	Os(VIII)	430			1.5–24				62
ESBAT	Pd(II)	420			1–7			Complex soluble in 30% ethanol	132
FAT	Pd(II)	364		4.5–10				Pd determined in coatings on Ag and Cu electric conductors, baths, and wastewater	134
FAcT	Pd(II)	390	3.95		3.4–53			Pd determined in catalysts	135
	Pt(II)	390	6.25		6.2–78				135
	Pd(II)	390	0.41	2–10				Simultaneous determination is possible at two different wavelengths	136
	Pt(IV)	390	0.57	2.5-3.5					136
FPT	Pd(II)	413	0.62	3–10	2–10 μM			Pd determined in catalysts; simultaneous determination of Pd and Pt(IV) at pH 3.75 and measuring at 413 and 480 nm	137
FT	Ni(II)	360	1.54	9	0.8–3.4		1:2	Instability const., 6.5×10^{11}	138
	Pd(II)	360	1.98	6	2–4		1:2	Pd determined in palladized carbon	138
	Cu(II)	355	1.45	3	1–2.8		1:1		138
	Co(II)	365	0.80	7.5	Up to 10		1:2	F masks Mn and Fe(III); Ni interferes	139
GDT	Ag(I)	335	4.3	1–7	0.05–0.4 μM		1:1	In presence of EDTA	140
	Hg(II)	335	4.4	1.01–7	0.05–0.4 μM		1:1	In presence of EDTA	140
	Pd(II)	600	0.25	1–9.5			1:1	Method selective in presence of EDTA, in 60% DMF medium	141
	Ni(II)	405	1.2	3.5–11			1:1	In 60% DMF medium	141
GBPT	Cu(II)	513	1.18		0.3–4		1:1	Cu determined in alloys, minerals	143
	Pd(II)	635	0.32	pH 9.6–11			1:1	Pd determined in catalysts	142
		700	0.15						
HAPT	Cu(II)	360	0.95	5.5	0.42–6.6		2:3	Stability const., 6.6×10^{12}	144
	Co(II)	360	1.0	5–7.5	0.4–66			Co determined in steel samples	145
HIDDT	Ni(II)	440	0.72	5–8	0.36–6			Ni determined in steel	147
HMAPT	Cu(II)	430		0.2 M-HNO_3		$CHCl_3$	1:1		148
		640		0.2 M-HNO_3			1:2		148

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Fe(II)		645	2.2	0.5–8		1:1	Only V(V) and Cu(II) interfere	149
HMCOT	Fe(II)	700					1:3	Fe determined in homogeneous media; stability const., 3.2×10^{13}	151
HMSBPT	Mo	505	0.5		2–10		1:1	in presence of $SnCl_2$; many interferences removed by extraction with dithiazone	152
HNAPT	Au(III)	446	2.04		Up to 10	Ethyl acetate	1:3		155
	Cu(II)	412	2.25	1.8–7.1	Up to 3	$CHCl_3$		Cu determined in Al and Ag alloys	154
HPAT	Co(II)	450	0.78	1–3	1–6		1:2	Selective in acidic media	156
		450	2.35	7–9	0.3–3.5		1:2	pK values 3.48, 6.76, and 10.75	156
IDBT	IO_4^-	520	0.48	1.7–2.5	5–20				157
ITMS	Cu(II)	355	1.25		Up to 3.49		1:2	Cu determined in ferrous materials	158
	Pd(II)	360	0.72		Up to 7.66		1:1		158
ITMT	Os(VIII)	440	6.43	0.32–0.6 *M* HCl	0.056–1.35			Os determined in synthetic solution corresponding to osmiridium	159
	Cu(II)	430	1.76	0.32 *M* HCl	0.25–2		1:2	Cu determined in alloy steel, sheep liver	160
	Pd(II)	460	1.5	3	0.42–3.63		1:1		160
IT	Cu(II)	380	1.7	4.5–6.2	0.5–3	$CHCl_3$	1:2		161
LS	Th(IV)	560	0.38	2–3	0.3–3.7		1:1	In 80% alcohol medium; stability const., 2.4×10^4	162
MCPDDT	Ag(I)	430	1.0	8	0–8		1:1	Apparent stability const., 6.9×10^4	165
	Pb(II)	420	0.47	8.2	0–60			Pb can be determined in aqueous solution	166
MCHDBPT	Cu(II)	480		1–2	2–10		1:1	50–70% DMF, first-order reaction with respect to Cu	167
MGBPT	Zn(II)	455	2.1	6–8.5	0.5–4		1:1	Zn determined in drinking water	168
	In(III)	465	2.71	3.5–6			1:1	60% DMF; 1:2 complex in basic medium	169
MNQT	Os(VIII)	470	5.76		0.5–3.3	Isoamyl alcohol	1:2	Only Ru(III) and SCN^- interfere	171
	Pd(II)	500	4.2	8.2–9.5	0.3–2.29		1:1		172
MPAT	Hg(II)	363	2.28	4.7	Up to 9				125
	Os(IV)	480				Benzyl alc.	1:2	5-Fold excess of Pt metals do not interfere	173
	Os(IV)	460	1.3	1.5			1:2	In homogeneous solution in presence of reductant	173
	Os(VI)	420	0.93	1.5			1:2	In homogeneous solution	173
	Cu(II)	410	2.6	9.15–10.1	1–7		1:1		173
	Pd(II)	420	0.43	2	3–16		1:1	Absorbance measured after 1 h	174
	Rh(III)	430	0.92	2	1–10		2:1	90 min heating needed	174
MPKT	Re(V)	430	0.455	2.4–6 *M* HCl	36-388 μM		1:1	Re(VII) reduced to Re(V) by $SnCl_2$	175
MTKT	Ni(II)	375	1.0	8–9	1.2–4.1		1:2	In 40% alcohol medium	177
MSAS	Fe(III)	550	0.53	4–5.7			2:1		176
MSAT	Fe(III)	590	0.50	2–5.1			1:2		176
1,2-NQT-4S	Cu(II)	555		Acidic	2–20		1:2		178
	Zn(II)	520	2.0	6–7			1:2	Formation const. determined	183

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Cd(II)	520	1.9	6-7			1:2		183
	Cu(II)	550	1.33	3–7			1:2		183
	Pb(II)	540	1.56	6–7			1:1		183
1,2-NQS-4S	Zr(IV)	510–520	2.7	2–3	Up to 3.9		1:2		286
	Th(IV)	490-500	2.44	2.8–4.5	Up to 13		1:2		286
	UO_2(II)	500	1.78	4.8–6.2	1–21		1:2		286
	Sc(III)	500	3.71	5–7.3	Up to 1.35		1:2		286
	Y(III)	490	3.13	6.7–8.3	Up to 2.9		1:2		286
	Pb(II)	500	1.72	5.5–7.3	Up to 8.5		1:1		286
	Bi(III)	515		Acidic	16–133		1:1		179
NDIDDT	Co(II)		1.50		0.24–2.4			Co determined in alloys	187
	Pd(II)		1.60		0.42–3.8				187
	Os(VIII)		1.30		0.76–7.6				187
NFAS	Hg(II)	420–480		3.5–7.5	Up to 0.2 m*M*			Method applicable to ore analysis	188
	Hg(II)	420		3.5–7.5	1×10^{-6}–2×10^{-4} *M*		1:2	Instability const., 2.75×10^{-10}	189
		420		10.5			2:3	Instability const., 3.71×10^{-10}	189
NSAPT	Cu(II)	390	2.5	2.3–6.2	Up to 2.5	$CHCl_3$		Applicable to analysis of Al and Mg alloys	190
OCT	Cu(II)	465	0.55		0.12–11		1:4	Cu determined in Al alloys and Sn-based white metal	191
	Fe(II)	516	0.60	1–1.5	1–8.5		1:3	Fe determined in Portland cement, magnesite, spinach, and lentil	196
	Mn(II)		0.30	Alkaline medium	9–400		1:3	Mn(II) determined in Lincolnshire iron ore	193
ODDT	Co(II)	400	1.0	Acidic medium	0–80	Isoamyl alcohol	1:3	Simultaneous determination of Co, Fe, and Co in steels	195
	Fe(II)	565	0.98	Strongly acidic	0–22	Isoamyl alcohol		Fe determined in wine, foods, minerals, and water	196
	Cr(VI)	485	0.56	Strongly acidic	Up to 9.5			Simultaneous determination of Cr and Fe; Cr and Fe determination in ceramic samples	197
	IO_4^-	400		Strongly acidic	0.24–5				198
	BrO_3^-	400		Strongly acidic	0.16–3.6				198
PQMT	Cu(II)	540	1.16	4.8–5.5	0.25–3.5	$CHCl_3$	1:2	Cu determined in steel, brass, gunmetal, Ni-Ag, and Al alloys	200
	Ni(II)	520	2.52	7–9.2	0.43–2.1		1:3	Ni determined in Brightray G wire	201
	Fe(II)	510	1.3	6.1–7.7	Up to 3.9			Fe determined in alloys; soluble in CH_3OH	202
	Fe(III)	510	0.83	8.1–8.9	Up to 6.8			Soluble in DMF	202
	Pt(IV)	495	1.63	0.8–4.4	0.75–15	$CHCl_3$	1:2	Complex extracted into molten naphthalene, solidified, and dissolved in $CHCl_3$; Pt determined in synthesized mixture	203
	Zn(II)	510	3.52	7–8.6	0.22–1.7		1:3	Zn determined in 6% Zn-Al alloy	204
	Pd(II)	590	0.42	Low pH	3.4–18.2			Both complexes soluble in DMF	205
		540	1.27	High pH	1.3–6.7				205
	Co(II)	500	1.20	6.8–8.5	0.8–4	Water-methanol	1:3	Co determined in vitamins, alloys, and ores	206
PhADT	Co(II)	385	0.56	8–10.5	Up to 11		1:1		207

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Ni(II)	400	0.80	7.5–9	Up to 4.5		1:1		207
		450		8.8	Up to 4.5	$CHCl_3$	1:1	Selectivity improved by extraction	207
	Pd(II)	405	1.10	3–4.5	5–25		1:1	In 50% DMF medium	208
PIDT	IO_4^-	410	2.84	1.6–2.7	1–60		1:3		157
	Pt(IV)	450	1.50	4–6.5	1–10		1:1	Pt can be determined in presence of equal amount of Au(III) by adding 10% NaCN before complexation	209
	Rh(III)	420	3.10	6–7			1:1	20 min heating	210
	Pd(II)	415	2.80	5.2–6.2	1–3.5		2:3		210
	Bi(III)	430		2.2		$CHCl_3$		3-Fold excess of Zn does not interfere	211
	Zn(II)	450	1.20	8–10	25–100		2:3	λ_{max} depends upon foreign ion present	211
	Os(VIII)	450	1.3	3.3–4.5	1–12		1:1	Oxidation of PIDT with Ce(IV) catalyzed by Os(VIII), allows a sensitive determination of Os	212
	Ni(II)	440	1.13	10	0.11–3.21		1:1	Stability const., 6.26×10^9	213
PAS	Cu(II)	350		9.5	0.3–1.5				50
	Ni(II)	350		10	0.2–1.2				50
	Co(II)	345		10	0.5–2.2				50
PAT	Co(II)	356	1.40	5.6–9.5			1:2		214
		410	0.74	5.6–9.5					214
	Fe(II)	610	0.58	9.4–12.3		$CHCl_3$	1:3		215
	Fe(III)	360	1.4	3.9–7.9			1:2		215
	Ni(II)	385	1.9	7.2–10.7	Up to 5		1:2	Cu can be determined at pH 2.8 and the sum of Ni and Cu at pH 9.6, the content of Ni being found by difference	216
	Ag(I)	316	1.3	5–6			1:1	In presence of EDTA	218
	Hg(II)	363	2.38	4.7	Up to 7				125
PAPT	Fe(II)	625	0.64		1–6	C_6H_6	1:2	Blue-green complex changes to red at pH 3	221
	Fe(III)	390	3.56		0.2–1.8		1:2	Fe determined in water samples	221
	Ni(II)	390		8–9	0.8–1.5		1:2	Determination of Ni and Co in mixtures	222
		400		8–9	0.8–2	$CHCl_3$	1:2		222
	Co(II)	390	2.99	4.4–6.9	0.2–2		1:2		223
	Co(II)	430	1.51	1.0	0.2–2		1:2	1 ppm Co determined in presence of 1000 ppm Fe; Co determined in steels	223
	Co(II)	400	2.95	4.7	0.2–2	$CHCl_3$	1:2	In presence of $NaClO_4$	223
PADS	Cu(II)	380	1.8	4–6.5	0.14–2.88 ng		1:1	Ni and Co at 3-fold and Fe at 10-fold level interfere	224
PT	Co(II)	440		4.7	1–4			Pd, Au(III), and V(V) interfere	225
	Fe(III)	430		4.7	0.5–4			Ag, Zn, Au(III), and V(V) interfere	225
PyAT	Os(VIII)	430			1.5–2.4				62
QAT	Ni(II)	460	1.58	7.5	5–25	$CHCl_3$	1:2	Extracted from aqueous pyridine solution	227
	Cu(II)	430	1.33	7.5	0.5–2.5	$CHCl_3$		Dissociation const., 3.89×10^{-10}	228

TABLE 2 (continued)
Spectrophotometric Data

Compound	Ion	λ_{max} (nm)	10^4 l mol^{-1} cm^{-1}	pH[a]	Limit of determination (ppm)[a]	Extraction	M:L	Remarks	Ref.
	Fe(II)	600	0.39	7.5	0.5–6	$CHCl_3$		Dissociation const., 5.32×10^{-7}, Cu and Fe determined in steel, brass, Ni-Ag alloy	228
SAS	Cu(II)	685		3–5	1–20 mg/50 ml				229
	U(VI)		7.9	3.7–3.9	0.001–2.5		1:4	U determined in ore samples in presence of Fe	231
	UO_2(II)	230		3.8	0.001–2.5		1:4		232
SAT	Mo(VI)	520	0.48	1.5	1–18		1:1	In presence of $SnCl_2$; Mo and Fe can be determined in presence of each other; used in analysis of Ni-Cr, Mo-Fe alloys	233
	Fe(III)	590	0.17	10	1–28		1:2	Fe can be determined in 2-fold excess of Mo	233
	Co(II)	400	1.10	4.5–6	0.3–6		1:2	Co determined in steel after removal of Fe with PO_4	234
	V(V)	375	0.43	0.06–0.08 *M*	0.5-6.5	n-Butanol	1:1	Stability const., 1.8×10^5	235
	Fe(III)	370	1.2	4.5–5.5	0.3–4.5		1:2	Stability const., 0.64×10^{10}	236
	V(V)	370	0.63	5–6.5	0.5–4		1:1	Heating and extraction not needed; stability const., 2.1×10^5	237
	Ni(II)	370	0.98	6.5–7	0.4–4		1:1		238
	Cu(II)	375	0.92	5–7	0.5–6			*Cu determined in* plant samples containing Zn, Fe, and Mn	239
	Hg(II)	363	1.69	4.7	Up to 9				125
	Rh(III)			0.1–0.3 *M* KOH	0.45–4.2			Complex formed on heating at 95°C; Rh determined in alloys and catalysts; analog derivative spectrum increases sensitivity 10-fold	241
	Pd(II)	405	0.64	1.8–4	1–7.2	Cyclohexanol	1:1	*Pd determined in* hydrogenated catalysts, dental alloys, and jewelry	240
	Cu(II)	364	2.0		0.25–7.5		1:1	In 20% acetone; Cu determined in Al and Zn, respectively	243
	Pt(II)	388	0.65	3.5	0.5–8	n-Butanol	1:1	Pt determined in alloys and catalysts	242
SAPT	Co(II)	390		4–6			1:2	Stability const., 1.1×10^{10}	244
	Ni(II)	380		4.5–8.5			1:1	Stability const., 2.5×10^6	244
	Cu(II)	395		4–7			1:1		244
SDMOT	Pd(II)				0.09–3.6		1:2	Stability const., 8.33×10^{10}	245
SBPT	Fe(II)	620	0.90	5–12			1:2		246, 247
	Fe(III)	380	1.24	5–8			1:2		246, 247
	Co(II)	370	1.2	2–10.5			1:2	pK values given	246, 247
	Ni(II)	400	2	6–10			1:2		246, 247
TAT	Cu(II)	372	3.9	4.5–8.2	0.2–1.2	$CHCl_3$		$pK_1 = 10.6$	248
TPGBPT	Fe(II)	640	0.79	4.7	1–7		1:2	Fe determined in minerals, Al, and cement	249
VT	Mo(VI)	470	1.98	HCl medium	0.1–4.6	$CHCl_3$	1:4:2 Mo:SCN:VT		250

[a] Unless otherwise indicated.

VI. FLUORIMETRIC STUDIES

Salicylaldehyde SC[264] gives a fluorescence reaction at pH 5.6 with salts of scandium, the sensitivity of which is 0.01 μg/5 ml. SAT[261,265] has been used for the fluorimetric determination of gallium.

A kinetic method is described for the fluorimetric determination of trace amounts of Mn(II)[266] and Fe(III),[267] based on their catalytic effect on the oxidation of 2-hydroxybenzaldehyde thiosemicarbazone (BAT) by H_2O_2. The reaction is followed by measuring the rate of change of fluorescence (λ_{ex} 365 nm and λ_{em} 440 nm). The calibration is linear over an Mn range of 2 to 9 ng/ml and an Fe range of 10 to 60 ng/ml. The simultaneous spectrofluorimetric determination of Fe(III) and Mn(II),[268] based on the different reaction rates resulting from the catalytic effect of these two metal ions on the oxidation of 2-hydroxy-BAT has also been reported. An indirect kinetic determination of Ag[269] is based on its inhibitory effect on Fe(III)-catalyzed oxidation of 2-hydroxy-BAT. The calibration is linear over the Ag range of 50 to 400 ng/ml.

Moreno et al.[270] reported on the fluorimetric control of semiautomatic catalytic titrations. This technique was applied to the direct determination of EDTA (2.25 to 3.75×10^{-5} *M*), which acts as an inhibitor of the oxidation of 2-hydroxy-BAT by H_2O_2, catalyzed by Fe(III).

A kinetic method is reported for the determination of trace Mn(II) based on its effect on the oxidation of 3-hydroxy-2-pyridinecarboxaldehyde TSC[271] by H_2O_2. The Mn calibration curve for this reaction was linear over the range of 5 to 50 ng Mn/ml. The determination of 1,2-diaminocyclohexanetetraacetic acid[272] involves dissolution in EtOH, addition of 2-hydroxy-BAT, H_2O_2, NH_3, and Mn^{2+}, and measurement of the resultant fluorescence (excitation 365 nm) at 440 nm. The same principle can be used for determining trace amounts of Mn^{2+} or Ni^{2+}.

Holzbecker and Lipska reported on the effects of surfactants on the fluorescence spectra of Al and Zn[273] complexes of SAS in aqueous alcoholic solution, and on their extractability into ethylacetate. The complexes are extractable into ethylacetate in the presence of anionic surfactants (sodium lauryl sulfate). Optimum conditions for the extraction and fluorimetric determination of Al and Zn have been evaluated. Gallium in Al metal has been determined fluorimetrically at 445 nm as the Ga-HBAS complex, after the extraction of Ga with isopropyl ether.[274]

A fluorimetric method for the determination of microamounts of Ga[275] with HMBAS was developed and applied to the analysis of Ga in Al and Al alloy. The method was extended to the determininationi of Sc in mineral samples.[276] A fluorimetric method for the determination of trace Sc[277] in minerals and Ga[278] in Al metal using HNAS also has been reported.

Uses of these compounds in fluorimetric analysis are summarized in Table 3.

VII. USES AS INDICATORS

One of the most common applications is the direct titration of metal against EDTA solution using SCs and TSCs as indicators. Gandhi and Lahiri[279] used four phenolic TSCs for direct titration of Fe(III) with five polyaminopolycarboxylic acids as titrants. The sodium salt of 1,2-NQT-4-sulfonic acid-2-phenylthiosemicarbazone was used as a sensitive indicator for the determination of Bi, Cd, Pd, and Zn with EDTA; the color changes from raspberry to yellow at the endpoint.[280] Methyl-2-pyridylketone TSC[281] was used as an indicator for the titration of Re(VII) with Sn(II). The method was also used to determine ≈9% of Re in wire containing Fe and Ni. Phenanthraquinone monothiosemicarbazone[284] was used to determine Cu in brass and gunmetal, and Ni in monel K and Brightway G. The addition of acid or alkali has no effect on the complex formation with *o*-thymoldosemicarbazone.[285] Phosphate, molybdate, tungstate, and sulfate were titrated with lead nitrate using 1,2-NQT-2-semicarbazone as an indicator.[287] A number of metal ions and anions have been determined using 1,2-NQT-2-thiosemicarbazone-4-sulfonic acid.[286,288] Aluminum and copper were determined together by breaking the Al-EDTA complex by addition of NaF and titrating the released EDTA with zinc.[286] Iodide has been determined by mercurimetric titration in isopropyl alcohol with the same reagent.[286]

1,2-NQT was proposed as an acid-base in-

TABLE 3
Fluorimetric Applications

Compound	Metal	pH	Excitation max. (nm)	Fluorescence max. (nm)	Range of determination	M:L	Remarks	Ref.
DBAS	Sc	6.0	360	425			0.002–0.3% of Sc determined in alanite, mica, etc.	251
	Be	9.7	343	430	0.4–200 ng			252
	Sc	6.0	360	425	0.2–400 ng			252
	Al	5.5	353	413			Al determined in seawater samples	253
HNAS	Al	5.9	365	435	0.005–5		Al determined in limestone	254
HNAT	Ga	3.2	412	472	1 μ*M*	1:1	10-fold excess of Al did not interfere	255
SAS	Sc		370	455	0.002–0.02 ppm	1:1		256, 257
	Zn	6.1–6.4			Up to 3 μg	1:1	0.002–0.004% of Zn(II) was determined in $CdSO_4$ and $CdCl_2$, after separation of Zn(II) as thiocyanate into isoamyl alcohol	258
	Ga	2-6	365	450			Ga determined in Mg base alloy, semiconductors, and silicate rocks	259
	Ga	4.5	365	440			Ga (0.0001%) was determined in Al metal and Al alloys	260
	Ga	3.5			2.5–65 μg	1:2		261
	Al	5.4			3.5–17 μg	1:2		261
	Y	6.9			15–125 μg	1:2		261
	In	5–5.5	365	450	0.7–10 μg			263
SAT	Ga	3.7–4.2	365	450	0.1 m*M*		~0.3–6% of Ga determined in Mg-Ga, Mg-Ga-Tl, or Mg-Ga-Hg alloys, and in silicate rocks	262

dicator in isopropyl and tertiary butyl alcohol medium.[335]

Uses of these compounds as indicators are summarized in Table 4.

VIII. USES AS GRAVIMETRIC REAGENTS

Only a few SCs and TSCs have been used in the gravimetric determination of metal ions. Cano Pavon et al.[289] used TAT and benzaldehyde thiosemicarbazone (BAT) as gravimetric reagents for the determination of Pd(II) in the 10 to 50 mg range. Precipitation is carried out at pH 3 to 6, and, after aging 2 h, the precipitate is collected, washed, dried at 60° to 120°, and weighed as the 1:2 metal:ligand complex.

Hovorka and Holzbecker[290] used SAT to precipitate Cd as the yellow crystalline $Cd(C_8H_8ON_3S)_2$ (containing 22.4% Cd) from nitrate or sulfate solution. Precipitation is incomplete from solutions containing chloride, fluoride, or oxalate.

In another study, 2,4-DBAT has been employed for the estimation of Cd[291] and Ni.[292] In the Cd estimation (5 to 100 mg) at pH 6.5 to 8.5 Fe^{3+}, Al^{3+}, Cr^{3+}, CN^-, citrate, traces of F^- and

TABLE 4
Use as Indicators

Compound	Ion	pH	Limit of determination	Color change	Remark	Ref.
NFAS	Hg(II)		1 mg	Orange to yellow	EDTA direct	282
HMAPS	Fe(III)	2.0	Up to 100 mg	Green to colorless	EDTA direct	283
HMAPT	Fe(III)	2.0	1–70 mg	Bluish green to colorless	EDTA direct	283
PQMT	Cu(II)	0.5–4.0	1 μg–30 mg	Red to colorless	EDTA direct, also used for Cu in brass and gunmetal	284
	Zn(II)	4.4–7.5	65 μg–65 mg	Red to colorless	and Ni in monel K and Brightway G	284
	Cd(II)	4.8–6.0	20 μg–110 mg	Red to colorless	EDTA direct	284
	Hg(II)	5.3–6.8	40 μg–100 mg	Red to colorless	EDTA direct	284
	Ni(II)	4.5–7.0	12 μg–60 mg	Red to colorless	EDTA direct	284
TAS	Fe(II)	Addition of		Deep red to colorless	EDTA direct	285
	Fe(III)	NaOH		Deep violet to colorless	EDTA direct	285
	Ni(II)	or HCl		Yellowish green to colorless	EDTA direct	285
	Cu(II)	has no		Deep green to blue	EDTA direct	285
	Co(II)	effect		Yellow to colorless	EDTA direct	285
	UO_2(II)	on com-		Blood red to light yellow	EDTA direct	285
	V(V)	plex		Deep mustard to greenish yellow	EDTA direct	285
NQS-4S	Pb(II)	5.5–6.8	0.1–207 mg	Purple to greenish yellow	Hexamine/HNO_3 buffer, EDTA	286
	Bi(III)	1.7–2.8	0.1–20 mg	Purple to greenish yellow	Dil.NH_3/HNO_3 buffer, EDTA	286
	PO_4^{3-}	6.0–7.3	0.5–28.5 mg	Greenish yellow to pink	Pyridine/HNO_3, or hexamine/HNO_3	287
	MoO_4^{2-}	6.0–7.0	1–80 mg	Greenish yellow to pink	Pyridine/HNO_3 or hexamine/HNO_3	287
	WO_4^{2-}	6.0–7.0	12–50 mg	Greenish yellow to pink	Titration with $Pb(NO_3)_2$	287
	SO_4^{2-}	4.8–5.5	1.7–6.9 mg	Greenish yellow to pink	Titration with $Pb(NO_3)_2$ in 30% methanol medium	287
	I^-	2.5–4.5	0.6–4 mg	Purple to yellow	Titration with $Hg(NO_3)_2$ in isopropyl alcohol medium	286
NQT-4S	Ni(II)	5.0–6.5	0.6–30 mg	Yellow to purple	EDTA direct, nonreversible AcOH/HAc	286
	Cu(II)	3.7–6.8	0.6–15.8 mg	Yellow to purple	EDTA direct, nonreversible AcOH/HAc	286
	Zn(II)	5.5–6.8	0.6–65 mg	Yellow to purple	EDTA direct, nonreversible AcOH/HAc	286
	Cd(II)	5.5–6.8	1–56 mg	Yellow to purple	EDTA direct, nonreversible AcOH/HAc	286
	In(III)	4.0–6.0	0.2–36 mg	Purple to yellow	NaAC/HAC, EDTA direct	288
	Pb(II)	5.5–6.8	0.1–207 mg	Purple to yellow	Hexamine/HNO_3, direct	288
	Bi(III)	1.7–2.8	0.1–105 mg	Purple to yellow	Hexamine/HNO_3, direct	288
	Al(III)				Detected by back-titration with Zn(II)	288
	Ga(III)				Detected by back-titration with Zn(II)	288
	Fe(III)				Detected by back-titration with Zn(II)	288
	Al + Cu				Al-EDTA complex broken by NaF; and released EDTA titrated with Zn(II); also Al in bauxite	288
	Al + Fe					
	Al + Ga				Al-EDTA complex broken by NaF; and released EDTA titrated with Zn(II); also Al in bauxite	288
NQT-4S (cont.)	PO_4^{3-}	5.0–7.0	0.5–28.5 mg	Yellow to purple	Hexamine/HNO_3, titration with $Pb(NO_3)_2$	287
	MoO_4^{2-}	5.0–6.0	1–80 mg	Yellow to purple	Hexamine/HNO_3, titration with $Pb(NO_3)_2$	287
	WO_4^{2-}	6.0–7.0	12–50 mg	Yellow to purple	Hexamine/HNO_3, titration with $Pb(NO_3)_2$	287
	I^-	2.5–4.0	0.6–4 mg	Yellow to purple	Isopropyl alcohol, titration of I^- with Hg(II)	286
	$Fe(CN)_6^{4-}$	0.3–2 ml of 0.2 *M* hexamine	2–42 mg	Purple to yellow	$(NH_2)_4SO_4$ or KCl or K_2SO_4 necessary, titration of standard Zn(II) solution with ferrocyanide	286

TABLE 4 (continued)
Use as Indicators

Compound	Ion	pH	Limit of determination	Color change	Remark	Ref.
		2–6 ml of 0.2 *M* hexamine	1.6–159 mg	Purple to yellow	NH_4Cl or $(NH_4)_2SO_4$ necessary; titration of standard Cd(II) solution with ferrocyanide	286

>2.5 mg/ml of other halides interfere. In the estimation of Ni (5 to 60 mg) the green complex, $(C_8H_8N_3O_2S)_2$ Ni·$6H_20$, was precipitated in the pH range 5 to 6.5. Komatsu and Hiroaki[293] used a 0.1% ethanolic solution of ESBAT for the gravimetric determination of Hg^{2+} in acidic medium (<2.5 N); it gives a yellow precipitate, which is washed with 1 *M*-HCl and dried at 110° to 120°. This method is also used for the estimation of Pd(II),[294] giving an orange-yellow precipitate in 5 *M*-HCl, washed with 1% HCl and water, and dried at 110° to 120°.

FAT has been used by Cano Pavon and Pino[295] as a gravimetric reagent for palladium at pH 2 to 6. The orange precipitate is washed with 10% aqueous ethanol and dried at 60° to 120°. They claim that the use of this reagent is preferable to either dimethylglyoxime or 2-furaldoxime. The same authors[296] used 2% ethanolic solution of FAPT for the gravimetric estimation of Pd in the pH range of 3 to 5.

BAMOS was used for estimating Pd[297] (10 to 35 mg) in 1 *M* HCl solution. The reaction mixture is heated for 45 min at 50°, the precipitate is collected, washed, dried at 110°, and weighed as $(BAMOS)_2$ Pd.

Laly and Parameswaran[298] used SAT for the estimation of Ni in the pH range 6.5 to 8.2. The reddish-brown precipitate was dried at 100° to 120° and weighed as $(C_8H_8N_3OS)_2Ni$. The method was applied for the determination of Ni in stainless steel.

IX. POTENTIOMETRIC STUDIES

5,5'-Dimethyl-1,3-cyclohexanedione bis (TSC)[299] forms a very stable 1:1 complex with Hg(II) in acid medium. Hg can be determined potentiometrically in the range 10^{-4} to 10^{-3} *M* with high precision ($\sigma = \pm 1.24 \times 10^{-3}$) and accuracy (0.22%). Bhatt et al.[300] studied the complexes of HNAPT with Cu^{2+}, Ni^{2+}, Co^{2+}, and VO^{2+} by pH-titration. The proton-ligand stability constants were determined by several methods in 70% dioxan medium. All four metals formed 1:2 complexes. The order of stability constants was found to be $VO^{2+} > Cu^{2+} \simeq CO^{2+} > Ni^{2+}$.

Complexation by SAPT was also studied potentiometrically by Bhatt et al.[301] at 25°C and ionic strength 0.1 in 50% aqueous dioxan. The proton-ligand stability constant was 7.94×10^9, and the log β values for the complexes were 9.44, 10.28, 9.84, and 8.94 for VO^{2+}, Cu^{2+}, Co^{2+}, and Ni^{2+}, respectively. Similar studies were carried out on HMAPT complexes[302] of Cu(II), Mn(II), Zn(II), and Ni(II) at 40° in water-dioxan (1:3). The observed order of the stability constants was Zn < Cu > Co > Ni > Mn.

Lazaro et al.[303] studied the pH metric determination of BAT, SAT, and *p*-hydroxybenzaldehyde TSCs with Hg(II) in excess, and the titration of Hg(II) with the last-named reagent in excess. With Hg(II) in excess, *p*-hydroxybenzaldehyde TSC releases two protons from each molecule of ligand, whereas SAT and BAT release only one proton from each molecule under the same conditions. Lazaro et al.[304] used iodide ion-selective and amalgamated gold electrodes for the titration of BAT, SAT, and HBAT using Hg(II) as titrant. The apparent formation constants are reported.

Titration of seven TSCs by complexation with cupric ion (and vice versa by use of a copper-selective electrode) in an acetone-water mixture has been carried out by Argüeso et al.[305] The conditional formation constants for these chelates were determined.

A potentiometric titrimetric method for the determination of TSC[306] also was developed, based on the titration of the protons released upon

reaction of the TSCs with $Cu(NO_3)_2$ in water-methylcyanide medium with $NaHCO_3$. In addition, a potentiometric method for the determination of 2-hydroxycyclohexanone TSC[146] using a copper ion-selective electrode was elaborated.

X. OTHER USES

Complexes of BABPT[307] with divalent metal ions (Zn, Cd, Pb, Ni, Cu, and Hg) were separated using reversed-phase high performance liquid chromatography (HPLC) with aqueous acetone containing sodium acetate as the mobile phase. Niederschulte and Ballschmiter[308] and Ballschmiter[309] have studied BABPT and GBPT as reagents for the separation of metal ions in the form of their chelates by thin-layer chromatography (TLC) on aluminum oxide and by liquid chromatography (LC). The Cu and Hg complexes of QAT[310] were separated by TLC. Pt group metal ions were quantitatively separated by sorption on ion-exchange resin containing thiosemicarbazide, followed by selective elution.[311]

Complexes of Mn(II) and Mn(III) with BAMOT have been investigated polarographically.[312] Verma et al.[313] reported polarographic studies on the complexes of Ni(II), Cu(II), and Zn(II) with 1-benzylidene-4-(2-pyridyl) TSC. Amperometric determinations of Hg(II) with 5,5′-dimethyl-1,3-cyclohexanedione bis(TSC) hydrochloride (D-1,3-CDBT·HCl)[314] have been proposed. In acidic medium, three waves (one cathodic and two anodic) can be distinguished on the polarographic curve for D-1,3-CDBT·HCl. The half-wave potentials are approximately -1.20, -0.10, and $+0.20$ V vs. SCE. Corresponding electrode processes for these waves have been proposed. Voltammetry with a Pt electrode gave a wave with a peak at 1.00 V vs. SCE due to the oxidation of 5,5′-D-1,3-CDBP. Polarography of the perchlorate of this TSC[315] showed only one anodic wave. There is evidence for the formation of two compounds with Hg(II): one stoichiometric (1:1), and one unidentified with a higher Hg content. Based on these results an amperometric method for determination of Hg(II) was proposed. Linares et al.[316] studied PAT, DPKT, and BPT by differential pulse polarography with the aim of elucidating the influence of the ring bonded to the TSC group, on its electroreduction. Nieto et al.[317] studied the polarographic behavior of BPT.

In the presence of cyanide and tartrate, manganese can be detected by a spot test[318] on paper by spraying with an ethanolic solution of BAMOT. A 0.5% solution of resorcyl-4-aldehyde thiosemicarbazone[319] in 55% acetone is suitable for the detection of Fe^{3+}, Co^{2+}, Hg^{2+}, Ag^{+}, Mn^{2+}, and Mo^{6+}.

Acid-base equilibrium of GBPT[320] has been reported. SAS[321] was used as an acid-base indicator and as a reagent for the determination of Zn using a luminescence method. SAS, SAT, and their 5-chloro derivatives were tested as reagents for the determination of Ga by luminescence.[322,323] The detection limits are better with semicarbazones, but the selectivity is superior with the corresponding TSCs.

Solvent extraction of Ag(I) into ethylacetate with DAPT was studied with 110 m Ag as a tracer.[104]

Vlacil et al. used SAT to study the synergistic extraction of Ni(II)[324] and Mn(II)[325] with respect to tributylphosphate. In addition, the solvent extraction of Co(II) with 2,4-DAPT has been reported by Reddy et al.[103] Silva and Valcarcel[326] reported on the liquid-liquid extraction of Zn and Cd with 1,2-NQT into methylisobutyl ketone and their simultaneous determination by atomic absorption spectrometry (AAS). They also used the same reagent for the determination of Cu[327] in waters, food, and analytical reagents, again using liquid-liquid extraction combined with AAS. Wang[328] studied the extraction of Cr(VI) with thiosemicarbazide and its subsequent determination by AAS. Takada and Fujita[329] employed thiosemicarbazide as a masking agent for the direct determination of Bi in copper metal by the atomic absorption method.

Molybdenum in natural water was determined by Ternero and Garcia.[330] The method involves the preconcentration of Mo on a Chelex-100 chelating resin and subsequent elution with NH_3 solution, followed by extraction with the complexing agent DPIDT dissolved in DMF:isoamyl alcohol (1:4), and a final, direct determination by AAS. The addition of ascorbic acid prior to extraction eliminates the interfering effect of several ions at the concentration level that are normally found in natural water.

Stankoviansky et al.[331] have used 2,4-DBAT for potentiometric, spectrophotometric, and polarographic studies. The data obtained were discussed in relation to (1) the calculation of pK values for three dissociation constants; (2) the dependence of the absorption maximum (at 330, 352, and 375 nm) on pH (2.5 or 12.1) in titrations with NaOH; and (3) cathodic waves characteristic of the reduction stages between pH 0.5 and 10.0.

AST[332] was used for the determination of Pt(II) in the range 0.05 to 8.0 m*M* by d.c. polarography. A reversible, diffusion-controlled, two-electron reduction wave was obtained when AST quantitatively reacted with Pt(II) in 0.1 to 0.25 *M* HCl to form a yellow complex. Various polarographic parameters were examined. An indirect polarographic method for the determination of Cu(II) has been reported[333] based on the highly stable complex formation between Cu(II) and EDTA.

An indirect differential pulse polarographic determination of Pt(II) using SAT also has been reported.[334] This method was applied to the determination of dental alloy, dehydrogenated catalyst, and electrical materials.

XI. CONCLUSIONS

This systematic study leads to the conclusion that approximately 90% of the reagents discussed have been employed in spectrophotometric analysis. This confirms the general belief that these reagents are good chromogenic reagents for metal ions, probably due to the strong chelating ability of the hydrazine nitrogen atom (>C=N) and thioketone sulfur (>C=S).

A statistical examination of the spectrophotometric reagents covered in this article for the determination of metal ions indicates that 50% of the reagents have been used in the determination of Cu (followed by Ni (30%), Fe (27%), Co (25%), Pd (19%), and Os (10%), which leads to the conclusion that many of these reagents are promising chromogenic agents for Cu. In all, 27 metal ions were determined using these reagents; however, there is only one reagent each reported for Zr, Sc, Y, Ru, Ir, Re, In, and Th.

Finally, a survey of Table 3 reveals that SCs and TSCs zones containing an −OH group in the ortho-position are good fluorimetric reagents.

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