ORGANIC REAGENTS FOR SPECTROPHOTOMETRIC DETERMINATION OF ANIONS

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INTRODUCTION

Organic reagents have a remarkable versatility for inorganic and organic analysis. The most important among the organic reagents are those which form 'chelates' with the metal ion. The chelating agents should possess two functional groups which may be either acidic, coordinating or a combination of both. The acidic character may be due to the presence of a hydroxy (-OH), mecapto (-SH), or imino (=NH) group, including forms such as enol ($\text{HO}-\overset{\text{O}}{\underset{|}{\text{C}}}=\text{CH}-$), oxime (=N-OH), acid nitro (=N-OH), sulphinic (-SO-OH), sulphonic (-SO₂-OH) or their thio analogue. On the other hand, groups like carbonyl (>C=O), thio (>C=S), primary amino (-NH₂), secondary amino (-NH-) or tertiary amino (-NR'), nitrosyl (-NO), etc. coordinate by virtue of the lone pair of electrons on the N, S or O atoms. Systematical study of organic reagents containing the aforesaid groups for the development of analytical methods for anions is scanty. Generally the organic reagents have been used widely for anions by indirect methods, either forming ion pair complexes or adding to the metal complexes to make metal chelates, from which the anion concentration is determined. A large number of organic reagents have been used for the extraction spectrophotometric determination

of several anions. The use of organic reagents for the determination of various anions has been summarised in Tables 1-8 / 1- 254/. Some frequently used spectrophotometric methods for the most common anions are discussed in the subsequent paragraphs.

SUMMARIZING TABLES FOR VARIOUS ANIONS

In Tables 1-8, the following abbreviations are used:

Ac ₂ O	Acetic anhydride	EtOH	Ethyl alcohol
AcOH	Acetic acid	ex	excitation wavelength
BG	Brilliant Green	HDTMA	hexadecyl trimethyl ammonium
BPG	Bromopyrogallol	IAA	Isoamyl alcohol
BuAc	Butyl acetate	MB	Methylene Blue
iBuAc	Isobutylacetate	Me	Methyl
CA	Chromotropic acid	Me ₂ CO	Acetone
CAS	Chrome Azurol S	MePh	Toluene
CP	Cetyl pyridinium	MG	Malachite Green
CPB	Cetyl pyridinium bromide	MIBK	Methyl isobutyl ketone
CPC	Cetyl pyridinium chloride	MTB	Methyl Thymol Blue
CTMA	Cetyl trimethyl ammonium	NaHMP	Sodium hexametaphosphate
CV	Crystal Violet	OAc ⁻	Acetate
D2EHP	bis (2-ethyl hexyl) phosphate	PAR	4,2-pyridylazoresorcinol
DMF	Dimethylformamide	PhCl	Chlorobenzene
DMSO	Dimethyl sulfoxide	Phen	1,10,phenanthroline
EBT	Eriochrome Black T	PV	Pyrocatechol Violet
ECAB	Eriochrome Azurol B	SPACA	3-(4-sulphophenylazo) chromotropic acid
ECR	Eriochrome Cyanin R	TBP	Tri(n) butyl phosphate
EDTA	Ethylenediaminetetracetic acid disodium salt	TOA	Trioctyl amine
em	emission wavelength	TOMA	Trioctyl methyl ammonium
ETAc	Ethyl acetate	XO	Xylenol Orange
Et ₂ O	Diethyl ether		

Table 1
BORATE (BO_3^{3-})

Organic Reagent	pH/ molarity	Solvent	λ_{max} nm	Molar Absorptivity $\text{l. mol}^{-1} \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
Azomethine-H	5	H_2O	420	— (0.1-6.0)	—	1-9
Carminic acid	7	H_2O	ex-467 em-556	(0-3.0)	—	10,11
Catechol Violet	8.4	H_2O	492	5000 (0.4-5.6)	—	12
Chromotropic acid	6.7	H_2O	ex-313 em-380	—	—	13
CA + Zephiramine	3.5	$\text{C}_2\text{H}_4\text{Cl}_2$	351	14000	—	14
Dibenzoylmethane	H_2SO_4 Conc.	H_2SO_4	ex-365	—	—	15
2,6-dihydrobenzoic acid + 4,4- diethylamino-phenylazo)-N- methyl pyridinium	H_2SO_4 0.025M	CHCl_3	570	66000 (0-16)	—	16
2,4-dihydroxybenzophenone	H_2SO_4 Conc.	— H_2SO_4	— em-503	— (0.03-0.16)	—	17
1,8-dihydroxynaphthalene-4- sulphonic acid + Zephiramine	—	$\text{C}_2\text{H}_4\text{Cl}_2$	341	25400	—	18,19

Table 1 continued

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity $\text{l.mol}^{-1}\text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Reference
2,4-dinitro naphthalene-1,8-diol + BG	HOAc	PhMe	—	103000	—	20,21
2-ethyl-1,3-hexane diol	—	CHCl_3	555	—	—	22,23
Hydrobenzoin + CV	9.2-9.6	C_3H_6	600	— (0.03-3.0)	Ca^{2+} , Mg^{2+} , Fe^{3+} , Al^{3+} , I^- , Br^- , Cl^- , SCN^- , ClO_4^- , SO_4^{2-}	24
2-Methyl-2,4-pentanediol + Curcumin	—	MIBK	518	—	NH_4^+ , Na^+ , K^+ , Ca^{2+} , Al^{3+} , Mn^{2+} , Mg^{2+} , Cl^- , N^{3-} , N^{2-} , Cl_2^{2-} , Fe^{3+} , Cl^- , NO_3^- , SO_4^{2-} , FO_4^{3-} , SiO_3^{2-}	25 26
Morin + oxalic acid	—	Ac_2O	—	— (0-10)	—	27,28
2,5 Naphthalene diol + CV	3.0-7.0	PhCl	610	10000 (0.01-0.1)	—	29-32
Pyrocatecholic acid	—	H_2O	ex-312 em-355	— (0.5-10)	—	33

H-resorcinol + diphenylguanidine	5-6	Higher alcohol	510	— (0.2-2)	—	34,35
H-resorcinol + thiazine	5	H ₂ O	510	11000 (0.04-0.4)	—	36
H-resorcinol + thionine	5	H ₂ O	510	1000	—	37
Sodium salicylate + ferroin	6-7	CHCl ₃	516	—	—	38-40
Tetraacycline	H ₂ SO ₄ Conc.	H ₂ SO ₄	570	5800 (2.1-12.6)	F ⁻ , NO ₃ ⁻	41
1,2,5,8-Tetrahydroxy-anthraquinone-3-methyl-amine-N,N-diacetic acid	H ₂ SO ₄ Conc.	H ₂ SO ₄	570	18000	—	42-44
Azurol C (N ₇ methylthionine)	H ₂ SO ₄ + HF	C ₂ H ₄ Cl ₂	540	— (0.2-10)	—	45
Iodine Green	Acidic	C ₃ H ₆	575	6500 (0.2-1.0)	—	46
Methylene Green	Acidic	PhCl	620	10600	—	46
Victoria Blue 4R	Acidic	PhCl	620	1000	—	45
Red Violet	—	C ₆ H ₆	554	— (0-0.42)	—	47,48

Table 2
FLUORIDE (F⁻)

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity $l \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
Alizarin	—	H ₂ O	610	10900	—	49
Alizarin complexone + diphenylguanidine	4.7	MePh + EtOH	575	—	—	50
Al + ECR + CPB	5.9	H ₂ O	620	—	—	51
Borax + Alizarin complexone	HNO ₃ 1N	Et ₂ O	630	—	—	52
Ce-Alizarin complexone	4.4	H ₂ O	612	— (0.06-0.8)	Al ³⁺ , Fe ³⁺ , Zn ²⁺ , Cu ²⁺	53-55
Ce-Alizarin Red S + diphenylguanidine	5.2	MeOH + CCl ₄	626	—	—	56
Ce/La-Quinalizarin complexone	4.0	DMF (30%)	660	—	—	57
Fe-4-Methyl resaceto- phenone (Paeonol) oxime	3.0	H ₂ O	Bluish- violet	15000 (1-10)	Cu ²⁺ , Ni ²⁺ , Co ²⁺ , Mo ⁶⁺ , W ⁶⁺ , CO ₃ ²⁻ , CrO ₄ ²⁻	58
Fe-5,5'-Methylene-bisallylic acid	1/6	H ₂ O	Violet	— (13-39)	—	59
La-Alizarin complexone	4.3	Me ₂ CO 20%	610	— (0.5-10)	—	60-70

La-Alizarin complexone + diphenylamine	4.3	IAA + Me ₂ CO	580	-	-	71
La-Alizarin complexone + TOMA chloride	4.3	IAA	625	-	-	72
Ta-oxalate-catechol	-	H ₂ O	400	-	-	73
Th-Alizarin Red S	2.9-3.0	H ₂ O	535	(10-80)	-	61, 69, 74, 75
Th-Arsenazo I	-	H ₂ O	575	-	-	76-80
U-2,2'-bis(phenylene oxime)	5.9	H ₂ O	400	-	Al ³⁺ , Fe ³⁺ , Oxalate, Citrate, C ₂ O ₄ ²⁻ , PO ₄ ³⁻ , SO ₄ ²⁻ , HCO ₃ ⁻	81
Zr-Alizarin Red S complexone	HCl 1.2M	H ₂ O	520-525	(0.40)	-	82
Zr-Arsenazo III	2.2-2.3	H ₂ O	665	(0.2-2.0)	-	83
Zr-chlorosulphophenol S	2.8	H ₂ O	640	8000 (0.1-1.4)	-	84
Zr-Thoron-Cl	HCl	H ₂ O	482	30000 (0.04-0.25)	Fe ³⁺ , Al ³⁺ , C ³⁺ , S ²⁻ , S ₂ O ₃ ²⁻ , PO ₄ ³⁻	85
Zr - XO	1.9	H ₂ O	560	(2.25)	-	86-98
Al(III)-calcon-carboxylic acid	-	-	-	(0.07-0.3)	-	89
Al(III)-quercetin	4.0	-	-	(5.160)	Ba, Ti, W, Th	90
1,3-N'-bis-4-(4'-nitro-benzene-diazo) phenyl squaraine	3.6-8.0	DMSO-H ₂ O	632	1.7 x 10 ⁴ (0-0.5)	-	91

Table 3
HYDROGEN PEROXIDE (H₂O₂)

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity l.mol ⁻¹ cm ⁻¹ (Beer's Law, ppm)	Interference	Refer- ences
Co ²⁺ + HCO ₃ ⁻ + NaHMP	HCO ₃ ⁻ (satur- ated)	H ₂ O	250	—	NO ₃ ⁻ , C O ₃ ⁻	92
Co ³⁺ + EDTA + NaOH	—	H ₂ O	567	—	—	93
CrSO ₄ + Phenol-phthalein	—	H O	540	—	—	94, 95
TBP + K ₂ Cr ₂ O ₇	—	C ₃ H ₆	584	— (0-136)	—	97
K ₂ T O (C ₂ O ₄) ₂ .2H ₂ O	—	H O	400	—	—	98
Tl ⁴⁺ + AcOH	—	H O	408	— (1-3)	—	99
Tl ⁴⁺ + PAR	8.6	H O	508	— (0.034-1.02)	—	100, 101
Tl ⁴⁺ + CAS	—	H ₂ O	557	— (0.07-1.8)	—	103, 104
Tl ⁴⁺ + EDTA	—	H ₂ O	365	— (0.34-68)	—	103

$Ti^{4+} + XO$	2-4	H ₂ O	562	13200 (0.1-3.4)	-	103-105
$V^{5+} + PAR$	1.0	H ₂ O	510	12800 (0.4-2.2)	-	105
$V^{5+} + XO$	4.0	H ₂ O	582	-	-	100, 101, 107, 108
Acetaminophenol	-	-	ex-290 em 333	-	-	109

Table 4
NITRATE (NO_3^{1-})

Organic Reagent	pH/ molarity	Solvent	λ_{max} nm	Molar Absorptivity $1. \text{mol}^{-1} \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
4'-amino acetophenone hydrochloride + <i>m</i> -phenylene diammonium hydrochloride	2.0-2.5	-	460	47,000	-	110
4'-amino acetophenone hydrochloride + <i>m</i> -phenylene diammonium hydrochloride	9.0	Toluene	450	- (0.0-0.03)	-	111
4'-amino acetophenone hydrochloride + N-1-naphthyl ethylene diamine	-	Butanol	550	48000	-	112
4'-amino acetophenone hydrochloride + resorcinol	-	-	435	52700 (0-0.33)	-	113
4'-amino acetophenone hydrochloride + resorcinol	-	-	435	-	S^2 , $\text{S}_2\text{O}_3^{2-}$, SO_3^{2-} , $\text{S}_2\text{O}_8^{2-}$	114
<i>p</i> -Amino acetophenone	-	-	570	6900 (0.08-0.48)	SCN^{2+} , $\text{S}_2\text{O}_3^{2-}$, Cu^{2+}	115
4-amino benzoic acid + 1- naphthol	-	DMF	519	35000 (0.1-1.3)	-	116

4-amino benzoic acid + 1-hydroxy-2-naphthoic acid	0.02-0.12 M HCl	-	525	0-2.5	-	117
4-amino-5-hydroxy-naphthalene-1-sulphonic acid	4.0	-	550	2500	-	118
4-amine benzoic acid + quinolin-8-ol	-	-	499	32000	-	119
Derivatives of 5-amino-naphthalene	5% HOAC	-	525	- (0-16.5)	-	120
Na-4-amino-naphthalene-1-sulphonate	4.0	-	485	10932 (0.1-5.0)	-	121
4-amino-naphthalene-1-sulphonic acid	-	-	530	30500 (0.16-4.9)	-	122
4-amino phenol + catechol	HOAC medium	-	530	23000 (0.6-1.6)	-	123
Anilinium chloride + 1-naphthol	2.0M HCl	Toluene	560	- (0.150.0)	-	124
Barbituric acid	9.0-11.0	-	340	- (4.0-60.0)	-	125
C ₂ ⁴⁺ + Promethazine	0.5 M H ₂ SO ₄	-	518	10000 (0.05-1.72)	-	126
Cyclohexane-1,3-dione + bis-(4-methyl thio semi carbazono)-mono-hydrochloride	-	-	365	11000 (0.5-5.0)	-	127

Table 4 (continued)

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity $l \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
3-6-diamino acridine	1.0	-	330	- (2.0-88.0)	-	128
4-5-dihydroxy coumarin	5.0 M HCl	Benzene	410	- (0.14-3.5)	I^{-} , SO_3^{2-} , Cr^{3+} , Fe^{2+}	129
4-5-dihydroxy coumarin	5.0 M HCl	EtAc	382	- (0.75)	I^{-} , SO_3^{2-} , Cr^{3+} , Fe^{2+}	130
5-7 dihydroxy-4-imino-2-oxochroman	1.6	-EtAc	361	23200 (0.05-0.28)	-	131
4-dimethyl amino benzaldehyde	3.00 M HCl	-	-	- (0.5-25.0)	-	132
2,3-dimethyl-1-phenyl-pyrazolin-5-one	-	-	345 or 600	- (6.3-16.5)	-	133

Fluorene-2,7-diamine	1.0-4.0 M HCl	-	355	- (0.1-2.3)	-	134
Guaiacol and 4-nitro aniline	11.0	IAA	540	62000 (0.3-0.9) 48000 (0.03-0.6)	-	135
5-Hydroxy indole-2-carboxylic acid	H ₂ SO ₄ medium	-	465	3540 (1.0-12.0)	-	136
5-Hydroxy-indole-2-carboxylic acid	11.8	-	476	- (0.5-6.0)	-	137
8-Hydroxy quinoline-5-sulphonic acid	HCl medium	-	530	- (5.0-30.0)	-	138
8-Hydroxy quinoline + 4-nitro aniline	-	-	550	- (0.08-1.12)	Cu ²⁺ , Fe ³⁺ , S ²⁻	139
Indole	5.00M HCl	-	530	- (0-2.0)	-	140
2-mercapto ethanol	0.4M HCl	-	-	-	Oxidizing agents	141
4-Nitro aniline + 1-naphthol	-	Hexanol	610	52400 (0.02-0.14)	-	142
4-Nitro aniline + 1-naphthol	0.4M HCl	Hexanol	610	- (0.02-0.14)	-	143

Table 4 (continued)

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity l.mol ⁻¹ cm ⁻¹ (Beer's Law, ppm)	Interference	Refer- ences
1-naphthol + sulphanilic acid	—	—	515	— (0.1-0.5)	F ³⁺	144
1-naphthol + 4-trifluoromethyl aniline	—	—	510	39100 (0.03-0.8)	F ³⁺ , Al ³⁺	145
1-naphthyl amine + sulphanilic acid	—	—	—	— (0-1.2)	—	146
N-naphthyl)-ethylene diamine + sulphanil amide	—	—	538	— (1.8-2.7)	—	147
N-(1-naphthyl)ethylene diamine + sulphanilic acid	—	—	525	— (1.0-40.0)	—	148
N-(1-naphthyl) ethylene diamine + sulphanilic acid	1.7-1.9	—	545	— (0.001-0.01)	—	149
N-(1-naphthyl)ethylene dia- mine dihydrochloride + sulpha- nilic acid + formaldehyde	—	—	540	— (0.08-0.6)	—	150
N-(1-naphthyl) ethylene diamine + sulphanilic acid	6,9	—	480	— (1.0-75.0)	Fe ³⁺	151

4-Nitro aniline + quinoline-8-ol	10.4	IAA	570	58500	-	152
4-Nitro aniline + 2-methyl quinoline 8-ol in HD PMAA bromide	-	-	585	47000 (0.1-0.95)	-	153
o-nitro aniline + N-(1-naphthyl) ethylene diamine	HCl medium	IAA	545	6000 (0.1-0.6)	-	154
Orthanillic acid + resorcinol	-	-	426	- (0.1-1.2)	-	155
Promazine hydrochloride	0.5-2.0N H ₂ SO ₄	-	505	- (0.04-1.68)	-	156
Quercetin-Zr complex	0.4M HCl	-	430	- (0.2-1.6)	F ²	157
Quinoline-8-ol + sulphanilamide (azo dye)	0.04M HCl	-	500	3000 (0.16-1.2)	-	158
Resorcinol + Zr ⁴⁺	-	-	347	- (0.3-5.0)	-	159
Saltzman Reagent	-	-	355	-	-	160

Table 5
PHOSPHATE (PO_4^{3-})

Organic Reagent	pH/ molarity	Solvent	λ_{max} nm	Molar Absorptivity $1. \text{mol}^{-1} \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
Ba^{2+} Thoron complex in hydroalcoholic medium	8.0	—	500	—	—	161
Ca^{2+} + N-phenylbenzo- hydroxamic acid	11.3	Chloro- form	340	— (0.5-10.0)	Mg^{2+} , F^{-}	162
Calciochrome + capriquat	NaOH medium	—	545	— (0.02-0.6)	—	163
Ce^{4+} + Arsenazo (III)	5.0-5.5	—	655	—	—	164
Ce^{4+} + XO	—	—	580	— (0-1.0)	—	165
Ce^{4+} + XO	5.8 $(\text{Na}_2\text{B}_4\text{O}_7 \cdot \text{H}_2\text{O})_3$	—	575	— (1.86-12.3)	—	165
Dinonylamine (I)- $\text{H}_2\text{O}(\text{Mo}_3\text{O}_{10})_4$	—	1,2 di- chloro- ethane	312	26000 (0.1-1.0) (0-1.0)	—	167
Ethyl Violet + MG	—	MIBK	602	27000 (0-0.001)	—	168
		MIBK- Toluene	650	23000	—	168

Back extraction of Mo^{6+} , reduction to Mo^{3+} with Fe^{2+} and Ferrozine	3.0-3.5	iBuAc	562	— (0.0-0.004)	—	169
Coumar quenching by FO_3^- on Fe^{3+} + Salicyldoxime(I)	6.5-7.0	480	480	— (0.9-4.5)	—	170
2-[1]hydroxy-1-naphthyl dicotinine (II)	1.0-2.5	—	400	— (1.8-9.0)	—	
Paeonoloxime(III)	1.0-3.5	—	400	—	—	
Fe^{3+} + Paeonoloxime	3.0	—	400	1550 (0.9-4.5)	—	58
Fe^{3+} + Tetraphenylporphine trisulphonic acid	4.0	—	512 434	82000 (10.0-50.0) 22000 (4.0-20.0)	—	171 172
Hydroxanthine + H_2C_2 + 4-aminoanthipyrine-N'-acetyl-N-ethyl-N-m-tolylethylenediamine	—	—	555	— (0-240.0)	—	172
MG + Urea	—	—	630	—	—	173
Mo^{6+} + MB	—	—	660	— (0.31-3.1)	As^{3+} , Ge^{3+} , Si^{4+}	174
Mo^{6+}	—	MIBK	435	—	—	175

Table 5 (continued)

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity $1. \text{mol}^{-1} \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
Mono methyl-p-amidophenol (4-hydroxy-N-methyl benzamide)	Conc. HCl medium	-	525	- (0.4-1.3)	$\text{Cr}_2\text{O}_7^{2-}$, SO_3^{2-} , HSO_3^{1-} , Citrate Oxalate	176
Mo^{6+} + Ascorbic acid	-	-	590	- (0-20.0)	-	177
Mo^{6+} Ascorbic acid + Sb K tartrate	-	-	780	- (0.2-0)	-	178
Mo^{6+} Ascorbic acid + Sb K tartrate	7.0	-	880	- (0.2-1.0)	-	179
Mo^{6+} Ascorbic acid + Sb K tartrate	-	-	710	-	-	180
Mo^{6+} + Antimony + Ascorbic acid	H_2SO_4 medium	-	880	- (0.005-0.8)	-	181
Mo^{6+} + Benzoylphenone + SnCl_2 + Methyl p-aminopropionate	HNO_3 medium	-	720	- (0-0.08)	-	182
Mo^{6+} + bis-(2-ethyl hexyl)- amine	7.0	1,2 di- chloro- ethane	400	-	-	183
Mo^{6+} + Butyl Rhodamine B	0.6M HNO_3	-	580	45000 (0.2-0.4)	-	184

as Mo_2F_{10} (C.I. Solvent Orange-15)	1.0	-	495	-	AsO_4^{3-}	185
$\text{Mo}^{6+} + \text{CuSO}_4 + \text{Methanol}$ NH_4SCN	3.0M HCl	-	470	-	-	186
$\text{Mo}^{6+} + \text{CV}$	H_2SO_4 medium	-	545	-	-	187
$\text{Mo}^{6+} + \text{CV}$ in Poly (vinyl alcohol)	-	-	560	-	-	188
$\text{Mo}^{6+} + \text{Diphenyl Amine}$	8.0mM HNO_3	1,2-di-chloro-ethane	312	-	-	164
$\text{Mo}^{6+} + \text{Dinonylamine} + \text{SnCl}_2$	1.0 M H_2SO_4	Chloroform Butanol (2:1)	710	-	-	189
$\text{Mo}^{6+} + \text{Fe}^{2+}$ in phosphoric acid medium	H_2SO_4 medium	MIBK	752	-	$\text{V}^{5+}, \text{U}^{6+}, \text{IO}_3^{1-}$	190
$\text{Mo}^{6+} + \text{Gelatin} + \text{Sb K tartrate} + \text{ascorbic acid} + \text{amberlite A-27}$	H_2SO_4 medium	-	695	-	$\text{As}^{3+}, \text{SiO}_3^{1-}$	191
$\text{Mo}^{6+} + \text{Hydrazine sulphate}$	0.5- 1.2N H_2SO_4	-	830	-	-	192
$\text{Mo}^{6+} + \text{Hydrazine sulphate}$	H_2SO_4 medium	-	825	-	-	193

Table 5 (continued)

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity $\text{l. mol}^{-1}\text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
$\text{Mo}^{6+} + \text{KCl}$	HCl medium	Chloro- form: Butanol (7:13)	310	— (0-20 0)	—	194
$\text{Mo}^{6+} + \text{MG}$	H_2SO_4	Benzene + MIBK	610	— (0.001-0.3)	—	195
$\text{Mo}^{6+} + \text{MG}$	—	2- methoxy- ethanol	627	—	—	196
$\text{Mo}^{6+} + \text{MG}$	H_2SO_4 medium	2- methoxy- ethanol	627	— (0.003-0.03)	—	197
$\text{Mo}^{6+} + \text{MG}$	—	MIBK: Toluene (1:3)	630	230000 (0-0.1)	—	198
$\text{Mo}^{6+} + \text{MG} + \text{Poly(vinylalcohol)}$	2.0-3.0	—	650	78000 (0.1-5.0)	—	199
$\text{Mo}^{6+} + \text{MB}$	HNO_3 medium	MIBK	655	— (0.08-0.8)	As^{3+} , Ge^{3+} , Si^{4+} , V^{5+} , Cr^{6+}	200

as Molybdophosphoric acid	0.5M HCl	BuAc	315	— (0.1-6)	—	201
as Molybdophosphate blue	HOAc medium	—	700	200-0 (1.0-30.0)	—	202
Mo^{6+} + Phenasafranine	—	MIBK	529	— (0.1-1.5)	—	203
Mo^{6+} + Ethyl Violet	H_2SO_4	Cyclohexane: MIBK	602	270-00 (0-0.6)	As^{3+} , Zn^{3+} , Sn^{4+} , Sn^{2+}	204
Reduction of Mo^{6+} with Amberlysi-15	—	—	700	— (0.001-0.005)	AsO_4^{1-} , SiO_4^{1-}	205
Mo^{6+} + Methyl Green	—	—	660	— (0-100.0)	—	206
as MB	0.28H HClO_4	—	825	— (0.16-70.0)	Fe^{3+}	207
Mo^{6+} + Resazurin(I) + Pararosaniline acid(II)	—	—	495 470	— (0.2-0.6) — (0.2-0.8)	— —	208 208
Mo^{6+} + Rhodamine 6G	H_2SO_4	—	565	— (0.04-0.2)	—	209
Mo^{6+} + SnCl_2	HClO_4 med ur.	BuAc	725	— (0-10.0)	—	210
Mo^{6+} + SnCl_2 + ascorbic acid	H_2SO_4 medium	—	715	— (0.02-1.3)	—	211

Table 5 (continued)

Organic Reagent	pH/ molarity	Solvent	λ_{max} nm	Molar Absorptivity $l \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
$\text{Mo}^{6+} + \text{SnCl}_2$ in Glycerol	—	—	640	— up to 2.0	—	212
$\text{Mo}^{6+} + \text{Triethylamine}$	5.0	Methanol	320	209000 (5.0-260)	—	213
$\text{Mo}^{6+} + \text{trilon}$	H_2SO_4 medium	—	410	—	—	214
$\text{Mo}^{6+} + \text{triethanolamine}$ dodecylsulphate	Con. HNO_3 medium	—	390	— (0-80.0)	—	215
$\text{Mo}^{6+} + \text{V}^{5+} + \text{CV}$	0.4M HCl	$\text{EtAc} + \text{Me}_2\text{CO}$	590	310000 (0.02-0.12)	—	216
$\text{Mo}^{6+} + \text{V}^{5+} + \text{bis-(2-ethylhexyl)-amine}$	—	1,2-dichloroethane	400	—	—	217
$\text{Mo}^{6+} + \text{V}^{5+} + \text{BG}$	0.2-1.8	—	626	288000 (0.06-0.1)	$\text{Ni}^{2+}, \text{Co}^{2+}, \text{Cr}^{6+}, \text{Si}^{4+}, \text{W}^{6+}, \text{Fe}^{3+}$	218

Rhodamine 6G	0.1-1.0	-	-	-	-	219
Rhodamine B	1.1	Acetone	560	62500 (3.0-42.0)	As ³⁺	220
as tungstophosphoric acid	0.3-1.9	Pentanol	267	51000 (0.0250)	-	221
as tungstophosphoric acid	1.6	Pentanol	265	- (0.04-0.8)	Si ⁴⁺	222
Colour quenching by PO ₄ ³⁻ on UO ₂ ²⁺ + salicylaldehyde(I)	5.0-11.0	-	400	- (4.0-20.0)	-	167
2-hydroxy-1-naphthal- dehyde(II)			-	- (1.9-9.0)	-	
Zn ²⁺ -XO + HDTMA	0.02M HCl	-	605	- (0.01-0.3)	F ¹⁻ , SO ₄ ²⁻	223
Antimonophosphomolybdenum blue	-	-	720	-	-	224
Phosphomolybdate Blue	-	Butanol	-	2.2 x 10 ⁴	-	225

Table 6
SULPHIDE (S²⁻)

Organic Reagent	pH/ molarity	Solvent	λ_{max} nm	Molar Absorptivity l.mol ⁻¹ cm ⁻¹ (Beer's Law, ppm)	Interference	Refer- ences
<i>p</i> -Phenylenediamine + [Fe(NH ₄)] (SO ₄) ₂ + MB	—	H ₂ O	656	—	—	226-232
<i>p</i> -Phenylenediamine + [Fe(NH ₄)] (SO ₄) ₂ perchlorate	—	C ₂ H ₄ Cl ₂	656	— (0.5-100)	—	233
Hg fluorescein	—	—	—	—	—	234
VO ₃ ⁻ + Lumirone	H ₂ SO ₄ 0.1M	H ₂ O	—	—	Fe ³⁺ , Mn ²⁺ , Co ²⁺ , Sn ²⁺ , Pt ⁴⁺	235
Sodium Nitroprusside	—	—	536	—	—	236

Table 7
SULPHITE, SULPHATE AND THIOSULPHATE

Organic Reagent	pH/ molarity	Solvent	$\lambda_{\max}$ nm	Molar Absorptivity l. mol ⁻¹ cm ⁻¹ (Beer's Law ppm)	Interference	References
(SO₃²⁻)						
Cr ₂ O ₇ ²⁻ + diphenyl carbazide	-	-	-	-	-	237
Fe(III) + Bipyridyl	5	-	525	- (0.0-2)	-	238
Na ₂ HgCl ₄	-	-	288	-	-	239
(SO₃²⁻)						
2-Aminopiperidine hydrobromide	HNO ₃	-	540	- (3-55)	-	240
Ba-chloranilate + SO ₃ ²⁻	1.5	C ₂ H ₅ Cl	595	68000	-	241
BaCl ₂ + chlorophosphonazo(III)	1.8-2.0	EtOH + H ₂ O (1:1)	645	- (0.3-2)	-	242-244
Ba ²⁺ + dimethyl Sulfanazo(III)	-	-	655	-	-	245
Ba ²⁺ + MTB	12.7	-	460	-	-	246,247
Ba ²⁺ + Orthanilic acid	-	H ₂ O + Me ₂ CO	645	-	-	248
Th-quercetin	2.5	EtOH	-	-	Fe ³⁺ , PO ₄ ³⁻	249
S₂O₃²⁻						
Chromopyrazole(II)	-	CHCl ₃	548	40000	-	250

Table 8
THIOCYANATE (SCN^{1-})

Organic Reagent	pH/ molarity	Solvent	λ_{max} nm	Molar Absorptivity $l \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ (Beer's Law, ppm)	Interference	Refer- ences
Barbituric acid in pyridine- HCl-water medium	-	-	586	- (0-6.0)	-	251
Barbituric acid in pyridine- HCl-water medium	-	-	580	- (up to 200.0)	-	252
Chloramine T + barbituric acid in pyridine-HCl-water medium	5.0	-	583	- (0.5-2.5)	-	253
Chloramine T- FeCl_3 + barbituric acid in 4-picoline- HCl-water medium	-	-	605	- (2.0-20.0)	-	254

FREQUENTLY USED SPECTROPHOTOMETRIC DETERMINATIONS OF COMMON ANIONS

1. Borate

Borate is extracted as the borate benzohydroxamate-phenylfluorone complex from 20% tributylphosphate in isoamyl alcohol. The reddish yellow coloured complex has a maximum absorbance at 510 nm and molar absorptivity $0.5 \times 10^3 \text{ l mol}^{-1} \text{ cm}^{-1}$. It obeys Beer's law in the range of 5-25 ppm /255/.

2. Bromate

An equimolar mixture of isonicotinic acid hydrazide and 2,3,5-triphenyltetrazolium chloride in dilute hydrochloric acid (pH 0.86, adjusted with 0.5 NHCl) gives a pink colour with bromate on heating for 10 min at 60°C, λ_{max} 480 nm /256/.

o-aminobenzoic acid reacts with bromate in the presence of hydrochloric acid (pH 1.5) to give a reddish brown colour on heating the solution at 80-90°C /257/.

3. Bromide

Phenol red upon treatment with hyhpochlorite in the presence of small amounts of bromide forms bromophenol blue. Into a neutral bromide solution add phenol red and borax solution. Add 0.01 N-hypochlorite and allow to stand exactly 4 min with occasional shaking and add 0.1 N sodium arsenite followed by acetate buffer (4.6 - 4.7 pH). The colour obtained will be yellow with less than 1 µg of bromide, reddish from 1.5 to 2 µg and bluish violet above 2.5 µg /258/.

4. Chlorate

Chlorate forms a yellow coloured complex with *o*-toluidine in strong hydrochloric acid solution. The absorbance can be measured at 448 nm and 490 nm for low and high concentrations of chlorate ion respectively /259/.

5. Chromate

Diphenylcarbazide reacts with chromium(VI) in acidic medium to give an intense red-violet complex cation, λ_{max} 540 nm, molar absorptivity 3.14×10^4 , Beer's Law (0.1 - 0.005 ppm). Mo(VI) can be masked by oxalate /260/.

6. Citrate

Citrate can be determined fluorimetrically at 450 nm by addition to 1:1 tungstate flavanal complex, Beer's Law 0-200 ppm /261/.

7. Cyanide

The cyanide is determined by reacting it first with excess bromine to give cyanogen and then reacting the cyanogen bromide with a pyridine-benzidine reagent. It also reacts with *p*-phenylenediamine and 0.1 mg cyanide can be determined /262/.

8. Fluoride

Fluoride gives a stable complex with scandium and, hence, by determining the unreacted scandium, the fluoride content is calculated. Excess scandium is reacted with ethanolic solution of N-phenylbenzohydroxamic acid (PBHA) at pH 6.0 and the scandium-PBHA complex is extracted into isoamyl alcohol. Scandium is determined after adding xylenol orange. Beer's Law for fluoride is obeyed in the range of 0.05 - 1.5 ppm; the molar absorptivity is $1.94 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ /263/.

9. Formate

Reduction of formate by magnesium ribbon to formaldehyde, followed by condensation of the latter with chromotropic acid, enables amounts of formic acid to be determined. 6-Amino-1-naphthol-3-sulphonic acid may also be used /264/.

10. Hydrogen Peroxide

Hydrogen peroxide reacts with vanadium(V)-N-phenylbenzohydroxamic acid (PBHA) complexes at 2 M HCl. The colour of the complexes is

bleached due to formation of a V(V)-peroxide complex, decreasing the concentration of vanadium(V)-PBHA complex. The hydrogen peroxide is determined at 520 nm in the range of 0.5 - 5.0 ppm /255/.

11. Iodate

Iodate gives a pink colour complex when mixed with an equimolar proportion of isonicotinic acid hydrazide and 2,3,5 triphenyltetrazolium chloride in dilute hydrochloric acid at room temperature. The λ_{max} is 480 nm /256/.

12. Nitrite

Nitrite reacts with salbutamol sulphate in acidic medium, which gives a yellow colour in alkaline solution. Beer's Law is obeyed in the range of 1.8 - 27.6 ppm of nitrite with molar absorptivity $1.8 \times 10^3 \text{ l mol}^{-1} \text{ cm}^{-1}$ at 410 nm /265/.

13. Oxalate

Oxalic acid (in 1N sulphuric acid) reacts with indole (heating on water bath at 80 - 90°C for 45 min) to form a red or pink-coloured compound, λ_{max} 525 nm, Beer's Law obeyed for 0.050 to 1.00 mg ml⁻¹ /266/.

14. Perchlorate

Perchlorate can be determined by extracting it from aqueous solution by n-butyro-nitrite as ferrous 1, 10-phenanthroline perchlorate at pH 5. The λ_{max} is 510 nm and molar absorptivity is $1.08 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ /267/.

15. Perrhenate

Perrhenate is first reduced by tin(II) chloride and forms a rhenium dioxime complex with a large excess of α -furildioxime. The absorbance of the complex is measured at 532 nm. Beer's Law is obeyed for 20 - 300 mg of rhenium per 50 ml /268/.

16. Phosphate

Phosphate is selectively separated from associated elements by reacting it with calcium and extracting excess calcium with N-phenylbenzohydroxamic acid (PBHA) at pH 11.3. The excess calcium was determined in the UV and visible region and hence the phosphate content was calculated by difference. Beer's Law is obeyed in the range of 0.5 - 10.0 ppm of phosphate at 340 nm and 0.25 - 8.0 ppm at 560 nm for a fixed amount of calcium (20.0 ppm). It can also be determined by AAS /269/.

17. Sulphate

Sulphate reacts with barium chloroanilate at pH 4 in a 50% ethyl alcohol solution, giving a highly coloured acid chloranilate ion. This has maximum absorbance at 530 nm /270/.

18. Sulphide

Sulphide is separated from other ions by reacting with mercury and the excess of mercury is determined using N-phenyl-benzohydroxamic acid (PBHA) at pH 6.8; the sulphide concentration is calculated by difference. Mercury forms a complex with PBHA in ethanol, which is extracted into chloroform. Beer's Law is obeyed for sulphide in the range 0.05 - 3.0 ppm at 550 nm for a fixed amount, 20 ppm, of mercury /255/.

19. Thiocyanate

Thiocyanate is selectively separated from its cation by reaction with mercury(II). The excess mercury(II) is determined using N-phenyl benzohydroxamic acid (PBHA) at pH 6.8 and the thiocyanate concentration calculated. Mercury(II) complexes rapidly with PBHA in ethanol and is further extracted into chloroform. Beer's Law is obeyed for thiocyanate in the range of 0-10 ppm at 550 nm for a fixed amount, 20 ppm, of mercury(II) /271/.

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