Table 1

Applications of Polarography to Plating Bath Analysis

BASE METALS	PRECIOUS METALS	ORGANICS	ANIONS
Cd^{2+}	Au^{1+}, Au^{3+}	BENZALDEHYDE	CHLORIDE
Cu^{1+}, Cu^{2+}	Pd^{2+}	BENZOQUINONE	CYANIDE
Ni^{2+}	Pt^{4+}	CYSTINE	SULFITE
Pb^{2+}	Rh^{3+}	FORMALDEHYDE	
Sn^{2+}, Sn^{4+}		NAPHTHALENE-1-SULFONIC ACID	
Zn^{2+}		THIOUREA	

Table 2

Instrumental Parameters for the Determination of Cu^{2+}

E initial:	-0.35 V
E final:	-0.70 V
Scan Rate:	5 mV/sec
Drop Time:	1.0 sec
Pulse Amplitude:	50 mV
Supporting Electrolyte:	0.5 M NaOH, 0.05 M EDTA
pH:	13

Table 3

Instrumental Parameters for the Determination of Pb^{2+} and Sn^{2+}

E initial:	-0.30 V
E final:	-0.80 V
Scan Rate:	5 mV/sec
Drop Time:	1.0 sec
Pulse Amplitude:	30 mV
Supporting Electrolyte:	2 M Na Acetate, 2 M Acetic Acid
pH:	4

Table 4

Instrumental Parameters for the Determination of Formaldehyde

E Initial:	-1.42 V
E Final:	-1.82 V
Scan Rate:	5 mV/sec
Drop Time:	1.0 sec
Pulse Amplitude:	50 mV
Supporting Electrolyte:	0.5 M NaOH, 0.05 M EDTA
pH:	13

Table 5

Instrumental Parameters for the Determination of CN^-

E Initial:	-0.37 V
E Final:	-1.00 V
Scan Rate:	5 mV/sec
Drop Time:	0.5 sec
Pulse Amplitude:	50 mV
Supporting Electrolyte:	0.2 M KOH, 0.05 M EDTA
pH:	13

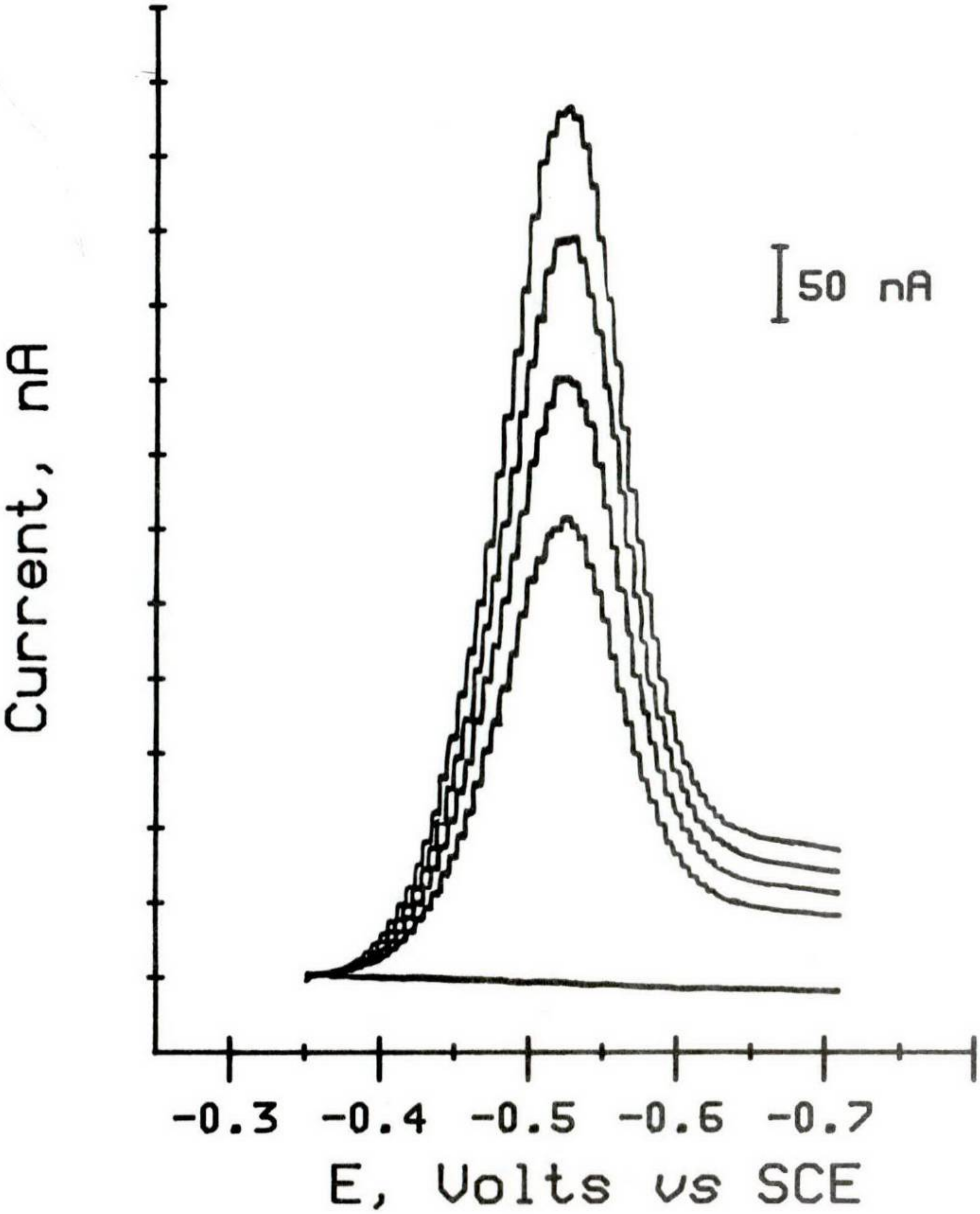

Fig. 1 - Determination of Cu^{2+} in an acid sulfate bath.

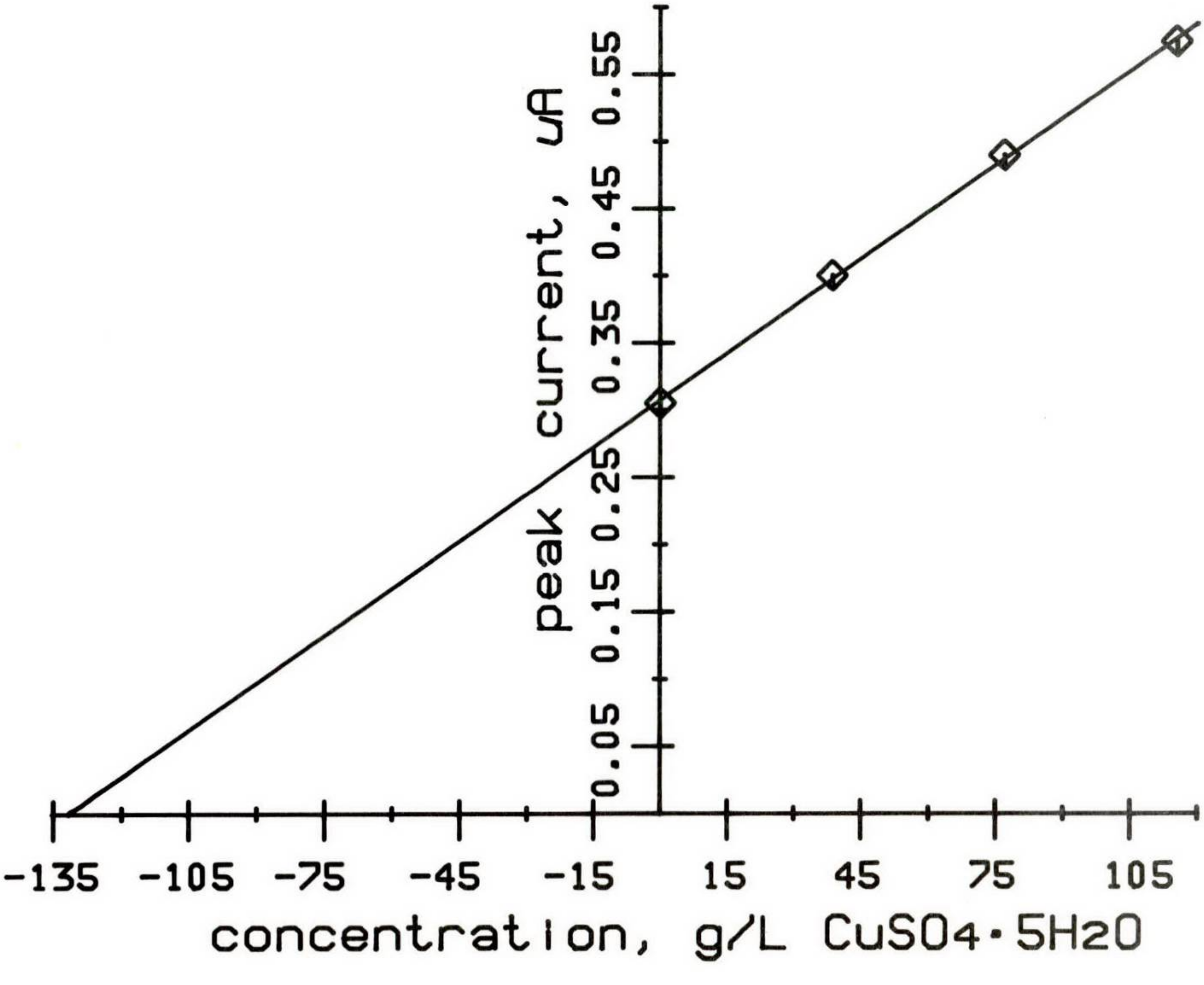

Fig. 2 - Standard addition plot for Cu^{2+} in an acid copper bath.

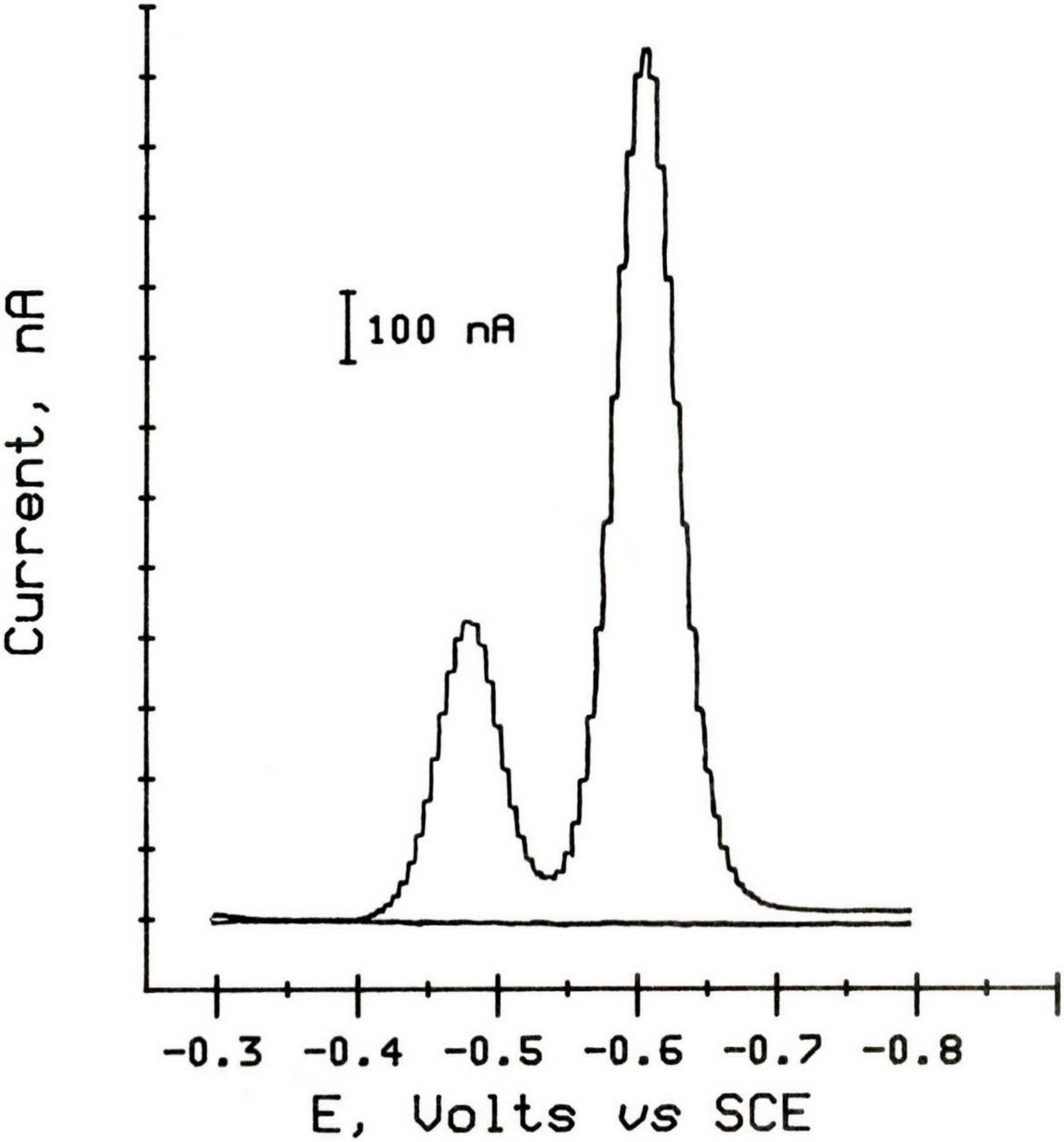

Fig. 3 - Simultaneous determination of Pb^{2+} and Sn^{2+} in a plating bath.

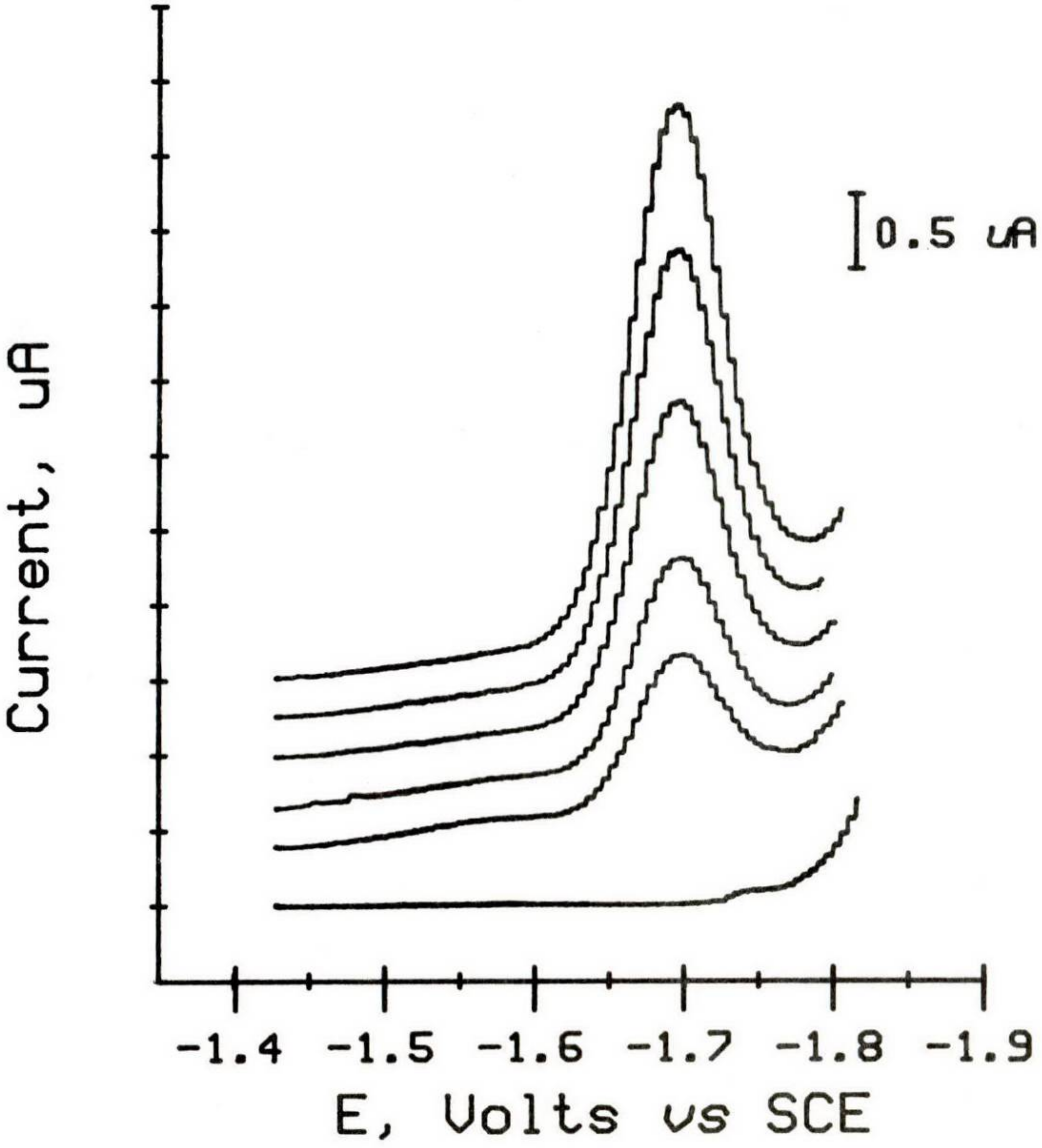

Fig. 4 - Determination of formaldehyde in an electroless copper bath.

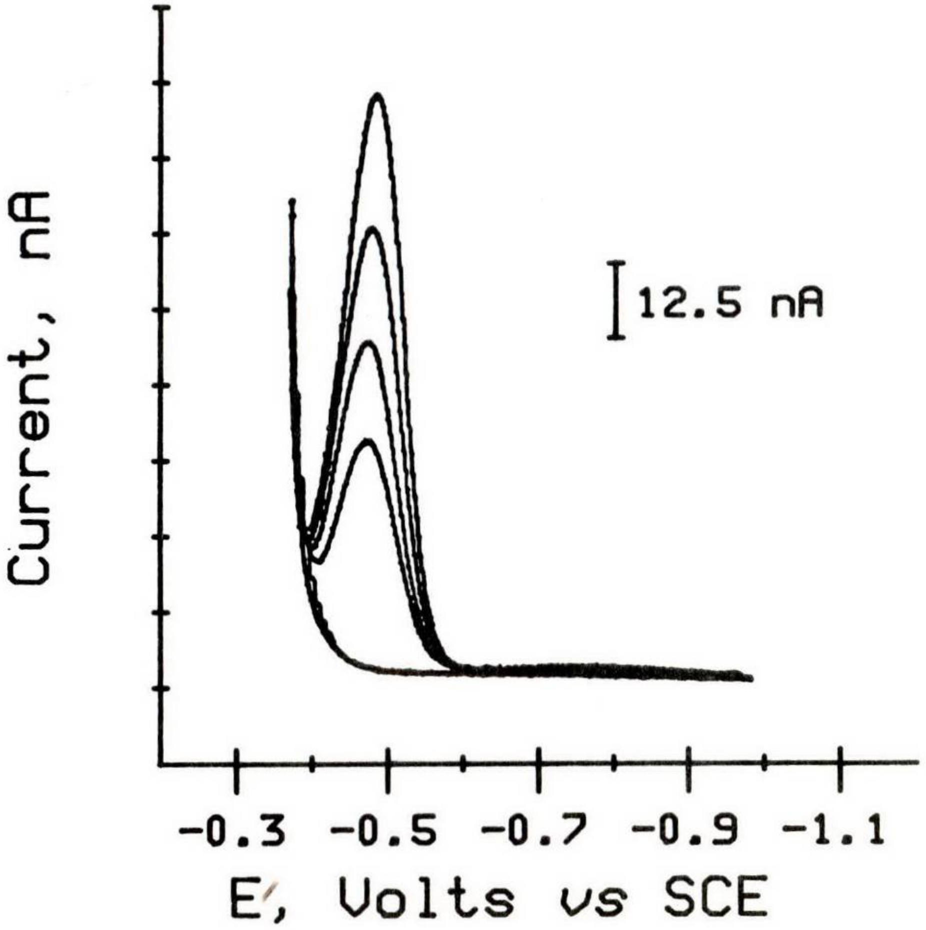

Fig. 5 - Determination of cyanide in a plating bath.

I-6

General Interest
Session I

Rapid Analysis and Troubleshooting of Gold, Copper and Nickel Plating Bath Chemistry by Ion Chromatography

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RAPID ANALYSIS AND TROUBLESHOOTING OF GOLD, COPPER AND NICKEL PLATING BATH CHEMISTRY BY ION CHROMATOGRAPHY

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INTRODUCTION

The use of high speed plating processes impose higher demands on plating baths which in turn, require more rapid monitoring and control for optimum performance. Electroless processes also require frequent monitoring for replenishment purposes. Analytical techniques offering speed, versatility, and ease of operation are needed. The purpose of this paper is to demonstrate how Ion Chromatography has been and can be employed to monitor, control and troubleshoot critical chemical balances in plating solutions.

Ion Chromatography (IC) is a technique which can be used to rapidly detect, identify, and quantify a wide variety of plating solution constituents. Some species of special interest include metal complexes ($Au(CN)_2^-$, $Au(CN)_4^-$, $Co(CN)_6^{3-}$, $Fe(CN)_6^{3-}$ $Fe(CN)_6^{4-}$, $Ni(EDTA)^{2-}$, $Cu(EDTA)^{2-}$); common anions (Cl^-, Br^-, NO_3^-, HPO_4^{2-}, PO_2^{3-}, SO_4^{2-}, BF_4^-); cations (Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}); organic acids (citric, formic, succinic) as well as carbonate and borate; transition metals (Fe^{3+}, Fe^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, Co^{2+}, Pb^{2+}); and surfactants such as sodium lauryl sulfate.

Ion Chromatography has been extensively employed for analysis of various plating solutions, acid etchants, conversion coatings, and cleaning solutions. Although IC analysis is applicable to the control and troubleshooting of a wide variety of plating solutions, the specific examples in this paper will address only gold, copper, and nickel solutions.

PRINCIPLES OF THE TECHNIQUE

Ion Chromatography (IC) is a unique analytical technique which specifically addresses the chromatography of ionic species. The system, which is schematically depicted in Figure 1, consists of an eluent delivery mode, a spearation mode, and a detection mode. An eluent is pumped through the system and a liquid sample is introduced into the flowing eluent stream via an injection valve situated upstream of the separator column. The eluent flow carries the ionic species through an analytical column that contains a neutral, anionic or cationic resin. Since the respective ionic species present in the sample have different affinities for the resin, they are separated into discrete bands before entering the detector. Identification and quantification are determined by comparing the retention times and peak heights with those of known standard solutions. Data reduction is accomplished manually or by use of electronic integration.

The separation mode employed depends upon the ionic nature of the sample ions of interest. Three distinct separation modes can be employed and are summarized in Table I. High Performance Ion Chromatography (HPIC) is the mode of choice for common inorganic ions such as F^-, Cl^-, NO_3^-, SO_4^{2-}, Na^+, NH_4^+, Mg^{2+}, Ca^{2+}, Fe^{3+}, and Zn^{2+}. The separation, based on ion exchange, is accomplished on a low capacity ion exchange resin. The theory of HPIC has been discussed by Small et al.[1]

Mobile Phase Ion Chromatography (MPIC) is the optimal separation mode for hydrophobic ions such as alkyl and aryl sulfonates and sulfates, metal cyanide complexes, SCN^-, BF_4^-, and quaternary and alkyl amines. The mechanism of separation is ion-pairing and adsorption which occurs on a neutral resin. This separation theory has been discussed in depth by Iskandarani[2] and Bidlingmeyer[3].

High Performance Ion Chromatography Exclusion (HPICE) is commonly used for the separation of weak organic acids such as formic, succinic, and citric. The mechanism is predominately dependent on pKa values and ion exclusion, however, adsorption also plays a role. Wheaton and Bauman[4] have discussed ion exclusion theory in detail.

The column packing material utilized in all of the separation modes, regardless of the resin functionality, is based on a polystyrene-divinylbenzene (PS/DVB) support. Unlike silica based packing, these resins are stable from pH 1-13. The excellent durability of the column packing material allows easy column clean-up with either strong acid or strong base.

Detection of the ions of interest can be accomplished in a variety of ways depending upon the chemical and optical properties of the sample ions. Four distinct detection modes are employed and these are summarized in Table II.

Suppressed conductivity is the most common detection mode used for ions with pK values <7. Highly conductive eluents are required to elute the sample ions from the separator column. If a conductivity detector cell was placed immediately after the separator column, the sample ions would be detected as small conductivity changes in a large background signal. Insertion of a suppressor, a second ion exchange device connected in series after the separator column, chemically converts the eluent to a less conductive form. The sample ions are thus detected as large changes in a low background conductivity. This increases sensitivity, extends the linear range, and promotes selectivity in detection. The actual suppression mechanisms differ depending on the chemical content of the eluent. Small et al[1] should be consulted regarding the actual mechanisms.

Three other detection modes are available in addition to suppressed conductivity. A UV/VIS detector can be used to detect ultraviolet or visible absorbing species in combination with any separation mode. Combined with a post column reactor utilizing a metallochromic indicator, this detector is used in the visible region for selective detection of the transition and heavy metals such as Fe^{3+}, Fe^{2+}, and Co^{2+}.[5] Amperometric detection is employed for species which can be oxidized or reduced, such as CN^-, HS^-, hydrazine, phenolics, carbohydrates, and mercaptans.[6 & 7] A fluorescence detector can also be used following post column reaction for the sensitive and selective determination of amino acids. By use of multiple separation and detection modes, a wide variety of inorganic and organic anions and cations can be determined in complex matrices. Furthermore, trace level (ppm and ppb) analysis can be accomplished in the presence of high concentrations of other species, as is often the case in plating analysis where minor components or contaminants are of major concern.

PLATING BATH ANALYSIS

For plating bath applications, the sample preparation generally involves no more than a simple dilution step followed by, at times, a filtration. All the separation and detection techniques utilized in Ion Chromatography apply to plating analysis, depending upon the particular species of interest.

GOLD PLATING BATHS

Cyanide based plating solutions containing phosphate and/or citrate buffers are common gold plating solutions. Within the bath, the Au exists as stable gold cyanide complexes, with $Au(CN)_2^-$ the most prominent. With use, the Au^{3+} content increases forming a $Au(CN)_4^-$ complex and causes plating efficiency to decline significantly. Consequently, a convenient method of differentiating between the two gold cyanide complexes is a key monitoring tool. Although important, total gold values shed no light on oxidation states. Figure 2 illustrates a determination of the two gold cyanide complexes in a phosphate/citrate buffered gold plating bath. Sample preparation involved only dilution 1/200 with deionized water. Since the Au^{3+} levels in this sample were relatively low (100 ppm), the detector attenuation was changed to a more sensitive range. In this way, both gold species are determined from a single injection.

The cobalt content is also of interest in the gold baths that are alloyed with cobalt to impart hardness. The cobalt is usually added as the $CoSO_4$ or in chelated forms. If free CN^- is generated in the bath during plating, the Co^{2+} can easily convert to Co^{3+} and be present as the $Co(CN)_6^{3-}$ complex. In this form, the cobalt no longer functions as intended and the plate is affected. The $Co(CN)_6^{3-}$ is also a very strong complex and is easily determined in gold cyanide baths as illustrated in Figure 3. Other strong cyanide complexes which can be determined in Au cyanide baths include $Fe(CN)_6^{3-}$ and $Fe(CN)_6^{4-}$.

Carbonate (Figure 4) present as a buffering agent in alkaline baths, is also easily determined using an alternate set of conditions. Other ions of interest in gold cyanide based baths that are readily determined by IC include the PO_4^{2-} and citrate content and trace Cl^- contaminents. Free cyanide is also easily determined by using the amperometric detector. If this detector is placed in series with the conductivity detector during the cyanide complex analysis, the free CN^- level would be determined simultaneously from a single injection. Although the majority of the methods developed to-date involve cyanide based baths, applications are being developed for sulfite, thiomalate and electroless gold baths.

COPPER PLATING BATHS

Copper Sulfate

When plating bright copper from a H_2SO_4 bath, a relatively small amount of Cl^- is necessary, as is commonly known, to increase the throwing power of bright copper baths. As illustrated in Figure 5, IC can be utilized for the determination of the low Cl^- level as well as the major constituent (sulfate) level from the same sample injection.

Many copper baths contain ppm levels of organic brighteners, levelers, or carriers. In some cases, these are anionic or cationic and are amenable to separation and detection by IC. Figure 6 illustrates the determination of an anionic brightener in a copper sulfate bath. In this particular case, UV detection was used due to excess sulfate which made conductivity detection impractical. The other peaks were not identified, however as bath use increased, the magnitude of these peaks also increased, possibly due to breakdown of some bath constituents or organic contamination. Although the chemical nature of many additives is proprietary, if some knowledge of the structure can be obtained and the additive is amenable to IC analysis, IC can be used in a monitoring capacity to quickly troubleshoot additive imbalances. The analysis of organic additives can also be applied to other types of plating baths.

In addition to copper, other metals can affect bath performance and plate quality. Figure 7 illustrates the determination of Zn^{2+} and Ni^{2+} contaminants in a $CuSO_4$ plating bath. By further dilution, the major metal content (Cu^{2+}) could also have been determined.

Copper Pyrophosphate

Ammonia improves the uniformity of the deposit in copper pyrophosphate baths as well as assisting in anode corrosion.[8] Since NH_3 is lost by evaporation, due to the bath's elevated temperature, frequent additions of NH_3 are necessary to maintain optimum conditions. Ammonia levels are rapidly determined as illustrated in Figure 8. The levels can then be adjusted if necessary.

Another common component of copper pyrophosphate baths is NO_3^- which increases the allowable maximum current density.[8] Figure 9 illustrates the determination of NO_3^- as well as the HPO_4^{2-} breakdown product of $P_2O_7^{4-}$ formed during the plating process. Using IC, a single sample analysis can determine the NO_3^- level as well as ensuring that the phosphate level is not excessive. Work is under way to develop a method for the determination of Cu^{2+} to $P_2O_7^{4-}$ ratios or the Cu pyrophosphate complex.

Electroless Copper

Major components of an electroless copper bath include formaldehyde and various chelators such as tartrate. During the plating process the formaldehyde is oxidized to formic acid. Both formic and tartaric acids are easily determined by IC (Figure 10). In this way, proper tartrate levels can be maintained, as well as the detection of premature bath aging which is associated with rapidly increasing formate levels. IC is also commonly employed for industrial hygiene monitoring of formaldehyde which is measured as formate following a rapid oxidation with H_2O_2.[9] Although this method has not been applied to the determination of formaldehyde in an electroless copper bath, the approach appears feasible. Other applications that are under development for electroless copper baths include triethanolamine (Figure 11), EDTA, and the $Cu(EDTA)^{2-}$ complex.

NICKEL BATHS

Nickel baths investigated by IC to-date include nickel sulfamate, alloyed Ni/Fe and both electroless Ni and Ni/Co.

Nickel Sulfamate

Nickel sulfamate baths often contain chloride, sometimes added intentionally and other times present as a contaminent. Figure 12 illustrates the determination of Cl^- in a bath containing HCl. Note that the sulfamic acid as well as the SO_4^{2-} breakdown product of the sulfamic acid can be monitored in the same injection with 10 minutes. Trace level chloride determinations would be easily obtained by changing the eluent composition slightly and hence completely resolving chloride from sulfamate. If required, the NH_4^+ breakdown product of the sulfamic acid could also be monitored under different analysis conditions.

Alloyed Nickel Sulfate-Chloride

As well as containing significant levels of Cl^- and SO_4^{2-}, many of the Ni and Ni alloy baths contain H_3BO_3 as a mild buffer. The analytical methods of analysis for borate were previously lacking and most commonly, time consuming titrations with ill-defined endpoints were used. Figure 13 illustrates the determination of borate in a Ni/Fe bath. This technique is applicable to low level analysis as required for waste water. The concentration in the figure corresponds to 3.5 ppm, and the bath concentration is 35 g/L. If analysis of metals is employed, the Ni/Fe ratios can be determined under one set of conditions, or trace contaminents such as Cu^{2+} can be monitored as in Figure 14 and 15 respectively.

Electroless Nickel

Hypophosphite is used as the reducing agent in many pure or alloyed electroless Ni processes. As the bath is plated, the hypophosphite is depleted and phosphite is generated. Analytical methods for differentiation of phosphorous species have not been available and actual levels were commonly determined by difference. Figure 16 contrasts levels of the different phosphorous species in new and used electroless nickel baths. As expected, the hypophosphite level decreased and the phosphite level increased after a period of use. Hence, IC offers a rapid means for process monitoring of these species.

Organic acids are present in electroless nickel baths as mild chelators. Figure 17 illustrates the ease with which these organic acids can be determined. When plating from an alloy bath, the ratio of the metallic ions is an important parameter. Figure 18 illustrates the determination of the Ni/Co ratio in an electroless alloy bath.

Additives

Nickel bath additives include aromatic or alky sulfonic acids or sulfates, such as sodium lauryl sulfate. Although an IC method of analysis exists for these types of species, it has not yet been applied to a plating sample. Figure 19 illustrates a standard separation.

CONCLUSION

Ion Chromatography offers a fast, versatile and simple analytical procedure for determining a multitude of key plating components. Minimal sample preparation and rapid analysis times permit fast process troubleshooting. Furthermore, the comprehensive data generated by IC promotes a sound understanding of gold, copper, and nickel bath chemistry which can be used to optimize process control and economics. IC also permits the analysis of ionic components that were previously difficult or impossible to monitor, which may play a key role in bath performance.

TABLE I

SUMMARY OF SEPARATION MODES

	Predominant Separation Mechanism	Resin Functionality	Standard Eluents	Useful For:
HPIC	Ion Exchange			
Anion	"	$-N^+-R_3$	$Na_2CO_3/NaHCO_3$	Cl^-, NO_3^-, Br^-, SO_4^{2-}, PO_2^{3-}, PO_3^{3-}, CrO_4^{2-}, AsO_4^{3-}
Cation	"	$-SO_3^-$	HCl	Li^+, Na^+, NH_4^+, Rb^+, Cs^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}
Metals	"	$-N^+-R_3$ or $-SO_3^-$	Oxalic and Citric Acid	Fe^{3+}, Fe^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, Co^{2+}, Pb^{2+}, Mn^{2+}, Cd^{2+}, Al^{3+}
MPIC	Ion Pairing and Adsorption	Neutral		
Anion	"	"	Tetrabutyl NH_4OH (TBAOH) CH_3CN	$Au(CN)_2^-$, $Au(CN)_4^-$, $Co(CN)_6^{3-}$ Alkyl sulfonates and sulfates BF_4^-
Cation	"	"	Hexane sulfonic acid (HSA) CH_3CN	Alkyl and quaternary amines
HPICE	Ion Exclusion	Totally functional $-SO_3^-$	HCl	Formate, citrate, tartrate, succinate, lactate, carbonate, borate

TABLE II

DETECTION MODES

	Principle	Most Useful For:
Suppressed Conductivity	Electrical conductivity	Anions or cations with $pK_{a\ or\ b}$ values <7 eg. Cl^-, SO_4^-, Na^+
Amperometric	Oxidation or reduction at Ag or Pt electrodes	CN^-, S_2^-, mercaptans, OCl^-, hydrazine
Pulsed Amperometric	Oxidation or reduction at Ag, Pt, Au, or glassy carbon electrodes	Phenolics, carbohydrates, nitro aromatics
Direct UV VIS after PCR	Ultraviolet light absorption Visible light absorption after reaction with a post column reagent	Aromatic organics and select anions Transition and heavy metals after post column reaction with 4-(2-pyridylazo)-resorcinol (PAR) Polyvalents after reaction with $Fe(NO_3)_3$
Fluorescence after PCR	Excitation and Emission	Amino acids after reaction with o-phthalaldehyde

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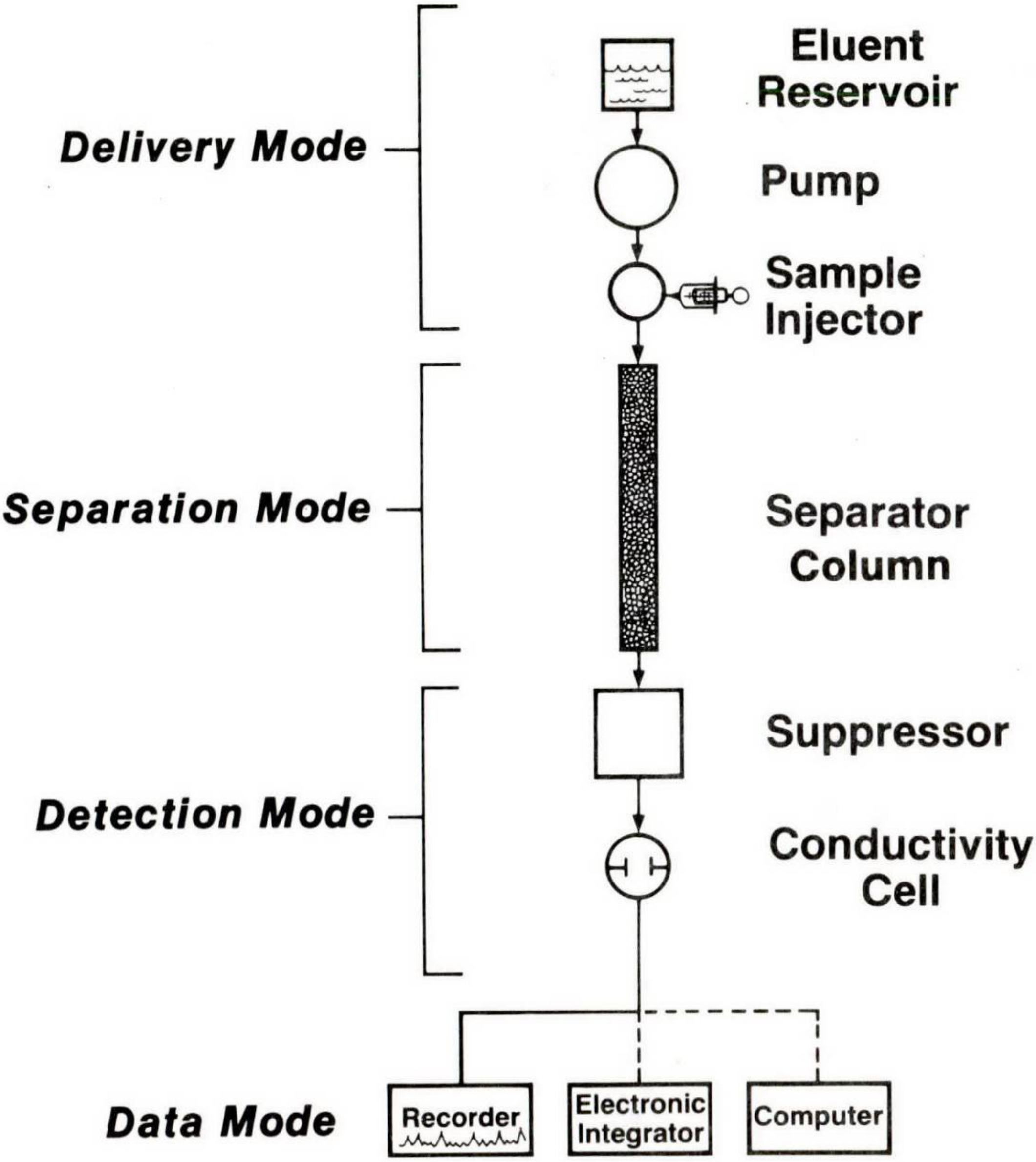


Figure 1. Schematic representation of the Ion Chromatographic system.

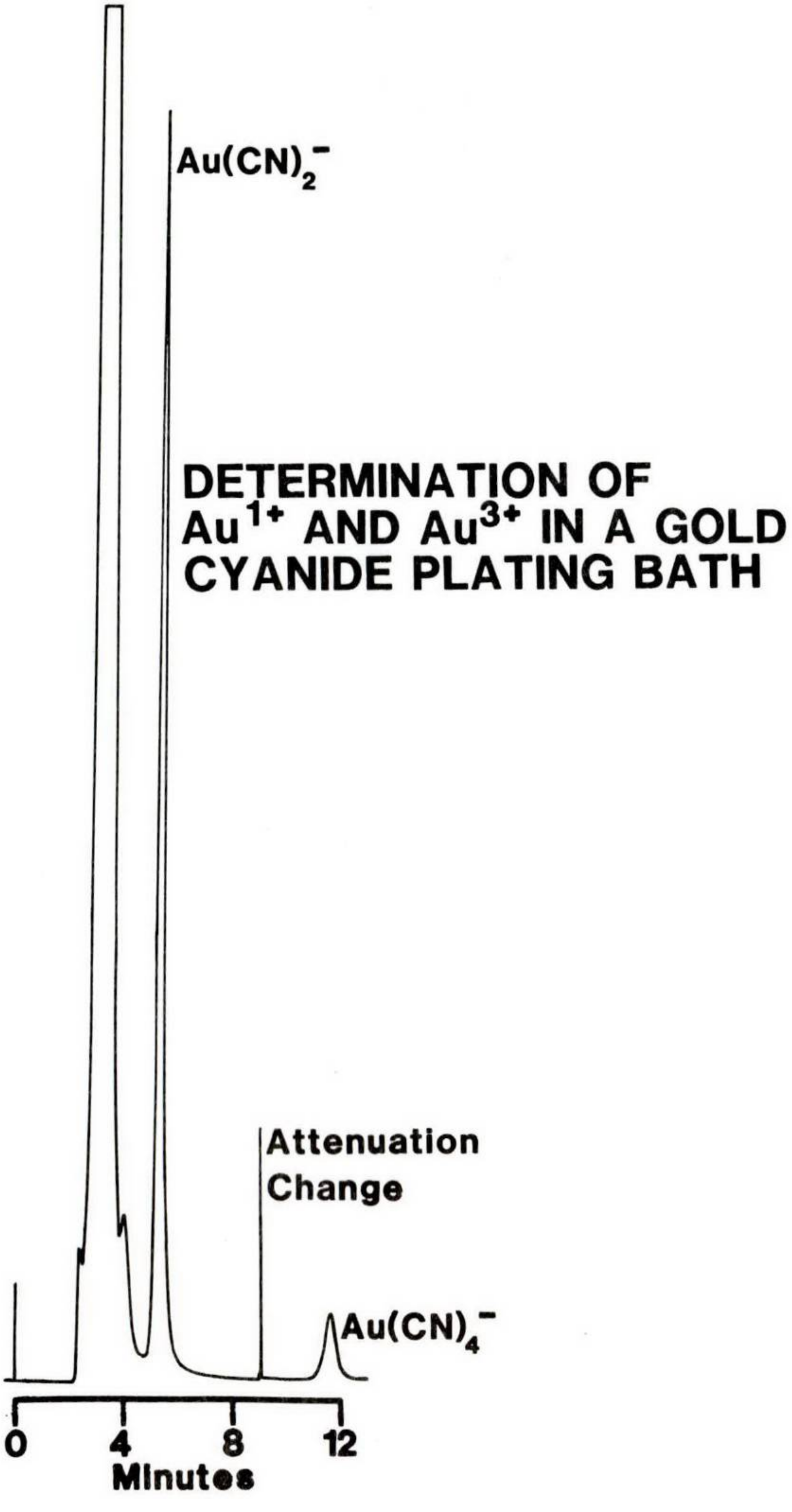
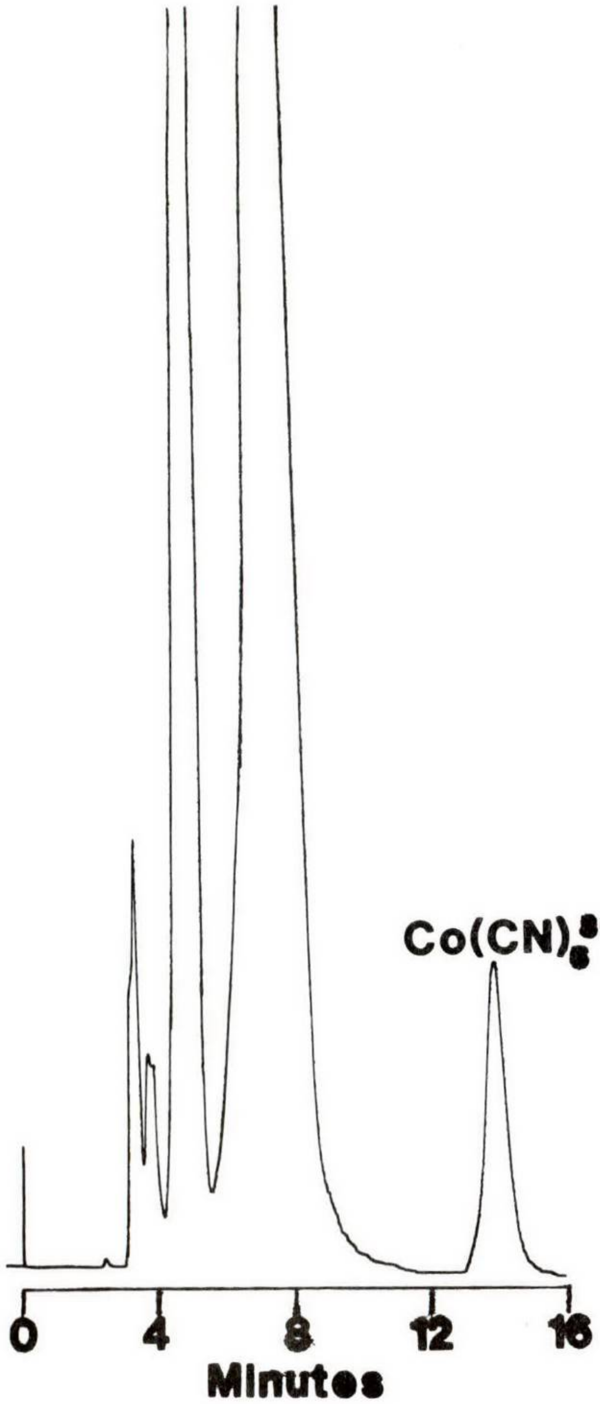
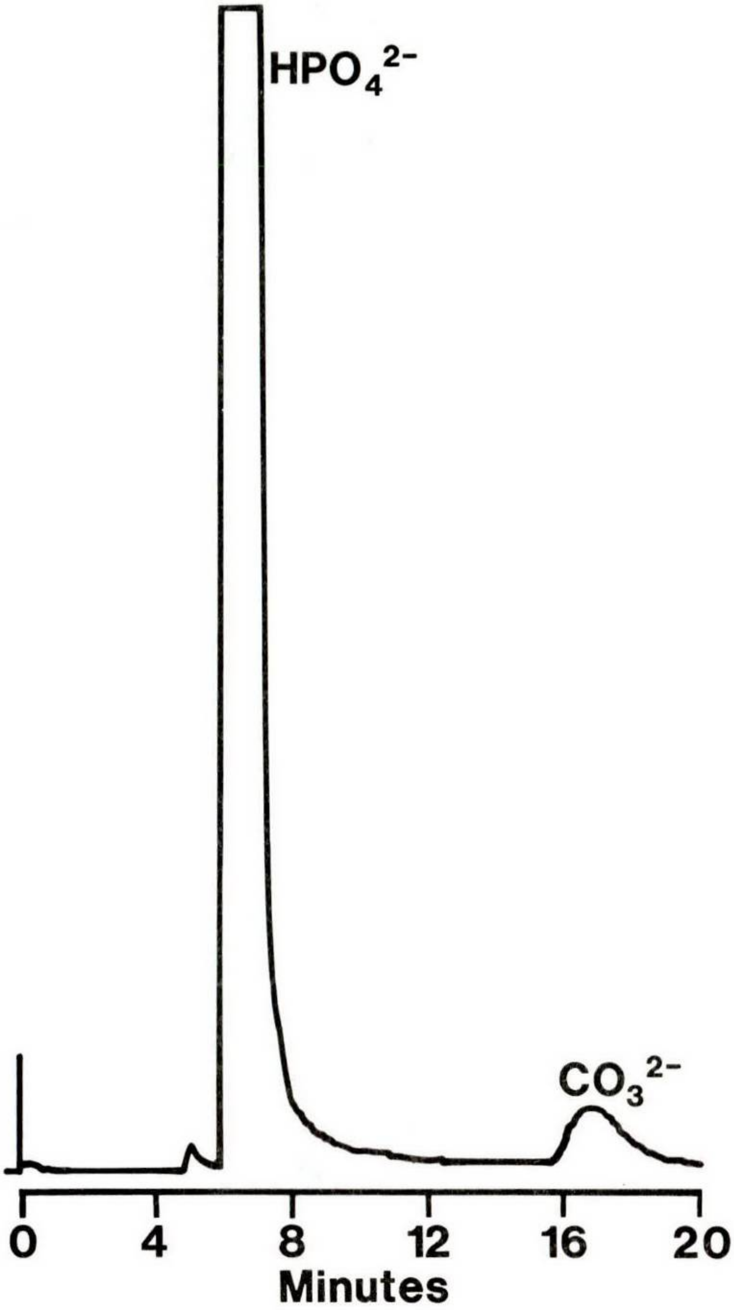


Figure 2. Columns: 1 MPIC-NG1 and 1 MPIC-NS1. Detection: ASC-1, conductivity at 30 and 3 µS. Sample: 50 µl of .5/100 dilution of bath. Eluent: 2 mM TBAOH, 40% CH_3CN, 1 mM Na_2CO_3.



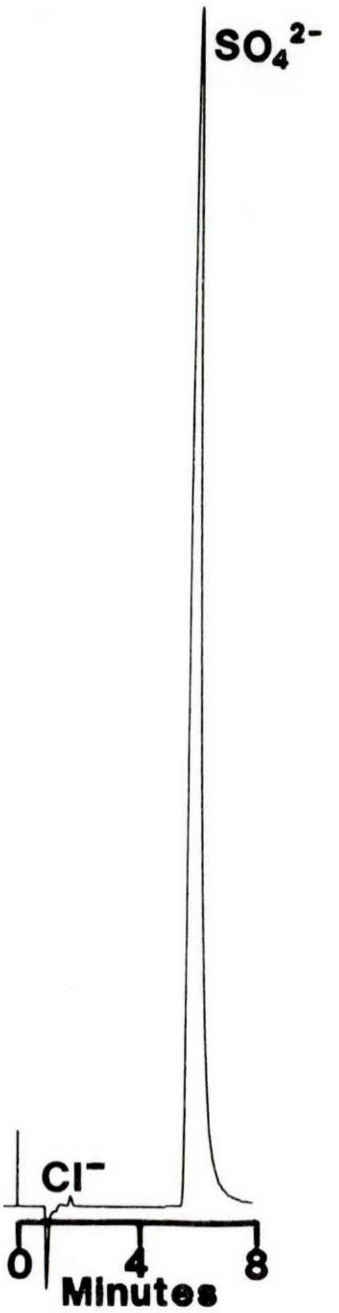
$Co(CN)_6^{3-}$ IN A PHOSPHATE-CITRATE BUFFERED GOLD PLATING BATH

Figure 3. Columns: 1 MPIC-NG1, 1 MPIC-NS1. Detection: ASC-1, conductivity at 10 μS. Sample: 50 μl of .25/100 dilution of bath. Eluent: 2 mM TBAOH, 10% CH_3CN.



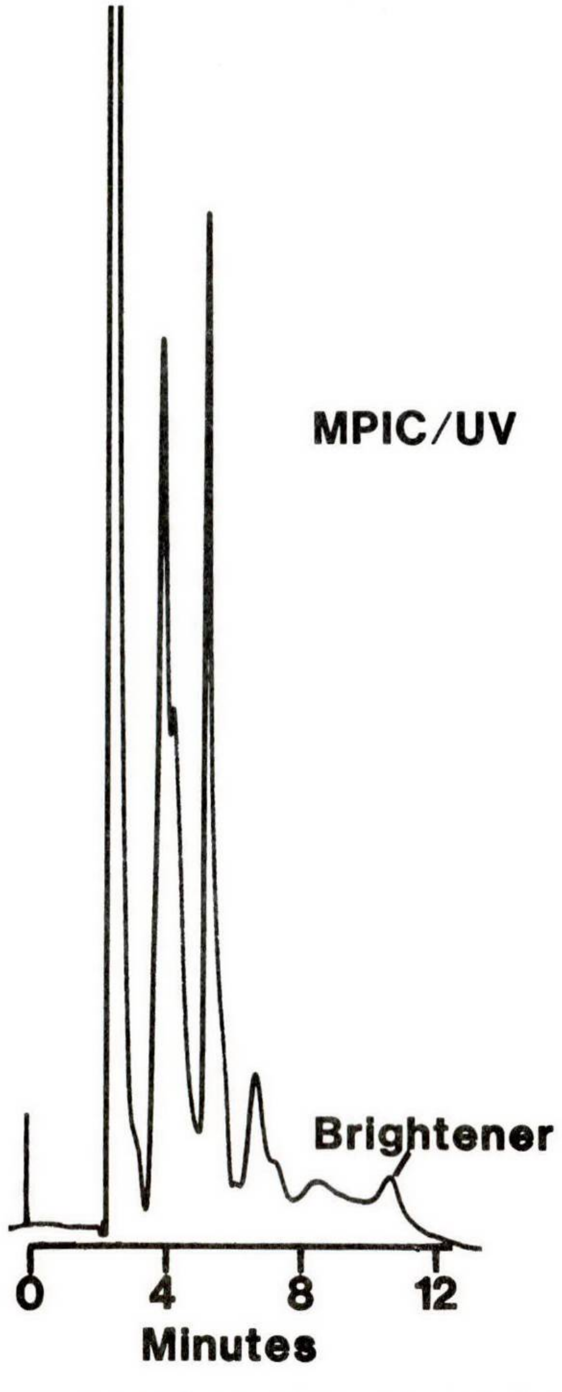
CARBONATE IN A
GOLD CYANIDE PLATING BATH

Figure 4. Columns: 1 HPICE-AS1. Detection: ISC condictivity 10 μS. Sample: 50 μl of 1/100 dilution of bath. Eluent: .005 M HCl, .8 ml/min.



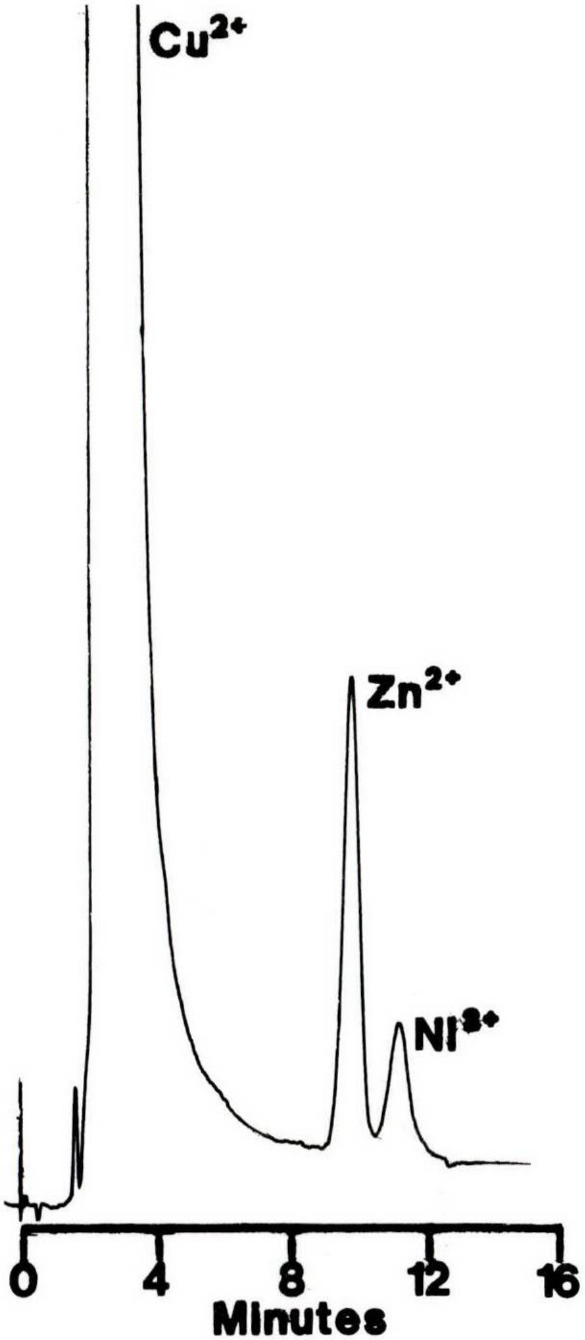
TRACE Cl^- IN A
$CuSO_4$ PLATING BATH

Figure 5. Columns: 1 HPIC-AG4, 1 HPIC-AS4. Detection: AFS, conductivity at 30 μS. Sample: 10 μl of 1/1000 dilution of bath. Eluent: standard anion, .0028 M $NaHCO_3$/.0023 M Na_2CO_2.



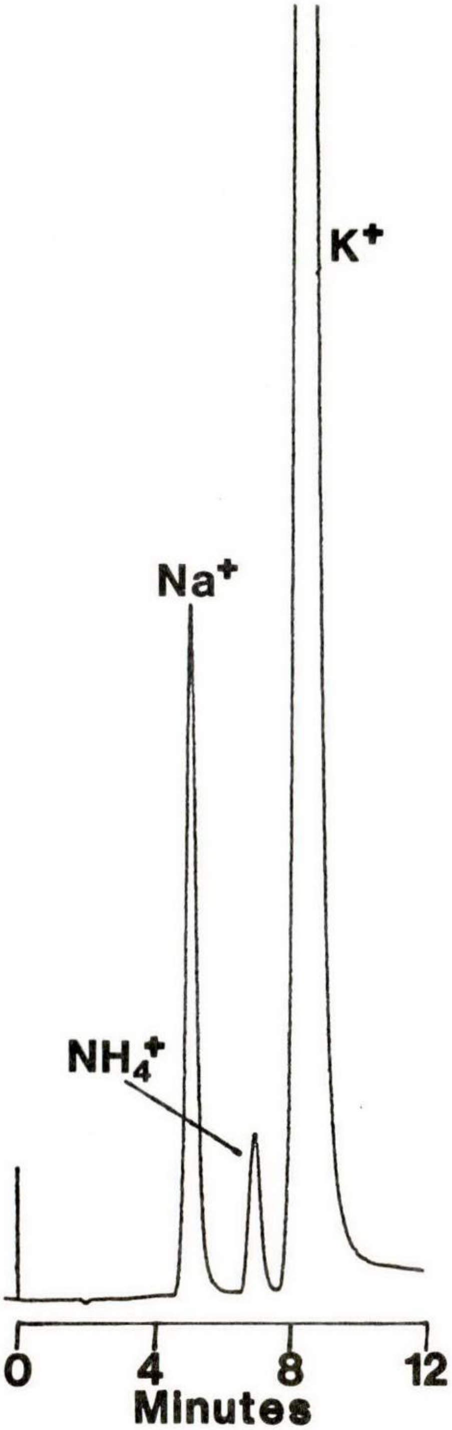
DETERMINATION OF A DISULFIDE ANIONIC BRIGHTENER IN A $CuSO_4$ PLATING BATH

Figure 6. Columns: 1 MPIC-NG1, 1 MPIC-NS1. Detection: UV, 215 nm .02 AUFS. Sample 50 µl of 1/2 dilution of bath. Eluent: 2 mM TBAOH, 25% CH_3CN, .2 mM Na_2CO_2.



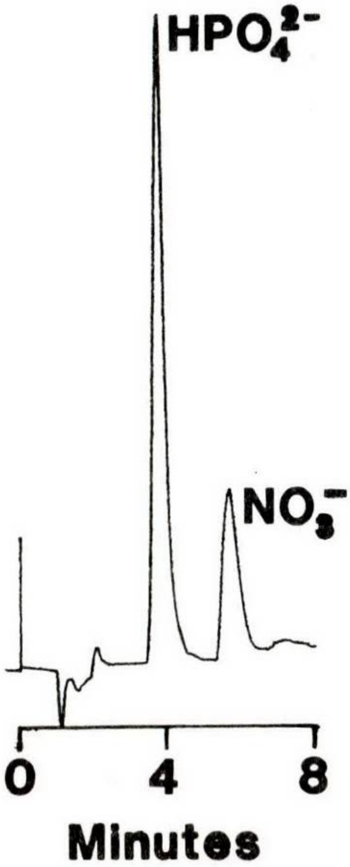
METAL CONTAMINANTS IN AN ACID COPPER PLATING BATH

Figure 7. Columns: 1 HPIC-AS4, Detection: Visible, 520 nm after post column reaction with PAR, 1 AUFS. Sample: 50 µl of 1/10 dilution of the bath. Eluent: 25 mM oxalic acid.



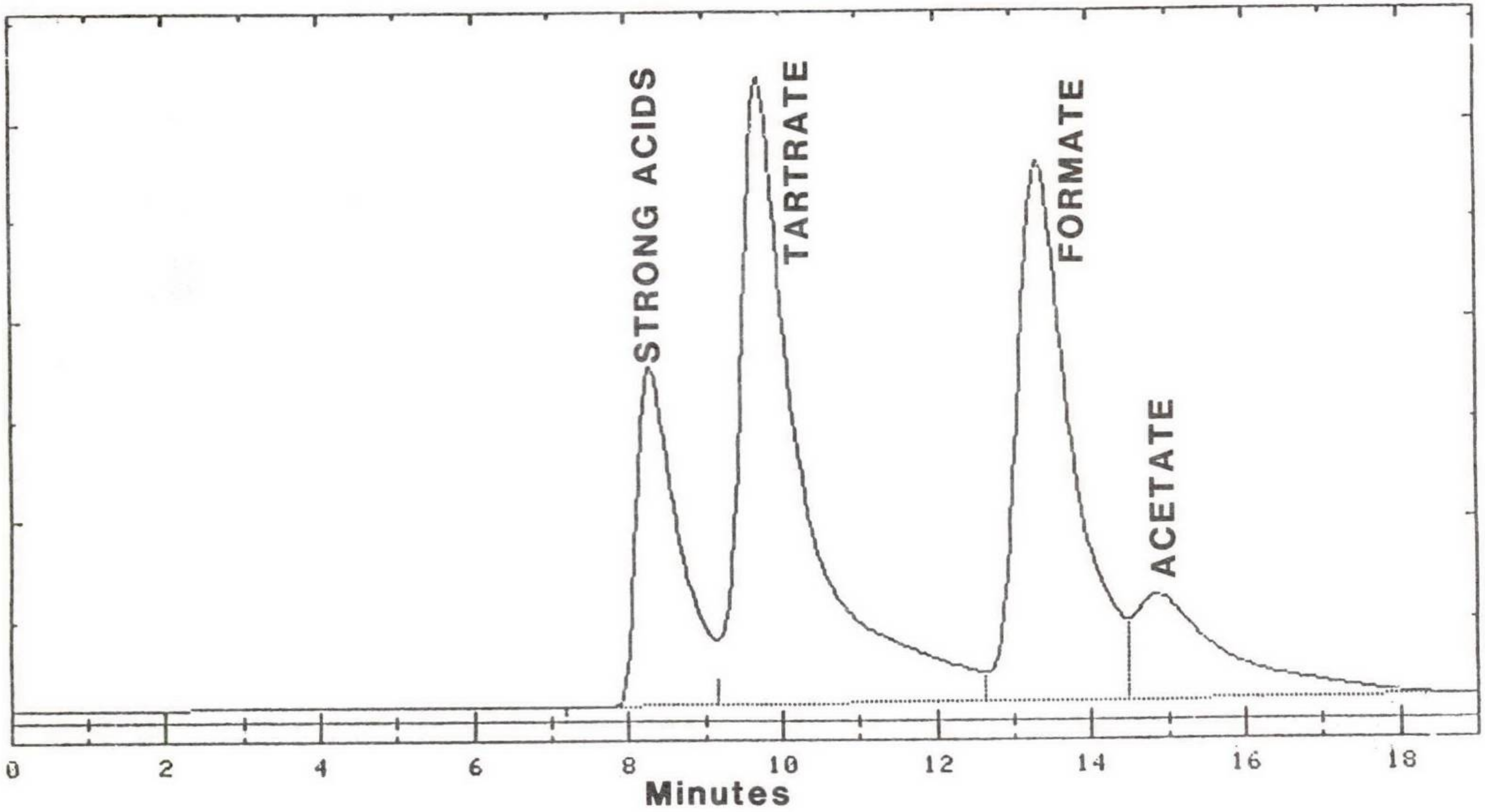
AMMONIA IN A COPPER PYROPHOSPHATE PLATING BATH

Figure 8. Columns: 1 HPIC-CS1, Detection: 1 CSC-1, 10 µS. Sample: 50 µl of 1/2500 dilution, Eluent: .005 M HCl.



ANIONS IN A COPPER PYROPHOSPHATE PLATING BATH

Figure 9. Columns: 1 HPIC-AG3, 1 HPIC-AS3. Detection: AFS, 10 µS. Sample: 10 µl of 1/1000 dilution. Eluent: .003 M $NaHCO_3$/ .0024 M Na_2CO_3.



ORGANIC ACIDS IN AN ELECTROLESS COPPER BATH

Figure 10. Columns: 1 HPICE-AS1, Detection: ISC, conductivity at 10 µS. Sample: 50 µl of 1/500 dilution. Eluent: .001 M HCl.

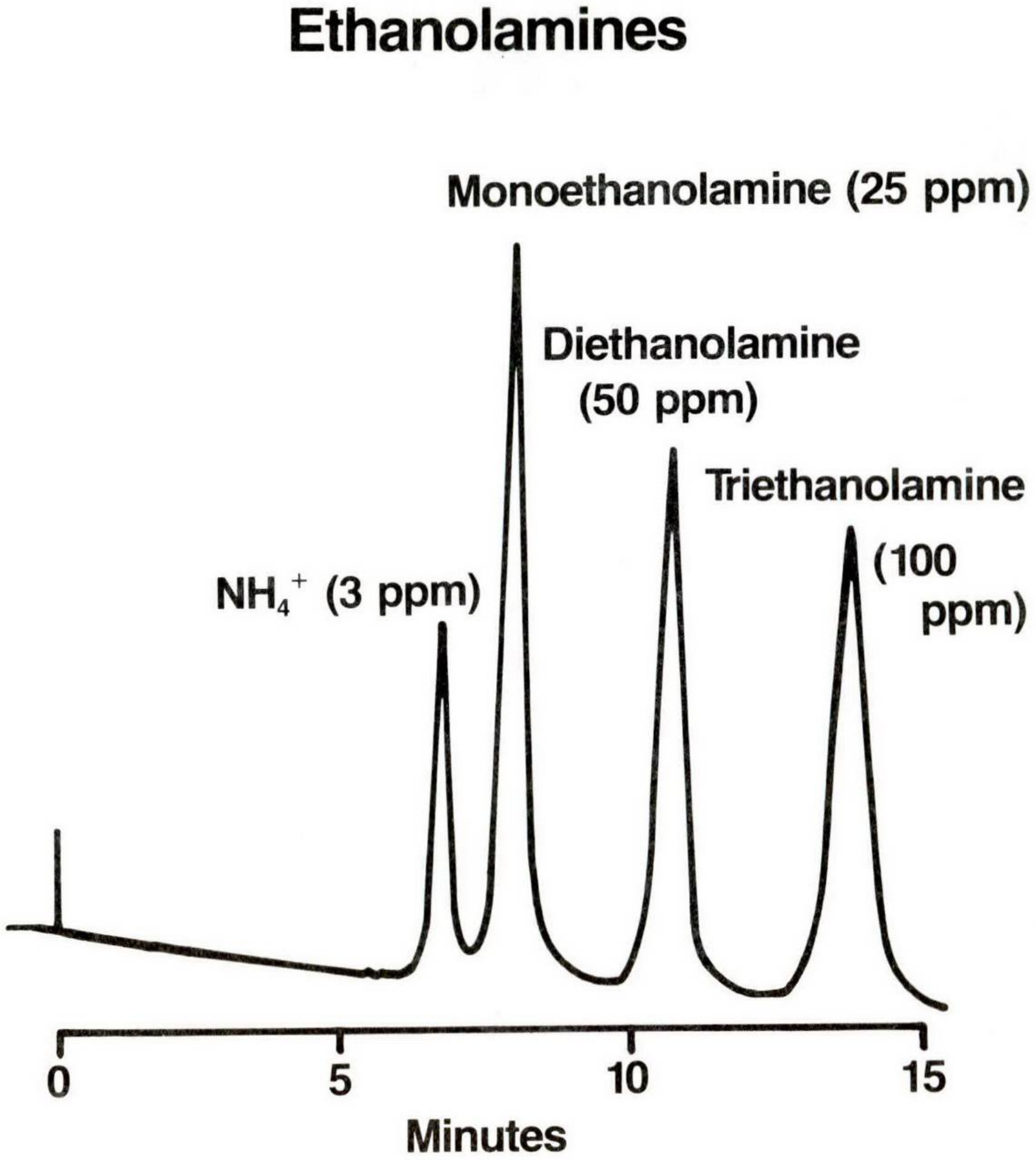
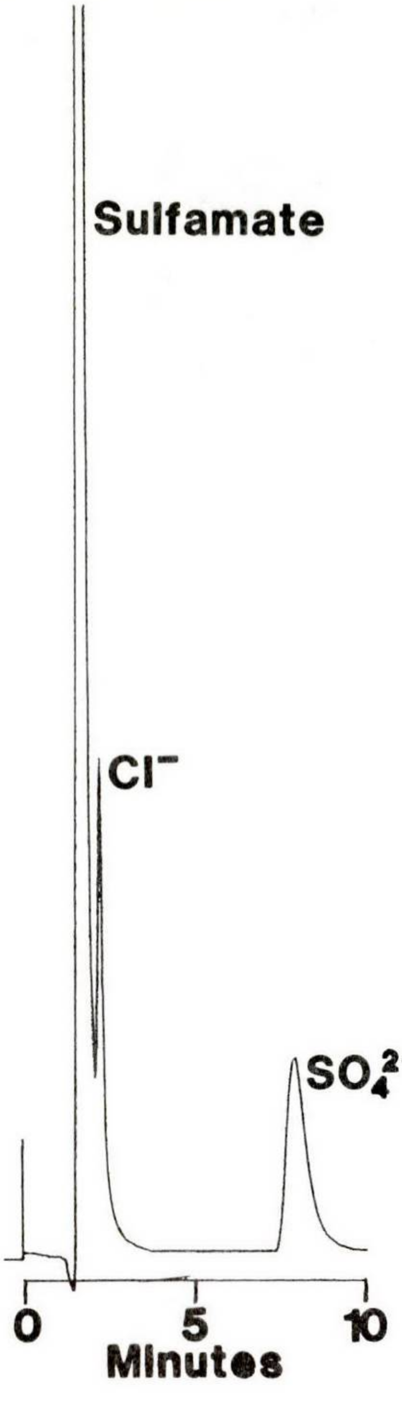


Figure 11. Columns: 1 MPIC-NG1, 1 MPIC-NS1, Detection: 1 CSC-1, conductivity 10 μS. Sample: 50 μl of standard ethanolamines. Eluent: 2 mM hexane sulfonic acid.



ANIONS IN A NICKEL SULFAMATE PLATING BATH

Figure 12. Columns: 1 HPIC-AG4, 1 HPIC-AS4, Detection: AFS, conductivity 10 μS. Sample: 50 μl of 1/500 dilution of bath. Eluent: .0028 M $NaHCO_3$/.0023 M Na_2CO_3.

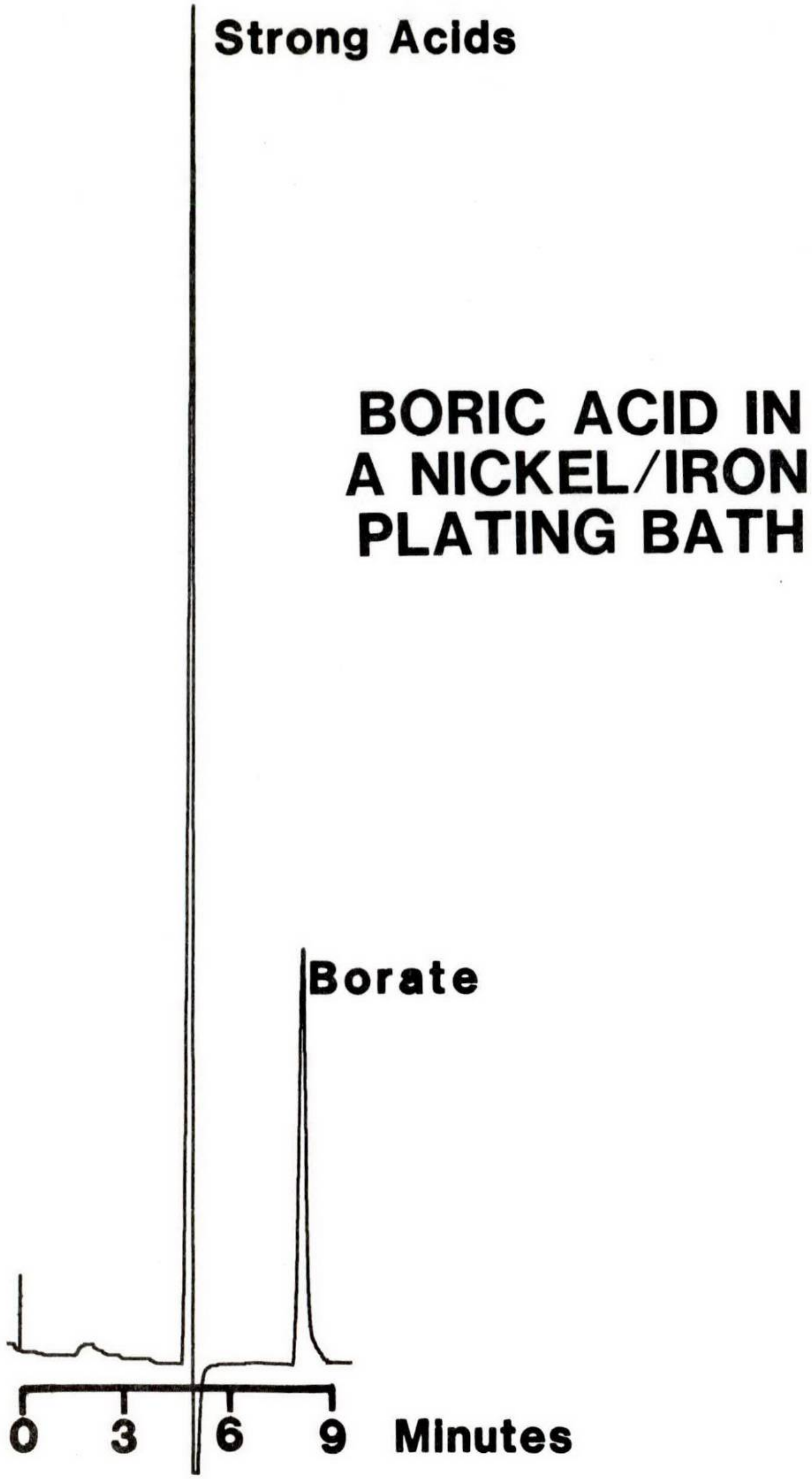
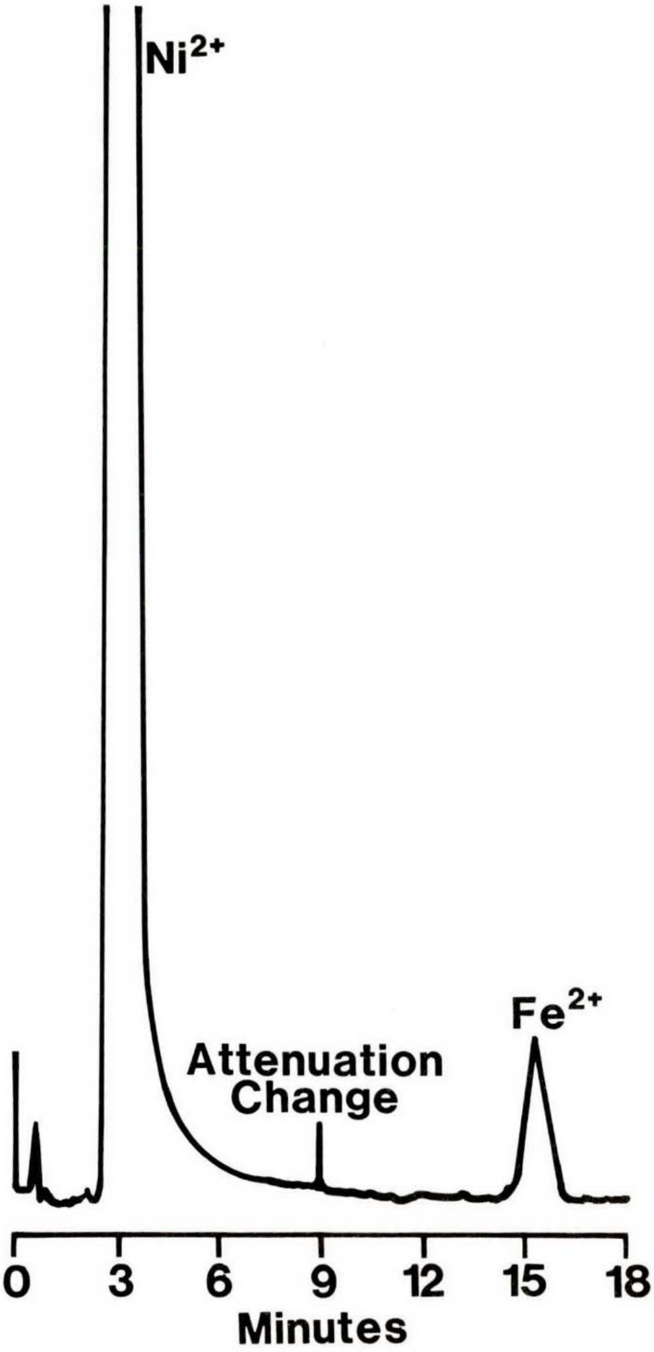
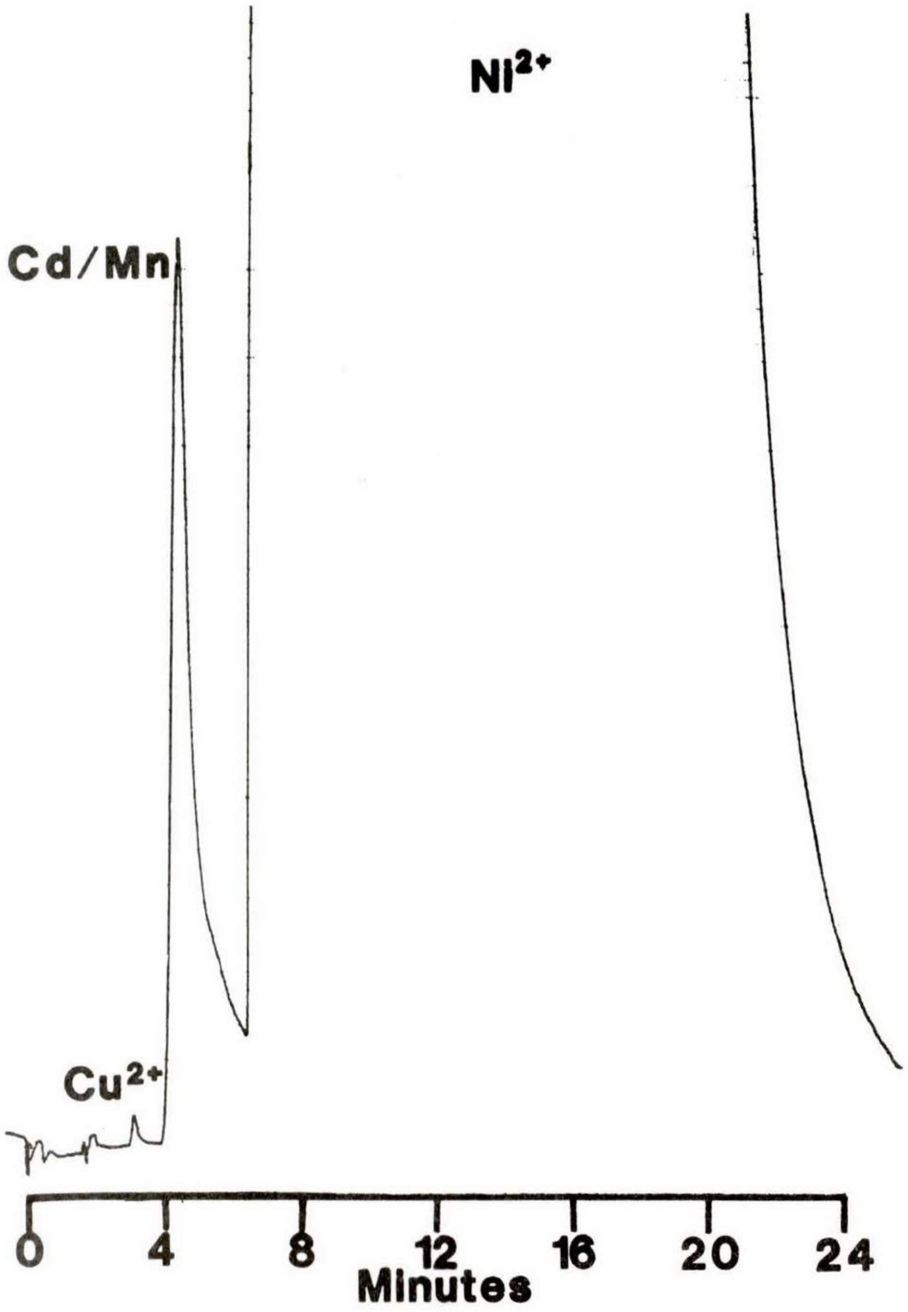


Figure 13. Column: 1 HPICE-1, suppressed conductivity, 10 µS. Sample: 50 µl of 1/1000 dilution.



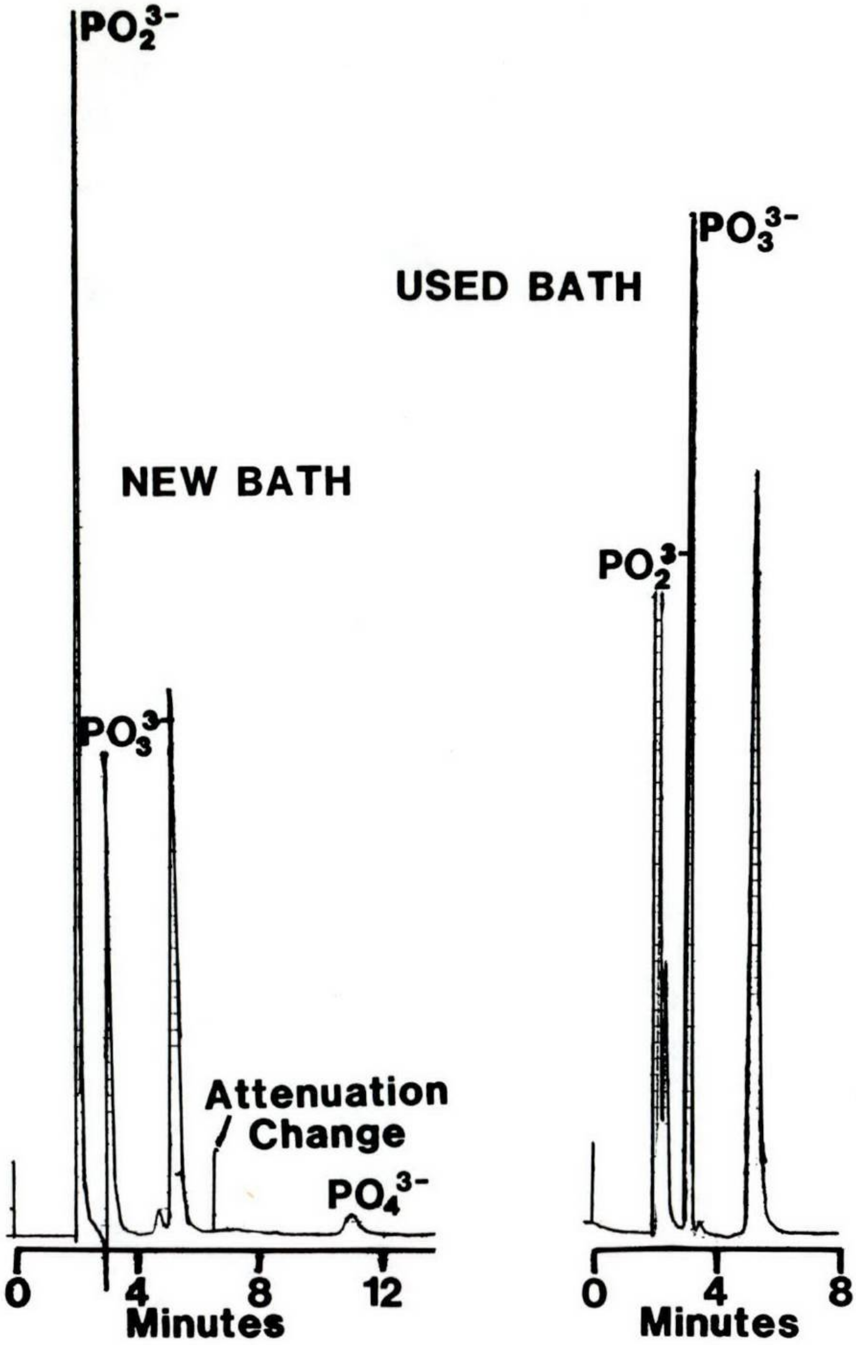
TRANSITION METALS IN
A NICKEL-IRON PLATING BATH

Figure 14. Columns: 1 HPIC-CG2, 1 HPIC-CS2. Detection: Visible, 520 nm after post column reaction with PAR. .2 AUFS, .1 AUFS. Sample: 50 µl of 1/500 dilution of bath. Eluent: 10 mM oxalic acid/7.5 mM citric acid, pH 4.30.



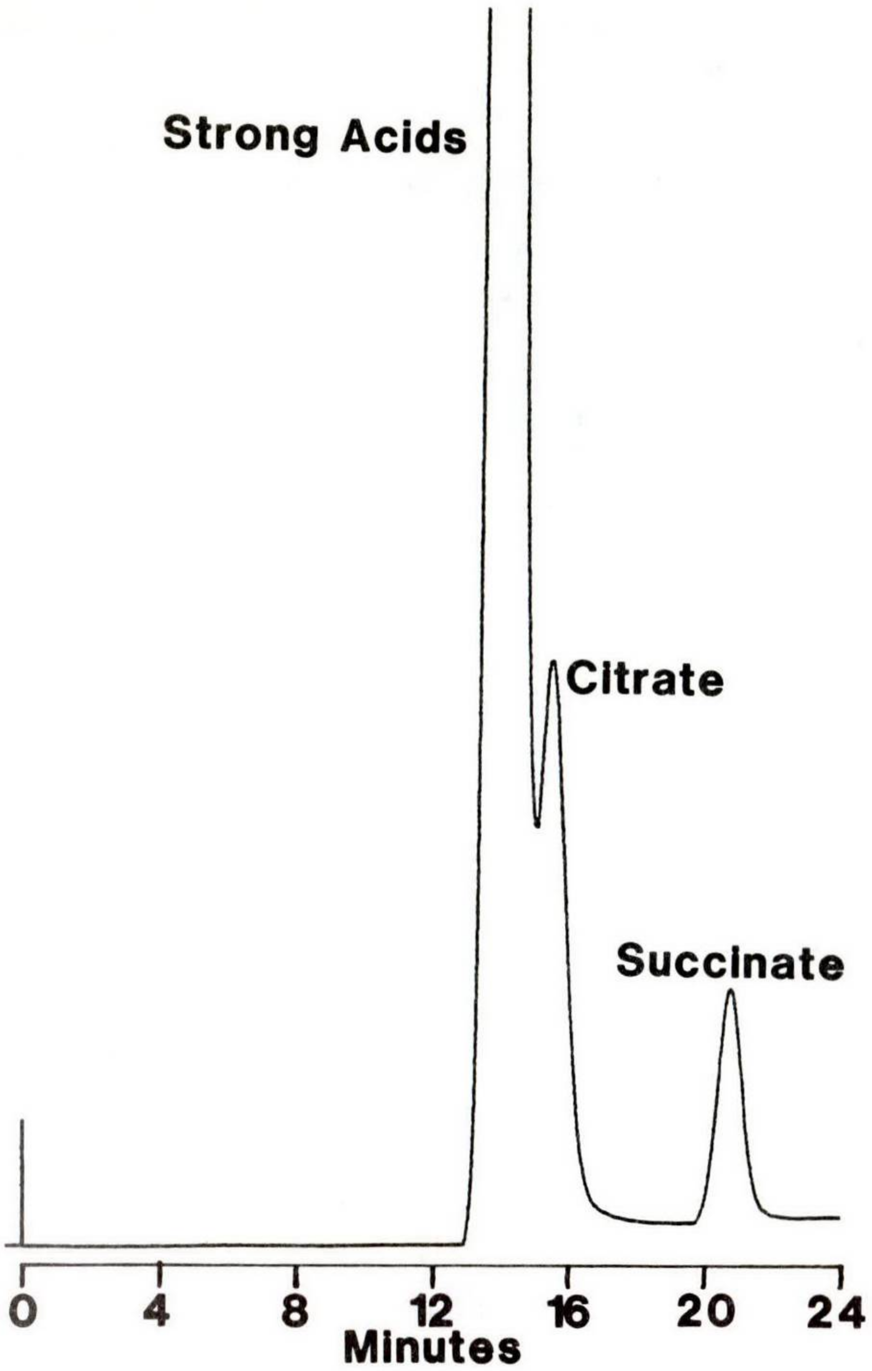
METAL CONTAMINANTS IN A NICKEL/IRON PLATING BATH

Figure 15. Column: 1 HPIC-AS4, Detection: Visible, 520 nm after post column reaction with PAR. Sample: 50 µl of 1/10 dilution of bath .1 AUFS. Eluent: 25 mM oxalic acid.



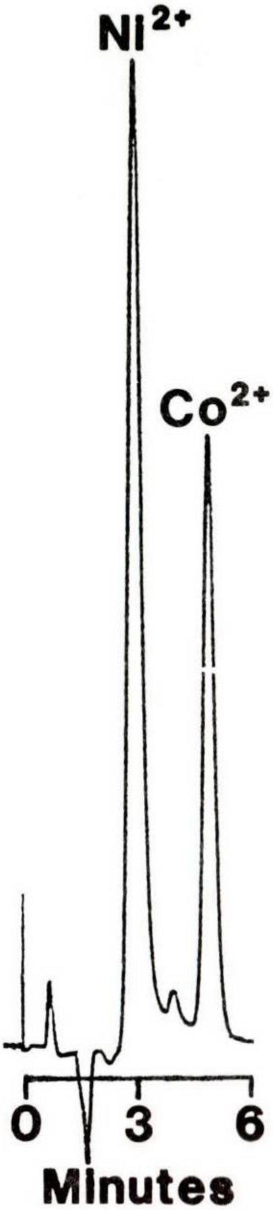
THE DETERMINATION OF HYPOPHOSPHITE, PHOSPHITE, AND PHOSPHATE IN ELECTROLESS NICKEL BATHS

Figure 16. Column: 1 HPIC-AG3, 1 HPIC-AS3. Detection: ASC-2 conductivity 30 µS. Sample: 50 µl of 1/5000 dilution, Eluent: 3 mM Na_2CO_3, pH 12 with NaOH.



ORGANIC ACIDS IN AN ELECTROLESS NICKEL PLATING BATH

Figure 17. Column: 1 HPICE-AS2, Detection: ISC, conductivity 10 µS. Sample: 50 µl of 1/1000 dilution. Eluent: .001 M HCl.



NICKEL & COBALT IN AN ELECTROLESS Ni/Co PLATING BATH

Figure 18. Column: 1 HPIC-CS2, Detection: Visible, 520 nm, after post column reaction with PAR. Sample: 10 µl of 1/1000 dilution. Eluent: 25 mM oxalic acid.

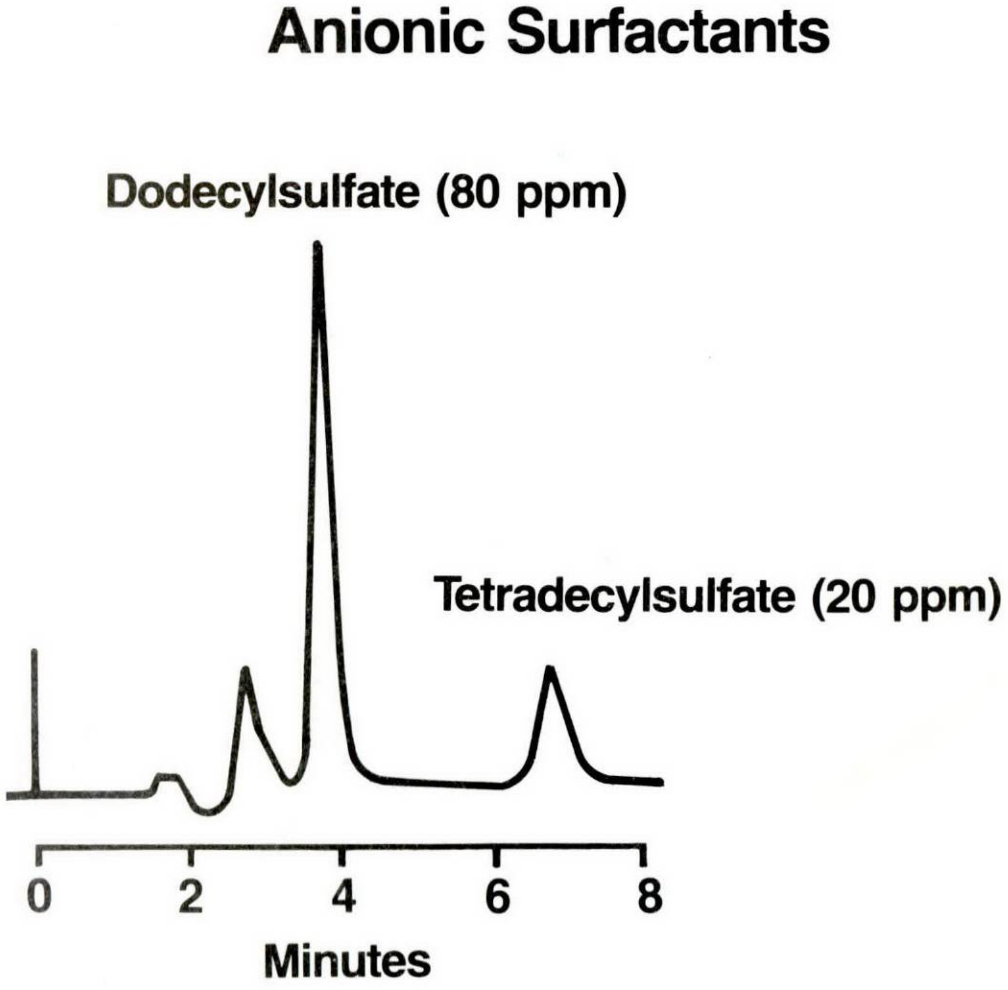


Figure 19. Column: 1 MPIC-NS1, Detection: 1 ASC-1, conductivity at 10 µS. Sample: 50 µl of standard solution. Eluent: 10 mM NH_4OH/32% CH_3CN.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