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THE DETERMINATION OF ALUMINUM, COPPER, IRON AND LEAD IN GLYCOL FORMULATIONS BY ATOMIC ABSORPTION SPECTROSCOPY

Prepared by

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Division of PPG Industries, Inc.
One Gateway Center
Corpus Christi, Texas

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16. ABSTRACT

This report describes initial screening tests and the results obtained in developing procedures to determine Al, Cu, Fe, and Pb in glycol formulations. Atomic absorption completion was selected for Cu, Fe and Pb, and after comparison with emission spectroscopy, was selected for Al also. Before completion, carbon, iron, and lead are extracted with diethyl-thiocarbamate (DDC) into methyl isobutyl ketone (MIBK). Aluminum was also extracted into MIBK using 8-hydroxyquinoline as a chelating agent. As little as 0.02 mg/ℓ carbon and 0.06 mg/ℓ lead or iron may be determined in glycol formulations. As little as 0.3 mg/ℓ aluminum may be determined.

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The Determination of Al, Cu, Fe and Pb in Glycol Formulations by Atomic Absorption Spectroscopy

I. INTRODUCTION

The Research Analytical Department of the Corpus Christi Technical Center has been requested to develop procedures for the determination of low levels of aluminum, copper, iron, and lead in aqueous glycol formulations. It was hoped that the analysis of these solutions would permit monitoring of the extent of corrosion occurring in tests being carried out by Houston Chemical Corporation to evaluate various materials of construction for use in solar panel systems without requiring the dismantling of panels for examination.

This report shows the results obtained in developing procedures for Al, Cu, Fe, and Pb determinations in glycol formulations and in their application to a series of samples from preliminary corrosion tests.

II. ABSTRACT

This report describes initial screening tests and the results obtained in developing procedures to determine Al, Cu, Fe, and Pb in glycol formulations. Atomic absorption completion was selected for Cu, Fe and Pb and, after comparison with emission spectroscopy, was selected for Al also. Prior to completion Cu, Fe, and Pb are extracted with diethyldithiocarbamate (DDC) into methyl isobutyl ketone (MIBK). Aluminum was also extracted into MIBK using 8-hydroxyquinoline as a chelating agent. As little as 0.02 mg/l Cu and 0.06 mg/l Pb or Fe may be determined in glycol formulations. As little as 0.3 mg/l Al may be determined.

III. EXPERIMENTAL AND RESULTS

An initial survey of a series of four glycol formulation samples was performed by filtering solids from the samples and diluting the filtrate (5 ml to 50 ml) with deionized water. The diluted solutions were read for Cu, Fe, Pb, and Al on the AA spectrophotometer. The solid portions of these samples were then fired on the emission spectrograph to obtain a semiquantitative analysis of the elements present. The results of the AA determinations are presented in Table I.

TABLE I

Dissolved Phase Analysis in Initial Screening by AA

Sample	Description	Concentration (mg/l) Cu	Fe	Pb	Al
I-6067	50% Zerex Before Static Test	<1	2	<5	<5
I-6068	50% 420 AF Before Static Test	<1	2	<5	<5
I-6069	50% Zerex From Panel Test	<1	<1	<5	<5
I-6070	50% 420 AF From Panel Test	<1	<1	<5	<5

The results of the emission spectrographic determinations are presented in Table II.

TABLE II

Solid Phase Determination in Initial Screening by Emission Spectrograph

Sample	Description	Estimated Concentration Ranges, (Wt. %) 10+	1 - 10	0.1 - 1	0.01 - 0.1
I-6073	Residue From I-6067	Fe and Si major (insufficient sample for est.)			
I-6074	Residue From I-6068	Si major (insufficient sample for est.)			
I-6075	Residue From I-6069	Fe, Al, Na	P, Sn, Pb, Cu, Si, Zn Ca		B, Cr, Mn, Ti, Mg, Ni
I-6076	Residue From I-6070	Si, Fe, Cu Zn	P, Sn, Pb, Ca	Mn, Al, Na	Mg, Cr

Following the initial screening determinations it was decided to develop extraction procedures to remove the metal ions from their glycol matrix and to concentrate them prior to their analysis. Cu, Fe and Pb appeared to best lend themselves to AA determination so a procedure was developed in which the cations are complexed with DDC and are extracted into MIBK, with the MIBK phase then being read on the AA spectrophotometer (CCTC Method AA-4D). This procedure was applied to a series of solutions from accelerated glycol corrosion tests and the results are presented in Table III, together with the weight loss results from the corrosion test.

TABLE III

Results of CCTC Method AA-4D and Sample Weight Losses for Accelerated Corrosion Tests

		Cu			Pb			Fe		
Sample	Description	mg/l	mg from Analysis	mg from Wt. Loss	mg/l	mg from Analysis	mg from Wt. Loss	mg/l	mg from Analysis	mg from Wt. Lo
Ethylene Glycol Formulations										
I-6089	LG048 Unexposed	0.007	0.069	6.9	0.03	0.11	14.1	1.00	-0.71	0.7
I-6082	LG048 Exposed	0.10			0.18			0.05		
I-6090	LG049 Unexposed	0.01	0.068	5.7	0.03	0.17	10.5	0.90	-0.65	2.1
I-6083	LG049 Exposed	0.10			0.25			0.07		
Propylene Glycol Formulations										
I-6091	LG052 Unexposed	0.02	8.5	16.0	0.02	0.08	1.2	0.20	-0.11	20.5
I-6078	LG052 Exposed	11.3			0.12			0.06		
I-6085	LG053 Unexposed	0.02	6.2	8.8	0.05	0.24	1.9	0.18	0.20	2.4
I-6079	LG053 Exposed	8.3			0.37			0.45		
Triethylene Glycol Formulation										
I-6086	LG054 Unexposed	0.007	3.3	13.5	0.05	0.05	2.3	0.28	-0.17	8.2
I-6080	LG054 Exposed	4.4			0.12			0.06		

The negative values shown for Fe in the " mg from Analysis" column indicate that the solutions actually lost Fe concentration during the test. The " mg from Wt. Loss" column shows the amount of metal lost from the corrosion coupons during the tests and should equal the " mg from Analysis" column for a given pair of samples if all the metal lost from the coupons was recovered in the solution analysis. No significant amount of solids was present in any of these samples as presented for analysis, although it is understood that solids were scrubbed from the corrosion coupons after the exposure and were discarded prior to reweighing the corrosion coupons to determine weight losses.

It was observed in developing procedure CCTC AA-4D that the efficiency of extraction of the metal ions into the MIBK phase was strongly dependent on the glycol concentration of the aqueous phase. As the glycol content of the aqueous phase increases, the chelated metal ion distribution shifts toward the aqueous phase during the extraction. For this reason it is very important to establish calibrations for each sample dilution by making an appropriate standard addition.

Aluminum is reportedly most sensitively determined by emission spectroscopy.[1] Accordingly, an initial attempt was made to determine aluminum in glycol by chelating into carbon tetrachloride with 8-hydroxyquinoline and evaporating the CCl_4 onto NaCl which was subsequently fired in the emission spectrograph. This procedure yielded no significant correlation of aluminum content with emission spectrographic results.

Next, aluminum in 40-ml portions of 50% ethylene glycol formulation was chelated with 8-hydroxyquinoline and extracted into MIBK. This extract was then read directly on the AA spectrophotometer and, after addition of chromium as an internal standard and evaporation on an electrode, was fired on the emission spectrograph. Results are presented in Table IV.

TABLE IV

Comparison of Emission Spectrographic and AA Results for Al in MIBK Extracts

Conc. of Al Added (mg/l)	AA Response (Absorbance)	Emission Spec. Response (Al, λ = 3092Å vs. Cr, λ = 3091Å)
0	0.001	1.63
0.5	0.002	3.4
1.0	0.005	3.2
2.0	0.010	4.2

While the AA response for Al shown in Table IV is weak, it is clearly preferable to the emission spectrographic responses. Accordingly, a procedure involving the AA completion of MIBK extracts for Al was developed (CCTC Method AA-9A) and applied to the same series of samples presented earlier in Table III. The results of this application are shown in Table V.

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[1] "Treatise on Analytical Chemistry" , Kolthoff and Elving, Part II, Volume 4, Page 394.

TABLE V

Results of CCTC Method AA-9A and Sample Weight Losses on Accelerated Corrosion Tests

Sample	Description	Al mg/1	mg from Analysis	mg from Wt. Loss
Ethylene Glycol Formulations				
I-6089	LG048 Unexposed	<0.2	<-0.08	8.2
I-6082	LG048 Exposed	<0.1		
I-6090	LG049 Unexposed	<0.2	<-0.08	9.2
I-6083	LG049 Exposed	<0.1		
Propylene Glycol Formulations				
I-6091	LG052 Unexposed	<0.5	<-0.2	14.5
I-6078	LG052 Exposed	<0.2		
I-6085	LG053 Unexposed	<0.5	----	21.0
I-6079	LG053 Exposed	0.5		
Triethylene Glycol Formulation				
I-6086	LG054 Unexposed	<0.3	<-0.2	9.4
I-6080	LG054 Exposed	<0.1		

As pointed out for Table III, the " mg from Analysis" and " mg from Wt. Loss " results in Table V would be the same if the physical loss from the coupons and the weight of metal picked up by the solutions were the same. The negative values again indicate a loss of metal from solution during the test, although in general these results only reflect the different limits of detection for the various solutions, and definite losses or gains in the metal contents of the solutions could not be identified although they were shown to be very small in comparison with the physical losses observed from corrosion coupons.

The limit of detection variations for Al shown in Table V reflect different recovery efficiencies of Al standard additions made to various samples to establish calibration for the respective samples. These differences did not appear connected to glycol content in the extraction media and the reason for the observed variations is not known.

IV. DISCUSSION

Tables III and V show there is very little correlation between the weight losses observed for the corrosion coupons and the dissolved metals found in the corrosion test fluids. Further, the fact that very few solids were present in the fluids does not encourage speculation that a means of collecting undissolved corrosion products in test fluids might offer a means of monitoring the progress of corrosion during tests. Discussions with the technician who performed the accelerated corrosion tests indicate that most of the corrosion products adhered to the test coupons and were washed and scrubbed free (and discarded) prior to taking final weights of the coupons.

The data in Tables III and V suggest that Cu, Fe, Pb and Al may have low solubilities in many aqueous glycol solutions. This is particularly true for Fe which apparently actually dropped out of solution during the test. Fe appears to be more soluble in the essentially pure glycol formulation sampled before the test than in the aqueous solution sampled afterward. Cu appears to be an exception to the low solubility in aqueous solutions indicated for the other elements, being soluble in the aqueous propylene glycol formulation and to a lesser extent the aqueous triethylene glycol formulation. It should be mentioned that a pure ethylene glycol formulation stored in a metal container was found to contain 3.5 mg/1 Pb, indicating considerable solubility of lead in the pure glycol. Similar formulations stored in plastic all contained less than 0.1 mg/1 Pb.

V. CONCLUSIONS

1. There is little correlation between corrosion of metal test coupons and a metal analysis of the test fluid.

2. Most corrosion products must have remained on or near the actual site of corrosion until physically removed during coupon cleaning.

3. Cu at levels down to 0.02 mg/1 and Fe and Pb at levels down to 0.06 mg/1 in glycol formulations can be determined by extraction into MIBK with diethyldithiocarbamate and AA completion (CCTC Method AA-4D).

4. Al at levels as low as 0.3 mg/1 in glycol formulations can be determined by extraction into MIBK with 8-hydroxyquinoline followed by AA completion (CCTC Method AA-9A).

VI. RECOMMENDATIONS

1. Little confidence should be placed in the analysis of test fluids as a means of evaluating the extent of metal corrosion during tests.

2. Solids obtained from test fluids during corrosion tests should be weighed and analyzed qualitatively by emission spectroscopy and when the amount and analysis warrant, they should be dissolved and determined quantitatively.

3. Alternative means of determining the extent of corrosion during testing should be considered, such as inserting test samples into the system which would be sacrificed periodically by removing for examination.

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APPENDIX I

METHOD AA-9A

THE AA DETERMINATION OF ALUMINUM IN AQUEOUS GLYCOL SOLUTIONS

March 4, 1977

SUMMARY

As little as 0.3 mg/l Al can be determined in aqueous glycol samples by complexing with 8-hydroxyquinoline and extracting into methylisobutylketone (MIBK). The MIBK extract is then read for aluminum on the AA spectrometer using a nitrous oxide flame.

KEYWORDS

Aluminum
Glycol
Atomic absorption
Corrosion
Testing
Houston Chemical Company
Solar cells

EQUIPMENT

Atomic absorption spectrometer, Perkin-Elmer Model 306, or equivalent, with Pb hollow cathode tube and nitrous oxide burner.

Volumetric flasks, 200-ml.

SOLUTIONS AND REAGENTS

(All solutions are prepared with double deionized water.)

Hydrochloric acid, 1:1, prepare with analytical reagent HCl.

Hydrochloric acid, 1%, prepare with analytical reagent HCl.

Nitric acid, 1:1, prepare with analytical reagent grade HNO_3.

Ammonium hydroxide, reagent grade.

Ammonium acetate, reagent grade.

Glacial acetic acid, reagent grade.

Aluminum, 1xxx. mg/l standard solution: Dissolve 1.xxxx g of aluminum wire or foil in a minimum amount of 1:1 HCl, adding a small drop of mercury as a catalyst. Dilute to 1 liter with 1% HCl. Filter the solution to remove the mercury.

Aluminum, 1x.xx ug/ml working standard: Dilute 10.0 ml of the 1xxx. mg/l standard to 1.000 liter.

Ammonium hydroxide-ammonium acetate buffer: Dissolve 200 g of $NH_4C_2H_3O_2$ and 70 ml of concentrated NH_4OH in water and dilute to 1 liter.

8-Hydroxyquinoline solution: Dissolve 20 g of 8-hydroxyquinoline (8-quinolinol) in 57 ml of glacial acetic acid and water. Dilute to 1 liter.

Methylisobutylketone (MIBK): Reagent grade, saturated with water.

PROCEDURE

NOTE: Clean all glassware immediately before use with warm 1:1 nitric acid and rinse with deionized water.

1. Pipet a volume of sample containing from 20 to 40 μg Al (less than 75 ml), 50 ml of deionized water (blank) and the same volume of sample as taken above plus 4 ml of 1x.xx μg/ml working Al standard into 3 separate 150-ml graduated beakers.

2. Adjust the solution volumes from Step 1. to 100 ml using deionized water and transfer each to a 200-ml volumetric flask.

3. Add 2.0 ml of 8-hydroxyquinoline, 10 ml of ammonium hydroxide-ammonium acetate buffer and 10 ml of MIBK to each flask from Step 2. Shake vigorously for 15 seconds. NOTE: This step must be carried out without interruption for each flask separately.

4. Allow the layers to separate and add sufficient deionized water to bring the MIBK layer entirely into the flask neck.

5. Read in triplicate each sample, water blank and sample plus standard addition on the AA spectrograph, aspirating the MIBK phase from the volumetric flask neck and using the following conditions:

Aluminum hollow cathode lamp.

Nitrous Oxide Burner Operation: See the start-up and shut-down instructions for nitrous oxide on pp. 2-10 of the Perkin-Elmer 306 Instruction Manual 990-9789.

Mode: Absorbance.

Wavelength: 309.3 nm (set for maximum energy at 309 using the UV scale).

Slit: 4.

Air Pressure: 40 psig (for use during start-up and shut-down).

Nitrous Oxide Pressure: 40 psig.

Acetylene Pressure: 12 psig.

Source Current: As instructed for 306 operating conditions on the hollow cathode tube.

Gain: As required to bring the Energy Meter in the working range.

Aspiration Rate: 5-5.5 ml/min for MIBK. NOTE: Read the absorbance of each sample using the same conditions. Do not aspirate water in place of MIBK while nitrous oxide and acetylene rates are unchanged; i.e., aspirate water-saturated MIBK as rinse between samples. Read sample and sample plus standard addition preparation immediately following each other.

Flame Type: Nitrous oxide-acetylene adjusted to a rose-red inner cone, 1/2 to 3/4-inch high. The outer flame will be 12 to 14 inches high.
Filter: Off.
Signal: Integrate 2.

CALCULATION

1. Average all absorbance readings.

2. Determine the net sample absorbance, A_{sample}, by subtracting the average blank absorbance from the average sample absorbance.

3. Determine a calibration factor, F, for each sample using the relationship:

$$F,\ \mu g\ Al/Abs\ unit = \frac{4(\mu g/ml\ Al\ in\ Working\ Std)}{(Avg.\ A\ of\ Sple + 4\ ml\ Std) - (Avg.\ A\ of\ Sple)}$$

4. Determine the concentration of aluminum in the sample using the relationship:

$$Al,\ mg/l = \frac{A_{sample}{}^{(1)}F}{ml\ sample}$$

Where: A_{sample} = as defined in Step 2.
F = as defined in Step 3.
ml sample = that sample taken in Step 1. under PROCEDURE.

(1) If A_{sample} is greater than the denominator in Step 3, the determination should be repeated with a smaller sample size.

ACCURACY AND PRECISION

Recoveries of standard additions of 20 µg of Al to a sample of propylene glycol formulation and two ethylene glycol formulations were 94, 110, and 94%, respectively, using the procedure specified in this method for calibration and calculation.

DISCUSSION

The sensitivity factor, F, (see Calculations) varies significantly from sample to sample and should, therefore, be determined for each sample.

REFERENCES

Atomic Absorption Newsletter, Vol. 11, No. 2, pp. 46-47, The Determination of Al in Water, M. J. Fishman.

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METHOD AA-4D

DETERMINATION OF COPPER, IRON AND LEAD IN GLYCOL SOLUTIONS

February [illegible], 1977

SUMMARY

As little as 0.02 mg/l Cu and 0.06 mg/l Fe or Pb may be determined by adjusting the pH of aqueous glycol solutions to 3.6 and extracting the respective cations as their diethyl dithiocarbamate (DDC) complexes into a methyl isobutylketone (MIBK) phase. Following extraction the organic phase is analyzed for the respective elements by atomic absorption (AA).

KEYWORDS

Copper
Iron
Lead
Glycol
Corrosion
Atomic absorption
Analytical methods

EQUIPMENT

Atomic absorption spectrometer, Perkin-Elmer Model 306, or equivalent, with an air/acetylene burner and Cu, Fe and Pb hollow cathode tubes.

Separatory funnels, 125- and 250-ml with Teflon stopcocks.

pH meter.

Millipore filtering apparatus, 25-mm diameter filter with 0.45-μ pore diameter, or equivalent.

SOLUTIONS AND REAGENTS

Methyl isobutyl ketone, MIBK, analytical reagent saturated with deionized water.

Hydrochloric acid, HCl, 1 M: Carefully dilute 9.6 g (~8 ml) of concentrated HCl to 100 ml with deionized water.

Hydrochloric acid, HCl, concentrated analytical reagent.

Sodium hydroxide, NaOH, 1 M: Carefully dissolve 4 g of analytical reagent NaOH and dilute to 100 ml with deionized water.

Diethyl dithiocarbamate (DDC): Dissolve 10 g of diethyl dithiocarbamic acid-sodium salt in 190 ml of deionized water and filter through a 0.45-μ Millipore filter. Extract the filtrate twice using 15-ml portions of MIBK and a 50-ml separatory funnel.

Phthalate buffer: Dissolve 10.2 g of potassium biphthalate (potassium hydrogen phthalate) in 50 ml of deionized water; add 1.4 ml of 1 M HCl and dilute to 100 ml with deionized water.

Nitric acid, HNO_3, analytical reagent grade.

Nitric acid, 1:1 HNO_3: Carefully add 50 ml concentrated HNO_3 to 50 ml deionized water.

Nitric acid, 1% HNO_3: Carefully add 30 g ($\sim$ 21 ml) concentrated HNO_3 to 100.0 ml deionized water. Dilute to 3.0 liters.

Copper, standard solution, 1.00xx gpl: Dissolve 1.00xx g copper metal in a minimum volume of 1:1 HNO_3, dilute to 1.000 liter with 1% HNO_3.

Copper, working standard, 12.5x μg/ml, Cu: Dilute 1.00xx g/l Cu standard solution 10.00 ml to 100.0 ml out 25.0 ml to 200.0 ml. Prepare fresh daily.

Iron, standard solution 1.00xx gpl: Dissolve 1.00xx g of iron wire in 50 ml of 1:1 HNO_3. Dilute to 1.000 liter with deionized water.

Iron, working standard, 25.x μg/ml Fe: Dilute 1.00xx g/l Fe standard solution 10.00 ml to 100.0 ml out 25.0 ml to 100.0 ml.

Lead, standard solution, 1.00xx gpl Pb: Dissolve 1.59xx g $Pb(NO_3)_2$ in 1% HNO_3, dilute to 1.000 liter with 1% HNO_3.

Lead, working standard, 25.x μg/ml Pb: Dilute 10.00 ml of 1.00xx gpl lead standard to 100.0 ml out 25.0 ml to 100.0 ml using deionized water.

PROCEDURE

NOTE: Immediately before use, rinse all glassware with 1:1 HNO_3 and then deionized water.

1. Deliver into 3 separate 150-ml beakers from 5 to 50 ml of deionized water (blank), from 5 to 50 ml of sample, and sample (the same volume as selected above) plus 1.00 ml each of the working standards of Cu, Fe and Pb. Adjust each solution to approximately 80 ml with deionized water.

2. Acidify the solutions from Step 1. with concentrated HNO_3, add 2 ml of phthalate buffer and adjust the pH to 3.6 ± 0.1. Adjust the final volume to about 100 ml.

3. Transfer the solutions from Step 2. above to 3 separatory funnels, add 7 ml DDC solution, 15 ml of MIBK and shake vigorously for 30 seconds. Allow the phases to separate and draw off the MIBK phase into a 15-ml beaker

4. Read the absorbance of each MIBK extract three times for each element on the AA spectrophotometer using the following conditions:

A. For Copper:

Cu Hollow Cathode Lamp.
Wavelength: 324.7 mm (set for maximum energy at approximately 325 using the UV scale).
Slit: 4.
Air Pressure: 30 psig.
Acetylene Pressure: 12 psig.
Mode: Absorbance.
Source Current: See lamp for Perkin-Elmer Model 306 operating current.
Gain: As required to bring Energy Meter in working range.
Aspiration Rate: 5.0-5.5 ml/minute for MIBK.
Signal: Integrate 2.
Filter: Off.

NOTE: Read the Absorbance of each sample using the same conditions. NEVER aspirate water in place of MIBK while air and acetylene flow rates are unchanged; i.e., aspirate MIBK (water saturated) as rinse between samples. Read standards immediately after samples to which standard additions are made.

B. For Iron:

Fe Hollow Cathode Lamp.
Wavelength: 248.3 mm (set for maximum energy at approximately 248 using the UV scale).
Slit: 3.
Air Pressure: 30 psig.
Acetylene Pressure: 12 psig.
Mode: Absorbance.
Source Current: See lamp for Perkin-Elmer Model 306 operating current.
Gain: As required to bring Energy Meter in working range.
Aspiration Rate: 5.0-5.5 ml/minute for MIBK.
Signal: Integrate 2.
Filter: Off.

NOTE: See instruction under Copper conditions concerning MIBK/water aspiration.

C. For Lead:

Pb Hollow Cathode Lamp.
Wavelength: 283.3 mm (set for maximum energy at approximately 283 using the UV scale).
Slit: 4.
Air Pressure: 30 psig.
Acetylene Pressure: 12 psig.
Mode: Absorbance.
Source Current: See lamp for Perkin-Elmer Model 306 operating current.
Gain: As required to bring Energy Meter in working range.

Aspiration Rate: 5.0-5.5 ml/minute for MIBK.
Signal: Integrate 2.
Filter: Off

NOTE: See instruction under Copper conditions concerning MIBK/water aspiration.

CALCULATION

1. Average all absorbance readings.

2. Determine the net sample absorbance, A_{sample}, by subtracting the average blank absorbance from the average sample absorbance.

3. Determine a calibration factor, F, for each sample using the relationship:

$$F\ (\mu g\ \text{metal/Abs unit}) = \frac{\mu g/\text{ml Metal in Working Std}}{(\text{Avg A of Sple + Std}) - (\text{Avg A of Sple})}$$

4. Determine the concentration of metal in each sample using the relationship:

$$M\ (\text{milligrams/liter, metal}) = \frac{A_{sample}{}^{(1)}F}{\text{ml sample}}$$

Where: M = the Cu, Fe or Pb content in mg/l.
A_{sample} = as defined in Step 2. above.
F = as defined in Step 3. above
ml sample = that sample taken in Step 1 under PROCEDURE

(1) If A_{sample} is greater than 3 times the denominator in Step 3. above, the determination should be repeated with a smaller sample size.

ACCURACY AND PRECISION

Recoveries of standard additions to typical glycol samples are summarized in Table I.

TABLE I

Recovery of Standard Additions of Cu, Fe and Pb to Glycol Samples

	Recoveries, %					
	Cu, ug		Fe, ug		Pb, ug	
Sample	12.5	37.5	12.5	37.5	12.5	37.5
I-6084 - Ethylene Formulation	110	102	114	106	87	95
I-6085 - Propylene Glycol Formulation	100	97	93	103	98	103
I-6086 - Triethylene Glycol Formulation	98	96	103	106	98	105

DISCUSSION

The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. It is, therefore, important that a calibration factor, F, be established for each sample size taken; i.e., the same volume of unknown must be taken for both the sample and the sample plus standard addition and carried through the procedure.

REFERENCES

Perkin-Elmer Analytical Method EN-8, September, 1976.

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