Glycol Formulations by Atomic Absorption Spectroscopy
The Determination of Aluminum, Copper, Iron, and Lead In
DOE/NSF CR-130520
DOE/NSF Contractor Report

Prepare by

Under Contract NAS8-32229 with
Corpus Christi, Texas
One Gateway Center
Division of PPG Industries, Inc.
Houston Chemical Company

For the Department of Energy
Department of Energy
George C. Marshall Space Flight Center, Alabama 35812
National Aeronautics and Space Administration

Department of Energy
Prepared by

Glycol Formulations by Atomic Absorption Spectroscopy
The Determination of Aluminum, Copper, Iron, and Lead In
DOE/NSF CR-130520
DOE/NSF Contractor Report

Prepare by

Under Contract NAS8-32229 with
Corpus Christi, Texas
One Gateway Center
Division of PPG Industries, Inc.
Houston Chemical Company
This report describes initial screening tests and the results obtained in developing procedures to determine Al, Cu, Fe, and Pb in glycol formulations. As little as 0.02 mg of carbon and 0.4 mg of Pb can be determined in glycol formulations. As little as 0.5 mg of Al may be determined in glycol formulations.

Aluminum 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 diethylenetriamine (DTE) into methyl isobutyl ketone (MIBK). Aluminum was also extracted into MIBK using 8-hydroxyquinoline as a chelating agent. As little as 0.05 mg of carbon and 0.05 mg of Pb can be determined in glycol formulations. As little as 0.3 mg of aluminum may be determined.

This work was done under the technical management of John J. Caudle, Marshall Space Flight Center. This report was submitted to the National Aeronautics and Space Administration.

<table>
<thead>
<tr>
<th>1. Rept. Date</th>
<th>16-Apr-1977</th>
</tr>
</thead>
<tbody>
<tr>
<td>2. Eprint. Accession No.</td>
<td>DOE/MSA CR-150260</td>
</tr>
<tr>
<td>4. Title and Subtitle</td>
<td>The Determination of Aluminum, Copper, Iron, and Lead in Glycol Formulations</td>
</tr>
<tr>
<td>5. Author(s)</td>
<td>Houston Chemical Company, Div of PPG Industries, Inc.</td>
</tr>
<tr>
<td>6. Performing Organization Name and Address</td>
<td>One Gateway Center, Corpus Christi, Texas</td>
</tr>
<tr>
<td>7. Sponsoring Agency Name and Address</td>
<td>National Aeronautics and Space Administration, Washington, D.C.</td>
</tr>
<tr>
<td>8. Performing Organization Code</td>
<td>8905.02.02</td>
</tr>
<tr>
<td>9. Sponsoring Agency Code</td>
<td>150.02</td>
</tr>
<tr>
<td>10. Work Unit No.</td>
<td>1430-260</td>
</tr>
<tr>
<td>11. Rept. No.</td>
<td>2517</td>
</tr>
<tr>
<td>12. Contract No.</td>
<td>0904-2600</td>
</tr>
<tr>
<td>13. Task Order No.</td>
<td>0404-2600</td>
</tr>
<tr>
<td>14. Grant No.</td>
<td>2/04-01</td>
</tr>
<tr>
<td>15. Program Element</td>
<td>0404-2600</td>
</tr>
<tr>
<td>Section</td>
<td>Page</td>
</tr>
<tr>
<td>---------</td>
<td>------</td>
</tr>
<tr>
<td>I. Introduction</td>
<td>1</td>
</tr>
<tr>
<td>II. Abstract</td>
<td>1</td>
</tr>
<tr>
<td>III. Experimental and Results</td>
<td>2</td>
</tr>
<tr>
<td>IV. Discussion</td>
<td>5</td>
</tr>
<tr>
<td>V. Conclusions</td>
<td>6</td>
</tr>
<tr>
<td>VI. Recommendations</td>
<td>6</td>
</tr>
<tr>
<td>APPENDIX I (Method AA-9A)</td>
<td>9</td>
</tr>
<tr>
<td>APPENDIX II (Method AA-4D)</td>
<td>10</td>
</tr>
</tbody>
</table>
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 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. 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

The determination of Al, Cu, Fe, and Pb in glycol formulations has been requested to develop procedures for the determination of low levels of aluminum, copper, iron, and lead in aqueous glycol formulations. Atomic absorption spectroscopy 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). Pb is also extracted into MIBK using HCl. Prior to completion Cu, Fe, and Pb are extracted from aqueous solution into methyl isobutyl ketone using diethyldithiocarbamate (DDC) and analyzed by atomic absorption spectroscopy. The determination of Al, Cu, Fe, and Pb in glycol formulations has been requested to develop procedures for the determination of low levels of aluminum, copper, iron, and lead in aqueous glycol formulations.
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 results of these determinations are presented in Table I.

### Table I

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Cu</th>
<th>Fe</th>
<th>Pb</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6076</td>
<td>50% Zerex Before Static Test</td>
<td>&lt;1</td>
<td>2</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6077</td>
<td>50% Zerex Before Static Test</td>
<td>&lt;1</td>
<td>2</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6078</td>
<td>50% Zerex Before Static Test</td>
<td>&lt;1</td>
<td>2</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6079</td>
<td>50% Zerex Before Static Test</td>
<td>&lt;1</td>
<td>2</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
</tbody>
</table>

Following the initial screening determinations, it was decided to develop extraction procedures to remove the metal ions from the glycol matrix and to concentrate the samples prior to their analysis. Cations of Cu, Fe and Pb appeared to be most suitable for the AA determination. A procedure was developed in which the cations are complexed with DTPA, extracted into NUK, with the AAs phase then being read on the AA spectrophotometer (CC Method AN-4). This procedure was applied to a series of solutions from accelerated glycol corrosion tests.

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

### Table II

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Zn</th>
<th>Cu</th>
<th>Fe</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6070</td>
<td>Bestrust from I-6070</td>
<td>1 - 10</td>
<td>&gt;5</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
<tr>
<td>I-6071</td>
<td>Bestrust from I-6071</td>
<td>1 - 10</td>
<td>&gt;5</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
<tr>
<td>I-6072</td>
<td>Bestrust from I-6072</td>
<td>1 - 10</td>
<td>&gt;5</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
</tbody>
</table>

**Solid Phase Determination by Emission Spectrography**

### Table I

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Cu</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6070</td>
<td>50% Zerex Before Static Test</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
<tr>
<td>I-6071</td>
<td>50% Zerex Before Static Test</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
<tr>
<td>I-6072</td>
<td>50% Zerex Before Static Test</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
</tbody>
</table>

**Dissolved Phase Analysis in Initial Screening**

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

---

**III. EXPERIMENTAL AND RESULTS**
The negative value shown for Fe in the "m m fro= Analysis" column indicates that the solutions actually lost Fe concentration during the test. The "m m fro= Ott. Loss" column shows the amount of metal lost from the corrosion coupons during the tests and should equal the "m m fro= 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 solid 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 CCТА-40 that the efficiency of extraction of the metal ions into the M13K 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. Accordingly, an initial attempt was made to determine aluminum in glycol by chelating into carbon tetrachloride with 8-hydroxyquinoline and evaporating the metal onto NaCl which was subsequently fired in the emission spectrograph. This procedure yielded no significant correlation of aluminum content with emission spectrographic results. For this reason it was decided to develop a procedure involving the completion of 155 extracts for Al, as the efficiency of the procedure was then increased, to determine aluminum by emission spectroscopy. This procedure was subsequently applied to the same series of samples presented earlier in Table III.

TABLE IV

<table>
<thead>
<tr>
<th>Added (mg/l)</th>
<th>AA Response</th>
<th>Emission Spectrographic Response</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.001</td>
<td>1.63</td>
</tr>
<tr>
<td>0.5</td>
<td>0.002</td>
<td>3.4</td>
</tr>
<tr>
<td>1.0</td>
<td>0.005</td>
<td>3.2</td>
</tr>
<tr>
<td>2.0</td>
<td>0.010</td>
<td>4.2</td>
</tr>
</tbody>
</table>

While the AA response for Al shown in Table IV is weak, it is clearly preferable to the emission spectrographic response. Accordingly, a procedure involving the completion of 155 extracts for Al was developed (CCTА-9A) and applied to the same series of samples presented earlier in Table III.

The results of the first application are shown in Table V.
As pointed out for Table III, the mg from Analysis and the mg from Weight 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 detection losses or gains in the metal contents of the solutions cannot be considered artifacts of the extraction media. Recovery efficiencies of Al standard additions made to various samples to establish these differences did not appear significant. These results only reflect the different limits of detection for the various solutions, and the reason for the variations is not known.

The differences found for Table V reflect different recovery efficiencies of Al standard additions made to 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.

**TABLE V**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Weight Loss (mg)</th>
<th>Analyzed Weight Loss (mg)</th>
<th>Mg from Analysis</th>
<th>Mg from Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>0054</td>
<td>L0054 Exposed</td>
<td>0.3</td>
<td>0.3</td>
<td>0.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L0054 Unexposed</td>
<td>-0.2</td>
<td>-0.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**TABLE VI**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Weight Loss (mg)</th>
<th>Analyzed Weight Loss (mg)</th>
<th>Mg from Analysis</th>
<th>Mg from Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>0053</td>
<td>L0053 Exposed</td>
<td>0.5</td>
<td>0.5</td>
<td>0.3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L0053 Unexposed</td>
<td>-0.2</td>
<td>-0.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**TABLE VII**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Weight Loss (mg)</th>
<th>Analyzed Weight Loss (mg)</th>
<th>Mg from Analysis</th>
<th>Mg from Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>0054</td>
<td>L0054 Exposed</td>
<td>0.3</td>
<td>0.3</td>
<td>0.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L0054 Unexposed</td>
<td>-0.2</td>
<td>-0.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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/l Pb, indicating considerable solubility of lead in the pure glycol. Similar formulations stored in plastic all contained less than 0.1 mg/l Pb.

VI. CONCLUSIONS

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

2. Most corrosion products must have remained on or near the actual site of metal corrosion and a metal analysis should be placed in the system at regular intervals.

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

4. 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 analyzed quantitatively by emission spectroscopy.

5. Accelerated corrosion tests on test fluids without the test coupon who performed the corrosion tests products in test fluids might offer a means of monitoring the process.
As little as 0.3 mg/l Al can be determined in aqueous solutions by complexing with sulfhydrates and extracting into methylisobutylketone (MIBK). The zinc with sulfhydrates and extracting into methylisobutylketone (MIBK). The Al extract is then read for aluminum on the Al specio meter using a nitrous oxide flame.

**KEYWORDS**
- Aluminum
- Glycol
- Atomic absorption
- Corrosion
- Testing
- Houston Chemical Company
- Solar cells
- Ammonium acetate, reagent grade
- Hydrochloric acid, 1:1, prepare with analytical reagent HCl.
- Hydrochloric acid, 1:1, prepare with analytical reagent HCl.
- Hydrochloric acid, 1:1, prepare with analytical reagent HCl.

**SOLUTIONS AND REAGENTS**
- Volumetric flasks, 200-mL.
- Ammonium acetate, reagent grade
- Hydrochloric acid, 1:1, prepare with analytical reagent HCl.
- Hydrochloric acid, 1:1, prepare with analytical reagent HCl.
- Ammonium acetate, reagent grade

**EQUIPMENT**
- Solar cells
- Houston Chemical Company
- Test cells
- Corrosion testing
- Amalgometer
- Absorption spectrometer
- Alumnum

**SUMMARY**

As little as 0.3 mg/l Al can be determined in aqueous aqueous solutions by complexing with sulfhydrates and extracting into methylisobutylketone (MIBK). The Al extract is then read for aluminum on the Al specio meter using a nitrous oxide flame.

**APPENDIX I**

March 4, 1977

**THE ALUMINUM IN AQUEOUS GLYCOL SOLUTIONS**

**SECTION AA-9A**
I. Preparation

PROCEDURE

1. Pipet a volume of sample containing from 20 to 50 ug Al (less than 75 ug) into 3 separate 150-mL graduated beakers.

2. Add 2.0 mL of 8-Hydroxyquinoline, 10 mL of ammonium hydroxide-acetate buffer and 10 mL of MIBK to each beaker from Step 1. Shaking vigorously for 15 seconds. NOTE: This step must be carried out without interruption for each beaker. After shaking, transfer the solution volumes from Step 2, shaking vigorously for each beaker, into a 200-mL volumetric flask.

3. Address the solution volumes from Step 2 to 100 mL using deionized water to bring the solution to the 100 mL mark.

4. Transfer each to a 200-mL volumetric flask.

5. Add the solution volumes from Step 3 to 200 mL of demineralized water.

6. Refer to the demineralized water in the standard procedure. Prepare a calibration curve using 20 to 150 mL of non-demineralized water in 5 mL increments. A standard curve is prepared at two standard concentrations, 20 mL of non-demineralized water and 150 mL of demineralized water.

7. Address the solution volumes from Step 3 to 100 mL of the blank, and the remaining solutions to the appropriate beakers.

8. Address the solution volumes from Step 3 to 100 mL of the blank, and the remaining solutions to the appropriate beakers.

9. Read in triplicate each sample, water blank, and standard on the X-ray spectrophotometer, aspirating the MIBK phase from the volumetric flask, 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 1106 Instruction Manual.
   - Mode: Absorbance.
   - Wavelength: 309.3 nm (set for maximum energy at 90 vol% of the UV range).

10. NOTE: Clean all glassware immediately before use with warm 11% nitric acid and water.
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 24 inches high.

Filter: Off.

Signal: In grate 2.

REFERENCES

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

DISCUSSION

A blank of standard addition of 20 mg of Al to a sample of propylene glycol and two ethylene glycol formulations were 94, 110, and 94%, respectively.

ACCURACY AND PRECISION

Recoveries of standard additions of 20 mg of Al to a sample of propylene glycol formulation and two ethylene glycol formulations were 94, 110, and 94%, respectively.

ACCURACY AND PRECISION

The accuracy of the procedure is indicated by the recoveries of standard additions of 20 mg of Al to a sample of propylene glycol formulation and two ethylene glycol formulations. These recoveries were 94, 110, and 94%, respectively.

DISCUSSION

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

or accuracy

Atomic Absorption Newsletter, Vol. 11, No. 2, pp. 46-47, The Determination of Al

Charles L. Holifield
Research Chemist
and dilute to 100 ml with demineralized water.

Sodium hydroxide, HNO₃, concentrated analytical reagent.

Hydrochloric acid, HCl, concentrated analytical reagent.

100 ml with demineralized water.

Hydrochloric acid, HCl, concentrated HCl to

20 ml of concentrated HCl to

Method: Select any reagent, HNO₃, analytical reagent standard with demineralized water.

SOLUTIONS AND REAGENTS

Nitric acid. 

pH meter.

Separatory funnels, 15- and 250-ml with PTFE stopcocks.

Artificial aerodynamic burner and Cu, Fg and Pb hollow cathode tubes.

Atomic absorption spectrometer, Perkin-Elmer model 506, or equivalent, with an

Acme Analytical method. 

Sample, refer to the user manual.

Iron

Copper

TMS

Equipment

Analytical methods.

CORRESPONDENCES

d Lead

Li

Co

EXPERIMENTAL

The elements by atomic absorption (AA).

The AA phase. Following extraction the atomic phase is manipulated for the recapture

as their deuterated ethyl ether derivative (DER) complexes into a methyl ethyl ether/ether.

the pH of aqueous ethyl ether solutions to 7.0 and extraction the respective carbonate

as little as 0.02 mg/l Cu and 0.06 mg/l Fe or Pb may be determined by absorption.

SUMMARY

DETERMINATION OF COPPER, LEAD AND LITHIUM IN AQUEOUS SOLUTIONS

METHOD AA-40

APPENDIX II
II

Diethyl dithiocarboxylate sodium salt in 190 ml of a 1:1 nitric acid and make up exactly to 200 ml with deionized water.

PROCEDURE

Immediately before use, rinse all glassware with 1:1 HNO₃ 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. Add each solution (blank), from 5 to 50 ml of sample, and sample (the same volume as selected above) to a 150-ml beaker.

2. Add 10 ml of DDC solution, 15 ml of 1:1 nitric acid, and 2 ml of deionized water.

3. Transfer the solutions from Step 1 to 3 separate 150-ml beakers. Add 2 ml of deionized water.

1. Add 10 ml of DDC solution, 15 ml of 1:1 nitric acid, and 2 ml of deionized water.

2. Add 100 ml of DDC solution, 15 ml of 1:1 nitric acid, and 2 ml of deionized water.

3. 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. Add each solution (blank), from 5 to 50 ml of sample, and sample (the same volume as selected above) to a 150-ml beaker.

2. Add 10 ml of DDC solution, 15 ml of 1:1 nitric acid, and 2 ml of deionized water.

3. Transfer the solutions from Step 1 to 3 separate 150-ml beakers. Add 2 ml of deionized water.
C Copper: As required to bring energy meter to working range.

Source current: See Lamp for Perkin-Elmer Model 406 Operating current.

Mode: Absorbance.

Acetone pressure: 12 psi.

Art pressure: 30 psi.

Slit: 4. 

the UV scale.

Wave length: 280.7 nm (see for maximum energy at approximately 225 nm)

Acetone: 10 psis.

For Hollow Cathode Lamp.

B Lead: 

immediately after samples to which standards additions are made.

LIA, aspirate N(2) (water saturated) at twice between samples, read standards.

aspirate water in place of N(2) while air and acetylene flow rates are unchanged.

C For Lead:

N(2): Read the absorbance of each sample using the same conditions.

NOTE: See instructions under Copper conditions concerning N(2)/water application.

Signal: Increase 5.

Aspirator rate: 5.0-5.5 mL/minute for N(2).

equal as required to bring energy meter to working range.

Source current: See Lamp for Perkin-Elmer Model 406 Operating current.

Mode: Absorban ce.

Acetone pressure: 12 psi.

Art pressure: 30 psi.

Slit: 4. 

the UV scale.

Wave length: 320.0 nm (see for maximum energy at approximately 225 nm)

Acetone: 10 psis.

For Hollow Cathode Lamp.
Aspiration Rate: 5.0-5.5 ml/min

FILTER: Off

NOTE: See instruction under copper conditions concerning nitric acid application.

Table 1

Recoveries of standard additions of Cu, Fe and Pb to glycol samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cu, mg/l</th>
<th>Fe, mg/l</th>
<th>Pb, mg/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6086 - 1,000 ppm Cu</td>
<td>1.5</td>
<td>0.1</td>
<td>0.5</td>
</tr>
<tr>
<td>I-6087 - 500 ppm Cu</td>
<td>0.7</td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>I-6088 - 100 ppm Cu</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
</tr>
</tbody>
</table>

DISCUSSION

The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. Therefore,

Correlation should be repeated with a smaller sample size.

Where: N = the Cu, Fe or Pb content in mg/l.

The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. Therefore,

The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. Therefore,

The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. Therefore,

The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. Therefore,
It is 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.

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

References