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

DOE/NA-84 CR-150520
DOE/NA-84 CR-150520

Prepared by

Houston Chemical Company

Under Contract NAS-382295 with

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

George C. Marshall Space Flight Center, Alabama 35812
National Aeronautics and Space Administration

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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 carbon and 0.05 mg Pb or Fe may be determined in glycol formulations. As little as 0.05 mg Ni was selected for Cu, Fe, and Pb after comparison with emission spectroscopy. An atomic absorption spectrophotometer was selected for Al also. Before completion, emission, atomic absorption, and atomic emission spectroscopy were compared to determine Al, Cu, Fe, and Pb in glycol formulations. Atomic absorption spectrophotometry was found to be the most suitable for Al, Cu, Fe, and Pb, and atomic absorption after completion of the research. 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 carbon and 0.05 mg Pb or Fe may be determined in glycol formulations. As little as 0.05 mg Ni was selected for Cu, Fe, and Pb after comparison with emission spectroscopy. An atomic absorption spectrophotometer was selected for Al also. Before completion, emission, atomic absorption, and atomic emission spectroscopy were compared to determine Al, Cu, Fe, and Pb in glycol formulations. Atomic absorption spectrophotometry was found to be the most suitable for Al, Cu, Fe, and Pb, and atomic absorption after completion of the research.
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<th>Page</th>
<th>Section</th>
</tr>
</thead>
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The Determination of \( \text{Cu}, \text{Fe}, \text{Al} \) in Glycol Formulations

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 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 were extracted with diethyldithiocarbamate (DDC) into methyIsobutyl ketone and Pb are extracted with dithyldithiocarbamate (DDC) into methyIsobutyl ketone. 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.
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 spectrophotograph to obtain a significant quantitative analysis of the elements present. The results of the determination are presented in 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-6073</td>
<td>Residue from I-6070</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6074</td>
<td>Residue from I-6069</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6075</td>
<td>Residue from I-6068</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6076</td>
<td>Residue from I-6071</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
</tbody>
</table>

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

<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-6070</td>
<td>50% 420 AP from Panel Test</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>I-6071</td>
<td>50% Zerex from Panel Test</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>I-6072</td>
<td>50% 420 AP before Static Test</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>I-6073</td>
<td>50% Zerex before Static Test</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

The results of the AA determinations are presented in Table III.
<table>
<thead>
<tr>
<th>Sample Description</th>
<th>Pe</th>
<th>0.07</th>
<th>0.09</th>
<th>0.10</th>
<th>0.11</th>
<th>0.12</th>
<th>0.13</th>
<th>0.14</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid Val Test</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wet Tn from</td>
<td>mg/l</td>
<td>1.00</td>
<td>0.71</td>
<td>0.50</td>
<td>0.25</td>
<td>0.13</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>Dry Tn from</td>
<td>mg/l</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>4.4% 14009 Exposed</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4.4% Ulexposed</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.6978</td>
<td>I-6098</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.6978</td>
<td>I-6098</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.6978</td>
<td>I-6098</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Results of Cyclic Method AA-4 and Sample Weight Losses for Accelerated Corrosion Testing
The negative values shown for Fe in the "Analysis" column indicate that the solutions actually lost Fe concentration during the test. The "Loss Fro Analysis" column shows the amount of metal lost during the tests. This column should equal the "Loss 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 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-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 tetrahydroxylamine and evaporating the MIBK onto NaCl which was subsequently fired in the emission spectrograph. This procedure yielded no significant correlation of aluminum content with emission spectrographic results. It was then realized that the chelated metal ion distribution is dependent on the aqueous phase concentration of the aqueous phase. As the glycol content of the aqueous phase decreased, the metal ions transferred into the MIBK phase in a very dependent manner.

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

<table>
<thead>
<tr>
<th>TABLE IV</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Comparison of Emission Spectrographic and AA Results for Al</strong></td>
</tr>
<tr>
<td><strong>Conc. of Al (mg/l)</strong></td>
</tr>
<tr>
<td><strong>Added to MIBK (mg/l)</strong></td>
</tr>
<tr>
<td>0</td>
</tr>
<tr>
<td>0.5</td>
</tr>
<tr>
<td>1.0</td>
</tr>
<tr>
<td>2.0</td>
</tr>
</tbody>
</table>

The results of this application are shown in Table IV. While the AA response for Al shown in Table IV is weak, it is clearly preferable to the emission spectrographic responses presented in Table III. Accordingly, a procedure involving the completion of 155 extracts for Al was developed (CCTC Method AA-40) and applied to the same series of samples presented earlier in Table III. The results are shown in Table V.
The results of the CCTC test on samples from various glycol formulations are shown in Table V. As pointed out for Table III, the mg from Analysis and 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 do not indicate losses of metal in the solution or in the test apparatus.

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

**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 test fluids. Further, the fact that very few solids were present in the test fluids does not encourage speculation that a means of collecting undissolved solids could be developed although they were shown to be very small in comparison to the weight losses. Various other factors, such as differences in the starting concentrations of the solutions, may affect these results. General trends were observed to the same effect the loss of metal from solution during the test. Although it is not evident from the results in Table V, where the weight of metal picked up by the solutions were the same, the net mass change in the solutions would be equal to the mass of metal picked up from the coupons. As pointed out for Table III, the mg from Analysis and mg from Weight Loss results in Table V show the same effect for different samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>mg from Analysis</th>
<th>mg from Weight Loss</th>
<th>Sample</th>
<th>Description</th>
<th>mg from Analysis</th>
<th>mg from Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6098</td>
<td>Al from Solution</td>
<td>0.2</td>
<td>-0.1</td>
<td>I-6094</td>
<td>Al from Solution</td>
<td>0.2</td>
<td>-0.1</td>
</tr>
<tr>
<td>I-6097</td>
<td></td>
<td></td>
<td></td>
<td>I-6095</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>I-6096</td>
<td></td>
<td></td>
<td></td>
<td>I-6096</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>I-6098</td>
<td></td>
<td></td>
<td></td>
<td>I-6098</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Tables**

Table V shows the results of the CCTC test on samples from various glycol formulations.
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 if the coupon.

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 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. There is little correlation between corrosion of metal test coupons and 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/l and Fe and Pb at levels down to 0.06 mg/l in glycol formulations can be determined by extraction into MIB K with dithiocarbamate and AA completion (CCTC Method A-7A).
4. Cu at levels down to 0.02 mg/l and Pb at levels down to 0.06 mg/l in glycol formulations can be determined by extraction into MIB K with dithiocarbamate and AA completion.

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 washed and analyzed quantitatively by emission spectroscopy and when the amount and analysis warrant. Solid corrosion products should be weighed.
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 sacrificial periodically by removal for examination.

Charles L. Kauffman
Research Chemist
As little as 0.3 mg/l of Al can be determined in aqueous solutions by complexation with ethylenediaminetetra-acetic acid (EDTA). The Al fraction is determined by atomic absorption spectrometry. The Al standard solution is prepared by dissolving 1.000 g of aluminum wire or foil in a minimum amount of 1:1 HCl, adding a small drop of mercury as a catalyst, and heating until a solution of aluminum nitrate [1.5% Al] is obtained.

**Keywords:**
- Atomic absorption
- Corrosion
- Testing
- Houston Chemical Company
- Solar cells
- Hydrochloric acid, 1:1
- Hydrochloric acid, 1:1
- Nitric acid, 1:1
- Ammonium hydroxide, reagent grade
- Glacial acetic acid, reagent grade
- Ammonium acetate, reagent grade
- Volumetric flasks, 200-ml.

**Equipment**
- Solar cells
- Houston Chemical Company
- Test tubes
- Corrosion test solutions
- Volumetric flasks
- Spectrophotometer
- Corrosion test solutions
- Alumina

**Reagents**
- Volumetric flasks, 200-ml.
- Hydrochloric acid, 1:1
- Hydrochloric acid, 1:1
- Nitric acid, 1:1
- Ammonium hydroxide, reagent grade
- Ammonium acetate, reagent grade
- Glacial acetic acid, reagent grade

**Standard solution of aluminum**
Dissolve 1.000 g of aluminum wire or foil in a minimum amount of 1:1 HCl, adding a small drop of mercury as a catalyst, and heat until a solution of aluminum nitrate [1.5% Al] is obtained. Filter the solution to remove the mercury.

**Solutions and Reagents**

**Summary**

March 4, 1977

**The AA Determination of Aluminum in Aqueous Clay Slurries**

Section AV-9A

**Appendix I**
PROCEDURE

1. Pipet a volume of sample containing from 20 to 100 µg Al (less than 75 ml) and 50 ml of deionized water (blank) into a 150-ml graduated beaker.

2. Adjust the solution volumes from step 1 to 100 ml using deionized water.

3. Add 2.0 ml of 8-N-hydroxyquinolinate, 10 ml of ammonium hydroxide-buffer,

and transfer each to a 200-ml volumetric flask.

4. Add water to complete the 200-ml volumetric flasks.

5. Read in triplicate each sample, water blank and sample plus standard on the Al spectrograph, aspirating the AlBK phase from the volumetric flasks.

6. Plot a volume of sample containing from 20 to 75 µl less than 75 ml.

7. Clean all glassware immediately before use with a mixture of concentrated nitric acid and distilled water.

8. Heat to 1000 ml.

9. Dilute to 1 liter.

PROCEDURE

1. Pipet a volume of sample containing from 20 to 100 ml of the Alx of the Alx, Alx/standard.

2. Add 2.0 ml of 8-N-hydroxyquinolinate, 10 ml of ammonium hydroxide-buffer, and 10 ml of dilute ammonium acetate buffer into a 200-ml volumetric flask and adjust the solution volumes from step 1 to 100 ml using deionized water.

3. Add 2.0 ml of 8-N-hydroxyquinolinate to each flask from step 2, shaking vigorously for 10 seconds. NOTE: This step must be carried out without interruption for each sample.

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

5. Read in triplicate each sample, water blank and sample plus standard on the Al spectrograph, aspirating the AlBK phase from the volumetric flasks.

6. Plot a volume of sample containing from 20 to 75 µl less than 75 ml.

7. Clean all glassware immediately before use with a mixture of concentrated nitric acid and distilled water.

8. Heat to 1000 ml.

9. Dilute to 1 liter.

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In water, M. J. Pitsman.

**Calculation**

1. Average all absorbance readings.

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

3. Determine a calibration factor, \( F \), using the relationship:

\[
F = \frac{\text{Abs} \text{ of Std}}{\text{Abs} \text{ of Sample}}
\]

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

\[
\text{Al} \text{, mg/l} = \frac{A_{net} \times F}{\text{ml sample}}
\]

Where: \( A_{net} \) = net absorbance of sample, \( F \) = calibration factor, \( \text{ml sample} \) = sample size as defined in Step 1, under Procedure.

If \( A_{net} \) 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 mg of Al to a sample of propylene glycol formulation were 94, 110, and 94%, respectively, using the procedure specified in this method for calibration and calculation.

**Discussion**

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

**References**

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and dilute to 100 ml with deionized water.

Sodium hydroxide, KOH, 1 M. Carefully dissolve 4 g of analytical reagent-grade sodium hydroxide in 100 ml of deionized water.

Hydrochloric acid, HCl, concentrated analytical reagent.

100 ml with deionized water.

Hydrochloric acid, HCl, 1 M. Carefully dilute 9.6 g of concentrated HCl to 100 ml with deionized water.

Method 1: Sodium Ketene, NBS, analytical reagent grade, 1 M. Add a few drops of concentrated HCl to 100 ml of deionized water.

SOLUTIONS AND REAGENTS

With thorium apparatus, 25-mm diameter cells, with 0.45-μm pore diameter, or acrylic observation spectrometer. pH meter.

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

Mix, acetone buffer, and Cu, Fe, and Pb hollow cathode tubes.

Acryl observation spectrometer, Perkin-Elmer Model 900, or equivalent, with an automatic aspiration system.

EQUIPMENT

Analytical reagents

Infra-red analysis

Corrosion

Atomic absorption

Lead

Iron

Copper

KEYWORDS

The elements by atomic absorption (AA).

This phase, following extraction the atomic phase is analyzed for the respective element.

As their chloroethyl derivatives (CE) complexes into a methyl tautomeric.

The pH of aqueous ethyl solutions to pH 2.5 and extraction the respective elements as little as 0.02 mg/l Cu and 0.06 mg/l Fe or Pb may be determined by absorption

RECOMMENDATION OF COPPER, IRON AND LEAD IN AQUEOUS SOLUTIONS

METHOD AV-75

APENDIX II

February 1971
Diethyl dithiocarbamate sodium salt in 190 ml lore filter. 

1. Deliver into 3 separate 150-ml beakers from 5 to 50 ml of deionized water and then detonized water.

PROCEDURE

NOTE: Immediately before use, rinse all glassware with 1:1 HNO₃ and then detonized water.

Lead, working standard, 2.0 µg/ml Pb solution. Prepare fresh daily.

Iron, standard solution 1.000 g/l Fe. Dilute 1.000 ml to 100.0 ml and dilute to 3.0 liters.

Lead, working standard, 1.000 g/l Pb. Dilute 1.000 ml to 100.0 ml and dilute to 3.0 liters.

Iron, working standard, 0.05 µg/ml Fe. Dilute 1.000 ml to 100.0 ml to 5.0 ml to 100.0 ml.

Copper, working standard, 1.000 g/l Cu. Dilute 2.5 ml to 50.0 ml. Prepare fresh daily.

Copper, standard solution, 1.000 g/l Cu. Dilute 5.0 ml to 100.0 ml.

Phthalate buffer: Dissolve 1.25 g of sodium hydrogen phthalate (pH 3.6 ± 0.1) and dilute to 100.0 ml with 1:1 HNO₃ and then detonized water.

Nitric acid, 1:1 HNO₃: Analytical reagent grade.

Nitric acid, 1% HNO₃: Carefully add 20 g of 50.0 ml of detonized water. Dilute to 100.0 ml and dilute to 3.0 liters.

PROCEDURE

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) (blank).

2. Acidify the solutions from Step 1. with concentrated HNO₃, add 2 ml of phthalate buffer and adjust the pH to 3.6 ± 0.1. Adjust the final volume to about 100 ml with deionized water.

3. Transfer the solutions from Step 2. above to 3 separatory funnels, add 7 ml of DDC solution, 15 ml of 1:1 HNO₃, and shake vigorously for 20 seconds. Allow the phases to separate and draw off the HNO₃ 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: 325 nm (set for maximum energy at approximately 325 using the UV scale),
- Air Pressure: 12 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: Integrator 2,
- Filter: OLR,
- 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 close between samples. Read standards immediately after samples to which standard additions are made.

**B. For Iron:**
- Fe Hollow Cathode Lamp,
- Wavelength: 248.3 nm (set for maximum energy at approximately 248 using the UV scale),
- Air Pressure: 12 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: Integrator 2,
- Filter: Off,
- NOTE: See instructions under Copper conditions concerning MIBK/water aspiration.

**C. For Lead:**
- Pb Hollow Cathode Lamp,
- Wavelength: 283.3 nm (set for maximum energy at approximately 283 using the UV scale),
- 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.
The efficiency of extraction of copper, iron and lead is dramatically reduced by the glycol content in the aqueous phase during the extraction. Therefore:

**Aspiration Rate:** 5.0-5.5 ml/min

**Shunt:** On

**Filter:** Off

**NOTE:** See instruction under copper concentration concerning HNO3/water aspiration.

**Calculation**

1. Average all absorbance readings.
2. Determine the net sample absorbance, absorbance by subtracting the average blank absorbance from the average sample absorbance.
3. Determine a calibration factor, F, for each sample using the relationship:

   \[ F = \frac{C_{\text{std}}}{A_{\text{std}}} \]

   where:
   - F is the calibration factor (mg/ml)
   - C is the concentration of metal (mg/ml)
   - A is the absorbance

   \[ F (\text{mg/ml}) = \frac{\text{milliters of metal}}{\text{milliters of reagent sample}} \]

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

   \[ C (\text{mg/ml}) = \frac{A \times V_{\text{sample}}}{V_{\text{total}} \times F} \]

   where:
   - C is the concentration of metal (mg/ml)
   - A is the absorbance
   - V is the volume of sample
   - V is the total volume
   - F is the calibration factor

**Table 1**

<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>Sample 1</td>
<td>3.75</td>
<td>10.6</td>
<td>100.3</td>
</tr>
<tr>
<td>Sample 2</td>
<td>3.75</td>
<td>10.6</td>
<td>100.3</td>
</tr>
<tr>
<td>Sample 3</td>
<td>3.75</td>
<td>10.6</td>
<td>100.3</td>
</tr>
<tr>
<td>Sample 4</td>
<td>3.75</td>
<td>10.6</td>
<td>100.3</td>
</tr>
<tr>
<td>Sample 5</td>
<td>3.75</td>
<td>10.6</td>
<td>100.3</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.
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. Plus, standard addition and carried through the procedure, $\pm$ the same volume of unknown must be taken for both the sample and the sample plus standard addition. It is important that a calibration factor, $F$, be established for each sample size taken.

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

References

Charles L. Aiello
Research Chemist
March 7, 1976