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much information as possible.
Glycol Formulations by Atomic Absorption Spectroscopy
The Determination of Aluminum, Copper, Iron, and Lead in

DOE/NASA CR-150520

DOE/NASA Contractor Report
This report describes initial screening tests and the results observed in developing procedures to determine Al, Cu, Fe, and Pb in glycol formulations. Atomic absorption spectrometry was selected to determine Cu, Fe, and Pb in glycol formulations. Before completion, carbon, iron, and lead are extracted with diethyldithiocarbamate (DDC) into methyl isobutyl ketone (MIBK). Aluminum was also extracted with diethyldithiocarbamate using 3-hydroxypyrididine as a chelating agent. As little as 0.02 mg of carbon and 0.06 mg of lead or iron may be determined in glycol formulations. As little as 0.3 mg of aluminum may be determined.
TABLE OF CONTENTS

I. Introduction .......................... 1
II. Abstract .............................. 1
III. Experimental and Results .......... 2
IV. Discussion ............................ 5
V. Conclusions ........................... 6
VI. Recommendations .................... 6
APPENDIX I (Method AA-9A) .......... 7
APPENDIX II (Method AA-4D) .......... 10
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 spectroscopy was selected for Al also. Prior to completion Cu, Fe, and Pb are extracted with diethyldithiocarbamate (DTC) 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.
An initial surrey 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 semiquantitative analysis of the elements present. The results of the determinations are presented in Table I.

TABLE I
Dissolved Phase Analysis in Initial Screening

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Cu (ppm)</th>
<th>Fe (ppm)</th>
<th>Pb (ppm)</th>
<th>Al (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6076</td>
<td>Best 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-6077</td>
<td>Best residue from I-6071</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6078</td>
<td>Best residue from I-6072</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;5</td>
<td>&lt;5</td>
</tr>
<tr>
<td>I-6079</td>
<td>Best residue from I-6073</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 spectrographic determinations are presented in Table II.

TABLE II
Solid Phase Determination in Initial Screening by Emission Spectrograph

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>0-0.1</th>
<th>0.1-1</th>
<th>1-10</th>
<th>10+</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6073</td>
<td>Residue from I-6070</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
</tr>
<tr>
<td>I-6074</td>
<td>Residue from I-6071</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
</tr>
<tr>
<td>I-6075</td>
<td>Residue from I-6072</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
</tr>
<tr>
<td>I-6076</td>
<td>Residue from I-6073</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
<td>50%</td>
</tr>
</tbody>
</table>

Following the initial screening determinations it was decided to develop extraction procedures to remove the metal ions from their glycol matrix and to concentrate the solutions to remove the metal ions from their glycol matrix and to concentrate them prior to their analysis. Cu, Fe and Pb appeared to be the most suitable for AA determination so a procedure was developed in which the cations are complexed with DTPA and then extracted into NUK, with the NUK phase then being read on the AA spectrophotometer (CC: Method AV-4D), and Fe and Pb appearing to best lend themselves to an extraction procedure so a procedure was developed in which the cations are complexed with DTPA and then extracted into NUK, with the NUK phase then being read on the AA spectrophotometer (CC: Method AV-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>
<thead>
<tr>
<th>Sample Description</th>
<th>1,0504 Exposed</th>
<th>1,0504 Unexposed</th>
<th>1,0608 Exposed</th>
<th>1,0608 Unexposed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenyle Glycerol Formulation</td>
<td>0.037</td>
<td>0.05</td>
<td>0.02</td>
<td>0.01</td>
</tr>
<tr>
<td>Propylene Glycerol Formulation</td>
<td>0.037</td>
<td>0.05</td>
<td>0.02</td>
<td>0.01</td>
</tr>
</tbody>
</table>

Table III

Results of COTC Method AA-4B end Sample Weight Loss for Accelerated Corrosion Testing.
The negative value shown for Fe in the "mass fro= Analysis" column indicate that the solutions actually lost Fe concentration during the test. The "mass fro= Ott. Loss" column shows the amount of metal lost from the corrosion coupons during the tests and should equal the "mass 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 TT-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 calibrations for each sample dilution must be made to ascertain appropriate concentrations of the aqueous phase. After correction of the aqueous phase, the glycol concentration of the aqueous phase was determined by spectrophotometry using 8-hydroxyquinoline and evaporating the M13K 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 M13K. This extract was then read directly in the 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>Concentration (mg/mL)</th>
<th>AA Response</th>
<th>Emission Spectroscopic</th>
<th>Emission Spectroscopic &amp; AA Results for Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.001</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>0.002</td>
<td>0.004</td>
<td>0.004</td>
<td>0.004</td>
</tr>
<tr>
<td>0.005</td>
<td>0.008</td>
<td>0.008</td>
<td>0.008</td>
</tr>
<tr>
<td>0.010</td>
<td>0.012</td>
<td>0.012</td>
<td>0.012</td>
</tr>
</tbody>
</table>

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 completion of 55 extracts for Al was developed (CCT-9A) and applied to the same series of samples presented earlier in Table III. The results of this application are shown in Table V.
TABLE V

<table>
<thead>
<tr>
<th>Sample</th>
<th>Description</th>
<th>Analysis of Glycol Formulations</th>
<th>mg from Sample</th>
<th>mg from Analysis</th>
<th>Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-6089</td>
<td>LG048 Unexposed</td>
<td>I-6089 Exposed</td>
<td>0.1</td>
<td>0.08</td>
<td>8.2</td>
</tr>
<tr>
<td>I-6082</td>
<td>LG048 Exposed</td>
<td>I-6082 Exposed</td>
<td>0.1</td>
<td>0.08</td>
<td>8.2</td>
</tr>
<tr>
<td>I-6090</td>
<td>LG049 Unexposed</td>
<td>I-6090 Exposed</td>
<td>0.1</td>
<td>0.08</td>
<td>9.2</td>
</tr>
<tr>
<td>I-6083</td>
<td>LG049 Exposed</td>
<td>I-6083 Exposed</td>
<td>0.2</td>
<td>0.08</td>
<td>8.1</td>
</tr>
<tr>
<td>I-6091</td>
<td>LG052 Unexposed</td>
<td>I-6091 Exposed</td>
<td>0.5</td>
<td>0.2</td>
<td>14.5</td>
</tr>
<tr>
<td>I-6078</td>
<td>LG052 Exposed</td>
<td>I-6078 Exposed</td>
<td>0.2</td>
<td>0.2</td>
<td>14.5</td>
</tr>
<tr>
<td>I-6085</td>
<td>LG053 Unexposed</td>
<td>I-6085 Exposed</td>
<td>0.5</td>
<td>0.2</td>
<td>21.0</td>
</tr>
<tr>
<td>I-6079</td>
<td>LG053 Exposed</td>
<td>I-6079 Exposed</td>
<td>0.2</td>
<td>0.2</td>
<td>21.0</td>
</tr>
<tr>
<td>I-6086</td>
<td>LG054 Unexposed</td>
<td>I-6086 Exposed</td>
<td>0.3</td>
<td>0.2</td>
<td>9.4</td>
</tr>
<tr>
<td>I-6080</td>
<td>LG054 Exposed</td>
<td>I-6080 Exposed</td>
<td>0.1</td>
<td>0.2</td>
<td>9.4</td>
</tr>
</tbody>
</table>

As pointed out for Table III, the mg from Analysis and the mg from Sample results in Table V would be the same if the physical loss from the coupon and the weight of metal picked up by the solutions were the same. The negative values again indicate a loss of metal from the solution during the test, although in general these results only reflect the different limits of detection for the various solutions, and do not reflect the difference in the recovery efficiencies of the respective ars. The observed loss results in Table V would be the same if the physical loss from the coupon and the weight of metal picked up by the solution were the same. The difference in Table V reflects different recovery efficiencies of the respective ars. The reason for this is not known.

IV. DISCUSSION

Tables III and V show there is very little correlation between the weight loss of the corrosion coupons and the dissolved metals found in the corrosion 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 materials could be developed. The negative results for the corrosion coupons are not known.

The limit of detection variations for Al shown in Table V reflect different recovery efficiencies of the respective ars. These differences do not appear to be connected to the glycol content in the extraction media and the reason for the observed variations is not known.
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 the extent of metal corrosion during tests and metal analysis of the test fluid.

2. Most corrosion products must have remained on or near the actual site of metal and a metal analysis of the test fluid.

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 MIK with 8-hydroxyquinoline followed by AA completion (CCTC Method AA-9A).

4. Al at levels as low as 0.02 mg/l in glycol formulations can be determined by extraction into MIK with 8-hydroxyquinoline followed by AA completion (CCTC Method AA-9A).

VII. 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, weighed and analyzed quantitatively by emission spectroscopy and when the amount and analyses warrant, they should be weighed out and 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 provide a means of determining the extent of corrosion during testing.

VI. CONCLUSIONS

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

2. Most corrosion products must have remained on or near the actual site of metal corrosion and a metal analysis of the test fluid.

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 MIK with 8-hydroxyquinoline followed by AA completion (CCTC Method AA-9A).

4. Al at levels as low as 0.3 mg/l in glycol formulations can be determined by extraction into MIK with 8-hydroxyquinoline followed by AA completion (CCTC Method AA-9A).
Dilute to 1 liter with 1% HCL. Filter the solution to remove the mercury.

The ammonium hydroxide, reagent grade.

Solutions and Reagents

Volumetric flasks, 200-mL.

All solutions are prepared with double distilled water.

Nitric acid, 1:1, prepare with analytical reagent HNO3.

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

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

All solutions are prepared with double distilled water.

EQUIPMENT

Solar cell
Houston Chemical Company
Testume
Corrosion
Atomic absorption
Optical
Alumnum

KEYWORDS

oxide film

METHOD

After extraction is then read for absorption on the atomic absorption using a nitrous oxide-acetylene flame and extraction into methylisobutylketone (MIBK). The

The as little as 0.5 mL/L can be determined in aqueous Al samples by complex-

SUMMARY

March 6, 1977

THE AA DETERMINATION OF ALUMINUM IN AQUEOUS GLYCOL SOLUTIONS

APPENDIX I
PROCEDURE

NOTE: Clean all glassware immediately before use with warm 3% nitric acid and

PROCEDURE

Note that the preparation immediately following each other.

Note that the preparation immediately following each other.

Note that the preparation immediately following each other.

Note that the preparation immediately following each other.

Note that the preparation immediately following each other.

Note that the 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.

In water, H. J. Prisman.


REFERENCES

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

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DISCUSSION

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

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ACCURACY AND PRECISION

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

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

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CALCULATION

1. Average all absorbance readings.

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

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

\[
F = \frac{A_{\text{sample}}}{V_{\text{sample}}} \cdot \frac{1}{\text{mg/mL}}
\]

Where:

- \( V_{\text{sample}} \) = volume of sample taken in Step 1, under Procedure.
- \( A_{\text{sample}} \) = absorbance of sample at wavelength of interest, determined in Step 2.

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

\[
\text{Al, mg/L} = \frac{A_{\text{sample}}}{V_{\text{sample}}} \cdot \frac{1}{\text{mg/mL}}
\]

I. Used with a smaller sample size.

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

I 1. Mix \( m/g \) of sample taken in Step 1, under Procedure.

Where:

- \( m/g \) = mass of sample.
- \( A_{\text{sample}} \) = absorbance of sample at wavelength of interest, determined in Step 2.

I 2. Determine the concentration of aluminum in the sample using the relationship:

\[
\text{Al, mg/L} = \frac{A_{\text{sample}}}{V_{\text{sample}}} \cdot \frac{1}{\text{mg/mL}}
\]

I 3. Determine a calibration factor, \( F \), for each sample using the relationship:

\[
F = \frac{A_{\text{sample}}}{V_{\text{sample}}} \cdot \frac{1}{\text{mg/mL}}
\]

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

\[
\text{Al, mg/L} = \frac{A_{\text{sample}}}{V_{\text{sample}}} \cdot \frac{1}{\text{mg/mL}}
\]

I 5. Average all absorbance readings.

I 6. Invert, if required.
Appendix II: Determination of Copper, Iron and Lead in Citric Solutions

February 24, 1977

METHOD AA-40

Solutions and Reagents

Solutions

Methyl isobutyl ketone, 99% or equivalent.

pH meter.

Separatory funnels, 150- and 250-ml with Teflon stopcocks.

Acrylic absorption spectrometer, Perkin-Elmer Model 300, or equivalent, with an argon/acetylene burner and Cu, Fe and Pb hollow cathode tubes.

Equipment

Analytical methods

Corrosion

Copper

Iron

Silver

The elements by atomic absorption (AA).

The elements by potassium dichromate (K2Cr2O7) complexes into a metallospecies and extraction of the respective complexes into 0.02 M HCl and 0.05 M Fe or Pb may be determined by adsorption on the elements by atomic absorption.

Determination of Copper, Iron and Lead in Citric Solutions

METHOD AA-40
Diethyl dithiocarbamate sodium salt in 190 ml lore filter. 

Extract 1.0 ml of filtrate from Step 1, with concentrated HNO₃, and adjust the final volume to about 100 ml.

2. Add 2 ml of concentrated HNO₃ to the solutions from Step 1, with concentrated HNO₃.

To approximately 50 ml of the working standards of Cu, Fe and Pb (plus, if necessary, the same volume of sample) (blank) from 5 to 50 ml of sample, and sample (the same volume as selected above) deliver into 3 separate 150-ml beakers. From 5 to 50 ml of detemination water.

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

### PROCEDURE

Lead, working standard, 25 mg/l Pb: Dissolve 1.5902 g Pb(NO₃)₂ in 1% HNO₃, dilute to 1.000 liter with 1% HNO₃, and adjust the final volume to 100 ml.

Lead, standard solution, 1.000 mg/l Pb: Dissolve 1.5902 g Pb(NO₃)₂ in 1% HNO₃, dilute to 1.000 liter with 1% HNO₃, and adjust the final volume to 100 ml.

Iron, standard solution 1.000 mg/l Fe: Dissolve 1.000 g Fe standard solution, 1.000 mg/l Fe, in 50 ml of 1% HNO₃, dilute to 100 ml with deionized water.

Iron, working standard, 12.5 mg/l Fe: Dilute 10.00 ml of 1.000 mg/l iron standard solution to 100 ml.

Copper, working standard, 12.5 mg/l Cu: Dilute 10.00 ml of 1.000 mg/l copper standard solution to 100 ml.

Copper, standard solution, 1.000 mg/l Cu: Dissolve 1.000 g Cu standard solution, 1.000 mg/l Cu, in a Mini-mula volume of 1:1 HNO₃, dilute to 1.000 liter with 1% HNO₃, and adjust the final volume to 100 ml.

Copper, working standard, 0.50 mg/l Cu: Dilute 10.00 ml of 1.000 mg/l copper standard solution to 100 ml.

0.00 ml with detemination water.

PROCEDURE: Immediately before use, rinse all glassware with 1% HNO₃ and then detemination water.

1. Deliver into 3 separate 150-ml beakers from 5 to 50 ml of detemination water (blank), from 5 to 50 ml of sample, and sample (the same volume as selected above).

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.

3. Transfer the solutions from Step 2 above to 3 separate 150-ml beakers. Allow the phases to separate, and draw off the H₂₁²K phase into a 15-ml beaker, and place in a separate filter.
4. Read the absorbance of each MISK 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 nm)
- 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.
- Filter: 254
- Aspirate water in place of MISK until air and acetylene flow rates are unchanged.
- Aspirate MISK (water saturated) as close between samples. Read standards immediately after samples to which standard additions are made.

NOTE: Read the absorbance of each sample using the same conditions. NEVER aspirate water in place of MISK while air and acetylene flow rates are unchanged.

B. For Iron:
- Fe Hollow Cathode Lamp
- Wavelength: 248.3 nm (set for maximum energy at approximately 248 nm)
- 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.
- Filter: 313
- Aspirate water in place of MISK until air and acetylene flow rates are unchanged.
- Aspirate MISK (water saturated) as close between samples. Read standards immediately after samples to which standard additions are made.

C. For Lead:
- Pb Hollow Cathode Lamp
- Wavelength: 283.3 nm (set for maximum energy at approximately 283 nm)
- 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.

NOTE: See instruction under Copper conditions concerning MISK/water aspiration.
Aspiration Rate: 5.0-5.5 ml/min

**Filter:** Off

NOTE: See instructions under Conditions concerning Make/Inter.:

**Precipitation:**
- Test 3 - Propylene Formulation
- Test 4 - Ethylene Formulation

**Calculation**

1. Average all absorbance readings.
2. Determine the net sample absorbance, 
   \[ A_{sample} = A_{blank} - A_{sample} \]
   by subtracting the average blank absorbance from the average sample absorbance.
3. Determine a calibration factor, \( F \), for each sample using the relationship:
   \[ F = \frac{A_{net}}{A_{sample}} \]
4. Determine the concentration of metal in the sample using the relationship:
   \[ \frac{N (\text{mM/L})(\text{Inter. conv.})}{A_{sample}} = \] (1)

**Accuracy and Precision**

**Recoveries of Standard Additions of Cu, Fe and Pb to Glycol Samples**

**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>I-6084</td>
<td>Ethylene Formulation</td>
<td>1.0</td>
<td>3.0</td>
</tr>
<tr>
<td>I-6085</td>
<td>Propylene Formulation</td>
<td>2.0</td>
<td>4.0</td>
</tr>
<tr>
<td>I-6086</td>
<td>Ethylene Formulation</td>
<td>1.5</td>
<td>3.5</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,

**Note**: See instructions under Conditions concerning Make/Inter.:

**Precipitation**
- Test 3 - Propylene Formulation
- Test 4 - Ethylene Formulation

**Aspiration Rate**: 5.0-5.5 ml/min
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. The same volume of unknown must be taken for both the sample and the sample plus standard addition, and the sample size taken.

References

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