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Stability of Aqueous Inorganic Lead Solutions in Polycarbonate Containers

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ABSTRACT

The stability of lead(II) acetate solutions at 50, 250 and

1000 mg/L (ppm as lead) in polycarbonate containers has been

studied to assure reliable monitoring. The concentrations of

aqueous solutions of lead maintained at pH 4.5 were determined at

both short term (hours) and long term (months) exposures.

Employing acid washed polycarbonate containers, no

significant loss in lead concentration was detected for up to 100

hours after initial exposure to 50, 250 and 1000 ppm lead

solutions. When containers were pretreated with lead solutions

at comparable concentrations that would ultimately be contained

within them, no significant loss in concentration was detected

over more than 50 days.

1.0 INTRODUCTION

Lead and its compounds pose a significant threat to human beings. However, mechanisms of harmful interactions resulting in toxic and carcinogenic effects are not well understood. In collaboration with the University of Maryland at Baltimore (UMAB), NIST is engaged in a novel research project which is designed to evaluate the molecular mechanisms and tumorigenic response of low-dose lead exposure (50 ppm to 1000 ppm) in the male Fischer-344 rat. A key element of this program requires that well-characterized water soluble lead solutions be used as a quality controlled drinking water supply for the test animals.

Lead(II) compounds normally have limited or poor solubility in aqueous solution. This presents problems in preparing stable monitoring solutions. The reliable stability of low concentrations of inorganic and organotin compounds in a variety of container materials has been previously studied (1). In those experiments, polycarbonate bottles were found to be the best containers for maintaining low concentrations of metal ions. The above tin study has been used as a springboard for this current lead research program. In this study, polycarbonate containers have been evaluated for their effect on both long term and short term storage stability of aqueous inorganic lead solutions.

Other factors may also affect the long and short term stability of lead solutions. These factors must be regulated, but not at the expense of the well-being of the test animals that will be used. Thus, pH and ligand strength of aqueous lead solutions at desired concentrations of 50 ppm, 250 ppm, and 1000 ppm must be such that they will have no deleterious effects on the experimental animals consuming the lead solutions as drinking water. In this report, we evaluate those properties within recognized safe doses for the animals.

Finally, we evaluate the use of highly concentrated lead solutions (20,000 ppm) to reproducibly provide, upon dilution, moderately low concentrations (50 to 1000 ppm) of lead solutions. This method has been demonstrated to provide lead acetate monitoring solutions with concentrations within 5 percent of the intended concentration.

2.0 EXPERIMENTAL

2.1 Chemicals and Materials (2)

Lead(II) acetate (>99.5 %) for solution preparation and 1000 ppm lead(II) nitrate solution for atomic absorption spectrophotometric (AAS) instrument calibration were purchased from commercial sources (Fluka Chemical Corp, Ronkonkoma, New

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York) and used without any further treatment. All polycarbonate bottles (Nalge Co., Sybron Corp., Rochester, New York) were unused prior to their use in these experiments. All activities that required the use of deionized water, i.e., preparation of lead acetate solutions, AAS calibration solutions and rinsing of polycarbonate bottles, used deionized water of 13 megohm-centimeter, or greater, specific resistance (Aqua Suma Reagent Water System, Culligan, Northbrook, IL).

The polycarbonate bottles were pretreated in one of two ways. The first method involved a Sparkleen soap and hot water wash followed by soaking the bottles for approximately 24 hours in 5% aqueous nitric acid prepared with deionized water. Following nitric acid soaking, the bottles were thoroughly rinsed (4 to 5 times) with deionized water. The second method involved repeating the above steps and then conditioning or seasoning these bottles by filling them with lead(II) acetate solutions at the concentrations to be used in stability experiments.

2.2 Measurement of Lead in Solution

All lead concentration determinations were performed with Perkin-Elmer Corp. (Norwalk, Connecticut) atomic absorption spectrophotometers; either a Perkin-Elmer 2380 atomic absorption spectrophotometer fitted with a burner-nebulizer (FAAS) or a Perkin-Elmer 460 AAS fitted with an HGA-500 graphite furnace (GFAA). Thus, samples were either determined at parts per million (ppm) concentration levels by FAAS or at parts per billion (ppb) concentrations by GFAA. Table 1 lists the operational parameters for the two AAS instruments.

3.0 RESULTS AND DISCUSSION

3.1 Analyses by Atomic Absorption Spectroscopy

Atomic absorption spectroscopy is a rapid, sensitive and reproducible technique that is effective in measuring low levels of metal and metalloid species in complex matrices. Figures 1 and 2 illustrate typical calibration curves for GFAA and FAAS obtained prior to making measurements of unknown concentrations of lead. Over the ranges displayed, good linearity is observed. By tailoring the concentration of the analyte solution to fall approximately at the middle of the range displayed in either of the figures, excellent analyses are readily obtained at high (99%) confidence levels.

The AAS instruments were calibrated with freshly diluted calibration solutions each time experimental data were generated, confirming that instrumental response to lead remained fairly constant over many weeks. Table 2 provides slope and intercept calibration data for an 11 week period. The statistically determined slopes for days 58 and 79 indicate that instrumental

response was less sensitive at those times. Although the accuracy of the analysis of unknown lead concentrations is related to the sensitivity of the analytical technique, no significant effects on determination of lead solution concentrations are observed because of these perturbations.

3.2 Lead Analysis of Laboratory Water at UMAB

Samples of water were obtained from three UMAB sources and the lead concentration determined by graphite furnace atomic absorption spectroscopy (GFAA). Each sample was determined 8 times and the GFAA was calibrated with dilutions of a lead(II) nitrate standard AAS calibration solution. Absorbance signals from NIST lead-free, deionized water, used to establish the calibration baseline, were subtracted from the measured absorbance signals produced by the UMAB water samples.

Table 3 summarizes the results of these GFAA determinations of lead concentration in the UMAB water samples. Determinations were initially made within 3 hours after the samples were collected and again one week later. Either of the purified water sources, identified as (C and D or E and F), having nearly undetectable amounts of lead, appears adequate for the dilution of the lead concentrates that will be supplied by NIST. The trace quantities of lead determined in samples A and B (1 to 4 ppb) approach the GFAA detection limit for the operational parameters employed. Since the concentration of lead detected in samples A and B is less than 0.01% of the lead concentration in the least concentrated dosing solution (50 ppm), these values may be considered low enough to be of no consequence in preparation of the lead containing drinking water solutions. However, periodic monitoring of the water used for preparing lead containing drinking water solutions is recommended. Lead concentrations in other containers such as the animals' water bottles should also be periodically tested to verify dosing strength.

Although no conventional procedures (acidification, refrigeration) were used to stabilize the water samples from UMAB, our determinations suggest that no short term surface absorption effects are in operation. Consequently, the initial amounts of lead detected in samples A and B should reflect the actual amount of lead present.

3.3 Solution Stability - pH Considerations

A convenient method of feeding test animals reproducible concentrations of lead would be to draw drinking water solutions from relatively large reservoirs that will maintain concentration stability for reasonably long periods of time. Thus, solution stability becomes a key issue.

Many lead(II) compounds have only little to almost no solubility in water. Even fairly soluble compounds may undergo ligand modifications via decomposition processes to produce less soluble products. For example, $Pb(OH)_2$ has a solubility of 0.0155 g/100 mL and $Pb(OAc)_2$ is 44.3 g/100 mL. Lead(II) acetate, while highly soluble, emits the characteristic odor of acetic acid which suggests that decomposition is occurring by the following pathway:

$$Pb(OAc)_2 + 2 H_2O \longrightarrow Pb(OH)_2 + 2 HOAc .$$
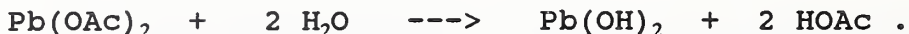
As the acetic acid evaporates from solution, equilibrium is driven to the right, the solution becomes more basic, and the homogeneity of lead in the container becomes suspect. This may be partially due to $Pb(OH)_2$ having a greater affinity for container walls than does $Pb(OAc)_2$. By increasing the acetate ion concentration through the addition of more acetic acid, long term stability of lead(II) may be accomplished. The drinking water solutions must be acidified to stabilize soluble lead. Preliminary experiments suggest that lead(II) acetate remains in solution when acidified to a pH of 4.5 with acetic acid. This amount of acetic acid required may be readily consumed by the test animals without deleterious effects.

As expected, the lead salt and acetic acid form a buffer that mediates the amount of acetic acid that must be added to each of the three concentrations of lead containing drinking water solutions. Figure 3 indicates the amount of glacial acetic acid that must be added to 20 L of 50, 250 and 1000 ppm lead(II) solutions to achieve a pH of 4.5, when the diluent water has an initial pH of 5.1 to 5.8. We may now reproducibly generate solutions at the specified pH values.

Once the chosen pH of 4.5 was reached, no change in pH was observed with time. Thirty-three days after the initial pH adjustment of the 50, 250 and 1000 ppm lead solutions, each lead solution was still found to have the initial pH value of 4.5.

3.4 Stability of High Concentrations of Aqueous Lead(II) Acetate Solutions

A convenient method for the delivery of lead solutions to UMAB involves delivering a lead concentrate to the laboratory which can then be diluted according to a well defined procedure. This procedure negates the task of sending three to four 20 L containers of lead solutions to UMAB on a weekly or biweekly basis. Containers of concentrated lead solutions ranging from 50 to 1000 mL could be more easily shipped.

The lead concentration of the master drinking water solution

was accurately determined by measuring the concentrations of four independent serial dilutions of the master solution by AAS. The lead concentration was found to be 20,000 ± 300 ppm. The relative standard deviation of the data was 1.5 per cent. After 5 months of storage at ambient laboratory temperature (20 °C), in the dark, the concentration of lead was found to be 19,000 ± 100 ppm with a relative standard deviation of 0.5 percent. Thus, over 5 months there was a 5 percent loss in concentration. Lead acetate solutions stable to this extent should be acceptable for the preparation of dilute lead drinking water solutions.

3.5 Long Term Stability of Lead Solutions in "Seasoned" Containers

The stability of lead solutions at nominally 50, 250 and 1000 ppm, were examined for more than 11 weeks. In each case the polycarbonate containers used in these experiments were exposed to one of the aforementioned lead solutions for 3 days prior to the onset of the experiment. The seasoning solutions were discarded from the containers and replaced with the appropriate freshly diluted lead solutions. These fresh solutions then had their total lead concentration determined by atomic absorption spectrophotometry. The seasoning procedure was used to avoid any initial diminution in lead concentration that may occur with unseasoned containers having the property of adsorbing on their walls metal ions from solution.

Three polycarbonate bottles for each lead concentration were used to assess bottle to bottle variations in stability results. Figures 4, 5, and 6 illustrate the changes in concentration as a function of time for 50, 250 and 1000 ppm concentrations of lead acetate solutions acidified to pH 4.5 with acetic acid. At each concentration, data from the three replicate bottles used are distinguished from one another by the solid or dashed lines shown in the figures. The error bar at the lower right part of each figure represents an average of two standard deviations for repeat measurements of the same sample. The ordinate for each figure indicates ten standard deviations.

One observes that for most data sets, the results for the various lead concentration determinations seldom varies beyond 2 standard deviations. Moreover, a linear regression analysis indicates that the error in the slope is normally greater than the calculated value of the slope. These findings suggest that there is no diminution of lead concentrations in these containers.

It must be pointed out, however, that seasoned containers were used in these experiments. Unseasoned containers may initially adsorb lead, thereby causing a much greater solution stability problem. Moreover, the water bottles that the test

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animals will be using will be periodically washed. Labels
indicating the original lead concentrations may also be lost in
the wash process and adsorption properties may become a more
serious problem. These issues are addressed, in part, in the
next Section.

3.6 Short Term Stability Studies

The question now arises as to the stability of diluted lead
solutions in the water bottles from which the test animals must
drink. These bottles will undergo periodic laboratory cleaning,
thereby altering the surface character. An experiment was
therefore designed to determine the adsorption of a 50 ppm lead
solution with an unseasoned bottle.

A 1000 mL polycarbonate bottle, given a hot soapy water
washing and deionized water rinsing but no nitric acid leaching,
was filled with a solution of 50 ppm lead acetate and adjusted to
pH 4.5. The concentration was monitored for up to 100 hours.
Figure 7 shows data obtained for the first 10 hours. No
immediate uptake in lead was observed. When the solution
concentration was measured at 53 and 100 hours, no significant
changes from the data shown in Figure 7 were found. The
stability of lead under these conditions may be partially due to
the strong chelation of lead(II) ions by acetic acid.

3.7 Effectiveness of Dilution Procedure

The quantities of deionized water needed to produce lead
concentrations within 5 percent of their target values were
empirically validated. Table 4 summarizes data from the
aforementioned determinations. Solutions were originally made 5
percent more concentrated by preparing 19 L of the diluted
solution instead of 20 L. Data sets 1 and 2 indicate AAS lead
determinations made at 2 hours and at 30 days after the solutions
were first mixed. Data set 3 indicates the accuracy of our
approach in reaching the desired concentrations of 50, 250 and
1000 ppm. In the Appendix to this report are given general
instructions regarding the preparation of lead containing
drinking water solutions and specific instructions for the
dilution of concentrated 20,000 ppm lead solutions to produce 50,
250 and 1000 ppm solutions.

4.0 CONCLUSIONS

A study was performed to develop a procedure whereby aqueous
solutions of lead at concentrations of 50, 250, and 1000 ppm can
be made to within 5 percent of the designated target value and
have relatively long term (> 2 months) stability. To accomplish
this goal, the effects of several factors were determined.
Polycarbonate solution storage bottles were found to have

negligible lead adsorption on their walls, with unseasoned bottles being indistinguishable from seasoned bottles in solution stability evaluations. Aqueous solutions of lead acetate buffered with acetic acid maintained a pH of 4.5 with no variation, for a 33 day monitoring period. Buffered aqueous lead solutions, stored in polycarbonate bottles at 20,000, 1000, 250, and 50 ppm were found to have acceptable long-term stabilities, showing little or no loss in solution strength over two and a half months.

5.0 REFERENCES:

1. W. R. Blair, G. J. Olson, F. E. Brinckman, R. C. Paule, and D. A. Becker, "An International Butyltin Measurement Methods Intercomparison: Sample Preparation and Results of Analyses" NBSIR 86-3321, National Bureau of Standards, Gaithersburg, MD (1986).

2. Certain trade names and company products are mentioned for identification purposes only. In no case does this imply recommendation or endorsement by the National Institute for Standards and Technology.

TABLE 1

Instrument Parameter	GFAA	FAAS
Pb absorbance line (nm)	217.0	283.3
Slit width (nm)	0.7 alternate	0.7 normal
Lamp	Electrodeless Discharge, 10W	Hollow Cathode, 10mA
Integration time	8 s	1 s
Repeat determinations	8 per sample	8 per sample
Fuel/Oxidizer	-----	acetylene/air

GFAA Furnace settings

Step	Temperature, °C	Ramp, s	Hold @ Temp, s
1	120	3	30
2	400	5	10
3	2300	0	8

TABLE 2

LONG TERM STABILITY CALIBRATION DATA[*]

Time (Days)	Slope	SD	Intercept	SD
2	0.1600	0.0010	0.0664	0.0163
9	0.1557	0.0010	0.0799	0.0305
7	0.1600	0.0010	0.0506	0.0163
9	0.1600	0.0014	0.0454	0.0257
14	0.1589	0.0010	-0.0211	0.020
18	0.1578	0.0010	0.0664	0.0168
23	0.0010	0.0010	0.0799	0.0168
37	0.1626	0.0010	0.0799	0.0192
37	0.1600	0.0010	0.0315	0.0248
51	0.1589	0.0010	0.0454	0.00222
58	0.08418	0.0004		
79	0.08476	0.00039	0.0031	0.0069

[*]Flame atomic absorption data.

TABLE 3

GRAPHITE FURNACE ATOMIC ABSORPTION DETERMINATION OF
LEAD IN UMAB WATER SAMPLES

SAMPLE[1,]	AVERAGE ± STD DEV.[2,3,4] <4 hours	(μg/L or ppb) 7 days
A	3.1 ± 0.8	1.6 ± 0.1
B	1.9 ± 0.5	1.2 ± 0.2
C	ND	ND
D	ND	0.1 ± 0.2
E	ND	0.2 ± 0.2
F	ND	0.2 ± 0.3

[1]SAMPLES

    A and B -- replicate samples of distilled water piped to
animal cages.

    C and D -- distilled water from 6th floor Bressler Hall.

    E and F -- distilled water from Millipore system.

[2]Samples 7 days old were analyzed at greater sensitivity than the
samples 4 hours old.

[3]Background absorptions were subtracted from the sample
absorptions.

[4]ND--Not detected.

TABLE 4

Effectiveness of Lead Concentrate Mixing Procedures
and Solution Stability

[Pb] calculated	[Pb] observed	Variation Percentage
DATA SET 1[*]		
52.6	55.1	4.8
263	275	4.6
1050	1040	4.8
DATA SET 2[*]		
52.6	51.9	1.3
263	261	1.3
1050	1040	1.0
DATA SET 3[*]		
50	49.7	0.6
250	245	2.0
1000	963	3.7

[*]DATA SET 1 - Measurements made within 2 hours of mixing.

[*]DATA SET 2 - Measurements made on same solution 30 days later.

[*]DATA SET 3 - DATA SET 2 further diluted to desired
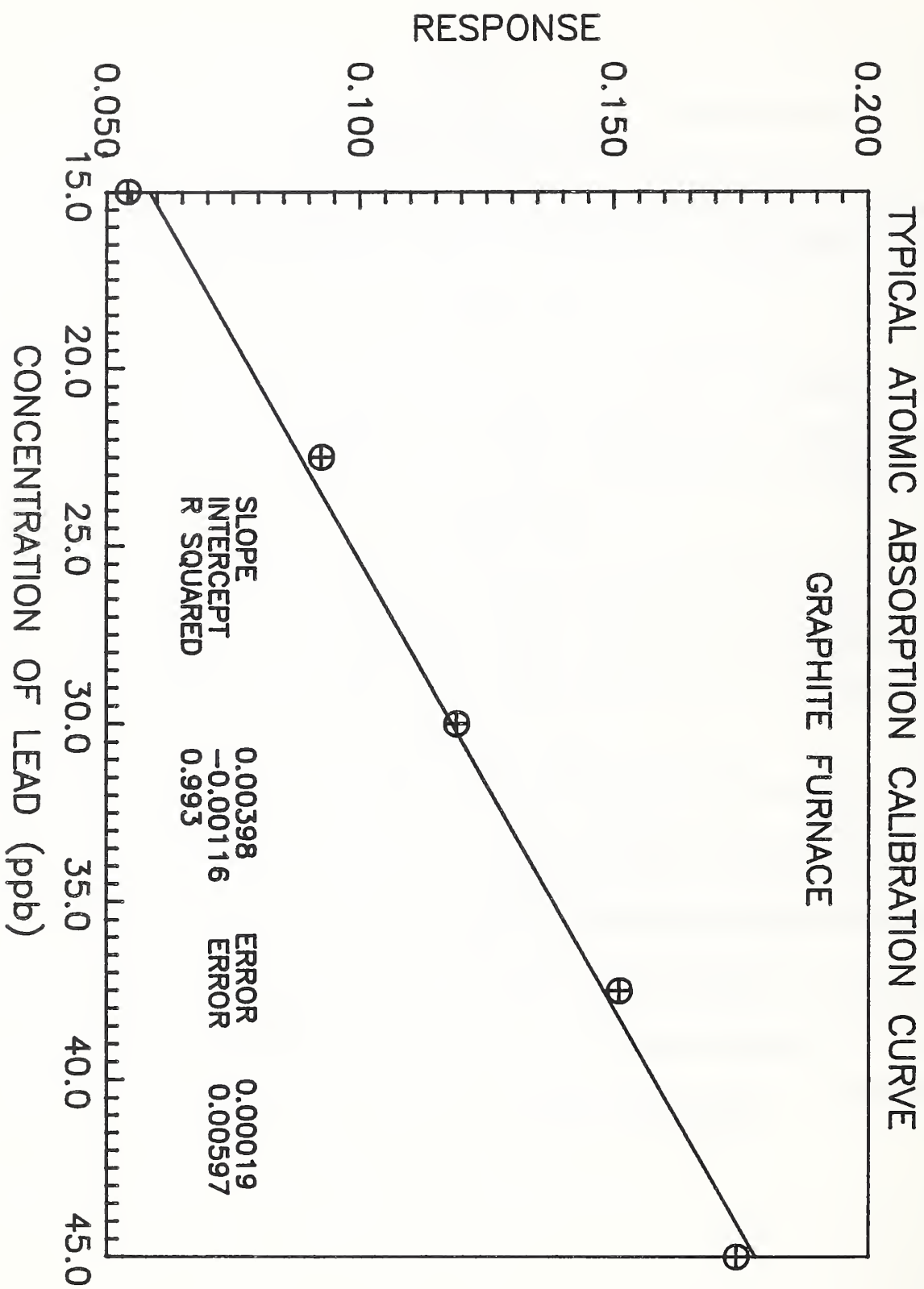   concentrations.

FIGURE 1

TYPICAL ATOMIC ABSORPTION CALIBRATION CURVE
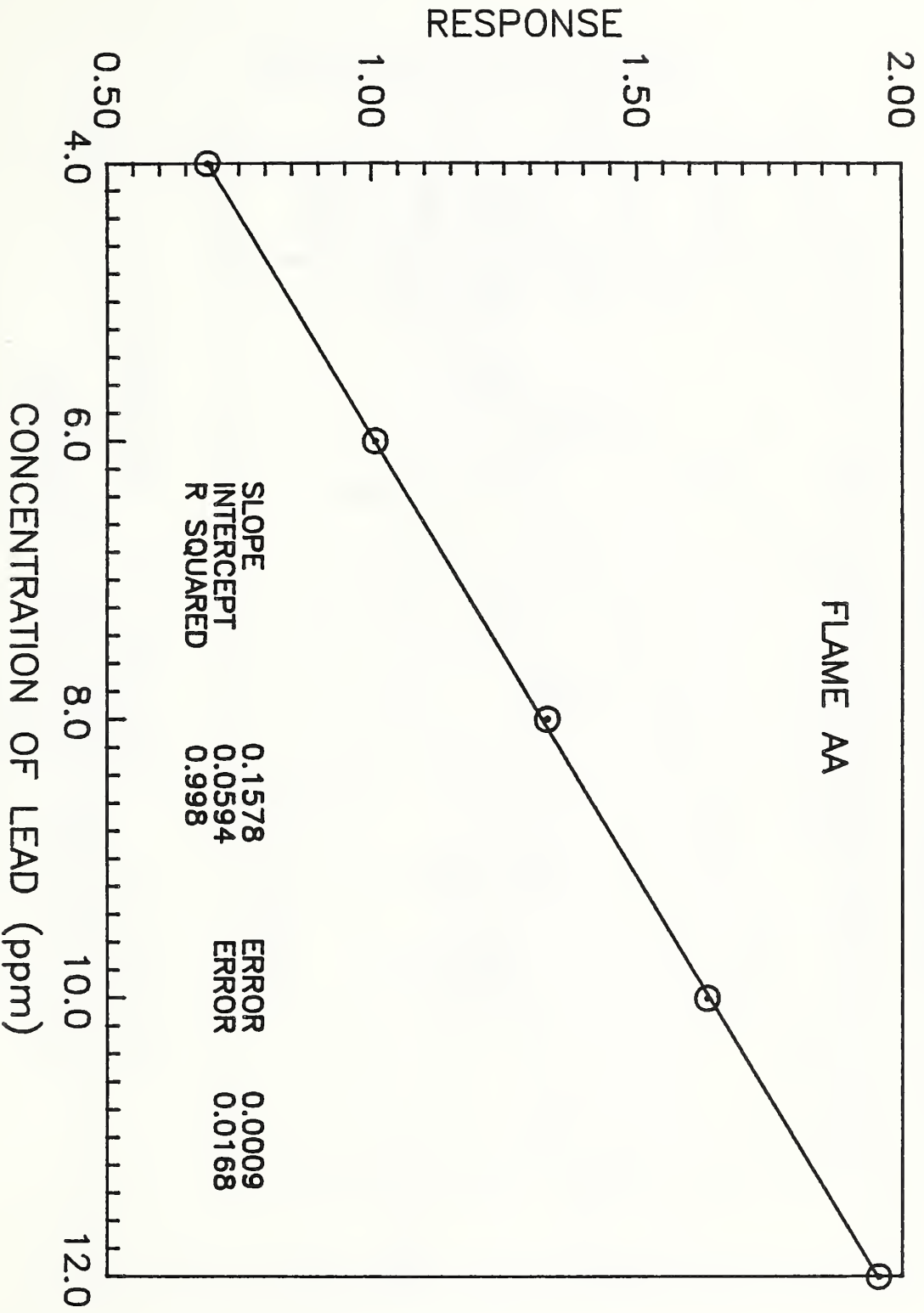
GRAPHITE FURNACE

SLOPE	0.00398	ERROR	0.00019
INTERCEPT	-0.00116	ERROR	0.00597
R SQUARED	0.993		

FIGURE 2

TYPICAL ATOMIC ABSORPTION CALIBRATION CURVE

FLAME AA

SLOPE	0.1578	ERROR	0.0009
INTERCEPT	0.0594	ERROR	0.0168
R SQUARED	0.998		

RESPONSE

2.00

1.50

1.00

0.50

CONCENTRATION OF LEAD (ppm)

4.0   6.0   8.0   10.0   12.0

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FIGURE 3

BUFFERING CAPACITY OF LEAD SOLUTIONS
20 L PER CONCENTRATION

o——o 1000 ppm   ●--●250 ppm   ▲--▲50 ppm

SOLUTION pH

GLACIAL ACETIC ACID ADDED (mL)

14

FIGURE 4

CHANGE IN SOLUTION CONCENTRATIONS VS TIME
50 PPM

2 STD DEV

CONCENTRATION (PPM)

CONTAINER EXPOSURE TIME (DAYS)

15

CHANGE IN SOLUTION CONCENTRATIONS VS TIME
250 PPM

FIGURE 5

FIGURE 6

CHANGE IN SOLUTION CONCENTRATIONS VS TIME
1000 PPM

CONCENTRATION (PPM)

CONTAINER EXPOSURE TIME (DAYS)

2 STD DEV

FIGURE 7

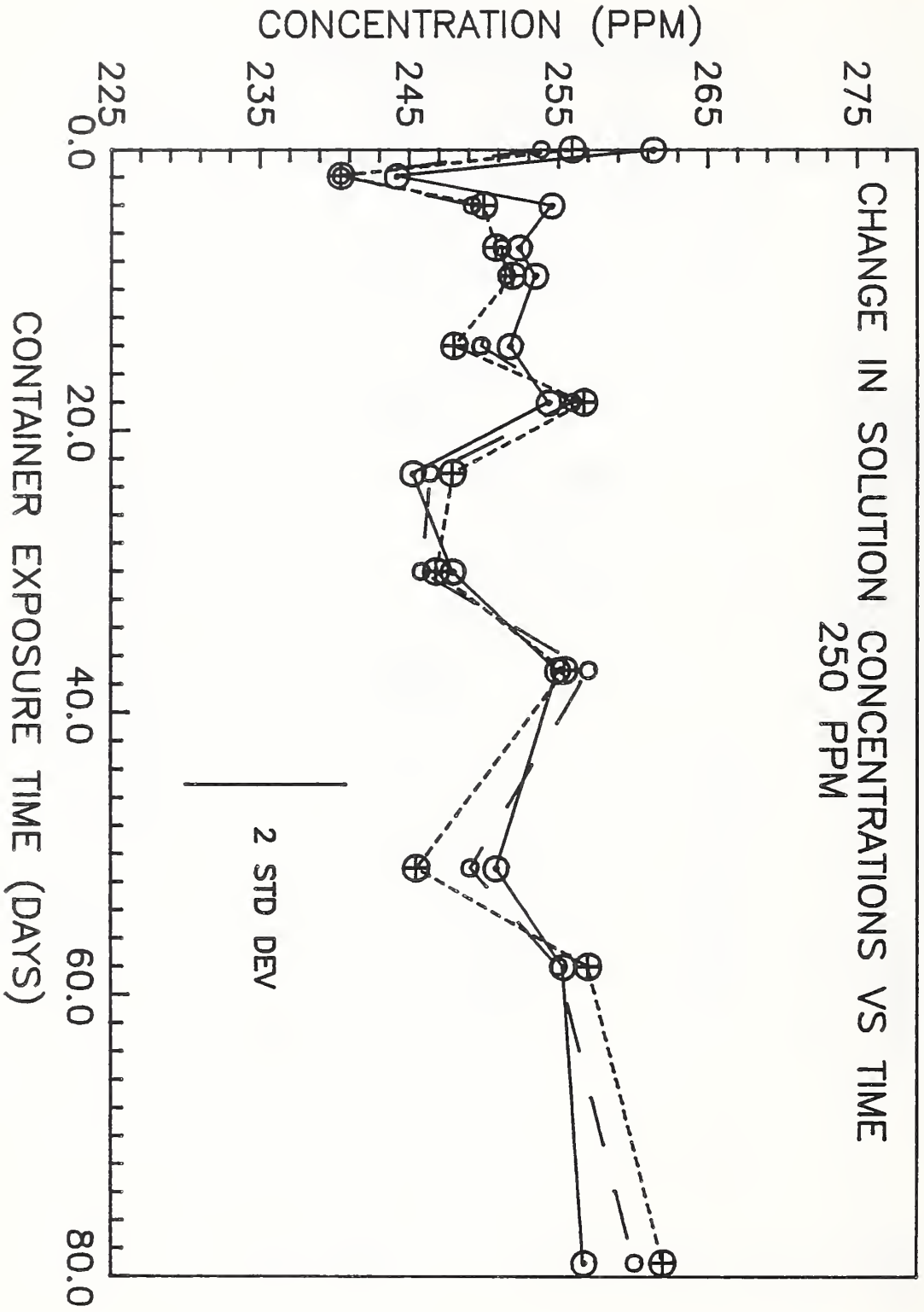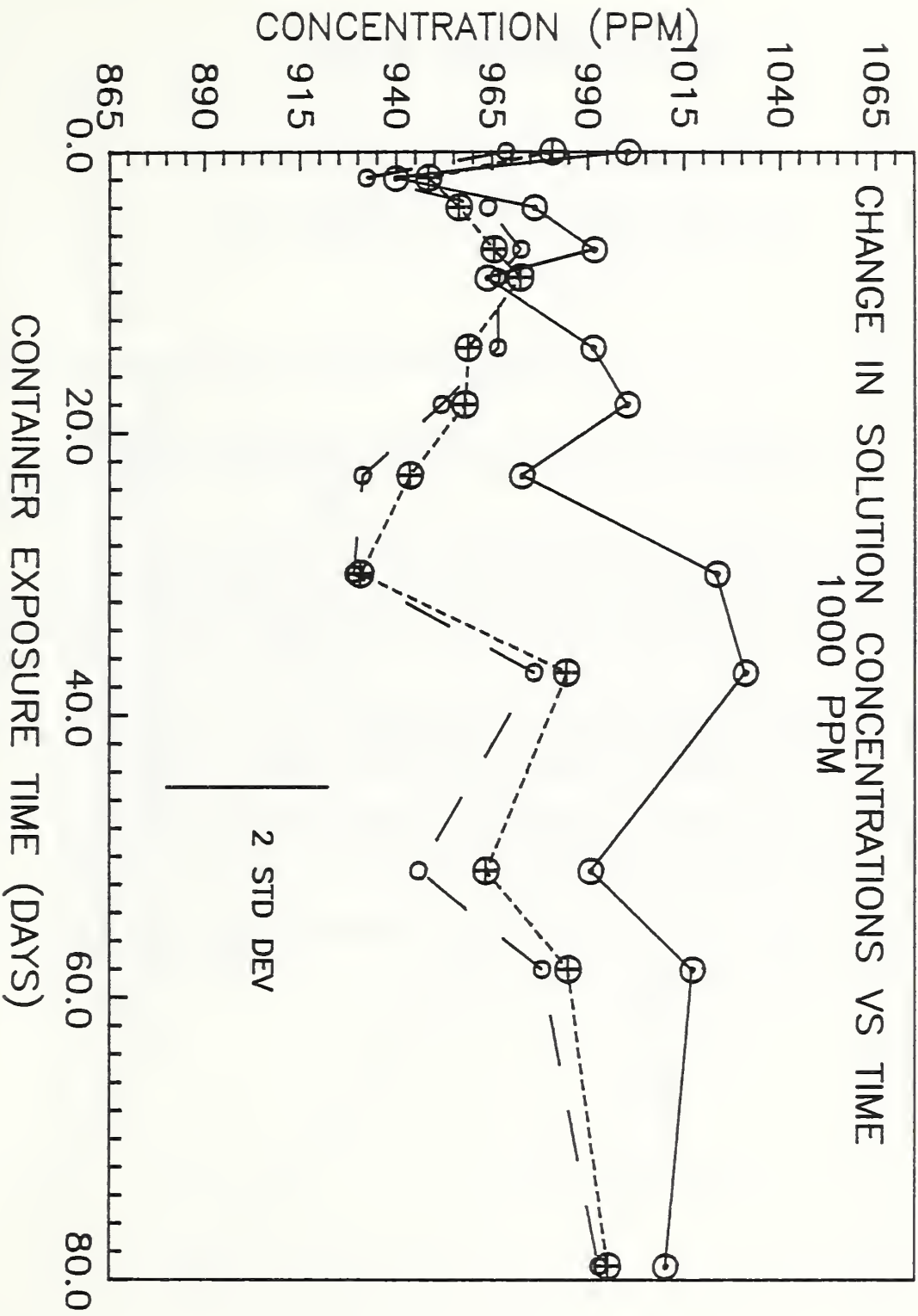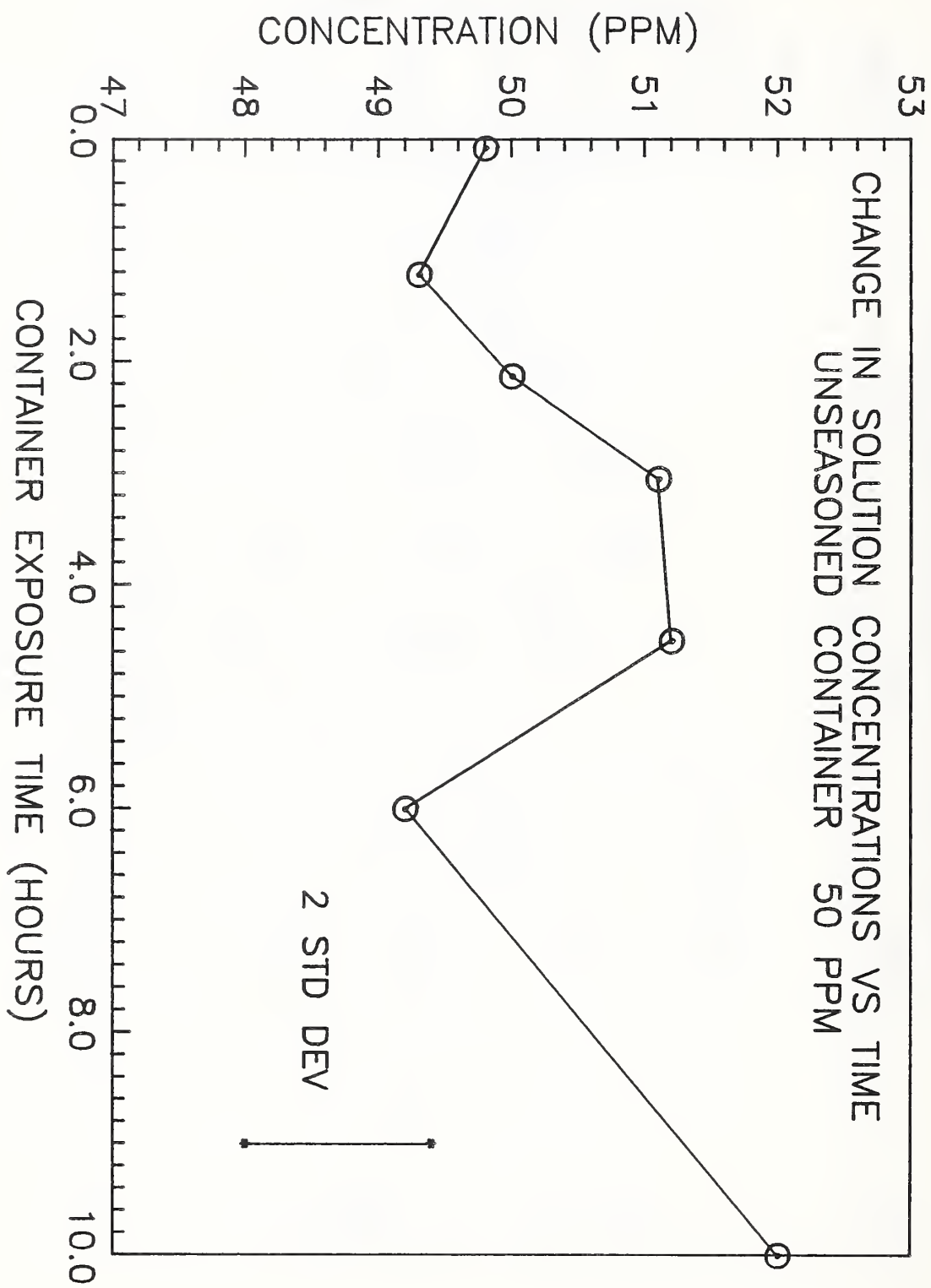CHANGE IN SOLUTION CONCENTRATIONS VS TIME
UNSEASONED CONTAINER 50 PPM

2 STD DEV

APPENDIX

DILUTION INSTRUCTIONS FOR
LEAD ACETATE DRINKING WATER SOLUTIONS

General Information and Instructions:

Use clean graduated cylinders for all volumetric measurements. Cylinders of 1000 or 2000 mL volumes are suggested.

After a soap and water wash and rinse, further clean graduated cylinders by allowing them to stand overnight full of 5% aqueous nitric acid. Rinse 4 to 5 times with distilled water. Keep the tops of the cylinders covered when they are not in use.

Carboys of 20 L capacity will be used to contain lead acetate drinking water solutions. These carboys will be prepared at NIST and may be used as received.

The concentrated lead solutions have been pH adjusted at NIST to result in a pH of 4.5 in the diluted lead solutions.

All empty polycarbonate bottles should be returned to NIST for recycling.

DILUTION INSTRUCTIONS FOR
LEAD ACETATE DRINKING WATER SOLUTIONS

50 ppm Concentration

1) Fill 20 L carboy with 15 L of distilled water.

2) Pour the contents of a 50 mL bottle containing concentrated lead solution into the carboy.

3) Add 25 mL of distilled water to the 50 mL bottle, put on the cap and shake the bottle. Pour this rinse water into the 20 L carboy. Repeat this rinsing operation four times using 25 mL of distilled water for each rinse and add the rinse water to the carboy each time.

4) The carboy now contains: 15 L of distilled water
                                 50 mL of conc. lead solution
                                100 mL of rinse water

         Volume in Carboy: 15.15 L

5) Add 4 L plus 850 mL of distilled water to bring the final carboy volume to 20 L.

6) After thorough mixing, the solution is ready for use.

DILUTION INSTRUCTIONS FOR
LEAD ACETATE DRINKING WATER SOLUTIONS

250 ppm Concentration

1)   Fill 20 L carboy with 15 liters of distilled water.

2)   Pour the contents of a 250 mL bottle containing concentrated lead solution into the carboy.

3)   Add 125 mL of distilled water to the 250 mL bottle, put on the cap and shake the bottle. Pour this rinse water into the carboy. Repeat this rinsing operation a total of four times using 125 mL of distilled water for each rinse and add the rinse water to the carboy each time.

4)   The carboy now contains: 15 L of distilled water
                                    250 mL of conc. lead solution
                                    500 mL of rinse water

          Volume in Carboy: 15.75 L

5)   Add 4 L plus 250 mL of distilled water to bring the final carboy volume to 20 L.

6)   After thorough mixing, the solution is ready for use.

DILUTION INSTRUCTIONS FOR
LEAD ACETATE DRINKING WATER SOLUTIONS

1000 ppm Concentration

1)   Fill 20 L carboy with 15 L of distilled water.

2)   Pour the contents of a one liter bottle containing
concentrated lead solution into the carboy.

3)   Add 250 mL of distilled water to the one liter bottle,
put on the cap and shake the bottle. Pour this rinse water
into the 20 liter carboy. Repeat this rinsing operation
four times using 250 mL of distilled water for each rinse
and add the rinse water to the carboy each time.

4)   The carboy now contains: 15 L of distilled water
                                1 L conc. lead solution
                             1 L of rinse water

          Volume in Carboy: 17 L

5)   Add 3 L of distilled water to bring final carboy
volume to 20 L.

6)   After thorough mixing, the solution is ready for use.

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11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

The stability of lead(II) acetate solutions at 50, 250 and 1000 mg/L (ppm as lead) in polycarbonate containers has been studied to assure reliable monitoring. The concentrations of aqueous solutions of lead maintained at pH 4.5 were determined at both short term (hours) and long term (months) exposures.

Employing acid washed polycarbonate containers, no significant loss in lead concentration was detected for up to 100 hours after initial exposures to 50, 250 and 1000 ppm lead solutions. When containers were pretreated with lead solutions at comparable concentrations that would ultimately be contained within them, no significant loss in concentration was detected over more than 50 days.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

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