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Staten Island/New Jersey Urban Air Toxics Assessment Project Report

Volume III
Part B

Results and Discussion of the Metals, Benzo[a]pyrene, and Formaldehyde in Ambient Air

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This report is a collaborative effort of the staffs of the Region II Office of the U.S. Environmental Protection Agency (EPA), the New Jersey Department of Environmental Protection and Energy, the New York State Department of Environmental Conservation, the New York State Department of Health, the University of Medicine and Dentistry of New Jersey and the College of Staten Island. The project was undertaken at the request of elected officials and other representatives of Staten Island concerned that emissions from neighboring industrial sources might be responsible for suspected excess cancer incidences in the area.

Other EPA offices that provided assistance included the Office of Air Quality Planning and Standards, which provided contract support and advice; and particularly the Atmospheric Research and Exposure Assessment Laboratory, which provided contract support, quality assurance materials, and sampling and analysis guidance, and participated in the quality assurance testing that provided a common basis of comparison for the volatile organic compound analyses. The Region II Office of Policy and Management and its counterparts in the States of New York and New Jersey processed the many grants and procurements, and assisted in routing funding to the project where it was needed.

The project was conceived and directed by Conrad Simon, Director of the Air and Waste Management Division, who organized and obtained the necessary federal funding.

Oversight of the overall project was provided by a Management Steering Committee and oversight of specific activities, by a Project Work Group. The members of these groups are listed in Volume II of the report. The Project Coordinators for EPA, Robert Kelly, Rudolph K. Kapichak, and Carol Bellizzi, were responsible for the final preparation of this document and for editing the materials provided by the project subcommittee chairs. William Baker facilitated the coordinators' work.

Drs. Edward Ferrand and, later, Dr. Theo. J. Kneip, working under contract for EPA, wrote several sections, coordinated others, and provided a technical review of the work.

The project was made possible by the strong commitment it received from its inception by Christopher Daggett as Regional Administrator (RA) for EPA Region II, and by the continuing support it received from William Muszynski as Acting RA and as Deputy RA, and from Constantine Sidamon-Eristoff, the current RA. The project has received considerable support from the other

project organizations via the Management Steering Committee, whose members are listed in Volume II.

PREFACE - DESCRIPTION OF THE STATEN ISLAND/NEW JERSEY URBAN AIR TOXICS ASSESSMENT PROJECT REPORT

This report describes a project undertaken by the States of New York and New Jersey and the United States Environmental Protection Agency with the assistance of the College of Staten Island, the University of Medicine and Dentistry of New Jersey and, as a contractor, the New Jersey Institute of Technology.

Volume I contains the historical basis for the project and a summary of Volumes II, III, IV, and V of the project report.

Volume II of the report lists the objectives necessary for achieving the overall purpose of the project, the organizational structure of the project, and the tasks and responsibilities assigned to the participants.

Volume III of the report presents the results and discussion of each portion of the project for ambient air. It includes monitoring data, the emission inventory, the results of the source identification analyses, and comparisons of the monitoring results with the results of other studies. Volume III is divided into Part A for volatile organic compounds, and Part B for metals, benzo[α]pyrene (BaP), and formaldehyde. Part B includes the quality assurance (QA) reports for the metals, BaP, and formaldehyde.

Volume IV presents the results and discussion for the indoor air study performed in this project. It contains the QA reports for the indoor air study, and a paper on the method for sampling formaldehyde.

Volume V presents the results of the detailed statistical analysis of the VOCs data, and the exposure and health risk analyses for the project.

Volume VI, in two parts, consists of information on air quality in the project area prior to the SI/NJ UATAP; quality assurance (QA) reports that supplement the QA information in Volume III, Parts A and B; the detailed workplans and QA plans of each of the technical subcommittees; the QA reports prepared by the organizations that analyzed the VOC samples; descriptions of the sampling sites; assessment of the meteorological sites; and a paper on emissions inventory development for publicly-owned treatment works.

The AIRS database is the resource for recovery of the daily data for the project. The quarterly summary reports from the sampling organizations are available on a computer diskette from the National Technical Information Service.

STATEN ISLAND/NEW JERSEY
URBAN AIR TOXICS ASSESSMENT PROJECT

VOLUME III, PART B.
RESULTS AND DISCUSSION OF THE METALS, BENZO[α]PYRENE, AND FORMALDEHYDE IN AMBIENT AIR

EPA/902/R-93-001d

TABLE OF CONTENTS

1. INTRODUCTION 1
2. SAMPLING AND ANALYSIS 2
   2.1 Particulates 2
   2.2 Formaldehyde 3
3. RESULTS AND DISCUSSION 3
   3.1 Concentration Data 3
   3.2 Comparisons to Concentration Data from Other Locations 3
   3.3 Temporal Patterns 5
   3.4 Spatial Patterns 6
      3.4.2 Iron 6
      3.4.3 Nickel 6
      3.4.4 Cobalt 7
      3.4.5 Vanadium and chromium 7
      3.4.6 Lead 7
      3.4.7 Copper and zinc 8
      3.4.8 Cadmium 8
      3.4.9 Mercury 8
      3.4.10 BaP 8
4. CONCLUSIONS 9
5. ACKNOWLEDGEMENT 9
6. REFERENCES 10

TABLES IIIB-1 through 16 11
TABLES IIIB-17a through 18b 27
FIGURES IIIB-1 through 10 33
FIGURES IIIB-15 through 26 47
FIGURES IIIB-27 through 53 59
APPENDICES 88
   APPENDIX A - QUALITY ASSURANCE SUMMARY A-1
   APPENDIX B - DATA SUMMARIES BY QUARTERLY AVERAGE B-1
   APPENDIX C - DATA SUMMARIES BY ANNUAL AVERAGE C-1
   APPENDIX D - QUALITY ASSURANCE REPORT FROM NEW JERSEY INSTITUTE OF TECHNOLOGY D-1
   APPENDIX E - QUALITY ASSURANCE REPORT FROM NEW YORK STATE DEPARTMENT OF HEALTH E-1

1. INTRODUCTION

The Staten Island/New Jersey Urban Air Toxics Assessment Project (SI/NJ UATAP) included sampling for airborne particulate matter and for formaldehyde at four sites, two in Staten Island and two in New Jersey. The sampling period included October 6, 1988, through September 19, 1989. The work was intended to provide data for use in estimating the health risks arising from inhalation exposure to the toxic substances transported by the particles, and to formaldehyde.

The particulate samples were prepared and analyzed at two of the participating laboratories; and the formaldehyde samples, at an EPA contract laboratory. The results were compiled, then reviewed for validation.

The elements and compounds (analytes) selected for the project were regarded as potentially hazardous materials likely to be present at measurable concentrations in the samples, and measurable with the methods available to the laboratories. The potential sources of the materials include resuspended soils, industrial emissions, incinerators, autos and trucks, power plants, and home heating systems. While some of the analytes selected may be characteristic of particular source categories, it was not an objective of the project to fully characterize the potential sources.

The data, summaries of which are available on computer diskettes through the National Technical Information Service (NTIS), are useful for (1) determining the average airborne concentrations for various time periods, such as annual averages; (2) seeking patterns in the concentrations versus time, space, source emissions, or meteorological parameters; and (3) estimating possible health risks resulting from inhalation exposure.

This volume of the project report presents the concentrations and patterns observed, and possible relations to other variables. A risk assessment is presented in Volume V of the project report.

2. SAMPLING AND ANALYSIS

2.1 Particulates

The particulate samples were taken with Hi-Vol (high-volume) samplers. These machines collect the particulate matter on highly efficient filters and collect all particles up to about 50 microns (micrometers) in diameter. The respirable portion of the samples is contained in particles with aerodynamic diameters of about 10 microns or less; based on literature data[1], the respirable portion often constitutes approximately 30 to 60% of the total sample concentration.

The initial stage in sample preparation was dissolution of the analytes from the particulate matter on the filter in an acid solution for metals analysis, or, for benzo[α]pyrene (BaP), in a solvent. There were differences between the acid dissolution methods of the two laboratories. The solutions obtained were analyzed by either the atomic absorption or Inductively Coupled Plasma-Optical Emission methods.

A summary of the results of the quality assurance (QA) evaluation of the submitted data follows; it states whether the data have been included in or excluded from the project data base, and, if included, if there is an attending caveat on use of the data. A more detailed QA discussion of the data is in an appendix of this volume.

The NJIT and NYSDOH data for cadmium, copper, lead, manganese, and zinc were accepted.

The NJIT data for chromium, iron, and nickel were accepted.

The NYSDOH data for arsenic and barium were accepted; NJIT did not provide data for these.

The NJIT data for mercury were accepted; there is no standard to assess accuracy, however. NYSDOH did not provide mercury data.

The NYSDOH data for beryllium, cobalt, and molybdenum were accepted, but there is no standard to assess their accuracy; recoveries were good for solution spikes. Beryllium was never detected by NYSDOH. NJIT did not provide data for

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[1] This assertion is based on information provided in an EPA criteria document addressing the development of the PM-10 standard (for respirable-size particles) from the TSP standard (for total suspended particulates) (U.S. EPA, 1986).

beryllium or molybdenum; reported data for cobalt were insufficient.

The NYSDOH data for chromium were rejected; recovery was 18%. The NYSDOH data for iron and nickel were accepted; but with a caveat that they be regarded as minimum values since recoveries were less than 80%.

NJIT data for BaP were accepted. NYSDOH data for BaP were accepted but with a caveat, since recoveries averaged 49%.

2.2 Formaldehyde

The formaldehyde samples were collected by NJIT and NYSDEC on 2,4-dinitophenylhydrazine-coated silica cartridges prepared and analyzed by NSI, an EPA contract laboratory. Ozone interferes with quantitation of formaldehyde by the method used; no correction factor is available for use of the reported concentrations as other than minimum values.

Formaldehyde data from the samples analyzed by NSI were included in the project data base. Samples analyzed by NJIT were not included due to the unavailability of QA information.

3. RESULTS AND DISCUSSION

3.1 Concentration Data

Quarterly and annual average concentration data are presented in Tables IIIB-1 through 16. Tables in the appendix of this volume order the sites by annual average concentration and by quarterly average concentration.

3.2 Comparisons to Concentration Data from Other Locations

Ranges and medians for annual average concentrations for selected chemicals[2] from the SI/NJ UATAP and the U.S. EPA Urban Air Toxics Monitoring Project (UATMP) studies (U.S. EPA, 1989)

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[2] Generally, the metals selected for these comparisons were those for which information for quantitative risk assessment was available.

are provided in Tables IIIB-17a and 18a. Minimum detection limits (mdls) for the metals and BaP for the two studies are listed in Table IIIB-17b. The annual average concentrations for each compound at each of the 17 UATMP sites are listed in Tables IIIB-17c and 18b, and compared graphically to the median annual average concentration for the SI/NJ UATAP sites, or to concentrations for individual SI/NJ UATAP sites, in Figures IIIB-1 through 14. On the basis of these annual averages, the SI/NJ UATAP data are comparable largely to the data for the UATMP study. Interlaboratory comparisons with samples of known composition would be necessary before any conclusions could be drawn regarding possible differences in the data sets.

For copper, iron, lead, manganese, zinc, and BaP, the SI/NJ UATAP annual average concentrations are at or below the median concentration for the UATMP sites.

Cadmium concentrations at the SI/NJ UATAP sites are higher than those at all but four of 17 UATMP sites. SI/NJ UATAP chromium concentrations, available only for the three New Jersey sites, were higher than those for all but three of 17 UATMP sites.

The annual average concentrations of nickel and vanadium at the SI/NJ UATAP sites data are, respectively, higher than those at all but one of the UATMP sites, and higher than those at all the UATMP sites. Vanadium and nickel are emitted by large oil-burning sources in the northeastern United States, a region which uses crude oils containing these elements.[3]

The graphs for cobalt and molybdenum show two unusual characteristics. The SI/NJ UATAP data are unusually high compared to the UATMP results; and identical concentrations are reported for a number of the UATMP sites. These are likely to be consequences of the differences in detection limits for the two studies. The UATMP mdl for cobalt is 1.8 ng/m^3; while the SI/NJ UATAP mdl is 5 ng/m^3. The UATMP mdl for molybdenum is 2.3 ng/m^3; while the SI/NJ UATAP mdl is 24 ng/m^3. The reporting convention for this project was that if the measured concentration was below the mdl, the sample concentration was to be reported as half the mdl. The combination of higher detection limits with readings less than the mdl might account for the apparent higher concentrations in the SI/NJ UATAP study area. In addition, the chemical analyses for these two elements may be the cause for apparent differences in concentrations at the SI/NJ UATAP sites

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[3] The airborne concentrations of nickel and vanadium decreased markedly in New York City from the late 1960s to the mid-1970s as the sulfur content of oils was reduced to meet SO_2 standards (Kleinman et al., 1977).

and the UATMP sites; cobalt and molybdenum are difficult to dissolve and to analyze with accuracy. Thus, the reported concentrations for these two elements should be given very cautious consideration.

The data for arsenic appear to place the SI/NJ UATAP results in the midrange of the UATMP data; again, however, a difference in mdls might account for the apparent difference in concentrations for the two studies. The mdls were 30, and later, 2 ng/m^3 for the SI/NJ UATAP sites; and 5.5 ng/m^3 for the UATMP sites.

While the results for formaldehyde at two SI/NJ UATAP sites are in the midrange of the annual average concentrations reported for the UATMP sites, the consequence of the ozone interference affecting both sets of data is that little information can be derived regarding actual site-to-site differences in formaldehyde concentration.[4]

3.3 Temporal Patterns

Concentration versus sampling date was plotted for multiple analytes at single sites in a search for covariation of particulate concentrations, and hence, suggestion of common sources. The results are Figures IIIB-15 through 25.

Figures IIIB-15 to 18 for Carteret, NJ, exhibit the normal variation in concentration with time found in data of this nature. Variations in source emission rates and in meteorological variables such as wind speed and direction affect the airborne concentrations and cause the apparently irregular variations in concentration. Visual comparisons of the results for the ferrous metals in Figure IIIB-15 (nickel, chromium, manganese, iron) with those for the non-ferrous metals in Figure IIIB-16 (cadmium, copper, zinc, lead) indicate that they are not closely related in their patterns. The dissimilarity of the concentration patterns for lead and BaP (a polynuclear aromatic hydrocarbon) in Figure IIIB-17 suggests that these two substances are not from a common source.

Similar conclusions can be drawn from the results for the Elizabeth site in Figures IIB-19 through 22, the Susan Wagner

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[4] Samplers in use for more recent years of the UATMP studies were modified to reduce/remove the ozone interference regarded as not amenable to use of a correction factor, and leading to an underreporting of formaldehyde concentrations.

site in Figures IIIB-23 and 25, and the PS-26 site in Figures IIIB-26 through 28. In the latter two cases, some similarity in patterns for some of the elements indicates that a complete statistical analysis would be necessary to determine whether any correlations did or did not exist in the results.

The complexity of the variables that create and disperse the particles in the atmosphere has been known for many years. Statistical methods can be applied to the data with the aid of a computer to obtain an understanding of the impacts of meteorological variables and source emissions on the airborne concentration patterns of the particles. However, because of the cost of the computer analyses, the statistical approaches are usually reserved for cases where significant health risks or potential regulatory violations must be addressed.

3.4 Spatial Patterns

Figures IIIB-29 through 42 provide a comparisons of concentration patterns (concentration versus sampling date) for a single analyte at multiple sites. The graphs have been sorted into the order of barium, ferrous metals, non-ferrous metals, and BaP. In cases for which two laboratories generated the reported concentrations, graphs representing another level of sorting are provided lest interlaboratory differences confound the observation of similarity in patterns.

3.4.1 Barium

The data for barium at Susan Wagner and PS-26 in Figure IIIB-29 suggest the likelihood of a relationship between concentrations at these two sites.

3.4.2 Iron

Figures IIIB-30 through 32 for iron show that the patterns for the four sites are similar and no major difference exists between the data for the sites in the two states. The results are the same for manganese (Figures IIIB-33 through 35); note that the scales for the x- and y-axes in Figures 34 and 35 differ by a factor of 2, resulting in an apparent visual difference that does not appear in Figure IIIB-33.

3.4.3 Nickel

The results for nickel are different in that the two sites in each state are similar, but the seasonal trends differ for the

two sets of monitors. See Figures IIIB-36 through 38. Peak concentrations in New Jersey occur in the period from April to August 1989, while the peak concentrations in New York occur in the period from January to February 1989. While insufficient information is available to provide a definitive explanation of these patterns, rationalization of the observations might involve such variables as seasonal variations in wind direction or velocity, number of sources and their locations relative to the monitors, and interlaboratory differences.

3.4.4 Cobalt

No conclusions can be drawn for cobalt (New York sites only), for which concentrations were less than the mdl of 5 ng/m^3.

3.4.5 Vanadium and chromium

For vanadium and chromium there are data for only one state in each case--vanadium for New York, and chromium for New Jersey. For both cases, Figures IIIB-39 and 40, the pairs of sites appear to show the same temporal variations. The period of higher concentrations for chromium occurs from April to June of 1989; source emissions, source versus sampler locations, and/or meteorological variations are possible causes of the concentration differences.

While average wind speeds are lower in the summer and higher in the winter in the region, it is possible that the drying of soils in the spring could permit resuspension of contaminated soil with increased airborne concentrations of chromium as seen in Figure IIIB-40. The concentrations would decrease when vegetative cover grows over the soil, or when the soil is wet or frozen and less easily resuspended. Resuspension occurs mostly for the larger particles, with a relatively small fraction in the respirable-size range which may reach the deep lung (alveoli).

3.4.6 Lead

The patterns for lead in Figures IIIB-42 through 44 show distinct differences between the data for the two states. The concentrations in New Jersey are about 40 ng/cu.m from October to December of 1988, followed by a drop to about 20 ng/cu.m from January to mid-March of 1989, and a return to about 40 to 50 ng/cu.m from late March to early June. The data for New York are around 40 ng/cu.m from October 1988 to March of 1989, decreasing to around 20 ng/cu.m from April to mid-June.

Drops in average concentrations are often related to seasonal shifts in average wind speeds; however, an average windspeed variation would be regional in nature and affect all stations in a similar manner. Thus the shifts in lead concentrations in these data must be related to variables that are not regional in nature, but more local to the sampling sites. Such variables could be a combination of the geometry of source and sampler locations with the average wind direction for the various sites and time periods. Note also that the time period of the higher chromium concentrations does not coincide with the return of higher lead concentrations, nor do the other changes in the concentrations of these two elements show any similarities.

3.4.7 Copper and zinc

The data for copper and zinc (Figures IIIB-44 through 49) show little or no difference between the two states, and suggest no apparent relationships to potential emission sources, sites, or time periods.

3.4.8 Cadmium

For the New Jersey sites in Figure IIIB-50, there appears to be a trend towards higher cadmium concentrations over the one-year sampling period. The concentration increased from about 10 ng/m3 in October 1988, to about 50 ng/m3 in September 1989.

3.4.9 Mercury

Mercury data were available only at the New Jersey sites, where there was an apparent decreasing trend in concentrations to less than 0.5 ng/cu.m for the period from January to September 1989. See Figure IIIB-51.

3.4.10 BaP

BaP was measured at five sites including the background site. The ranges of concentrations and temporal variations are essentially the same for all five sites (Figures IIIB-52 through 55). This compound serves as an indicator compound for all of the polynuclear aromatic hydrocarbons--a class of compounds emitted by all fossil fuel burning sources, with auto and truck traffic often the predominant factors. The similarity in the data for all five sites indicates that the mobile sources predominate in this area and reflects the regional influences of traffic variation with season, as well as regional meteorological effects.

The lack of similarity of the patterns of lead and BaP indicates that the mobile sources are no longer significant in the overall lead emissions in the area.

4. CONCLUSIONS

The data sets for analytes found in the particulate matter samples for the SI/NJ UATAP show that many of these toxic substances are in the same concentration ranges as those found for a number of sites in the EPA UATMP program during the same time period. Where the SI/NJ UATAP data appear to be high, as is the case for cadmium, vanadium (New York sites only, no valid data for New Jersey sites), and nickel, there is an unfortunate lack of certainty regarding accuracy of the reported SI/NJ UATAP results. Vanadium and nickel concentrations have been higher in the northeast than in other regions of the U.S. for many years. There is little indication of unusual impacts from industrial sources in the area, with only cadmium showing an increasing concentration trend. Chromium concentrations were generally higher at the New Jersey sites than at most of the UATMP sites; no valid data were available for the New York sites.

There are a number of interesting and potentially useful temporal and spatial patterns in the data, with some substances showing differences between the sampling sites in the two states, but not between sites within the states. Determination of the likely causes of the patterns in the results would require a major effort using multivariate statistical methods. Separating source and meteorological effects might require concentration data for additional chemical species, and meteorological data not currently in the data base. Such a complex, costly program might be justified in cases where the likelihood of a significant health risk or of a regulatory violation has been demonstrated.

Since an ozone interference negatively biased the formaldehyde results, little information is derived from the apparent site-to-site differences for this compound.

5. ACKNOWLEDGEMENT

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Table IIIB-1

ARSENIC

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	FIRST YEAR OCT 1987 - SEPT 1988 ARITH. MEAN (ng/m3)
CARTERET	B										
ELIZABETH	A										
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	1.9	14	2.6	14	9.5	10	12.9	49	6.5
PS 26	2	13	2.7	14	3.5	14	11.1	11	11.6	52	7.1

ARSENIC

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	SECOND YEAR OCT 1988 - SEPT 1989 ARITH. MEAN (ng/m3)
CARTERET	B										
ELIZABETH	A										
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	9	14.5	13	1.4	11	1.1	15	1.0	48	3.7
PS 26	2	10	11.4	9	5.1	11	1.4	14	1.0	44	4.3

Table IIIB-2

CADMIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	1.6	15	12.6	10	4.5	14	2.7	52	5.6
ELIZABETH	A					3	1.6	13	2.4	16	2.3
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	2.7	14	2.5	14	2.9	10	3.2	49	2.8
PS 26	2	13	3.0	14	3.0	14	3.2	11	2.2	52	2.9

CADMIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	9.3	15	1.8	15	1.9	15	4.1	60	4.3
ELIZABETH	A	15	0.8	15	1.5	13	1.8	11	2.7	54	1.6
HIGHLAND PARK	E			15	2.3	15	1.7	12	2.2	42	2.1
SUSAN WAGNER HS	1	9	2.5	13	2.3	11	2.5	15	2.5	48	2.4
PS 26	2	10	2.0	9	2.0	12	2.5	15	3.0	46	2.5

Table IIIB-3

CHROMIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	22.7	15	37.9	10	42.7	14	3.1	52	22.2
ELIZABETH	A					3	33.9	13	2.3	16	8.2
HIGHLAND PARK	E										

CHROMIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B			15	13.3	15	51.8	15	16.5	46	26.8
ELIZABETH	A	15	3.9	15	12.9	11	35.8	11	8.4	56	15.6
HIGHLAND PARK	E			15	9.5	12	20.5	12	5.2	42	12.3

Table IIIB-4

COBALT

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	FIRST YEAR OCT 1987 - SEPT 1988 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	11	2.7	14	2.5	14	2.9	10	2.6	49	2.7
PS 26	2	13	3.0	14	3.0	14	3.2	11	2.2	52	2.9

COBALT

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	SECOND YEAR OCT 1988 - SEPT 1989 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	9	2.5	13	2.5	11	2.5	15	2.5	48	2.5
PS 26	2	10	2.0	9	2.0	11	2.5	15	3.0	45	2.5

Table IIIB-5

COPPER

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	47.0	15	78.4	10	113.5	14	105.4	52	84.6
ELIZABETH	A					3	76.4	13	22.9	16	32.9
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	83.8	14	90.7	14	121.1	10	95.3	49	98.8
PS 26	2	13	59.2	14	45.9	14	50.5	11	75.4	52	56.7

COPPER

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B			15	83.7	15	69.1	15	88.1	45	80.3
ELIZABETH	A	15	26.6	15	42.8	13	25.6	11	44.3	54	34.5
HIGHLAND PARK	E			15	22.6	15	32.2	12	49.6	42	33.8
SUSAN WAGNER HS	1	9	79.4	13	74.1	11	86.3	15	91.3	48	83.3
PS 26	2	10	39.6	9	47.4	11	38.9	15	39.4	45	40.9

Table IIIB-6

IRON

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	519.7	15	752.6	10	805.4			38	686.8
ELIZABETH	A					3	661.0	13	495.6	16	526.6
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	511.9	14	522.8	14	807.8	10	888.0	49	676.3
PS 26	2	13	1168.0	14	875.7	14	1468.0	11	1433.0	52	1226.1

IRON

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	426.0	15	508.1	15	689.7	15	795.4	60	604.8
ELIZABETH	A	15	308.4	15	401.5	13	493.1	11	552.8	54	428.5
HIGHLAND PARK	E			15	376.4	13	656.7	12	406.3	40	476.5
SUSAN WAGNER HS	1	9	537.3	13	690.0	11	610.0	15	710.0	48	649.3
PS 26	2	10	738.0	9	970.0	11	800.0	15	1110.0	45	923.6

Table IIIB-7

LEAD

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	69.0	15	118.7	10	30.8			38	78.6
ELIZABETH	A					3	21.6	13	31.3	16	29.5
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	58.2	14	31.1	14	44.6	10	49.3	49	44.7
PS 26	2	13	82.5	14	46.4	14	44.8	11	55.3	52	56.9

LEAD

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	38.1	15	26.4	15	42.8	15	38.3	60	36.4
ELIZABETH	A	15	38.1	15	22.6	13	38.1	11	23.5	54	30.8
HIGHLAND PARK	E			15	91.1	13	26.6	12	20.5	40	49.0
SUSAN WAGNER HS	1	9	36.1	13	39.5	11	14.4	15	33.7	48	31.3
PS 26	2	9	35.7	9	40.1	11	24.5	15	45.7	44	37.2

Table IIIB-8

MANGANESE

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	29.5	15	27.0	10	32.2			38	29.2
ELIZABETH	A					3	23.1	13	17.9	16	18.9
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	15.1	14	15.1	14	21.8	10	22.1	49	18.5
PS 26	2	13	26.6	14	21.0	14	33.6	11	34.5	52	28.7

MANGANESE

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	18.9	15	20.3	15	25.7	15	22.1	60	21.8
ELIZABETH	A	15	14.9	15	15.6	13	15.0	11	13.4	54	14.8
HIGHLAND PARK	E			15	12.4	15	15.6	12	11.2	42	13.6
SUSAN WAGNER HS	1	9	13.9	13	18.6	11	12.2	15	15.2	48	15.2
PS 26	2	10	15.9	9	22.3	11	15.5	15	21.1	45	18.8

Table IIIB-9

MERCURY

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B			15	0.5	10	0.6			25	0.5
ELIZABETH	A					3	0.9	13	1.2	16	1.1
HIGHLAND PARK	E										
SUSAN WAGNER HS	1										
PS 26	2										

MERCURY

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	0.8	15	0.4	15	0.4	15	0.4	60	0.5
ELIZABETH	A	15	0.7	15	0.3	13	0.5	14	0.4	57	0.5
HIGHLAND PARK	E			15	0.5	15	0.5	15	0.3	45	0.4
SUSAN WAGNER HS	1										
PS 26	2										

Table IIIB-10

MOLYBDENUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	11	11.1	14	10.8	14	11.0	10	10.8	49	10.9
PS 26	2	13	12.4	14	12.3	14	12.9	11	8.9	52	11.8

MOLYBDENUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	9	10.7	13	9.2	11	9.5	15	9.5	48	9.6
PS 26	2	10	7.9	9	7.7	11	9.8	15	12.1	45	9.7

Table IIIB-11

NICKEL

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	FIRST YEAR OCT 1987 - SEPT 1988 ARITH. MEAN (ng/m3)
CARTERET	B	13	30.6	15	76.3	10	29.3			38	48.3
ELIZABETH	A					3	13.6	13	44.5	16	38.7
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	15.6	14	26.4	14	13.6	10	14.7	49	17.9
PS 26	2	13	29.4	14	27.7	14	16.6	11	16.6	52	22.8

NICKEL

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	SECOND YEAR OCT 1988 - SEPT 1989 ARITH. MEAN (ng/m3)
CARTERET	B			15	13.9	15	32.4	15	32.1	45	26.1
ELIZABETH	A			15	13.5	13	30.2	11	30.7	39	23.9
HIGHLAND PARK	E			15	17.3	13	29.0	12	22.2	40	22.6
SUSAN WAGNER HS	1	9	13.4	13	32.2	11	12.3	15	16.1	48	19.1
PS 26	2	10	15.1	9	33.7	11	18.0	15	17.1	45	20.2

Table IIIB-12

VANADIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	FIRST YEAR OCT 1987 - SEPT 1988 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	11	12.8	14	19.4	14	11.6	10	9.3	49	13.6
PS 26	2	13	14.8	14	23.9	14	15.5	11	14.9	52	17.5

VANADIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	SECOND YEAR OCT 1988 - SEPT 1989 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	9	9.3	13	22.5	11	10.1	15	16.2	48	15.2
PS 26	2	10	15.2	9	19.8	11	16.0	15	17.0	45	16.9

Table IIIB-13

ZINC

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	182.0	15	89.8	10	90.3			38	121.5
ELIZABETH	A					3	78.4	13	83.8	16	82.8
HIGHLAND PARK	E										
SUSAN WAGNER HS	1	11	112.4	14	89.5	14	95.1	10	176.1	49	113.9
PS 26	2	13	145.5	14	107.8	14	106.8	11	160.8	52	128.2

ZINC

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	93.0	15	101.5	15	93.2	15	173.1	60	115.2
ELIZABETH	A	15	117.9	15	88.1	13	105.9	11	131.3	54	109.5
HIGHLAND PARK	E			15	77.8	15	134.9	12	76.5	42	97.8
SUSAN WAGNER HS	1	9	88.4	13	117.5	11	54.2	10	194.7	43	113.2
PS 26	2	10	83.8	9	120.2	11	70.4	15	109.3	45	96.3

Table IIIB-14

BENZO(A)PYRENE

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	13	0.36	14	0.15	10	0.07	14	0.09	51	0.17
ELIZABETH	A					3	0.06	13	0.03	16	0.04
HIGHLAND PARK	E										

BENZO(A)PYRENE

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	15	0.24	15	0.35	15	0.07	15	0.15	60	0.20
ELIZABETH	A	15	0.22	15	0.29	14	0.11	11	0.12	55	0.19
HIGHLAND PARK	E	10	0.31	15	0.15	15	0.06	13	0.07	53	0.14

Table IIIB-15

FORMALDEHYDE - HCHO (METHANAL)

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	ARITH. MEAN (ppb)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	ARITH. MEAN (ppb)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	ARITH. MEAN (ppb)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	ARITH. MEAN (ppb)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	ARITH. MEAN (ppb)
CARTERET	B	8	2.91	10	3.38			7	4.81	25	3.63
ELIZABETH	A										
PISCATAWAY	D							10	3.30	10	3.30
SUSAN WAGNER HS	1							1	4.05	1	4.05
PORT RICHMOND PO	5										

FORMALDEHYDE - HCHO (METHANAL)

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	ARITH. MEAN (ppb)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	ARITH. MEAN (ppb)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	ARITH. MEAN (ppb)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	ARITH. MEAN (ppb)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	ARITH. MEAN (ppb)
CARTERET	B										
ELIZABETH	A			6	2.89					6	2.89
PISCATAWAY	D	7	1.78							7	1.78
SUSAN WAGNER HS	1	8	1.62	14	1.98	10	2.13	13	2.06	44	2.02
PORT RICHMOND PO	5	9	2.03	14	1.80	6	1.69	6	1.04	35	1.71

Table IIIB-16

BARIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1987 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1988 # OF SAMPLES	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1988 # OF SAMPLES	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1988 # OF SAMPLES	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)	FIRST YEAR OCT 1987 - SEPT 1988 # OF SAMPLES	FIRST YEAR OCT 1987 - SEPT 1988 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	11	15.8	14	14.4	14	13.2	10	17.8	49	15.1
PS 26	2	13	28.4	14	21.7	14	24.2	11	27.5	52	25.3

BARIUM

SITE	SITE #	QUARTER BEGINNING OCTOBER 1988 # OF SAMPLES	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JANUARY 1989 # OF SAMPLES	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING APRIL 1989 # OF SAMPLES	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	QUARTER BEGINNING JULY 1989 # OF SAMPLES	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)	SECOND YEAR OCT 1988 - SEPT 1989 # OF SAMPLES	SECOND YEAR OCT 1988 - SEPT 1989 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	9	14.5	13	22.5	11	13.4	18	4.9	51	12.9
PS 26	2	10	22.2	9	29.8	11	16.8	15	27.8	45	23.5

Table IIIB-17a: Comparison of 1988 UATMP and 1989 SI/NJ UATAP metals and benzo[a]pyrene

	Concentrations, ng/m³							
	Range for SI/NJ UATAP annual avgs., 10/88-10/89 (excluding Highland Park) (Ref: Data summaries of 1/90/92.)				SI/NJ UATAP for Highland Park, 1/89-10/89	Range for 1988 UATMP annual averages for 17 urban areas		
	min.	max.	median	(# of sites)	annual avg.	min.	max.	median
Lead	14.4▲ 31.3	45.7▲ 37.2	34.7▲ 33.9	(4)	min. 20.5/max. 91.1▲ 47.8	10*	440*	40*
Chromium	15.6	26.8	21.2	(2)**	12.3**	1.5	25.0	5.6
Nickel	19.1	28.2	21.9	(4)***	22.4	2.8	34.0	3.8
Arsenic	3.7	4.3	4.0	(2)	-	2.8	8.4	3.3
Cadmium	1.8	4.2	2.1	(4)	2.1	0.5	13.3	0.9
Mercury	0.5	0.5	0.5	(2)	0.4	-	-	-
Manganese	14.8	21.6	15.0	(4)	13.3	20.4	491.7	28.8
Cobalt	2.5	2.5	2.5	(2)	-	0.9	1.5	0.9
Copper	36.0	83.3	60.1	(4)	34.5	2.0	1913.0	77.5
Iron	445.6	923.6	624.1	(4)	476.3	554.9	9154.0	1182.0
Molybdenum	9.6	9.7	9.6	(2)	-	1.2	3.6	1.2
Vanadium	15.2	16.9	16.0	(2)	-	4.9	14.3	5.2
Zinc	96.3	116.1	113.6	(4)	97.8	24.8	1084.0	89.1
Benzo[a]pyrene	0.15	0.21	0.17	(4)	0.14	0.032	5.212	0.183

▲ Quarterly averages, not annual averages.
* Not annual averages; based on 50 quarterly averages.
** Annual average was computed without having data for 10/88 to 1/89.
*** The annual averages for two of the four sites were computed without having data for 10/88 to 1/89.

Table IIIB-17b: Detection limits for the metals and benzo[a]pyrene in the 1988 UATMP and the SI/NJ UATAP

Chemical	Minimum detection limit, ng/m3		
	1988 UATMP	SI/NJ UATAP	
		NYSDOH	NJIT
Lead	-	-	-
Chromium, total	10	-	10
Nickel	5.6	5	7.5
Arsenic	5.5	30, 2*	-
Cadmium	1.1	5	2.5
Mercury	-	-	0.01
Manganese	40.8	5	5
Cobalt	1.8	5▲	-
Copper	3.2	5	7.5
Iron	20.3	11	10
Molybdenum	2.3	24	-
Vanadium	9.9	5	-
Zinc	13.3	10	3.5
Benzo[a]pyrene	0.2	-	-

- Not available.
* The minimum detection limit changed in mid-1988. Ref: Personal communication of S. Koblenz, NYSDOH to C. Bellizzi, U.S. EPA Region II, ca. February 1992.
▲ The minimum detection limit was not constant; sometimes it was lower than 5 ng/m^3.

Table IIIB-17c: Metals and benzo[α]pyrene data from 1988 UATMP*

	(18) Cleveland, OH	(20) Sauget, IL	(20) Lansing, MI	(19) Midland, MI	(20) Port Huron, MI	(19) Detroit, MI	(9) Dearborn, MI
Lead, quarterly avgs., ng/m3							
1st qtr	150	100	30	20	40	60	70 (110)***
2nd qtr	410	270	20	10	30	70	80 (110)***
3rd qtr	440	270	20	20	40	80	130 (100)***
4th qtr	-	-	-	-	-	-	- (90)***
Annual avg., ng/m3**							
Chromium	25.0	9.8	5.2 (1.5)***	5.2	5.2	7.7	23.9
Nickel	8.8	3.5	3.5	2.8	4.2	4.1	8.4
Arsenic	7.0	8.4	2.9	2.8	2.8	3.3	5.6
Cadmium	10.0	13.3	0.5	0.7	0.7	3.4	4.7
Mercury	-	-	-	-	-	-	- (0.3)***
Manganese	194.6	49.2	23.8	23.1	21.5	79.2	491.7
Cobalt	1.3	0.9	1.1	0.9	0.9	1.5	1.0
Copper	102.5	520.9	49.3	10.0	20.0	50.3	77.5
Iron	4279.0	1182.0	824.1 (431.8)***	554.9	609.5 (343.7)***	2047.0	9154.0
Molybdenum	3.6	1.6	1.2	1.2	1.2	1.3	3.1
Vanadium	7.0	4.9	5.2	4.9	10.8	5.2	9.5
Zinc	459.8	506.1	61.0 (81.4)***	33.8	74.5	512.6	1084.0
Benzo[α]pyrene	5.212	0.183	0.434	0.171	0.100	0.319	0.374

* AIRS database print-out of 8/22/90. Data coded as 1988, organization code 800 (USEPA Monitoring Support Lab). Hammond, IN, and Portland, OR, data were not available.

** Arithmetic mean

*** As measured by another organization.

Example of how to read this table:

	(18)	<--------Total number of valid samples
	Cleveland, OH	<---Monitor site name
Chromium	25.0	<--------Annual average concentration of chromium
Nickel	8.8	
Arsenic	7.0	
Cadmium	10.0	
Mercury	-	<-------- Chemical was not included at this site.

Table IIIB-17c, continued: Metals and benzo[α]pyrene data from 1988 UATMP*, continued

	(16) Jacksonville, FL	(21) Miami, FL	(21) Houston, TX	(22) Dallas, TX	(23) Atlanta, GA	(23) Burlington, VT	(11) Chicago, IL
Lead, quarterly avgs., ng/m3							
1st qtr	40	40	20 (40)***	30	50	40	80
2nd qtr	30	40	20 (70)***	20	50	30	-
3rd qtr	30	20	20 (30)***	20	30	30	40
4th qtr	-	-	- (40)***	-	-	-	-
Annual avg., ng/m3**							
Chromium	6.0	5.6	5.6	5.2	5.5	5.5	12.6
Nickel	6.1	4.7	6.7	2.8	2.8	3.8	2.8 (13.1)***
Arsenic	4.3	3.1	2.8	2.8	3.5	3.2	3.3
Cadmium	0.7	0.5	0.6	0.8	1.4	0.7	1.1
Mercury	-	-	-	-	-	-	-
Manganese	20.4	20.4	25.8	20.4	28.8	30.4	87.2
Cobalt	1.1	0.9	0.9	0.9	0.9	0.9	0.9
Copper	50.1	49.3	80.0	51.4	65.0	253.9	114.7
Iron	721.7	556.0	685.3	681.4	1296.0	1285.0	1588.0 (862.0)***
Molybdenum	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Vanadium	14.3	5.3	6.7	4.9	4.9	6.7	4.9
Zinc	88.2	89.1	66.2	24.8	96.9	73.1	148.2
Benzo[α]pyrene	0.222	0.100	0.117	0.100	0.229	0.147	0.357

* AIRS database print-out of 8/22/90. Data coded as 1988, organization code 800 (USEPA Monitoring Support Lab). Hammond,IN, and Portland, OR, data were not available.
** Arithmetic mean
*** As measured by another organization.

Table IIIB-17c, continued: Metals and benzo[α]pyrene data from 1988 UATMP*, continued

	(21) Birmingham, AL	(22) Louisville, KY	(21) Baton Rouge, LA
Lead, quarterly avgs., ng/m3			
1st qtr	80	80	20
2nd qtr	50	90	20
3rd qtr	30	60	10
4th qtr	-	-	-
Annual avg., ng/m3**			
Chromium	6.1	9.8	5.2
Nickel	2.8	34.0	3.5
Arsenic	3.6	4.5	3.1
Cadmium	0.9	1.9	1.0
Mercury	-	-	-
Manganese	34.2	64.5	21.4
Cobalt	0.9	1.5	0.9
Copper	1913.0	102.2	107.0
Iron	1078.0	1595.0	603.7
Molybdenum	1.5	2.4	1.2
Vanadium	4.9	4.9	8.8
Zinc	244.2	171.5	47.3
Benzo[α]pyrene	1.448	0.332	0.173

* AIRS database print-out of 8/22/90. Data coded as 1988, organization code 800 (USEPA Monitoring Support Lab). Hammond,IN, and Portland, OR, data were not available.
** Arithmetic mean
*** As measured by another organization.

Table IIIB-18a: Comparison of 1989 UATMP and SI/NJ UATAP formaldehyde data*

	Range for SI/NJ UATAP annual avgs., 10/88-10/89 (S.W. and Port Rich) (Ref: Data summaries of 1/90/92.)				SI/NJ UATAP for Piscataway, 1/89-10/89	Range for 1989 UATMP annual averages for 14 urban areas		
	min.	max.	median	(# of sites)	annual avg.	min.	max.	median
Formaldehyde, ppbv	1.71	2.02	1.86	(2)	- **	1.41	3.81	2.04

Table IIIB-17b: Formaldehyde data from the 1989 UATMP*

	(32) Camden, NJ	(30) Sauget, IL	(28) Washington,D.C.-1	(30) Washington,D.C.-2	(31) St. Louis, MO	(7) Pensacola, FL	(32) Miami, FL
Formaldehyde, ppbv	2.419	1.45	3.768	3.148	2.465	1.674	1.763

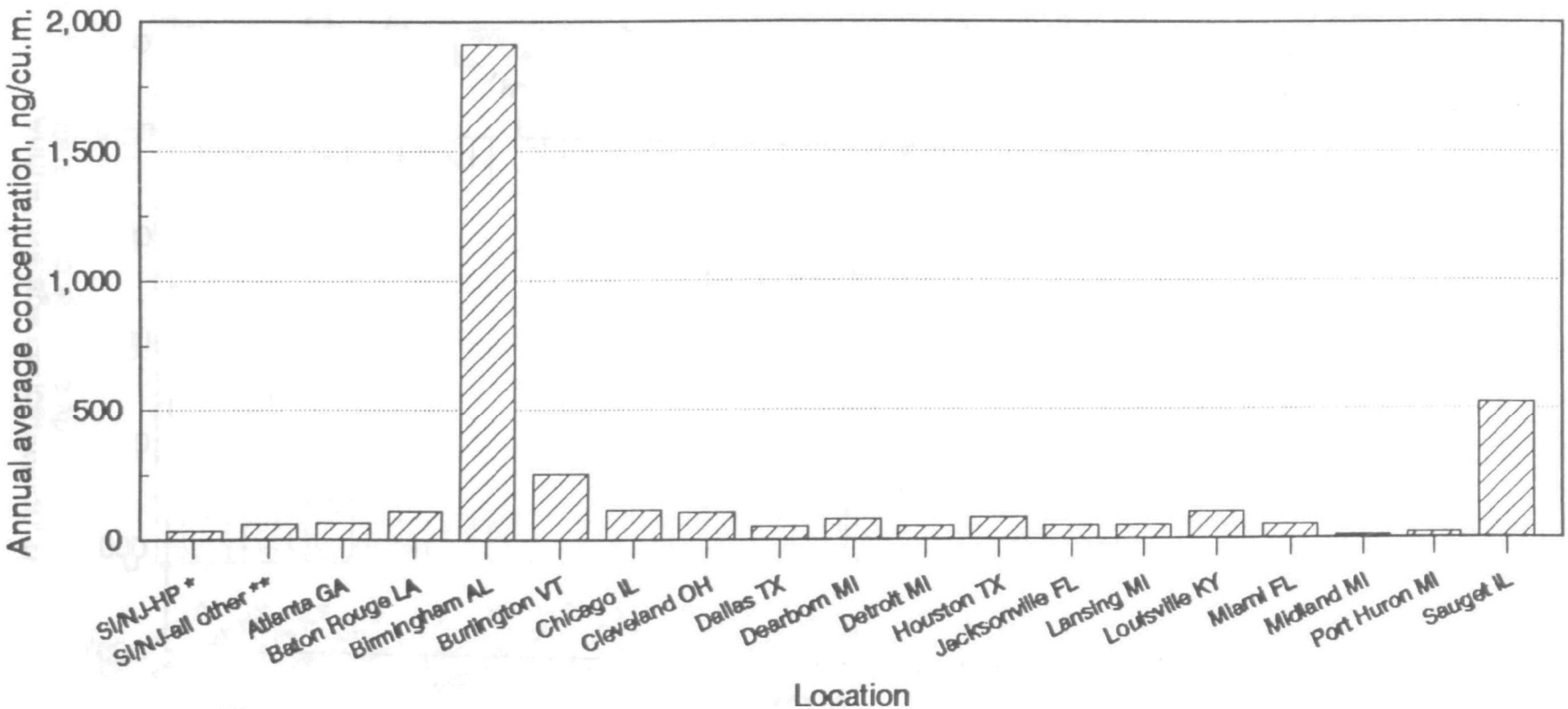
	(38) Houston, TX	(29) Dallas, TX	(32) Ft. Lauderdale, FL	(30) Chicago, IL	(30) Wichita, KS-1	(36) Wichita, KS-2	(31) Baton Rouge, LA
Formaldehyde, ppbv	2.319	2.010	2.243	2.073	1.471	1.409	1.525

* From EPA-450/4-91-006, January 1991, 1989 Urban Air Toxics Monitoring Program Aldehyde Results. U.S. EPA/Office of Air Quality Planning and Standards. Research Triangle Park.
Both the UATMP and SI/NJ UATAP acknowledge an ozone interference with the sampling method.
The ozone interference results in the reporting of concentrations as lower than actual.
** Insufficient data; the average for seven samples during 10/88 to 1/89 was 1.78 ppbv.

FIGURE IIIB-1

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

COPPER



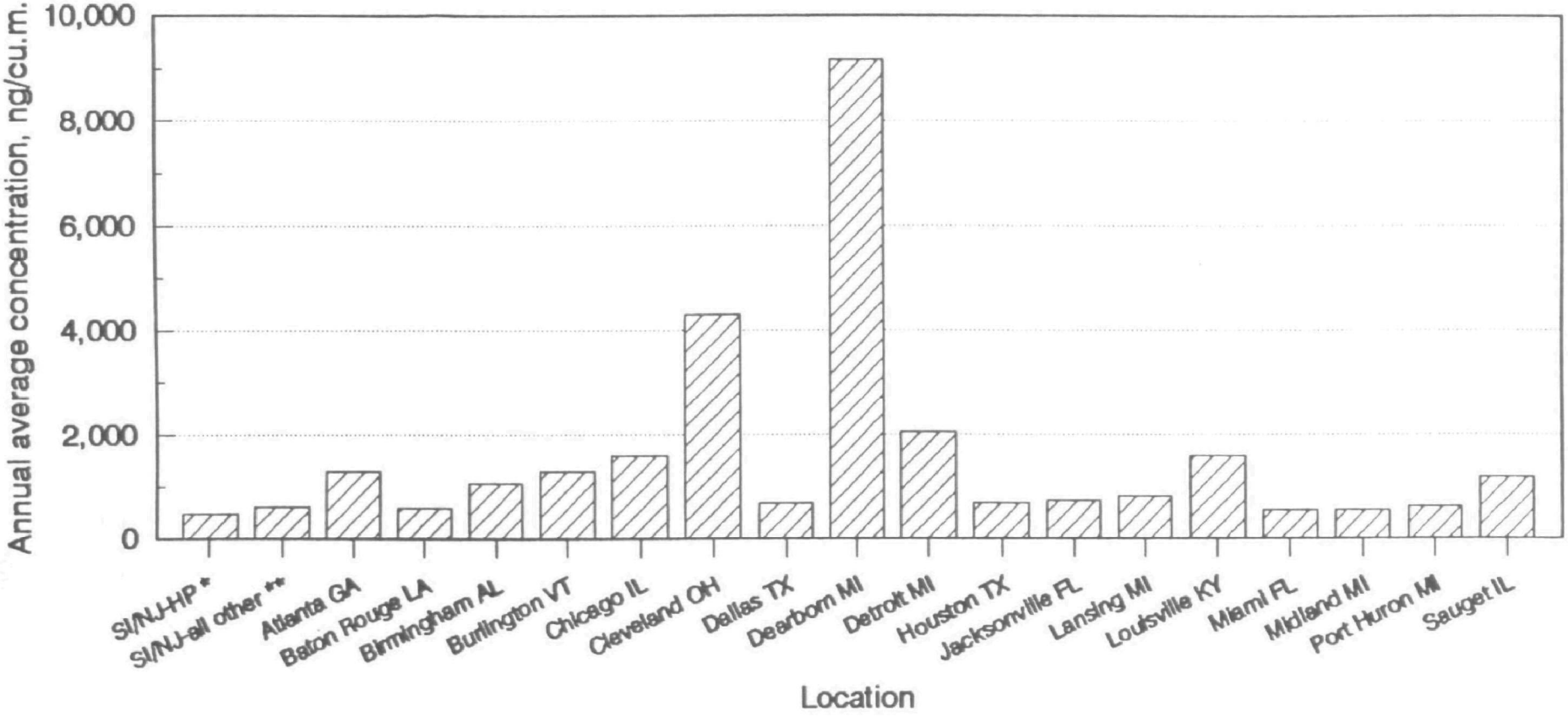
* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26, and Susan Wagner H.S.

FIGURE IIIB-2

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

IRON

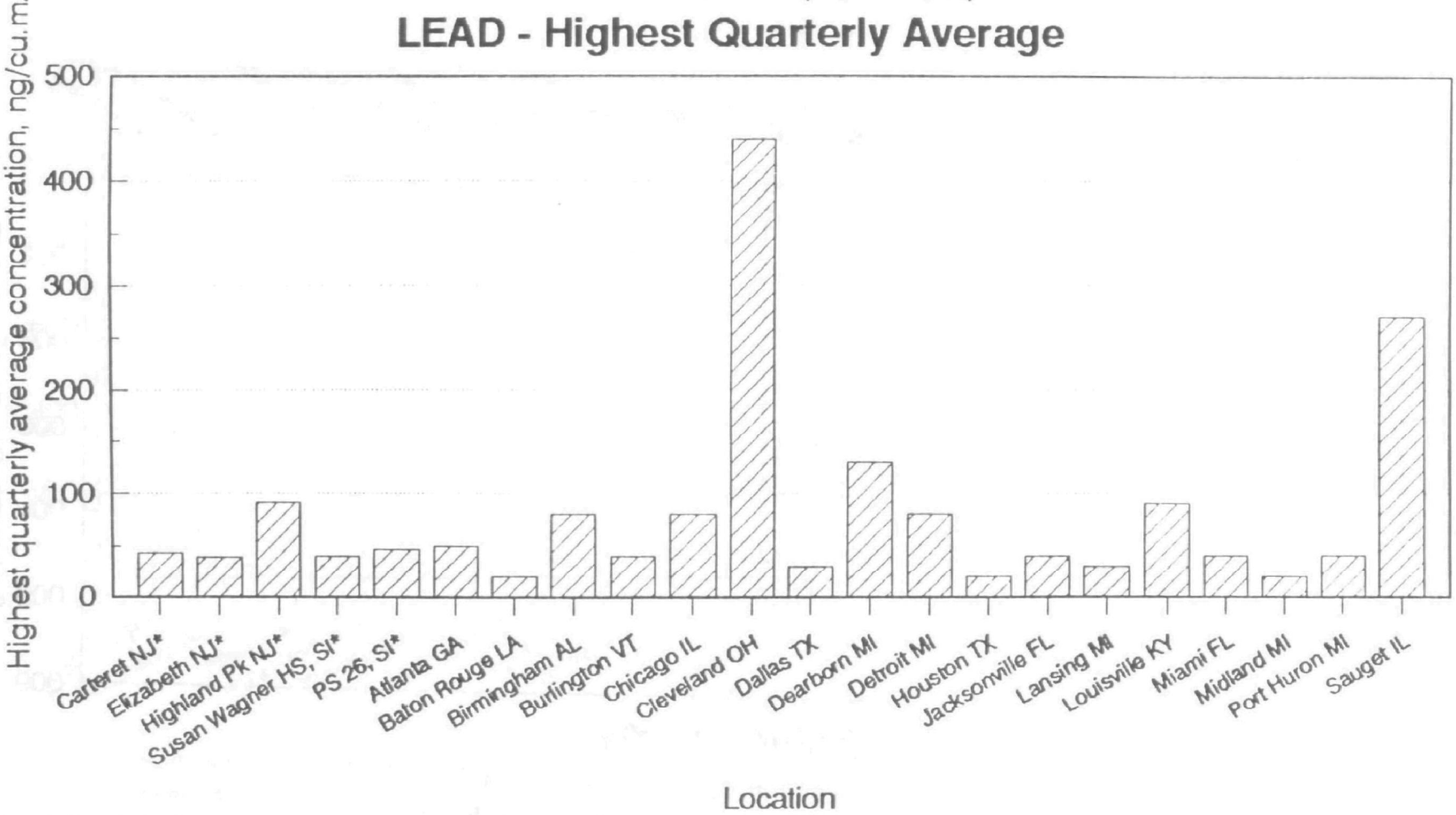


* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26, and Susan Wagner H.S.

FIGURE IIIB-3

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

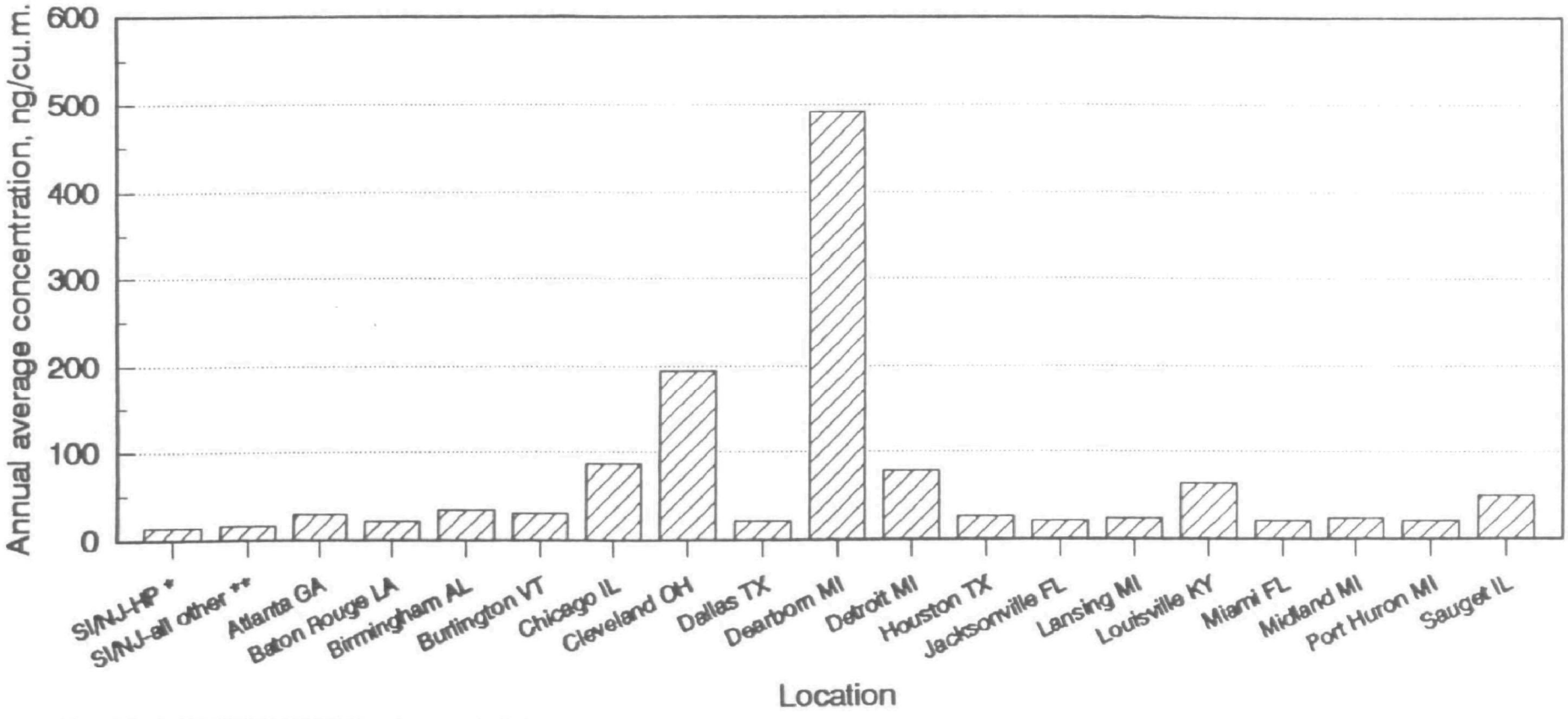


* SI/NJ UATAP site

FIGURE IIIB-4

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

MANGANESE

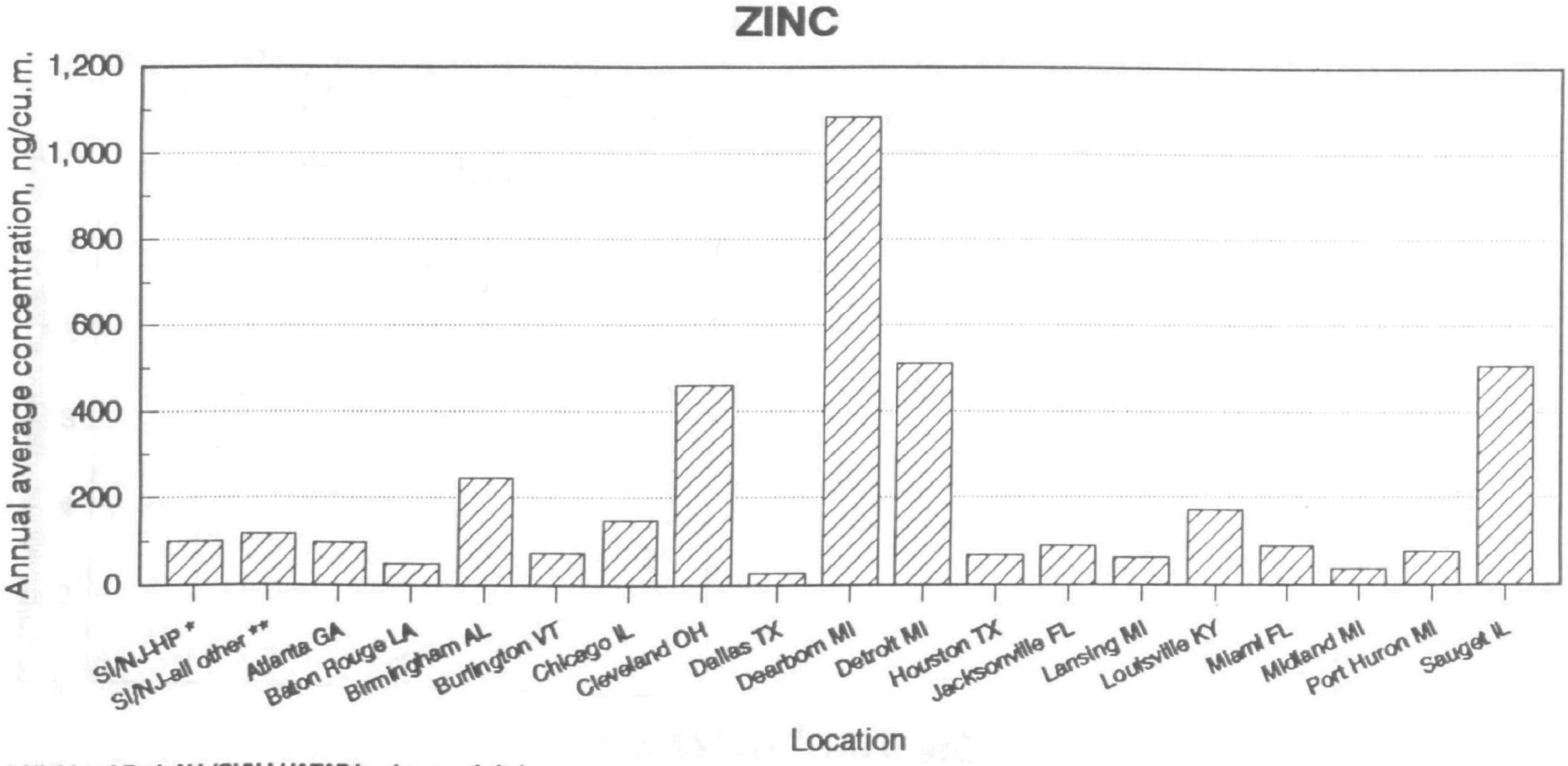


* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26, and Susan Wagner H.S.

FIGURE IIIB-5

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)



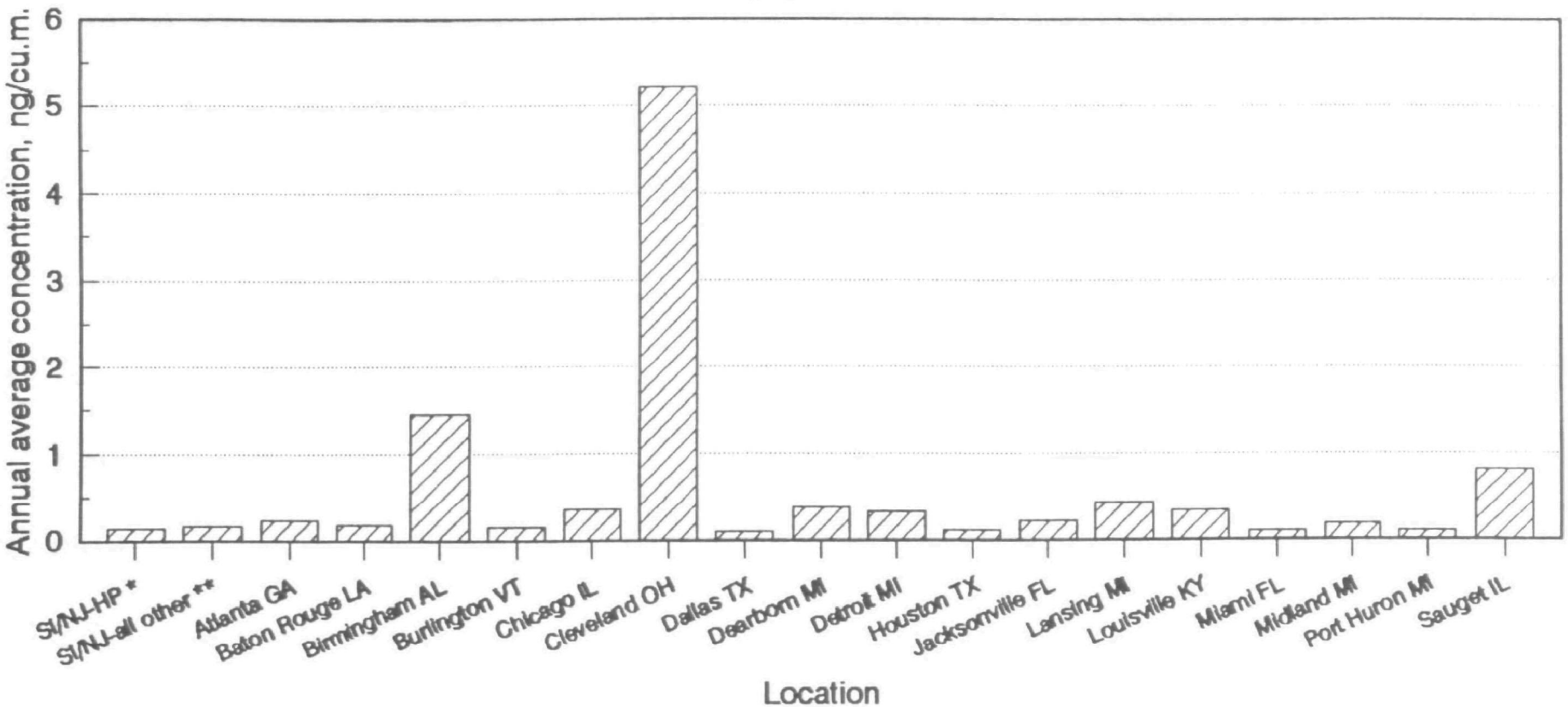
* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26, and Susan Wagner H.S.

FIGURE IIIB-6

Comparison of SI/NJ UATAP Data (10/88-10/89)
with1988 UATMP Data (10/87-10/88)

BENZO(A)PYRENE



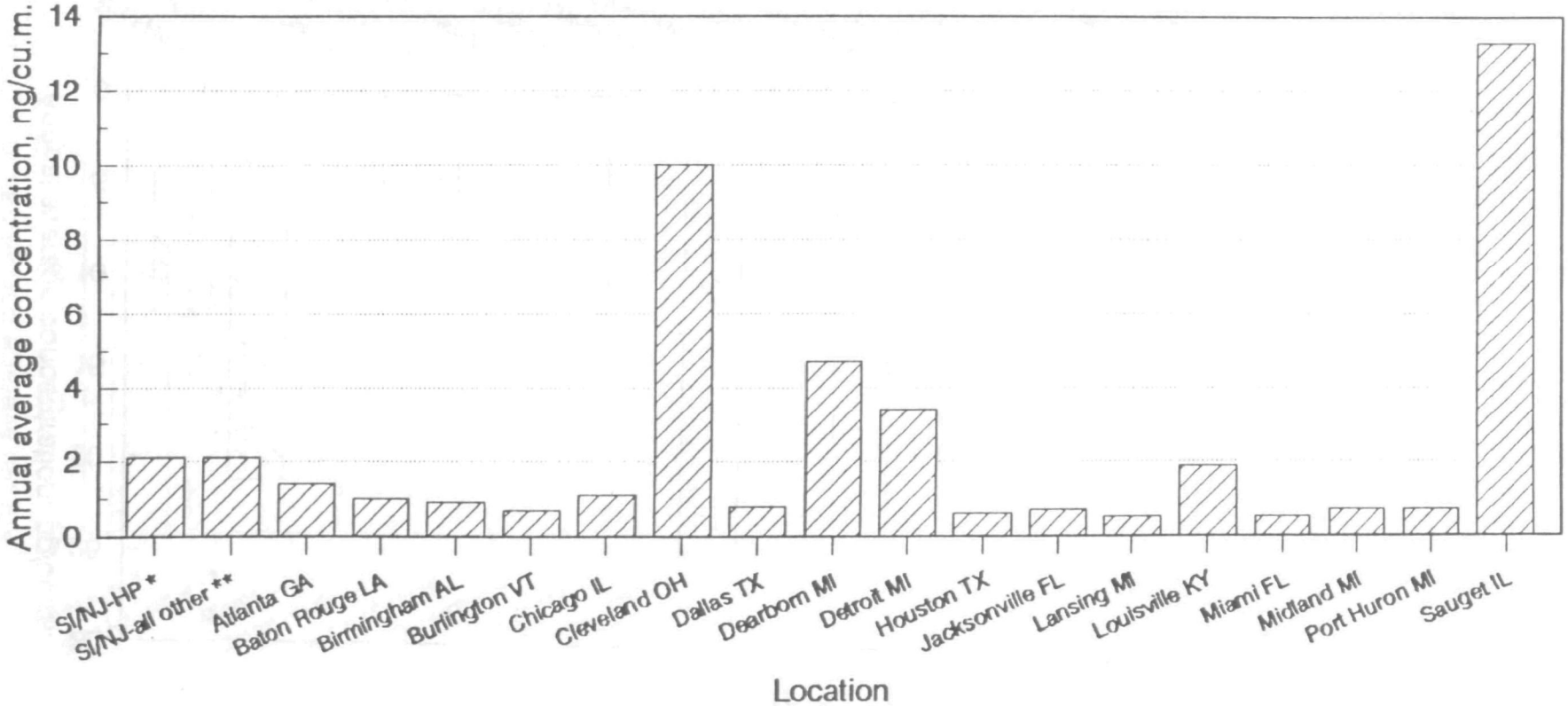
* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26, and Susan Wagner H.S.

FIGURE IIIB-7

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

CADMIUM



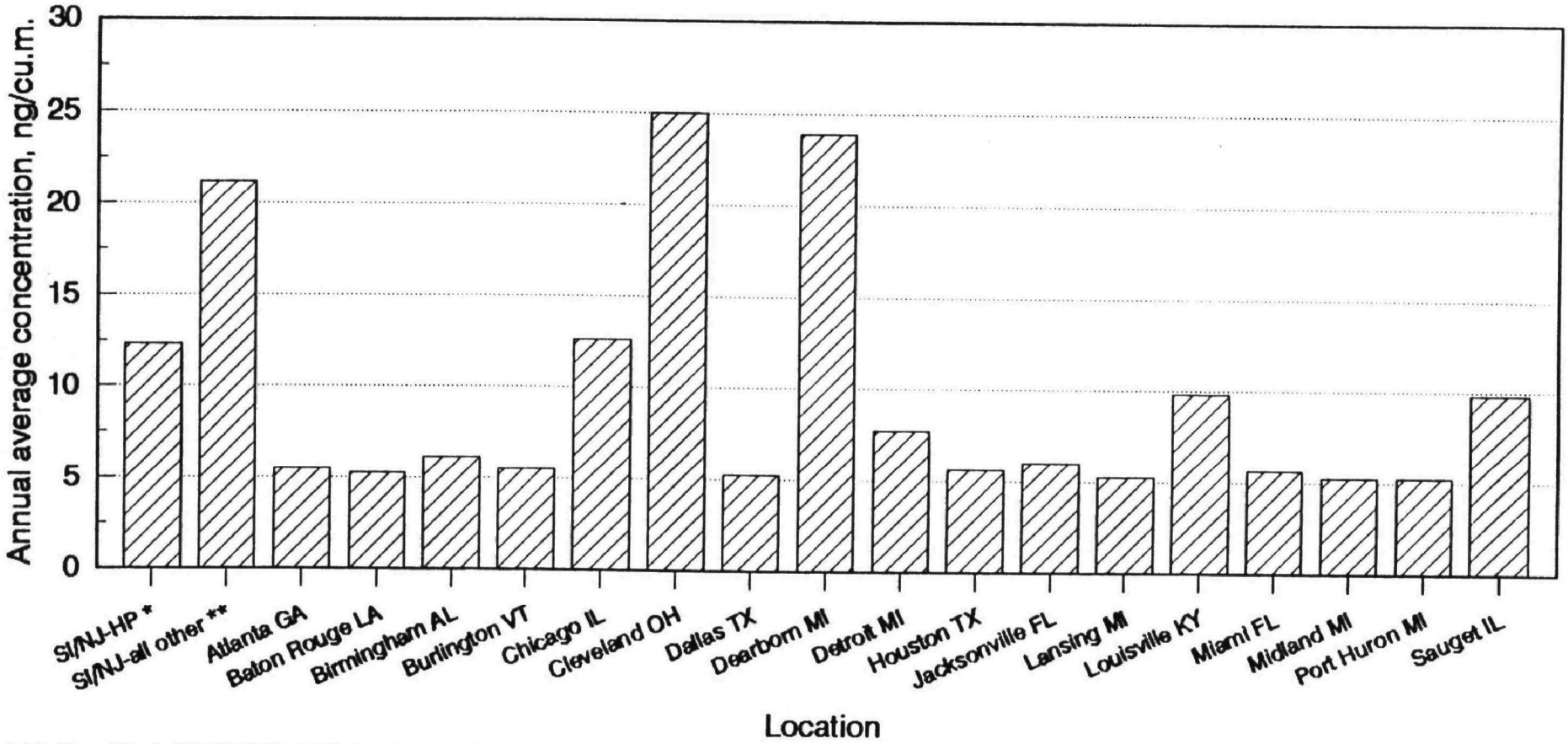
* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26 sites, and Susan Wagner H.S.

FIGURE IIIB-8

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

CHROMIUM



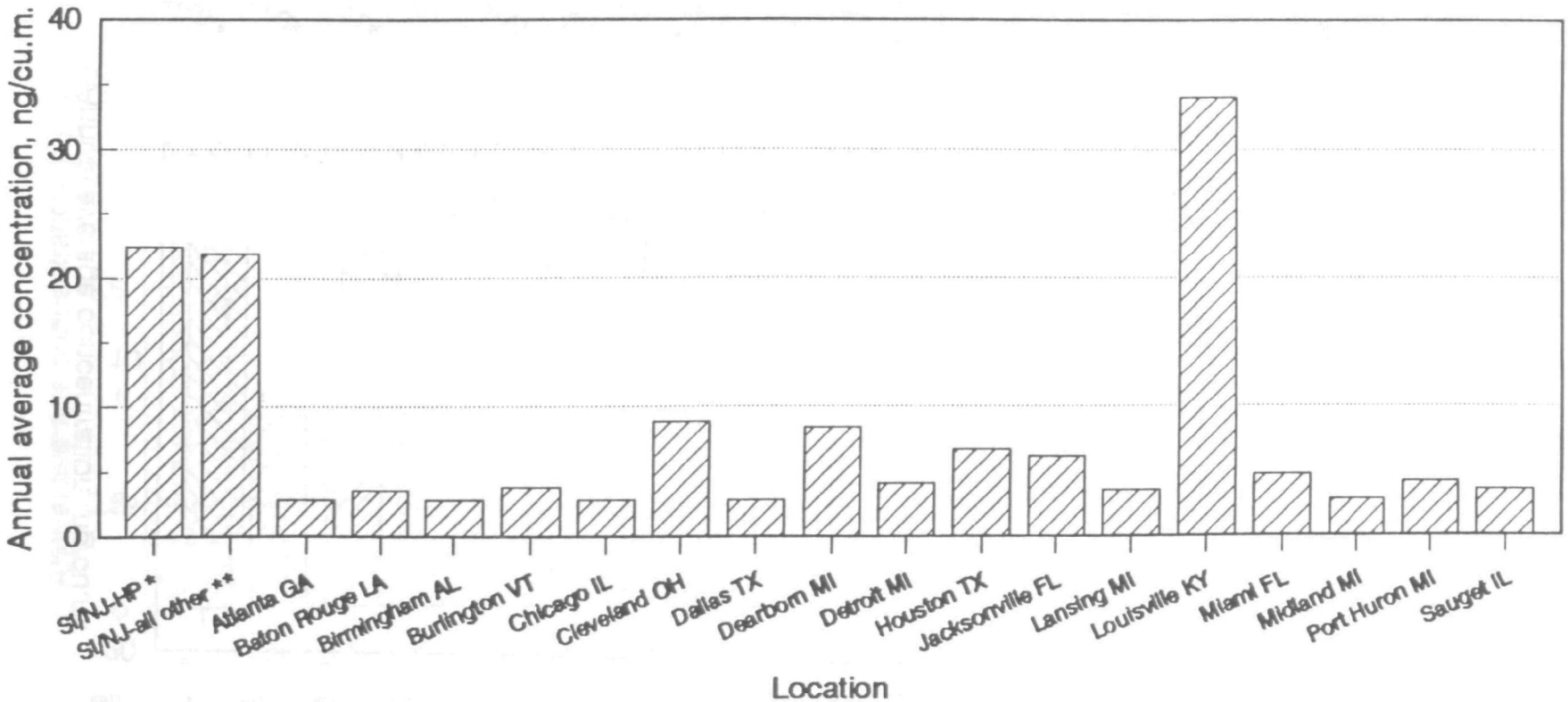
* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret and Elizabeth

FIGURE IIIB-9

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

NICKEL



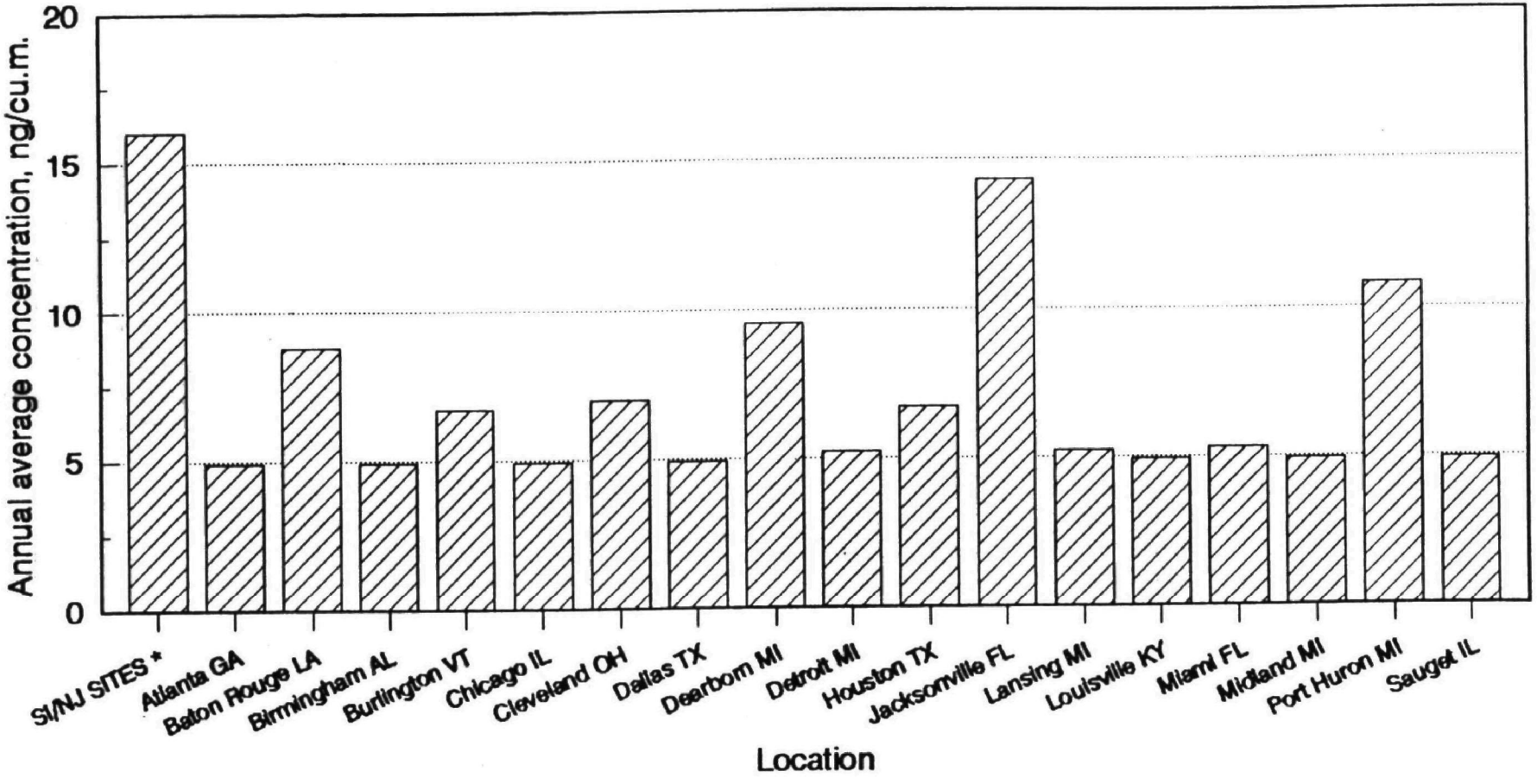
* Highland Park NJ (SI/NJ UATAP background site)

** Median for Carteret, Elizabeth, PS-26, and Susan Wagner H.S.

FIGURE IIIB-10

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

VANADIUM

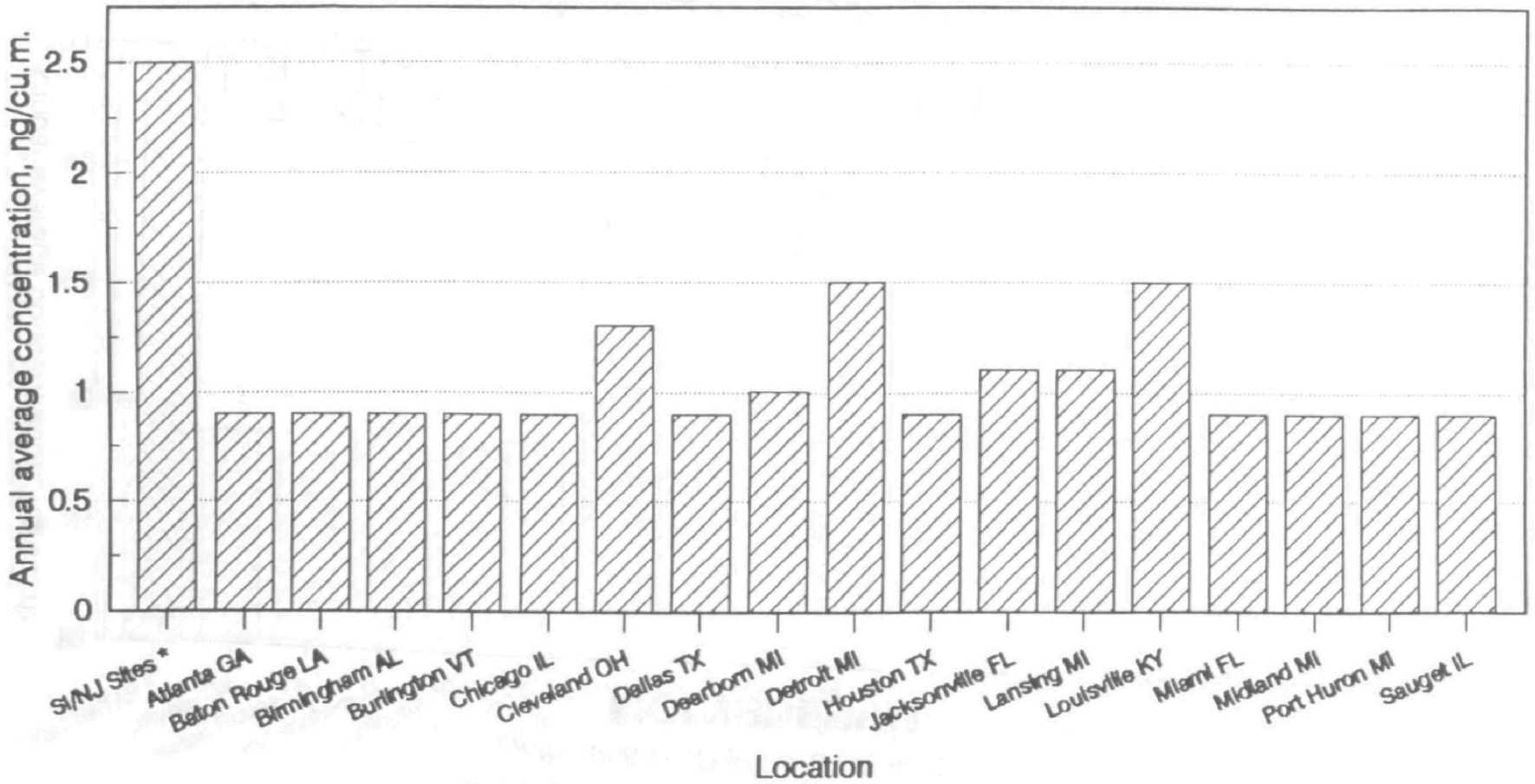


* Median for Susan Wagner H.S. and PS-26

FIGURE IIIB-11

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

COBALT

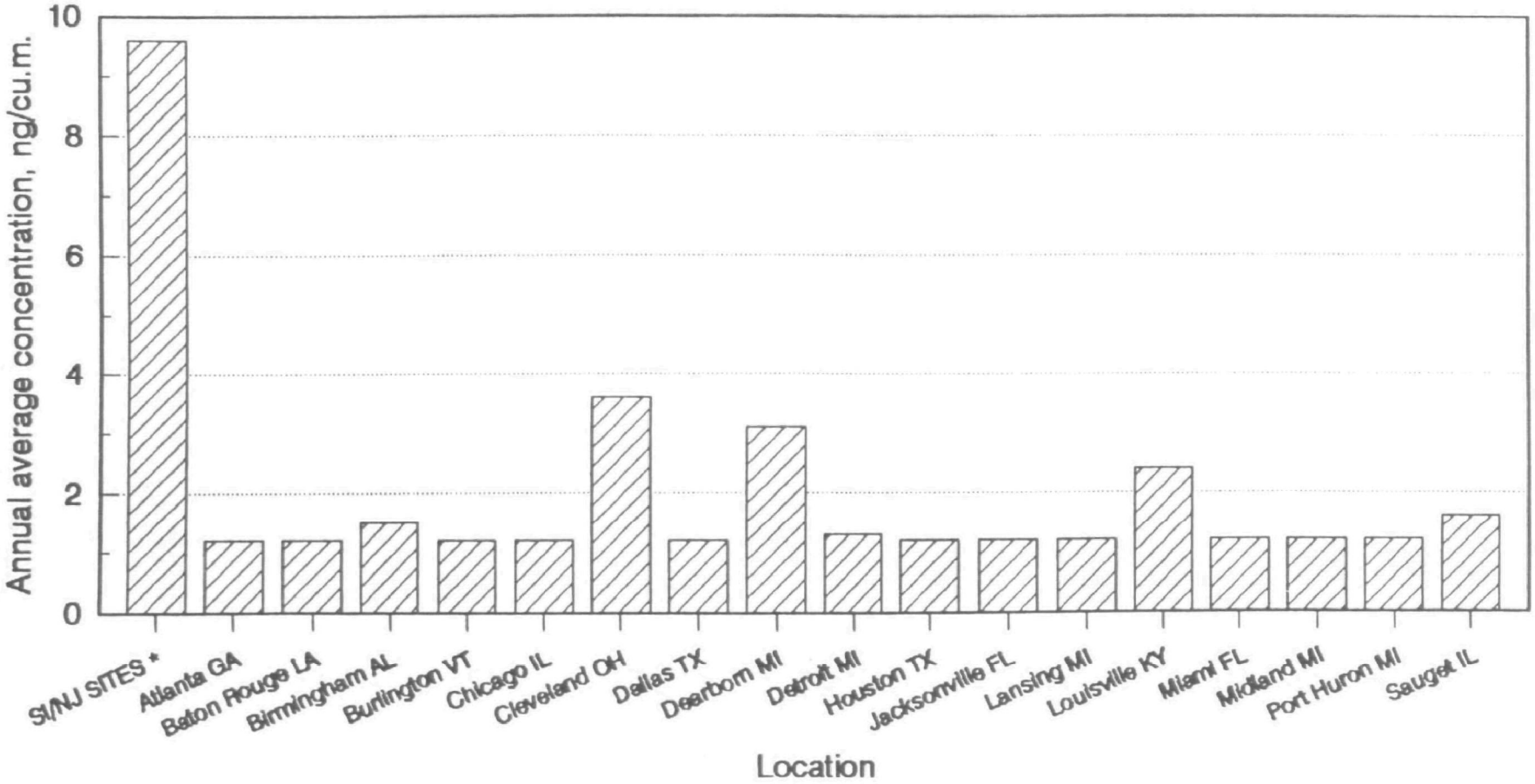


* Median for Susan Wagner H.S. and PS-26

FIGURE IIIB-12

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

MOLYBDENUM

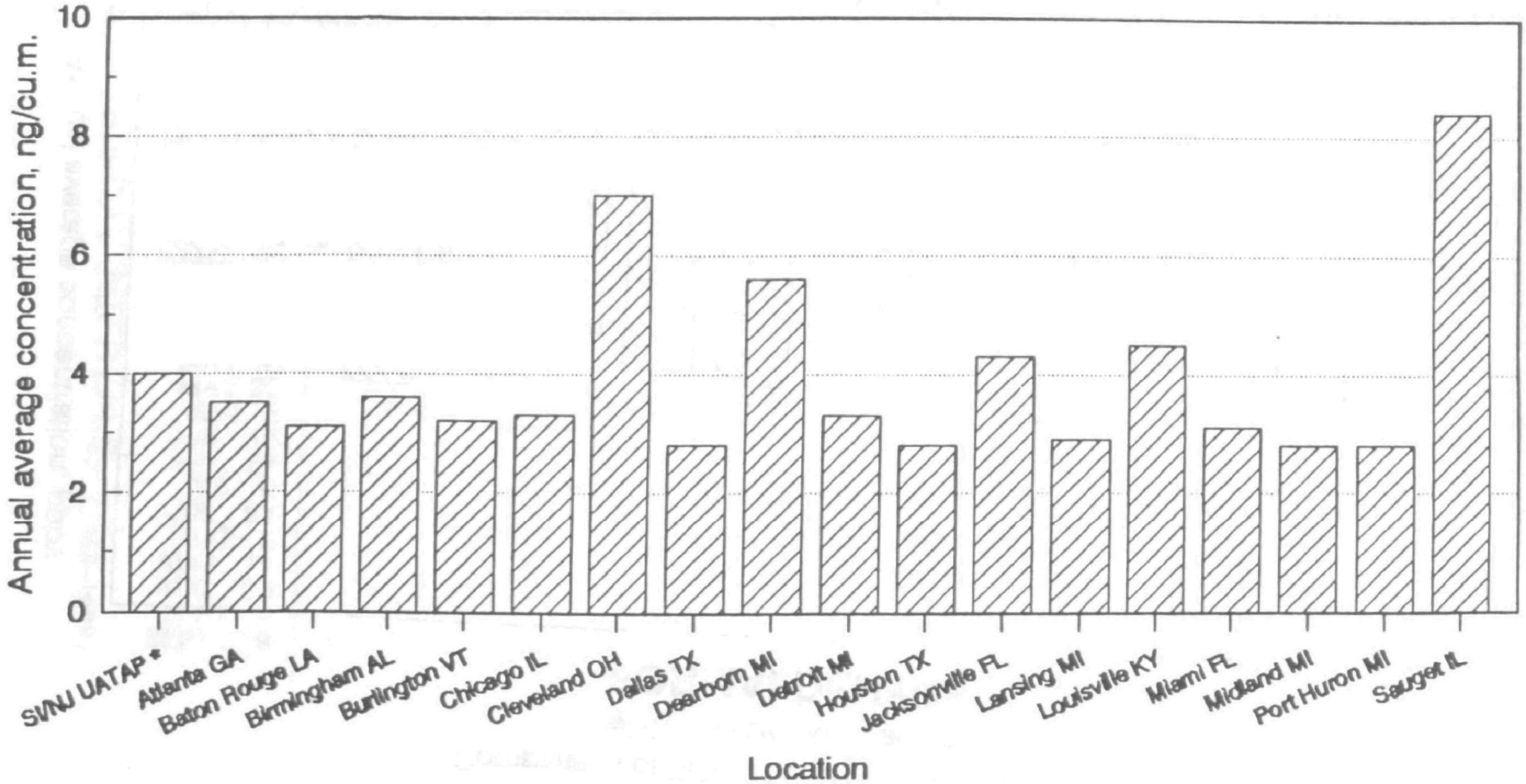


* Median for Susan Wagner H.S. and PS-26

FIGURE IIIB-13

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1988 UATMP Data (10/87-10/88)

ARSENIC

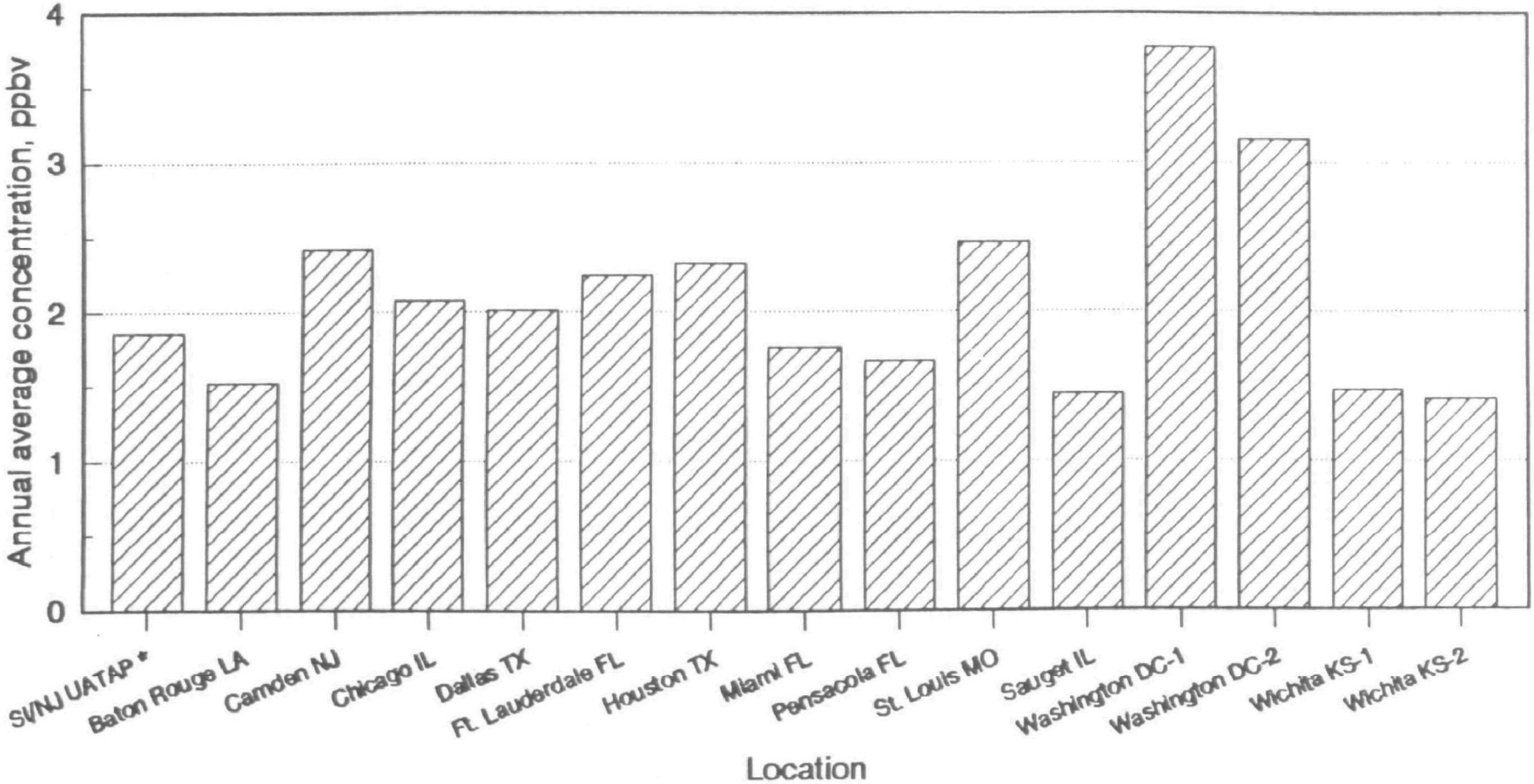


* Median for Susan Wagner H.S. and PS-26

FIGURE IIIB-14

Comparison of SI/NJ UATAP Data (10/88-10/89)
with 1989 UATMP Data for

FORMALDEHYDE



* Median for Susan Wagner H.S. and Port Richmond

Figure IIIB-15

Nickel, Chromium, Manganese, and Iron at Carteret, NJ

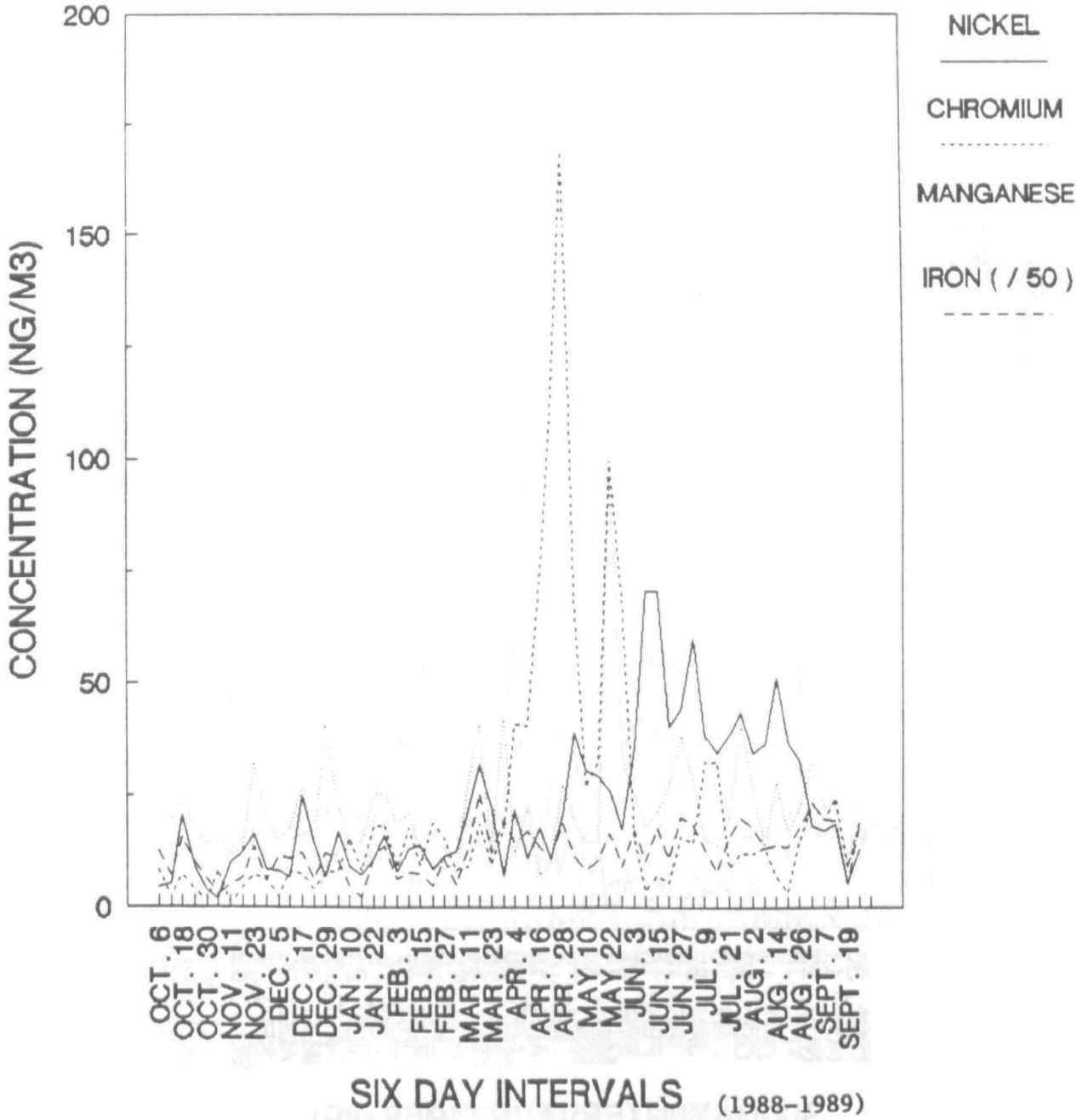


Figure IIIB-16

Cadmium, Copper, Zinc, and Lead at Carteret, NJ

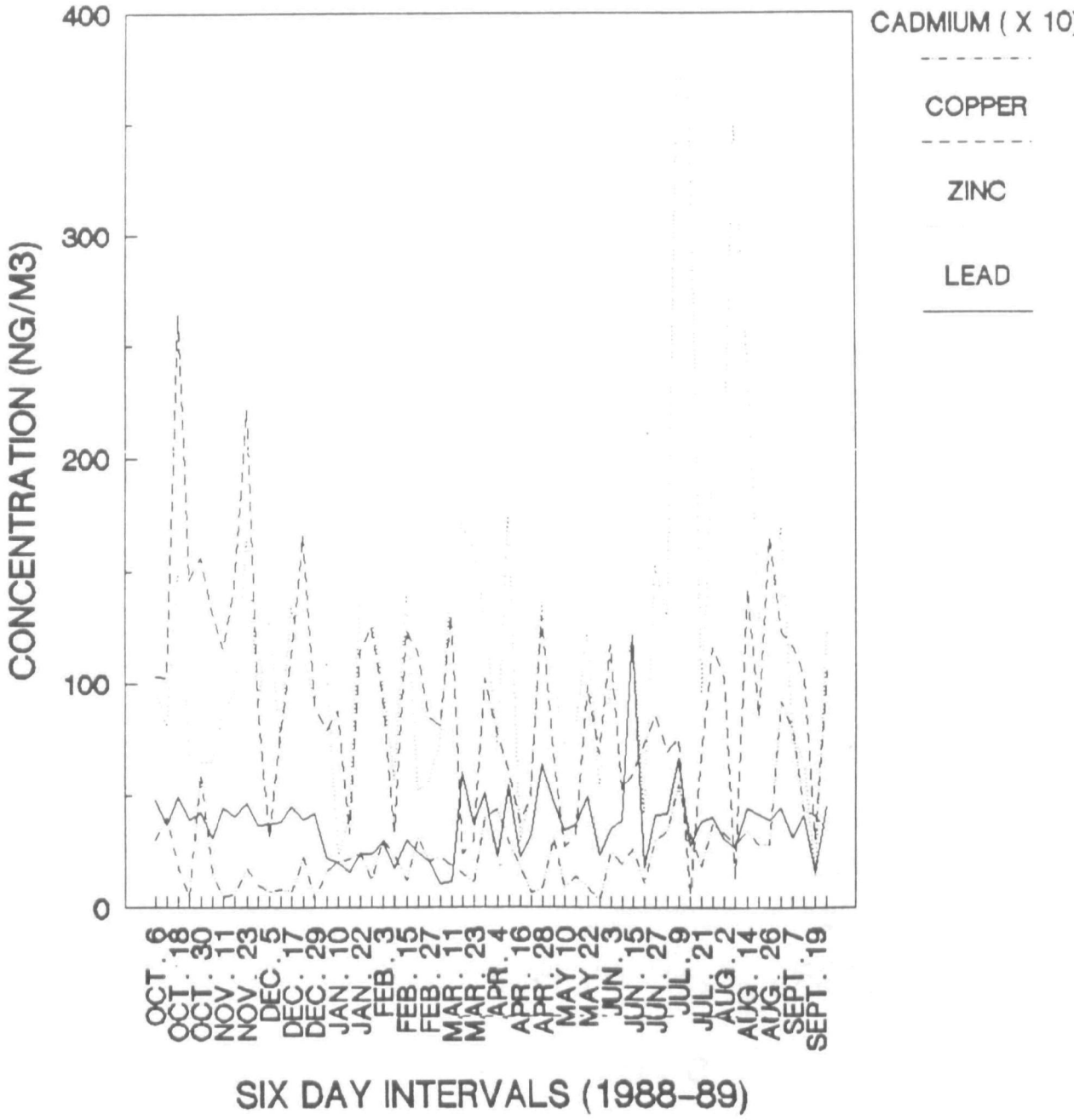


Figure IIIB-17

Benzo[α]pyrene and Lead at Carteret, NJ

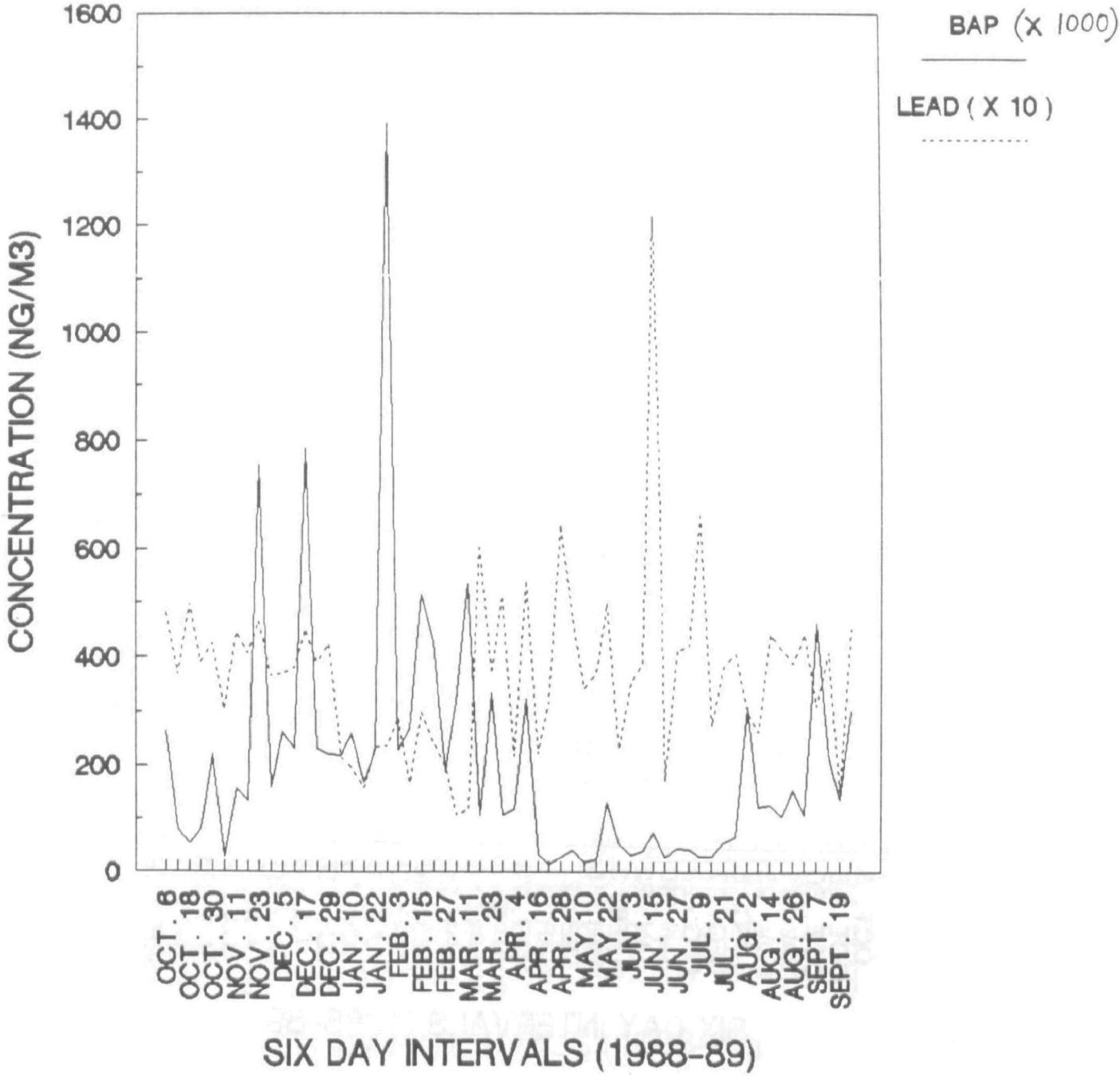


Figure IIIB-18

Mercury
at Carteret, NJ

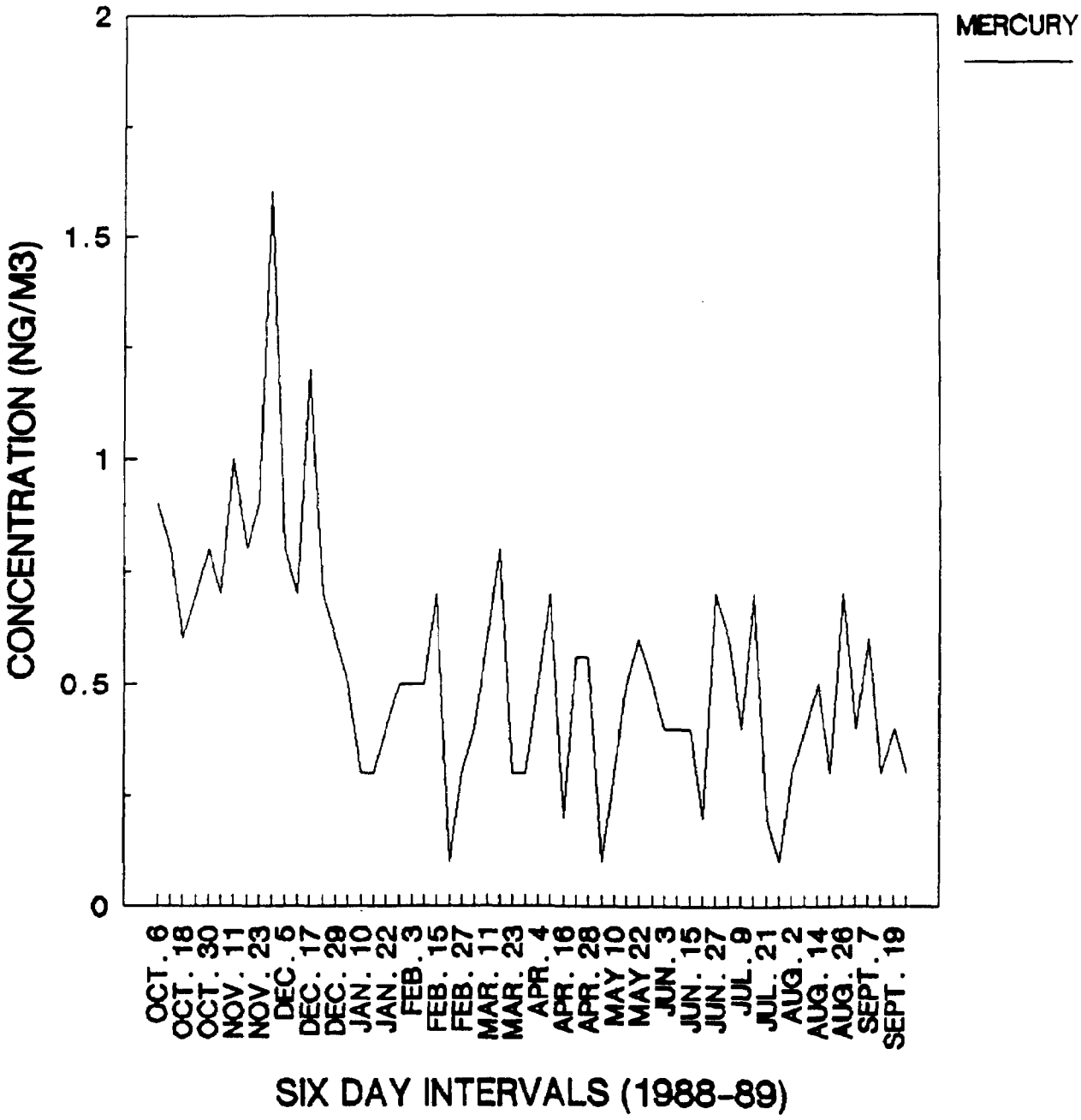


Figure IIIB-19

Nickel, Chromium, Manganese, and Iron at Elizabeth, NJ

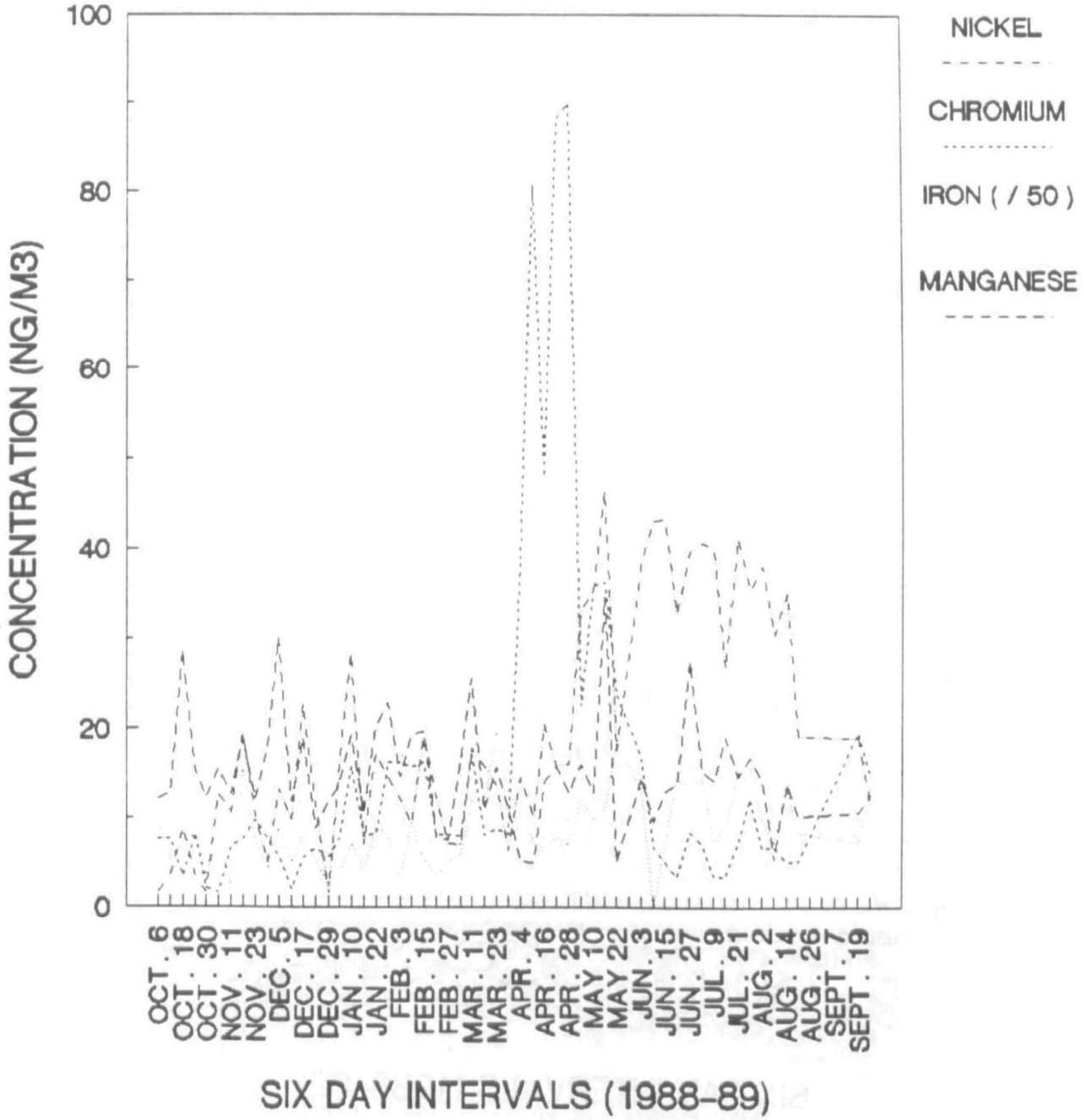


Figure IIIB-20

Cadmium, Copper, Zinc, and Lead at Elizabeth, NJ

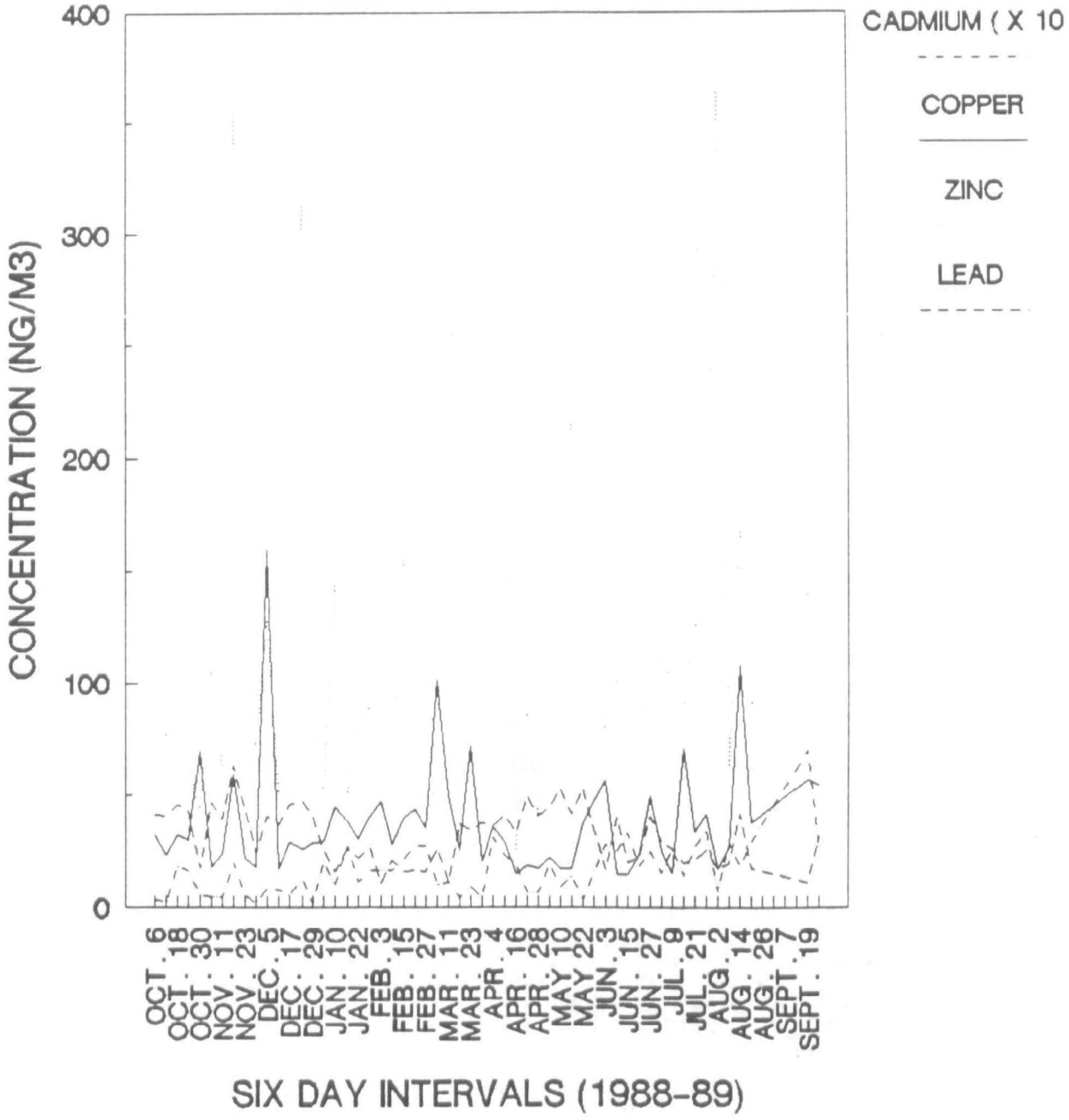


Figure IIIB-21

Benzo[α]pyrene and Lead at Elizabeth, NJ

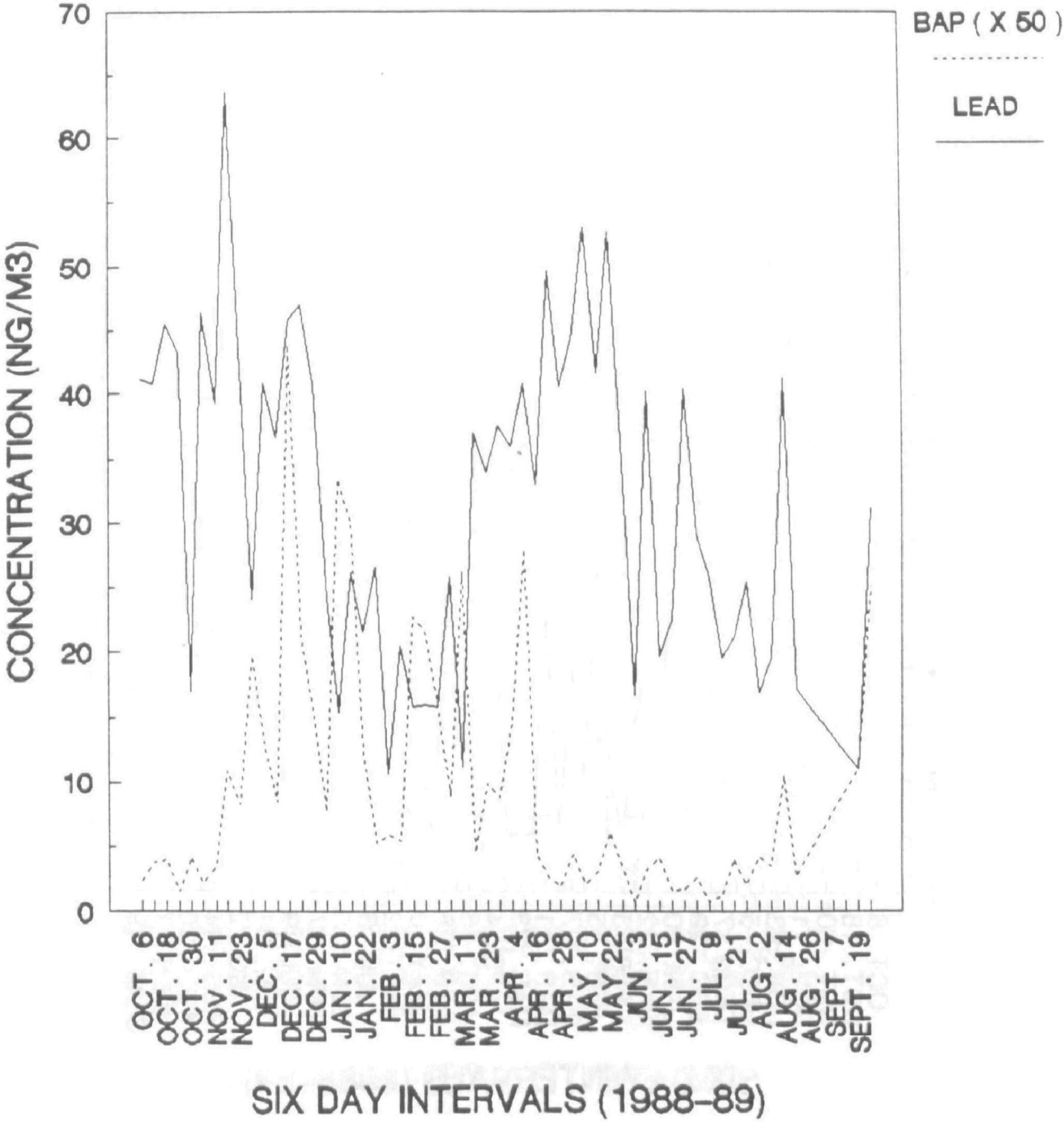


Figure IIIB-22

Mercury
at Elizabeth, NJ

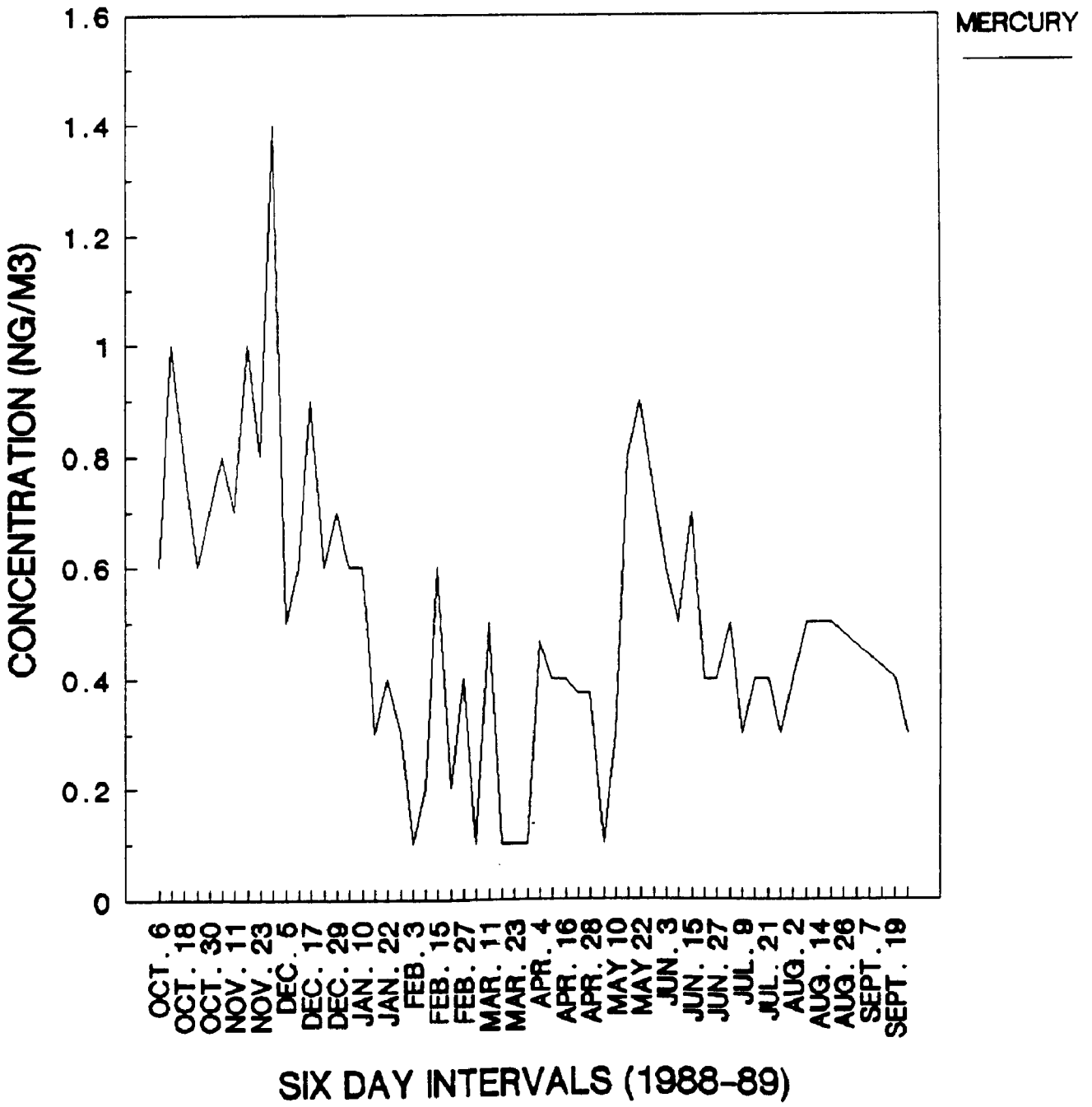


Figure IIIB-23

Molybdenum, Nickel, Cobalt, Iron, and Manganese at Susan Wagner H.S.

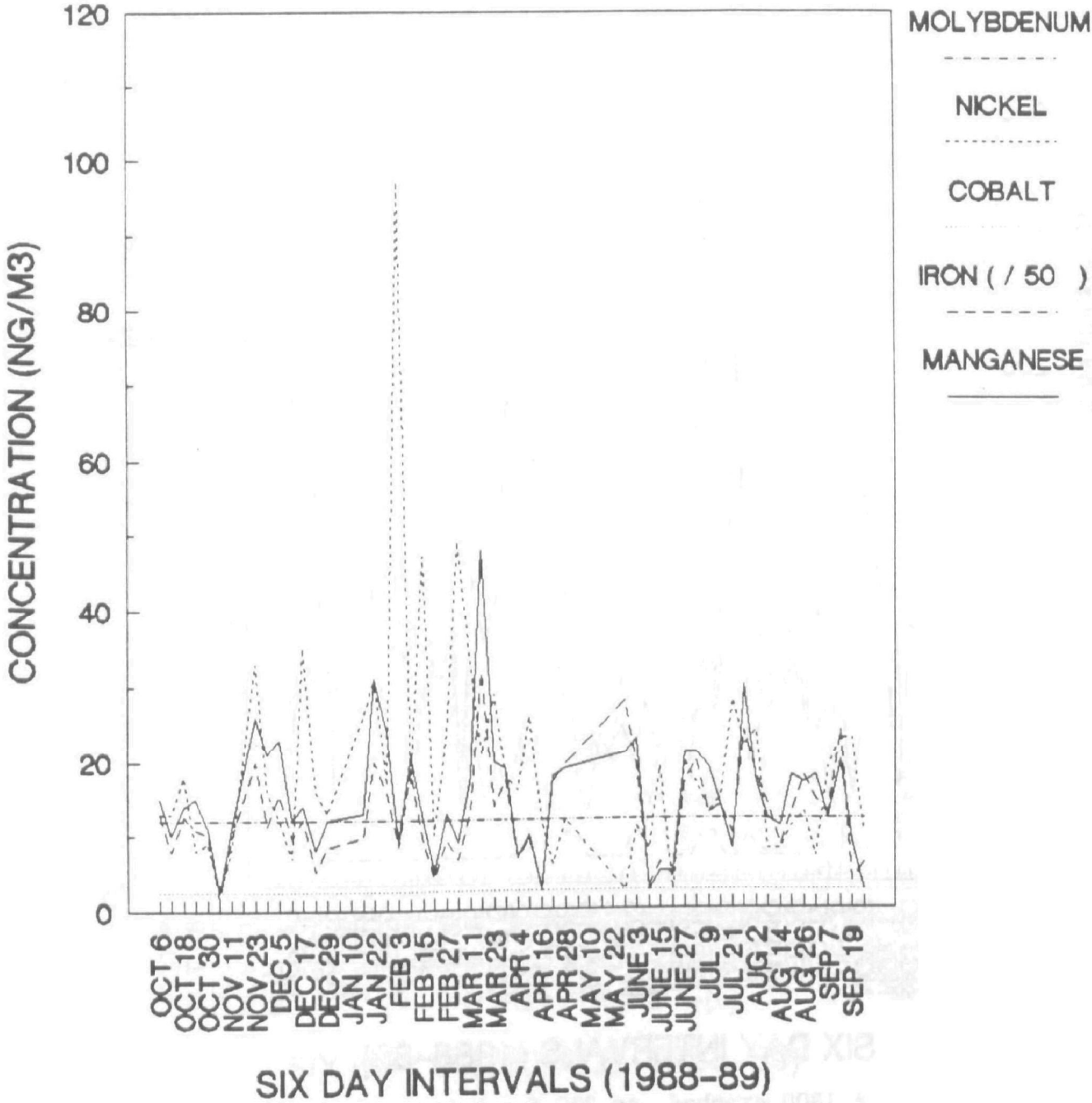
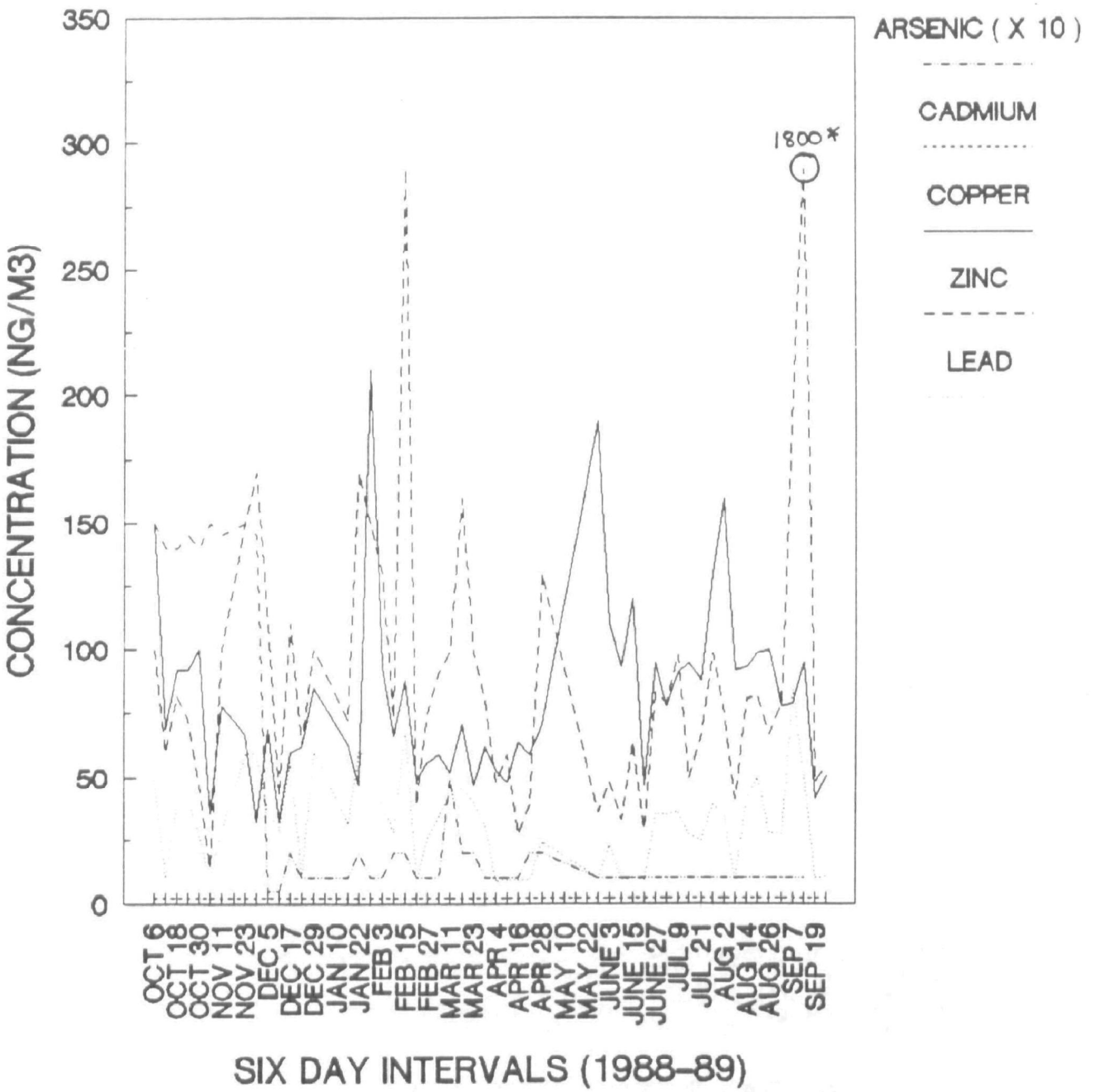


Figure IIIB-24

Arsenic, Cadmium, Copper, Zinc, and Lead at Susan Wagner H.S.



* 1800 graphed as 290 for better resolution of the various curves

Figure IIIB-25

Benzo[α]pyrene, Vanadium, and Lead at Susan Wagner H.S.

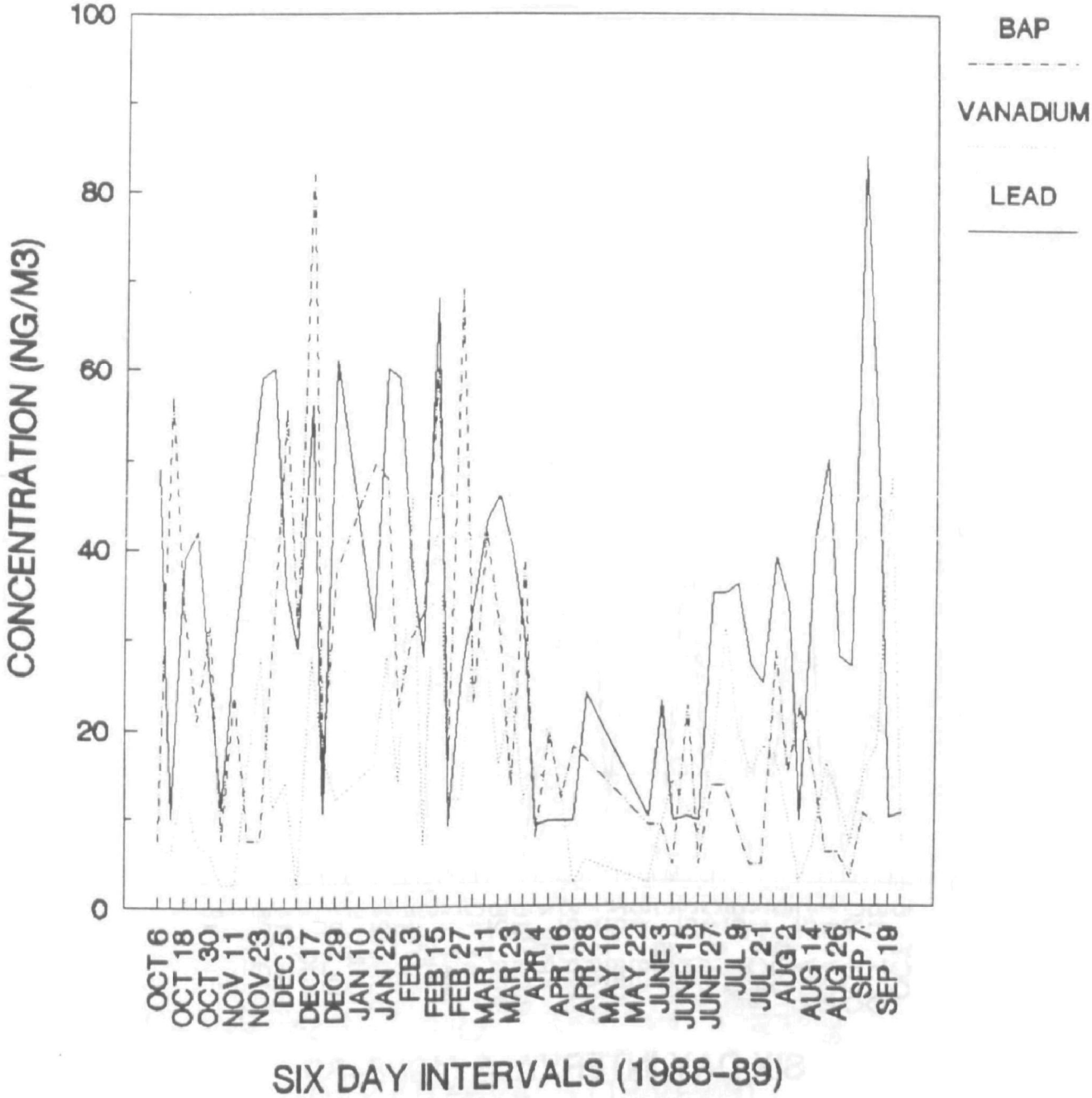


Figure IIIB-26

Molybdenum, Nickel, Copper, Iron, and Manganese at PS-26

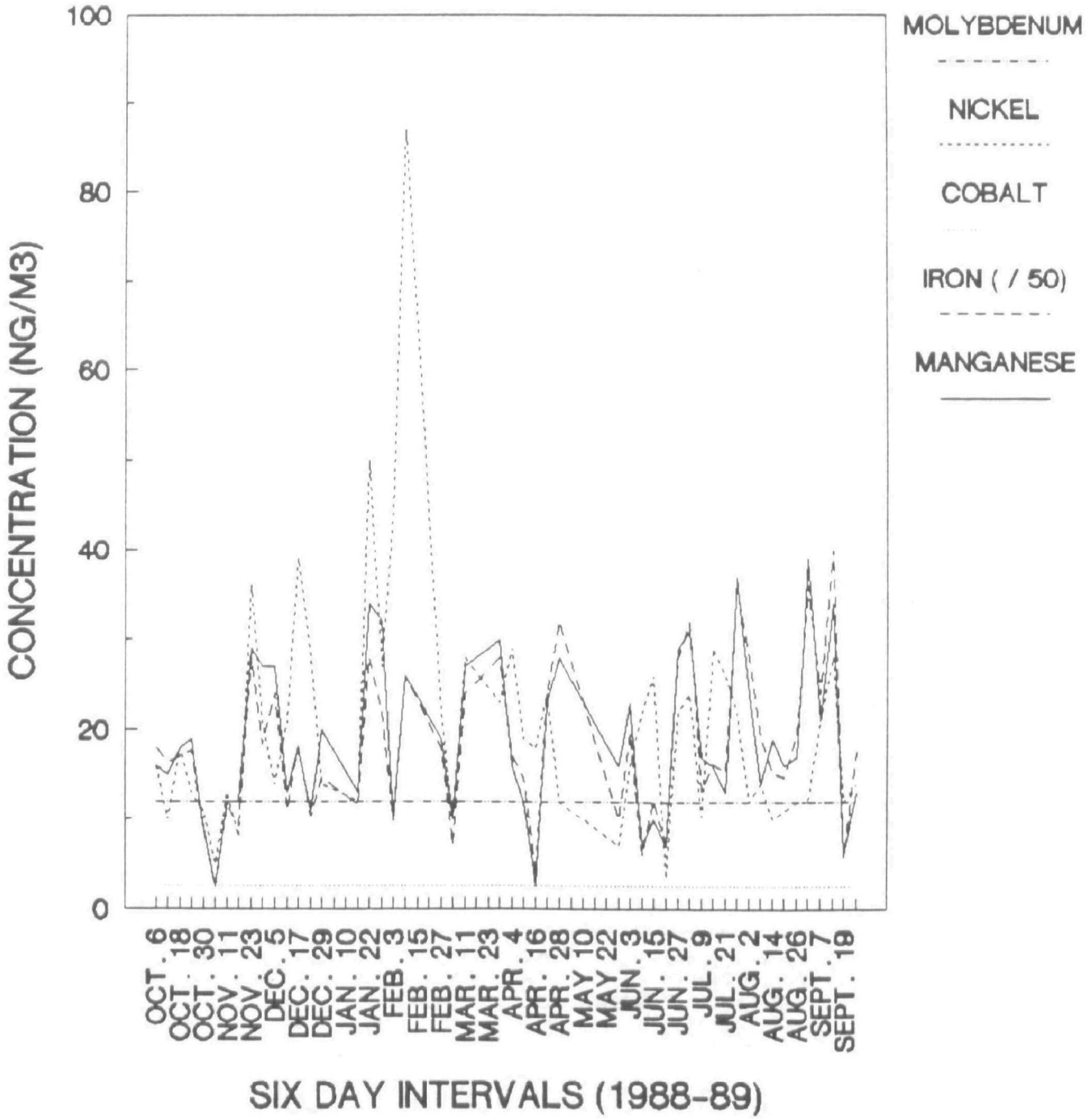


Figure IIIB-27

Arsenic, Cadmium, Copper, Zinc, and Lead at PS-26

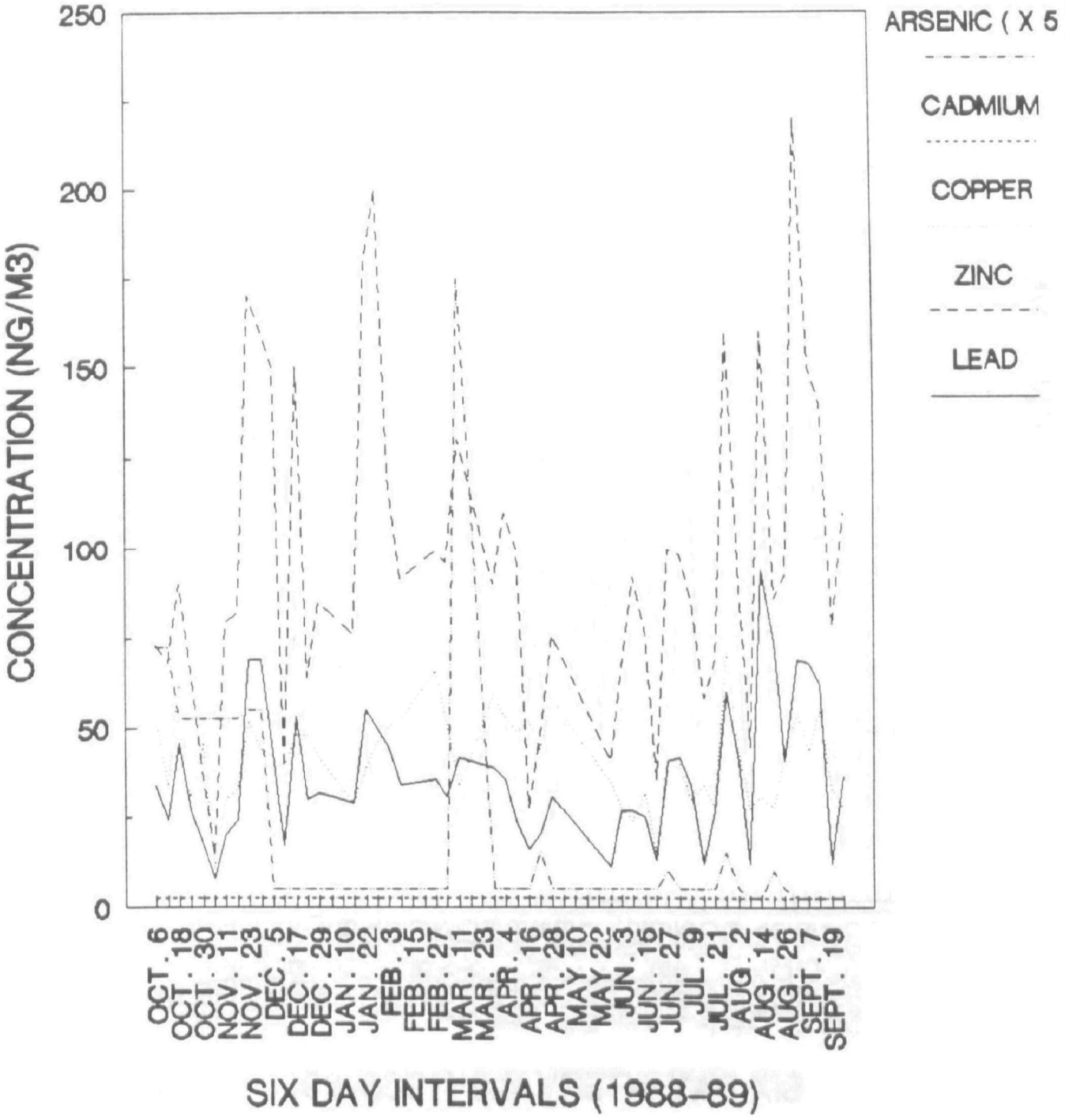


Figure IIIB-28

Benzo[α]pyrene, Vanadium, and Lead at PS-26

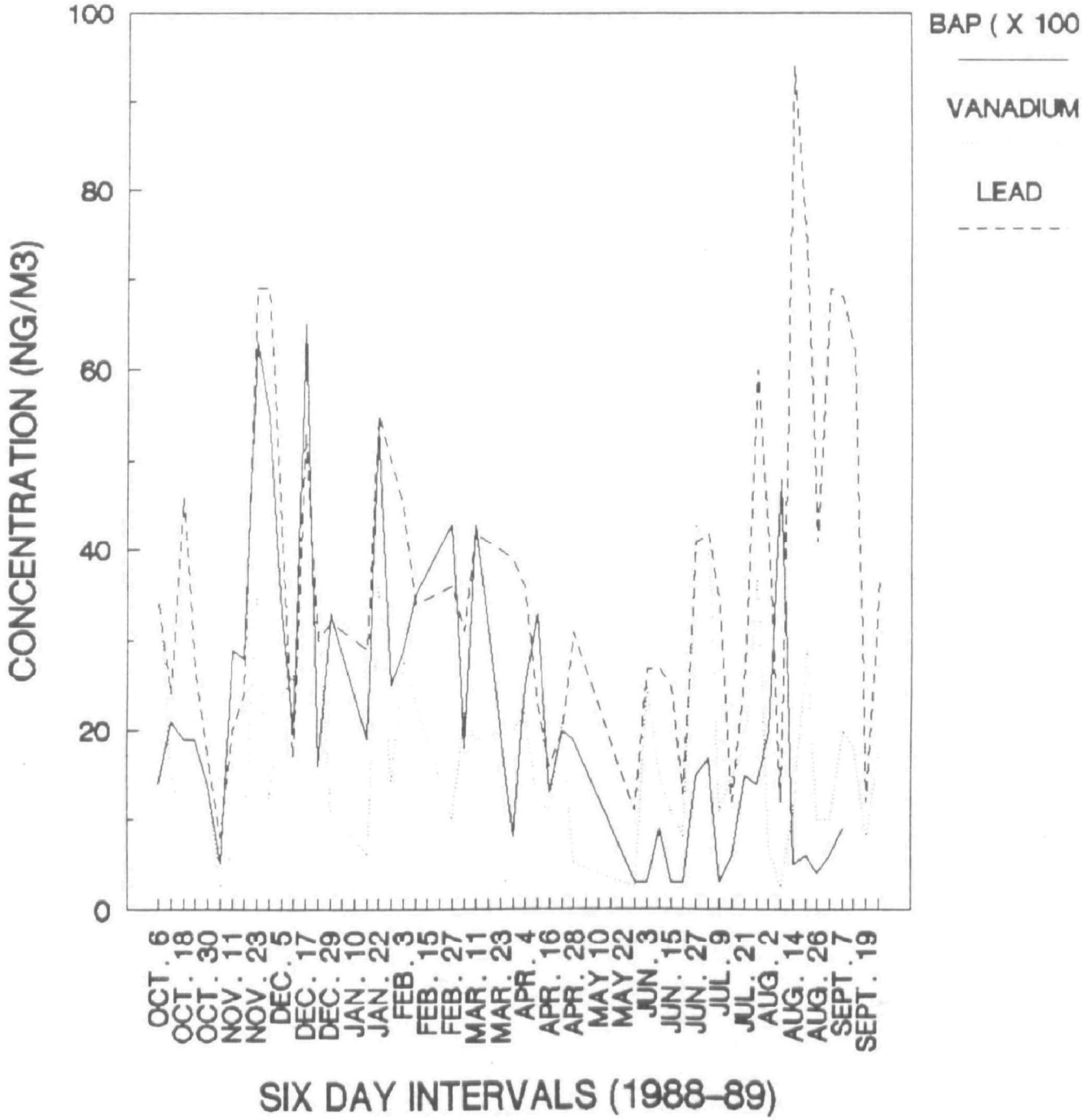


FIGURE IIIB-29

BARIUM
TWO SITES

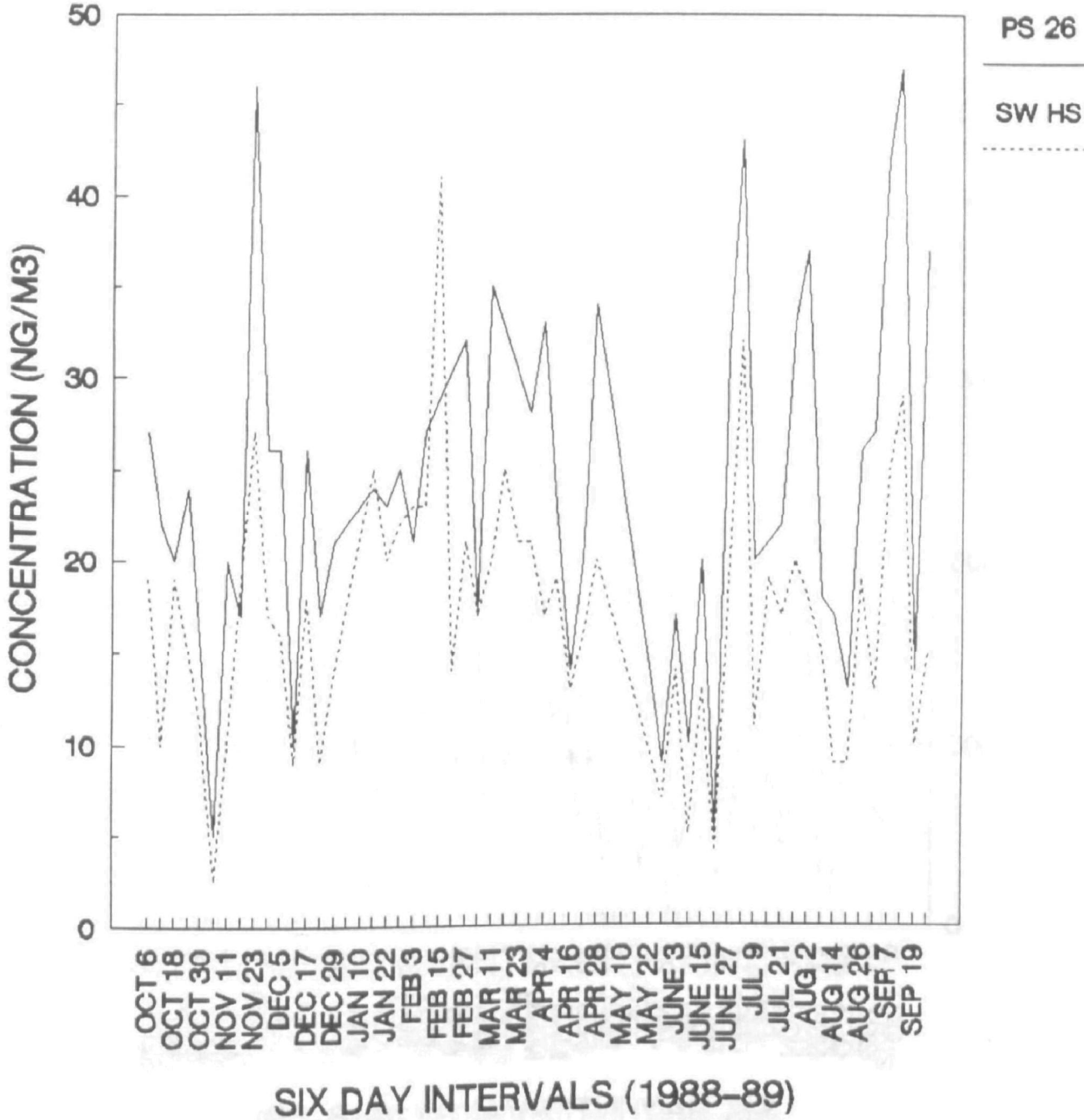


FIGURE IIIB-30

IRON

4 SITES

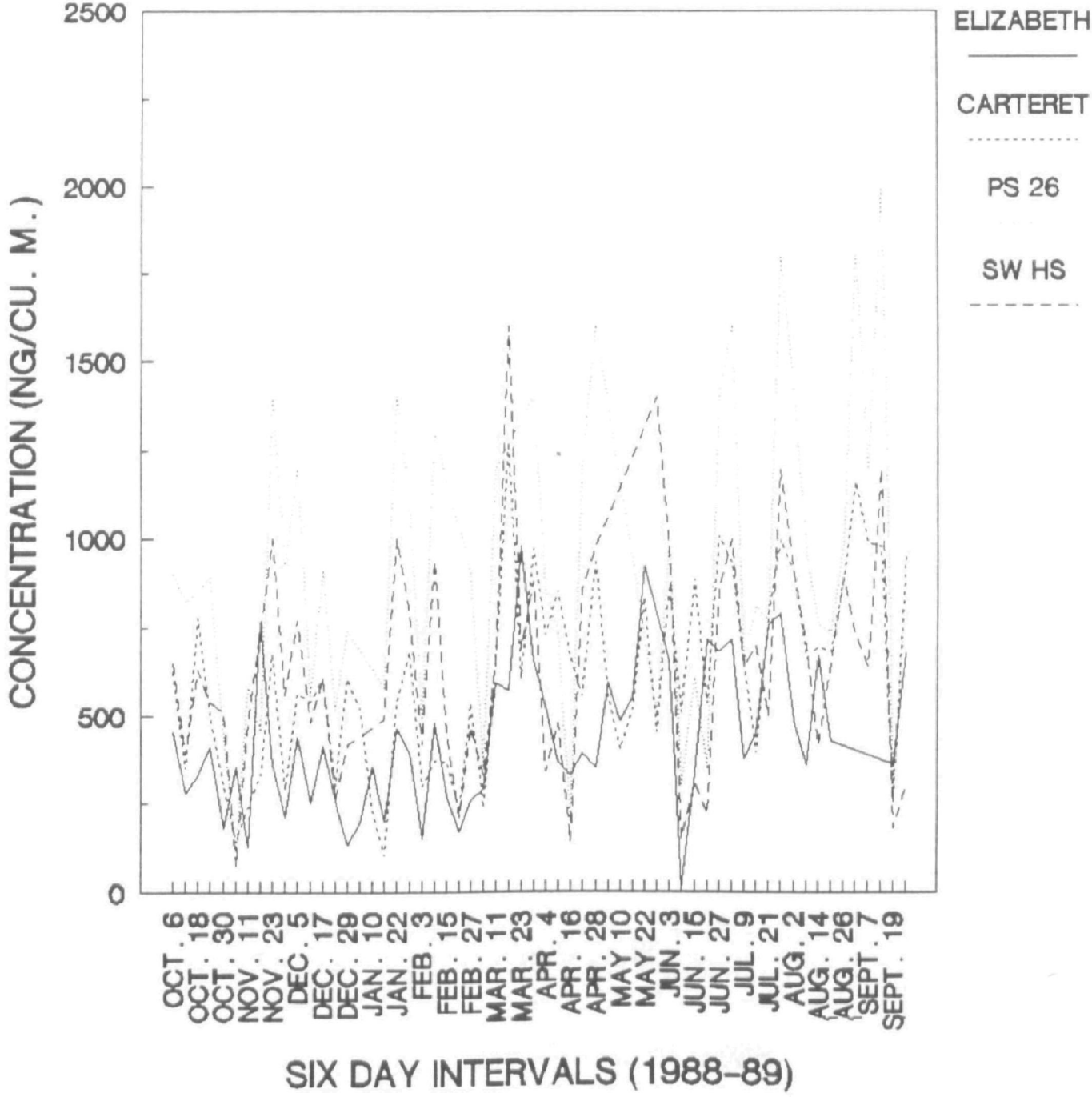


FIGURE IIIB-31

IRON

2 SITES (NJ)

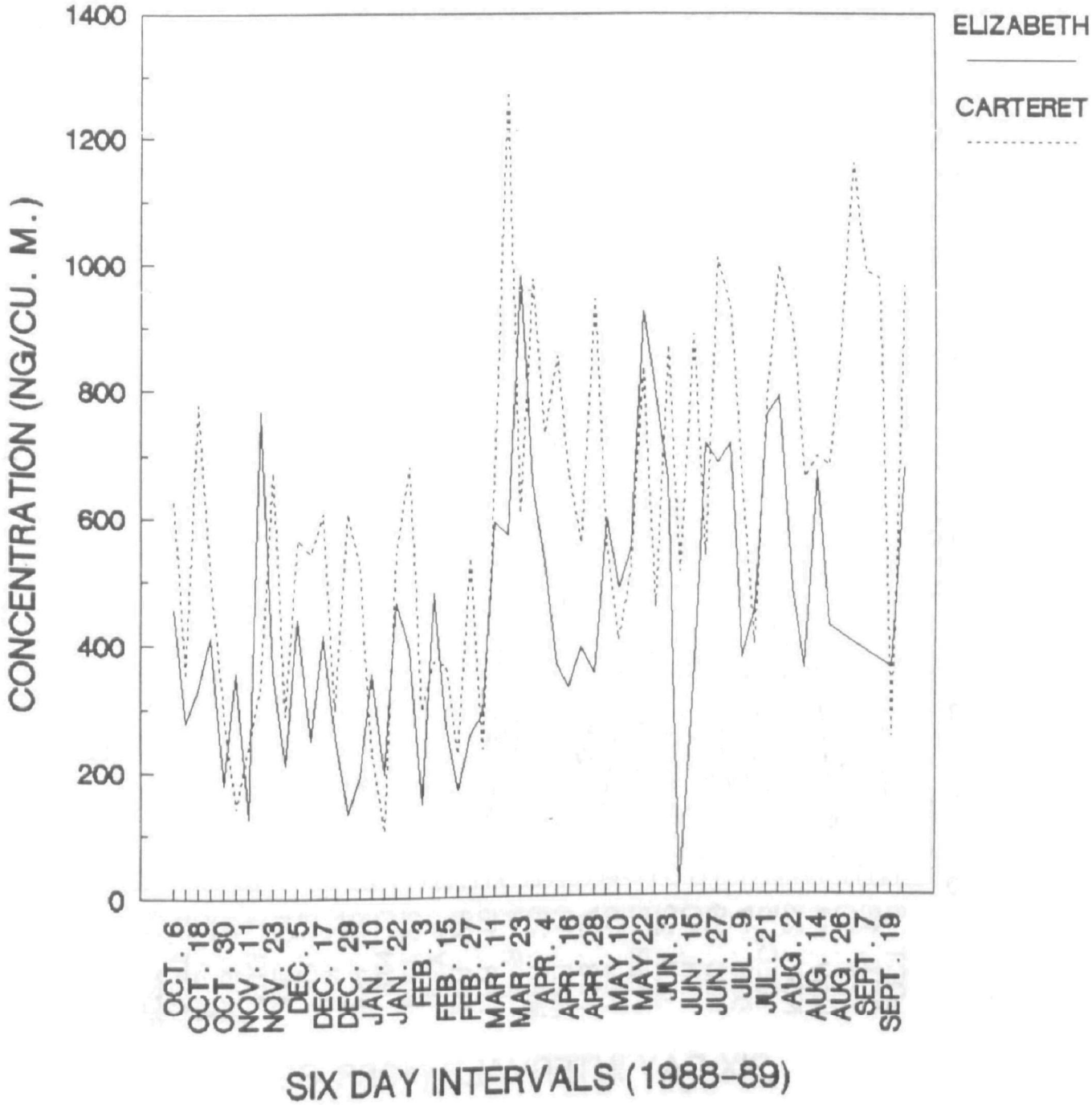


FIGURE IIIB-32

IRON

2 SITES (NY)

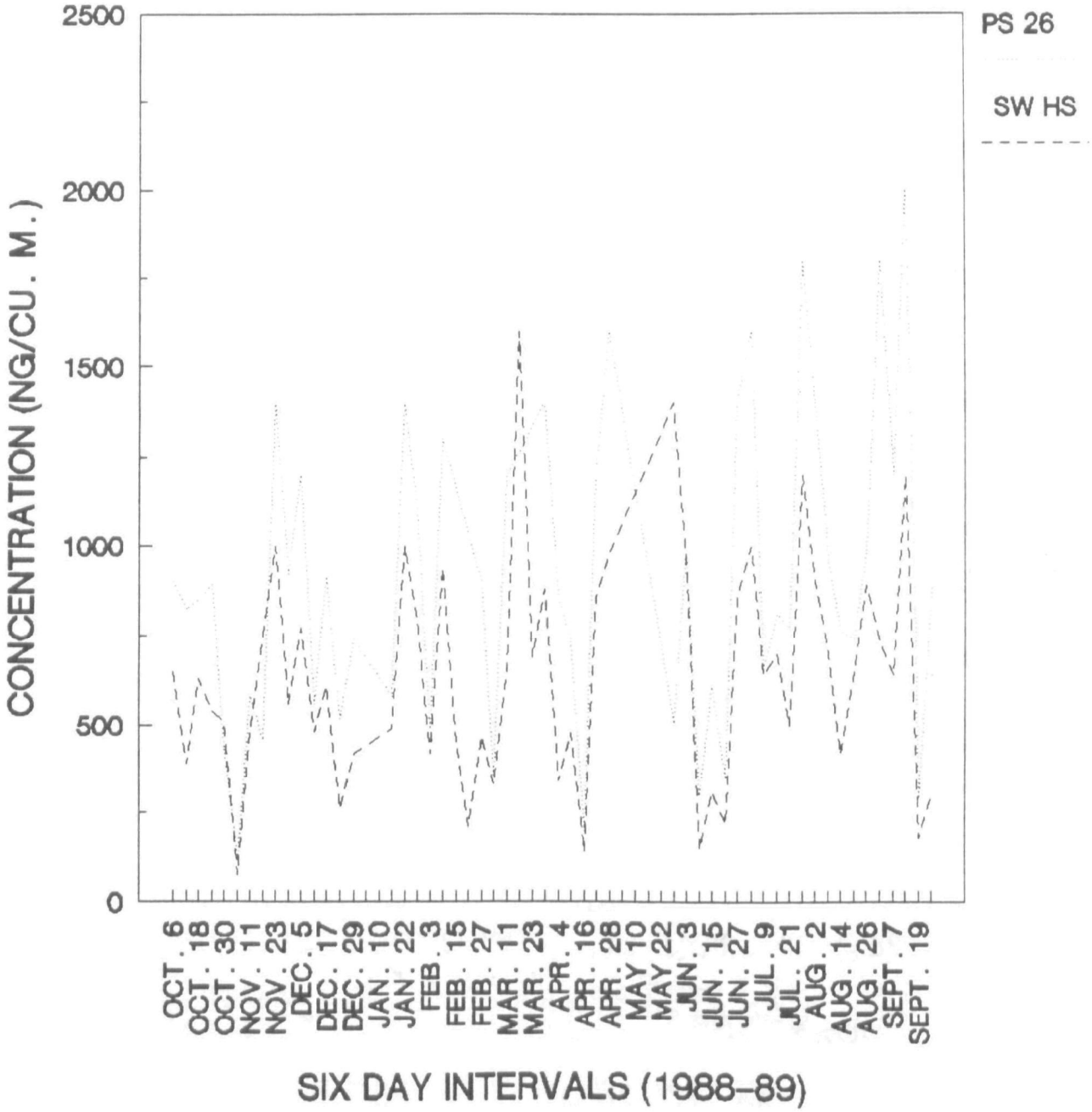


FIGURE IIIB-33

MANGANESE

4 SITES

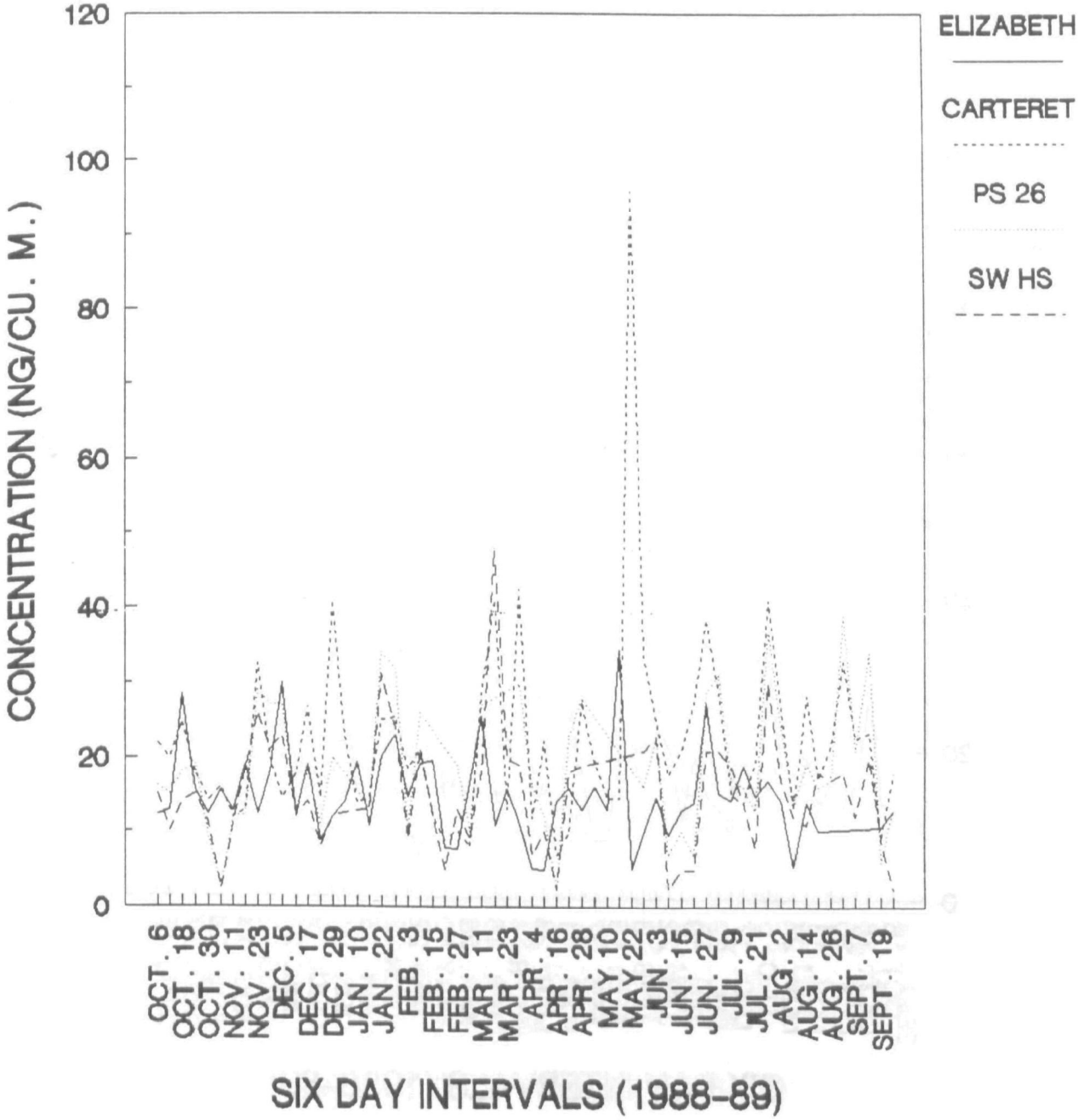


FIGURE IIIB-34

MANGANESE

2 SITES (NJ)

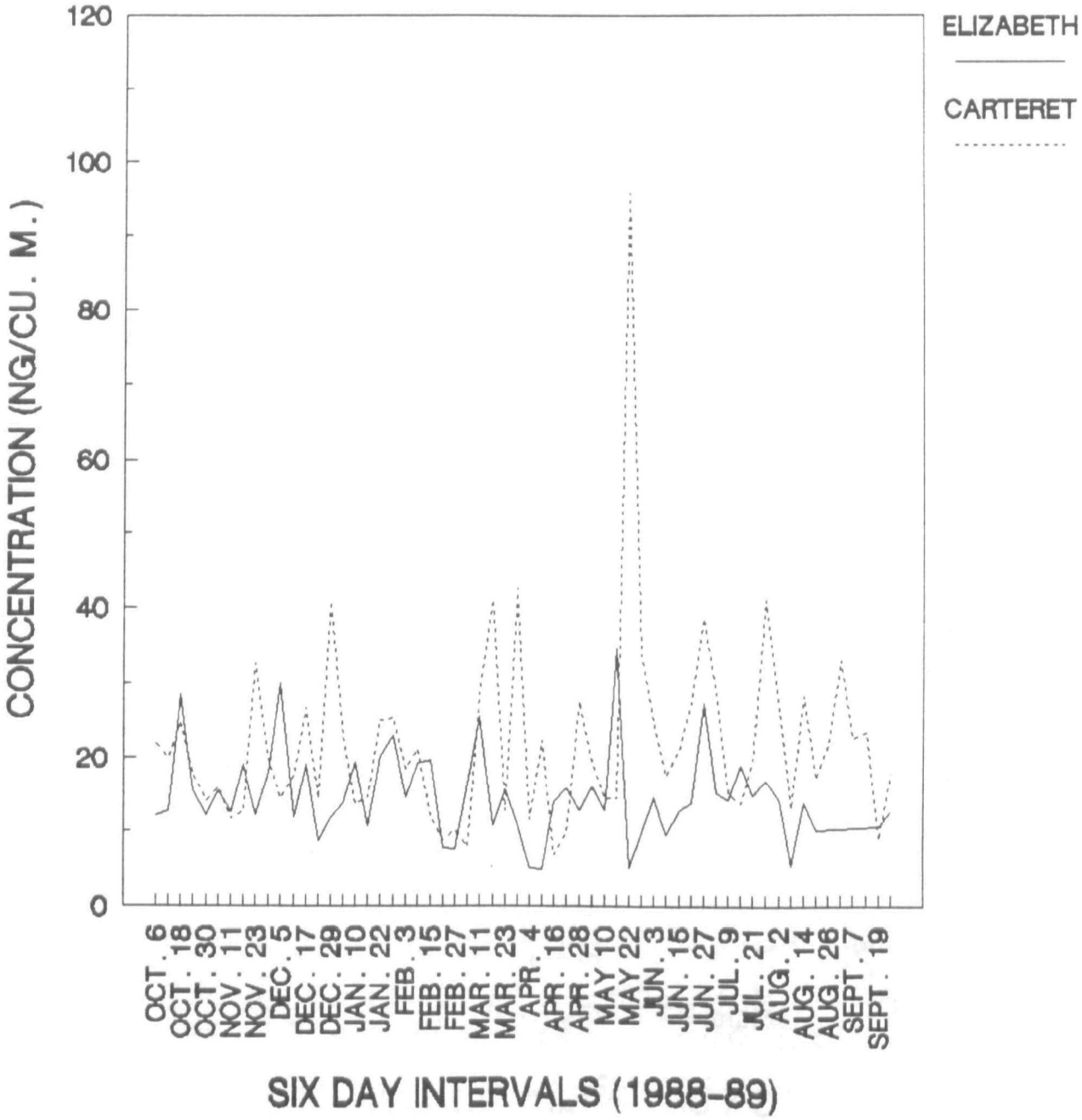


FIGURE IIIB-35

MANGANESE

2 SITES (NY)

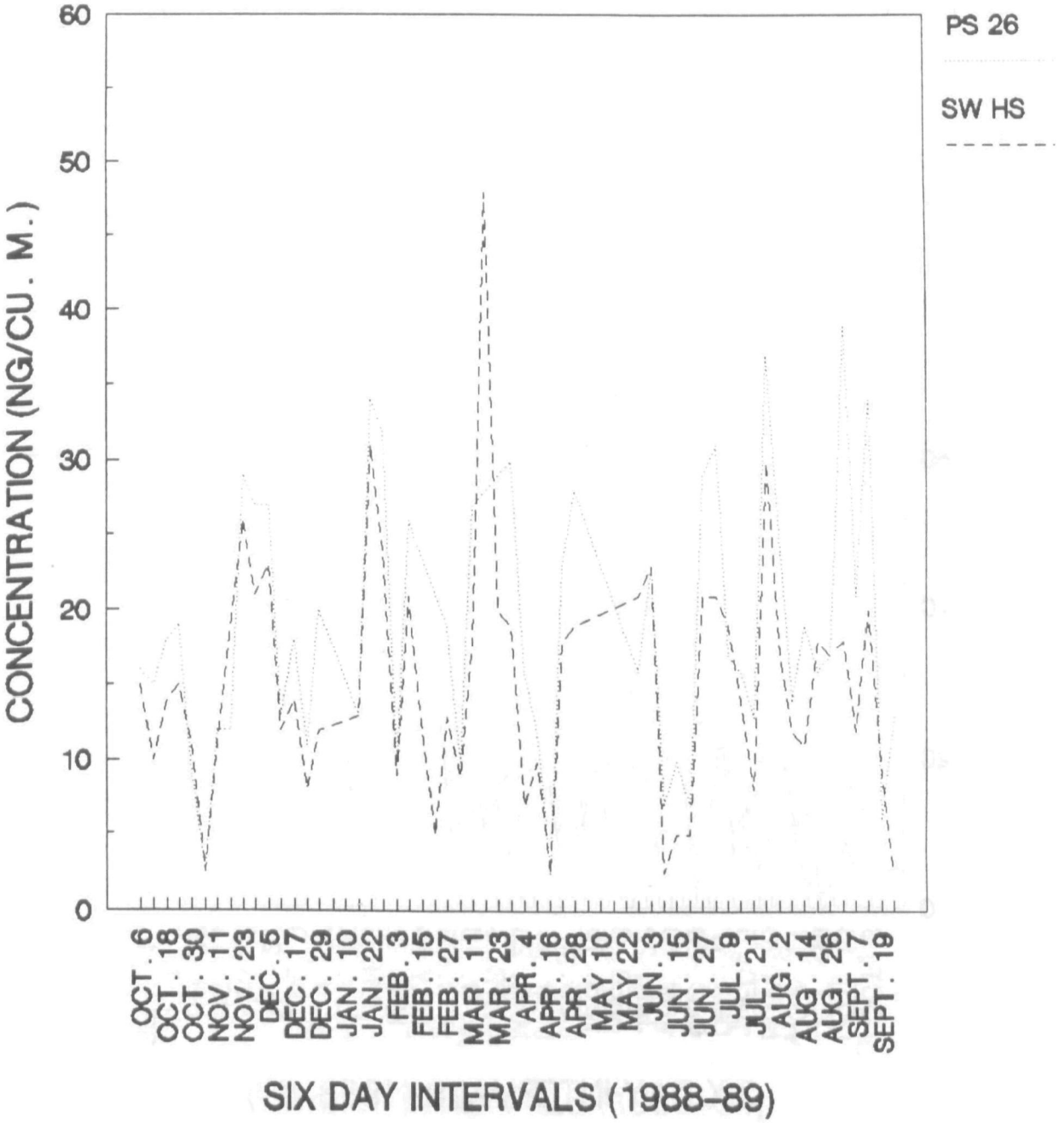


FIGURE IIIB-36

NICKEL

4 SITES

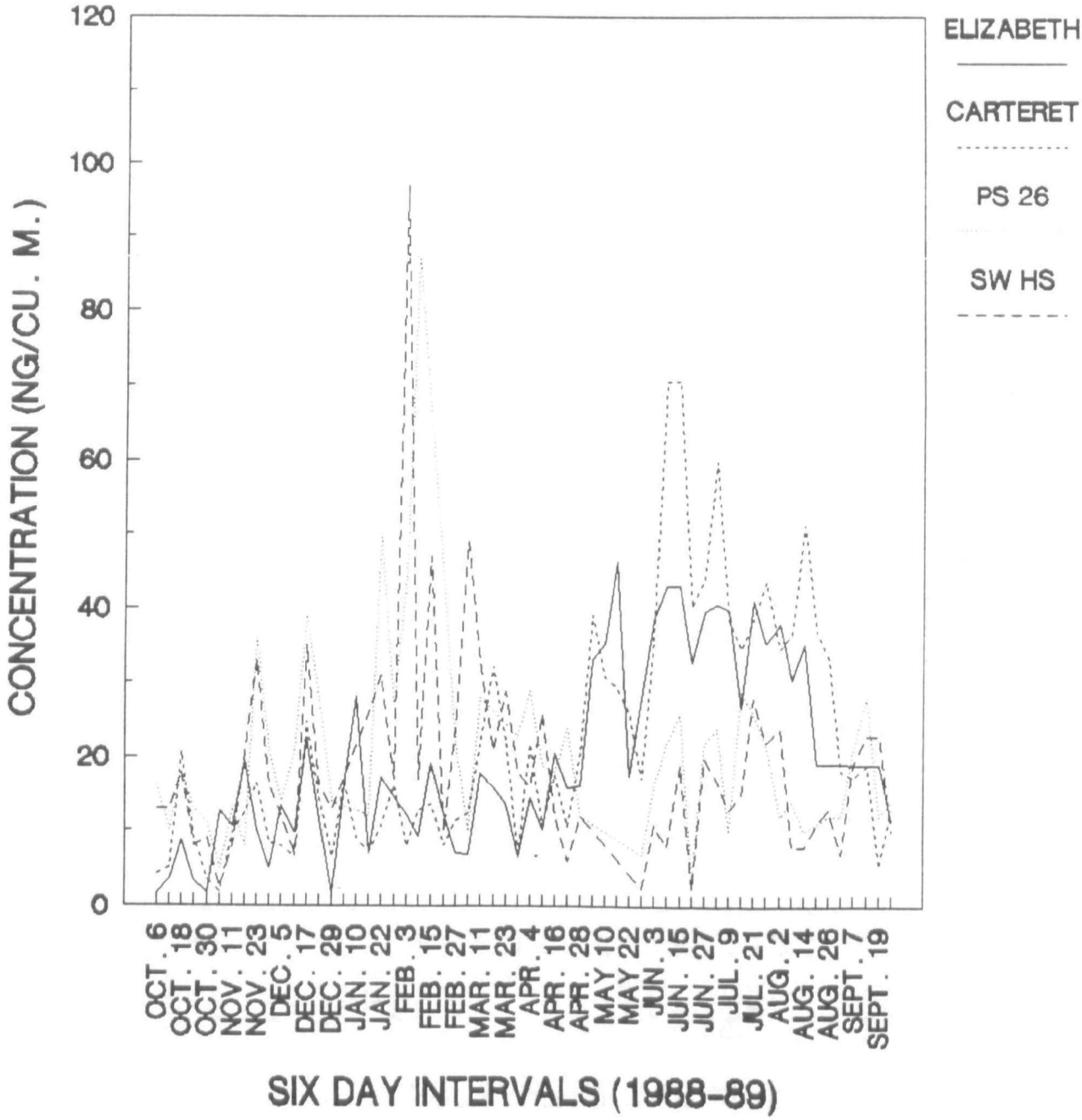


FIGURE IIIB-37

NICKEL

2 SITES (NJ)

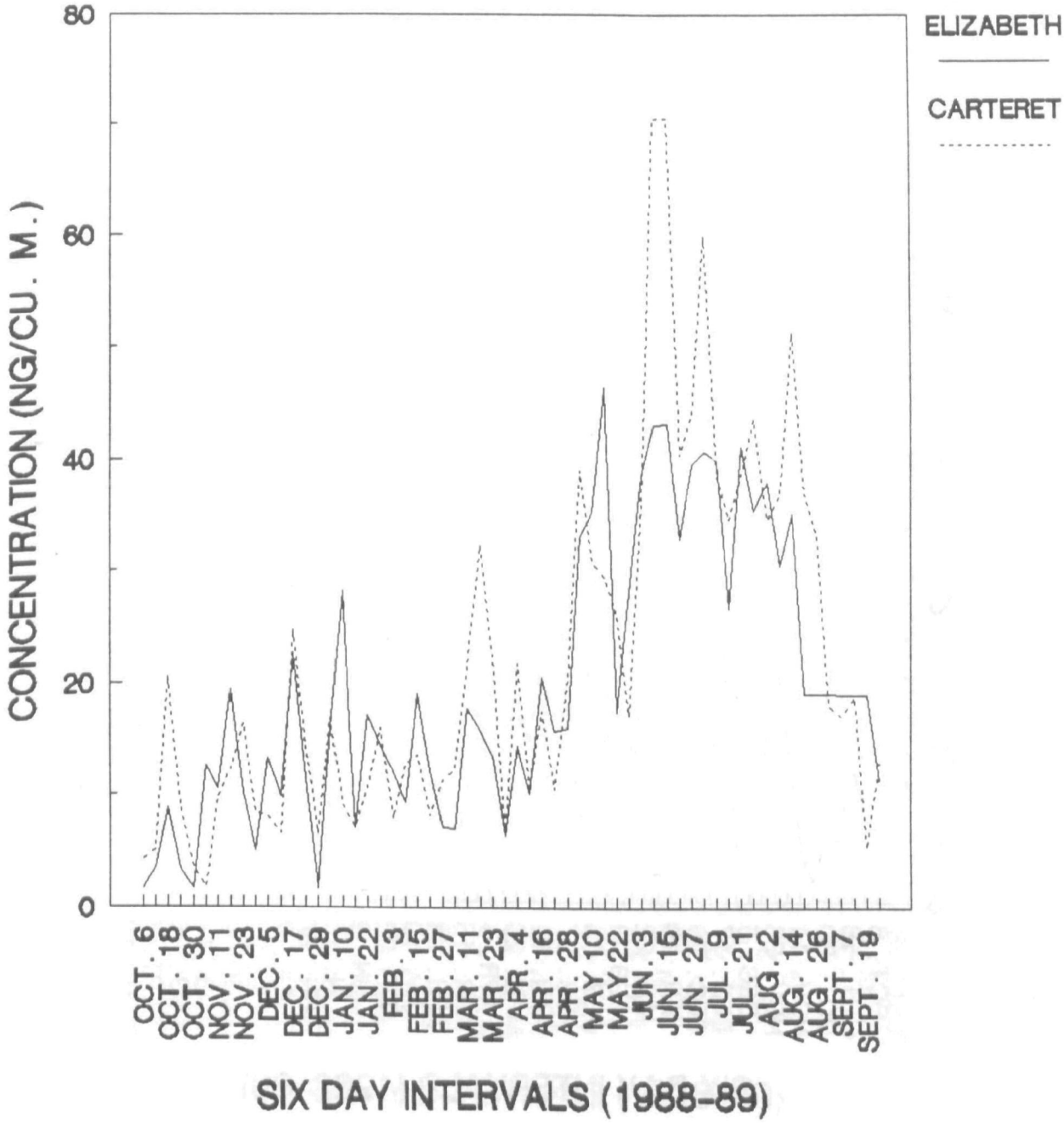


FIGURE IIIB-38

NICKEL

2 SITES (NY)

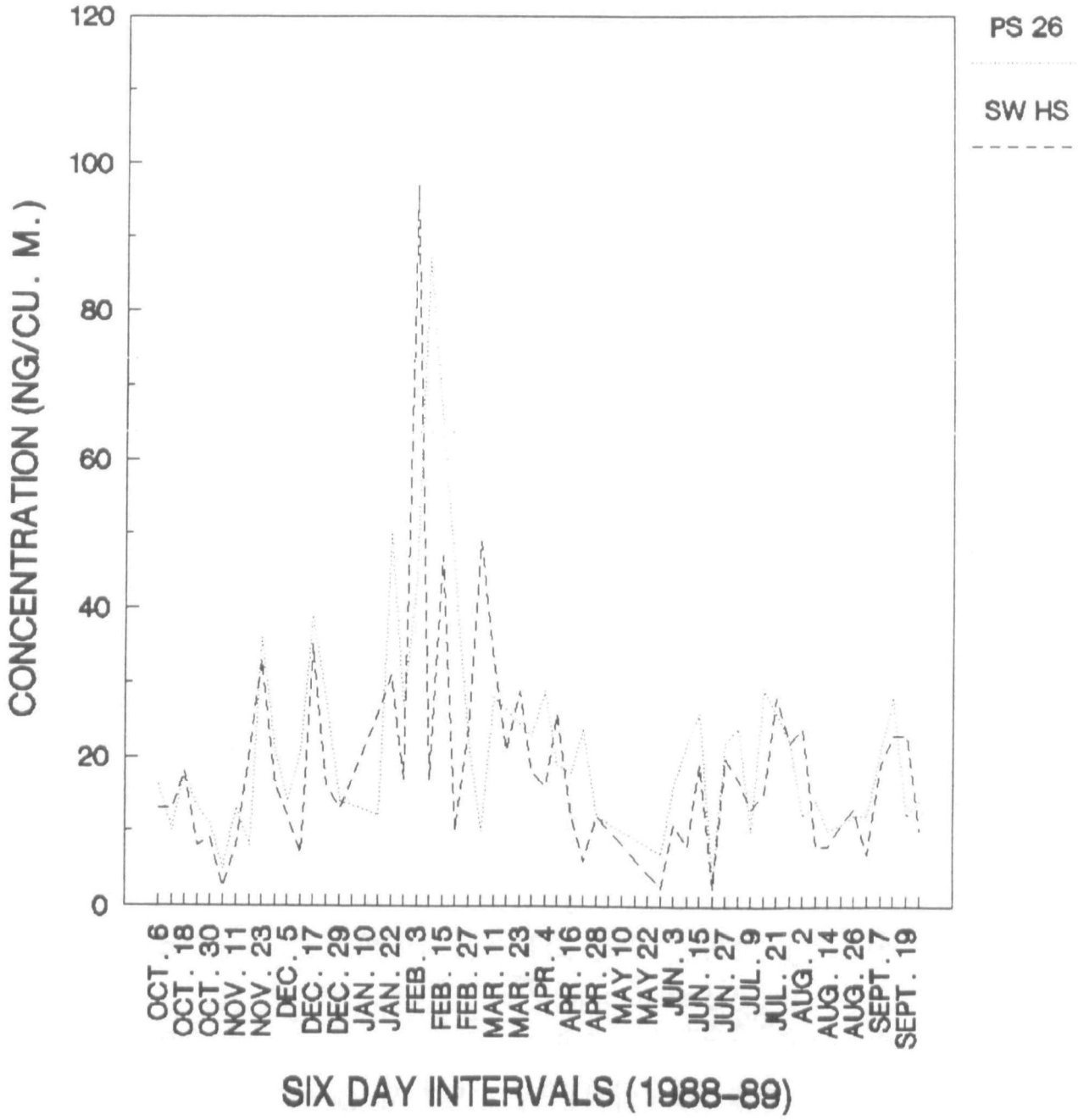


FIGURE IIIB-39

VANADIUM

2 SITES

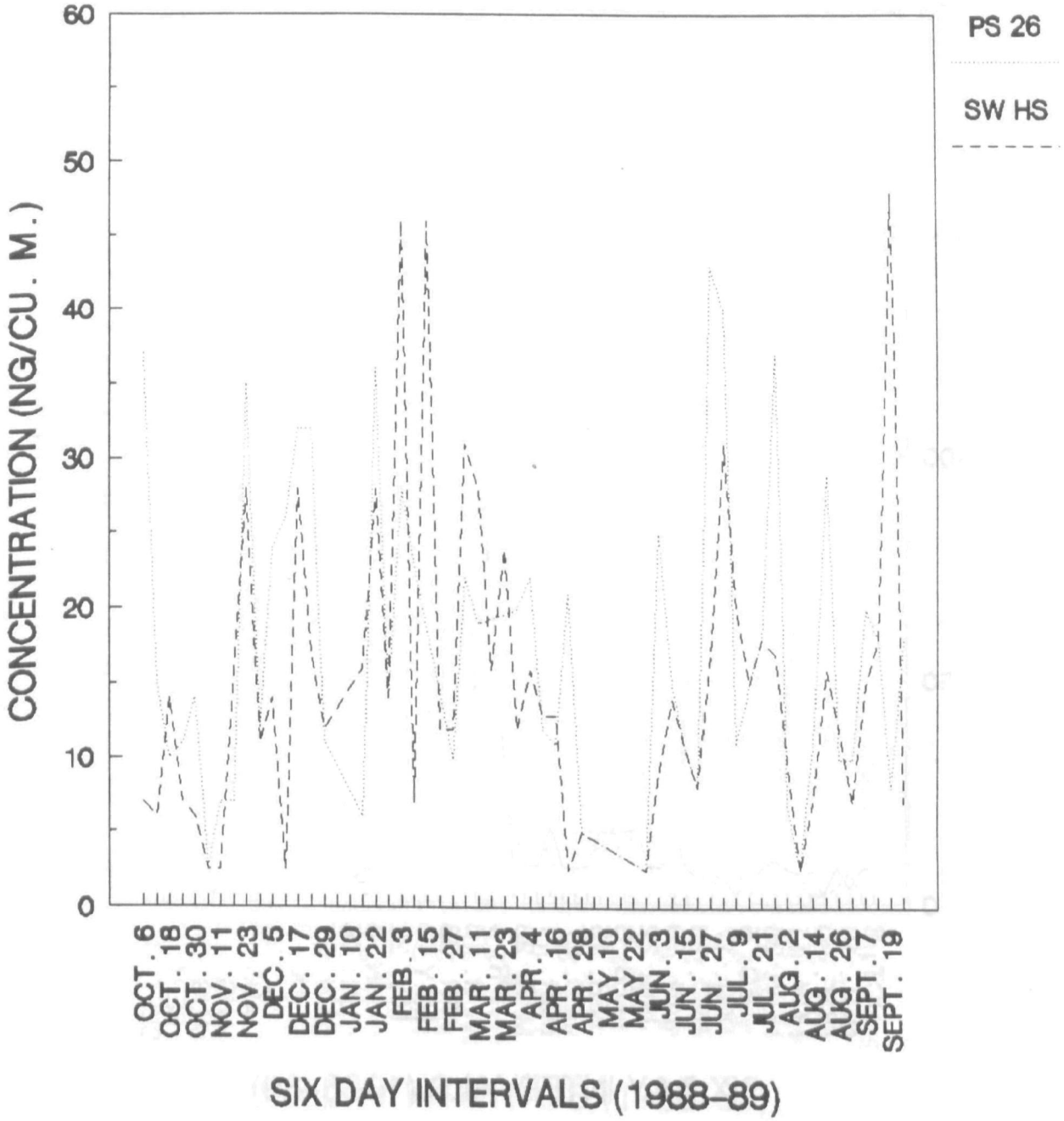


FIGURE IIIB-40

CHROMIUM

2 SITES

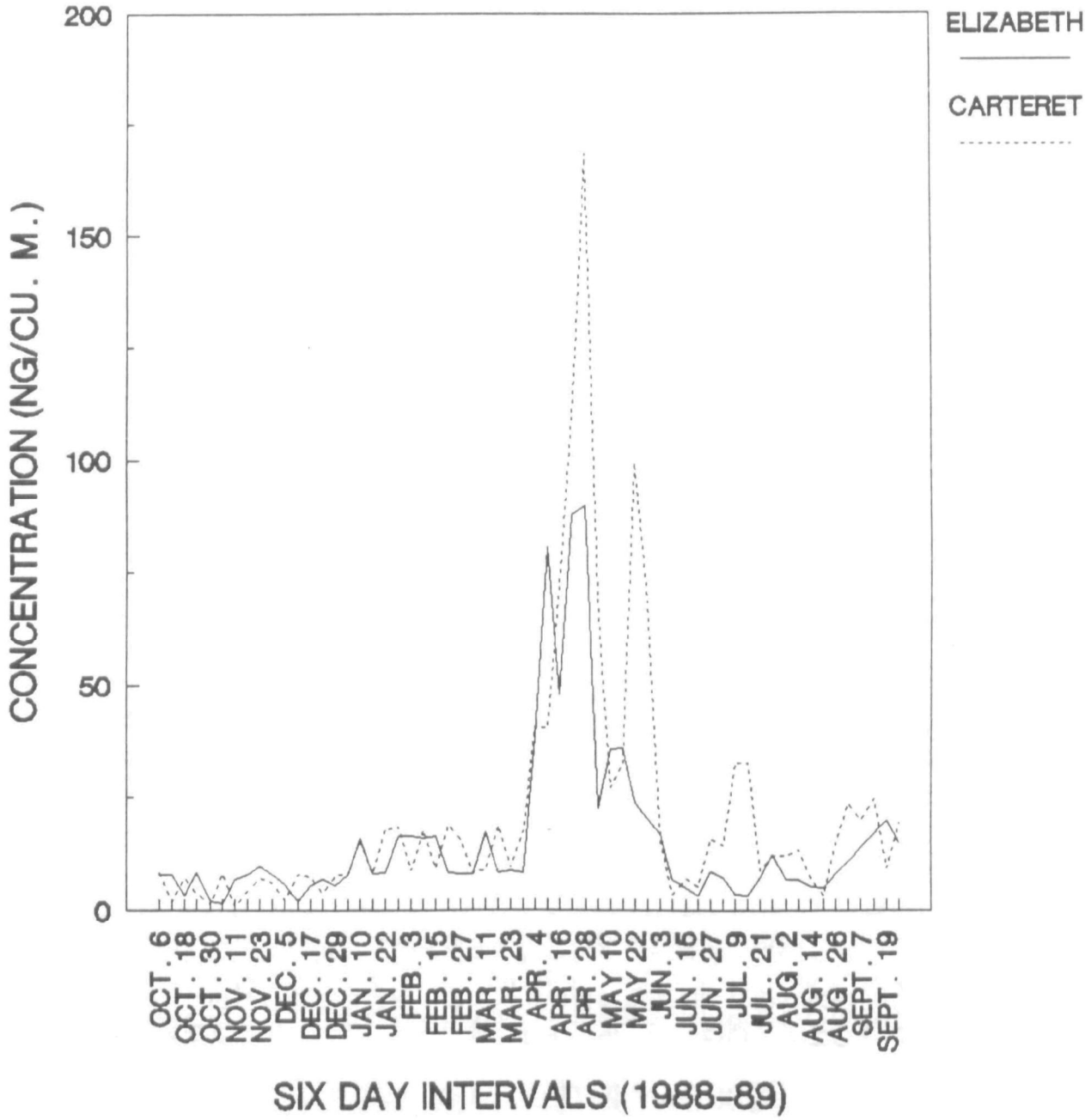


FIGURE IIIB-41

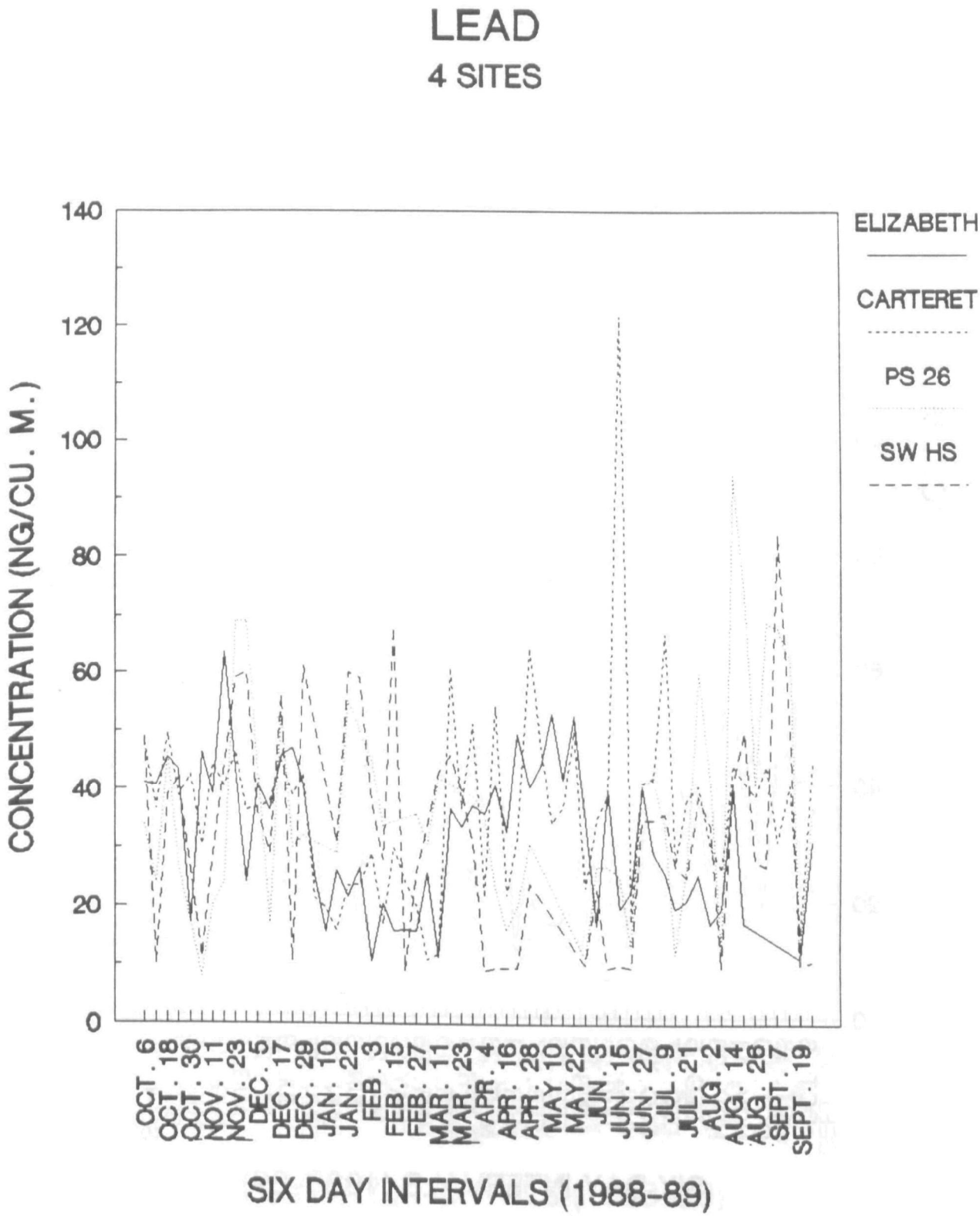


FIGURE IIIB-42

LEAD

2 SITES (NJ)

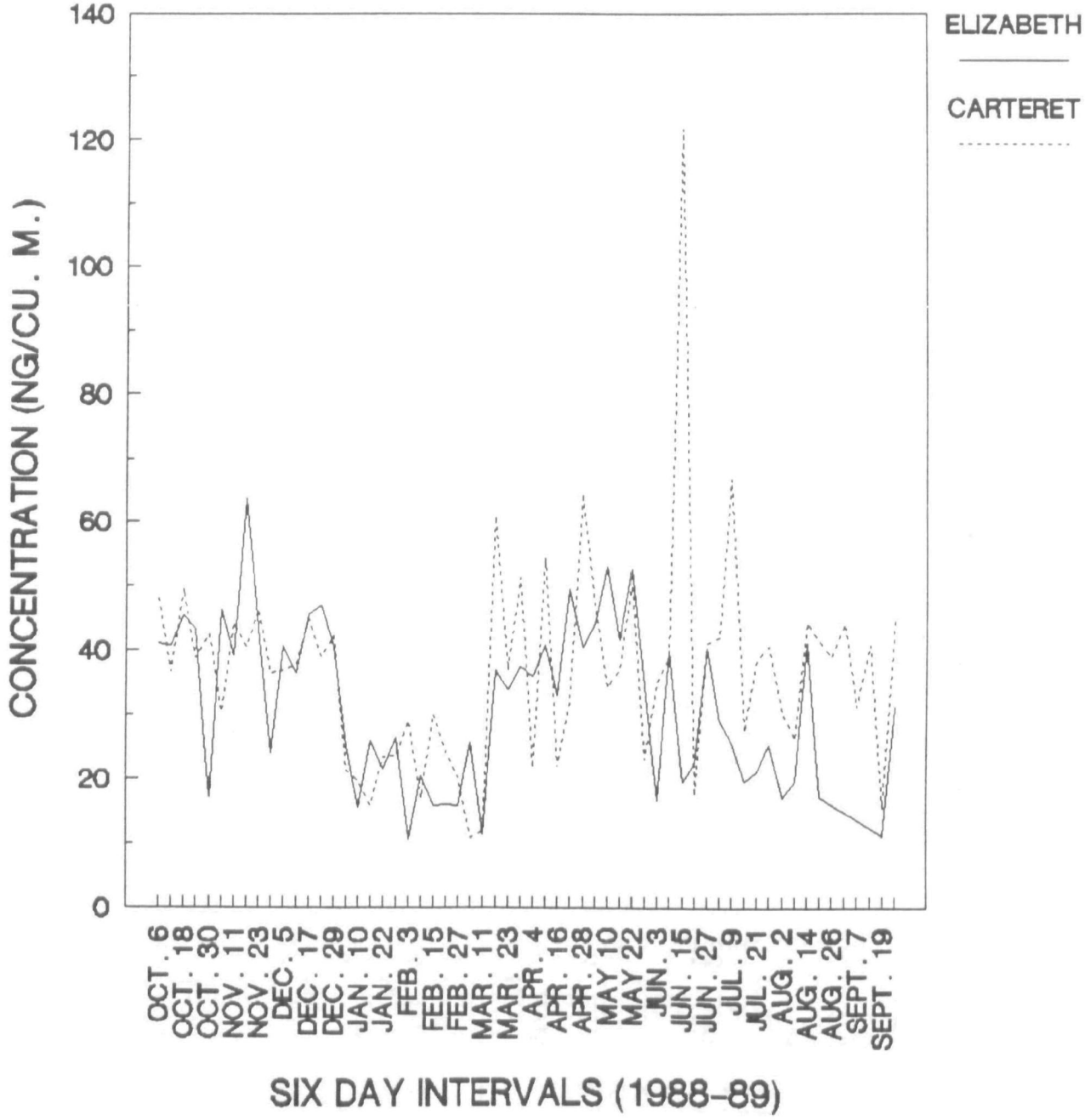


FIGURE IIIB-43

LEAD

2 SITES (NY)

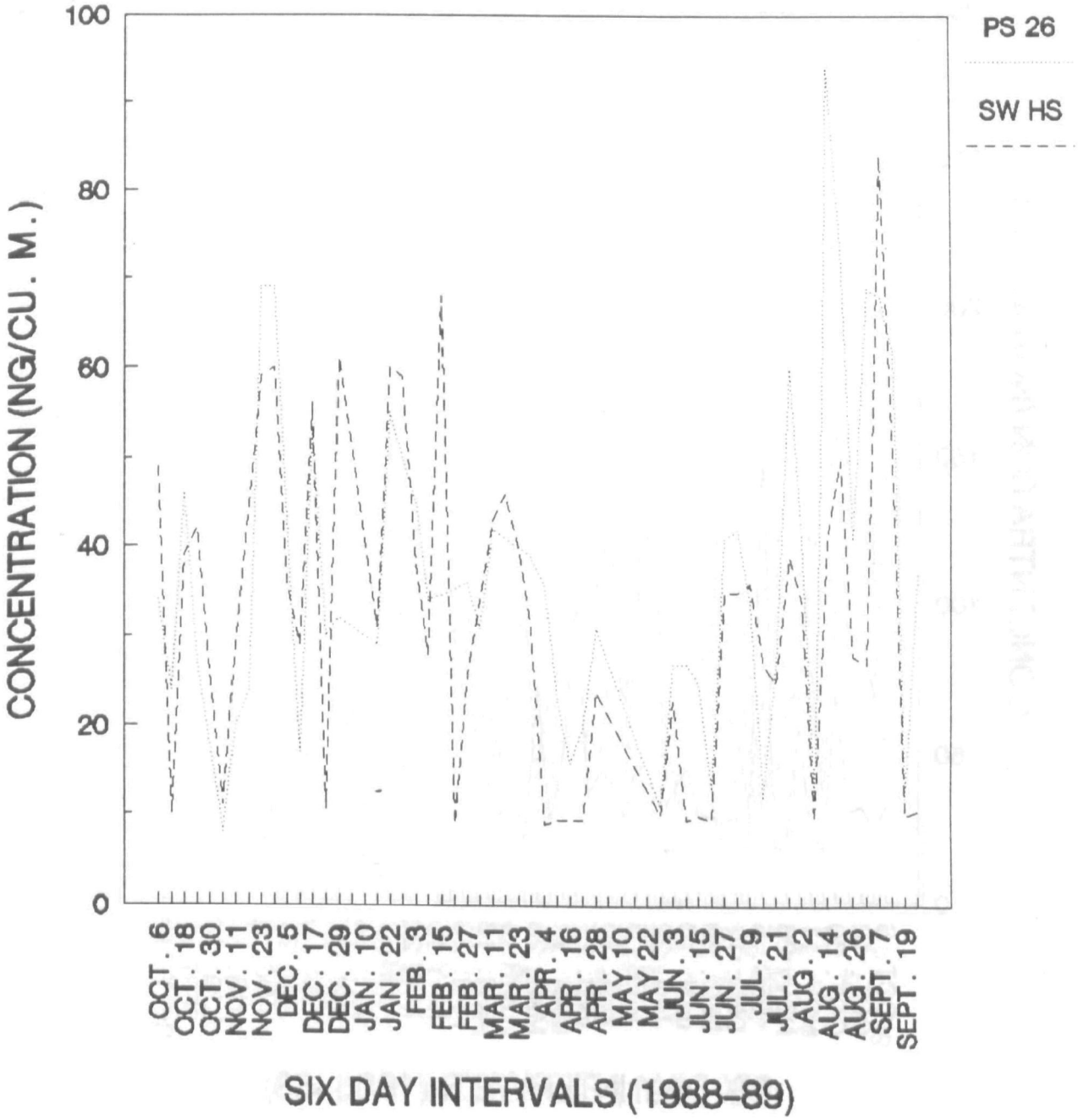


FIGURE IIIB-44

COPPER

4 SITES

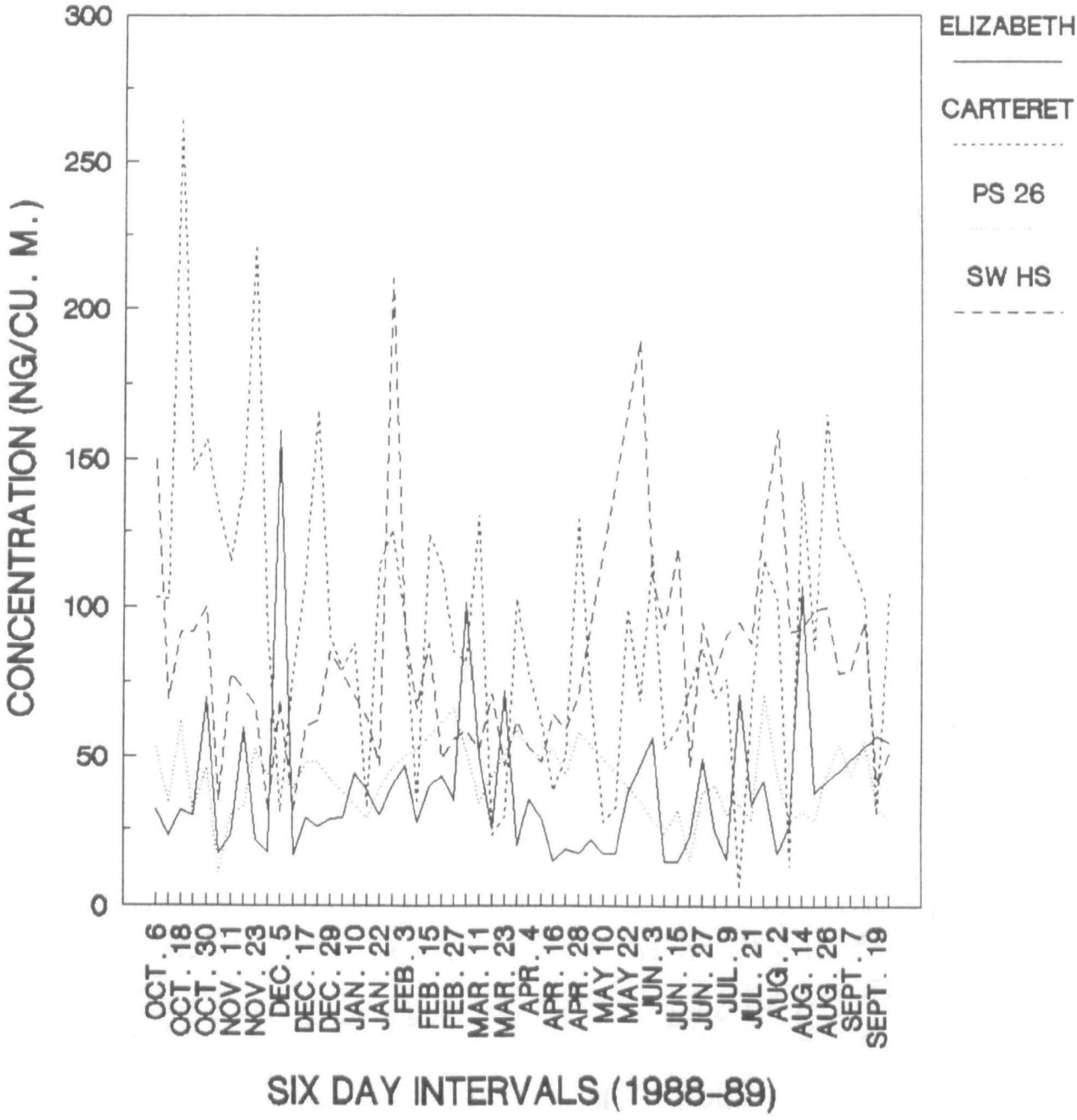


FIGURE IIIB-45

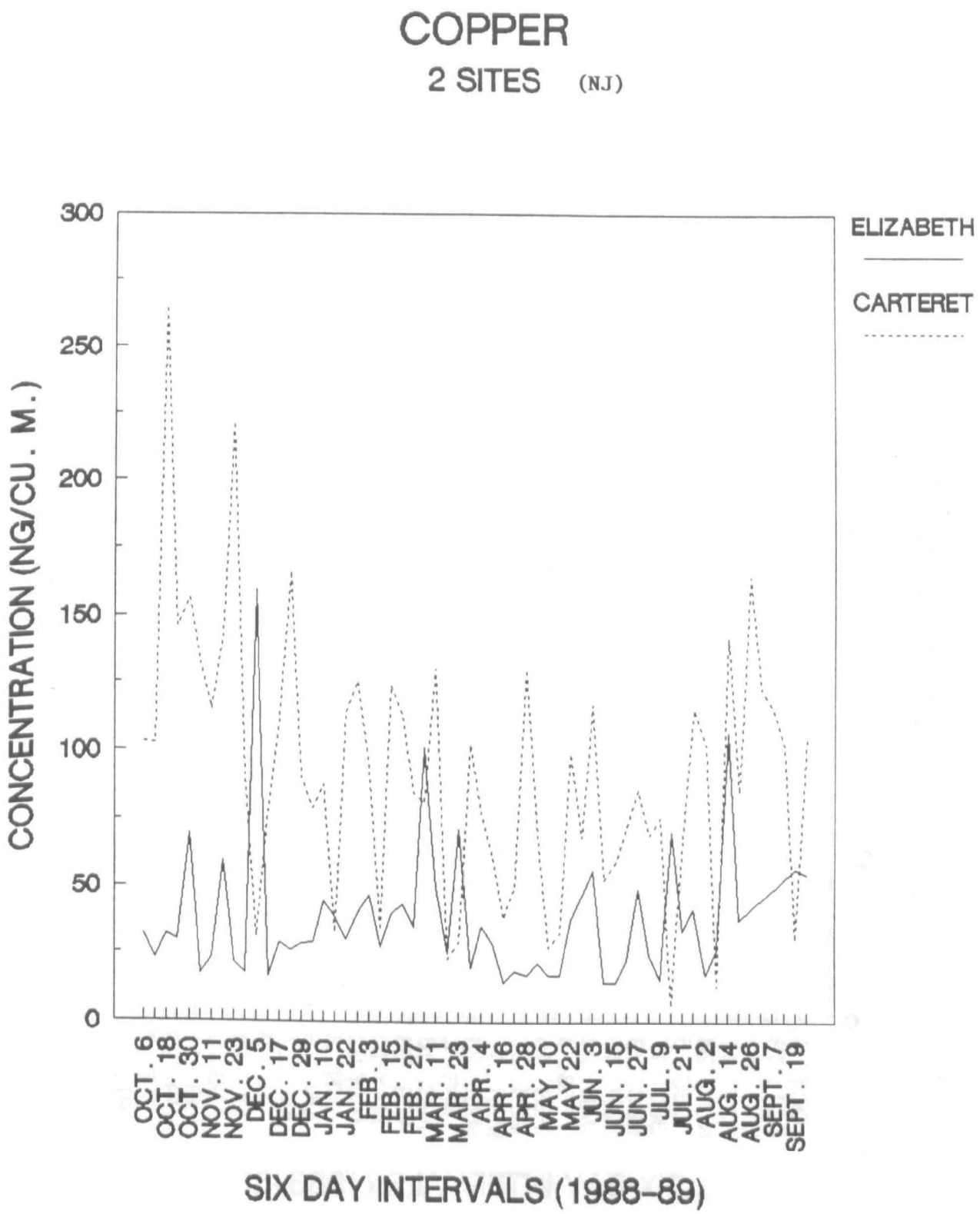


FIGURE IIIB-46

COPPER

2 SITES (NY)

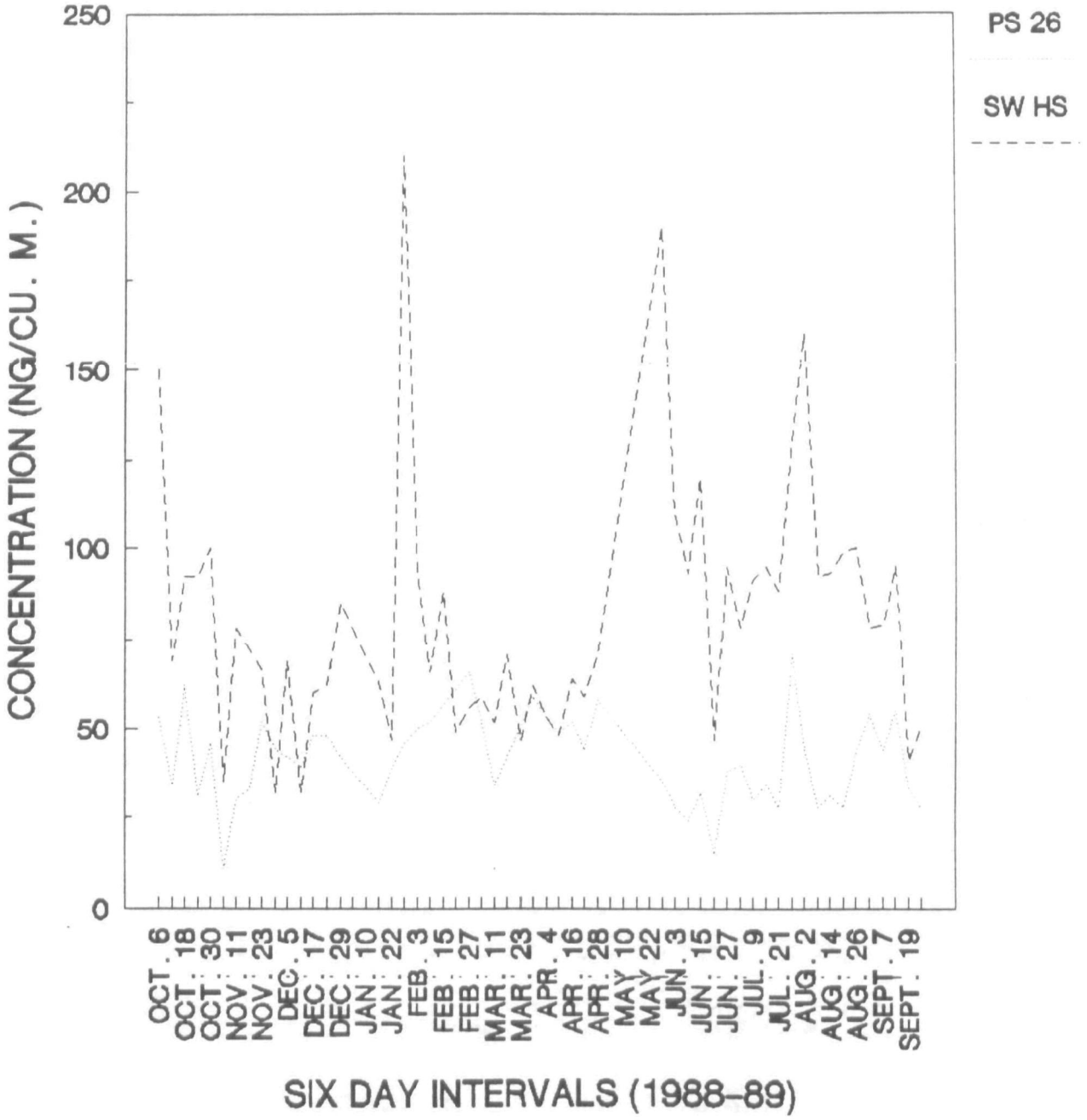


FIGURE IIIB-47

ZINC

4 SITES

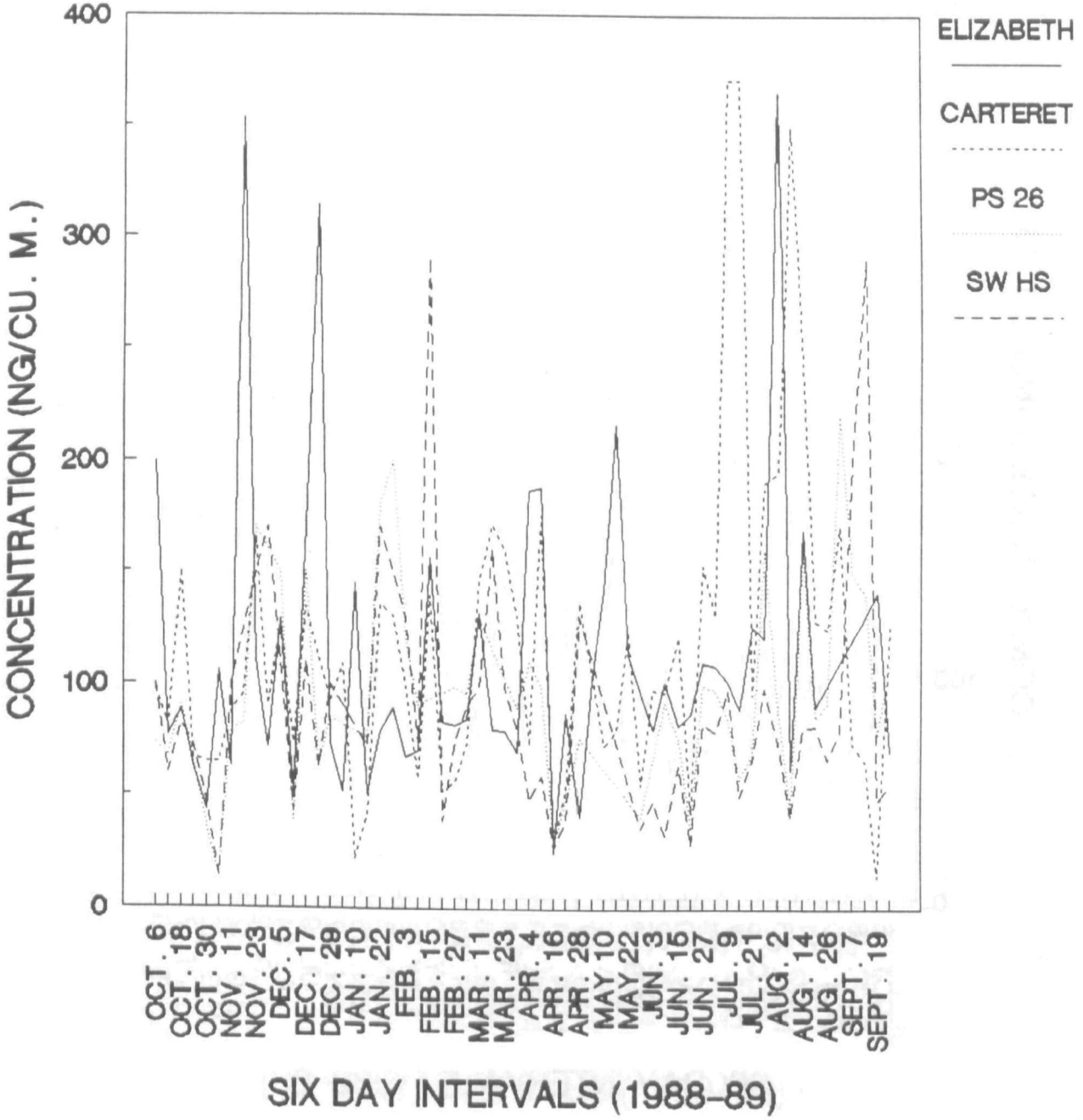


FIGURE IIIB-48

ZINC

2 SITES (NJ)

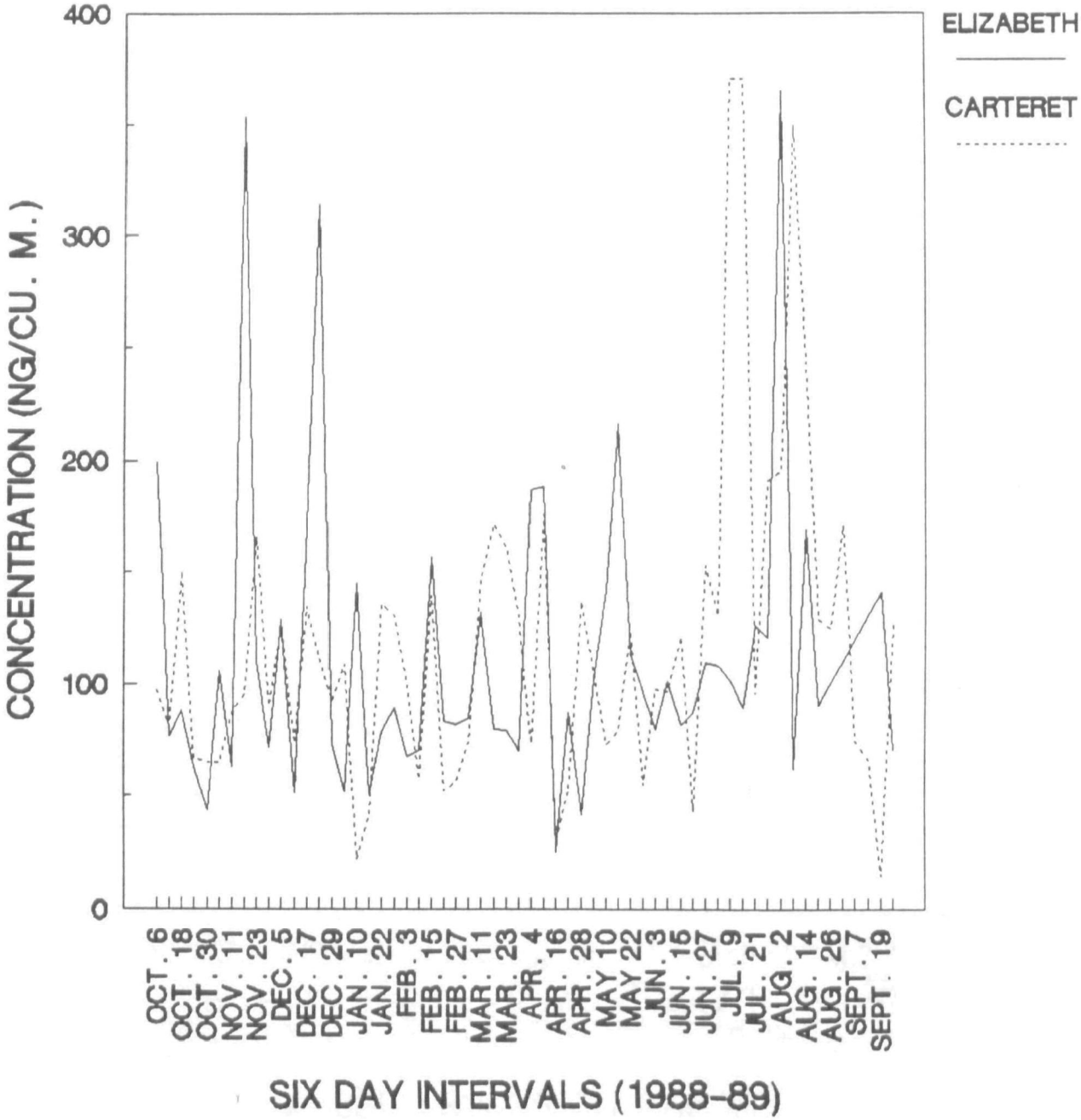


FIGURE IIIB-49

ZINC

2 SITES (NY)

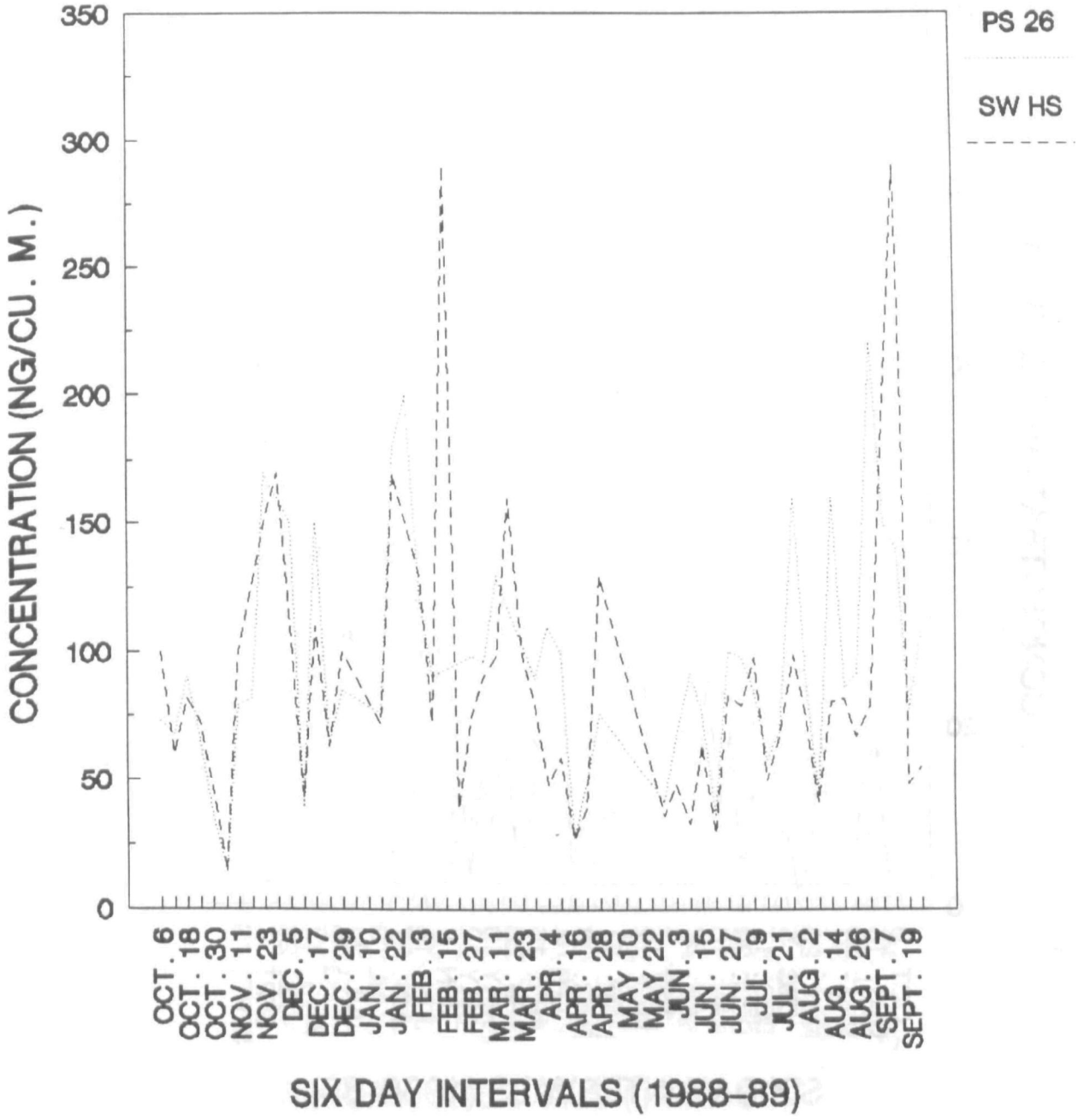


FIGURE IIIB-50

CADMIUM

4 SITES

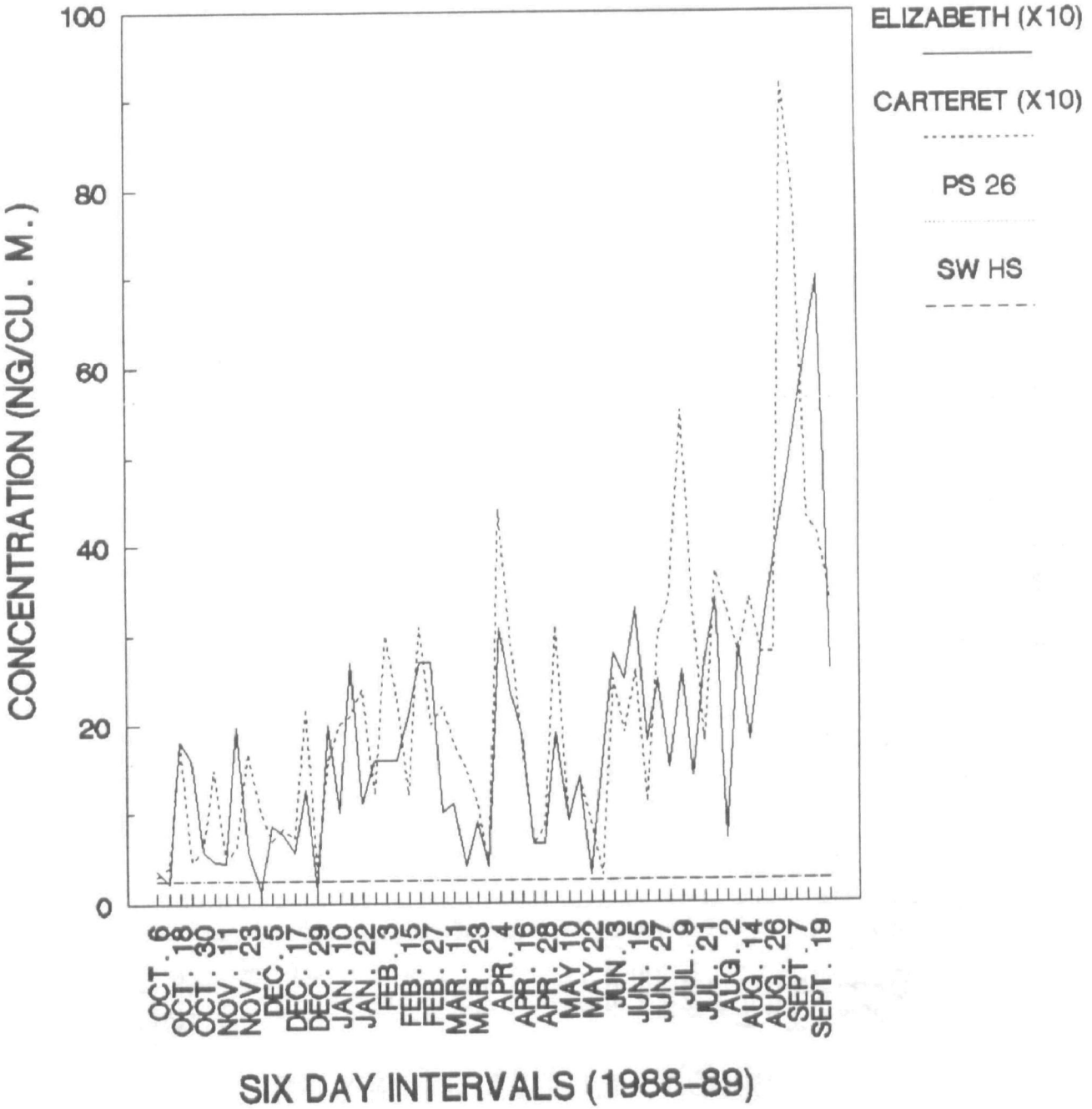


FIGURE IIIB-51

MERCURY

2 SITES

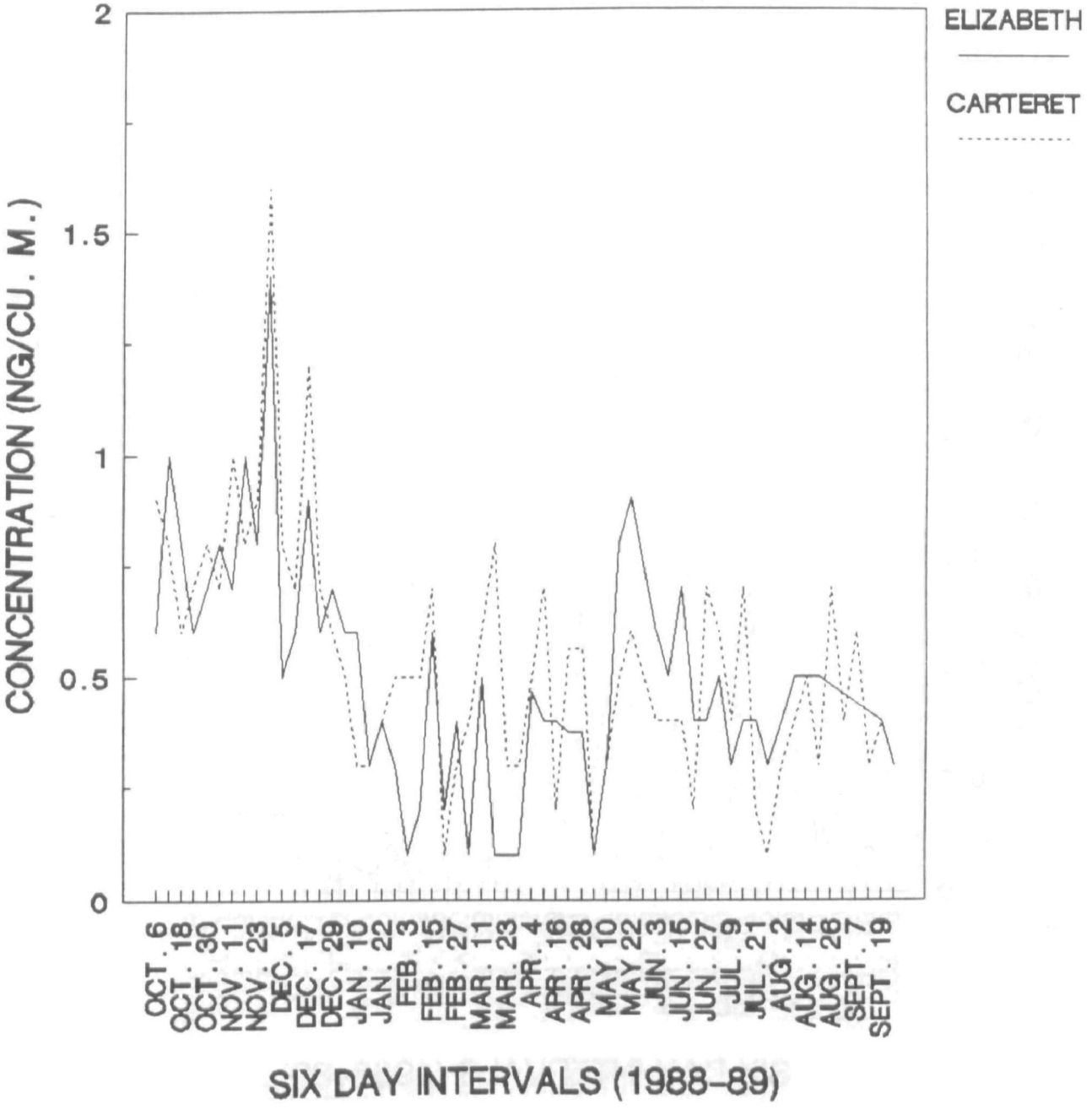


FIGURE IIIB-52

BAP

5 SITES

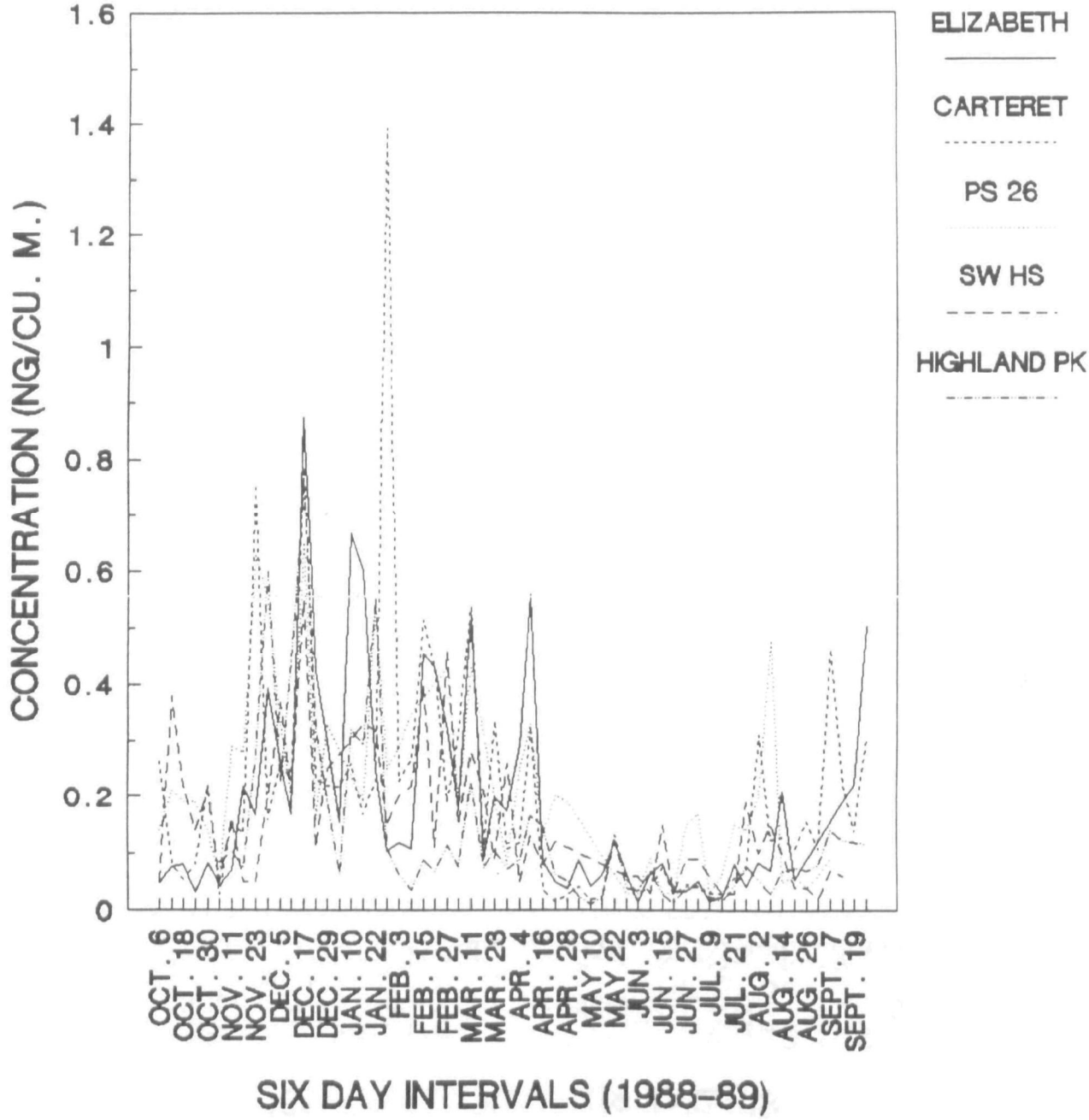


FIGURE IIB-53

BAP

3 SITES (NJ)

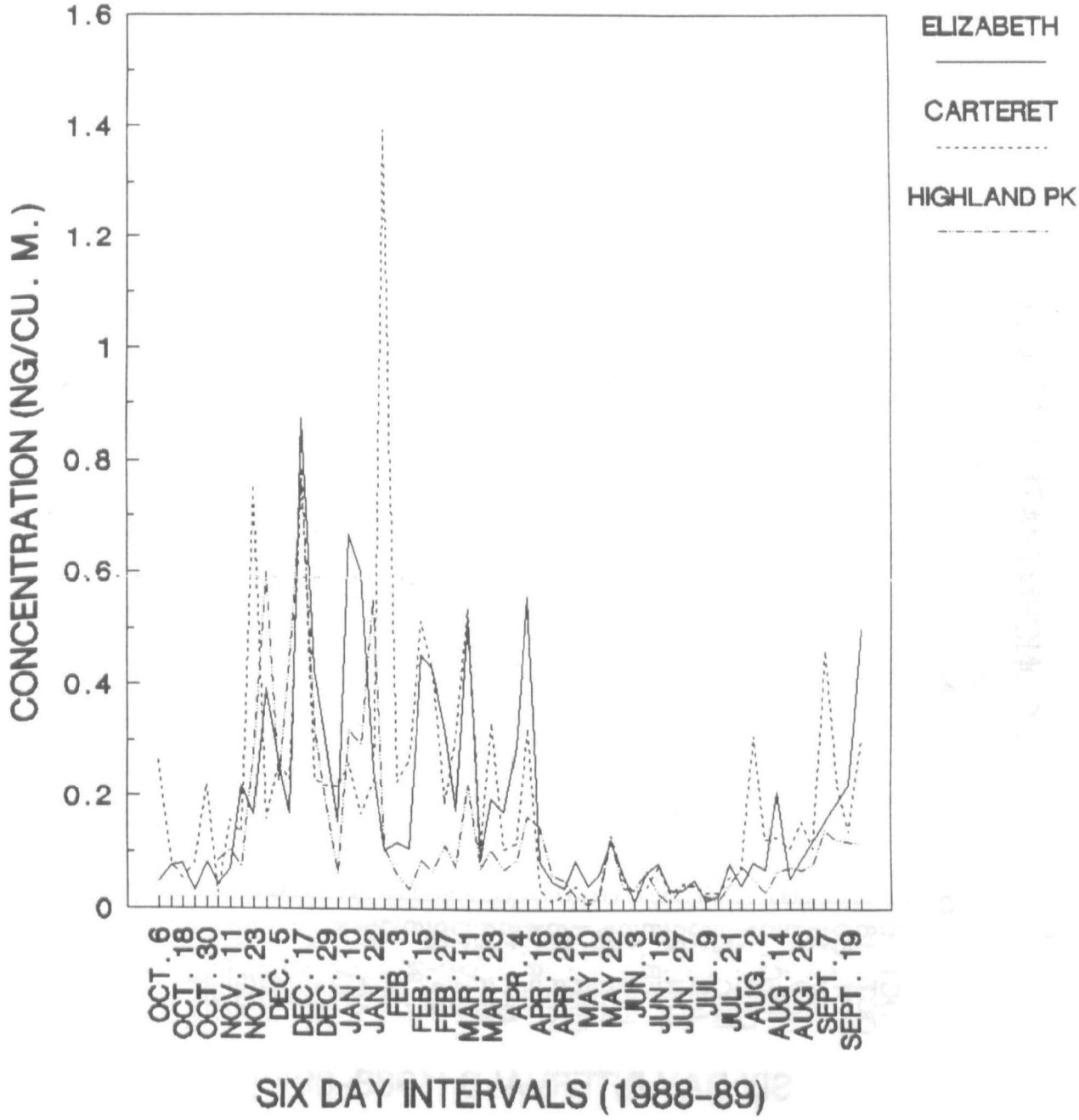


FIGURE IIIB-54

BAP

HIGHLAND PARK

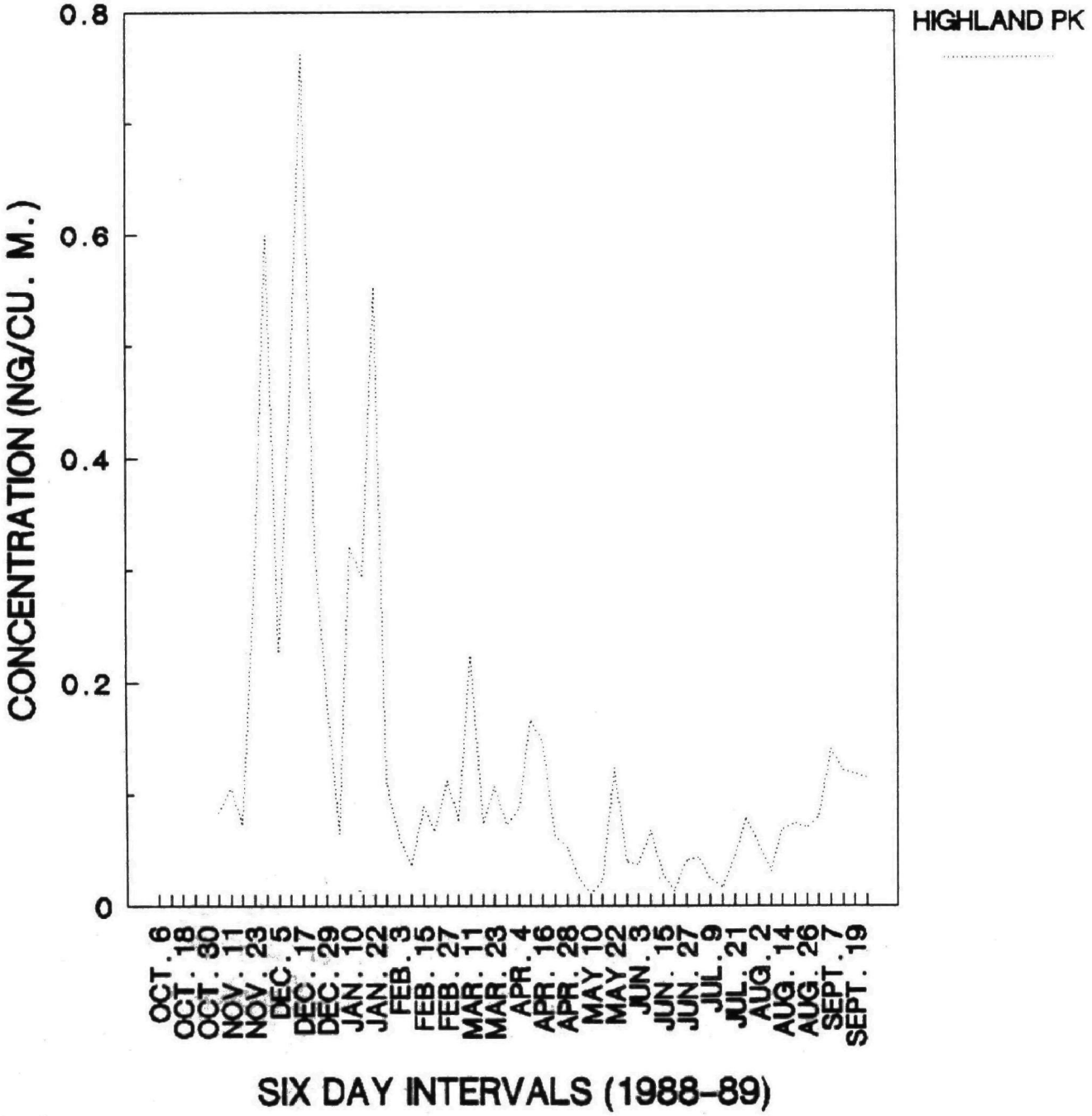
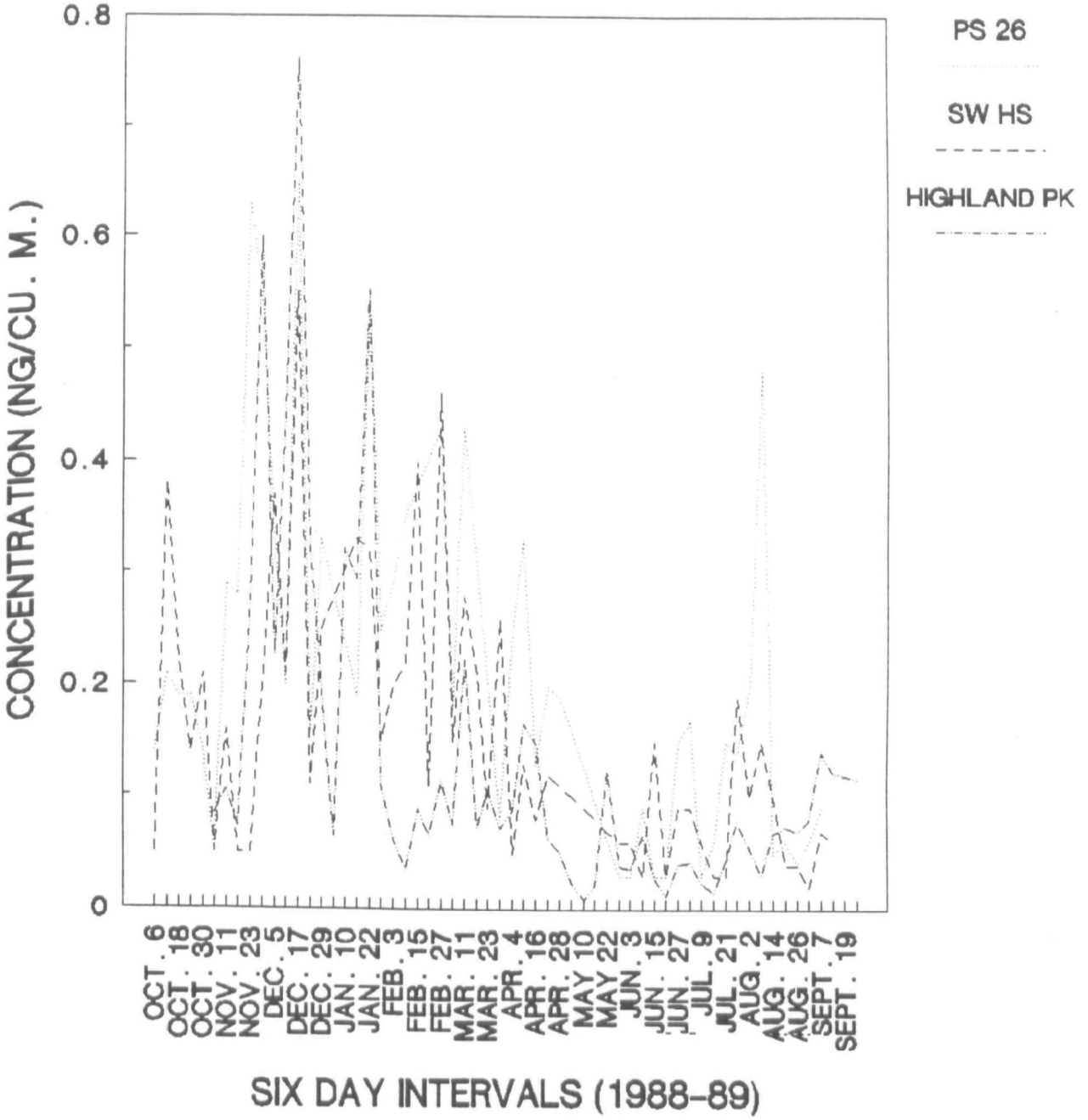


FIGURE IIIB-55

BAP

3 SITES



APPENDICES

APPENDIX A

QUALITY ASSURANCE SUMMARY FOR METALS, BENZO[α]PYRENE, AND FORMALDEHYDE

1. INTRODUCTION

Each sampling organization was responsible for its own Quality Assurance (QA). However, to ensure that appropriate quality assurance methods were selected and followed, the QA procedures of the organizations submitting monitoring data for the project were reviewed by the QA Subcommittee.

2. METALS

Quality Assurance reports were received from both the New Jersey Institute of Technology (NJIT) and the New York State Department of Health (NYSDOH). NJIT's samples were collected by its own staff, whereas the NYSDOH samples were collected by the New York State Department of Environmental Conservation (NYSDEC).

2.1 Sample Collection

2.1.1 NYSDEC/NYSDOH

Sample collection and sampler calibration were conducted by NYSDEC under the guidelines and procedures established in its Ambient Air Quality Assurance Manual.

2.1.2 NJIT

NJIT's samplers were calibrated by the New Jersey Department of Environmental Protection according to the methods outlined in its Ambient Air Monitoring Quality Assurance Manual. Sample collection was done by NJIT staff.

2.2 Analytical Results

Table IIIB-A1 provides a summary of the QA disposition of the metals data from NYSDEC/NYSDOH and from NJIT.

2.2.1 NYSDOH/NYSDEC

The analytical QA data provided by NYSDOH showed excellent precision and recovery results for all compounds tested. However, these data were generated using dilute solutions of metals spiked onto filters. Field samples, however, consist of particulates and not liquids, and the % recovery of metals from particulates may be lower than % recoveries from liquid standards.

To ascertain percent recoveries for particulate metals, an urban dust Standard Reference Material (SRM) can be run. This SRM, obtainable from the National Institute for Standards and Technology (NIST), contains known trace amounts of many of the metals monitored in the SI/NJ UATAP. However, NYSDOH did not run these standards until recently.

NYSDOH results with the NIST urban dust SRM showed recoveries of cadmium, copper, zinc, and lead to be 95% of the SRM concentration. Barium and manganese recoveries were within 82% of the standard. Iron and nickel recoveries were within 75% of the standard, and vanadium was within 63% of the NIST SRM. Chromium recoveries were only 18% of the standard. Precision of analysis for all compounds was uniformly excellent.

As a result of these findings, the data were treated as follows:

1. All NYSDOH chromium data were rejected and removed from the project data base.

2. The vanadium, iron, and nickel data were accepted, but were caveated since their recoveries were below 80%. The values reported for these compounds should be viewed as the minimum values known to be present.

3. The cadmium, copper, zinc, barium, lead, and manganese data were accepted. The results for arsenic, beryllium, cobalt, mercury, and molybdenum were approved with the understanding that their accuracy can not be verified with NIST particulate SRMs because the SRMs do not exist.

The percent recovery data for NYSDOH results with SRMs are presented in Table IIIB-A2, % Recovery From Urban Dust - Ultrasonic Bath Digestion.

2.2.2 NJIT

NJIT submitted QA data for 10 compounds: cadmium, chromium, cobalt, copper, iron, lead, mercury, manganese, nickel, and zinc. A serious concern with the NJIT report was the lack of specific information pertaining to background contamination levels in blank samples or the results of calibration checks. Also, data to support estimates of precision were provided only for mercury. Furthermore, NJIT asserted that absorbance vs. response curves were generated for all compounds analyzed, but only provided these curves for mercury and lead.

Mitigating these concerns, however, were NJIT's acceptable analytical results for EPA particulate lead standards and the NIST urban dust particulate SRMs. The NIST samples were analyzed for 8 compounds: cadmium, chromium, copper, iron, lead, nickel, manganese, and zinc. Recoveries using these standards were typically in the range 85-100%. Therefore, NJIT's QA, though somewhat lacking in documentation, was regarded as sufficient for inclusion of data for the eight compounds named above into the project data base.

Cobalt, although present in the NIST standard, was reported by NJIT as below its detection limit. Since no other data, including precision, calibration, and blank level data, were included in the NJIT report, the cobalt data were excluded from the project data base.

The submitted mercury data, although somewhat lacking in detail, did include acceptable precision and calibration curve data. Therefore, the mercury data were accepted for use in the project data base. No standard was available from NIST or EPA for mercury.

NJIT collected and reported data for vanadium, selenium, and molybdenum during the course of the project. As stated in its QA report, NJIT was unable to provide QA information for vanadium. Selenium and molybdenum were not mentioned in NJIT's QA report at all. Therefore monitoring data for these three compounds were excluded from the project data base.

3. BENZO[α]PYRENE

Benzo[α]pyrene (BaP) sampling in the project was conducted by NJIT and NYSDEC. Sample analysis was done by NJIT and the NYSDOH.

3.1 Sample Collection

3.1.1. NYSDEC/NYSDOH

Calibration of BaP samplers was done by NYSDEC in accordance with the NYSDEC guidance for flow calibration of high-volume particulate samplers as outlined in its Ambient Air Monitoring Quality Assurance Manual.

3.1.2 NJIT

Calibration of B(a)P samplers was done by the NJDEP according to the procedures outlined in its Ambient Air Monitoring Quality Assurance Manual.

3.2 Analytical Results

3.2.1 NYSDOH

Analysis of all NYSDEC BaP samples was done by NYSDOH. The results of NYSDOH's analyses are summarized below.

1. Filters spiked with 100 ng of BaP had percent recoveries averaging 49%, a sample standard deviation of 15.0, and a coefficient of variation of 30.6.

2. Filters spiked with 200 ng of BaP had percent recoveries averaging 84%, a standard deviation of 19.0, and a coefficient of variation of 22.6.

3. The detection levels for BaP were not presented.

BaP concentrations for the summer months were reported by NYSDOH to average 0.10 ng/m^3. Assuming a 1600-m^3 sample, typical for a hi-vol sampler, there would be 160 ng of BaP collected on a filter during an average summer day. In its procedure, NYSDOH digests half of each filter; therefore, the total BaP extracted from the half-filter would be 80 ng. Since NYSDOH's recoveries at this level averaged 49%, it appears that NYSDOH's results are negatively biased by at least 51% on average.

Furthermore, NYSDOH's results are derived from liquid standards spiked onto filters. The hi-vol/glass fiber filter collection method used in the SI/NJ UATAP, however, gathers particulate matter, where BaP recovery is often less than that obtained when using liquid standards. Results with particulate standards, such as the NIST Urban Dust Standard Reference Material, were not provided by NYSDOH.

As a result of these findings, the NYSDOH BaP data is included in the project data base with a caveat that the data provided reflect the minimum concentration of BaP present.

3.2.2 NJIT

The QA data submitted by NJIT showed ± 20% recoveries of BaP from the Urban Dust Standard Reference Material (SRM's - obtained from the National Institiute for Technology and Standards). NJIT's detection limits were well into the sub-part per trillion range. Recoveries at these low concentrations were >95% using liquid SRM's. Simulated samples using liquids spiked onto filters at concentrations of approximately 0.05 ppt yielded 87-90% recovery.

Duplicate sample analyses showed standard deviations of 1.12%.

In view of these results, the NJIT BaP data were accepted for inclusion in the project data base.

4. FORMALDEHYDE

Formaldehyde sampling in the project was conducted by the New Jersey Institute of Technology (NJIT) and the New York State Department of Environmental Conservation (NYSDEC). Sample analysis was done by NSI, an EPA contractor, and by NJIT.

4.1 Sample Collection

4.1.1 NYSDEC

Calibration of formaldehyde sample flow was done in accordance with the NYSDEC procedures for flow calibration as outlined in its Ambient Air Monitoring Quality Assurance Manual.

4.1.2 NJIT

Calibration of NJIT formaldehyde samplers was done in accordance with the same protocols used for its volatile organic compound samplers.

4.2 Analytical Results

4.2.1 NYSDEC samples

Analysis of all NYSDEC formaldehyde samples was done by NSI under contract with EPA at Research Triangle Park. Quality Assurance data were provided by Dr. Silvestre Tejada at EPA/AREAL, who had the oversight responsibility for formaldehyde analysis conducted by NSI for EPA. NSI analyzed SI/NJ UATAP samples concurrently with other samples as part of a national EPA formaldehyde monitoring effort. The QA data presented by Dr. Tejada did not come from SI/NJ UATAP samples directly, but rather from NSI's results for the national studies that were done concurrently with the SI/NJ UATAP.

Since the analytical protocols, personnel, instrumentation, management, and EPA oversight were the same for these national projects as for the SI/NJ UATAP, this QA information is considered valid for assessing the quality of the analytical data provided for the SI/NJ UATAP. Direct QA information for the SI/NJ UATAP formaldehyde data is unavailable due to unresolved sample log number difficulties.

The QA information provided by Dr. Tejada shows the following:

1. Blank levels in formaldehyde tubes were always below 0.15 ppb.

2. NSI analytical accuracy was assessed by EPA/AREAL in a cross check with 2 other laboratories. Results of the study showed a 2.1 % relative standard deviation among the laboratories.

3. Precision was estimated by evaluating collocated samples from field studies. In most cases, the collocated samples were within 10% of each other. In cases where this was not the case, EPA concluded that the results were due to sampler miscalibration or sample misidentification. The data provided lends support to this view.

As a result of these findings, all of the NYSDEC-collected/NSI-analyzed formaldehyde data were accepted for inclusion in the project data base.

4.2.2 NJIT samples

Analyses of NJIT's formaldehyde samples were conducted partly by NSI and partly in-house by NJIT. NSI's analytical QA was addressed in detail above; the same findings apply to the NJIT-gathered/NSI-analyzed samples. However, no QA information was provided for samples analyzed in-house by NJIT.

Therefore, (1) the NJIT-gathered/NSI-analyzed samples were accepted for inclusion in the project data base; however, (2) samples analyzed by NJIT were rejected from inclusion in the project data base since appropriate QA information is not available.

Table IIIB-A1: SI/NJ UATAP Metals Data QA Status as of 12/4/91

Metal	QA Status	
	NJIT	NYSDOH/NYSDEC
Arsenic[1]	No analysis for this compound	Approved
Barium	No analysis for this compound	Approved
Beryllium[1]	No analysis for this compound	Approved[2]
Cadmium	Approved	Approved
Chromium	Approved	Rejected
Cobalt[1]	Insufficient data	Approved
Copper	Approved	Approved
Iron	Approved	Approved[3]
Lead	Approved	Approved
Manganese	Approved	Approved
Mercury[1]	Approved	No analysis for this compound
Molybdenum[1]	Insufficient data	Approved
Nickel	Approved	Approved[3]
Vanadium	Insufficient data	Approved[3]
Zinc	Approved	Approved
Benzo[α]pyrene	Approved	Approved[4]

1 No particulate Standard Reference Material (SRM) was available.

2 This compound was not detected in samples.

3 Recoveries of these compounds from the SRM were in the 63%-76% range. They were acceptable for use in the project, however they represent a minimum of the amount that may actually have been present.

4 Recoveries of this compound from the SRM averaged 49%. They reported concentrations are acceptable for use in the project, however they represent a minimum of the amount that may actually have been present.

Table IIIB-A2

% RECOVERY FROM URBAN DUST - ULTRASONIC BATH DIGESTION

SAMPLE	CADMIUM % RECOVERY	COPPER % RECOVERY	ZINC % RECOVERY	IRON % RECOVERY	BARIUM % RECOVERY
1	96.9	94.5	97.1	74.8	82.6
2	95.1	97.0	98.4	76.6	83.8
3	91.5	94.7	96.6	76.0	82.2
4	95.0	96.1	98.3	76.1	81.7
5	109.2	95.7	98.0	75.6	83.5
6	96.9	95.0	97.1	74.8	81.7
7	94.5	96.6	97.3	75.7	81.9
8	97.1	97.1	97.9	76.0	82.3
9	96.3	96.6	98.2	75.5	82.9
10	97.2	94.9	100.5	75.8	81.8
MEAN	97.0	95.8	97.9	75.7	82.4
STD. DEV.	4.40	0.94	1.03	0.53	0.71
U 95% CL	99.7	96.4	98.6	76.0	82.9
L 95% CL	94.2	95.2	97.3	75.4	82.0

SAMPLE	NICKEL % RECOVERY	VANADIUM % RECOVERY	LEAD % RECOVERY	CHROMIUM % RECOVERY	MANGANESE % RECOVERY
1	76.3	62.7	100.6	18.4	86.6
2	80.5	64.7	101.4	18.1	87.4
3	74.2	65.2	101.1	18.2	84.8
4	74.6	62.8	102.4	18.3	86.3
5	75.8	62.4	102.4	18.8	87.2
6	76.7	62.7	100.6	18.4	85.6
7	73.9	62.2	101.6	18.2	86.4
8	79.0	63.2	102.6	18.1	86.9
9	77.3	63.5	102.3	18.8	87.1
10	73.9	62.7	101.7	18.2	87.0
MEAN	76.2	63.2	101.7	18.4	86.5
STD. DEV.	2.12	0.94	0.71	0.25	0.76
U 95% CL	77.5	63.8	102.1	18.5	87.0
L 95% CL	74.9	62.6	101.2	18.2	86.1

APPENDIX B

DATA SUMMARIES BY QUARTERLY AVERAGE

Table IIIB-B1

ARSENIC

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
PS 26	2.7	PS 26	3.5	PS 26	11.1	SUSAN WAGNER HS	12.9
SUSAN WAGNER HS	1.9	SUSAN WAGNER HS	2.6	SUSAN WAGNER HS	9.5	PS 26	11.6
HIGHLAND PARK		HIGHLAND PARK		HIGHLAND PARK		CARTERET	
CARTERET		CARTERET		CARTERET		ELIZABETH	
ELIZABETH		ELIZABETH		ELIZABETH		HIGHLAND PARK	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	14.5	PS 26	5.1	PS 26	1.4	SUSAN WAGNER HS	1.0
PS 26	11.4	SUSAN WAGNER HS	1.4	SUSAN WAGNER HS	1.1	PS 26	1.0
CARTERET		HIGHLAND PARK		HIGHLAND PARK		CARTERET	
ELIZABETH		CARTERET		CARTERET		ELIZABETH	
HIGHLAND PARK		ELIZABETH		ELIZABETH		HIGHLAND PARK	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B2

CADMIUM

QUARTER BEGINNING OCTOBER 1987		QUARTER BEGINNING JANUARY 1988		QUARTER BEGINNING APRIL 1988		QUARTER BEGINNING JULY 1988	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
PS 26	3.0	CARTERET	12.6	CARTERET	4.5	SUSAN WAGNER HS	3.2
SUSAN WAGNER HS	2.7	PS 26	3.0	PS 26	3.2	CARTERET	2.7
CARTERET	1.6	SUSAN WAGNER HS	2.5	SUSAN WAGNER HS	2.9	ELIZABETH	2.4
HIGHLAND PARK		HIGHLAND PARK		ELIZABETH	1.6	PS 26	2.2
ELIZABETH		ELIZABETH		HIGHLAND PARK		HIGHLAND PARK	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

QUARTER BEGINNING OCTOBER 1988		QUARTER BEGINNING JANUARY 1989		QUARTER BEGINNING APRIL 1989		QUARTER BEGINNING JULY 1989	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
CARTERET	9.3	SUSAN WAGNER HS	2.3	PS 26	2.5	CARTERET	4.1
ELIZABETH	0.8	HIGHLAND PARK	2.3	SUSAN WAGNER	2.5	PS 26	3.0
HIGHLAND PARK		PS 26	2.0	CARTERET	1.9	ELIZABETH	2.7
SUSAN WAGNER HS		CARTERET	1.8	ELIZABETH	1.8	SUSAN WAGNER HS	2.5
PS 26		ELIZABETH	1.5	HIGHLAND PARK	1.7	HIGHLAND PARK	2.2
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B3

COBALT

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
PS 26	3.0	PS 26	3.0	PS 26	3.2	SUSAN WAGNER HS	2.6
SUSAN WAGNER HS	2.7	SUSAN WAGNER HS	2.5	SUSAN WAGNER HS	2.9	PS 26	2.2
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	2.5	SUSAN WAGNER HS	2.5	SUSAN WAGNER HS	2.5	PS 26	3.0
PS 26	2.0	PS 26	2.0	PS 26	2.5	SUSAN WAGNER HS	2.5
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B4

COPPER

QUARTER BEGINNING OCTOBER 1987		QUARTER BEGINNING JANUARY 1988		QUARTER BEGINNING APRIL 1988		QUARTER BEGINNING JULY 1988	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	83.8	SUSAN WAGNER HS	90.7	SUSAN WAGNER HS	121.1	CARTERET	105.4
PS 26	59.2	CARTERET	78.4	CARTERET	113.5	SUSAN WAGNER HS	95.3
CARTERET	47.0	PS 26	45.9	ELIZABETH	76.4	PS 26	75.4
ELIZABETH		HIGHLAND PARK		PS 26	50.5	ELIZABETH	22.9
HIGHLAND PARK		ELIZABETH		HIGHLAND PARK		HIGHLAND PARK	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

QUARTER BEGINNING OCTOBER 1988		QUARTER BEGINNING JANUARY 1989		QUARTER BEGINNING APRIL 1989		QUARTER BEGINNING JULY 1989	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	79.4	CARTERET	83.7	SUSAN WAGNER HS	86.3	SUSAN WAGNER HS	91.3
PS 26	39.6	SUSAN WAGNER HS	74.1	CARTERET	69.1	CARTERET	88.1
ELIZABETH	26.6	PS 26	47.4	PS 26	38.9	HIGHLAND PARK	49.6
CARTERET		ELIZABETH	42.8	HIGHLAND PARK	32.2	ELIZABETH	44.3
HIGHLAND PARK		HIGHLAND PARK	22.6	ELIZABETH	25.0	PS 26	39.4
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B5

IRON

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
PS 26	1168.0	PS 26	875.1	PS 26	1468.0	PS 26	1433.0
CARTERET	519.7	CARTERET	752.6	SUSAN WAGNER HS	807.8	SUSAN WAGNER HS	888.0
SUSAN WAGNER HS	511.9	SUSAN WAGNER HS	522.8	CARTERET	805.4	ELIZABETH	495.6
HIGHLAND PARK		HIGHLAND PARK		ELIZABETH	661.0	HIGHLAND PARK	
ELIZABETH		ELIZABETH		HIGHLAND PARK		CARTERET	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
PS 26	738.0	PS 26	970.0	PS 26	800.0	PS 26	1110.0
SUSAN WAGNER HS	537.3	SUSAN WAGNER HS	690.0	CARTERET	689.7	CARTERET	795.4
CARTERET	426.0	CARTERET	508.1	HIGHLAND PARK	656.7	SUSAN WAGNER HS	710.0
ELIZABETH	308.4	ELIZABETH	401.5	SUSAN WAGNER HS	610.0	ELIZABETH	552.8
HIGHLAND PARK		HIGHLAND PARK	376.4	ELIZABETH	493.1	HIGHLAND PARK	406.3
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B6

LEAD

QUARTER BEGINNING OCTOBER 1987		QUARTER BEGINNING JANUARY 1988		QUARTER BEGINNING APRIL 1988		QUARTER BEGINNING JULY 1988	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
PS 26	82.5	CARTERET	118.7	PS 26	44.8	PS 26	55.3
CARTERET	69.0	PS 26	46.4	SUSAN WAGNER HS	44.6	SUSAN WAGNER HS	49.3
SUSAN WAGNER HS	58.2	SUSAN WAGNER HS	31.1	CARTERET	30.8	ELIZABETH	31.3
HIGHLAND PARK		HIGHLAND PARK		ELIZABETH	21.6	HIGHLAND PARK	
ELIZABETH		ELIZABETH		HIGHLAND PARK		CARTERET	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

QUARTER BEGINNING OCTOBER 1988		QUARTER BEGINNING JANUARY 1989		QUARTER BEGINNING APRIL 1989		QUARTER BEGINNING JULY 1989	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
CARTERET	38.1	HIGHLAND PARK	91.1	CARTERET	42.8	PS 26	45.7
ELIZABETH	38.1	PS 26	40.1	ELIZABETH	38.1	CARTERET	38.3
SUSAN WAGNER HS	36.1	SUSAN WAGNER HS	39.5	HIGHLAND PARK	26.6	SUSAN WAGNER HS	33.7
PS 26	35.7	CARTERET	26.4	PS 26	24.5	ELIZABETH	23.5
HIGHLAND PARK		ELIZABETH	22.6	SUSAN WAGNER HS	14.4	HIGHLAND PARK	20.5
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B7

MANGANESE

QUARTER BEGINNING OCTOBER 1987		QUARTER BEGINNING JANUARY 1988		QUARTER BEGINNING APRIL 1988		QUARTER BEGINNING JULY 1988	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
CARTERET	29.5	CARTERET	27.0	PS 26	33.6	PS 26	34.5
PS 26	26.6	PS 26	21.0	CARTERET	32.2	SUSAN WAGNER HS	22.1
SUSAN WAGNER HS	15.1	SUSAN WAGNER HS	15.1	ELIZABETH	23.1	ELIZABETH	17.9
HIGHLAND PARK		HIGHLAND PARK		SUSAN WAGNER HS	21.8	HIGHLAND PARK	
ELIZABETH		ELIZABETH		HIGHLAND PARK		CARTERET	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

QUARTER BEGINNING OCTOBER 1988		QUARTER BEGINNING JANUARY 1989		QUARTER BEGINNING APRIL 1989		QUARTER BEGINNING JULY 1989	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
CARTERET	18.9	PS 26	22.3	CARTERET	25.7	CARTERET	22.1
PS 26	15.9	CARTERET	20.3	HIGHLAND PARK	15.6	PS 26	21.1
ELIZABETH	14.9	SUSAN WAGNER HS	18.6	PS 26	15.5	SUSAN WAGNER HS	15.2
SUSAN WAGNER HS	13.9	ELIZABETH	15.6	ELIZABETH	15.0	ELIZABETH	13.4
HIGHLAND PARK		HIGHLAND PARK	12.4	SUSAN WAGNER HS	12.2	HIGHLAND PARK	11.6
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B8

MERCURY

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
CARTERET		CARTERET	0.5	ELIZABETH	0.9	ELIZABETH	1.2
ELIZABETH		ELIZABETH		CARTERET	0.6	CARTERET	
HIGHLAND PARK		HIGHLAND PARK		SUSAN WAGNER HS		HIGHLAND PARK	
SUSAN WAGNER HS		SUSAN WAGNER HS		HIGHLAND PARK		SUSAN WAGNER HS	
PS 26		PS 26		PS 26		PS 26	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
CARTERET	0.8	HIGHLAND PARK	0.5	HIGHLAND PARK	0.5	CARTERET	0.4
ELIZABETH	0.7	CARTERET	0.4	ELIZABETH	0.5	ELIZABETH	0.4
HIGHLAND PARK		ELIZABETH	0.3	CARTERET	0.4	HIGHLAND PARK	0.3
SUSAN WAGNER HS		SUSAN WAGNER HS		SUSAN WAGNER HS		SUSAN WAGNER HS	
PS 26		PS 26		PS 26		PS 26	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B9

MOLYBDENUM

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
PS 26	12.4	PS 26	12.3	PS 26	12.9	SUSAN WAGNER HS	10.8
SUSAN WAGNER HS	11.1	SUSAN WAGNER HS	10.8	SUSAN WAGNER HS	11.0	PS 26	8.9
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	10.7	SUSAN WAGNER HS	9.2	PS 26	9.8	PS 26	12.1
PS 26	7.9	PS 26	7.7	SUSAN WAGNER HS	9.5	SUSAN WAGNER HS	9.5
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B10

NICKEL

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
CARTERET	30.6	CARTERET	76.3	CARTERET	29.3	ELIZABETH	44.5
PS 26	29.4	PS 26	27.7	PS 26	16.6	PS 26	16.6
SUSAN WAGNER HS	15.6	SUSAN WAGNER HS	26.4	ELIZABETH	13.6	SUSAN WAGNER HS	14.7
HIGHLAND PARK		HIGHLAND PARK		SUSAN WAGNER HS	13.6	HIGHLAND PARK	
ELIZABETH		ELIZABETH		HIGHLAND PARK		CARTERET	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
PS 26	15.1	PS 26	33.7	CARTERET	32.4	CARTERET	32.1
SUSAN WAGNER HS	13.4	SUSAN WAGNER HS	32.2	ELIZABETH	30.2	ELIZABETH	30.7
HIGHLAND PARK		HIGHLAND PARK	17.3	HIGHLAND PARK	29.0	HIGHLAND PARK	22.2
CARTERET		CARTERET	13.9	PS 26	18.0	PS 26	17.1
ELIZABETH		ELIZABETH	13.5	SUSAN WAGNER HS	12.3	SUSAN WAGNER HS	16.1
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B11

VANADIUM

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
PS 26	14.8	PS 26	23.9	PS 26	15.5	PS 26	14.9
SUSAN WAGNER HS	12.8	SUSAN WAGNER HS	19.4	SUSAN WAGNER HS	11.6	SUSAN WAGNER HS	9.3
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
PS 26	15.2	SUSAN WAGNER HS	22.5	PS 26	16.0	PS 26	17.0
SUSAN WAGNER HS	9.3	PS 26	19.8	SUSAN WAGNER HS	10.1	SUSAN WAGNER HS	16.2
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B12

ZINC

QUARTER BEGINNING OCTOBER 1987		QUARTER BEGINNING JANUARY 1988		QUARTER BEGINNING APRIL 1988		QUARTER BEGINNING JULY 1988	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
CARTERET	182.0	PS 26	107.8	PS 26	106.8	PS 26	128.2
PS 26	145.5	CARTERET	89.8	SUSAN WAGNER HS	95.1	CARTERET	121.5
SUSAN WAGNER HS	112.4	SUSAN WAGNER HS	89.5	CARTERET	90.3	SUSAN WAGNER HS	113.9
HIGHLAND PARK		HIGHLAND PARK		ELIZABETH	78.4	ELIZABETH	82.8
ELIZABETH		ELIZABETH		HIGHLAND PARK		HIGHLAND PARK	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

QUARTER BEGINNING OCTOBER 1988		QUARTER BEGINNING JANUARY 1989		QUARTER BEGINNING APRIL 1989		QUARTER BEGINNING JULY 1989	
SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)	SITE	ARITH. MEAN (ng/m3)
CARTERET	117.9	PS 26	120.2	HIGHLAND PARK	134.9	SUSAN WAGNER HS	194.7
ELIZABETH	93.0	SUSAN WAGNER HS	117.5	ELIZABETH	105.9	CARTERET	173.1
SUSAN WAGNER HS	88.4	CARTERET	101.5	CARTERET	93.2	ELIZABETH	131.3
PS 26	83.8	ELIZABETH	88.1	PS 26	70.4	PS 26	109.3
HIGHLAND PARK		HIGHLAND PARK	77.8	SUSAN WAGNER HS	54.2	HIGHLAND PARK	76.5
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B13

CHROMIUM

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
CARTERET	22.7	CARTERET	37.9	ELIZABETH	33.9	CARTERET	3.1
HIGHLAND PARK		HIGHLAND PARK		CARTERET	24.7	ELIZABETH	2.3
ELIZABETH		ELIZABETH		HIGHLAND PARK		HIGHLAND PARK	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
ELIZABETH	3.9	CARTERET	13.3	CARTERET	51.8	CARTERET	16.5
HIGHLAND PARK		ELIZABETH	12.9	ELIZABETH	35.8	ELIZABETH	8.4
CARTERET		HIGHLAND PARK	9.5	HIGHLAND PARK	20.5	HIGHLAND PARK	5.2

Table IIIB-B14

BENZO(A)PYRENE

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
CARTERET	0.36	CARTERET	0.15	CARTERET	0.07	CARTERET	0.09
ELIZABETH		ELIZABETH		ELIZABETH	0.06	ELIZABETH	0.03
HIGHLAND PARK		HIGHLAND PARK		HIGHLAND PARK		HIGHLAND PARK	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
HIGHLAND PARK	0.31	CARTERET	0.35	ELIZABETH	0.11	CARTERET	0.15
CARTERET	0.24	ELIZABETH	0.29	CARTERET	0.07	ELIZABETH	0.12
ELIZABETH	0.22	HIGHLAND PARK	0.15	HIGHLAND PARK	0.06	HIGHLAND PARK	0.07
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

Table IIIB-B15

FORMALDEHYDE - HCHO (METHANAL)

QUARTER BEGINNING OCTOBER 1987		QUARTER BEGINNING JANUARY 1988		QUARTER BEGINNING APRIL 1988		QUARTER BEGINNING JULY 1988	
SITE	ARITH. MEAN (ppb)	SITE	ARITH. MEAN (ppb)	SITE	ARITH. MEAN (ppb)	SITE	ARITH. MEAN (ppb)
CARTERET	2.91	CARTERET	3.38	CARTERET		CARTERET	4.81
ELIZABETH		ELIZABETH		ELIZABETH		SUSAN WAGNER HS	4.05
PISCATAWAY		PISCATAWAY		PISCATAWAY		PISCATAWAY	3.30
SUSAN WAGNER HS		SUSAN WAGNER HS		SUSAN WAGNER HS		ELIZABETH	
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

QUARTER BEGINNING OCTOBER 1988		QUARTER BEGINNING JANUARY 1989		QUARTER BEGINNING APRIL 1989		QUARTER BEGINNING JULY 1989	
SITE	ARITH. MEAN (ppb)	SITE	ARITH. MEAN (ppb)	SITE	ARITH. MEAN (ppb)	SITE	ARITH. MEAN (ppb)
PORT RICHMOND PO	2.03	ELIZABETH	2.89	SUSAN WAGNER HS	2.13	SUSAN WAGNER HS	2.06
PISCATAWAY	1.78	SUSAN WAGNER HS	1.98	PORT RICHMOND PO	1.69	PORT RICHMOND PO	1.04
SUSAN WAGNER HS	1.62	PORT RICHMOND PO	1.80	CARTERET		CARTERET	
CARTERET		CARTERET		ELIZABETH		ELIZABETH	
ELIZABETH		PISCATAWAY		PISCATAWAY		PISCATAWAY	

Table IIIB-B16

BARIUM

SITE	QUARTER BEGINNING OCTOBER 1987 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1988 ARITH. MEAN (ng/m3)
PS 26	28.4	PS 26	21.7	PS 26	24.2	PS 26	27.5
SUSAN WAGNER HS	15.8	SUSAN WAGNER HS	14.4	SUSAN WAGNER HS	13.2	SUSAN WAGNER HS	17.8
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

SITE	QUARTER BEGINNING OCTOBER 1988 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JANUARY 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING APRIL 1989 ARITH. MEAN (ng/m3)	SITE	QUARTER BEGINNING JULY 1989 ARITH. MEAN (ng/m3)
PS 26	22.2	SUSAN WAGNER HS	25.8	PS 26	16.8	PS 26	27.8
SUSAN WAGNER HS	14.5	PS 26	22.5	SUSAN WAGNER HS	13.4	SUSAN WAGNER HS	4.9
PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO		PORT RICHMOND PO	

APPENDIX C

DATA SUMMARIES BY ANNUAL AVERAGE

Table IIIB-C1

ARSENIC

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	7.1
SUSAN WAGNER HS	1	49	6.5
ELIZABETH	A		
CARTERET	B		
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	44	4.3
SUSAN WAGNER HS	1	48	3.7
ELIZABETH	A		
CARTERET	B		
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

Table IIIB-C2

CADMIUM

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	52	5.6
PS 26	2	52	2.9
SUSAN WAGNER HS	1	49	2.8
ELIZABETH	A	16	2.3
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	60	4.3
PS 26	2	46	2.5
SUSAN WAGNER HS	1	48	2.5
HIGHLAND PARK	E	42	2.1
ELIZABETH	A	54	1.6
PORT RICHMOND PO	5		

Table IIIB-C3

CHROMIUM

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	52	22.2
ELIZABETH	A	16	8.2
HIGHLAND PARK	E		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	46	26.8
ELIZABETH	A	56	15.6
HIGHLAND PARK	E	42	12.3

Table IIIB-C4

COBALT

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	2.9
SUSAN WAGNER HS	1	49	2.7
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	48	2.5
PS 26	2	45	2.5
PORT RICHMOND PO	5		

Table IIIB-C5

COPPER

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	49	98.8
CARTERET	B	52	84.6
PS 26	2	52	56.7
ELIZABETH	A	16	32.9
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
SUSAN WAGNER HS	1	48	83.3
CARTERET	B	45	80.3
PS 26	2	45	40.9
ELIZABETH	A	54	34.5
HIGHLAND PARK	E	42	33.8
PORT RICHMOND PO	5		

Table IIIB-C6

IRON

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	1226.1
CARTERET	B	38	686.8
SUSAN WAGNER HS	1	49	676.3
ELIZABETH	A	16	526.6
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	45	923.6
SUSAN WAGNER HS	1	48	649.3
CARTERET	B	60	604.8
HIGHLAND PARK	E	40	476.5
ELIZABETH	A	54	428.5
PORT RICHMOND PO	5		

Table IIIB-C7

LEAD

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	38	78.6
PS 26	2	52	56.9
SUSAN WAGNER HS	1	49	44.7
ELIZABETH	A	16	29.5
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
HIGHLAND PARK	E	40	49.0
PS 26	2	44	39.2
CARTERET	B	60	36.4
ELIZABETH	A	54	30.8
SUSAN WAGNER HS	1	48	31.3
PORT RICHMOND PO	5		

Table IIIB-C8

MANGANESE

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	38	29.2
PS 26	2	52	28.7
ELIZABETH	A	16	18.9
SUSAN WAGNER HS	1	49	18.5
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	60	21.8
PS 26	2	45	18.8
SUSAN WAGNER HS	1	48	15.2
ELIZABETH	A	54	14.8
HIGHLAND PARK	E	42	13.5
PORT RICHMOND PO	5		

Table IIIB-C9

MERCURY

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	25	5.4
ELIZABETH	A	16	2.7
HIGHLAND PARK	E		
SUSAN WAGNER HS	1		
PS 26	2		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	60	0.5
ELIZABETH	A	57	0.5
HIGHLAND PARK	E	45	0.4
SUSAN WAGNER HS	1		
PS 26	2		
PORT RICHMOND PO	5		

Table IIIB-C10

MOLYBDENUM

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	11.8
SUSAN WAGNER HS	1	49	10.9
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	45	9.7
SUSAN WAGNER HS	1	48	9.6
PORT RICHMOND PO	5		

Table IIIB-C11

NICKEL

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	38	48.3
ELIZABETH	A	16	38.7
PS 26	2	52	22.8
SUSAN WAGNER HS	1	49	17.9
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	45	26.1
ELIZABETH	A	39	23.9
HIGHLAND PARK	E	40	22.6
PS 26	2	45	20.2
SUSAN WAGNER HS	1	48	19.1
PORT RICHMOND PO	5		

Table IIIB-C12

VANADIUM

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	17.5
SUSAN WAGNER HS	1	49	13.6
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	45	16.9
SUSAN WAGNER HS	1	48	15.2
PORT RICHMOND PO	5		

Table IIIB-C13

ZINC

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	128.2
CARTERET	B	38	121.5
SUSAN WAGNER HS	1	49	113.9
ELIZABETH	A	16	82.8
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	60	115.2
ELIZABETH	A	54	109.5
SUSAN WAGNER HS	1	43	113.2
HIGHLAND PARK	E	42	97.8
PS 26	2	45	96.3
PORT RICHMOND PO	5		

Table IIIB-C14

BENZO(A)PYRENE

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	51	0.17
ELIZABETH	A	16	0.04
HIGHLAND PARK	E		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
CARTERET	B	60	0.20
ELIZABETH	A	55	0.19
HIGHLAND PARK	E	53	0.14
PORT RICHMOND PO	5		

Table IIIB-C15

FORMALDEHYDE - HCHO (METHANAL)

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ppb)
SUSAN WAGNER HS	1	1	4.05
CARTERET	B	25	3.63
PISCATAWAY	D	10	3.30
ELIZABETH	A		
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ppb)
ELIZABETH	A	6	2.89
SUSAN WAGNER HS	1	44	2.02
PISCATAWAY	D	7	1.78
PORT RICHMOND PO	5	35	1.71
CARTERET	B		

Table IIIB-C16

BARIUM

FIRST YEAR
OCT 1987 - SEPT 1988

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	52	25.3
SUSAN WAGNER HS	1	49	15.1
PORT RICHMOND PO	5		

SECOND YEAR
OCT 1988 - SEPT 1989

SITE	SITE #	# OF SAMPLES	ARITH. MEAN (ng/m3)
PS 26	2	55	19.2
SUSAN WAGNER HS	1	51	12.9
PORT RICHMOND PO	5		

APPENDIX D

QUALITY ASSURANCE REPORT FROM NEW JERSEY INSTITUTE OF TECHNOLOGY

METALS IN AIRBORNE PARTICULATE

Joseph W. Bozzelli, Dept. of Chemistry and Chemical Engineering
New Jersey Institute of Technology, Newark, NJ 07102.

Quality Assurance Report

Submitted to

Steven Quan
Air Quality Division
New Jersey Department of Environmental Protection
State St
Trenton, NJ 08625
609 633 1110

Submitted by:

Joseph W. Bozzelli,
Dept. of Chemistry and Chemical Engineering
New Jersey Institute of Technology,
Newark, NJ 07102.
201 596 3459

METALS IN AIRBORNE PARTICULATE

Joseph W. Bozzelli, Dept. of Chemistry and Chemical Engineering
New Jersey Institute of Technology, Newark, NJ 07102.

The sample preparation and analysis procedures are designed to provide optimum collection efficiency and accuracy in determining levels of toxic metals in the ambient airborne particulate sampled. Atomic Absorption spectrometry utilizing air acetylene flame was used for all metals except Mercury, where Cold Vapor AA was used.

ANALYTICAL PROCEDURES

The analysis of the airborne particulate sample was performed by dissolving (digesting) the particulate from the filter paper in an acid solution[1], quantitatively transferring the solution into a volumetric flask, and diluting it to exactly 50 ml volume. The analysis was then performed by atomic absorption spectroscopy. The spectrometer was set to the optimum operating parameters for the analysis of each specific metal before analysis of that metal was performed.

A group of samples (usually 6 plus 1 blank filter for filter and acid background correction) were all digested during the same time period. Analyses for the metals in this digestion batch were then done within three days after digestion. The analytical techniques used in these determinations are similar to those described in references 13.

The analysis of metals from airborne particulate consists of several steps. These include:

1 - Preparation - Mounting of the Filter

2 - Particulate collection

3 - Digestion of the particulate to dissolve metals of interest

4 - Calibration of the AA instrument using standards

5 - Analysis of Metal

---

1 - Preparation - Mounting of the Filter

Filters were supplied by NJDEP. They were kept in a desiccator prior to weighing on an analytical balance capable of 100 microgram measurements. The balance was serviced once per year and calibrated with weights traceable to the National Bureau of Standards. The weighing was only useful for total particulate measurement.

The filters were placed in the desiccator for a minimum of 3 days prior to weighing after sample collection to eliminate errors in particulate weights from moisture.

The desiccant was monitored with color indicating silica gel and was regenerated when required via heating to 200 C in a vacuum oven.

2 - Particulate Collection

The 8 x 10 inch glass fiber filter (Whatman) was placed into a sampler filter holder assembly and mounted onto the sampling blower motor assembly at 0900 five days prior to automatic sampler turn on. The sampler was manually checked at 0900 hours on the day of sampling (midnight to midnight sampling) for flow calibration. The manual check verified and served to calibrate the continuous flow (pressure monitor) recorder measurements.

The sampler / filter holder assembly was removed from the Hi Vol blower motor assembly at 0900 hours on the day after the sampling midnight turn-off. The total time (hours, minutes) of sample collection was recorded by NJIT and reported to NJDEP.

The filter holder assembly was then returned to the laboratory. The filter was removed from the filter holder and placed into the desiccator prior to weighing.

NJDEP calibrated the Hi Vol samplers versus Flow monitor (pressure) and provided the total flow to NJIT, after total time of operation were reported to NJDEP by NJIT.

3 - Digestion of the Particulate to Dissolve (DIGEST) the Metals

The analysis of the metals in the particulate consists of the digestion and the second stage, which is the quantitative analysis of the specific metals in the solution.

A section which represents 50 percent of the 8 x 10 inch filter paper was digested, 8 by 5 inch piece. This piece was cut up into small (less than 2 cm x 2 cm) pieces and placed into a 250 ml round bottom flask for digestion.

Six sample filters and 1 blank filter were digested during the same time period. Analysis for metals in the digestion batch was always performed within 3 days of the digestion to minimize sample loss of the analyte on the container walls.

The digestion acid, 50 ml per filter sample, consists of 50% nitric, 10% hydrochloric acids and 40% high purity super water (deionized and then doubly distilled), plus 3 ml of 30% hydrogen peroxide. The hydrogen peroxide volume was adjusted to eliminate (oxidize) carbon solids so that no metals remained adsorbed within the carbon particulate. The Peroxide was added dropwise after the solution was boiling and then allowed to cool to just below boiling. Boiling was then continued after completion of the clarification of the carbon soot.

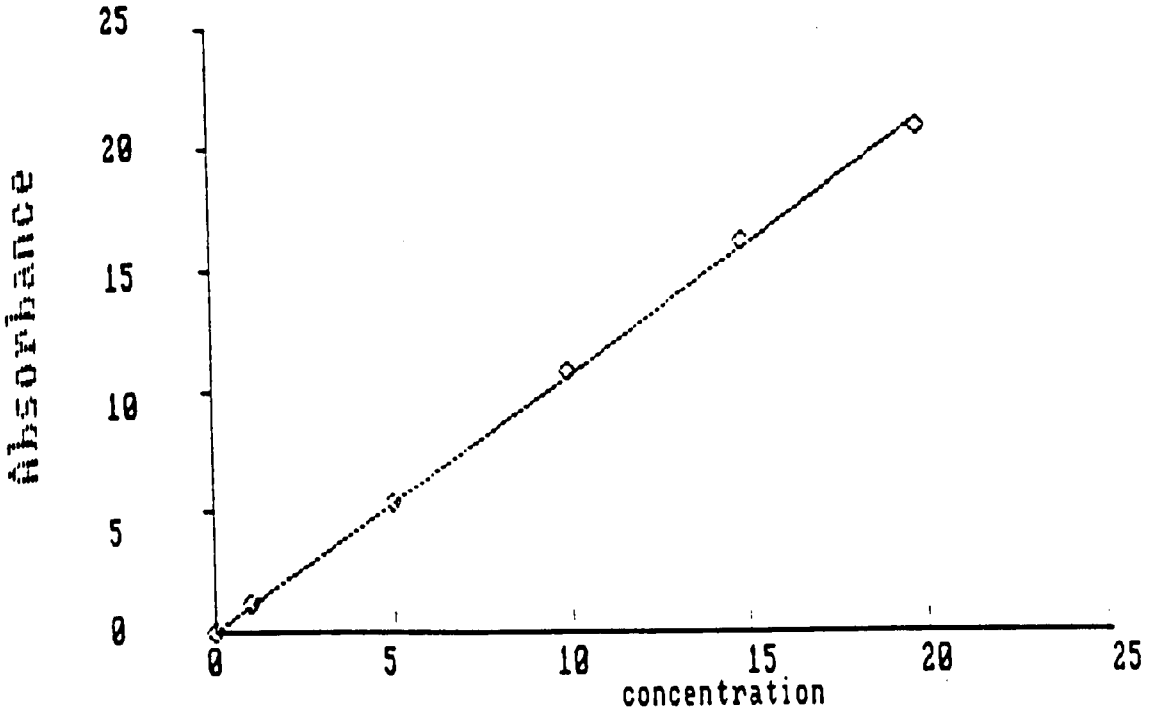
The solution was held at or near boiling in round bottom flasks with water cooled condensers for approximately 8 hours. Some filters were digested for 16 hours, but it was discerned that the longer digestion time did not improve the analysis. All quality assurance data on the EPA and NBS (NIST) standard filters and urban dust particulate materials respectively, were performed using the 8 hour digestion time period.

Upon completion of the heating digestion period the solution was removed from the digestion flasks and diluted to 50 ml volume. It was poured into a volumetric flask through a filter paper - funnel assembly to remove glass fibers from the solution, which would serve to block or partially obstruct the liquid flow into the aspirator of the AA, if not removed. The 50 ml volume was made up with rinse from the digestion flasks and condensers and then from distilled water.

Commercial standards for each metal were purchased (Baxter Health Care Inc.) either in 1000 ppm concentration or in high purity solid form and then dissolved in acid solution to a known concentration. The standards were diluted with doubly distilled deionized water using calibrated pipets and volumetric flasks. Typical levels of standard solution were between 0.1 and 10 mg/ml. A least squares fit to absorbance vs. concentration line was calculated using the standards data and the point (0,0) The slope from this least squares calculation was then used to determine the concentration (mg/ml) from the sample absorbance readings. A typical plot for Lead standards is shown in Figure 1.

Minimum Detection Limits (MDL's) were determined by two methods. One method was that used at NJIT routinely, the second was a method provided by the USEPA Region II. The NJIT method was simply a signal required to provide 4 times the Signal to noise of the AA instruement absorbance reading for standard solutions. The EPA provided method yielded somewhat lower MDL's and the

Calibration Curve for the Lead



Concentration in ug/ml
Figure I

reader should be referred to MDL reporting requirements for this Project, supplied by the USEPA Region II for further specific information on this method.

These EPA - MDL values are determined from the Standard Curve run on each day of analysis -- each metal. They provide, quality assurance data as per EPA requirement. this includes quality assurance to specific evaluation on each set of values supplied to EPA in the data report. These MDL's incorporate values of the absolute values (responses), of the standards for each metal for each set of analysis (standards run before and after each set of analysis for each metal on the AA instruments - Each batch of 6 runs plus blank). The MDL's are therefore calculated and reported separately for each batch of analysis and incorporated into the data sheets and data format disks provided to USEPA as required part of the EPA data reporting format. Since this method was developed reviewed and recommended by EPA it is not further discussed here.

MERCURY COLD VAPOR APPARATUS

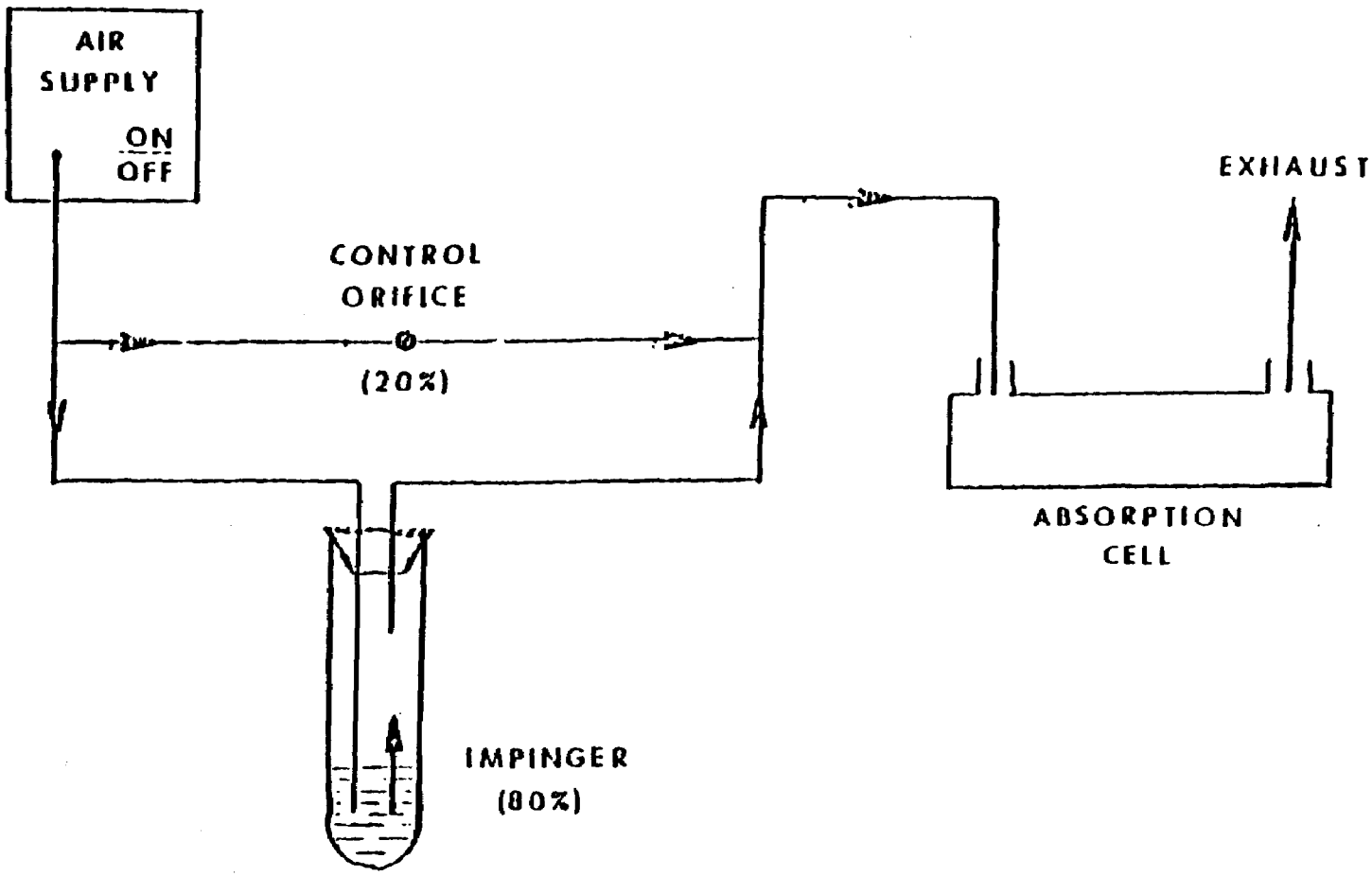


FIGURE - 2

TABLE I MINIMUM DETECTABLE QUANTITIES

METAL	Concentration (ug/ml)	EPA** (ug/ml)	Air Levels (Ng/M^3)*
Pb	0.2	0.042	10.
Mn	0.1	0.017	5.
Ni	0.15	0.027	7.5
Cd	0.05	0.0035	2.5
Hg	0.05(ng/ul)	0.057 (ng/ul)	0.01#
Co	0.2	0.48	10.
Zn	0.07	0.26	3.5
Cr	0.2	0.13	10.0
Fe	0.2	0.14	10.
Cu	0.15	0.28	7.5

* Assuming a 2000 m^3 sample volume collected, with half (1000 m^3) being analyzed and the NJIT detection limits. (Nanogram/m^3)

Cold Vapor Technique (all units - nanograms)

** Determined by methods provided by USEPA (Region II). These lower MDl values are determined from the Standard Curve run on each day of analysis of each metal. They provide, as per EPA requirement, quality assurance data - specific evaluation on each set of values supplied to EPA in the data report. These MDL's incorporate values of the absolute values - responses, of the standards run before and after each set of analysis for each metal on the AA instruments. Each batch of 6 runs plus blank.

NJIT - The total amount (concentration in ug/ml) of each metal is required to give a signal of 4 times the noise level for a 50 ml volume of analyte solution. Occasionally, a value less than this minimum is reported. These low values, due to spectrometer readings of less than 4 x noise or due to subtraction of a large blank, have a larger margin of error than discussed (see later).

This table only represents minimum detection limits as evaluated by NJIT. The separate Quality assurance minimum detection limits as determined using the USEPA supplied formula were rigorously reported with all of the sample data supplied to NJDEP and to the USEPA.

Mercury Analysis by Cold Vapor Atomic Analysis

The Hatch and Ott Cold Vapor technique used for mercury analysis on an AA has been modified in order to improve both precision and accuracy. The drying filter for elimination of water vapor has been removed from the Hg vapor inlet line absorption cell and the activated charcoal mercury removal trap has been eliminated. A diagram of the present mercury cold vapor apparatus is shown in Figure 2. It consists of an air supply pump an impinger and an impinger pass line, an inlet to the absorption cell and exhaust from the cell directly to a fume hood.

A constant fraction, 20 percent, of the air flow from the pump by-passes the impinger assembly and flows directly into the absorption cell. This is a sufficient flow of dry air to prevent water vapor from condensing on the cell windows, body, or tubing lines.

The digestion of the filter containing the particulate, storage under acidified conditions, and treatment of the digestion solution with stannous chloride just prior to analyses, remain identical to the previous method. Using this method the absorbance reading peaks about ten seconds after the air supply pump is turned on, and the peak width is approximately 15 seconds. The sample flows through the absorption cell and is exhausted into a fume hood. An illustration of the reproducibility of the method is shown in Figure 3, where replicate samples gave a standard deviation of 0.5%. The volume of the sample used in

the impinger, 5ml, permits up to 8 analyses on the same air sample, if required. All absorbance readings are output on a recorder for display and data measurement.

A typical plot of absorbance versus micrograms of Hg per ml using a 5ml volume of standard solution is shown in Fig 4. The minimum detectable amount is 0.05 ng of Hg per ul, using a 5ml sample into the saturator. Using a 50 ml volume of solution from digestion this corresponds to 10 ng of Hg per filter, minimum detectable limit -- 0.01ng.m^3 for 1000 m^3 sample.

The analyte solution was made by taking a 20 ml portion of the digested solution and 2 ml of concentrated nitric acid in a capped plastic vial. The nitric acid was added in order to stabilize the Mercury as the HgO. 10 ml of this solution was placed in a 50 ml aerator tube just before the analysis. 1 ml of 10% (saturated) stannous chloride solution was then added to the liquid in the aerator tube and standard cold vapor analysis performed. The Stannous chloride converted the HgO into Hg vapor, which was circulated into the path of the AA lamp (light source).

A separate AA instrument was set up for the Hg analysis and dedicated to this analysis. This instrument was not used for any other analysis.

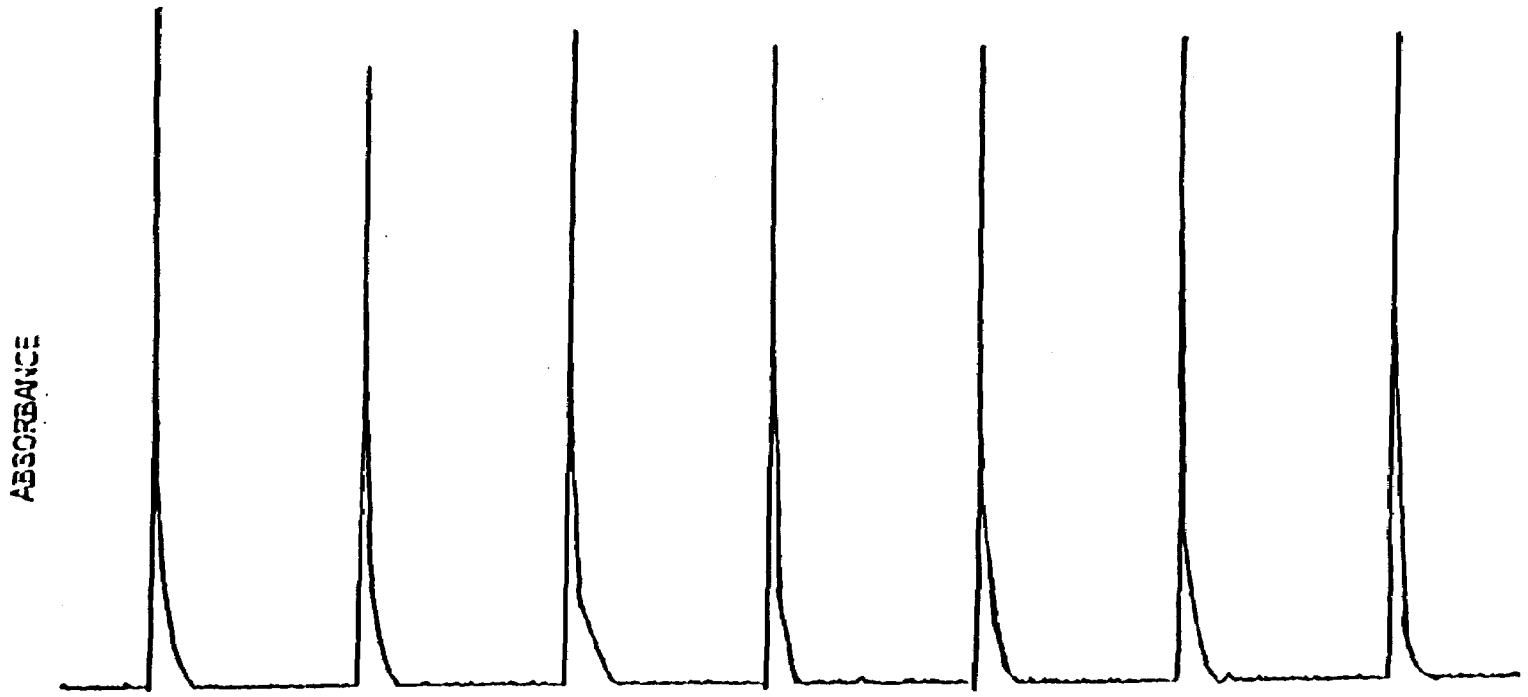
FIGURE - III

PRECISION OF MERCURY ANALYSIS

STANDARD DEVIATION - 0.5%

5.0 ML. OF 0.05 UG./ML. HG STANDARD

1 MINUTE

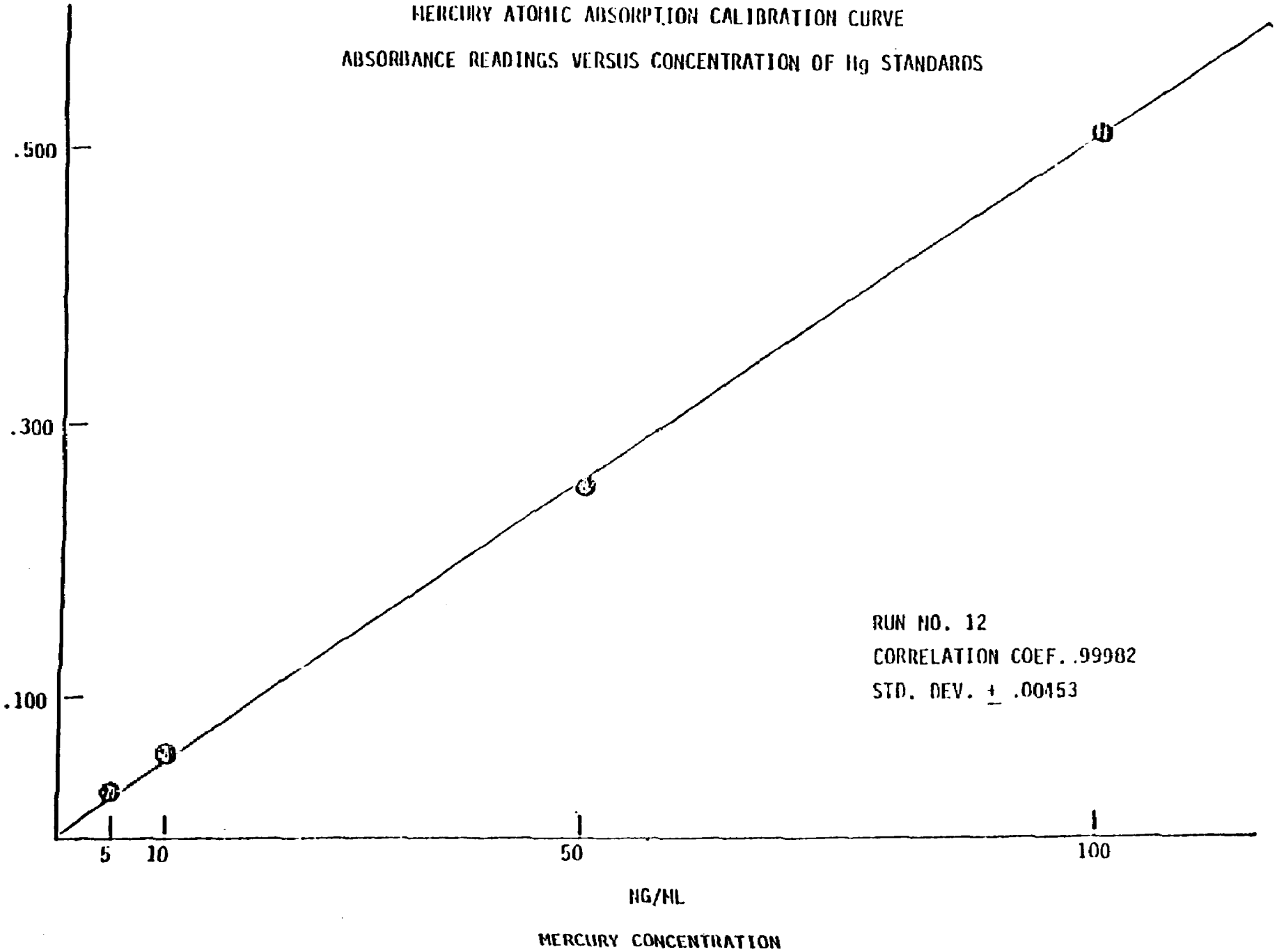


7 REPLICATE MERCURY DETERMINATIONS

FIGURE - IV

MERCURY ATOMIC ABSORPTION CALIBRATION CURVE

ABSORBANCE READINGS VERSUS CONCENTRATION OF Hg STANDARDS



Analysis of USEPA Lead Standards

Glass fiber filter strips impregnated with known amounts of lead material were obtained from the United States Environmental Protection Agency, Quality Assurance Branch, Research Triangle Park, North Carolina. These filter strips were analyzed with identical procedures to those used in determinations in this project on airborne particulate. A comparison of the EPA supplied values with the results obtained in this laboratory showed our analysis was routinely within 95% of the known standards. Table II illustrates the agreement for lead.

Table II

ANALYSIS OF USEPA LEAD STANDARDS

USEPA ID. #	LEAD(EPA)	LEAD (NJIT)	%NJIT/EPA
Pb 831-4135	900	910.9	101.2
Pb 831-5145	1300	1239.4	95.3
Pb 831-6024	1100	1055.2	95.9
Pb 831-7150	2000	1841.6	92.1
Pb 831-8153	1800	1682.3	93.5
Pb 831-9148	1600	1473.3	92.8

Analysis of National Bureau of Standards Urban Particulate

The National Bureau of Standards (NBS) urban particulate standard, No. 1648, was purchased and analyzed in these laboratories for quality assurance determinations. The standard was analyzed for lead and cadmium nickel and manganese in the initial determinations and for chromium, iron, copper and zinc in the last four determinations. The results are listed in Table III. This particulate required extensive drying in an oven before a sample of it could be accurately weighed and analysis performed. The drying step was done in an oven at 150°C for a period of 16 hours or longer. The drying is necessary to remove water vapor which had adsorbed on the standards and is part of the NBS recommended procedure.

Table III

COMPARISON OF NJIT METALS ANALYSIS WITH

NATIONAL BUREAU OF STANDARDS

PARTICULATE STANDARD NO. 1648

Analysis	NBS - 3		
Metal	NJIT	NBS	% (NJIT/NBS)
Cadmium	56.9	75 ±7	76
Lead	6,024	6,550 ±8	92
NBS - 4			
Cadmium	65	75 ±7	87
Lead	5,546	6,550 ±8	85
Nickel	90	82 ±3	110
NBS - 5			
Cadmium	78.8	75	105
Lead	5,654	6,550	86.3
Nickel	72.4	82	88.3
Manganese	562	800[a]	71
NBS - 6			
Cadmium	76	75	101
Lead	6,244	6,550	95.3
Nickel	152	82	185**
Manganese	603	800[a]	75.4

* Concentrations in ug metal per gram of dry particulate.

** Possible contamination of sample.

TABLE III (Con't)

NBS - 7D

Metal	NJIT	NBS	% (NJIT/NBS)
Cadmium	69.4	75	92.5
Lead	7,310	6,550	111.6
Nickel	74.4	82	90.7
Manganese	695	800[a]	86.9
Chromium	348	403	86.4
Iron - Fe	38,23	39,100	97.8
Copper	523	609	85.9
Zinc	4,49	4,760	94.3

NBS - 8D

Cadmium	77.6	75	103.4
Lead	6,589	6,550	100.6
Nickel	88.8	82	108.3
Manganese	717	800[a]	89.6
Chromium	371	403	92.1
Iron - Fe	37,47	39,100	95.8
Copper	567	609	93.1
Zinc	4,59	4,760	96.4

* Concentrations in ug metal per gram of dry particulate.

Table III (con't)

NBS - 9D

Cadmium	68.6	75	91.5
Lead	6,930	6,550	105.8
Nickel	75.6	82	92.2
Manganese	703	800[a]	87.9
Chromium	339	403	84.1
Iron - Fe	37,6	39,100	96.2
Copper	541	609	88.8
Zinc	4,87	4,760	102.1

NBS - 10D

Cadmium	68.3	75	91.0
Lead	6,380	6,550	97.4
Nickel	84.9	82	103.5
Manganese	686	800[a]	85.8
Chromium	377	403	93.5
Iron - Fe	36,3	39,100	92.8
Copper	555	609	91.1
Zinc	4,17	4,760	87.6

a - Not a NBS certified value.

- Cobalt levels too low to quantitize with size of our samples.

- Mercury is not reported in the NIST Standard.

* Concentrations in ug metal per gram of dry particulate.

Metals Except Mercury, i.e. Pb, Cd, Mn, ... etc.

The digested samples are kept in polyethylene volumetric flasks or vials until they were analyzed by atomic absorption spectroscopy. The solutions are thoroughly mixed to assure uniform concentration before the analyses are performed. The digested sample is then aspirated into the air-acetylene flame of the atomic absorption instrument and the absorbance monitored.

The atomic absorption spectrometer is tuned each day before analyses are run (burner alignment, optimization of lamp alignment and frequency). Calibration absorbance curves are prepared each day from standard metal solutions. When samples are run, the standards are checked at the beginning and end of the analysis of each metal. In the analysis of a group of samples for one metal, all the spectrometer conditions are first optimized, the standards run, and a graph of absorbance versus concentration (ug/ml) is plotted to verify the linear relationship. This is done for each metal in the group of six samples plus blank. The samples are then analyzed for the metal, 3 readings are taken in a time frame of 5 to 10 seconds per reading. It should be noted that usual Flame AA analysis takes readings in time frames of 1 - 2 seconds per reading. The zero reading is checked after each absorbance measurement, and standards are rerun to check instrument drift after approximately every seven samples.

Commercial standards for each metal are purchased either in

1000 ppm concentrations or in high purity solid form and then prepared in acid solution to a known concentration. The standards are diluted with the "super water" using calibrated pipets and volumetric flasks. Typical levels are between 0.1 and 10 ug/ml. An example of the procedures and calculations for making a known lead standard from a solid aliquot of $PbCl_2$ is illustrated in the attachemnt following this section of the report.

The absorbance is directly proportional to the standard, showing a linear relationship in accordance with Beers Law. A least squares fit to this line is calculated using the standards data and the point (0,0) which is valid for these AA plots. The slope from this least squares calculation is then used to determine the concentration (ug/m) from the sample absorbance readings. Figure 1, as mentioned previously, shows a typical absorption curve for lead standards. Samples found at higher concentrations than these values are diluted and rerun in order to locate the concentration within the linear portion of the curve. The spectrometer conditions for each metal and lamp are listed in Table IV and are for the wavelength at which the AA is most sensitive to the metal under analysis.

Vanadium was initially analyzed using a nitrous oxide - acetylene flame. After two blowouts of the flame it was determined that continued use of our AA with this method was unsafe. Vanadium analysis was discontinued. While it is felt that the Vanadium results reported are of reasonable accuracy and qualility, proper quality assurance procedures and calibrations can not be reported.

Table IV

Operating Parameters for Atomic Absorption Analysis
Metals Air-Acetylene Flame.

Element		Wavelength (nm)	Spectral Band Pass (nm)	Lamp Current ma
1)	Lead	217.0	1	4
2)	Nickel	232.0	1	5
3)	Copper	324.8	2	4
4)	Cobalt	240.7	1	6
5)	Iron	248.3	2	6
6)	Zinc	213.9	1	5
7)	Chromium	357.9	2	6
8)	Mercury*	253.7	1	3
9)	Manganese	279.5	2	5
10)	Cadmium	228.8	1	3

* Cold Vapor Method

Spectro photometric Conditions for Each Metal

Vanadium was initially analyzed using a nitrous oxide - acetylene flame. After two blowouts of the flame it was determined that continued use of our AA with this method was unsafe. Vanadium analysis was discontinued. While it is felt that the Vanadium results reported are of reasonable accuracy and qualilty, proper quality assurance procedures and calibrations can not be reported.

ANALYSIS OF PRECISION AND ACCURACY

1. Error Analysis

The relative errors associated with each of the steps in the collection and analysis of airborne particulate for metals are presented below.

Standard Solutions

Standard solutions of 1000 ppm (1000 ug/ml) were purchased from Baxter Health Care, Edison, NJ. The accuracy of these atomic absorption reagents was National Bureau of Standards certified (±0.2% of the reported concentration). The standards are diluted using precision pipets, burettes and volumetric flasks. Slated errors from reading volume levels in volumetric flasks and pipets is less then 1%, while error for reading a small difference from a burette may be as high as 2 percent. On the basis of the burette error the accuracy of the standard solution is placed within 2.0 percent of the nominal value. The burets are not used frequently and most of the standard solutions are therefore considered to have an even smaller error limit.

It is valuable to note that agreement with the USEPA lead standards - filter strips provides reinforcement of the accuracy in the standard make up. This is because there is no digestion or extraction problem here, i.e. all the metal is easily extracted from the filter. It is then, primarily, lab techniques and standard accuracy which dictate whether one achieves agreement with the EPA standard filter values.

Standard Solutions - Storage

Standard solutions are stored in polyethylene bottles and are routinely checked for stability. New standard solutions are made up for these studies approximately once every four weeks. A comparison of the AA signals from the old and new standards provides information on stability of the standard solutions. This corresponds, in this study, to once every two times samples are analyzed. New solutions are always stored in the bottles which had previously contained the same concentrations of the same metal. This eliminates adsorption effects on the walls of the plastic containers. Errors which arose from slight changes in standard concentrations due to storage are Further monitored by observing the behavior of the standard curves. The loss of a metal to the vessel surfaces is amplified greatly on the very low concentration standards. This results in a low value for the lower concentration standard absorbance readings, and a corresponding higher value for the higher concentration standards. Average standard deviations from standard curves correspond to less than 5%, thus we estimate this error at 5%.

AA Analysis:

The Absolute accuracy of the atomic absorption analysis results from sensitivity specific to each element being analyzed. Please see table I.

Digestion

The digestion step used is a standard method accepted by the United States Environmental Protection Agency and a thorough study of its effectiveness is not undertaken. The results from digestion and analysis of the NIST Standard Particulate (road Dust) sample provides a good indication of the accuracy of our digestion, with the exception of tests NBS 1-3 which were not performed on thoroughly dried material our average error is within 10% for the three metals (Cd, Pb, Ni, Fe, Zn) tested routinely and within 23 % for Manganese. The error for Manganese improved to within 20 percent on the last 4 particulate analysis. An approximately error limit of 10% is assigned to this step by assuming that it contributes half of the total error (±20%) accepted by the U.S. EPA> This estimate correlates well with the data obtained in these labs on metal standards, supplied by the USEPA and the National Bureau of Standards described earlier. Chromium and Copper analysis were with in 15%.

Collection

The collection efficiency of the glass fiber filters is 99.9% for 0.3 micron particles and greater than 99.9% for larger particles, as reported by the manufacturer, Whatman in this study.

Volume of Air Samples

A detailed discussion of the volume flow calibration of the sampling pumps is included in a separate section supplied by

A summary of these errors is illustrated in Table V

Table V

Summary of Error Analysis for Collection and Measurement

Airborne Particulate for Toxic Metals.

ERROR LEVELS

PROCEDURE	ACCURACY(%)	ERROR LEVEL (%)
1. Standards	98.	±2.0
2. Standard Storage	95.	±5.0
3. Digestion completeness	90.	±10.0
4. Collection Efficiency[c]	99.	±1.0
5. Volume of air sample	90.	±10.0
Overall (Root mean square)		±17

c. Within the size range 0.3 - 30 um (micro meters).

The estimate of error associated with AA instrument instability is determined by the average instability observed for a series of readings near blank level. This is dependent on the specific metal being evaluated and on te condition of the emission lamp of that metal. It corresponds to 0.2 micrograms per milliliter for the metal (Pb), which we determine to exhibit this problem to the largest extent. This is equivalent to 10.0 micrograms total in the 50 ml volume of analyte solution and equates to ± 10. ng/m^3 air sample - 1000 m^3 volume. The percent error is difficult to fully evaluate because different metals have different concentrations and thus different % error for each case. The EPA has indicated to NJIT that the best way to evaluate this is to utilize the USEPA Minimum Detection Limit Value (MDL) as we report in Table I for a select set of studies. These values have been reported to the USEPA for each metal, each set of analysis.

The EPA - MDL values are determined from the Standard Curve run on each day of analysis -- each metal. They provide, quality assurance data as per EPA requirement. this includes quality assurance to specific evaluation on each set of values supplied to EPA in the data report. These MDL's incorporate values of the absolute values (responses), of the standards for each metal for each set of analysis (standards run before and after each set of analysis for each metal on the AA instruments - Each batch of 6 runs plus blank). The MDL's are therefore calculated and reported separately for each batch of analysis and incorporated into the data sheets and data format disks provided to USEPA as required part of the EPA data reporting format. Since this method was developed reviewed and recommended by EPA it is not further discussed here.

Atomic Absorption Analysis

Introduction

Since determination of metallic elements in a sample matrix is a complex process, a sensitive and selective method of analysis was desirable. The steadily growing list of atomic absorption analytical applications now covers almost all of the elements. The major breakthrough in the development of the use of measurement of atomic absorbance as an analytical technique came in the 1950s when Sir Alan Walsh realized that it was possible to use a line source which emitted very narrow lines at the same wave length in place of a continuum source.

Operating Principles

As the instrument is turned on and the necessary lamp currents, wavelength, slit widths and PMT voltages are set to the required levels, a light source (hollow cathode lamp) emits the spectrum of the metal selected for analysis. A specific line of the target metal spectrum is capable of being absorbed by atoms of the same metal if they are present in the sample. The liquid sample is converted to a fine aerosol by a nebulizer and the aerosol is then reduced to the constituent atoms in a flame. The flame lies in the light path between the lamp and the line detector (monochromator and photomultiplier tube). If any of the metal is present in the sample, absorption of the characteristic line will provide an accurate measure of the concentration of the metal. The analytical precision is typically around 1% relative.

Principle of the method -- Atomic Absorption

In atomic absorption, the element of interest in the sample is not present in an excited state. Rather, it is in an elemental form dissociated from its chemical bonds and placed into an unexcited, un-ionized "ground" state. This dissociation is most commonly achieved by placing the sample into a high temperature flame. The element is then capable of absorbing radiation at discrete lines of narrow bandwidth.

A hollow cathode lamp usually provides the narrow emission lines which are to be absorbed by the same element. The lamp contains a cathode made of the same element being determined and is filled with an inert atmosphere at low pressure. Such a lamp emits the spectrum of the desired target element.

The atomic absorption spectrophotometer was tuned each day before the analysis was carried out. The tuning process involves burner alignment, optimization of lamp alignment and wavelength. A Varian atomic absorption spectrophotometer, model 12 was used.

The calibration absorbance curve for each metal was prepared from standard metal solutions each time the analysis was carried out. When samples were run, the standards were checked at the beginning and end of each set of metal analyses. In an analysis of a group of samples for a metal, all the spectrophotometric conditions, wavelength, alignment, lamp current and zero reading were first optimized. The standards were then run

and a graph of absorbance versus concentration is plotted to verify the linear relationship. The zero reading was checked for instrumental drift. Figure 1 shows a typical absorption curve for the lead standard. The spectro-photometric conditions for each metal and lamp are listed in Table IV. The line source commonly used is a hollow-cathode lamp and the flame is air-acetylene.

Standard Solution Concentration Example Calculation

Lead chloride $Pb\ Cl_2$ Molecular Weight $PbCl2 = 278$

Atomic Weights $Pb = 207$ $Cl_2 = 71$

Wt of Pb in $PbCl_2$ = 207/278 x Grams $PbCl_2$ weighed out

Definition 1000 PPM Pb = 1gm/1000g = 1gm/liter = 0.1 gm/100ml

Assume Weighed 0.1300 Gms $PbCl_2$ on analytical balance

Dissolve 0.13 gms PbCl2 x 207/278 = 0.0975 gms Pb

Solution made by heating Sample $PbCl_2$ in 10 % nitric acid solution in super water.

0.0975 gms Pb in 100ml = 975. PPM_w Mix Well Before Use

Lower concentrations of lead standard are made by serial dilution of this 975 ppm solution.

the formula to use for determining quantities of this 975 ppm standard and the distilled water diluent is:

Vi x Ci = Vf x Cf

where V is volume C is concentration in ppm
i stands for initial f stands for final

Choose Vf to be a convenient volume, where there is a volumetric flask available, typically 250 to 500 ml is utilized. Pick 500.

Ci is fixed at 975 ppm in this example until a lower concentration is made up.

Cf should be an intermediate concentration, that requires a volume of Ci, which can be measured accurately. (0.01 micro liters is, for example, difficult to measure quantitatively using a pipette. Typically one might choose 50 or 100 ppm for Cf.

Choosing 50 ppm for Cf, one calculates Vi should be 25.6 ml; that is 25.6 milliliters of 975 ppm standard needs to be added to the 500 ml volumetric and then the volumetric filled to the 500 ml mark with distilled or super water to make up a 50 ppm standard.
This solution also needs to be very well mixed before using for a standard or an initial concentration to make up a lower concentration solution.

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Appendix B

Quality Assurance

Glass fiber filters from E.P.A. (calendar years 1989 and 1990), manufactured by Whatman, were used at all sites.

Metals by Inductively Coupled Plasma (ICP)

Twenty sample filters, one acid blank, one filter blank, one spike, and one duplicate filter aliquot were analyzed per batch.

Arsenic by Graphite Furnace

Twenty sample filters, one filter blank, and one N.B.S. Urban Dust aliquot were analyzed per batch.

Mercury by Cold Vapor Atomic Absorption

Twenty sample filters, one filter blank, and two known mercury reference solutions were analyzed per batch.

Average acid blanks, filter blanks, known Q.C. recoveries, spike recoveries, and duplicate precision are presented in the following tables.

Table B-I Average Acid Blanks

Metals	ng/ml Extract
Barium	<20
Beryllium	< 4
Cadmium	<20
Chromium	<20
Cobalt	<20
Copper	<20
Iron	<40
Lead	<80
Manganese	<20
Molybdenum	<80
Nickel	<20
Vanadium	<20
Zinc	<40

Tables B-II Average Filter Blanks

Metal	ng/ml extract	ng/filter
Arsenic	<10	< 3000
Barium	36 92	17000 (CY 1989) 44000 (CY 1990)
Beryllium	< 4	< 2000
Cadmium	<20	<10000
Chromium	<20	<10000
Cobalt	<20	<10000
Copper	<20	<10000
Iron	117	56200
Lead	<80	<40000
Manganese	<20	<10000
Mercury	<0.2	< 60
Molybdenum	<80	<40000
Nickel	<20	<10000
Vanadium	<20	<10000
Zinc	59	28000

Computer Printouts

B-III Known QC Recoveries (Urban Dust, Hg Solutions)

B-IV Spike Recoveries

B-V Duplicate Precision

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QUALITY CONTROL SUMMARY SHEET ARSENIC (URBAN DUST)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL	%107.7520	----------
UPPER 95% CL	%103.3397	----------
MEAN	89.3909	----------
LOWER 95% CL	75.4422	----------
LOWER 99% CL	71.0298	----------

Batch Number 1 20 25

BIAS = %-10.609%

99% CONFIDENCE LEVEL =± 20.54026 %
95% CONFIDENCE LEVEL =± 15.60423 %

Batch Number	OPERATOR	QUALITY CONTROL % Recovery DATA
1	23	88.7
2	23	96.5
17	23	102.0
18	23	86.6
19	23	84.0
20	23	79.1
21	23	85.6
22	23	96.7
23	23	82.4
24	23	94.9
25	23	86.6

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET MERCURY (REFERENCE SOL.)

TARGET 0.830 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 12.5 %

UPPER 99% CL	0.9311
UPPER 95% CL	0.9107
MEAN	0.8464
LOWER 95% CL	0.7820
LOWER 99% CL	0.7617

Batch Number 1 20 25

BIAS = +1.972%

99% CONFIDENCE LEVEL =± 10.00632 %
95% CONFIDENCE LEVEL =± 7.601704 %

Batch Number	OPERATOR	QUALITY CONTROL ng/ml	DATA
1	23	0.805	0.826
2	23	0.805	0.826
17	23	0.867	[illegible]
18	23	0.822	[illegible]
19	23	0.837	[illegible]
20	23	0.874	[illegible]*
21	23	0.831	[illegible]
22	23	0.857	[illegible]
23	23	0.847	[illegible]
24	23	0.847	[illegible]
25	23	0.822	[illegible]

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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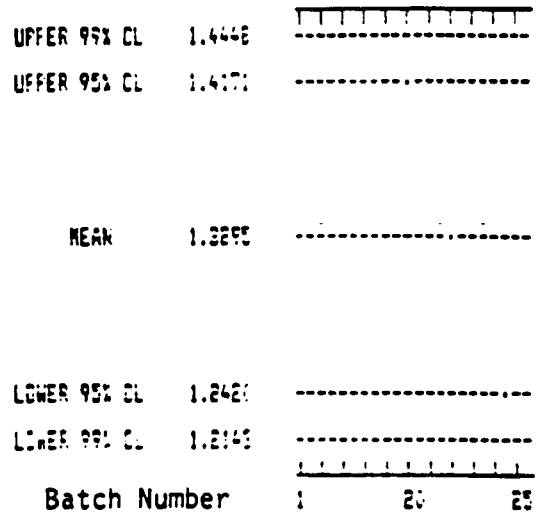
QUALITY CONTROL SUMMARY SHEET MERCURY (REFERENCE SOL.)

TARGET 1.33 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 10 %



BIAS = -0.034%

99% CONFIDENCE LEVEL =± 6.667175 %
95% CONFIDENCE LEVEL =± 6.594361 %

Batch Number	OPERATOR	QUALITY CONTROL ng/ml	%
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

NEW YORK STATE DEPARTMENT OF HEALTH
Wadsworth Center for Laboratories and Research
Laboratory of Inorganic Analytical Cemistry

QUALITY CONTROL SUMMARY SHEET BARIUM (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %133.2371 ||||||||||

UPPER 95% CL %125.9075 ----------

MEAN %102.7364 ----------

LOWER 95% CL 79.5653 ----------

LOWER 99% CL 72.2356 ||||||||||

Batch Number 1 20 25

BIAS = +2.736%

99% CONFIDENCE LEVEL =± 29.68834 %
95% CONFIDENCE LEVEL =± 22.55394 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	116.7
2	23	109.3
17	23	108.2
18	23	127.0*
19	23	97.2
20	23	101.8
21	23	84.9
22	23	97.6
23	23	92.3
24	23	96.5
25	23	98.6

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET BERYLIUM (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 17.5 %

UPPER 99% CL	%104.2669	----------
UPPER 95% CL	%102.0007	----------
MEAN	94.8364	----------
LOWER 95% CL	87.6720	----------
LOWER 99% CL	85.4058	----------

Batch Number 1 20 25

BIAS = -5.164%

99% CONFIDENCE LEVEL =± 9.944049 %
95% CONFIDENCE LEVEL =± 7.554395 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	93.6
2	23	92.0
17	23	90.4
18	23	100.0
19	23	101.6
20	23	96.0
21	23	92.0
22	23	92.8
23	23	92.8
24	23	93.6
25	23	98.4

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET CADMIUM (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %121.7562

UPPER 95% CL %115.6415

MEAN 96.3111

LOWER 95% CL 76.9807

LOWER 99% CL 70.8660

Batch Number 1 20

BIAS = -3.689%

99% CONFIDENCE LEVEL =± 26.41967 %
95% CONFIDENCE LEVEL =± 20.07076 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	87.1
2	23	95.4
17	23	103.7
18	23	91.3
19	23	87.1
20	23	103.7
23	23	95.4
24	23	116.0*
25	23	87.1

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET CHROMIUM (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %141.8105

UPPER 95% CL %133.1175

MEAN %105.6364

LOWER 95% CL 78.1552

LOWER 99% CL 69.4622

Batch Number 1 20 25

BIAS = +5.636%

99% CONFIDENCE LEVEL =± 34.24404 %
95% CONFIDENCE LEVEL =± 26.01486 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	119.6
2	23	110.0
17	23	108.4
18	23	138.4*
19	23	108.0
20	23	103.6
21	23	89.2
22	23	101.2
23	23	95.2
24	23	93.6
25	23	94.8

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET COBALT (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 17.5 %

UPPER 99% CL %128.3585

UPPER 95% CL %124.1478

MEAN %110.8364

LOWER 95% CL 97.5250

LOWER 99% CL 93.3142

Batch Number 1 20 25

BIAS = %+10.836%

99% CONFIDENCE LEVEL =± 15.80904 %
95% CONFIDENCE LEVEL =± 12.00997 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	113.6
2	23	110.4
17	23	112.0
18	23	102.4
19	23	112.0
20	23	124.8*
21	23	113.6
22	23	110.4
23	23	97.6
24	23	112.0
25	23	110.4

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET COPPER (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL	%105.1523	---------
UPPER 95% CL	%101.7914	---------
MEAN	91.1667	---------
LOWER 95% CL	80.5420	---------
LOWER 99% CL	77.1811	---------

Batch Number 1 21

BIAS = -8.833%

99% CONFIDENCE LEVEL =± 15.34068 %
95% CONFIDENCE LEVEL =± 11.65416 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	93.2
2	23	82.1
17	23	92.1
18	23	93.6
20	23	100.4
21	23	86.9
23	23	95.2
24	23	86.5
25	23	90.5

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET IRON (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 20 %

UPPER 99% CL %118.4512

UPPER 95% CL %114.0533

MEAN %100.1500

LOWER 95% CL 86.2468

LOWER 99% CL 81.8488

Batch Number 1 20

BIAS = +0.150%

99% CONFIDENCE LEVEL =± 18.27381 %
95% CONFIDENCE LEVEL =± 13.88243 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	103.7
2	23	104.1
17	23	86.8
18	23	111.1
19	23	92.1
20	23	105.0
21	23	95.2
22	23	103.7
23	23	100.6
25	23	99.2

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

NEW YORK STATE DEPARTMENT OF HEALTH
Wadsworth Center for Laboratories and Research
Laboratory of Inorganic Analytical Cemistry

QUALITY CONTROL SUMMARY SHEET LEAD (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %120.8043 ||||||||

UPPER 95% CL %114.0879 --------

MEAN 92.8556 ---------

LOWER 95% CL 71.6232 ---------

LOWER 99% CL 64.9068 ||||||||

Batch Number 1 20

BIAS = -7.144%

99% CONFIDENCE LEVEL =± 30.09916 %
95% CONFIDENCE LEVEL =± 22.86603 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	102.3
2	23	96.2
17	23	87.7
18	23	116.2*
19	23	82.7
20	23	90.8
22	23	87.9
23	23	90.4
25	23	81.5

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET MANGANESE (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %155.2937 TTTTTTTTTT

UPPER 95% CL %143.0650 ----------

MEAN %104.4000 ----------

LOWER 95% CL 65.7350 ----------

LOWER 99% CL 53.5043 TTTTTTTTTT

Batch Number 1 21

BIAS = +4.400%

99% CONFIDENCE LEVEL =± 48.75068 %
95% CONFIDENCE LEVEL =± 37.0354 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	124.0
17	23	92.0
18	23	132.0
19	23	76.0
20	23	108.0
21	23	80.0
22	23	104.0
23	23	116.0
24	23	124.0
25	23	88.0

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET MOLYDEN (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 17.5 %

UPPER 99% CL %106.9847
UPPER 95% CL %104.1936

MEAN 95.3700

LOWER 95% CL 86.5464
LOWER 99% CL 83.7553

Batch Number 1 20

BIAS = -4.630%

99% CONFIDENCE LEVEL =± 12.17859 %
95% CONFIDENCE LEVEL =± 9.251957 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	102.5
2	23	92.2
17	23	92.6
18	23	89.5
19	23	89.9
20	23	96.8
21	23	98.7
22	23	99.8
23	23	93.0
25	23	98.7

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

NEW YORK STATE DEPARTMENT OF HEALTH
Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET NICKEL (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %124.6008

UPPER 95% CL %116.9238

MEAN 92.6545

LOWER 95% CL 68.3853

LOWER 99% CL 60.7082

Batch Number 1 20 25

BIAS = -7.345%

99% CONFIDENCE LEVEL =± 34.47894 %
95% CONFIDENCE LEVEL =± 26.19331 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	118.40
2	23	94.4
17	23	83.2
18	23	104.0
19	23	99.2
20	23	89.6
21	23	81.6
22	23	97.6
23	23	88.0
24	23	91.2
25	23	72.0

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET VANADIUM (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 RANGE MEAN +- 20 %

UPPER 99% CL	%108.1138	----------
UPPER 95% CL	%104.9493	----------
MEAN	94.9455	----------
LOWER 95% CL	84.9416	----------
LOWER 99% CL	81.7771	----------

Batch Number 1 20 25

BIAS = -5.055%

99% CONFIDENCE LEVEL =± 13.86934 %
95% CONFIDENCE LEVEL =± 10.5364 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	98.9
2	23	86.9
17	23	86.7
18	23	100.3
19	23	95.7
20	23	96.3
21	23	90.9
22	23	100.5
23	23	93.3
24	23	94.4
25	23	100.5

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET ZINC (SPIKE)

TARGET 100 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL %148.1756

UPPER 95% CL %136.9563

MEAN %101.4889

LOWER 95% CL 66.0215

LOWER 99% CL 54.8022

Batch Number 1 21

BIAS = +1.489%

99% CONFIDENCE LEVEL =± 46.00176 %
95% CONFIDENCE LEVEL =± 34.94708 %

Batch Number	OPERATOR	QUALITY CONTROL % recovery DATA
1	23	94.5
2	23	133.1
17	23	69.3
18	23	100.0
20	23	109.4
21	23	89.0
22	23	115.7
23	23	93.7
25	23	108.7

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET BARIUM (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL 9.9797 ||||||||||

UPPER 95% CL 8.3898 ----------

MEAN 3.3637 ----------

LOWER 95% CL -1.6624 ----------

LOWER 99% CL -3.2523 ||||||||||

Batch Number 1 20 25

BIAS = %+3.363E+05%

99% CONFIDENCE LEVEL =± 196.687 %
95% CONFIDENCE LEVEL =± 149.4211 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	2.8
2	23	1.4
17	23	4.9
18	23	3.4
19	23	0.001
20	23	2.7
21	23	8.6*
22	23	2.4
23	23	7.0
24	23	2.8
25	23	1.0

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET CHROMIUM (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 24 SCALED BY OUTLIER

UPPER 99% CL 6.3497 ||||||||

UPPER 95% CL 5.1284 ---------

MEAN 1.2673 ---------

LOWER 95% CL -2.5937 ---------

LOWER 99% CL -3.8151 ||||||||

Batch Number 1 21

BIAS = %+1.266E+05%

99% CONFIDENCE LEVEL =± 401.0313 %
95% CONFIDENCE LEVEL =± 304.6594 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	0.001
2	23	0.001
17	23	0.001
18	23	4.3
19	23	4.5
21	23	0.001
22	23	2.6
23	23	0.001
24	23	0.001

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET COPPER (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL 26.8144 TTTTTTTTTT

UPPER 95% CL 22.4061 ----------

MEAN 8.4700 ----------

LOWER 95% CL -5.4661 ----------

LOWER 99% CL -9.8745 TTTTTTTTTT

Batch Number 1 20

BIAS = %+8.469E+05%

99% CONFIDENCE LEVEL =± 216.5815 %
95% CONFIDENCE LEVEL =± 164.5348 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	8.0
2	23	3.6
17	23	3.7
18	23	11.1
19	23	1.5
20	23	1.0
21	23	20.6
23	23	6.2
24	23	8.5
25	23	20.5

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET IRON (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL 12.8861

UPPER 95% CL 10.9407

MEAN 4.7909

LOWER 95% CL -1.3589

LOWER 99% CL -3.3043

Batch Number 1 20 25

BIAS = %+4.790E+05%

99% CONFIDENCE LEVEL =± 168.9692 %
95% CONFIDENCE LEVEL =± 128.3642 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	5.7
2	23	1.8
17	23	5.3
18	23	3.1
19	23	6.9
20	23	2.6
21	23	4.8
22	23	12.6*
23	23	0.9
24	23	4.6
25	23	4.4

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
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QUALITY CONTROL SUMMARY SHEET LEAD (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 2 TO 25 SCALED BY OUTLIER

UPPER 99% CL 24.0716 ||||||||||

UPPER 95% CL 20.2383 ----------

MEAN 8.1201 ----------

LOWER 95% CL -3.9981 ----------

LOWER 99% CL -7.8314 ||||||||||

Batch Number 2 21

BIAS = %+8.119E+05%

99% CONFIDENCE LEVEL =± 196.4452 %
95% CONFIDENCE LEVEL =± 149.2374 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
2	23	4.7
17	23	15.4
18	23	0.001
19	23	1.0
20	23	7.3
21	23	15.2
22	23	6.9
23	23	13.9
24	23	2.2
25	23	14.6

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET MANGANES (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL 10.5017 ||||||||||

UPPER 95% CL 8.7974 ----------

MEAN 3.4094 ----------

LOWER 95% CL -1.9786 ----------

LOWER 99% CL -3.6830 ||||||||||

Batch Number 1 20 25

BIAS = %+3.408E+05%

99% CONFIDENCE LEVEL =± 208.0258 %
95% CONFIDENCE LEVEL =± 158.0351 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	0.001
2	23	0.001
17	23	4.2
18	23	2.5
19	23	4.3
20	23	3.7
21	23	7.8
22	23	7.4
23	23	0.001
24	23	2.6
25	23	5.0

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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Wadsworth Center for Laboratories and Research
Laboratory of Inorganic Analytical Cemistry

QUALITY CONTROL SUMMARY SHEET NICKEL (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL 16.9175 ||||||||||

UPPER 95% CL 13.9071 ----------

MEAN 4.3903 ----------

LOWER 95% CL -5.1265 ----------

LOWER 99% CL -8.1369 ||||||||||

Batch Number 1 21

BIAS = %+4.389E+05%

99% CONFIDENCE LEVEL =± 285.3377 %
95% CONFIDENCE LEVEL =± 216.7682 %

Batch Number	OPERATOR	% QUALITY CONTROL difference DATA
1	23	1.1
2	23	0.001
17	23	2.9
18	23	6.4
20	23	6.7
21	23	0.001
22	23	15.6*
23	23	4.3
24	23	6.9
25	23	0.001

DATA WHICH EICEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

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QUALITY CONTROL SUMMARY SHEET VANADIUM (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 95% CL 10.9880

MEAN 2.6205

LOWER 95% CL -5.7470

Batch Number 1 20

BIAS = %+2.619E+05%

99% CONFIDENCE LEVEL =± 420.3157 %
95% CONFIDENCE LEVEL =± 319.3097 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	2.3
2	23	0.001
17	23	5.6
18	23	2.2
19	23	13.6*
20	23	0.001
21	23	0.001
22	23	0.001
24	23	2.5
25	23	0.001

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

NEW YORK STATE DEPARTMENT OF HEALTH
Wadsworth Center for Laboratories and Research
Laboratory of Inorganic Analytical Cemistry

QUALITY CONTROL SUMMARY SHEET ZINC (DUPLICATES)

TARGET 0.001 MEAN COMPUTED FROM 1 TO 25 SCALED BY OUTLIER

UPPER 99% CL 18.8475

UPPER 95% CL 15.7922

MEAN 6.1333

LOWER 95% CL -3.5255

LOWER 99% CL -6.5809

Batch Number 1 20

BIAS = %+6.132E+05%

99% CONFIDENCE LEVEL =± 207.2966 %
95% CONFIDENCE LEVEL =± 157.4812 %

Batch Number	OPERATOR	QUALITY CONTROL % difference DATA
1	23	2.5
2	23	7.1
17	23	3.0
18	23	0.4
19	23	5.6
20	23	1.5
21	23	8.0
23	23	12.5
25	23	14.6

DATA WHICH EXCEED 95% CONFIDENCE LEVEL MARKED BY (*); 99% CONFIDENCE LEVEL (**)
95% CONFIDENCE LEVEL = 1.96σ 99% CONFIDENCE LEVEL = 2.58σ

Appendix C

Methodology

Method Summary

Arsenic

One sixth of each filter was digested in an acid bomb according to EPA Stack method 108, and analyzed by graphite furnace, EPA Method 206.2, EPA 600/4-79-020.

Metals by ICP

One twelfth of each filter was extracted with a nitric acid-hydrochloric acid mixture in an ultrasonic bath (final acid concentration of 1.6% nitric acid - 5% hydrochloric acid). Barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, nickel, vanadium, and zinc were digested using EPA method EQL-0380-043 and determined by ICP, EPA Method 200.7, EPA 600/4-79-020.

Mercury

One sixth of each filter was digested and analyzed by method 3112B, Cold Vapor Atomic Absorption after persulfate-permanganate digestion, Standard Methods for the Examination of Water and Wastewater, 17th Edition.

The minimum reportable concentrations of metals in the extracts are listed in Table C-I.

Analytical Methods are included at the end of this appendix.

Table C-I Minimum Reportable limits

Metal	ng/ml extract	ng/filter	ng/M^{3*}
Arsenic	10	3000	2
Barium	20	10000	5
Beryllium	4	2000	1
Cadmium	20	10000	5
Chromium	20	10000	5
Cobalt	20	10000	5
Copper	20	10000	5
Iron	40	20000	10
Lead	80	40000	20
Manganese	20	10000	5
Mercury	0.2	60	0.1
Molybdenum	80	40000	20
Nickel	20	10000	5
Vanadium	20	10000	5
Zinc	40	20000	10

* assuming 2000 M^3 air sampled.

Arsenic Digestion Procedure - Graphite Furnace Analysis

Each batch will contain 1 filter blank and 1 - 50 mg urban dust sample.

1. Place 2 strips (3/4" x 8") in the Teflon liner of the Parr Bomb (one sixth of sample).
2. Add 10 ml of concentrated nitric acid.
3. Close bomb and liner and place in 150°C oven for at least 5 hours.
4. Remove from oven and cool.
5. Open bomb and liner and pour off acid from the filter into a 50 ml volumetric flask. Rinse filter with several portions of distilled deionized water, pour rinse water into the volumetric flask. Bring to volume with distilled deionized water. Shake.
6. Evaporate a 10 ml portion of the solution to dryness.
7. Bring back to 10 ml volume with 0.5% nitric acid.
8. Pour in vial and give it to be analyzed using the graphite furnace.

<u>REAGENT</u>

o.5% nitric acid: dilute 5.0 ml concentrated acid to 1 liter.

Reference, Digestion Method - EPA Stack Method 108.

Reference, Graphite Furnace Method - EPA 600/4-79-020, 206.2.

ARSENIC

Method 206.2 (Atomic Absorption, furnace technique)

STORET NO. Total 01002
Dissolved 01000
Suspended 01001

Optimum Concentration Range: 5–100 ug/l
Detection Limit: 1 ug/l

Preparation of Standard Solution

1. Stock solution: Dissolve 1.320 g of arsenic trioxide, As_2O_3 (analytical reagent grade) in 100 ml of deionized distilled water containing 4 g NaOH. Acidify the solution with 20 ml conc. HNO_3 and dilute to 1 liter. 1 ml = 1 mg As (1000 mg/l).

2. Nickel Nitrate Solution, 5%: Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ in deionized distilled water and make up to 100ml.

3. Nickel Nitrate Solution, 1%: Dilute 20 ml of the 5% nickel nitrate to 100 ml with deionized distilled water.

4. Working Arsenic Solution: Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 ml of conc. HNO_3, 2ml of 30% H_2O_2 and 2ml of the 5% nickel nitrate solution. Dilute to 100 ml with deionized distilled water.

Sample Preservation

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample Preparation

1. Transfer 100 ml of well-mixed sample to a 250 ml Griffin beaker, add 2 ml of 30% H_2O_2 and sufficient conc. HNO_3 to result in an acid concentration of 1%(v/v). Heat for 1 hour at 95°C or until the volume is slightly less than 50 ml.

2. Cool and bring back to 50 ml with deionized distilled water.

3. Pipet 5 ml of this digested solution into a 10-ml volumetric flask, add 1 ml of the 1% nickel nitrate solution and dilute to 10 ml with deionized distilled water. The sample is now ready for injection into the furnace.

Approved for NPDES and SDWA
Issued 1978

NOTE: If solubilization or digestion is not required, adjust the HNO_3 concentration of the sample to 1% (v/v) and add 2 ml of 30%H_2O_2 and 2 ml of 5% nickel nitrate to each 100 ml of sample. The volume of the calibration standard should be adjusted with deionized distilled water to match the volume change of the sample.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec–125°C.
2. Ashing Time and Temp: 30 sec–1100°C.
3. Atomizing Time and Temp: 10 sec–2700°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 193.7 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Analysis Procedure

1. For the analysis procedure and the calculation, see "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 ul injection, continuous flow purge gas and non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.
2. The use of background correction is recommended.
3. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
4. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
5. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
6. Data to be entered into STORET must be reported as ug/l.

Precision and Accuracy

1. In a single laboratory (EMSL), using a mixed industrial-domestic waste effluent containing 15 ug/l and spiked with concentrations of 2, 10 and 25 ug/l, recoveries of 85%, 90% and 88% were obtained respectively. The relative standard deviation at these concentrations levels were ±8.8%, ±8.2%, ±5.4% and ±8.7%, respectively.
2. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 20, 50 and 100 ug As/l, the standard deviations were ±0.7, ±1.1 and ±1.6 respectively. Recoveries at these levels were 105%, 106% and 101%, respectively.

NEW YORK STATE DEPARTMENT OF HEALTH
DIVISION OF LABORATORIES AND RESEARCH

DETERMINATION OF LEAD CONCENTRATION IN AMBIENT PARTICULATE MATTER BY FLAME ATOMIC ABSORPTION SPECTROMETRY FOLLOWING ULTRASONIC EXTRACTION WITH HEATED HNO_3-HCl

EPA DESIGNATED EQUIVALENT METHOD NO. EQL-0380-043

1. Principle and Applicability

1.1 Ambient air suspended particulate matter is collected on a glass-fiber filter for 24-hours using a high volume air sampler. The analysis of the 24-hour samples may be performed for either individual samples or composites of the samples collected over a calendar month or quarter, provided that the compositing procedure has been approved in accordance with section 2.8 of Appendix C to Part 58 of Chapter I of Title 40, Code of Federal Regulations (40 CFR 58 -- 44 FR 27585, May 10, 1979).

1.2 Lead in the particulate matter is solubilized by ultrasonic extraction with a heated mixture of nitric acid (HNO_3) and hydrochloric acid (HCl).

1.3 The lead content of the sample is analyzed by atomic absorption spectrometry using an air-acetylene flame, the 283.3 or 217.0 nm lead absorption line, and the optimum instrumental conditions recommended by the manufacturer.

1.4 The ultrasonication extraction with heated HNO_3/HCl will extract metals other than lead from ambient particulate matter. (Do not use for Cr, Sn or Ti.)

2. Range, Sensitivity, and Lower Detectable Limit

The values given below are typical of the method's capabilities. Absolute values will vary for individual situations depending on the type of instrument used, the lead line, and operating conditions.

Accepted 7/80

2.1 Range. The typical range of the method is 0.07 to 7.5 μg Pb/m^3 assuming an upper linear range of analysis of 15 μg/ml and an air volume of 2400 m^3.

2.2 Sensitivity. Typical sensitivities for a 1% change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 Lower detectable limit (LDL). A typical LDL is 0.07 μg Pb/m^3. The above value was calculated by doubling the between-laboratory standard deviation obtained for the lowest measurable lead concentration in a collaborative test of a similar method.[15] An air volume of 2400 m^3 was assumed.

3. Interferences

Two types of interferences are possible: chemical, and light scattering.

3.1 Chemical. Reports on the absence [1,2,3,4,5] of chemical interferences far outweigh those reporting their presence,[6] therefore, no correction for chemical interferences is given here. If the analyst suspects that the sample matrix is causing a chemical interference, the interference can be verified and corrected for by carrying out the analysis with and without the method of standard additions.[7]

3.2 Light scattering. Nonatomic absorption or light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations.[2] The interference is greater at the 217.0 nm line than at the 283.3 nm line. No interference was observed using the 283.3 nm line with a similar method.[1]

Light scattering interferences can, however, be corrected for instrumentally. Since the dissolved solids can vary depending on the origin of the sample, the correction may be necessary, especially when using the 217.0 nm line. Dual beam instruments with a continuum source give the most

accurate correction. A less accurate correction can be obtained by using a nonabsorbing lead line that is near the lead analytical line. Information on use of these correction techniques can be obtained from instrument manufacturers' manuals.

If instrumental correction is not feasible, the interference can be eliminated by use of the ammonium pyrrolidinecarbodithioate-methylisobutyl ketone, chelation-solvent extraction technique of sample preparation.[8]

4. Precision and Bias

4.1 The high-volume sampling procedure used to collect ambient air particulate matter has a between-laboratory relative standard deviation of 3.7 percent over the range 80 to 125 $\mu g/m^3$.[9] The combined extraction-analysis procedure for a similar method[15] has an average within-laboratory relative standard deviation of 5 to 6 percent over the range 1.5 to 15 μg Pb/ml, and an average between-laboratory relative standard deviation of 7 to 9 percent over the same range.

5. Apparatus

5.1 Sampling.

5.1.1 High-volume sampler. Use and calibrate the sampler as described in reference 10.

5.2 Analysis.

5.2.1 Atomic absorption spectrophotometer. Equipped with lead hollow cathode or electrodeless discharge lamp.

5.2.1.1 Acetylene. The grade recommended by the instrument manufacturer should be used. Change cylinder when pressure drops below 50-100 psig.

5.2.1.2 Air. Filtered to remove particulate, oil, and water.

5.2.2 Labware.

5.2.2.1 Centrifuge tubes. 50-ml polypropylene tubes with polypropylene screw tops. Nalgene* 3119-0050 polyallomor or equivalent.

5.2.2.2 Volumetric flasks. (Class A borosilicate glass). 100-ml, 200-ml, 1000-ml.

5.2.2.3 Pipettes. (Class A borosilicate glass). To deliver 1, 2, 4, 8, 10, 15, 30, 50-ml. An automatic dispensing pipette capable of delivering 12.0 and 14.0 ml with an accuracy of 0.1 ml or better and a repeatability of 20 µl may be substituted for Class A pipettes used in sample preparation. Grumann* ADP-30T1 or equivalent.

5.2.2.4 Cleaning. All labware should be scrupulously cleaned. Wash with laboratory detergent (or ultrasonicate for 30 minutes in laboratory detergent), rinse, soak for a minimum of 4 hours in 20 percent (w/w) HNO_3, rinse 3 times with distilled-deionized water, and dry in a dust free manner.

5.2.3 Centrifuge. Capable of holding 50-ml centrifuge tubes and speed of 2500 RPM.

5.2.4 Ultrasonication water bath, heated. Commercially available laboratory ultrasonic cleaning baths of 450 watts or higher "cleaning power", (i.e., actual ultrasonic power output to the bath) and capable of maintaining 100°C have been found satisfactory, e.g., Branson Cleaning Equipment Co., model EMA-70-36 ultrasonicator.

5.2.5 Template. To aid in sectioning the glass-fiber filter. See Figure 1 for dimensions, or 1.75" punch.

5.2.6 Pizza cutter. Thin wheel. Thickness <1 mm.

5.2.7 Polyethylene bottles. For storage of samples. Linear polyethylene gives better storage stability than other polyethylenes and is preferred.

6. Reagents

6.1 Sampling.

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*Mention of commercial products does not imply endorsement by the U.S. Environmental Protection Agency.

6.1.1 Glass fiber filters. The specifications given below are intended to aid the user in obtaining high quality filters with reproducible properties. These specifications have been met by EPA contractors.

6.1.1.1 Lead content. The absolute lead content of filters is not critical, but low values are, of course, desirable. EPA typically obtains filters with a lead content of <75 μg/filter.

It is important that the variation in lead content from filter to filter, within a given batch, be small.

6.1.1.2 Testing.

6.1.1.2.1 For large batches of filters (> 500 filters) select at random 20 to 30 filters from a given batch. For small batches (< 500 filters) a lesser number of filters may be taken. Cut one 3/4" x 8" or 1" x 8" strip or 2 discs (8% total) from each filter anywhere in the filter. Analyze all strips or 2 discs, separately, according to the directions in Sections 7 and 8.

6.1.1.2.2 Calculate the total lead in each filter as

$$F_b = \mu g\ Pb/ml \times \frac{40\ ml}{strip} \times \frac{n\ strips}{filter}$$

where:

F_b = Amount of lead per 72 square inches of filter, μg.

n = 12 (for 3/4" x 8" strip) or 9 (for 1" x 8" strip), or $\frac{100\%}{8\%}$ for discs.

6.1.1.2.3 Calculate the mean, $\overline{F}_b$, of the values and the relative standard deviation (standard deviation/mean x 100). If the relative standard deviation is high enough so that, in the analysts' opinion, subtraction of $\overline{F}_b$ (Section 10.2) may result in a significant error in the μg Pb/m^3, the batch should be rejected.

6.1.1.2.4 For acceptable batches, use the value of $\overline{F}_b$ to correct all lead analyses (Section 10.2) of particulate matter collected using that batch of filters. If the analyses are below the LDL (Section 2.3) no correction is necessary.

6.2 Analysis.

6.2.1 Concentrated (16.0 M) HNO_3. ACS reagent grade HNO_3 and commercially available redistilled HNO_3 have been found to have sufficiently low lead concentrations.

6.2.2 Concentrated (12.3 M) HCl. ACS reagent grade.

6.2.3 Distilled-deionized water. (D.I. water).

6.2.4 Extracting acid (1.03 M HNO_3 + 2.23 M HCl). This solution is used in the extraction procedure. To prepare, place 500 ml of D.I. water in a 1000-ml volumetric flask and add 64.6 ml of concentrated HNO_3 and 182 ml of concentrated HCl. Shake well, cool, and dilute to volume with D.I. water. Caution: Acid fumes are toxic. Prepare in a well ventilated fume hood.

6.2.5 Calibration matrix (0.31 M HNO_3 + 0.67 M HCl). This solution is used as the matrix for calibration standards. To prepare, place 500 ml of D.I. water in a 1000-ml volumetric flask and add 19.4 ml of concentrated HNO_3 and 54.6 ml of concentrated HCl. Shake well, cool, and dilute to volume with D.I. water.

6.2.6 Lead nitrate, $Pb(NO_3)_2$. ACS reagent grade, purity 99.0 percent. Heat for 4-hours at 120°C and cool in a desiccator.

6.3 Calibration standards.

6.3.1 Master standard, 1000 µg Pb/ml in HNO_3/HCl. Dissolve 1.598 g of $Pb(NO_3)_2$ in 0.31 M HNO_3 + 0.67 M HCl (Section 6.2.5) contained in a 1000-ml volumetric flask and dilute to volume with 0.31 M HNO_3 + 0.67 M HCl. Store standard in a polyethylene bottle. Commercially available certified lead standard solutions may also be used.

7. Procedure

7.1 Sampling. Collect samples for 24-hours using the procedure described in reference 10 with glass-fiber filters meeting the specifications in 6.1.1. Transport collected samples to the laboratory taking care to minimize contamination and loss of sample.[16]

7.2 Sample preparation.

7.2.1 Extraction procedure.

7.2.1.1 Cut a 3/4" x 8" or 1" x 8" strip or 2 discs (8%) from the exposed filter using a template and a pizza cutter as described in Figures 1 and 2 or 1.75 punch. Other cutting procedures may be used.

Lead in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter.[1,3,11] Another study[12] has shown that when sampling near a roadway, strip position contributes significantly to the overall variability associated with lead analyses. Therefore, when sampling near a roadway, additional strips or discs shc be analyzed to minimize this variability.

7.2.1.2 Using vinyl gloves or plastic forceps, accordion fold or tightly roll the filter strip and place on its edge in a 50-ml polypropylene centrifuge tube. Add 12.0 ml of the extracting acid (Section 6.2.4) with pipettes or the automatic dispensing pipette. The acid should completely cover the sample. Cap the tube loosely (finger tight) with the polypropylene screw top. Caution: Centrifuge tubes must be loosely capped to prevent elevated pressures during ultrasonication at elevated temperatures and will not withstand repeated cycling to elevated pressures.

7.2.1.3 Label the centrifuge tube, place in a sample rack, and place upright in the preheated (100°C) ultrasonic water bath (in fume hood) so that the water level is slightly above the acid level in the centrifuge tubes but

well below the centrifuge tube caps. This will prevent contamination of the samples during ultrasonication. Ultrasonicate the sample at 100°C for 50 minutes.

7.2.1.4 Remove the centrifuge tube from the ultrasonic bath and allow to cool.

7.2.1.5 Uncap the centrifuge tube in the fume hood and add 28.0 ml of D.I. water with pipettes or the automatic dispensing pipette. Recap the tube tightly, shake well, and centrifuge for 20 minutes at 2500 RPM.

7.2.1.6 Decant the extract into a clean polyethylene storage bottle bearing the sample I.D. Be careful not to disturb any solids in the bottom of the tube. Cap the bottle tightly and store until analysis. The final extract is now in 0.31 M HNO_3 + 0.67 M HCl.

8. Analysis

8.1 Set the wavelength of the monochromator at 283.3 or 217.0 nm. Set or align other instrumental operating conditions as recommended by the manufacturer.

8.2 The sample can be analyzed directly from the polyethylene storage bottle, or an appropriate amount of sample can be transferred to a sample analysis tube.

8.3 Aspirate samples, calibration standards, and blanks (Section 9.2) into the flame and record the equilibrium absorbance.

8.4 Determine the lead concentration in µg Pb/ml, from the calibration curve, Section 9.3.

8.5 Samples that exceed the linear calibration range should be diluted with acid of the same concentration (Section 6.2.5) as the calibration standards and reanalyzed.

9. Calibration

9.1 Working standard, 20 μg Pb/ml. Prepared by diluting 2.0 ml of the master standard (Section 6.3.1) to 100 ml with acid of the same concentration (Section 6.2.5) as used in preparing the master standard.

9.2 Calibration standards. Prepare daily by diluting the working standard, with the same acid matrix, as indicated below. Other lead concentrations may be used.

Volume of 20 μg/ml Working Standard, ml	Final Volume, ml	Concentration μg Pb/ml
0	100	0.0
1.0	200	0.1
2.0	200	0.2
2.0	100	0.4
4.0	100	0.8
8.0	100	1.6
15.0	100	3.0
30.0	100	6.0
50.0	100	10.0
100.0	100	20.0

9.3 Preparation of calibration curve. Since the working range of analysis will vary depending on which lead line is used and the type of instrument, no one set of instructions for preparation of a calibration curve can be given. Select standards (plus the reagent blank), in the same acid concentration as the samples, to cover the linear absorption range indicated by the instrument manufacturer. Measure the absorbance of the blank and standards as in Section 8.0. Repeat until good agreement is obtained between replicates. Plot absorbance

(y-axis) versus concentration in μg Pb/ml (x-axis). Draw (or compute) a straight line through the linear portion of the curve. Do not force the calibration curve through zero. Other calibration procedures may be used.

To determine stability of the calibration curve, remeasure - alternately - one of the following calibration standards for every 10th sample analyzed: concentration $\leq$ 1 μg Pb/ml; concentration $\leq$ 10 μg Pb/ml. If either standard deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

10. Calculation.

10.1 Measured air volume. Calculate the measured air volume at standard temperature and pressure as described in reference 10.

10.2 Lead concentration. Calculate lead concentration in the air sample.

$$C = \frac{(\mu g\ Pb/ml \times 40\ ml/strip \times n\ strips/filter) - \bar{F}_b}{V_{STP}}$$

where:

C = Concentration, μg Pb/sm^3.

μg Pb/ml = Lead concentration determined from Section 8.

40 ml/strip = Total sample volume.

n = 12 (for 3/4" x 8" strip) or 9 (for 1" x 8" strip) strips per filter, or $\frac{100\%}{8\%}$ for discs.

$\bar{F}_b$ = Mean lead concentration of blank filter, μg, from Section 6.1.1.2.3.

V_{STP} = Air volume from 10.1.

11. Quality Control

Glass-fiber filter strips (3/4" x 8" or 1" x 8") containing 80 to 2000 μg Pb/strip (as lead salts) and blank strips with zero Pb content should be used to determine if the method - as being used - has any bias. Quality control charts should be established to monitor differences between measured and true values. The frequency of such checks will depend on the local quality control program.

To minimize the possibility of generating unreliable data, the user should follow practices established for assuring the quality of air pollution data,[13] and take part in EPA's semiannual audit program for lead analyses.[16]

12. Trouble Shooting

1. The sample acid concentration should minimize corrosion of the nebulizer. However, different nebulizers may require lower acid concentrations. Lower concentrations can be used provided samples and standards have the same acid concentration.

2. Ashing of particulate samples has been found, by EPA and contractor laboratories, to be unnecessary in lead analyses by atomic absorption. Therefore, this step was omitted from the method.

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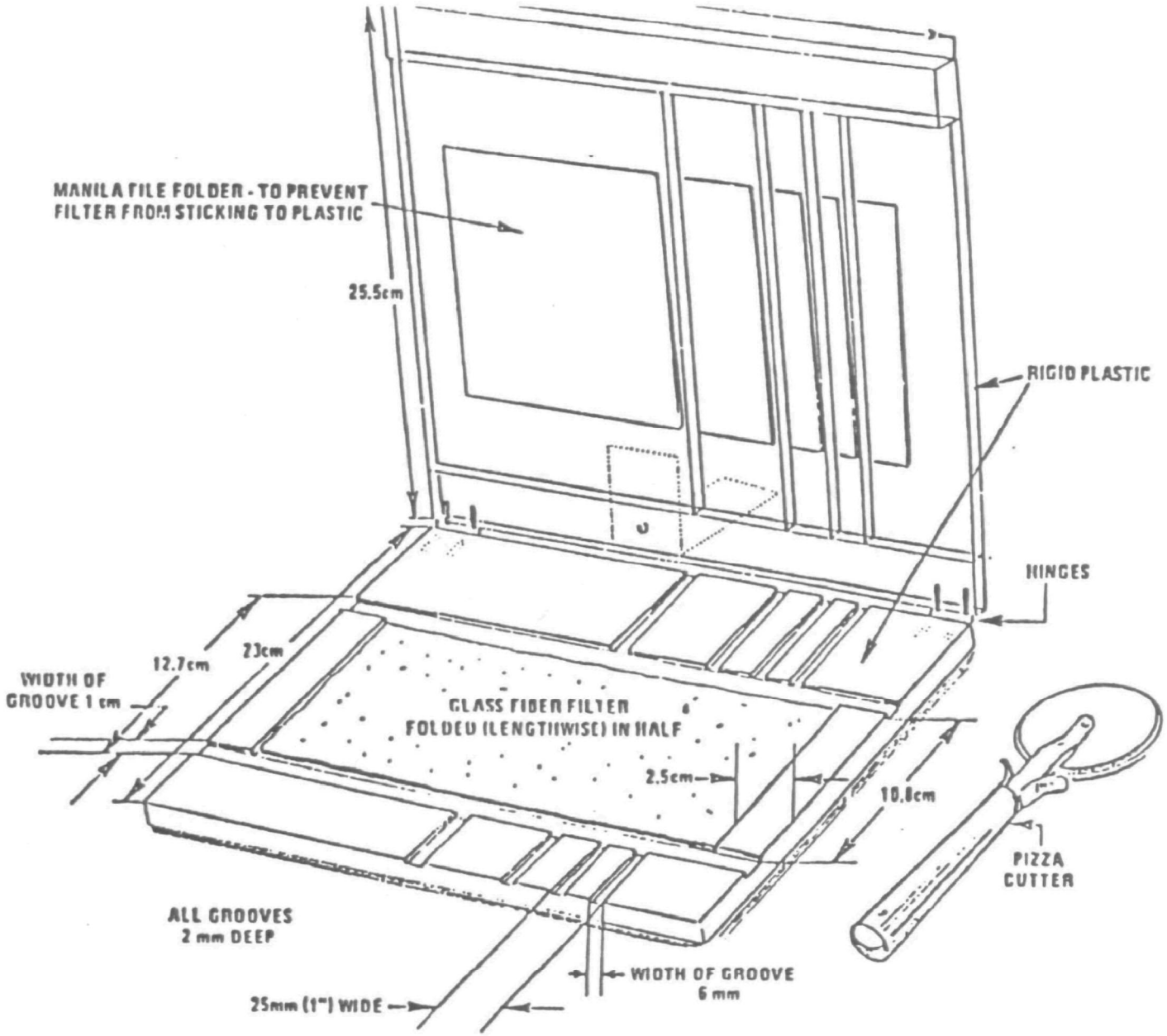
MANILA FILE FOLDER - TO PREVENT
FILTER FROM STICKING TO PLASTIC
25.5cm
RIGID PLASTIC
HINGES
12.7cm
23cm
WIDTH OF
GROOVE 1 cm
GLASS FIBER FILTER
FOLDED (LENGTHWISE) IN HALF
2.5cm
10.8cm
PIZZA
CUTTER
ALL GROOVES
2 mm DEEP
WIDTH OF GROOVE
6 mm
25mm (1") WIDE

Figure 1

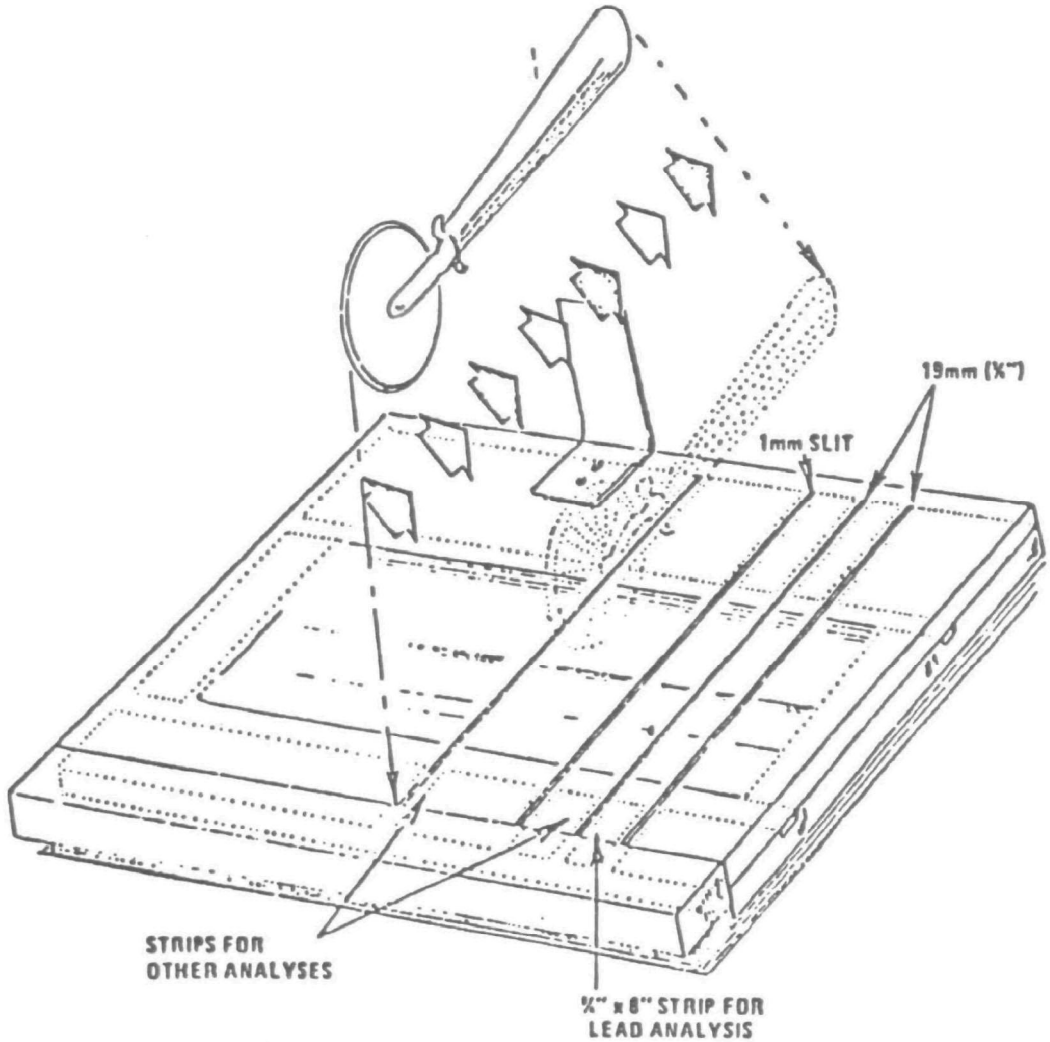
19mm (¾")
1mm SLIT
STRIPS FOR
OTHER ANALYSES
¾" x 8" STRIP FOR
LEAD ANALYSIS

Figure 2

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Inductively Coupled Plasma—Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes—Method 200.7

1. Scope and Application

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, domestic and industrial wastewaters

1.2 Dissolved elements are determined in filtered and acidified samples Appropriate steps must be taken in all analyses to ensure that potential interference are taken into account This is especially true when dissolved solids exceed 1500 mg/L. (See 5)

1.3 Total elements are determined after appropriate digestion procedures are performed Since digestion techniques increase the dissolved solids content of the samples, appropriate steps *must* be taken to correct for potential interference effects (See 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary In time, other elements may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument

2. Summary of Method

2.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the

determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

3. Definitions

3.1 Dissolved — Those elements which will pass through a 0.45 μm membrane filter.

3.2 Suspended — Those elements which are retained by a 0.45 μm membrane filter.

3.3 Total — The concentration determined on an unfiltered sample following vigorous digestion (9.3), or the sum of the dissolved plus suspended concentrations (9.1 plus 9.2).

3.4 Total recoverable — The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (9.4).

3.5 Instrumental detection limit — The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 Sensitivity — The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 Instrument check standard — A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 7.6.1)

3.8 Interference check sample — A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors. (See 7.6.2)

3.9 Quality control sample — A solution obtained from an outside source having known concentration values to be used to verify the calibration standards. (See 7.6.3)

3.10 Calibration standards — a series of know standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 7.4)

3.11 Linear dynamic range — The concentration range over which the analytical curve remains linear.

3.12 Reagent blank — A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 7.5.2)

3.13 Calibration blank — A volume of deionized, distilled water acidified with HNO_3 and HCl. (See 7.5.1)

3.14 Method of standard addition — The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard. (See 10.6.1)

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified (14.7, 14.8 and 14.9) for the information of the analyst.

5. Interferences

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

5.1.1 *Spectral interferences* can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multielement instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

The interference information, which was collected at the Ames Laboratory,[1] is expressed at analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interferent element. The suggested use of this information is as follows. Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of aluminum. According to Table 2, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system.

Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed for the interferent concentrations listed in Table 3. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2-5% of the peaks generated by the

[1] Ames Laboratory, USDOE, Iowa State University, Ames, Iowa 50011.

analyte concentrations also listed in Table 3

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that second order energy from the magnesium 383 231 nm wavelength interferes with the listed potassium line at 766.491 nm

5.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aersol flow-rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

5.1.3 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

5.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 5.2.1 through 5.2.4, will ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.2.1 Serial dilution—If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 5 % of the original determination (or within some acceptable control limit (14.3) that has been established for that matrix). If not, a chemical or physical interference effect should be suspected.

5.2.2 Spike addition—The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect. *Caution:* The standard addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended. (See 5.2.3)

5.2.3 Comparison with alternate method of analysis—When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.

5.2.4 Wavelength scanning of analyte line region—If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

6. Apparatus

6.1 Inductively Coupled Plasma-Atomic Emission Spectrometer.

6.1.1 Computer controlled atomic emission spectrometer with background correction.

6.1.2 Radiofrequency generator.

6.1.3 Argon gas supply, welding grade or better.

6.2 Operating conditions — Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

7. Reagents and standards

7.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

7.1.1 Acetic acid, conc. (sp gr 1.06).

7.1.2 Hydrochloric acid, conc. (sp gr 1.19)

7.1.3 Hydrochloric acid, (1+1). Add 500 mL conc. HCl (sp gr 1.19) to 400 mL deionized, distilled water and dilute to 1 liter.

7.1.4 Nitric acid, conc. (sp gr 1.41)

7.1.5 Nitric acid, (1+1). Add 500 mL conc. HNO_3 (sp. gr 1.41) to 400 mL deionized, distilled water and dilute to 1 liter.

7.2 *Dionized, distilled water:* Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (14.6).

7.3 *Standard stock solutions* may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 h at 105°C unless otherwise specified.
(CAUTION. Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.) Typical stock solution preparation procedures follow:

7.3.1 Aluminum solution, stock, 1 mL = 100 µg Al: Dissolve 0.100 g of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 mL of (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.2 Antimony solution stock, 1 mL = 100 µg Sb: Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 mL (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

7.3.3 Arsenic solution, stock 1 mL = 100 µg As Dissolve 0.1320 g of As_2O_3 in 100 mL of deionized distilled water containing 0.4 g NaOH Acidify the solution with 2 mL conc HNO_3 and dilute to 1.000 mL with deionized distilled water

7.3.4 Barium solution, stock 1 mL = 100 µg Ba Dissolve 0.1516 g $BaCl_2$ (dried at 250°C for 2 hrs) in 10 mL deionized distilled water with 1 mL (1+1) HCl Add 10.0 mL (1+1) HCl and dilute to 1.000 mL with deionized, distilled water

7.3.5 Beryllium solution, stock, 1 mL = 100 µg Be *Do not dry* Dissolve 1.966 g $BeSO_4 \cdot 4\ 4H_2O$, in deionized distilled water add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.6 Boron solution, stock 1 mL = 100 µg B *Do not dry* Dissolve 0.5716 g anhydrous H_3BO_3 in deionized distilled water dilute to 1.000 mL Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture

7.3.7 Cadmium solution, stock, 1 mL = 100 µg Cd Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3 Heat to increase rate of dissolution Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.8 Calcium solution, stock, 1 mL = 100 µg Ca Suspend 0.2498 g $CaCO_3$ dried at 180°C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3 Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized distilled water

7.3.9 Chromium solution, stock, 1 mL = 100 µg Cr Dissolve 0.1923 g of CrO_3 in deionized, distilled water. When solution is complete, acidify with 10 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water.

7.3.10 Cobalt solution, stock, 1 mL = 100 µg Co Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) HNO_3 Add 10.0 mL (1+1) HCl and dilute to 1.000 mL with deionized, distilled water

7.3.11 Copper solution, stock, 1 mL = 100 µg Cu Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3 Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.12 Iron solution, stock 1 mL = 100 µg Fe Dissolve 0.1430 g Fe_2O_3 in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc HNO_3 Cool add an additional 5 mL of conc HNO_3 and dilute to 1000 mL with deionized distilled water

7.3.13 Lead solution, stock 1 mL = 100 µg Pb Dissolve 0.1599 g $Pb(NO_3)_2$ in minimum amount of (1+1) HNO_3. Add 10.0 mL conc HNO_3, and dilute to 1.000 mL with deionized distilled water

7.3.14 Magnesium solution, stock, 1 mL = 100 µg Mg Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3 Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.15 Manganese solution, stock, 1 mL = 100 µg Mn Dissolve 0.1000 g of manganese metal in the acid mixture 10 mL conc HCl and 1 mL conc HNO_3, and dilute to 1.000 mL with deionized distilled water

7.3.16 Molybdenum solution, stock, 1 mL = 100 µg Mo Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized distilled water and dilute to 1.000 mL

7.3.17 Nickel solution, stock 1 mL = 100 µg Ni Dissolve 0.1000 g of nickel metal in 10 mL hot conc HNO_3, cool and dilute to 1.000 mL with deionized, distilled water

7.3.18 Potassium solution, stock, 1 mL = 100 µg K Dissolve 0.1907 g KCl dried at 110°C, in deionized distilled water dilute to 1.000 mL

7.3.19 Selenium solution, stock 1 mL = 100 µg Se *Do not dry* Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in deionized, distilled water and dilute to 1.000 mL

7.3.20 Silica solution, stock, 1 mL = 100 µg SiO_2 *Do not dry* Dissolve 0.4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water. Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.21 Silver solution, stock, 1 mL = 100 µg Ag Dissolve 0.1575 g $AgNO_3$ in 100 mL of deionized, distilled water and 10 mL conc HNO_3 Dilute to 1.000 mL with deionized, distilled water

7.3.22 Sodium solution, stock, 1 mL = 100 µg Na Dissolve 0.2542 g NaCl in deionized, distilled water Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.23 Thallium solution, stock 1 mL = 100 µg Tl Dissolve 0.1303 g $TlNO_3$ in deionized distilled water Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.24 Vanadium solution, stock 1 mL = 100 µg V Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc HNO_3 Heat to increase rate of dissolution Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.3.25 Zinc solution, stock, 1 mL = 100 µg Zn Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3 Add 10.0 mL conc HNO_3 and dilute to 1.000 mL with deionized, distilled water

7.4 Mixed calibration standard solutions—Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (See 7.4.1 thru 7.4.5) Add 2 mL of (1+1) HCl and dilute to 100 mL with deionized distilled water (See Notes 1 and 6.) Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities Care should be taken when preparing the mixed standards that the elements are compatible and stable Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging Calibration standards must be initially verified using a quality control sample and monitored weekly for stability (See 7.6.3) Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 1

7.4.1 Mixed standard solution I—Manganese, beryllium, cadmium, lead, and zinc

7.4.2 Mixed standard solution II—Barium, copper, iron, vanadium, and cobalt

7.4.3 Mixed standard solution III—Molybdenum, silica, arsenic, and selenium

7.4.4 Mixed standard solution IV—Calcium, sodium, potassium, aluminum, chromium and nickel

7.4.5 Mixed standard solution V— Antimony, boron, magnesium, silver, and thallium

NOTE 1 If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of deionized distilled water and warm the flask until the solution clears Cool and dilute to 100 mL with deionized, distilled water For this acid combination the silver concentration should be limited to 2 mg/L Silver under these conditions is stable in a tap water matrix for 30 days Higher concentrations of silver require additional HCl

7.5 Two types of blanks are required for the analysis. The calibration blank (3 13) is used in establishing the analytical curve while the reagent blank (3 12) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing

7.5.1 The calibration blank is prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL with deionized, distilled water (See Note 6) Prepare a sufficient quantity to be used to flush the system between standards and samples

7.5.2 The reagent blank must contain all the reagents and in the same volumes as used in the processing of the samples The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.6 In addition to the calibration standards, an instrument check standard (3.7), an interference check sample (3 8) and a quality control sample (3.9) are also required for the analyses

7.6.1 The *instrument check standard* is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. (See 12.1.1)

7.6.2 The *interference check sample* is prepared by the analyst in the following manner. Select a representative sample which contains minimal concentrations of the analytes of interest by known concentration of interfering elements that will provide an adequate test of the correction factors Spike the sample with the elements of interest at the approximate concentration of either 100 µg/L or 5 times the estimated detection limits given in Table 1 (For effluent samples of expected high concentrations, spike at an appropriate level) If the type of samples analyzed are varied, a synthetically prepared sample may be used if the above criteria and intent are met A limited supply of a synthetic interference check sample will be available from the Quality Assurance Branch of EMSL-Cincinnati (See 12 1 2)

7.6.3 The *quality control sample* should be prepared in the same acid matrix as the calibration standards at a concentration near 1 mg L and in accordance with the instructions provided by the supplier The Quality Assurance Branch of EMSL-Cincinnati will either supply a quality control sample or information where one of equal quality can be procured (See 12.1 3)

8. Sample handling an preservation

8.1 For the determination of trace elements, contamination and loss are of prime concern Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether polyethylene, polypropylene or FEP-fluorocarbon) should be thoroughly washed with detergent and tap water; rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap and finally deionized, distilled water in that order (See Notes 2 and 3).

NOTE 2: Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid. Chomic acid should not be used with plastic bottles

NOTE 3 If it can be documented through an active analytical quality control program using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure

8.2 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter

8.2.1 For the determination of dissolved elements the sample must be filtered through a 0.45-µm membrane filter as soon as practical after collection (Glass or plastic filtering apparatus are recommended to avoid possible contamination) Use the first 50-100 mL to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH of 2 or less. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

8.2.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-µm membrane filter as soon as practical after collection The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

8.2.3 For the determination of total or total recoverable elements, the sample is acidified with (1+1) HNO_3 to pH 2 or less as soon as possible, preferable at the time of collection. The sample is not filtered before processing.

9. Sample Preparation

9.1 For the determinations of dissolved elements, the filtered, preserved sample may often be analyzed as received. The acid matrix and concentration of the samples and calibration standards must be the same. (See Note 6.) If a precipitate formed upon acidification of the sample or during transit or storage, it must be redissolved before the analysis by adding additional acid and/or by heat as described in 9.3.

9.2 For the determination of suspended elements, transfer the membrane filter containing the insoluble material to a 150-mL Griffin beaker and add 4 mL conc HNO_3. Cover the

beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane.

Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated cool the beaker and watch glass and add another 3 mL of conc HNO_3. Cover and continue heating until the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (2 mL), cool, add 10 mL HCl (1+1) and 15 mL deionized, distilled water per 100 mL dilution and warm the beaker gently for 15 min to dissolve any precipitated or residue material. Allow to cool, wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined. (See Note 6.) The sample is now ready for analysis. Concentrations so determined shall be reported as "suspended."

NOTE 4. In place of filtering, the sample after diluting and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

9.3 For the determination of total elements, choose a measured volume of the well mixed acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 3 mL of conc HNO_3. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil and that no area of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 5 mL portion of conc HNO_3. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.) Again, evaporate to near dryness and cool the beaker. Add 10 mL of 1+1 HCl and 15 mL of deionized, distilled water per 100 mL of final solution and warm the beaker gently for 15 min to dissolve any precipitate or residue resulting from evaporation. Allow to cool, wash down the beaker walls and watch glass with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the sample to a predetermined volume based on the expected concentrations of elements present. The sample is now ready for analysis. (See Note 6.) Concentrations so determined shall be reported as "total."

NOTE 5. If low determinations of boron are critical, quartz glassware should be use.

NOTE 6. If the sample analysis solution has a different acid concentration from that given in 9.4, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

9.4 For the determination of total recoverable elements, choose a measured volume of a well mixed, acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to the sample and heat on a steam bath or hot plate until the volume has been reduced to near 25 mL making certain the sample does not boil. After this treatment, cool the sample and filter to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume to 100 mL and mix. The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

10. Procedure

10.1 Set up instrument with proper operating parameters established in 6.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 min of operation prior to calibration.

10.2 Initiate appropriate operating configuration of computer.

10.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in 7.4. Flush the system with the calibration blank (7.5.1) between each standard. (See Note 7.) (The use of the average intensity of multiple exposures for both standardization and sample analysis has been found to reduce random error.)

NOTE 7. For boron concentrations greater than 500 µg/L extended flush times of 1 to 2 min may be required.

10.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than ± 5 percent (or the established control limits whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

10.5 Begin the sample run flushing the system with the calibration blank solution (7.5.1) between each sample. (See Note 7.) Analyze the instrument check standard (7.6.1) and the calibration blank (7.5.1) each 10 samples.

10.6 If it has been found that method of standard addition are required, the following procedure is recommended.

10.6.1 The standard addition technique (14.2) involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive interference which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a small volume V_s of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculated:

$$c_x = \frac{S_B V_s c_s}{(S_A - S_B) V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.

3 The interference effect must be constant over the working range of concern
4 The signal must be corrected for any additive interference

11. Calculation

11.1 Reagent blanks (7 5.2) should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

11.2 If dilutions were performed, the appropriate factor must be applied to sample values

11.3 Data should be rounded to the thousandth place and all results should be reported in mg L up to three significant figures.

12. Quality Control (Instrumental)

12.1 Check the instrument standardization by analyzing appropriate quality control check standards as follow

12.1.1 Analyze an appropriate instrument check standard (7 6 1) containing the elements of interest at a frequency of 10% This check standard is used to determine instrument drift. If agreement is not within ±5% of the expected values or within the established control limits, whichever is lower, the analysis is out of control The analysis should be terminated, the problem corrected, and the instrument recalibrated

Analyze the calibration blank (7.5.1) at a frequency of 10%. The result should be within the established control limits of two standard deviations of the mean value. If not, repeat the analysis two more times and average the three results. If the average is not within the control limit, terminate the analysis, correct the problem and recalibrate the instrument.

12.1.2 To verify interelement and background correction factors analyze the interference check sample (7.6.2) at the beginning, end, and at periodic intervals throughout the sample run. Results should fall within the established control limits of 1.5 times the standard deviation of the mean value. If not, terminate the analysis, correct the problem and recalibrate the instrument.

12.1.3 A quality control sample (7.6.3) obtained from an outside source must first be used for the initial verification of the calibration standards. A fresh dilution of this sample shall be anlayzed every week thereafter to monitor their stability. If the results are not within ±5% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

Precision and Accuracy

13.1 In an EPA round robin phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been dosed with various metal concentrates. Table 4 lists the true value, the mean reported value and the mean % relative standard deviation

References

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Table 1. Recommended Wavelengths [1] and Estimated Instrumental Detection Limits

Element	Wavelength, nm	Estimated detection limit, µg/L[2]
Aluminum	308.215	45
Arsenic	193.696	53
Antimony	206.833	32
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	see[3]
Selenium	196.026	75
Silica (SiO_2)	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[1] *The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 5.1.1.).*

[2] *The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines, "EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.*

[3] *Highly dependent on operating conditions and plasma position*

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION ANALYSIS OF DRINKING WATER

APPENDIX TO METHOD 200.7

"Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes"

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Physical and Chemical Methods Branch
Environmental Monitoring and Support Laboratory

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1. Scope and Application

1.1 This procedure is designed to be a supplement to Method 200.7 (1) and is to be used in processing drinking water supply samples prior to inductively coupled plasma-atomic emission spectrometric (ICP-AES) analysis. This appendix does not supercede Method 200.7, but provides elaboration on the analysis of drinking water using Method 200.7. For a listing of the recommended wavelengths, Definitions, and discussions on Safety, Reagents and Standards, and Sample Handling and Preservation see the appropriate Sections of Method 200.7.

1.2 This procedure is to be used for the total element determination of primary and secondary elemental drinking water contaminants included in Method 200.7. It is only to be used for compliance monitoring when the determined method detection limit (MDL) (2) for a particular contaminant is no greater than 1/5 its respective maximum contaminant level (MCL) concentration. For these reasons, mercury and selenium have been omitted from this edition of the appendix. A listing of the contaminants for which the procedure is applicable along with their MCLs and MDLs is given as Table 1.

1.3 This procedure is to be used in all pneumatic nebulization ICP analyses for compliance monitoring of drinking water, and is recommended for the analysis of ground and surface water where determination at the drinking water MCL is requested.

1.4 This procedure also can be used to determine the concentration of calcium (Ca) for calculating corrosivity and for the required monitoring of sodium (Na). Since these two elements can occur in waters at concentrations greater than 25 mg/L, particular care must be taken that concentrating the sample does not cause the analysis of these two elements to exceed the calibration limit of linearity. If standardization of the instrument does not include provision for non-linear calibration, a more convenient and allowable determination of these two elements is the direct aspiration analysis of the acidified unprocessed sample.

2. Summary of Method

2.1 For a description of the analytical technique and method summary see Section 2 of Method 200.7.

2.2 Analytical Discussion

2.2.1 The analysis of drinking water for elemental contaminants requires that a "total" element determination be made. Irrespective of the valence state or chemical species, the term "total" refers to the sum of the elemental concentration in the dissolved and suspended fractions of the sample. The sample is not filtered, but immediately preserved with nitric acid to pH of less than 2 at the time of collection.

2.2.2 Although most finished drinking waters are free of suspended matter, all samples must be subjected to a pretreatment acid dissolution to solubilize that portion of the contaminant that may be occluded or adhering to minute suspended matter. This is especially true for water supplies that receive only chlorination pretreatment. Once solubilized, the energy of the plasma is sufficient that all species in the nebulized droplets are desolvated, dissociated and raised to an energetic excited state for atomic emission spectrometric analysis.

2.2.3 Method 200.7 describes two acceptable sample preparation procedures for "total" element analyses. One is a vigorous nitric acid digestion (Section 9.3), while the other is a total recoverable acid solubilization procedure (Section 9.4). These procedures are essentially the same as those used for flame atomic absorption analysis, except the final acid concentration has been changed to match the ICP calibration standards. The total recoverable procedure is preferred for drinking water analyses because there is less chance of losses from volatilization, the formation of insoluble oxides or occlusion in precipitated silicates.

2.2.4 Data that are to be used for compliance monitoring should be reported with a known estimate of uncertainty. The uncertainty of the analysis should be determined at the critical MCL concentration and should be a precision of small enough variance to determine that the contaminant is either in-or-out of compliance. A guide for evaluating data to be reported can be described as data with sufficient precision at the MCL, that when two standard deviations are either added to or subtracted from the MCL concentration, the value is not changed by more than 10%. An example is As (MCL = 0.05 mg/L) where data reported with a precision of two standard deviations equal to less than 0.005 mg/L would be acceptable as shown in the preconcentration data of Table 2 with the interval values of 0.048 to 0.052 mg/L.

2.2.5 As indicated in Table 1, the MCLs for As and Pb are close to their estimated instrumental detection limits. A single analysis of these two elements using the total recoverable procedure 9.4 of Method 200.7 lacks the precision needed for compliance monitoring at their respective MCLs. As a consequence inaccurate determinations can result. Only with repeated analyses of the sample can an average value with acceptable precision be determined. The number of analyses required can be specified by the following equation:

$$n = \left(\frac{Sa}{Sx}\right)^2$$

where: n = the number of replicate analyses required,

Sa = the determined standard deviation of a single observation, and

Sx = the standard deviation deemed acceptable around the mean value for n determinations.

Using the preceding equation the number of repeated analyses required for the procedure 9.4 can be calculated from the direct analysis standard deviation data given in Table 2. For each element the listed determined standard deviation is Sa and the acceptable standard deviation is Sx. From the calculation the number (n) of repeated analyses required for As is 8, while for Pb the number is 6. (Note: From the standard deviation data listed for analysis after 4X concentration, the number for both elements is 1.)

2.2.6 The drinking water procedure that follows (5.1) is a modification of the total recoverable procedure 9.4 Method 200.7 that provides for improved precision and accuracy by concentrating the contaminants 4X prior to ICP analysis. With preconcentration the determination is made on a more reliable portion of the calibration curve. Also, since the variability over the narrow concentration range in question is nearly constant and does not change significantly by concentrating the sample 4X, the precision of the determination improves when the concentrated value is divided by 4 to calculate the analyte concentration in the original sample. Table 2 gives a comparison of precision and accuracy for the two elements As and Pb as determined by direct analysis and after preconcentration. The data for the direct analysis were determined from seven replicate analyses of a single unconcentrated aliquot while the preconcentration data were determined from the analysis of seven aliquots after preparation using the procedure described in 5.1. The percent recovery range data are the spread of the average percent recoveries from the seven replicate analyses determined on four separate days. The mean value is the average of the spread. The listed standard deviation is from the set of replicate analyses having the greatest variance.

3. INTERFERENCES

3.1 Concentration of surface, ground and drinking water supply samples can produce slight spectral and matrix interferences in ICP analysis. Reported effects have not been severe with the spectral

interference being an elevated shift in background intensity, while the matrix interference causes the signal intensity of some analytes to be reduced. In both cases the alkaline earth elements, calcium (Ca) and magnesium (Mg), are the primary interferents. For a complete description of interferences affecting ICP analysis see Section 5 of Method 200.7.

3.2 Spectral Interference

3.2.1 The technique of "off-the-line background correction adjacent to the wavelength peak," as required in Method 200.7, is usually adequate to compensate for shifts in background intensity. To test the spectral location selected for background correction, analyze analytically pure, single element Ca and Mg solutions of high concentration (>500 mg/L) and compare the data to the instrumental detection limit from acid blank determinations. If a value falls outside a confidence interval of ±2 standard deviations around the instrumental detection limit, the wavelength should be spectrally scanned for selection of a different background location. If it is not feasible to change the background correction location, an interelement correction factor can sometimes be used. An example is the effect of Ca on the recommended wavelength for Pb (220.353 nm). A non-uniform background shift occurs on the low side of the wavelength peak; however, the location is not changed because of a possible severe spectral interference from Al on the high side of the wavelength peak. For the situation described only a very small correction factor (-0.00002) is required for the EMSL-Cincinnati instrument. When using interelement correction for this purpose, the correction should not be completed when the determined interferent concentration deviates from linearity by more than 10% or unless the equation used in standardization includes terms for non-linear calibration.

3.2.2 Although no significant interelement spectral line interferences have been reported from the alkali and alkaline-earth elements on the wavelengths specified for the contaminants listed in Table 1, the EMSL-Cincinnati instrument does experience a weak Mg interference at 0.037 nm below the recommended Zn wavelength (213.856 nm) read in the second order. To avoid a possible Mg spectral interference, background intensity should be read on the high side of the Zn wavelength peak. Another possible spectral interferent whose effect should be determined is that of Al on the recommended wavelengths for As, Mn and Pb. Also, care must be taken that spectrally interfering elements are not mixed in the same calibration standard unless the computer program provides for their correction during standardization.

3.3 Matrix Interference

3.3.1 As the dissolved solids in the solution to be nebulized increase to exceed a concentration of 1500 mg/L, a suppressive effect on the analyte signal can occur. The most noticeable effect has been observed on certain analytes where a characteristic ion line is the preferred wavelength for the analysis. To determine the presence of a suppressive interference because of concentrating the matrix, a second aliquot of the sample should be spiked with each element to a concentration above 10X its determined MDL (but not to exceed its MCL), concentrated and analyzed. Recoveries outside the interval of 90% to 110% of the expected value can be used to indicate the presence of a matrix interference.

3.3.2 At EMSL-Cincinnati, using a fixed crossflow nebulizer with the instrument conditions given in Section 4.2, it has been observed that high concentrations of Ca (>400 mg/L) can cause a 5% suppressive effect on the emision signal of certain analytes; Cd and Pb experience the greatest suppression. As the concentration of Ca increases, its suppressive effect becomes more pronounced. Also, Mg has an additive suppressive effect on Pb, and this combined effect must be recognized when considering matrix interferences.

3.3.3 When the concentration of a primary contaminant is determined to be within 10% of its MCL or above, and the Ca concentration exceeds 400 mg/L (100 mg/L in the original sample concentrated 4X) or the combined Mg and Ca concentration equals 500 mg/L, a matrix matched calibration standard must be used. Otherwise the sample should be analyzed by the standard addition technique (see Section 10.6 of Method 200.7).

4. APPARATUS

4.1 In addition to the minimum requirements listed in Section 6 of Method 200.7, the use of mass flow controllers to regulate the argon flow rates, especially through the nebulizer, provide more exacting control and reproducible plasma conditions. Their use is highly recommended, but not required.

4.2 Operating conditions — Because of differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. However, the following instrument conditions were used in conjunction with a fixed crossflow nebulizer in developing the analytical data contained in this appendix:

Operating Conditions

Forward rf power	1100 watts
Reflected rf power (4)	< 5 watts
Viewing height above work coil	15 mm
Argon supply	Liquid Argon
Argon pressure	40 psi
Coolant argon flow rate	19 L min^{-1}
Aerosol carrier argon flow rate	630 cc min^{-1}
Auxilliary (plasma) argon flow rate	300 cc min^{-1}
Sample uptake rate controlled to	1.2 mL min^{-1}

5. SAMPLE PREPARATION

5.1 Transfer a 200 mL aliquot of a well mixed acid preserved sample to a Griffin beaker. Add 1.0 mL of (1+1) HNO_3 and 5.0 mL (1+1) HCL to the sample and heat on a steam bath or hot plate until the volume has been reduced to near 20 mL making certain the sample does not boil. Allow the sample to cool, transfer to a 50 mL volumetric flask, dilute to the mark with deionized-distilled water and mix. The sample is now ready for analysis. If after preparation the sample contains particulate matter, an aliquot should be centrifuged or the sample allowed to settle by gravity before aspiration.

6. QUALITY CONTROL

6.1 Instrumental

6.1.1 For required instrumental quality control see Section 12 of Method 200.7.

6.1.2 (Optional) To monitor nebulizer performance and aerosol effects in the plasma, a surrogate spike of a noncontaminant element (Au) is added at a concentration of 2 mg/L (1 mL of 100 mg/L Au per 50 mL sample) to each sample after dissolution, but before final dilution. If the analyzed Au value is not within ±5% of the true value, either the nebulizer or torch has become partially clogged or a suppressive matrix effect has occurred. An analysis of the instrument check standard will indicate if shutdown and cleaning is required. (Note: EMSL-Cincinnati has been able to use the "high surge" argon flow when the mass flow controller is first opened, to flush clean the argon port of the nebulizer. This purging is usually done during the print-out of analytical data and has proven in almost all instances to restore calibration drift back to its original calibration.)

3112 B. Cold-Vapor Atomic Absorption Spectrometric Method

1. General Discussion

This method is applicable to the determination of mercury.

2. Apparatus

a. Atomic absorption spectrometer and associated equipment: See Section 3111A.6. Instruments and accessories specifically designed for measurement of mercury by the cold vapor technique are available commercially and may be substituted.

b. Absorption cell, a glass or plastic tube approximately 2.5 cm in diameter. An 11.4-cm-long tube has been found satisfactory but a 15-cm-long tube is preferred. Grind tube ends perpendicular to the longitudinal axis and cement quartz windows in place. Attach gas inlet and outlet ports (6.4 mm diam) 1.3 cm from each end.

c. Cell support: Strap cell to the flat nitrous-oxide burner head or other suitable support and align in light beam to give maximum transmittance.

d. Air pump: Use any peristaltic pump with electronic speed control capable of delivering 2 L air/min. Any other regulated compressed air system or air cylinder also is satisfactory.

e. Flowmeter, capable of measuring an air flow of 2 L/min.

f. Aeration tubing, a straight glass frit having a coarse porosity for use in reaction flask.

g. Reaction flask, 250-mL erlenmeyer flask or a BOD bottle, fitted with a rubber stopper to hold aeration tube.

h. Drying tube, 150-mm × 18-mm-diam, containing 20 g $Mg(ClO_4)_2$. A 60-W light bulb with a suitable shade may be substituted to prevent condensation of moisture inside the absorption cell. Position bulb to maintain cell temperature at 10°C above ambient.

i. Connecting tubing, glass tubing to pass mercury vapor from reaction flask to absorption cell and to interconnect all other components. Clear vinyl plastic* tubing may be substituted for glass.

3. Reagents†

a. Metal-free water: See 3111B.3c.

b. Stock mercury solution: Dissolve 1.354 g mercuric chloride, $HgCl_2$, in about 700 mL water, add 10 mL conc HNO_3, and dilute to 1000 mL with water; 1.00 mL = 1.00 mg Hg.

c. Standard mercury solutions: Prepare a series of standard mercury solutions containing 0 to 5 µg/L by appropriate dilution of stock mercury solution with water containing 10 mL conc HNO_3/L. Prepare standards daily.

d. Nitric acid, HNO_3, conc.

e. Potassium permanganate solution: Dissolve 50 g $KMnO_4$ in water and dilute to 1 L.

f. Potassium persulfate solution: Dissolve 50 g $K_2S_2O_8$ in water and dilute to 1 L.

g. Sodium chloride-hydroxylamine sulfate solution: Dissolve 120 g NaCl and 120 g $(NH_2OH)_2 \cdot H_2SO_4$ in water and dilute to 1 L. A 10% hydroxylamine hydrochloride solution may be substituted for the hydroxylamine sulfate.

h. Stannous chloride solution: Dissolve 100 g $SnCl_2$ in water containing 200 mL conc HCl and dilute to 1 L. On aging, this

* Tygon or equivalent.
† Use specially prepared reagents low in mercury.

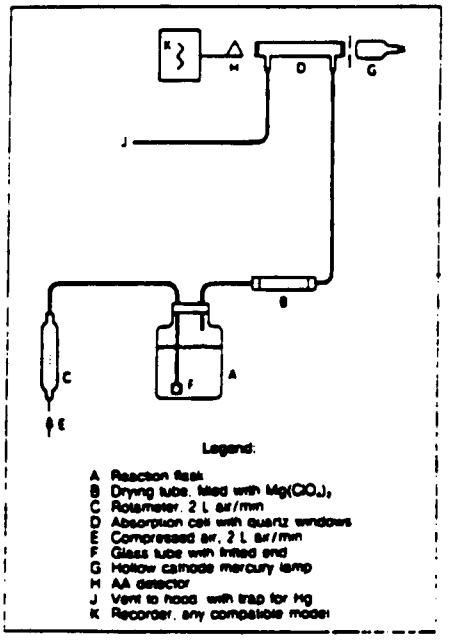

Figure 3112:1. Schematic arrangement of equipment for measurement of mercury by cold-vapor atomic absorption technique.

solution decomposes. If a suspension forms, stir reagent continuously during use.

i. Sulfuric acid, H_2SO_4, conc.

4. Procedure

a. Instrument operation: See Section 3111B.4*b*. Install absorption cell and align in light path to give maximum transmission. Connect associated equipment to absorption cell with glass or vinyl plastic tubing as indicated in Figure 3112:1. Turn on air and adjust flow rate to 2 L/min. Allow air to flow continuously. Alternatively, follow manufacturer's directions for operation. NOTE: Fluorescent lighting may increase baseline noise.

b. Standardization: Transfer 100 mL of each of the 1.0, 2.0, and 5.0 µg/L Hg standard solutions and a blank of 100 mL water to 250-mL erlenmeyer reaction flasks. Add 5 mL conc H_2SO_4 and 2.5 mL conc HNO_3 to each flask. Add 15 mL $KMnO_4$ solution to each flask and let stand at least 15 min. Add 8 mL $K_2S_2O_8$ solution to each flask and heat for 2 h in a water bath at 95°C. Cool to room temperature.

Treating each flask individually, add enough NaCl-hydroxylamine sulfate solution to reduce excess $KMnO_4$, then add 5 mL $SnCl_2$ solution and immediately attach flask to aeration apparatus. As Hg is volatilized and carried into the absorption cell, absorbance will increase to a maximum within a few seconds. As soon as recorder returns approximately to the base line, remove stopper holding the frit from reaction flask, and replace with a flask containing water. Flush system for a few seconds and run the next standard in the same manner. Construct a standard curve by plotting peak height versus micrograms Hg.

c. Analysis of samples: Transfer 100 mL sample or portion diluted to 100 mL containing not more than 5.0 µg Hg/L to a reaction flask. Treat as in ¶ 4*b*. Seawaters, brines, and effluents high in chlorides require as much as an additional 25 mL $KMnO_4$ solution. During oxidation step, chlorides are converted to free chlorine, which absorbs at 253 nm. Remove all free chlorine before the Hg is reduced and swept into the cell by using an excess (25 mL) of hydroxylamine sulfate reagent.

Remove free chlorine by sparging sample gently with air or nitrogen after adding hydroxylamine reducing solution. Use a separate tube and frit to avoid carryover of residual stannous chloride, which could cause reduction and loss of mercury.

4. Calculation

Determine peak height of sample from recorder chart and read mercury value from standard curve prepared according to ¶ 4*b*.

TABLE 3112:I. INTERLABORATORY PRECISION AND BIAS OF COLD-VAPOR ATOMIC ABSORPTION SPECTROMETRIC METHOD FOR MERCURY[1]

Form	Conc. µg/L	SD µg/L	Relative SD %	Relative Error %	No. of Participants
Inorganic	0.34	0.077	22.6	21.0	23
Inorganic	4.2	0.56	13.3	14.4	21
Organic	4.2	0.36	8.6	8.4	21

6. Precision and Bias

Data on interlaboratory precision and bias for this method are given in Table 3112:I.

7. Reference

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