of

OCEANOGRAPHY

OREGON STATE UNIVERSITY

LABORATORY TECHNIQUES USED FOR ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSIS OF GEOLOGIC SAMPLES

by
Stan Fukui

Reference 76-10 August 1976

Laboratory Techniques Used for Atomic Absorption Spectrophotometric Analysis of Geologic Samples

by

Stan Fukui

School of Oceanography
Oregon State University
Corvallis, Oregon 97331

August 1976

Reference 76-10

CONTENTS

Contents, continued:

LIST OF ILLUSTRATIONS AND TABLES

INTRODUCTION

Precise chemical analyses of geological samples continue to play an important role in geochemical research. During the past 10 years atomic absorption spectrophotometry has become increasingly prominent as an accurate and relatively simple analytical technique. The marine geochemistry group at OSU has used a Jarrel-Ash 82-820 atomic absorption spectrophotometer (AAS) for the past four years to determine the elemental composition of deep-sea sediments and oceanic rocks. This document is meant to serve as a summary of the procedures which have been developed and adopted during this time.

THEORY OF ATOMIC ABSORPTION SPECTROMETRY

When light of a specific wavelength is incident upon a free ground state atom, that light will be absorbed causing excitation of the electrons. In the reverse process, light of the same wavelength is emitted.

Atomic absorption spectroscopy is based on the principle that the amount of light absorbed is exponentially related to the population of free atoms. The following equation describes this relationship:

$$C = \text{Log} \frac{I_o}{I}$$

where C is the concentration of free atoms,

I_o is the incident radiation and

I is the transmitted light.

Since each element absorbs and emits light at its own characteristic wavelengths, it is possible to analyze for the concentration of one metal in a solution of many elements and eliminate the need for chemical separation.

The instrumental components which are required for analysis are: 1) a light source (a hollow-cathode tube); 2) a method of producing atomic vapor (a flame-aspirator system, carbon rod, tantalum ribbon, etc.); 3) a method of isolating the desired wavelength of light (a monochromator) 4) a detector (a photomultiplier); and 5) a d-c amplifier and read-out system.

OPERATION OF THE J. A. 82-820 A.A.S.

Introduction

The J. A. 82-820 is the same instrument as the J. A. 82-810 except for some minor electronic modifications. The 82-820 is a dual channel, double beam instrument. It is essentially two complete spectrophotometers sharing the same flame and fore-optics. The dual channel feature will allow the user to analyze for two elements simultaneously or to operate in one of two compensated modes; 1) background correction, or 2) an internal standard. It has been found, however, that only the background correction feature is useful in our application. Since both channels use the same set of fore-optics (mirrors, beam splitters, etc.), both monochromators will receive light from both lamps. Therefore, background correction can be done with a non-absorbing line of the analyzed element or a non-absorbing line of a second element.

For a more detailed explanation of the Jarrel Ash 82-810, the reader is referred to the instruction manual (11).

Preliminary Discussion

Before beginning this section of the manual, the reader should be familiar with the JA 82-810 Instruction manual (11). Many of the instructions and precautions included in the instruction manual have not been duplicated here.

Some of the procedures described here are considerably different from those described in the instruction manual. In these cases, the procedure described in this manual should be used. In many cases the procedures described here were obtained orally from the manufacturer and supersede the direction described in the manual.

The reader is specifically directed to:

Section	
1	Theory of Operation
3	Controls and Indicators
4	Single Channel Operation
6	Background Correction

TABLE 1. INSTRUMENT SETTINGS

Element	Sci. Abbrev.	Wave-length	Slit Width	Slit Setting	Lamp Current	Oxidant/ Fuel	Comments
Aluminum	1	3093 Å	4 Å	4	10 ma	N_2O_2/C_2H_2	
Barium	Ba	5536	2	3	15	N_2O_2/C_2H_2	
Calcium	Ca	4227	10	5	10	N_2O_2/C_2H_2	
Cobalt	Co	2407	2	3	12	Air/C_2H_2	
Copper	Cu	3247	4	4	7	Air/C_2H_2	
Iron	Fe	2483	2	3	8	Air/C_2H_2	
Magnesium	Mg	2852	10	5	10	N_2O_2/C_2H_2	
Manganese	Mn	2795	4	4	10	Air/C_2H_2	
Nickel	Ni	2320	1	2	10	Air/C_2H_2	Bkgd 2316
Potassium	K	7665	10	5	10	Air/C_2H_2	
Silicon	Si	2516	2	3	12	N_2O_2/C_2H_2	
Titanium	Ti	3653	10	5	12	N_2O_2/C_2H_2	
Zinc	Zn	2139	10	5	7.5	Air/C_2H_2	Bkgd 2100

*This is a tabulation of the instrument settings found in the Fisher Scientific Company's Atomic Absorption Analytic Methods Manual (10). This list also includes any changes made by the geochemistry laboratory to the Analytic Methods manual.

General Stepwise Operating Procedure

1. Turn on the instrument and allow it to warm-up for several minutes.

2. Plug it in the desired hollow cathode lamp, set the correct current and allow it to warm up.

3. Turn on the acetylene gas main cylinder valve.

4. Set the pressure to 14 psig. (The pressure should never be higher than this because acetone will become entrained in the acetylene and acetylene becomes unstable above 15 psig.)

5. Set the acetylene flow rate to approximately 5 scfh.

6. If nitrous oxide is to be used:

   Open the main supply tank valve fully.

   Set the regulator to approximately 125 psig and plug in the heater cord.

   Open the secondary tank valve fully.

   Set the regulator to 50 psig.

7. Set the oxidant fuel rate to one or two marks below the maximum. This will insure a steadier flow rate.

8. Be sure you are using the correct burner head.

   The air/acetylene burner head is slotted and approximately 10 cm long.

The nitrous oxide/acetylene burner head is slotted and approximately 5 cm long.

9. Switch on the air, then the acetylene and push the ignitor button.

10. Adjust the acetylene flow rate so that the flame is a clear blue.

    If the flame is uneven, the burner head and/or aspirator system must be cleaned.

11. Allow the burner to warm up for several minutes.

    This allows the adjacent electronic system to reach an equilibrium temperature and the burner head to expand to its operating configuration.

12. Adjust the slit width control to the proper setting.

13. Turn the monitor switch to HVA.

14. Move the wavelength control to the approximate setting and adjust until the digital voltmeter (DVM) indicates a minimum value.

15. If background correction is to be used, repeat steps 12-14 for Channel B. The monitor switch should be set to HVB.

    When setting the proper wavelength, care must be taken to avoid tuning into an emission peak which is adjacent to the correct value. This is important for iron and nickel which have many emission peaks close together.

16. Turn the flame off and be sure that the burner head is below the light path.

17. Turn the monitor switch to RA. Push the (A) display button, the light should go on.

18. While holding the autozero button down adjust the lamps until the absolute value of the display is less than 2.00.
This indicates that the measure and reference beams appear approximately equal in intensity to the detector.

19. If background correction is to be done, the same procedure must be done for Channel B. The monitor switch is turned to RB and the (B) button must be pushed. Remember to check RA if you must adjust the lamp. Repeat until the absolute value of both RA and RB is less than 2.00.

20. Turn the monitor switch to OPERATE.

21. Turn the mode switch to %A and be sure the (A) button light is on.

22. Turn the integrate switch to 1 sec.

23. Autozero channel A.

24. Now move the burner head up until it is in the light path. This will be indicated by an increase in the DVM readings.

25. Turn the burner head down until the DVM readings return to zero. The light will now pass through the most stable part of the flame.

26. Turn on the gases and ignite the flame.

27. Autozero aspirating the zero standard.

28. Aspirate a solution which will give an absorbency of between 30-60%.

29. Adjust the burner horizontal and rotational positions until a maximum absorbency is reached.

30. Adjust the nebulizer for a maximum reading.

31. Finally adjust the acetylene flow rate for maximum readings on the DVM.

32. Turn the mode switch to ABS and autozero, aspriating the zero standard.

33. If background correction is required, push the (B) button, turn the mode switch to ABS and autozero as above.
Now push the (A-B) button and autozero using the channel A autozero button, and while aspirating the zero standard.

34. Aspirate the samples and record an approximate absorbency value for each one.

35. Aspirate the standards and record an approximate absorbency value for each one.

36. Decide which standards are required to bracket each sample - one higher and one lower.

37. Turn the mode switch to CONC.

38. Set the THRESHOLD control to the approximate % absorption value where the relationship between absorbency and concentration becomes nonlinear.
    This value may be obtained from the curves in Appendix A. The absorbancy values must be converted to % absorption. This process must be followed in any case since % absorption and concentration are not linearly related.

39. Autozero aspirating the zero standard.

40. Aspirate the lowest standard (not zero) and set the CONCENTRATION controls so that an appropriate value is displayed on the DVM.
    NOTE: The maximum number that can be displayed by the DVM is 1999.

41. Now aspirate the highest standard required and set the CURVATURE control until the appropriate value is displayed on the DVM.

42. Check all of the standards for the appropriate values and repeat steps 39-41 until the proper values are obtained.

43. The AAS is now ready to collect data.

NOTE: If the CURVATURE control has been turned to zero and the readout is still too high, the THRESHOLD control must be set to a higher value.

Shutdown of the AAS

1. Turn off the lamps.

2. If nitrous oxide is being used, switch to air then turn down the acetylene flow rate. This is important to avoid flashback.

3. Aspirate distilled water for at least 15 minutes. This is done to remove any accumulated salts from the aspirator system.

4. Turn off the air.

5. Turn off the acetylene.

6. Close all main tank valves.

7. Unplug the nitrous oxide regulator heater.

8. Drain all of the gas lines except the air line.

9. Close all of the regulator valves except the air regulator.

10. Empty the bucket which catches the overflow from the aspirator chamber.

11. Turn off the power.

Maintenance of the AAS

The only routine maintenance required on the AAS is the cleaning of the burner-aspirator system. A good description of this procedure is described in Section 4.2.4 Burner Cleaning, on page 40 of the Instruction Manual (11).

An electronic checkout procedure has been obtained from Jarrel Ash and is available in the laboratory.

The only other maintenance required is the cleaning of the exterior windows and lenses. This should be done with optical glass cleaning tissue and never with regular laboratory wipes. These contain too many abrasives.

PREPARATION OF REAGENTS AND STANDARDS

Introduction

Standard solution preparation techniques were developed with four major objectives in mind. These were:

1) To match the samples in reagent composition,
2) To compensate for any inter-elemental interferences,
3) Produce solutions which had utility for a large variety of samples with varying concentrations of minerals,
4) Ease of preparation.

To date the following procedure has proven to be very efficient and no systematic errors have been detected for this method.

This section will describe the procedures used in the preparation of the reagents and standards used in the analysis of geochemical samples.

Cesium Chloride

Introduction

Certain easily excitable elements, such as potassium and sodium, are readily ionized in the flame. This will reduce the population of ground state atoms of these elements and alter their absorption values. In addition if these elements are present in varying amounts in samples and standards, their ionization can affect the analysis of other elements.

To reduce these effects a standard amount of cesium chloride is added to each solution. Cesium is more easily ionized than the other elements which are present and the amount of cesium added is enough to mask out any naturally present cesium. Therefore, any ionization effects are approximately the same for all the solutions.

Preparation

The Cesium Chloride (CsCl) solution contains 25,000 ppm of Cs.

To make 1.0 liter:

1. Weigh out 31.66886 gms of CsCl.
2. Transfer to a 1.0 liter volumetric flask.
3. Partially fill with D. D. water.
4. Shake until CsCl dissolves.
5. Bring to volume with D. D. water. Shake well and transfer back to the poly bottles, rinsing once with a small amount of the solution.

For smaller quantities:

500 ml	15.83443 gms CsCl
250	7.91722
200	6.33377
100	3.16689
50	1.58344

Aqua Regia

Aqua regia is a highly oxidizing solution made from one part concentrated nitric acid and three parts concentrated hydrochloric acid.

The reaction of HCl and HNO_3 forms three products, two gases, Cl_2 and NOCl, which will eventually escape from solution and water. This reaction gives aqua regia its oxidizing characteristics. Therefore, for this solution to be most effective in dissolving the sample, it should be freshly prepared. In instances where a sample will not dissolve, a new solution of aqua regia may solve the problem.

In addition care should be exercised when handling aqua regia since one of the gases evolved is chlorine (Cl_2).

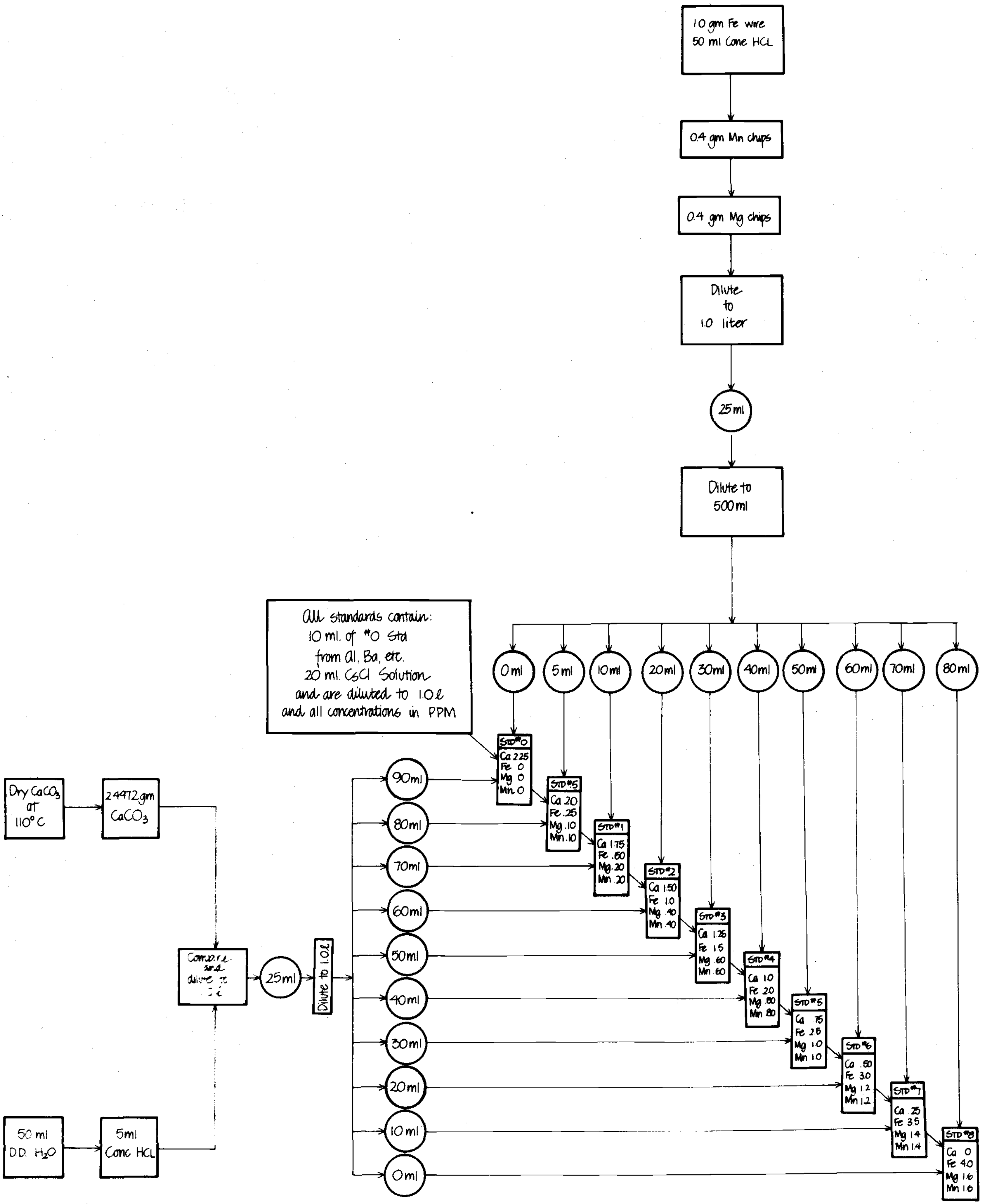


Figure 1. Preparation of Ca, Fe, Mg, Mn Standards.

Preparation of Stock Standards

Iron, Manganese, Magnesium

1. Add 50 ml of Conc. HCl to 1.0 gm of iron wire in a teflon beaker.

2. Let set until the wire dissolves (overnight).

3. Add 0.4 gm of Manganese chips (be careful - reaction is very violent).

4. Add 0.4 gm of Magnesium chips (be careful - reaction is very violent).

*5. Transfer the solution to a 1.0 liter flask. Rinse beaker several times with D. D. water.

*6. Fill the flask to just below the mark.

*7. Let set to equilibrate to room temp. (Room temp. should be 18-22°C).

*8. Fill to mark.

*9. Shake well.

*10. Transfer to poly bottle.

*Do these steps for each stock standard.

Calcium

1. Add 5 ml of conc. HCl to 50 ml D. D. water.

2. Slowly add to 2.49724 gm of $CaCO_3$* in a 1.0 liter vol. flask.

3. Go to step six of Iron, Manganese, Magnesium stock preparation.

* $CaCO_3$ (Calcium Carbonate) should be thoroughly dried in a drying oven at 110°C before weighing.

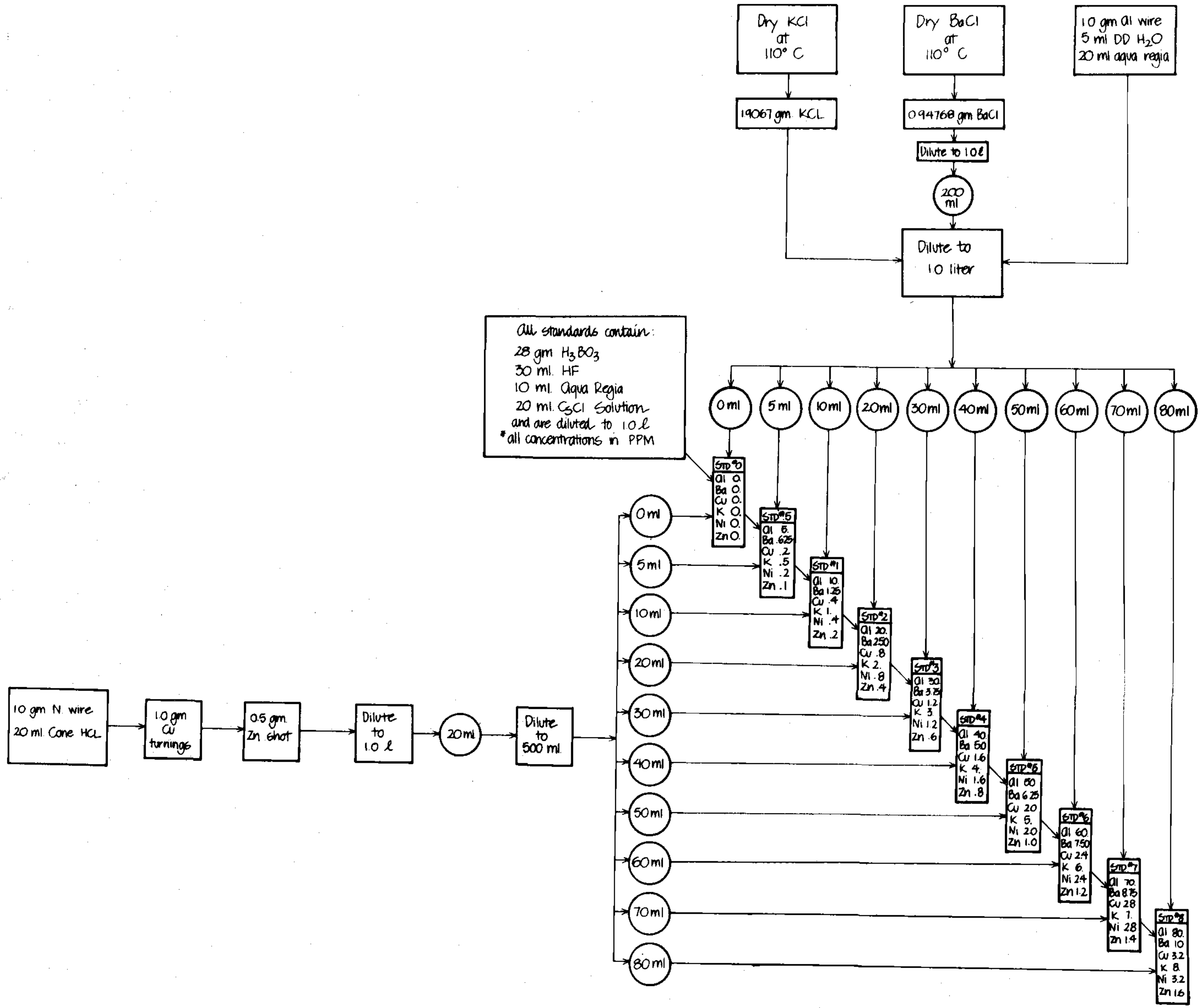


Figure 2. Preparation of Al, Ba, Cu, K, Ni, Zn Standards.

Aluminum, Barium, Potassium

1. Add 20 ml aqua regia and 5 ml D. D. H_2O to 1.0 gm Aluminum wire in a teflon beaker.

2. Let set until Aluminum dissolves.

3. Pour into 1.0 liter volumetric flask which contains

    *a) 0.19067 gm Potassium Chloride

    *b) 0.60651 gm Barium Chloride

4. Go to step six of Iron, Manganese, Magnesium stock preparation.

*Potassium Chloride and Barium Chloride should be thoroughly dried in a drying oven at 110°C before weighing.

Copper, Nickel, Zinc

1. Add 20 ml of conc. HCl to 1.0 gm of Nickel wire in a teflon beaker.

2. Set on a hot plate at the 1 setting until nickel dissolves (overnight).

3. Add 1.0 gm Copper turnings.

4. Add 0.5 gm zinc shot.

5. Go to step five of Iron, Manganese, Magnesium stock preparation.

Preparation of Working Standards

Ca, Fe, Mn, Mg

1. Pipette 25 ml of Fe, Mn, Mg stock solution into a 500 ml volumetric flask. Bring to volume with D. D. water.

2. Pipette 50 ml of Ca stock solution into a 500 ml volumetric flask. Bring to volume with D. D. water.

3. Into each of ten 1.0-liter volumetric flasks add 20 ml of CsCl solution and 10 ml of the 0 standard or equivalent from the Al, Ba, etc. standard.

4. Add to the flask the following:

Flask #	ml of Fe, Mn, Mg solution	ml of Ca solution
0	0	90
.5	5	80
1	10	70
2	20	60
3	30	50
4	40	40
5	50	30
6	60	20
7	70	10
8	80	0

5. Bring to volume and transfer to polyethelene bottles.

Al, Ba, Cu, K, Ni, Zn

1. Pipette 20 ml of Cu, Ni, Zn stock into a 500 ml volumetric flask.

2. Dilute with D. D. water.

3. Into ten 30-oz. polyethelene bottles, add:

   28.0 gm Boric acid (H_3BO_3)

   30.0 ml Hydrofluoric acid (HF)

   10.0 ml Aqua regia

   20.0 ml Cesium chloride (CsCl) solution

4. Shake well and transfer to ten 1.0-liter volumetric flasks.

5. To each of the flasks add the following amounts of the recently prepared Cu, Ni, Zn solution and the Al, Ba, K stock solution.

Flask #	ml of Al, Ba, K stock solution	ml of Cu, Ni, Zn solutions
0	0	0
.5	5	5
1	10	10
2	20	20
3	30	30
4	40	40
5	50	50
6	60	60
7	70	70
8	80	80

6. Dilute with D. D. water and transfer back to the poly. bottles.

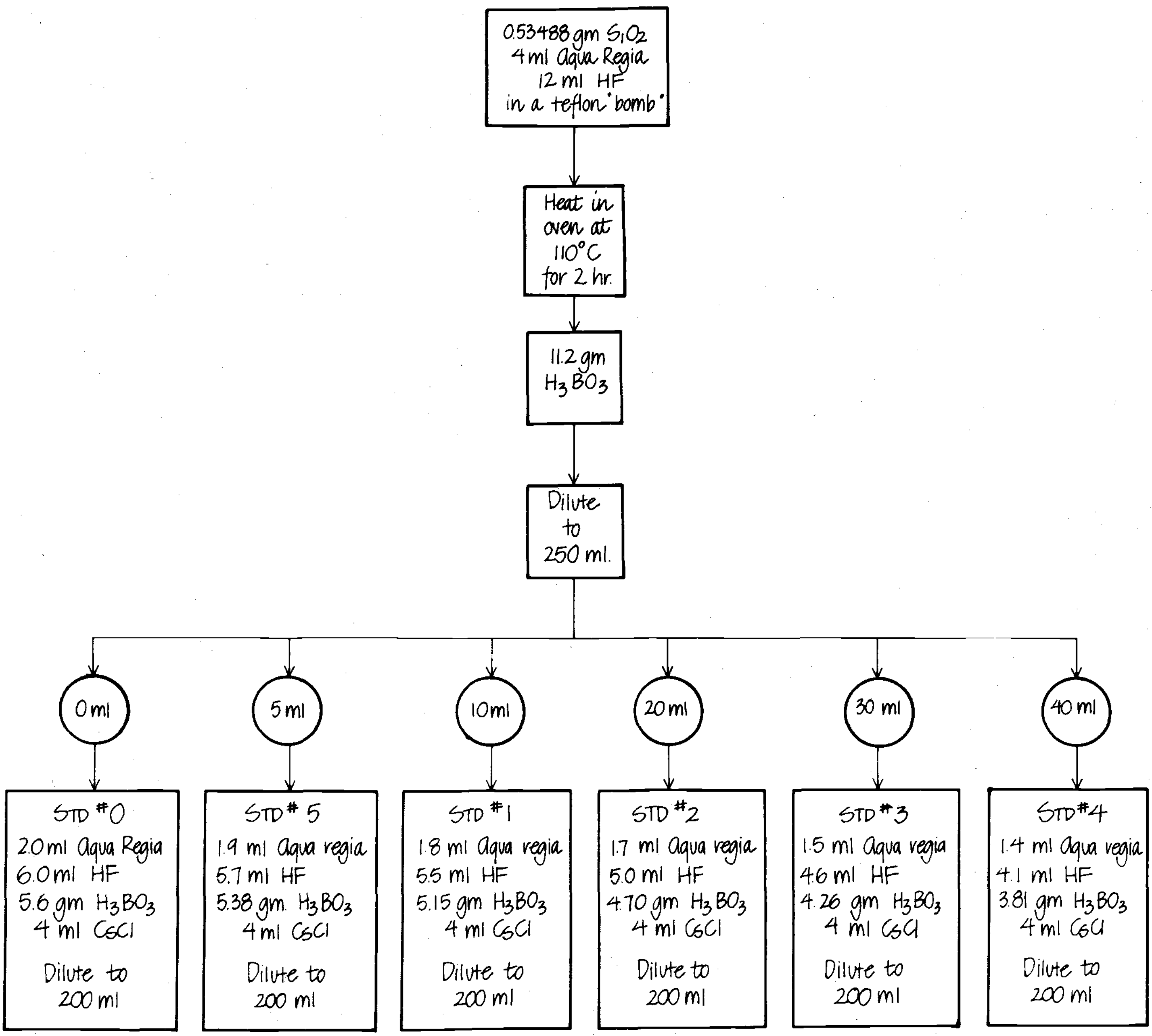


Figure 3. Preparation of Silicon Standards.

Preparation of Silicon Standards

1. Weigh out 0.53488 gm of silicon dioxide into the Parr teflon bomb (14, 15).

2. Add 4 ml of aqua regia.

3. Add 12 ml of HF.

4. Cap and insert into metal body. Hand tighten.

5. Heat in a 110°C oven for 1.5 to 2 hours.

6. Remove bomb from oven and let cool to room temperature. This is very important because of dangerous fumes and the loss of silicon-fluoride compounds at higher temperatures.

7. Pour the slurry into a poly bottle containing 11.2 gm of boric acid.

8. Shake well and transfer to a 250 ml vol. flask.

9. Bring to volume with D. D. water.

10. Rinse the poly bottle with a small portion of the solution and transfer the remainder.

11. To each of five 8-oz. poly bottles add the following:

Bottle no.	Stock sol'n.	Aqua regia	HF	H_3BO_3	CsCl
0	00 ml	2.0 ml	6.0 ml	5.6 gm	4 ml
.5	5	1.9	5.7	5.38	4
1	10	1.8	5.5	5.15	4
2	20	1.7	5.0	4.70	4
3	30	1.5	4.6	4.26	4
4	40	1.4	4.1	3.81	4

12. Pour into 200 ml vol. flasks.

13. Rinse poly bottles with small amounts of D. D. water.

14. Bring to volume with D. D. water and transfer the solutions back to the appropriate poly bottle.

Preparation of Titanium Standards

1. Pipette 50 ml of 1000 ppm Ti standard into a 250 ml volumetric flask. Bring to volume with D. D. water.

2. To each of six 8-oz. poly bottles add:

   5.6 gm Boric acid

   2.0 ml Aqua regia

   6.0 ml HF

   4.0 ml CsCl solution

3. Shake well and transfer to six 200 ml vol. flasks. Rinse twice.

4. To each of the above vol. flasks add:

Flask #	mls. of Ti solution
.5	5
1	10
2	20
3	30
4	40
5	50

5. Bring to volume with D. D. water. Shake well and transfer back to the poly bottles rinsing once with a small amount of the solution.

Preparation of Cobalt Standards

1. Pipette 5 ml of 1000 ppm Co standard into a 250 ml vol. flask. Bring to volume with D. D. water.

2. To each of six 8-oz. poly bottles add:

   5.6 gm Boric acid

   2.0 ml Aqua regia

   6.0 ml HF

   4.0 ml CsCl solution

3. Shake well and transfer to six 200 ml vol. flasks. Rinse twice.

4. To each of the above flasks add:

Flask #	mls. of Co solution
.5	5
1	10
2	20
3	30
4	40
5	50

5. Bring to volume with D. D. water. Shake well and transfer back to the poly bottles rinsing once with a small amount of the solution.

SAMPLE PREPARATION

Introduction

Samples are dissolved in a teflon crucible using hydrofluoric acid and aqua regia. The technique is similar to that described by Bernas (1). This method has proven successful for the dissolution of sediments, basalts and manganese nodules.

Sediments are dryed at 100 °C to remove absorbed water and insure a consistent weight of sample. A suitable amount of sample is weighed out; generally 400 mg. for sediments and basalts and 200 mg. for manganese nodules. The sample is then transferred to a teflon crucible and reweighed.

Two milliliters of aqua regia are then added to the sample in the teflon crucible. The aqua regia must be recently prepared to insure the dissolution of the sample. (See discussion on aqua regia.) Then 6 ml of HF are added.

A teflon lid is placed over the crucible and it is sealed into the stainless steel casing. The "bombs" are heated in a drying oven at 100 °C for 1.5 to 2 hours. The longer heating time is recommended for the darker colored sediments containing larger amounts of iron and manganese oxides, which are more difficult to dissolve. Heating times of three hours are recommended for manganese nodules.

The decomposition vessel is then removed and allowed to cool to room temperature.

The sample is transferred to a polyethylene bottle containing 5.6 gms of boric acid. The boric acid performs two functions; one is to neutralize the excess HF so the solution can be accurately diluted in glass vessels and second to dissolve the fluorides which have precipitated in the crucible.

Bernas determined that in the presence of excess boric acid, the attack on glass vessels by HF was insignificant over a period of two hours (1). However, since the accuracy of a volumetric flask would be significantly changed over a period of several years, the solution should be transferred to polyethylene bottles as soon as possible and the glass vessels rinsed (see below).

Finally, four milliliters of 25000 ppm cesium solution is added and the mixture is diluted to 200 ml. The cesium is added to the sample to act as an ionization suppressant. (See discussion on cesium chloride solution.) All samples contain approximately 500 ppm of cesium.

From this solution other dilutions are made as necessary to analyze the desired elements.

Hydrofluoric acid is prevented from attacking glass by the formation of fluoroboric acid with boric acid in a two step reaction:

$$H_3BO_3 + 3HF \quad HBF_3OH + 2H_2O$$

$$HBF_3OH + HF \quad HBF_4 + H_2O$$

Fluoroboric acid will then hydrolyze to hydroxyfluoroborate ions and hydrofluoric acid, hence the time limit the solution can remain in contact with the glass.

Step-by-Step Sample Preparation Procedures

1. Pour out the entire sample onto a piece of weighing paper.

2. Weigh out approximately 400 mg of the sample (200 mg for manganese nodules). Small portions should be taken from different sections of the pile to prevent size particle fractionation.

   a. Metalliferous sediments are transferred to a black capped vial and dried in a drying oven at 110°C overnight, then cooled in a dessicator to room temperature.

   b. 2-20 μ fractions must be sampled using a micro-splitter.

3. Brush the teflon portion of the dissolution bomb with the "Staticmaster" brush to remove some of the static charges which tend to accumulate on teflon.

4. Weigh the teflon liner.

5. Pour the sample into the teflon liner. Use a piece of rolled weighing paper as a cylindrical funnel to keep the sample from flying around and adhering to the side of the teflon crucible. (This is caused by the electrostatic charges.)

6. Weigh the sample and crucible.

7. Carefully add 2 ml of aqua regia. A light colored sample indicates high carbonate content and extreme care must be used to prevent

bubble formation and spattering. It is suggested in this case that the analyst wet the sample with approximately .5 ml of double distilled water (DDW) then add the aqua regia in .1 ml aliquots for the first .5 mls, then add .5 ml and finally a full milliliter. (Use the pipette gun.)

8. Add 6 ml of HF.

9. Put on the teflon lid and insert into the metal bomb.

10. Tighten the metal cover to 250 in-lbs. of torque.

11. Put the bomb into a drying oven at 110°C for 1.5 to 2 hours. The longer times are suggested for the darker sediments. (Up to 3 hours are recommended for manganese nodules.)

12. Remove the bombs from the oven and allow to cool to room temperature. While the bombs are cooling, they must be retightened to 250 in-lbs. of torque every 10 minutes to reduce leakage. Leakage is caused by the cold-flowing of teflon under pressure and loss of the seal on cooling.

13. After cooling remove the teflon liners from the metal body and pour the dissolved sample into a polyethylene bottle containing 5.6 gms of boric acid (H_3BO_3). Be sure to rinse the liner and the lid carefully with DDW.

14. Shake the mixture; the solution should be yellowish to clear. If brown or a dark color, the sample was not dissolved.

15. Pour the solution into a 200 ml volumetric flask. Rinse the poly bottle with a small amount of DDW and pour into the volumetric flask. NOTE: All flasks and pipettes must be Class A.

16. Repeat the rinsing.

17. Pipette 4 mls of cesium chloride (CsCl) solution into the vol. flask.

18. Bring to volume with DDW.

19. Rinse the poly bottle with a small amount of sample solution and transfer the remainder.

20. Pipette 10 mls of the solution into a one liter vol. flask.

21. Add 20 mls of CsCl solution.

22. Bring to volume with DDW.

23. Rinse a poly bottle with a portion of the solution and transfer the remainder.

SAMPLE DECOMPOSITION HARDWARE

The acid decomposition vessels were machined from 1 3/4" dia. virgin teflon rods and the metal casings from 2 in. and $2\frac{1}{4}$ in. dia. 304 stainless steel rods.

The teflon crucibles and caps were "roughed out" to the approximate size and shape. They were then annealed at 220°C overnight. This was done to relieve stresses caused in the teflon by the cutting and machining process (12).

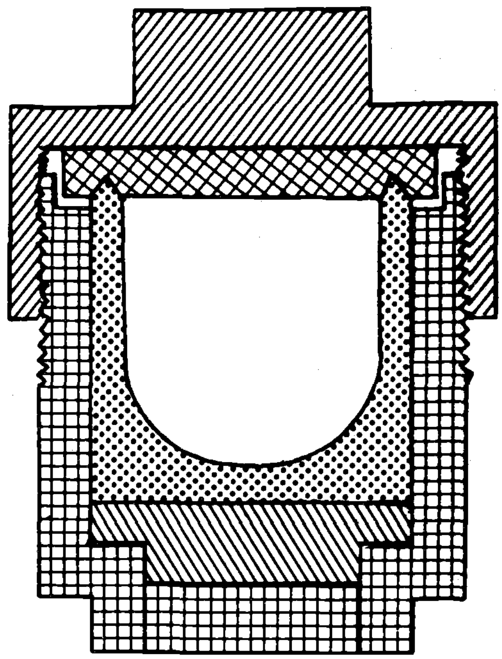
The stainless steel shell was machined with a 1 1/8" hexhead nut on the cap to facilitate tightening. It was felt that the use of a torque wrench to seal the vessles would insure more uniform results than "hand tightening" which had been suggested by all of the other designers.

It was found that 250 in-lbs. of torque could be developed by "hand tightening" and this was the value that was decided as sufficient to seal the vessels.

The aluminum plate used to hold the "bombs" while they are being tightened and cooled has proven to be a very useful item. In addition to increasing the efficiency of the tightening operations. The holder will also act as a heat radiator to increase the rate of cooling.

There are several things which could be done to improve the design of the vessels. The most important would be to increase the depth of the crucibles. This should be done to decrease the chance of losing the sample when it is being transferred to the vessel and when the acid is being added. Also a more acid-resistant grade of stainless-steel (316 s. s.) should be used for the metal casings.

SAMPLE DECOMPOSITION VESSEL



STAINLESS STEEL CAP, WITH 1 1/8" HEXHEAD NUT

STAINLESS STEEL PRESSURE CASING, MAIN BODY

STAINLESS STEEL REMOVABLE PLUG

TEFLON CAP

TEFLON CRUCIBLE

Figure 4. Sample Decomposition Vessel.

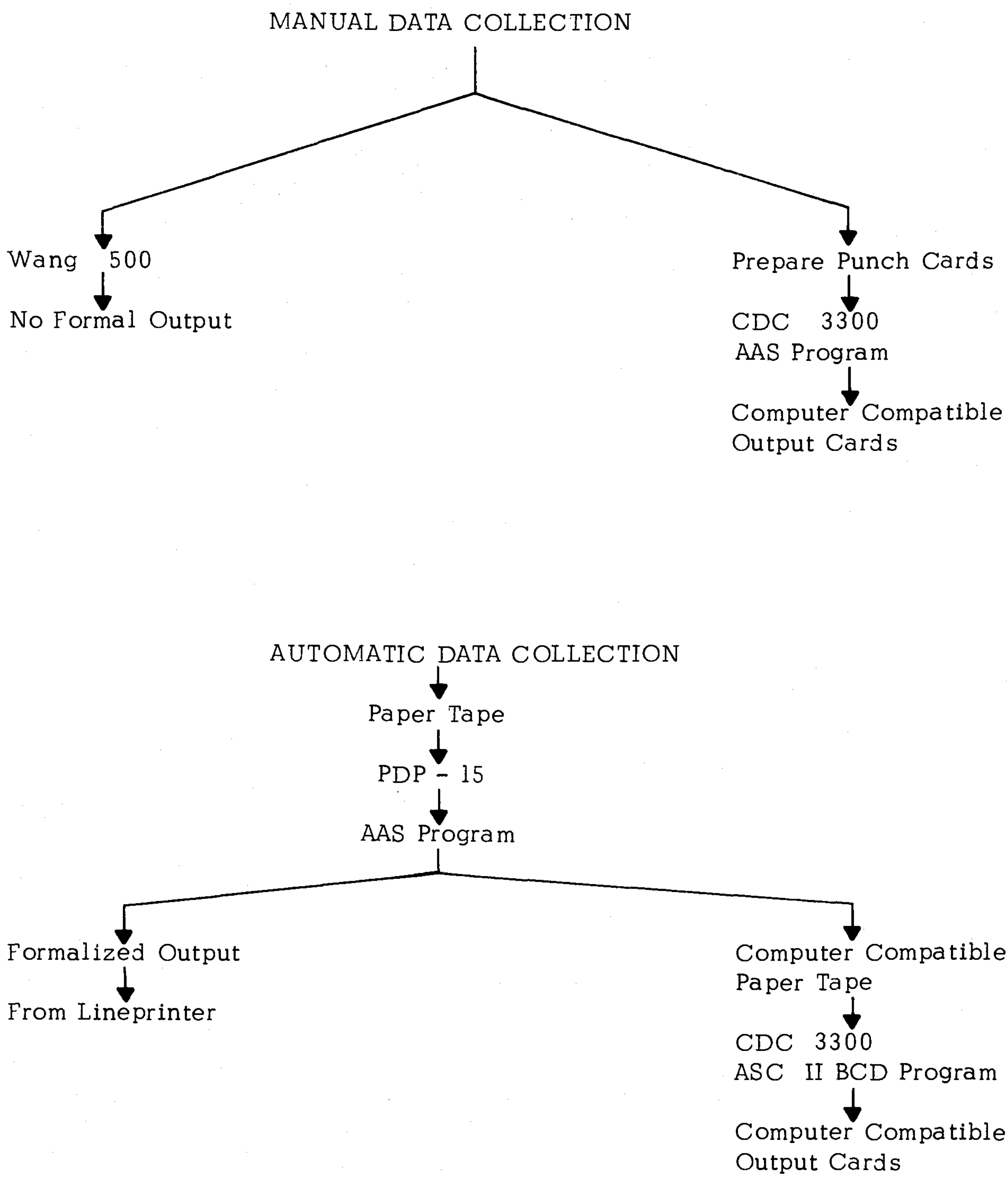

MANUAL DATA COLLECTION
Wang 500
No Formal Output
Prepare Punch Cards
CDC 3300
AAS Program
Computer Compatible
Output Cards
AUTOMATIC DATA COLLECTION
Paper Tape
PDP - 15
AAS Program
Formalized Output
From Lineprinter
Computer Compatible
Paper Tape
CDC 3300
ASC II BCD Program
Computer Compatible
Output Cards

Figure 5

DATA COLLECTION AND REDUCTION

Introduction

Data may be collected manually by the operator or automatically using papertape produced by an ASR-33 teletype.

Data reduction procedures are related to the method of data collection. If the data are collected manually there are two devices for which programs have been developed to reduce the data. One is the Wang 500 programmable calculator and the other is the CDC 3300 computer. Using the teletype and papertape system for data collection, the operator must use the School of Oceanography's PDP-15 computer.

As shown in the schematic drawing, with the Wang 500 only non-computer compatable hard copy is obtained, whereas the other two methods of reducing the AAS data result in a standard formatted, computer compatable data card.

All of these systems are presently workable; however, the automatic system seems to be the most satisfactory. Using this system a larger amount of data can be collected more quickly than using the manual systems. The data are also collected on papertape which can be immediately fed into the computer without further preparation.

Proceedings for Using the Wang 500 for AAS Data Reduction

Preliminary Discussion

The program to reduce atomic absorption data on the Wang 500 is available on a magnetic tape cassette. The reader is referred to the Wang 500 reference manual (17) for operating instructions.

Utilizing this method of data reduction, the analyst may input any amount of data for each sample or standard. The procedure is fairly simple and reasonably fast.

The major drawback is that no computer compatable output is produced by the calculator and must be produced manually by the operator.

This process is probably best suited for check calculations and the stray analysis which must be performed occasionally.

The output is interpreted as follows:

A - average

E - variance

D - standard deviation

J - percent metal in the sample

M - percent error

Stepwise Operating Procedure

1. Be sure the PRINTER is OFF before turning the calculator on.

2. Turn the calculator on. The switch is on lower left, backside of the calculator.

3. Depress RUN.

4. Insert the tape, push REWIND.

5. Key PRIME.

6. Depress TAPE READY.

7. Key LOAD PROGRAM.

8. Key VERIFY PROGRAM (1738 should appear in the display, if not, repeat steps 5-8).

9. Depress f(x).

10. Key PRIME.

11. Key 01.

12. Enter the LOW STANDARD READOUT VALUES.

13. Key GO.

14. Repeat steps 12 & 13 for all values.

15. Key 00 after entering all of the data.

16. Repeat steps 12 to 15 for all of the UNKNOWN READOUT VALUES.

17. Repeat steps 12 to 15 for all of the HIGH STANDARD READOUT VALUES.

18. Key in the LOW STANDARD CONCENTRATION VALUE in P. P. M., GO.

19. Key in the HIGH STANDARD CONCENTRATION VALUE in P. P. M., GO.

20. Key in the SAMPLE WGT in milligrams, GO.

21. Key in the DILUTION FACTOR, GO (the no. which would raise the actual dilution to 1000 ml, i.e. if the actual dilution is 500 ml the factor is 2).

22. For further tests begin at STEP 12.

23. Be sure the PRINTER is OFF before turning the calculator off.

24. Switch the calculator OFF.

Program Listing For
The Wang 500 Data Reduction Program

000	09	00	* M
001	10	01	f 1
002	00	14	E14
003	00	02	E2
004	02	15	+15
005	09	00	* M
006	00	01	E1
007	09	03	* SP
008	08	02	* W
009	00	10	E10
010	10	02	f 2
011	08	00	* S
012	00	01	E1
013	09	00	* M
014	00	02	E2
015	01	00	T0
016	03	10	−10
017	02	11	+11
018	01	01	T1
019	02	12	+12
020	02	13	+13
021	09	00	* M
022	00	03	E3
023	09	03	* SP
024	08	02	* W
025	01	10	T10
026	10	02	f 2
027	08	00	* S
028	00	03	E3
029	09	00	* M
030	00	04	E4
031	01	00	T0
032	02	10	+10
033	01	01	T1
034	02	12	+12
035	09	00	* M
036	00	05	E5
037	09	03	* SP
038	08	02	* W
039	02	10	+10
040	10	02	f 2
041	08	00	* S
042	00	05	E5
043	09	00	* M
044	10	02	f 2
045	06	14	ST14
046	02	00	+0
047	01	14	T14
048	08	12	* x^2
049	02	01	+1
050	00	01	E1
051	02	02	+2
052	09	15	* RT
053	09	00	* M
054	10	03	f 3
055	06	04	ST4
056	05	10	÷10
057	07	04	RE4
058	05	03	÷3
059	01	04	T4
060	05	00	÷0
061	09	15	* RT
062	09	00	* M
063	10	04	f 4
064	08	02	* W
065	00	15	E15
066	08	02	* W
067	00	15	E15
068	09	15	* RT
069	09	00	* M
070	10	00	f 0
071	08	02	* W
072	00	15	E15
073	07	02	RE2
074	05	00	÷0
075	08	02	* W
076	03	10	−10

077	08	12	* x^2
078	06	03	ST 3
079	07	02	RE 2
080	04	03	× 3
081	03	01	− 1
082	00	01	E 1
083	03	02	− 2
084	01	02	T 2
085	05	01	÷ 1
086	08	02	* W
087	07	10	RE 10
088	08	13	* $\sqrt{x}$
089	08	02	* W
090	06	10	ST 10
091	10	04	f 4
092	00	01	E 1
093	03	15	− 15
094	08	05	* J
095	08	00	* S
096	00	06	E 6
097	09	04	* J
098	08	00	* S
099	00	04	E 4
100	08	00	* S
101	00	02	E 2
102	09	00	* M
103	00	06	E 6
104	01	00	T 0
105	02	11	+ 11
106	01	01	T 1
107	02	13	+ 13
108	08	02	* W
109	00	15	E 15
110	09	03	* SP
111	08	02	* W
112	14	10	C 10
113	06	03	ST 3
114	03	00	− 0
115	09	03	* SP
116	08	02	* W
117	10	10	f 10
118	02	00	+ 0
119	04	10	× 10
120	09	03	* SP
121	08	02	* W
122	09	10	* LG
123	10	03	f 3
124	09	03	* SP
125	08	02	* W
126	08	10	* LN
127	10	03	f 3
128	10	04	f 4
129	07	11	RE 11
130	05	10	÷ 10
131	02	03	+ 3
132	00	01	E 1
133	00	00	E 0
134	00	00	E 0
135	04	03	× 3
136	08	02	* W
137	12	10	A 10
138	07	11	RE 11
139	05	10	÷ 10
140	08	12	* x^2
141	04	13	× 13
142	07	11	RE 11
143	05	00	÷ 0
144	08	12	* x^2
145	04	12	× 12
146	02	13	+ 13
147	08	13	* $\sqrt{x}$
148	06	13	ST 13
149	00	01	E 1
150	00	00	E 0
151	00	00	E 0
152	04	13	× 13
153	08	02	* W
154	15	10	D 10
155	10	04	f 4
156	10	04	f 4
157	10	04	f 4
158	10	04	f 4
159	10	04	f 4
160	10	04	f 4
161	08	00	* S
162	10	01	f 1

An Example of the Data Input to the Wang 500 and the Results from the Calculator

201.0000000	X
202.0000000	X
199.0000000	X
197.0000000	X
203.0000000	X
199.0000000	X
200.1666666	A
4.966670800	E
2.228602880	D
231.0000000	Y
233.0000000	Y
235.0000000	Y
231.0000000	Y
232.0000000	Y
236.0000000	Y
233.0000000	A
4.400000000	E
2.097617696	D
410.0000000	Z
411.0000000	Z
408.0000000	Z
407.0000000	Z
409.0000000	Z
410.0000000	Z
409.1666666	A
2.166674000	E
1.471962635	D
1.020000000	L
2.040000000	H
397.5400000	G
5.000000000	F
.0593771310	J
.0007584712	M

Procedures for Using the CDC-3300 for AAS Data Reduction

Preliminary Discussion

Atomic absorption data may be reduced using the Computer Center's CDC-3300 computer operating under the OS-3 operating system.

Data must be taken manually then punched onto computer cards for processing.

This is an intermediate step between the Wang 500 and the PDP-15. The major disadvantage is that data must be punched onto computer cards manually. This offers no real advantage over the Wang. However, all results are punched onto computer cards which are immediately available for further work.

Data Card Formats

The program used to reduce the data performs all of the calculations for one sample before proceeding to the next. Therefore, the data must be arranged to conform to these requirements.

The formatting for each type of card is given and then a more detailed description of the order follows.

	Data Type	Columns (inclusive)
Card 1:	Sample name	1-24
Card 2:	Geochemistry Ascession no.	1-6
	IDK Ascession no.	8-14
	Subsampling typing code	16-18
	Sample weight (in mg. with a decimal)	20-30

Card 3:	Element analyzed (scientific abbreviation)	1-2
	Low standard concentration (ppm)	*
	High standard concentration (ppm)	*
	Dilution (ml.)	*
Card 4:	Low standard readout values	
	1st value (with decimal)	1-8
	2nd value (with decimal)	9-16
	3rd value (with decimal)	17-24
	etc. to a maximum of ten values	
Card 5:	Sample readout values same format as card 4	
Card 6:	High standard readout values same format as card 4	
Card 7:	Blank card to separate samples	

* These three values are read by the computer using freeform input. They may be typed in any column of Card 3 (following the Element name) in the given order and must be separated by a space.

The data deck will consist of the above seven types of cards repeated in a specific sequence.

For the first sample the operator will punch cards 1 and 2. Then for each element analyzed for that sample, cards 3, 4, 5, and 6 must be repeated sequentially. After all of the data for sample one has been punched, a blank card is inserted into the deck. This signals the computer that calculations have been completed for sample one. For further samples the sequence is repeated.

It must be noted that if the decimal is forgotten in values for the sample weight or the readout values, the computer will interpret an erroneous value.

Most of the common typing errors can be corrected while running the data reduction program. However, care in typing the data deck will save a great deal of time.

The program <u>cannot</u> operate correctly if any of the cards are out of sequence or missing. If this type of error should occur, it may be corrected by fixing the data deck and re-entering the deck to the computer.

Alternately, the data file may be corrected using the OS-3 Editor (4).

To enter the data deck into the computer, the following cards must be added:

Card 1: Cover card (from Computer Center)

Card 2: $^{7}_{8}$ JOB,.......card

Card 3: $^{7}_{8}$ COPY, 0 = (datafile name)

(the data deck)

Card 4: $^{7}_{8}$ $^{7}_{8}$

Card 5: $^{7}_{8}$ LOGOFF

Running the Program

Data is reduced on the CDC-3300 using a program called ATOMS and subprograms AVE, VARI and DEKODE plus routines from library files *CHEMLIB and *REGLIB. (16)

These programs have been compiled and an overlay program called *AAS has been produced. (For instructions on these and other procedures refer to the Control Mode Manual for OS-3 (13), the CDC FORTRAN reference manual (2) and Dayton and Massic (3).)

The procedure from this point will be described.

NOTE: When a name is in all capitals and enclosed by parenthesis, it denotes a single key.

1. After getting the I/O center to hook-up your teletype to the computer, type (CONTR)(A), the computer will respond with a #.

2. Type in your user number and validity code and type (RETURN). The computer will respond by masking out your number and typing the date and a #.

3. Type *AAS, (RETURN).

4. The computer will begin by asking you for your name, then proceed with further queries and information. The operator must supply the correct answers (usually a YES or NO).

5. After you have completed the data reduction process you must type LOGOFF after the computer types a #.

The results will be punched on computer cards in the correct format.

To correct typing errors on the teletype the following procedures may be used:

1. For a typing error in your name
   a. Type (CONTR)(A), the computer will respond with a #.
   b. Type GO, (RETURN), (LINE FEED)

c. Type in your name

2. An error in the data input file name will cause the computer to ask you for the name again. (Unless the name also exists - in which case you must reinitialize the program)

3. For errors in other alphanumeric names type (SHIFT)(L) for each mistake. The computer will respond with a \ for each character deleted counting from right to left.

4. For any numeric errors, type any letter except E. The computer will respond with ERR. Then type in the entire number.

```
      PROGRAM ATOMS
      DIMENSION X(10),Y(10),Z(10),XA(10),YA(10),ZA(10),IDEN(3),
     7IERMES(6),CHANGE(20),CORRECT(10)
      REAL IDEN,LOSTO,NAME,OPERATOR
      INTEGER ELEM,CHANGE,ERROR,ELEMNO
      PRINT 1
    1 FORMAT(≠ GUTEN TAG∨∨  LASS UNS ZUSAMMEN RECHNEN.≠/
     1≠ EXCEPT FOR YES-NO ANSWERS, ALWAYS END YOUR RESPONSES≠/
     2≠ BY TYPING THE RETURN KEY.≠//≠ PLEASE TYPE YOUR LAST NAME.≠)
      READ(60,2)OPERATOR,ADDRESS
    2 FORMAT(2A5)
    9 PRINT 10
   10 FORMAT(≠ WHAT IS THE DATA FILE NAME ^≠)
      READ(60,11)NAME
   11 FORMAT(A8)
      CALL UNEQUIP(5)
      IF(NEQUIP(5,NAME,IERMES))GO TO 14
      PRINT 15,IERMES
   15 FORMAT(1X,6A4)
      GO TO 9
   14 CALL UNEQUIP(62)
      CALL EQUIP(62,5HPUN  )
      CALL LABELP(62,OPERATOR)
      CALL UNEQUIP(10)
      CALL EQUIP(10,5HFILE )
C
C  DATA INPUT SECTION
C
  510 PRINT 16
   16 FORMAT(/////)
      READ (5,100)IDEN,ASCNO,ASCNID,IFRAC,WGT
  100 FORMAT(3A8/A6,1X,A7,1X,A3,F11.2)
      IF(EOF(5))GO TO920
      PRINT 105,IDEN,ASCNO,ASCNID,WGT,IFRAC
  105 FORMAT(≠ IDEN= ≠,3A8/≠ GEO= ≠,A6,5X,≠IDK= ≠,A7,5X,≠WGT= ≠,
     2F7.2,5X,≠FRAC= ≠,A3)
  104 GO TO(500,111)TELOS(≠OK^$≠)
  111 PRINT 106
  106 FORMAT(≠ WHICH VARIABLE IS INCORRECT^≠)
  114 READ(60,5)CHANGE
    5 FORMAT(20A4)
      CALL CANCEL(CHANGE)
      ERROR=CHANGE(1)
      IF(ERROR.EQ.3HWGT.OR.ERROR.EQ.3HGEO.OR.ERROR.EQ.3HIDK.OR.
     1ERROR.EQ.4HFRAC.OR.ERROR.EQ.4HIDEN)GO TO 117
      PRINT 116
  116 FORMAT(≠ WHAT^≠)
      GO TO 114
  117 IF(ERROR.EQ.3HWGT)GO TO 110
      PRINT 107
  107 FORMAT(≠ TYPE IN THE CORRECT IDENTIFIER.≠)
      IF(ERROR.NE.4HFRAC)GO TO 112
      READ(60,5)CHANGE
      CALL CANCEL(CHANGE)
      IFRAC=CHANGE(1)
      GO TO 104
  112 READ(60,109)CORRECT
  109 FORMAT(10A8)
      CALL CANCEL(CORRECT)
      IF(ERROR.EQ.3HGEO)ASCNO=CORRECT(1)
      IF(ERROR.EQ.3HIDK)ASCNID=CORRECT(1)
      IF(ERROR.NE.4HIDEN)GO TO 104
```

```
      DO 108 I=1,3
  108 IDEN(I)=CORRECT(I)
      GO TO 104
  110 WGT=TELOS(≠THE CORRECT WGT=$≠)
      GO TO 104
  500 IRUN=TELOS(≠TRIAL RUN=$≠)
  505 READ(5,101)ELEM
  101 FORMAT(A2)
      IF(ELEM.EQ.2H  )GO TO 910
      BACKSPACE 5
      LOSTD=FFIN(5)
      HISTD=FFIN(5)
      DILUTE=FFIN(5)
      READ(5,102)XA
      READ(5,102)YA
      READ(5,102)ZA
  102 FORMAT(10A8)
      CALL DEKODE(XA,NX,X)
      CALL DEKODE(YA,NY,Y)
      CALL DEKODE(ZA,NZ,Z)
      DO 120 I=1,10
      IF(X(I).GE.2000..OR.Y(I).GE.2000..OR.Z(I).GE.2000.)GO TO 121
  120 CONTINUE
      GO TO 140
  121 PRINT 122,ELEM
  122 FORMAT(≠ ERROR IN DATA FOR ≠,A2,≠ CALCULATIONS.≠)
      GO TO 300
C
C  CALCULATE AVERAGE VALUES USING SUBPROGRAM AVE
C
  140 XAVE=AVE(X,NX)
      YAVE=AVE(Y,NY)
      ZAVE=AVE(Z,NZ)
C
C  CALCULATE VARIANCES USING SUBPROGRAM VARI
C
      XVARI=VARI(X,NX,XAVE)
      YVARI=VARI(Y,NY,YAVE)
      ZVARI=VARI(Z,NZ,ZAVE)
C
C  CALCULATE PER CENT METAL AND ERROR
C
      FACTR=1000/DILUTE
      YX=YAVE-XAVE
      ZX=ZAVE-XAVE
      HL=HISTD-LOSTD
      FG=FACTR*WGT
      AMTMET=((LOSTD+HL*YX/ZX)/FG)*100.
      ERR=100*SQRT((HL/(FG*ZX))**2*(XVARI+YVARI)+(HISTD*YX/(FG*ZX**2
     1))**2*(XVARI+ZVARI))
      PER=(ERR/AMTMET)*100
C
C  PRINTS RESULTS AND ASKS OPERATOR TO CHECK THEM
C
      WRITE(61,190)ASCNO,ELEM,AMTMET,ERR
  190 FORMAT(1X,A6,5X,A2,2(5X,F12.4))
      WRITE(61,301)LOSTD,HISTD,DILUTE
  301 FORMAT(1X,4HLO= ,F10.4, 5X,4HHI= ,F10.4,5X,8HDILUTION,2H= ,F11.3/)
      GO TO(200,300)TELOS(≠OK^$≠)
C
C  PRINTS OUT DATA IF ERROR IS SUSPECTED AND ALLOWS FOR CORRECTION
C
  300 WRITE (61,303)
  303 FORMAT (2X,1HN,12X,1HX,13X,1HY,13X,1HZ/)
```

```
      DO 310 NI=1,10
      IF(NI.GT.NX)GO TO 303
      IF(NI.GT.NY)GO TO 307
      IF(NI.GT.NZ)GO TO 360
      WRITE(61,364)NI,X(NI),Y(NI),Z(NI)
  364 FORMAT(1X,I2,3(8X,F6.0))
      GO TO 310
  303 IF(NI.GT.NY)GO TO 305
      IF(NI.GT.NZ)GO TO 312
      PRINT 304,NI,Y(NI),Z(NI)
  304 FORMAT(1X,I2,14X,2(8X,F6.0))
      GO TO 310
  305 IF(NI.GT.NZ)GO TO 314
      PRINT 306,NI,Z(NI)
  306 FORMAT(1X,I2,36X,F6.0)
      GO TO 310
  307 IF(NI.GT.NZ)GO TO 309
      PRINT 308,NI,X(NI),Z(NI)
  308 FORMAT(1X,I2,8X,F6.0,22X,F6.0)
      GO TO 310
  309 PRINT 311,NI,X(NI)
  311 FORMAT(1X,I2,8X,F6.0)
      GO TO 310
  312 PRINT 313,NI,Y(NI)
  313 FORMAT(1X,I2,22X,F6.0)
      GO TO 310
  360 PRINT 361,NI,X(NI),Y(NI)
  361 FORMAT(1X,I2,2(8X,F6.0))
  310 CONTINUE
  314 PRINT 315
  315 FORMAT(≠ WHAT VARIABLE IS INCORRECT ^≠)
      READ(60,5)CHANGE
      CALL CANCEL(CHANGE)
      IF(CHANGE(1).EQ.4HNONE)GO TO 200
      IF(CHANGE(1).EQ.2HLO)GO TO 323
      IF(CHANGE(1).EQ.2HHI)GO TO 324
      IF(CHANGE(1).EQ.4HDILU)GO TO 319
      IF(CHANGE(1).EQ.1HX)GO TO 320
      IF(CHANGE(1).EQ.1HY)GO TO 321
      IF(CHANGE(1).EQ.1HZ)GO TO 322
      GO TO 350
  319 DILUTE=TELDS(≠DILUTION=$≠)
      GO TO 350
  320 I=TELDS(≠WHAT N EQUALS THE BAD DATA POINT^$≠)
      X(I)=TELDS(≠THE CORRECT X=$≠)
      IF(I.GT.NX)NX=I
      GO TO 350
  321 J=TELDS(≠WHAT N EQUALS THE BAD DATA POINT^$≠)
      Y(J)=TELDS(≠THE CORRECT Y=$≠)
      IF(J.GT.NY)NY=J
      GO TO 350
  322 K=TELDS(≠WHAT N EQUALS THE BAD DATA POINT^$≠)
      Z(K)=TELDS(≠THE CORRECT Z=$≠)
      IF(K.GT.NZ)NZ=K
      GO TO350
  323 LOSTD=TELDS(≠LO=$≠)
      GO TO 350
  324 HISTD=TELIO(8HHI=00000)
  350 GO TO (314,140)TELDS(≠ARE THERE ANY MORE ERRORS^$≠)
C
C  ASSIGNS ATOMIC NO TO THE ELEMENT
C
  200 ELEMNO=0
      IF(ELEM.EQ.2HCU)ELEMNO=29
```

```
      IF(ELEM.EQ.2HFE)ELEMNO=26
      IF(ELEM.EQ.2HMN)ELEMNO=25
      IF(ELEM.EQ.2HNI)ELEMNO=28
      IF(ELEM.EQ.2HSI)ELEMNO=14
      IF(ELEM.EQ.2HZN)ELEMNO=30
      IF(ELEM.EQ.2HAG)ELEMNO=47
      IF(ELEM.EQ.2HCA)ELEMNO=20
      IF(ELEM.EQ.2HMG)ELEMNO=12
      IF(ELEM.EQ.2HAL)ELEMNO=13
      IF(ELEM.EQ.1HK)ELEMNO=19
      IF(ELEM.EQ.2HBA)ELEMNO=56
      IF(ELEM.EQ.2HCO)ELEMNO=27
      IF(ELEMNO.NE.0)GO TO 900
      PRINT 210,ELEM
  210 FORMAT( ≠ WHAT IS THE ATOMIC NO. OF ≠,A2)
      READ(60,220)ELEMNO
  220 FORMAT(I2)
C
C   OUTPUT TO CARDS
C
  900 AMTMET=AMTMET*10000
      ERR=ERR*10000
      WRITE(62,901)ASCNO,IFRAC,ELEM,ELEMNO,AMTMET,ERR,PER,IDEN,
     1IRUN,ASCNIO
  901 FORMAT( 4HAASU,X,A6,X,A3,X,A2,X,I2,X,F12.4,X,F10.4,X,F5.2,X,
     12A8,A1,X,I2,X,A7)
      GO TO 505
  910 IF(IRUN.GT.1)GO TO 510
      WRITE(10,911) IDEN,IFRAC,ASCNID,ASCNO
  911 FORMAT( 2A8,A7,10X,A3,5X,A7,5X,A6)
      GO TO 510
C
C   FINAL DIRECTIONS AND OUTPUT OF CROSS REFERENCE CARDS.
C
  920 PRINT 921,OPERATOR
  921 FORMAT(≠ YOUR CARDS WILL BE SAVED FOR YOU UNDER THE NAME    ≠,A5/
     1≠ REMEMBER TO LOGOFF≠///≠ MERCI, NOUS SOMMES FINISvvv≠)
      ENDFILE 10
      REWIND 10
  924 READ(10,925)IDEN,IFRAC,ASCNID,ASCNO
  925 FORMAT(2A8,A7,10X,A3,5X,A7,5X,A6)
      IF(EOF(10))GO TO 930
      WRITE(62,911)IDEN,IFRAC,ASCNID,ASCNO
      GO TO 924
  930 CONTINUE
      END
      FUNCTION AVE(W,N)
      DIMENSION W(1)
      SUM=0
      DO 1 L=1,N
    1 SUM=SUM+W(L)
      AVE=SUM/N
      RETURN
      END
      FUNCTION VARI(W,N,AVENO)
      DIMENSION W(1)
      SUM=0
      DO 1 L=1,N
    1 SUM=SUM+((W(L)-AVENO)**2)
      VARI=SUM/(N-1)
      RETURN
      END
      SUBROUTINE DEKODE(DATA,N,DAT)
      DIMENSION DATA(1),DAT(1)
```

```
      BLANKS=8H
      N=0
      DO 1 I=1,10
      IF(DATA(I).EQ.BLANKS)RETURN
      N=N+1
      DECODE(8,2,DATA(I))DAT(I)
    2 FORMAT(F8.0)
    1 CONTINUE
      RETURN
      END
```

An Example of the Data Input Format for the CDC 3300 Data Reduction Program

```
OC73-3-17P 75-80 CM
MS0812          AAA 375.41
FE 1.50 2.00 20000
473.      470.      475.      470.      474.      478.
644.      642.      645.      648.      650.      651.
664.      663.      659.      666.      665.      667.
MN 3.0 4.0 20000.
585.      584.      584.      686.      589.      592.
627.      625.      631.      626.      632.      627.
784.      783.      782.      788.      788.      785.
CU 0.40 0.80 200.
139.      138.      137.      137.      138.      139.
252.      254.      257.      254.      257.      255.
264.      263.      265.      264.      266.      267.
NI 0.40 0.80 200.
212.      215.      212.      216.      212.      215.
277.      276.      281.      280.      279.      282.
437.      438.      435.      435.      438.      437.

DSDP 34-319-8-5 114-116
MS1214          AAA 401.32
FE 0.25 0.50 20000.
106.      108.      107.      108.      108.      109.
187.      185.      187.      188.      189.      187.
208.      209.      213.      207.      207.      213.
CU 0.20 0.40 200.
102.      104.      102.      101.      105.      104.
164.      165.      160.      159.      163.      159.
207.      210.      207.      207.      207.      212.
SI 0.0 50.37 200.
-1.       0.        0.        0.        0.        0.
38.       38.       39.       41.       39.       40.
148.      151.      150.      152.      152.      152.
MN 0.10 0.20 20000.
106.      008.      108.      110.      111.      106.
127.      131.      131.      129.      131.      131.
221.      223.      222.      221.      222.      219.

DDDP 34-319-10-2 86-88CM
MS1215          AAX 010.00
MN 0.01 0.20 20000.
121.      120.      121.      125.      122.      121.
152.      149.      121.      154.      148.      151.
228.      229.      231.      233.      232.      231.
FE 0.25 5.00 200000.
106.      108.      107.      108.      108.      109.
187.      185.      187.      188.      189.      187.
208.      209.      213.      207.      207.      213.
CA 6.0 7.0 20000.
634.      640.      647.      640.      636.      638.
744.      735.      742.      737.      743.      740.
745.      749.      745.      751.      747.      744.
```

An Example of the Operation of the CDC-3300 Program

[redacted]

```
AUGUST 2Ø, 1975  11:Ø4:Ø7 AM TERMINAL Ø73-Ø45B

#**AAS
GUTEN TAG!!  LASS UNS ZUSAMMEN RECHNEN.
EXCEPT FOR YES-NO ANSWERS, ALWAYS END YOUR RESPONSES
BY TYPING THE RETURN KEY.

PLEASE TYPE YOUR LAST NAME.
FUKUI
WHAT IS THE DATA FILE NAME ?
XXSDATA
NAME NOT FOUND
WHAT IS THE DATA FILE NAME ?
AASDATA




IDEN= OC73-3-17P 75-80 CM
GEO= MSØ812        IDK=                    WGT=  375.41       FRAC= AAA
OK?YES
TRIAL RUN=1
MSØ812        FE            1Ø.4129                 .2223
LO=       1.5ØØØ      HI=        2.ØØØØ      DILUTION=     2ØØØØ.ØØØ

OK?YES
MSØ812        MN            16.7Ø59                1.3588
LO=       3.ØØØØ      HI=        4.ØØØØ      DILUTION=     2ØØØØ.ØØØ

OK?NO
 N              X                 Y                 Z

 1             585               627               784
 2             584               625               783
 3             584               631               782
 4             686               626               788
 5             589               632               788
 6             592               627               785
WHAT VARIABLE IS INCORRECT ?
X
WHAT N EQUALS THE BAD DATA POINT?4
THE CORRECT X=586
ARE THERE ANY MORE ERRORS?NO
MSØ812        MN            17.Ø928                 .1468
LO=       3.ØØØØ      HI=        4.ØØØØ      DILUTION=     2ØØØØ.ØØØ

OK?YES
MSØ812        CU              .Ø4Ø9                 .ØØØ6
LO=        .4ØØØ      HI=         .8ØØØ      DILUTION=       2ØØ.ØØØ

OK?YES
MSØ812        NI              .Ø276                 .ØØØ3
LO=        .4ØØØ      HI=         .8ØØØ      DILUTION=       2ØØ.ØØØ

OK?YES
```

```
IDEN= DSDP 34-319-8-5 114-116
GEO= MS1214      IDK=                   WGT=  401.32      FRAC= AAA
OK?YES
TRIAL RUN=1
MS1214      FE           2.2185             .0608
LO=       .2500     HI=       .5000     DILUTION=    20000.000

OK?YES
MS1214      CU            .0155             .0004
LO=       .2000     HI=       .4000     DILUTION=      200.000

OK?YES
MS1214      SI            .6539             .0218
LO=           0     HI=     50.3700     DILUTION=      200.000

OK?YES
MS1214      MN            .6461             .1829
LO=       .1000     HI=       .2000     DILUTION=    20000.000

OK?NO
 N           X             Y            Z

 1          106           127          221
 2            8           131          223
 3          108           131          222
 4          110           129          221
 5          111           131          222
 6          106           131          219
WHAT VARIABLE IS INCORRECT ?
X
WHAT N EQUALS THE BAD DATA POINT?3N ERR 2
THE CORRECT X=108
ARE THERE ANY MORE ERRORS?NO
MS1214      MN            .5945             .0124
LO=       .1000     HI=       .2000     DILUTION=    20000.000

OK?YES




IDEN= DDDP 34-319-10-2 86-88CM
GEO= MS1215      IDK=                   WGT=   10.00      FRAC= AAX
OK?NO
WHICH VARIABLE IS INCORRECT?
IDEN
TYPE IN THE CORRECT IDENTIFIER.
DSDP 34-319-10-2 86-88CM
OK?NO
WHICH VARIABLE IS INCORRECT?
WGT
THE CORRECT WGT=410.98
OK?YES
TRIAL RUN=1
MS1215      MN            .2537             .1059
LO=       .0100     HI=       .2000     DILUTION=    20000.000
```

```
OK?NO
 N              X              Y              Z

 1             121            152            228
 2             120            149            229
 3             121            121            231
 4             125            154            233
 5             122            148            232
 6             121            151            231
WHAT VARIABLE IS INCORRECT ?
LO
LO=.1
ARE THERE ANY MORE ERRORS?YES
WHAT VARIABLE IS INCORRECT ?
Y
WHAT N EQUALS THE BAD DATA POINT?3
THE CORRECT Y=151.
ARE THERE ANY MORE ERRORS?NO
MS1215      MN             .6169              .0138
LO=       .1000     HI=        .2000     DILUTION=    20000.000

OK?YES
MS1215      FE          192.6257             6.7675
LO=       .2500     HI=       5.0000     DILUTION=   200000.000

OK?NO
 N              X              Y              Z

 1             106            187            208
 2             108            185            209
 3             107            187            213
 4             108            188            207
 5             108            189            207
 6             109            187            213
WHAT VARIABLE IS INCORRECT ?
HI
HI=.5
ARE THERE ANY MORE ERRORS?NO
MS1215      FE           21.6639              .5937
LO=       .2500     HI=        .5000     DILUTION=   200000.000

OK?NO
 N              X              Y              Z

 1             106            187            208
 2             108            185            209
 3             107            187            213
 4             108            188            207
 5             108            189            207
 6             109            187            213
WHAT VARIABLE IS INCORRECT ?
DILUTION
DILUTION=20000.
ARE THERE ANY MORE ERRORS?NO
MS1215      FE            2.1664              .0594
LO=       .2500     HI=        .5000     DILUTION=    20000.000

OK?YES
MS
```

```
MS1215     CA          33.7636          1.5787
LO=      6.0000     HI=      7.0000     DILUTION=     20000.000

OK?YES




YOUR CARDS WILL BE SAVED FOR YOU UNDER THE NAME    FUKUI
REMEMBER TO LOGOFF


MERCI, NOUS SOMMES FINIS!!!




END OF FORTRAN EXECUTION

#LOGOFF
COST $1.30
CPU TIME SEC.     9.6
MFBLKS              2
SFBLKS            255
WC TIME  MIN.    13.8
PUN RECORDS        15
```

A Listing of the Data Cards Output by the CDC-3300 Data Reduction Program

```
AASU MS0812 AAA FE 26  104128.5770   2222.6104  2.13 OC73-3-17P 75-80    1
AASU MS0812 AAA MN 25  170927.9647   1467.5397   .86 OC73-3-17P 75-80    1
AASU MS0812 AAA CU 29     409.3991      6.4278  1.57 OC73-3-17P 75-80    1
AASU MS0812 AAA NI 28     275.6925      3.1220  1.13 OC73-3-17P 75-80    1
AASU MS1214 AAA FE 26   22185.3807    607.9628  2.74 DSDP 34-319-8-5 1   1
AASU MS1214 AAA CU 29     155.1841      4.0407  0.60 DSDP 34-319-8-5 1   1
AASU MS1214 AAA SI 14    6533.7532    217.9448  3.33 DSDP 34-319-8-5 1   1
AASU MS1214 AAA MN 25    5945.6353    123.5006  2.08 DSDP 34-319-8-5 1   1
AASU MS1215 AAX MN 25    6158.5926    137.6410  2.23 DSDP 34-319-10-2    1
AASU MS1215 AAX FE 26   21663.9179    593.6727  2.74 DSDP 34-319-10-2    1
AASU MS1215 AAX CA 20  337635.9188  15787.0701  4.68 DSDP 34-319-10-2    1
OC73-3-17P 75-80 CM           AAA                   MS0812
DSDP 34-319-8-5 114-116       AAA                   MS1214
DSDP 34-319-10-2 86-88C       AAX                   MS1215
```

The BCD-ASCII Converter - Teletype System

Introduction

The BCD-ASCII converter used to obtain teletype compatable output from the AAS was designed by Mike Cranford and assembled and installed by Ron Stillinger and Milo Clausen. It will take the parallel, BCD output signal from the spectrophotometer's digital voltmeter and convert it to a serial, ASCII signal which is teletype compatable.

The speed of the system is limited by the teletype which can handle approximately one six-character output every second.

This system of data handling has proven to be very efficient if the directions are followed completely. The operator should be thoroughly familiar with the following directions before attempting to use this system because the correction of errors must be done on the papertape before being introduced to the computer. If these types of errors are not corrected immediately following their occurrence, the process to remedy the situation will become long and tedious.

Operation

The BCD-ASCII converter is switched on and off by the AAS on-off switch. Therefore, the converter is operable any time the AAS is on.

Startup and shutdown of the system must be done in the following order to avoid miscellaneous characters on the tape. The characters are caused by electrical transients which occur when a switch is turned on or off.

Startup

1. Turn on the AAS

2. Turn the teletype switch to (ONLINE)

3. Push the tape-punch (ON) button

Shutdown

1. Push the tape punch (OFF) button

2. Turn the teletype switch to (OFF)

3. Turn off the AAS

In addition to the three switches above, an additional push button is located on the right side of the teletype keyboard. This will disable the converter so that data may be typed onto the paper tape from the keyboard.

As an added precaution the tape punch should be turned off before turning any switches on the AAS. The flame igniter in particular will produce transients which will cause the teletype to print miscellaneous characters.

Special Editing and Code Characters

To give the analyst a data reduction program which is flexible enough to handle a variable number of samples and data points and allow him to easily correct typing errors, the following characters are used to indicate to the computer that it should perform the specified operation.

These characters should not be used as part of the sample name or subsample typing code. Their use in these instances would cause the data reduction program to malfunction.

In addition to these special characters the computer will ignore rubouts, line feeds and nulls.

• Backslash (\)

This character will delete the last character typed in a line. A series of backslashes will delete characters from right to left until a carriage return is encountered. The backslash is formed by typing (SHIFT)(L).

• Commercial at sign (@)

This character will delete the entire line up to the previous carriage return.

NOTE: The automatic carriage return produced by the teletype is not punched onto the papertape and has no effect on the character or line delete symbols.

NOTE: The backslash and commercial at sign do not affect the following code characters except for the asterisk and the first pound sign in the two set series. To erase the other characters, the tape must be manually backspaced to the correct position then a (RUBOUT) must be typed.

• Asterisk (*)

Two asterisks typed in succession indicates the end of the sample identification data to the computer.

• Pound Sign (#)

A single pound sign, when first encountered by the computer indicates to it that all of the following data are <u>numbers</u> which originated from the AAS. The next pound sign encountered by the computer, signifies the end of the data from the AAS. The sequence is then repeated.

• Ampersand (&)

The ampersand signals the end of the data for a particular element. It is used <u>in place of the second pound sign</u> after all data has been taken for a particular element.

• Dollar Sign ($)

The dollar sign is used to indicate the physical end-of-tape to the computer. It must always be the last character on all tape segments. Any data following a dollar sign on a tape will be ignored by the computer.

<u>Tape Format</u>

The formatting required on the paper tape is very restrictive and any deviations from the prescribed patterns will cause the data reduction program to malfunction.

Mistakes made in the tape format must be corrected by repunching the tape and editing out errors manually - a very tedious process. It cannot be performed on the computer as with the CDC-3300 program. This is due to the lengths of some of the data lines and the internal programming of the PDP-15.

Since these types of errors do occur, even when great care is taken, it is strongly suggested that the data tape be divided into several segments. This is done by use of the dollar sign ($) which is described later.

Tapes should be segmented between each element and in addition the sample identification data should be punched on a separate tape. NOTE: All tapes should have a long leader to make it easier to load into the computer. Leaders and trailers are made by typing (HERE IS).

The sample identification tape must be formated as follows:

	DATA	COLUMNS (inclusive)
LINE 1:	Sample name	1-24
LINE 2:	Ascession No.	1-6
	Subsample typing code	8-10
	Sample weight (in mg. with a decimal)	12-20
LINE 3:	Trial Run number (right adjusted)	1-2

The above sequence is repeated for all of the samples run in a particular experiment, up to a maximum of twenty samples. The last sample is followed by two asterisks (*) and then a dollar sign ($) on the next line. The tape may then be removed from the teletype.

The order of the samples in the above list is very important since the first one is labeled unknown one (U01) by the computer, the second unknown two (U02), etc.

The description of the element - data tapes are more difficult since many different possibilities exist for the formating of each line.

Line 1:

- Line one must always contain the element name in its scientific abbreviation in columns 1 and 2.

Line 2 - (4 to 9):

- The next set of data will describe the first set of solutions to be run through the AAS and their sequence.
- In one group, two standards and from one to seven unknowns are run.
- Standards are identified with an "S" and a two digit number to identify which standard. i.e. S02 would indicate standard number two to the computer. This data must be in columns 1-3. The number will normally match the number on the standard solution bottle.
- Unknowns are identified with a "U" and a two digit number which will coincide with its position on the sample identification tape. i.e. U01 would indicate the first sample in the list. This data must also be in columns 1-3.
- Following all unknown listings and on the same line is the dilution number. This must include a decimal and be in columns 5-13.
- The standards and unknowns may be listed in any order, but they must be analyzed in that order.
- One of the standards should have a higher concentration of the element of interest and the other should have a lower

concentration than the sample being analyzed.

- After all samples and standards are listed for this group, a single pound sign (#) should follow in column one.

The following data lines are automatically formated by the teletype.

The operator should begin aspirating the first solution listed above.

Data are obtained by pressing the BCD-ASCII converter enable-disable button on the teletype keyboard.

A maximum of 125 data points (10+ lines) are allowed at one time for one solution.

After enough data points have been obtained the operator will again push the BCD-ASCII converter enable-disable button.

Then he must type (RETURN)(LINE FEED). This signals the computer that the next number is for the next solution in the sequence.

NOTE: The automatic return - line feed is not punched onto the paper-tape and has no effect on the program.

The operator will then repeat the above sequence for all of the solutions in the first list.

He may then repeat the sequence starting with the first solution and obtain up to 125 more numbers.

The sequence may be repeated seven times for each group.

After the first set of data has been obtained, the operator may stop anywhere in the sequence.

After enough data have been collected, the operator must type a pound sign (#) in column one.

The next group of samples is listed and run as above. This sequence is repeated until all of the samples have been run for this element.

The ampersand (&) is then typed in place of the pound sign (#) at the end of the data. The ampersand is then followed by the dollar sign ($) in column one of the next line.

This tape may now be removed and the entire process repeated for another element.

Operation of the PDP-15

Introduction

The PDP-15 computer utilizes a main program called AAS and fourteen subroutines to complete its tasks. Most of the routines, including AAS, have been written in DEC Fortran IV. (6) Subroutines Editor and Erase have been coded in Macro-15 assembly language.(7)

All of these programs have been compiled or assembled and the binary routines have been combined under the name AAS using the UPDATE utility program. (9)

For a more complete explanation of the DEC PDP-15 system the reader is referred to the Digital Equipment Corporation reference manuals (5, 6, 7, 8, 9).

Stepwise Operating Procedure

NOTE: When a name is in capitals and enclosed by parenthesis, it denotes a single key or switch.

1. Mount the Dectape marked Geochem on to one of the drive units. Turn the cogwheel to 5 and move the two switches on the drive unit to WRITE LOCK and REMOTE.

2. Mount a scratch tape onto another drive unit. Turn the cogwheel to 4 and move the two switches to WRITE ENABLE and REMOTE.

3. Switch Dectape unit 0 to WRITE LOCK and REMOTE.

NOTE: If this tape is not marked B/F MONITOR (background/foreground), it must be removed and the proper tape must be mounted. The system then must be loaded into the computer. Directions for this procedure are located in the PDP-15 Operations Notebook.

4. Turn on the line printer. Push the (SELECT) button (the light should go on) and be sure the paper is mounted correctly.

5. Turn on the Tekscope. This is done with the key.

6. Push the (ASCII-TTY) button. The light should be off.

7. Push the (ONLINE-LOCAL) button. The light should be off.

8. Turn on teletype 0 and type (CTRL)(C). The computer should respond by typing a $. If the computer does not respond, see the directions for reloading the system in the PDP-15 operations notebook.

9. After the computer responds properly, type BCONTROL 3 (CR). This transfers control of the background portion of the computer to the tekterminal.

10. Mount the paper tape with your sample identification data onto the high speed tape reader.

11. Push the Reader (FEED) button to clear the reader. Be sure that none of your data has passed by the read head.

12. On the tekterminal:

    Push (ERASE) to clear the screen and type (ETX). This is equivalent to typing (CTRL)(C).

13. After the computer responds with a $, type in the following information:

    ```
    A TT3 4,5/TT2 3 (CR)
    GLOAD (CR)
    ```

    The first statement assigns the tekterminal to logical units 4 and 5 and the line printer to unit 3. The second statement invokes the program loader. After the first line the computer will respond with $. After the second line the computer will type BGLOAD V2A.

14. Now type

(_)AAS(?)

This will load the data reduction program into the computer. The papertape should be read in and the computer should ask

MORE DATA?

15. Mount the next paper tape and then type

YES(CR)

This should be repeated until all tapes have been read.

16. The rest of the program is operated by answering queries from the computer.

17. After you have finished, type BCONTROL 0. The computer will respond as before.

18. Push the (TOP OF FORM) button on the line printer until you can remove your data.

19. Push the paper tape punch's (FEED) switch until you have a tail of blanks on the tape. Then remove the tape.

20. Turn off the line printer, tekterminal, teletype and all of the tape drives.

21. Remove the Geochem tape from the drive.

22. Record the time you used the computer into the log book.

The computer has output all of the final data onto the line printer and punched the same information in card image form onto the paper tape.

This tape must now be taken to the computer center and copied into their computer. This ASCII coded information is then converted to BCD code and punched onto cards.

Program Listing For
The PDP-15 Data Reduction Program

```
CC
C
C********************ATOMIC ABSORBTION SPECTROPHOTOMETRY*****************
C          ***************************(AAS)*****************************
C
C
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                                       C
C       C                                                       C
C       C     AAS IS A PROGRAM WRITTEN EXPRESSLY  FOR THE       C
C       C  PURPOSE OF REDUCING DATA FROM THE JARREL-ASH 820     C
C       C  ATOMIC ABSORPTION SPECTROPHOTOMETER USING THE        C
C       C  BCD-ASCII CONVERTER AND PAPER TAPE.                  C
C       C  AAS CALLS THE MACRO PROGRAM EDITOR TO READ IN THE     C
C       C  PAPER TAPE AND TRANSFER IT TO DEKTAPE.   THEN AAS     C
C       C  PROCESSES THE DATA-OUTPUTS THE RESULTS TO THE PAPER  C
C       C  TAPE PUNCH IN CARD IMAGE FORM SO THAT A COPY ON OS3  C
C       C  WILL PRODUCE THE DESIRED CARDS.   AAS WILL ALSO PRINT C
C       C  ALL OF THE RESULTS ON THE LINEPRINTER FOR FILING.    C
C       C                                                       C
C       C                                                       C
C       C     THE ASSIGNMENTS IN THE .DAT WHICH HAVE TO BE      C
C       C  MADE ARE:                                            C
C       C                                                       C
C       C        DEVICE             .DAT                        C
C       C                                                       C
C       C   DEKTAPE UNIT 4          1                           C
C       C   DEKTAPE UNIT 5          2                           C
C       C   LINEPRINTER (TT2)       3                           C
C       C   TEKTRM (TT3) OUTPUT     4                           C
C       C   TEKTRM (TT3) INPUT      5                           C
C       C   PAPERTAPE READER        6                           C
C       C   PAPERTAPE PUNCH         7                           C
C       C                                                       C
C       C                                                       C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
C
C
      INTEGER SRT,RUN
      REAL NB
C
      DIMENSION SNM(120),ASN(40),SSC(20),WGT(20),  ELM(20),STD(10),
     1BT(9),NB(9),DIL(9),X(125),AVE(72),VAR(72),FMT(24),RUN(20),
     2CON(20,20),ERR(20,20),PC(20,20),DT1(2),DT2(2),QMKFLE(2)
C
      COMMON STD,X,QMK,NRD,SNM,ASN,SSC,RUN,WGT,CON,ERR,PC
C
      DATA YES/3HYES/,PD/1HD/,S/1HS/,BTL/3HBTL/,DI/3HDIL/,
     1R1/3HR1 /,R2/3HR2 /,R3/3HR3 /,R4/3HR4 /,R5/3HR5 /,R6/3HR6 /,
     2R7/3HR7 /,R8/3HR8 /,DT1(1),DT1(2)/5HAASDA,4HTAED/,CA/2HCA/,
     3U/1HU/,U1/2HU1/,U0/2HU0/,U2/2HU2/,S0/2HS0/,S1/2HS1/
     4,SP/2HS /,QMKFLE(1),QMKFLE(2)/5HQUEST,4HNMRK/,D0/2HD0/
C
C
```

```
C         ZERO ALL OF THE ELEMENTS OF ARRAY CONC
C
C
      DO 2 I=1,15
      DO 1 J=1,20
    1 CON(I,J)=0
    2 CONTINUE
      NRD=NOLIST=0
C
C
C         READ CODE FOR QUESTION MARK(?)
C
C
      CALL SEEK(2,QMKFLE)
      READ(2,901)QMK
  901 FORMAT(A1)
      CALL CLOSE(2)
      CALL ERASE
C
C
C         CALL THE MACRO SUBPROGRAM
C
C
      WRITE(4,895)QMK
  895 FORMAT(1H ,31HDO YOU WISH TO USE THE STANDARD/
     11H ,28HCONCENTRATION VALUES ON FILE,A1)
      CALL YESNO(ANS)
      IF(ANS.NE.YES)NRD=1
      WRITE(4,890)QMK
  890 FORMAT(1H ,34HDO YOU WISH TO READ IN PAPER TAPES,A1)
      CALL YESNO(ANS)
      IF(ANS.NE.YES)GO TO 3
      CALL EDITOR
C
C
C         SEEK THE DATA FILE JUST CREATED BY EDITOR AND OPEN
C         IT SO THAT IT CAN BE USED.
C
C
    3 CALL SEEK(1,DT1)
C
C         READ IN THE SAMPLE NAME, ACSESSION NUMBER, SUBSAMPLE CODE,
C         WEIGHT OF THE SAMPLE AND THE NUMBER OF TIMES THE SAMPLE HAS
C         BEEN RUN.
C
C
      CALL SAMPLE(KNT)
C
C
C         READ IN THE ELEMENT NAME, READ IN THE STANDARD VALUES FOR THAT
C         ELEMENT AND ASSIGN IT A PLACE IN THE CON, ERR, AND PC ARRAYS
C         FOR OUTPUT TO THE LINEPRINTER.
C
C
   24 READ(1,930)ENM
  930 FORMAT(A2)
      IF(ENM.EQ.DO)GO TO 175
      CALL ELMSRT(ENM,SRT,NEL,NOLIST)
```

```
      ELM(SRT)=ENM
C
C
C        READ IN THE ORDER OF SAMPLES AND STANDARDS WHICH HAVE
C        BEEN RUN, AND THE DILUTION OF THE UNKNOWNS.
C
C
   40 DO 60 I=1,9
      READ(1,935)BT(I),NB(I),DIL(I)
  935 FORMAT(A1,A2,F10.2)
      IF(BT(I).EQ.PD)GO TO 62
   60 CONTINUE
   62 NSM=I-1
C
C
C        READ IN THE VALUES FROM THE SPECTROPHOTOMETER.
C
C
      DO 80 I=1,72
      READ(1,937)N
  937 FORMAT(I4)
      IF(N.GT.590)GO TO 82
      READ(1,940)(X(J),J=1,N)
  940 FORMAT(125F5.0)
C
C
C        CALCULATE THE MEAN AND THE STANDARD DEVIATION.
C
C
      AV1=AVERAG(N)
      VA1=VARIAN(N,AV1)
      SG2=2.*SQRT(VA1)
C
C
C        ELIMINATE ALL VALUES GREATER THEN TWO STANDARD DEVIATIONS
C        AWAY FROM THE MEAN.
C
AV1+SG2
ES GREATER THEN TWO STANDARD DEVIATIONS
YS
,
      TRG=AV1+SG2
      BRG=AV1-SG2
      DO 76 K=1,N
      IF(X(K).GT.BRG.AND.X(K).LT.TRG)GO TO 76
      X(K)=9999.
   76 CONTINUE
C
C
C        RECALCULATE THE MEAN AND CALCULATE THE VARIANCE.
C
C
      AVE(I)=AVERAG(N)
      VAR(I)=VARIAN(N,AVE(I))
   80 CONTINUE
   82 NRP=I-1
C
C
```

```
C         PRINTS THE SAMPLE OR STANDARD BOTTLE NUMBER, THE DILUTION,
C         AND THE AVERAGE OF EACH OF THE GROUPS. THEN ASKS THE
C         OPERATOR TO CHECK THEM.
C
C
      NST=NSR=1
   84 CALL ERASE
   85 CALL ERPWRT(BT,NB,DIL,NRP,NSM,AVE,ELM(SRT))
      IF(BTLCK.EQ.0.)GO TO 90
      CRT=BTL
      GO TO 105
   90 WRITE(4,945)QMK
  945 FORMAT(//1H ,2HOK,A1)
      CALL YESNO(ANS)
      IF(ANS.EQ.YES)GO TO 132
C
C
C         THE PROGRAM NOW ALLOWS FOR THE CORRECTION OF A BOTTLE
C         NUMBER OR  DILUTION NUMBER AND ALLOWS THE OPERATOR TO
C         DELETE ANY AVERAGES WHICH HE FEELS ARE INVALID.
C
C
   95 WRITE(4,955)QMK
  955 FORMAT(1H ,26HWHICH VARIABLE IS IN ERROR,A1,31H  (BTL, DIL
     1UTION, R1, R2, ETC.))
   97 READ(5,960)CRT
  960 FORMAT(A2)
      IF(CRT.EQ.BTL.OR.CRT.EQ.DI.OR.CRT.EQ.
     1R1.OR.CRT.EQ.R2.OR.CRT.EQ.R3.OR.CRT.EQ.R4.OR.
     2CRT.EQ.R5.OR.CRT.EQ.R6.OR.CRT.EQ.R7.OR.CRT.EQ.
     3R8)GO TO 105
      WRITE(4,915)QMK
  915 FORMAT(1H ,4HWHAT,A1)
      GO TO 97
  100 WRITE(4,962)QMK
  962 FORMAT(1H ,20HWHAT IS THE IND. NO.,A1)
      READ(5,965)IND
  965 FORMAT(I1)
      IF(CRT.NE.BTL)GO TO 115
      WRITE(4,967)QMK
  967 FORMAT(1H ,30HWHAT IS THE CORRECT BTL NUMBER,A1)
      READ(5,935)BT(IND),NB(IND)
      IF(BTLCK.EQ.1.)GO TO 136
      IF(BTLCK.EQ.2.)GO TO 152
      GO TO 125
  115 IF(CRT.NE.DI)GO TO 120
      WRITE(4,972)QMK
  972 FORMAT(1H ,29HWHAT IS THE CORRECT DILUTION,A1)
      READ(5,975)DIL(IND)
  975 FORMAT(F10.2)
      GO TO 125
  120 IF(CRT.EQ.R2)IND=IND+NSM
      IF(CRT.EQ.R3)IND=IND+2*NSM
      IF(CRT.EQ.R4)IND=IND+3*NSM
      IF(CRT.EQ.R5)IND=IND+4*NSM
      IF(CRT.EQ.R6)IND=IND+5*NSM
      IF(CRT.EQ.R7)IND=IND+6*NSM
      IF(CRT.EQ.R8)IND=IND+7*NSM
```

```
      AVE(IND)=9999.
  125 WRITE(4,945)OMK
      CALL YESNO(ANS)
      IF(ANS.EQ.YES)GO TO 84
      GO TO 95
C
C
C       AVERAGE THE MEAN OF THE GROUPS FOR EACH SAMPLE
C
C
  132 DO 135 I=1,NSM
      AVR=VAN=REP=0.
      DO 134 J=1,NRP,NSM
      K=I+J-1
      IF(K.GT.NRP)GO TO 134
      IF(AVE(K).GE.2000.)GO TO 134
      AVR=AVR+AVE(K)
      VAN=VAN+VAR(K)
      REP=REP+1.
  134 CONTINUE
      AVE(I)=AVR/REP
  135 VAR(I)=VAN/REP
C
C
C       ROUTINE TO DETERMINE WHICH BOTTLES IN THE LIST ARE
C       STANDARDS.
C
C
      FLG=0.
  136 BTLCK=0.
      DO 150 I=NSR,NSM
      NSR=I
      IF(BT(I).EQ.U)GO TO 150
      IF(FLG.EQ.1.)GO TO 137
      CALL MATCH(NB(I),NS1,BTLCK)
      IF(BTLCK.EQ.1.)GO TO 85
      NP1=I
      FLG=1.
      GO TO 150
  137 CALL MATCH(NB(I),NS2,BTLCK)
      IF(BTLCK.EQ.1.)GO TO 85
      NP2=I
      IF(NS1.LT.NS2.AND.ELM(SRT).NE.CA)GO TO 152
      IF(NS1.GT.NS2.AND.ELM(SRT).EQ.CA)GO TO 152
      NTP=NS2
      NS2=NS1
      NS1=NTP
      NTP=NP2
      NP2=NP1
      NP1=NTP
      GO TO 152
  150 CONTINUE
C
C
C       CALCULATE THE CONCENTRATION, ERROR AND PERCENT ERROR
C
C
  152 BTLCK=0.
```

```
      DO 170 J=NST,NSM
      NST=J
      IF(BT(J).EQ.5)GO TO 170
      CALL UNKNOW(NB(J),NUN,BTLCK)
      IF(BTLCK.EQ.2.)GO TO 85
      FG=WGT(NUN)*1000./DIL(J)
      YX=AVE(J)-AVE(NP1)
      ZX=AVE(NP2)-AVE(NP1)
      HL=STD(NS2)-STD(NS1)
      CON(SRT,NUN)=((STD(NS1)+HL*YX/ZX)/FG)*1.0E06
      ERR(SRT,NUN)=1.E06*SQRT((HL/(FG*ZX))**2*(VAR(NP1)+
     1VAR(J))+(STD(NS2)*YX/(FG*ZX**2))**2*(VAR(NP1)+VAR(NP2)))
      PC(SRT,NUN)=ERR(SRT,NUN)/CON(SRT,NUN)*100.
C
C
C        WRITE THE RESULTS ONTO PAPER TAPE
C
C
      LL=NUN*2
      L=LL-1
      MM=(NUN*6)-2
      M=MM-3
      WRITE(7,985)(ASN(K),K=L,LL),SSC(NUN),ELM(SRT),
     1NEL,CON(SRT,NUN),ERR(SRT,NUN),PC(SRT,NUN),
     2(SNM(K),K=M,MM),RUN(NUN)
  985 FORMAT(1H ,4HAASU,1X,A2,A4,1X,A3,1X,A2,1X,I2,1X,F12.4,1X,
     1F10.4,1X,F5.2,1X,A2,3A5,1X,I2)
  170 CONTINUE
      IF(N.EQ.9999)GO TO 40
      IF(N.EQ.8888)GO TO 24
C
C
C        WRITE ALL OF THE FINAL RESULTS TO THE LINE PRINTER
C        AND ALL OF THE CROSS REFERENCE INFORMATION TO THE
C        PAPER TAPE PUNCH.
C
C
  175 CALL LPLIST(KNT,ELM)
      CALL CREF(KNT)
  800 CONTINUE
      STOP
      END
```

```
      FUNCTION AVERAG(N)
C
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                             C
C       C        AVERAG CALCULATES THE MEAN OF N      C
C       C   NUMBERS AND WILL ELIMINATE ANY NUMBERS    C
C       C   GREATER THAN 1999 (THE LARGEST POSSIBLE   C
C       C   NUMBER FROM THE JARREL ASH 820 SPECTRO-   C
C       C   PHOTOMETER.)                              C
C       C                                             C
C       C                                             C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      DIMENSION X(125),DUM(10)
      COMMON DUM,X
      SUM=COUNT=0
      DO 1 I=1,N
      IF(X(I).GE.2000.)GO TO 1
      COUNT=COUNT+1.
      SUM=SUM+X(I)
    1 CONTINUE
      AVERAG=SUM/COUNT
      RETURN
      END
```

```
      SUBROUTINE CREF(KNT)
C
C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C        C                                           C
C        C                                           C
C        C        THIS SUBROUTINE WRITES THE CROSS REF-    C
C        C    ERENCE INFORMATION ON THE PAPER PUNCH.      C
C        C                                           C
C        C                                           C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      INTEGER RUN
      DIMENSION DUM(10),DX(125),SNM(120),ASN(40),SSC(20)
     1,RUN(20)
C
      COMMON DUM,DX,DQ,NRD,SNM,ASN,SSC,RUN
C
      N=M=0
      DO 10 I=1,KNT
      NN=N+1
      N=NN+5
      MM=M+1
      M=MM+1
      IF(RUN(I).GT.1)GO TO 10
      WRITE(7,900)(SNM(J),J=NN,N),SSC(I),(ASN(J),J=MM,M)
  900 FORMAT(1H ,A2,4A5,A1,10X,A3,17X,A2,A4)
   10 CONTINUE
      RETURN
      END
```

```
        .TITLE EDITOR
/
/
/       ////////////////////////////////////////////////////////////////
/       /                                                              /
/       /                                                              /
/       /         SUBROUTINE EDITOR WAS WRITTEN TO TRANSFER            /
/       /     DATA FROM PAPERTAPE TO DEKTAPE IN A FORM WHICH           /
/       /     COULD BE USED BY AAS.                                    /
/       /         IN THE PROCESS OF TRANSFERRING THE DATA              /
/       /     EDITOR WILL DELETE INDIVIDUAL CHARACTERS OR              /
/       /     ENTIRE LINES, REMOVE SPACES FROM THE DATA                /
/       /     RECIEVED FROM THE BCD-ASCII CONVERTER, AND               /
/       /     OUTPUT SPECIAL CODES WHICH WILL CONTROL AAS.             /
/       /                                                              /
/       /         THE SPECIAL CHARACTERS USED BY EDITOR ARE:           /
/       /                                                              /
/       /              \       DELETE THE PREVIOUS CHARACTER           /
/       /              @       DELETE THE ENTIRE LINE                  /
/       /              #       DENOTES THE BEGINNING AND END           /
/       /                          OF DATA FROM THE BCD-ASCII          /
/       /                          CONVERTER                           /
/       /              &       DENOTES THE END OF DATA FOR             /
/       /                          THE PRESENT ELEMENT (USE            /
/       /                          IN PLACE OF THE #)                  /
/       /              $       DENOTES THE END OF THE TAPE             /
/       /                                                              /
/       /                                                              /
/       /         EDITOR WILL IGNORE CARRIAGE RETURNS AND              /
/       /     BACKSLASHS (\) AT THE BEGINNING OF LINES, RUBOUTS,       /
/       /     NULLS, AND LINEFEEDS.                                    /
/       /                                                              /
/       /                                                              /
/       ////////////////////////////////////////////////////////////////
/
/
        .GLOBL EDITOR
EDITOR  0
PTR=6
DTP=1
TT3O=4
TT3I=5
IN=0
OUT=1
IOPS=2
IMAGE=3
EOF=04
        .IODEV PTR,DTP,TT3O,TT3I
START   .INIT PTR,IN,RESTRT
        .INIT DTP,OUT,RESTRT
        .INIT TT3O,OUT,RESTRT
        .INIT TT3I,IN,RESTRT
        .ENTER DTP,NAME /WRITE FILENAME ON DECTAPE
        DZM REPEAT#
        DZM CFLAG#
/
/
```

```
/        FILL THE BUFFERS CODE, ELEM, AND DONE WITH HEADER WORDS
/        AND THE PROPER CODES:       CODE=9999
/                                    ELEM=8888
/                                    DONE=DONE
/
/
         LAC (2002          /FIRST HEADER WORD
         DAC* CODEPT
         DAC* ITRPT
         DAC* ELEMPT
         DAC* DONEPT
         ISZ ELEMPT
         ISZ DONEPT
         ISZ ITRPT
         ISZ CODEPT
         DZM* CODEPT        /SECOND HEADER WORD
         DZM* ITRPT
         DZM* ELEMPT
         DZM* DONEPT
         ISZ CODEPT
         LAC (345627
         DAC* CODEPT
         ISZ CODEPT
         LAC (134432
         DAC* CODEPT
         ISZ ELEMPT
         LAC (341607
         DAC* ELEMPT
         ISZ ELEMPT
         LAC (34032
         DAC* ELEMPT
         ISZ DONEPT
         LAC (422371
         DAC* DONEPT
         ISZ DONEPT
         LAC (642432
         DAC* DONEPT
         ISZ ITRPT
         ISZ ITRPT
         JMS STROVR
/
/
/        READ FROM THE PAPER TAPE
/
/
READR    .READ PTR,IMAGE,RDBUFF,52
         .WAIT PTR
/
/
/        GET THE WORD PAIR COUNT, MULTIPLY BY TWO AND SETUP THE
/        READ COUNTER (RDCNTR).
/
/
         LAC RDBUFF
         SWHA
         AND (377
         RCL
         TAD (-1
```

```
        TCA
        DAC RDCNTR#
        LAC RDTOP
        DAC RDPTER
        ISZ RDPTER#
/
/
/       CHECK READ BUFFER TO SEE IF IT IS EMPTY.
/
/
NEXT    ISZ RDPTER
        ISZ RDCNTR
        JMP LOAD
        JMP READR
/
/
/       GET A CHARACTER FROM THE READ BUFFER AND CHECK IT.
/
/
LOAD    LAC* RDPTER
        AND (177
        DAC CHECK#
        SAD (43             /IS THE CHARACTER A #?
        JMP CKCODE          /YES
        DZM REPEAT
        LAC CFLAG           /REMOVES SPACE WHEN
        SNA                 /CFLAG IS SET
        JMP EDIT
        LAC CHECK
        SAD (40             /IS THE CHARACTER A SPACE?
        JMP NEXT            /YES
EDIT    LAC CHECK
        SAD (46             /IS THE CHARACTER A &?
        JMP NELM            /YES
        DZM REPT#
        SAD (100            /IS THE CHARACTER AN @?
        JMP BADLN           /YES
        SAD (15             /IS THE CHARACTER A [CR]?
        JMP CKSPOT          /YES
        SAD (12             /IS THE CHARACTER A [LF]?
        JMP NEXT            /YES
        SAD (177            /IS THE CHARACTER A RUBOUT?
        JMP NEXT            /YES
        SAD (44             /IS THE CHARACTER A $?
        JMP FINISH          /YES
        SAD (134            /IS THE CHARACTER A \?
        JMP CKSPOT          /YES
        SAD (0              /IS THE CHARACTER A NULL?
        JMP NEXT            /YES
/
/
/       LOCATE THE POSITION WHERE THE NEXT WORD IS TO BE PACKED.
/
/
PACKR   LAC PACKPT
        SNA
        JMP ZERO
        SAD (1
```

```
        JMP ONE
        SAD <2
        JMP TWO
        SAD <3
        JMP THREE
/
/
/       PACK IN LOCATION 4
/
/
        LAC CHECK           /PACK IN LOCATION 4
        RAL
        AND <376
        TAD PACK
        DAC* BUFFPT         /DEPOSIT WORD IN STORAGE BUFFER
        ISZ WDPRCT#
        ISZ BUFFPT#
        DZM PACKPT          /RESET THE PACKING POINTER
        ISZ ITRCNT
        LAC WRIT
        SZA
        JMP ENDLN
        JMP NEXT
/
/
/       PACK IN LOCATION 0
/
/
ZERO    LAC CHECK
        SWHA
        RTL
        AND <774000
        DAC PACK#
        ISZ PACKPT
        LAC WRIT
        SZA
        JMP ENDLN1
        JMP NEXT
/
/
/       PACK IN LOCATION 1
/
/
ONE     LAC CHECK
        RTL
        RTL
        AND <3760
        TAD PACK
        DAC PACK
        ISZ PACKPT
        LAC WRIT
        SZA
        JMP ENDLN1
        JMP NEXT
/
/
/       PACK IN LOCATION 2
/
```

```
/
TWO     LAC CHECK
        RTR
        RAR
        AND <17
        TAD PACK
        DAC* BUFFPT
        ISZ BUFFPT
        LAC CHECK
        RTR
        RTR
        AND <700000
        DAC PACK
        ISZ PACKPT
        LAC WRIT
        SZA
        JMP ENDLN2
        JMP NEXT
/
/
/       PACK IN LOCATION 3
/
/
THREE   LAC CHECK
        SWHA
        RAR
        AND <77400
        TAD PACK
        DAC PACK
        ISZ PACKPT
        LAC WRIT
        SZA
        JMP ENDLN2
        JMP NEXT
/
/
/       FINDS THE LOCATION OF THE CHARACTER TO BE ERASED
/       WHEN A \ IS ENCOUNTERED.
/
/
ERASE   LAC PACKPT
        SNA
        JMP PACPT0
        SAD <1
        JMP PACPT1
        SAD <2
        JMP PACPT2
        SAD <3
        JMP PACPT3
/
/
/       ERASE CHARACTER IN LOCATION 3
/
/
        LAC <3
        DAC PACKPT
        LAC PACK
        AND <700000
```

```
         DAC PACK
         JMP NEXT
/
/
/        ERASE CHARACTER IN LOCATION 4
/
/
PACPT0   LAC <4
         DAC PACKPT
         LAC BUFFPT
         TAD <-1
         DAC BUFFPT
         LAC WDPRCT
         TAD <-1
         DAC WDPRCT
         JMP NEXT
/
/
/        ERASE CHARACTER IN LOCATION 0
/
/
PACPT1   DZM PACKPT
         JMP NEXT
/
/
/        ERASE CHARACTER IN LOCATION 1
/
/
PACPT2   LAC <1
         DAC PACKPT
         LAC PACK
         AND <774050
         DAC PACK
         JMP NEXT
/
/
/        ERASE CHARACTER IN LOCATION 2
/
/
PACPT3   LAC <2
         DAC PACKPT
         LAC BUFFPT
         TAD <-1
         DAC BUFFPT
         LAC* BUFFPT
         AND <777760
         DAC PACK
         JMP NEXT
/
/
/        CHECKS THE CODE CHARACTER #.  IF IT IS THE FIRST # ENCOUNTERED
/        IT WILL SET CFLAG AND REPEAT TO ONE   REPEAT WILL CAUSE ALL
/        SUCCESSIVE #'S TO BE IGNORED UNTIL ANOTHER CHARACTER IS ENCOUNTERED.
/        THE SECOND SET OF #'S TO BE ENCOUNTERED WILL CAUSE 9999 TO BE
/        WRITTEN, CFLAG WILL BE CLEARED, AND REPEAT WILL BE
/        RESET UNTIL ANOTHER CHARACTER IS ENCOUNTERED.
/
/
```

```
CKCODE  LAC REPEAT#
        SZA
        JMP NEXT
        ISZ REPEAT
        LAC CFLAG
        SZA
        JMP CWRITE
        ISZ CFLAG
        JMP PACKR
/
/
/       CAUSES (CR) AND (\) TO BE IGNORED IF THEY ARE AT THE BEGINNING
/       OF A LINE.
/
/
CKSPOT  LAC PACKPT
        SZA
        JMP CK
        LAC TOPSTR
        AAC +2
        SAD BUFFPT
        JMP NEXT
CK      LAC CHECK
        SAD (134
        JMP ERASE
        ISZ WRIT#
        JMP PACKR
/
/
/       ERASES AN ENTIRE LINE.
/
/
BADLN   JMS STROVR
        JMP NEXT
/
/
/       INITIALIZES THE POINTERS AND COUNTERS.
/
/
STROVR  0                       /GET ADDRESS OF STORAGE
        LAC TOPSTR              /BUFFER
        DAC TOPBUF#
        AAC +2
        DAC BUFFPT
        DZM ITRCNT#
        DZM PACKPT#
        DZM WDPRCT
        ISZ WDPRCT              /ACCT FOR HDR WORD PAIR
        DZM WRIT#
        JMP* STROVR
/
/
/       COMPLETES THE WORD PAIR IF THE LAST CHARACTER OF THE LINE
/       WAS PACKED IN THE FIRST WORD OF THE PAIR.
/
/
ENDLN1  LAC PACK
        DAC* BUFFPT
```

```
        ISZ BUFFPT
        DZM* BUFFPT
        ISZ WDPRCT
        JMP ENDLN
/
/
/       COMPLETES THE SECOND WORD OF THE WORD PAIR.
/
/
ENDLN2  LAC PACK
        DAC* BUFFPT
        ISZ WDPRCT
ENDLN   JMS HDER
        LAC CFLAG
        SNA
        JMP WRITR
/
/
/       PACKS THE DATA PT.  COUNT INTO BUFFER ITRATE.
/
/
        SAD (2
        JMP BINDEC
        ISZ CFLAG
        JMP WRITR
BINDEC  LAC (32         /BIN TO DEC CONV.
        DAC PACK
        LAC ITRCNT
        LMQ
        CLA
        CLL
        JMS DIVIDE
        SWHA
        RAR
        AND (77400
        TAD PACK
        DAC PACK
        CLA
        CLL
        JMS DIVIDE
        DAC TEMP#
        RTR
        RTR
        AND (700000
        TAD PACK
        DAC* ITRPT
        LAC ITRPT
        TAD (-1
        DAC ITRPT
        LAC TEMP
        RTR
        RAR
        AND (17
        DAC PACK
        CLA
        CLL
        JMS DIVIDE
        RTL
```

```
        RTL
        AND (3760
        TAD PACK
        DAC PACK
        CLA
        CLL
        JMS DIVIDE
        SWHA
        RTL
        AND (774000
        TAD PACK
        DAC* ITRPT
        ISZ ITRPT
        JMP IWRITR
/
/
/       DIVIDES A BINARY NUMBER BY OCTAL 12 (BINARY 1010) TO GET
/       THE DECIMAL EQUIVALENT.
/
/
DIVIDE  0
        DIV
        12
        AND (17
        TAD (260
        JMP* DIVIDE
/
/
/       FORMS THE HEADER WORDS FOR THE BUFFER STORB1
/
/
HDER    0
        LAC WDPRCT
        SWHA
        AND (377000
        AAC +2
        DAC* TOPBUF
        ISZ TOPBUF
        DZM* TOPBUF
        JMP* HDER
CWRITE  .WRITE DTP,IOPS,CODE,4
        .WAIT DTP
        DZM CFLAG
        JMP NEXT
NELM    LAC REPT
        SZA
        JMP NEXT
        ISZ REPT
        .WRITE DTP,IOPS,ELEM,4
        .WAIT DTP
        DZM CFLAG
        JMP NEXT
IWRITR  .WRITE DTP,IOPS,ITRATE,4
        .WAIT DTP
WRITR   .WRITE DTP,IOPS,STORB1,254
        .WAIT DTP
        JMS STROVR
        JMP NEXT
```

```
CODEPT  CODE
ITRPT   ITRATE
ELEMPT  ELEM
DONEPT  DONE
RDTOP   RDBUFF
TOPSTR  STORB1
CODE    .BLOCK 4
ITRATE  .BLOCK 4
ELEM    .BLOCK 4
DONE    .BLOCK 4
MSG     ANSWER-MSG/2*1000
        0
        .ASCII "MORE DATA?"<15>
        0
ANSWER  .BLOCK 4
RDBUFF  .BLOCK 64
STORB1  .BLOCK 376
RESTRT  .CLOSE PTR
        .CLOSE DTP
        JMP START
NAME    .SIXBT "AASDATAED"
/
/
/       CHECKS TO SEE IF THE OPERATOR HAS FINISHED READING
/       ALL OF THE PAPER TAPES.
/
/
FINISH  .WRITE TT30,IOPS,MSG,34
        .WAIT TT30
        .READ TT3I,IOPS,ANSWER,4
        .WAIT TT3I
        LAC ANSWER+2
        AND (777760
        SAD (472360        /IS THE ANSWER NO?
        JMP FINI           /YES
        LAC ANSWER+2
        AND (777777
        SAD (546132        /IS THE ANSWER YES?
        JMP WD2            /POSSIBLY
        JMP FINISH         /NO
WD2     LAC ANSWER+3
        AND (790000
        SAD (300000        /IS THE ANSWER YES?
        JMP READR          /YES
        JMP FINISH         /NO
FINI    .WRITE DTP,IOPS,DONE,4
        .WAIT DTP
        .MTAPE DTP,EOF
        .CLOSE DTP
        .CLOSE PTR
        .CLOSE TT30
        .CLOSE TT3I
        JMP* EDITOR
        .END
```

```
      SUBROUTINE ELMSRT(ELEM,SRT,NOEL,NOLIST)
C
C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C        C                                         C
C        C                                         C
C        C          THIS SUBROUTINE MATCHES THE ELEMENT    C
C        C     NAME TO THE ATOMIC NUMBER AND CALLS SUB-    C
C        C     ROUTINE STAND TO READ THE STANDARD VALUES   C
C        C     FROM A FILE.  ELMSRT ALSO ASSIGNS EACH      C
C        C     ELEMENT A UNIQUE NUMBER (SRT) SO THAT IT    C
C        C     CAN BE RELOCATED.                           C
C        C                                         C
C        C                                         C
C        C          ELEM     ELEMENT NAME(ABREV)           C
C        C          SRT      NUMBER ASSIGNED TO EACH       C
C        C                   ELEMENT                       C
C        C          NOEL     ATOMIC NUMBER                 C
C        C                                         C
C        C                                         C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
C
      INTEGER SRT
      REAL K,KSTD,MG,MGSTD,MN,MNSTD,NA,NASTD,NI,NISTD
C
      DIMENSION STD(10),ALSTD(2),BASTD(2),CASTD(2),COSTD(2),
     1CUSTD(2),FESTD(2),HGSTD(2),KSTD(2),MGSTD(2),MNSTD(2),
     2NASTD(2),NISTD(2),SISTD(2),TISTD(2),ZNSTD(2),PBSTD(2),
     3RNDM(2),DX(125)
C
      COMMON STD,DX,QMK,NRD
C
      DATA AL/2HAL/,BA/2HBA/,CA/2HCA/,CO/2HCO/,CU/2HCU/,
     1K/2HK /,FE/2HFE/,HG/2HHG/,MG/2HMG/,MN/2HMN/,NA/2HNA/,
     2NI/2HNI/,PB/2HPB/,SI/2HSI/,TI/2HTI/,ZN/2HZN/,
     3ALSTD(1),ALSTD(2)/5HALSTD,4HFILE/,BASTD(1),BASTD(2)/
     45HBASTD,4HFILE/,CASTD(1),CASTD(2)/5HCASTD,4HFILE/
      DATA COSTD(1),COSTD(2)/5HCOSTD,4HFILE/,CUSTD(1),CUSTD(2)/
     15HCUSTD,4HFILE/,FESTD(1),FESTD(2)/5HFESTD,4HFILE/,
     2HGSTD(1),HGSTD(2)/5HHGSTD,4HFILE/,MGSTD(1),MGSTD(2)/
     35HMGSTD,4HFILE/,MNSTD(1),MNSTD(2)/5HMNSTD,4HFILE/,
     4NASTD(1),NASTD(2)/5HNASTD,4HFILE/,NISTD(1),NISTD(2)/
     55HNISTD,4HFILE/,PBSTD(1),PBSTD(2)/5HPBSTD,4HFILE/
      DATA SISTD(1),SISTD(2)/5HSISTD,4HFILE/,TISTD(1),TISTD(2)/
     15HTISTD,4HFILE/,ZNSTD(1),ZNSTD(2)/5HZNSTD,4HFILE/,
     2KSTD(1),KSTD(2)/5HK STD,4HFILE/,RNDM(1),RNDM(2)/5HRNDMF,4HFILE/,
     3YES/3HYES/
C
      CALL ERASE
    1 WRITE(4,900)ELEM,QMK
  900 FORMAT(1H ,3HIS ,A2,25H THE CORRECT ELEMENT NAME,A1)
      CALL YESNO(ANS)
      IF(ANS.EQ.YES)GO TO 3
      WRITE(4,910)QMK
  910 FORMAT(1H ,24HWHAT IS THE CORRECT NAME,A1)
```

```
      READ(5,915)ELEM
  915 FORMAT(A2)
    3 IF(ELEM.NE.AL)GO TO 5
      CALL STAND(ALSTD,ELEM)
      SRT=1
      NOEL=13
      RETURN
    5 IF(ELEM.NE.BA)GO TO 10
      CALL STAND(BASTD,ELEM)
      SRT=2
      NOEL=56
      RETURN
   10 IF(ELEM.NE.CA)GO TO 15
      CALL STAND(CASTD,ELEM)
      SRT=3
      NOEL=20
      RETURN
   15 IF(ELEM.NE.CO)GO TO 20
      CALL STAND(COSTD,ELEM)
      SRT=4
      NOEL=27
      RETURN
   20 IF(ELEM.NE.CU)GO TO 25
      CALL STAND(CUSTD,ELEM)
      SRT=5
      NOEL=29
      RETURN
   25 IF(ELEM.NE.FE)GO TO 30
      CALL STAND(FESTD,ELEM)
      SRT=6
      NOEL=26
      RETURN
   30 IF(ELEM.NE.HG)GO TO 35
      CALL STAND(HGSTD,ELEM)
      SRT=7
      NOEL=80
      RETURN
   35 IF(ELEM.NE.K)GO TO 40
      CALL STAND(KSTD,ELEM)
      SRT=8
      NOEL=19
      RETURN
   40 IF(ELEM.NE.MG)GO TO 45
      CALL STAND(MGSTD,ELEM)
      SRT=9
      NOEL=12
      RETURN
   45 IF(ELEM.NE.MN)GO TO 50
      CALL STAND(MNSTD,ELEM)
      SRT=10
      NOEL=25
      RETURN
   50 IF(ELEM.NE.NA)GO TO 55
      CALL STAND(NASTD,ELEM)
      SRT=11
      NOEL=11
      RETURN
   55 IF(ELEM.NE.NI)GO TO 60
```

```
      CALL STAND(NISTD,ELEM)
      SRT=12
      NOEL=28
      RETURN
   60 IF(ELEM.NE.PB)GO TO 65
      CALL STAND(PBSTD,ELEM)
      SRT=13
      NOEL=82
      RETURN
   65 IF(ELEM.NE.SI)GO TO 70
      CALL STAND(SISTD,ELEM)
      SRT=14
      NOEL=14
      RETURN
   70 IF(ELEM.NE.TI)GO TO 75
      CALL STAND(TISTD,ELEM)
      SRT=15
      NOEL=22
      RETURN
   75 IF(ELEM.NE.ZN)GO TO 80
      CALL STAND(ZNSTD,ELEM)
      SRT=16
      NOEL=30
      RETURN
   80 WRITE(4,950)ELEM
  950 FORMAT(1H ,A2,33H IS NOT ON THE PROGRAM FILE LIST./
     11H ,38HIT WILL BE ASSIGNED THE ATOMIC NO. 00.)
      DO 500 IX=1,50000
      CONTINUE
  500 CONTINUE
      NOLIST=NOLIST+1
      IF(NOLIST.GT.1)GO TO 200
      CALL STAND(RNDM,ELEM)
      SRT=17
      NOEL=0
      RETURN
  200 IF(NOLIST.GT.2)GO TO 210
      CALL STAND(RNDM,ELEM)
      SRT=18
      NOEL=0
      RETURN
  210 IF(NOLIST.GT.3)GO TO 220
      CALL STAND(RNDM,ELEM)
      SRT=19
      NOEL=0
      RETURN
  220 IF(NOLIST.GT.4)GO TO 230
      CALL STAND(RNDM,ELEM)
      SRT=20
      NOEL=0
      RETURN
  230 WRITE(4,955)
  955 FORMAT(1H ,40HYOU HAVE EXCEEDED THE LIMITS SET BY THIS/
     11H ,43HPROGRAM.   THE RESULTS OF YOUR LAST ELEMENT /
     21H ,43HNOT FOUND ON THE PROGRAM LIST WILL NOT APPEAR ON /
     31H ,46HTHE LINEPRINTER OUTPUT, BUT ON PAPERTAPE ONLY.)
      DO 501 IX=1,50000
      CONTINUE
```

```
501 CONTINUE
    CALL STAND(RNMD,ELEM)
    SRT=20
    NOEL=0
    RETURN
    END
```

```
         .TITLE ERASE
/
/
/        ////////////////////////////////////////////////////////////
/        /                                                          /
/        /                                                          /
/        /       SUBROUTINE ERASE WILL ERASE THE                    /
/        /    TEKTERMINAL SCREEN, DELAY FOR .5+                     /
/        /    SECONDS, THEN HOME THE POINTER.                       /
/        /                                                          /
/        /                                                          /
/        ////////////////////////////////////////////////////////////
/
/
         .GLOBL ERASE
ERASE    0
         .IODEV 4
START    .INIT 4,1,RESTRT
         .WRITE 4,2,CLEAR,4
         .WAIT 4
         LAC (501500
         DAC WASTE#        /WASTE .5+ SECONDS
         ISZ WASTE
         JMP .-1
         .WRITE 4,2,HOME,4
         .WAIT 4
         .CLOSE 4
         JMP* ERASE
CLEAR    HOME-CLEAR/2*1000
         0
         .ASCII <30>
         .ASCII <15>
HOME     RESTRT-HOME/2*1000
         0
         .ASCII <1>
         .ASCII <15>
RESTRT   .CLOSE 4
         JMP START
         .END
```

```
      SUBROUTINE ERRWRT(BT,NB,DIL,NRP,NSM,AVE,ELM)
C
C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C        C                                               C
C        C                                               C
C        C         THIS SUBROUTINE WRITES ALL OF THE     C
C        C    PRELIMINARY RESULTS TO THE CONTROL TTY     C
C        C    FOR THE OPERATOR TO CHECK.                 C
C        C                                               C
C        C                                               C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      REAL NB
      DIMENSION BT(9),NB(9),DIL(9),AVE(72)
C
      DATA S/1HS/
C
      WRITE(4,900)ELM
  900 FORMAT(1H ,A2//)
      WRITE(4,910)
  910 FORMAT(1H0,16HIND BTL DILUTION,4X,2HR1,4X,2HR2,4X,
     12HR3,4X,2HR4,4X,2HR5,4X,2HR6,4X,2HR7,4X,2HR8)
      DO 10 I=1,NSM
      IF(BT(I).EQ.S)GO TO 5
      WRITE(4,920)I,BT(I),NB(I),DIL(I),(AVE(J),J=I,NRP,NSM)
  920 FORMAT(1H ,I2,2X,A1,A2,1X,F9.2,1X,8F6.0)
      GO TO 10
    5 WRITE(4,930)I,BT(I),NB(I),(AVE(J),J=I,NRP,NSM)
  930 FORMAT(1H ,I2,2X,A1,A2,11X,8F6.0)
   10 CONTINUE
      RETURN
      END
```

```
      SUBROUTINE LPLIST(KNT,ELM)
C
C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C        C                                               C
C        C                                               C
C        C        THIS SUBROUTINE LISTS ALL OF THE FINAL C
C        C    RESULTS ON THE LINEPRINTER.                C
C        C                                               C
C        C                                               C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      INTEGER RUN
      REAL NO
      DIMENSION DUM(10),DX(125),SNM(120),ASN(40),
     1SSC(20),WGT(20),CON(20,20),ERR(20,20),PC(20,20),
     2RUN(20),ELM(20)
C
      COMMON DUM,DX,QMK,NED,SNM,ASN,SSC,RUN,WGT,CON,ERR,PC
C
      DATA NO/3HNO /
C
    3 WRITE(4,800)QMK
  800 FORMAT(1H ,33HHOW MANY LP COPIES DO YOU REQUIRE,A1)
      READ(5,810)NCP
  810 FORMAT(I1)
      IF(NCP.EQ.0)GO TO 30
      DO 20 NT=1,NCP
      M=MM=0
      DO 10 I=1,KNT
      N=M+1
      M=N+5
      WRITE(3,910)(SNM(J),J=N,M)
  910 FORMAT(1H1////1H ,5X,A2,5A5)
      NN=MM+1
      MM=NN+1
      WRITE(3,920)(ASN(J),J=NN,MM),SSC(I),WGT(I)
  920 FORMAT(1H0,7X,A2,A4,5X,A3,5X,F7.2,3H MG)
      WRITE(3,930)
  930 FORMAT(////1H ,7X,7HELEMENT,8X,10HCONC (PPM),10X,5HERROR,
     16X,7H% ERROR/)
      DO 5 K=1,20
      IF(CON(K,I).EQ.0.)GO TO 5
      WRITE(3,940)ELM(K),CON(K,I),ERR(K,I),PC(K,I)
  940 FORMAT(1H ,9X,A2,9X,F12.4,6X,F10.4,6X,F5.2)
    5 CONTINUE
   10 CONTINUE
   20 CONTINUE
   30 WRITE(4,870)QMK
  870 FORMAT(1H ,2HOK,A1)
      CALL YESNO(ANS)
      IF(ANS.EQ.NO)GO TO 3
      RETURN
      END
```

```
      SUBROUTINE MATCH(NB,K,BTLCK)
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                            C
C       C                                            C
C       C       SUBROUTINE MATCH CONVERTS THE BOTTLE C
C       C   NUMBERS READ IN A FORMAT TO AN INTEGER   C
C       C   NUMBER WHICH CAN BE USED IN AN ARRAY.    C
C       C                                            C
C       C                                            C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      REAL NB
C
      DATA ZERO/2H00/,HALF/2H.5/,ONE/2H01/,TWO/2H02/,THREE
     1/2H03/,FOUR/2H04/,FIVE/2H05/,SIX/2H06/,SEVEN/2H07/,
     2EIGHT/2H08/
C
      K=1
      IF(NB.EQ.ZERO)RETURN
      K=2
      IF(NB.EQ.HALF)RETURN
      K=3
      IF(NB.EQ.ONE)RETURN
      K=4
      IF(NB.EQ.TWO)RETURN
      K=5
      IF(NB.EQ.THREE)RETURN
      K=6
      IF(NB.EQ.FOUR)RETURN
      K=7
      IF(NB.EQ.FIVE)RETURN
      K=8
      IF(NB.EQ.SIX)RETURN
      K=9
      IF(NB.EQ.SEVEN)RETURN
      K=10
      IF(NB.EQ.EIGHT)RETURN
      CALL ERASE
      WRITE(4,900)
  900 FORMAT(1X,43HTHERE IS AN ERROR IN THE BTL NOS. , RECHECK.)
      BTLCK=1.
      RETURN
      END
```

```
      SUBROUTINE SAMPLE(KNT)
C
C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C        C                                            C
C        C                                            C
C        C         THIS SUBROUTINE READS IN THE SAMPLE   C
C        C    NAME, ASCESSION NO., SUBSAMPLE TYPING    C
C        C    CODE, WEIGHT, AND THE NO. OF TIMES THE   C
C        C    SAMPLE HAS BEEN RUN AND THEN ALLOWS THE  C
C        C    OPERATOR TO MAKE CORRECTIONS.            C
C        C                                            C
C        C                                            C
C        CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      INTEGER RUN
      REAL NME
C
      DIMENSION SNM(120),ASN(40),SSC(20),WGT(20),RUN(20),
     1STD(10),X(125)
C
      COMMON STD,X,QMK,NRD,SNM,ASN,SSC,RUN,WGT
C
      DATA AST/2H**/,YES/3HYES/,NME/5HNAME /,GNO/5HGEONO/,
     1FRC/5HFRAC /,WT/5HWGT  /,RN/5HRUN  /
      M=MM=KNT=0
      DO 20 I=1,20
      N=M+1
      M=N+5
      READ(1,902)(SNM(J),J=N,M)
  902 FORMAT(A2,5A5)
      IF(SNM(N).EQ.AST)GO TO 24
      NN=MM+1
      MM=NN+1
      READ(1,905)(ASN(J),J=NN,MM),SSC(I),WGT(I)
  905 FORMAT(A2,A4,1X,A3,1X,F10.2)
      READ(1,906)RUN(I)
  906 FORMAT(I2)
C
C
C        WRITES OUT THE INFORMATION JUST READ IN AND ASKS THE OPERATOR
C        TO CHECK IT.
C
C
    4 CALL ERASE
      WRITE(4,907)(SNM(J),J=N,M),(ASN(J),J=NN,MM),SSC(I),WGT(I),RUN(I)
  907 FORMAT(1H ,6HNAME= ,A2,5A5/1H ,7HGEONO= ,A2,A4,5X,6HFRAC= ,A3,
     15X,5HWGT= ,F7.2,5X,5HRUN= ,I2)
      WRITE(4,908)QMK
  908 FORMAT(//1H ,2HOK,A1)
      CALL YESNO(ANS)
      IF(ANS.EQ.YES)GO TO 20
C
C
C        THE PROGRAM NOW ALLOWS FOR THE CORRECTION OF ANY ERRORS FOUND.
C
```

```
C
    6 WRITE(4,910)QMK
  910 FORMAT(1H ,28HWHICH IDENTIFIER IS IN ERROR,A1)
   12 READ(5,912)CRT
  912 FORMAT(A5)
      IF(CRT.EQ.NME.OR.CRT.EQ.GNO.OR.CRT.EQ.FRC.OR.
     1CRT.EQ.WT.OR.CRT.EQ.RN)GO TO 14
      WRITE(4,915)QMK
  915 FORMAT(1H ,4HWHAT,A1)
      GO TO 12
   14 WRITE(4,917)CRT,QMK
  917 FORMAT(1H ,20HWHAT IS THE CORRECT ,A5,A1)
      IF(CRT.EQ.NME)READ(5,902)(SNM(J),J=N,M)
      IF(CRT.EQ.GNO)READ(5,905)(ASN(J),J=NN,MM)
      IF(CRT.EQ.FRC)READ(5,922)SSC(I)
      IF(CRT.EQ.WT)READ(5,923)WGT(I)
      IF(CRT.EQ.RN)READ(5,906)RUN(I)
  922 FORMAT(A3)
  923 FORMAT(F10.2)
      WRITE(4,908)QMK
      CALL YESNO(ANS)
      IF(ANS.EQ.YES)GO TO 4
      GO TO 6
   20 KNT=KNT+1
   24 CONTINUE
      RETURN
      END
```

```
      SUBROUTINE STAND(FILE,ELEM)
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                            C
C       C                                            C
C       C       SUBROUTINE STAND INPUTS THE CONCEN-  C
C       C   TRATIONS OF THE STANDARDS FROM A FILE    C
C       C   OR WILL ALLOW THE USER TO INPUT THE VALUES C
C       C   FROM THE TELETYPE IF NO FILE EXISTS      C
C       C   FOR THAT ELEMENT.                        C
C       C                                            C
C       C                                            C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      DIMENSION FILE(2),STD(10),DX(125)
      COMMON STD,DX,QMK,NRD
      DATA YES/3HYES/
      IF(NRD.EQ.1)GO TO 5
      CALL FSTAT(2,FILE,I)
      IF(I.EQ.-1)GO TO 40
    5 CALL ERASE
      WRITE(4,10)ELEM
   10 FORMAT(71H *******************************************************
     1***************/2H *,68X,1H*/2H *,8X,44HTHE STANDARD VALUES HAVE
     2 NOT BEEN FILED FOR ,A2,1H.,13X,1H*/2H *,9X,22HPLEASE ENTER THEM
     3 NOW.,38X,1H*/2H *,68X,1H*/2H *,4X,58HYOU MUST ENTER VALUES FOR
     4 TEN STANDARDS BEGINNING WITH 500,6X,1H*/2H *,7X,57HAND INCLUDING
     5 5.5.  EACH VALUE MUST CONTAIN A DECIMAL PT.,4X,1H*/2H *,7X,54HAND
     6 BE TERMINATED WITH A CARRIAGE RETURN(CR).  FOR ANY,7X,1H*/2H *,7X
     7,58HSTANDARD WITHOUT A VALUE, A CARRIAGE RETURN IS SUFFICIENT.,3X,
     81H*/2H *,68X,1H*/71H *********************************************
     9*************************)
      DO 20 I=1,10
   20 READ(5,25)STD(I)
   25 FORMAT(F6.2)
      DO 30 I=1,10
   30 WRITE(4,35)STD(I)
   35 FORMAT(1H ,F6.2)
      WRITE(4,900)QMK
  900 FORMAT(//1H ,2HOK,A1)
      CALL YESNO(ANS)
      IF(ANS.NE.YES)GO TO 5
      RETURN
   40 CALL SEEK(2,FILE)
      DO 50 I=1,10
   50 READ(2,25)STD(I)
      CALL CLOSE(2)
      RETURN
      END
```

```
      SUBROUTINE UNKNOW(NB,K,BTLCK)
C
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                               C
C       C                                               C
C       C        SUBROUTINE UNKNOW CONVERTS THE BOTTLE  C
C       C   NUMBERS READ IN A-FORMAT TO AN INTEGER      C
C       C   NUMBER.                                     C
C       C                                               C
C       C                                               C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      REAL NB,NINE
C
      DATA ONE/2H01/,TWO/2H02/,THREE/2H03/,FOUR/2H04/,
     1FIVE/2H05/,SIX/2H06/,SEVEN/2H07/,EIGHT/2H08/,
     2NINE/2H09/,TEN/2H10/,ELEVEN/2H11/,TWELVE/2H12/,
     3TEEN3/2H13/,TEEN4/2H14/,TEEN5/2H15/,TEEN6/2H16/,
     4TEEN7/2H17/,TEEN8/2H18/,TEEN9/2H19/,TWENTY/2H20/
C
      K=1
      IF(NB.EQ.ONE)RETURN
      K=2
      IF(NB.EQ.TWO)RETURN
      K=3
      IF(NB.EQ.THREE)RETURN
      K=4
      IF(NB.EQ.FOUR)RETURN
      K=5
      IF(NB.EQ.FIVE)RETURN
      K=6
      IF(NB.EQ.SIX)RETURN
      K=7
      IF(NB.EQ.SEVEN)RETURN
      K=8
      IF(NB.EQ.EIGHT)RETURN
      K=9
      IF(NB.EQ.NINE)RETURN
      K=10
      IF(NB.EQ.TEN)RETURN
      K=11
      IF(NB.EQ.ELEVEN)RETURN
      K=12
      IF(NB.EQ.TWELVE)RETURN
      K=13
      IF(NB.EQ.TEEN3)RETURN
      K=14
      IF(NB.EQ.TEEN4)RETURN
      K=15
      IF(NB.EQ.TEEN5)RETURN
      K=16
      IF(NB.EQ.TEEN6)RETURN
      K=17
      IF(NB.EQ.TEEN7)RETURN
      K=18
```

```
      IF(NB.EQ.TEEN8)RETURN
      K=19
      IF(NB.EQ.TEEN9)RETURN
      K=20
      IF(NB.EQ.TWENTY)RETURN
      CALL ERASE
      WRITE(4,900)
  900 FORMAT(1X,43HTHERE IS AN ERROR IN THE BTL NOS., RECHECK.)
      BTLCK=2.
      RETURN
      END
```

```
      FUNCTION VARIAN(N,AVE)
C
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                           C
C       C                                           C
C       C        VARIAN CALCULATES THE VARIANCE     C
C       C   OF A GROUP OF NUMBERS AND WILL ELIMINATE  C
C       C   ANY NUMBERS GREATER THEN 1999 (THE LARGEST C
C       C   POSSIBLE NUMBER FROM THE JARREL ASH 820   C
C       C   SPECTROPHOTOMETER)                      C
C       C                                           C
C       C                                           C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      DIMENSION X(125),DUM(10)
      COMMON DUM,X
      COUNT=SUM=0
      DO 1 I=1,N
      IF(X(I).GE.2000.)GO TO 1
      COUNT=COUNT+1.
      SUM=SUM+((X(I)-AVE)**2)
    1 CONTINUE
      VARIAN=SUM/(COUNT-1.)
      RETURN
      END
```

```
      SUBROUTINE YESNO(ANSWER)
C
C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C       C                                             C
C       C                                             C
C       C           SUBROUTINE YESNO WAITS FOR A YES  C
C       C     OR NO ANSWER.                           C
C       C                                             C
C       C                                             C
C       CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
C
C
      REAL NO
      DIMENSION DUM(10),DX(125)
      COMMON DUM,DX,QMK
      DATA YES/3HYES/,NO/3HNO /
    1 READ(5,910)ANSWER
  910 FORMAT(A3)
      IF(ANSWER.EQ.YES.OR.ANSWER.EQ.NO)RETURN
      WRITE(4,920)QMK
  920 FORMAT(1H ,4HWHAT,A1)
      GO TO 1
      RETURN
      END
```

A Listing of the Formatting Used on the Data Tapes for the PDP-15

```
Y74-3-58P 18-23CM
HN1422 AAA 468.41
01

Y74-3-58P 18-23CM
HN1422 AAA 445.16
02

Y74-3-58P 118-123CM
HN1423 AAA 403.45
01

Y74-3-58P 118-123CM
HN1423 AAA 396.97
02

Y74 3-58P 218-223CM
HN1424 AAA 448.93
01

Y74-3-58P 218-223CM
HN1424 AAA 427.67
02

**
$$
```

```
CU

S01
U01 200.
S02
U02 200.
#

+0194 +0194 +0197 +0198 +0194 +0195 +0199 +0200 +0191 +0194 +0190 +0188
+0189 +0190 +0188 +0189 +0191 +0187 +0190 +0189

+0305 +0348 +0346 +0345 +0341 +0340 +0340 +0342 +0339 +0345 +0340 +0336
+0338 +0334 +0340 +0338 +0336 +0335 +0339 +0339

+0387 +0389 +0387 +0387 +0387 +0390 +0385 +0385 +0383 +0384 +0383 +0385
+0382 +0383 +0382 +0381 +0380 +0382 +0383 +0382

+0238 +0331 +0328 +0327 +0328 +0326 +0325 +0328 +0325 +0327 +0329 +0326
+0325 +0320 +0320 +0321 +0325 +0328 +0326 +0326

#

S01
U03 200.
S02
U04 200.
#

+0183 +0198 +0198 +0195 +0197 +0198 +0194 +0195 +0199 +0193 +0196 +0195
+0193 +0195 +0193 +0195 +0192 +0194 +0193 +0195

+0371 +0367 +0371 +0369 +0365 +0365 +0363 +0364 +0361 +0363 +0367 +0366
+0367 +0365 +0366 +0363 +0363 +6367 +0366 +0367

+0354 +0388 +0385 +0388 +0384 +0389 +0393 +0392 +0390 +0392 +0390 +0388
+0389 +0389 +0392 +0389 +0392 +0388 +0389 +0387

+0299 +3364 +0358 +0357 +0359 +0357 +0360 +0355 +0356 +0360 +0353 +0353
+0356 +0359 +0355 +0354 +0351 +0349 +0350 +0346

#

S02
U05 200.
S03
U06 200.
#

+0401 +0399 +0404 +0401 +0397 +0406 +0399 +0403 +0398 +0400 +0393 +0402
+0394 +0399 +0399 +0402 +0392 +0398 +0397 +0402 +0395 +0399

+0589 +0605 +0608 +0606 +0603 +0602 +0602 +0601 +0601 +0604 +0603 +0605
+0601 +0604 +3598 +0597 +0598 +0590 +0593 +0591

+0592 +0601 +0593 +0598 +0596 +0585 +0582 +0575 +0581 +0571 +0580 +0578
+0581 +0575 +0581 +0574 +0575 +0582 +0574 +0578

+0584 +0589 +0592 +0587 +0588 +0591 +0593 +0593 +0594 +0588 +0587 +0592
+0590 +0588 +0588 +0591 +0595 +0594 +0592 +0595

&&
$$
```

```
FE
S02
U01 20000.
S03
U02 20000.
#

+0396 +0402 +0397 +0398 +0405 +0397 +0406 +0394 +0403 +0402 +0398 +0401
+0401 +0399 +0406 +0395 +0403 +0395 +0402 +0394 +0406 +0396 +0405 +0393

+0509 +0520 +0510 +0507 +0518 +0516 +0514 +0509 +0517 +0515 +0542 +0542
+0537 +0538 +0532 +0529 +0510 +0515 +0513 +0519 +0518

+0582 +0595 +0597 +0598 +0600 +0593 +0594 +0601 +0584 +0595 +0597 +0599
+0593 +0601 +0592 +0589 +0591 +0594 +0594 +0592 +0596

+0483 +0494 +0490 +0487 +0495 +0495 +0486 +0488 +0489 +0482 +0488 +0485
+0490 +0488 +0490 +0489 +0489 +0488 +0493 +0490 +0488 +0493 +0493

#

S02
U03 20000.
SO\03
U04 20000.
#

+0185 +0401 +0392 +0400 +0385 +0394 +0393 +0395 +0393 +0400 +0386 +0399
+0396 +0397 +0397 +0393 +0398 +0402 +0390 +0389 +0393

+0530 +0535 +0537 +0543 +0532 +0534 +0529 +0539 +0540 +0534 +0539 +0538
+0533 +0540 +0534 +0534 +0540 +0530 +0534 +0536 +0537

+0596 +0595 +0597 +0599 +0597 +0600 +0590 +0597 +0605 +0594 +0591 +0597
+0598 +0595 +0601 +0596 +0588 +0593 +0593 +0600 +0586

+0436 +0439 +0438 +0434 +0434 +0438 +0431 +0434 +0433 +0437 +0433 +0433
+0435 +0432 +0435 +0440 +0428 +0436 +0442 +0429 +0433

#

S02
U05 20000.
S03
U06 20000.
#

+0408 +0402 +0410 +0397 +0412 +0406 +0404 +0401 +0403 +0412 +0408 +0406
+0405 +0398 +0412 +0405 +0403 +0402 +0408 +0406 +0408

+0564 +0554 +0551 +0551 +0538 +0556 +0550 +0550 +0554 +0548 +0547 +0548
+0547 +0548 +0554 +0553 +0554 +0549 +0545 +0542 +0542 +0542

+0599 +0604 +0602 +0603 +0608 +0599 +0605 +0605 +0601 +0595 +0594 +0590
+0579 +0588 +0583 +0583 +0576 +0582 +0583 +0581 +0576 +0582 +0583 +0583

+0451 +0488 +0495 +0503 +0498 +0497 +0505 +0494 +0499 +0504 +0501 +0500+
+0493 +0499 +0509 +0500 +0499 +0501 +0497 +0496 +0503

&&
$$
```

```
MN

S.5
U01 20000.
S01
U02 20000.
#

+0098 +0098 +0099 +0097 +0100 +0094 +0099 +0099 +0102 +0096 +0103 +0099
+0100 +0093 +0102 +0096 +0100 +0096 +0102 +0096

+0210 +0203 +0208 +0203 +0204 +0203 +0207 +0207 +0209 +0208 +0205 +0203
+0204 +0205 +0209 +0206 +0207 +0206 +0208 +0204

+0202 +0200 +0206 +0204 +0202 +0206 +0206 +0205 +0203 +0208 +0207 +0202
+0203 +0207 +0205 +0208 +0203 +0203 +0207 +0204

+0182 +0192 +0191 +0187 +0195 +0196 +0197 +0197 +0197 +0200 +0192 +0196
+0195 +0197 +0198 +0199 +0201 +0195 +0197 +0198

#

S01
U03 20000.
S02
U04 20000.
#

+0197 +0201 +0195 +0197 +0200 +0194 +0200 +0196 +0197 +0198 +0198 +0201
+0200 +0195 +0195 +0199 +0197 +0198 +0197 +0199 +0199

+0264 +0263 +0264 +0264 +0264 +0262 +0268 +0269 +0265 +0272 +0266 +0266
+0266 +0267 +0268 +0266 +0270 +0267 +0275 +0271 +0265

+0433 +0430 +0425 +0433 +0436 +0435 +0433 +0435 +0432 +0435 +0430 +0432+
+0433 +0435 +0436 +0437 +0434 +0435 +0436 +0432 +0435

+0263 +0270 +0267 +0264 +0260 +0263 +0265 +0261 +0264 +0264 +0263 +0267
+0272 +0270 +0264 +0266 +0265 +0267 +0272 +0272 +0266

+0225 +0222 +0228 +0230 +0228 +0224 +0221 +0222 +0224 +0224 +0223 +0223
+0223 +0222 +0222 +0229 +0228 +0224 +0226 +0227 +0227 +0226 +0230 +0227+
+0224 +0220

+0273 +0275 +0271 +0270 +0271 +0271 +0268 +0270 +0271 +0268 +0276 +0275
+0275 +0272 +0273 +0275 +0275 +0278 +0270 +0272 +0276

+0439 +0446 +0440 +0436 +0445 +0440 +0437 +0443 +0443 +0441 +0438 +0447
+0444 +0438 +0443 +0444 +0439 +0443 +0445 +0442

+0280 +0277 +0285 +0273 +0272 +0264 +0268 +0270 +0267 +0277 +0275 +0275
+0276 +0273 +0272 +0269 +0271 +0270 +0274 +0278

#

S02
U05 20000.
S03
U06 20000.
#
```

+0399 +0399 +0413 +0402 +0398 +0403 +0395 +0404 +0398 +0411 +0398 +0403
+0397 +0404 +0399 +0409 +0405 +0401 +0400 +0399 +0414 +0398 +0408 +0404
+0404 +0405

+0431 +0430 +0429 +0431 +0433 +0434 +0438 +0433 +0437 +0436 +0437 +0430
+0429 +0429 +0427 +0432 +0430 +0428 +0434 +0433 +0435

+0610 +0606 +0615 +0608 +0606 +0609 +0609 +0609 +0605 +0607 +0610 +0605
+0608 +0614 +0606 +0612 +0604 +0609 +0611 +0606

+0413 +0416 +0413 +0420 +0413 +0418 +0414 +0409 +0412 +0411 +0409 +0414
+0414 +0412 +0409 +0415 +0399 +0398 +0391 +0393 +0412 +0404

&&
$$

An Example for the Operation of the PDP-15 Program

```
↑C
BKIH15 V4D
$A TT3 4,5/TT2 3
$GLOAD


BGLOAD V2A
>-AAS




DO YOU WISH TO USE THE STANDARD
CONCENTRATION VALUES ON FILE?

YES
DO YOU WISH TO READ IN PAPER TAPES?
YES


MORE DATA?
YES
MORE DATA?
YES
MORE DATA?
YES
MORE DATA?
NO




NAME= Y74-3-58P 18-23CM
GEONO= HHI422       FRAC= AAA       WGT=  468.41       RUN=  1


OK?
YES



NAME= Y74-3-58P 18-23CM
GEONO= HHI422       FRAC= AAA       WGT=  445.16       RUN=  2


OK?
YES



NAME= Y74-3-58P 118-123CM
GEONO= HHI423       FRAC= AAA       WGT=  493.45       RUN=  1


OK?
YES
```

```
NAME= Y74-3-58P 118-123CM
GEONO= HNI 423       FRAC= AAA      WGT=  396.97      RUN=  2


OK?
NO
WHICH IDENTIFIER IS IN ERROR?
FRAC
WHAT IS THE CORRECT FRAC ?
ABC


OK?
YES


NAME= Y74-3-58P 118-123CM
GEONO= HNI 423       FRAC= ABC      WGT=  396.97      RUN=  2


OK?
YES


NAME= Y74 3-58P 217-223CM
GEONO= HNI 424       FRAC= AAA      WGT=  44[illegible].93      RUN=  1


OK?
YES


NAME= Y74-3-58P 218-223CM
GEONO= HNI 424       FRAC= AAA      WGT=  427.67      RUN=  2


OK?
NO
WHICH IDENTIFIER IS IN ERROR?
WGT
WHAT IS THE CORRECT WGT  ?
428.[illegible]


OK?
YES
```

NAME= Y74-3-5SP 218-223CM
GEONO= HNI 424 FRAC= AAA UGT= 428.00 RUN= 2

OK?
YES

IS MN THE CORRECT ELEMENT NAME?
YES

MN

IND	BTL	DILUTION	R1	R2	R3	R4	R5	R6	R7	R8
1	S.5		98.							
2	U01	20000.00	206.							
3	S01		205.							
4	U02	20000.00	196.							

OK?
YES

MN

IND	BTL	DILUTION	R1	R2	R3	R4	R5	R6	R7	R8
1	S01		198.	225.						
2	U03	20000.00	266.	273.						
3	S02		434.	442.						
4	U04	20000.00	266.	273.						

OK?
NO
WHICH VARIABLE IS IN ERROR? (BTL, DILUTION, R1, R2, ETC.)
R1
WHAT IS THE IND. NO.?
1

OK?
YES

MN

IND	BTL	DILUTION	R1	R2	R3	R4	R5	R6	R7	R8

```
 1  S01                9999.  225.
 2  U03  20000.00       266.  273.
 3  S02                 434.  442.
 4  U04  20000.00       266.  273.

OK?
YES


MN

IND BTL DILUTION    R1    R2    R3    R4    R5    R6    R7    R8
 1  S02             432.
 2  U05  20000.00   432.
 3  S03             608.
 4  U06  20000.00   411.

OK?
YES


IS CU THE CORRECT ELEMENT NAME?
YES


CU

IND BTL DILUTION    R1    R2    R3    R4    R5    R6    R7    R8
 1  S01             192.
 2  U01    200.00   343.
 3  S02             384.
 4  U02    200.00   326.

OK?
YES
```

CU

IND	BTL	DILUTION	R1	R2	R3	R4	R5	R6	R7	R8
1	S01		195.							
2	U03	200.00	366.							
3	S02		389.							
4	U04	200.00	355.							

OK?
YES

CU

IND	BTL	DILUTION	R1	R2	R3	R4	R5	R6	R7	R8
1	S02		399.							
2	U05	200.00	601.							
3	S03		582.							
4	U06	200.00	591.							

OK?
YES

IS FE THE CORRECT ELEMENT NAME?
YES

FE

IND	BTL	DILUTION	R1	R2	R3	R4	R5	R6	R7	R8
1	S02		400.							
2	U01	20000.00	520.							
3	S03		595.							
4	U02	20000.00	490.							

OK?
NO
WHICH VARIABLE IS IN ERROR? (BTL, DILUTION, R1, R2, ETC.)
BTL
WHAT IS THE IND. NO.?
2
WHAT IS THE CORRECT BTL NUMBER?
U03

OK?

```
YES


FE

IND BTL DILUTION     R1     R2     R3     R4     R5     R6     R7     R8
 1  S02              470.
 2  U23  20000.00    520.
 3  S03              595.
 4  U22  20000.00    490.

OK?
YES


FE

IND BTL DILUTION     R1     R2     R3     R4     R5     R6     R7     R8
 1  S02              395.
 2  U23  20000.00    536.
 3  S03              596.
 4  U24  20000.00    434.

OK?
YES\\\NO


FE

IND BTL DILUTION     R1     R2     R3     R4     R5     R6     R7     R8
 1  S02              406.
 2  U25  20000.00    549.
 3  S03              591.
 4  U26  20000.00    499.

OK?
YES
HOW MANY LP COPIES DO YOU REQUIRE?
1
OK?
NO
HOW MANY LP COPIES DO YOU REQUIRE?
1
OK?
YES

STOP 000000
↑C
MM15 WAD
```

A Sample of Data Output to the Papertape Punch by the PDP-15 Program

```
AASU HN1422 AAA MN 25    8595.8674   324.4747  3.77 Y74-3-58P 18-23CM  1
AASU HN1422 AAA MN 25    8614.5723   332.5441  3.86 Y74-3-58P 18-23CM  2
AASU HN1423 AAA MN 25   11988.9543   198.3679  1.65 Y74-3-58P 118-123  1
AASU HN1423 ABC MN 25   12176.7864   238.2333  1.96 Y74-3-58P 118-123  2
CASU HN1424 AAA MN 25   19132.9849   241.4360  1.26 Y74 3-58P 218-223  1
AASU HN1424 AAA MN 25   19120.0415   313.1579  1.64 Y74-3-58P 218-223  2
CASU HN1422 AAA CU 29     302.3809     8.0218  2.65 Y74-3-58P 18-23CM  1
CASU HN1422 AAA CU 29     304.8522     7.6034  2.49 Y74-3-58P 18-23CM  2
CCSU HN1423 AAA CU 29     372.7051     6.5834  1.77 Y74-3-58P 118-123  1
AASU HN1423 ABC CU 29     367.9527     7.3615  2.00 Y74-3-58P 118-123  2
CASU HN1424 AAA CU 29     553.1872    28.1724  5.09 Y74 3-58P 218-223  1
AASU HN1424 AAA CU 29     570.2504    27.8523  4.88 Y74-3-58P 218-223  2
AASU HN1423 AAA FE 26   64973.4053  2002.7383  3.09 Y74-3-58P 118-123  1
AASU HN1422 AAA FE 26   55247.3203  1058.9392  1.92 Y74-3-58P 18-23CM  2
AASU HN1423 AAA FE 26   66957.5695  1711.5405  2.56 Y74-3-58P 118-123  1
AASU HN1423 ABC FE 26   55363.8643   841.4104  1.52 Y74-3-58P 118-123  2
AASU HN1424 AAA FE 26   61735.4678  3215.5471  5.21 Y74 3-58P 218-223  1
AASU HN1424 AAA FE 26   58483.8154  2273.3007  3.89 Y74-3-58P 218-223  2
Y74-3-58P 18-23CM              AAA                HN1422"
Y74-3-58P 118-123CM            AAA                HN1423
Y74 3-58P 218-223CM            AAA                HN1424
```

A Sample of Data Output to the Line Printer by the PDP-15 Program

```
Y74-3-58P 118-123CM
  HN1423      ABC        396.97 MG


  ELEMENT          CONC (PPM)           ERROR      % ERROR

    CU               367.9527          7.3615        2.00
    FE             55363.8643        841.4104        1.52
    MN             12176.7864        238.2333        1.96
```

CALCULATIONS

The algorithm used to calculate abundances is a simple linear interpolation:

$$M = 100 \left[\frac{H - (H-L)\left(\frac{Z - Y}{Z - X}\right)}{F\ G} \right]$$

where

M= % metal in sample

H= High standard value

L= Low standard value

X= Avg of AAS readouts for the low standard

Y= " " " " " sample

Z= " " " " " high standard

F= the dilution factor (1000/ dilution of sample)

G= wgt of sample

The error is propagated with the assumption that the only variation is in the digital readout of the AAS. With this in mind, the following equation was used to calculate the total error for each calculation.

$$RSD = Sx^2 \left(\frac{M}{X}\right)^2 + Sy^2 \left(\frac{M}{Y}\right)^2 + Sz^2 \left(\frac{M}{Z}\right)^2$$

where RSD = % Relative Standard Deviation

Sx^2 = the sample variance of X

Sy^2 = the sample variance of Y

Sz^2 = the sample variance of Z

and M is the linear interpolation equation above.

Sample variance is calculated using the following formula:

$$S^2 = \frac{\sum (Xi-x)}{n-1}$$

where Xi is one of the data points,
x is the mean of the sample
n is the number of data points in the sample.

Acknowledgments

The author would like to thank John Toth for his contribution in "Cold Vapor Atomic Absorption Techniques" and to Mitch Lyle for "Pb Analysis by Carbon Rod."

This work was supported by the National Science Foundation as a contribution of the International Decade of Ocean Exploration (Nazca Plate Project), Grant # IDO 71-04208.

REFERENCES

(1) Bernas, Bedrich, 1968, A new method for the decomposition and comprehensive analysis of silicates by atomic absorption spectrometry: Analytical Chemistry, v. 40, p. 1682-1686.

(2) Control Data Corporation, 1969, 3300 Computer Systems Fortran reference manual, Control Data Corporation Pub. No. 60057600, Rev. B, 116 p.

(3) Dayton, Fred and Massie, Walter, 1968, Free form input for OS-3 Fortran, Oregon State University Computer Center Pub. No. CC-68-27, 9 p.

(4) Dayton, Fred, 1970, OS-3 Editor manual, Oregon State University Computer Center Pub. No. CCM-70-7.

(5) Digital Equipment Corporation, 1974, PDP-15 Edit Utility program reference manual, Digital Equipment Corporation Pub. No. DEC-15-YWZB-DN6.

(6) Digital Equipment Corporation, 1972, PDP-15 Fortran IV programmers reference manual, Digital Equipment Corporation Pub. No. DEC-15-KFZD-D.

(7) Digital Equipment Corporation, 1972, PDP-15 MACRO-15 assembler programmers reference manual, Digital Equipment Corporation Pub. No. DEC-15-APIZB-D.

(8) Digital Equipment Corporation, 1969, PDP-15 Systems reference manual, Digital Equipment Corporation Pub. No. DEC-15-BRZA-D.

(9) Digital Equipment Corporation, 1972, PDP-15 UPDATE utility program reference manual, Digital Equipment Corporation Pub. No. DEC-15-YWZB-DN7.

(10) Fisher Scientific Company, 1972, Atomic absorption analytical methods, Fisher Scientific Company, Atomic Absorption Analytical Laboratory Publication.

(11) Fisher Scientific Company, 1971, JA82-810 instruction manul, Fisher Scientific Company, Jarrel Ash Division Engineering Pub. No. 82-810, 54 p.

(12) Krogh, T. E., , A simplified technique for the dissolution of zircons and the isolation of uranium and lead, Carnegie Institution, Annual Report of the Director, Geophysical Laboratory, 1969-1970, p. 341-344.

(13) Massie, Walter, 1968, A control mode manual for OS-3 (vers. 2.0), Oregon State University Computer Center Publ. No. CC-68-21.

(14) Parr Instrument Company, 1969, Acid digestion bomb, Parr Instrument Company Product Sheet 4745, 2 p.

(15) Parr Instrument Company, 1972, Instructions for Parr 4745 Acid Digestion Bomb, Parr Instrument Company, 4 p.

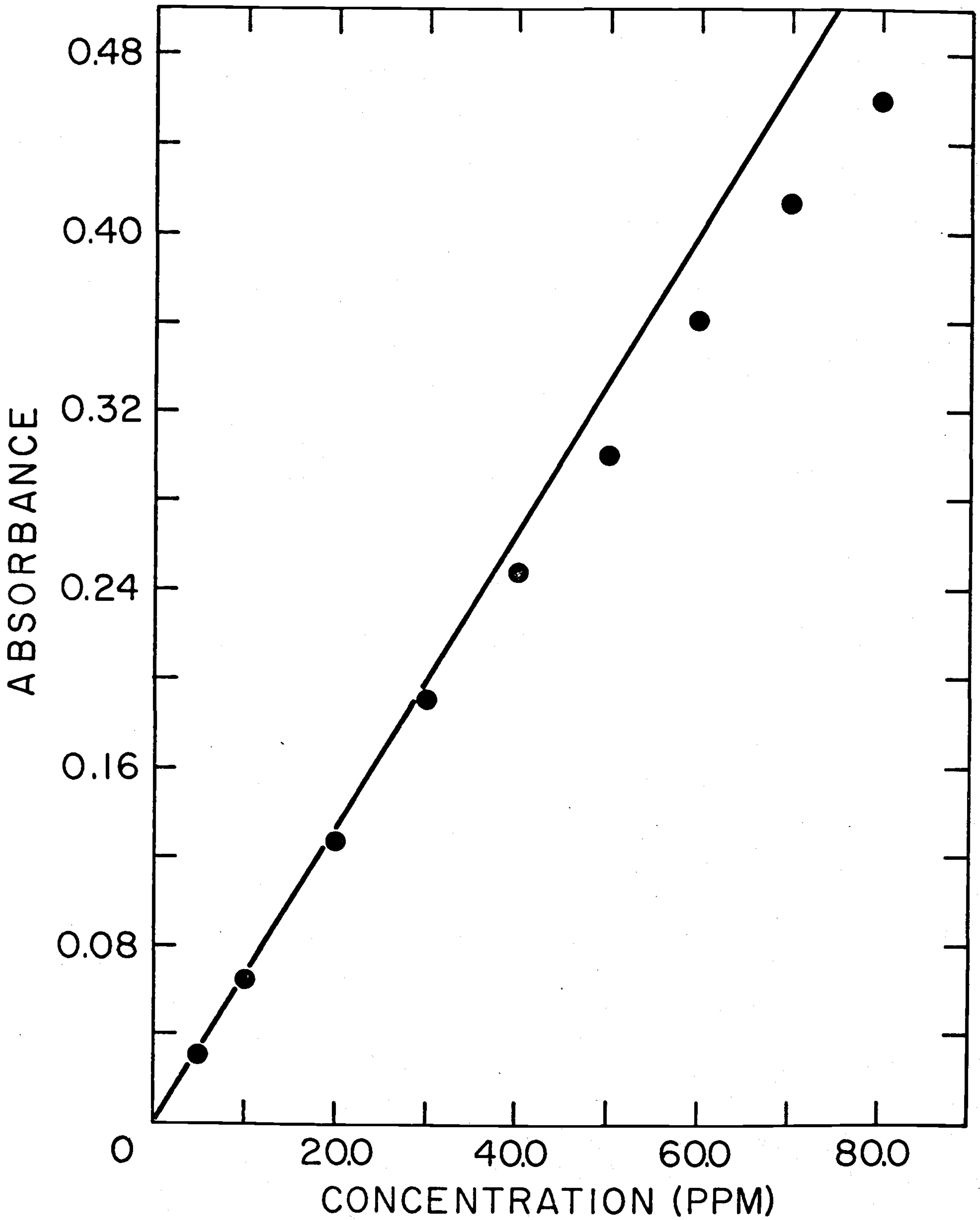
(16) Standley, David R., 1972, Digital recording and analysis of mass spectra, Master's degree thesis, Oregon State University, 84 p.

(17) Wang Laboratories, Inc., 1971, Wang Model 500 reference manual, Wang Laboratories Pub. No. 700-0352, 37 p.

APPENDIX A

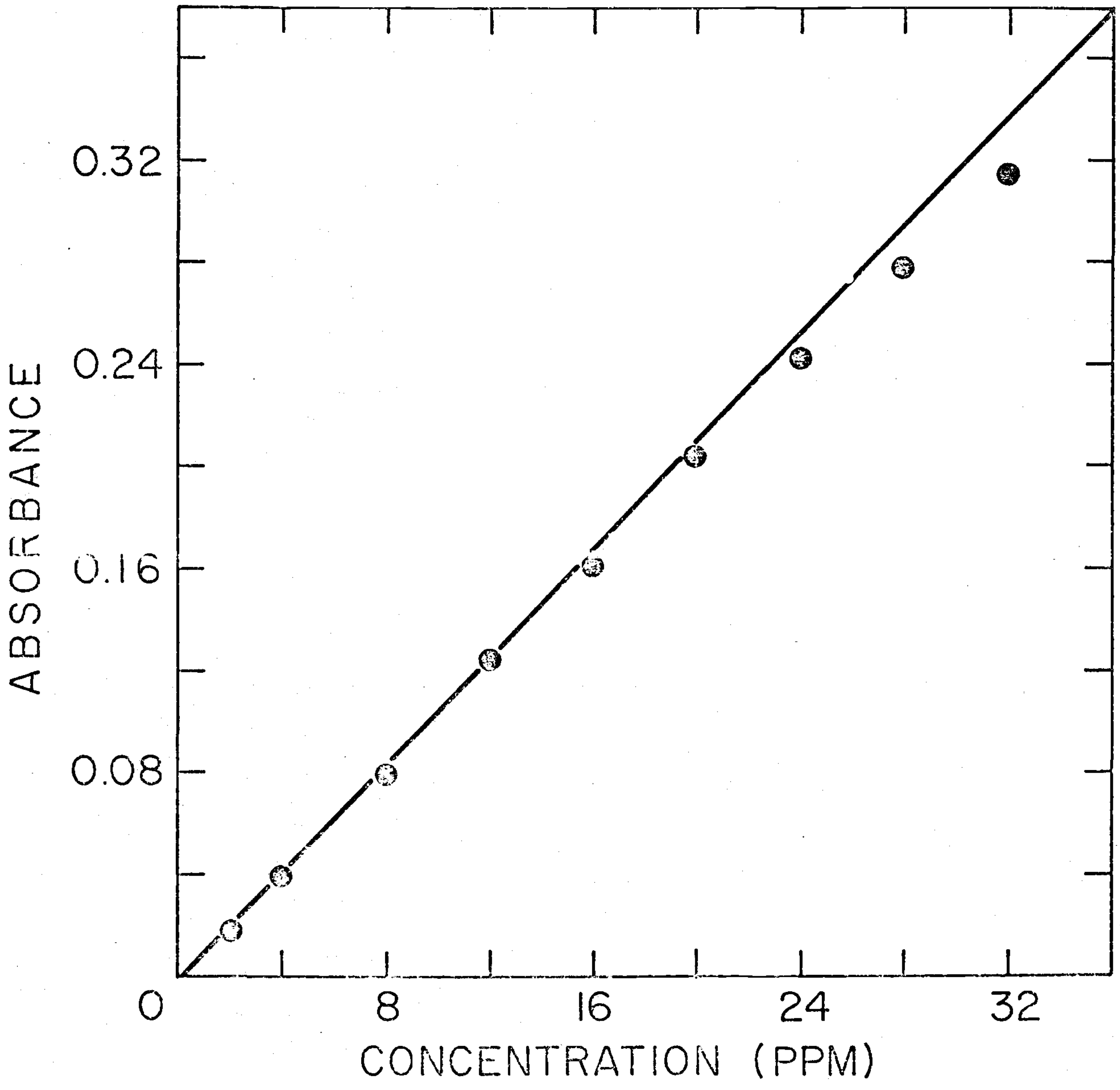
BEER'S LAW CURVES FOR THE MOST FREQUENTLY ANALYZED ELEMENTS

ALUMINUM

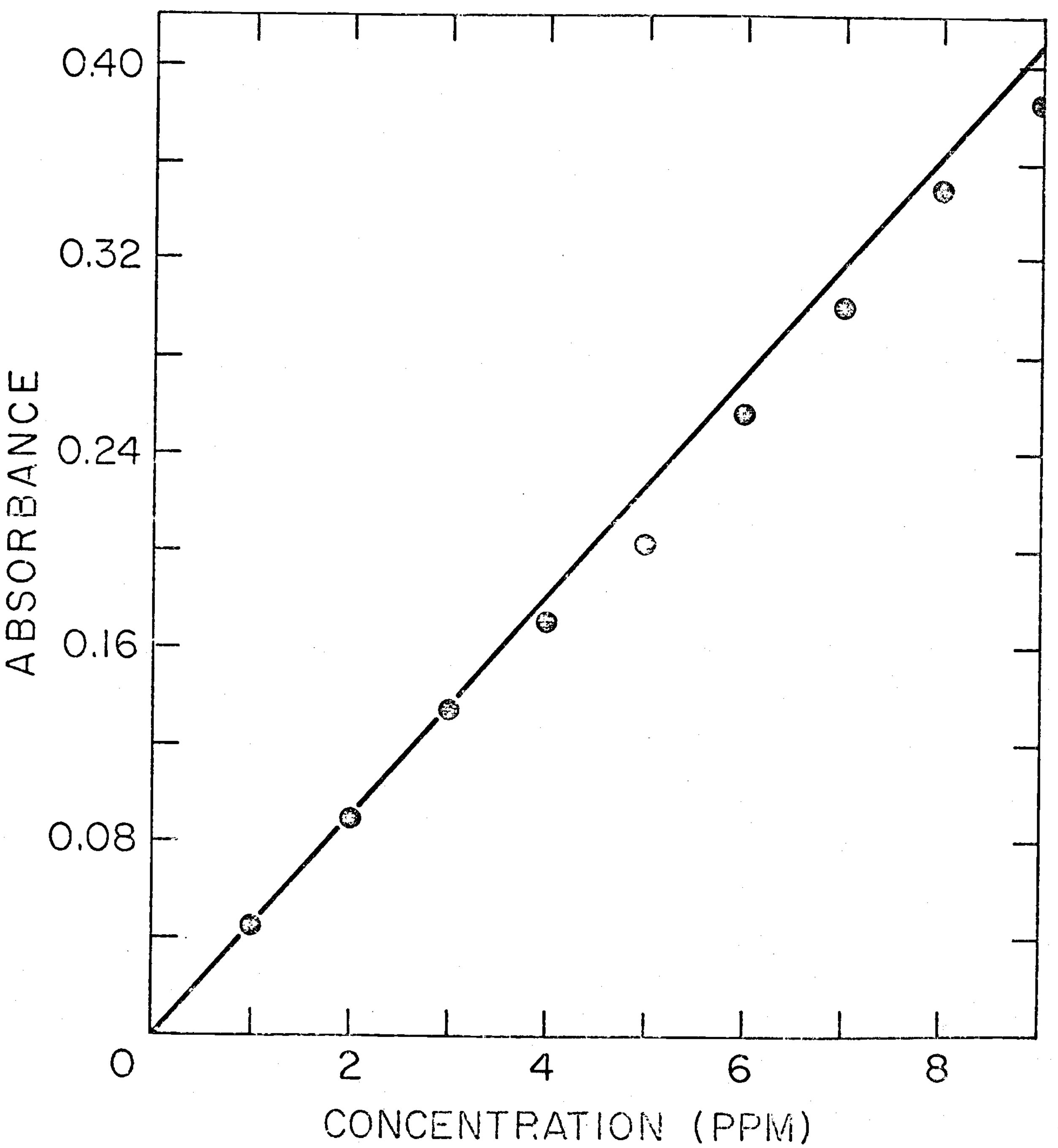


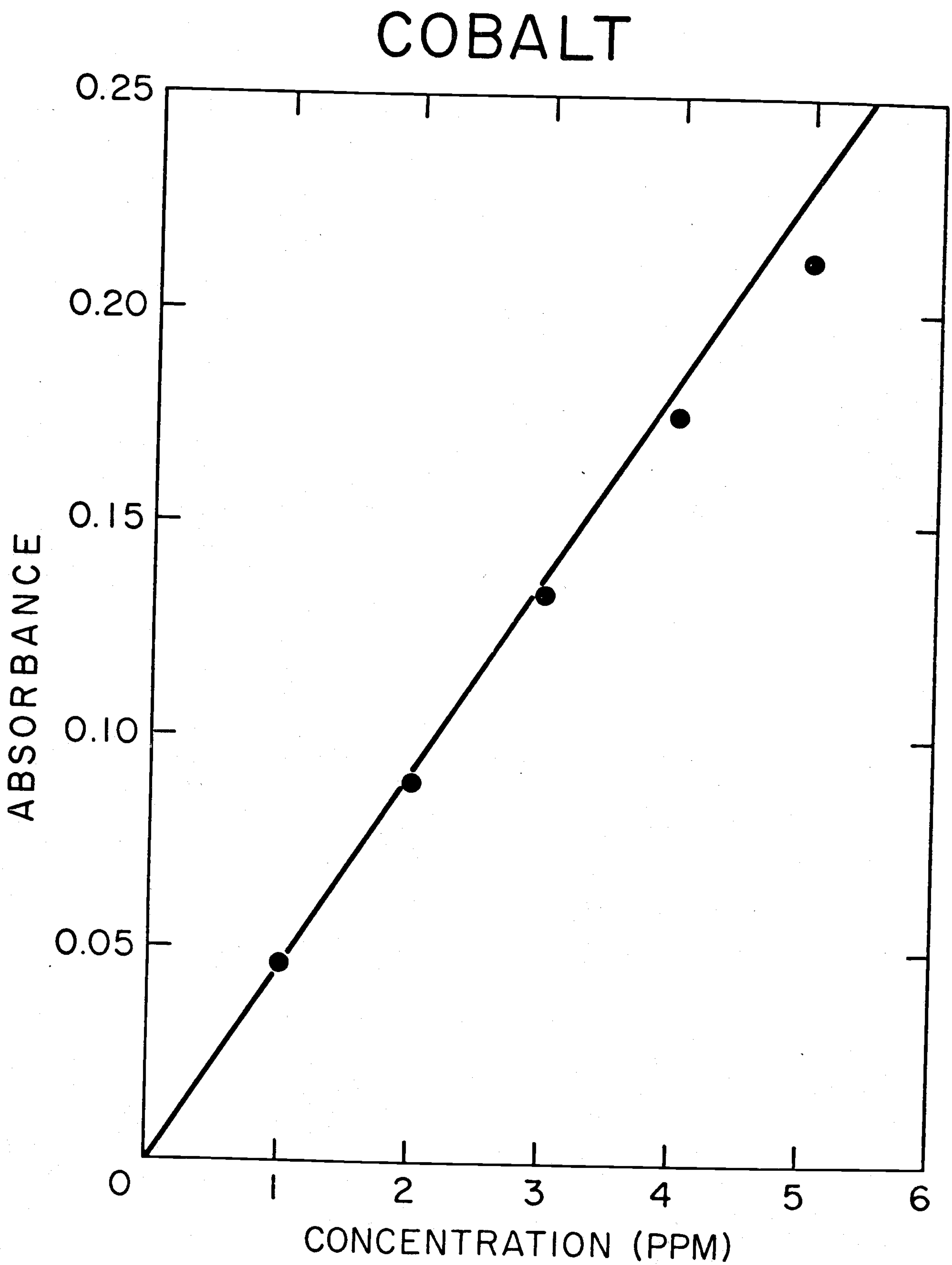
0.48
0.40
0.32
0.24
0.16
0.08
0
ABSORBANCE
20.0
40.0
60.0
80.0
CONCENTRATION (PPM)

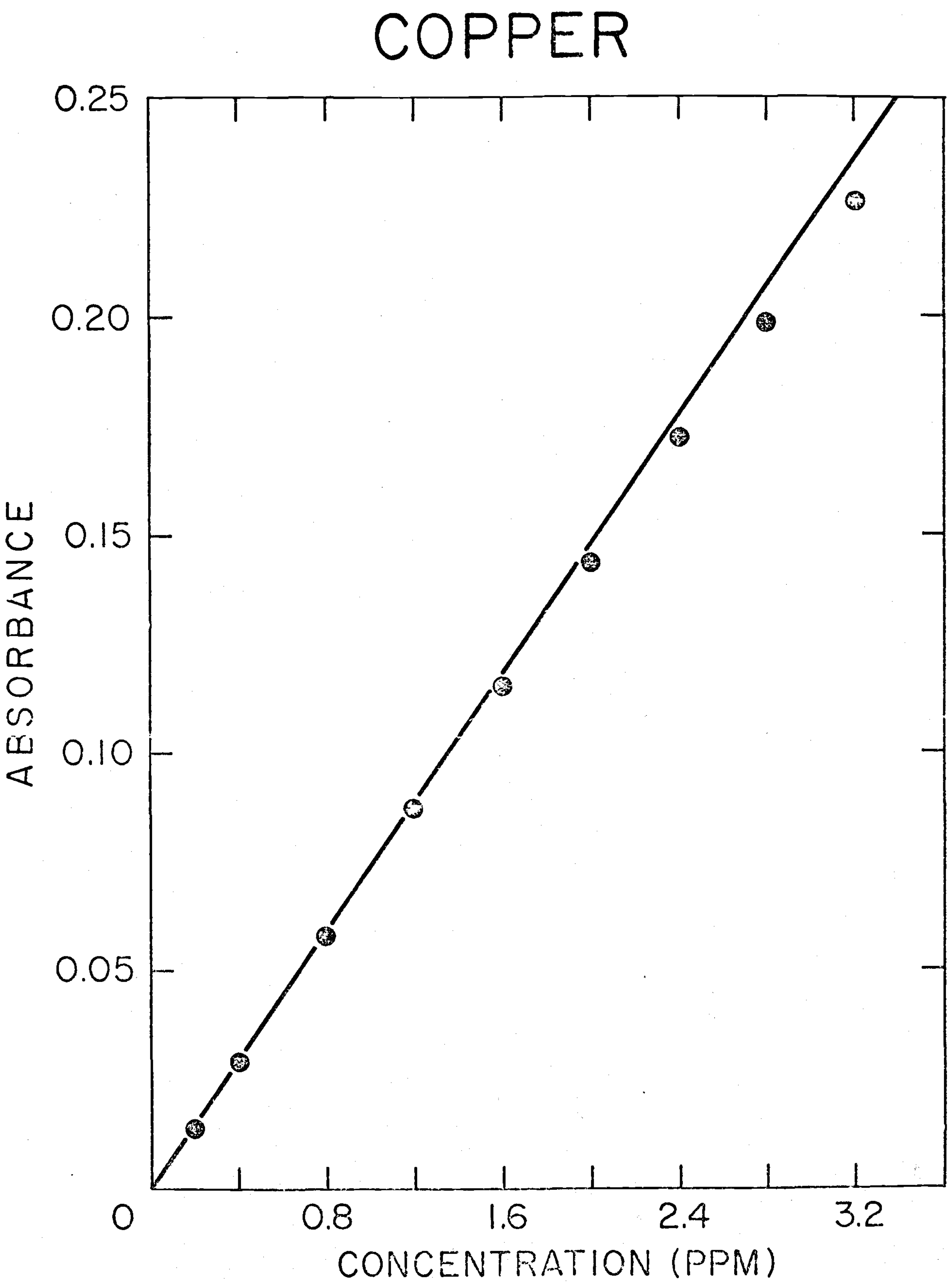
BARIUM



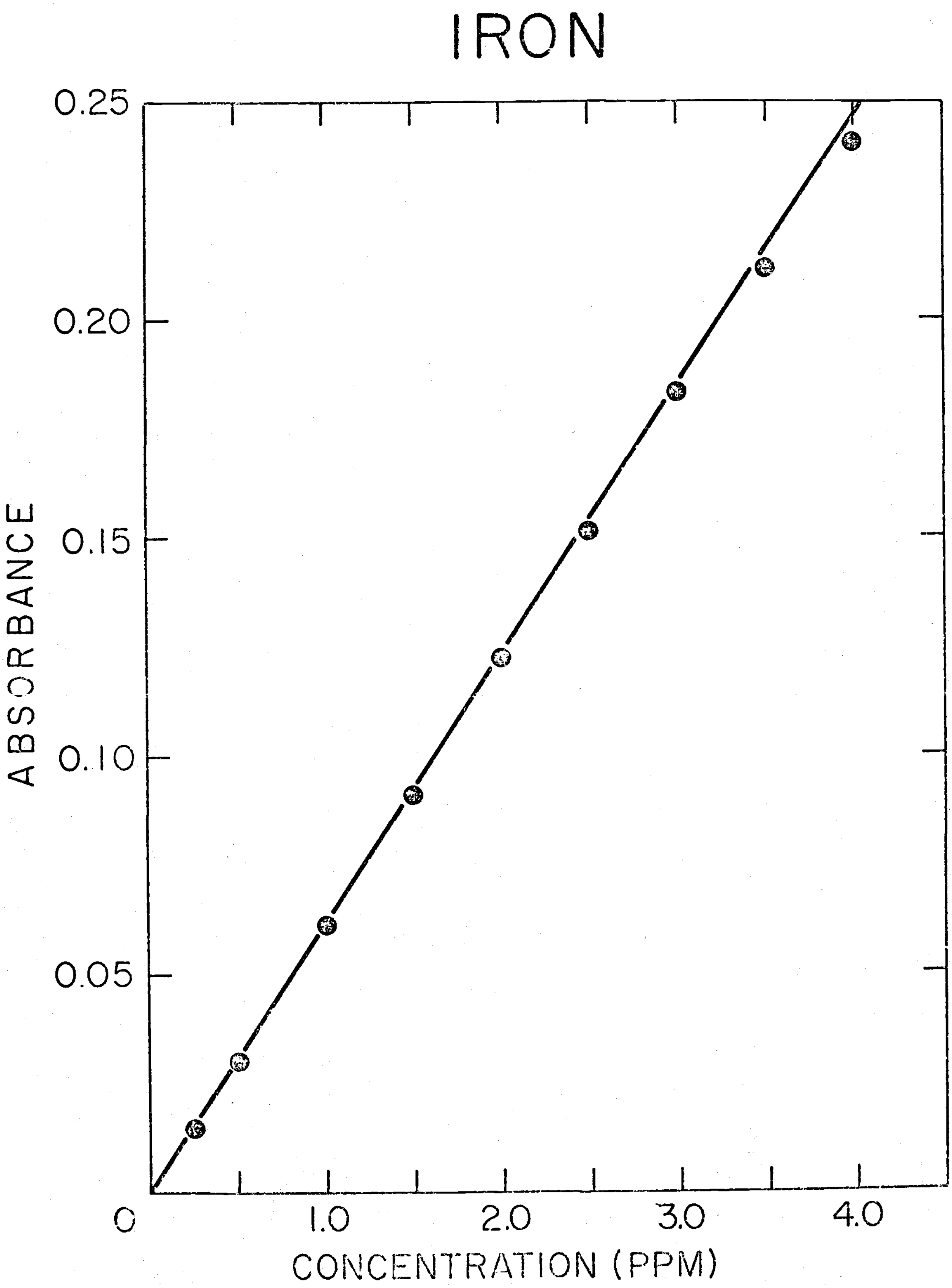
CALCIUM

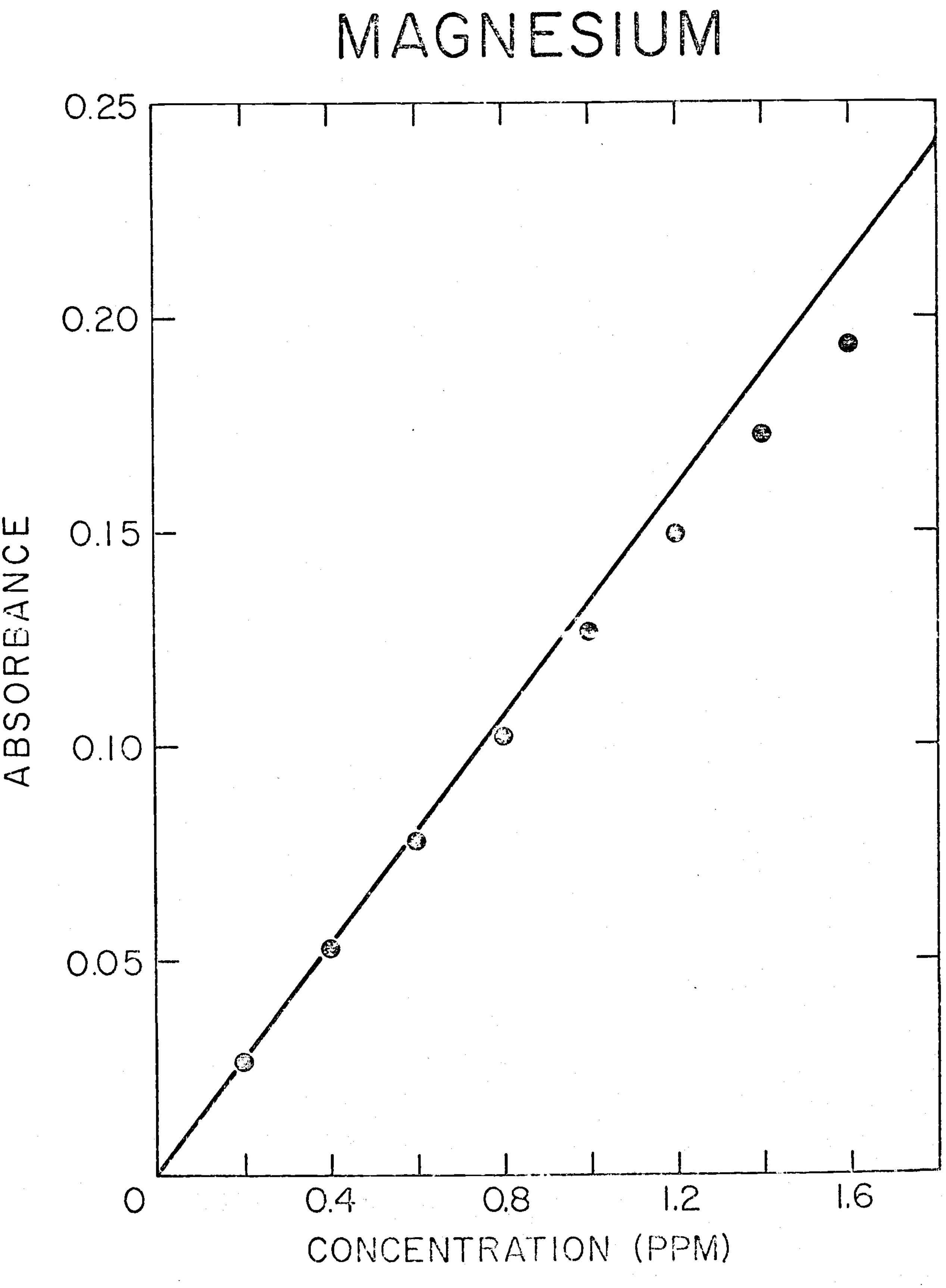


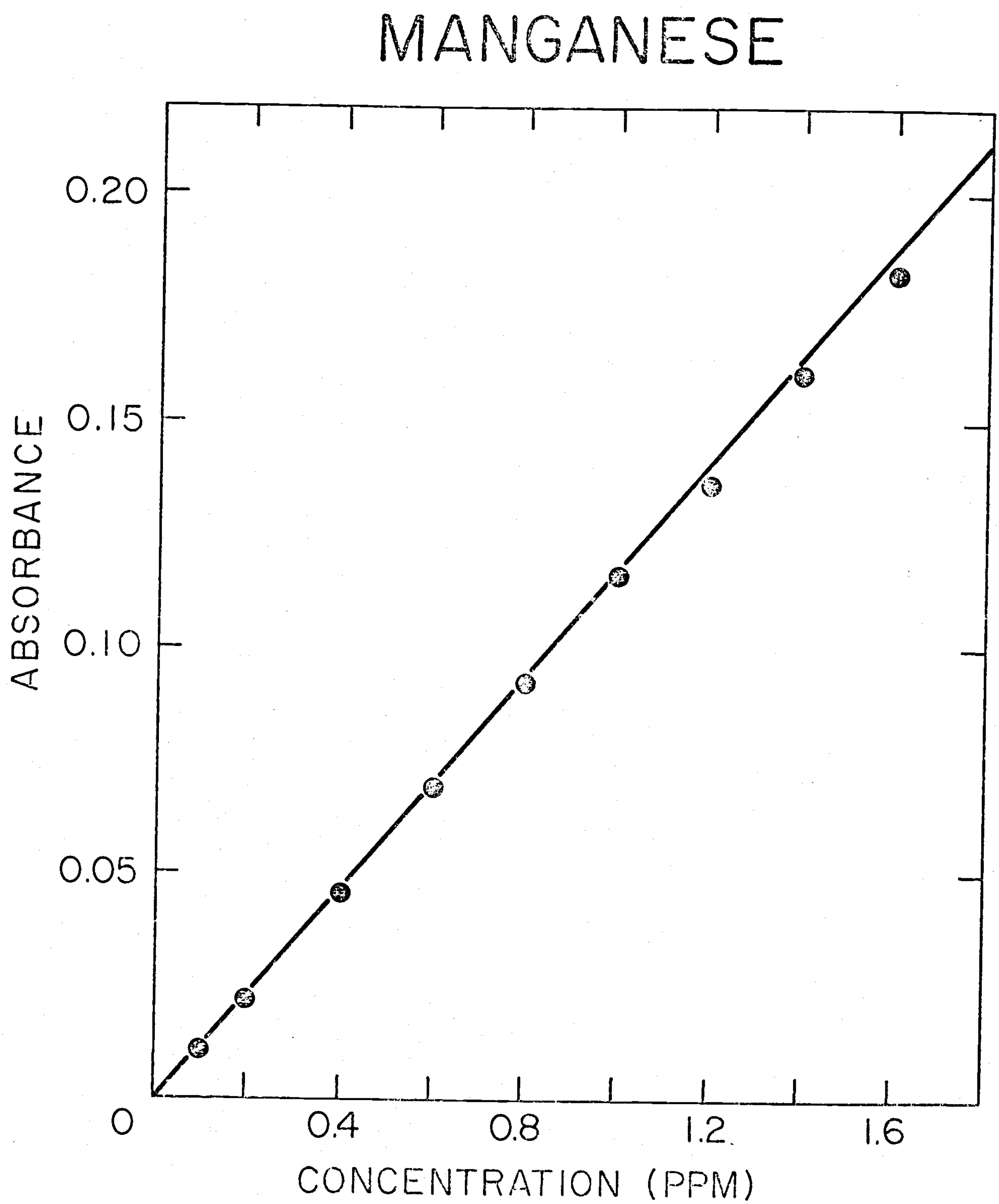

COBALT
0.25
0.20
0.15
0.10
0.05
0
ABSORBANCE
1
2
3
4
5
6
CONCENTRATION (PPM)



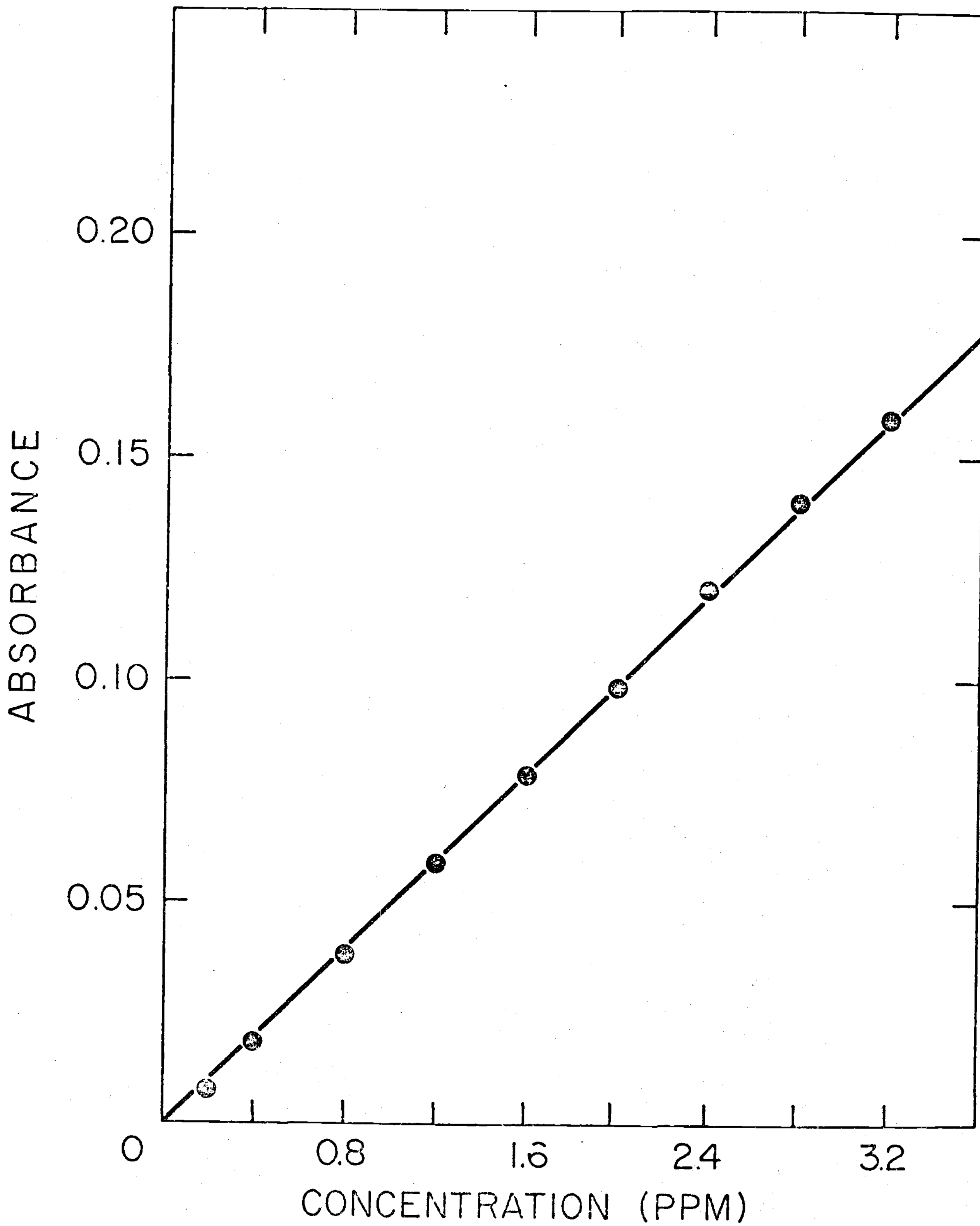
COPPER
0.25
0.20
0.15
0.10
0.05
0
0.8
1.6
2.4
3.2
ABSORBANCE
CONCENTRATION (PPM)

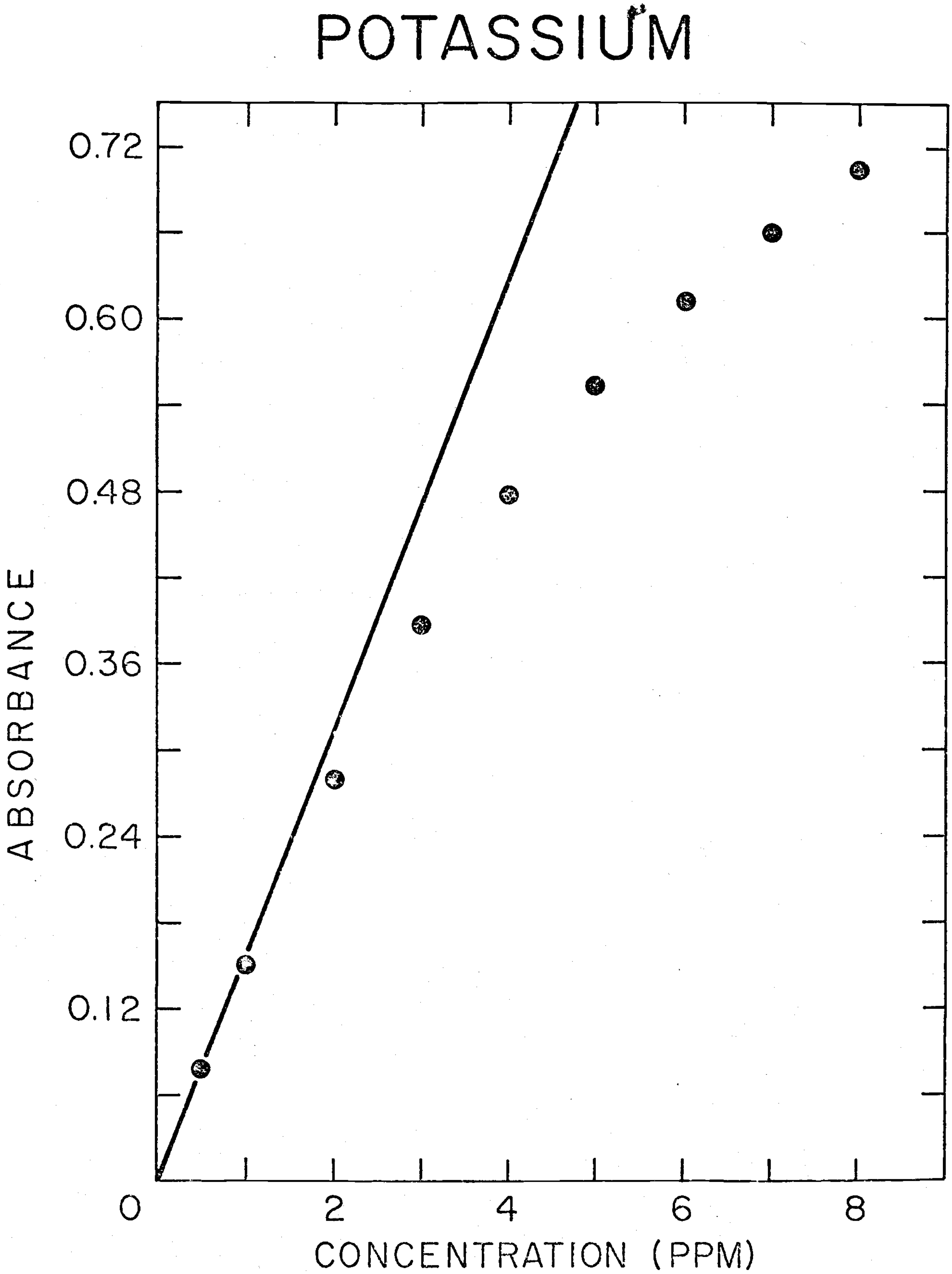

IRON
ABSORBANCE
0.25
0.20
0.15
0.10
0.05
0
1.0
2.0
3.0
4.0
CONCENTRATION (PPM)

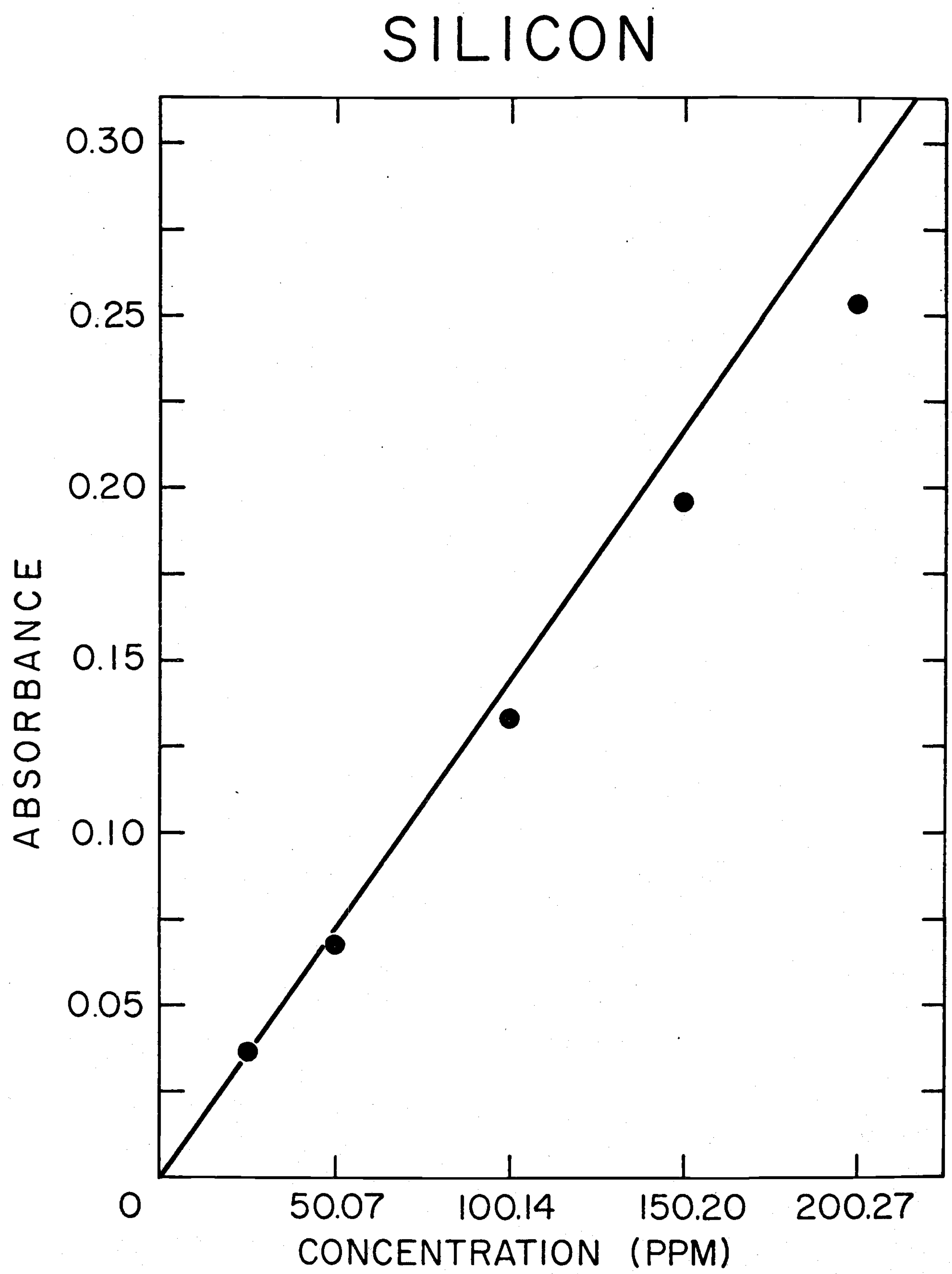

MAGNESIUM
0.25
0.20
0.15
0.10
0.05
ABSOREANCE
0
0.4
0.8
1.2
1.6
CONCENTRATION (PPM)

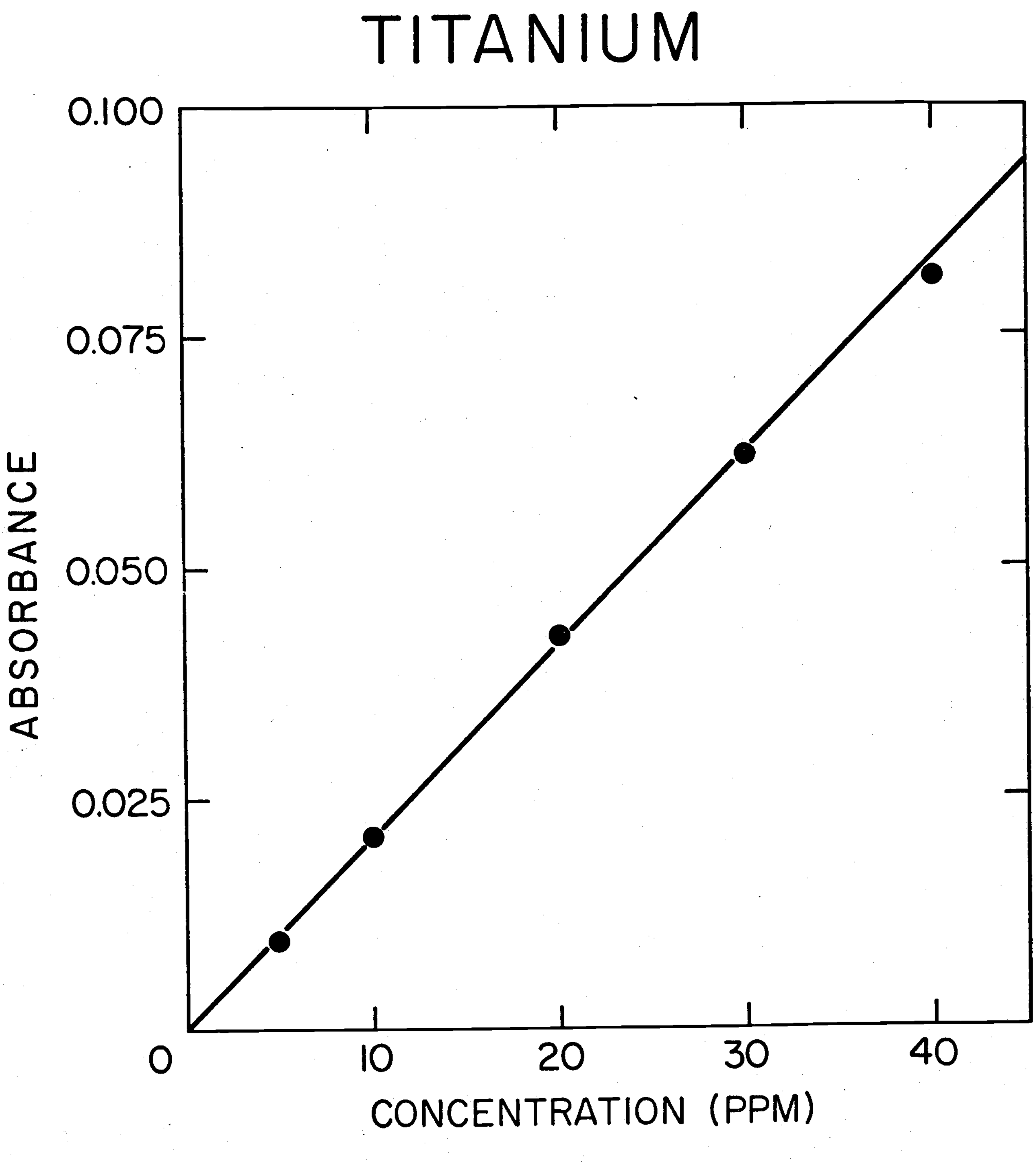

MANGANESE
ABSORBANCE
0.20
0.15
0.10
0.05
0
0.4
0.8
1.2
1.6
CONCENTRATION (PPM)

NICKEL

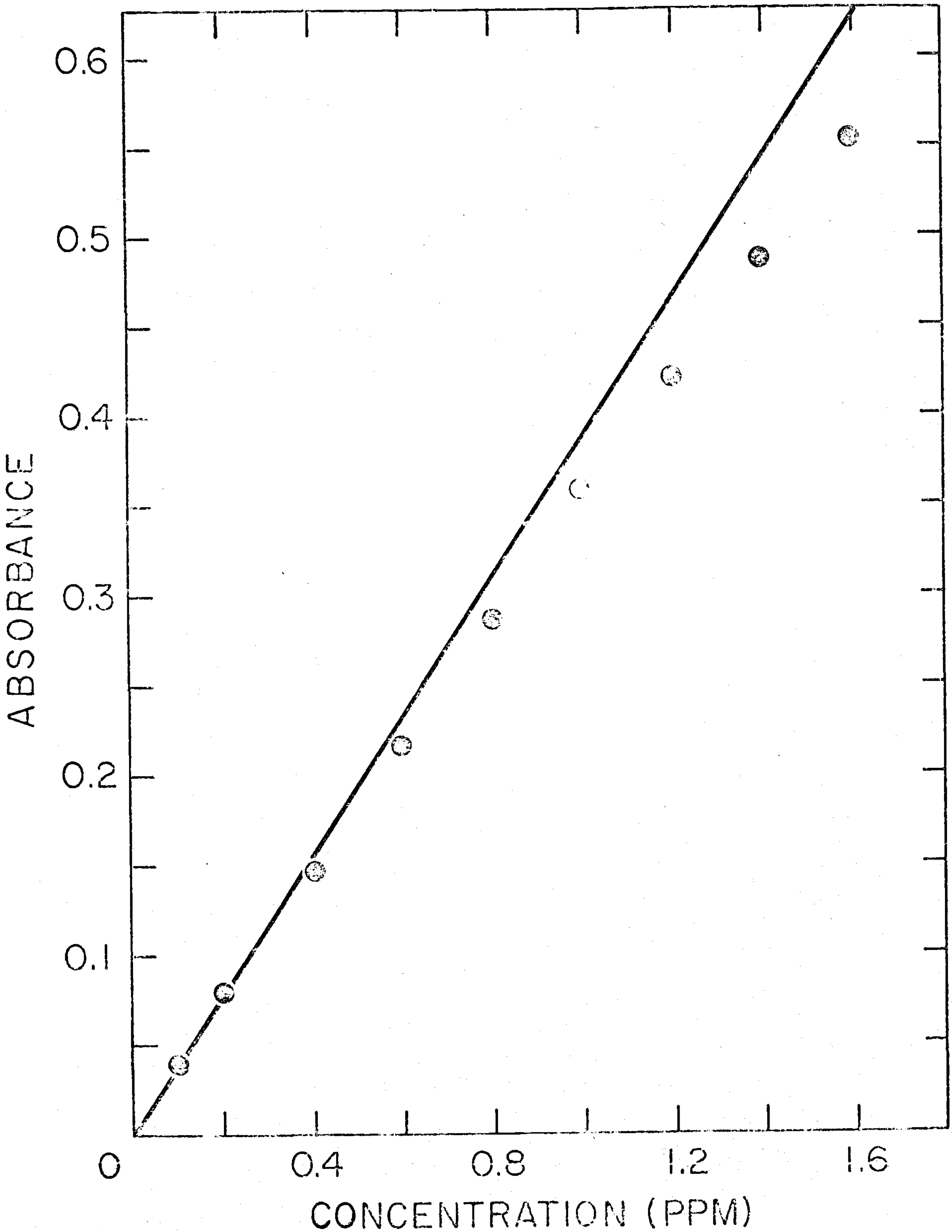



POTASSIUM
ABSORBANCE
0.72
0.60
0.48
0.36
0.24
0.12
0
2
4
6
8
CONCENTRATION (PPM)


SILICON
ABSORBANCE
0.30
0.25
0.20
0.15
0.10
0.05
0
50.07
100.14
150.20
200.27
CONCENTRATION (PPM)


TITANIUM
0.100
0.075
0.050
0.025
ABSORBANCE
0
10
20
30
40
CONCENTRATION (PPM)

ZINC



APPENDIX B

COLD VAPOR ATOMIC ABSORPTION OF MERCURY

COLD VAPOR ATOMIC ABSORPTION OF MERCURY

The reduction-aeration cold vapor technique for the determination of mercury involves the chemical reduction of Hg (II) ions in solution to neutral mercury atoms by a suitable reducing agent. The sample solution is then aerated with a carrier gas which carries the mercury atoms into an absorption cell where the absorbance is measured at the 2537 A mercury resonance line.

The reduction-aeration apparatus consists of a reduction vessel, an aeration device, a water mist vaporizer, and an absorption cell. The reduction vessel is made from a 10mm diameter glass tube fitted with a porous glass frit near its base. (Fig. B-1). Aeration is from below up through the glass frit and the solution. The small volume of the vessel, efficient aeration, and large effective solution surface area created by the bubbling cause rapid diffusion of Hg (o) out of solution and into the carrier gas. This enables a very sharp peak absorbance to be observed. Peak absorbance is also related directly to the length of absorption cell and inversely to its diameter (within limits). A cell 60cm in length and 2mm in diameter was found to be optimum for the sample size and mercury concentrations used.

Light scattering by water mist in the absorption cell is the main source of spectral interference with this system. This water is vaporized

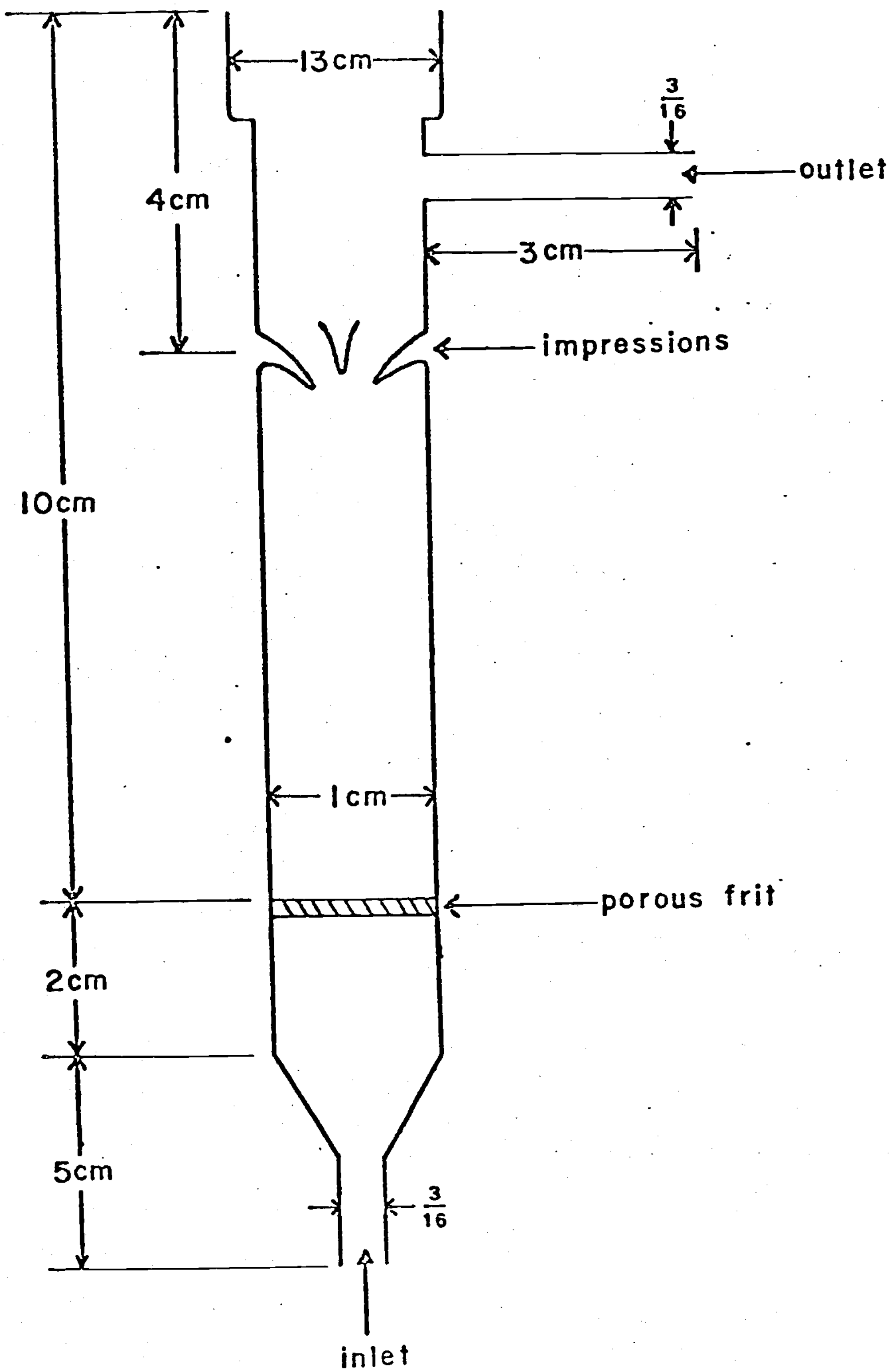


Figure B-1. Reduction vessel

by passing through a heating chamber, a glass tube filled with glass beads, at 200 °C, and by maintaining the absorption cell temperature above 100° C.

The mercury absorbance is measured by a double beam system. The mercury lamp beam is split and partially reflected to a reference photomultiplier tube and compared to the absorbed beam through a log ratio amplifier. The resulting absorbance is recorded on a pen recorder. Mercury concentration is linearly related to peak absorbance height in the range 0-5 ppb. See Figure B-2 for a diagram of the apparatus.

This cold-vapor atomic absorption apparatus was designed and built by Dr. Ingel of the O.S.U. chemistry department[1] and is described in detail in the MS thesis of James Hawley[2]. It has a detection limit of 4×10^{-12} gm Hg (4 ppt Hg in solution) and an effective range of .004-10 ppb in solution.

Hg ANALYSIS

Solution and Glassware Preparation

All solutions are prepared from reagent grade chemicals and double-distilled water.

Solutions

Reductant: 1 gm $SnCl_2$ 1 ml $HCl_{(c)}$ diluted to 100ml (1% ($\frac{w}{v}$) $SnCl_2$)

Oxidant: 0.2 gm $KMnO_4$ diluted to 100 ml (0.2% ($\frac{w}{v}$) $KMnO_4$)

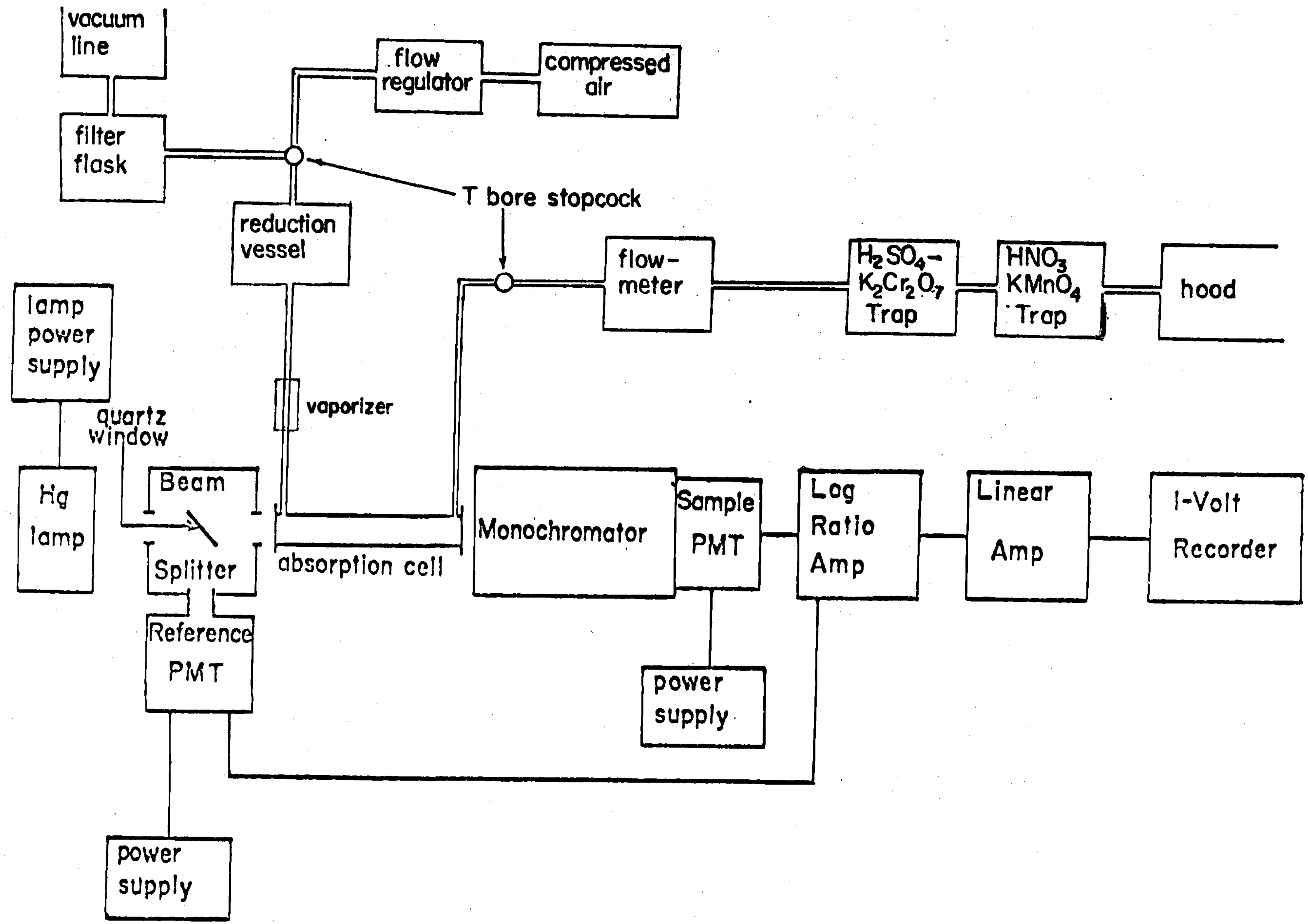


Figure B-2. Double beam AA

Mercury: 0.1354 gm $HgCl_2$ 50 ml HNO_3 (c) diluted to 1 liter (100 ppm Hg (II) stock sol.)

All standards are prepared by dilution of the 100 ppm Hg (II) stock solution and contain 1% $(\frac{v}{v})$ HNO_3 (c) and 0.002% $(\frac{w}{v})$ $KMnO_4$ as a preservative. This acidic and oxidative solution is necessary to prevent loss of mercury by 1) reduction to Hg (o) or Hg (I), which readily dissociates to Hg (II) and Hg (o), or 2) cation-exchange type adsorption onto the walls of the container. Dilute mercury solutions should still be analyzed within eight hours after preparation since mercury loss is still significant over long periods of time.

All glassware must be stored for 24 hours with a 1% HNO_3-0.002% $KMnO_4$ solution to remove traces of mercury adhering to the glass surface. Afterwards the glassware must be washed with HCl (c), rinsed with double-distilled water, washed with HNO_3 and rinsed with double-distilled water. Beware mercury contamination is everywhere, so all flasks, beakers, pipets, etc., must be initially cleaned with the acid-permanganate solution and should be rinsed with nitric acid before each use.

Sample Digestion and Analysis

Geologic samples are digested by the standard HF-pressure bomb technique and brought to a volume to contain less than 5ppb Hg. Teflon liners are rinsed just prior to digestion with acid-permanganate solution.

Manganese nodules and crusts are digested using 50mg samples that have been crushed in an agate mortar and dried in a dessicator at least 24 hours. A drying oven is not used because of the potential loss of mercury due to its high volatility. The material is weighed into the teflon liner and the following reagents are added:

1) 0.1 ml HNO_3 (c)

2) 0.3 ml HCl (c)

3) 2.0 ml HF (c)

After sealing, heating for $1\frac{1}{2}$-2 hours at 110 C, and cooling, 1.9 gm H_3BO_3 is carefully added directly into the Teflon liner. The solution is brought to a final volume of 25ml and immediately analyzed. Standards are either prepared from the Hg (II) stock solution in a HF-boric acid matrix, or a known amount of Hg (II) solution is added to a duplicate digested sample as a standard addition. Blanks are determined by adding only the reagents to the Teflon liner and taking this through the digestion procedure.

Analysis of the solutions is carried out as follows. Instrument variables are adjusted to optimal conditions as shown in Table B-1 from the Hawley M.S. thesis). After carrier gas flow through the frit is initiated, 0.1ml of the $SnCl_2$ reductant solution is injected by syringe into the reduction vessel. After a baseline is established on the pen recorder, 1.0 ml of sample or standard solution is injected. The peak absorbance and return to baseline occur in less than 20 seconds. The solution is then

Table B-1. Optimal variables for analysis

Variable	A. A.	A. F.
Flow rate	140 ml/min	140 ml/min
Frit grade	medium	medium
Drying tube	5 cm long x 12 mm dia $Mg(ClO_4)_2$	5 cm long x 12 mm dia $Mg(ClO_4)_2$
Gas carrier	air	argon
Volume of reductant	0.1 ml	0.1 ml
Volume of sample	1.0 ml	1.0 ml
Slit width	1000 um	2000 um
Absorption cell	20 cm long x 2 mm id 60 cm long x 2 mm id	--
Fluorescence cell	--	1 cm x 9 mm x 6 mm
Radiation source	Hg pen lamp (DC)	Hg pen lamp (AC)
Lamp current	9-10 ma	17 ma
RC time constant	0.32 sec	1 sec
System	Double beam	--
Photoanodic current	10^{-5} A	--
Photomultiplier supply[1] voltage	500-600 V	700-800 V
R_f[1]	--	10^6 Ω

[1]Adjusted as described in procedure

evacuated in preparation for the next analysis. The reduction vessel is cleaned after each day's use by flushing with HNO_3(c), rinsing with distilled water, flushing with $KMnO_4$ solution, and rinsing with distilled water.

REFERENCES

1 Hawley, J. E. and J. D. Ingle, Jr. (1975) Improvements in cold vapor atomic absorption determination of mercury, Anal. Chem., 47, 719.

2 Hawley, James E. (1975) Cold vapor atomic absorption and fluorescence analysis of mercury. Unpublished M.S. Thesis, Oregon State University, Dept. of Chemistry, 85 p.

APPENDIX C

PB ANALYSIS BY CARBON ROD

Pb ANALYSIS BY CARBON ROD

General Considerations

Pb tends to plate out of the dissolved sample. It should therefore be analyzed quickly after sample dissolution (within about a day). In the short testing I have done, it appears that the standards are more stable. They probably should be made up fresh for each analysis, however. Samples are dissolved by the standard HF-Aqua regia treatment. Because of the generally low Pb levels, we use 200 ml dilution for analysis. Standards are therefore made up using this matrix in 0, 0.05, 0.1, 0.2, and 0.3 ppm Pb concentrations.

Setting Up For Carbon Rod Analysis

We generally use the carbon cup for Pb analysis. It has lower sensitivity, necessitating larger sample size, but seems to be more easily reproducible.

A) Mounting the Cup - See Varian Techtron Manual

1) One support block for the electrodes has a click stop built in. Slide rod in until one of the slots machined in the rod clicks into place.

2) Slide other rod in. Put cup in place. Align light path holes with hole in masking plate. It is best done with thin rod.

3) Make sure cam lever is down (spreads support blocks slightly.) Fit electrode snugly against cup.

4) Fasten electrodes in place by clamping screws. Put cam lever back up again

B) Preparation of Carbon Rod Assembly for AA Work

1) Mount carbon rod assembly in AA work head holder

2) Warm up Pb lamp and adjust for Pb wavelength. We use 2833A plus 2820A as background non-absorbing line.

3) Align carbon rod in light beam with the burner head controls.

4) With chart recorder on 5 m_v scale, set A channel (2833A) and B channel (2820A) to cover same chart distance when light path is blocked. This should be done in per cent absorption. Switch to absorbance mode. Switch to 2m_v scale (expands scale of chart recorder 2.5 times.)

5) Turn on water supply to carbon rod assembly. (recommended is 0.5 liters/min.)

6) Turn on argon supply to carbon rod. Recommended is 10 psi gauge pressure at tank, and 7.0 setting on needle valve.

7) Set all power supply voltages to 0. Set ash time to 20, leave dry and atomize at 0. Set hydrogen gauge pressure to 10 psi. Cycle through power sequence. While machine is in ash cycle, set hydrogen flow to 1.0 on needle gauge. (Must be done this way because solenoid controlling hydrogen flow will not turn on until ash stage).

8) Set power controls as follows:

Stage	Voltage Setting	Time (in seconds)
dry	3.5	40
ash	2.5	20
atomize	9.0	3.5

Running the Carbon Rod:

1) All operations run on a 90 second interval between firings to allow rod conditions to be as reproducible as possible. Timer is necessary.

2) Fire about 5 times in sequence with no analyte. This cleans the rod and warms it up.

3) Pipette 20 ml of first standard into cup. Be careful to wipe excess off pipette, and that you pipette into the bottom of the cup. Be sure to rinse tip 3 to 4 times with new solution when changing to new standard.

4) Cycle through power sequence. Wait 90 seconds after atomize stage to run again. At about 15 seconds before firing time, pipette in new 20 ml aliquot of standard. Cycle it through at 90 seconds. Measure peak height of channel A and subtract from channel B to get concentration peak height.

5) If they reproduce, change to new standard, repeat (4). Do this for all standards. If they do not reproduce, keep running standards until they agree.

6) Run 1st sample four times.

7) Run through standards twice again. If you know all samples will fall within a small range, you can limit your standards to that range.

8) Repeat step 5 and 6 for all samples.

9) IF there is a systematic decrease in peak height of sample or

standard, your rods have probably worn out. Check to make sure decrease is not measurement error. If not, replace rods, go through procedure again for samples not yet analyzed. If you think rods are getting old, disconnect H_2, connect up lab gas (mainly CH_4). Give the rods a few burns with the lab gas. This will increase their life by replacing some of the lost carbon.

10) Analysis time: 30 to 40 minutes per sample.