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RESEARCH AND DEVELOPMENT DIVISION
ORGANIC CHEMICALS DEPARTMENT
E. I. DU PONT DE NEMOURS AND COMPANY

IMPROVEMENT IN THE DETERMINATION OF TRACE LEAD IN BODY FLUIDS AND TISSUES PART III - INVESTIGATION OF FLAME SPECTROSCOPY

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Project: 090009
Recent Previous Reports: ORD 68-352, 69-91

ABSTRACT

Atomic absorption and flame fluorescence were investigated to develop a more sensitive and a more accurate method for determining trace lead in body fluids and tissues. Work with long path atomic absorption, which has adequate sensitivity to determine 0.2 ppm lead in a 1-gram sample, was discontinued in favor of anodic stripping voltammetry. A suitable procedure was developed using short path atomic absorption for determining > 1 ppm lead.

Flame fluorescence was also discontinued because light sources lacked sufficient intensity to determine < 1 ppm lead in a 1-gram sample.

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OBJECTIVE

To develop a method for determining lead in biological samples which is more sensitive, accurate and precise than existing methods. To examine flame spectroscopy as a means to achieve this sensitivity.

BACKGROUND

The necessity for a more sensitive and accurate method for determining lead in samples of 1.0 gram or less has been stated in a previous report (Ref. 1).

Long path atomic absorption was shown to have the required sensitivity to determine 0.2 ppm Pb in a 1.0 gram sample of blood (Ref. 2). However, large amounts of other metals present in body fluids interfere and must be removed prior to the determination of lead.

An intense light source of Pb resonance energy for use with atomic fluorescence was sought unsuccessfully (Ref. 2). The same report showed that an examination of a microwave-excited discharge lamp for the intense source was justified.

CONCLUSIONS

1. Long-path (90-cm) atomic absorption has adequate sensitivity for determining 0.2 μg of Pb, but the procedures used to separate lead from interfering metals in blood have given low recovery of lead. An alternative procedure, anodic stripping voltammetry, (Ref. 3) is workable. No further work on the long path is planned.

2. A short-path (10-cm) atomic absorption procedure was developed to determine $\geq$ 1 ppm lead.

3. Microwave-excited discharge lamps gave insufficient Pb resonance radiation for sensitive fluorescence determinations of Pb.

PATENT SITUATION

The patent status of this work will not be investigated, because of the nature of the work.

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SAFETY

Flame spectroscopy involved the use of high temperature flames and the use of acetylene and hydrogen. The operator must be thoroughly familiar with the valving system controlling these highly flammable gases. A written procedure must be provided for a safe start-up and operation of the apparatus. No copper lines or fittings shall be used in acetylene service. The light sources, especially the electrodeless discharge lamps, should not be viewed without dark glasses with good absorption in the ultraviolet.

WASTE DISPOSAL

Only laboratory quantities of chemicals are used. Regular solvent disposal provided by the laboratory is used for non-aqueous materials.

PUBLICATION STATUS

Publication of this work is not planned at this time.

FUTURE WORK

1. Use anodic stripping voltammetry for determining less than 0.5 μg of lead.
    a. Search for a longer-lived working electrode.
    b. Determine digestion procedures for samples compatible with the method (blood, soil, bones).
    c. Investigate means to improve precision.
2. Determine if solvent extraction followed by atomic absorption can be utilized to determine Pb in urine, thus eliminating time-consuming evaporation and wet oxidation of these samples.

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I. DISCUSSION

A. Introduction

The need for more sensitive methods for Pb in biological samples has been shown previously (Ref. 1). A specific goal is to be able to determine routinely 0.2 ± 0.01 ppm Pb (normal level) in a 1-gram sample of blood. For special cases, a method must be available for determining Pb in 0.1 g samples. Methods are also required for determining Pb in urine to better than 0.01 ppm and in bones (> 2 ppm). The methods covered in this report are atomic absorption, both long-path (90-cm) and short-path (10-cm), and atomic fluorescence.

B. Long-path Atomic Absorption

A previous report (Ref. 2) showed that the major metals present in blood deteriorated the quartz long tube and scattered the light beam. Scattering of the light decreased the signal just as if Pb were present, thus interfering in the determination of Pb. The ratio of these metals to Pb in normal blood (Ref. 4) is: K-13,000:1; Na-11,000:1; Fe-2,300:1; Ca-500:1; Mg- 200:1. Two approaches, anion exchange and solvent extraction, were tried to effect an efficient separation.

1. Anion Exchange Separation

The work of Kraus and Moore (Ref. 5) showed that Pb could be separated from the interfering metals in hydrochloric acid by anion exchange. We checked the equilibrium distribution of Pb between the presently available Dowex-1, X-8 (Dow Chemical Company) anion exchange resin and various concentrations of HCl (Table I). A K value of 21 in 1 N HCl indicates a moderate absorption of Pb by the resin. Reasonable agreement was obtained between this work and that of Kraus and Moore (Ref. 5). In checking out the proper size of resin column and volumes of eluate to effect a separation, we found that this method of separation was much too slow if a large number of samples was to be analyzed. This approach was abandoned without trying to obtain recoveries of Pb better than those shown in the Experimental Section II, A, 2a).

2. Solvent Extraction Separation

The work by Stary and Smizanska (Ref. 6) showed that lead could be separated from the major metals in blood by extracting the aqueous phase with cupferron in $CHCl_3$. Na, K, Ca and Mg are not extracted from the aqueous phase. Iron is quantitatively extracted at all pH's between 0 and 13; all of the lead remains in the aqueous phase at pH <1 but it is quantitatively extracted into the $CHCl_3$ phase at pH >3. The procedure described in the Experimental Section was used as a basic procedure. A special flask was used so that the acid digestion of the sample and subsequent extraction could be carried out without a transfer (Figure 1), thus reducing the probability of

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contamination or loss of Pb. The aqueous solution from acid digestion is neutralized and pH is adjusted >8 with NH_4OH. Pb and Fe are extracted into the organic phase leaving the alkali and alkaline earth metals in the aqueous phase. One wash of water is used to rinse the vessel of these metals. Neither of these two aqueous phases contained a significant amount of Pb. Also a second extraction of the $CHCl_3$ extract with 0.5 N HCl showed that no Pb was present in the second acid wash. These tests proved that 100% of the Pb present in the original sample ended up in the 0.5 N HCl extract.

In applying this method of separation to a digested blood sample, modifications were necessary. The amount of acid in the digest is variable and therefore must be neutralized to an indicator change rather than by addition of a known amount of base. The precipitation of $Fe(OH)_3$ is an indication of proper pH of the solution, but is a detriment to the extraction of Pb by cupferron. Blood samples thus treated yielded low results for Pb due to coprecipitation of Pb with $Fe(OH)_3$. Good recovery of Pb was obtained when the cupferron was added prior to neutralization to slightly acidic solution. When the cupferron is added first to the strongly acid solution, two possible difficulties may be met. Cupferron is unstable in strong acid. The rapidity of oxidation of the cupferron was not investigated. Second, determination of when the pH is greater than 3 is difficult in the presence of the voluminous precipitate of Fe cupferrate. If acid-base indicators are used, care in their selection is imperative. Almost 100% recovery of lead from HCl was obtained when phenolsulphonephthalein was used as an indicator. Using bromocresol green gave 0% recovery. The use of pH paper or electrodes to determine the extent of neutralization was considered to be a significant source of loss of the small volumes of solutions being used.

The successful development anodic stripping voltammetry technique, which was being sought concurrently, (Ref. 3) caused the work toward solution of this separation problem to cease.

C. Atomic Fluorescence

In the previous report (Ref. 2) atomic fluroescence conditions were given which had a detection limit for Pb of 3 ppm in the solution aspirated into the flame. A hollow cathode lamp was used to obtain this detection limit. In order to be useable in attaining the goals of this project, a detection limit at least as low as 0.01 ppm Pb is required. The exciting radiation (283.3 nm) needs to be 300 times that of the hollow cathode lamp to achieve this detection limit. The intensities of various electrodeless discharge lamps were measured versus the hollow cathode lamp used previously for the fluorescence detection limit work. The intensities of the discharge lamps fell far short of the 300-fold increase needed. No set of conditions gave a measurement of intensity greater than 50 times that of the hollow cathode lamp. The details of the measurements are in the Experimental Section. A general discussion of electrodeless discharge lamps as well as a study of various parameters in their construction is given by J. M. Mansfield et al, in Reference 8.

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D. Lead in Bone

An atomic absorption spectroscopy procedure using a 10-cm slot burner was developed for use with bone samples. C. Chakrabarti (Ref. 7) had reported the use of methyl isopropyl ketone to extract Pb from an aqueous KI-HCl solution and aspiration of the organic phase into an atomic absorption flame for determination of Pb. Methyl isobutyl ketone was used in this investigation because of its lesser tendency toward forming emulsions with the aqueous phase. The calibration graph (Figure 2) shows that adequate sensitivity is present to determine Pb in the range of 5 to 80 ppm in a 0.5 g sample. Interferences were studied and it was found that calcium phosphate as well as other trace metals (Fe, Cu, Zn) to the extent present in bones does not interfere in this determination of Pb.

Samples of beef bones received from Haskell Laboratory were analyzed for Pb by both extraction-atomic absorption and anodic stripping voltammetry procedure (Ref. 3). Although the atomic absorption procedure is not as accurate as anodic stripping voltammetry for the low level determination of Pb, those samples analyzed both ways showed good agreement between the methods (Table II).

Values of trace metals in bone are reported on two bases in the literature. "Ash" at some temperature and "fat-free, dry bone" are used as reporting bases. In order that the results from this laboratory may be compared readily with other work, the work-up of the bones has been planned so that the Pb content will be reported on the "fat-free, dry" basis and the percent ash at 550°C in the "fat-free dry" bone will be reported. The procedure used for the work-up of the bone samples is presented in the Appendix.

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II. EXPERIMENTAL

A. Atomic Fluorescence

A Magnitron, Model 2000L, microwave generator (Aztec Instrument Company, South Norwalk, Conn.) was used to power electrodeless discharge lamps (EDL). The electrodeless discharge lamps were obtained with argon filling gas from Aztec Instrument Company and EDL's with neon filling gas were obtained from Ophthos Instrument Company, 9600 Overlea Dr., Rockville, Maryland. The lamps from Aztec were standard production items; those from Ophthos were made specially with PbI_2 and neon in unspecified quantities. A reflected power meter for the above generator from Aztec Instrument Company was used to adjust the tuning of the microwave cavities. Two microwave cavities,#210 L and #214, were used to couple the generator to the lamps, both from Aztec Instrument Company.

A lamp was operated by placing it within a cavity, turning on the generator and initiating the discharge within the lamp by allowing the high voltage discharge from a Telsa coil to touch the lamp. Adjustments were made with the positioning of the lamp within the cavity and the tuning stubs on the cavities to give a minimum reflected power reading. That is, maximum percentage of power coupled from the generator to the lamp.

Relative brightness of the EDL's was determined versus a hollow cathode lamp (Atomic Spectral Lamp - small bore, HC-Pb from Varian, Palo Alto, Calif.) operated at 285 c/sec and 10 milliamperes. The EDL's and the hollow cathode lamps were positioned at separate times the same distance from the slit of a Techtron AA-4 atomic absorption monochromator. Minor adjustment of the lamp position was made to give maximum signal from the photomultiplier tube (standard Techtron tube, HTV type R213). An oscilloscope was used to measure the photomultiplier tube output which was D.C. with the EDL's and A.C. with the hollow cathode lamp. Both microwave cavities were used to couple the power source to to each of the two types of discharge lamps, argon-filled and neon-filled.

Repeatable readings were not possible with the EDL due to a very critical dependence of the brightness of the lamps on the position of the lamps within the cavities. At no time was a brightness of an EDL at 283.3 nm measured which was more than 50 times that of the hollow cathode lamp.

B. Atomic Absorption

1. Apparatus

The long path atomic absorption tube (90-cm) was used as described previously (Ref. 2). The 10-cm burner, Techtron burner AB 41, was set up with the Techtron AA-4 for atomic absorption as described in the Techtron manual.

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2. Separation of Pb

a. Anion Exchange

The distribution of Pb between anion exchange resin and aqueous solution was determined by adding weighed amounts of dry (110°C for 3 hours) resin (Dowex-1, X-8)(J.T. Baker, No. 1906) in the chloride form to 10 ml of various HCl concentrations, each of which contained 1.00 ppm Pb. The mixture was shaken for 2 hours at room temperature. One ml of the clear supernatant liquid was pipeted into a 10-ml volumetric flask. Hydrochloric acid was added to make 1.0 N when diluted with water to 10 ml volume. The Pb concentration of these solutions was determined using the long tube. The results are shown in Table I.

A resin column of 8-mm x 100-mm long was used to test the separation of Pb from the alkali metals. Ten ml of 1 N HCl containing 15 mg of NaCl was passed through the column followed by a wash of 1 N HCl. All of the Na was eluted in the first 10 ml of the wash solution, as determined by a qualitative flame test. Ten μg of Pb was added to the column in 10 ml of 1 N HCl and the column washed with 10 ml of 1 N HCl. Each ml was analyzed for Pb using long tube atomic absorption. These washes contained a total of 0.23 μg of Pb or a total of 2.3% of that added. Washing the column with water eluted the Pb with a maximum concentration in the fourth and fifth ml portions with no Pb detected (<0.01 μg) after the 8-ml fraction.

Recovery runs were made by placing 0.50 μg of Pb onto the column in 10 ml of 1 N HCl, washing the column with 10 ml of 1-N HCl and eluting the Pb with 10 ml of water. The time for a column run was from one to two hours.

μg Pb Added	μg Pb Found
0.00	0.12
0.00	0.25
0.50	0.57
0.50	0.66

b. Cupferron Extraction

The reagents for the extraction were prepared as follows. Chloroform was extracted in turn with 3 N HCl, H_2O, cupferron reagent, 0.5 N HCl and H_2O. Absolute ethyl alcohol was then added to make a 1% solution to inhibit hydrolysis. NH_4OH and HCl were purified by isothermal diffusion into redistilled water. Cupferron reagent was prepared by dissolving 15.5g of the ammonium salt in water, adding 115 ml of NH_4OH (14 N) and diluting to 1.0 liter. Filter through paper and extract twice with purified $CHCl_3$.

The following is the basic extraction procedure used for standard solutions in developing the separation of Pb from the major metals present

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in blood.

Add the Pb standard in 1 N HCl to the extraction tube (Figure 1). Pipet 5.0 ml of cupferron reagent into the tube. Add $CHCl_3$ to bring the interface into the restricted neck. Add purified NH_4OH to neutralize the sample. Shake well and let the phases separate. Remove and discard the top (aqueous) phase. Add 2-ml of water and shake well. Discard this aqueous wash. Pipet 3.0 ml of 0.5 N HCl into the tube. Shake well. Let the phases separate and aspirate the HCl phase into the long tube for determination of Pb by atomic absorption.

In order to use Pb in 0.5 N HCl as a standard for the long tube atomic absorption determination of Pb in the extract from the above separation, the sensitivities for Pb were compared for (1) Pb in 0.5 N HCl, (2) 0.5 N HCl saturated with $CHCl_3$ and (3) 0.5 N HCl saturated with $CHCl_3$-cupferron solution. There was no difference in sensitivity.

Extractions were run using 10-second equilibration times for each time of shaking required in the above procedure. These were compared with extractions using 30-second shaking times. No difference in recovery was seen (Table III).

3. 10-Cm Burner

The conditions to give the best signal to noise ratio were found by aspirating into the flame the organic phase from an extraction of 800 ml water containing 32 ml HCl (conc.), 17 g KI and 250 μg Pb with 130 ml of methyl isobutyl ketone (MIBK). The variables examined were: (1) fuel gas (H_2 or C_2H_2), (2) fuel flow rate, (3) height of light beam above the burner, (4) wavelength (283.3 or 217.0 nm). The best conditions were found to be H_2/air: (4.8 and 5.1 l/min), beam:7 mm above the burner, and 283.3 nm. With these conditions the amounts of HCl and KI necessary for optimum sensitivity were sought. The extractions were made from 40 ± 2 ml containing 21.4 μg Pb and various amounts of HCl and KI. The MIBK volume added was 10.0 ml. The mean values at each condition are shown in Table IV.

Using 2.5 g of KI and 4.0 ml of HCl in 40 ml of aqueous phase, extractions were made to establish the extent of various possible interferences. These are shown in Table V. The extraction under these conditions was found to be rapid. There was no difference in the recovery of Pb between equilibration times of 10 and 30 seconds.

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IV. APPENDIX

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APPENDIX

DETERMINATION OF Pb IN BONES

A. Extraction of Fat From Bone

1. Remove flesh, tendons and marrow from the bone.

2. Weigh (± 0.001 g) 2 to 5 grams of bone and wrap it in filter paper.

3. Identify the paper with pencil and place in a Sohxlet extractor and extract using benzene-ethanol (1 + 1) for at least 7 hours.

4. Place the bone in a glass tube and draw filtered laboratory air (MSA Ultra Air Filter 15-82144) over it to remove residual solvent; then dry for 2 hours in an oven at 110°C.

5. Cool in a desiccator and weigh (± 0.001 g) the bone.

Calculations:

$$\frac{W_2 \times 100}{W_1} = \% \text{ dry, fat-free bond}$$

where: W_1 = weight of bone, Step 2

W_2 = weight of bone, Step 5

Report to the nearest 0.1%.

B. Determination of Ash

1. Weigh (± 0.001 g) 0.3 to 1.0 of dry, fat-free bone into an ignited, tarred 30-ml Vycor crucible.

2. Place in a furnace at 550°C for 3-4 hours.

3. Cool in a desiccator and reweigh (± 0.001 g).

Calculations:

$$\frac{W_2 \times 100}{W_1} = \% \text{ Ash, based on dry, fat-free bone}$$

where: W_1 = weight of bone, Step 1

W_2 = weight of bone, Step 3

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C. Digest and Determination of Pb

1. Weigh (± 0.0001 g) 0.3 to 0.5 g of dry, fat-free bone into a 50-ml Erlenmeyer flask. Carry two blanks through the procedure also.

2. Add 5 ml of HNO_3 (conc.). Heat gently on a hot plate. The acid must boil off slowly. Take to dryness.

3. Repeat Step 2 until there is no further darkening of the residue when dried.

4. Add 1 ml of HCl (conc.) and evaporate to dryness. Repeat once.

5. Add 10 ml HCl (5 N), cover and warm to dissolve completely. The HCl must not be lost during the dissolution step.

6. Make up two standards and a blank in 50-ml Erlenmeyer flasks.

   a. Pipet 10.0 ml of 0.2 μg Pb/ml standard into each of 2 flasks.

   b. Pipet 4 ml HCl (conc.) into each of the 3 flasks.

7. Dilute with H_2O to about 30 ml in all flasks.

8. Add 5.0 ml KI solution (0.5 g/ml). Dilute to 40 ± 2 ml with H_2O.

9. Pipet 10.0 ml of methyl isobutyl ketone into each flask.

10. Stopper and shake for at least 15 seconds.

11. Let settle and aspirate the organic phase into the flame for measurement of percent absorption at 283.3 nm.

    Burner conditions: H_2 4.8 l/Min.; Air 5.1 l/Min.; light beam 7 mm above the burner.

    Calculations:

    Convert percent absorption to absorbance

    $$\frac{S}{A_o} \times A_s = C = \mu g \text{ Pb in sample}$$

    where: S = μg of Pb in the standard, Step C-6.
    A_o = net absorbance (less blank, Step C-6) of the standard.
    A_s = net absorbance (less blank, Step C-6) of the sample.

    $$\frac{C - B}{W} = \text{ppm Pb, based on dry, fat-free bone.}$$

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where: C = μg Pb in the sample.
B = μg Pb in the digested blank.
W = weight, in g, of sample, Step C-1.

Report the results to the nearest 0.1 ppm.

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FIGURE 1

QUARTZ DIGESTION-EXTRACTION VESSEL FOR BLOOD ANALYSIS

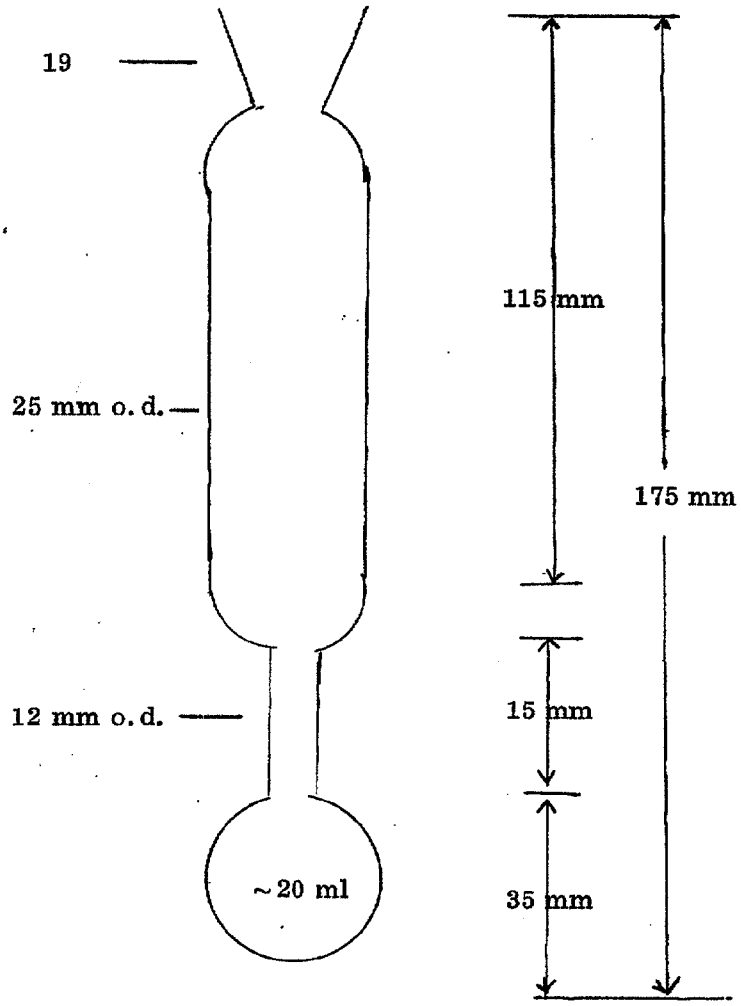

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FIGURE 2

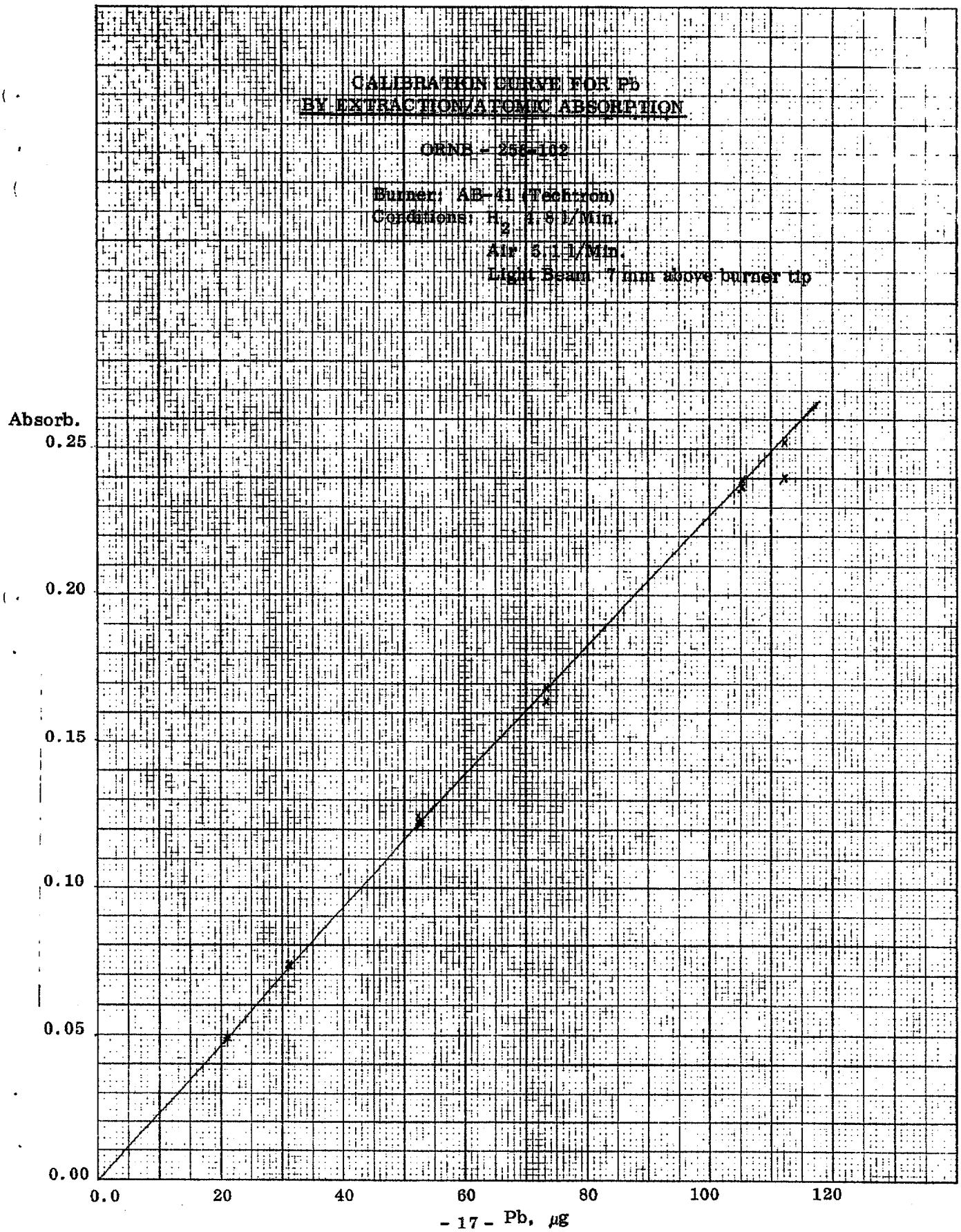

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Table I

ANION EXCHANGE OF Pb
ON DOWEX-1, X-8

(ORNB 256-35)

N HCl, 10 ml	g Of Resin	ppm Pb In Solution	μg Pb In Resin	K*
0.018	5.08	0.87	1.3	0.29
0.102	5.21	0.22	7.8	6.8
0.96	2.20	0.175	8.25	21.0
0.192	1.20	0.37	6.3	14.0
3.84	0.91	0.61	3.9	7.0
6.0	4.17	0.43	5.7	3.2
8.0	5.00	0.58	4.2	1.4

$$* K = \frac{\mu g \text{ Pb in resin}}{\text{g of resin}} \times \frac{1}{\mu g \text{ Pb/ml in solution}}$$

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Table II

DETERMINATION OF Pb IN BONES - COMPARISON OF METHODS

(ORNB 853-3, 5, 23)

Sample No.	Atomic Absorption, ppm Pb	Anodic Stripping, ppm Pb
469-1-3L	1.9	1.9
	2.0	1.7
469-1-3R	2.3	2.3
	2.5	2.2

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Table III

EQUILIBRATION TIMES FOR EXTRACTING Pb FROM $CHCl_3$-CUPFERRON INTO 0.5 N HCl

(ORNB 256-78)

10 Seconds		30 Seconds	
μg Pb Taken	% Recovery	μg Pb Taken	% Recovery
0.230	84	0.230	101
0.230	84	0.230	97
0.230	85	0.230	97
0.265	102	0.230	88
0.265	102	0.230	88
0.265	102	0.230	95
0.577	94	0.230	98
0.577	90	0.230	99
0.577	91	0.230	97
0.662	100	0.230	100
0.662	99	0.230	95
0.662	99	0.230	95
		0.577	94
		0.577	94
		0.577	95
		0.577	94
		0.577	95
		0.577	94
		0.577	95
		0.577	94
		0.577	95
		0.577	96
		0.577	95
		0.577	95

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Table IV

PERCENT ENERGY ABSORPTION (283.3 nm) vs.
CONDITIONS FOR EXTRACTING Pb INTO MIBK

(ORNB 256-93)

	HCl, ml			
KI, g	1	3	5	7
0.5	9.3%	8.2%	8.4%	-
1.5	10.1	9.8	9.3	8.3%
2.5	9.9	9.9	9.8	9.1
5.0	8.7	9.0	9.5	-

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Table V

POSSIBLE INTERFERENCES - MIBK EXTRACTION OF Pb

(ORNB 256-95, 99)

Addition	% Recovery of Pb (21.0 μg Added)
None	98.2, 100.4, 99.4, 100.4, 101.6
$Ca(H_2PO_4)_2 \cdot 2H_2O$, 0.5 g	94.4, 96.5, 91.7
HNO_3, 1.0 ml	86
H_2SO_4, 0.8 g	96.9
Sn, 19.2 mg	101.0
Bi, 20.2 mg	99.3
Sb, 20.0 mg	92.8
Zn, 20.6 mg	101.0
Fe, 19.0 mg	78.6
Fe, 11.4 mg	80.0
Fe, 2.3 mg	99.5
Cu, 20.2 mg	70.0
Cu, 2.0 mg	97.8

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