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SUBJECT: A CURRENT REVIEW OF BODY FLUID ANALYSIS,
PROCEDURES, AND METHOD OF REPORTING - JANUARY 1957

TO: Distribution

FROM: P. E. Brown, D. M. Davis, L. C. Henley

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OAK RIDGE NATIONAL LABORATORY
Applied Health Physics Sections
Health Physics Division

A Current Review of Body Fluid Analysis, Procedures, and
Method of Reporting - January 1957

- by -

P. E. Brown
D. M. Davis
L. C. Henley

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ACKNOWLEDGEMENTS

Analytical procedures outlined in this report are based on the work of Mr. L. B. Farabee of the Applied Radiobiology Section of the Health Physics Division.

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- VI. Analytical Procedures.
- VII. Forms Used in Requesting Samples, Recording Data, and Submitting Results.

I. Introduction

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Body fluid analysis are made routinely by the Bio-Assays Unit for H^3 , Po, Pu, Ra, Sr, U, Gross Alpha (Th, Pu, Am, Cm) and Gross Beta (including K^{40}).

The purpose of this report is to indicate the sampling procedures currently in use (January 1957) and to summarize the maximum permissible body burden and concentration data as given in Handbook 52 and the ICRP reports.

II. Maximum permissible amount of radioisotope in total body and maximum permissible concentration in air and water for continuous exposure. (ICRP 54-4)

<u>Element</u>	<u>Organ</u>	<u>μc in Total Body</u>	<u>$\mu c/ml$ H_2O</u>	<u>$\mu c/ml$ Air</u>
Po ²¹⁰ (sol)	Spleen	.04	3×10^{-5}	5×10^{-10}
Pu ²³⁹ (sol)	Bone	.04	6×10^{-6}	2×10^{-12}
Ra ²²⁶	Bone	.1	4×10^{-8}	8×10^{-12}
Unat (sol)	Kidneys	.04	10^{-4}	3×10^{-11}
Sr ⁸⁹	Bone	2	7×10^{-5}	2×10^{-8}
Sr ⁹⁰ + Y ⁹⁰	Bone	1	8×10^{-7}	2×10^{-10}
H^3	Total Body	10^4	0.2	10^{-5}

III. Elimination rates for chronic exposures to maximum permissible concentrations.

<u>Element</u>	<u>d/m/24 hr. Urine Sample for 100% MPC</u>
Po	530
Pu	7
Ra	7
Sr ⁸⁹	800
Sr ⁹⁰ + Y ⁹⁰	400
U	75
	<u>$\mu c/liter$ of Sample</u>
H^3	200

IV. Single exposure values for inhalation of soluble radioactive material when body organs other than GI Tract or lungs are the critical body organs. (1)

<u>Isotope</u>	<u>Organ</u>	MPI or μ c inhaled in 8 hours that will present a dose of:		
		<u>.3 rem/wk.</u>	<u>15.7 rem/yr.</u>	<u>150 rem/70 yr.</u>
H ³	Total Body	1.5×10^4	1.7×10^5	1.7×10^6
Po ²¹⁰	Spleen	.60	3.7	33
Pu	Bone	.13	.12	2.0×10^{-2}
Ra	Bone	1.4	1.4	0.32
Sr ⁸⁹	Bone	10	47	4.3×10^2
Sr ⁹⁰ + Y ⁹⁰	Bone	5.3	5.7	5
U(nat)	Kidney	3.7×10^{-2}	0.28	2.7

V. Elimination Rates for Acute Exposures.

The elimination rates in terms of "Per Cent of Initial Body Burden Eliminated" vs "Days After Exposure" for Po²¹⁰, Pu²³⁹, Sr⁹⁰, and U(nat). are given in Graphs I-IV, pages 28-31. The data for the Pu, Sr, and Po curves were obtained from Dr. Langham of Los Alamos. The data for the U(nat) curve were obtained from Mr. Bernard of Oak Ridge. The elimination rate is for urine only.

VI. Analytical Procedures - (Outlined in following order)

- A. LaF₃ Determination of Pu in Urine.
- B. Laboratory Directions and Reagents for Ra Analysis.
- C. Polonium Analysis by Internal Electrolysis.
- D. Reagents, Equipment, and Procedures for Radiochemical Analysis of Strontium and Barium in Human Urine.

(1) "Maximum Permissible Concentration of Radioisotope in Air and Water For Short Period Exposure", K. Z. Morgan, E. S. Snyder, M. R. Ford.

- E. Preparation of Reagents and Extraction of Uranium from Urine.
- F. Analytical Procedure and Reagents for Gross Alpha Analysis.
- G. Analytical Procedures and Reagents for Dithiozone Extraction and Colormetric Determination of Lead in Blood and Urine.

VII. Forms Used in Requesting Samples, Recording Data, and Submitting Results.

A. Health Physics Request for Body Fluid Specimen (Appendix I)

The forms shown in Appendix I are filled in by the Health Physics representative in the field. All information requested is necessary to obtain dose data. Copies are distributed as indicated on the blue and yellow sheets.

B. Internal Exposure Work Sheet and Permanent Record (Appendix II)

The form shown in Appendix II is filled in by the Bio-Assays Unit and filed at the Bio-Assays Laboratory. There is a record sheet for each employee submitting a sample.

C. Internal Dose Report for Body Fluid Analysis (Appendix III)

The form shown in Appendix III is filled in by the Bio-Assays Unit and sent to the Personnel Monitoring Group for inclusion in the employees Personnel Monitoring folder. Distributions are made according to information at the bottom of the sheet.

A. LaF_3 Determination of Plutonium in Urine

<u>Reagents</u>	<u>Preparation of Reagents</u>
I. La Carrier (Purified)	1. Dissolve 1.56 grams of purified $\text{La}(\text{NO}_3)_3 \cdot 6 \text{H}_2\text{O}$ in 63 ml of HNO_3 , adding water as necessary and dilute to 1 liter.
II. 3M $\text{Ca}(\text{NO}_3)_2$	2. Dissolve 708 grams $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$ in distilled water and build to 1 liter.
III. 1M Oxalic Acid	3. Dissolve 90.4 grams oxalic acid crystals in distilled water and build to 1 liter.
IV. HClO_4	4. Stock
V. NH_4OH	5. Stock
VI. 10M HF	6. Dilute 37 ml of conc. HF to 100 ml with distilled water.
VII. 1N HNO_3	7. Dilute 62.5 ml of conc. HNO_3 to 1 liter with distilled water.
VIII. HCl	8. Stock

Determination of Plutonium in Urine

- I. Sampling
 1. Measure and record volume of urine specimen and transfer to a 1500 ml. beaker.
 2. Adjust pH to a value of 1.2 to 1.3
- II. Calcium Oxalate Precipitation
 3. Add 0.5 ml of La carrier.
 4. Add 5 ml of 3M $\text{Ca}(\text{NO}_3)_2$.
 5. Add 15 ml of 0.10 M oxalic acid.
 6. Adjust pH to 3 with NH_4OH . Allow precipitate to digest overnight at room temperature.
- III. Separation of Precipitation
 7. Draw off as much of supernate as possible without disturbing precipitate into a vacuum flask. (Save supernate for fission product analysis if requested.)
 8. Slurry calcium oxalate precipitate and transfer to a 100 ml centrifuge cone.
 9. With the aid of a closed end rubber policeman, wash beaker three times with small amount of distilled water and add washing to centrifuge cone.
 10. Centrifuge for 5 to 10 minutes and add supernate to the supernate in step number 7.
- IV. Destruction of Organic HClO_4 and Na BrO_3
 11. Add to the precipitate in the 100 ml centrifuge cone 2 ml of conc. HClO_4 and heat for several minutes in a water bath 100°C .
 12. Add 3 ml of 1M NaBrO_3 .
 13. Heat in boiling water for 1 hour. (After about 20 minutes add 1 ml of water to compensate for evaporation.)
 14. At end of oxidation period solution should be colorless. (It may be clear or slightly cloudy.) (See note.)

- V. $\text{La}(\text{OH})_3$ Precipitation
15. Wash down wall of centrifuge cone with distilled water to a volume of about 12 ml and add 3 ml of NH_4OH . Stir.
 16. Allow $\text{La}(\text{OH})_3$ precipitate to digest 15 to 20 minutes and centrifuge for 5 minutes.
 17. Pour off supernate and transfer precipitate to 40 ml centrifuge cone.
 18. Add 1 drop of NH_4OH . Stir and centrifuge for 5 minutes. Pour off supernate.
- VI. LaF_3 Precipitation
19. Dissolve precipitate with 0.5 ml of HCl and build volume to 10 ml with distilled water.
 20. Add 8 drops of 6M $\text{NH}_2\text{OH}:\text{HCl}$ and allow 10 to 15 minutes for reduction of Pu VI to IV.
 21. Add 0.5 ml of HCl and 2 ml of 10M HF and stir. Allow 5 minutes for digestion.
 22. Centrifuge for 5 minutes and decant supernate.
 23. Wash precipitate with 2 ml of 1N HNO_3 and 1 drop of HF . Centrifuge and decant.
- VII. Mounting the sample for counting
24. Use enough 1N HNO_3 to transfer sample to a platinum disc quantitatively.

NOTE: If solution is not colorless, add 2 ml more of 1M NaBrO_3 and reheat. Should color persist after second addition of NaBrO_3 , add 2 ml of HClO_4 and heat carefully over a flame.

B. Laboratory Directions and Reagents for Radium Analysis

<u>Reagents</u>	<u>Preparation of Reagents</u>
I. Nitric Acid, conc.	1. Stock
II. Nitric Acid, 0.1 M	2. Dilute 6.25 ml of conc. HNO_3 to 1 liter.
III. Lead Carrier solution	3. Dissolve 32 grams of $\text{Pb}(\text{NO}_3)_2$ in water and dilute to 100 ml.
IV. Barrium Carrier	4. Dissolve 1.9 grams of $\text{Ba}(\text{NO}_3)_2$ in water and dilute to 100 ml.
V. Sulfuric Acid, conc.	5. Stock
VI. Sulfuric Acid, 0.5 M	6. Add slowly 278 ml of H_2SO_4 to 500 ml of distilled water. Cool and dilute to 1 liter.
VII. HCl - ether	7. Dissolve 15 ml of diethyl ether in 85 ml of HCl.
VIII. Zapon lacquer	8. Stock

Radium Analysis

- I. Sample Preparation
 - (a) Made acid
 - (b) Evaporated to dryness
 - (c) Destruction of organic material
1. Transfer 50 to 2000 ml of sample to beaker.
2. Rinse original container with 100 ml of conc. HNO_3 and add to sample.
3. Evaporate to dryness.
4. Cool and cover residue with conc. HNO_3 and evaporate slowly. Repeat until only a white residue remains.
5. Add 150 ml of .1 M HNO_3 and warm for 5 minutes.
- II. Separation of solution from residue
6. Transfer to a 250 ml centrifuge bottle and save beaker.
7. Centrifuge and decant supernatant solution to a second 250 ml centrifuge bottle. Save both residue and solution.
8. Rinse original beaker with 50 ml of .1 M HNO_3 and pour washings into first centrifuge bottle. Stir, then centrifuge and decant supernatant solution to second centrifuge bottle.
- III. Lead sulfate Precipitation
 - (a) H_2SO_4
 - (b) Lead Carrier
 - (c) Cool in ice bath
9. Stir solution with a mechanical stirrer. Add 3 ml of H_2SO_4 .
10. Add dropwise 0.5 ml of lead carrier solution.
11. Cool in ice bath for 15 minutes with stirring.
12. Centrifuge and discard solution.
- IV. Barium Sulfate Precipitation
 - (a) Dissolve $\text{Pb}(\text{SO}_4)$ in HCl -ether
 - (b) Cool and add barium carrier
13. Add 15 ml of HCl -ether reagent to lead sulfate precipitate and warm gently to hasten solution.

14. Transfer solution to a 50 ml centrifuge tube using a total of 10 ml of HCl-ether reagent to complete transfer.
15. Cool in an ice bath and add 0.4 ml of Barrium carrier dropwise while stirring. Continue to stir for 5 minutes.
16. Centrifuge for 5 minutes and discard solution.

V. Mounting of sample for counting

- (a) Slurry precipitate with
 .1 M HNO_3
- (b) Transfer to platinum disc
- (c) Evaporate with H_2SO_4
- (d) Flame disc
- (e) Count

17. Add 1 ml of 0.1 HNO_3 and make slurry.
18. Transfer slurry to a platinum disc ringed with Zapon lacquer.
19. Evaporate under an infra-red lamp for 2 to 3 minutes.
20. Add 4 drops of 0.5 M H_2SO_4 and evaporate to dryness.
21. Flame the disc carefully over a small flame to burn off Zapon, then flame to a red heat.
22. Count, if activity is greater than 1 count per minute, count at one or two day interval to determine which isotope is present.

C. Polonium Analysis by Internal Electrolysis

Urine samples are made 1 N in HCL, and the polonium is plated onto clean copper discs suspended in the solutions. The alpha activity is determined by counting both sides of the discs.

REAGENTS

Copper disc - $3/4$ inch in diameter with a $3/16$ hole near the circumference.

Ethyl Alcohol

HCL, 6N

I. Preparation of copper disc for plating.

Clean with alcohol.

1. Heat copper disc to redness and drop into a beaker of ethyl alcohol.
2. Allow complete reaction.
3. Remove from alcohol and allow disc to dry.

II. Preparation of urine specimen for analysis.

Make specimen 1N in HCL.

4. Measure 50 ml of urine into a 100 ml beaker.
5. Add 10 ml of 6N HCL.
6. Suspend disc in solution.
7. Stir by means of a magnetic stirrer for 2 hours.
8. Remove disc from solution and rinse with distilled water.
9. Dry under an infra-red lamp.

III. Counting

10. Count both sides of disc.
Sum of two sides is equal to gross counts per minute.

D. Reagents, Equipment and Procedures for Radiochemical Analysis of Strontium and Barium in Urine.

Reagents

- I. Concentrated HCl (12M)
- II. 6M Phosphoric Acid
- III. 6M Sodium Hydroxide
- IV. Concentrated HNO_3
- V. 1M Sodium Hydroxide
- VI. Eriochrome Black T.
- VII. Versene Solution (7.5%)
- VIII. 1.0N Hydrochloric Acid
- IX. Citric Acid - Versene Solution
- X. 6N Nitric Acid
- XI. 1N Nitric Acid
- XII. Dowex-50 x 12 50-100 mesh

Preparation of Reagents

- 1. Stock item.
- 2. 434 ml. of 85% H_3PO_4 made to 1 liter.
- 3. 240 grams Sodium Hydroxide pellet dissolved in 600 ml. of distilled water. Cool and make to 1 liter.
- 4. Stock item.
- 5. 40 grams Sodium Hydroxide pellets dissolved in 600 ml of distilled water and made to 1 liter.
- 6. Mix 0.5 grams of Eriochrome Black T with 4.5 grams Hydroxylamine Hydrochloride and dissolve in 100 ml alcohol. Filter.
- 7. Dissolve 75 grams of Tetrasodium Ethylenediamine Tetracetic Acid in 800 ml distilled water, filter and make to 1 liter.
- 8. Dilute 83.4 ml of concentrated HCl to 1 liter.
- 9. 10.93 grams of Citric Acid Monohydrate and 100 ml of 7.5% Versene solution are made to 1 liter and pH is adjusted to 5.0 with 6N Sodium Hydroxide.
- 10. Dilute 375 ml of concentrated HNO_3 to 1 liter.
- 11. Dilute 62.5 ml of concentrated HNO_3 to 1 liter.
- 12. See column preparation for conditioning of resin.

Column Preparation

- I. Conditioning of the Resin
 1. Wash dowex-50 with 5% NaCl solution and 5% HCl solution. Repeat this step several times allowing most of resin to settle before decanting washes.
- II. Resin converted to sodium cycle.
 2. About 250 grams of conditioned resin is put into a pyrex tube 4 ft. long, 2.5 cm diameter.
 3. Wash resin in pyrex tube with 6 liter of 5% NaCl at a flow rate of 4.7 ml/min/cm².
 4. Transfer resin from tube to a large beaker and wash free of NaCl with distilled water.
- III. Preparation of column.
 5. Transfer 16.3 grams of air dried resin or 25 ml. wet form to a column prepared as follows:
 - (a) The bottom of a pyrex glass tube, 18.5 cm long and 1.8 cm in diameter is filled with a one way stop cock in a one hole rubber stopper. A six inch pyrex glass funnel is fitted to the top with rubber tubing.

Procedure for Strontium and Barium Analysis

- I. Sample preparation:
Make acid with HCl and heat
to clear solution.

1. Make solution 0.1 N in HCl to a 1500 ml. sample. Add 12.5 ml. concentrated HCl, that is 0.85 ml. per 100 ml of urine.
2. Heat sample to a temperature of 85° - 90° C.

- II. Co-precipitation of
Strontium and Barium with
alkaline earth phosphates
from an alkaline solution.
Add excess of H_2PO_4 .
Adjust pH to 8-10 with
6N NaOH.

3. Add 6 ml of 6M phosphoric acid with mechanical stirring.
4. Add slowly 6M sodium hydroxide to a pH of 8.0 - 10.0 using universal pH paper as an indicator. Continue stirring with addition of sodium hydroxide.
5. Remove stirrer and allow precipitate to settle for 2 hours or longer.

- III. Isolation of Sr, Ba alkaline
earth precipitate.
Decant.
Transfer to centrifuge tube.
Centrifuge discarding supernate.

6. Decant, by means of suction, as much of supernate as possible without disturbing precipitate. Discard supernate.
7. Transfer slurry to a centrifuge cone and centrifuge at 1500 rpm for 5 minutes. Decant and discard supernate liquid.

IV. Destruction of the remaining organic material.

HNO_3 - H_2O_2 treatment.

8. Dissolve precipitate in approximately 15 ml. of concentrated HNO_3 .
9. The solution is poured into the two liter beaker in which phosphate precipitation was made.
10. Heat on hot plate adding concentrated HNO_3 and 30% H_2O_2 alternately until only a white residue remains. Take residue to dryness.
11. Remove beaker from hot plate and wash down walls with about 20 ml. of distilled water. Add 2 to 3 ml. of concentrated HCl and heat until inorganic salts dissolve. Make volume to 800 ml. with distilled water.

V. Preparation of solution for ion exchange separation.

Adjust pH to 9.0.

Chelate alkaline earths with versene.

Adjust pH to 5.5

12. Adjust pH to about 9.0 with 1.0 N sodium hydroxide.
13. Add 2 ml. of eriochrome black T indicator solution.
14. Add versene solution (7.5%) until indicator changes from wine red to blue at a pH of 10.5, this indicates complete chelation of all calcium and magnesium.
15. Adjust pH of solution to 5.5 with 1.0 N HCl .
16. Pass solution through resin column (dowex-50) at a flow rate not greater than 8 ml/min/cm².
17. Wash down the walls of funnel with 50 ml. of distilled water and discard the effluents.

VI. Elution of adsorbed calcium and magnesium from resin column. 0.75% versene - 1% citric acid elutant.

VII. Elution of Sodium from calcium. 0.5 N HCl elutant.

VIII. Elution of Barium and Strontium from resin column. 6 N HNO₃ elutant.

IX. Preparation of sample for counting:
Transfer after evaporation to 304 type stainless steel disc for conventional beta counting.

X. Standardization of counter.

18. Pass 800 ml. 4.0 = .8 ml/min/cm² of citric acid-versene solution through column. Discard elutant.

19. Pass 800 ml. of 0.5 N HCl through resin column at a flow rate of 4.0 = 0.8 ml/min/cm². Discard effluents wash.

20. Pass 200 ml. of 6 N HNO₃ through the resin column at a flow rate of 2 ml/min/cm². Collect eluate in a 400 ml beaker.

21. Evaporate HNO₃ eluate to almost dryness on hot plate.

22. Wash into a 50 ml beaker with distilled water. Wash sides of 400 ml. beaker with HNO₃ and water adding wash to 50 ml. beaker.

23. Evaporate to dryness in 50 ml. beaker.

24. Dissolve residue in .5 ml. of 1 N HNO₃ and transfer to a counting dish.

25. Wash sides of beaker with another .5 ml of 1 N HNO₃ adding this to counting dish.

26. Dry under an infra-red lamp.

27. a. Count Sr standard sample 20 min. on first shelf.

b. Count background 20 minutes.

c. Determine counting efficiency $C = \text{cm/dm}$ of standard.

28. Sample counts for 20 minutes on first shelf of beta counter and determine net counts.

E. Preparation of Reagents and Extraction of Uranium From Urine

<u>Reagents</u>	<u>Preparation of Reagents</u>
I. 3% TBP in Hexane	<ol style="list-style-type: none">1. Add 30 ml of tributyl phosphate to a 2 liter separatory funnel.2. Add 970 ml of Hexane.3. Shake three times with 500 ml. 0.2 N HNO_3 discarding HNO_3 wash each time.4. Shake once with 500 ml. distilled water. Discard wash water.5. Shake TBP with 500 ml. of 0.25 N Na_2CO_3 three times, discarding carbonate each time.6. Wash TBP five or six times with 500 ml. portions of distilled water. Discard wash water.7. Store in convenient bottle.

Extraction of Uranium from Urine Using TBP

- I. Sampling:
100 ml. urine
20 ml. HNO_3
 - II. Destruction of organic.
 HNO_3 ; H_2O_2 treatment.
 - III. Preparation of solution for
TBP extraction.
Make 2 M in $\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$
and 1 M in HNO_3 .
 - IV. Extraction of Uranium with TBP.
Three extractions with 20 ml. of
3% TBP in Hexane.
1. Measure 100 ml. of urine into a 400 ml. beaker.
 2. Add 20 ml. of conc. HNO_3 .
 3. Cover with a speedvap watchglass and heat to dryness.
 4. Add 2 to 3 ml. of conc. HNO_3 , take to dryness.
 5. Add 2 to 3 ml. of H_2O_2 , take to dryness.
 6. Repeat steps 4 and 5 until there is only a white ash residue.
 7. Wash down side of beaker and watch glass with distilled water.
 8. Add 0.75 ml. of conc. HNO_3 and evaporate to a volume of 4 to 5 ml.
 9. Add 15 grams of $\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$ and warm. DO NOT BOIL.
 10. Transfer to a 50 ml glass stoppered graduate. Rinse beaker with .75 ml of HNO_3 and 2 to 3 ml. of distilled water. Volume should be 20 ml. $\pm$ 2 ml.
 11. Add 20 ml of 3% TBP in Hexane and shake for 2 minutes.
 12. Allow 15 minutes for phase separation.
 13. Transfer organic phase by means of a suction pipette to a 125 ml separatory funnel.
 14. Repeat steps 12 and 13 twice.

V. Stripping Uranium from TBP solution. Make four strips with 20 ml. portion of 0.1 N HNO_3 .

15. Add to the 60 ml. TBP solution in 125 ml. separatory funnel 20 ml. of 0.1 N HNO_3 .
16. Shake for 1/2 minute and allow 2 minutes for phase separation and draw off aqueous phase into a 100 ml. beaker.
17. Repeat steps 15 and 16 three times.

VI. Preparation of sample for counting.

18. Evaporate aqueous solution to near dryness.
19. Wash down wall of beaker with 2 to 3 ml. of HNO_3 and evaporate to near dryness.
20. Transfer to stainless steel disc. Rinse beaker with 10 drops of .1 HNO_3 .
21. Dry under heat lamp and flame to a dull red color.
22. Count alpha activity in sample.

F. Analytical Procedure and Reagents for Gross Alpha Analysis

<u>Reagents</u>	<u>Preparation of Reagents</u>
I. HNO_3 , conc.	1. Stock
II. HNO_3 , 6 M	2. Dilute 37.5 ml of conc. HNO_3 to 100 ml.
III. HNO_3 , 2 M	3. Dilute 12.5 ml of conc. HNO_3 to 100 ml.
IV. SO_2 water	4. Saturate a volume of distilled water with SO_2 by bubbling SO_2 gas through it.
V. .1 gms/ml Bi solution	5. Dissolve 232 grams of $\text{Bi}(\text{NO}_3)_3 \cdot 5 \text{H}_2\text{O}$ in 630 ml. of HNO_3 and dilute to 1 liter with distilled water.
VI. H_3PO_4	6. Stock
VII. HCl	7. Stock
VIII. 1:1 HF	8. Dilute 50 ml. of conc. HF to 100 ml with distilled water.
IX. 10% NH_4OH	9. Dilute 10 ml of NH_4OH to 100 ml. with distilled water.
X. 0.5 mg/ml La solution	10. Dissolve 1.56 grams of $\text{La}(\text{NO}_3)_3 \cdot 6 \text{H}_2\text{O}$ in 63 ml of HNO_3 , adding water as necessary. Dilute to 1 liter.

Analytical Procedure for Gross Alpha

I. Preparation of sample:

- (a) Measure and record volume of sample.
 - (b) Add HNO_3 and evaporate to dryness.
 - (c) Destroy organic with HNO_3 .
1. Transfer 300 to 2000 ml. of urine sample to beaker, rinse original container with 100 ml. of HNO_3 and add to beaker.
 2. Cover with watch and evaporate to dryness.
 3. Cool and cover with HNO_3 , evaporate to dryness slowly. Repeat until only white residue remains.
 4. Add 2 ml. of HNO_3 to white residue, allow to soak in, and 35 ml. of water to dissolve residue. A small amount of insoluble matter remains suspended in solution.
 5. Transfer to a 50 ml. centrifuge cone and centrifuge at 2000 RPM for 5 minutes.
 6. Wash down sides of 50 ml. cone with 1 ml. of HNO_3 and 2 ml. of H_2O . Slurry the residue, centrifuge and decant washing to 90 ml. centrifuge cone.

II. BiPO_4 precipitation:

- (a) Warm to $85-90^\circ \text{C}$
- (b) Add Bi carrier
- (c) Add H_3PO_4
- (d) Digest at $85-90^\circ \text{C}$

7. Dilute the solution to 80 ml, add 2 ml. of SO_2 water, stir and let stand for 20 minutes.
8. Place in water bath at $85-90^\circ \text{C}$. Stir vigorously with stirring motor, allow solution to reach bath temperature and add dropwise 1 ml. of Bi carrier solution.
9. Add dropwise 1 ml. of H_3PO_4 .
10. Allow BiPO_4 precipitate to digest with constant stirring at $85-90^\circ$ for 1 hour.
11. Remove from bath and let BiPO_4 settle. Centrifuge if necessary. Discard supernatant solution.

III. LaF_3 precipitation

- (a) Transfer to 15 ml. cone.
- (b) Dissolve in HCl
- (c) Add La carrier
- (d) Add 1:1 HF

- 12. Transfer the BiPO_4 and remaining 1/2 ml. of liquid to a 15 ml. centrifuge cone.
- 13. Wash sides of 90 ml. tube with distilled water and add to 15 ml. cone.
- 14. Centrifuge 15 ml. centrifuge cone and discard supernatant.
- 15. Wash the 90 ml. tube with 10 drops of HCl and 10 drops of water and add to precipitate in 15 ml. cone.
- 16. Repeat step 14 and stir until BiPO_4 is dissolved.
- 17. Dilute solution to 5 ml. and add 0.5 ml. of $\text{La}(\text{NO}_3)_3$ to solution. Stir.
- 18. Add 0.5 ml. of 1:1 HF and stir. Centrifuge 5 minutes and discard supernatant.
- 19. Wash precipitate with 5 ml. of a solution 1 M in HF and 1 M HNO_3 . Centrifuge and discard washings.

IV. Plate the sample for counting.

- 20. Transfer precipitate to stainless steel counting disc using 2 M HNO_3 .
- 21. Dry under an infra-red lamp and flame.
- 22. Count for alpha.

G. Analytical Procedure and Reagents for Dithiozone Extraction and Colormetric Determination of Lead in Blood and Urine

<u>Reagent</u>	<u>Preparation of Reagent</u>
I. 25% Sodium Citrate Solution.	<ol style="list-style-type: none"> 1. Dissolve 250 grams of sodium citrate (C.P.) in demineralized water, dilute to 1 liter, filter and purify by extraction with dithiozone. (a) Purification by extraction. <ol style="list-style-type: none"> 1. Filter sodium citrate into a 2 liter separatory funnel. 2. Add 1 ml. of conc. NH_4OH. 3. Add 10 ml. of dithiozone and shake, discard dithiozone, repeat until color of dithiozone remains green after extraction. 4. Add 10 ml. of conc. HCl. 5. Wash with 20 ml. portions of chloroform until no more dithiozone (green color) appears in chloroform. 6. Add 10 ml. of conc. NH_4OH, and shake several times with 20 ml. portions of chloroform, discard chloroform. 7. Let stand overnight until solution clears.
II. Phenol Red Indicator	<ol style="list-style-type: none"> 2. Dissolve 1 gram of phenol red in 50 ml. of .05 N NaOH and dilute to 100 ml.
III. 10% Potassium Cyanide	<ol style="list-style-type: none"> 3. Dissolve 100 grams of KCN in demineralized water, make to 1 liter and filter.
IV. Dithiozone 1 gram/liter purified.	<ol style="list-style-type: none"> 4. (a) Dissolve 400 to 500 mg of dithiozone in 200 ml. of carbon tetrachloride in a 500 ml. glass stoppered graduate cylinder. (b) Shake with three 200 ml. portion of 1% NH_4OH and filter aqueous into a 1 liter beaker. (c) Make aqueous slightly acid with 1 N HCl. (d) Filter through three filter paper in a Buchner funnel with vacuum. (e) Wash filtrate one time with water and dry overnight in a desicator. (f) Weight and dissolve in enough chloroform to make a solution of 1 mg/ml.

V. Dithiozone: 20 mg/ml	5. Dilute 20 ml of 1 mg/ml dithiozone to 1 liter with CHCl_3 . (Store in refrigerator.)
VI. 1% HNO_3 with methyl orange indicator.	6. Dilute 10 ml. of conc. HNO_3 and 2 ml. of 1% methyl orange to 1 liter.
VII. Methyl orange indicator	7. Dissolve 1 gram of methyl orange in 100 ml. (filter).
VIII. 1 N HNO_3	8. Dilute 6.25 ml. of conc. HNO_3 to 100 ml. with demineralized water.
IX. 1 N NH_4OH	9. Dilute 6.70 ml. of conc. NH_4OH to 100 ml. with demineralized water.
X. Solution A	10. Mix 1430 ml. of 14% NH_4OH with 286 ml. of 1% sodium sulfate and 286 ml. of 2% potassium cyanide.
XI. 14% ammonium hydroxide	11. Mix equal quantities of 28% NH_4OH and demineralized water.
XII. 1% sodium sulfide	12. Dissolve 1 gram of sodium sulfide in 100 ml. of demineralized water and filter.
XIII. 2% potassium cyanide	13. Dilute 200 ml. of purified 10% KCN solution to 1 liter with demineralized water.
XIV. 10% calcium chloride	14. Dissolve 100 grams of CaCl_2 in 1 liter of demineralized water, purify by shaking with dithiozone. Wash out dithiozone with chloroform.
XV. Saturated Mono Basic Ammonium Phosphate	15. Dissolve 58 grams of $(\text{NH}_4)_2\text{HPO}_4$ in 100 ml. of demineralized water.
XVI. Phenolphthalien 1%	16. Dissolve 1 gram of phenolphthalien in 50 ml. of absolute alcohol and dilute to 100 ml. with demineralized water.
XVII. Sulferdioxide	17. Stock

Preparation of Urine Sample for Dithiozone Extraction

- I. Sampling:
 - (a) measure volume
 - (b) Take specific gravity
 - II. Phosphate Precipitation
 - (a) mono-ammonium phosphate
 - (b) make alkaline
 - (c) digest 1 hr.
 - (d) discard supernatated solution
 - III. Destruction of Organic $\text{HNO}_3 - \text{H}_2\text{O}_2$
1. Measure 100 ml. of urine.
 2. Take specific gravity.
 3. Transfer sample to 250 ml. centrifuge cup.
 4. Add 3 or 4 drops of conc. HNO_3 .
 5. Add 1 ml. of 10% calcium chloride.
 6. Heat to approximately 60°C , stir and add 1 ml of saturated mono-ammonium phosphate.
 7. Add 2 drops of phenolphthalien and make alkaline with NH_4OH .
 8. Digest 1/2 to 1 hour, centrifuge and decant supernatant by suction.
 9. Add 3 to 4 ml of conc HNO_3 and heat on hot plate.
 10. Add 30% H_2O_2 occasionally until digestion of organic is complete.
 11. Make volume to approximately 35 ml with demineralized water.
 12. Cool and pass SO_2 through solution for 5 minutes. Let stand for 30 minutes or longer.
 13. Analyze for lead by dithiozone extraction procedure.

Preparation of Blood for Dithiozine

Extraction of Lead

- I. Sampling:
 - (a) weight specimen
 - (b) transfer to beaker
 - II. Oxidation of Organic
 - (a) HNO_3 to a yellow color
 - (b) 2 ml H_2SO_4 and char
 - (c) HNO_3 dropwise until charring disappears
 - (d) HClO_4 for final oxidation
 - III. Reduction of Fe^{+3}
 - (a) pass SO_2 through solution
 - (b) let stand for 2 hours
1. Weigh whole blood, approximately 10 grams, transfer to 250 ml beaker. Was weighing vessel with demineralized water and add ashings to sample.
 2. Add 20 ml of conc. HNO_3 .
 3. Heat at low temperature on hot plate. Caution: oxidation of organic material in blood will generate excess heat and may cause spattering.
 4. Add 10 ml more of HNO_3 and continue heating until solution becomes a yellowish color.
 5. Add 2 ml of H_2SO_4 and heat until material chars.
 6. Add HNO_2 drop wise until charring disappears.
 7. Cool and add 10 drops of perchloric acid, shake and heat cautiously. Continue adding perchloric acid until organic is destroyed.
 8. Cool and wash down side of beaker with 30 ml of demineralized water.
 9. Heat to dissolve all solids.
 10. Pass sulfur dioxide through solution for 5 minutes and let stand for 2 hours.
 11. Analyze for lead by dithiozone extraction method.

Dithizone Procedure for Lead

- I. Extraction of Lead:
 - (a) Sodium citrate buffer
 - (b) Make alkaline
 - (c) Potassium cyanide complex
 - (d) Extract with dithiozone
1. Add 25 ml of 25% solution of sodium citrate to a 125 ml. separatory funnel.
2. Add sample and 2 drops of phenol red indicator.
3. Make alkaline to phenol red with NH_4OH .
4. Add 3 ml. of 10% potassium cyanide.
5. Add 10 ml. of 20 mg/ml dithiozone in chloroform and shake for 1 minute.
6. Drain dithiozone layer in another funnel with 25 ml. of 1% HNO_3 with methyl orange indicator.
7. Extract original solution with 5 ml. more of dithiozone and add to step 6.
- II. Stripping Lead back to Aqueous with Acid Solution (HNO_3)
 - (a) Adjust pH slightly acid
 - (b) Shake
 - (c) Discard dithiozone
 - (d) Wash with CHCl_3 and discard CHCl_3 .
8. Adjust pH of HNO_3 acid strip as follows:
 - (a) Add conc. NH_4OH to a yellow color.
 - (b) Make final adjustment with 1 N HNO_3 such that one drop of HNO_3 gives a definite pink and 1 drop NH_4OH gives a definite yellow.
 - (c) Make acid with 1 drop HNO_3 and add 2 drops excess.
9. Shake for 1 minute, allow phases to separate and discard dithiozone.
10. Add 5 ml. of CHCl_3 , shake and discard CHCl_3 .
- III. Final Extraction of Lead in Dithiozone:
 - (a) Add reducing agent. and complex. (Solution A)
 - (b) Extract with 10 ml. dithiozone.
 - (c) Filter and determine absorbency of transmittance of solution.

11. Add 50 ml. of solution A and 10 ml. of dithiozone.
12. Shake for one minute.
13. Filter dithiozone phase through No. 1 paper into a clean, dry, curvette.
14. Read against a similarly prepared blank one Beckman Model D.U. spectrophotometer at 510 M.

28

DAYS AFTER EXPOSURE

% OF INITIAL BODY BURDEN ELIMINATED PER 24 Hr SPECIMEN
SINGLE EXPOSURE EXCRETION DATA FOR Po^{210}

ORNL-18-DWG.-23106 UNCLASSIFIED

280

GROUP 1

29

-29- ORNL-IR-D.S.-23105 UNCLASSIFIED

GRAPH II

30

•30•
ORNL-IR-DWG-23106
UNCLASSIFIED

GRAPH III

100

10

1

31

ORNL-R-DWG.-23107
UNCLASSIFIED

-31-

% OF INITIAL BODY BURDEN ELIMINATED PER 24 HR SPECIMEN
SINGLE EXPOSURE EXCRETION DATA FOR U(NOT)

X-463 (Rev. 1-57)

HEALTH PHYSICS REQUEST FOR BODY FLUID SPECIMEN

Name _____	Payroll No. _____
Division or Dept. _____	Supervisor _____

It is recommended that the above named individual submit a _____ A.M.
specimen, to be collected for a _____ hour period ending by _____ P.M.
on _____. The specimen should be left at _____ as
soon as practical after the sampling period.

Requested by _____ Date _____
Health Physics Representative

Analyze for:

- ☐ H³
☐ Po
☐ Pu
☐ Ra
☐ Sr
☐ U
☐ _____
☐ Gross α
(Th, Pu, Am, Cm)
☐ Gross β
(including K⁴⁰)

Requested because of:

- ☐ Chronic exposure: from _____ to _____
☐ Single exposure of _____ hours beginning
_____ A.M.
_____ P.M. on _____
☐ Routine control measure
☐ Other (explain) _____

☐ Resample on specimen submitted _____
(date)

The method of intake (if any) was probably due to:

- ☐ Inhalation ☐ Injection
☐ Ingestion ☐ _____

- (1) White copy to person submitting sample
(2) Blue copy to body fluid analysis
(3) Yellow copy to file

535-036

Division or Department: _____ Supervisor: _____

[illegible]³Inhalation (A) Ingestion (B) Injection (C).

1037-85-27

INTERNAL DOSE REPORT FOR BODY FLUID ANALYSIS

Name: _____	Payroll No: _____
Division or Dept: _____	Supervisor: _____

Internal dose data accrued for the above named individual should be added to the Personnel Exposure Record in accordance with the calculations shown in the table below:

Period	Dose in Mrems				Remarks
	D _s	D _m	D _l	D _p	

Special Instructions: _____

Assays-Instruments Supervisor

Noted: _____ _____	Personnel Exposure Record Adjusted _____ (Date)
	Approved as Noted: _____ (Pers. Mon. Supv.)

Copies: Requestor (2), AHP-Personnel Monitoring File.

535-038

I-IV AND
1-34