

December 27, 1957

Health Protection Branch
Division of Biology and Medicine
U. S. Atomic Energy Commission
Washington 25, D. C.

Attention: Mr. Allen Brodsky

Dear Mr. Brodsky:

As per the request of J. C. Pollard, Assistant Manager, San Francisco Operations Office to Mr. Stafford L. Warren, we have compiled the following bioassay procedures that we use as needed in our operations. The procedures were taken from existing urinalysis reports and modified to meet the needs and capabilities of this project. In each case a preliminary study of available procedures were made to enable urinalysis to be done as needed.

On this project nearly all of the bioassay activities have involved urinalyses with only a few fecal analyses being done as courtesy consultations for other government agencies. Although we have not done any activity blood analyses a background of experience in this phase of bioassay procedures was obtained during the Manhattan project period and could be done here if needed.

The attached compilation will be numbered to correspond with the item numbers in your letter. We hope that a copy of the compilation from all the projects will be available or put out as a TID report since they may save time and duplication of effort on the problems of bioassay techniques.

Sincerely yours,

BEST COPY AVAILABLE

Louis S. Silverman, Chief
Health Physics Section

LUS:lmw

cc: Dr. J. C. Pollard
Mr. S. L. Warren ✓
Mr. H. E. Huslaun
file

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NOTES	4-5-DEL
FOLDER NAME	1000-ACC-FBI-UCI-K-GENERAL-1057
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SERIES NUMBER	SERIES 300, SUBSERIES 1000
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1. bioassay procedures for the elements Pu, Na, Sr, and U are as follows:

Analytical Methods

(A) For Plutonium a modification of the method described in report No. DP-146 (1) is used which enables this project to run Pu²³⁹ analysis with a 65% recovery factor.

The steps we follow in the analytical procedures are:

1. Place a 24 hour urine specimen into 2 L. beaker and make 0.2N with HNO₃.
2. Heat to 65° C.
3. Make 0.1N in H₃PO₄.
4. Add 10 ml Bi(NO₃)₃ dropwise from sep. funnel and stir for 30 min.
5. Allow to stand overnight at room temperature.
6. Aspirate all but approx. 25 ml. of supernatant.
7. Add 23.5 ml HNO₃ (Conc.).
8. Add 10 ml 30% H₂O₂ and cover with watchglass.
9. Heat until reaction stops, adding more H₂O₂ if needed.
10. Cool and then add 2 ml H₂SO₄, allow to stand for 15 min.
11. Add 1500 ml (65°C) distilled H₂O.
12. Add dropwise 7.5 ml 85% H₃PO₄ while stirring.
13. Stir for 30 min. then allow to stand for 2 hrs.
14. Aspirate all but 25 ml of supernatant.
15. Slurry and transfer to 50 ml centrifuge tube.
16. Centrifuge at 2000 rpm for 10 min., decant supernatant.
17. Add 4 ml (conc) HCl to orig. 2 L. beaker, cover with watchglass and heat to wash sides.
18. Pour contents into cent. tube and wash down sides with 1 ml h.l.
19. Add 1 mg La carrier and 1 ml 40% H₂ to each 6 ml liquid.

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20. Stir 5 min, centrifuge at 2000 rpm 10 min., decant.
21. Add 0.2 ml 60% HClO₃ fume cautiously over burner while shaking.
22. Cool and add 0.5 ml conc. HCl, increase volume to 5 ml with H₂O
23. Add 0.4 ml NH₂OH·HCl, allow to stand 15 min.
24. Make 2N in HF and stir occasionally for 5 min.
25. Centrifuge at 2000 rpm for 10 min.
26. Wash once with 1 N. HNO₃-HF solution.
27. Slurry ppt. and transfer to planchet for counting.
28. Flame and then cool.
29. Count in low background alpha counter.

Reagents for Pu Analysis

HNO₃

H₃PO₄

Rf (NO₃)₃ (232 gm. Bi(NO₃)₃·5H₂O in 1L 10N HNO₃)

H₂O₂

H₂SO₃ (freshly prepared by saturating cold H₂O with SO₂)

HCl

Ia carrier (1 mg Ia per ml H₂O = 3.12 gm Ia(NO₃)₃·6H₂O per l. H₂O)

HClO₃

NH₂OH·HCl (6N Hydroxylamine Hydrochloride)

- (R) For Radium essentially the methods described in Report No. ANL 4509 (2) on pages 12-14 inclusive are used at this laboratory.
- (C) For Strontium a modification of the method described in Report No. ORNL-155 is used which enables this project to run urine analysis for Sr with a recovery factor of 75%.

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The steps we follow in the analytical procedures are:

1. A 750 ml aliquot of a 24 hr. urine specimen is placed in a 2 l. beaker.
2. Add dropwise 75 ml Sulkowitch Reagent from a sep. funnel while stirring.
3. Continue to stir 15 min. after all the reagent is added.
4. Cover the beaker with watch glass and allow to stand overnight.
5. Aspirate the supernatant.
6. Slurry ppt., transfer to special centrifuge tube. *
7. Centrifuge at 2000 rpm for 15 min., discard supernatant.
8. Wash down beaker with 0.05% NH_4OH solution and policeman.
9. Add washings to same centrifuge tube and repeat #7.
10. Rinse ppt. twice in cent. tube with 0.05% NH_4OH and repeat #7.
11. Remove bottom from special cent. tube * and dry slowly with infrared lamp.
12. Count in a standard type beta gamma counter.

Reagents for Sr Analysis

Sulkowitch Reagent: 25 gm oxalic acid
 25 gm ammonium oxalate
 50 ml glacial acetic acid
 Stir with 600 ml distilled water at 40-50°C
 until dissolved, cool, make up to 750 ml and filter

0.05% NH_4OH Solution: 0.5 ml conc. ammonia diluted to 1 liter.

* The special centrifuge tubes in which the bottom of the tube is a removable counting planchet. Available from The Atomic Center New York, N. Y.

(D) For Uranium we are currently using a modification of the method suggested by private letter from George W. Royster, Jr. of ORNL Health Physics Division. His method is a modification of

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a method reported in NLC0-595 (4). Our modification consisted mainly in modifying and simplifying the electrodeposition apparatus needed. Results to date have been better than 95% from spiked samples.

The following are the steps of this analysis:

1. To the urine sample in the collection bottle add NaHCO_3 2 grams to 100 ml. of urine.
2. Raise pH to 9-10 with NaOH.
3. Allow to stand for 2-3 hrs, remove 20 ml. aliquot.
4. Centrifuge 15 min. at 2000 rpm.
5. Decant into 150 ml. beaker.
6. Wash ppt. with 20 ml. 5% NH_4OH - 2% NaHCO_3 solution.
7. Centrifuge 15 min. at 2000 rpm, combine supernatants, discard ppt.
8. Add 20 ml. conc. HNO_3 and 5 ml. conc. HCl to supernatants.
9. Evap. to dryness on slow heat.
10. Cool and add 5-10 ml. H_2O_2 and evap. to dryness.
11. Cool and add 5-10 ml. conc. HNO_3 evap. to dryness, repeat until white.
12. Dissolve white ash in 0.1N HNO_3 .
13. Add 20 ml. of Special Plating Buffer solution * to plating cell ** and then transfer sample to cell.
14. Adjust pH to 4.5 to 5.5 (green to brown creosol green)
15. Plate at 2.0 amp until solution reaches 97°C.
16. Lower to 1.0 amp and plate for 1 hr., maintain temp. at 97°C with external electric heater. ***
17. Remove plate, rinse with distilled H_2O , dry under infrared lamp.
18. Count in low background alpha counter.

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Reagents for Uranium Analysis:

NaHCO_3

NaOH

NH_4OH

HNO_3

HCl

H_2O_2

* Special Plating Buffer Solutions:

29 grams $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$

25 grams NaH_2PO_4

0.218 gms $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$

1 gm brom cresol green

1 liter H_2O (distilled)

Special Apparatus:

** The Plating cell consists of an 8 oz. wide mouth bottle from which the bottom half is cut off. The plastic screw cap is drilled to allow a brass cathode connection. Gaskets are cut from 5 mil. polyethylene sheeting and standard flat stainless steel 2 in. diam. counting discs are used for cathode. A rotating Pt anode is used as a stirrer in this cell.

*** The external electric heater needed to maintain the solution temperature at 97°C . was made by bending a light piece of copper sheet around the cell in the form of a jacket. Wrapping it with asbestos paper for insulating the chromal resistance wire winding which in turn is covered with more asbestos paper and tied down with asbestos string. This jacket is connected to a Variac which controls the current and resulting temperature.

In addition, a stable and sufficient D.C. power supply was obtained from a standard Heathkit Battery Eliminator with a more sensitive milli-amp meter connected in the plating circuit.

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2. Sample collection procedures are enclosed for the standard type of "spot" urine specimen. When twenty-four hour specimen collections are required a standard 2 liter pyrex Erlenmeyer flask with a Mylar wrapped rubber stopper is issued to the individual concerned and he is informed according to circumstances as to method and period of time for the collection. In most cases this type of sample is requested on a weekend with the collection period starting after the first voiding on Sunday morning and collecting all voidings to and including the first voiding on Monday morning. All samples are checked for pH with alkacid test paper upon arrival at the laboratory and acidified so the pH remains on the acid side. The kind of acid added depends upon the subsequent analysis to be performed and should be compatible with the procedure used.

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GENERAL INSTRUCTIONS FOR COLLECTING URINE SPECIMENS
FOR RADIOACTIVITY ANALYSIS

The following instructions must be followed carefully in order that the activity of the urine sample will represent actual excretion rather than external contamination of the sample:

1. Monday morning, before voiding specimen, scrub both hands with an abrasive soap (such as Lava or Boraxo) and hand brush. At least two complete scrubs of about three-minute duration, with complete and thorough rinses.
2. If available, put on clean or new light-weight cotton gloves.
3. For a container, use a clean or new bottle that has not been handled or used near any radioactive materials.
4. The first urine voided on Monday morning should be collected in the clean bottle and sent in for analysis.
5. Be careful not to contact or touch the "lip" or bottle opening at any time (especially during the collection of the specimen).
6. The specimen bottle should be capped and labeled with your name and date of collection and brought into the laboratory for analysis before noon of the same day.
7. The time for collecting the specimen for analysis is set for Monday morning as below, the longest period away from work. Also it is assumed that at least one shower or bath has been taken between the last work period and the time of the specimen collection.
8. Special conditions requiring special precautions will be investigated where indicated and advised accordingly.

L. B. Silverman
L. B. Silverman, Chief,
Health Physics Section

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3. Permissible concentrations of the above elements in urine are calculated from the N.R.C. Handbook 52 data and other A.F.C. reports. Here again, due to the low exposure potential at this Project even when monitoring personnel exposed to minor accidents, spills, etc., the amounts of material which are involved are in nearly all cases far below the maximum permissible levels. We have been using the Los Alamos suggested maximum permissible concentrations as follows for activity urinalyses:

- (a) Plutonium (239) 7 dis/min/24 hr sample
- (b) Strontium (90) 660 dis/min/Liter
- (c) Radium (226) 22 dis/min/24 hr sample
- (d) Uranium 100 dis/min/24 hr sample

The urine to feces ratio as published by Jack Shubert (5) in *Nucleonics* has been used to calculate permissible concentrations for feces as needed.

4. The levels of activity determined at this Project have in all cases except one accidental exposure been at the natural background level or below 5% of tolerance. At 20% of the maximum permissible excretion concentration we prohibit the individual from working in an area where he handles radioactive materials. Our one instance when this level was exceeded (21% of tolerance) was due to an accident with Sr^{90} where the level dropped within one week after the incident to 5% of tolerance.

We have arbitrarily set these levels here because (1) they allow a realistic safety factor with changing tolerance levels and (2) they are easy to achieve in our operations.

5. The relating of bioassay results to concentration of radioactive materials in the body have only been done on the few positive results above the natural background level of urinary excretions and in all cases these have been far less than 0.1 of the body burden permissible level of the isotope involved. No attempts have been made on relating these minute amounts to the internal dose rate.

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REPORTS REFERRED TO IN COMPILATION

1. Determination of Plutonium in Urine
By S. Marshall Sanders, Jr. Report DF-146 (March 1956)
2. The Analytical Procedures of the Bioassay Group at the Argonne National Laboratory
By Jack Schubert, J. S. Myers, Jr. and Jean A. Jackson
Report ANL-4509 (March 1951)
3. The Estimation of Radioactive Strontium and Other Fission Products in Urine and Water
By C. A. Mawson and I. Fischer Report CRM-455 (1950)
4. Proceedings of Bio-Assay and Analytical Meeting Oct. 6-7, 1955
By R. L. Hoover Report NLGO 595
Page 35-44 Electrodeposition of Enriched Uranium in Urine
By Fred Williams (ORNL)
5. Estimating Radioelements in Exposed Individuals -
II - Radiation Dosage and Permissible Levels
By Jack Schubert
Nucleonics Vol 8 No. 3 page 74-77 (March 1951)