

APPENDIX A
RADIOCHEMICAL ANALYTICAL PROCEDURES



ANALYTICAL PROCEDURES MANUAL

ENVIRONMENTAL, Inc.
MIDWEST LABORATORY

prepared for

NUCLEAR MANAGEMENT Co, LLC
KEWAUNEE NUCLEAR POWER PLANT

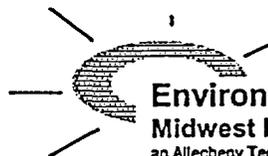
Revised 05-07-02

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WPS

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SAMPLE PREPARATION

PROCEDURE NO. EIML-SP-01

Prepared by

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<u>Revision #</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
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SAMPLE PREPARATION

Principle of Method

Different classes of samples require different preparations. In general, food products are prepared as for home use, while others are dried and ashed as received.

Reagents

Formaldehyde

Apparatus

Balance
Ceramic Dishes
Counting Containers
Cutting Board
Drying Oven
Drying Pans
Grinder
High Temperature Marking Pen
Knives
Muffle Furnace
Plastic Bags
Pulverizer
Scissors
Spatulas

PROCEDURE FOR PACKING STANDARD CALIBRATED COUNTING CONTAINERS

- A. 1.0 L, 2.0 L, 3.5 L: Pour 1.0, 2.0, or 3.5 liters of water into corresponding container. Mark the level and empty the container. Fill with the sample to the mark, except for grass. Pack as much as will fit into the container.
- B. 250 mL, and 500 mL: Fill to the rim on the inside wall, which is 1/4" from the top.
- C. 4 oz: Fill to the 100 mL mark.

Pack the sample tightly. When filling with soil and bottom sediments, make sure they are level.

NOTE 1: For Exelon samples, use a **NEW** counting container for each sample.

NOTE 2: For tritium analysis, transfer approximately 100 g of wet sample to a 4 oz. container. Label with the sample number and seal.

NOTE 3: US Ecology Inc. samples: record total weight received.

NOTE 4: US Ecology Inc. and Maxey Flats samples are **DRIED** before gamma spectroscopic analysis.

NOTE 5: If I-131 analysis is required, the sample must be prepared and submitted to the counting room immediately. Mark "I-131" on the tape.

A. Vegetables and Fruits

1. Cut vegetables and hard fruits into small pieces (about 1/4" cubes). Mash soft fruits.
2. Transfer the sample to a standard calibrated container. Use the largest size possible for the amount of sample available. **DO NOT FILL ABOVE THE MARK.** Record the wet weight.
3. Add a few mL of formaldehyde to prevent spoilage.
4. Seal with cover. Attach label to the top of the cover and record sample number, net weight, and date and time collected.
5. Submit to the counting room for gamma spectroscopic analysis without delay or store in a cooler, (for short period), until counting
6. After gamma scanning is completed, transfer the sample to a drying pan and dry at 110°C.

NOTE 1: If only gamma scan is required, skip drying and ashing (Steps 6 through 10). Transfer the sample to a plastic bag, seal, label, and store in a cooler until disposal.

NOTE 2: If there is sufficient quantity, use surplus sample for drying and ashing instead of waiting for gamma scanning to be completed.

7. Cool, weigh, and record dry weight. Grind.
8. Transfer to a tared ceramic dish. Record dry weight for ashing. Ash in a muffle furnace by gradually increasing the temperature to 600°C. Ash overnight.

NOTE: If ashing is incomplete, (black carbon remains), cool the dish, crush the ash with spatula, and continue ashing overnight at 600°C. At this stage, it is not necessary to increase the temperature gradually. Set the temperature for 600°C and turn on the furnace.
9. Cool and weigh the ashed sample and record the ash weight. Grind and sieve through a 30 mesh screen. Transfer to a 4 oz. container, seal, and record sample number, weight, analysis required, and date of collection. The sample is now ready for analysis.
10. Store remaining ground sample in a plastic bag for possible future reanalysis.

B. Grass, Green Leafy Vegetation and Cattle Feed

1. Take enough sample to fill 3.5 L or 2.0 L Marinelli beaker to the top.
NOTE: Do not wash the sample.
2. Cut grass and green leafy vegetation into approximately 1-2" long stems and pack into a 3.5 L or 2.0 L container. Pack cattle feed and silage as is. Use larger container if sufficient amount of sample is available. **FILL TO THE TOP OF THE CONTAINER.** Record the wet weight.
3. Add a few mL of formaldehyde to prevent spoilage.
4. Seal with cover. Attach label to the top of the cover and record the sample number, net weight, and date collected.
5. Submit to the counting room for gamma spectroscopic analysis without delay or store in a cooler, (for short period), until counting
6. After gamma scanning is completed, transfer the sample to a drying pan and dry at 110°C.

NOTE 1: If only gamma scan is required, skip drying and ashing (Steps 6 through 10). Transfer the sample to a plastic bag, seal, label, and store in the cooler until disposal.

NOTE 2: If there is sufficient quantity, use surplus sample for drying and ashing instead of waiting for gamma scanning to be completed.
7. Cool, weigh, and record dry weight. Grind.
8. Transfer to a tared ceramic dish. Record dry weight for ashing. Ash in a muffle furnace by gradually increasing the temperature to 600°C. Ash overnight.

NOTE: If ashing is incomplete, (black carbon remains), cool the dish, crush the ash with spatula, and continue ashing overnight at 600°C. At this stage, it is not necessary to increase the temperature gradually. Set the temperature at 600°C and turn on the furnace.
9. Cool and weigh the ashed sample and record the ash weight. Grind and sieve through a 30 mesh screen. Transfer to 4 oz. container, seal, and record sample number, weight, analyses required, and date of collection. The sample is now ready for analysis.
10. Store the remaining ground sample in a plastic bag for possible future reanalysis.

C. Fish

1. Wash the fish.
2. Fillet and pack the fish immediately (to prevent moisture loss) in a 250 mL, 500 mL, or 4 oz. standard calibrated container. Use 500 mL size if enough sample is available. **DO NOT FILL ABOVE THE RIM.** Record the wet weight.
3. Add a few mL of formaldehyde.
4. Seal with cover. Attach label to the on top of the cover and record the sample number, weight, and date of collection.

NOTE: If bones are to be analyzed, boil remaining fish in water for about 1 hour. Clean the bones. Air dry, weigh, and record as wet weight. Dry at 110°C. Record dry weight. Ash at 800°C, cool, weigh, and record the ash weight. Grind to a homogeneous sample. The sample is ready for analysis.

5. Submit to the counting room for gamma spectroscopic analysis without delay or store in a refrigerator, (for short period), until counting.
6. After gamma spectroscopic analysis is completed, transfer the sample to a drying pan and dry at 110°C.

NOTE 1: If only gamma scan is required, skip drying and ashing (Steps 6 through 10). Transfer the sample to a plastic bag, seal, label, and store in the freezer until disposal.

NOTE 2: If there is sufficient quantity, use surplus flesh for drying and ashing instead of waiting for gamma scanning to be completed.

7. Cool, weigh, and record dry weight.
8. Transfer to a tared ceramic dish. Record dry weight for ashing.
9. Ash in a muffle furnace by gradually increasing the temperature to 450°C. If considerable amount of carbon remains after overnight ashing, the ash should be crushed with a spatula and placed back in the muffle furnace until ashing is completed.
10. Cool and weigh the ashed sample and record the ash weight. Grind and sieve through a 30 mesh screen. Transfer to a 4 oz. container, seal, and record sample number, weight, analyses required, and date of collection. The sample is now ready for analysis.

D. Waterfowl, Meat, and Wildlife

1. Skin and clean the animal. Remove a sufficient amount of flesh to fill an appropriate standard calibrated container (500 mL, 250 mL, or 4 oz). Weigh without delay (to prevent moisture loss). **DO NOT FILL ABOVE THE RIM.** Record the wet weight.
2. Add a few mL of formaldehyde.

NOTE: If bones are to be analyzed, boil remaining flesh and bones in water for about 1 hour. Clean the bones. Air dry, weigh, and record as wet weight. Dry at 110°C. Record dry weight. Ash at 800°C, cool, weigh, and record the ash weight. Grind to a homogeneous sample. The sample is ready for analysis.

3. Seal with the cover. Attach paper tape on top of the cover and label with sample number, wet weight, and date of collection.
4. Submit to the counting room for gamma spectroscopic analysis without delay or store in a refrigerator, (for short period), until counting
5. After the gamma scanning is completed, transfer the sample to a drying pan and dry at 110°C.
6. Cool, weigh, and record dry weight.
7. Transfer to a tared ceramic dish. Record dry weight for ashing.
8. Ash in a muffle furnace by gradually increasing the temperature to 450°C. If considerable amounts of carbon remain after overnight ashing, the sample should be crushed with a spatula and placed back in the muffle furnace until ashing is completed.
9. Cool and weigh the ashed sample and record the ash weight. Grind to pass a 30 mesh screen. Transfer to a 4 oz container. Seal and record sample number, weight, analyses required, and date of collection. The sample is now ready for analysis.

E. Eggs

1. Remove the egg shells and mix the eggs with a spatula.
2. Transfer the mixed eggs to a standard calibrated 500 mL container. Record the wet weight. **DO NOT FILL ABOVE THE RIM.**
3. Add a few mL of formaldehyde.
4. Seal with cover. Attach label to the top of the cover and record the sample number, wet weight, and date and time of collection.
5. Submit to the counting room for gamma spectroscopic analysis without delay or store in a refrigerator, (for short period), until counting.
6. After gamma spectroscopic analysis is completed, transfer the sample to a plastic bag, seal, label, and store in a freezer until disposal.

NOTE: If only a gamma scan is required, skip Steps 7 through 11.

7. After the gamma scanning is completed, transfer the sample to a drying pan and dry in an oven at 110°C.
8. Cool, weigh, and record dry weight.
9. Transfer to tared ceramic dish. Record dry weight for ashing.
10. Cool and weigh the ashed sample and record the weight. Grind and sieve through a 30 mesh screen. Transfer to a 4 oz. container, seal, and record sample number, weight, analyses required, and date of collection. The sample is now ready for analysis.
11. Store the remaining dry sample in a plastic bag for possible future rechecking.

F. Slime and Aquatic Vegetation

1. Remove foreign materials.
2. Place the sample in a sieve pan and wash until all sand and dirt is removed (turn the sample over several times).
3. Squeeze out the water by hand.
4. Place the sample in a standard calibrated container. Use the largest size possible for the amount of sample available. Weigh and record wet weight. **DO NOT FILL ABOVE THE RIM.**
5. Add a few mL of formaldehyde.
6. Seal with cover. Attach label to the top of the cover and record the sample number, weight, and date and time of collection.
7. Submit to the counting room without delay. Slime decomposes quickly even with formaldehyde. If gamma scanning must be delayed, freeze.
8. After gamma scanning is completed, transfer the sample to a drying pan and dry at 110°C.

NOTE: If only gamma scan is required, skip drying and ashing (Steps 8 through 11). Transfer the sample to a plastic bag, seal, label, and store in the freezer until disposal.

9. Cool, weigh, and record dry weight.
10. Transfer to a tared ceramic dish, and record dry weight for ashing. Ash in a muffle furnace by gradually increasing the temperature to 600°C.

NOTE: If ashing is incomplete (black carbon remains), cool the dish, crush the ash with spatula, and continue ashing overnight at 600°C. At this stage, it is not necessary to increase the temperature gradually. Set the temperature at 600°C and turn on the furnace.

11. Cool and weigh the ashed sample and record ash weight. Grind and sieve through a 30 mesh screen. Transfer to a 4 oz. container, seal, and label with sample number, weight, analyses required, and date of collection. The sample is now ready for analysis.

G. Bottom Sediments and Soil

1. Remove rocks, roots, and any other foreign materials.
2. Place approximately 1 kg of sample on the drying pan and dry at 110°C.
3. Seal, label, and save remaining sample.
4. Grind or pulverize the dried sample and sieve through a No. 20 mesh screen.
5. For gamma spectroscopic analysis, transfer sieved sample to a standard calibrated 500 mL, 250 mL, or 4 oz. container. **DO NOT FILL ABOVE THE RIM.** Record dry weight.
6. Seal with cover. Attach label to the top of the cover and record the sample number, weight, and date of collection.
7. Submit to the counting room for gamma spectroscopic analysis without delay.
8. For gross alpha and beta analysis transfer 1-2 g of sample to a 4 oz. container, seal and label with the sample number. For other analysis (i.e., radiostrontium, transuranics etc.) transfer to a ceramic dish and ash in a muffle furnace at 600°C. Cool and transfer to a 4 oz. container, seal and label with the sample number.
9. Store the remaining sieved sample in a plastic bag for possible future rechecking.
10. After the gamma scanning is completed, transfer the sample to a plastic bag, seal, label, and store until disposal.

H. Milk

1. Transfer 25 mL of milk for gross alpha and beta analysis or 100-1000 mL for other analysis into a glass beaker.
2. Dry at 110°C.
3. Ash in the muffle furnace by gradually increasing the temperature to 600°C. If a considerable amount of carbon remains (black) cool the beaker, crush the ash with a spatula and continue ashing until ashing is completed (white or light gray).
4. Cool and weigh the ashed sample and record the ash weight. Grind and transfer to a 4oz. container, seal and record the sample number. The sample is now ready for analysis.

I. Feces

NOTE: Perform Transfer operation in the hood. Wear new plastic gloves and face mask.

1. Take 600 mL beaker, clean acid etched area and write sample # using HI-Temp marker.
2. Cover the beaker with parafilm and weigh. Record the weight.
3. Transfer the whole sample to the beaker using a new plastic spoon.
4. Cover the beaker with the same parafilm and weigh. Record total weight.
5. Transfer the beaker to the drying oven, turn the oven on, remove parafilm and dry the sample overnight at 110°C.
6. In the morning, turn the heater off and let the exhaust fan run until the sample is cooled to room temperature.
7. Transfer the beaker to the muffle furnace. Set temperature to 175°C. Gradually increase the temperature to 450°C and ash the sample overnight.

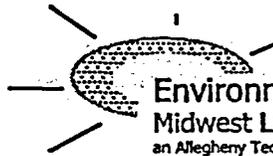
NOTE: In the morning, carefully open the door and visually inspect the sample. Do not touch or remove the beaker from the furnace. If ashing is incomplete, (black carbon remains), continue ashing for another 24 hours or until the ash is grey-white.

8. When ashing is complete, turn the temperature dial down. Let the exhaust fan run until beaker is cool.
9. Remove the beaker from the furnace and cover with parafilm. The sample is ready for analysis.

NOTE: Digest the whole ash sample in the same beaker before taking aliquot for analysis. Do not weigh the beaker.

J. Bottom Sediments and Soil, Analysis for Ra-226 by Gamma Spectroscopy

1. Remove rocks, roots and any other foreign materials.
2. Place approximately 1 kg of sample in a drying pan and dry at 110°C.
3. Seal, label and save remaining sample.
4. Grind or pulverize the dried sample and sieve through a No. 20 mesh screen.
5. Transfer sieved sample to a standard calibrated 500 mL or 250 mL container. **DO NOT FILL ABOVE THE RIM.** Record dry weight.
6. Seal with cover and electric tape. Attach label to the top of the cover and record the sample number, weight, and date of collection and date and time the container was sealed.
7. Store sealed sample for a minimum of 20 days to allow for Pb-214 to come to equilibrium with Ra-226.
8. Submit to counting room for gamma spectroscopic analysis. Use Pb-214 peak to calculate Ra-226 concentration.
9. Store the remaining sieved sample in a plastic bag for possible future reanalysis.
10. After the gamma scanning is completed, transfer the sample to a plastic bag, seal, label and store until disposal.



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**MEASUREMENT of AMBIENT GAMMA RADIATION by
 THERMOLUMINESCENT DOSIMETRY (CaSO₄:Dy)**

PROCEDURE NO. EIML-TLD-01

Prepared by

**Environmental, Inc.
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<u>5</u>	<u>01-08-90</u>	<u>6</u>	<u>B Grob</u>	<u>LG Huebner</u>
<u>6</u>	<u>04-24-95</u>	<u>6</u>	<u>B Grob</u>	<u>LG Huebner</u>
<u>7, Reissue</u>	<u>06-07-01</u>	<u>3</u>	<u>SA Coorlim</u>	<u><i>[Signature]</i></u>

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MEASUREMENT of AMBIENT GAMMA RADIATION by THERMOLUMINESCENT DOSIMETRY (CaSO₄:Dy)

Principle of Method

The cards are spread out in a single layer on a perforated metal tray and annealed for two hours at 250-260 °C. After annealing, the cards are packaged and sent to the field.

Once the cards are returned from the field they are read as soon as possible. After reading, several cards are chosen, annealed and irradiated with a known dose using a Ra-226 source encapsulated in an iridium needle to calculate efficiency. The net exposure is calculated after in-transit exposure is subtracted.

I. Equipment & Materials:

TLD Reader: (Teledyne Isotopes Model 8300)	Annealing oven
TLD Cards (CaSO ₄ :Dy phosphor)	Forceps
TLD Card Holder with copper shielding	Black Plastic bags (pouches)
Transparent plastic bags (6oz and 8oz puncture proof Whirl-Pak)	
Heat sealer	Scotch tape
Labels	Recording sheet
Ra-226 Needle: ("American Radium" No. 37852)	Turntable

II. Preparation

1. Enter location I.D, dosimeter (card) number, and date annealed on the readout recording sheet. As per project requirements, include cards for in-transits and spares.
2. Spread the cards in a single layer on the perforated tray.
3. Preheat the annealing oven to 250-260 °C
4. Set the alarm and anneal for two hours. Remove tray from the oven and let cool.
5. Place each card in a black plastic bag (pouch), seal the flap with scotch tape, and place in the card holder.
6. Attach a label identifying the station, location, and exposure period, on each holder. Place the holders into a transparent plastic bag and heat seal.
7. Ship without delay. Place a "Do Not X-Ray" sticker on the mailing container.

III. Reader Calibration

1. Adjust the nitrogen flow control to 6 SCF per hour.
2. Open the card drawer.
3. Turn "FUNCTION" switch to "CALIBRATE". The "WAIT" sign will be illuminated and the reading will change every three seconds. The reading should be 1000 ±10. If not, adjust using the "CALIBRATE" dial.

III. Reader Calibration (continued)

4. Turn "FUNCTION" switch to "OPERATE". Press "START". When the "READ" signal appears, the reading should be as posted. If not, adjust with "Sensitivity" dial. (Turn clockwise if reading is low, counterclockwise if reading is high).
5. Wait for "START" button to light before continuing. Press "START". Continue adjusting "SENSITIVITY" until the reading is as posted. Make and record 5 readings.
6. When the "START" button lights, push in the card drawer to position No. 3. Press "START". Wait for the "READ" signal and record the reading. (dark current / background)
7. Repeat this step four more times (total of five readings) and record the results.

NOTE: The reading should be as posted on the reader. If not, notify the Lab supervisor.

IV. Readout of TLD Cards

1. After the "START" button lights, pull out card drawer. Take the card out of the holder and insert in the drawer with printed card number facing down and to the back (away from you).
2. Push drawer into position No. 1. Push "START" button.
3. When "READ" sign appears, record the reading.
4. When "START" button lights up, push the drawer to position No. 2. Push "START" button. Repeat steps 2.3 and 2.4 until all positions are read out.
5. Read out and record the reading for the rest of the cards in the same manner.

V. Efficiency Determination

NOTE: Perform an efficiency calibration after each field cycle. (i.e. random TLDs from each project are calibrated after every readout of that project.)

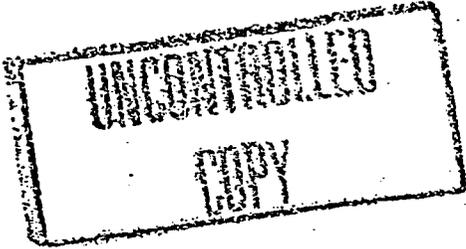
1. After readout of a project is completed, select two to three cards at random.
2. Anneal and package as described in Part II, Steps 2 thru 8.
3. Clip the holders (with the freshly annealed cards) on the irradiation turntable. Start rotation.
4. Attach the Ra-226 needle to center of the turntable. Record the time. Irradiate overnight.
5. Remove the needle, record the time, and read out the cards as in Part III.
6. Average all the readings, and subtract average dark current reading (Part III, Step 6-7).
7. Calculate efficiency (light response) as follows:

$$\text{Efficiency} = \frac{\text{Net Average Reading (from step 6.)}}{\text{Hours of exposure} \times 2.097}$$

8. Submit the field data and efficiency data sheets to data clerk for calculations.

NOTE:

The calculation program will automatically subtract the in-transit exposure and prorate exposure to a selected number of days (usually 30 or 91). Occasionally, some TLDs are placed and/or removed at different times resulting in a different number of exposure days in the field. Exposure will be prorated for the selected number of days.



DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA
IN AIR PARTICULATE FILTERS

PROCEDURE NO. TIML-AP-02

Prepared by
Teledyne Isotopes Midwest Laboratory

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	0	07-11-86	3	<i>B. Job</i>	<i>L. H. Huebner</i>
2	1	07-15-91	3	<i>B. Job</i>	<i>L. H. Huebner</i>

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DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA
IN AIR PARTICULATE FILTERS

Principle of Method

Air particulate filters are stored for at least 72 hours to allow for the decay of short-lived radon and thoron daughters and then counted in the proportional counter.

Apparatus

Forceps
Loading Sheet
Proportional Counter
Stainless Steel Planchets (standard 2" x 1/8")

Procedure

1. Store the filters for at least 72 hours from the day of collection.
2. Place filters on a stainless steel planchet.
3. Fill out a sample loading sheet. Fill in the date, counter number, counting time, sample identification number, sample collection date, and initials.

NOTES: When loading samples in the holder, load blanks (unexposed filter paper) in positions 1, 12, 23, 34, 45, etc.

If filters from more than one project are loaded, make sure that the appropriate blanks are loaded with each batch. Load the counter blank planchet as a last sample.

4. Count in a proportional counter long enough to obtain the required LLDs.
5. After counting is completed, return the filters to the original envelopes.
6. Submit the counter printout, field collection sheet, and the loading sheet to the data clerk for calculations.

Calculations

Gross alpha (beta) concentration:

$$(\text{pCi/liter}) = \frac{A}{B \times C \times 2.22} \pm \frac{2 \sqrt{E_{sb}^2 + E_b^2}}{B \times C \times 2.22}$$

Where:

- A = Net alpha (beta) count (cpm)
- B = Efficiency for counting alpha (beta) activity (cpm/dpm)
- C = Volume of sample
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background

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PROCEDURE for COMPOSITING AIR PARTICULATE FILTERS
for GAMMA SPECTROSCOPIC ANALYSIS

PROCEDURE NO. AP-03

Prepared by

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_____	0	12-15-89	3	B. Grob	L.G. Huebner
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PROCEDURE FOR COMPOSITING AIR PARTICULATE FILTERS
FOR GAMMA SPECTROSCOPIC ANALYSIS

Principle of Method

AP filters are placed in a Petri dish in chronological order, labeled and submitted to the counting room for analysis.

Materials

Tweezers (long)
Blank filter paper
Small Petri Dish (50 x 9 mm)
Scotch Tape

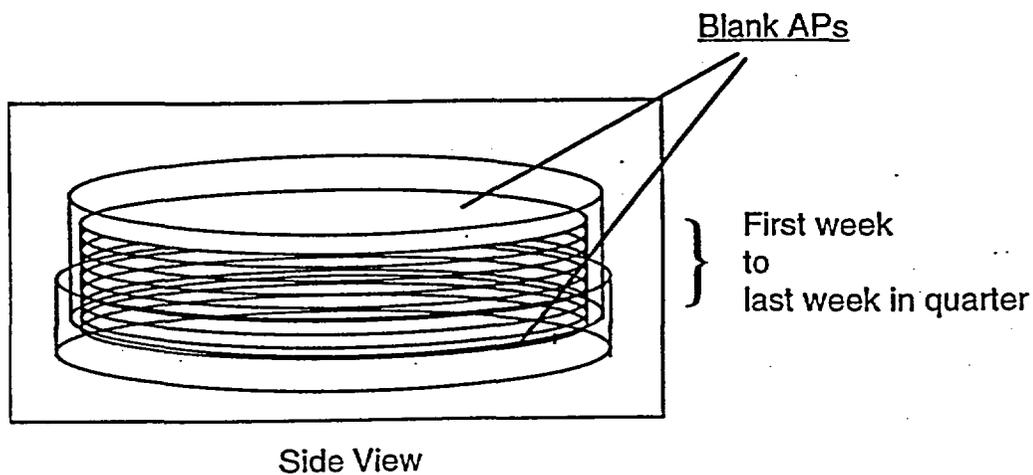
Procedure

1. In the Recording Book enter:
 - Sample ID (project)
 - Sample No.
 - Location
 - Collection Period
 - Date Composited
2. Obtain sample numbers from Receiving Clerk.
3. Stack the envelopes with APs from each location in chronological order, starting with the earliest date on the bottom. After you are done, flip the stack over.
4. Place blank filter paper, "fluffy" side down, in deep half of Petri dish.
5. Beginning from the top of the stack, remove each AP from its envelope and place in the Petri Dish with the deposit facing down.
6. Continue transferring AP's from envelopes into the Petri Dish.
7. Place blank filter, "fluffy" side down, on top of APs.
8. Cap the Petri Dish using the shallow half (you may use Scotch tape to hold cap in place, (if needed). Turn the Petri dish over.
9. On the Petri dish and each stack of glassine envelopes (each location kept together by either paperclips or rubber bands) using a black marker write:
 - Sample ID
 - Sample No.
 - Last date of collection
 - Collection Period
10. Submit the samples to the counting room.
11. After counting, samples are stored in the warehouse, according to client's requirements.

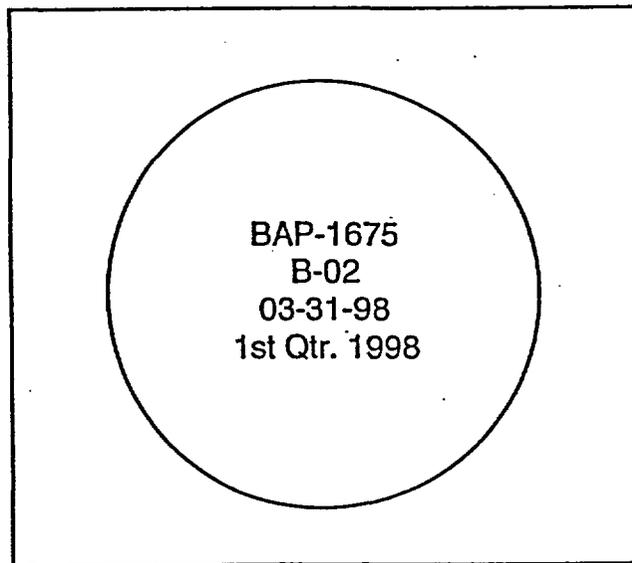
PROCEDURE for COMPOSITING AIR PARTICULATE FILTERS
FOR GAMMA SPECTROSCOPIC ANALYSIS

Example

- Sample ID (project) BAP
- Sample No. 2
- Location 1675
- Last Collection Date 03-31-98
- Collection Period 1st Qtr. 1998



Side View



Top View

| 2

| 2

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ENVIRONMENTAL SERVICES
MIDWEST LABORATORY

DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA IN WATER
(DISSOLVED SOLIDS OR TOTAL RESIDUE)

PROCEDURE NO. TIML-W(DS)-01

Prepared by

Teledyne Brown Engineering Environmental Services
Midwest Laboratory

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<u>Revision #</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
<u>0</u>	<u>11-25-85</u>	<u>4</u>	<u>B. Grob</u>	<u>L.G. Huebner</u>
<u>1</u>	<u>02-28-91</u>	<u>4</u>	<u>B. Grob</u>	<u>L.G. Huebner</u>
<u>2</u>	<u>05-03-91</u>	<u>4</u>	<u>B. Grob</u>	<u>L.G. Huebner</u>
<u>3</u>	<u>08-14-92</u>	<u>4</u>	<u>B. Grob</u>	<u>L.G. Huebner</u>
<u>4</u>	<u>07-21-98</u>	<u>4</u>	<u>D. Richter</u>	<u>[Signature]</u>

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DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA IN WATER

(Dissolved Solids or Total Residue)*

Principle of Method

Water samples containing suspended matter are filtered through a membrane filter and the filtrate is analyzed. The filtered water sample is evaporated and the residue is transferred to a tared planchet for counting gross alpha and gross beta activity.

Reagents

All chemicals should be of "reagent-grade" or equivalent whenever they are commercially available.

Lucite: 0.5 mg/ml in acetone

Nitric acid, HNO₃: 16 N (concentrated), 1 N (62 ml of N HNO₃ diluted to 1 liter)

Apparatus

Filter, membrane Type AA, 0.08

Filtration equipment

Planchets (Standard 2"x1/8" stainless steel, ringed planchet)

Electric hotplate

Heat lamp

Drying oven

Muffle furnace

Analytical Balance

Dessicator

Proportional counter

Procedure

1. Filter a volume of sample containing not more than 100 mg of dissolved solids for alpha assay, or not more than 200 mg of dissolved solids for beta assay.^a

NOTE: For gross alpha and gross beta assay in the same sample, limit the amount of solids to 100 mg.

2. Filter sample through a membrane filter. Wash the sides of the funnel with deionized (D. I.) water. Discard the filter, unless determining suspended solids also. See procedure TIML-W(SS)-02.
3. Evaporate the filtrate to NEAR dryness on a hot plate.
4. Add 20 ml of concentrated HNO₃ and evaporate to NEAR dryness again.

NOTE: If water samples are known or suspected to contain chloride salts, these chloride salts should be converted to nitrate salts before the sample residue is transferred to a stainless steel planchet. (Chlorides will attack stainless steel and increase the sample solids. No correction can be made for these added solids.) Chloride salts can be converted to nitrate salts by adding concentrated HNO₃ and evaporating to near dryness.

5. Transfer quantitatively the residue to a TARED PLANCHET, using an unused plastic disposable pipette for each sample, (not more than 1 or 2 ml at a time) evaporating each portion to dryness under the lamp. Spread residue uniformly on the planchet.

NOTE: Non-uniformity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

6. Wash the beaker with DI water several times and combine the washings and the residue in the planchet, using the rubber policeman to wash the walls. Evaporate to dryness.

NOTE: Rinse the rubber policeman with DI water between samples.

7. Bake in muffle furnace at 400 ° C for 45 minutes, cool and weigh.

NOTE: If the sample is very powdery, add a few drops (6-7) of the Lucite solution and dry under the infrared lamp for 10-20 minutes.

8. Store the sample in a dessicator until ready to count since vapors from the moist residue can damage the detector and the window and can cause erratic measurements.

9. Count the gross alpha and/or the gross beta activity in a low background proportional counter.

NOTE: If the gas-flow internal proportional counter does not discriminate for the higher energy alpha pulses at the beta plateau, the activity must be subtracted from the beta plus alpha activity. This is particularly important for samples with high alpha activity.

Samples may be counted for beta activity immediately after baking; alpha counting should be delayed at least 72 hours (until equilibrium has occurred).

- a For analysis of total residue (for clear water), proceed as described above but do not filter the water. Measure out the appropriate amount and proceed to Step 3.

Calculations

Gross alpha (beta) activity:

$$pCi/L = \frac{A}{B \times C \times D \times 2.22} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{B \times C \times D \times 2.22}$$

Where:

- A = Net alpha (beta) count (cpm)
- B = Efficiency for counting alpha (beta) activity (cpm/dpm)
- C = Volume of sample (liters)
- D = Correction factor for self-absorption (See Proc. TIML-AB-02)
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background

- References: Radio assay Procedures for Environmental Samples, US. Department of Health, Education and Welfare. Environmental Health Series, Jan. 1967.
- EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water. August 1980

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DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA IN WATER
(SUSPENDED SOLIDS)

PROCEDURE NO. TIML-W(SS)-02

Prepared by

Teledyne Brown Engineering Environmental Services
Midwest Laboratory

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0	10-21-86	4	L.G. Huebner	L.G. Huebner
1	08-14-92	4	B. Grob	L.G. Huebner
2	07-21-98	3		

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DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA IN WATER
(SUSPENDED SOLIDS)

Principle of Method

The sample is filtered through a tared membrane filter. The filter containing the solids is placed on a ringless, stainless steel planchet and air dried, then placed in a dessicator until ready for weighing. The gross alpha and gross beta activities are measured in a low background internal proportional counter.

Reagent

Acetone

Apparatus

Filter, membrane Type AA 0.08

Filtration equipment

Planchets (Standard 2"x1/8" stainless steel, ringless planchet)

Heat lamp

Analytical Balance

Dessicator

Proportional counter

Procedure

1. Filter one liter of sample through a TARED membrane filter. Wash the sides of the funnel with deionized water.

NOTE: If the sample contains sand, place it in the separatory funnel, allow the sand to settle for 30 minutes, then drain off the sand at the bottom. Shake the funnel and repeat as above two (2) more times.

2. Place the filter on a ringless planchet and air dry for 24 hours.
3. Dry under the infrared lamp for 20-30 minutes. Desiccate to constant weight and weigh.
4. Count for gross alpha and gross beta activity using a proportional counter.
5. Calculate the activity in pCi/L, using the computer program designed for this analysis.

Calculations

Gross alpha (beta) activity:

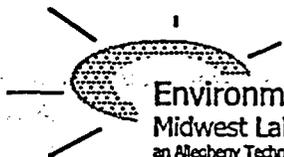
$$\text{pCi/L} = \frac{A}{B \times C \times D \times 2.22} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{B \times C \times D \times 2.22}$$

Where:

- A = Net alpha (beta) count (cpm)
- B = Efficiency for counting alpha (beta) activity (cpm/dpm)
- C = Volume of sample (liters)
- D = Correction factor for self-absorption (See Proc. TIML-AB-02)
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background

References: Radio assay Procedures for Environmental Samples, U.S. Department of Health, Education and Welfare. Environmental Health Series, January 1967.

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**DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA
IN SOLID SAMPLES**

PROCEDURE NO. EIML-AB-01

Prepared by

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<u>0</u>	<u>08-04-86</u>	<u>4</u>	<u>B Grob</u>	<u>LG Huebner</u>
<u>1</u>	<u>08-14-92</u>	<u>5</u>	<u>B Grob</u>	<u>LG Huebner</u>
<u>2</u>	<u>06-11-01</u>	<u>3</u>	<u>SA Coorlim</u>	<u><i>[Signature]</i></u>
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DETERMINATION OF GROSS ALPHA AND/OR GROSS BETA
IN SOLID SAMPLES

Principle of Method

100 mg to 200 mg of sample is distributed evenly on a 2" ringed planchet, counted in a proportional counter, and concentrations of gross alpha and /or gross beta are calculated.

A. Vegetation, Meat, Fish, and Wildlife

Procedure

1. Weigh out accurately in a planchet no more than 100 mg of ashed or dried and ground sample for gross alpha assay and no more than 200 mg for gross beta assay.

NOTE: If both gross alpha and gross beta analyses are required, do not use more than 100mg.

2. Add a few drops of water and spread uniformly over area of the planchet. Dry under a heat lamp.

NOTE: If necessary, a few drops (6-7) of a lucite solution (0.5 mg/ml in acetone) may be added to keep residue in place. Dry under an infrared lamp for 10-20 minutes.

4. Store the planchets in a dessicator until counting.
5. Count the gross alpha and gross beta activity in a low background proportional counter.

Calculations

Gross alpha / gross beta activity:

$$(\text{pCi/g wet}) = \frac{A}{B \times C \times D \times F \times 2.22} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{B \times C \times D \times F \times 2.22}$$

Where:

- A = Net alpha / beta counts (cpm)
- B = Efficiency for counting alpha / beta activity (cpm/dpm)
- C = Weight of sample (grams), ash or dry
- D = Correction factor for self absorption (See Proc. TIML-AB-02)
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background
- F = Ratio of wet weight to ashed or dry weight

REFERENCES: Radioassay Procedures for Environmental Samples, U.S. Department of Health, Education and Welfare. Environmental Health Series, January 1967.

B. Gross Alpha and/or Gross beta in Soil and Bottom Sediments**Procedure**

1. Weigh out accurately in a planchet no more than 100 mg of a pulverized sample for a gross alpha assay and no more than 200 mg for a gross beta assay.

NOTE: If both gross alpha and gross beta analyses are required, do not use more than 100mg.

2. Add a few drops of water and spread uniformly over area of the planchet. Dry under a heat lamp.

NOTE: If necessary, a few drops (6-7) of a lucite solution (0.5 mg/ml in acetone) may be added to keep residue in place. Dry under an infrared lamp for 10-20 minutes. | 2

3. Store the planchets in a dessicator until counting.
4. Count the gross alpha and gross beta activity in a low background proportional counter.

Calculations

Gross alpha / gross beta activity:

$$(\text{pCi/g dry}) = \frac{A}{B \times C \times D \times F \times 2.22} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{B \times C \times D \times F \times 2.22}$$

Where:

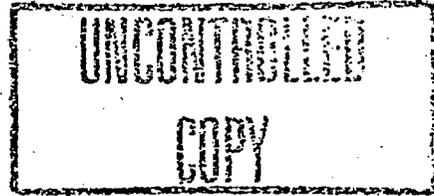
- A = Net alpha / beta counts (cpm)
- B = Efficiency for counting alpha / beta activity (cpm/dpm)
- C = Weight of sample (grams)
- D = Correction factor for self absorption (See Proc. EIML-AB-02)
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background
- F = Ratio of wet weight to ashed or dry weight

REFERENCES: Radioassay Procedures for Environmental Samples, U.S. Department of Health, Education and Welfare. Environmental Health Series, January 1967,



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**DETERMINATION OF GAMMA EMITTERS
 BY GAMMA SPECTROSCOPY
 (GERMANIUM DETECTORS)**

PROCEDURE NO. GS-01

Prepared by

Environmental Inc., Midwest Laboratory

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<u>Revised Pages</u>	<u>Revision #</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
<u>Reprint</u>	<u>2</u>	<u>07-01-98</u>	<u>3</u>	<u>F. G. Shaw</u>	<u>S. A. Coorlim</u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>	<u> </u>	<u> </u>
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DETERMINATION OF GAMMA EMITTERS
BY GAMMA SPECTROSCOPY
(GERMANIUM DETECTORS)

Principle of Method

The sample is placed in a calibrated container and counted for a length of time required to reach the required LLD. The results are decay corrected to the sampling time, where appropriate, using a dedicated computer and software.

Apparatus

Counting Containers
Counting Equipment
Cylinders
Marking Pens
Recording Books

A. Milk, Water, and other Liquid Samples

1. Measure accurately 500 mL, 1.0 L, 2.0 L or 3.5 L of sample and put it in the calibrated counting container (Marinelli beaker). Always use largest volume if sample is of sufficient quantity.
2. Cover and attach a gummed label to the cover; write the sample number, volume and date and time of collection on the label. Mark "I-131" if analysis for I-131 is required by gamma spectroscopy.
3. Count without delay for estimated time required to meet the client's technical requirements. Record file number, sample identification number, date and time counting started, detector number, geometry, sample size, and date and time of collection.
4. Stop counting; transfer spectrum to the disk, and print out the results.
5. Check results before taking the sample off. If client's technical requirements are not met, continue counting.
6. After counting is completed, record the counting time.
7. Return the sample to the original container and mark with a red marker.

B. Airborne Particulates

1. Place air filters in a small Petrie dish following Procedure AP-03.
2. Place Petrie dish (with marked side up) on the detector and count long enough to meet the client's technical requirements. Record the file number, sample identification number, date and time counting started, detector number, geometry, sample size, and date and time collected.

NOTE: When counting individual filter, place it in the Petrie dish with active (with deposit) side up. Mark the Petrie dish and place it on the detector with the active side up.

3. Stop counting and transfer spectrum to the disk. Print out and check the results before taking the sample off. If client's technical requirements are not met, continue counting.
4. After counting is completed, record the counting time.
5. Replace air filters in the original envelopes for storage or further analyses.

C. Other Samples

NOTE: Sample, e.g. soil, vegetation, fish, etc., are prepared in the prep lab and delivered to the counting room.

1. Place the sample on the detector and count long enough to meet client's technical requirements. Record the file number, sample identification number, date and time counting started, detector number, geometry, sample size, and date and time of collection.
2. Stop counting and transfer spectrum to the disk. Print out the results and check the results before taking the sample off. If client's technical requirements are not met, continue counting.
3. After counting is completed, record counting time. Mark the container with red marker and return to the prep lab for transfer to the plastic bag for storage or further analyses.

D. Charcoal Cartridges

For counting charcoal cartridges, follow Procedure I-131-02, I-131-04 or I-131-05.

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DETERMINATION OF TRITIUM IN WATER
(DIRECT METHOD)

PROCEDURE NO. EIML-T-02

Prepared by

Environmental Inc., Midwest Laboratory

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<u>0</u>	<u>11-22-85</u>	<u>5</u>	<u>B Grob</u>	<u>L. G. Huebner</u>
<u>1</u>	<u>09-27-91</u>	<u>4</u>	<u>B Grob</u>	<u>L. G. Huebner</u>
<u>2</u>	<u>04-24-95</u>	<u>4</u>	<u>B Grob</u>	<u>L. G. Huebner</u>
<u>3</u>	<u>07-07-98</u>	<u>4</u>	<u>D. Rieter</u>	<u>B Grob</u>
<u>4</u>	<u>06-06-00</u>	<u>4</u>	<u>R. Amroftin</u>	<u>B Grob</u>
<u>5</u>	<u>01-29-02</u>	<u>4</u>	<u><i>[Signature]</i></u>	<u><i>[Signature]</i></u>

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DETERMINATION OF TRITIUM IN WATER (DIRECT METHOD)Principle of Method

The water sample is purified by distillation, a portion of the distillate is transferred to a counting vial and the scintillation fluid added. The contents of the vial are thoroughly mixed and counted in aliquot scintillation counter.

Reagents

Scintillation medium, Ultima-Gold LLT, Packard Instruments Co.
Tritium standard solution
Dead water
Ethyl alcohol
Sodium Hydroxide (pellets)
Potassium permanganate (crystals)

Apparatus

Condenser
Distillation flask, 250-mL capacity
Liquid scintillation counter
Pipette and disposable tips (0.1ml., 5-10 ml.)
Kimwipes

Procedure

NOTE: All glassware must be dry. Set drying oven for 100-125°C.

1. Place 60-70 mL of the sample in a 250-mL distillation flask. Add a boiling chip to the flask. Add one NaOH pellet and about 0.02g KMnO₄. Connect a side arm adapter and a condenser to the outlet of the flask. Place a receptacle at the outlet of the condenser. Set variac at 70 mark. Heat to boiling to distill. Discard the first 5-10mL of distillate. Collect next 20-25mL of distillate for analysis. Do not distill to dryness.
2. Mark the vial caps with the sample number and date.

NOTE: Use the same type of vial for the whole batch (samples, background and standard.)
3. Mark three vial caps "BKG-1", " BKG-2", " BKG-3", and date.
4. Mark three vial caps "ST-1", " ST-2", " ST-3"; standard number, and date.
5. Dispense 13 mL of sample into marked vials and "dead" water into vials marked BKG-1, BKG-2, BKG-3.

NOTE 1: The Pipette is set (and calibrated) to deliver 6.5 mL, so pipette twice into each vial. Use new tip for each sample and new tip (one) for three background samples.

NOTE 2: Make sure the pipette has not been reset. If it has been reset, or if you are not sure, do not use it; check with your supervisor.

NOTE 3: Make sure the plastic tip is pushed all the way on the pipette and is tight. If it is not, the air will be draw in and the volume withdrawn will not be correct (it will be smaller).

6. Dispense 13 mL (see Notes 1, 2, and 3, above) of "dead" water into each vial marked "ST-1", "ST-2" and "ST-3."
7. Using a 0.1 mL pipette, withdraw water from each of the three standard vials. Discard this 0.1 mL of water.
8. Take a new 0.1 mL tip. Dispense 0.1 mL of standard into each of the three vials marked "ST-1," "ST-2," and "ST-3."
9. Take all vials containing samples, background, and standard to the counting room.

NOTE: To avoid spurious counts, scintillation fluid should not be added under fluorescent light.

10. Dispense 10 mL of scintillation fluid into each vial (one at a time), cap tightly, and shake VIGOROUSLY for at least 30 seconds. Recheck the cap for tightness.
11. Wet a Kimwipe with alcohol and wipe off each vial in the following order:
 - Background
 - Samples
 - Standard
12. Load the vials in the following order:
 - BKG-1
 - ST-1
 - Samples
 - BKG -2*
 - ST -2*
 - Samples
 - BKG-3
 - ST -3

*BKG-2 and ST-2 should be approximately
in the middle of the batch
13. Let the vials dark- and temperature-adapt for about one hour.

NOTE 1: To check if vials have reached counter temperature, inspect one vial (Bkg). The liquid should be transparent. If the temperature is too high (or too low), the liquid will be white and very viscous.

NOTE 2: The temperature inside the counter should be between 10° and 14°C (check thermometer). In this temperature range, the liquid is transparent.

14. Set the counter for 100-minute counting time and infinite cycles. (Follow manufacturer's procedure for setting the counter.)

15. Fill out the loading sheet, being sure to indicate the date and time counting started, and your initials.

NOTE 1: Do not count prepared background and standard sets with another batch of samples if plastic vials are used. Prepare new backgrounds and standards for each batch.

NOTE 2: If glass vials are used, the prepared background and standard sets can be counted with other batches up to one month after preparation, provided they are not taken out of the counter (not warmed up) and the same vial type from the same manufacturing batch (the same carton) is used. After one month prepare new sets of backgrounds and standards.

Calculations

$$\text{pCi/L} = \frac{\frac{A}{t_1} - \frac{B}{t_2}}{2.22EVe^{-\lambda t_3}} + \frac{2\sqrt{\frac{A}{t_1^2} + \frac{B}{t_2^2}}}{2.22EVe^{\lambda t_3}}$$

Where:

- A = Total counts, sample
- B = Total counts, background
- E = Efficiency, (cpm/dpm)
- V = Volume (liter)
- e = Base of the natural logarithm = 2.71828
- $\lambda = \frac{0.693}{12.26} = 0.5652$
- t₁ = Counting time, sample
- t₂ = Counting time, background
- t₃ = Elapsed time from the time of collection to the time of counting (in years)



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**DETERMINATION OF I-131 IN MILK
 BY ANION EXCHANGE
 (BATCH METHOD)**

PROCEDURE NO. I-131-01

Prepared by

Environmental Inc., Midwest Laboratory

Copy No. _____

<u>Revised Pages</u>	<u>Revision #</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
Reprint	5	09-24-92	5	B. Grob	L. G. Huebner
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Determination of I-131 in milk by Ion Exchange(Batch Method)Principle of Method

Iodine, as the iodide, is concentrated by adsorption on an anion resin. Following a NaCl wash, the iodine is eluted with sodium hypochlorite. Iodine in the iodate form is reduced to I₂ and the elemental iodine extracted into CHCl₃, back-extracted into water, then finally precipitated as palladium iodide.

Chemical recovery of the added carrier is determined gravimetrically from the PdI₂ precipitate. I-131 is determined by beta counting the PdI₂.

Reagents

Anion Exchange Resin, Dowex 1-X8 (20-50 mesh) chloride form

Chloroform, CHCl₃ - reagent grade

Hydrochloric Acid, HCl, 1N

Hydrochloric Acid, HCl, 3N

Wash Solution: H₂O - HNO₃ - NH₂OH HCl, 50 mL H₂O; 10 mL 1M - NH₂OH-HCl;
10 mL conc. HNO₃

Hydroxylamine Hydrochloride, NH₂OH HCl - 1 M

Nitric Acid, HNO₃ - concentrated

Palladium Chloride, PdCl₂, 7.2 mg Pd⁺⁺/mL (1.2 g PdCl₂/100 mL of 6N HCl)

Sodium Bisulfite, NaHSO₃ - 1 M

Sodium Chloride, NaCl - 2 M

Sodium Hypochlorite, NaOCl - 5% (Clorox)

Potassium Iodide, KI, ca 29 mg KI/mL (See Proc. CAR-01 for preparation)

Special Apparatus

Chromatographic Column, 20 mm x 150 mm (Reliance Glass Cat. #R2725T)

Vacuum Filter Holder, 2.5 cm² filter area

Filter Paper, Whatman #42, 21 mm

Mylar

Polyester Gummed Tape, 1 1/2", Scotch #853

Heat Lamp

Determination of I-131 in milk by Ion Exchange(Batch Method)Part AIon Exchange Procedure

1. Transfer 2 liters (if available) of sample to the beaker. Add 1.00 mL of standardized iodide carrier to each sample.
2. Add a clean magnetic stirring bar to each sample beaker. Stir each sample for 5 minutes or longer on a magnetic stirrer. Allow sample to equilibrate at least 1/2 hour. If a milk sample is curdled or lumpy, vacuum filter the sample through a Buchner funnel using a cheesecloth filter. Wash the curd thoroughly with deionized water, collecting the washings with the filtrate. Pour the filtrate back into the original washed and labeled 4 liter beaker and discard the curd.
3. Add approximately 45 grams of Dowex 1X8 (20-50 mesh) anion resin to each sample beaker and stir on a magnetic stirrer for at least 1 hour. Turn off the stirrer and allow the resin to settle for 10 minutes.
4. Gently decant and discard the milk or water sample taking care to retain as much resin as possible in the beaker. Add approximately 1 liter of deionized water to rinse the resin, allow to settle 2 minutes, and pour off the rinse. Repeat rinsing in the case of milk samples until all traces of milk are removed from the resin.
5. Using a deionized water wash bottle, transfer the resin to the column marked with the sample number. Allow resin to settle 2 minutes and drain the standing water. Wash the resin with 100 mL of 2 M NaCl.
6. Measure 50 mL 5% sodium hypochlorite in a graduated cylinder. Add sodium hypochlorite to column in 10-20 mL increments, stirring resin as needed to eliminate gas bubbles and maintain a flow rate of 2 mL/min. Collect eluate in 250 mL beaker and discard the resin.

Part BIodine Extraction Procedure

CAUTION: Perform the following steps in the fume hood.

1. Acidify the eluate from Step 6 by adding ca. 15 mL of concentrated HNO₃ to make the sample 2-3 N in HNO₃ and transfer to a 250 mL separatory funnel. (Add the acid slowly with stirring until the vigorous reaction subsides).
2. Add 50 mL of CHCl₃ and 10 mL of 1 M hydroxylamine hydrochloride (freshly prepared). Extract iodine into organic phase (about 2 minutes equilibration). Draw off the organic phase (lower phase) into another separatory funnel.
3. Add 25 mL of CHCl₃ and 5 mL of 1 M hydroxylamine hydrochloride to the first separatory funnel and again equilibrate for 2 minutes. Combine the organic phases. Discard the aqueous phase (upper phase).
4. Add 20 mL H₂O-HNO₃-NH₂OH HCl wash solution to the separatory funnel containing the CHCl₃. Equilibrate 2 minutes. Allow phases to separate and transfer CHCl₃ (lower phase) to clean separatory funnel. Discard the wash solution.
5. Add 25 mL H₂O and 10 drops of 1 M sodium bisulfite (freshly prepared) to the separatory funnel containing the CHCl₃. Equilibrate for 2 minutes. Discard the organic phase (lower phase). Drain aqueous phase (upper phase) into a 100 mL beaker. Proceed to the Precipitation of PdI₂.

Determination of I-131 in milk by Ion Exchange

(Batch Method)

Part CPrecipitation of Palladium IodideCAUTION: AMMONIUM HYDROXIDE INTERFERES WITH THIS PROCEDURE

1. Add 10 mL of 3 N HCl to the aqueous phase from the iodine extraction procedure in Step 5.
2. Place the beaker on a stirrer-hot plate. Using the magnetic stirrer, boil and stir the sample until it evaporates to 30 mL or begins to turn yellow.
3. Turn the heat off. Remove the magnetic stirrer, rinse with deionized water.
4. Add, dropwise, to the solution, 2.0 mL of palladium chloride.
5. Cool the sample to room temperature. Place the beaker with sample on the stainless steel tray and put in the refrigerator overnight.
6. Weigh a clean 21 mm Whatman #42 filter which has been dried under a heat lamp.
7. Place the weighed filter in the filter holder. Filter the sample and wash the residue with water and then with absolute alcohol.
8. Remove filter from filter holder and place it in the labeled petri dish.
9. Dry under the lamp for 5-10 minutes.
10. Weigh the filter with the precipitate and calculate the carrier recovery.
11. Cut a 1-1/2" strip of polyester tape and lay it on a clean surface, gummed side up. Place the filter, precipitate side up, in the center of the tape.
12. Cut a 1-1/2" wide piece of mylar. Using a spatula to press it in place, put it directly over the precipitate and seal the edges to the polyester tape. Trim to about 5 mm from the edge of the filter with scissors.
13. Mount the sample on a plastic disc and write the sample number on the back side of the disc.
14. Count the sample on a proportional beta counter.

Determination of I-131 in milk by Ion Exchange(Batch Method)Calculations

Calculate the sample activity using computer program I131.

I-131 concentration:

$$(\text{pCi/L}) = \frac{A}{2.2 \times B \times C \times D \times R} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{2.22 \times B \times C \times D \times R}$$

where:

A = Net cpm, sample

B = Efficiency for counting beta I-131 (cpm/dpm)

C = Volume of sample (liters)

D = Correction for decay to the time of collection = $e^{-\lambda t} =$

$$\text{Exp}\left(-\frac{0.693 \times t}{8.04}\right) = e^{-0.0862t}$$

where t = elapsed time from the time of collection to the counting time (in days)

E_{sb} = Counting error of sample plus background

E_b = Counting error of background

R = Carrier recovery

2.22 = dpm/pCi

Reference: "Determination of I-131 by Beta-Gamma coincidence Counting of Pd12 ". Radiological Science Laboratory. Division of Laboratories and Research, New York State Department of Health, March 1975, Revised February 1977.

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DETERMINATION OF AIRBORNE I-131 IN CHARCOAL CARTRIDGES BY GAMMA SPECTROSCOPY (BATCH METHOD)

PROCEDURE NO. TIML-I-131-02

Prepared by
Teledyne Isotopes Midwest Laboratory

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Revised Pages	Revision #	Date	Pages	Prepared by	Approved by
	0	07-04-86	3	<i>B. Job</i>	<i>LJ Huebner</i>
1234	1	08-01-92	4	<i>B. Job</i>	<i>LJ Huebner</i>

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DETERMINATION OF AIRBORNE I-131 IN CHARCOAL CARTRIDGES
BY GAMMA SPECTROSCOPY
(BATCH METHOD)

Principle of Method

Five or six cartridges are mounted in a specially designed holder and counted. A peak of 0.36 MeV is used to calculate the concentration at counting time. The concentration at the end of collection is then calculated.

NOTE: This procedure is used for screening only. If I-131 is detected, each cartridge from the batch is analyzed individually.

Materials

Charcoal Cartridges

Apparatus

Counting Container
Germanium Detector
Rubber Band

Procedure

NOTE: Because of the short half-life of I-131, count the samples as soon as possible after receipt, but no later than 8 days after collection.

1. Load the charcoal cartridges in a specially designed holder with the rim facing the detector and the arrow (if there is one - not all cartridges have arrows) pointing away from the detector (see Figure 1). Use rubber band to hold side mounted cartridges in place.
2. Place the holder on the detector and count for a period of time that will meet the required Lower Limit of Detection (LLD).
3. Calculate concentration of I-131 at counting time by inputting sample ID, volume (use 1m^3) and date and time (midpoint) of counting. Submit printout to data clerk for final calculations without delay.

NOTE: If I-131 is detected, (positive result) count each cartridge from the batch individually in accordance with Procedure TIML-I-131-04 and notify supervisor immediately.

Calculations:

$$A_1 = \text{I-131 concentration (pCi/sample)} = \frac{A}{2.22 \times B} \text{ (at counting time)}$$

Where:

A = Net count rate of I-131 in the 0.36 MeV peak (cpm)

B = Efficiency for the I-131 in 0.36 MeV peak (cpm/dpm)

2.22 = dpm/pCi

I-131 concentration at the time of collection:

$$(\text{pCi/m}^3) = \frac{A_1}{C \times D} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{C \times D}$$

where:

C = Volume of sample (m³)

D = Correction for decay to the time of collection = $e^{-\lambda t}$

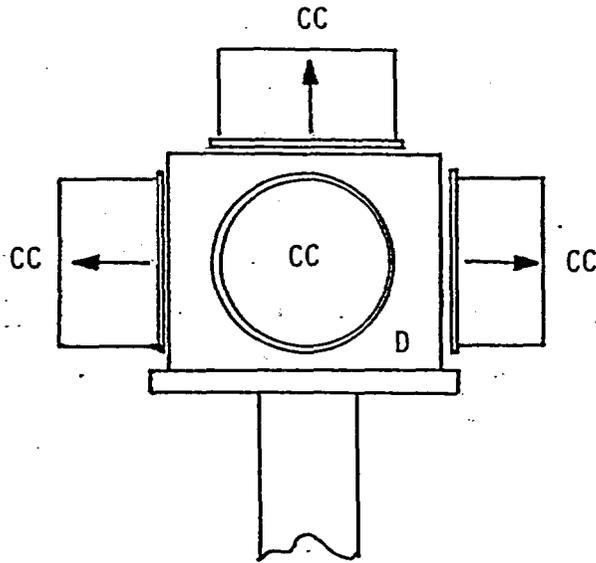
$$\text{Exp}\left(-\frac{0.693 \times t}{8.04}\right) = e^{-0.0862t}$$

where t = elapsed time from the time of collection to the counting time (in days)

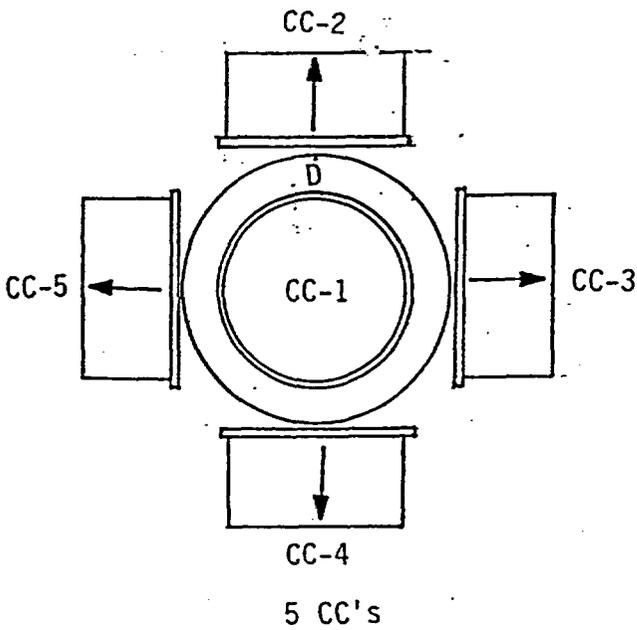
E_{sb} = Counting error of sample plus background

E_b = Counting error of background

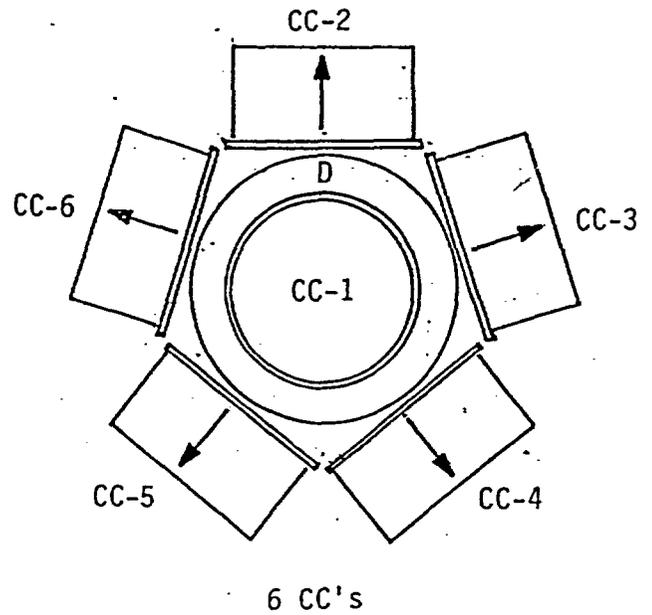
Figure 1.



Side View (5 CC's)



5 CC's



6 CC's

Top View

Charcoal Cartridge: CC
Germanium Detector: D

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DETERMINATION OF SR-89 AND SR-90 IN WATER
(CLEAR OR DRINKING WATER)

PROCEDURE NO. TIML-SR-02

Prepared by
Teledyne Isotopes Midwest Laboratory

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<u>Revision No.</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
0	03-21-86	7	J. Kattner	<i>J. Kattner</i>
_____	_____	_____	_____	_____
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Determination of Sr-89 and Sr-90 in WaterPrinciple of Method

The acidified sample of clear water with stable strontium, barium, and calcium carriers is treated with oxalic acid at a pH of 3.0 to precipitate insoluble oxalates. The oxalates are dissolved in nitric acid, and strontium nitrate is separated from calcium as a precipitate in 70% nitric acid. The residue is purified by adding iron and rare earth carriers and precipitating them as hydroxides. After a second strontium nitrate precipitation from 70% nitric acid, the nitrates are dissolved in acid with added yttrium carrier and are stored for ingrowth of yttrium-90. The yttrium is again precipitated as hydroxide and separated from strontium with the strontium being in the supernate. Each fraction is precipitated separately as an oxalate (yttrium) and carbonate (strontium) and collected on No. 42 (2.4 cm) Whatman filter for counting.

Reagents

Ammonium acetate buffer: pH 5.0

Ammonium hydroxide, NH_4OH : concentrated (15N), 6 N

Ammonium oxalate, $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$: 0.5% w/v

Carrier solutions:

Ba^{+2} as barium nitrate, $\text{Ba}(\text{NO}_3)_2$: 20 mg Ba^{+2} per ml

Ca^{+2} as calcium nitrate, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$: 40 mg Ca^{+2} per ml

Sr^{+2} as strontium nitrate, $\text{Sr}(\text{NO}_3)_2$: 20 mg Sr^{+2} per ml

Y^{+3} as yttrium nitrate, $\text{Y}(\text{NO}_3)_3$: 10 mg Y^{+3} per ml

Hydrochloric acid, HCl : concentrated (3 N)

Nitric acid, HNO_3 : Fuming (90%), concentrated (16 N), 6 N

Oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$: Saturated at room temperature

Scavenger solutions: 20 mg Fe^{+3} per ml, 10 mg each Ce^{+3} and Zr^{+4} per ml

Fe^{+3} as ferric chloride, $\text{FeCl}_3 \cdot \text{H}_2\text{O}$

Ce^{+3} as cerous nitrate, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$

Zr^{+4} as zirconyl chloride, $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$

Sodium carbonate, Na_2CO_3 : 3N, 0.1N

Sodium chromate, Na_2CrO_4 : 3N

Apparatus

Analytical balance

Low background beta counter

pH meter

Procedure

1. Measure 1 liter of acidified water into a 2 liter beaker.

NOTE: If the sample contains foreign mater, such as sand, dirt, etc., filter through a 47 mm glass fiber filter using suction flask.
2. To acidified clear water in a 2 liter beaker, add 1 ml of strontium carrier solution, 1 ml barium carrier solution, and if necessary, 1 ml of calcium carrier solution. (Improved precipitation may be obtained by adding calcium to soft waters.) Stir thoroughly, and while stirring add 125 ml of saturated oxalic acid solution.
3. Using a pH meter, adjust the pH to 3.0 with 15N NH_4OH and allow the precipitate to settle for 5 - 6 hours or overnight.
4. Decant to waste most of the supernate (liquid) and transfer the precipitate to a 250 ml centrifuge bottle using D.I. water. Discard the supernate to waste.
5. Dissolve the precipitate with 10 ml of 6N HNO_3 and transfer to a 250 ml beaker. Then use 20 ml of 16N HNO_3 to rinse the centrifuge tube and combine it to the solution in the 250 ml beaker.
6. Evaporate the solution to dryness. Cool; then add 50 ml 16N HNO_3 and repeat the acid addition and evaporation until the residue is colorless.
7. Transfer the residue to a 40 ml centrifuge tube, rinsing with a minimum volume of 16N HNO_3 . Cover with parafilm and cool in an ice bath. Centrifuge at 1500 - 1800 rpm for 10 minutes, and discard the supernate to waste.
8. Dissolve the precipitate in 5 ml of 6N HNO_3 and then add 30 ml of fuming nitric acid. Cover with parafilm, cool in the ice bath, centrifuge, and discard the supernate to waste.
9. Dissolve the nitrate precipitate in about 10 ml of D.I. water (perform under the hood). Add 1 ml of scavenger solution. Adjust the pH of the mixture to 7 with 6N HN_4OH . Heat in hot water bath for 10 minutes, stir, and filter through a Whatman No. 541 filter into another 40 ml centrifuge tube. Discard the mixed hydroxide precipitate (filter paper).
10. To the filtrate, add 5 ml of ammonium acetate buffer. Adjust the pH with 3N HNO_3 or NH_4OH to pH 5.5.

NOTE: The pH of the solution at this point is critical.

Add dropwise with stirring 1 ml of 3N Na_2CrO_4 solution, stir, and heat in a water bath.

11. Cool and centrifuge. Decant the supernate into another 40 ml centrifuge tube. (Save the precipitate for Ba analysis if needed.)

Procedure

12. Heat the supernate in a water bath. Adjust the pH to 8 - 8.5 with NH_4OH . With continuous stirring, cautiously add 5 ml of 3N Na_2CO_3 solution. Heat gently for 10 minutes. Cool, centrifuge, and decant the supernate to waste. Wash the precipitate with 0.1N Na_2CO_3 . Centrifuge again and decant the supernate to waste.
13. Dissolve the precipitate in no more than 4 ml of 3N HNO_3 . Then add 20 - 30 ml of fuming HNO_3 , cover with parafilm, cool in a water bath, and centrifuge. Decant and discard the supernate.
14. Repeat Step 13. Then RECORD THE TIME AND DATE AS THE BEGINNING OF YTTRIUM-90 INGROWTH.
15. Dissolve precipitate in 4 ml of 6N HNO_3 and add 1 ml of yttrium carrier solution.
16. Cover with parafilm and store for 7 - 14 days.

NOTE: At this point, the sample can be transferred to a glass scintillation vial for the ingrowth storage. Use several portions of 6N HNO_3 (a total of not more than 4 ml); then add 1 ml of yttrium carrier to the vial.)

Separation

NOTE: If the sample was stored in the scintillation vial, transfer back into 40 ml centrifuge tube using a few drops of 6N HNO_3 as a rinse.

1. After storage (ingrowth period), heat the 40 ml centrifuge tube containing the sample in the hot water bath (approximately 90°C) for 10 minutes.
2. Adjust pH to 8 with NH_4OH , stirring continuously.
3. Cool in a cold water bath and centrifuge for 5 minutes.
4. Decant the supernate into a 40 ml centrifuge tube marked with the sample number and "SR-89." RECORD THE DATE AND TIME OF DECONTAMINATION AS THE END OF Y-90 ingrowth in SR fraction and the beginning of its decay in Y-90 fraction.
5. Redissolve the precipitate by adding 3 - 4 drops of 6N HCl and add 5 - 10 ml of D.I. water with stirring.
6. Repeat Steps 1, 2, and 3.
7. Combine supernate with the one in Step 4.

DeterminationA. Strontium-90 (Yttrium-90)

1. Add 3 drops of 6N HCl to dissolve the precipitate; then add 5 - 10 ml of water. Heat in a water bath at approximately 90°C. Add 1 ml of saturated oxalic acid solution dropwise with vigorous stirring. Adjust to a pH of 2 - 3 with NH₄OH. Allow the precipitate to digest for about an hour.

NOTE: Do Part "B" while precipitate is digesting.

2. Cool to room temperature in a cold water bath. Centrifuge for 10 minutes and decant most of the supernate. filter by suction on a weighed 2.5 cm filter paper. Wash the precipitate with water and alcohol.
3. Dry the precipitate under the lamp for 30 minutes. Cool and weigh. Mount and count without delay in a proportional counter. (See Part C for mounting.)

B. Strontium-89 (Total Strontium)

1. Heat the solution from Step 7 in water bath.
2. Adjust the pH to 8 - 8.5 using NH₄OH.
3. With continuous stirring, add 5 ml of 3N Na₂CO₃ solution. Stir until precipitate appears. Heat gently for 10 minutes.
4. Cool and filter on a weighed No. 42 (2.4 cm) Whatman filter paper.
5. Wash thoroughly with water and alcohol.
6. Mount and count without delay its beta activity as "total radio-strontium" in a proportional counter.

C. Filtering and Mounting

1. Place filters under heat lamps for 30 minutes before weighing.
2. Use Mettler balance (Serial No. 343112) for weighing.
3. Label a clean petri dish with the weight of the filter paper. (After samples are filtered, the filter paper will again be dried and weighed to determine weight of precipitate before mounting.)

C. Filtering and Mounting (continued)

4. Mount weighed filter paper and precipitate on nylon disk using 1" transparent tape to hold filter paper and 2" mylar foil placed over precipitate and held in place with slip-ring. Trim off excess mylar foil and place the mounted sample in a labeled petri dish.
5. Fill out corresponding loading sheets and place samples in counting room.

CalculationsPart A.

$$\text{Strontium-90 Concentration (pCi/liter)} = \frac{A}{B \times C \times D \times E \times F}$$

Where:

- A = Net beta count rate of yttrium 90 (cpm)
- B = Recovery of yttrium carrier
- C = Counter efficiency for counting yttrium-90 or yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/pCi)
- D = Sample volume (liters)
- E = Correction factor $e^{-\lambda t}$ for yttrium-90 decay, where t is the time from the time of decantation (Step 4, Separation) to the time of counting
- F. Correction factor $1 - e^{-\lambda t}$ for the degree of equilibrium attained during the yttrium-90 ingrowth period, where t is the time from collection of the water sample to the time of decantation (Step 4, Separation)

Part B

$$\text{Strontium-89 Concentration (pCi/liter)} = \frac{1}{B \times C} \cdot \frac{A}{D \times E} - F (G \times H + I \times J)$$

Where:

- A = Net beta count rate of "total radiostrontium" (cpm)
- B = Counter efficiency for counting strontium-89 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/pCi)
- C = Correction factor $e^{-\lambda t}$ for strontium-89 decay, where t is the time from sample collection to the time of counting
- D = Recovery of strontium carrier
- E = Volume of water sample (liters)
- F = Strontium-90 concentration (pCi/liter) from Part A
- G = Self-absorption factor for strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter, obtained from a self-absorption curve prepared by plotting the fraction of a standard activity absorbed against density thickness of the sample (mg/cm²)
- H = Counter efficiency for counting strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/pCi)
- I = Counter efficiency for counting yttrium-90 as yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/pCi)
- J = Correction factor $1 - e^{-\lambda t}$ for yttrium-90 ingrowth, where t is the time from the last decantation of the nitric acid (Step 4, Separation)

REFERENCE: Radioassay Procedures for Environmental Samples, U. S. Department of Health, Education, and Welfare. Environmental Health Series, January 1967.

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DETERMINATION OF SR-89 AND SR-90 IN
ASHED SAMPLES (VEGETATION, FISH, ETC.)

PROCEDURE NO. TIML-SR-05

Prepared by
Teledyne Isotopes Midwest Laboratory

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<u>Revision No.</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
0	07-23-86	7	<i>J. Grob</i>	<i>L. J. Huebner</i>

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DETERMINATION OF SR-89 AND SR-90 IN
ASHED SAMPLES (VEGETATION, FISH, ETC.)

Principle of Method

The sample with stable strontium and barium carriers added is leached in nitric acid and filtered. After filtration, filtrate is reduced in volume by evaporation. The residue is purified by adding iron and rare earth carriers and precipitating them as hydroxides. After a second strontium nitrate precipitation from 70% nitric acid, the nitrates are dissolved in acid again with added yttrium carrier and are stored for ingrowth of yttrium-90. The yttrium is precipitated as hydroxide and separated from strontium with the strontium being in the supernate. Each fraction is precipitated separately as an oxalate (yttrium) and carbonate (strontium) and collected on No. 42 (2.4 cm) Whatman filter for counting.

Reagents

Ammonium acetate buffer: pH 5.0

Ammonium hydroxide, NH_4OH : concentrated (15N), 6 N

Carrier solutions: Ba^{+2} as barium nitrate, $\text{Ba}(\text{NO}_3)_2$: 20 mg Ba^{+2} per ml

Sr^{+2} as strontium nitrate, $\text{Sr}(\text{NO}_3)_2$: 20 mg Sr^{+2} per ml

Y^{+3} as yttrium nitrate, $\text{Y}(\text{NO}_3)_3$: 10 mg Y^{+3} per ml

Hydrochloric acid, HCl : 6 N

Nitric acid, HNO_3 : Fuming (90%), concentrated (16 N), 6 N

Oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$: Saturated at room temperature

Scavenger solutions: 20 mg Fe^{+3} per ml, 10 mg each Ce^{+3} and Zr^{+4} per ml -

Fe^{+3} as ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

Ce^{+3} as cerous nitrate, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$

Zr^{+4} as zirconyl chloride, $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$

Sodium carbonate, Na_2CO_3 : 3N, 0.1N

Sodium chromate, Na_2CrO_4 : 3N

Apparatus

Analytical balance

Low background beta counter

pH meter

Procedure

1. Weigh 3 g of ash and transfer to the 250 ml beaker.
2. Add 50 ml concentrated nitric acid.
3. Add 1 ml strontium and 1 ml barium carrier solutions.
4. Place the sample on the moderate hot plate under the hood and cover with the watch glass.
5. Allow to leach for 2 hours or longer.
6. Remove sample beaker from the hot plate and allow to cool to room temperature.
7. Add deionized water, filling to 100 ml; mark on the beaker.
8. Filter the sample through Whatman No. 541 filter paper.
9. Place the filtrate on the moderate hot plate under the hood and gently evaporate to 5 ml.
10. Transfer the sample into 40 ml centrifuge tube. Rinse the beaker with 16N HNO₃. Add rinsing to the tube.
11. Centrifuge for 10 minutes and discard the supernate to waste.
12. Carefully add 30 ml of concentrated HNO₃ to the precipitate. Heat in a hot water bath for about 30 minutes, stirring occasionally. Cool the sample in an ice water bath for about 5 minutes. Centrifuge and discard the supernate.
13. Repeat Step 12.
14. Dissolve the nitrate precipitate in about 10 ml of D.I. water (perform under the hood). Add 1 ml of scavenger solution. Adjust the pH of the mixture to 7 with 6N NH₄OH. Heat in hot water bath for 10 minutes, stir, and filter through a Whatman No. 541 filter into another 40 ml centrifuge tube. Discard the mixed hydroxide precipitate (filter paper).
15. To the filtrate, add 5 ml of ammonium acetate buffer. Adjust the pH with 6N HNO₃ or NH₄OH to pH 5.5.

NOTE: The pH of the solution at this point is critical.

Add dropwise with stirring 1 ml of 3N Na₂CrO₄ solution, stir, and heat in a water bath.

16. Cool and centrifuge. Decant the supernate into another 40 ml centrifuge tube. (Save the precipitate for Ba analysis if needed.)

Procedure (continued)

17. Heat the supernate in a water bath. Adjust the pH to 8 - 8.5 with NH_4OH . With continuous stirring, add 5 ml of 3N Na_2CO_3 solution. Heat gently for 10 minutes. Cool, centrifuge, and decant the supernate to waste. Wash the precipitate with 0.1N Na_2CO_3 . Centrifuge again and decant the supernate to waste.
18. Dissolve the precipitate in no more than 4 ml of 3N HNO_3 . Then add 20 - 30 ml of fuming HNO_3 , cover with parafilm, cool in a water bath, and centrifuge. Decant and discard the supernate.
19. Repeat Step 13. Then RECORD THE TIME AND DATE AS THE BEGINNING OF YTTRIUM-90 INGROWTH.
20. Dissolve precipitate in 4 ml of 6N HNO_3 and add 1 ml of yttrium carrier solution.
21. Cover with parafilm and store for 7 - 14 days.

NOTE: At this point, the sample can be transferred to a glass scintillation vial for the ingrowth storage. Use several portions of 6N HNO_3 (a total of not more than 4 ml); then add 1 ml of yttrium carrier to the vial.)

Separation

NOTE: If the sample was stored in the scintillation vial, transfer back into 40 ml centrifuge tube using a few drops of 6N HNO_3 as a rinse.

1. After storage (ingrowth period), heat the 40 ml centrifuge tube containing the sample in the hot water bath (approximately 90°C) for 10 minutes.
2. Adjust pH to 8 with NH_4OH , stirring continuously.
3. Cool in a cold water bath and centrifuge for 5 minutes.
4. Decant the supernate into a 40 ml centrifuge tube marked with the sample number and "SR-89." RECORD THE DATE AND TIME OF DECANTATION AS THE END OF Y-90 INGROWTH in SR fraction and the beginning of its decay in Y-90 fraction.
5. Redissolve the precipitate by adding 3 - 4 drops of 6N HCl and add 5 - 10 ml of D.I. water with stirring.
6. Repeat Steps 1, 2, and 3.
7. Combine supernate with the one in Step 4.

DeterminationA. Strontium-90 (Yttrium-90)

1. Add 3 drops of 6N HCl to dissolve the precipitate; then add 5 - 10 ml of water. Heat in a water bath at approximately 90°C. Add 1 ml of saturated oxalic acid solution dropwise with vigorous stirring. Adjust to a pH of 2 - 3 with NH₄OH. Allow the precipitate to digest for about an hour.

NOTE: Do Part "B" while precipitate is digesting.

2. Cool to room temperature in a cold water bath. Centrifuge for 10 minutes and decant most of the supernate. Filter by suction on a weighed 2.5 cm filter paper. Wash the precipitate with water and alcohol.
3. Dry the precipitate under the lamp for 30 minutes. Cool and weigh. Mount and count without delay in a proportional counter. (See Part C for mounting.)

B. Strontium-89 (Total Strontium)

1. Heat the solution from Step 7 in water bath.
2. Adjust the pH to 8 - 8.5 using NH₄OH.
3. With continuous stirring, add 5 ml of 3N Na₂CO₃ solution. Stir until precipitate appears. Heat gently for 10 minutes.
4. Cool and filter on a weighed No. 42 (2.4 cm) Whatman filter paper.
5. Wash thoroughly with water and alcohol.
6. Mount and count without delay its beta activity as "total radio-strontium" in a proportional counter.

C. Filtering and Mounting

1. Place filters under heat lamps for 30 minutes before weighing.
2. Use Mettler balance (Serial No. 343112) for weighing.
3. Label a clean petri dish with the weight of the filter paper. (After samples are filtered, the filter paper will again be dried and weighed to determine weight of precipitate before mounting.)

C. Filtering and Mounting (continued)

4. Mount weighed filter paper and precipitate on nylon disk using 1" transparent tape to hold filter paper and 2" mylar foil placed over precipitate and held in place with slip-ring. Trim off excess mylar foil and place the mounted sample in a labeled petri dish.
5. Fill out corresponding loading sheets and place samples in counting room.

CalculationsPart A

Strontium-90 Concentration (pCi/g wet) =

$$\frac{A}{2.22 \times B \times C \times D \times E \times F \times G} \pm \frac{2 \sqrt{E_{sb}^2 + E_b^2}}{2.22 \times B \times C \times D \times E \times F \times G}$$

Where:

- A = Net beta count rate of yttrium 90 (cpm)
- B = Recovery of yttrium carrier
- C = Counter efficiency for counting yttrium-90 or yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- D = Sample size (grams), ash
- E = Correction factor $e^{-\lambda t}$ for yttrium-90 decay, where t is the time from the time of decantation (Step 4, Separation) to the time of counting
- F = Correction factor $1 - e^{-\lambda t}$ for the degree of equilibrium attained during the yttrium-90 ingrowth period, where t is the time from collection of the water sample to the time of decantation (Step 4, Separation)
- G = Ratio of wet weight to ashed weight
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background

Part B

Strontium-89 Concentration (pCi/g wet) =

$$\frac{1}{2.22 \times B \times C} \left[\frac{A}{D \times E \times K} - F (G \times H + I \times J) \right] \pm \frac{2 \sqrt{E_{sb}^2 + E_b^2}}{2.22 \times B \times C \times D \times E \times F \times K}$$

Where:

- A = Net beta count rate of "total radiostrontium" (cpm)
- B = Counter efficiency for counting strontium-89 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- C = Correction factor $e^{-\lambda t}$ for strontium-89 decay, where t is the time from sample collection to the time of counting
- D = Recovery of strontium carrier
- E = Sample size (grams), ash
- F = Strontium-90 concentration (pCi/g wet) from Part A
- G = Self-absorption factor for strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter, obtained from a self-absorption curve prepared by plotting the fraction of a standard activity absorbed against density thickness of the sample (mg/cm^2)
- H = Counter efficiency for counting strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- I = Counter efficiency for counting yttrium-90 as yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- J = Correction factor $1 - e^{-\lambda t}$ for yttrium-90 ingrowth, where t is the time from the last decantation of the nitric acid (Step 4, Separation)
- K = Ratio of wet weight to ashed weight

REFERENCE: Radioassay Procedures for Environmental Samples, U. S. Department of Health, Education, and Welfare. Environmental Health Series, January 1967.

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DETERMINATION OF SR-89 AND SR-90 IN
SOIL AND BOTTOM SEDIMENTS

PROCEDURE NO. TIML-SR-06

Prepared by
Teledyne Isotopes Midwest Laboratory

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DETERMINATION OF SR-89 AND SR-90 IN
SOIL AND BOTTOM SEDIMENTS

Principle of Method

The sample with stable strontium and barium carriers added is leached in hydrochloric acid. After separation from calcium, the residue is purified by adding iron and rare earth carriers and precipitating them as hydroxides. After a second strontium nitrate precipitation from 70% nitric acid, the nitrates are dissolved in acid again with added yttrium carrier and are stored for ingrowth of yttrium-90. The yttrium is precipitated as hydroxide and separated from strontium with the strontium being in the supernate. Each fraction is precipitated separately as an oxalate (yttrium) and carbonate (strontium) and collected on No. 42 (2.4 cm) Whatman filter for counting.

Reagents

Ammonium acetate buffer: pH 5.0

Ammonium hydroxide, NH_4OH : concentrated (15N), 6N

Carrier solutions: Ba^{+2} as barium nitrate, $\text{Ba}(\text{NO}_3)_2$: 20 mg Ba^{+2} per ml

Sr^{+2} as strontium nitrate, $\text{Sr}(\text{NO}_3)_2$: 20 mg Sr^{+2} per ml

Y^{+3} as yttrium nitrate, $\text{Y}(\text{NO}_3)_3$: 10 mg Y^{+3} per ml

Hydrochloric acid, HCl : 6 N

Nitric acid, HNO_3 : Fuming (90%), concentrated (16N), 6N, 1:1

Oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$: Saturated at room temperature

Scavenger solutions: 20 mg Fe^{+3} per ml, 10 mg each Ce^{+3} and Zr^{+4} per ml

Fe^{+3} as ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

Ce^{+3} as cerous nitrate, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$

Zr^{+4} as zirconyl chloride, $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$

Sodium carbonate, Na_2CO_3 : 3N, 0.1N

Sodium chromate, Na_2CrO_4 : 3N

Apparatus

Analytical balance

Centrifuge

Hot plate

Low background beta counter

pH meter

Plastic disc and ring

Stirrer

Procedure

1. Weigh out a 100 g sample into a 1 liter beaker. Add 1 ml of strontium carrier and 1 ml of Ba carrier.
2. Stir mechanically while slowly adding 200 ml of 6N HCl. (It may be necessary to add a few drops of octyl alcohol to prevent excessive frothing.) Continue stirring for about 30 minutes. Allow a minimum of two hours for the insoluble material to settle.
3. Stir the mixture and filter with suction through a 24 cm Whatman No. 42 filter paper using a Buchner funnel. Wash the residue with hot water. Wash with 6N HCl and again with hot water until the yellow color of ferric chloride is removed. Discard the residue.
4. Transfer the filtrate to a 1 liter beaker and evaporate to approximately 200 ml. Cool and slowly add 200 ml of concentrated HNO₃. (If there is excessive frothing, add a few drops of octyl alcohol.) Evaporate to 100-200 ml.
5. Add 500 ml of water and stir.
6. Add 25 grams of oxalic acid with magnetic stirring until it is completely dissolved.
7. Adjust the pH to 5.5 - 6.0 with concentrated NH₄OH. (If the brown color of ferric hydroxide persists, add more oxalic acid and readjust the pH.) The optimum condition is an excess of oxalic acid in solution without causing crystallization of ammonium oxalate upon cooling.
8. Allow precipitate to settle for 5 - 6 hours or overnight.
9. Decant most of the supernate (liquid) and transfer the precipitate to a 250 ml centrifuge tube using deionized water for rinsing. Add rinsing to the tube. Centrifuge and decant supernate.
10. Wash the precipitate with 50 - 100 ml portion of water and centrifuge again.
11. Repeat washing as needed until all the yellow color of the solution has been removed.
12. Cool the precipitate and dissolve it with 6N HNO₃ and transfer it in a 250 ml beaker. Rinse the tube with 6N HNO₃, making the total volume to 50 - 100 ml. Add about 6 drops of H₂O₂ (30%) to facilitate dissolution.
13. Cool to room temperature. If insoluble material is present at this point, filter by suction through a glass fiber filter. Discard the filter and residue.

Procedure (continued)

14. Transfer the solution to an appropriate size beaker and evaporate to dryness. The evaporation must be done slowly to avoid spattering.
15. Dissolve the salt in water and perform successive fuming nitric acid separations (the first two separations at concentration slightly greater than 75%) until the strontium has been separated from the bulk of the calcium. Samples with a high calcium content will require five or more separations.
16. The volumes of 75% HNO_3 vary (fuming solutions may be changed as required by the mass of calcium present, keeping in mind that minimum volumes are always best).
17. If calcium content is still thick, evaporate the solution to dryness and bake.
18. Dissolve the residue with 50 ml boiling water and filter. Discard residue.
19. Evaporate the solution to dryness again.
20. Cool and dissolve the residue in a minimum amount of water and add 50 ml of fuming HNO_3 .
21. Continue the fuming nitric acid separations until the strontium has been separated from the bulk of calcium.
22. Transfer the solution to a 40 ml conical, heavy-duty centrifuge tube, using a minimum of concentrated HNO_3 to effect the transfer. Cool the centrifuge tube in an ice bath for about 10 minutes. Centrifuge and discard the supernatant.

NOTE: The precipitate consists of calcium, strontium, and barium-radium nitrate.

The supernatant contains part of the sample's calcium and phosphate content.

23. Add 30 ml of concentrated HNO_3 to the precipitate. Heat in a hot water bath with stirring for about 10 minutes. Cool the solution in an ice bath, stirring for about 5 minutes. Centrifuge and discard the supernatant.

NOTE: Additional calcium is removed from the sample.

Nitrate precipitations with 70% HNO_3 will afford a partial decontamination from soluble calcium, while strontium, barium, and radium are completely precipitated.

Procedure (continued)

23. NOTE: (continued)

The separation of calcium is best at 60% HNO₃; however, at 60% the precipitation of strontium is not complete. Therefore, it is common practice to precipitate Sr(NO₃)₂ with 70% HNO₃, which is the concentration of commercially available 16N HNO₃.

Most of the other fission products, induced activities, and actinides are soluble in concentrated HNO₃, affording a good "gross" decontamination step from a wide spectrum of radio-nuclides. The precipitation is usually repeated several times.

24. Repeat Step 23 two (2) more times.

25. Dissolve the nitrate precipitate in about 20 ml distilled water. Add 1 ml of scavenger solution. Adjust the pH of the mixture to 7 with 6N NH₄OH. Heat, stir, and filter through a Whatman No. 541 filter. Discard the mixed hydroxide precipitate.

26. To the filtrate, add 5 ml of ammonium acetate buffer. Adjust the pH with 6 N HNO₃ or NH₄OH to pH 5.5.

NOTE: The pH of the solution at this point is critical.

Add dropwise with stirring 1 ml of 3N Na₂CrO₄ solution, stir, and heat in a water bath.

27. Cool and centrifuge. Decant the supernate into another 40 ml centrifuge tube. (Save the precipitate for barium analysis if needed.)

28. Heat the supernate in a water bath. Adjust the pH to 8 - 8.5 with NH₄OH. With continuous stirring, add 5 ml of 3N Na₂CO₃ solution. Heat gently for 10 minutes. Cool, centrifuge, and decant the supernate to waste. Wash the precipitate with 0.1N Na₂CO₃. Centrifuge again and decant the supernate to waste.

29. Dissolve the precipitate in no more than 4 ml of 3N HNO₃. Then add 20 - 30 ml of fuming HNO₃, cover with parafilm, cool in a water bath, and centrifuge. Decant and discard the supernate.

30. Repeat Step 13. Then RECORD THE TIME AND DATE AS THE BEGINNING OF YTTRIUM-90 INTROWTH.

31. Dissolve precipitate in 4 ml of 6N HNO₃ and add 1 ml of yttrium carrier solution.

Procedure (continued)

32. Cover with parafilm and store for 7 - 14 days.

NOTE: At this point, the sample can be transferred to a glass scintillation vial for the ingrowth storage. Use several portions of 6N HNO₃ (a total of not more than 4 ml); then add 1 ml of yttrium carrier to the vial.)

Separation

NOTE: If the sample was stored in the scintillation vial, transfer back into 40 ml centrifuge tube using a few drops of 6N HNO₃ as a rinse.

1. After storage (ingrowth period), heat the 40 ml centrifuge tube containing the sample in the hot water bath (approximately 90°C) for 10 minutes.
2. Adjust pH to 8 with concentrated NH₄OH, stirring continuously.
3. Cool in a cold water bath and centrifuge for 5 minutes.
4. Decant the supernate into a 40 ml centrifuge tube marked with the sample number and "SR-89." RECORD THE DATE AND TIME OF DECANTATION AS THE END OF Y-90 INGROWTH in SR fraction and the beginning of its decay in Y-90 fraction.
5. Redissolve the precipitate by adding 3 - 4 drops of 6N HCl and add 5 - 10 ml of D.I. water with stirring.
6. Repeat Steps 1, 2, and 3.
7. Combine supernate with the one in Step 4.

DeterminationA. Strontium-90 (Yttrium-90)

1. Add 3 drops of 6N HCl to dissolve the precipitate; then add 5 - 10 ml of water. Heat in a water bath at approximately 90°C. Add 1 ml of saturated oxalic acid solution dropwise with vigorous stirring. Adjust to a pH of 2 - 3 with concentrated NH₄OH. Allow the precipitate to digest for about an hour.

NOTE: Do Part "B" while precipitate is digesting.

Determination (continued)A. Strontium-90 (Yttrium-90) (continued)

2. Cool to room temperature in a cold water bath. Centrifuge for 10 minutes and decant most of the supernate. Filter by suction on a weighed 2.5 cm filter paper. Wash the precipitate with water and alcohol.
3. Dry the precipitate under the lamp for 30 minutes. Cool and weigh. Mount and count without delay in a proportional counter. (See Part C for mounting.)

B. Strontium-89 (Total Strontium)

1. Heat the solution from Step 7 in water bath.
2. Adjust the pH to 8 - 8.5 using concentrated NH_4OH .
3. With continuous stirring, add 5 ml of 3N Na_2CO_3 solution. Stir until precipitate appears. Heat gently for 10 minutes.
4. Cool and filter on a weighed No. 42 (2.4 cm) Whatman filter paper.
5. Wash thoroughly with water and alcohol.
6. Mount and count without delay its beta activity as "total radio-strontium" in a proportional counter.

C. Filtering and Mounting

1. Place filters under heat lamps for 30 minutes before weighing.
2. Use Mettler balance (Serial No. 343112) for weighing.
3. Label a clean petri dish with the weight of the filter paper. (After samples are filtered, the filter paper will again be dried and weighed to determine weight of precipitate before mounting.)
4. Mount weighed filter paper and precipitate on nylon disk using 1" transparent tape to hold filter paper and 2" mylar foil placed over precipitate and held in place with slip-ring. Trim off excess mylar foil and place the mounted sample in a labeled petri dish.
5. Fill out corresponding loading sheets and place samples in counting room.

CalculationsPart A

Strontium-90 Concentration (pCi/g dry) =

$$\frac{A}{2.22 \times B \times C \times D \times E \times F} \pm \frac{2\sqrt{E_{sb}^2 + E_b^2}}{2.22 \times B \times C \times D \times E \times F}$$

Where:

- A = Net beta count rate of yttrium 90 (cpm)
- B = Recovery of yttrium carrier
- C = Counter efficiency for counting yttrium-90 or yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- D = Sample weight (grams), dry
- E = Correction factor $e^{-\lambda t}$ for yttrium-90 decay, where t is the time from the time of decantation (Step 4, Separation) to the time of counting
- F = Correction factor $1 - e^{-\lambda t}$ for the degree of equilibrium attained during the yttrium-90 ingrowth period, where t is the time from collection of the water sample to the time of decantation (Step 4, Separation)
- E_{sb} = Counting error of sample plus background
- E_b = Counting error of background

Part B

Strontium-89 Concentration (pCi/g dry) =

$$\frac{1}{2.22 \times B \times C} \left[\frac{A}{D \times E} - F (G \times H + I \times J) \right] \pm \frac{2 \sqrt{E_{sb}^2 + E_b^2}}{2.22 \times B \times C \times D \times E \times F}$$

Where:

- A = Net beta count rate of "total radiostrontium" (cpm)
- B = Counter efficiency for counting strontium-89 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- C = Correction factor $e^{-\lambda t}$ for strontium-89 decay, where t is the time from sample collection to the time of counting
- D = Recovery of strontium carrier
- E = Sample weight (grams), dry
- F = Strontium-90 concentration (pCi/g dry) from Part A
- G = Self-absorption factor for strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter, obtained from a self-absorption curve prepared by plotting the fraction of a standard activity absorbed against density thickness of the sample (mg/cm²)
- H = Counter efficiency for counting strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- I = Counter efficiency for counting yttrium-90 as yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/dpm)
- J = Correction factor $1 - e^{-\lambda t}$ for yttrium-90 ingrowth, where t is the time from the last decantation of the nitric acid (Step 4, Separation).

REFERENCE: Radioassay Procedures for Environmental Samples, U. S. Department of Health, Education, and Welfare. Environmental Health Series, January 1967.



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DETERMINATION OF SR-89 AND SR-90 IN MILK

(ION EXCHANGE BATCH METHOD)

PROCEDURE NO. TIML-SR-07

Prepared by
 Teledyne Isotopes Midwest Laboratory

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<u>Revision #</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
0	06-15-88	9		
1	11-20-90	10		
2	06-12-92	10		
3	12-07-93	10		
4	08-18-94	10		

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DETERMINATION OF SR-89 AND SR-90 IN MILK (ION EXCHANGE BATCH METHOD)Principle of Method

A citrate complex of strontium carrier at the pH of milk is added to the milk sample. Strontium, barium, and calcium are absorbed on the cation-exchange resin.

Strontium, barium, and calcium are eluted from the cation-exchange resin with sodium chloride solution. Following dilution of the eluate, the alkaline earths are precipitated as carbonates. The carbonates are then converted to nitrates. Strontium is purified by Argonne method (modified at Teledyne Isotopes Laboratory in Westwood, NJ, and TIML) using three grams of extraction material in a chromatographic column. Yttrium carrier is added and a sample is stored for ingrowth of yttrium-90. The yttrium is again precipitated as hydroxide and separated from strontium with the strontium being in the supernate. Each fraction is precipitated separately as an oxalate (yttrium) and carbonate (strontium) and collected on No. 42 (2.4 cm) Whatman filter for counting.

The concentration of Sr-89 is calculated as the difference between the activity for "total radiostrontium" and the activity due to Sr-90.

Reagents

Ammonium hydroxide, NH₄OH: concentrated (15N)

Carrier solutions:

Sr⁺² as strontium nitrate, Sr(NO₃)₂: 20mg Sr⁺² per mL

Y⁺³ as yttrium nitrate, Y(NO₃)₃: 10 mg Y⁺³ per mL

Cation-exchange resin: Dowex 50W-X8 (Na⁺ form, 50-100 mesh)

Citrate solution: pH 6.5

DI water

Ethyl alcohol, C₂H₅OH: 95%

Hydrochloric acid, HCl: 6N

Nitric acid, HNO₃: 3N

Oxalic acid, H₂C₂O₄·2H₂O: 2N

Sodium carbonate, Na₂CO₃: 3N

Sodium chloride, NaCl: 4N

Silver nitrate, AgNO₃: 1N

Strontium Spec Resin

Apparatus

Ion-exchange system: The apparatus for this system is illustrated in Figure Sr-07-1. At the top is a 1-liter glass separatory funnel which serves as the reservoir. Below it is connected a 250 mL glass column, 5 cm in diameter and 25 cm long, which services as the cation column. Column has extra coarse, fritted glass disc at the bottom.

Millipore filtering apparatus (Pyrex Hydrosol Microanalysis Filter Holder)

Chromatographic Column

|3

Preparation and regeneration of cation resin:

1. Wash 170 mL of Dowex 50W resin to fill the cation column.
2. Pass 500 mL of 1N NaOH through the column at a flow rate of 10 mL/minute.
3. Rinse with 500-1000 mL of H₂O.
4. Test effluent with AgNO₃. If effluent is clear, the resin is ready for milk.

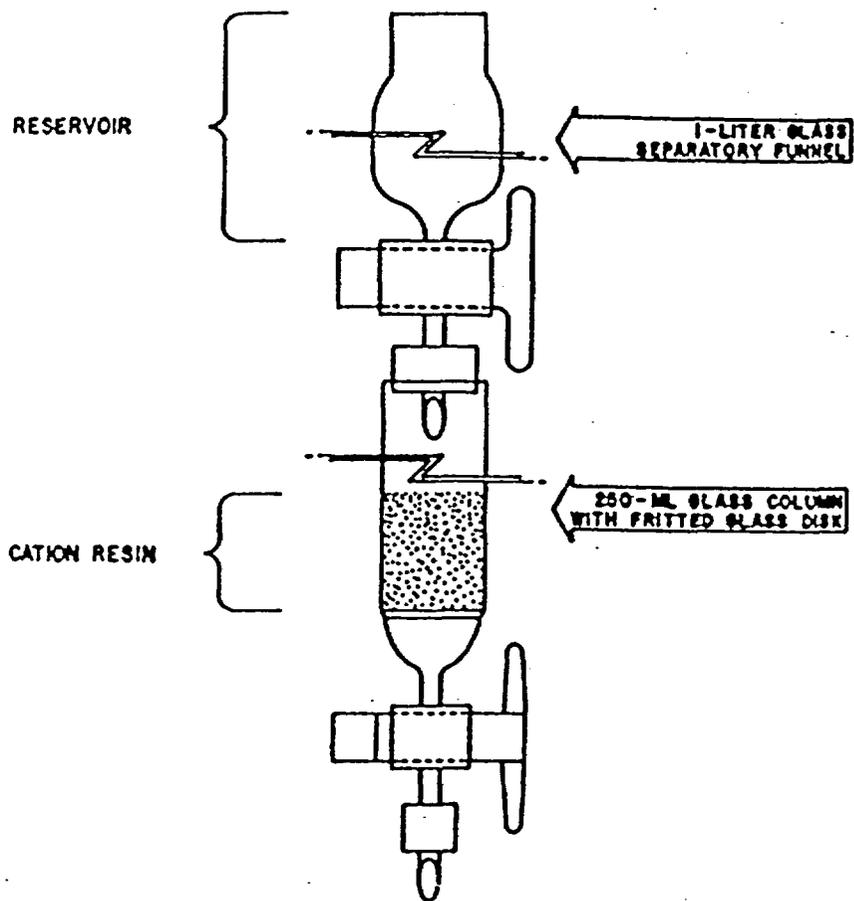


Figure SR-07-01

Part ATotal Radiostrontium (Sr-89, -90 Separation)Procedure

1. Place 1 liter of milk in 4 liter beaker.
2. Pipette 1.0 mL of strontium carrier solution into 10 mL of citrate solution. Swirl to mix. | 3
3. Transfer the mixture quantitatively to the milk with 5 mL of DI water.
4. Add a clean magnetic stirring bar to each sample beaker. Stir each sample for 5 minutes or longer on a magnetic stirrer. Allow sample to equilibrate at least 1/2 hour. If a milk sample is curdled or lumpy, vacuum filter the sample through a Buchner funnel using a cheesecloth filter. Wash the curd thoroughly with deionized water, collecting the washings with the filtrate. Pour the filtrate back into the original washed and labeled 4-liter beaker and discard the curd.
5. Add approximately 170 mL of Dowex 50Wx8 (50-100 mesh) cation resin to each sample beaker and stir on a magnetic stirrer for 2 hours. Turn off the stirrer and allow the resin to settle for 10 minutes. | 3
6. Gently decant and discard the milk sample, taking care to retain as much resin as possible in the beaker. Add approximately 1 liter of deionized water to rinse the resin, allow to settle 2 minutes, and pour off the rinse. Repeat rinsing until all traces of milk are removed from the resin.
7. Using a DI water wash bottle, transfer the resin to the column marked with the sample number. Allow resin to settle 2 minutes and drain the standing water.
8. Connect 1-liter separatory funnel containing 1 liter of 4N NaCl to the cation column. Allow the solution to flow at 10 mL/minute to elute the alkali metal and alkaline earth ions and to recharge the column. Collect 1 liter of eluate into a 2-liter beaker, but leave the resin covered with 2-3 mL of solution.
9. Wash the column with 500 mL of H₂O or more to remove excess NaCl. Discard the wash.
10. Remove 20 mL of the NaCl eluate into a small bottle for the determination of stable calcium, if required (see procedure on calcium determination).
11. Dilute the eluate to 1500 mL with DI water. | 3
12. Heat the solution to 85-90°C (near boiling on a hot plate) and add, with constant stirring, 100 mL of 3N Na₂CO₃. Cover with watch glass. Let stand overnight.
13. Decant most of the supernate to waste. Transfer the precipitate to a 250 mL centrifuge bottle with DI water.
14. Centrifuge. Pour off the supernate to waste. Dry the precipitate in an oven at 100°C for 1-2 hours. Cool. | 3
15. Dissolve the precipitate in 30 mL 3M HNO₃.

16. Place each sample centrifuge tube in front of a SR extraction column. Write sample numbers on gummed labels and attach to the corresponding columns.
17. Condition columns by passing 30 mL 3M HNO₃ through them with the stopcocks fully open. Catch effluent in a waste beaker.
18. Add sample from the beaker into the correspondingly numbered column.

NOTE: Use no water to make this transfer. Use only 3M HNO₃ to rinse out the beaker.

Allow the sample to pass through the column. Catch effluent in a waste beaker.

19. When the column reservoir is drained, measure 70 mL 3M HNO₃ in a graduated cylinder and pass through the column to rinse. Catch effluent in a waste beaker. When the column is drained, RECORD THE DATE AND TIME ON THE WORK SHEET AS THE BEGINNING OF Y-90 INGROWTH.
 20. Write the sample number on a clean 150 mL beaker. Place it under the column after the rinse solution has drained. Discard the contents of the waste beaker.
 21. Elute strontium by adding 70 mL DI water to the column. Catch effluent in the 150 mL beaker.
 22. When the elution is complete, add 1.00 mL standardized yttrium carrier to the numbered sample beaker using an Eppendorf pipet.
 23. Place sample beaker on a moderate hotplate and evaporate gently to approximately 10 mL volume. Remove beaker from hotplate and allow to cool.
- NOTE: If the sample accidentally evaporates to dryness, allow it to cool, then add a few drops HNO₃ and approximately 10 mL DI water. Warm gently and swirl to dissolve residue.
24. Mark the sample number on a 40 mL centrifuge tube. Transfer the sample using the minimum amount of DI water.
 25. Seal the sample tube with parafilm and place in a rack to stand for a minimum 5-day period for Y-90 ingrowth.
 26. Rinse the SR extraction columns with an additional 70 mL DI water. Catch effluent in a waste beaker. Leave the columns wet with DI water, with the stopcocks closed.
 27. Enter column number, date, and sample number in the SR Column Log.

Separation

1. After storage (ingrowth period), heat the 40mL centrifuge tube containing the sample in the hot water bath (approximately 90°C) for 10 minutes.
2. Adjust pH to 8.0-8.5 with NH₄OH, stirring continuously.
3. Cool in a cold water bath and centrifuge for 5 minutes.
4. Decant the supernate into a 40mL centrifuge tube marked with the sample number and "SR-89." RECORD THE DATE AND TIME OF DECANTATION AS THE END OF Y-90 INGROWTH IN SR FRACTION AND THE BEGINNING OF ITS DECAY IN Y-90 FRACTION.
5. Redissolve the precipitate by adding 3-4 drops of 6N HCl and add 5-10 mL of DI water with stirring.
6. Repeat Steps 1, 2, and 3.
7. Combine supernate with the one in Step 4:
8. Wash the precipitate twice with 20mL portions of DI Water. Centrifuge each time and discard supernate.
9. Proceed with determination.

DeterminationA. Strontium-90 (Yttrium-90)

1. Add 3 drops of 6N HCl to dissolve the precipitate from Step 4, Separation; then add 5-10 mL of DI water. Heat in a water bath at approximately 90°C for about 10 minutes. Add 1 ml of saturated oxalic acid solution dropwise with vigorous stirring. Adjust to a pH of 2-3 with NH₄OH. Allow the precipitate to digest for approximately one hour. | 3

NOTE: Do Part "B" while precipitate is digesting.

2. Cool to room temperature in a cold water bath. Centrifuge for 10 minutes and decant most of the supernate to waste. Filter by suction on a weighed 2.5 cm filter paper. Wash the precipitate with water and alcohol. | 3
3. Dry the precipitate under the lamp for 30 minutes. Cool and weigh. Mount and count in a proportional counter. (See Part C for mounting.)

B. Strontium-89 (Total Strontium)

1. Heat the solution from Step 7, Separation, in water bath.
2. Adjust the pH to 8-8.5 using NH₄OH.
3. With continuous stirring, add 5 mL of 3N Na₂CO₃ solution. Stir until precipitate appears. Heat gently for 10 minutes.
4. Cool and filter on a weighed No. 42 (2.4 cm) Whatman filter paper.
5. Wash precipitate with water and alcohol. | 3
6. Dry the precipitate under the lamp for 30 minutes. Cool and weigh. Mount and count in a proportional counter. (See Part C for mounting.)

C. Filtering and Mounting

1. Place filters under heat lamps for 30 minutes before weighing.
2. Use Mettler balance (Serial No. 343112) for weighing.
3. Label a clean petri dish with the weight of the filter paper. (After samples are filtered, the filter paper will again be dried and weighed to determine weight of precipitate before mounting.)
4. Mount weighed filter paper and precipitate on nylon disc using 1" transparent tape to hold filter paper and 2" mylar foil placed over precipitate and held in place with slip-ring. Trim off excess mylar foil and place the mounted sample in a labeled petri dish.
5. Fill out corresponding loading sheets and place samples in counting room.

Calculations

Part A

$$\text{Strontium-90 Concentration (pCi/L)} = \frac{A}{2.22 \times B \times C \times D \times E \times F \times G}$$

Where:

$$2.22 = \text{dpm/pCi}$$

A = Net beta count rate of yttrium-90 (cpm)

B = Recovery of yttrium carrier

C = Recovery of strontium carrier

D = Counter efficiency for counting yttrium-90 as yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/dpm) | 4

E = Sample volume (liters)

F = Correction factor $e^{-\lambda t}$ for yttrium-90 decay, where t is the time from the time of decantation (Step 4, Separation) to the time of countingG = Correction factor $1 - e^{-\lambda t}$ for the degree of equilibrium attained during the yttrium-90 ingrowth period, where t is the time from the beginning of ingrowth (Step 19, Total Radiostrontium Separation) to the time of decantation (Step 4, Separation) | 4Lower Limit of Detection (LLD), at 4.66 sigma

LLD for Sr-90: 1 pCi/L. LLD is based on the following typical parameters:

Sample Size: 1 L

Recovery (Sr and Y): 0.6

Decay Factor (Y-90): 0.8

Ingrowth Factor (Y-90): 0.6

Counter Efficiency: 0.4

Counter Background: 0.3cpm | 4

Counting Time: 100 minutes

(Changes in any of the above parameters will change LLD correspondingly.)

Part B

$$\text{Strontium-89 Concentration (pCi/L)} = \frac{1}{2.22 \times B \times C} \left[\frac{A}{D \times E} - 2.22 \times F(G + H \times I) \right]$$

Where:

2.22 = dpm/pCi

A = Net beta count rate of "total radiostrontium" (cpm)

B = Counter efficiency for counting strontium-89 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/dpm) | 4

C = Correction factor $e^{-\lambda t}$ for strontium-89 decay, where t is the time from sample collection to the time of counting

D = Recovery of strontium carrier

E = Sample volume (liters)

F = Strontium-90 concentration (pCi/liter) from Part A

G = Counter efficiency for counting strontium-90 as strontium carbonate mounted on a 2.4 cm diameter filter paper (cpm/dpm) | 4

H = Counter efficiency for counting yttrium-90 as yttrium oxalate mounted on a 2.4 cm diameter filter paper (cpm/dpm) | 4

I = Correction factor $1 - e^{-\lambda t}$ for yttrium-90 ingrowth, where t is the time from the last decantation of the nitric acid (Step 4, Separation) to the time of countingLower Limit of Detection (LLD), at 4.66 sigma

LLD for Sr-89: 2.0 pCi/L. LLD is based on the following typical parameters:

Sample Size: 1 L

Recovery: 0.7

Decay Factor: 0.5

Counter Efficiency: 0.3

Counter Background: 0.3 cpm | 4

Counting Time: 100 minutes

LLD for Sr-90: 1 pCi/L

(Changes in any of the above parameters will change LLD correspondingly.)

REFERENCES: Radioassay Procedures for Environmental Samples, U. S. Department of Health, Education, and Welfare. Environmental Health Series, January 1967.Horwitz, Dietz, Fisher, Analytical Chemistry, 63 (5), March 1991.

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PROCEDURE FOR COMPOSITING
WATER AND MILK SAMPLES

PROCEDURE NO. TIML-COMP-01

Prepared by
Teledyne Isotopes Midwest Laboratory

Copy No. _____

Revised Pages	Revision No.	Date	Pages	Prepared by	Approved by
_____	0	11-07-88	2	<i>J. Gorb</i>	<i>R. G. Heesbeen</i>
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

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Procedure for Compositing Water and Milk Samples

1. At the beginning of each composite period, (month, quarter, semi-annual), prepare a one-gallon cubitainer for a specific location and time-period.
2. Remove an equal aliquot of original sample (for example, one liter) and transfer to prepared cubitainer. Do this for each week, month, etc. Mark date of original sample on prepared cubitainer.
3. When prepared container is complete, give the sample to the recording clerk for assigning a number.
4. Analyze according to the client requirement.

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DETERMINATION OF STABLE CALCIUM IN MILK

PROCEDURE NO. TIML-CA-01

Prepared by

Teledyne Isotopes Midwest Laboratory

Copy No.

<u>Revision No.</u>	<u>Date</u>	<u>Pages</u>	<u>Prepared by</u>	<u>Approved by</u>
0	07-08-88	4	<i>B. Gorb</i>	<i>L. J. Huchman</i>

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TIML-CA-01Determination of Stable Calcium in MilkPrinciple of Method

Strontium, barium, and calcium are absorbed on the cation-exchange resin, then eluted with sodium chloride solution. An aliquot of the eluate is diluted to reduce the high sodium ion concentration. From this diluted aliquot, calcium oxalate is precipitated, dissolved in dilute hydrochloric acid, and the oxalate is titrated with standardized potassium permanganate.

ReagentsAmmonium hydroxide, NH₄OH: 6NAmmonium oxalate, (NH₄)₂C₂O₄·H₂O: 0.03NCarrier solutions:Ba⁺² as barium nitrate, Ba(NO₃)₂: 20 mgBa⁺² per mlSr⁺² as strontium nitrate, Sr(NO₃)₂: 20 mg Sr⁺² per mlCation-exchange resin: Dowex 50W-X8 (Na⁺ form, 50-100 mesh)Citrate solution: 3N (pH 6.5)Hydrochloric acid, HCl: 6NOxalic acid, H₂C₂O₄·2H₂O: 1NPotassium permanganate, KMnO₄: 0.05N standardizedSodium chloride, NaCl: 4NSodium oxalate, Na₂C₂O₄:Apparatus

Burette

Procedure

1. Follow the TIML-SR-01 or SR-07 procedures, Steps 1-10.
2. Into a 40 ml glass centrifuge tube, pipette 10 ml aliquot of the initial eluate collected in Step 10.
3. Dilute the 10 ml aliquot to approximately 20 ml with D.I. water.
4. Heat in a hot water bath. Add 5 ml of 1N oxalic acid, and stir. While hot, adjust to pH 3 with 6N NH₄OH (use a pH meter) to precipitate calcium oxalate. Cool slowly to room temperature, centrifuge, and discard the supernate.

TIML-CA-01Procedure (continued)

5. Thoroughly wash the precipitate and the wall of the centrifuge tube, using not more than 5 ml of 0.03N ammonium oxalate. Centrifuge, and discard the supernatant.
6. Wash the precipitate with 10 ml of hot D.I. water. Cool to room temperature, centrifuge, and discard the supernate. (A stirring rod may be used to agitate the precipitate while it is being washed. It is important to remove all excess oxalic acid from the precipitate.)
7. Dissolve the precipitate in approximately 2.5 ml of 6N HCl. Heat in hot water bath for 5 minutes.
8. Dilute the acid solution to approximately 10 ml with D.I. water. Quantitatively transfer it to a 125 ml Erlenmeyer flask, rinsing the centrifuge tube with D.I. water.
9. Add an additional 1 ml of 6N HCl, and adjust the volume of solution to approximately 25 ml with D.I. water. Heat to near boiling.
10. While hot, titrate with standardized 0.05N KMnO_4 to the first faint pink endpoint which persists for at least 30 seconds.

Calculations

$$\text{Calcium (g/liter)} = \frac{A \times B \times C}{D}$$

Where:

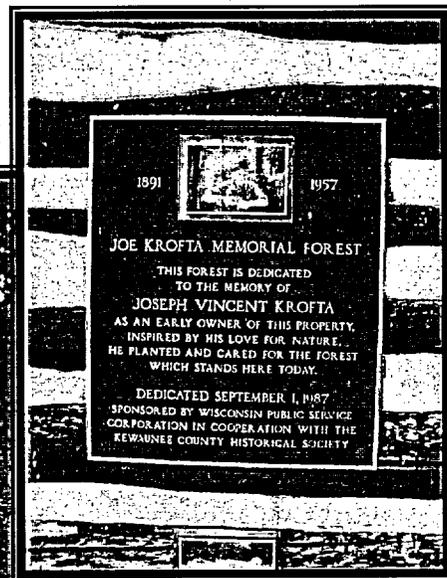
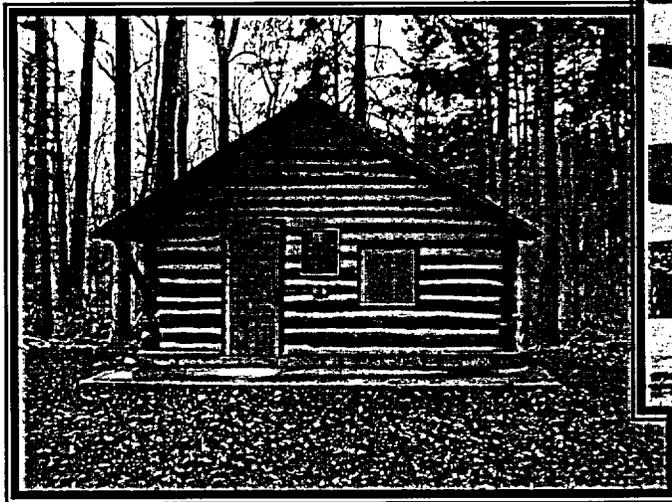
- A = Volume of KMnO_4 solution used for titration (ml)
- B = Normality of standardized KMnO_4 solution (mg/ml)
- C = Milli-equivalent weight of calcium (mg/meg)
- D = Sample volume (ml)

Since the sample size is 10 ml and the milli-equivalent weight of calcium is 20 mg, the equation reduces to:

$$\text{Calcium (g/liter)} = A \times B \times 2$$

KEWAUNEE NUCLEAR POWER PLANT

ANNUAL REPORT PART III PROGRAM SELF-ASSESSMENT AND PROGRAM CHANGES



Kewaunee's School Forest and
wetlands restoration project south of the plant

Kewaunee Nuclear Power Plant

Radiological Environmental Monitoring Manual (REMM)

*Revision 7
May 28, 2002*

Reviewed by: *D Braun* Date: *5-28-02*
Plant Operations Review Committee

Approved by: *Tom Webb* Date: *5-30-02*
Site Licensing Director

Approved by: *Stanley F. Baker* Date: *6-3-02*
Radiation Protection Manager

Approved by: *Wally F. Cook* Date: *6-4-02*
Superintendent - Plant Radiochemistry

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1.0 Introduction

1.1 Purpose

The purpose of this document is to define the Radiological Environmental Monitoring Program (REMP) for the Kewaunee Nuclear Power Plant (KNPP). The REMP is required by KNPP Technical Specification (TS) 6.16.b.2, "Radiological Environmental Monitoring Program".

This document is known as the Radiological Environmental Monitoring Manual (REMM) and is intended to serve as a tool for program administration and as a guidance document for contractors which implement the monitoring program.

1.2 Scope

This program defines the sampling and analysis schedule which was developed to provide representative measurements of radiation and of radioactive materials in those exposure pathways and for those radionuclides that lead to the high potential radiation exposures of MEMBERS OF THE PUBLIC resulting from plant operation. This monitoring program implements Section IV.B.2 of Appendix I to 10 CFR Part 50 and thereby verifies that the measurable concentrations of radioactivity and levels of radiation are not higher than expected on the basis of the effluent measurements and the modeling of the environmental exposure pathways. Guidance for the development of this monitoring program is provided by the Radiological Assessment Branch Technical Position on Environmental Monitoring. This program has been developed in accordance with NUREG 0472.

The program will provide field and analytical data on the air, aquatic, and terrestrial radioecology of the area near the Kewaunee Nuclear Power Plant so as to:

1. Determine the effects of the operation of the Kewaunee Nuclear Power Plant on the environment;
2. Serve as a gauge of the operating effectiveness of in-plant control of waste discharges; and
3. Provide data on the radiation dose to the public by direct or indirect pathways of exposure.

1.3 Implementation

This document is considered, by reference, to be part of the Offsite Dose Calculation Manual. This is as required by KNPP TS 6.16.b.2. The REMM is controlled as a separate document for ease of revision, use in the field and use by contractors. This format was approved by the NRC as part of TS Amendment No. 64, which provided Radiological Effluent Technical Specifications (RETS) for KNPP.

The REMP is setup to be implemented by a vendor and controlled by the Kewaunee Nuclear Power Plant (KNPP) in accordance with Nuclear Administrative Directive (NAD) 1.20, "Radiological Environmental Monitoring Program". Monthly reviews of the vendor's progress report are checked

and approved by KNPP in accordance with Surveillance Procedure SP 63-276. Annual reviews and submittals of the vendor's report and raw data are checked and approved by KNPP in accordance with Surveillance Procedure SP 63-280. All sample collection, preparation, and analysis are performed by the vendor except where noted. Surveillance Procedure SP 63-164 outlines the environmental sample collection performed by KNPP. Current vendor Quality Control Program Manuals and implementing procedures shall be kept on file at KNPP.

Periodic reviews of monitoring data and an annual land use census will be used to develop modifications to the existing monitoring program. Upon approval, these modifications will be incorporated into this document so that it will accurately reflect the current radiological environmental monitoring program in effect for the Kewaunee Nuclear Power Plant.

The remainder of this document is divided into two sections. The first section, 2.0 REMP Requirements, describes the different TS and REMM requirements associated with the REMP. The second section, 3.0 REMP Implementation, describes the specific requirements used to implement the REMP.

2.0 REMP Requirements

KNPP TS Amendment No. 104 implemented the guidance provided in Generic Letter 89-01, "Implementation of Programmatic Controls for Radiological Effluent Technical Specifications (RETS)". These changes included: 1) incorporation of *programmatic controls* in the Administrative Controls section of the TS to satisfy existing regulatory requirements for RETS, and 2) relocation of the *procedural details* on radioactive effluents monitoring, radiological environmental monitoring, reporting details, and other related specifications from the TS to the ODCM. Relocating the procedural details to the ODCM allows for revising these requirements using the 10 CFR 50.59 process instead of requiring prior NRC approval using the TS Amendment process.

The RETS requirements were incorporated verbatim into the ODCM, Revision 6. Several of these requirements pertain only to the environmental monitoring program and therefore have been relocated into this document (REMM, Revision 3 and 4) and are identified as REMM requirements.

2.1 Technical Specification Requirements

Technical Specification 6.16.b.2 provides the programmatic control, which requires a program to monitor the radiation and radionuclides in the environs of the plant. This is the reason for the existence of the REMP. TS 6.16.b.2 also provides the programmatic control which requires: (a) the program to perform the monitoring, sampling, analysis, and reporting in accordance with the methodology and parameters in the ODCM, (b) a land use census to be performed, and (c) participation in an Interlaboratory Comparison Program. The details of each requirement are described in the REMM requirements stated below.

Technical Specification 6.9.b.1 requires an "Annual Radiological Environmental Monitoring Report" be submitted to the NRC each year. The specific contents of this report are detailed in REMM 2.4.1. Additional specific reporting requirements are listed in the other REMM requirements.

2.2 REMM Requirements

The following REMM requirements include the procedural details that were originally located in the KNPP RETS section and then relocated into Revision 6 of the ODCM, as discussed above. These requirements are specific to the radiological environmental monitoring program and have been relocated into this document for ease of use and completeness.

The REMM requirements for the Monitoring Program, Land Use Census, and the Interlaboratory Comparison Program include a detailed specification (numbered 2.2.1, 2.2.2, & 2.2.3 respectively) and an associated surveillance requirement (numbered 2.3.1, 2.3.2, & 2.3.3 respectively), along with the basis for the requirement. Reporting requirements are listed in specification REMM 2.4.1.

General requirements also apply to all ODCM and REMM requirements (specifications 3.01, 3.02, 3.03, 4.01, 4.02, and 4.03). The requirements are located in the ODCM and are repeated here for convenience.

GENERAL SPECIFICATIONS

- 3.0.1 Compliance with the specifications contained in the succeeding text is required during the conditions specified therein; except that upon failure to meet the specifications, the associated ACTION requirements shall be met.
- 3.0.2 Noncompliance with a Specification shall exist when its requirements and associated ACTION requirements are not met within the specified time intervals. If the Specification is restored prior to expiration of the specified time intervals, completion of the Action requirements is not required.
- 3.0.3 When a Specification is not met, except as provided in the associated ACTION requirements, reporting pursuant to TS 6.9.b and REMM 2.4.1 will be initiated.

SURVEILLANCE REQUIREMENTS

- 4.0.1 Surveillance Requirements shall be met during the conditions specified for individual Specifications unless otherwise stated in an individual Surveillance Requirement.
- 4.0.2 Each Surveillance Requirement shall be performed within the specified time interval with a maximum allowable extension not to exceed 25 % of the surveillance interval.
- 4.0.3 Failure to perform a Surveillance Requirement within the specified time interval shall constitute a failure to meet the OPERABILITY requirements for a Specification. Exceptions to these requirements are stated in the individual Specification. Surveillance Requirements do not have to be performed on inoperable equipment.

REMM 2.2.1/2.3.1 Monitoring Program

SPECIFICATION

2.2.1 The radiological environmental monitoring program shall be conducted as specified in Table 2.2.1-A.

APPLICABILITY

At all times.

ACTION

- a. With the radiological environmental monitoring program not being conducted as specified in Table 2.2.1-A, in lieu of a Licensee Event Report, prepare and submit to the Commission, in the Annual Radiological Environmental Monitoring Report required by TS 6.9.b.1 and REMM 2.4.1, a description of the reasons for not conducting the program as required and the plans for preventing a recurrence.
- b. With the level of radioactivity as the result of plant effluents in an environmental sampling medium at a specified location exceeding the reporting levels of Table 2.2.1-D when averaged over any calendar quarter in lieu of a Licensee Event Report, prepare and submit to the Commission within 30 days, pursuant to TS 6.9.b.3, a Special Report that identifies the cause(s) for exceeding the limit(s) and defines the corrective actions to be taken to reduce radioactive effluents so that the potential annual dose¹ to A MEMBER OF THE PUBLIC is less than the calendar year limits of specifications ODCM 3.3.2, 3.4.2, and 3.4.3. When more than one of the radionuclides in Table 2.2.1-D are detected in the sampling medium, this report shall be submitted if:

$$\frac{\text{concentration}(1)}{\text{reporting level}(1)} + \frac{\text{concentration}(2)}{\text{reporting level}(2)} + \dots \geq 1.0$$

When radionuclides other than those in Table 2.2.1-D are detected and are the result of plant effluents, this report shall be submitted if the potential annual dose¹ to a MEMBER OF THE PUBLIC is equal to or greater than the calendar year limits of specifications ODCM 3.3.2, 3.4.2 and 3.4.3. This report is not required if the measured level of radioactivity was not the result of plant effluents; however, in such an event the condition shall be reported and described in the Annual Radiological Environmental Monitoring Report.

- c. With milk or fresh leafy vegetable samples unavailable from one or more of the sample locations required by Table 2.2.1-A, a sample from an alternative location will be substituted, noting the reason for the unavailability in the Annual Radiological Environmental Monitoring Report. When changes in sampling locations are permanent, the sampling schedule in the RADIOLOGICAL ENVIRONMENTAL MONITORING MANUAL (REMM) will be updated to reflect the new routine and alternative sampling locations and this revision will be described in the Annual Radiological Environmental Monitoring Report.

¹The methodology and parameters used to estimate the potential annual dose to a member of the public shall be indicated in this report.

SURVEILLANCE REQUIREMENTS

- 2.3.1 The radiological environmental monitoring samples shall be collected pursuant to Table 2.2.1-A from the specific locations given in the table and figure(s) in the REMM, and shall be analyzed pursuant to the requirements of Table 2.2.1-A and the detection capabilities required by Table 2.3.1-A.
-

BASIS

The radiological environmental monitoring program required by this specification provides representative measurements of radiation and of radioactive materials in those exposure pathways and for those radionuclides that lead to the highest potential radiation exposures of MEMBERS OF THE PUBLIC resulting from the station operation. This monitoring program implements Section IV.B.2 of Appendix I to 10 CFR Part 50 and thereby supplements the radiological effluent monitoring program by verifying that the measurable concentrations of radioactive materials and levels of radiation are not higher than expected on the basis of the effluent measurements and the modeling of the environmental exposure pathways. Guidance for this monitoring program is provided by the Radiological Assessment Branch Technical Position on Environmental Monitoring. Program changes may be initiated based on operational experience.

The required detection capabilities for environmental sample analyses are tabulated in terms of the lower limits of detection (LLDs). The LLDs required by Table 2.3.1-A are considered optimum for routine environmental measurements in industrial laboratories. It should be recognized that the LLD is defined as a priori (before the fact) limit representing the capability of a measurement system and not as an a posteriori (after the fact) limit for a particular measurement.

Detailed discussion of the LLD, and other detection limits, can be found in HASL Procedures Manual, HASL-300 (revised annually), Currie, L.A., "Limits for Qualitative Detection and Quantitative Determination - Application to Radiochemistry", Anal. Chem. 40, 586-93 (1968), and Hartwell, J.K., "Detection Limits for Radioanalytical Counting Techniques", Atlantic Richfield Hanford Company Report ARH-SA-215 (June 1975).

Discussion

KNPP TS 6.16.b.2(a) requires that the monitoring, sampling, analysis, and reporting of radiation and radionuclides in the environment be done in accordance with the methodology and parameters in the ODCM.

REMM 2.2.2/2.3.2 Land Use Census

SPECIFICATIONS

- 2.2.2 A land use census shall be conducted and shall identify within a distance of 8 km (5 miles) the location in each of the 10 meteorological sectors of the nearest milk animal, the nearest residence and the nearest garden² of greater than 50 m² (500 ft²) producing broad leaf vegetation.

APPLICABILITY

At all times

ACTION

- a. With a land use census identifying a location(s) that yields a calculated dose or dose commitment greater than the values currently being calculated in specification ODCM 4.4.3, in lieu of a Licensee Event Report, identify the new location(s) in the next Annual Radiological Environmental Monitoring Report pursuant to TS 6.9.b.1 and REMM 2.4.1.
- b. With a land use census identifying a location(s) that yields a calculated dose or dose commitment (via the same exposure pathway) 20% greater than at a location from which samples are currently being obtained in accordance with specification REMM 2.2.1, add the new location(s) to the radiological environmental monitoring program within 30 days. The sampling location(s), excluding the control station location, having a lower calculated dose or dose commitment(s), via the same exposure pathway, may be deleted from this monitoring program. In lieu of a Licensee Event Report, identify the new location(s) in the next Annual Radiological Environmental Monitoring Report pursuant to TS 6.9.b.1 and REMM 2.4.1 and also include in the report a revised figure(s) and table for the REMM reflecting the new location(s).

SURVEILLANCE REQUIREMENT

- 2.3.2 The land use census shall be conducted during the growing season once per 12 months using reasonable survey methods, such as by a door-to-door survey, aerial survey, or by consulting local agriculture authorities. The results of the land use census shall be included in the Annual Radiological Environmental Monitoring Report pursuant to TS 6.9.b.1 and REMM 2.4.1.

BASIS

This specification is provided to ensure that changes in the use of areas at and beyond the SITE BOUNDARY are identified and that modifications to the radiological environmental monitoring program are made if required by the door-to-door survey, from aerial survey or from consulting with local agricultural authorities. This census satisfies the requirements of Section IV.B.3 of Appendix I to 10 CFR Part 50. Restricting the census to gardens of greater than 50 m² provides assurance that significant exposure pathways via leafy vegetables will be identified and monitored since a garden of this size is the minimum required to produce the quantity (26 kg/yr) of leafy vegetables assumed in Regulatory Guide 1.109 for consumption by a child. To determine this minimum garden size, the following assumptions were made: 1) 20% of the garden was used for growing leafy vegetation (i.e., similar to lettuce and cabbage), and 2) a vegetation yield of 2 kg/m².

²Sampling of leaf vegetation may be performed at the site boundary in each of two different direction sectors with the highest predicted D/Qs in lieu of the garden census. Specifications for broad leaf vegetation sampling in Table 2.2.1-A item 4c shall be followed, including analysis of control samples.

Discussion

KNPP TS 6.16.b.2(b) requires that a land use census be performed to ensure that changes in the use of areas at and beyond site boundary are identified and that modifications to the radiological environmental monitoring program are made if required by the results of this census.

REMM 2.2.3/2.3.3 Interlaboratory Comparison Program

SPECIFICATIONS

- 2.2.3 Analyses shall be performed on radioactive materials supplied as part of an Interlaboratory Comparison Program that has been approved by the Commission.

APPLICABILITY

At all times.

ACTION

- a. With analyses not being performed as required above, report corrective actions taken to prevent a recurrence to the Commission in the Annual Radiological Environmental Monitoring Report pursuant to TS 6.9.b.1 and REMM 2.4.1.

SURVEILLANCE REQUIREMENTS

- 2.3.3 The Interlaboratory Comparison Program shall be described in the REMM. A summary of the results obtained as part of the above required Interlaboratory Comparison Program shall be included in the Annual Radiological Environmental Monitoring Report pursuant to TS 6.9.b.1 and REMM 2.4.1.
-

BASIS

The requirement for participation in an approved Interlaboratory Comparison Program is provided to ensure that independent checks on the precision and accuracy of measurements of radioactive material in environmental sample matrices are performed as part of the quality assurance program for environmental monitoring in order to demonstrate that the results are valid for the purposes of Section IV.B.2 of Appendix I to 10 CFR Part 50.

Discussion

KNPP TS 6.16.b.2(c) requires participation in an approved Interlaboratory Comparison Program to ensure that an independent check is performed of the precision and accuracy of radioactive materials measurements. This will demonstrate that the results are valid for the purposes of Section IV.B.2 of Appendix I to 10 CFR Part 50.

REMM 2.4.1 Reporting Requirements

- 2.4.1 The Annual Radiological Environmental Monitoring Report shall include:
- a. Summaries, interpretations, and an analysis of trends of the results of the radiological environmental surveillance activities for the report period, including a comparison with pre-operational studies, with operational controls as appropriate, and with previous environmental surveillance reports, and an assessment of the observed impacts of the plant operation on the environment. The reports shall also include the results of land use censuses required by specification REMM 2.2.2.
 - b. The results of analyses of radiological environmental samples and of environmental radiation measurements taken during the period pursuant to the locations specified in the table and figures in the Radiological Environmental Monitoring Manual (REMM), as well as summarized and tabulated results of these analyses and measurements in the format of the table in the Radiological Assessment Branch Technical Position, Revision 1, November 1979. In the event that some individual results are not available for inclusion with the report, the report shall be submitted noting and explaining the reasons for the missing results. The missing data shall be submitted as soon as possible in a supplementary report when applicable.
 - c. A summary description of the radiological environmental monitoring program; legible maps covering all sampling locations keyed to a table giving distances and directions from the centerline of one reactor; the results of licensee participation in the Interlaboratory Comparison Program, required by specification REMM 2.2.3; discussion of all deviations from the sampling schedule of Table 2.2.1-A; and discussion of all analyses in which the LLD required by Table 2.3.1-A was not achievable.
-

Discussion

KNPP TS 6.9.b.1 provides the programmatic control, which requires that an Annual Radiological Environmental Monitoring Report be submitted to the NRC. It also states that this report shall include summaries, interpretations, and analysis of trends of the results of the REMP for the reporting period.

The procedural details of this report are included in this specification. Specifications REMM 2.2.1, 2.2.2/2.3.2, and 2.2.3/2.3.3 also include specific reporting requirements. These specifications reference this REMM specification, along with TS 6.9.b.1, as the method for reporting deviations from the current program during the reporting period, and require that this information be included in the Annual Radiological Environmental Monitoring Report.

3.0 REMP Implementation

The Radiological Environmental Monitoring Program for KNPP is currently under the direction of Environmental, Inc. Midwest Laboratory (EIML). This section describes this program, as required by REMM 2.2.1 and the process EIML uses to perform it.

3.1 *Sampling Requirements*

Table 2.2.1-A identifies the various samples required by the REMP. Identified in the "available sample locations" column in Table 2.2.1-A are the sample locations selected, in conjunction with the vendor, to meet or exceed the REMP requirements. Table 2.2.1-B includes the same requirements as in Table 2.2.1-A but presents the information in a different format by identifying the type of samples required at each location and the collection frequency. Table 2.2.1-C identifies the location and description of each sample location. Figure 1 shows the physical location of each sample point on an area map.

3.2 *Analysis Methodology*

Analytical procedures and counting methods employed by EIML will follow those recommended by the U.S. Public Health Service publication, Radioassay Procedures for Environmental Samples, January 1967; and the U.S. Atomic Energy Commission Health and Safety Laboratory, HASL Procedures Manual (HASL-300), 1972. The manual is also available on-line at www.eml.doe.gov/publications/procman.

Updated copies will be maintained in KNPP's vault.

3.3 *Detection Capability (LLD) Requirements*

The required detection capabilities for environmental sample and analysis are tabulated in terms of lower limits of detection (LLDs) in Table 2.3.1-A. The LLDs required by Table 2.3.1-A are considered optimum for routine environmental measurements in industrial laboratories. It should be recognized that the LLD is defined as a priori (before the fact) limit representing the capability of a measurement system and not as an a posteriori (after the fact) limit for a particular measurement.

Detailed discussion of the LLD, and other detection limits, can be found in HASL Procedures Manual, HASL-300 (revised annually), Currie, L.A., "Limits for Qualitative Detection and Quantitative Determination - Application to Radiochemistry", *Anal. Chem.* 40, 586-93 (1968), and Hartwell, J.K., "Detection Limits for Radioanalytical Counting Techniques", Atlantic Richfield Hanford Company Report ARH-SA-215 (June 1975).

3.4 Environmental, Inc. Midwest Laboratory (EIML) Reporting Requirements

Monthly Progress Reports

Monthly progress reports will include a tabulation of completed analytical data on samples obtained during the previous 30-day period together with graphic representations where trends are evident, and the status of field collections. One copy of the reports will be submitted within 30 days of the reporting month.

Annual Reports

Annual reports will be submitted in two parts. Part I, to be submitted to the NRC, will be prepared in accordance with NRC Regulatory Guide 4.8. It will contain an introductory statement, a summary of results, description of the program, discussion of the results, and summary table. Part II of the annual report will include tables of analytical data for all samples collected during the reporting period, together with graphic presentation where trends are evident and statistical evaluation of the results. Gamma scan data will be complemented by figures of representative spectra. Draft copies of each annual report will be due 60 days after completion of the annual period. After final review of the draft document, one photoready copy of the revised annual report will be sent to KNPP for printing.

Non-Routine Reports

If analyses of any samples collected show abnormally high levels of radioactivity, KNPP will be notified by telephone immediately after data becomes available.

Action Limits

EIML should report any radioactive concentrations found in the environmental samples which exceed the reporting levels shown in Table 2.2.1-D, EIML to KNPP column. These levels are set below the NRC required reporting levels (KNPP to NRC column) so actions can be initiated to prevent exceeding the NRC concentration limits.

3.5 Quality Control Program

To insure the validity of the data, EIML maintains a quality control (QC) program, which employs quality control checks, with documentation, of the analytical phase of its environmental monitoring studies. The program is defined in the EIML QC Program Manual, and procedures are presented in the EIML QC Procedures Manual. The program shall be reviewed and meet the requirements of 10 CFR 50 Appendix B and 10 CFR 21. All data related to quality control will be available for review by WPS upon reasonable prior notification. Proprietary information will be identified so that it may be treated accordingly.

Updated copies of the Quality Control Program Manual and the Quality Assurance Program Manual will be maintained in KNPP's vault.

3.6 *Sample Descriptions*

A description of each of the samples required by this program follows:

Airborne Particulates

Airborne particulates are collected at six locations (K-1f, K-2, K-7, K-8, K-16, K-31) on a continuous basis on a 47 mm diameter membrane filter of 0.8 micron porosity at a volumetric rate of approximately one cubic foot per minute (CFM). The filters are changed weekly, placed in glassine protective envelopes, and dispatched by U.S. Mail to EIML for Gamma Isotopic Analysis (ref. SP 63-164). Filter samples are analyzed weekly for gross beta activity after sufficient time (usually 3 to 5 days) has elapsed to allow decay of Radon and Thoron daughters. If gross beta concentration in air particulate samples are greater than ten (10) times the yearly mean of the control samples, gamma isotopic analysis shall be performed on the individual samples. Quarterly composites from each location receive Gamma Isotopic Analysis using a Germanium detector. All identifiable gamma-emitters are quantified. Reporting units are pCi/m³.

Airborne Iodine

All air samplers are equipped with charcoal traps installed behind the particulate filters for collection of airborne I-131. The traps are changed once every two weeks. Iodine-131 is measured by Gamma Isotopic Analysis.

Periphyton (Slime) or Aquatic Vegetation

Periphyton (slime) or aquatic plant samples are collected at or near locations used for surface water sampling. They are collected twice during the year (2nd and 3rd quarter), if available. The samples are analyzed for gross beta activity and, if available in sufficient quantity, for Sr-89, Sr-90, and by Gamma Isotopic Analysis. Reporting units are pCi/g wet weight.

Fish

Fish is collected three times per year (second, third, and fourth quarters) near the discharge area (K-1d) (ref. RC-C-207). Flesh is separated from the bones and analyzed for gross beta activity and by Gamma Isotopic Analysis. The bones are analyzed for gross beta activity and Sr-89 and Sr-90. Reporting units are pCi/g wet weight.

Domestic Meat

Domestic meat (chickens) is collected once a year during the 3rd quarter, at five locations in the vicinity of the plant (K-20, K-24, K-27, K-29 and K-32). Samples may not be available every year at every location due to farmer preference. At least one control and one indicator should be collected. The flesh is analyzed for gross alpha, gross beta, and by Gamma Isotopic Analysis to identify and quantify gamma-emitting radionuclides. Reporting units are pCi/g wet weight.

Ambient Radiation

Two packets of thermoluminescent dosimeters (CaSO₄: Dy cards) are placed at fourteen locations, six of which are air sampling locations (K-1f, K-2, K-7, K-8, K-16 and K-31) and four of which are milk sampling locations (K-3, K-5, K-25 and ~~K-37~~); the remaining four locations are K-15, K-17, K-27, and K-30. One packet is changed quarterly and one annually. Annual TLDs will serve as an emergency set to be read when needed. They will be exchanged annually (without reading) if not read during the year. To insure the precision of the measurement, each packet will contain two cards with four dosimeters each (four sensitive areas each for a total of eight). For protection against moisture each set of cards is sealed in a plastic bag and placed in a plastic container.

Each card is individually calibrated for self-irradiation and light response. Fading is guaranteed by the manufacturer (Teledyne Isotopes) not to exceed 20% in one year. Minimum sensitivity for the multi-area dosimeter is 0.5 mR defined as 3 times the standard deviation of the background. Maximum Error (1 standard deviation) - ⁶⁰Co Gamma +/-0.2 mR or +/-3%, whichever is greater. The maximum spread between areas on the same dosimeter is 3.5% at 1 standard deviation.

Reporting units for TLDs are mR/91 days for quarterly TLDs and mR/exposure period for annual TLDs.

Tests for uniformity and reproducibility of TLDs as specified in ANSI N545-1981 and NRC Regulatory Guide 4.13, are performed annually.

Well Water

One-gallon water samples are taken once every three months from four off-site wells, (K-10, K-11, K-13 and ~~K-25~~) and two on-site wells (K-1h and K-1g). All samples are analyzed for gross beta in the total residue, K-40 and by Gamma Isotopic Analysis. Samples from one on-site well are analyzed for Sr-89, Sr-90, and for tritium. Samples from K-1h and K-1g are also analyzed for gross alpha. Reporting units are pCi/l.

Precipitation

A monthly cumulative sample of precipitation is taken at Location K-11. This sample is analyzed for tritium. Reporting units are pCi/l.

Milk

Milk samples are collected from two herds that graze within three miles of the reactor site (K-25 and K-34); from four herds that graze between 3-7 miles of the reactor site (K-3, K-5, ~~K-37~~ and ~~K-38~~); and one from a dairy in Green Bay (K-28), 26 miles from the reactor site.

The samples are collected twice per month during the grazing period (May through October) and monthly for the rest of the year. To prevent spoilage the samples are treated with preservative. All samples are analyzed by Gamma Isotopic Analysis and for iodine -131 immediately after they are received at the laboratory. To achieve required minimum sensitivity of 0.5 pCi/l, iodine is separated on an ion exchange column, precipitated as palladium iodide and beta counted. Monthly samples and monthly composites of semimonthly samples are then analyzed for Sr-89 and Sr-90. Potassium and calcium are determined and the $^{137}\text{Cs/gK}$ and $^{90}\text{Sr/gCa}$ ratios are calculated. Reporting units are pCi/l except for stable potassium and calcium, which are reported in g/l.

If milk samples are not available, green leafy vegetables will be collected on a monthly basis (when available) from Locations K-10, K-11, and K-26.

Grass

Grass is collected three times per year (2nd, 3rd, and 4th quarters) from the five dairy farms (K-3, K-5, K-25, K-34 and ~~K-37~~) and from two on-site locations (K-1b and K-1f). The samples are analyzed for gross beta activity, for Sr-89 and Sr-90, and Gamma Isotopic Analysis to identify and quantify gamma-emitting radionuclides. Reporting units are pCi/g wet weight.

Cattlefeed

Once per year, during the first quarter when grass is not available, cattlefeed (such as hay or silage) is collected from the five dairy farms. The analyses performed are the same as for grass. Reporting units are pCi/g wet weight.

Vegetables and Grain

Annually, during the 3rd quarter, samples of five varieties of vegetables grown and marketed for human consumption are collected from K-17 and/or K-26, depending upon the availability of samples. In addition, two varieties of grain, if available, are collected annually from the farmland owned by WPS (K-23) and rented to a private individual for growing crops. The analyses performed are the same as for grass. Reporting units are pCi/g wet weight.

Eggs

Quarterly samples of eggs can be taken from K-24, K-27, and K-32. At least one control and one indicator should be collected. The samples are analyzed for gross beta activity, for Sr-89 and Sr-90, and Gamma Isotopic Analysis to identify and quantify gamma-emitting radionuclides. Reporting units are pCi/g wet weight.

Soil

Twice during the growing season samples of the top two inches of soil are collected from the five dairy farms and from an on-site location (K-1f). The soil is analyzed for gross alpha and gross beta

activities, for Sr-89 and Sr-90, and Gamma Isotopic Analysis to identify and quantify gamma-emitting manmade radionuclides. Reporting units are pCi/g dry weight.

Surface Water

Surface water is sampled monthly from Lake Michigan at the KNPP discharge (K-1d), and at Two Creeks Park, 2.5 miles south of the reactor site (K-14). Samples are collected monthly at the Green Bay Municipal Pumping station between Kewaunee and Green Bay (K-9). Raw and treated water is collected. Monthly samples are also taken, when available, from each of the three creeks (K-1a, K-1b, K-1e) that pass through the reactor site and from the drainage pond (K-1k) south of the plant. The samples are taken at a point near the mouth of each creek and at the shore of the drainage pond. The water is analyzed for gross beta activity in: (a) the total residue, (b) the dissolved solids, and (c) the suspended solids.

The samples are also analyzed for K-40 and by Gamma Isotopic Analysis. Quarterly composites from all locations are analyzed for tritium, Sr-89 and Sr-90. Reporting units are pCi/l.

Bottom Sediments

Five samples of Lake Michigan bottom sediments, one at the discharge (K-1d), one from 500 feet north of the discharge (K-1c), one from 500 feet south of the discharge (K-1j), and one at the Two Creeks Park (K-14), one at the Green Bay Municipal Pumping Station (K-9) are collected semi-annually (May and November). The samples are collected at the beach in about 2-3 feet of water. All samples are analyzed for gross beta activity, for Sr-89 and Sr-90 and by Gamma isotopic Analysis. Since it is known that the specific activity of the sediments (i.e., the amount of radioactivity per unit mass of sediment) increases with decreasing particle size, the sampling procedure will assure collection of very fine particles. Reporting units are pCi/g dry weight.

Table 2.2.1-A Radiological Environmental Monitoring Program

Exposure Pathway And/Or Sample	Minimum Required Samples *	Available Sample Locations ^b	Sampling, Collection and Analysis Frequency	Type of Analysis
1. Direct Radiation ^c	5 Inner Ring locations	K-5, K-25, K-27, K-7, K-1F, K-30	See Table 2.2.1-B	Gamma dose
	6 Outer Ring locations	K-2, K-3, K-15, K-17, K-8, K-31, K-37		
	1 Control location	K-16		
	1 Population center	K-7		
	1 Special interest location	K-8		
	1 Nearby resident	K-27		
2. Airborne Radioiodine and Particulates	3 samples close to the site boundary in highest average X/Q	K-1f, K-2, K-7, K-8, K-31	See Table 2.2.1.B Continuous sampler operation Iodine: charcoal	Iodine (I-131) by Gamma Isotopic ^f
	1 sample from the closest community having the highest X/Q	K-7	Particulates See Table 2.2.1-B	Particulates: gross beta analysis ^f Gamma isotopic of composite (by location) ^f
	1 sample from a control location	K-16 ^d	See Table 2.2.1-B	
3. Waterborne a. Surface ^a	1 Upstream sample	K-1a, K-9, K-1d	Grab sample See Table 2.2.1-B	Gross Beta, Sr 89/90 Gamma isotopic ^f Composite of grab samples for tritium.
	1 Downstream sample	K-1e, K-14, K-1k, K-1b		
	b. Ground	1-2 location likely to be affected ^a	Grab sample See Table 2.2.1-B	Gamma isotopic ^f , tritium analysis Gross Beta, Sr 89/90
	c. Drinking	1-3 samples of nearest water supply	K-10, K-11, K-12, K-13	Grab sample See Table 2.2.1-B
d. Sediment from shoreline	1 sample from downstream area with potential for recreational value	K-14, K-1c, K-1d, K-1j, K-9	Grab sample See Table 2.2.1-B	Gamma isotopic ^f analysis Gross Beta, Sr 89/90
4. Ingestion a. Milk	Samples from milking animals in 3 locations within 5 km having the highest dose potential.	K-5, K-25, K-34	See Table 2.2.1-B	I-131 Gamma Isotopic ^f SR 89/90
	1 alternate location	K-37, K-38		
	1 control location	K-3, K-28		
b. Fish	3 random samplings of commercially and recreationally important species in the vicinity of the discharge.	K-1d	See Table 2.2.1-B	Gamma isotopic ^f and edible portions Gross Beta Sr 89/90 on bones

Table 2.2.1-A Radiological Environmental Monitoring Program

Exposure Pathway And/Or Sample	Minimum Required Samples ^a	Available Sample Locations ^b	Sampling, Collection and Analysis Frequency	Type of Analysis
c. Food Products	Samples of leaf vegetables grown nearest each of two different offsite locations within 5 miles of the plant if milk sampling is not performed.	2 samples nearest highest predicted annual average ground level D/Q. K-10, K-11 1 sample 15-30 km distant if milk sampling is not performed. K-26	See Table 2.2.1-B	Gamma isotopic ^f and I-131 Analysis.
5. Miscellaneous samples not identified in NUREG-0472				
a. Aquatic Slime	None required	K-1k K-1a, K-1b, K-1e K-14, K-1d K-9 (control)	See Table 2.2.1-B	Gross Beta activity and if available Sr-89, Sr-90 and Gamma Isotopic ^f
b. Soil	None required	K-1f, K-5, K-25, K-31 K-34 K-3, (control)	See Table 2.2.1-B	Gross Alpha/Beta Sr-89 and Sr-90 Gamma Isotopic ^f
c. Cattlefeed	None required	K-5, K-25, K-37 K-34 K-3,(control)	See Table 2.2.1-B	Gross Beta Sr-89 and Sr-90 Gamma Isotopic ^f
d. Grass	None required	K-1b, K-1f, K-25, K-31 K-5, K-12, K-34 K-3,(control)	See Table 2.2.1-B	Gross Beta Sr-89 and Sr-90 Gamma Isotopic ^f
e. Domestic Meat	None required	K-20, K-24, K-27, K-29 K-32 (control)	See Table 2.2.1-B	Gross Alpha/Beta Gamma Isotopic ^f
f. Eggs	None required	K-27,K-33 K-32 K-24	See Table 2.2.1-B	Gross Beta Sr-89/90 Gamma Isotopic ^f
g. Precipitation	None required	K-11	See Table 2.2.1-B	Tritium
h. Vegetables/ Grain	None required	K-17, K-23 K-26 (control)	See Table 2.2.1-B	Gross Beta Sr-89/90 Gamma Isotopic ^f

Table 2.2.1-A Radiological Environmental Monitoring Program

Exposure Pathway And/Or Sample	Minimum Required Samples *	Available Sample Locations ^b	Sampling, Collection and Analysis Frequency	Type of Analysis
Table Notations				
a	The samples listed in this column describe the minimum sampling required to meet REMP requirements.			
b	Additional details of sample locations are provided in Table 2.2.1-C and Figure 1. The REMP requires that samples to be taken from each of the "available sample locations" listed (see section 3.1). Deviations from the required sampling schedule will occur if specimens are unobtainable due to hazardous conditions, seasonal unavailability, malfunction of automatic sampling equipment and other legitimate reasons. If specimens are unobtainable due to sampling equipment malfunction, reasonable efforts shall be made to complete corrective actions prior to the end of the next sampling period. All deviations from the sampling schedule shall be documented, as required by REMM 2.4.1.c, in the Annual Radiological Environmental Monitoring Report. It is recognized that, at times, it may not be possible or practicable to continue to obtain samples of the media of choice at the most desired location or time. In these instances suitable alternative media and locations may be chosen for the particular pathway in question and appropriate substitutions made within 30 days in the REMM. The cause of the unavailability of samples for that pathway and the new location(s) for obtaining replacement samples will be identified in the Annual Radiological Environmental Monitoring Report.			
c	For the purposes of this table, each location will have 2 packets of thermoluminescent dosimeters (TLDs). The TLDs are CaSO ₄ : Dy cards with 2 cards/packet and 4 dosimeters/card (four sensitive areas each for a total of eight dosimeters/packet). The NRC guidance of 40 stations is not an absolute number. The number of direct radiation monitoring stations has been reduced according to geographical limitations; e.g., Lake Michigan. The frequency of analysis or readout for TLD systems depends upon the characteristics of the specific system used and selection is made to obtain optimum dose information with minimal fading.			
d	The purpose of this sample is to obtain background information. If it is not practical to establish control locations in accordance with the distance and wind direction criteria, other sites that provide valid background data may be substituted.			
e	Airborne particulate sample filters shall be analyzed for gross beta radioactivity 24 hours or more after sampling to allow for radon and thoron daughter decay. If gross beta activity in air particulate samples is greater than ten times the yearly mean of control samples, gamma isotopic analysis shall be performed on the individual samples.			
f	Gamma isotopic analysis means the identification and quantification of gamma-emitting radionuclides that may be attributable to the effluents from the facility.			
g	The "upstream sample" shall be taken at a distance beyond significant influence of the discharge. The "downstream" sample shall be taken in an area near the mixing zone.			
h	Ground water samples shall be taken when this source is tapped for drinking or irrigation purposes in areas where the hydraulic gradient or recharge properties are suitable for contamination.			

Table 2.2.1-B Type and Frequency of Collection

Location	Weekly	Biweekly	Monthly	Quarterly			Semi-Annually		Annually
K-1a			SW				SL		
K-1b			SW	GR*			SL		
K-1c							BS ^b		
K-1d			SW	FT*			BS ^b	SL	
K-1e			SW					SL	
K-1f	AP	AI		GR*	TLD		SO		
K-1g				WW					
K-1h				WW					
K-1j							BS ^b		
K-1k			SW					SL	
K-2	AP	AI			TLD				
K-3			MF*	GR*	TLD	CF ^d	SO		
K-4									
K-5			MF*	GR*	TLD	CF ^d	SO		
K-6									
K-7	AP	AI			TLD				
K-8	AP	AI			TLD				
K-9			SW				BS ^b	SL	
K-10			GLV ^f	WW					
K-11			PR, GLV ^f	WW					
K-12									
K-13				WW					
K-14			SW				BS ^b	SL	
K-15					TLD				
K-16	AP	AI			TLD				
K-17					TLD			VE	
K-19									
K-20								DM	
K-23								GRN	
K-24				EG				DM	
K-25*			MF*	GR*	TLD	CF ^d	SO		
K-26			GLV ^f					VE	

Table 2.2.1-B Type and Frequency of Collection

Location	Weekly	Biweekly	Monthly	Quarterly	Semi-Annually	Annually
K-27			EG	TLD		DM
K-28			MI ^f			
K-29						DM
K-30				TLD		
K-31	AP	AI		TLD		
K-32				EG		DM
K-33						
K-34			MI ^f	GR ^a CF ^d	SO	
K-35						
K-36						
K-37			MI ^f	TLD GR ^a CF ^d	SO	
K-38			MI ^f	TLD GR ^a CF ^d	SO	

- a Three times a year, second (April, May, June), third (July, August, September), and fourth (October, November, December) quarters
- b To be collected in May and November
- c Monthly from November through April; semimonthly from May through October
- d First (January, February, March) quarter only
- e Replaced by K-29 in summer of 1990
- f Alternate if milk is not available

<u>Code</u>	<u>Description</u>	<u>Code</u>	<u>Description</u>	<u>Code</u>	<u>Description</u>
AI	Airborne Iodine	FI	Fish	SO	Soil
AP	Airborne Particulate	GR	Grass	SW	Surface Water
BS	Bottom Sediment	GRN	Grain	TLD	Thermoluminescent Dosimeter
CF	Cattlefeed	MI	Milk	VE	Vegetables
DM	Domestic Meat	PR	Precipitation	WW	Well Water
EG	Eggs	SL	Slime	GLV	Green Leafy Vegetables

Table 2.2.1-C Sampling Locations, Kewaunee Nuclear Power Plant

Code	Type ^a	Distance (Miles) ^b and Sector	Location
K-1			Onsite
K-1a	I	0.62 N	North Creek
K-1b	I	0.12 N	Middle Creek
K-1c	I	0.10 N	500' North of Condenser Discharge
K-1d	I	0.10 E	Condenser Discharge
K-1e	I	0.12 S	South Creek
K-1f	I	0.12 S	Meteorological Tower
K-1g	I	0.06 W	South Well
K-1h	I	0.12 NW	North Well
K-1j	I	0.10 S	500' south of Condenser Discharge
K-1k	I	0.60 SW	Drainage Pond, south of plant
K-2	C	9.5 NNE	WPS Operations Building in Kewaunee
K-3	C	6.0 N	Lyle and John Siegmund Farm, N2815 Hy 42, Kewaunee
K-4(h)	I	3.0 N	Tom Stangel Farm, E4804 Old Settlers Rd, Kewaunee
K-5	I	3.5 NNW	Ed Papham Farm, E4160 Old Settlers Rd, Kewaunee
K-6(e)	C	6.7 WSW	Novitsky Farm, E1870 Cty Tk BB, Denmark
K-7	I	2.75 SSW	Ron Zimmerman Farm, 17620 Nero Rd, Two Rivers
K-8	C	5.0 WSW	Saint Mary's Church, 18424 Tisch Mills Rd, Tisch Mills
K-9	C	11.5 NNE	Green Bay Municipal Pumping Station, six miles east of Green Bay (sample source is Lake Michigan from Rostok Intake 2 miles north of Kewaunee)
K-10	I	1.5 NNE	Turner Farm, Kewaunee Site
K-11	I	1.0 NW	Harlan Ihlenfeld Farm, N879 Hy 42, Kewaunee
K-12(i)	I	1.5 WSW	LeCaptain Farm, N491 Woodside Rd, Kewaunee
K-13	C	3.0 SSW	Rand's General Store, Two Creeks
K-14	I	2.5 S	Two Creeks Park, 2.5 miles south of site
K-15	C	9.25 NW	Gas Substation, 1.5 miles north of Stangelville
K-16	C	26 NW	WPS Division Office Building, Green Bay, Wisconsin

Table 2.2.1-C Sampling Locations, Kewaunee Nuclear Power Plant

Code	Type ^a	Distance (Miles) ^b and Sector	Location
K-17	I	4.25 W	Jansky's Farm, N885 Cty Tk B, Kewaunee
K-19(f)	I	1.75 NNE	Wayne Paral Farm, N1048 Lakeview Dr., Kewaunee
K-20	I	2.5 N	Carl Struck Farm, N1596 Lakeshore Dr., Kewaunee
K-23	I	0.5 W	0.5 miles west of plant, Kewaunee site
K-24	I	5.45 N	Fectum Farm, N2653 Hy 42, Kewaunee
K-25	I	2.75 S \bar{W}	Wotachek Farm, E3968 Cty Tk BB, Two Rivers
K-26(d)	C	10.7 SSW	Bertler's Fruit Stand (8.0 miles south of "BB")
K-27	I	1.5 NW	Schlies Farm, E4298 Sandy Bay Rd
K-28	C	26 NW	Hansen Dairy, 1742 University Ave., Green Bay, Wisconsin
K-29	I	5.75 W	Kunesh Farm, E3873 Cty Tk G, Kewaunee
K-30	I	1.00 N	End of site boundary
K-31	I	6.25 NNW	E. Krok Substation, Krok Road
K-32	C	11.50 N	Piggly Wiggly, 931 Marquette Dr., Kewaunee
K-33(g)	I	4.25 W	Gary and Lynn Holly Farm, E2885 Holly Lane, Tisch Mills
K-34	I	2.5 N \bar{N}	Leon and Vicky Struck Farm, N1549 Lakeshore Drive, Kewaunee
K-35(j)	C	6.75 WNW	Jean Ducat Farm, N1215 Sleepy Hollow, Kewaunee
K-36(j)	I		Fiala's Fish Market, 216 Milwaukee, Kewaunee
K-37	I	4.00 N	Gary and Ann Hardtke Farm, E4282 Old Settlers Road, Kewaunee
K-38	I	3.8 WNW	Dave Sinkula Farm, N890 Town Hall Road, Kewaunee

- a I = indicator; C = control.
- b Distances are measured from reactor stack.
- c Deleted
- d Location K-18 was changed because Schmidt's Food Stand went out of business. It was replaced by Bertler's Fruit Stand (K-26).
- e Replaced by K-33 in summer of 2000. Retired from farming.
- f Replaced by K-34 in summer of 2000. Retired from farming.
- g. Replaced by K-35 in fall of 2000.
- h. Sold farm in summer of 2000, replaced by K-25
- i. Retired from farming in summer of 2000
- ~~Removed from the program in Fall of 2001~~

Table 2.2.1-D Reporting Levels for Radioactivity Concentrations in Environmental Samples

Medium	Radionuclide	Reporting Levels	
		<u>EIML to KNPP^a</u>	<u>KNPP to NRC^b</u>
Airborne Particulate or Gases (pCi/m ³)	Gross Beta	1	--
	I-131 (Charcoal)	0.1	0.9
	Cs-134	1	10
	Cs-137	1	20
Precipitation (pCi/l)	H-3	1,000	--
Water (pCi/l)	Gross Alpha	10	--
	Gross Beta	30	--
	H-3	10,000	20,000 ^c
	Mn-54	100	1,000
	Fe-59	40	400
	Co-58	100	1,000
	Co-60	30	300
	Zr-Nb-95	40	400
	Cs-134	10	30
	Cs-137	20	50
	Ba-La-140	100	200
	Sr-89	10	--
	Sr-90	10	--
	Zn-65	30	300
	Milk (pCi/l)	I-131	1.0
Cs-134		20	60
Cs-137		20	70
Ba-La-140		100	300
Sr-89		10	--
Grass, Cattle Feed, and Vegetables (pCi/g wet)	Gross Beta	30	--
	I-131	0.1	0.1
	Cs-134	0.2	1
	Cs-137	0.2	2
	Sr-89	1	--
	Sr-90	1	--
Eggs (pCi/g wet)	Gross Beta	30	--
	Cs-134	0.2	1
	Cs-137	0.2	2
	Sr-89	1	--
	Sr-90	1	--

Table 2.2.1-D Reporting Levels for Radioactivity Concentrations in Environmental Samples

Medium	Radionuclide	Reporting Levels	
		EIML to KNPP	KNPP to NRC ^b
Soil, Bottom Sediments (pCi/g)	Gross Beta	50	--
	Cs-134	5	--
	Cs-137	5	--
	Sr-89	5	--
	Sr-90	5	--
Meat (pCi/g wet)	Gross Beta (Flesh, Bones)	10	--
	Cs-134 (Flesh)	1.0	1.0
	Cs-137 (Flesh)	2	2.0
	Sr-89 (Bones)	2	--
	Sr-90 (Bones)	2	--
Fish (pCi/g wet)	Gross Beta (Flesh, Bones)	10	--
	Mn-54	--	30.0
	Fe-59	--	10.0
	Co-58	--	30.0
	Co-60	--	10.0
	Cs-134 (Flesh)	1	1.0
	Cs-137 (Flesh)	2	2.0
	Sr-89 (Bones)	2	--
	Sr-90 (Bones)	2	--
	Zn-65 (Bones)	--	20

- a) Radionuclides will be monitored by ~~EIML~~ and concentrations above the listed limits will be reported to ~~KNPP~~.
- b) Concentrations above the listed limits will be reported to NRC as required by REMM 2.4.1.
- c) For drinking water samples, this is 40 CFR Part 141 value. If no drinking water pathway exists, a value of 30,000 pCi/l may be used.

Table 2.3.1-A
Detection Capabilities for Environmental Sample Analysis^a
Lower Limit of Detection (LLD)^{b,c}

Analysis	Water (pCi/l)	Airborne Particulate or Gases (pCi/m ³)	Fish (pCi/kg, wet)	Milk (pCi/l)	Food Products (pCi/kg, wet)	Sediment (pCi/kg, dry)
Gross Beta	4	0.01				
H-3	2000 ^d					
Mn-54	15		130			
Fe-59	30		260			
Co-58, 60	15		130			
Zr-Nb-95	15					
I-131	1 ^e	0.07		1	60	
Cs-134	15	0.05	130	15	60	150
Cs-137	18	0.06	150	18	80	180
Ba-La-140	15			15		
Zn-65	30		260			

Table Notations for Table 2.3.1-A

- a. This list does not mean that only these nuclides are to be considered. Other peaks that are identifiable, together with those of the above nuclides, shall also be analyzed and reported in the Annual Radiological Environment Monitoring Report.
- b. Required detection capabilities for thermoluminescent dosimeters used for environmental measurements are given in Regulatory Guide 4.13.
- c. The LLD is defined, for purposes of these specifications, as the smallest concentration of radioactive material in a sample that will yield a net count, above system background, that will be detected with 95% probability with only 5% probability of falsely concluding that a blank observation represents a "real" signal.

For a particular measurement system, which may include radiochemical separation:

$$LLD = \frac{4.66s_b}{E \times V \times 2.22 \times Y \times \exp(-\gamma\Delta t)}$$

Where:

LLD is the a priori lower limit of detection as defined above, as picocuries per unit mass or volume,

S_b is the standard deviation of the background counting rate or of the counting rate of blank sample as appropriate, as counts per minute,

E is the counting efficiency, as counts per disintegration,

V is the sample size in units of mass or volume,

2.22 is the number of disintegrations per minute per picocurie,

Y is the fractional radiochemical yield, when applicable,

γ is the radioactive decay constant for the particular radionuclide, and

Δt for environmental samples is the elapsed time between sample collection, or end of the sample collection period, and time of counting,

Typical values of E, V, Y, and Δt should be used in calculation.

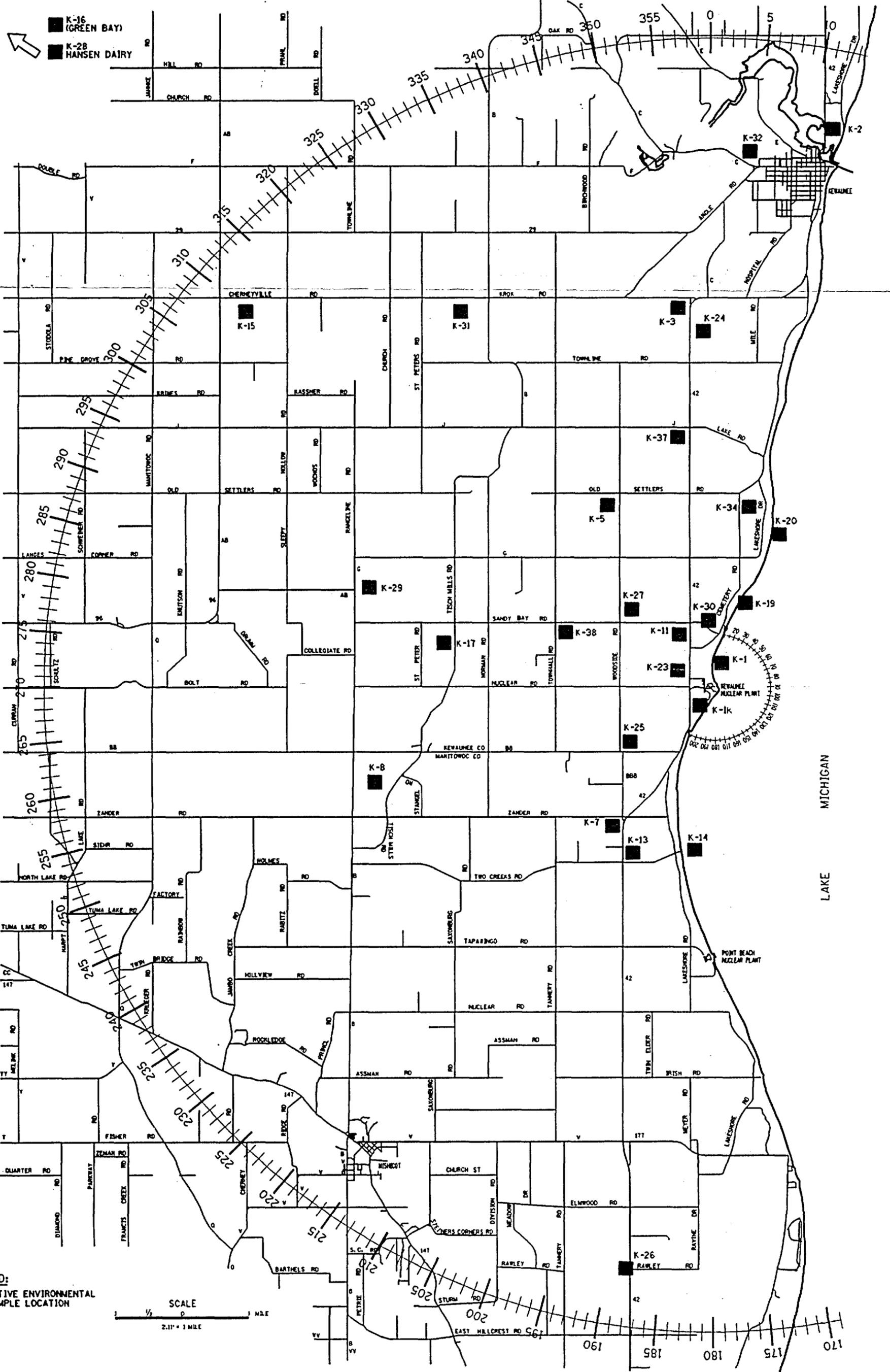
Table Notations for Table 2.3.1-A (con't)

It should be recognized that the LLD is defined as a priori (before the fact) limit representing the capability of a measurement system and not as an posteriori (after the fact) limit for a particular measurement. Analyses shall be performed in such a manner that the stated LLDs will be achieved under routine conditions. Occasionally background fluctuations, unavoidable small sample sizes, the presence of interfering nuclides, or other uncontrollable circumstances may render these LLDs unachievable. In such cases, the contributing factors shall be identified and described in the Annual Radiological Environmental Monitoring Report.

- d. If no drinking water pathway exists, a value of 3,000 pCi/l may be used.
- e. LLD for drinking water samples. If no drinking water pathway exists, the LLD of gamma isotopic analysis may be used.

K-9 INTAKE

K-16 (GREEN BAY)
K-28 HANSEN DAIRY

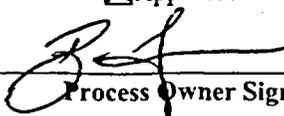
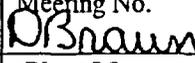


LEGEND:
ACTIVE ENVIRONMENTAL
SAMPLE LOCATION

SCALE
2.11 = 1 MILE

LAKE MICHIGAN

TRACKING AND PROCESSING RECORD

A	Initiated By: <u>Brad Gauger</u> Date: <u>02/13/02</u> Dept: <u>RP</u> Ext.: <u>8242</u>	
	Current	
	Document No.: <u>REMM</u>	Rev. No.: <u>6</u> New Rev. No.: <u>7</u>
	Title: <u>Radiological Environmental Monitoring Manual</u>	
B	Describe Change <small>(New Document or Revision- attach commitments, KAPs, markup, etc.)</small>	Describe Reason Requested Due /Required Date →
	Changed WPSC to KNPP in various areas throughout the document	Generic term referring to Kewaunee since it's split between the NMC and WPSC
	Changed from Teledyne Isotopes Midwest Lab (TIML) to Environmental Inc, Midwest Lab (EIML) throughout the document.	Vendor changed their name
	Section 3.2, pg 3-1, Noted that the HASL manual is available on-line	Referenced the new technology
	Fish, Page 3-3, Table 2.2.1-A, and Table 2.2.1-B removed K-36 from the program	Per KAP 01-009973, the location is not a representative sample
	Page 1 of 2	Continued on Next Page <input checked="" type="checkbox"/>
C	Expiration Requirement (if applicable)	Expiration Date <small>(IPTE, Vendor, Other) →</small>
D	Activity: <input type="checkbox"/> New <input checked="" type="checkbox"/> Revision <input type="checkbox"/> Admin Hold <input type="checkbox"/> Vendor <input type="checkbox"/> Deletion Priority: <input checked="" type="checkbox"/> Immediate Action <input type="checkbox"/> Non-Urgent - Perform Later <input type="checkbox"/> Rejected - See Comments Doc Type: <input type="checkbox"/> Nuclear Safety Related <input type="checkbox"/> PORC Review <input type="checkbox"/> SRO Approval - Temp Changes Level of Use: <input type="checkbox"/> Continuous Use <input type="checkbox"/> Reference Use <input checked="" type="checkbox"/> Information Use Comments:	
E	<input checked="" type="checkbox"/> Technical <input type="checkbox"/> Cross Discipline <input type="checkbox"/> Editorial <input type="checkbox"/> Minor <input checked="" type="checkbox"/> 50.59 <input type="checkbox"/> Validation <input type="checkbox"/> Oversight (QC) <input type="checkbox"/> Other	
F	Safety Review Attached? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No Safety Evaluation Report Attached? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	
G	1) <input checked="" type="checkbox"/> ⇒ Process Owner Review Recommendation <input checked="" type="checkbox"/> Approval <input type="checkbox"/> Disapproval  Process Owner Signature Date	2) <input checked="" type="checkbox"/> ⇒ PORC Review Recommendation <input checked="" type="checkbox"/> Approval #02-045 <input type="checkbox"/> Disapproval Meeting No. <u>02-045</u>  Plant Manager Signature Date <u>5-28-02</u>
H	Effective Date:	

TRACKING AND PROCESSING RECORD

CONTINUATION SHEET

Document No.: REMM

Current Rev. No.: 6

New Rev. No.: 7

Page 2 of 2

Describe Change (New Document or Revision)	Describe Reason
Ambient Radiation(pg 3-4), Milk(pg 3-5),Grass(pg 3-5), Table 2.2.1-A replaced K-35 with K-37	K-35 no longer wanted to participate in the sampling program and was replaced by K-37 (KAP 01-014973)
Well Water, pg 3-4, replaced K-12 with K-25	K-12 has retired from farming and the sampling program
Table 2.2.1-D, delete I-131 from Water reporting levels	Per KAP 01-8281, and the fact that the analysis is not done or required
Milk(pg 3-5), Table 2.2.1-A, Table 2.2.1-B added K-38 to milk sampling location	K-12 retired from farming and was replaced by K-38
Table 2.2.1-A, change the K-35/37 designation from control to indicator for Soil, Cattlefeed, and Grass	K-35/37 is an indicator location
Table 2.2.1-B, added location K-37 and associated samples	K-35 no longer wanted to participate in the sampling program and was replaced by K-37 (KAP 01-014973)
Table 2.2.1-B, removed the associated sample designations from K-35	K-35 no longer wanted to participate in the sampling program and was replaced by K-37 (KAP 01-014973)
Table 2.2.1-C and attached map, added location K-37 and K-38	New sample locations replacing K-35 and K-12 respectively.

DOCUMENT REVIEW DATA SHEET

Document Number: REMM Revision Number: 7 Required Return Date: _____

Title: Radiological Environmental Monitoring Manual

Author/RI: B. Gauger Reviewer: THOMAS P. SCHMIDL

Technical Review Writer's Guide Review Other _____ Other _____
 Cross Discipline Review Oversight (QC) Review Other _____ Other _____

IF the review is for a Minor Revision, **THEN** ensure the following criteria are met.

1. Will not increase the safety risk to personnel YES NO
2. Will not alter a source document requirement YES NO
3. Will not alter the purpose or scope of the procedure YES NO
4. Will not eliminate any required reviews or approvals YES NO
5. Will not alter the operating, technical, design, process, regulatory, or quality control requirements of a procedure. YES NO

> **IF** any of the above questions are answered NO, **THEN** record detailed comment and suggested resolution (either marked-up in the document review copy or in the space below), attach to the review document, and return to Writer for resolution.

> **IF** the above questions are answered YES, **THEN** sign below and return the review package to the Writer.

Technical Review:
Refer to Attachment B, "Technical Attributes," and answer the following questions.

1. Is the technical content adequate? YES NO
2. Is procedure adequate to cover task? YES NO
3. Does procedure satisfy current flow & logic diagrams? YES NO NA
4. Is the Infrequently Performed Test and Evolutions determination per NAD-03.01 still correct? YES NO NA
5. Are the references and/or cross references correct? YES NO NA

Editorial Review:

6. Does the procedure conform to the applicable sections of the Procedure Writer's Guide or the IPEOP Writer's Guide? YES NO

IF any of the above questions are answered NO, **THEN** record detailed comment and suggested resolution (either marked-up in the review copy of the document or in the space below), attach to the review document, and return to Writer for resolution.

7. Are there process improvements that may be appropriate to implement at this time? YES NO

IF the above question is answered YES, **THEN** return the review copy of the document to Writer for resolution.

Step or Section No.	Comments and Suggested Resolution	Response

IF additional pages are needed, **THEN** note the procedure number on the page(s) and attach to this Form.

When the review is complete, sign and date below. Return the Review Package to the Writer.

Thomas P. Schmidl 3-18-02
 Reviewer Signature Date

50.59 APPLICABILITY REVIEW

1. Document/Activity number: REMM Rev. 7
2. Brief description of proposed activity (what is being changed and why):
Revisions to the Radiological Environmental Monitoring Manual.
See Form GNP-03.01.01-1 for details
3. Does the proposed activity involve or change any of the following documents or processes? Check YES or NO for each applicability review item. [Ref. NMC 50.59 Resource Manual, Section 4]

NOTE: If you are unsure if a document or process may be affected, contact the process owner.

	Yes ✓	No ✓	Document or Process	Applicable Regulation	Contact/Action
a	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Technical Specifications or Operating License	10CFR50.92	Process change per NAD-05.14. Contact Licensing.
b	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Activity/change previously approved by NRC in license amendment or NRC SER	10CFR50.90	Identify NRC letter in comments below. Process change. Contact Licensing for assistance.
c	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Activity/change covered by an existing 10CFR50.59 screening or evaluation.	10CFR50 Appendix B	Identify screening or evaluation in comments below. Process change.
d	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Quality Assurance Program (OQAP/OQAP)	10CFR50.54(a)	Contact QA. Refer to NAD-01.07.
e	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Emergency Plan	10CFR50.54(q)	Contact EP. Refer to NAD-05.15.
f	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Security Plan	10CFR50.54(p)	Contact Security. Refer to NAD-05.17.
g	<input type="checkbox"/>	<input checked="" type="checkbox"/>	IST Plan	10CFR50.55a(f)	Contact IST process owner. Refer to NAD-01.24.
h	<input type="checkbox"/>	<input checked="" type="checkbox"/>	ISI Plan	10CFR50.55a(g)	Contact ISI process owner. Refer to NADs 01.03, 01.05, and 05.11.
i	<input type="checkbox"/>	<input checked="" type="checkbox"/>	ECCS Acceptance Criteria	10CFR50.46	Contact Licensing.
j	<input type="checkbox"/>	<input checked="" type="checkbox"/>	USAR - Check YES only if change is editorial (see Attachment A).	10CFR50.71	Process USAR change per NEP-05.02. Contact USAR process owner for assistance.
k	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Commitment - Check NO if associated with a response to Generic Letters and Bulletins, or if described in the USAR.	10CFR50 Appendix B	Contact Licensing. Refer to NAD-05.25.
l	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Maintenance activity/procedure - Check YES only if equipment will be restored to its as-designed condition within 90 days (see Attachment C).	10CFR50.65	Evaluate under Maintenance Rule. Refer to NAD-08.20 and NAD-08.21.
m	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Degraded/Non-conforming plant condition - Check YES if returned to as-designed condition in a timely manner consistent with safety.	10CFR50 Appendix B	Evaluate under GL 91-18, Revision 1. Contact licensing for assistance. Refer to GNP 11.08.03.
n	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Administrative or managerial directive/procedure (e.g., NAD and GNP) or other controlled document - See Attachment B.	10CFR50 Appendix B	Process procedure/document revision.

4. Conclusion. Check one of the following:
 - All documents/processes listed above are checked NO. 10CFR50.59 applies to the proposed activity. A 50.59 pre-screening shall be performed.
 - One or more of the documents/processes listed above are checked YES, AND controls all aspects of the proposed activity. 10CFR50.59 does NOT apply. Process the change under the applicable program/process/procedure.
 - One or more of the documents/processes listed above are checked YES, however, some portion of the proposed activity is not controlled by any of the above processes. 10CFR50.59 applies to that portion. A 50.59 pre-screening shall be performed.

5. Comments:

6. Print name followed by signature. Attach completed form to document/activity/change package.

Prepared by: BRGawger Date: 4/15/02
 Reviewed by: THOMAS P. SCHMIDL Thomas P. Schmidl Date: 4-19-02

50.59 PRE-SCREENING

1. Document/Activity number: REMM Rev. 7
2. Brief description of proposed activity (what is being changed and why):
Replacing milk sample locations, delete fish sample location, change well water location
3. Does the proposed activity involve or change any of the following documents or processes?
 Check YES or NO for each pre-screening item. [Ref. NMC 50.59 Resource Manual, Section 5.1]
 NOTE: If you are unsure if a document or process may be affected, contact the process owner.

NOTE: An asterisk (*) indicates that the document is incorporated by reference in the USAR or is implicitly considered part of the USAR.

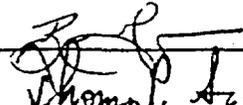
NOTE: Check NO if activity/change is considered editorial, administrative, or routine maintenance as defined in Attachments A, B, and C. Explain in Comments if necessary.

	Yes /	No /	Document/Process	Directive/Procedure
a	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Updated Safety Analysis Report (USAR)	NEP-05.02
b	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Technical Specifications Bases	NAD-05.14
c	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Commitments made in response to NRC Generic Letters and Bulletins, and those described in the USAR	NAD-05.25
d	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Environmental Qualification (EQ) Plan	NAD-01.08
e	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Regulatory Guide 1.97 (RG 1.97) Accident Monitoring Instrumentation Plan	NAD-05.22
f	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Fire Plan	NAD-01.02
g	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Appendix R Design Description	NAD-01.02
h	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Fire Protection Program Analysis (FPPA)	NAD-01.02
i	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Offsite Dose Calculation Manual (ODCM)	NAD-05.13
j	<input checked="" type="checkbox"/>	<input type="checkbox"/>	* Radiological Environmental Monitoring Manual (REMM)	NAD-05.13
k	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Station Blackout Design Description	
l	<input type="checkbox"/>	<input checked="" type="checkbox"/>	* Control Room Habitability Study	
m	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Plant Drawing Discrepancies	NAD-05.01
n	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Engineering Calculations/Evaluations - Check YES only if directly or indirectly involves SSC described in the USAR, or affects a methodology or safety analysis described in the USAR.	GNP-04.03.04
o	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Permanent Plant Physical Changes	NAD-04.03
p	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Temporary Plant Physical Changes	NAD-04.03
q	<input type="checkbox"/>	<input checked="" type="checkbox"/>	QA Typing Determinations - Check YES only if reduction in classification, or affects design function as described in USAR.	NAD-01.01
r	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Plant Setpoint Document - Check YES only if change affects plant monitoring, performance, or operation.	NAD-04.06
s	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Plant Procedures/Revisions - Check YES if change directly or indirectly involves operation, control, or configuration of SSCs described in USAR (see Attachment B).	NAD-03.01
t	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Engineering Specifications - Check YES only if a design function or design requirement may be affected.	NAD-05.03
u	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Operations Night Orders or Operator Work Arounds - Check YES only if SSCs are operated or configured differently than described in USAR.	NAD-12.08
v	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Temporary plant alterations (e.g., jumpers, scaffolding, shielding, barriers) - Check YES only if installed (or in effect) for maintenance for longer than 90 days at power conditions.	NAD-08.14, GMP-127 HP-04.002, FPP-08-09
w	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Temporary plant alterations - Check YES only if not associated with maintenance.	
x	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Corrective/Compensatory Actions - Check YES only if degraded/non-conforming plant condition accepted "as-is" or compensatory action taken.	GNP-11.08.03
y	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Non-identical replacements (like-for-like or alternate) - Check NO for part number changes.	NAD-06.02

4. Conclusion. Check one of the following:
- All of the documents or processes listed above are checked NO. A 50.59 screening is NOT required. Process change in accordance with the applicable program/process/procedure.
- One or more of the documents or processes listed above are checked YES. A 50.59 screening shall be performed.

5. Comments:

6. Print name followed by signature. Either the preparer or reviewer shall be 50.59 screening qualified. Attach completed form to document/activity/change package.

Prepared by: Brad Gauger ,  Date: 5/21/02

Reviewed by: THOMAS P. SCHMIDLI ,  Date: 5-22-02

10CFR50.59 SCREENING

Page 1 of 7

Document/Activity Number: REMM Rev.7 SCRN# 02-048

PART I: Describe the Proposed Activity and Search the KNPP USAR
(Refer to NMC 50.59 Resource Manual Section 5.3.1)

- I.1. Describe the proposed activity, and scope of the activity covered by this screening. Appropriate descriptive materials may be attached.

Editorial changes including changing WPSC to KNPP throughout the document due to the NMC operating license change, changed from Teledyne Isotopes Midwest Lab (TIML) to Environmental Inc, Midwest Lab (EIML) due to a corporate name change and changed a reference to the HASL manual which is available on-line. Removed K-36, Fiala's Fish Market, from the fish sampling program. Per CAP002064, it can not be guaranteed that the sample from this location is from the vicinity of the plant discharge area as described in NUREG 0472. This location was an alternate to K-1d, "Condensor Discharge". The REMM continues to meet minimum locations as described in NUREG 0472. This change will not adversely affect the REMP. Replaced sample location K-35, "Ducat Farm", with K-37, "Hardtke Farm". All samples obtained from K-35 were transferred to K-37. The Ducat's no longer wanted to participate in the sampling program for the KNPP (ref CAP001586). Added sample location K-38, "Sinkula Farm" to the program to return the number of sample locations to pre-1998 levels. For Well Water samples, K-12 was replaced with K-25. K-12 has retired from farming and no longer wished to participate in the program. Deleted I-131 from the water reporting levels in Table 2.2.1-D. Per CAP002203, and the fact that the analysis is not currently performed by the vendor.

- I.2. Search the Updated Safety Analysis Report (USAR) including those documents incorporated by reference. Describe the pertinent design function(s), performance requirements, and methods of evaluation of the affected SSCs, and where this information is described in the USAR. It is acceptable to attach and highlight applicable portions of the USAR.

The REMM was incorporated into the Technical Specifications (TS) by TS Amendment No. 64 which took effect on 1/1/86. They were subsequently removed from TS Section 7/8 and made stand alone documents on 12/9/93 by TS Amendment No. 104 in response to NRC Generic Letter 89-01. USAR Sections 1.8 (NRC General Design Criteria) and 2.8 (Environmental Radioactivity Program) were also reviewed. Page 1.8-8 of the USAR answers the NRC GDC-17 by saying that the environmental radiation monitoring system is described in Section 2. Page 2.8-1 discusses the preoperational environmental radiological monitoring program, discusses the removal of the ODCM from TS, and discusses that the REMM defines the program for environmental radioactivity sampling. No changes to the formal USAR are required. This program defines the sampling and analysis schedule which are developed to provide representative measurements of radiation and of radioactive materials in those exposure pathways and for those radionuclides that lead to the high potential radiation exposures of MEMBERS OF THE PUBLIC resulting from plant operation. This monitoring program implements Section IV.B.2 of Appendix I to 10 CFR Part 50 and thereby verifies that the measurable concentrations of radioactivity and levels of radiation are not higher than expected on the basis of the effluent measurements and the modeling of the environmental exposure pathways. Guidance for the development of this monitoring program is provided by the Radiological Assessment Branch Technical Position on Environmental Monitoring. This program has been developed in accordance with NUREG 0472.

- I.3. Does the activity involve a change to the Technical Specifications?
(Changes to the Technical Specifications require a License Amendment request.)

Yes No

10CFR50.59 SCREENING

Page 2 of 7

SCRN# 02-048

PART II: Determine if the Activity Involves a Design Function
(Refer to NMC 50.59 Resource Manual Section 5.3.2)

Compare the proposed activity to the relevant portions of the USAR and answer the following questions:

- | YES | NO | QUESTION |
|-------------------------------------|-------------------------------------|---|
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Does the proposed activity involve Safety Analyses or an SSC(s) credited in the Safety Analyses? |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Does the proposed activity involve SSCs that support SSC(s) credited in the Safety Analyses? |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Does the proposed activity involve SSCs whose failure could initiate a transient (e.g., reactor trip, loss of feedwater, etc) or accident? |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Does the proposed activity involve SSCs whose failure could impact SSC(s) credited in the Safety Analyses? |
| <input checked="" type="checkbox"/> | <input type="checkbox"/> | Does the proposed activity involve USAR-described SSCs or procedural controls that perform functions that are required by, or otherwise necessary to comply with, regulations, license conditions, orders, or Technical Specifications? |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Does the activity involve a method of evaluation described in the USAR? |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Is the activity a test or experiment? (i.e., a non-passive activity which gathers data) |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | Does the activity exceed or potentially affect a design basis limit for a fission product barrier (DBLFPB)?
If this question is answered YES, this activity requires a 10CFR50.59 Evaluation. |

If the answer to all of these questions is NO, answer PART III as Not Applicable, and proceed to PART IV. A 10CFR50.59 evaluation is not required.

If any of the above questions are checked YES, identify the specific design function, method of evaluation, or DBLFPB involved:

The REMM defines the sampling and analysis schedule which are developed to provide representative measurements of radiation and of radioactive materials in those exposure pathways and for those radionuclides that lead to the high potential radiation exposures of MEMBERS OF THE PUBLIC resulting from plant operation.

10CFR50.59 SCREENING

Page 3 of 7

SCRN# 02-048

PART III: Determine Whether the Activity Involves Adverse Effects
(Refer to NMC 50.59 Resource Manual Section 5.3.3)

If all the questions in Part II were answered NO, then Part III is:

Not Applicable

Answer the following questions to determine if the activity has an adverse effect on a design function. Any YES answer means that a 10CFR50.59 Evaluation is required, except where noted in Question III.3.

III.1. Changes to the Facility or Procedures

YES	NO	QUESTION
<input type="checkbox"/>	<input checked="" type="checkbox"/>	Does the activity adversely affect the design function(s) identified in Part II?
<input type="checkbox"/>	<input checked="" type="checkbox"/>	Does the activity adversely affect the method of performing or controlling the design function(s) identified in Part II?

If either answer is YES, a 10CFR50.59 Evaluation is required. If both answers are NO, describe the basis for the conclusion (attach additional discussion, as necessary):

See Attachment pages 6 and 7

III.2. Changes to a Method of Evaluation

If the activity does not involve a method of evaluation, these questions are:

Not Applicable

YES	NO	QUESTION
<input type="checkbox"/>	<input type="checkbox"/>	Does the activity use a revised or different method of evaluation for performing safety analyses than that described in the USAR?
<input type="checkbox"/>	<input type="checkbox"/>	Does the activity use a revised or different method of evaluation for evaluating SSCs credited in safety analyses than that described in the USAR?

If either answer is YES, a 10CFR50.59 Evaluation is required. If both answers are NO, describe the basis for the conclusion (attach additional discussion, as necessary):

10CFR50.59 SCREENING

Page 4 of 7

SCRN# 02-048

III.3. Tests or Experiments

If the activity is not a test or experiment, the questions in III.3.a and III.3.b are:

Not Applicable

a. Answer these two questions first:

YES NO QUESTION

Is the proposed test or experiment bounded by other tests or experiments that are described in the USAR?

Are the SSCs affected by the proposed test or experiment isolated from the facility?

If the answer to both questions is NO, continue to III.3.b. If the answer to either question is YES, then briefly describe the basis:

b. Answer these additional questions only for tests or experiments which do not meet the criteria given above. If the answer to either question in III.3.a is YES, then these three questions are:

Not Applicable

YES NO QUESTION

Does the activity use or control an SSC in a manner that is outside the reference bounds of the design bases as described in the USAR?

Does the activity use or control an SSC in a manner that is inconsistent with the analyses or descriptions in the USAR?

Does the activity place the facility in a condition not previously evaluated or that could affect the capability of an SSC to perform its intended functions?

If any answer in III.3.b is YES, a 10CFR50.59 Evaluation is required. If the answers are all NO, describe the basis for the conclusion (attach additional discussion, as necessary):

10CFR50.59 SCREENING

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PART IV: Conclusion
(Refer to NMC 50.59 Resource Manual Section 5.3.4)

Check all that apply:

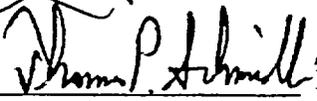
1. A 10CFR50.59 Evaluation is
 required,
OR
 NOT required

2. A change to the USAR and/or any document incorporated by reference is
 required (Process change in accordance with applicable plant program/process/procedure.),
OR
 NOT required

Additional comments:

Print name followed by signature. The preparer and reviewer shall be 50.59 screening or evaluation qualified. The completed screening is part of the document/activity/change package. Provide a copy of 50.59 screening to the 50.59 Process Owner/Program Coordinator.

Prepared By: Brad Gauger  Date: 5/21/02

Reviewed By: THOMAS P. SCHMIDL  Date: 5-22-02

May 21,2002

Subject: Attachment to REMM Rev-7 CFR50.59 Screening Review

Reference: NMC 50.59 Resource Manual

Removed K-36, Fiala's Fish Market, from the fish sampling program. Per CAP002064, it can not be guaranteed that the sample from this location is from the vicinity of the plant discharge area as described in NUREG 0472. This location was an alternate to K-1d, "Condensor Discharge". The REMM continues to meet minimum locations as described in NUREG 0472. This change will not adversely affect the REMP.

Replaced sample location K-35, "Ducat Farm", with K-37, "Hardtke Farm". All samples obtained from K-35 were transferred to K-37. The Ducat's no longer wanted to participate in the sampling program for the KNPP (ref CAP001586). Added sample location K-38, "Sinkula Farm" to the program to return the number of sample locations to pre-1998 levels. Per NUREG 0472, the minimum locations should include 3 locations within 5 km having the highest dose potential. If there are none, then, 1 sample in each of 3 areas between 5 and 8 km distance where doses are calculated to be greater than 1 mrem per year. The REMM currently has identified locations at K-5 (3.5mi NNW), K-25 (2.75mi WSW), and K-34 (2.5mi N). This change added locations K-37 (4.0mi N) and K-38 (3.8mi WNW). Those locations and dose potentials (XOQ) based on the NRCs XOQ/DOQ Computer Program are shown in the Tables below for two meteorological data sets. The sectors are sorted by the highest XOQ values. The bolded sectors are those sectors not over water.

Jan-Dec 1999 Met Data Chi/Q				K=KNPP E=PBNP
1999 mi		1999 2-3mi Seg		
Nne	4.62E-07	Nne	6.64E-07	K-34
N	4.08E-07	N	5.84E-07	K-37
E	3.93E-07	E	5.62E-07	
Ne	3.60E-07	Ne	5.13E-07	
Ese	3.50E-07	Ese	5.02E-07	
Ene	3.49E-07	Ene	4.97E-07	
Se	3.22E-07	Se	4.61E-07	
Nnw	3.15E-07	Nnw	4.51E-07	K-5
Sse	2.57E-07	Sse	3.71E-07	
S	1.78E-07	S	2.57E-07	E-21
Nw	1.68E-07	Nw	2.41E-07	
Ssw	1.50E-07	Ssw	2.19E-07	E-19
W	1.45E-07	W	2.10E-07	
Sw	1.42E-07	Sw	2.06E-07	K-25,E-11
Wnw	1.05E-07	Wnw	1.52E-07	K-38
Wsw	7.23E-08	Wsw	1.06E-07	

Jan-Dec 1991,92,&93 Met Data Chi/Q					K=KNPP E=PBNP
1999 3mi		1999 2-3mi Seg			
N	4.67E-07	N	6.71E-07		K-37
Nne	4.53E-07	Nne	6.48E-07		K-34
Nnw	4.15E-07	Nnw	5.91E-07		K-5
E	3.94E-07	E	5.67E-07		
Ene	3.82E-07	Ene	5.48E-07		
Ne	3.40E-07	Ne	4.87E-07		
Ese	2.58E-07	Ese	3.72E-07		
Nw	2.13E-07	Nw	3.04E-07		
Se	1.87E-07	Se	2.70E-07		
W	1.85E-07	W	2.66E-07		
Wnw	1.61E-07	Wnw	2.31E-07		K-38
Ssw	1.36E-07	Ssw	1.98E-07		E-19
Sse	1.33E-07	Sse	1.91E-07		
Sw	1.07E-07	Sw	1.55E-07		K-25,E-11
Wsw	9.81E-08	Wsw	1.41E-07		
S	7.64E-08	S	1.13E-07		E-21

As you can see, in each meteorological data set, the KNPPs Milk Samples cover the three sectors with the highest dose potential as recommended by NUREG-0472. These changes will not adversely affect the REMP.

For Well Water samples, K-12 was replaced with K-25. K-12 has retired from farming and no longer wished to participate in the program. For ground water, NUREG-0472 recommends 1 or 2 samples if likely to be affected. The KNPPs REMP samples two wells located on site (K-1g and K-1h). For drinking water, NUREG-0472 recommends 1 to 3 samples of the nearest water supply. The KNPP REMP program has K-10,K-11,K-25 and K-13. The REMP also samples water from the Green Bay Municipal Water Pumping Station which draws from Lake Michigan. This changes will not adversely affect the program.

Deleted I-131 from the water reporting levels in Table 2.2.1-D. Per CAP002203, and the fact that the analysis is not currently performed by the vendor. This change will not adversely affect the REMP.

Editorial changes including changing WPSC to KNPP throughout the document due to the NMC operating license change, changed from Teledyne Isotopes Midwest Lab (TIML) to Environmental Inc, Midwest Lab (EIML) due to a corporate name change and changed a reference to the HASL manual which is available on-line.