UNC MINING A[illegible] MILLING SERVICES, INC.

UNC

Subsidiary of United Nuclear Corporation
A **UNC RESOURCES** Company

P. O. Box 2996
Casper, Wyoming 82602

Telephone 307/358-5112

Docket 40-8602

PDR

Return to D. Cramer

396

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June 16, 1981

CERTIFIED MAIL NO. 314678
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U.S. Nuclear Regulatory Commission
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U. S. NUCLEAR REGULATORY COMMISSION
Uranium Recovery Licensing Branch
Washington, D. C. 20555

Attn: Mr. R. A. Scarano

RE: Docket No. 40-8602
Source Material License SUA-1356
Amendment No. 2
License Condition No. 44-A&B

Gentlemen:

Enclosed herein are the responses to the Source Material License SUA-1356 Amendment No. 2, License Condition No. 44-A&B. The following information is herewith enclosed:

1. Water Quality Program
2. Air Quality Program
3. Vegetation Sampling
4. Soil Sampling
5. Laboratory Quality Assurance Program - Camp Dresser & McKee Inc.
6. Laboratory Quality Assurance Program - UNC Mining and Milling Services, Inc.
7. Appendices
8. UNC Map No. 2038, entitled "U.S. NRC SUA-1356 Condition 44-B Restricted Area Boundary Map of Morton Ranch Project," dated June 1, 1981

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Please advise if further information is desired.

Very truly yours,

C. E. Wolff
Project Manager
Morton Ranch

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FEE EXEMPT

response to NRC Order dated 3/26/81

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cc: A. W. Woods
A. S. Chater
F. L. Uhrich
R. L. Morley
R. J. Mullin
M. R. Central File
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UNC MINING AND MILLING SERVICES, INC.

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6.1.A.3.9 WATER SAMPLING PROCEDURE - NRC WELLS AND SURFACE WATER SITES

A. PREPARATION

1. Clean sample containers, caps, filtration apparatus, bailer, and any necessary glassware with rad-cleaner (10% FL-70 Detergent/90% de-ionized water).
2. Rinse all equipment after washing, with de-ionized water, three rinses, then sample water.
3. Clean cells for conductivity meter with appropriate brush and de-ionized water.
4. If using a pH meter, rinse the electrode and thermometer with de-ionized water. If pH is to be measured with a color comparator, clean the optical tubes.
5. Check batteries in conductivity, pH, dissolved oxygen and turbidity meters.
6. Make sure all sample containers have lids securely in place. Pack all sampling equipment and make sure that all glassware and fragile materials are protected from breakage. All equipment should be packed to avoid dust contamination.

B. SURFACE WATER SITES

1. The field notebook should be prepared prior to sampling. Information that should be included is sample code number, sample

location, date, time, and flow rate if applicable. Space should be made for temperature, pH, specific conductance, dissolved oxygen, turbidity, filtering information, preservation information, and shipping information.

2. Rinse sample container three times with water to be sampled. This water should be poured out, away from actual sample point. This should be done either on stream or pond bank, or down stream of sample point.
3. Fill sample containers. This should be done against the natural flow of the water. If possible, the sample should be taken at least six inches below the surface of the water. Care should be taken to avoid contact of sample container with the bottom of stream bed.
4. Immediately fill and rinse small beaker with collected sample. Place thermometer in beaker and record temperature. Use this beaker for pH measurement. Record pH measurement immediately.
5. Take appropriate conductivity, turbidity, and dissolved oxygen cells and rinse three times with collected sample. Obtain reading and record immediately.
6. Filter and preserve sample if needed, following designated procedures.

7. Always take extreme care to avoid cross contamination of samples (i.e. switching container lids, or reusing equipment without cleaning it properly).
8. Always follow all applicable safety procedures.
9. Mark sample containers with appropriate code number in at least two places using a water-proof pen.
10. Make sure there is an airspace in the sample container and that the lid is secure.
11. Rinse pH probe and thermometer and other probes with de-ionized water.

E. PREPARING SAMPLES FOR SHIPPING

1. Check all sample containers for proper information and legibility.
2. Tighten all caps securely.
3. Fill out shipping orders; use one set for each different sample.
4. Place yellow copy inside the shipping box with the sample.
5. Insert green copy into "Packing List Enclosed" envelope and attach envelope to outside of box.
6. Seal shipping box and attach mailing label to top of box.
7. The white copy of the shipping order is filed in the active analysis file.
8. Submit pink copy to the Purchasing Department.

F. MONITORING PARAMETERS

Monitoring parameters for water samples are presented in Tables 1 and 2.

G. SEDIMENT SAMPLES

Sediment samples are collected at surface water sampling sites after spring run-off and late summer following a period of extended low water flow.

Sediment samples are collected from up-stream and down-stream of each site then composited for proper analysis.

UNC MINING AND MILLING SERVICES, INC.

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6.1.A.1.6 WATER SAMPLING PROCEDURES

A. PREPARATION

1. Sample Containers
   - a. Wash all sample containers, with caps on, with rad-cleaner (10% FL-70 detergent/90% de-ionized water.)
   - b. Rinse all containers in three rinses of de-ionized water. (If the amount of de-ionized water available is insufficient, the sample containers may be rinsed three times with the sample water which will be collected prior to filling the containers.)
   - c. Cap all containers securely to avoid contamination.
2. Filter
   - a. Remove hand air pump from top of filter.
   - b. Thoroughly clean all parts of the filter with rad-cleaner solution.
   - c. Rinse three times with de-ionized water.
   - d. Re-assemble filter and keep securely closed until ready to use.
3. Meters
   - a. Check batteries in pH and conductivity meters.
   - b. Check piezometer to be sure it is operating properly.

B. SAMPLE COLLECTION

1. Take piezometer reading to 100ths foot accuracy. Record data in the field notebook.
2. Use bailer or pump to collect the water from the well. The first bailer full, or the first few pump cycles, should be used to rinse the collection vessel. (Be sure to dump any waste water at least five feet away from well casing to prevent contamination.)
3. Collect enough water to fill all sample containers (about 4 gallons). Five gallon Gerry Jugs, cleaned at the same time as the sample containers, may be used to collect the required amount.
4. Set up and calibrate dissolved oxygen meter. Take D-O reading and record in field notebook. If several samples are to be tested, calibration need be done only once and the meter may be left on between readings.
5. Make pH and conductivity measurements on the collected water.
6. Calibrate pH meter with buffered standards closest to the expected pH of the sample.
7. Fill a small previously cleaned beaker with sample water and take temperature. Adjust pH meter for the sample temperature then take pH reading. Record both temperature and pH in the field notebook, as well as the time these readings were taken.

Rinse pH meter probe and thermometer with de-ionized water after each reading is taken.

8. Rinse cell of conductivity meter (Lab line Lectro MHO-Meter, or equivalent) three times with sampler water. Discard rinse water then fill cell and take conductivity reading. Readings should be taken when sample temperature is between 20°C and 25°C. Record this reading in the field notebook.

C. <u>SAMPLE PREPARATION AND PRESERVATION</u>

1. Fill two (2) gallon containers with unfiltered sample water. Label with sample designation number; example: MW-14-1, the date the sample was pulled, and the words "Unfiltered and Unpreserved." Refrigerate until shipped.
2. Filter one (1) pint of sample water into a pint container. Preserve with 2 ml Sulfuric Acid (H_2SO_4). Label the sample container with the sample location, month, S(H_2SO_4) and date sample was taken; example: MW-14-1-S.
3. Filter three (3) gallons of sample into separate gallon containers. Preserve each with 15 - 20 ml Nitric Acid (HNO_3). Label with sample location, month, N(HNO_3) and date sample was taken.

Two gallons will be submitted for analysis; the other gallon is retained for six months.

D. NOTES

1. Use of pre-filter may be necessary prior to use of the 0.45 micron final filter in particularly cloudy samples.
2. If several samples are to be handled at the same time, care must be taken to prevent cross contamination between samples.
   a. Never switch caps on containers.
   b. Wash and rinse the filter thoroughly between samples.
   c. Always complete work on one sample before starting a different sample.
   d. Always have samples clearly labeled.
   e. If more than one preservative is used, make sure that the graduated cylinder used for measuring and dispensing of acids is cleaned between each type of preservative.
3. Samples which require filtration may not sit in excess of six hours prior to filtration.
4. Be sure all necessary information for each sample has been recorded in the field notebook. This includes sample designation number,

time, date, pH, conductivity, dissolved oxygen, location, and static water level. Any other pertinent information regarding a sample should be recorded at this time.

5. Sample designation number is standardized in the following manner:

Location	Month	Preservation
MW-14	1	N or S

In this case the information reflects that this sample was pulled from Monitor Well-14 in January and preserved with Nitric Acid (HNO_3) or Sulfuric Acid (H_2SO_4).

6. Preservation of samples for radiological analysis are tabulated and presented in Table 1.

7. A list of chemical analyses required on all water samples is presented in Table 2.

TABLE 1
PROCEDURES FOR PRESERVATION OF SAMPLES FOR RADIOLOGICAL ANALYSIS

Measurement	Filter	Vol Req. (ml)	Container	Preservation	Holding Time
Gross Alpha	Not Filtered	500	P,G	HNO_3	depends upon half-life of the radionuclide
Dis. Unat	Filtered	1000	P,G	HNO_3	6 Months
Dis. Ra-226	Filtered	1000	P,G	HNO_3	6 Months
Dis. Th-230	Filtered	500	P,G	HNO_3	6 Months
Dis. Pb-210	Filtered	1000	P,G	HNO_3	6 Months
Dis. Po-210	Filtered	250	P,G	HNO_3	10 Days
Sus. Unat	Not Filtered	1000	P,G	----	6 Months
Sus. Ra-226	Not Filtered	1000	P,G	----	6 Months
Sus. Pb-210	Filtered	1000	P,G	HNO_3	6 Months
Sus. Po-210	Filtered	250	P,G	HNO_3	10 Days
Sus. Th-230	Not Filtered	1000	P,G	----	6 Months

TABLE 2
CHEMICAL ANALYSIS
REQUIRED ON ALL WATER SAMPLES

2 Gallon Sample - Filtered and Preserved with Nitric Acid.

Aluminum
Arsenic
Barium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Mercury
Molybdenum
Nickel
Potassium
Selenium
Silver
Sodium
Vanadium
Zinc

2 Gallon Sample - Not Filtered. No Preservation.

Bicarbonate
Carbonate
Boron
Chloride
Fluoride
Sulfate
TDS
TSS

1 Pint Sample - Filtered and Preserved with Sulfuric Acid.

Ammonia (as N)
Nitrate (as N)

1 Gallon Sample - Not Filtered. Preserved with Nitric Acid.

Gross Alpha Activity
If Gross Alpha Activity Exceeds 3 pCi/l
Analyze for Ra-228

Field Measurements

Turbidity
Conductivity
pH
Dissolved Oxygen

TABLE 2 (CONTINUED)

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO MEASUREMENT(1)

Measurement	Vol. Req. (ml)	Container(2)	Preservative	Holding Time(3)
100 Physical Properties				
Color	50	P,G	Cool, 4°C	24 Hrs.
Conductance	100	P,G	Cool, 4°C	24 Hrs.(4)
Hardness	100	P,G	Cool, 4°C HNO_3 to pH <2	6 Mos.(5)
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	25	P,G	Det. on site	6 Hrs.
Residue				
Filterable	100	P,G	Cool, 4°C	7 Days
Non-Filterable	100	P,G	Cool, 4°C	7 Days
Total	100	P,G	Cool, 4°C	7 Days
Volatile	100	P,G	Cool, 4°C	7 Days
Settleable Matter	1000	P,G	None Req.	24 Hrs.
Temperature	1000	P,G	Det. on site	No Holding
Turbidity	100	P,G	Cool, 4°C	7 Days
200 Metals				
Dissolved	200	P,G	Filter on site HNO_3 to pH <2	6 Mos.(5)
Suspended	200		Filter on site	6 Mos.
Total	100	P,G	HNO_3 to pH <2	6 Mos.(5)

TABLE 2 (CONTINUED)

Measurement	Vol. Req. (ml)	Container[2]	Preservative	Holding Time[3]
Mercury				
Dissolved	100	P,G	Filter on site HNO_3 to pH <2	38 Days (Glass) 13 Days (Hard Plastic)
Total	100	P,G	HNO_3 to pH <2	38 Days (Glass) 13 Days (Hard Plastic)
300 Inorganics, Non-Metallics				
Acidity	100	P,G	None Req	24 Hrs.
Alkalinity	100	P,G	Cool, 4°C	24 Hrs.
Bromide	100	P,G	Cool, 4°C	24 Hrs.
Chloride	50	P,G	None Req.	7 Days
Chlorine	200	P,G	Det. on site	No Holdi
Cyanides	500	P,G	Cool, 4°C NaOH to pH 12	24 Hrs.
Fluoride	300	P,G	None Req.	7 Days
Iodide	100	P,G	Cool, 4°C	24 Hrs.
Nitrogen				
Ammonia	400	P,G	Cool,4°C H_2SO_4 to pH <2	24 Hrs.
Kjeldahl, Total	500	P,G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.[4]
Nitrate plus Nitrite	100	P,G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.[4]
Nitrate	100	P,G	Cool, 4°C	24 Hrs.
Nitrite	50	P,G	Cool, 4°C	48 Hrs.

TABLE 2 (CONTINUED)

Measurement	Vol. Req. (ml)	Container(2)	Preservative	Holding Time(3)
Dissolved Oxygen				
Probe	300	G only	Det. on site	No Holding
Winkler	300	G only	Fix on site	4-8 Hours
Phosphorus				
Ortho-phosphate, Dissolved	50	P,G	Filter on site Cool, 4°C	24 Hrs.
Hydrolyzable	50	P,G	Cool, 4°C H_2SO_4 to $pH < 2$	24 Hrs.(6)
Total	50	P,G	Cool, 4°C H_2SO_4 to $pH < 2$	24 Hrs.(6)
Total, Dissolved	50	P,G	Filter on site Cool, 4°C H_2SO_4 to $pH < 2$	24 Hrs.(6)
Silica	50	P only	Cool, 4°C	7 Days
Sulfate	50	P,G	Cool, 4°C	7 Days
Sulfide	500	P,G	2 ml zinc acetate	24 Hrs.
Sulfite	50	P,G	Det. on site	No Holding
400 Organics				
BOD	1000	P,G	Cool, 4°C	24 Hrs.
COD	50	P,G	H_2SO_4 to $pH < 2$	7 Days(6)
Oil & Grease	1000	G only	Cool, 4°C H_2SO_4 or HCl to $pH < 2$	24 Hrs.
Organic carbon	25	P,G	Cool, 4°C H_2SO_4 or HCl to $pH < 2$	24 Hrs.
Phenolics	500	G only	Cool, 4°C H_3PO_4 to $pH < 4$ 1.0 g $CuSO_4$/l	24 Hrs.
MBAS	250	P,G	Cool, 4°C	24 Hrs.

E. PREPARING SAMPLES FOR SHIPPING

1. Check all sample containers for proper information and legibility.
2. Tighten all caps securely.
3. Fill out shipping orders; use one set for each different sample.
4. Place yellow copy inside the shipping box with the sample.
5. Insert green copy into "Packing List Enclosed" envelope and attach envelope to outside of box.
6. Seal shipping box and attach mailing label to top of box.
7. The white copy of the shipping order is filed in the active analysis file.
8. Submit the pink copy to the Purchasing Department.

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6.1.A.1.7 OPERATION INSTRUCTIONS: PIEZOMETER

A. PREPARATION

1. Before going into the field, test piezometer batteries. Open housing lid to allow meter to function. Dip electrode in water; if meter needle registers, batteries are operational.

B. OPERATION

1. Lower cable into well. When electrode reaches water lever, meter will register a steady, positive reading.
2. Using a 10 foot engineer's tape marked in 100ths foot, measure the necessary distance from the nearest brass marker to determine the exact depth to water.
3. Record the well designation number, date, time, and static water level in the field notebook.

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6.1.A.1.8 PNEUMATIC WELL PUMP SYSTEM OPERATING INSTRUCTIONS

A. PREPARATION

1. Check air compressor, pump, hose, regulator, and pneumatic pump for damage or defect. Appendix 1.8.0 presents detailed instructions for setup and use of pump.
2. Clean pump interior and exterior thoroughly with rad-cleaner. Rinse with de-ionized water.

B. OPERATION

1. Set delivery pressure at approximately 100 psi.
2. Lower pump head into well until completely immersed.
3. Secure hose to prevent slippage.
4. Connect air hose to regulator, then attach regulator to air compressor supply line.
5. Pump is ready to operate. Adjustment instructions are presented in Appendix 1.8.0.
6. Run pump to purge, until no less than one gallon has been pumped. Discard water at least five feet from well casing.
7. Collect the required volume of water sample.
8. Once sampling has been completed, disconnect air compressor supply line from regulator, then disconnect regulator from air hose.

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9. Retrieve air hose and pump head from well.
10. Drain and clean pump head and air hose with deionized water.
11. Repeat preparation and operation steps for each well if more than one well is sampled.

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6.1.A.1.9 BAILER USE INSTRUCTIONS

A. PREPARATION

1. Thoroughly clean bailer bucket with rad-cleaner solution.
2. Rinse bucket three times with de-ionized water.
3. Replace bailer in carrying case to avoid contamination during transport.

B. USE OF BAILER

1. Uncap well.
2. Lower bailer bucket by attached cord until bucket reaches water and fills.
3. Raise bailer and dump first bucket of water well away from casing.
4. Refill bailer and use water to rinse sample containers; dump water well away from casing.
5. Refill bailer and start filling sample containers.

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6.1.A.1.10 DISSOLVED OXYGEN METER OPERATING INSTRUCTIONS

A. PREPARATION

Check probe sensor. Replace membrane and KCl solution if necessary using the following steps:

1. Prepare the KCl solution.
2. Remove "O" ring and old membrane. Thoroughly rinse the sensor with KCl solution (electrolyte).
3. Fill sensor body with electrolyte while gently pumping the diaphram with a blunt object (eraser end of pencil) to remove any air bubbles.
4. Replace the membrane and "O" ring (check for wrinkles in membrane or trapped air bubbles).

B. OPERATING PROCEDURES

1. With switch in the "off" position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed.
2. Switch to RED LINE and adjust RED LINE knob until the meter needle aligns with the red mark at the 31° position.
3. Switch to ZERO and adjust to zero with zero control knob.

4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
5. Before calibrating allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been off or the probe has been disconnected.

C. CALIBRATION

1. Instrument should be placed in the intended operating position (vertical, tilted or on its back) before it is calibrated.
2. Draw a volume of water from a common source and carefully divide into four samples. Determine the oxygen in three samples using the Winkler Titration technique and average the three values. If one of the values differs from the other two by more than 0.5 ppm, discard that value and average the remaining two.
3. Place the probe in the fourth sample and stir.
4. Set the Salinity control to zero or the appropriate salinity value of the sample.
5. Switch the desired ppm range and adjust the calibration control to the average value determined in Step 2. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional two minutes to verify stability. Readjust if necessary.

D. MEASUREMENT

1. Place probe and stirrer in the sample and switch stirrer control to "ON."
2. Adjust salinity control knob for the correct salinity.
3. When meter has stabilized, switch to the appropriate range and read D.O.
4. Leave instrument on between readings to avoid the necessity of repolarizing the probe.

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6.1.A.1.11 OPERATING INSTRUCTIONS: CONDUCTIVITY METER

A. OPERATING PROCEDURE

1. Detach the measuring cell from the instrument. Clean it thoroughly with a bottle brush and rinse with distilled water. Use a liquid detergent solution initially, and occassionally thereafter, to remove grease and tenacious deposits. Rinse the cell with a small quantity of the solution to be sampled before filling for measurement.
2. Turn the switch to the anticipated range of measurement.
3. Hold the "On" button down and slowly rotate the measuring dial pointer until the balance indicator is central.
4. Read the measuring dial and multiply by the range factor. The result is the specific conductivity of the sample.
5. When a number of samples are compared for accuracy, measurements should be be made at the same temperature. Place the thermometer in the cell and measure the conductivitiy when the sample temperature falls to the required degree, generally 20°C or 25°C.

B. CALIBRATION

To check the calibration, turn the range selector to "test." Depress the "on" button and note that balace occurs when the measuring pointer is at midscale, marked thus ▼. An indecisive balance, or partial deflection of the indicator when the cell is empty, shows that the battery should be replaced. To replace the "Ever Ready" type PP4 battery remove the undercover of the instrument by withdrawing six screws. Disconnect the battery wires and slide the battery from beneath its retainer.

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6.1.A.2.0 AIR QUALITY PROGRAM (DEQ-AQD & U.S. NRC)

A. REGULATIONS

The statutory requirements of the Air Quality Division may be found in the Wyoming Enviornmental Quality Act, as amended in 1975, and in the 1978 Wyoming Air Quality Standards and Regulations. Futhermore, additional requirements are dictated by the Morton Ranch Source Material License SUA-1356 issued by the U. S. Nuclear Regulatory Commission.

B. INSPECTIONS

Inspections will be conducted by the NRC Enforcement Division and the DEQ-AQD representative.

C. REPORTING

Quarterly Air Quality Reports must be submitted to the DEQ-AOD no later than the end of the second month following the reporting quarter. The reporting quarters end on the last day of March, June, September, and December of each calendar year. Semi-annual reports must be submitted to the NRC in January and July of each year.

D. PERMITS

UNC Mining and Milling Services, Inc., currently operates under the Amended Air Quality Permit CT-238 covering mill operation and expanded mining operations, stripping, etc. This permit must be up-dated only if construction and/or operations will exceed those limitations which have already been approved. Also, UNC Mining and Milling Services, Inc., holds Source Material License SUA-1356 issued by the U. S. Nuclear Regulatory Commission.

E. MONITORING REQUIREMENTS

The parameters which shall be monitored to satisfy the air quality monitoring requirements include supsended particulates, meteorological parameters, and radiological concentrations. Data collection is accomplished by the operation of Hi-Volume air samplers, Low-Volume air samplers, and the Morton Ranch Meteorological Station. Fugitive dust emissions shall be monitored whenever stripping and/or mining activities are in progress. Additional monitoring programs are outlined in the Morton Ranch Source Material License Application as presented in Appendix 1.C.0.

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6.1.A.2.1 REPORTING PROCEDURES

A. GENERAL

Air Quality Reports must contain the particulate data collected from the samplers ASVX1 through ASVX4 and the hourly meteorological data for the three months of each quarter. Particulate data must be recorded in micrograms per cubic meter of air on the state AQDHS report forms. Data must be rounded to the nearest whole number and recorded in the first column marked "reading" opposite the correct date. The starting time must be recorded in military time; example, a start at midnight is recorded as 0-0-0-0.

Meteorological data may be reported in the form of the Morton Ranch Weather Station meteorological data printout as approved by the DEQ-AQD. The only other acceptable means of reporting this data is on the AQDHS report forms. The coding requirements are presented in Table I.

All air quality data is reported to the Nuclear Regulatory Commission semi-annually. These data include meteorological data, particulate data and radiological data collected during the first two quarters, reported in July; and the last two quarters, reported in January of each year.

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TABLE 1

CODING - Code the Air Quality AQDHS forms As follows:

ITEM	CODE
State	52
Area	0180
Site ASVX1	810
Site ASVX2	811
Site ASVX3	813
Site ASVX4	812
Weather Station	805
Project	J02
Time	8
Year and Month (7-9-0-9) for September, 1979, etc.)	
Parameter Codes	
Wind Speed	61101
Wind Direction	61102
Particulates	11101
Method	40 (Visual average)
Units mph	12
Units degrees	14
DP (decimal point)	0 (No tenths)
Status	5
Action	2

UNC MINING AND MILLING SERVICES, INC.

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6.1.A.2.2 PROCEDURES FOR INSPECTIONS

A. GENERAL

The Manager of Environmental/Radiation Department or an assigned representative shall accompany any DEQ-AQD or NRC inspectors whenever an inspection of the Morton Ranch is in progress. Any samples collected during the inspection tour shall be duplicated by the department representative. Notes should be kept of all comments and answers made during the inspection for future reference.

Once the inspection has been completed and prior to departure from the property, the inspectors shall meet with the Project Manager or Acting Project Manager to discuss their findings.

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6.1.A.2.3 RADON MONITORING UTILIZING PASSIVE MONITORS

A. OPERATING PROCEDURES

Passive Radon Monitor operating procedures are presented in detail in Appendix 2.3.0.

B. CALIBRATION

The system is calibrated by exposing the dosimeter to a known amount of radiation.

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6.1.A.2.4 RADON MONITORING UTILIZING TEDLAR BAG METHOD

A. STARTING PROCEDURE

1. Label bag with location, starting date and time. Use military time.
2. Remove white lid from sampler.
3. Connect small filter holder which holds 5.0 micron membrane filters. This filter holder should be connected with the side marked inlet toward the air intake. Replace white lid.
4. Remove the top of sampler.
5. Turn switch to "On" position.
6. Check timing on pulses. The sampler should pulse every 10 seconds. If the timing needs to be adjusted, this can be accomplished by turning the screw by the switch; turning clockwise increases the pulse rate.
7. Attach the bag by connecting the plastic nipples. Make sure the clasp is open on plastic tubing.
8. Secure the lids.
9. Be sure sampler is pulsing before leaving.

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B. STOPPING PROCEDURE

1. Remove the top of sampler.
2. Turn radon sampler to "Off" position.
3. Close clasp on plastic tubing.
4. Remove radon bag.
5. Secure the lid.
6. Record the radon bag pick-up time and date.

C. REPLACING BATTERIES AND FILTERS

1. Batteries need to be replaced quarterly.
2. To replace batteries, remove lid on sampler.
3. Take out battery pack containing four 12-volt batteries.
4. Replace batteries and place back in sampler.
5. Check to see that the sampler cord is connected and working.
6. Check to see that the sampler is pulsing.
7. Change 5.0 membrane filter in samplers monthly.

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6.1.A.2.5 GAMMA DOSE SURVEY

A. PROCEDURE

The Gamma Dose Survey is conducted quarterly at all air sampler sites ASVX1 through ASVX6. The Ludlum Model 19 Micro R Meter or equivalent is utilized to take eight readings, two facing each compass point, at each sample site. These readings are taken at one-minute intervals at an elevation of one meter above the ground surface.

Readings taken on site, including gamma dose rate, ambient air temperature, barometric pressure and wind speed are recorded on the Field Gamma Dose Survey form, presented in Figure 2.

B. INSTRUMENT OPERATION

1. Ludlum Scintillometer (Primary Instrument)
    a. Operating instruction for the Ludlum 19 Micro R Meter are presented in "Instruction Manual - Model 19 Micro R Meter," Appendix 2.5.0.
2. SC-132 Scintillometer (Alternate Instrument)
    a. Preparation
        1. Check batteries prior to use.

b. Operating Procedure

1. Turn "Function" switch to battery position. Meter should read in green. Failure to read in green (below 0.6) indicates insufficient battery strength.
2. If necessary, replace batteries.
3. Set "Function" switch to 1 sec. or 4 sec.
4. Adjust Meter Scale Multiplier control so the meter deflection for the background will be on the upper half of the meter scale.
5. Take readings and record.

c. Additonal Information

1. Due to energy dependent characteristics of the SC-132 Scintillometer survey meter, it is necessary to establish a cross calibration curve to allow correction of readings to the actual gamma dose readings as illustrated in Table 2, and Figure 1.

   Gamma Source with an activity of 1.7276 ± 0.0864µCi was applied in cross calibration of the SC-132 Scintillometer vs. the Pressurized Ionization Chamber (PCI).

2. Pressurized Ionization Chamber (Primary Instrument)
   - a. Preparation
     1. Check batteries.
     2. Prior to use, place sensor head upright and directly above the designated sample point. The sensor head must be stable and balanced before connection of cable to control and readout housing.
   - b. Operating Procedure
     1. Turn "Mode" switch to battery position.
     2. Turn "Electometer" switch to "Zero" and wait 60 seconds to allow transient charging current to die out before proceeding.
     3. Turn "Electrometer" switch to "Read." After a 10-second delay, the signal will appear on the chart recorder.
     4. To display signal, turn "Display/Recorder" switch to "On" position. Depress "Push-to-read" switch. Signal will appear on display for approximately 30 seconds.
     5. Continue to record background gamma dose rate for at least 4 minutes.

c. Stopping Procedure

1. Turn "Display/Recorder" switch to "Off" position.
2. Turn "Electrometer" switch to "Off" position.
3. Turn "Mode" switch to off position.

d. Additional Information

1. PIC should be calibrated annually by the Manufacturer.

C. CALIBRATION

All instruments should be calibrated semi-annually by the manufacturer.

TABLE 2 - CROSS CALIBRATION OF SC-132 vs. PIC WITH CS-137 STANDARD GAMMA SOURCE

DATE	SOURCE DISTANCE (FT.)	SC-132 CPS	SC-132 µR/hr	PIC µR/hr
01.10-79	10	105 ± 3.52	13.34 ± 0.46	17 ± 1.46
01-10-79	9	115 ± 3.79	14.61 ± 0.48	18 ± 1.50
01-10-79	8	125 ± 3.95	15.88 ± 0.50	20 ± 1.58
01-10-79	7	140 ± 4.18	17.78 ± 0.53	22 ± 1.66
01-10-79	6	160 ± 4.47	20.32 ± 0.57	25 ± 1.77
01-10-79	5	190 ± 4.87	24.13 ± 0.62	28 ± 1.87
01-10-79	4	260 ± 5.70	33.02 ± 0.72	35 ± 2.09

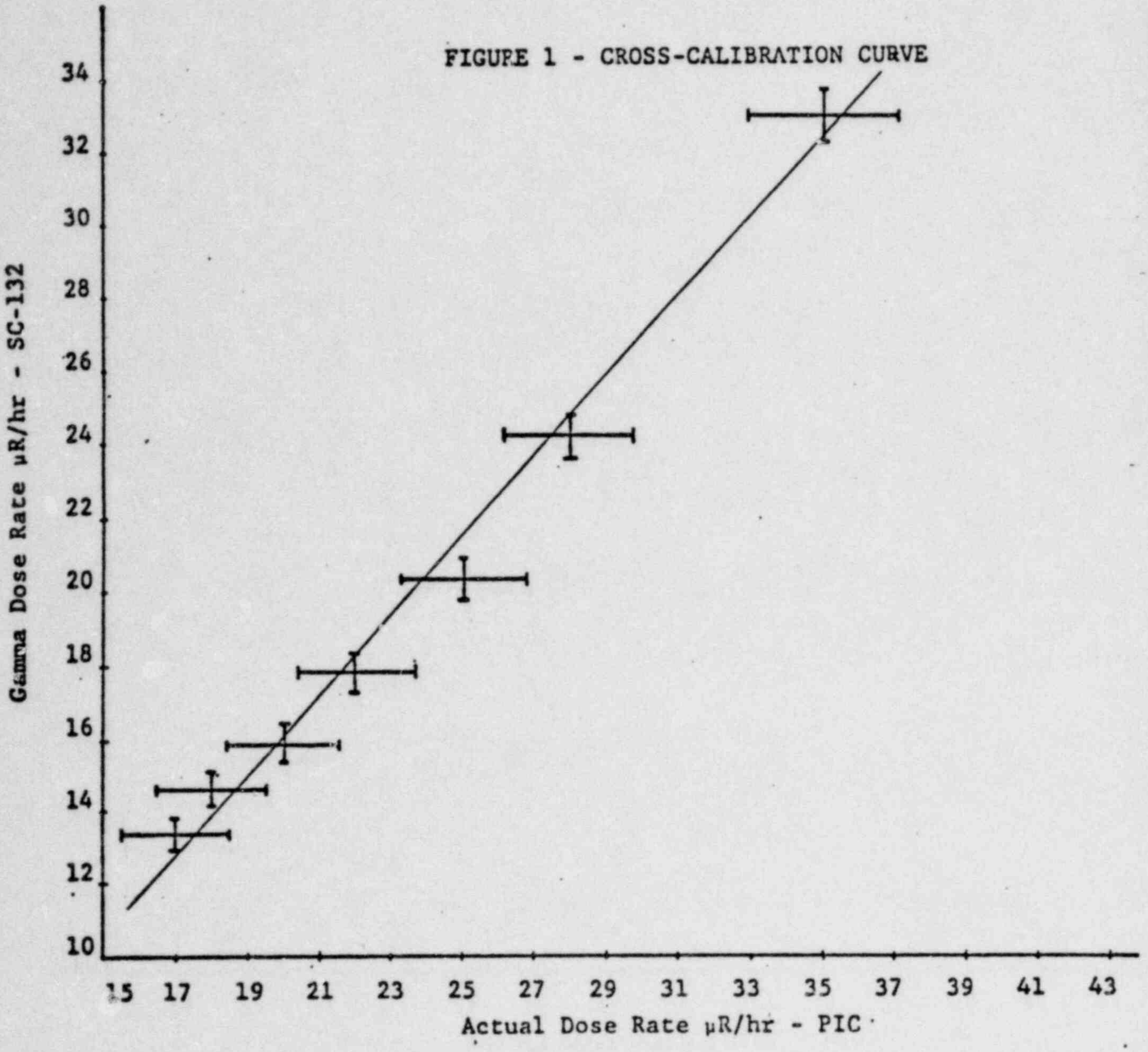
FIGURE 1 - CROSS-CALIBRATION CURVE

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FIELD GAMMA DOSE SURVEY **FIGURE 2**

TECHNICAN: ______________________________

Instruments	Date & Time	Location	Weather Condition	Dose Rate μR/hr	True Dose-Rate μR/hr
Pressurized Ionization Chamber (PIC)					
Portable Scintillometer L-19					
Portable Scintillometer SC-132					

REMARK:

TECHNICAN: ______________________________

Pressurized Ionization Chamber (PIC)					
Portable Scintillometer L-19					
Portable Scintillometer SC-132					

REMARK:

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6.1.A.2.7 MONITORING PROCEDURES

A. GENERAL

The Air Quality Permit CT-238 requires Hi-Volume air particulate sampling on a six-day schedule, as shown in Table 1 for 1981, on a state wide basis. The particulate data collected from the Hi-Volume samplers, as well as the meteorological data collected from the Morton Ranch Meteorological Station, are reported to the DEQ-AQD quarterly. The Nuclear Regulatory Commission requires semi-annual reporting of the Hi-Volume air particulate data, Low-Volume air sampler operational data air filter analysis data, meteorological data, and Radon 222 data.

Fugitive dust emissions must be monitored visually whenever stripping and/or mining activities are in progress. If high visible dust emissions are evident, corrective actions must be undertaken in accordance with the established Morton Ranch Dust Abatement Program.

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TABLE 1
1981 AIR SAMPLING SCHEDULE

1st Quarter	January	February	March
	4 Su	3 T	5 Th
	10 Sa	9 M	11 W
	16 F	15 Su	17 T
	22 Th	21 Sa	23 M
	28 W	27 F	29 Su

2nd Quarter	April	May	June
	4 Sa	4 M	3 W
	10 F	10 Su	9 T
	16 Th	16 Sa	15 M
	22 W	22 F	21 Su
	28 T	28 Th	27 Sa

3rd Quarter	July	August	September
	3 F	2 Su	1 T
	9 Th	8 Sa	7 M
	15 W	14 F	13 Su
	21 T	20 Th	19 Sa
	27 M	26 W	25 F

4th Quarter	October	November	December
	1 Th	6 F	6 Su
	7 W	12 Th	12 Sa
	13 T	18 W	18 F
	19 M	24 T	24 Th
	25 Su	30 M	30 W
	31 Sa		

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6.1.A.2.8 TOTAL SUSPENDED PARTICULATE (TSP) MONITORING

A. HI-VOLUME AIR SAMPLER OPERATION

1. Filter Preparation
    a. Drying Filters
        1. Place appropriate number of 8" X 10" air filters on shelves in vacuum desiccator; close door.
        2. Fasten door latches to secure door.
        3. Check oil level gauge on side of vacuum pump for proper oil level. Add oil if required.
        4. Insert copper tubing through rubber stopper in flask prior to starting pump.
        5. Allow vacuum pump to operate 10 to 15 seconds, close valve on right, then turn off pump.
        6. Unlatch door and check seal.
        7. Repeat steps 5 and 6 if necessary to obtain proper seal.
        8. If door has sealed properly, tightly close valve located on right hand side of desiccator.
        9. Turn off vacuum pump.
        10. Record time.
        11. Remove filters from desiccator after 24 hours drying time.
        12. Weigh filters immediately after removal from desiccator.

b. Weighing Filters

1. Prior to use of balance, check all zero points. Zero points include two front weight dials and the micrometer weight dial on the right hand side of the balance.
2. Fully release balance beam and adjust the zero point by turning the zero point adjustment knob until reading mark coincides with zero line of the optical scale. Arrest balance.
3. Complete steps 1 and 2 prior to removing filters from desiccator.
4. Place filter on balance pan in such a way as to avoid contact with body of balance.
5. Rotate release lever counter-clockwise to "Pre-Weighing" position. The coarse weight, to the nearest gram, will appear on the optical scale.
6. Set weight dials at coarse weight reading, below index mark.
7. Rotate release lever counter-clockwise to fully release balance beam.
8. Once optical scale has stabilized, rotate micrometer dial until reading mark coincides with one line on the optical scale.
9. Record the tare (dry) weight on plastic bag.

10. Arrest balance.
11. Remove filter from balance pan and transfer to labeled plastic bag.

c. Set-Up

1. Place filter, rough side up, on bottom half of filter frame. Put top half of frame over filter and hand tighten the round brass nuts.
2. Cover filter and frame with protective cover.
3. Record filter tare weight, sampling date and sampler name in field log book and on plastic bag.
4. On the back of a recorder chart record tare weight, sampling date and sampler name.
5. Repeat steps 1 through 4 for each filter used.

d. Filter Pick-up and Replacement

1. Lift sample saver cover from filter frame in sampler.
2. Loosen the four corner wing nuts which secure the filter frame and remove frame. Immediately place protective cover over filter and filter frame.
3. Place fresh filter and filter frame in sampler and secure the frame with the wing nuts
4. Remove protective cover from fresh filter and lower sample saver to cover filter. Align filter frame with sample saver cover.

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5. Open recorder door, lift pen from chart and remove chart.
6. Place new, properly numbered chart in recorder being careful to secure the edges of the chart under the retainer tabs.
7. Rotate chart, using a screwdriver in the slotted central pin, until the correct time is indicated by the pointer at the right.
8. Lower the pen until it contacts the chart and adjust the zero, using "zero adjust," if necessary.
9. Set timer dial at the correct time and day (e. g.: if it is 9:00 AM on Thursday and the sampler is required to start at 12:00 PM Saturday, the timer must be set to allow the "ON" trip tab to travel for 39 hours prior to contacting the "On" switch).

   NOTE: Timer dial may only be rotated clockwise.
10. Close and latch all doors. Secure sampler lid with pin through hasp.
11. Return to vehicle with used filter and recorder chart.
12. Carefully remove filter from frame and fold, dirty sides together, in quarters.
13. Place filter and chart in properly identified plastic bag.

14. Record date, military time, sampler run time (as recorded by the digital recorder) and any comments in the field log book.

e. Handling of Used Filters

   1. Place filter, with corresponding recorder chart on top, in vacuum desiccator following steps a.1 through a.12 above.
   2. Weigh filters, after desiccation, following steps b.1 through b.11 above. Tare weight for second weighing will include weight of particulates collected.

f. Preparing Filters for Analyses

   1. At the end of each quarter, separate the filters for each sampler by month. Place all filters for each month in a plastic bag labeled with the sampler designation number, the month during which sampling occurred, the total volume of air sampled (m^3), and the total corrected weight for the month.
   2. Place all samples for each sampler in a manila envelope labeled with the air sampler designation number and the months during which the samples were collected.
   3. Submit for analysis.

B. HI-VOLUME AIR SAMPLER CALIBRATION

1. Quarterly In-House Calibration
   - a. Place clean filter in air sampler and allow to run for approximately 30 minutes for warm up prior to calibration.
   - b. Stop motor and remove filter
   - c. Place calibration cover over filter holder and secure.
   - d. Place #18 calibration plate, seal side down, over opening in calibration cover and attach calibration orifice.
   - e. Place chart in recorder, lower pen to paper and zero pen.
   - f. Attach manometer to side of air sampler housing. Adjust measure in center to read zero at fluid level miniscus.
   - g. Start air sampler and allow to run a few seconds before attaching manometer tube to brass nipple on orifice. (Attaching too quickly may cause some manometer fluid to be sucked into orifice.)
   - h. Allow sampler to run for five (5) minutes; then, just before shut-off, turn recorder chart enough to allow a distinct ascending line and a distinct descending line.
   - i. Take manometer reading and chart reading and record.
   - j. Stop air sampler. Rotate chart to allow space between readings.
   - k. Replace #18 plate with #13 plate. Repeat steps 1.d through 1.j.

l. Replace #13 plate with #10 plate. Repeat steps 1.d through 1.j.

m. Replace #10 plate with #7 plate. Repeat steps 1.d through 1.j.

n. Replace #7 plate with #5 plate. Repeat steps 1.d through 1.j.

o. Run calibration with plates #18 and #13, respectively, a second time after completion of the first run.

NOTE: Temperature and barometric pressure for calculations shall be obtained from the Morton Ranch Meterological station.

2. Orifice Calibration

a. Each Hi-Volume calibration orifice and manometer shall be sent to the EPA for proper calibration once a year.

C. LOW-VOLUME AIR SAMPLER OPERATION

Each Low-Volume sampler site has two samplers which operate continuously in series. Low-Volume sampler sites shall be visited at least once a week at which time the filters in both samplers are replaced.

1. Filter Replacement

   Each Low-Volume sampler requires replacement of the 102 mm diameter circular filter once per week. Two fresh filters are required at each sample site.

   a. Place clean filter in plastic bag which is labeled with air sampler designation number.
   b. Upon arrival at site, throw circuit breaker on power pole to "OFF" position.
   c. Loosen wing nuts on filter frame and remove top portion of frame.
   d. Remove filter, fold in quarters, dirty sides together, and place in plastic bag which was stored in the sampler housing, weighted down by the filter frame.
   e. Place fresh filter, rough side up, in frame. Replace top portion of frame and secure wing nuts.
   f. Record start time (military time), date and digital recorder reading on plastic bag. Secure under filter frame.
   g. Record start time, date, sampler designation number and digital recorder reading in log book.
   h. Repeat steps c through g for the second sampler at the site.

i. Before leaving sampler site, throw circuit breaker on power pole to "ON" position. There will be a short time lag before the sampler starts.

2. Preparing Filters for Analysis

   a. At the end of each quarter, separate the filters for each sampler by month. Place all filters for each month in a plastic bag labeled with the sampler designation number, the month during which sampling occurred, the total volume of air sampled (m^3), and the total corrected weight for the month.

   b. Place all samples for each sampler in a manila envelope labeled with the air sampler designation number and the months during which the samples were collected.

   c. Submit for analysis.

D. LOW-VOLUME AIR SAMPLER CALIBRATION

1. Monthly In-House Calibration

   a. Install new air sampler motor. ("New" means new or repaired.)

   b. Place a clean filter in the sampler.

   c. Start sampler and allow to run for approximately 30 minutes, then stop.

d. Install calibration orifice and attach manometer to air sampler housing. Do not attach manometer tubing at this time.

e. Adjust manometer rule so that zero point is in line with fluid miniscus.

f. Start sampler and allow to run several seconds before attaching manometer tube to brass nipple on side of calibration orifice. Attach manometer tube after initial start-up surge has passed.

g. Allow manometer liquid to stabilize before taking reading.

h. Make 3 readings,one every 15 minutes. Record time and reading in field log book after each reading.

NOTE: Temperature and barometric pressure for calculations shall be obtained from the Morton Ranch Meteorological Station.

2. Annual Orifice Calibration

a. Each Low-Volume calibration orifice shall be returned to the manufacturer for calibration once per year.

E. REPLACING AND SEATING BRUSHES

1. Remove the motor from its housing.
2. Remove brackets and brushes.
3. Replace with new brackets and brushes.
4. Reassemble motor and housing.
5. Connect voltage variator to motor.
6. Set voltage dial on 45 volts for 30 minutes.
7. Turn up voltage dial to 75-90 volts and reseat new brushes for 10 minutes.
8. Brushes on Hi-Volume samplers require replacement semi-annually or quarterly, depending on temperature and dust loading.
9. Brushes on Low-Volume samplers require replacement bi-weekly.

F. CALCULATION OF RESULTS

The particulate data and calibration data should be computed in accordance with the EPA and manufacturer's recommended procedures as presented in Appendix 2.8.0.

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6.1.A.2.9 METEOROLOGICAL STATION OPERATING INSTRUCTIONS

A. GENERAL

The Morton Ranch Meteorological Station consists of a 60-meter tower composed of two instrument levels, one at 10 meters and the other at approximately 60 meters; signal conditioners; programmable data acquisition system; keyboard/printer; cassette tape recorder; and strip chart recorders for each instrument level.

The daily summary print-out provides daily average, hourly average, minimum and maximum values for each meteorological parameter. In addition, the system is fully capable of providing additional pertinent information such as frequency distribution and atmospheric stability (Pasquill Stability Categories A-G) based on wind direction sigma (σ_Θ) at wind speed above 1.5 m/s, and delta temperature (ΔT).

The technique and computation of atmospheric stability (Pasquill Stability Categories A-G) is in accordance with U. S. Nuclear Regulatory Commission Regulatory Guide 1.23, Onsite Meteorological Programs presented in Appendix 2.9.0

B. OPERATING PROCEDURES

Detailed instructions are presented in Appendix 2.9.1.

C. CALIBRATION PROCEDURE

1. Semi-annual system calibration shall be conducted by Technical Environmental Enterprises, or an equivalent service.
2. Daily auto-calibration result shall be reviewed for instrument drift.
3. Signal conditioners shall be recalibrated quarterly, or more frequently if instrument drift occurs.

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6.1.A.2.6 VEGETATION SAMPLING

During the grazing period each year, vegetation samples are collected and analyzed for radiological parameters. These analytical results are reported in the January submittal of the semi-annual report to NRC.

A. PROCEDURES

Vegetation samples are collected three times during the Morton Ranch grazing period, usually in June, August, and October at each air sampler sites ASVX1 through ASVX4 using the following procedure:

1. Randomly place a one-foot-square plot within the area of the air sampler site.
2. Clip all current above-ground vegetative growth enclosed within the plot.
3. Place all clippings in a 12" X 18" plastic sample bag which has been marked with the date, air sampler site designation number, and "V" for vegetation.
4. Repeat plot placement and clipping until a minimum of 1 kg of vegetation has been collected.
5. Record field data in field notebook.
6. Allow sample to dry 1 - 2 weeks prior to anaylsis.

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6.1.A.2.6.1 SOIL SAMPLING

Surface soil samples are collected at the same locations as used for collection of air particulates (Sites ASVX1 - ASVX6).

Soil samples are taken using a plug sampler which is driven into the ground to a depth of five (5) centimeters at each site and then composited for proper analysis.

INTRODUCTION

It is well understood that a quality assurance program, such as the one described herein, is designed to minimize errors and to help produce reliable data. However, a worthwhile quality assurance program also has other definite benefits. It provides for the substantiation of data to meet evidential requirements, since a laboratory which produces reliable data that cannot be substantiated in court is of minimal value. In addition, a quality assurance program also provides a definite cost savings. It is less expensive to do a procedure correctly the first time rather than to repeat it. By providing for written procedures, training of new personnel is carried out most effectively in a minimum of time.

In order to meet these goals a laboratory must maintain certain standards:

1. Adequately educated and trained personnel.
2. Properly calibrated and maintained equipment.
3. Tested, appropriate, written procedures.
4. Quality control to establish the precision and accuracy of measurements.
5. Record keeping which meets evidential standards.

The quality assurance procedures described in this manual are designed to meet these requirements. The following sections provide detailed quality assurance procedures to be followed both in the field and in the laboratory.

It is the individual who is the key to a successful quality assurance program. It is thus essential that each person involved with this program is familiar with this manual and is an active supporter of the program which it describes.

John [illegible]
11/1/77

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GENERAL QA PERSONNEL CERTIFICATION

All scientific personnel should undergo an initial training before sampling or analyzing material for clients. Each individual's training extends to both field and/or laboratory procedures. The field training should include correct use of all equipment, proper procedures for field experiments, and instruction for handling storage, shipping, cleaning and preservation procedures.

The laboratory training should basically consist of: 1) The trainee reading the procedure and becoming familiar with the reagents and equipment used. 2) The trainee preparing and analyzing a set of standards in duplicate under the supervisor's direction. These should fall within the acceptable set of limits in the Quality Control Book. 3) The trainee independently preparing a set of known samples along with blanks and standards in duplicate without help from the other personnel or supervisor.

The date, assay, notebook location of the analysis, and the supervisor's initials are then logged on the Personnel Certification form, a copy of which is retained by the Quality Assurance Manager.

The certification may be renewed according to the supervisors discretion if new procedures or equipment are implemented, or over a certain period of time.

The Group Supervisors, or a delegate appointed specifically by memo, are personally responsible for the training of each technician in his group.

Written UE Date 9-28-77 Q.A. UE Date 9-29-77

Approved 9/30/77, JKW

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EQUIPMENT CALIBRATION AND MAINTENANCE

Instruments and other measuring and testing devices used in activities affecting quality are calibrated and maintained periodically to assure accuracy within specified limits.

Equipment Specifications for this equipment are filed in the Quality Assurance Department and in the files of the department responsible for the equipment. These specifications include:

1. Equipment identification (name)
2. Control number
3. Calibration/maintenance schedule
4. Equipment specifications
5. Specification verification (where applicable)

Written Calibration and Maintenance procedures are maintained for each piece of equipment affecting quality. A copy of these procedures is kept in the files of the Quality Assurance Department and in the files of the department using the procedure. These procedures include, but are not limited to:

1. Equipment idenficiation (name)
2. Control number
3. Calibration and/or maintenance schedule
4. Equipment necessary to accomplish calibration
5. Procedure for calibration and/or maintenance

Instruments requiring calibration or maintenance have a prominently displayed sticker containing the following information:

1. Date of calibration or maintenance

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2. Next due date for calibration or maintenance
3. Initials of agent performing calibration or maintenance

There is a log for each measuring and testing device used in activities affecting quality. This log is retained in departmental files and contains:

1. All data, charts, and curves pertaining to calibration or maintenance procedures
2. Date of calibration or maintenance
3. Next due date for calibration or maintenance
4. Initials of agent performing calibration or maintenance
5. Initials of agent's supervisor (where applicable)
6. Accuracy of equipment prior to and following calibration (where applicable)
7. Record of equipment failures or inability to meet specifications (where applicable)

If calibration schedule is not adequately maintained or accuracy as reported in specifications cannot be attained for a specific instrument, that instrument is labeled "HOLD" and is unavailable for use until the specifications are attained.

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METHOD SELECTION

The methods used at CDM/Accu-Labs must be defendable in court. In many cases this may mean selecting methods that are legally required. (As an example, the EPA often requires that specific analytical methods be employed). In other cases, methods will be used which are recommended by reputable organizations such as ASTM. Literature discussions should also be considered when selecting a method.

Since universal methods applicable in all situations do not exist, persons responsible for methodology selection must be familiar with legal requirements, the recommendations of scientific associations, and the appropriate literature. New methods developed in-house are verified and thoroughly tested by analyzing known standards, spiked samples, and duplicate samples in a variety of matrices. Records of this supporting information should be provided to the Quality Assurance Manager to be stored in the permanent archives.

The use of a well-accepted method by itself does not assure reputable results. Even the most well characterized determinations must be continually monitored with regard to their precision and accuracy as described in the Quality Control Section.

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QUALITY CONTROL

The quality control procedures used at CDM/Accu-Labs are designed to measure the consistency and quality of the data produced. Appropriate parameters are routinely recorded and elementary statistical interpretation of this data is then used to establish the precision and accuracy for each test. Based on these recorded parameters, acceptance limits are determined for individual assays in order to prevent the release of unacceptable data.

The quality control procedures vary considerably between departments. However, certain features are in common:

1. A specified number of samples are repeated at random in order to determine the precision (reproducibility) of the analysis.
2. The accuracy of the methods employed are determined by analyzing spiked samples, by participating in "round robins", and by analyzing known check samples such as those provided by NBS or EPA.
3. The results of standards are charted each time an assay is performed. These results must fall within numerically specified acceptance limits in order for the assay to be accepted.

Each department has a quality control procedure specifying such items as the number of samples to be repeated and the specific acceptance limits (e. g., ±2 standard deviations) appropriate to their type of samples.

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ADEQUATE RECORDS

Controlled Documents

All activities affecting quality are described in writing so that any individual procedure can be fully described and supported at a later date. Because any changes in these written documents must be made in the formalized manner described below, they are referred to as "controlled documents". Controlled documents include, but are not limited to, analytical procedures, descriptions of computer programs, and equipment specifications and calibration and maintenance procedures. Every controlled document is issued a unique document number and is released for use by the following procedure:

1. The author of the document initials and dates his work.
2. The Technical Director or the appropriate supervisory personnel review, date, and sign the procedure.
3. The Quality Assurance Manager reviews the document for completeness, signs and dates it, and assigns it a document number.

Initials indicate approval unless otherwise indicated. Approval of forms is noted on a cover sheet and does not require approval of the Technical Director.

Document numbers for data routing procedures, equipment specifications, and operational procedures consist of seven digits. The first digit designates the class of document; the following two digits, the regional laboratory; the next digit, the department involved; and the last three digits, the order of submission to the Quality Assurance Department:

CLASS OF DOCUMENT

1 = Equip. Specifications
2 = Data Routing Procedures
3 = Operational Procedures

REGIONAL LABORATORY

80 = CDM/Accu-Labs

WRITTEN JRW DATE 11/1/77 Q. A. VE DATE 11-2-77
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DEPARMENT CODE

1 = Lab wide
2 = WA
3 = FC
4 = RC
5 = MS
6 = Overburden

In addition, a revision letter is assigned in order to indicate revisions. A suffix "T" indicates a temporary procedure which is still undergoing development.

Document numbers for forms are assigned a prefix F and four digits which designate the order in which the documents were submitted to the Q. A. Department.

The Quality Assurance department is responsible for distributing controlled documents to the appropriate activity centers.

When a document or form is changed, the document number is altered correspondingly with a revision letter suffix. To initiate a change in a controlled document, a change order form (F8019) is completed, signed by the group leader, dated, and submitted to the Quality Assurance Department. Non-substantive changes such as the correction of typographical errors do not require a change in the revision number. Substantive changes such as changes in procedure require change in the revision letter, and must be reviewed and initialed by the Technical Director or the appropriate supervisory personnel. In either case, a copy of the original and revised document is kept by the Quality Assurance Department in permanent archives.

Occasionally, CDM/Accu-Labs deals with contracts in which the required work is tightly specified in a Scope of Work. Such contracts require additional input from the Quality Assurance Manager in order to assure that specifications in the Scope of Work are met. This includes the review of Request for Purchase documents by the program management and the Quality Assurance Department.

Any deviations from the procedures required in the Scope of Work are referred to as a variance. The Quality Assurance Manager and Program Manager must approve any variances which occur.

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Data Retention

Any data recorded manually in the laboratory is written in ink into cloth bound notebooks or pre-printed forms. Any errors in the laboratory notebooks should be crossed out, not erased or whited out and a short explanation included. The Quality Assurance Manager assigns a unique number to each notebook and records which department has which notebooks. The notebooks may be retained in a secure location in each section until they are no longer needed, at which time they are returned to the Quality Assurance Manager. The Quality Assurance Manager will check periodically to see that all notebooks can be accounted for.

An automatic printer or strip chart recorder can be substituted for a hand-written notebook. However, such tapes should be dated, initialed, and easily traceable to the correct sample and analysis.

Raw data is normally retained for a minimum of two years from the completion of the project; final reports, for a minimum of three years. Data for court cases is retained until otherwise directed by the General Manager of CDM/Accu-Labs.

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No. 3801009 Rev.

QUALITY ASSURANCE ORGANIZATION

The Quality Assurance Department acts as a monitor for all Quality related matters concerning the laboratory, and is independent of all project activities. The Quality Assurance Manager reports to the General Manager of Accu-Labs and also conducts meetings with the management and group supervisors to review any quality related problems.

The Manager's responsibilities include:

1. Auditing and reviewing on a formal basis the implementation of the quality assurance program.
2. Maintaining permanent records as described in this document.
3. Reviewing the scope of work of projects that require specially designed quality assurance specifications.
4. Assigning control numbers to procedures and forms.
5. Educating laboratory personnel in general quality assurance requirements and in the quality assurance requirements of specific agencies.
6. Preparing a monthly written report for management on quality related activities.

The Quality Assurance Department audits all departments on a regular basis (annually at a minimum) for compliance to the quality assurance program. The audit will include a review of record keeping, instrument calibration, personnel certification, and quality control procedures. The Quality Assurance Manager prepares a written report of the audit which is discussed with the group supervisor and other involved management. At this time a copy of this report is filed with the quality assurance archives and a schedule of corrective action (if necessary) is agreed upon.

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No. 3801012 Rev.

QUALITY CONTROL - RADIOCHEMISTRY

Quality control is accomplished in the Radiochemistry section in the following manner:

1. Precision. The precision of each analysis is calculated based on counting statistics whenever possible. In addition, random samples are repeated, as sample volume permits, to further establish the precision.

2. Accuracy. The accuracy of radiochemical analyses is determined by participation in the EPA's Cross Check Program and by the analysis of standards with each set of samples.

3. Consistency. The results of standards are tabulated with each determination. Control limits are set at ± 3 standard deviations (99. 7 confidence limits). Proportional counters are calibrated for efficiency on a regular basis using certified standard sources. Background determinations are performed and tabulated on a regular basis in order to check for instrument contamination. (See instrument calibration and maintenance procedures.)

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PROCEDURE TO ENSURE QUALITY OF DATA PRODUCED

An evaluation is made to document daily laboratory performance in determining if valid data are being produced.

First, valid precision and accuracy data should be available on the method and analyst. Thereafter, systematic daily checks are done to show that responsible results are being obtained, and that the methodology is actually measuring what is in the sample. These items are discussed in detail in this section.

Precision and Accuracy

Precision refers to the reproducibility among replicate observations. The precision is determined on reference standards which cover the expected range of concentrations. For colorimetric analyses, the initial standard curve includes a blank and a series of at least four standards incompassing the full concentration range to be used for routine sample analyses. Subsequently, three standards are analyzed in duplicate to verify the original standard curve. For other measurements, such as pH, conductivity, turbidity, etc., instruments are standardized according to the procedure described in the methods section.

Accuracy refers to a degree of difference between observed and known or actual values. Accuracy is determined by the analysis of known standards and can be reported on a relative or absolute basis, depending on client preference.

WRITTEN TJA DATE 10/7/76 Q.A. TJA DATE 10/9/76

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Percent recovery at each concentration is expressed as the mean of the two replicate results.

Valid precision and accuracy data on the method are available to the analyst, and daily checks are made to ensure that valid data are being generated.

Three (3) sets of duplicate standards are run with each colorimetric test for a total of 6 standards. In addition to standards and spikes, reagent blanks are analyzed in duplicate for each colorimetric test performed. Background data are collected and plotted on a graph of time vs. test value. These data are entered in the appropriate Quality Control data and chart notebook (F8001, F8002, F8003). The range and mean are plotted on the graph until enough background data are recorded to achieve an upper and lower control limit. Once the limits are determined (± 2 standard deviation), the standards run with each analysis must lie between the upper and lower limits. If they do not, the analysis is stopped and the cause is determined before completing the analysis. <u>All</u> standards, spiked samples and/or blanks must lie on the charts.

Once the precision and accuracy charts are established, they are updated with each set of standards run with each analysis. The limits are recalculated periodically including the recent data.

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Duplicates of actual samples are intermittently dispersed throughout a series of samples to be analyzed without the analyst's knowledge. The results for the analysis of those "blind repeats" are recorded on form F8024.

In addition to analyzing standards, reference samples are analyzed that were formulated by the Environmental Protection Agency (unknown to the analyst). This outside sample confirms the accuracy of the method and analyst. The results of these analyses are recorded on form F8023.

Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1975, pages 26-33.

WRITTEN JJL DATE 10/7/76 Q.A. JJL DATE 10/8/76

APPROVED JRW 10/11/76

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WATER LABORATORY PERSONNEL CERTIFICATION

1.) The supervisor of the laboratory shall certify the analyst as competent to perform particular analyses.

2.) All scientific personnel should undergo an initial training period before analyzing samples for clients. This training should satisfy the supervisor of the department, or the project manager. It should basically consist of:
   1) The trainee reading the procedure and becoming familiar with the reagents and equipment used.
   2) The trainee preparing and analyzing a set of standards in duplicate under the supervisors direction. These should fall within the acceptable set of limits in the Quality Control Book.
   3) The trainee independently preparing a set of known samples along with blanks and standards in duplicate without help from the other personnel or supervisor.

3.) The date, assay, and location of the analysis for certification are logged on the Personnel Certification form(F8066). The training individual initials the entry, documenting that sufficient training has been done. The supervisor initials the entry to "certify" the analyst.

) The Group Supervisor is responsible for training but may as appropriate delegate the job of training to someone else. (This is documented by the initials of the trainer on the Personnel Certification Form)

Written DL Date ________ Q.A. VE Date 4-18-79

Approved DL 4-18-79

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GENERAL QA PERSONNEL CERTIFICATION

All scientific personnel should undergo an initial training before sampling or analyzing material for clients. Each individual's training extends to both field and/or laboratory procedures. The field training should include correct use of all equipment, proper procedures for field experiments and instruction for handling storage, shipping, cleaning and preservation procedures.

The laboratory training should basically consist of: 1) The trainee reading the procedure and becoming familiar with the reagents and equipment used. 2) The trainee preparing and analyzing a set of standards in duplicate under the supervisors direction. These should fall within the acceptable set of limits in the Quality Control Book. 3) The trainee independently preparing a set of known samples along with blanks and standards in duplicate without help from the other personnel or supervisor.

The date, assay, location of analysis and the supervisor's initials are then logged on the Personnel Certification form, a copy of which is retained by the Quality Assurance Manager.

The certification may be renewed according to the supervisors discretion if new procedures or equipment are implemented, or over a certain period of time.

The Group Supervisors, or a delegate appointed specifically by memo, are personally responsible for the training of each technician in his group.

Written UE Date 9-28-77 Q.A. UE Date 9-29-77

Approved J.RW 9/30/77

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QUALITY CONTROL - WATER

Quality Control in the Water Laboratory includes the following:

1. Replicate Analyses. 10% of all analyses are routinely repeated to determine precision; control charts are generated from these data.

2. Spiked Sample Analyses. 10% of all analyses are routinely spiked to determine recovery and accuracy; control charts are generated from these data.

3. Cross Check Samples. Known and unknown samples from USEPA, and standard samples from NBS are analyzed routinely to determine analytical accuracy.

4. Standards. The instrumental response of a Standard is charted with each analytical run to determine the comparability with past performance of the method.

5. Data Acceptance. Data which falls outside acceptance limits (± 3 sigma, 99% confidence level) will not be reported. Data which falls between ± 2 sigma and ± 3 sigma (95% confidence level) shall be immediately brought to the attention of the supervisor and reported at his discretion.

6. Control Parameters. Unless specifically ordered by the supervisor, data acceptability will be tested by the results of spiked sample analyses. For determinations for which this is not feasible, the results of Standards will typically be used.

Written C Gill Date 11/1/77 Q.A. VE Date 11-2-77

Approved J RW 11/1/77

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CDM
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planners, & management consultants

CAMP DRESSER & McKEE INC.

11455 West 48th Avenue
Wheat Ridge, Colorado 80033
303 422-0469

QUALITY ASSURANCE PROGRAM

Camp Dresser & McKee has an efficient and smoothly functioning Quality Assurance Program in order to ensure that accurate and precise laboratory data are given to the client. Our Quality Assurance Program was designed to comply with the Criteria for Nuclear Power Plants, set forth in 10 CFR 50. The Quality Assurance Program prepared by Camp Dresser & McKee is documented by written policies, procedures, and instructions. All personnel are responsible for conducting their activities in compliance with these procedures. The Quality Assurance Manager, who is responsible for carrying out periodic technical audits, reports directly to the Manager of the laboratory.

All laboratory analyses are performed utilizing appropriate written procedures which are referenced to standard methods or to literature citations from respected journals. At the completion of an analysis or test, the laboratory technician has his data sheet verified and initialed by his laboratory supervisor. All data are checked and approved by qualified personnel. Control charts are used to monitor the consistency of analysis. Data flow is sufficiently well documented so that results presented in a final report can be traced back to the original citation in the laboratory notebook or worksheet and to the receipt of the sample.

All instruments used in analysis have maintenance and calibration schedules which are rigorously followed. All instruments are checked and standardized whenever an analytical measurement is made. Wherever possible, instruments are calibrated against NBS Standards such as NBS Certified Thermometers.

Camp Dresser & McKee has programs for blind repeats, duplicate analyses and the use of standard samples such as NBS Certified Chemical Standards to ensure the precision and accuracy of the data.

Training procedures are documented in our Quality Assurance Manuals. All program personnel have scientific backgrounds compatible with their function. Camp Dresser & McKee has an active program for the continuing education of its personnel through college courses, short refresher courses, seminars and professional meetings. In this manner, the employees stay abreast of new developments.

The Quality Assurance Manual Documents all sampling techniques, storage and analysis procedures, as well as instrument calibration and maintenance procedures and is continually maintained. These procedures are not deviated from unless revisions are approved through proper channels. Camp Dresser & McKee Quality Assurance Manual, listing all these procedures, is proprietary; however, at the initiation of a project, the client can receive a copy of a Quality Assurance Manual specifically designed for the needs of his project.

Camp Dresser & McKee encourages client audits, inspection of its facilities and analyses of its programs.

CDM
environmental engineers, scientists,
planners, & management consultants

CAMP DRESSER & McKEE INC.

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RADIOCHEMISTRY SERVICE

Camp Dresser & McKee offers a comprehensive radiochemical analysis service specializing in analysis of water, soil, ores, vegetation and animal matter.

Samples are analyzed in accordance with methods presented in "Standard Methods for Examination of Water and Wastewater", 14 Edition, 1975, APHA, AWWA, WPCF; HASL Procedures Manual . J. H. Harley, Ed., Health and Safety Laboratory, U.S. Atomic Energy Commission, 1972; 1975 Annual Book of Standards, Water, Atmospheric Analysis, Part 23, ASTM, American Society for Testing Materials, 1971; Handbook of Analytical Procedures, J. N. Latimer, Et al., USAEC, 1970; Interim Radiochemical Methodology for Drinking Water, USEPA, 1975; and others.

Samples are analyzed for alpha and beta emitting nuclides using internal proportional counters.

Several features of our radiochemical analysis service are outlined below.

A. Quality Assurance

An active quality assurance program. documented by written policies and procedures insures that accurate and precise results are obtained.

Quality control is maintained by a program of analyzing standards, blanks, and repeat determinations with each set of samples. Accuracy of the results is further assured by participation in the EPA cross-check program. In this program, the EPA distributes samples of unknown radioactivity to the participating laboratories who then perform the required analysis and send their results to the EPA. The participating labs are then informed of the correct result for the analysis as well as the results obtained by the other labs.

The proportional counters used are calibrated for efficiency on a regular basis using certified standard sources. Background determinations are frequently performed to check for instrument contamination.

B. Procedure Development

Currently the EPA is engaged in a program of establishing standardized procedures for the analysis of various radioisotopes. Laboratories taking part in the EPA cross-check program are asked to submit copies of procedures they use for a specific analysis. The procedures are reviewed by the EPA and incorporated into a Tentative Reference Method procedure which is sent back to the participating labs for review and criticism. We take part in this program to the fullest extent possible.

C. Detection Limits

Detection limits, given adequate sample, are generally well within the limits established for drinking water by the EPA, State Health Departments and other agencies. All analysis are reported with an error term representing the variability of the radioactive disintegration process at the 95% confidence level, 1.96σ.

D. Technical Assistance

We welcome any inquiries concerning analytical problems with which we may be of assistance. Personnel are willing to discuss any problem in the technical area or in service scheduling.

E. Turn Around Time. . . . . . Or why Does It take So Long, But Not Always?

Completion time for water samples of drinking water quality will generally be two to three weeks. This is determined by the number of samples and analyses required and also by the activity of the sample. Certain determinations require, when radioactivity is present, the measurement of decay rates or ingrowth of daughter products over a period of time varying from one to three weeks. When this is necessary, turn-around time will, of course, suffer.

F. Sample Preservation and Preparation

Water samples for analysis of radium, thorium, gross alpha & beta, strontium, barium, lead, and polonium should be filtered at the time of collection, if possible, and acidified with 40 ml of 8M (1:1) nitric acid per gallon. Acidification of unfiltered samples may result in a redistribution of radioactivity between solid and liquid phases. (Some authorities state that any suspended radioactivity dissolved by such a dilute acid treatment should be considered potentially soluble radioactivity and included in the analysis.) Samples for tritium, cesium, iodine, and potassium should not be treated with nitric acid. All samples are filtered at the time of analysis. Solid samples, excluding vegetation and tissue, should be dried and crushed to -100 mesh if possible. A small sample preparation fee may be charged for those which are not.

G. Sample Size

The following volumes and weights are required in order to assure meeting of EPA, APHA, and State Health Department requirements.

Determination	Volume (ml)	Weight (g) for Solids*
Gross Alpha & Beta	500	0.1
Radium 226, 223, 224	1000	5
Thorium 230	500	5
Strontium 90	1000	5
Lead 210	1000	5
Polonium 210	250	2
Barium 140	1000	
Cesium 137	1000	
Tritium	100	

*For vegetation samples, we recommend collecting at least 500 grams of sample in order to assure a representative sample, and also to report better detection limits.

H. Sample Receiving and Disposition

All charges are for samples delivered to the laboratory or shipped to Denver.

Bus service is convenient and effective, Air Freight is preferred for samples sent from distant location. Samples are discarded 30 days after completion of analyses unless we are notified otherwise.

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UNC MINING AND MILLING SERVICES, INC.

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6.1.A.10.0 QUALITY CONTROL AND QUALITY ASSURANCE

A. PURPOSE

The role of an analytical laboratory is to provide qualitative and quantitive data that will assist in decision making. This is especially true in a program in which laboratory measurements are used to determine whether operation of a facility complies with applicable regulations.

The quality assurance program is designed to control analytical data so that it accurately describes the composition of the samples analyzed. The quality assurance program will continuously ensure the precision and accuracy of the data provided.

B. SCOPE

In order to properly fulfill its function, the quality assurance program will govern personnel training, methods selection, equipment operation, data handling, review of documentation and results, and auditing of the facility. Furthermore it will contain quality control procedures which allow continuous monitoring of the accuracy and precision of analytical results, and assure the proper functioning of laboratory equipment and instruments.

C. RESPONSIBILITIES AND QUALIFICATIONS

The Environmental/Radiation Department Manager, who will not perform laboratory analyses, will be the designated Quality Control Supervisor.

The qualifications and experience of the laboratory staff are listed in Appendix 10.0.0.

D. PROCEDURES

1. Personnel Training

All laboratory personnel will be trained by the Environmental Chemist in all aspects of every analysis in accordance with the procedure outlined in the Laboratory Training Manual. Included in the training will be extensive analysis of spiked samples to demonstrate the accuracy and precision of the individual's analytical performance from the receipt of the sample to the final analytical result. Instruction will also be given in the use and care of routine laboratory equipment and glassware, and routine instrument maintenance.

Retraining of laboratory personnel will be performed regularly, particularly in areas of laboratory work infrequently performed by the individual. Retraining of all individuals will be performed whenever an approved modification is made to an analytical procedure or when it is found that the precision or accuracy of analytical results can be improved.

During training, and thereafter, the analyst shall have in his possession a detailed procedure manual for the particular analysis being performed. It is the responsibility of the Environmental Chemist to ensure that the analyst rigidly adheres to the approved procedure at all times.

2. Methods Selection and Use

The methods used will be those which have been collaboratively studied and found to provide acceptable results for the type of work performed by the laboratory. In many cases this requires the use of methods which have been approved by the Environmental Protection Agency or other regulatory agencies. In cases where no specific method is mandated, documented methods which are in common use will be employed.

Modification of existing methods may be required in order to accommodate the available equipment or to accommodate a particular type of sample. In all such cases sufficient documentation will be obtained through analysis to determine that the modifications do not affect the accuracy of the result.

In order to ensure that the procedure manuals for the laboratory are kept up to date and all revisions are included in all copies, the procedure manuals must be controlled documents. All copies must be numbered and assigned to individuals who are responsible for them. Documentation that revisions have been included in each copy must be maintained. Upon the termination or transfer of any individual from the laboratory, all procedure manuals assigned to that individual must be returned to the Environmental Chemist. Additionally, each copy will be reviewed once a year to determine that it is complete and up to date. Documentation of this review will be kept in the individual copy of the procedure manual and in a separate file.

3. Equipment Operation

Equipment operation will be governed by operators manuals provided by the equipment manufacturer. Their procedure manuals are not controlled documents. Where necessary, procedure manuals for the use of equipment in specific analyses will be written. These procedure manuals will be controlled documents and will be regulated in the same manner as the analytical procedure manuals.

4. Data Handling

Adequate records of each analysis sufficient to recreate a particular result and demonstrate the accuracy shall be maintained. Laboratory worksheets, which must be completed, are provided for each analysis. The laboratory worksheet provides documentation of all data, such as sample volumes, instrument counting times, and calculation of results. Any analytical instrument print out which is obtained from the analysis shall be permanently attached to the laboratory worksheet.

Upon completion of the analysis the laboratory worksheet is submitted to the Environmental Chemist for review. After review the worksheet is retained on file for a period of not less than five years.

Separate log books will be maintained for the quality control records of each analysis and for each major instrument. These log books will contain the results of all control samples, background samples, and spikes. The control charts for each analysis will also be contained in the log book.

The instrument log books will contain all calibration and background determinations and control charts. Additionally, records of all malfunctions and corrective actions taken will be maintained in the log book.

5. Review of Analytical Results

On a quarterly basis the Quality Control Supervisor and the Environmental Chemist will review the analytical results on all samples and the associated spike and cross check samples. Samples will be reviewed through the use of control charts and will take into consideration previous results from the sample site, expected results, and changes in sampling conditions. Spike samples and cross check samples will be reviewed for accuracy and precision. If unreasonable or inaccurate results are found, the cause will be investigated and appropriate remedial action will be taken. Documentation of the cause and the remedial action will be maintained.

At the time of completion of each analysis, the spike sample results will be entered on the control charts. If indications of a problem exist, the analysis of all affected samples will be repeated.

The Environmental Chemist and the Quality Control Supervisor shall verify and recompute approximately 25 percent of all analytical results. Documentation of the recomputation will be maintained. If a computer or calculator program has been used in calculation, the program shall be verified with standard test data on a regular basis. Documentation of the program and the verification of results will be maintained.

6. Review of Procedures

On a yearly basis, the Quality Control Supervisor and the Environmental Chemist will review the analytical and operating procedures for the laboratory. Consideration will be given to new methodology and to the improvement of the existing procedures. Revisions to procedures will be issued as required. Documentation of reviews and revisions will be maintained.

If a new procedure is introduced, it will be documented and all affected personnel will be instructed in its performance. The effectiveness of the procedure will be documented through the analysis of spike samples before use on unknowns.

7. Auditing of the Laboratory

Internal - A quarterly audit of the laboratory facility will be made by the Quality Control Supervisor. Any deficiencies found will be identified in writing. The laboratory will provide written documentation of corrective actions taken in response to the audits. The auditing criteria will be those of the NRC Regulatory Guide 4.15 and such other criteria as are applicable.

External - On a yearly basis an audit will be performed by a qualified individual not involved in the operation of the laboratory. Any deficiencies found by the auditor must be identified in writing. The laboratory will provide written documentation of corrective actions taken in response to the audit. The auditing criteria will be those of the NRC Regulatory Guide 4.15 and such other criteria as are applicable.

8. Quality Control Procedures

Quality control procedures provide a means of checking the accuracy and precision of results and the proper functioning of analytical equipment. Quality control procedures involve the use of various control samples and the application of statistical analyses to analytical results. Not all of the statistical analyses are necessary for every type of control sample as they often represent duplicate means of achieving the same results. The quality control techniques which are to be applied to individual analyses and equipment will be specified in the analytical procedure manuals and equipment operating manuals.

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a. Duplicate Analyses

Sample analysis will be performed in duplicate on a minimum of one sample in every ten samples present in a sample run. In the event of less than ten samples being present in a run a minimum of one duplicate analysis will be performed. The sample analyzed in duplicate may be either a regular sample or a synthetic sample whose actual value is unkown to the analyst.

Accurate records will be kept for duplicate analyses. All factors which might affect the analytical performance will be recorded. Minimum information will include the date, the analyst, and instruments used.

After a minimum of 20 sets of duplicate data has been accumulated for a particular analysis, a statistical range analyses will be performed in which a mean range ($\overline{R}$) between duplicate analyses is calculated from the standard deviation of the analysis. The formula for this calculation is $\overline{R}=d_2\sigma$ where d_2 is a function of the replicates involved and σ is the standard deviation.

The control limits can be calculated by $\overline{R}+3\sigma_R=D_4\overline{R}=D_4d_2\sigma$ where σ_R is the standard deviation of the D_4 is a function of the number of replicates involved. The factor d_2 and D_4 are presented in the following table for different numbers of replicate samples (eg., duplicate, triplicate):

FACTORS FOR CALCULATING RANGE CONTROL CHART LINES

Number of observations	Central line factor (d_2)	Control limit factor (D_4)
2	1.128	3.267
3	1.693	2.575
4	2.059	2.282
5	2.326	2.115
6	2.534	2.004

After setting up range limits, results can be evaluated. The theoretical distribution is as follows:

Range	Percent of results
$\leq(\overline{R} + \sigma_R)$	84
$\leq(\overline{R} + 2\sigma_R)$	97.5
$\leq(\overline{R} + 3\sigma_R)$	100
$>(\overline{R} + 3\sigma_R)$	0

An acceptable distribution will either be close to that predicted by theory or shifted toward the σ_R and $2\sigma_R$ categories. An unacceptable distribution will be shifted toward the $2\sigma_R$, $3\sigma_R$, and $>3\sigma_R$ categories.

An "outlier" distribution follows the theoretical distribution, but has a few results in the $>3\sigma_R$ category. This indicates acceptable precision with occassional poor results. The "outliers" should be examined closely to determine their source.

b. Spike Samples

The accuracy of laboratory data can be established by preparing special standard samples containing known concentrations (spikes) of the constituents of interest and statistically studying the relation between the resulting analytical values and the known concentrations. The spikes will be analyzed in the ratio of one spike for every ten samples in a sample run. In the event that a sample run contains fewer than ten samples, no less than one spike will be run.

Accurate records will be kept for spike analyses. All factors which might affect the analytical performance will be recorded.

Minimum information will include the date, the analyst, and the instruments used.

One of the following types of control chart will be maintained for the spike samples related to each analytical procedure.

1. Means Control Chart (See Figure 1)

   The means control chart enables one to determine the amount of bias between the mean and the expected value. It is assumed that the resulting data will resemble a normal distribution over time. When preparing such a control chart one assumes the known activity to be the mean. The allowable standard deviation for the individual determinations is selected and the standard deviation of the mean is calculated by dividing the standard deviation of the individual measurements by the square root of the number of determinations ($\sigma m = \sigma/\sqrt{n}$). Acceptable results should fall within ± $2\sigma m$ for 95% of the results. Any bias will be evident as a consistent displacement from the mean.

2. Individual Results Control Chart (See Figure 1)

The control chart for individual results is essentially the same as the means chart except that individual values are used rather than the mean value. In this chart the standard deviation of the individual values, rather than for the mean, is used. The known value is assumed to be the central value. Both charts may be used to determine analytical trends and to correct problems before they reach a level which would seriously compromise the analytical results.

c. Cross-Check Analyses

Cross-check analyses are of essentially two types:

1. Cross-check samples supplied by an agency such as the Environmental Protection Agency to a large number of laboratories to test the ability of individual laboratories to perform accurate analyses. The results from individual laboratories are reported to the agency which, in turn, performs a statistical analysis on the results and reports to each individual laboratory its performance with respect to the known values and the performance of the other laboratories.

This laboratory will participate to the greatest extent possible in such cross-check programs as the Environmental Protection Agency - Las Vegas Incomparison Program for all applicable samples.

2. Split - Sample analyses between two or more laboratories where one of the participating laboratories supplies the sample to be analyzed. The sample may be an unknown, a synthetic spike or a sample whose values are known to the supplying laboratory through prior analysis. The use of actual samples is the only way for an environmental laboratory to estimate the accuracy of its results at true environmental levels. Programs such as those in part a, above, because they cannot be carried out at the low levels found in the environment, do not substitute for a split-sample program.

   This laboratory shall have an ongoing split sample program with the Tennessee Valley Authority analytical laboratory and one or more other laboratories. This laboratory will supply, at least once each year, a split sample for analysis for each analytical procedure routinely performed.

This laboratory shall participate to the extent requested in analyzing the split samples supplied by the Tennessee Valley Authority and other laboratories.

d. Blank Sample Analysis

Blank samples shall be analyzed with each set of samples. Blank samples can be used effectively to determine analytical problems and to correct the analytical results on samples.

All blanks analyzed with samples will be process blanks which shall be deionized water, analyzed in the same manner as an actual sample. This allows a check on the contaminants introduced by the laboratory analysis and chemical reagents. This also allows for the correction of sample results.

Additionally, deionized water blanks, to which no reagents have been added, will be used in instrumental analyses to check proper instrument function and to detect and correct for possible contaminants in the deionized water itself.

9. Radiation Detection Instrumentation Quality Control Procedures

a. Radioactive Standard Check Sources.

The resources chosen as check sources will be traceable to the National Bureau of Standards and will contain nuclides whose type and energy of radiation correspond to the type of analysis for which the system is being used. The sources will also closely approximate the geometry of the sample to be analyzed.

Radioactive Standard Check sources and radioactive solutions, because of their potential danger and because improper handling might destroy their calibration, will be kept in a locked cabinet when not in use. Their use will be governed and documented by the relevent procedures in the laboratory procedure manual.

Instrument effeciencies will be determined on all instruments at a frequency which will ensure that analytical results will not be effected by an instrument drift or malfunction. In most cases it will be possible to check the instrument efficiency immediately prior to counting of a sample. Instrument backgrounds will also be determined on a regular basis.

All efficiency and background determinations will be documented and entered on instrument control charts. (See Figures 1 and 2)

b. Instrument Control Charts

The statistical nature of radioactive decay will result in uncertainties in the determinations of check source and background count rates. Deviations from the "true" values that result from instrument malfunctions can be quickly detected through the use of control charts of the routine check and background determinations.

A control chart consists of a graph showing time on the abscissa and count rate, or total counts, on the ordinate. (See Figure 3). Lines drawn parallel to the time axis at values for the "true" count rate and for values of ± 2 and ± 3 standard deviations. The "true" count rate for a source is determined by at least 30 countings whose individual statistics are acceptable or from a single measurement of at least 10 hours.

When a point goes outside the limits a further check is necessary to determine whether service may or may not be required. A series of repeated measurements should be run and a Chi-square test applied to the results to determine the need for service.

Control charts will be received on a regular basis (the time period must be determined by the number of analyses performed in order to obtain a statistically valid base) to check for possible trends. If trends are present, the cause will be determined and appropriate action taken.

The detector plateau for all gas discharge and scintillation instruments shall be determined on a quarterly basis and before use after any modification or repair.

Multichannel analysis systems will have a quarterly energy calibration using either a mulit-isotope source or several single isotope sources.

10. Sample Handling and Treatment

Once a representative sample is collected and delivered to the laboratory the laboratory staff is responsible for properly treating and storing the sample. Often samples collected in an environmental survey require treatment because they are not physically ready for analysis. Treatment of the sample after it is received depends on the sample itself and the analyses to be performed on it. Most treatment and handling techniques have been established and well known for many years. Again, certain precautions must be taken in the radioanalytical laboratory.

a. Water Samples

Generally, water samples should be acidified when collected. Under certain conditions this procedure should be modified; for example, if total and dissolved fractions are to be analyzed, the samples should be filtered before acidification. If the samples are to be stored for any length of time, carrier or complexing agents should be added to prevent absorption of trace metals on the storage containers. Absorption is known to occur quite rapidly under some conditions. See Table 1 for recommended sample volumes and preservation.

b. Air Filters

The air filter should be handled with care when dust loading is observed because particulate matter is easily removed from the filter, thus invalidating the analysis. Air filters are often stored in the envelopes; some extremely low level analyses may require analysis of the envelope in which the sample was stored as well as the sample itself.

c. Soil

Soil samples shall be dried, pulverized, and sieved to pass a 200 mesh (this will vary according to the analyses to be performed) sieve before analysis. Further thorough mixing is required to ensure a homogeneous sample.

d. Other Samples

Perishable samples should be preserved by refrigeration or freezing. Vegetation and other samples may need to be dried, pulverized, or ashed before analysis.

11. Data Handling and Reporting

A system for controlling the passage of samples through the laboratory is required to assure that the proper analyses are assigned and completed.

a. As they arrive, samples are recorded in a log book. At this time, a laboratory number is assigned to the sample; and all descriptive information concerning the sample, including sample source, sample type, quantity, collection time, and sample collector is recorded.

b. An analytical request sheet, which lists the sample number and descriptive information about the sample, is prepared for the sample. All necessary pretreatments and analyses to be performed should appear on the sheet. Space is provided for completion dates.

c. As results are completed, more information, such as the name of the analyst who performed the work, his calculations for the sample, and all pertinent data that would allow recalculation of results at a later time if necessary, is noted on the result sheets.

d. As various analyses are performed and results are reviewed by the analyst, the request sheet is checked off and the result sheets are attached to the analytical request sheet.

e. After all analyses are completed, the results are sent to the laboratory supervisor. After the results are reviewed, they are transferred to a final report form.

f. The analysis request form and result sheets are appropriately filed for future reference. Results shall be kept for ready reference.

12. General Procedures

a. Individuals may not enter the laboratory area without first removing any significant contamination.

b. The adhesive floor mats at the laboratory entrances will be replaced as required to assure the removal of dust and soil from shoes.

c. The air filters on the laboratory air inlets will be inspected weekly for dust loading and cleaned as required.

d. All chemicals and reagents used in the laboratory will be of sufficient purity to prevent interference or introduction of contaminants into the samples. Reagent grade chemicals will be used except where higher purity is required or where reagent grade is not commercially available.

e. All primary laboratory standards and all radioactive standard solutions will be traceable to the National Bureau of Standards except in cases where the National Bureau of Standards does not provide certification.

f. The deionized water used in the laboratory will be monitored through the sample analysis blanks and calibration blanks analyzed in the normal course of laboratory work. Corrective action will be taken as required to prevent the introduction of contaminants.

TABLE 1

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO MEASUREMENT(1)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time
100 *Physical Properties*				
Color	50	P,G	Cool, 4°C	24 Hrs.
Conductance	100	P,G	Cool, 4°C	24 Hrs.
Hardness	100	P,G	Cool, 4°C HNO_3 to pH <2	6 Mos.
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	25	P,G	Det. on site	6 Hrs.
Residue				
Filterable	100	P,G	Cool, 4°C	7 Days
Non-Filterable	100	P,G	Cool, 4°C	7 Days
Total	100	P,G	Cool, 4°C	7 Days
Volatile	100	P,G	Cool, 4°C	7 Days
Settleable Matter	1000	P,G	None Req.	24 Hrs.
Temperature	1000	P,G	Det. on site	No Holding
Turbidity	100	P,G	Cool, 4°C	7 Days
200 *Metals*				
Dissolved	200	P,G	Filter on site HNO_3 to pH <2	6 Mos.
Suspended	200		Filter on site	6 Mos.
Total	100	P,G	HNO_3 to pH <2	6 Mos.

TABLE 1 (CONT)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time
Mercury				
Dissolved	100	P,G	Filter on site HNO_3 to pH <2	38 Days (Glass) 13 Days (Hard Plastic)
Total	100	P,G	HNO_3 to pH <2	38 Days (Glass) 13 Days (Hard Plastic)
300 Inorganics, Non-Metallics				
Acidity	100	P,G	None Req	24 Hrs.
Alkalinity	100	P,G	Cool, 4°C	24 Hrs.
Bromide	100	P,G	Cool, 4°C	24 Hrs.
Chloride	50	P,G	None Req.	7 Days
Chlorine	200	P,G	Det. on site	No Holding
Cyanides	500	P,G	Cool, 4°C NaOH to pH 12	24 Hrs.
Fluoride	300	P,G	None Req.	7 Days
Iodide	100	P,G	Cool, 4°C	24 Hrs.
Nitrogen				
Ammonia	400	P,G	Cool,4°C H_2SO_4 to pH <2	24 Hrs.
Kjeldahl, Total	500	P,G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
Nitrate plus Nitrite	100	P,G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
Nitrate	100	P,G	Cool, 4°C	24 Hrs.
Nitrite	50	P,G	Cool, 4°C	48 Hrs.

TABLE 1 (CONT)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time
Dissolved Oxygen				
Probe	300	G only	Det. on site	No Holding
Winkler	300	G only	Fix on site	4-8 Hours
Phosphorus				
Ortho-phosphate, Dissolved	50	P,G	Filter on site Cool, 4°C	24 Hrs.
Hydrolyzable	50	P,G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
Total	50	P,G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
Total, Dissolved	50	P,G	Filter on site Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
Silica	50	P only	Cool, 4°C	7 Days
Sulfate	50	P,G	Cool, 4°C	7 Days
Sulfide	500	P,G	2 ml zinc acetate	24 Hrs.
Sulfite	50	P,G	Det. on site	No Holding
400 Organics				
BOD	1000	P,G	Cool, 4°C	24 Hrs.
COD	50	P,G	H_2SO_4 to pH <2	7 Days
Oil & Grease	1000	G only	Cool, 4°C H_2SO_4 or HCl to pH <2	24 Hrs.
Organic carbon	25	P,G	Cool, 4°C H_2SO_4 or HCl to pH <2	24 Hrs.
Phenolics	500	G only	Cool, 4°C H_3PO_4 to pH <4 1.0 g $CuSO_4$/l	24 Hrs.
MBAS	250	P,G	Cool, 4°C	24 Hrs.

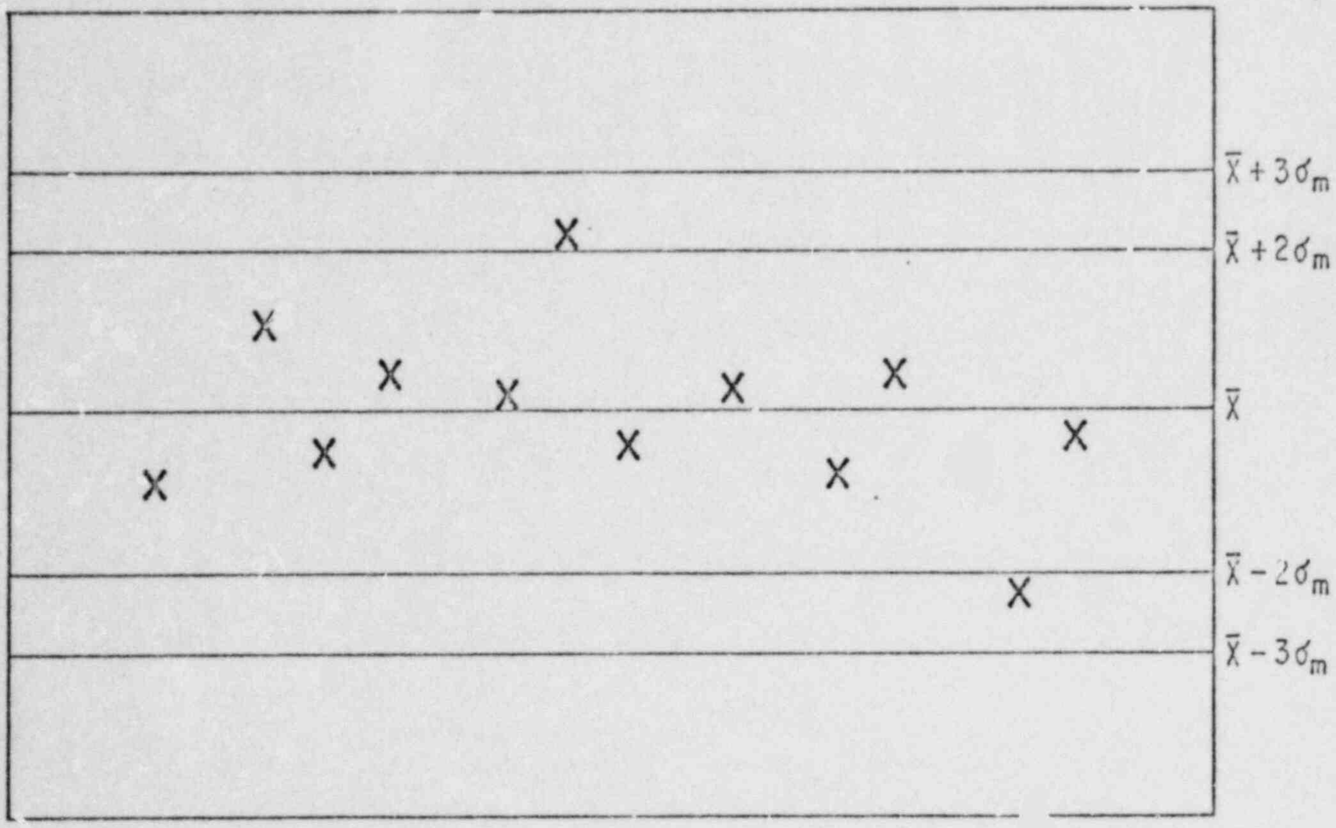

Figure 1 Means control chart. $\bar{X}$ is mean, $\sigma = \sigma/\sqrt{n}$, the ordinate is concentration, and the abscissa is time.

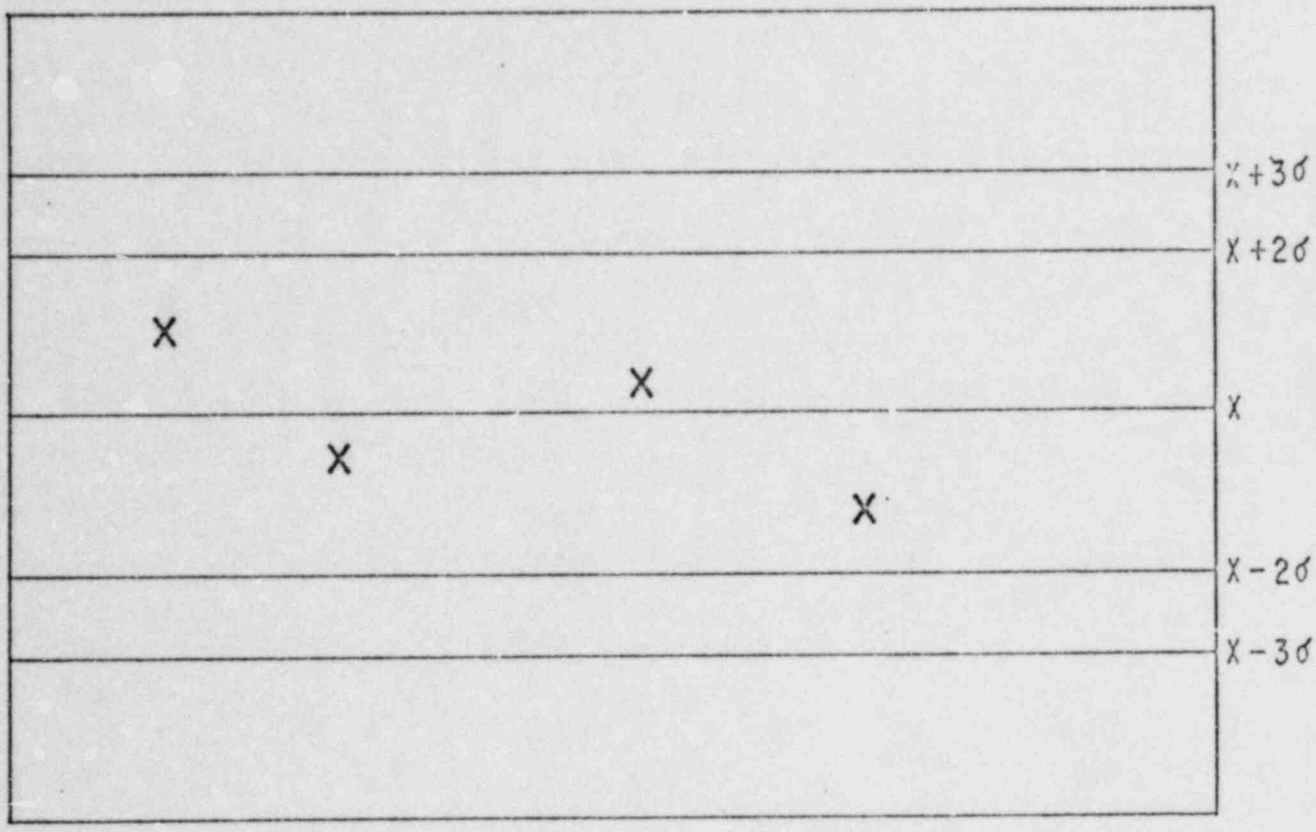

Figure 2 Individual results control chart. X is the known concentration (ordinate), σ is the standard deviation, and the abscissa is the run number.

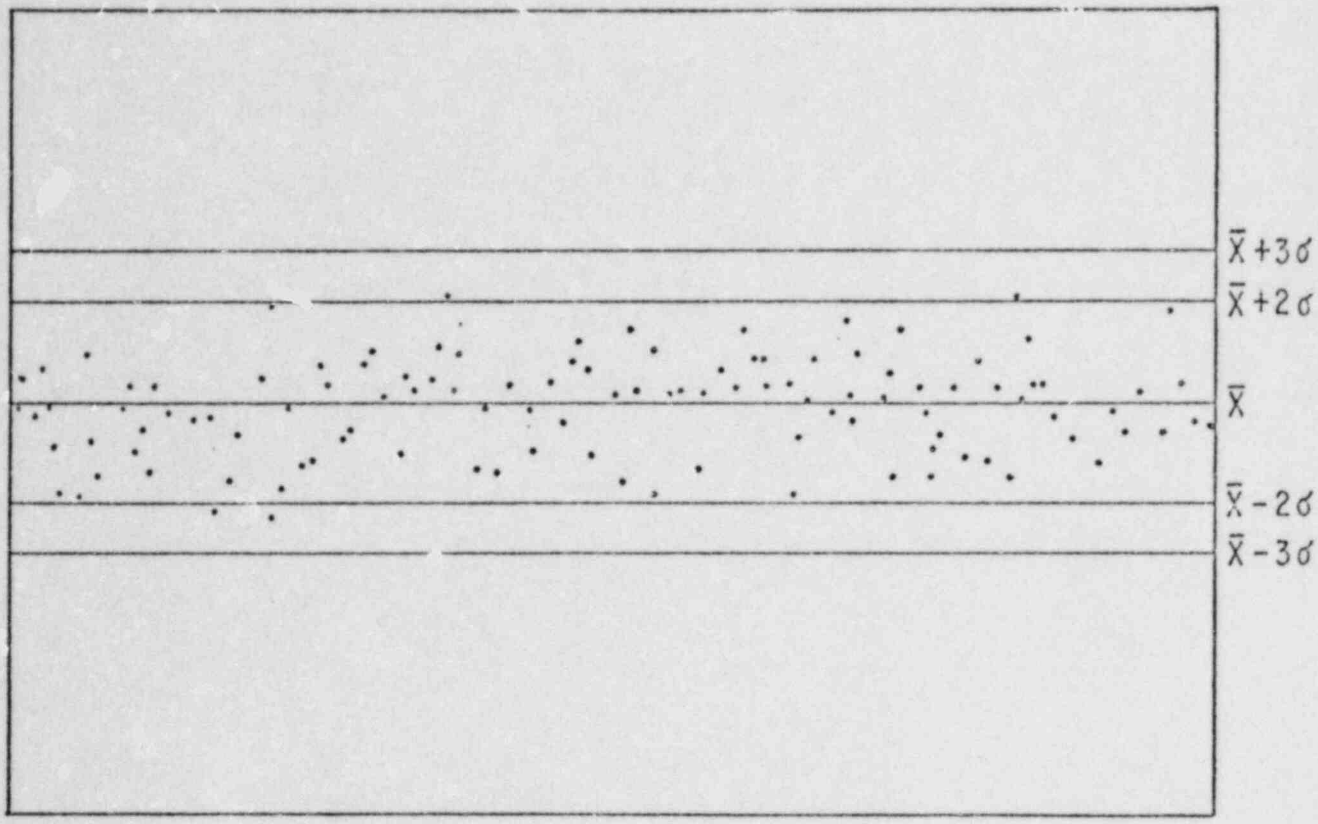

Figure 3 Control chart for measurement of background radiation.

13. Appendices

The following additional information is provided within the appendices:

1. Appendix 10.0.1 - Statistical Terminology
2. Appendix 10.0.2 - Significant Figures
3. Appendix 10.0.3 - Errors Caused by Computational Processes
4. Appendix 10.0.4 - Radiochemical Statistics and Counting Data
5. Appendix 10.0.5 - Bibliography

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6.1.A.11.0 LABORATORY SECURITY AND CONTROLLED ACCESS

A. PURPOSE

To maintain laboratory safety and security, access to the laboratory is restricted to prevent entry of unauthorized and/or unqualified personnel. Control of access is necessary to protect the visiting individual, the laboratory personnel, the laboratory equipment, and the laboratory environment from any potential hazards. The following steps have been implemented to maintain laboratory security and controlled access:

1. The entrances to the laboratory are to be kept locked at all times.
2. Authorization to enter the laboratory may be obtained only from the Environmental/Radiation Department Manager or the Environmental Chemist.
3. Authorization will be granted by the Environmental/Radiation Department Manager or the Environmental Chemist only if the individual has a specific need requiring his presence in the laboratory and is qualified to perform the required work.
4. The following individuals are routinely authorized to enter and work in the laboratory as required, provided that the Environmental/Radiation Department Manager is notified in advance:
    a. Project Manager
    b. Environmental/Radiation personnel

5. Visits to the laboratory by UNC personnel and outside individuals must be arranged in advance with the Environmental Radiation Department Manager, or in his absence, the Environmental Chemist.

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6.1.A.12.0 LABORATORY SAFETY

A. PURPOSE

Laboratory safety can be effectively implemented only by ensuring the proper use of the facilities and the personnel conduct in the laboratory. UNC MMSI has provided at the South Morton office a well-designed laboratory with the facilities and equipment necessary to perfom the analytical work as required. To ensure the effective and safe use of these facilities, the laboratory safety program encompasses the following areas:

1. Personnel conduct
2. Proper use of equipment and chemicals
3. Training
4. Decontamination of facilities and personnel
5. Personnel monitoring
6. Personnel evacuation

The more specific safety precautions required by particular procedures, chemicals, and equipment will be addressed within the individual analytical and equipment operating procedures in which they are used.

B. PERSONNEL CONDUCT

1. Persons working in the laboratory shall wear safety glasses and a lab coat at all times; in addition they shall wear clothing

and protective equipment dictated by the procedure being performed.

2. No lighted smoking materials shall be brought into or used in the laboratory.
3. No person shall bring food or beverages into the laboratory.
4. No person shall use laboratory chemicals or glassware outside of the laboratory except when immediate preservation of samples is required in the field.
5. Laboratory clothing and protective equipment shall not be removed from or worn outside the laboratory.
6. Protective equipment shall be maintained in proper condition for use.
7. No chemical or sample shall be placed in a container without first labeling the container as to the contents.
8. No reagent container shall be left without a lid securely in place.
9. All persons working in the laboratory shall be familiar with the locations and operation of the eye wash bottles, eye wash stand and safety showers.
10. No apparatus or equipment shall be placed so as to restrict access to the eye wash stand, safety showers, or exits.
11. All aisles and doorways shall be kept free of obstructions.
12. Any person working alone in the laboratory shall do so only after notifying the Environmental/Radiation Department Manager or the Environmental Chemist and ascertaining that he

or another authorized individual will be in the immediate area and will make periodic checks of the laboratory.

13. All persons working in the laboratory shall report all accidents or injuries, no matter how minor, to the Environmental Chemist immediately.

14. When two or more persons are working in the same laboratory area they shall wear the protective equipment necessary to protect them against the hazards of all analyses being performed.

15. Two or more incompatible analyses shall not be performed simultaneously in the same laboratory area.

16. All work involving volatile liquids or flammable liquids shall be performed in a properly operating fume hood.

17. All work which generates toxic or irritating fumes shall be performed in a properly operating fume hood or with a fume isolation system.

18. All work involving perchloric acid shall be performed only in the perchloric acid fume hood which shall be cleaned after each use.

19. To the extent practical, samples being analyzed outside of fume hoods shall be covered.

20. All other persons in the laboratory shall be notified when an individual intends to use an open flame or an electric heating device outside of a fume hood.

C. PROPER USE OF EQUIPMENT AND CHEMICALS

1. All broken glassware shall be discarded in a clearly marked receptacle.
2. Cracked glassware shall be discarded, and not used in the laboratory.
3. Empty chemical containers shall be rinsed, and the rinse properly discarded, before disposal of the container.
4. Equipment shall not be used for a purpose for which it was not designed.
5. If an instrument or equipment malfunctions, it shall be turned off immediately and the Environmental Chemist notified.
6. All instruments, equipment, and chemicals shall be properly stored in their assigned locations when not in use.

D. LABORATORY TRAINING

Laboratory training is required for each individual working in the laboratory regardless of previous background and experience. The equipment available in the laboratory necessitates the use of particular procedures in order to achieve the best analytical results. Therefore, the following training rules shall be enforced.

1. An analytical procedure shall have a detailed step by step instruction which must be adhered to at all times when an analysis is being performed.

2. Instruction for the proper use of each instrument will be given by the Environmental Chemist.
3. The Environmental Chemist shall demonstrate to each individual each analysis that will be performed, including the proper use of all protective equipment as required.
4. After a procedure is demonstrated, the individual in training shall perform the analysis in the presence of the Environmental Chemist.
5. The Environmental Chemist shall be present whenever the analysis is being performed until such time as the individual is able to perform the analysis in a proper manner.
6. The Environmental Chemist will certify in writing the individual's ability to perform the procedure without his direct supervision.

E. DECONTAMINATION OF FACILITIES AND PERSONNEL

The laboratory is subject to two (2) major types of contamination which must be effectively controlled in order to provide quality analytical results and to protect the personnel and equipment within the laboratory. The types of contamination fall into the following general catorgories.

1. Contamination from material introduced to the laboratory from the outside.

This type of contamination is the more easily handled of the two and involves means of controlling the entry of contaminants. In the case of individuals it involves having proper facilities to remove contaminants before entering into the laboratory.

Entry of contaminants into the facility by non-human means is controlled by the installation of air filters on all air inlets to prevent the entry of dust into the laboratory and by the availability of purified water, free of contaminants. Both the air filters and the water purification system are checked at frequent intervals to ensure continued efficiency.

Entry of contaminants by human means is in part controlled by the security and access portion of this manual which allows entry of authorized and trained indiviuals. Additionally, the following rules must be followed by those authorized individuals:

a. Persons entering the laboratory must wear uncontaminated clothing and shoes.

b. In certain cases where the person has been working outside the laboratory and might be highly contaminated (eg. the person had been collecting water samples and had become covered with mud), use of the decontamination room located outside of the laboratory and changing into clean clothes will be required.

c. Special adhesive floor mats are provided at the entrances to the laboratory to remove loose surface soil from the soles of shoes.

d. Individuals who work both inside and outside the laboratory shall keep suitable clothing available for wearing when working in the laboratory.

2. Contamination from spills or careless handling of materials within the laboratory.

To prevent contamination from within the laboratory, the following precautions must be observed:

a. All chemicals, standard solutions, and liquid samples shall be stored in containers with tightly fitting lids.

b. All containers should have clean exteriors and any leakage should be removed.

c. All solid samples shall be stored in containers with tightly fitting lids or in sealed plastic bags.

d. All spills of chemicals, standard solutions, and solid or liquid samples shall be cleaned up immediately using proper neutralization agents, cleaning solutions, and protective equipment. The Environmental Chemist shall be notified of any spill.

e. All spills of radioactive materials, after proper clean up shall be monitored for the appropriate type of radiation.

If the radiation exceeds background levels, further cleaning will be undertaken.

f. Tap water shall not be used for preparation of reagents, dilution of samples, final rinse of glassware, or in any other instance where the contaminants present in tap water might affect an analytical result.

g. Only reagent grade or better chemicals shall be used in the laboratory, except in the few cases where reagent grade chemicals are not commonly available.

Additionally, regular cleaning of laboratory work surfaces and equipment shall be undertaken. Monitoring of work surfaces for radioactive contaminants will be performed on a regular basis.

In some cases where an accident has occurred it may be necessary to decontaminate an individual. If the accident poses an immediate threat to health or safety of the individual, action should first be taken to counter that threat and decontamination would become secondary. If contamination such as by a strong acid, is part of the threat to health, removal of the contaminants is of primary importance. The following guidelines are designed to achieve decontamination; however, the procedures required are highly dependent on the circumstances of each individual case.

1. In the event that the contamination poses an immediate threat to the health or safety of the individual, the safety showers and/or eye wash station should be used to remove the contamination. Any affected clothing should be removed.
2. In the event of a spill or accident which is not immediately threatening but only poses a threat of the spread of contamination to other work areas, the decontamination shower outside of the laboratory may be used.
3. In the event of radioactive contamination of an individual, any affected clothing should be removed and decontaminated thoroughly or properly discarded and the individual should shower thoroughly. The individual should then be monitored by any necessary means to determine the effectiveness of decontamination and the amount, if any, of material ingested.

In many cases contamination of samples and personnel can occur in the routine operation of the laboratory through carelessness or improper handling of materials. The training received, the safety precautions in each analytical procedure, and design of the laboratory facilities should largely eliminate this type of contamination. There are, however, several rules which can aid significantly in reducing contamination.

1. Rubber gloves should be worn whenever practical and should be thoroughly washed and dried whenever they come in contact with samples or reagents.

2. The mouth should not be used to hold objects such as pens and pencils.
3. Pens and pencils should not be placed on work surfaces where samples or reagents might contaminate them.
4. Glassware shall be washed immediately prior to use.
5. Glassware shall not be moved from room to room within the laboratory except when required by the analysis.

Laboratories also have the potential to spread contamination through chemicals or fumes outside the laboratory area. The laboratory design, with proper fume hoods and a separate ventilation system, effectively controls the non-human spread of contaminants. Several regulations are required to prevent the possible spread of contamination outside of the laboratory by personnel.

1. Laboratory coats shall not be removed from the laboratory or worn outside of the laboratory.
2. Laboratory chemicals, glassware, and equipment shall not be removed from the laboratory.

F. PERSONNEL MONITORING

Due to the nature of the samples and radioactive standards present in the laboratory, it is necessary to monitor the exposure of all personnel in the laboratory. It is also necessary to monitor the potential ingestion of radioactive and other materials by laboratory personnel. Therefore, the following rules will be enforced.

1. All laboratory personnel shall wear TLD badges at all times when in the laboratory area. These badges will be read on a quarterly basis. An individual's results and an evaluation of their significance will be made available to that individual on request.
2. All laboratory personnel shall submit a urine sample for alpha and gross beta minus K-40 analysis on a quarterly basis. An individual's results and an evaluation of their significance will be made available to that individual on request.
3. In the event of a suspected ingestion of radioactive materials, the individual shall provide urine samples as required to determine the extent of the ingestion and exposure.
4. In the event of a suspected ingestion of non-radioactive material, the individual shall be monitored closely until the potential problems are eliminated.

G. PERSONNEL EVACUATION

It may, at some time, become necessary to evacuate personnel from the laboratory area due either to the release of a hazardous or irritating chemical or to a fire. In the event of the release of a chemical the following shall be done:

1. All persons shall leave the laboratory by the nearest exit closing the doors behind them.
2. The Environmental Chemist shall ensure that no individual remains in the laboratory.
3. All laboratory personnel shall proceed to a designated area and remain there until released by the Environmental Chemist.
4. If the nature of the chemical warrants, all electricity to the laboratory shall be shut off at the circuit breakers.
5. The Environmental Chemist shall initiate and direct the required clean up procedure.

In the event of a fire, the following shall be done:

1. If the fire is small and localized, the individual shall extinguish the fire using a dry chemical or a carbon dioxide fire extinguisher. Water shall not be used to fight fires.
2. If the fire is electrical, the affected equipment shall be turned off if possible. If the equipment cannot be turned off, the individual shall turn off the related circuit at the circuit breaker panel outside the laboratory. The fire may be extinguished using either a dry chemical or a carbon dioxide fire extinguisher.
3. The Environmental Chemist should be notified as soon as practical after the start of a fire.

4. Regardless of the size of the fire, all persons not directly involved with the laboratory should leave the laboratory area and proceed to a designated area. One person should notify the Environmental Chemist.

5. The Environmental Chemist shall ascertain that all persons are out of the laboratory, and shall initiate and direct fire fighting procedures.

6. Fire fighting procedures shall be continued on a limited basis even though the fire cannot be extinguished or contained with the equipment available.

7. As soon as possible after the start of a fire, the Safety Department and the Project Manager shall be notified.

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6.1.A.13.0 CARE AND HANDLING OF CALIBRATED SOURCES

There are two types of calibrated sources available in the laboratory: electroplated sources and foil-covered sources. The care and handling of each is similar but there are important differences as outlined in the following:

1. All sources should be handled only by their edges. Avoid touching the active surface.
2. Electroplated sources may be cleaned with alcohol and very light wiping with a cotton swab.
3. Foil-covered sources should not be cleaned except with a low pressure stream of air due to the extreme fragility of the foil covering.
4. All sources shall be checked for leakage on a quarterly basis using the following procedures:
   a. Gently wipe the surface of the source with a two-inch diameter filter paper moistened with alcohol. (Avoid touching the foil portion of a foil source.)
   b. The filter paper shall then be counted for the appropriate type of radiation.
   c. If the source shows leakage, it shall be placed in a sealed plastic bag. The manufacturer shall be contacted and the source returned for disposal by the manufacturer. The manufacturer's packaging and shipping instructions will be followed.
   d. If no leakage shows, the source shall be considered acceptable.

e. The source check form shall be filled out each time a leakage test is performed.

5. All sources shall be stored in the storage container supplied by the manufacturer when not in use.
6. Authorization to use a source must be obtained from either the Environmental/Radiation Manager or the Environmental Chemist.
7. Whenever a source is removed from storage, all information in the source log book must be filled out. (See sample page attached.)
8. Sources shall not be removed from the laboratory.
9. Sources shall not be carried on one's person.

SOURCE LEAKAGE TEST FORM

Source Identification

Isotope(s):

Serial No.

Activity

Date leakage test performed: ______________ By: ________________

Swipe Filter Information

Instrument Used:

Instrument Background: ______________cpm

Instrument Efficiency: ______________

Gross Sample Count: ______________cts

Sample Count Time: ______________minutes

Gross sample cpm (Gross sample counts/counts time): ______________cpm

Net Sample cpm (Gross sample cpm-background cpm): ______________cpm

Net Sample dpm (Net smaple cpm/instrument efficiency): ______________dpm

________ Source acceptable, retained in service.

Date ____________ Signature ______________

________ Source unacceptable,

Disposition of source:

Date ______________ Signature ________________

RADIOACTIVE SOURCE LOG

Source Indentification

Isotope(s):

Serial No:

Activity:

Active Diameter:

Date Used	By	Purpose of Use	Date Returned	By

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6.1.A.14.0 ORDERING RADIOACTIVE STANDARD SOLUTIONS

In accordance with its By-product Materials License, the laboratory is restricted in the quantity of radioactive materials it may have in possession. Futhermore, certain minimum information must be obtained from the supplier in order to ensure effective use of the radioisotopes. The following steps are, therefore, required in ordering Radioactive Standard Solutions:

1. Determine the amount of particular isotope currently on hand.
2. Subtract the amount on hand from the amount allowed by the license to determine the maximum amount which may be ordered.
3. The quantity desired may then be ordered only from a supplier who agrees to provide the following information with the solution in the form of a calibration certificate:
    a. A description of the solution
        1. Principal Radionuclide
        2. Mass or Volume
        3. Chemical Composition
    b. Reference time and date
    c. Activity per gram of solution (measured)
    d. Measurement method
    e. Statement of purity
    f. Decay information
    g. Estimate of error

4. Upon receipt of the solution, it should be checked to ensure proper labeling of the contents and stored in the radioisotope storage cabinet until use.
5. The certificate of calibration shall be filed in the radioactive solution file.
6. A page shall be opened in the radioisotope solution log book for recording the receipt of the solution and its dilution. (See procedure for dilution of Radioactive Standard Solution.)
7. A UNC solution number shall be assigned by the Environmental Chemist.

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6.1.A.15.0 DILUTION OF RADIOACTIVE STANDARD SOLUTIONS

A. SAFETY REQUIREMENTS

1. All dilutions shall be performed in the radiochemistry fume hood.
2. Rubber gloves shall be worn at all times.
3. No pipeting by mouth shall be performed.
4. All glassware shall be kept isolated until properly decontaminated.
5. The empty concentrated solution container shall be properly discarded as radioactive waste.
6. All necessary reagents shall be prepared and standardized in advance.

B. QUALITY CONTROL AND QUALITY ASSURANCE

1. Only reagent grade materials may be used.
2. Only high purity deionized water may be used.
3. The following forms should be complete and available before dilution:
    a. Authorization and instruction form
    b. Certificate of calibration (from supplier)
    c. Dilution worksheet
4. The above forms must be filed appropriately upon completion.

C. DILUTION OF RADIOACTIVE STANDARD SOLUTIONS

Virtually all radioactive solutions which will be used by the laboratory are supplied in concentrated forms in order to simplify shipment and enhance long term stability of the solution. For use in the laboratory, it is necessary to dilute the concentrated solution to convenient working level. The following procedure shall be followed in the dilution of Radioactive Standard Solutions:

1. No laboratory personnel may dilute a concentrated Radioactive Standard Solution without written authorization and instruction from the Environmental Chemist.
2. All glassware shall be class A and shall be thoroughly cleaned before use.
3. The storage container shall be thoroughly cleaned before use.
4. All glassware and utensils used shall be thoroughly decontaminated after use.
5. The solution used for dilution shall be compatible with the concentrated standard solution.
6. The diluted solution shall be calibrated before use in accordance with the Standard Solution Calibration procedure.
7. See the attached worksheet for calculation of the dilution required.
8. The completed worksheet shall be filed in the radioactive solution file and a copy filed in the solution log book.

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WORKSHEET-DILUTION OF RADIOACTIVE STANDARD SOLUTIONS

Standard Solution Identification

Supplier: ____________

Isotope: ____________

Stated Activity: ________ μCi = ________ pCi = ________ dpm

Stated Volume: ____________

Solution Composition: ____________

UNC Solution No.: ____________

Dilution Calculations:

Concentration Desired: pCi/ml = dpm/ml

Stated Activity in dpm: ____________

Stated Volume: ____________ ml

Stated activity in dpm/ml (^{dpm}/Volume in ml): ____________

To determine the total volume to which the solution should be diluted the following equation is solved:

$$\text{Volume required (ml)} = \frac{\text{Stated activity (dpm/ml X Stated Volume (ml)}}{\text{Concentration desired (dpm/ml)}}$$

This volume may not be a convenient volume for dilution and it should be checked to determine whether a convenient volume may be used in which case the following formula is used to calculate the diluted concentration:

$$\text{Diluted Concentration (dpm/ml)} = \frac{\text{Stated Activity (dpm/ml) X Stated Volume (ml)}}{\text{Diluted Volume (ml)}}$$

Calculated diluted concentration: ____________ dpm/ml = pCi/ml

Date ____________ Signature ____________

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6.1.A.16.0 CALIBRATION OF DILUTED RADIOACTIVE STANDARD SOLUTIONS

There are several sources of error in the preparation of diluted standard solutions which make necessary a very careful calibration of the solution after preparation. Sample dilution errors are unavoidable even with the best techniques and equipment. The calibration of the concentrated solution is rarely carried to enough figures to allow accurate calculations of the diluted activity. Complete transfer of the concentrated solution out of its container cannot be assumed. All these possible sources of errors necessitate the calibration of the diluted working solutions which shall be performed according to the following procedures. The particular procedures used will depend on the particular isotope. A listing of isotopes and procedures to be used for each is attached. At least two different procedures should be used for each isotope when possible.

A. PLANCHETTING AND CALIBRATING BY ALPHA PROPORTIONAL COUNTER

1. A minimum of three separate calibration samples should be used.
2. Clean three 2" planchettes with alcohol to remove grease and dirt.
3. Label each planchette.
4. Place the planchettes in a desiccator for a minimum of four hours or overnight.

5. Remove from desiccator and weigh. Record the weight on the calibration worksheet.
6. Using a Class A pippet, place 1.0 ml of solution on each planchette. Record the volume used on the work sheet.
7. Dry the solution under the heat lamp. (Polonium should be allowed to air dry).
8. When dry place the planchette in a desiccator for a minimum of four hours or overnight.
9. Remove from the desiccator and weigh. Record the weight on the worksheet.
10. Count each sample for a minimum of 12 hours and record the results on the work sheet.
11. Determine the instrument efficiency for the weight of sample on the planchette from the instrument alpha attenuation graph. Record on the worksheet.
12. Calculate the activity in accordance with the instructions on the worksheet.
13. Complete the worksheet and file it in the diluted radioactive solution calibration file.

    Note: Background count time should be the same as sample count time.

B. PLANCHETTING AND CALIBRATING BY LOW BACKGROUND BETA COUNTER

1. A minimum of three separate samples should be used.
2. Clean three 2" planchettes with alcohol to remove grease and dirt.
3. Label each planchette.
4. Place the planchette in a desiccator for a minimum of four hours or overnight.
5. Remove from desiccator and weigh. Record the weight on the calibration worksheet.
6. Using a Class A pipet, place 1.0 ml of solution on each planchette. Record the volume used on the worksheet.
7. When dry, place the planchettes in a desiccator for a minimum of four hours or overnight.
8. Remove from the desiccator and weigh. Record the weight on the worksheet.
9. Count each sample for a minimum of 12 hours and record the results on the worksheet.
10. Determine the instrument efficiency for the weight of sample on the planchette from the instrument beta attenuation graph. Record on the worksheet.
11. Compute the activity in accordance with the instructions on the worksheet.

12. Complete the worksheet and file it in the diluted radioactive solutions calibration file.

    Note: Background count time should be the same as sample count time.

C. ELECTROPLATING AND CALIBRATION BY ALPHA SPECTROMETER

1. A minimum of three separate samples should be used.
2. Label three 1" stainless steel discs using the carbide pencil.
3. Assemble the discs into the electroplating cells.
4. Clean thoroughly with acetone and cotton swab.
5. Add 1.0 ml of solution to each plating cell.
6. Add plating solution to the cell to fill. The following plating solutions should be used.
    a. For Thorium - 1% W/V Ammonium Oxalate in deionized water with 1 ml of 4N Hydrochloric acid. Plate at 210 mA.
    b. For Uranium - 1:1 solution of 1N HNO_3 and 1N NH_4OH. Plate at 300 [illegible]
7. The samples should be electroplated for a minimum of six hours. Additional electroplating solution should be added as required.
8. When electroplating is complete add 1 drop phenolphthalein solution and 1% NH_4OH until a red color appears in the electroplating cell.

9. Turn off current to the cell and remove from electroplating unit.
10. Save the electroplating solution from each sample in a labeled beaker.
11. Disassemble the cell and rinse each disc with alcohol and allow to dry.
12. When dry, heat each disc in a burner until red. Allow to cool.
13. Count each sample a minimum of 12 hours in the alpha spectrometer and attach the results to the worksheet.
14. Dry the electroplating solution from each cell on a hotplate.
15. Cool the beakers and add 10 ml of the appropriate electroplating solution and police the beaker thoroughly.
16. Add the solution to prepared electroplating cells.
17. Continue to add electroplating solution to the beakers, police, and add to the cells until the cells are full.
18. Electroplate for six hours at the appropriate amperage.
19. Prepare the electroplated discs and count for the same length of time as the first set of discs.
20. Attach the results to the worksheet.
21. If a significant number of counts appear in the counting of the second set of discs, repeat the procedure with a third set of discs.

22. Attach all results to the worksheet and follow the instructions to complete the solution calibration.

    Note: Background count time should be the same as sample count time.

D. <u>CALIBRATION BY RADON DE-EMANATION (for Ra 226)</u>

1. This calibration should be performed with a minimum of three samples.
2. Prepare a radon counting system in accordance with the Ra 226 procedure for each sample.
3. In a clean beaker containing 5 ml of 3N nitric acid, place 1.0 ml of Ra 226 solution using a class A pipet.
4. Transfer the sample to a radon bubbler using a minimum of 7 ml of distilled water.
5. Proceed as in the Ra 226 analysis procedure being sure to record all the times and dates required on the calibration worksheet.
6. When the ingrowth period is complete and the de-emanation is complete, count the Lucas Cell for a minimum of 12 hours and record the results on the calibrations worksheet.
7. Follow the instructions on the work sheet to complete the calibration.

E. CALIBRATION BY DEPOSITION FROM ACID SOLUTION (Polonium and Lead)

1. This procedure should be performed with a minimum of three samples.
2. Prepare nickel discs for each sample by labeling each using the carbide pencil and spraying the labeled side with shellac.
3. Mount the disc in an electrode holder.
4. Polish the exposed surface with 0.1 micron gamma alumina polishing cloth.
5. Prime the disc by suspending in 0.5 HCl for five minutes.
6. Add 1.0 ml of the solution to 50 ml of 3.0N HNO_3 in a 125 ml Erlenmeyer Flask.
7. Place a magnetic stirring bar in the flask and place on a magnetic stirring plate.
8. Add 2 ml of 40% W/V citric acid and 2 ml of hydroxylamine hydrochloride to the sample solution.
9. Suspend the nickel disc in the solution and allow the sample to plate for 1 hour with the stirrer running at a speed low enough to prevent splashing.
10. Remove the disc from solution and rinse with deionized water. Allow to dry.
11. Count the sample for a minimum of 12 hours in the alpha spectrometer and attach the results to the calibration worksheet.

12. Follow the instruction on the calibration worksheet to complete the calibration.

Note: In some instances more specialized calibration procedures may have to be used to correct for ingrowth. As these instances arise, procedures shall be written and approved before calibration begins.

F. RECOMMENDED CALIBRATION PROCEDURES

Standard Solution	Calibration Procedures
Th-228	A, C.
Th-229	A, C.
Th-230	A, C.
Th-232	A, C.
Nat U	A, C.
Depleted U	A, C.
U-232	A, C.
Po-208	A, E.
Po-210	A, E.
Ra-226	A^1,D.
Ra-228	B
Pb-210	A^2, B, E.

1. Ra-226 calibration by alpha proportional counter requires additional steps which are not listed in the procedure.
2. Pb-210 calibration may be performed by analysis of the Po-210 which will be at equililrium if the solution is greater than 30 days old.

Calculations: Activity and counting error at the 95% confidence level.

The following equation will be used to calculate the activity of each sample.

$$\text{dpm/ml} = \frac{S - B}{EVe^{-\lambda t/T_{\frac{1}{2}}}} \pm \frac{1.96\sqrt{S/T_S + B/T_B}}{EVe^{-\lambda t/T_{\frac{1}{2}}}}$$

Where

S = Sample gross cpm

B = background cpm

V = sample aliquot in ml

T_S = sample count time

T_B = background count time

E = instrument efficiency for the sample of interest

$e^{-\lambda t/T_{\frac{1}{2}}}$ = radioactive decay equation

t = time from the midtime of counting of the first sample to the midtime of counting of the sample of interest.

$T_{\frac{1}{2}}$ = half life of the isotope of interest

λ = constant = log 2.

Note: The expression $e^{-\lambda t/T_{\frac{1}{2}}}$ will only contribute significantly when the half life of the isotope is short, as in the case of Po-208.

UNC Lab Solution No.: ______________ ______________dpm/ml ± ______________dpm/ml

Calculations:

G. SOLUTION CALIBRATION WORKSHEET FOR ALPHA AND LOW BACKGROUND BETA PROPORTIONAL COUNTERS

UNC Lab Solution No.: ____________

Isotope(s): ________

Calculated Activity: __________

1. Amount used for each sample: ________ ml
2. Calculation of sample dry weight:

Sample No. ____ Sample No. ____ Sample No. ____

Planchette + Sample Weight

- Planchette Tare Weight - ______ - ______ - ______

Sample Weight

3. Counting Data:

Instrument Used: __________

Instrument Background: ______ cts/ ______ minutes = ______ cpm

Gross Counts; Sample No.: ______ ______ cts/ ______ minutes = ______ cpm

Gross Counts; Sample No.: ______ ______ cts/ ______ minutes = ______ cpm

Gross Counts; Sample No.: ______ ______ cts/ ______ minutes = ______ cpm

Instrument Efficiency; Sample No.: ______ ______

Instrument Efficiency; Sample No.: ______ ______

Instrument Efficiency; Sample No.: ______ ______

Midtime of Counting; Sample No.: ______ ______ hrs. ______ date

Midtime of Counting; Sample No.: ______ ______ hrs. ______ date

Midtime of Counting; Sample No.: ______ ______ hrs. ______ date

Midtime of counting of the first sample is arbitrarily t = 0.

Sample No. ____________ ____________ dpm/ml ± ____________ dpm/ml

Calculation:

Sample No. ____________ ____________ dpm/ml ± ____________ dpm/ml

Calculation:

4. Calculation of solution activity from the three samples

The best determination of sample activity uses the following equation:

$$x_{Best} = \frac{\sum_i x_i/\sigma_i^2}{\Sigma i/\sigma_i^2}$$

Where x_{Best} = the average value of the solution concentration
x_i = the individual sample concentration
σ_i = the standard deviation of the individual value.

The best determination of the standard deviation uses the following equation:

$$\sigma_{Best} = (\sum_i 1/\sigma i^2)^{-\frac{1}{2}}$$

Calculation:

UNC Lab Solution No.: ____________

Calibrated Activity = ____________ dpm/ml ± ____________ dpm/ml

Date and time of calibration ____________

Signature ____________ Date ____________

Channel of peak of interest: Start channel ____________

Stop channel ____________

Detector Background over peak of interest: ____________ ct/ ____________ minutes = ____________ cpm.

Sample count over peak of interest: ____________ cts/ ____________ minutes = ____________ cpm.

Midtime of counting, UNC Lab Solution No.: ____________ hrs. ____________ date

Midtime of counting, UNC Lab Solution No.: ____________ hrs. ____________ date

Midtime of counting, UNC Lab Solution No.: ____________ hrs. ____________ date

The midtime of counting of the first sample is arbitrarily $t = o$.

3. Calculations, activity and counting error at 95% confidence level.

The following equation will be used to calculate the activity of each sample:

$$\text{dpm} = \frac{S - B}{EVe^{-\lambda t/T_{\frac{1}{2}}}} \pm \frac{1.96\sqrt{S/T + B/T}}{EVe^{-\lambda t/T_{\frac{1}{2}}}}$$

where

S = sample gross cpm

B = background cpm

V = sample aliquot in ml

T_S = sample count time

T_B = background count time in minutes

$e^{-\lambda t/T_{\frac{1}{2}}}$ = radioactive decay equation

t = time from the midtime of counting of the first sample to the midtime of counting of the sample of interest

$T_{\frac{1}{2}}$ = half life of the istope of interest.

λ = a constant = $\log_{10}2$

Note: The expression $e^{-\lambda t/T_{\frac{1}{2}}}$ will only contribute significantly when the half life of the isotope is short, as in the case of Po 208.

UNC Sample Solution No.: ____________ ________________dpm/ml ±

____________ dpm/ml

Calculation:

H. SOLUTION CALIBRATION WORKSHEET

UNC Lab Solution No.: ____________________

Isotope(s): ____________________

Calibrated Activity: ____________________

Note: a separate worksheet shall be used for each istope of a multi-isotope solution.

1. ml used for each sample: __________ml

2. Counting Data:

Instrument used: ______________

Sample No.: ______________

Detector Used: ______________

Detector Efficiency: ______________

Channels of peak of interest: start channel ________ stop channel ________

Detector Background over peak of interest: ________cts/________minutes =

______________cpm.

Sample counts over peak of interest: ________cts/________minutes ________cpm

Sample No.: ______________

Detector Used: ______________

Detector Efficiency: ______________

Channels of peak of interest: start channel________stop channel ________

Detector background over peak of interest: ________cts ________minutes =

______________cpm.

Sample counts over peak of interest: ________cts________minutes =

__________cpm

Sample No.: ______________

Detector Used: ______________

Detector Efficiency: ______________

Sample No. ____________________ ______________dpm/ml ± ______________dpm/ml

Calculation:

5. Calculation of solution activity from the three samples

The best determination of sample activity use the following formula:

$$x_{Best} = \frac{\sum_i x_i/\sigma_i^2}{\sum 1/\sigma_i^2}$$

where x_{Best} = the average value of the solution concentration
x_i = the individual sample concentration value
σ_i = the standard deviation of the individual value

The best determination of the standard deviation uses the following formula:

$$\sigma_{Best} = (\Sigma_i\ 1/\sigma_i^2)^{-1/2}$$

Calculation:

UNC Lab Solution No.: ____________________
Calibrated activity = ____________________dpm/ml ± ____________________dpm/ml

Date and time of calibration ______________________________

Signature: ______________________________ Date ______________________________

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6.1.A.17.0 STORAGE, USE, AND HANDLING OF RADIOACTIVE STANDARD SOLUTIONS

The radioactive solutions present in the laboratory will be generally of two types: the concentrated solution as received from the supplier, and the diluted solutions for laboratory use. The following rules shall be adhered to at all times:

1. The concentrated solutions shall be stored in a locked cabinet in their original containers until their specific use.
2. When needed, the concentrated solutions will be diluted to working concentrations by the Environmental Chemist in accordance with the Radioactive Solution Dilution Procedure.
3. Laboratory personnel shall not dilute the concentrated solutions without specific written authorization and instruction from the Environmental Chemist. (See attachment)
4. Diluted solutions shall be stored in a locked cabinet in Teflon or polyethylene bottles having a maximum size of one liter.
5. Authorization to use the solutions must be obtained from the Environmental/Radiation Department Manager or the Environmental Chemist.
6. The person using a solution must fill out the Radioactive Solution log book each time a solution is used. (See attached sheet)
7. The solution must be returned to storage immediately after use.
8. Each diluted soluton will have a labeled pipet of the proper size which shall be used exclusively for dispensing of that solution.

RADIOACTIVE SOLUTION LOG

UNC Solution No.: ____________________

Isotope(s): ____________________

Calibrated activity: ____________________

Date of calibration: ____________________

Date Used	By	Purpose	Volume Before Use	Volume Used	Volume Returned

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AUTHORIZATION TO DILUTE A CONCENTRATED RADIOACTIVE SOLUTION

______________________ is specifically authorized to dilute the below indentified Radioactive Solution in accordance with the instructions given below and in accordance with the Radioactive Solution Dilution Procedure Manual.

Date ________ Signature ____________________

Solution Identifcation:

Supplier: ____________________

Isotope: ____________________

Stated Activity: ____________________

Stated Voume: ____________________

Serial of Lot No. (if any): ____________

Solution composition: ____________

UNC Solution No.: ____________

Dilution Instructions:

Diluted Volume (Volume of Concentrated sample & diluting liquid):

____

Dilute with: __ ____________

Store in: ____________________

Labeling of storage containers:

Isotope: ____________

Activity per ml (calculated): ____________________

Activity per ml (from calibration): ____________________

Date of calculation: ____________________

UNC Solution No.: ____________________

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6.1.A.18.0 PREPARATION AND STANDARDIZATION OF REAGENTS

The analytical procedures used in the laboratory require the use of diluted reagents of known concentrations. These reagents must be prepared from concentrated stock reagents. The specific reagents used in each analytical proceudre will be listed in the procedure manual. however, it is helpful to know the concentration units employed and how to prepare and standardize certain types of reagents.

A. CONCENTRATION UNITS

1. Percent by weight - The percentage given refers to the solute (the portion being dissolved). Example: 5 percent aqueous NaCl solution contains 5 percent, by weight, sodium chloride, the remaining 95 percent being water.
2. Percent by volume - Used to express the concentration of a solution of two liquids.
3. Molarity (M) - Moles of solute per liter of solution.
4. Normality (N) - The number of equivalent weights of solute per liter of solution.
5. Additive volumes - (a + b), the first number , a, refers to the volume of concentrated reagent; the second number, b, refers to the volume of distilled water required for dilution. Example: "1 + 9HCL" denotes 1 volume of concentrated HCl to 9 volumes

of water. Additive volumes are sometimes expressed as a: b rather than a + b.

B. PREPARATION OF REAGENTS

1. All reagents shall be prepared using reagent grade chemicals and high purity water.
2. If a solution of exact normality is to be prepared by dissolving a weighed amount of a primary standard or by diluting a stronger solution, bring it up to exact volume using a Class A volumetric flask.
3. If the concentration of a solution does not need to be exact, it is easier to mix the concentrated solution or the solid with measured amounts of water using graduated cylinders.
4. When preparing acid or alkali solutions add the acid or alkali to the water, with stirring, in a vessel which can withstand thermal shock. Dilute to the final volume after cooling to room temperature.

C. STANDARDIZATION OF REAGENTS

1. For the dilution of most solid material, adequate standardization is obtained by careful weighing of the solid on an analytical balance and the use of Class A volumetric glassware in

in the dilution.

2. For the dilution of acids and bases, whose concentrated forms are of variable concentration a more complex procedure is required. A dilute acid and dilute base whose concentrations are well defined must first be prepared. The unknown acid may then be standardized by measurement of its ability to neutralize the known base and the unknown base may be standardized by its ability to neutralize the known acid. The procedure is as follows.
   a. Prepare a quantity of Certified Potassium Hydrogen Phthalate Acidimetric Standard by lightly crushing and heating in an oven at 120°C for 1 hour. Allow to cool in a desiccator.
   b. Weigh out 20.423 grams of the Potassium Hydrogen Phthalate on the analytical balance and record the weight to four decimal places. The weight need not be exactly 20.4230 grams.
   c. Dilute the acid to 1.0 liter using a Class A volumetric flask and high purity water.
   d. Mix the solution well and transfer to a clean glass bottle.
   e. Calculate the Normality of the solution using the following formula:

$$N = \frac{\text{grams used}}{204.23 \text{ g/equivalent weight X 1 liter}}$$

In this case the Normality will be approximately 0.1N

f. Label the bottle with the contents and the concentration.

g. This solution represents the primary standard for determining the concentration of basic solutions. The determination of the concentration of a base is as follows:

1. Prepare the basic solution as instructed in the procedure manual. This will result in a solution of the nominal concentration specified.
2. Take a quantitiy of this solution that would be neutralized by approximately 20 ml of the Potassium Hydrogen Phthalate Solution and place in a clean beaker.
3. Standardize a pH meter and place the electrode in the beaker.
4. Fill a fifty milliliter burette with the Potassium Hydrogen Phthalate Solution. (KHP)
5. Titrate the base to the pH 7.0 endpoint and record the volume of KHP solution used.
6. Calculate the concentration of the base by the following formula:

$$N_{Base} = \frac{N_{KHP} \times V_{KHP}}{V_{Base}}$$

7. Label the base appropriately.

h. To standardize an acid solution a solution of approximately 0.1N Sodium Hydroxide is prepared and standardized as above. This base is now used to standardize the acid solution.

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6.1.A.19.0 EQUIPMENT MALFUNCTION PROCEDURE

This procedure and attached form are for the reporting of malfunction and necessary repairs in laboratory equipment such as hotplates and other apparatus which are used in the performance of analyses but which do not directly generate analytical data. For those instruments which generate data the instrument malfunction procedure should be followed.

If an apparatus malfunctions or breaks down it shall immediately be removed from service. A tag shall be attached, indicating that the apparatus has been removed from service and may not be used.

The attached form must then be filled out and submitted to the Environmental Chemist. The corrective actions taken must be entered on the form along with the result of those actions. The apparatus must then be checked for proper function. If function is acceptable, the apparatus may be returned to service.

Copies of the malfunction report shall be filed in the equipment malfunction file.

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EQUIPMENT MALFUNCTION REPORT

Equipment ____________

Serial No. or Morton Ranch No. ____________

Location of Equipment ____________

Instrument Removed from Service and Tagged Date ____________

Signature ____________

Description of Malfunction or required repair work:

Date Reported: ____________

Signature: ____________

Corrective Action Taken:

Date ____________

Signature ____________

Systems Checks Performed:

Date ____________

Signature ____________

Date Returned to Service ____________ Signature ____________

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6.1.A.20.0 INSTRUMENT MALFUNCTION PROCEDURE

This procedure is applicable to those laboratory instruments which directly generate analytical data and whose proper function can be tested by defined system checks.

In the event of a malfunction of a laboratory instrument, it shall be removed from service. A tag shall be attached indicating that the instrument has been removed from service and may not be used.

The attached form must then be filled out and submitted to the Environmental Chemist who will initiate corrective action. Do not attempt any corrective action until instructed to do so by the Environmental Chemist.

The corrective action shall be recorded on the form. If corrective action is taken by a manufacturer service representative, a copy of his repair report shall be attached to the malfunction report form.

After corrective action has been taken, relevant system checks must be performed before the instrument can be returned to service.

Copies of the systems check should be filed with the malfunction report in the instrument malfunction file and copies of the malfunction report should be filed with each systems check in the files for the instrument.

INSTRUMENT MALFUNCTION REPORT

Instrument ______________________________

Serial No. or Morton Ranch No. ______________________________

Location of Instrument (Room No.) ______________________________

Instrument Removed from Service and Tagged date ______________________

Signature ______________________________

Description of malfuntion:

Date Reported: ______________________

Signature ______________________

Corrective Action Taken:

Date: ______________________

Signature: ______________________

Systems Checks Required:

1. ______________________ Date ______________ Signature ______________
2. ______________________ Date ______________ Signature ______________
3. ______________________ Date ______________ Signature ______________
4. ______________________ Date ______________ Signature ______________
5. ______________________ Date ______________ Signature ______________

Date Insturment Returned to Service ______________ Signature ______________

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6.1.A.21.0 SAMPLE PREPARATION

A. PUPROSE

To provide instructions for preparing samples for either instrumental or chemical analysis.

B. SCOPE

This procedure details preparation methods for all sample types requiring any treatment. Any other sample type is analyzed as received.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the resulting data.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results or problems to the Environmental Chemist.

D. RECORD KEEPING

The analyst must enter in the sample preparation log book all weighings and must initial his entries to indicate that the procedure from the manual has been followed without deviation. The analyst must also enter all required information on the sample worksheet which shall then be attached to the anaylsis request sheet.

E. SAFETY

Safety glasses are required. The jaw crusher and sample grinder should be in proper working order. All loose clothing and jewelry (such as ties or necklaces) should be removed or restrained to prevent their catching in the equipment. Long hair should be tied back. Do not place hands in the grinders or open the grinders while in operation.

F. QUALITY CONTROL

The balance shall be checked before use and the proper portion of the worksheet filled out. All utensils, containers, and equipment shall be thoroughly cleaned before and after use. Oven temperatures shall be recorded in the log book.

G. SAMPLE PREPARATION

1. Soil, Sediment, and Rock
   a. Place the sample in a tared stainless steel pan. Weigh and record the sample Wet Weight.
   b. Place the pan in a drying oven for roughly 16 hours at approximately 110°C.
   c. Turn oven off and allow to cool. Remove the sample and check to see if it is completely dried. If it is dry, allow the

sample to cool to room temperature. If the sample is not dry return it to the oven and continue heating until dry; then cool.

d. Weigh and record the Dry Weight.

e. Grind the sample to approximately 6.5 mesh. Weigh and record the Ground Dry Weight.

f. Transfer the sample to a crucible or crucibles and place in a 500°C muffle furnace for roughly 16 hours.

g. Reduce the furnace temperature to roughly 100°C before attempting to remove the sample.

h. Remove the sample and allow to cool to room temperature. Weigh and record the Ash Weight.

2. Vegetation

   a. Place the sample in a tared stainless steel pan. Weigh and record the sample Wet Weight.

   b. Place the pan in a gravity convection drying oven for roughly 16 hours at approximately 110°C.

   c. Turn oven off and allow to cool. Remove the sample and check to see if it is completely dried. If it is dry, allow the sample to cool to room temperature. If the sample is not dry, return it to the oven and continue heating until dry; then cool.

d. Weigh and record the Dry Weight.

e. Grind the sample to approximately 6.5 mesh in the Wiley Mill or equivalent instrument. Weigh and record the Ground Dry Weight.

f. Transfer the sample to a crucible or crucibles and place in a 500°C muffle furnace for approximately 16 hours, until completely ashed.

g. Reduce the furnace temperature roughly to 100°C before attempting to remove the sample.

h. Remove the sample and allow to cool to room temperature. Weigh and record the Ash Weight.

i. Proceed to the desired analysis.

3. Water for Dissolved Analysis

   a. Mount a 0.45 micron filter in the filtration apparatus.

   b. Apply vacuum to the system and fill with the water sample. As the liquid level falls, add more sample, until the filtration rate slows significantly.

   c. At this point the filter may need to be changed. If so, change the filter and continue.

   d. Save the filters in a labled dish. They represent the suspended portion of the sample.

e. Transfer the water to a clean, dry container. This represents the dissolved portion of the sample.

f. Proceed to the desired analyses.

H. RELATED OPERATIONS MANUALS

1. Jaw Crusher
2. Pulverizer
3. Cutting Mill
4. Sieve Shaker
5. Forced Air Drying Oven
6. Gravity Convection Drying Oven
7. Muffle Furnace
8. Filtering Equipment

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SAMPLE PREPARATION WORKSHEET

Laboratory Sample Preparation: ____________________

Balance Check:

Balance Used: ____________

Known Weight Used: ____________ g

Balance Reading: ____________ g

Sample Wet Weight: ____________ g

Sample Dry Weight: ____________ g

Ground Dry Weight: ____________ g

Ash Weight: ____________ g

Comments:

Analyst: ____________________ Date: ____________________

I. CALIBRATION OF LUCAS CELLS

A. PURPOSE

To provide an analytical procedure for the calibration of Lucas Cells.

B. SCOPE

Applicable to all Lucas Cells.

C. RESPONSIBILITY

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. REAGENTS

1. Nitric Acid - concentrated, reagent
2. Ra 226 - Standard solution 100 pCi/ml. Traceable to National Bureau of Standards.

E. PROCEDURE

1. Add 1 ml of the 100 pCi/ml Ra 226 standard to an appropriate sample size for the type of sample to be analyzed and proceed with the Ra 226 method for that type of sample.
2. After counting the radon, calculate the scintillation cell factor.

D. CALCULATION

1. Cell Factors = $\frac{\text{cpm at equilibrium}}{\text{pCi of Standard}}$

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6.1.A.22.0 GROSS ALPHA AND GROSS BETA DETERMINATION

A. PURPOSE

To provide an analytical method for the determination of gross alpha and gross beta activity in environmental samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine environmental samples.

C. RESPONSIBILITIES

It is the reponsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the results.

It is the responsibiliby of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

A minimum of one duplicate, one spike and one blank are to be analyzed with each 10 samples in a sample run. If a sample run consists of fewer than ten samples, one duplicate, one spike, and one blank shall be analyzed.

F. PROCEDURE

1. Reagents
    a. None
2. Determination for Water
    a. Measure the conductivity of the sample.
    b. Estimate the solids content of the water. For most samples the conductivity times 0.8 will estimate the approximate solids content in milligrams of solids per liter of solution.
    c. Determine the volume of water that contains less than 150 mg of solids. The selected volume is never to be greater than 500 ml.
    d. If the volume containing 150 mg of solids is very small, an aliquot of the sample should be diluted sufficiently to give 250-500 ml volume for analysis.
    e. Measure the selected volume into a beaker.
    f. Record the volume of the sample taken and dilution information. Evaporate the sample to near dryness.

g. With deionized water, transfer the concentrate to a tared planchette. Using a rubber policeman, wash the beaker walls with a few drops of deionized water.

h. Combine the washings with the concentrate in the planchette and evaporate to near dryness.

i. Allow to cool and place in a desiccator for a minimum of 2 hours. Weigh the planchette.

j. Store the sample in a desiccator until it is to be counted.

k. Count the sample for alpha and/or beta in either a proportional counter (alpha) or a low-background counter (beta or alpha/beta).

l. Calculations are performed as described in the calculations section of the manual.

3. Solid Sample Determination

   a. After the sample has been dried and ashed in a muffle furnace, a 1 gram aliquot shall be weighed into a planchette.

   b. Distribute the ash evenly and spray lightly with Krylon.

   c. Store the sample in a desiccator until it is to be counted.

   d. Count the sample on a low-background counter for alpha and/or beta activity.

   e. Calculations are performed as described in the calculations section of the manual.

G. WATER CALCULATIONS

Activity and error at the 95% confidence level.

Gross alpha or gross beta (pCi/l) $= \frac{A - B}{(E)(V_s)\ 2.22} \pm \frac{1.96\sqrt{A/T_A + B/T_B}}{(E)(V_s)\ 2.22}$

where

A = Alpha or beta count rate (cpm)

B = Background count rate (cpm)

T_A = Sample count time

T_B = Background count time

E = Counting efficiency for alpha or beta corrected for self absorption

V_s = Volume of sample in liters.

Lower Limit of Detection

$$\text{Lower Limit of Detection} = \frac{4.66\sqrt{B/T_B}}{2.22\ EV_s}$$

H. SOLID SAMPLE CALCULATIONS

Activity and error at the 95% confidence level.

Gross alpha or gross beta (pCi/l) $= \frac{(A - B)}{(C)(D)(E)2.22} \pm \frac{1.96\sqrt{A/T_A + B/T_B}}{(C)(D)(E)\ 2.22}$

Where

A = alpha or beta count rate (cpm)

B = background count rate (cpm)

T_A = Sample count time

T_B = Background count time

C = weight of ash in planchette

D = dry weight (total) divided by ash weight (total)

E = efficiency corrected for self absorption

Lower Limit of Detection

$$\text{Lower Limit of Detection (pCi)} = \frac{4.66\sqrt{B/T_B}}{(C)(D)(E)\ 2.22}$$

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GROSS ALPHA AND GROSS BETA WORKSHEET FOR WATER

Laboratory Sample No.: _______________

Sample Type: _______________

Estimation of Aliquot Volume:

Conductivity = _______________ X 0.8 = _______________mg/liter

_______________mg/liter ÷ 150 mg = _______________liter = calculated aliquot

Actual aliquot used (V_s) = _______________liter. Diluted to _______________liter.

Sample Dry Weight:

Planchette plus Sample Weight _______________ gram

\- Planchette Tare Weight _______________ gram

Sample Weight _______________ gram = _______________mg

Counting Date:

Instrument Used: _______________

Instrument Background (B): _______________cpm alpha, _______________cpm beta

Background counting time (T_B): _______________minutes

Gross Sample Activity (A): _______________cpm alpha, _______________cpm beta

Sample counting time (T_A): _______________ minutes

Instrument efficiency (E): _______________alpha, _______________beta

Calculation:

$$\text{Gross alpha or beta} = \frac{A - B}{(E)(V_S)2.22} \pm \frac{1.96\sqrt{A/T_A + B/T_B}}{(E)(V_s)2.22}$$

Calculation:

alpha =

beta =

$$\text{Lower Limit of Detection} = \frac{4.66\sqrt{B/T_B}}{2.22(E)(V_s)}$$

LLD (alpha) =

LLD (beta) =

Result: Gross alpha = ____________ pCi/l ± ____________ pCi/l, LLD = ________ pCi/l

Gross beta = ____________ pCi/l ± ____________ pCi/l, LLD = ________ pCi/l

Analyst ______________________ Date ________________

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GROSS ALPHA AND GROSS BETA WORKSHEET FOR SOLIDS

Laboratory Sample No.: ____________

Sample Type: ____________

Total Dry Weight: ____________ grams

Total Ash Weight: ____________ grams

Aliquot Ash Weight (C): ____________ grams

Dry Weight/Ash Weight Ratio: ____________

Counting Data:

Instrument Used: ____________

Instrument background (B): ____________ cpm alpha, ____________ cpm beta

Background counting time (T_B): ____________ minutes

Gross Sample activity (A): ____________ cpm alpha, ____________ cpm beta

Sample counting time (T_A): ____________ minutes

Instrument efficiency (E): ____________ alpha, ____________ beta

Calculations:

$$\text{Gross alpha or beta (pCi)} = \frac{A - B}{(C)(D)(E)2.22} \pm \frac{1.96\sqrt{A/T_A + B/T_B}}{(C)(D)(E)2.22}$$

alpha =

beta =

$$\text{Lower Limit of Detection (pCi)} = \frac{4.66\sqrt{B/T_B}}{2.22\,(C)(D)(E)}$$

LLD alpha (pCi)

LLD beta (pCi)

Result

Gross alpha = ______________pCi/g ± ______________pCi/g,LLD = __________ pCi/g

Gross beta = ______________pCi/g ± ______________pCi/g,LLD = __________pCi/g

Analyst____________________ Date ________________________

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6.1.A.23.0 PROCEDURE FOR DETERMINING ALPHA AND BETA ATTENUATION AS A FUNCTION OF RESIDUE

A. PURPOSE

To provide a procedure to correct for the effects of sample residue on the instrument counting efficiency.

B. SCOPE

The efficiency curve produced may be applied to all routine environmental samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to perform the procedure without deviation and to generate the necessary graphs.

D. REFERENCE

L.L. Thatcher, and V. J. Janzer, U. S. G. S., and K. W. Edwards, Colorado School of Mines. 1977 Techniques of Water - Resources Investigations of the United States Geological Survey, Chapter A5 Methods for Determination of Radioactive Substances in Water and Fluvial Sediments. U. S. Government Printing Office, Washington D. C. 20402. Stock Number 024-001-02928-6.

E. SAFETY

All radioactive solutions to be handled wearing rubber gloves.

Work to be performed in a properly operating radioisotope fume hood

F. QUALITY CONTROL

Class A volumetric equipment to be used. All measurments of weight to be performed on an analytical balance and recorded to four decimal places.

G. PROCEDURE

1. Reagents
   - a. Calibration Solution A: Dissovlve 0.284 g $MgSO_4 \cdot 7H_2O$, 0.070 g NaCl, 0.026g $CaSO_4 \cdot 2H_2O$, 0.109 g $NaHCO_3$, and 0.245 g $CaCO_3$ in deionized water. Dilute to 2.00 liters.
   - b. Calibration Solution B: Dissolve 1.350 g $MgSO_4 \cdot 7H_2O$; 3.510 g NaCl, 1.550 g $CaSO_4 \cdot 2H_2O$, 0.508 g $MgCl_2 \cdot 6H_2O$ and 0.300 $CaCl_3$ in deionized water. Dilute to 2.00 liters.
   - c. Lead 210 standard solution - approximately 500 pCi/ml and acidified to approximately 1N with hydrochlori [illegible].
   - d. Uranium standard solution - Dissolve 0.177 g of [illegible] $_2H_3O_2)_2 \cdot 2H_2O$ in approximately 500 ml of deionized water. Add 15 ml of concentrated HNO_3 amd dilute to 1000 ml in a volumetric flask.

2. Preparation of Beta Calibration Curve

   a. Add the following amounts of calibration solutions A & B to 100 ml Teflon beakers.

Beaker No.	A(ml)	B(ml)	Approx. Residue (g)
1	25		10
2		5	20
3	80		30
4		10	40
5	140		50
6		20	60
7	200		70
8		25	80
9	250		90
10		30	100
11	300		110
12		35	120
13	350		130
14		40	140
15	400		150
16	-	-	0
17	-	-	0
18	-	-	0
19	-	-	0

b. To each beaker add 1.00 ml of lead standard solution. To beakers 18 and 19 add 5 drops concentrated NH_4OH.

c. Evaporate all solutions to dryness. When beakers 18 and 19 are dry, raise heat to 350° to volatilize NH_4Cl.

d. Quantitatively transfer the residues in the beakers to tared planchettes using rubber policemen and a minimum of deionized water.

e. Dry the planchettes under infrared heat lamps. Police down the beaker with a small additional amounts of de-ionized water and transfer to the planchettes.

f. Disperse the final liquid slurry in the planchettes as uniformly as possible, disrupting large aggregates. Evaporate to dryness.

g. Place the planchettes in a desiccator overnight. Weigh the planchette to determine the weight of residue.

h. Count the planchettes in a low background beta counter.

i. Proceed to the calculation portion of the procedure.

3. Preparation of Alpha Calibration Curve

   a. The preparation is identical to the beta calibration curve except that the standard uranium solution is used instead of the lead solution.

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H. CALCULATIONS

1. Knowing the standardized activity of the initial solution, calculate the apparent instrument efficiency for each planchette.
2. Graph, on linear paper, the instrument efficiency versus the weight of residue.

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6.1.A.24.0 FLUOROMETRIC DETERMINATION OF URANIUM IN ENVIRONMENTAL SAMPLES

A. PURPOSE

To provide an analytical method for total uranium in environmental samples where the determination of individual isotope concentration is not required.

B. SCOPE

The procedure applies to the analysis of all routine environmental sample types. Analysis must be limited to low levels of uranium. Higher activitity levels will require dilution.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist.

D. RECORD KEEPING

All required information shall be entered in the laboratory notebook and on the Uranium Analysis Worksheet which shall be attached to the sample analysis request form when complete.

E. SAFETY

Safety glasses required. Rubber gloves to be worn when handling any reagents. Didymium glassblowers goggles to be worn when the fusion unit is in operation. Diethyl ether and ethyl acetate to be used in a properly operating fume hood away from open flame or electrical sparks. Hydrofluoric acid to be used only when wearing rubber gloves, rubber apron, safety glasses, and full face shield.

F. QUALITY CONTROL

One spike sample and one duplicate to be analyzed with each ten samples. If fewer than ten samples are to be analyzed in a run, one spike sample and one duplicate shall be run.

All platinum fusion dishes should be treated equally in order to maintain uniformity. Therefore, all dishes in the set will be used for each fusion operation.

All platinum fusion dishes shall be checked for cleanliness between each sample run by fusing them with flux. These blank flux pellets are read in the fluorimeter. If the results show excessive background the dishes are cleaned and rechecked. Reagent grade chemicals shall be used for making all reagents.

G. PROCEDURE

1. Reagents:

   a. Aluminum Nitrate (Saturated) - Add aproximately 625 grams of reagent grade $Al(NO_3)_3 \cdot 9H_2O$ to 250 milliliters of 7% v/v nitric acid. Heat gently on a hotplate for one hour, stirring frequently. Cool to room temperature, allow the crystals to settle, and decant. Prepare two fluorimeter blanks by extracting 8 ml of the above reagent with 10 ml of ethyl acetate. If the blanks yield acceptable background readings on the fluorimeter, then the reagent may be used without further cleaning. Higher readings require that the saturated reagent be extracted three times with 15 ml portions of diethyl ether in a 500 ml separatory funnel. Store the extracted solution in a polyethylene bottle over crystals of uranium free aluminum nitrate.

   b. Aluminum Nitrate (0.2 Molar) - Dissolve 7.5 grams of $Al(NO_3) \cdot 9H_2O$ in deionized water. Add one drop of concentrated nitric acid and dilute to 100 ml. Test the uranium concentration as in the saturated aluminum nitrate solution.

   c. Ammonium Hydroxide (1:1) - Dilute 500 ml of concentrated ammonium hydroxide to 1 liter with deionized water.

d. Ammonium Nitrate Solution (1%) - Dissolve 5 grams of reagent grade ammonium nitrate in deionized water and dilute to 500 ml.

e. Diammonium Hydrogen Phosphate Solution - Dissolve 6 grams of reagent grade $(NH_4)_2HPO_4$ in deionized water and dilute to 500 ml.

f. Diethyl Ether - Reagent

g. Ethyl Acetate - Reagent

h. Flux - Using anhydrous reagent grade chemicals, prepare a flux consisting of 9% sodium fluoride, 45.5% sodium carbonate, and 45.5% potassium carbonate by weight. Dry mix the chemicals for approximately 12 hours in a ball mill. Fuse the resulting mixture in a platinum dish over a Fisher burner or in a muffle furnace. Cool the fused flux, grind it in a mortar and pestle, then pulverize it in a ball mill for 48-72 hours. Store in a desiccator until use.

i. Methyl - Red Indicator Solution - Dissolve 0.1 grams of methyl-red indicator in 18.6 ml of 0.02N sodium hydroxide solution and dilute to 250 ml with deionized water.

j. Nitric Acid - concentrated, reagent.

k. Potassium Pyrosulfate Crystals, reagent.

l. Sodium Hydroxide - Pellets, reagent.

m. Standard uranium stock solution (100 µg) - Dissolve 0.1782 grams of reagent grade uranyl acetate dihydrate in approximately 500 ml of deionized water. Add 2 ml of concentrated reagent grade nitric acid and dilute to 1 liter. Store in polyethylene bottle. From this stock solution, prepare working standards containing 2.5 µgU/ml, 1.0 µg U/ml, 0.2 µgU/ml, 0.1µgU/ml, and 0.05 µgU/ml.

2. Determination for Water
   a. Place 250 ml of each sample in a 400 ml beaker.
   b. Prepare two blanks by placing 250 ml of deionized water in each of two 400 ml beakers.
   c. Add to each sample and blank:
      1. 3 ml of concentrated nitric acid.
      2. 1 ml of 0.2 molar aluminum nitrate solution
      3. 5 ml of diammonium hydrogen phosphate.
   d. Transfer to a hotplate and heat to boiling point. Hold at boiling point for 10 minutes. Remove samples from hotplate.
   e. Add 3 drops methyl-red indicator and neutralize the sample with 1:1 ammonium hydroxide to the yellow endpoint. (If, on addition of the indicator, a pink color forms and disappears, the water may contain excessive iodide or bromide ions. In that event, add ammonium hydroxide, 2 or 3 drops

at a time; then add a drop of indicator. Repeat until the indicator instantly exhibits the yellow color.)

Caution: Do not add ammonium hydroxide past the endpoint. If the solution becomes too basic, no precipitate will form. In this event, the solution pH should be checked. If the pH is 8 or greater add 3 ml of concentrated nitric acid. Add 1 ml of 0.2 molar alumuinum nitrate solution and 5 ml of diammonium hydrogen phosphate solution. Heat the solution and neutralize with 1% ammonium hydroxide.

f. Digest the precipitate on the hotplate for 10-15 minutes. Allow the precipitate to cool and settle.

g. Using an aspirator, draw off as much of the supernatant liquid as possible without disturbing the precipitate.

h. Transfer the precipitate to a 50 ml centrifuge tube. Rinse the beaker and stirring rod with 1% ammonium nitrate solution from a wash bottle, adding the washing to the centrifuge tube. Centrifuge for about 10 minutes. Discard the liquid, wash the precipitate with a few ml of 1% ammonium nitrate solution, centrifuge and discard the liquid.

i. Add 8 ml of the saturated aluminum nitrate reagent and warm gently to dissolve the precipitate. If the precipitate does not dissolve completely add more aluminum nitrate until it does.

j. Transfer the solution to a 50 ml stoppered test tube. Drain the centrifuge tube as completely as possible but do not wash.

k. Add 15.0 ml of ethyl acetate and shake the mixture for 1-2 minutes. Vent frequently to relieve the pressure. Allow 15 minutes for the layers to separate. If necessary centrifuge the sample at low speed to separate the layers.

Caution: Do not let the samples stand for a long period at this point. Excessive pressure may build up and blow the stopper from the tube.

l. Draw off about 13 ml of the ethyl acetate and filter through a dry Whatman No. 42 filter paper into a dry test tube; stopper the tube.

m. To test each sample for the amount of spike and amount of sample to use, 0.5 ml of the filtered ethyl acetate is transferred to a platinum fusion dish. Place the dish on a large watchglass. Add water to the watchglass until the dish is about one-fourth submerged. Ignite the ethyl acetate and let it burn completely.

n. To each of the dishes add 0.5 grams of the flux mixture.

o. Place the dishes on the rotating fusion table. Press the start button on the fusion apparatus. Make sure the temperature reaches slightly higher that 605°C and heat the dishes until the flux is melted (approximately 4 minutes). The fusion apparatus will automatically reduce the heat and run for an additional 3 minutes. When fusion is complete allow the sample to cool for 8 minutes with the table rotating.

p. Place the dishes in a desiccator and cool for at least 30 minutes.

q. Allow the fluorimeter to warm up for at least ½ hour before use.

r. Remove the samples from the fusion dishes. Do not touch the sample with fingers in order to avoid contamination with skin oil which may quench the fluorimeter reading

s. Read each sample in the fluorimeter and record the result.

t. A spike size for each sample is selected which will approximately equal the fluorimeter readings obtained in step s.

Fluorimeter Reading	Spike Size
	0.05 μgU/ml
	0.20 μgU/ml
	1.0 μg/U/ml
	2.5 μgU/ml

u. If the sample gives a greater reading than above, the extraction must be diluted with reagent grade ethyl acetate.

v. The actual uranium concentration of the samples may now be determined. A typical analysis will consist of two blanks, two aliquots of each sample unspiked, and two aliquots of each sample spiked with the appropriate spike from step t. Transfer to platinum fusion dishes four 0.5 ml aliquots of each sample and two 0.5 ml aliquots of a blank.

w. Burn the ethyl acetate as in step m.

x. To the appropriate sample dishes add the spike. Place the samples under a heat lamp and dry.

y. To each fusion dish add at 0.5 grams of flux

z. Proceed as in steps o through s.

aa. The uranium concentrations are then calculated by the method described in the calculations section of this manual.

3. Determination for Soil, Sediment, and Vegetation

   a. Weigh out 1 gram of prepared sample.

   b. Transfer to a 100 ml Teflon beaker and evaporate twice to dryness with 15 ml portions of concentrated hydrochloric acid.

   c. Dry in oven at 100°C for a minimum of 2 hours to dehydrate the silica.

d. Add two ml of concentrated hydrochloric acid and 20 ml of water and heat, covered, for 30 minutes. Decant liquid into a 50 ml centrifuge tube. Centrifuge at 2000 RPM for 5-10 minutes. Decant the supernatant liquid into a 250 ml bottle and transfer the residue back to the Teflon beaker.

e. Add 15 ml of 48% hydrofluoric acid and 10 ml of concentrated hydrochloric acid to the residue and evaporate to dryness on a hotplate. Remove the fluoride by three successive evaporations to dryness with 5 ml portions of 6N hydrochloric acid.

f. Add 2 ml of concentrated hydrochloric acid and 20 ml of water. Cover and heat for 30 mintues and then transfer to the same 50 ml centrifuge tube used in step d. Centrifuge and decant the supernatant liquid into the bottle containing the first dissolved portion. Transfer the remaining residue to a 15 ml plantinum dish. Dry under a heat lamp.

g. Add one more gram of potassium pyrosulfate to the platinum dish and fuse over a burner. Cool, add 3 ml of concentrated hydrochloric acid and 2.5 ml of water, and heat until the residue has dissolved. Combine with the other dissolved portions.

h. Dilute to 250 ml and transfer to 400 ml beaker.

i. Go to step b of the determination for water and proceed from there.

4. Determination of Airborne Dust on Glass Fiber Filters

   a. Take a representative cutting from the total filter such that the suspended matter weighs approximately 0.1 gram. (See the procedure for representative cutting from air-filters).

   b. Proceed to step a of the soil and sediment sample determination.

H. CALCULATION OF RESULTS

The calculation of the uranium concentration is made with the following expression:

$$U = \frac{(A - B)S}{(D - A)V_S} \cdot F_D \cdot E_A$$

where

U = concentration of uranium, in micrograms (ug) per unit, in the sample

A = Fluorimeter reading of the sample

B = Fluorimeter reading of the blank

S = Micrograms of uranium in the spike

D = Fluorimeter reading of the sample spiked with 5 micrograms of uranium.

V_S = The sample size in liters, grams, etc.

F_D = Dilution factor, used to correct for the dilution in step u

E_A = Extraction volume factor

$$E_A = \frac{\text{Volume of ethyl acetate used in extraction}}{\text{Volume of ethyl acetate used in fusion}}$$

FLUORIMETRIC URANIUM WORKSHEET

Laboratory Sample Number: ______________________

Sample Type: ____________________

Aliquot Used for Analysis: ______________________ = V

Ml of ethyl acetate used for extraction: ______________________

Spike amount determination:

Ethyl acetate aliquot: ____________ fluorimeter reading: __________

Spike amount used: _____________ = S

Dilution required: ________________ Amount of ethyl acetate used

to dilute: _____________ ml

Fluorimeter reading of sample: _______ = A

Fluorimeter reading of blank: ________ = B

Fluorimeter reading of spiked sample: ___________ = D

Dilution factor: ____________ = F_D

Extraction factor: _____________ = E_A

Calculation: $U = \frac{(A - B)\,S}{(D - A)\,V_S} \cdot F_D \cdot E_A$ = Uranium concentration

Analysis result: ______________________________________

Analyst: ________________________________

Date: _____________________________

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6.1.A.25.0 RADIOCHEMICAL DETERMINATION OF RADIUM 226

A. PURPOSE

To provide an analytical method for Radium 226 in environmental samples.

B. SCOPE

This analytical procedure applies to all routine environmental samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that the procedure is followed by the analyst and to evaluate the analytical results. It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. When using hydrofluoric acid or hydrogen peroxide, rubber gloves, rubber apron, safety glasses, and full face shield shall be worn. Xylene shall be used only in a properly operating fume hood. Any spill of mercury shall be cleaned up immediately using the mercury spill kit and the Environmental Chemist notified. The high pressure helium tanks shall be secured at all times and shall be used only with a suitable two stage regulator.

E. QUALITY CONTROL

A spike sample, a duplicate sample, and a blank shall be analyzed with every ten samples in a sample run. If fewer than ten samples are analyzed in a single run, a spike sample, a duplicate sample, and a blank shall be analyzed.

A recovery spike sample shall be analyzed for each sample.

F. PROCEDURE

1. Reagents
    a. Ammonium Acetate (6 Molar) - Dissolve 462g of ammonium acetate in 500 ml of deionozed water and diluted to 1 liter.
    b. Ascarite - 8 to 20 mesh.
    c. EDTA - Sodium Carbonate Decontaminating Solution - Dissolve 10g of tetrasodium EDTA and 10g of sodium carbonate in deionized water and dilute to 1 liter.
    d. Hydrochloric Acid (6N) - Mix 1 volume of concentrated reagent hydrochloric acid with 1 volume of deionized water.
    e. Hydrofluoric Acid (48%) - reagent.
    f. Hydrogen Peroxide (30%) - reagent.

g. Lead Carrier Solution (100 mg/ml) - Dissolve 40g of $Pb(NO_3)_2$ plus a few drops of concentrated nitric acid in 200 ml of deionized water and diluted to 250ml. Store in a glass bottle.

h. Magnesium Perchlorate - Anhydrous, reagent.

i. Nitric Acid - concentrated, reagent.

j. Nitric Acid (3N) - Mix 188 ml of concentrated nitric acid in 600 ml of deionized water and dilute to 1 liter.

k. Sodium Carbonate - powder, reagent.

l. Sodium Carbonate (3N) - Dissolve 159g of anhydrous sodium carbonate in 500ml of deionized water and dilute to 1 liter.

m. Sodium hydroxide - Pellets, reagent.

n. Sufuric Acid - concentrated, reagent.

o. Sulfuric Acid (1N) - Mix 28 ml of concentrated sulfuric acid in 500 ml of deionized water and dilute to 1 liter.

p. Toluene - reagent.

2. Apparatus

a. Radon Counting System - An alpha scintillation system capable of accomidating Lucas cells.

b. Filter Flask - 2 liter volume.

c. Suction Pump

d. Radon De-emanation Apparatus - consists of open ended manometer, gas purification tube and stopcocks. See Attachment 1.

e. Radon Bubblers - See Attachment 2.

f. Helium Cylinder - Equipped with a two-stage regulator and a needle valve.

g. Pyseal or equivalent - for sealing bubbler joints.

h. Centrifuge

i. Lucas Cells - See Attachment 3.

j. Magnetic stirring hot plates.

k. Nickel crucibles.

3. Determination for Water

a. Transfer two 100ml aliquots of sample to 2 liter beakers. Add 1ml of Ra-226 solution to one of the beakers as a recovery spike. Label the spike beaker accordingly.

b. Adjust the pH to approximately 1.0 with concentrated nitric acid, add 10ml of lead carrier.

c. Add 35ml of concentrated sulfuric acid, and heat to about 70°C with stiring on the magnetic stirring hot plate.

d. Remove the sample from the hot plate and allow precipitate to settle overnight. Decant or aspirate, discard the supernate and transfer the precipitate to a 50ml centrifuge tube using 1N sulfuric acid.

e. Centrifuge, discard the supernate. Wash the precipitate with 10ml of water, centrifuge, discard the supernate.

f. Rinse the walls of the 2 liter beaker with 5ml of 6M ammonium acetate. Transfer this solution to the precipitate in the centrifuge tube. Bring the volume in the tube to about 20ml with 6M ammonium acetate.

g. Heat in a water bath with stirring until the precipitate dissolves.

h. Slowly add 20ml of 3N sodium carbonate, continue heating, and stir for 15 minutes. Centrifuge, discard the supernate. Wash the precipitate twice with 10ml of deionized water. Discard the washing.

i. Dissolve the carbonate precipitate with 10ml of 3N nitric acid, reprecipitate using 30ml of hot 3N sodium carbonate.

j. Heat and stir for approximately 15 minutes, centrifuge, discard the supernate. Wash the precipitate with 10ml of deionized water. Centrifuge, discard the supernate.

k. Dissolve the carbonate precipitate with 5ml of 3N nitric acid. Transfer sample to radon bubbler with a minimum of 7ml of deionized water. The bubbler frit should first be moistened with a drop of deionized water.

l. Seal the bubbler with pyseal.

m. Attach the bubbler to the helium gas line with a piece of tygon tubing. Adjust the helium regulator valve so that a very slow stream of gas will flow. Adjust the pressure so that a froth a few millimeters thick is produced. Purge the liquid for 10 minutes. Record the end of this time as the zero ingrowth time.

n. Close the inlet stopcock on the bubbler, remove the gas connection, and close the outlet stopcock on the bubbler.

o. Store the bubbler for a minimum of 14 days before collecting and counting the Radon-222.

p. Proceed to the de-emanation section of this procedure.

4. Determination for Vegetation

   a. Weigh out two 4 g aliquots of vegetation ash into two nickel crucibles.

      NOTE: It may be necessary to use less than 4g of ash in order to leave sufficient sample for the other required analyses.

      Add 1ml of Ra-226 spike solution to one of the crucibles. Mark this crucible as the recovery spike sample.

   b. Dry the samples in an oven for 1 hour at 100°C.

c. Add 30g of sodium hydroxide pellets and mix thoroughly with the ash. Fuse over a burner for 30 minutes and then slowly add 2.5g of anhydrous sodium carbonate. Heat the clear red melt for 30 minutes.

d. Remove the crucible from the flame immediately into a cold water bath to crack the mixture.

e. Put the mixture into a 1 liter beaker. If the mixture adheres to the crucible, fill with deionized water, and boil on a hot plate. Add the resulting solution to the 1 liter beaker.

f. Dilute the total volume in the beaker to 300ml with boiling deionized water. Place the beaker on a preheated hotplate and boil to disintegrate the fused mixture. Add deionized water as necessary to keep the volume between 150-200ml.

g. Cool then transfer the mixture to two 100ml centrifuge tubes.

h. Centrifuge for 5 - 10 minutes and discard the supernate. Wash with 50ml portions of hot deionized water. Centrifuge and discard the supernate.

i. Add 12ml of 6M hydrochloric acid to each centrifuge tube to dissolve the precipitate. Add 12ml of deionized water to each centrifuge tube.

j. Centrifuge the solution. Pour off the liquid into a clean 250ml beaker. Discard the solid residue.

k. Transfer 10ml of the solution to a radon bubbler. Measure the remaining volume of solution with a graduated cylinder. Record the total volume of solution (Including the 10ml aliquot) on the worksheet.

l. Proceed to step m of the determination for water.

5. Determination for Soil, Sediments and Glass Fiber Airfilters.

   a. Weigh 2g of the prepared sample into a 600ml beaker.

   b. Add 50ml of concentrated nitric acid to the sample and heat on a hot plate.

   c. Evaporate to near dryness but do not let the sample completely dry out.

   d. Add 50ml of concentrated nitric acid and 30ml of 30% hydrogen peroxide. Heat on the hot plate to near dryness.

   e. Transfer the solution to a 100ml centrifuge tube with deionized water. Centrifuge for 5 - 10 minutes.

   f. Pour the solution into a 2 liter beaker and transfer the residue to a teflon beaker using deionized water.

   g. Add 25ml of concentrated hydrofluoric acid to the teflon beaker and evaporate to dryness. Repeat.

   h. Add 25ml of concentrated nitric acid and evaporate to dryness. Repeat twice.

i. Cool the residue and add 25ml of 3N nitric acid to dissolve the residue. Scrub down the beaker walls with a rubber policeman. Add more 3N nitric acid as necessary.

j. Transfer the beaker contents to a 100ml centrifuge tube. Centrifuge.

k. Transfer the solution to the 2 liter beaker.

l. Wash the residue once with 3N nitric acid, adding the washings to the 2 liter beaker.

m. Discard any remaining residue.

6. Determination of Suspended Solids Collected on a Membrane Filter

   a. Place the sample in a 150ml borosilicate beaker and wet ash with repeated additions of concentrated nitric acid in the presence of 1ml of concentrated sulfuric acid.

   b. Transfer the solution and residue to a 15ml platinum dish, add 5ml of 48% hydrofluoric acid and evaporate until fumes of sulfuric acid are given off.

   c. Add 1g of potassium pyrosulfate, heat to volatilize the sulfuric acid and fuse the pyrosulfate.

   d. Disolve the residue in 3ml of concentrated nitric acid and 8ml of water. Transfer to a radon bubbler.

   e. Transfer to step m of the determination for water.

7. De-emanation and counting procedure

   a. Attach a Lucas cell as shown in Attachment 1. Substitute a glass tube with a stopcock for the bubbler so that the helium gas can be turned off and on conveniently.

   b. Open the stopcock on the Lucas cell; close the stopcock on the helium supply and gradually open the stopcock to the vacuum source to evacuate to the cell.

      NOTE: Too rapid a manipulation of the vacuum stopcock can either burst the manometer or introduce mercury into the vacuum system.

   c. Close the stopcock to the vacuum source and check the manometer reading over a period of several minutes. If there is no change in manometer reading after 5 minutes, the system is considered to be leak free.

   d. Open the stopcock to the helium gas and allow the gas to enter the Lucas cell until atmospheric pressure is reached.

   e. Return to step b and repeat step b only.

   f. Close the stopcock of the Lucas cell and then supply helium to return the de-emanation system to atmospheric pressure.

   g. Place the radon bubbler into the system. Before opening any stopcocks evacuate the de-emanation system. Open the stopcock of the Lucas cell.

h. Close the stopcock to the vacuum and check for leaks as in step c.

i. Adjust the helium flow so that a very slow stream of helium will flow with the needle valve open. Attach the gas line to the inlet of the bubbler.

j. Cautiously open the bubbler outlet stopcock to equalize pressure and to transfer all or most of the fluid in the inlet side arm to the bubbler chamber.

k. Close the outlet stopcock and cautiously open the inlet stopcock to flush the remaining fluid from the side area and fritted disk. Close the inlet stopcock.

l. Repeat steps j and k.

m. With the outlet stopcock fully open, cautiously open the inlet stopcock so that the gas flow produces a froth a few millimeters thick at the surface of the bubbler liquid. Maintain the flow rate by adjusting the inlet stopcock of the bubbler.

n. Continue the de-emanation until the manometer indicates atmospheric pressure has been obtained. The total time required should be 10 - 20 minutes.

o. In rapid succession, close the stopcock to the Lucas cell, close the bubbler inlet and outlet stopcocks, shut off and disconnect the gas supply.

p. Record the time and date as the end of ingrowth and the beginning of decay time.

q. Store the bubbler for possible future de-emanation of the same sample.

r. Place the Lucas cell in a dark area to light adapt.

s. Four hours after de-emanation, place the Lucas cell in the scintillation detector, wait 10 minutes, and count. Record the date and time for the counting period.

t. After counting is complete, remove the Lucas cell and reattach the cell to the de-emanation system as in Attachment 1.

u. To remove the radon-222, evacuate the Lucas cell and cautiously refill with helium as described in steps b and d. Repeat several times.

v. For those cells with high activity, a counting check should be made before the cell is used for another sample.

w. If counts above background are still registered, the cell (filled with helium) should be stored 3 to 4 days without use. Then step u should be performed again before the cell is returned to service.

x. All Lucas cells should be stored, after cleanup, filled with helium.

y. Before each use, the background of the particular Lucas cell must be determined. If time permits, the background should be counted for the same length of time as the sample. The minimum background time should be 50 minutes.

z. Calculate results as shown in the calculation section of this procedure.

8. Radon Bubbler Cleaning Procedure

a. After the Ra-226 results have been accepted, and no additional de-emanations of the sample are required, the bubbler may be be emptied and cleaned. If Ra-228 is to be determined, remove the solution from the bubbler, filter through a Whatman No. 2 filter paper, and transfer to step b of the Radium 228 procedure.

b. If the sample results show a Ra-226 concentration greater than 10pCi, the bubbler should be filled with EDTA - sodium carbonate decontamination solution.

c. Place the filled bubbler in a water bath and raise the temperature to the boiling point. Continue heating for 1 to 2 hours.

d. Cool and empty the bubbler; rinse with deionized water.

e. Fill the bubbler with 0.1N hydrochloric acid and supply suction to the gas inlet on the bubbler. Repeat twice.

f. Dry and remove any sealant residue with toluene. Store in a dust-free area until next use.

9. Calculation of Result:

The calculation of the Ra-226 concentration is made using the following expression

$$\text{Ra-226 (pCi/unit)} = \frac{(S-B)C}{(E)(Y)(V_S)(B^1)(A)(D)} \pm \frac{1.96\sqrt{S/T_S + B/T_B}\,(C)}{(E)(\gamma)(V_S)(B^1)(A)(D)}$$

S = Sample cpm

T_S = Sample Count Time

B = Background cpm

T_B = Background Count Time

C = Factor for the decay of Rn-222 during counting, (see Attachment 4)

E = Efficiency Factor = $\frac{\text{counts/min.}}{\text{pCi}}$ for a particular Lucas cell.

$$Y = \text{Chemical Yield} = \frac{\frac{\text{Recovery Spike Counts}}{E_R} - \frac{\text{Sample Counts}}{E_S}}{\text{Recovery Spike Decays}}$$

where E_R = Efficiency of the recovery spike Lucas cell.

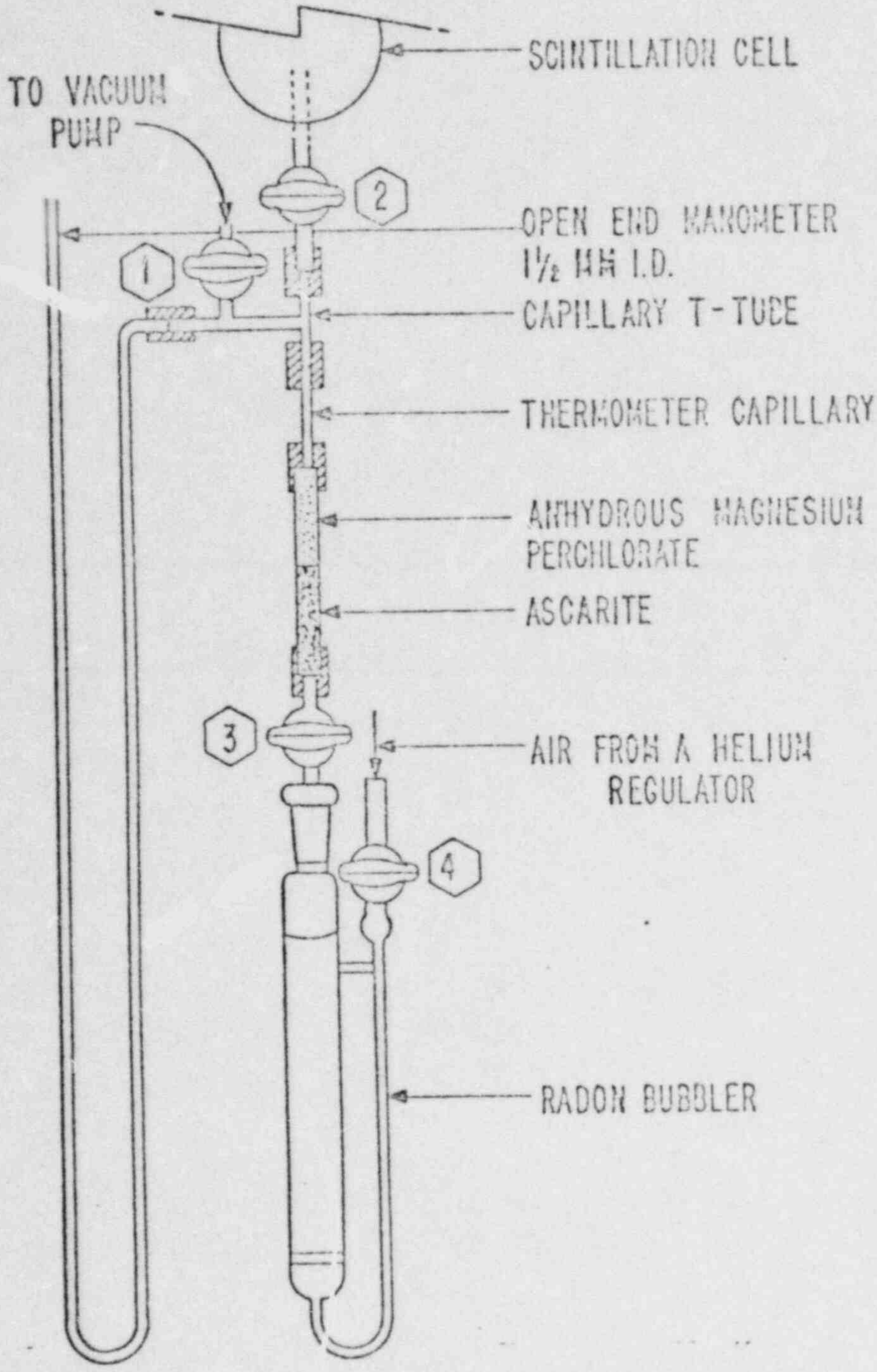
E_S = Efficiency of the sample Lucas cell.

V_S = Sample Volume (liters/grams)

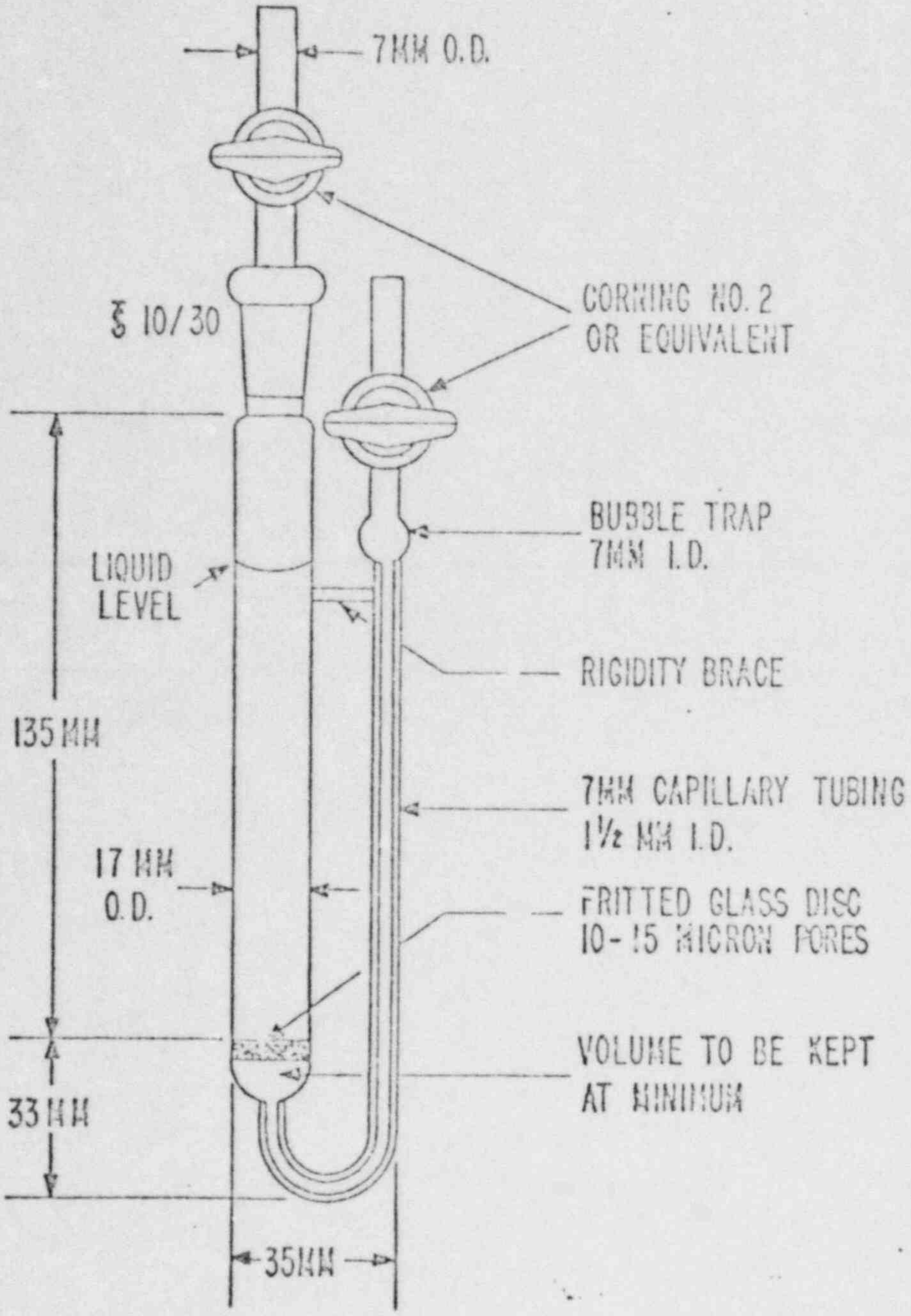
D = Dry weight ratio factor = $\frac{\text{dry weight}}{\text{ash weight}}$ = 1 for water samples.

B^1 = Factor for the ingrowth of Rn-222 from Ra-226 (See Attachment 4)

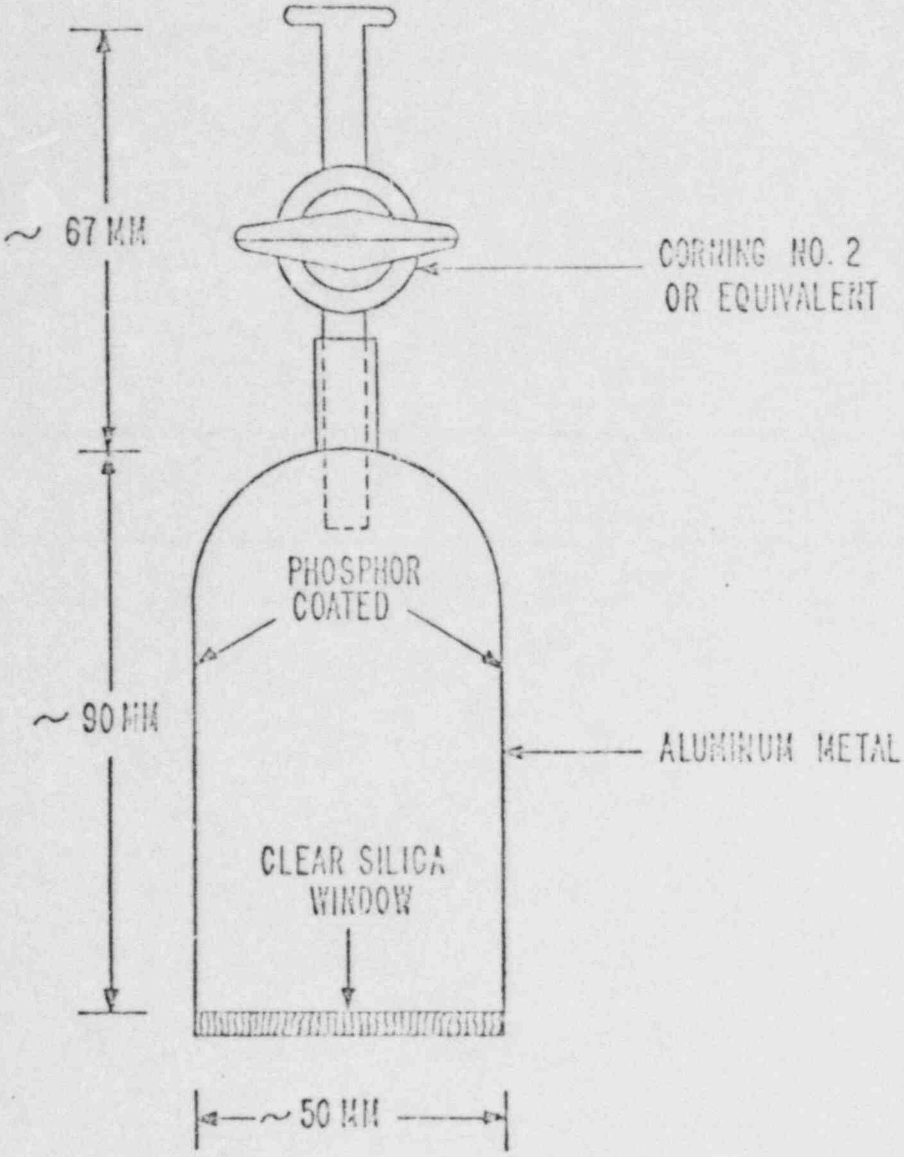
A = Factor for the decay of Rn-222 (See Attachment 4)



ATTACHMENT I. RADON DE-EMANATION APPARATUS



ATTACHMENT 2. RADON BUBBLER



ATTACHMENT 3. LUCAS SCINTILLATION CELL

ATTACHMENT 4

Factors for Decay of Padon-222, Growth of Radon-222 from Radium-226, and Correction of Radon-222 Activity for Decay During Counting

Time	Factor for Decay of Radon-222		Factor for Growth of Radon-222 from Radium-226		Factor for Correction of Radon-222 Activity for Decay during Counting
	$A=e^{-\lambda t}$		$B=1-e^{-\lambda t}$		$C=\lambda t/(1-e^{-\lambda t})$
	Hours	Days	Hours	Days	Hours
0.0	1.0000		0.000 00		1.000
0.2	0.9985		0.001 51		1.001
0.4	0.9970		0.003 01		1.001
0.6	0.9955		0.004 52		1.002
0.8	0.9940		0.006 02		1.003
1	0.9925	0.8343	0.007 52	0.1657	1.004
2	0.9850	0.6960	0.014 99	0.3040	1.008
3	0.9776	0.5807	0.022 40	0.4193	1.011
4	0.9703	0.4844	0.029 75	0.5156	1.015
5	0.9630	0.4041	0.037 05	0.5959	1.019
6	0.9557	0.3372	0.044 29	0.6628	1.023
7	0.9485	0.2813	0.051 48	0.7187	1.027
8	0.9414	0.2347	0.058 61	0.7653	1.031
9	0.9343	0.1958	0.065 69	0.8042	1.034
10	0.9273	0.1633	0.072 72	0.8367	1.038
11	0.9203	0.1363	0.079 69	0.8637	1.042
12	0.1134	0.1137	0.086 62	0.8863	1.046
13	0.9065	0.0948	0.093 49	0.9052	1.050
14	0.8997	0.0791	0.100 31	0.9209	1.054
15	0.8929	0.0660	0.107 07	0.9340	1.058
16	0.8862	0.0551	0.1138	0.9449	1.062
17	0.8795	0.0459	0.1205	0.9541	1.066
18	0.8729	0.0383	0.1271	0.9617	1.069
19	0.8664	0.0320	0.1336	0.9680	1.073
20	0.8598	0.0267	0.1402	0.9733	1.077

ATTACHMENT 4 (Continued)

Factors for Decay of Radon-222, Growth of Radon-222 from Radium-226, and Correction of Radon-222 Activity for Decay During Counting (continued)

Time	Factor for Decay of Radon-222		Factor for Growth of Radon-222 from Radium-226		Factor for Correction of Radon-222 Activity for Decay during Counting
	$A=e^{-\lambda t}$		$B=1-e^{-\lambda t}$		$C=\lambda t/(1-e^{-\lambda t})$
	Hours	Days	Hours	Days	Hours
21	0.8534	0.0223	0.1466	0.9777	1.081
22	0.8470	0.0186	0.1530	0.9814	1.085
23	0.8406	0.0155	0.1594	0.9845	1.089
24	0.8343	0.0129	0.1657	0.9871	1.093
25	0.8280	0.0108	0.1720	0.9892	1.097
26	0.8218	0.0090	0.1782	0.9910	1.101
27	0.8156	0.0075	0.1844	0.9925	1.105
28	0.8095	0.0063	0.1905	0.9937	1.109
29	0.8034	0.0052	0.1966	0.9948	1.113
30	0.7973	0.0044	0.2027	0.9956	1.118
31	0.7913	0.0036	0.2087	0.9964	1.122
32	0.7854	0.0030	0.2146	0.9970	1.126
33	0.7795	0.0025	0.2205	0.9975	1.130
34	0.7736	0.0021	0.2264	0.9979	1.134
35	0.7678	0.0018	0.2322	0.9982	1.138
36	0.7620	0.0015	0.2380	0.9985	1.142
37	0.7563	0.0012	0.2437	0.9988	1.146
38	0.7506	0.0010	0.2494	0.9990	1.150
39	0.7449	0.0009	0.2551	0.9991	1.154
40	0.7393	0.0007	0.2607	0.9993	1.159
41	0.7338	0.0006	0.2662	0.9994	1.163
42	0.7283	0.0005	0.2717	0.9995	1.167
43	0.7228	0.0004	0.2772	0.9996	1.171
44	0.7173	0.0003	0.2827	0.9997	1.175
45	0.7120	0.0003	0.2880	0.9997	1.179

ATTACHMENT 4 (Continued)

Factors for Decay of Radon-222, Growth of Radon-222 from Radium-226, and Correction of Radon-222 Activity for Decay During Counting (continued)

Time	Factor for Decay of Radon-222		Factor for Growth of Radon-222 from Radium-226		Factor for Correction of Radon-222 Activity for Decay during Counting
	$A=e^{-\lambda t}$		$B=1-e^{-\lambda t}$		$C=\lambda t/(1-e^{-\lambda t})$
	Hours	Days	Hours	Days	Hours
46	0.7066	0.0002	0.2934	0.9998	1.184
47	0.7013	0.0002	0.2987	0.9998	1.188
48	0.6960	0.0002	0.3040	0.9998	1.192
49	0.6908	0.0001	0.3092	0.9999	1.196
50	0.6856	0.0001	0.3144	0.9999	1.201
51	0.6804	0.0001	0.3196	0.9999	1.205
52	0.6753	0.0001	0.3247	0.9999	1.209
53	0.6702	0.0001	0.3298	0.9999	1.213
54	0.6652	0.0001	0.3348	0.9999	1.218
55	0.6602	0.0000	0.3398	1.0000	1.222
56	0.6552	0.0000	0.3448	1.0000	1.226
57	0.6503	0.0000	0.3497	1.0000	1.231
58	0.6454	0.0000	0.3546	1.0000	1.235
59	0.6405	0.0000	0.3595	1.0000	1.239
60	0.6357	0.0000	0.3643	1.0000	1.244

RADIUM 226 WORKSHEET

Laboratory Sample Number: ____________

Sample Type: ____________

Sample Dry Weight (Solid Samples): ____________

Sample Ash Weight (Solid Samples): ____________

Aliquot Weight or Volume (l/S): ____________

Times and Dates

Zero Ingrowth: ____________

End of Ingrowth: ____________

Start of Decay: ____________

Start of Count: ____________

Count Time: ____________

Factor Calculations

Decay of Rn-222

Start of Count - Start of Decay = ____________ hours

Factor from Chart (A) = ____________

Ingrowth of Rn-222

End of Ingrowth - Zero Ingrowth = ____________ hours

Factor (B^1) = ____________

Decay of Rn-222 During Counting

Count Time = ____________ hours

Factor (C) = ____________

Counting Data

Sample Lucas cell Background (B) ____________ cpm

Sample Lucas cell Background time (T_B) ____________ minutes

Spike Lucas cell Background ____________ cpm

Spike Lucas cell Background Count Time ____________ minutes

Sample Counts (S) ____________ cpm

Sample Count Time ____________ minutes

Sample Lucas cell Efficiency (E)(E_R) ____________ minutes

Spike Lucas cell Efficiency (E_S) ____________

Calculations

$$= \text{Chemical Yield} = \frac{\frac{\text{Recovery Spike Counts}}{E_R} - \frac{\text{Sample Counts}}{E_S}}{\text{Recovery Spike Decays}}$$

D Dry Weight Ratio Factor = $\frac{\text{Dry Weight}}{\text{Ash Weight}}$ = 1 for Water Samples

$$\text{Ra-226 (pci/unit)} = \frac{(S-B)C}{(E)(Y)(V_S)(B^1)(A)(D)} \pm \frac{1.96\sqrt{S/T_S + B/T_B}\ (C)}{(E)(Y)(V_S)(B^1)(A)(D)}$$

Calculation:

Lower Limit of Detection

$$\text{LLD Ra-226 (pCi/unit)} = \frac{4.66\sqrt{B/T_B}\ (C)}{E\ Y\ V_S\ B^1\ A\ D}$$

Result:

Ra226 (pCi/____________) = ____________pCi/_____ ± ____________pCi/_____

LLD (pCi/____________) + ____________pCi/_____

Analyst____Date______________________

6.1.A.26.0 RADIOCHEMICAL DETERMINATION OF RADIUM 228

A. PURPOSE

To provide an analytical method for Radium 228 in environmental samples.

B. SCOPE

This procedure applies to the analysis of all routine environmental samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Rubber gloves to be worn when handling reagents. Ammonium sulfate, ammonium sulfide and acetone to be used only in a properly operating fume hood.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank are to be analyzed with every ten samples in a sample run. If fewer than ten samples are to be analyzed in a sample run, one spike sample, one duplicate, and one blank are to be analyzed. A recovery spike sample is to be analyzed with each sample.

D. PROCEDURE

1. Reagents
   - a. Acetic acid (glacial) - Reagent grade
   - b. Ammonium Sulfate Solution (200 mg/ml) - Dissolve 200 g of ammonium sulfate in a minimum of water and dilute to 1 liter.
   - c. Ammonium Sulfide Solution (approx. 2%) - Dilute 4 ml of ammonium sulfide to 100 ml.
   - d. Barium carrier solution (16 mg/ml) - Dissolve 28.460 g of barium chloride di-hydrate in water, and 2 ml of concentrated nitric acid and dilute to 1 liter.
   - e. Binder Solution - Dissolve 1 ml of "Duco" cement or equivalent in 100 ml of acetone.
   - f. EDTA reagent - Dissolve 20 g of sodium hydroxide in 750 ml

water, heat, slowly add 93 g of disodium EDTA while stirring; dilute to 1 liter.

g. Lead Carrier Solution (1.5 mg/ml) - Dissovle 2.4 g of lead nitrate in water, add 2 ml of concentrated nitric acid and dilute to 1 liter.

h. Sodium Hydroxide Pellets - Reagent grade

i. Sodium Hydroxide (10N) - Carefully dissolve 400 g of sodium hydroxide pellets in a minimum of water, dilute to 1 liter.

j. Strontium-Yttrium carrier (0.9 mg/ml Sr and 0.9 mg/ml Y) - Dilute 10 ml of yttrium carrier solution (18 mg/ml) to 100 ml (Solution A). Dissolve 434.8 mg of strontium nitrate in water, dilute to 100 ml (Solution B). Combine solution A and B.

k. Yttrium Carrier Solution (18 mg/ml) - Add 22.85 g of yttrium oxide to a 250 ml Erlenmeyer Flask containing 20 ml of water, swirl, place on a hotplate and heat to boiling. About 30 ml of nitric acid is needed to dissolve the yttrium oxide. Small additions of water may also be required. After the yttrium oxide has dissolved, add 70 ml of concentrated nitric acid and then dilute to 1 liter.

2. Determination for Water

   a. If Ra 226 analysis was not requested for the sample, perform steps a through k of the Ra 226 procedure for water.

If Ra 226 analysis was requested, perform steps a through o of the Ra 226 procedure for water and step a of the Ra-226 procedure for radon bubbler cleaning.

b. Add 25 ml of EDTA reagent. Heat in a hot water bath and stir intermittently; add additional 10M sodium hydroxide in small amounts if the precipitate does not dissolve readily.

c. Add 1 ml of strontium-yttrium carrier solution. Stir thoroughly. Add a few drops of 10N sodium hydroxide if any precipitate forms.

d. Add 1 ml of ammonium sulfate solution (200 mg/ml) and stir thoroughly. Add glacial acetic acid dropwise until barium sulfate precipitates and then add 2 ml excess. Allow the precipitate to digest in a hot water bath until the precipitate has largely settled. Centrifuge; discard the supernatant liquid.

e. Add 20 ml of the EDTA reagent, heat, and stir until the barium sulfate precipitate dissolves. Repeat steps c and d. Record the time of the last barium sulfate precipitation.

f. Dissolve the barium sulfate precipitate in 20 ml of EDTA reagent; then add 1 ml of yttrium carrier solution (18 mg/ml) and 1 ml of lead carrier solution (1.5 mg/ml). If any precipitate forms, dissolve by adding a few drops of 10N sodium hydroxide. Transfer to a teflon or polyethylene container and age for a minimum of 36 hours (2200 minutes). The sample should be covered to prevent evaporation.

g. Transfer the aged solution to a 100 ml centrifuge tube; add 0.3 ml of ammonium sulfide solution. Add 10N sodium hydroxide dropwise while stirring until the lead sulfide precipitates, then add 10 drops excess, stir intermittently over a period of 10 minutes.

h. Centrifuge the solution and transfer the supernatant liquid to a clean centrifuge tube.

i. Place the centrifuge tube in a hot water bath and slowly add 10N sodium hydroxide while stirring until yttrium hydroxide precipitates. Add 1 ml excess and stir intermittently for several minutes. Record the time of precipitation. Centrifuge as soon as the yttrium hydroxide has largely settled.

j. Wash the precipitate thoroughly with 5 ml of water containing about 10 drops of 10N sodium hydroxide; centrifuge and discard the wash solution.

k. Transfer the precipitate to a tared 2 inch diameter counting planchette using small volumes of distilled water while drying under a heat lamp. Add 1 ml of binder solution to the last washing. Dry the sample and weigh.

l. Count the sample in a low-background beta counter for a minimum of 300 minutes.

m. Calculate result as described in the calculations section of this procedure.

3. Determination for Soil and Sediments

   a. Perform steps a through o of the Ra-226 procedure for soil and sediments.

   b. Perform steps a through m of the Ra-228 procedure for water.

4. Calculation of Results and Counting Error at the 95% Confidence Level

   The following formula is used to calculate the Ra-228 concentration:

$$\text{Ra (pCi/unit)} = \frac{(S - B)\lambda t_2}{2.22\, V_S E e^{-\lambda t_1}(1 - e^{-\lambda t_2})Y} \times \frac{1}{(1 - e^{-\lambda t_3})}$$

$$\pm \frac{1.96\sqrt{S/T_S + B/T_B}}{2.22 V_S E e^{-\lambda t_1}(1 - e^{-\lambda t_2})Y} \times \frac{1}{(1 - e^{-\lambda t_3})}$$

   where,

   S = gross sample count rate in cpm

   B = blank count rate including background

   T_S = sample count time in minutes

   T_B = blank count time in minutes

   T_1 = elasped time from yttrium hydroxide precipitation to the beginning of the sample counting period.

   T_2 = length of the sample counting period in minutes

T_3 = time of ingrowth for Ac-288 in minutes measured from the barium sulfate precipitation to the yttrium hydroxide precipitation

V_S = volume of the sample in liters or grams

λ = a decay constant for Ac-228 ($0.001885 \text{ min}^{-1}$)

E = detector efficiency

$$Y = \text{Chemical yield} = \frac{\text{Recovery spike cpm} - \text{Sample cpm}}{\text{E Spike dpm}}$$

Ra-228 ANALYSIS WORKSHEET

UNC Laboratory Sample No.: ____________

Sample Type: ____________

Is Ra 226 analysis requested? ______yes ______no

Aliquot Volume (V_S): ____________

Recovery spike solution used: ____________

Volume of recovery spike solutions used: ____________

Planchette + sample weight ____________

Planchette tare weight ____________

Sample weight ____________

Times and Dates (Use 24 hour clock)

Barium sulfate precipitation: ____________

Yttrium hydroxide precipitation: ____________

Beginning of sample counting: ____________

Instrument Data

Instrument used: ____________

Instrument efficiency, corrected for sample self absorption: ____________

Blank sample gross cpm (B): ____________

Blank sample count time (T_B): ____________ minutes

Sample gross cpm (S): ____________

Sample count time (T_S and t_2): ____________ minutes

Recovery Spike gross cpm: ____________

Recovery Spike count time: ____________

Calculation of Chemical Yield (Y)

$$Y = \frac{\text{Recovery Spike cpm - sample cpm}}{\text{E spike dpm}}$$ (must be corrected for decay)

Calculation of t_1 and t_3

t_1 = time of beginning of count period minus time of yttrium hydroxide precipitation

t_1 = ________________minutes

t_3 = time of yttrium hydroxide precipitation - time of barium sulfate precipitation

t_3 = ________________ $\frac{1}{(1 - e^{-\lambda t_3})} = 1$ if $t_3 > 2200$ minutes

Calculation of result and counting error at the 95% confidence level

$$\text{Ra 228 (pCi/unit)} = \frac{(S - B)\lambda t_2 x_2}{2.22\ V_S EY\ e^{-\lambda t_1}(1 - e^{-\lambda t}\ )} \quad X \quad \frac{1}{(1 - e^{-\lambda t_3})}$$

$$\pm \frac{1.96 \sqrt{S/T_S + B/T_B}\ \lambda t_2}{2.22\ V_S EYe^{-\lambda t_1}(1 - e^{-\lambda t_2})} \quad X \quad \frac{1}{(1 - e^{-\lambda t_3})}$$

Calculation:

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Lower Limit of Detection

$$LLD = \frac{4.66 \sqrt{B/T_B}\ \lambda t_2}{2.22 V_S E y e^{-\lambda t_1} (1- e^{-\lambda t_2})} \quad X \quad \frac{1}{(1 - e^{-\lambda t_3})}$$

Calculation:

Result

Ra 228 (pCi/_____) = ________________pCi/______ ± ________ pCi/_________

LLD(pCi) = ____________pCi/_________

Analyst ____________________________ Date ________________________

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6.1.A.27.0 DETERMINATION OF PO-210 IN ENVIRONMENTAL SAMPLES

A. PUPROSE

To provide an analytical method for Polonium 210 in environmental samples.

B. SCOPE

This procedure applies to the analysis of all routine environmental samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that the proceudre is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Rubber gloves, rubber apron, safety glasses, and full face shield to be worn when using hydrofluroic acid. Hydroxylamine hydrochloride to be diluted in a properly operating fume hood. Polonium 208 tracer to be used only when wearing rubber gloves.

E. QUALITY CONTROL

One spike, one duplicate, and one blank sample to be analyzed with every ten samples in a sample run. If there are fewer than ten samples in a sample run, one spike, one duplicate, and one blank sample are to be analyzed.

If Po-208 is not available as an internal tracer then a recovery spike sample shall be analyzed with each sample.

F. PROCEDURE

1. Reagents
    a. Citric acid (40% W/V) - Dissolve 40 g of reagent grade citric acid in deionized water and dilute to 100 ml.
    b. Hydrofluoric acid (48%) - Concentrated, reagent.
    c. Hydroxylamine hydrochloride (50% W/V) - Dissolve 50 g of reagent grade hydroxylamine hydrochloride in deionized water and dilute to 100 ml. Prepare only enough for one days usage.

d. Lead carrier (50 mg/ml) - Dissolve 20 g of reagent grade lead nitrate in 100 ml of 4 N nitric acid and dilute to 250 ml with 4 N nitric acid.

e. Nitric acid (16 N) - Concentrated, reagent.

f. Nitric acid (4 N) - Mix 250 ml of concentrated nitric acid with 600 ml of deionized water and dilute to 1 liter.

g. Po-208 tracer - Standard dilution in 4 N hydrochloric acid.

h. Sulfuric acid (36 N) - concentrated, reagent.

2. Apparatus

   a. Alpha spectroscopy system

      NOTE: If Po-208 tracer is not available, the sample and the recovery spike may be counted in an alpha proportional counter, although the alpha spectrometer is still preferred.

3. Determination for Water

   a. Perform steps a through c of the Pb-210 procedure for water.

   b. Plate the polonium from the supernate of step c according to steps a through k of the Pb-210 plating procedure.

4. Determination for Vegetation
    a. Perform steps a through d of the Pb-210 procedure for vegetation.
    b. Plate the polonium from the resulting supernate according to steps a through k of the Pb-210 plating procedure.
5. Determination for Soil and Sediment
    a. Perform steps a through f of the Pb-210 procedure for soils and sediment.
    b. Plate the polonium from the resulting supernate according to steps a through k of the Pb-210 plating procedure.
6. Counting
    a. Count in an alpha spectrometer for a minimum of 300 minutes.
7. Calculation (with Po-208 internal tracer)

Sample activity and counting error at the 95% confidence level.

$$\text{Po-210 (pCi/unit)} = \frac{(S - B_S)}{2.22\ AV_SD} \pm \frac{1.96\ \ S/T_S + B/T_B}{2.22\ RV_SD}$$

where:

S = Sample gross cpm in the Po-210 energy region

T_S = Sample count period in minutes

B_S = Background cpm in the Po-210 energy region

T_B = Background count period in minutes

V_S = Sample volume

D = The decay of Po-210 from the date of collection to the date of counting

$D = e^{-\lambda t/T_{\frac{1}{2}}} = e^{-\ln 2 t/T_{\frac{1}{2}}}$

where

t = time and date of counting minus time and date of collection.

$T_{\frac{1}{2}}$ = the half life of Po-210 = 138.4 days.

R = a factor which includes the detector efficiency, chemical yield and all other variables of the chemical process.

$$R = \frac{C - B_C}{V_F F H}$$

where

C = Gross tracer cpm in th Po-208 energy region

B_C = Background cpm in the Po-208 energy region

F = The standardized concentration of Po-208 in dpm/ml

V_F = the volume of Po-208 tracer used in ml

H = The decay of Po-208 from the time of standardization to the time of sample counting.

$H = e^{-\lambda t/T_{\frac{1}{2}}} = e^{-\ln 2 t/T_{\frac{1}{2}}}$

where

t = the time and date of sample counting minus the time and date of Po-208 standardization in days.

$T_{\frac{1}{2}}$ = the half life of Po-208 = 1069.45 days.

Po-210 ANALYSIS WORKSHEET

Laboratory Sample No.: ____________________

Sample Type: __________________________

Date and Time of Collection: __________________

Sample aliquot (V_S): ________________

Po-208 internal tracer

Activity (F): ____________

Date and Time of Standardization: ________________

Amount used (V_F): ____________________

Counting Data

Instrument used: __________________

Detector used: __________________

Po-210 Background cpm (B_S): ________________

Po-208 Background cpm (B_C): ____________________

Background count period (T_B): ____________________

Po-210 Sample cpm (S): ______________

Po-208 Sample cpm (C): ______________

Sample count period (T_S): ______________

Sample counting date and time: ________________

Decay calculation:

Po-210 Decay = D = $e^{-\ln 2t/T_{\frac{1}{2}}}$

t = counting date - date of collection = ____________ days

$T_{\frac{1}{2}}$ = 138.4 days

Calculation:

Po-208 Decay = H = $e^{-\ln 2t/T_{\frac{1}{2}}}$

t = counting date - standardization date = ____________ days

$T_{\frac{1}{2}}$ = 1069.45 days

Calculation:

Calulation of sample activity and counting error at the 95% confidence level.

$$\text{Po-210 (pCi/unit)} = \frac{(S - B)}{2.22RV_SD} \pm \frac{1.96\ S/T_S + B/T_B}{2.22RV_SD}$$

Calculation:

Calculation of Lower Limit of Detection

$$\text{LLD (pCi/unit)} = \frac{4.66\ B_S/T_S}{2.22\ RV_SD}$$

Calculation:

Result

Po-210 pCi/_____ = _______pCi/______ ± ________pCi/________

LLD pCi/______ = _________pCi/_____

Analyst ________________________ Date ___________________

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6.1.A.28.0 DETERMINATION OF Pb-210 in ENVIRONMENTAL SAMPLES

A. PURPOSE

To provide an analytical method for Pb-210 in environmental samples.

B. SCOPE

The analytical procedure applies to the analysis of all routine environmental samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Rubber gloves, rubber apron, safety glasses, and full face shield required when using hyrdofluoric acid. Hydroxylamine hydrochloride to be diluted only in a properly operating fume hood. All traces to be handled using rubber gloves.

E. QUALITY CONTROL

One spike, one duplicate, and one blank to be analyzed with every ten samples in a sample run. If there are fewer than ten samples in a sample run, one spike, one duplicate, and one blank are to be analyzed.

F. PROCEDURE

1. Reagents:
    a. Ammonuim Acetate (6M) - Dissolve 462g of ammonuim acetate in 500ml of deionized water and dilute to one liter.
    b. Citric Acid (40% W/V) - Dissolve 40g of reagent grade citric acid in dionized water and dilute to 100ml.
    c. Hydrochloric Acid (12N) - concentrated, reagent.
    d. Hydrofluoric Acid (48%) - concentrated, reagent.
    e. Hydroxylamine hydrochloride (50% W/V) - Dissolve 50g of reagent grade hydroxylamine hydrochloride in deionized water and dilute to 100ml. Prepare only enough for one days usage.

f. Lead carrier (50mg/ml) - Dissolve 20g of reagent grade lead nitrate in 100 ml of 4N nitric acid and dilute to 250ml with 4N nitric acid.

g. Nitric Acid (16N) - concentrated, reagent.

h. Nitric Acid (8N) - Mix 250ml of concentrated nitric acid with 200ml of deionized water and dilute to 500ml.

i. Nitric Acid (4N) - Mix 250ml of concentrated nitric acid with 600ml of deionized water and dilute to 1 liter.

j. Nitric Acid (3N) - Mix 190ml of concentrated nitric acid with 700ml of deionized water and dilute to 1 liter.

k. Po-208 Tracer - Standard dilution in 4N hydrochloric acid.

l. Potassium Nitrate (0.01M) - Dissolve 1g of reagent grade potassium nitrate in 500ml of deionized water and dilute to 1 liter.

m. Potassium Pyrosulfate - Crystals, reagent.

n. Sodium Carbonate (3N) - Dissolve 159g of anhydrous Na_2CO_3 in 500ml of deionized water and dilute to 1 liter.

o. Sulfuric Acid (36N) - concentrated, reagent.

p. Sulfuric Acid (dilute) - Dilute 35ml of concentrated sulfuric acid in 500ml of deionized water and dilute to 1 liter.

2. Apparatus
   a. Alpha Spectroscopy System.
   b. Nickel Electrode - 7/8" diameter commerical pure nickel disc.
3. Determination for Water
   a. To one liter of sample, add 50ml of concentrated HNO_3 and 3ml of 50mg/ml lead carrier solution. If Po-210 is to be determined, add 1 ml of Po-208 tracer.
   b. Add 35ml of concentrated sulfuric acid; heat to 70°C while stirring; remove from the stirrer and cool overnight.
   c. Decant supernate into a clean one liter beaker. If Po-210 analysis is requested, proceed to step 2 of the Po-210 procedure.
   d. Transfer the sulfate precipitate to a 50ml centrifuge tube with dilute sulfuric acid. Centrifuge, decant, and discard the supernate.
   e. Wash the precipitate with 30ml of dilute sulfuric acid and repeat. Discard the washings.
   f. Add 20ml of 6N Ammonium acetate and heat with stirring until the precipitate dissolves.
   g. Slowly add 20ml of 3N sodium carbonate and heat with stirring for 15 minutes. Centrifuge, decant, and dis-

card the supernate.

h. Wash the precipitate twice with 10ml of deionized water. Discard the washings.

i. Add 10ml of 3N nitric acid and stir until the precipitate dissolves.

j. Add 30ml of hot 3N sodium carbonate; heat with stirring for 15 minutes. Centrifuge, decant, and discard the supernate.

k. Add 5 ml of 3N nitric acid; stir until precipitate dissolves. Transfer the solution to a plastic deposition cell with 3N nitric acid and dilute to about 30ml with 3N nitric acid.

l. Cap the cell; label and store it for a minimum of 30 days.

m. After the above ingrowth period, plate the solution according to the plating of Polonium Section of this procedure.

n. After plating the solution, transfer it to a 50ml volumetric flask and dilute to volume with deionized water.

o. Analyze this solution using the Inductively Coupled Plasma System and determine the lead concentration.

Record the result on the work sheet. This will be used to determine the chemical recovery.

4. Determination for Vegetation.
   a. To one gram of vegetation sample in an 800 ml beaker, add 50 ml of concentrated nitric acid and 3 ml of 50 mg/ml lead carrier. If Po-210 is to be determined, add 1ml of Po-208 tracer.
   b. Digest the sample until the greenish-brown fumes of NO_2 are no longer emitted. Continue heating for at least 10 minutes adding concentrated nitric acid as needed to maintain volume.

      Note: Heat gently and do not boil the sample. Some forms of Po-210 are volatile.
   c. While the sample is still warm, dilute it with deionized water to 500ml. Stir the sample while diluting. Filter through a Whatman #2 filter into a clean 1 liter beaker.
   d. Transfer to step a of the Pb-210 determination for water.
5. Determination for Soil and Sediment.
   a. To 2g of sample in a teflon beaker, add 3ml of 50mg/ml of lead carrier. If Po-210 is to be determined add 1ml of Po-208 tracers.

b. Add 10ml of 4N nitric acid and 10ml of concentrated hydrofluoric acid. Reduce the volume to about 5ml.

   Note: Heat gently and never boil the sample during digestion.

c. Repeat step b twice.

d. Add 10ml of 4N nitric acid and reduce the volume to about 5ml.

e. Repeat step d once.

f. While the sample is still warm, dilute with deionized water to 500ml with stirring. Filter through a Whatman #2 filter into a clean 1 liter beaker.

g. Transfer to step b of the Pb-210 determination for water.

6. Determination for a Paper Filter.

   a. To the sample, in a glass beaker, add 10ml of nitric acid, and 1ml of sulfuric acid and evaporate to near dryness.

   b. Transfer the sample to a platinum crucible with very small volumes of nitric acid. Evaporate to dryness under a heat lamp.

   c. Add 5ml of hydrofluoric acid and evaporate to dryness under a heat lamp.

d. Fuse with 1g of potassium pyrosulfate. Dissolve the resulting salt with 8N nitric acid and dilute to 1 liter with de-ionized water.

e. Using an appropriate aliquot of this solution, proceed to step a of the determination for water.

7. Determination for Glass Fiber Filter.

   a. Place the sample in a teflon beaker and add de-ionized water to cover.

   b. Add 1ml of sulfuric acid and 15ml of hydrofluoric acid. Heat until white fumes of hydrofluoric acid appear.

   c. Add 10ml of 8N nitric acid and evaporate to near dryness

   d. Repeat step c twice.

   e. Transfer the sample to a platinum crucible with very small volumes of 8N nitric acid. Evaporate to dryness under a heat lamp.

   f. Fuse with 1g of potassium pyrosulfate.

   g. Dissolve the resulting salt with 8N nitric acid. Transfer the sample to a 50ml centrifuge tube, centrifuge and discard any residue.

   h. Dilute the supernate to 1 liter with deionized water.

i. Using an appropriate aliquot of this solution, proceed to step a of the determination for water.

8. Plating of Polonuim

a. Mount a nickel disc in the holder after writing the sample information on the back of the disc and spraying with lacquer.

b. Polish the exposed nickel surface to a mirror finish.

c. Rinse the electrode with deionized water and wipe with a lint free tissue.

d. Prime the nickel disc by immersion in 0.5N hydrochloric acid for five minutes.

e. Add 2ml of 40% citric acid and 2ml of hydroxylamine hydrochloride to the sample solution.

f. Insert the electrode holder into the sample solution.

g. Stir the solution using a magnetic stirrer (avoid splashing or vortexing).

h. Plate for one hour.

i. Remove the solution and rinse the nickel disc in deionized water.

j. Allow the disc to air dry.

k. Remove the disc from the holder and count in an alpha spectrometer.

1. Save the solution to determine the lead recovery (steps n and o of the determination for water.)

9. Calculations - The analysis and calculation utilize the ingrowth of Po 210 to determine the original concentration of Pb210.

$$\text{Pb210 (pCi/unit)} = \frac{(S-B_S)}{2.22\ RV_S DMP} \pm \frac{1.96\ \ S/T_S + B/T_B}{2.22\ RV_S\ DMP}$$

where

S =Sample gross cpm in the Po210 energy region.

T_S=Sample count period in minutes.

B_S=Background cpm in the Po210 energy region.

T_B=Background count period in minutes.

V_S=Sample volume.

D =The decay of Po210 from the date and time of plating to the date and time of counting.

$$D = e^{-\lambda t/T_{1/2}} = e^{-\ln 2 t/T_{1/2}}$$

where t= time and date of counting minus time and date of plating

$T_{1/2}$ = half-life of Po210 = 138.4 days.

R = A factor which includes the chemical yield of Po210 and the detector efficiency.

$$R = \frac{C - B_C}{V_F FH}$$

where C = Gross tracer cpm in the Po208 energy region

B_C = Background cpm in the Po208 energy region

F = The standardized concentration of Po208 in dpm/ml

V_F = The volume, in ml, of Po208 tracer used.

H = The decay of Po208 from the time of standardization to the time of sample counting.

$$H = e^{-\lambda t/T_{\frac{1}{2}}} = e^{-\ln 2t/T_{\frac{1}{2}}}$$

where T = The time and date of sample counting minus the time and date of Po208 standardization in days.

$T_{\frac{1}{2}}$ = The half-life of Po208 = 1069.45 days.

M = The lead recovery determined by the ICP analysis for lead.

P = The ingrowth of Po210 from Pb210

$$P = 1 - e^{-\lambda t/T_{\frac{1}{2}}}$$

where t = the time and date of sample plating minus the time and date of beginning of ingrowth.

$T_{\frac{1}{2}}$ = the half-life of Po210 = 138.4 days.

Pb210 Analysis Worksheet.

Laboratory Sample Number: ____________________

Sample Type: ____________________

Date and time of collection: ____________________

Sample aliquot (V_S): ____________________

Po208 internal tracer

Activity (F): ____________________

Date and time of Standardization: ____________________

Amount used (V_F): ____________________

Lead tracer.

Amount of lead added: ____________________ mg.

Times and Dates.

Beginning of Ingrowth: ____________________

Beginning of Plating: ____________________

Beginning of Counting: ____________________

Counting Data.

Instrument used: ____________________

Detector used: ____________________

Po210 Background cpm (B_S): ____________________

Po208 Background cpm (B_C): ____________________

Background count Period (T_B):________________

Po210 Sample cpm (S):________________

Po208 Sample cpm (C):________________

Sample count period (T_S):________________

Lead Analysis:________________mg/liter

Decay and Ingrowth Calculations:

Po210 Decay = D = $e^{-\ln 2\, t/T_{1/2}}$

t = counting date - plating date.

$T_{1/2}$ = half-life of Po210 = 138.4 days.

Calculation:

Po208 Decay = H = $e^{-\ln 2\, t/T_{1/2}}$

t = counting date - standardization date.

$T_{1/2}$ = the half-life of Po208 = 1069.45 days.

Calculation:

Po210 Ingrowth = $P = 1 - e^{\lambda t/T_{1/2}}$

t = time of plating - time of beginning of ingrowth

$T_{1/2}$ = the half-life of Po210 = 138.4 days.

Calculation:

Recovery calculations

Po208 Recovery = $R = \frac{C - B_C}{V_F F H}$

Calculation:

Lead Recovery = mg/l Pb x 0,05 l ÷ lead added in mg.

calculation:

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Calculation of Sample Activity and counting error at the 95% confidence level.

$$\text{Pb210 (pCi/unit)} = \frac{S - B_S}{2.22\ RV_S DMP} \pm \frac{1.96\sqrt{S/T_S + B_S/T_B}}{2.22\ RV_S DMP}$$

Calculation:

Calculation of Lower Limit of Detection

$$\text{LLD} = \frac{4.66\sqrt{B_S/T_B}}{2.22\ R_V SDMP}$$

calculation:

Result:

Pb210 pCi/_____ = ______ pCi/_____ ± ______ pCi/________

LLD pCi ______ = _________ pCi/__________

Analyst ______________ Date ____________

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6.1.A.29.0 RADIOCHEMICAL DETERMINATION OF THORIUM IN ENVIRONMENTAL SAMPLES

A. PURPOSE

To provide an analytical method for the isotopic analysis of alpha emitting thorium in environmental samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine environmental samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure from the manual has been followed without deviation.

D. RECORD KEEPING

All required information shall be entered in the analyst's laboratory notebook and on the Thorium analysis worksheet which shall be attached to the sample analysis request form when complete.

E. SAFETY

Safety glasses required. Rubber gloves to be worn when handling any reagents. Reagents containing xylene to be used in an operating fume hood. Hydrofluoric acid and hydrogen peroxide to be used only when wearing rubber gloves, rubber apron, safety glasses, and full face shield.

F. QUALITY CONTROL

One duplicate analysis, one spike analysis, and one blank to be run with every ten samples in a sample run. If there are fewer than ten samples in a run, one duplicate and one spike are to be run. A recovery spike shall be run with each sample.

G. PROCEDURE

1. Reagents
   a. Ammonium hydroxide - concentrated, reagent.
   b. Ammonium hydroxide (50%) - 50 ml of concentrated NH_4OH diluted to 100 ml with deionized water in a graduated cylinder. Store in a polyethylene wash bottle.
   c. Ascorbic Acid Solution - Approximately 0.5 g of ascorbic acid to 20 ml of deionized water. Make immediately prior to use and discard immediately after use.

d. Aluminum nitrate - crystals, reagent

e. Hydrochloric acid - concentrated, reagent

F. Hydrochloric acid (6N) - Add 250 ml of concentrated hydrochloric acid to 100 ml of deionized water and dilute to 500 ml.

g. Hydrofluoric acid 48% - reagent

h. Hydrogen Peroxide 30% - reagent

i. Iron Carrier (10 mg/ml) - Dissolve 48.27 g of $FeCl_3 \cdot 6H_2O$ in 200 ml of deionized water and dilute to 1 liter. Store in a glass [illegible].

j. Iron Carrier (1 mg/ml) - Dissolve 1.27 g of $FeCl_3 \cdot 6H_2O$ in 200 ml of deionized water and dilute to 250 ml. Store in a glass bottle.

k. Lanthanum Carrier (10 mg/ml) - Dissolve 15.60 g of $La(NO_3)_3 \cdot 6H_2O$ in 400 ml of deionized water and dilute to 500 ml. Store in a glass bottle.

l. Nitric Acid - concentrated, reagent.

m. Potassium Pyrosulfate - crystals, reagent.

n. Thenoyltrifluroacetone (TTA) - Dissolve 25 g of TTA in 200 ml of xylene and dilute to 250 ml with xylene. Store in a glass bottle.

o. Thymol blue indicator (0.1%) - Dissolve 0.100 g of indicator in 50 ml of ethyl alcohol and dilute to 100 ml with ethanol. Store in a glass bottle.

p. Wash Solution - Add 6 ml of concentrated nitric acid and 3 ml of concentrated hydrofluoric acid to 50 ml of deionized water. Dilute to 100 ml. Store in a polyethylene wash bottle.

q. Xylene - reagent.

r. Sulfuric acid - concentrated, reagent.

2. Determination for Water

a. For each sample, transfer two one-liter aliquots to one liter beakers. To each beaker add 20 ml of concentrated nitric acid, 10 mg/ml iron carrier solution and 3 ml of 10 mg/ml lanthanum carrier solution. To one of the beakers add 1 ml of Th-230 tracer solution. Mark this beaker as the recovery spike. Evaporate the samples to approximately 100 ml on a hotplate.

b. Add 10 ml of concentrated nitric acid and 100 ml of water. Cover and heat until the residue has dissolved.

c. Transfer sample to a 250 ml beaker and while swirling, add concentrated ammonium hydroxide, from a burette, to incipient precipitation of the iron (permanent amber color). Increase the volume to about 190 ml by adding deionized water. Transfer the sample equally to two 100 ml centrifuge tubes. To each tube add 8 ml of concentrated ammonium hydroxide while mixing. Allow to stand for one hour and centrifuge at about 1800 rpm. Decant and discard the supernatant liquid.

d. Add 10 ml of concentrated nitric acid to the one liter beaker that contained the sample. Cover, and heat on a hotplate until the acid refluxes to the top of the beaker and turns clear. Cool and wash down the sides of the beaker with about 10 ml of deionized water.

e. Pour the diluted acid from the one liter beaker into the 250 ml beaker in such a way as to wash down the sides of the beaker. Transfer the remaining acid to the 250 ml beaker with several water washes. Transfer from the 250 ml beaker to the two centrifuge tubes, washing down the sides of the tubes, and using several water rinses. Swirl the centrifuge tubes to dissolve the precipitate and dilute to about 50 ml.

f. Add concentrated ammonium hydroxide, from a burette, to incipient precipitation, dilute to about 80 ml, and add 10 ml of concentrated ammonium hydroxide slowly with mixing. Allow to stand one hour and centrifuge at about 1800 rpm. Decant and discard the supernatant liquid.

g. Slurry the precipitate and add water to 100 ml. Centrifuge at about 1800 rpm, decant and discard the wash.

h. Using a pipet, add 3 ml of concentrated nitric acid to each centrifuge tube in such a way that the precipitate adhering to the sides will dissolve. If the precipitate fails to dissolve completely with swirling, add 10 drops of 30% hydrogen peroxide.

i. Transfer the solution and any precipitate of silica from the two centrifuge tubes to a single 50 ml polycarbonate centrifuge tube with deionized water and dilute to about 30 ml.

j. Add 5 ml of 48% hydrofluoric acid, mix, and allow to stand for one hour.

k. Centrifuge at about 1600 rpm for 5 minutes. Decant and discard the supernatant liquid.

l. Add 5 ml of concentrated nitric acid to the precipitate. Disperse the precipitate and pour into a 100 ml glass beaker. Rinse the centrifuge tube with 2 or 3 ml of deionized water and add the washings to the beaker.

m. To the beaker add 0.3-0.4 g of $Al(NO_3)_3 \cdot 9H_2O$ and heat with stirring until the precipitate dissolves.

n. Slowly add concentrated ammonium hydroxide until no further precipitation occurs. Centrifuge and decant the supernatant and discard.

o. Add 5 ml of concentrated nitric acid to the precipitate and stir until dissolved.

p. Add one ml of ascorbic acid solution, add one drop of thymol blue indicator solution. Adjust the pH visually to the range pH 2.0 - pH 4.0 (golden color) with 50% ammonium hydroxide and 2N nitric acid.

q. Transfer the sample to a 125 ml separatory funnel with a wash solution adjusted to pH 1.5 (adjust 0.1N nitric acid to pH 1.5 with ammonium hydroxide using a pH meter). Use enough wash solution to end up with a total volume of 15 - 20 ml.

r. Add 20 ml of 10% TTA in xylene and shake vigorously for 35 minutes. Discard the aqueous layer.

s. Add 5 ml of 0.2N nitric acid and stir for 5 minutes. Discard the aqueous layer. Repeat.

t. Transfer the TTA layer to a 50 ml beaker. Rinse the separatory funnel with 3 ml of xylene and add to the beaker.

u. Allow the TTA/xylene to evaporate to dryness.

v. Place the sample in a muffle furnace at 550°C for 15-20 minutes. Allow to cool and remove from furnace.

w. Prepare an electroplating cell.

x. Add 10 ml of electroplating solution to the beaker and police thoroughly with a rubber policeman.

y. Rinse the beaker and policeman with additional electroplating solution. Transfer the rinses to the electroplating cell.

z. Add 4-6 drops of 6N hydrochloric acid to the electroplating cell and electroplate for 2 hours at 15-25 volts and 210 milliamps.

aa. When electroplating is complete add one drop of phenolphthalein indicator to the cell and add 50% ammonium hydroxide until the solution turns red. Turn off the current, remove the cell and discard the liquid. Disassemble the cell and rinse the plating disk with alcohol. Allow to dry.

bb. Flame the plating disk in a Fisher burner until red. Cool.

cc. Count the sample and the recovery spike in the alpha spectrometer. If possible use the same detector for both the sample and the recovery spike.

dd. Calculate as shown in the calculation section of the procedure manual.

3. Determination for Soil, Sediment, and Vegetation

   a. Weigh out 1 g of dried and ashed sample for each sample and 1 g for each recovery spike.

   Caution: particularly with vegetation, only small amounts of ashed sample may be available. Make sure that sufficient sample remains to perform all required analyses. This may make necessary the use of aliquots smaller than 1 g.

b. Transfer sample to a 100 ml Teflon beaker, add 1 ml of Th 230 spike solution to the recovery spike, evaporate twice to dryness with 15 ml portions of concentrated hydrochloric acid.

c. Dry in an oven at 100°C for 2 hours to dehydrate silica.

d. Add 2 ml concentrated hydrochloric acid and 20 ml of water and heat while covered for 30 minutes. Decant the liquid into a 50 ml centrifuge tube. Centrifuge at 2000 rpm for 5-10 minutes. Decant supernatant liquid into a 250 ml bottle and transfer the residue to the Teflon beaker.

e. Add 15 ml of 48% hydrofluoric acid and 10 ml of concentrated hydrochloric acid to the residue and evaporate to dryness on a hotplate. Remove fluoride by three successive evaporations to dryness with 5 ml portions of 6N hydrochloric acid.

f. Add 2 ml of concentrated hydrochloric acid and 20 ml of water. Cover and heat for 30 minutes and then transfer to the same 50 ml centrifuge tube used in step d. Centrifuge and decant supernatant liquid into the bottle containing the first dissolved portion. Transfer the remaining residue. Dry under a heat lamp.

g. Add one gram of potassium pyrosulfate to the platinum dish and fuse over a burner. Cool, add 3 ml of concentrated hydrochloric acid and 2-5 ml of water, and heat until residue has dissolved. Combine with the other dissolved portions.

h. Add one ml of 10 µg/ml iron carrier per gram of sample. If less than 1 gram of sample was used, add an additional 1 ml of iron carrier for each 100 mg of weight below one gram. Proceed with the hyroxide separation of thorium at step c of the determination for water.

4. Determination of Airborne Dust on Glass Fiber Filters

   a. Take a representative portion of the air filter in accordance with the procedure for representative portion of air filters.

   b. Proceed with step a of the determintation for soils and sediments.

F. CALCULATION

The calculation of Th-230 or other alpha emitting thorium concentration is made using the following expression:

$$\text{Th(pCi/unit)} = \frac{(S - B)}{2.22(E)(Y)(V_s)}$$

where

S = Sample counts per minute = $\frac{\text{sample counts}}{\text{Time}}$

B = Background counts per minute = $\frac{\text{Background counts}}{\text{Time}}$

E = Detector Efficiency

Y = Chemical Yield = $\frac{\text{spike cpm} - \text{sample cpm}}{(\text{spike dpm})(E)}$

V_s = Sample size in liters, grams, etc.

ISOTOPIC THORIUM WORKSHEET

Laboratory Sample No.: ____________

Sample Type: ____________

Sample Aliquot = Spike Aliquot: ____________

Tracer Soution No.: ____________

Tracer concentration: ____________

Tracer amount: ____________

Instrument data:

Detector Used: ____________

*Detector background: ________ cpm Background count time: ________ minutes

*Sample Gross cpm: ________ cpm Sample count time: ________ minutes

*Spike Gross cpm: ________ cpm Spike count time: ________ minutes

* The background, sample, and spike counts used are those for the peak of interest for the particular isotope being analyzed. See alpha spectrometer procedure manual for details on determining.

Calulation of chemical yield

$$Y = \frac{\text{spike cpm} - \text{sample cpm}}{(\text{dpm spike added})(E)} =$$

Calculation of result and counting error are at the 95% confidence level.

$$\text{Th(pCi/Unit)} = \frac{(S - B)}{2.22\ EY\ V_s} \pm \frac{1.96\sqrt{S/T_S + B/T_B}}{2.22\ EY\ V_s}$$

Calculation:

Note: The expression $e^{-\lambda t/T_{\frac{1}{2}}}$ normally found in this equation is ignored due to the long half lives of the Thorium isotopes.

Calculation of Lower Limit of Detection (LLD):

$$\text{LLD (pCi/Unit)} = \frac{4.66\sqrt{B/T_B}}{2.22\ EY\ V_s}$$

Calculation:

Result:

Isotope: __________ = ________pCi/____ ± _________pCi/________

LLD = _________pCi/ _______

Analyst ______________________ date ____________________

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Date Issued April 1, 1981

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APPROVED

6.1.A.30.0 DETERMINATION OF CHLORIDE IN WATER SAMPLES

A. PURPOSE

To provide an analytical method for determination of chloride in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Mercuric nitrate is poisonous, use only when wearing rubber gloves.

E. APPARATUS

Standard laboratory titrimetric equipment including a 1 ml or 5 ml microburet with 0.01 ml graduations.

F. REAGENTS

1. Standard sodium chloride, 0.025 N: Dissolve 1.4613 ± 0.0002 g (dried at 600°C for 1 hour) in chloride free water in a 1 liter volumetric flask and dilute to the mark.
2. Nitric acid, HNO_3 solution (3 + 997).
3. Sodium hydroxide solution, NaOH, (10 g/1).
4. Hydrogen peroxide (30%), H_2O_2.
5. Hydroquinone solution (10 g/liter): Dissolve 1 g of purified hydroquinone in water in a 100 ml volumetric and dilute to the mark.
6. Mercuric nitrate titrant (0.141 N): Dissolve 25 g $Hg(NO_3) \cdot H_2O$ in 900 ml of distilled water acidified with 5.0 ml conc. HNO_3 in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (F.1) using procedure 6. Adjust to exactly 0.141 N and check. Store in a dark bottle. A 1.00 ml aliquot is equivalent to 5.00 mg of chloride.

7. Mercuric nitrate titrant (0.025 N): Dissolve 4.2830 g $Hg(NO_3)_2 \cdot H_2O$ in 50 ml of distilled water acidified with 0.5 ml conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (1) using procedure 6. Adjust to exactly 0.025 N and check. Store in a dark bottle.

8. Mercuric nitrate titrant (p.0141 N): Dissolve 2.4200 g $Hg(NO_3)_2 \cdot H_2O$ in 25 ml of distilled water acidified with 0.25 ml of conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (1) using procedure 6. Adjust to exactly 0.014 N and check. Store in a dark bottle. A 1 ml aliquot is equivalent to 500 ug of chloride.

9. Mixed indicator reagent: Dissolve 0.5 g crystalline disphenylcarbazone and 0.05 g bromophenol blue powder in 75 ml 95% ethanol in a 100 ml volumetric flask and dilute to the mark with 95% ethanol. Store in brown bottle and discard after 6 months.

10. Alphazurine indicator solution: Dissolve 0.005 g of alphazurine blue-green dye in 95% ethanol or isopropanol in a 100 ml volumetric and dilute to the mark with 95% ethanol or isopropanol.

G. DETERMINATION

1. Place 50 ml of sample in a vessel for titration. If the concentration is greater than 20 mg/l chloride, use 0.141 N mercuric nitrate titrant (F.6) in step 6 or dilute. If the concentration is less than 2.5 mg/l of chloride, use 0.0141 N mercuric nitrate titrant (8) in step 6, a 1 ml or 5 ml microburet, and determine an indicator blank on 50 ml chloride-free water using procedure 6. If the concentration is less than 0.1 mg/l of chloride, concentrate an appropriate volume to 50 ml.
2. Add 5 to 10 drops of mixed indicator reagent (9), shake or swirl solution.
3. If a blue-violet or red color appears, add HNO_3 solution (2) dropwise until the color changes to yellow.
4. If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (3) dropwise until the color changes to blue-violet; than add HNO_3 solution (2) dropwise until the color changes to yellow.

5. Add 1 ml excess HNO_3 solution (2).

6. Titrate with 0.025 N mercuric nitrate titrant (7) until a blue-violet color persists throughout the solution. See 1 for choice of titrant normality. Alphazurine indicator solution (10) may be added with the indicator to sharpen the end point. This will change color shades. Practice runs should be made.

7. Additional steps to eliminate particular interferences:
   a. If chromate is present at <100 mg/l and iron is not present, add some alphazurine indicator solution (10) and acidify to pH 3 (indicating paper). End point will then be an olive-purple color.
   b. If chromate is present at >100 mg/l and iron is not present, add 2 ml of fresh hydroquinone solution (5).
   c. If ferric ion is present use volume containing no more than 2.5 mg of ferric ion or ferric ion plus chromate ion. Add 2 ml fresh hydroquinone solution (5).
   d. If sulfite ion is present, add 0.5 ml of H_2O_2 solution (4) to 50 ml sample and mix for 1 minute.

H. CALCULATION

$$\text{mg chloride/l} = \frac{(A - B)N \times 35{,}450}{\text{ml of sample}}$$

where:

A - ml titrant for sample

B = ml titrant for blank

N = normality mercuric nitrate titrant

mg NaCl/l = mg chloride/l X 1.65

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6.1.A.31.0 DETERMINATION OF TOTAL CYANIDE IN WATER SAMPLES

A. PURPOSE

To provide an analytical method for cyanide in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Due to the extreme hazard presented by the use of cyanide, the procedure will be performed only by the Environmental Chemist. The cyanide first aid kit shall be immediately available and at least one individual not present in the laboratory shall be notified prior to performance of the analysis. All portions of the analysis except the spectrophotometric reading shall be performed inside a properly operating fume hood.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. Interferences are eliminated or reduced by using the distillation procedure described in Procedure I.1 through I.5.
2. Sulfides adversely affect the colorimetric and titration procedures. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 ml more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution

does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide.

3. Fatty acids will distill and form soaps under the alkaline titration conditions, making the end point almost impossible to detect. Correct in the following manner:
   a. Acidify the sample with acetic acid (1 + 9) to pH 6.0 to 7.0.

      Caution: This operation must be performed in the hood and the sample left there until it can be made alkaline again after the extraction has been performed.
   b. Extract with iso-octane, hexane, or chloroform (preference in order named) with a solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the fatty acids below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When

the extraction is completed, immediately raise the pH of the sample to above 12 with NaOH solution.

G. APPARATUS

1. Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber.
2. Microburet, 5.0 ml (for titration).
3. Spectrophotometer suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger.

H. REAGENTS

1. Sodium hydroxide solution 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.
2. Cadmium carbonate: powdered.
3. Ascorbic acid: crystals
4. Dilute sodium hydroxide solution, 0.25N: Dilute 200 ml of sodium hydroxide solution to 1000 ml with distilled water.
5. Sulfuric acid: concentrated.
6. Sodium dihydrogenphosphate, 1 M: Dissove 148 g of NaH_2PO. H_2O in 1 liter of distilled water. Refrigerate this solution.
7. Stock cyanide solution: Dissolve 2.5 g of KCN and 2 g KOH

in 1 liter of of distilled water. Standardize with 0.0192 N $AgNO_3$. Dilute to appropriate concentration so that 1 ml = 1 mg CN.

8. Standard cyanide solution, intermediate: Dilute 50.0 ml of stock (1 ml = 1 mg CN) to 1000 ml with distilled water (1 ml = 50.0µg).
9. Standard cyanide solution: Prepare fresh daily by diluting 100.0 ml of intermediate cyanide solution to 1000 ml with distilled water and store in a glass stoppered bottle. 1 ml = 5.0µg CN (5.0 mg/l).
10. Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g $AgNO_3$ crystals and drying to constant weight at 40°C. Weigh out 3.2647 g of dried $AgNO_3$, dissolve in distilled water, and dilute to 1000 ml (1 ml = 1 mg CN).
11. Rhodanine indicator: Dissolve 20 mg of p-dimethyl-aminobenzalrhodanine in 100 ml of acetone.
12. Chloramine T solution: Dissolve 1.0 g of white, water soluble Chloramine T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh weekly.
13. Color Reagent - One of the following may be used:
    a. Pyridine-Barbituric Acid Reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just

enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of HCI (sp gr 1.19), mix and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

b. Pyridine-pyrazolone solution:

   1. 3-Methyl-1-phenyl-2-pyrazolin-5-one reagent, saturated solution: Add 0.25 g of 3-methyl-1-phenyl-2-pyrazolin-5-one to 50 ml of distilled water, heat to 60°C with stirring. Cool to room temperature.
   2. 3,3'Dimethyl-1'-diphenyl-[4,4'-bi-2 pyrazoline]-5-5'dione (bispyrazolone): Dissolve 0.01 g of bispyrazolone in 10 ml of pyridine.
   3. Pour solution (H.13.b.1) through non-acid-washed filter paper. Collect the filtrate. Through the same filter paper pour solution (H.13.b.2) collecting the filterate in the same container as filtrate from (H.13.b.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color but this does not affect the color production with cyanide if used within 24 hours of preparation.

14. Magnesium chloride solution: Weight 510 g of $MgCl_2 \cdot 6H_2O$ into a 100 ml flask, dissolve and dilute to 1 liter with distilled water.

I. DETERMINATION

1. Place 500 ml of sample or an aliquot diluted to 500 ml in the 1 liter boiling flask. Add 50 ml of sodium hydroxide (H.1) to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.
2. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

   Caution: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

3. Slowly add 25 ml conc. sulfuric acid (H.5) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 min. Pour 20 ml of magnesium chloride (H.4) into the air inlet and wash down with a stream of water.
4. Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.
5. Drain the solution from the absorber into a 250 ml volumetric flask and bring up to volume with distilled water washings from the absorber tube.
6. Withdraw 50 ml or less of the solution from the flask and transfer to a 100 ml volumetric flask. If less than 50 ml is taken, dilute to 50 ml with 0.25 n sodium hydroxide solution (H.4). Add 15.0 ml of Sodium phosphate solution (H.6) and mix.
    a. Pyridine-Barbituric Acid Method: Add 2 ml of chloramine T (H.12) and mix. After 1 to 2 minutes, add 5 ml of pyridine-bartituric acid solution (H.13.a) and mix.

Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

b. Pyridine-pyrazolone method: Add 0.5 ml of chloramine T (H.12) and mix. After 1 to 2 minutes add 5 ml of pyridine-pyrazolone solution (H.13.a) and mix. Dilute to mark with distilled water and mix again. After 40 minutes read absorbance at 620 nm in a 1 cm cell.

NOTE: More than 0.5 ml of Chloramine T will prevent the color from developing with pyridine-pyrazolone.

7. Prepare a series of standards by pipeting suitable volumes of standard solution into 250 ml volumetric flasks. To each standard add 50 ml of 1.25 sodium hydroxide and dilute to 250 ml with distilled water. Prepare as follows:

ML of Standard Solution (1.0 = 5 ug CN)	Conc. mgCN per 250 ml
0	BLANK
1.0	5
2.0	10
5.0	25
10.0	50
15.0	60
20.0	100

a. It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree within ± 10% of the undistilled standards the operator should find the cause of the apparent error before proceeding.

b. Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

c. To check the efficiency of the sample distillation add an increment of cyanide from either the intermediate standard (H.8) or the working standard (H.9) to insure a level of 200 ug/l or a significant increase in absorbance value. Proceed with the analysis as in Procedure (I.8.1) using the same flask and system from which the previous sample was distilled.

8. Alternatively, if the sample contains more than 1 mg of CN transfer the distillate, or a suitable aliquot diluted to 250 ml, to a 500 ml Erlenmeyer flask. Add 10-12 drops of the benzalrhodanine indicator.

9. Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.
10. The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 or 10 ml micro-buret may be conveniently used to obtain a more precise titration.

J. CALCULATION

If the colorimetric procedure is used, calculate the cyanide, in ug/l, in the original sample as follows:

$$\text{CN, } \mu g/l = \frac{A \times 1{,}000}{B} \times \frac{50}{C}$$

where:

A = ug CN read from standard curve

B = ml of original sample for distillation

C = ml taken for colorimetric analysis

Using the titrimetric procedure, calculate concentrations of CN as follows:

$$\text{CN, mg/l} = \frac{(A - B)1{,}000}{\text{ml orig. sample}} \times \frac{250}{\text{ml of aliquot titrated}}$$

where:

A = volume of $AgNO_3$ for titration of sample.

B = volume of $AgNO_3$ for titration of blank.

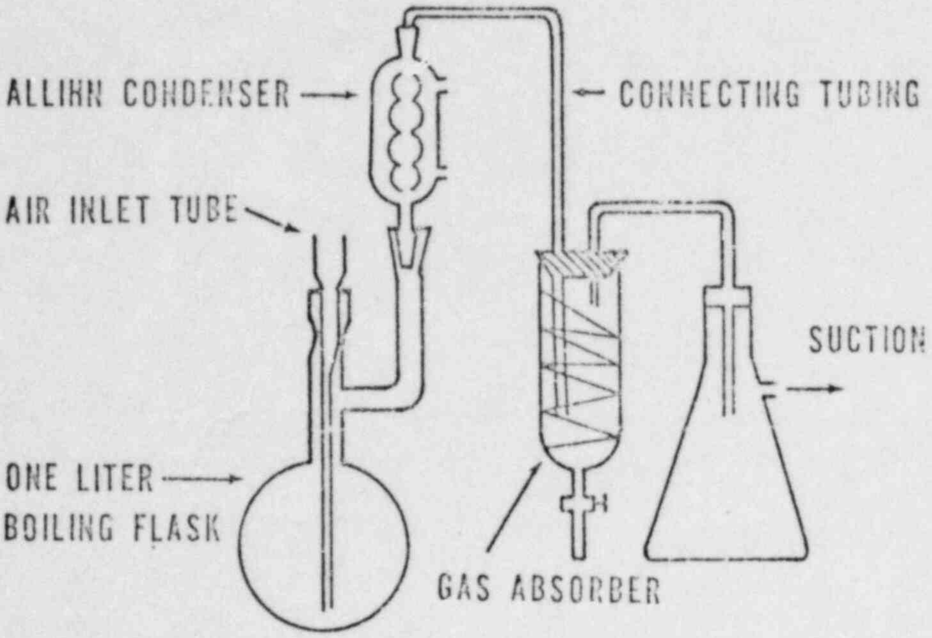

FIGURE 1

CYANIDE DISTILLATION APPARATUS

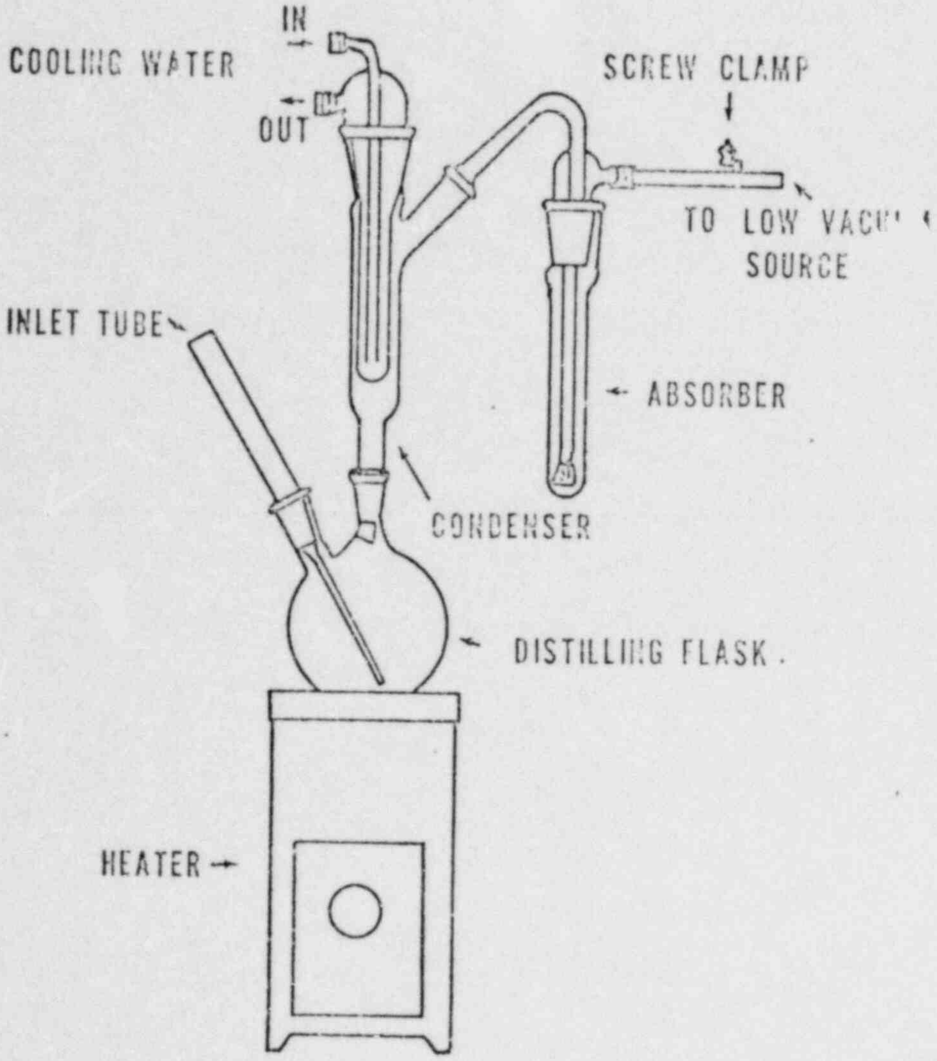

FIGURE 2

CYANIDE DISTILLATION APPARATUS

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6.1.A.32.0 POTENTIOMETRIC DETERMINATION OF FLUORIDE IN WATER

A. PURPOSE

To provide an analytical method for fluoride in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

Extremes of pH interfere; sample pH should be between 5 and 9. Polyvalent cations of Si^{+4}, Fe^{+3}, and Al^{+3} interfere by forming complexes with fluoride. The degree of interference depends upon the concentration of fluoride and the pH of the sample. The addition of a pH 5.0 buffer (described below) containing a strong chelating agent preferentially complexes aluminum (the most common interference), silicon and iron and eliminates the pH problem.

G. APPARATUS

1. Electrometer (pH meter), with expanded mv scale, or a selective ion meter.
2. Fluoride Ion Activity Electrode, such as Orion No. 94-09 or equivalent.
3. Reference electrode, single junction, sleeve-type such as Orion No. 90-01 or equivalent.
4. Magnetic Mixer, Teflon-coated stirring bar.

H. REAGENTS

1. Buffer solution, pH 5.0-5.5: To approximately 500 ml of distilled water in a 1 liter beaker add 57 ml of glacial acetic acid, 58 g of sodium chloride and 2 g of CDTA. Stir to dissolve and cool to room temperature. Adjust pH of solution to between 5.0 and 5.5 with 5 N sodium hydroxide (about 150 ml will be required). Transfer solution to a 1 liter volumetric flask and dilute to the mark with distilled water. For work with brines, additional NaCl should be added to raise the chloride level to twice the highest expected level of chloride in the sample.
2. Sodium fluoride, stock solution: 1.0 ml = 0.1 mg F. Dissolve 0.2210 g of sodium fluoride in distilled water and dilute to 1 liter in a volumetric flask. Store in chemical-resistant glass or polyethylene.
3. Sodium fluoride, standard solution: 1.0 ml = 0.01 mg F. Dilute 100.0 ml of sodium fluoride stock solution (H.2) to 1000 ml with distilled water.
4. Sodium hydroxide, 5N: Dissolve 200 g sodium hydroxide in distilled water, cool and dilute to 1 liter.

I. CALIBRATION

1. Prepare a series of standards using the fluoride standard solution (H.3) in the range of 0 to 2.00 mg/l by diluting appropriate volume to 50.0 ml. The following series may be used.

Millimeter of Standard (1.0 ml = 0.01 mg/F)	Concentration when Diluted to 50 ml. mg F/liter
0.00	0.00
1.00	0.20
2.00	0.40
3.00	0.60
4.00	0.80
5.00	1.00
6.00	1.20
8.00	1.60
10.00	2.00

2. Calibration of Electrometer: Proceed as described in (I.1). Using semilogarithmic graph paper, plot the concentration of fluoride in mg/liter on the log axis vs. the electrode potential developed in the standard on the linear axis, starting with the lowest concentration at the bottom of the scale. Calibration of a selective ion meter: Follow the directions of the manufacturer for the operation of the instrument.

J. DETERMINATION

Place 50.0 ml sample or standard solution and 50.0 ml of buffer (See Note) in a 150 ml beaker. Place on a magnetic stirrer and mix at medium speed. Immerse the electrodes in the solution and observe the meter reading while mixing. The electrodes must remain in the solution for at least three minutes or until the reading has stabilized. At concentrations under 0.5 mg/liter F, it may require as long as five minutes to reach a stable meter reading; high concentrations stabilize more quickly. If a pH meter is used, record the potential measurement for each unknown sample and convert the potential reading to the fluoride ion concentration of the unknown using the standard curve. If a selective ion meter is used, read the fluoride level in the unknown sample directly in mg/l on the fluoride scale.

NOTE: For industrial waste samples, this amount of buffer may not be adequate. Analyst should check pH first. If highly basic (>9), add 1 N HCl to adjust pH to 8.3.

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6.1.A.33.0 COLORIMETRIC DETERMINATION OF FLUORIDE IN WATER

A. PURPOSE

To provide an analytical method for fluoride in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. APPARATUS

1. Distillation apparatus: A 1-liter round-bottom, long-necked pyrex boiling flask, connecting tube, efficient condenser, thermometer adapter and thermometer reading to 200°C. All connections should be ground glass. Any apparatus equivalent to that shown in Figure 1 is acceptable.
2. Colorimeter: Spectrophotometer for use at 570 nm providing a light path of at least 1 cm.

G. REAGENTS

1. Sulfuric acid, H_2SO_4, conc.
2. Silver sulfate, Ag_2SO_4 crystals.
3. Stock fluoride solution: Dissolve 0.221 g anhydrous sodium fluoride, NaF, in distilled water in a 1-liter volumetric flask and dilute to the mark with distilled water; 1.00 ml = 0.1 mg F.

4. Standard fluoride solution: Place 100 ml stock fluoride solution (5.3) in a 1 liter volumetric flask and dilute to the mark with distilled water; 1.00 ml = 0.010 mg F.
5. SPADNS solution: Dissolve 0.958 g SPADNS, sodium 2-(parasulfophenylazo)-1.8-dihydroxy-3,6-naphtalene disulfonate, in distilled water in a 500 ml volumetric flask and dilute to the mark. Stable indefinitely if protected from direct sunlight.
6. Zirconyl-acid reagent: Dissolve 0.133 g zirconyl chloride octahydrate $ZrOCl_2 \cdot 8H_2O$ in approximately 25 ml distilled water in a 500 ml volumetric flask. Add 350 ml conc HCl and dilute to the mark with distilled water.
7. Acid-zirconyl-SPADNS reagent: Mix equal volume of SPADNS solution (G.5) and zirconyl-acid reagent (G.6). The combined reagent is stable for at least 2 years.
8. Reference solution: Add 10 ml SPADNS solution (G.5) to 10 ml distilled water. Dilute 7 ml conc. HCl to 10 ml and add to the dilute SPADNS solution. This solution is used for zeroing the spectrophotometer or photometer. It is stable and may be used indefinitely.
9. Sodium arsenite solution: Dissolve 5.0 g $NaAsO_2$ in distilled water in a 1-liter volumetric flask and dilute to the mark with distilled water (CAUTION· Toxic-avoid ingestion).

H. DETERMINATION

1. Preliminary Distillation

   a. Place 400 ml distilled water in the distilling flask.

   b. Carefully add 200 ml conc. H_2SO_4 and swirl until contents are homogenous.

   c. Add 25 to 35 glass beads, connect the apparatus (Figure 1) making sure all joints are tight.

   d. Heat slowly at first, then as rapidly as the efficiency of the condenser will permit (distillate must be cool) until the temperature of the flask contents reaches exactly $180^{\circ}C$. Discard the distillate. This process removes fluoride contamination and adjusts the acid-water ratio for subsequent distillations.

   e. Cool to $120^{\circ}C$ or below.

   f. Add 300 ml sample, mix thoroughly, distill as in step d until temperature reaches $180^{\circ}C$. Do not heat above $180^{\circ}C$ to prevent sulfate carryover.

   g. Add Ag_2SO_4 (G.2) at a rate of 5 mg/mg Cl when high chloride samples are distilled.

   h. Use the sulfuric acid solution in the flask repeatedly until the contaminants from the samples accumulate to such an extent that recovery is affected or interferences appear in the distillate. Check periodically by distilling standard fluoride samples.

   i. High fluoride samples may require that the still be flushed by using distilled water and combining distillates.

2. Colorimetric Determination:

   a. Prepare fluoride standards in the range 0 to 1.40 mg/l by diluting appropriate quantities of standard fluoride solution to 50 ml with distilled water.

   b. Pipet 5.00 ml each of SPADNS solution (G.5) and zirconyl-acid reagent (G 6) or 10.00 ml of the mixed acid-zirconyl-SPADNS reagent (G.7) to each standard and mix well.

   c. Set photometer to zero with reference solution (G.8) and immediately obtain absorbance readings of standards.

   d. Plot absorbance versus concentration. Prepare a new standard curve whenever fresh reagent is made.

   e. If residual chlorine is present, pretreat sample with 1 drop of $NaAsO_2$ solution(G.9) per 0.1 mg residual chlorine and mix. Sodium arsenite concentrations of 1300 mg/l produce an error of 0.1 mg/l at 1.0 mg/F.

   f. Use a 50 ml sample or a portion diluted to 50 ml. Adjust the temperature of the sample to that used for the standard curve.

   g. Perform steps b and c.

I. CALCULATIONS

Read the concentration in the 50 ml sample using the standard curve.

Calculate as follows:

$$\text{mg/l F} = \frac{\text{mgF} \times 1{,}000}{\text{ml sample}}$$

When a sample (ml sample) is diluted to a volume (B) and then a portion (C) is analyzed use:

$$\text{mg/lF} = \frac{\text{mgF} \times 1{,}000}{\text{ml sample}} \times \frac{B}{C}$$

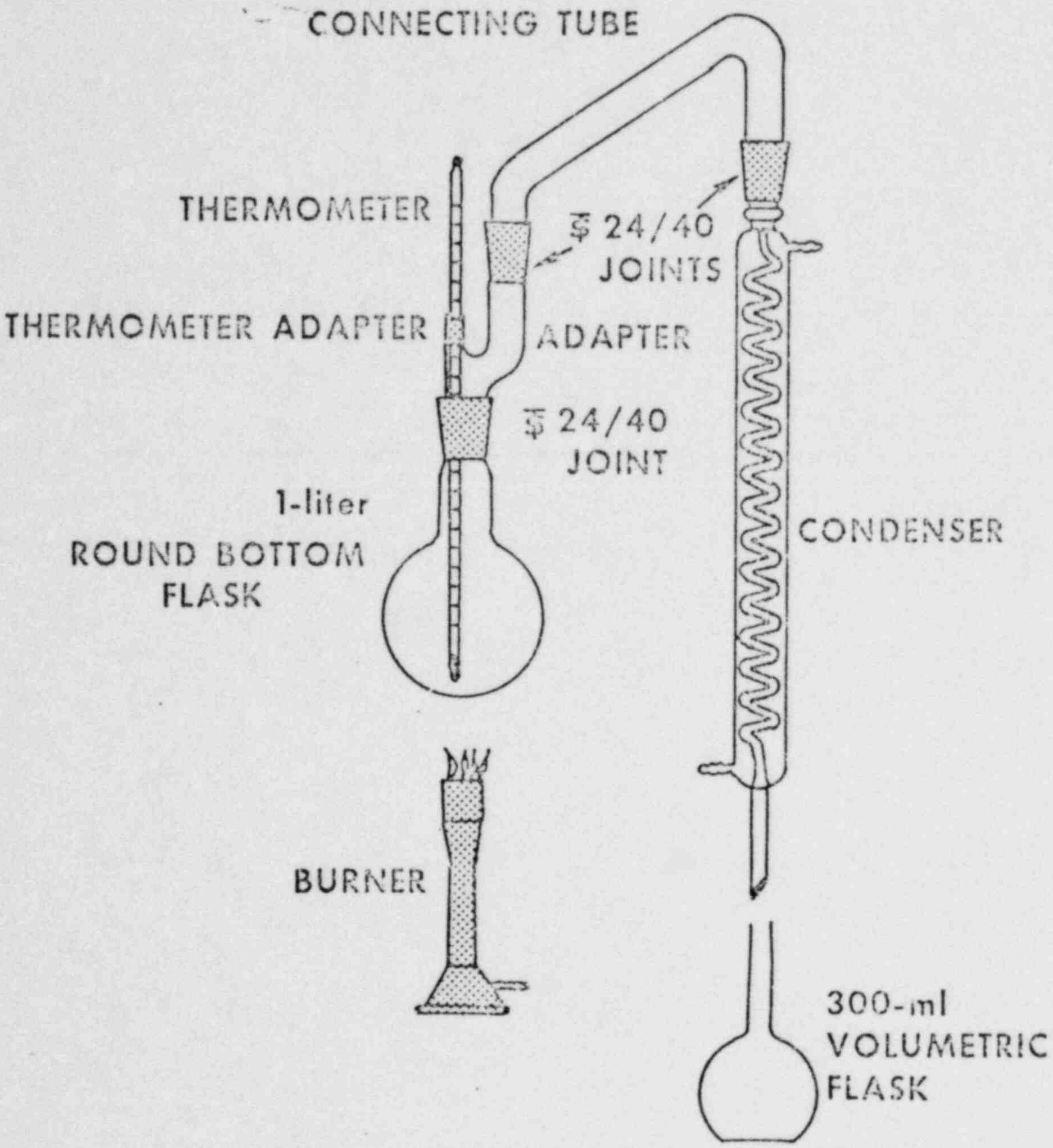

FIGURE 1 DIRECT DISTILLATION APPARATUS FOR FLUORIDE.

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APPROVED

6.1.A.34.0 DETERMINATION OF AMMONIA NITROGEN IN WATER

A. PURPOSE

To provide an analytical method for ammonia nitrogen in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

A number of aromatic and aliphatic amines, as well as other compounds both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, so direct nesslerization (i.e. without distillation) has been discarded as an official method.

Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile alkaline compounds such as certain ketones, aldehydes, and alcohols, may cause an off-color upon nesslerization in the distillation methods. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation and nesslerization.

Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

G. APPARATUS

1. An all-glass distilling apparatus with an 800-1000 ml flask.
2. Spectrophotometer or filter photometer for use at 425 nm and providing a light path of 1 cm or more.
3. Nessler tubes: Matched Nessler tubes (ALPHA Standard) about 300 mm long, 17 mm inside diameter, and marked at 225 mm ± 1.5 mm inside measurement from bottom.
4. Erlenmeyer flask: The distillate is collected in 500 ml glass-stoppered flasks. These flasks should be marked at the 350 and the 500 ml volumes. With such marking, it is not necessary to transfer the distillate to volumetric flasks.

H. REAGENTS

1. Distilled water should be free of ammonia. Such water is best prepared by passage through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.

   NOTE 1: All solutions must be made with ammonia-free water.
2. Ammonium chloride, stock solutions: 1.0 ml = 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1 liter volumetric flask.

3. Ammonium chloride, standard solution: 1.0 ml = 0.01 mg. Dilute 10.0 ml of stock solution (H.2) to 1 liter in a volumetric flask.

4. Boric acid solution (20 g/l): Dissolve 20 g H_3BO_3 in distilled water and dilute to 1 liter.

5. Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethyl alcohol with 1 volume of 0.2% methylene blue in 95% ethyl alcohol. This solution should be prepared fresh every 30 days.

   NOTE 2: Specially denatured ethyl alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue may be substituted for 95% alcohol.

6. Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 ml of water. Dilute the mixture to 1 liter. If this reagent is stored in Pyrex bottle out of direct sunlight, it will remain stable for a period of up to 1 year.

   NOTE 3: This reagent should give the characteristic color with ammonia within 10 minutes after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50 ml volume).

7. Borate buffer: Add 88 ml of 0.1 N NaOH solution to 500 ml of 0.025 M sodium tetraborate solution (5.0 g anhydrous $Na_2B_4O_7$ or 9.5 g $Na_2B_4O_7 \cdot 10H_2O$ per liter) and dilute to 1 liter.

8. Sulfuric acid, standard solution: (0.02 N, 1 ml = 0.28 mg NH_3-N) Prepare a stock solution of approximately 0.1 N acid by dilution 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2-free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2-free distilled water.

   NOTE 4: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 solution against a 0.100 N Na_2CO_3 solution. By proper dilution the 0.02 N acid can then be prepared.

   a. Standardize the approximately 0.02 N acid against the 0.200 N. Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g anhydrous Na_2CO_3, oven dried at 140°C and diluting to 1000 ml with CO_2-free distilled water.

9. Sodium hydroxide, 1 N: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 liter.

10. Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distllation. These include:

    a. Sodium thiosulfate (1/70 N): Dissolve 3.5 g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1 liter. One ml of this solution will remove 1 mg/l of residual chlorine in 500 ml of sample.

b. Soduim arsenite (1/70 N): Dissolve 1.0 g $NaAsO_2$ in distilled water and dilute to 1 liter.

I. DETERMINATION

1. Preparation of equipment: Add 500 ml of distilled water to an 800 ml Kjeldahl flask. The addition of boiling chips which have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia with Nessler reagent.
2. Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 400 ml of sample add 1 N NaOH (H.9), until the pH is 9.5, checking the pH during addition with a pH meter or by use of a short range pH paper.
3. Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 800 ml Kjeldahl flask and add 25 ml of the borate buffer (H.7). Distill 300 ml at the rate of 6-10 ml/min. into 50 ml of 2% boric acid (H.4) contained in a 500 ml Erlenmeyer flask.

   NOTE 5: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.

Dilute the distillate to 500 ml with distilled water and nesslerize an aliquot to obtain an approximate value of the ammonia-nitrogen concentration. For concentrations above 1 mg/l the ammonia should be determined titrimetrically. For concentrations below this value it is determined colorimetrically. The electrode method may also be used.

4. Determination of ammonia in distillate: Determine the ammonia content of the distillate titrimetrically, colorimetrically or potentiometrically as described below.
   a. Titrimetric determination: Add 3 drops of the mixed indicator to the distillate and titrate the ammonia with the 0.02 N H_2SO_4, matching the end point against a blank containing the same volume of distilled water and H_3BO_3 solution.
   b. Colormetric determination: Prepare a series of Nessler tube standards as follows:

ml of Standard 1.0 ml = 0.01 mg NH_3-N	mg NH_3-N/50.0 ml
0.0	0.0
0.5	0.005
1.0	0.01
2.0	0.02
3.0	0.03
4.0	0.04
5.0	0.05
8.0	0.08
10.0	0.10

Dilute each tube to 50 ml with distilled water, add 2.0 ml of Nessler reagent (H.6) and mix. After 20 minutes read the absorbance at 425 nm against the blank. From the values obtained plot absorbance vs mg NH_3-N for the standard curve. Determine the ammonia in the distillate by nesslerizing 50 ml or an aliquot diluted to 50 ml and reading the absorbance at 425 nm as described above for the standards. Ammonia-nitrogen content is read from the standard curve.

c. Potentiometric determination: Consult the method entitled Nirogen, Ammonia: Selective Ion Electrode Method.

5. It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree with undistilled standards the operator should find the cause of the apparent error before proceeding.

J. CALCULATIONS

Titrimetric

$$\text{mg/l } NH_3 - N = \frac{A \times 0.28 \times 1{,}000}{S}$$

where

A = ml 0.02 N H_2SO_4 used

S = ml sample

Spectrophotometric

$$\text{mg/l } NH_3 - N = \frac{A \times 1{,}000}{D} \times \frac{B}{C}$$

where

A = mg NH_3-N read from standard curve.

B = ml total distillate collected, including boric acid and dilution.

C = ml distillate taken for nesslerization

D = ml of original sample taken.

Potentiometric

$$\text{mg/}NH_3 - N = \frac{500}{D} \times A$$

where

A = mg NH_3-N/l from electrode method standard curve.

D = ml of original sample taken.

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Date Issued April 1, 1981

Supersedes New

APPROVED

6.1.A.35.0 COLORIMETRIC DETERMINATION OF NITRATE NITROGEN IN WATER

A. PURPOSE

To provide an analytical method for nitrate nitrogen in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Brucine Sulfate is toxic.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. Dissolved organic matter will cause an off color in 13 N H_2SO_4 and must be compensated for by additions of all reagents except the brucine-sulfanilic acid reagent. This also applies to natural color present not due to dissolved organics.
2. The effect of salinity is eliminated by addition of sodium chloride to the blanks, standards and samples.
3. All strong oxidizing or reducing agents interfere. The presence of oxidizing agents may be determined with a total residual chlorine test kit.
4. Residual chlorine interference is eliminated by the addition of sodium arsenite.
5. Ferrous and ferric iron and quadrivalent manganese give slight positive interferences, but in concentrations less than 1 mg/l these are negligible.

6. Uneven heating of the samples and standards during the reaction time will result in erratic values. The necessity for <u>absolute control</u> of temperature during the critical color development period cannot be too strongly emphasized.

G. APPARATUS

1. Spectrophotometer suitable for measuring absorbance at 410 nm.
2. Sufficient number of 40-50 ml glass sample tubes for reagent blanks, standards and samples.
3. Neoprene coated wire racks to hold sample tubes.
4. Water bath suitable for use at 100°C. This bath should contain a stirring mechanism so that all tubes are at the same temperature and should be of sufficient capacity to accept the required number of tubes without significant drop in temperature when the tubes are immersed.
5. Water bath suitable for use at 10-15°C.

H. REAGENTS

1. Distilled water free of nitrite and nitrate is to be used in preparation of all reagents and standards.
2. Sodium chloride solution (30%): Dissolve 300 g NaCl in distilled water and dilute to 1 liter.

3. Sulfuric acid solution: Carefully add 500 ml conc. H_2SO_4 to 125 ml distilled water. Cool and keep tightly stoppered to prevent absorption of atmospheric moisture.

4. Brucine-sulfanilic acid reagent: Dissolve 1 g brucine sulfate $(C_{23}H_{26}N_2O_4)\cdot H_2SO_4\cdot 7H_2O$ and 0.1 g sulfanilic acid $(NH_2C_6H_4SO_3H\cdot H_2O)$ in 70 ml hot distilled water. Add 3 ml conc. HCl, cool, mix and dilute to 100 ml with distilled water. Store in a dark bottle at 5°C. This solution is stable for several months; the pink color that develops slowly does not affect its usefulness. Mark bottle with warning CAUTION: Brucine Sulfate is toxic; take care to avoid ingestion.

5. Potassium nitrate stock solution: 1.0 ml = 0.1 mg NO_3-N. Dissolve 0.7218 g anhydrous potassium nitrate (KNO_3) in distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 ml chloroform per liter. This solution is stable for at least 6 months.

6. Potassium nitrate standard solution: 1.0 ml - 0.001 mg NO_3-N. Dilute 10.0 ml of the stock solution to 1 liter in a volumetric flask. This standard solution should be prepared fresh weekly.

7. Acetic acid (1 + 3): Dilute 1 volume glacial acetic acid (CH_3COOH) with 3 volumes of distilled water.

8. Sodium hydroxide (1N): Dissolve 40 g of NaOH in distilled water. Cool and dilute to 1 liter.

I. DETERMINATION

1. Adjust the pH of the samples to approximately 7 with acetic acid or sodium hydroxide. If necessary, filter to remove turbidity.
2. Set up the required number of sample tubes in the rack to handle reagent blank, standards and samples. Space tubes evenly throughout the rack to allow for even flow of bath water between the tubes. This should assist in achieving uniform heating of all tubes.
3. If it is necessary to correct for color or dissolved organic matter which will cause color on heating, a set of duplicate samples must be run to which all reagents except the brucine-sulfanilic acid have been added.
4. Pipette 10.0 ml of standards and samples or an aliquot of the samples diluted to 10.0 ml into the sample tubes.
5. If the samples are saline, add 2 ml of the 30% sodium chloride solution to the reagent blank, standards and samples. For fresh water samples, sodium chloride solution may be omitted. Mix contents of tubes by swirling and place rack in cold water bath (0-10°C).

6. Pipette 10.0 ml of sulfuric acid solution into each tube and mix by swirling. Allow tubes to come to thermal equilibrium in the cold bath. Be sure that temperatures have equilibrated in all tubes before continuing.

7. Add 0.5 ml brucine-sulfanilic acid reagent to each tube (except the interference control tubes) and carefully mix by swirling, then place the rack of tubes in the 100°C water bath for exactly 25 minutes.

   Caution: Immersion of the tube rack into the bath should not decrease the temperature of the bath more than 1 to 2°C. In order to keep this temperature decrease to an absolute minimum, flow of bath water between the tubes should not be restricted by crowding too many tubes into the rack. If color development in the standard reveals discrepancies in the procedure, the operator should repeat the procedure after reviewing the temperature control steps.

8. Remove rack of tubes from the hot water bath and immerse in the cold water bath and allow to reach thermal equilibrium (20-25°C).

9. Read absorbance against the reagent blank at 410 nm using a 1 cm or longer cell.

J. CALCULATION

1. Obtain a standard curve by plotting the absorbance of standards run by the above procedure against mg NO_3ON/l. (The color reaction does not always follow Beer's law.)
2. Subtract the absorbance of the sample without the brucine-sulfanilic reagent from the absorbance of the sample containing brucine-sulfanilic acid and determine mg NO_3-N/l. Multiply by an appropriate dilution factor if less than 10 ml of sample is taken.

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Date Issued April 1, 1981

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APPROVED

6.1.A.36.0 DETERMINATION OF NITRATE-NITRITE NITROGEN IN WATER

A. PURPOSE

To provide an analytical method for nitrogen in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered through a glass fiber filter or a 0.45μ membrane filter. Highly turbid samples may be pretreated with zinc sulfate before filtration to remove the bulk of particulate matter present in the sample.
2. Low results might be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the samples to eliminate this interference.
3. Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.
4. This procedure determines both nitrate and nitrite. If only nitrate is desired, a separate determination must be made for

nitrite and subsequent corrections made. The nitrite may be determined by the procedure below without the reduction step.

G. APPARATUS

1. Reduction column: The column in Figure 1 was constructed from a 100 ml pipet by removing the top portion. This column may also be constructed from two pieces of tubing joined end to end. A 10 mm length of 3 cm I.D. tubing is joined to a 25 cm length of a 3.5 mm I.D. tubing.
2. Spectrophotometer for use at 540 nm, providing a light path of 1 cm or longer.

H. REAGENTS

1. Granulated cadmium: 40-60 mesh (EM Laboratories, Inc. 500 Exec. Blvd. Elmsford, NY 10523, Cat 2001 Cadmium, Coarse Powder).
2. Copper-Cadmium: The cadmium granules (new or used) are cleaned with dilute HCl and copperized with 2% solution of copper sulfate in the following manner:
    a. Wash the cadmium with dilute HCl and rinse with distilled water. The color of the cadmium should be silver.
    b. Swirl 25 g cadmium in 100 ml portions of a 2% solution of copper sulfate for 5 minutes or until blue color partially

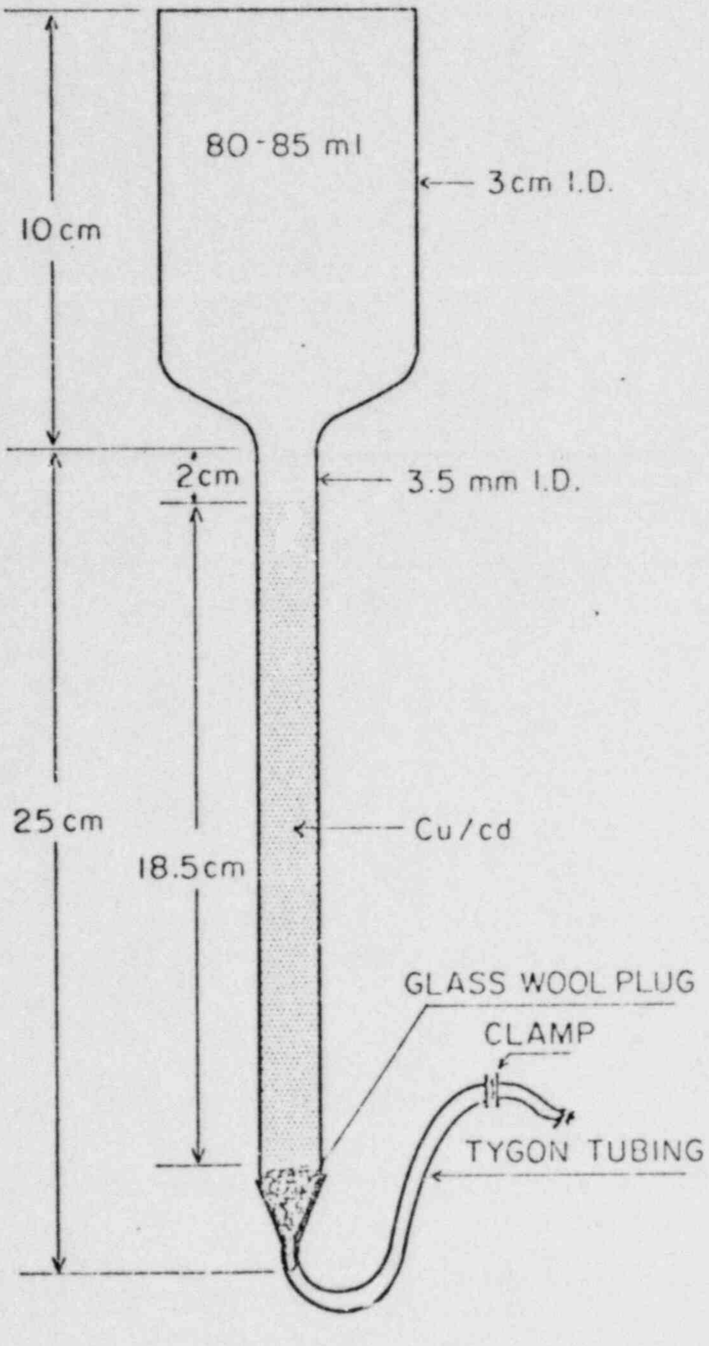

FIGURE 1. REDUCTION COLUMN

fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms.

c. Wash the copper-cadmium with distilled water (at least 10 times) to remove all the precipitated copper. The color of the cadmium so treated should be black.

3. Preparation of reaction column: Insert a glass wool plug into the bottom of the reduction column and fill with distilled water. Add sufficient copper-cadmium granules to produce a column 18.5 cm in length. Maintain a level of distilled water above the copper-cadmium granules to eliminate entrapment of air. Wash the column with 200 ml of diluted ammonium chloride solution. The column is then activated by passing through the column 100 ml of solution composed of 25 ml of a 1.0 mg/l NO_3-N standard and 75 ml of ammonium chloride - EDTA solution. Use a flow rate between 7 and 10 ml per minute.

4. Ammonium chloride - EDTA solution: Dissolve 13 g ammonium chloride and 1.7 g disodium ethylenediamine tetracetate in 900 ml of distilled water. Adjust the pH to 8.5 with conc. ammonium hydroxide and dilute to 1 liter.

5. Dilute ammonium chloride-EDTA solution: Dilute 300 ml of ammonium chloride-EDTA solution to 500 ml with distilled water.

6. Color reagent: Dissolve 10 g sulfanilamide and 1 g N(1-naphthyl)-ethylene-diamine dihydrochloride in a mixture of 100 ml conc. phosphoric acid and 800 ml of distilled water and dilute to 1 liter with distilled water.
7. Zinc sulfate solution: Dissolve 100 g $ZnSO_4 \cdot 7H_2O$ in distilled water and dilute to 1 liter.
8. Sodium hydroxide solution, 6N: Dissolve 240 g NaOH in 500 ml distilled water, cool and dilute to 1 liter.
9. Ammonium hydroxide, conc.
10. Dilute hydrochloric acid, 6N: Dilute 50 ml of conc. HCl to 100 ml with distilled water.
11. Copper sulfate solution, 2%: Dissolve 20 g of $CuSO_4 \cdot 5H_2O$ in 500 ml of distilled water and dilute to 1 liter.
12. Stock nitrate solution: Dissolve 7.218 g KNO_3 in distilled water and dilute to 1000 ml. Preserve with 2 ml of chloroform per liter. This solution is stable for at least 6 months. 1.0 ml - 1.00 mg NO_3-N.
13. Standard nitrate solution: Dilute 10.0 ml of nitrate stock solution to 1000 ml with distilled water 1.0 ml - 0.01 mg NO_3-N.
14. Stock nitrite solution: Dissolve 6.072 g KNO_2 in 500 ml of distilled water and dilute to 1000 ml. Preserve with 2 ml of chloroform and keep under refrigeration. Stable for approximately 3 months. 1.0 ml - 1.00 mg NO_2-N.

15. Standard nitrite solution: Dilute 10.0 ml of stock nitrite solution to 1000 ml with distilled water. 1.0 ml - 0.01 mg NO_2-N.

16. Using standard nitrate solution prepare the following standards in 100 ml volumetric flasks:

Conc., mg-NO_3-N/l	ml of Standard Solution/100.0 ml
0.00	0.0
0.05	0.5
0.10	1.0
0.20	2.0
0.50	5.0
1.00	10.0

I. DETERMINATION

1. Turbidity removal: One of the following methods may be used to remove suspended matter.
    a. Filter sample through a glass fiber filter or a 0.45μ membrane filter.
    b. Add 1 ml zinc sulfate solution to 100 ml of sample and mix thoroughly. Add 0.4-0.5 ml sodium hydroxide solution to obtain a pH of 10.5 as determined with a pH meter. Let the treated sample stand a few minutes to allow the heavy flocculent precipitate to settle. Clarify by filtering through a glass fiber filter or a 0.45μ membrane filter.

2. Oil and grease removal: Adjust the pH of 100 ml of filtered sample to 2 by addition of conc. HCl. Extract the oil and grease from the aqueous solution with two 25 ml portions of a non-polar solvent (Freon, chloroform or equivalent).
3. If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. HCl or conc. NH_4OH. This is done to insure a sample pH of 8.5 after step 4.
4. To 25.0 ml of sample or an aliquot diluted to 25.0 ml, add 75 ml of ammonium chloride-EDTA solution and mix.
5. Pour sample into column and collect sample at a rate of 7-10 ml per minute.
6. Discard the first 25 ml, collect the rest of the sample (approximately 70 ml) in the original sample flask. Reduced samples should not be allowed to stand longer than 15 minutes before addition of color reagent, step 7.
7. Add 2.0 ml of color reagent to 50.0 ml of sample. Allow 10 minutes for color development. Within 2 hours measure the absorbance at 540 nm against a reagent blank.

   NOTE: If the concentration of sample exceeds 1.0 mg NO_3-N/l, the remainder of the reduced sample may be used to make an appropriate dilution before proceeding with step 7.

8. Standards: Carry out the reduction of standards exactly as described for the samples. At least one nitrite standard should be compared to a reduced nitrate standard at the same concentration to verify the efficiency of the reduction column.

J. CALCULATION

1. Obtain a standard curve by plotting the absorbance of standards run by the above procedure against NO_3-N mg/l. Compute concentration of samples by comparing sample absorbance with standard curve.
2. If less than 25 ml of sample is used for the analysis the following equation should be used:

$$mgNO_2 + NO_3 - N/l = \frac{A \times 25}{ml\ sample\ used}$$

where:

A - Concentration nitrate from standard curve.

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6.1.A.37.0 DETERMINATION OF NITRITE NITROGEN IN WATER

A. PURPOSE

To provide an analytical method for nitrite nitrogen in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

There are very few known interferences at concentrations less than 1,000 times that of the nitrite; however, the presence of strong oxidants or reductants in the samples will readily affect the nitrite concentrations. High alkalinity (>600 mg/l) will give low results due to a shift in pH.

G. APPARATUS

1. Spectrophotometer equipped with 1 cm or larger cells for use at 540 nm.
2. Nessler tubes, 50 ml or volumetric flasks, 50 ml.

H. REAGENTS

1. Distilled water free of nitrite and nitrate is to be used in preparation of all reagents and standards.
2. Buffer-color reagent: To 250 ml of distilled water, add 105 ml conc. hydrochloric acid, 5.0 g sulfanilamide and 0.5 g N -

(1 naphthyl) ethylenediamine dihydrochloride. Stir until dissolved. Add 136 g of sodium acetate ($CH_3COONa \cdot 3H_2O$) and again stir until dissolved. Dilute to 500 ml with distilled water. This solution is stable for several weeks if stored in the dark.

3. Nitrite stock solution: 1.0 ml = 0.10 mg NO_2-N. Dissolve 0.4926 g of dried anhydrous sodium nitrite (24 hours in desiccator) in distilled water and dilute to 1000 ml. Preserve with 2 ml chloroform per liter.
4. Nitrite standard solution: 1.0 ml = 0.001 mg NO_2-N. Dilute 10.0 ml of the stock solution to 1000 ml.

I. DETERMINATION

1. If the sample has a pH greater than 10 or a total alkalinity in excess of 600 mg/l, adjust to approximately pH6 with 1:3 HCl.
2. If necessary, filter the sample through a 0.45 μ pore size filter using the first portion of filtrate to rinse the filter flask.
3. Place 50 ml of sample, or an aliquot diluted to 50 ml, in a 50 ml Nessler tube; hold until preparation of standards is completed.
4. At the same time prepare a series of standards in 50 ml Nessler tubes as follows:

ml of Standard Solution 1.0 ml = 0.001 mg NO_2-N	Conc., When Diluted to 50 ml, mg/l of NO_2-N
0.0	(Blank)
0.5	0.01
1.0	0.02
1.5	0.03
2.0	0.04
3.0	0.06
4.0	0.08
5.0	0.10
10.0	0.20

5. Add 2 ml of buffer-color reagent to each standard and sample, mix and allow color to develop for at least 15 minutes. The color reaction medium should be between pH 1.5 and 2.0.
6. Read the color in the spectrophotometer at 540 nm against the blank and plot concentration of NO_2-N against absorbance.

J. CALCULATION

1. Read the concentration of NO_2-N directly from the curve.
2. If less than 50.0 ml of sample is taken, calculate mg/l as follows:

$$NO_2 - N,\ mg/l = \frac{mg/l \text{ from std. curve } X\ 50}{ml \text{ sample used}}$$

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6.1.A.38.0 GRAVIMETRIC DETERMINATION OF SULFATE IN WATER

A. PURPOSE

To provide an analytical method for sulfate in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure. and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. High results may be obtained for samples that contain suspended matter, nitrate, sulfite and silica.
2. Alkali metal sulfates frequently yield low results. This is especially true of alkali hydrogen sulfates. Occlusion of alkali sulfate with barium sulfate causes the substitution of an element of lower atomic weight than barium in the precipitate. Hydrogen sulfate of alkali metal acts similarly and decomposes when heated. Heavy metals such as chormium and iron, cause low results by interfering with complete precipitation and by formation of heavy metal sulfates.

G. APPARATUS

1. Steam bath
2. Drying oven, equipped with thermostatic control.
3. Muffle furnace with heat indicator.

4. Desiccator
5. Analytical balance, capable of weighing to 0.1 mg.
6. Filter paper, acid-washed, ashless hard-finish filter paper sufficiently retentive for fine precipitates.

H. REAGENTS

1. Methyl red indicator solution: Dissolve 100 mg methyl red sodium salt in distilled water in a 100 ml volumetric flask and dilute to the mark with distilled water.
2. Hydrochloric acid, HCl, 1 + 1
3. Barium chloride solution: Dissolve 100 g $BaCl_2 \cdot 2H_2O$ in 1 liter of distilled water. Filter through a membrane filter or hard-finish filter paper. One ml of this reagent is capable of precipitating approximately 40 mg SO_4.
4. Silver nitrate nitric acid reagent: Dissolve 8.5 g $AgNO_3$ and 0.5 ml conc. HNO_3 in 500 ml distilled water.

I. DETERMINATION

1. Removal of silica: If silica concentration is greater than 25 mg/l.
    a. Evaporate sample nearly to dryness in a platinum dish on a steam bath.

- b. Add 1 ml HCl solution, tilt dish and rotate until acid contacts all of the residue.
- c. Continue evaporation to dryness.
- d. Complete drying in an oven at 180°C.
- e. If organic matter present, char over a flame.
- f. Moisten with 2 ml distilled water and 1 ml HCl solution.
- g. Evaporate to dryness on a steam bath.
- h. Add 2 ml HCl solution.
- i. Take up soluble residue in hot distilled water and filter.
- j. Wash the insoluble silica with several small portions of hot distilled water.
- k. Combine filtrate and washings.

2. Precipitation of barium sulfate
    - a. If necessary, treat clarified sample to remove interfering agents.
    - b. Adjust to contain approximately 50 mg SO_4 ion in a 250 ml volume.
    - c. Adjust acidity with HCl solution to pH 4.5 to 5.0, using pH meter or orange color of methyl red indicator.
    - d. Add an additional 1 to 2 ml HCl solution.

e. For lower concentrations of sulfate ion fix the total volume at 150 ml.

f. Heat to boiling and, while stiring gently, add warm $BaCl_2$ solution slowly, until precipitation appears to be complete; then add approximately 2 ml in excess.

g. If amount of precipitate is small, add a total of 5 ml $BaCl_2$ solution.

h. Digest the precipitate at 80 to $90^{o}C$ preferably overnight but for not less than 2 hours.

3. Filtration and weighing

   a. Mix a little ashless filter paper pulp with the $BaSO_4$ and filter at room temperature.

   b. Wash the precipitate with small portions of warm distilled water until the washings are free of chloride as indicated by testing with silver nitrate-nitric acid reagent.

   c. Dry the filter and precipitate.

   d. Ignite at $800^{o}C$ for 1 hour. DO NOT LET THE FILTER PAPER FLAME!

   e. Cool in desiccator and weigh.

J. CALCULATION

$$\text{mg/l } SO_4 = \frac{\text{mg } BaSO_4 \text{ X } 411.5}{\text{ml sample}}$$

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6.1.A.39.0 TURBIDIMETRIC DETERMINATION OF SULFATE IN WATER

A. PURPOSE

To provide an analytical method for sulfates in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been completed without deviation.

D. SAFETY

Safety glasses required.

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E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. APPARATUS

1. Magnetic stirrer, variable speed so that it can be held constant just below splashing. Use identical shape and size magnetic sirring bars.
2. Stopwatch, if the magnetic stirrer is not equipped with an accurate timer.
3. Measuring spoon, capacity 0.2 to 0.3ml.

G. REAGENTS

1. Conditioning reagent: Place 30ml conc. HCl, 300ml distilled water, 100ml 95% ethanol or isopropanol and 75g NaCl in solution in a container. Add 50ml glycerol and mix.
2. Barium chloride, $BaCl_2$ crystals, 20 to 30 mesh.
3. Sodium carbonate solution (approximately 0.05N): Dry 3 to 5g primary standard Na_2CO_3 at 250°C for 4 hours and cool in a desiccator. Weigh 2.5 ± 0.2g (to the nearest mg), transfer to a 1 liter volumetric flask and fill to the mark with distilled water.
4. Standard sulfate solution (1.00ml = 100ugSO_4).

H. DETERMINATION

1. Formation of barium sulfate turbidity
   - a. Place 100ml sample, or a suitable portion diluted to 100ml into a 250 Erlenmeyer flask.
   - b. Add exactly 5.0ml conditioning reagent.
   - c. Mix in the stirring apparatus.
   - d. While the solution is being stirred, add a measuring spoonful of $BaCl_2$ crystals and begin timing immediately.
   - e. Stir exactly 1.0 minutes at constant speed.
2. Measurement of barium sulfate turbidity
   - a. Immediately after the stirring period has ended, pour solution into absorbance cell.
   - b. Measure turbidity at 30 second intervals for 4 minutes.
   - c. Record the maximum reading obtained in the 4 minute period.
3. Preparation of calibration curve
   - a. Prepare calibration curve using standard sulfate solution.
   - b. Space standards at 5mg/l increments in the 0-40mg/l sulfate range.
   - c. Above 50mg/l the accuracy decreases and the suspensions lose stability.
   - d. Check reliability of calibration curve by running a standard with every 3 or 4 samples.

4. Correction for sample color and turbidity

   a. Run a sample blank using the procedure 1 and 2 without the addition of barium chloride.

I. CALCULATIONS

1. Read mg SO_4 from calibration curve.

$$\text{mg } SO_4/1 = \frac{\text{mg } SO_4 \text{ X } 1{,}000}{\text{ml sample}}$$

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6.1.A.40.0 DETERMINATION OF TOTAL DISSOLVED SOLIDS

A. PURPOSE

To provide an analytical method for Total Dissolved Solids in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Use stainless steel tongs to handle hot crucibles.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. Highly mineralized waters containing significant concentrations of calcium, mangesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
2. Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180° to insure that all the bicarbonate is converted to carbonate.
3. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

G. APPARATUS

1. Glass fiber filter discs 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E or equivalent.
2. Filter holder, membrane filter funnel or Gooch cruicible adapter.

3. Suction flask, 500 ml.
4. Gooch crucibles, 25 ml (if 2.1 cm filter is used).
5. Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
6. Steam bath.
7. Drying over, 180°C ± 2°C.
8. Desccator.
9. Analytical balance, capable of weighing to 0.1 mg.

H. DETERMINATION

1. Preparation of glass fiber disc. Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.
2. Preparation of evaporating dishes: If volatile residue is also to be measured heat the clean dish to 550 ± 50°C for one hour in a muffle furnace. If only filterable residue is to be measured heat the clean dish to 180°C ± 2°C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

3. Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If total filterable residue is low, a larger volume may be filtered.
4. Filter the sample through the glass fiber filter, rinse with three 10 ml portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.
5. Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.
6. Dry the evaporated sample for at least one hour at $180 \pm 2^{\circ}C$ in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

I. CALCULATION

Calculate Total Dissolved Solids as follows:

$$\text{Total Dissolved Solids, mg/l} = \frac{(A - B) \times 1,000}{C}$$

where:

A = weight of dried residue + dish in mg

B = weight of dish in mg

C = volume of sample used in ml.

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6.1.A.41.0 DETERMINATION OF TOTAL SUSPENDED SOLIDS

A. PURPOSE

To provide an analytical method for Total Suspended Solids in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Use stainless steel tongs to handle hot crucibles.

E. INTERFERENCES

1. Filtration apparatus, filter material, pre-washing, post-washing and drying temperature are specified because these variables have been shown to affect the results.
2. Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

F. APPARATUS

1. Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeve Angel 934-AH, Gelman type A/E, or equivalent. NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size," collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.
2. Filter support: filtering apparatus with reservoir and a coarse 40-60 microns) fritted disc as a filter support.

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

a. Suction flask

b. Drying Oven, 103-105°C

c. Desiccator

d. Analytical balance, capable of weighing to 0.1mg.

G. DETERMINATION

1. Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20ml volumes of deionized water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

2. Selection of Sample Volume

For a 4.7 cm diameter filter, filter 100 ml of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 ml/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five ml increments for timing are suggested. Continue to record the time and volume increments until filtration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration support.

3. Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

4. Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

5. With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

   NOTE: Total volume of wash water used should equal approximately 2 ml per cm^2. For a 4.7 cm filter the total volume is 30 ml.

6. Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103-105^{o}C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

H. CALCULATIONS

1. Calculate total suspended solids as follows:

$$\text{Non-filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

A = weight of filter (or filter and crucible) + residue in mg

B = weight of filter (or filter and crucible) in mg

C = ml of sample filtered.

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6.1.A.42.0 DETERMINATION OF TOTAL SOLIDS

A. PURPOSE

To provide an analytical method for Total Solids in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required. Use stainless steel tongs to handle hot crucibles.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. Non-representative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
2. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

G. APPARATUS

Evaporating dishes, procelain, 90 mm, 100 ml capacity. (Vycor or platinum dishes may be substituted and smaller size dishes may be used if required.)

H. DETERMINATION

1. Heat the clean evaporating dish to 103-105°C for one hour, if Volatile Residue is to be measured, heat at 550 ± 50°C for one

hour in a muffle furnace. Cool, desiccate, weigh and store in desiccator until ready for use.

2. Transfer a measured aliquot of sample to the pre-weighed dish and evaporate to dryness on a steam bath or in a drying oven.
    a. Choose an aliquot of sample sufficient to contain a residue of at least 25 mg. To obtain a weighable residue, successive aliquots of sample may be added to the same dish.
    b. If evaporation is performed in a drying oven, the temperature should be lowered to approximately 98°C to prevent boiling and splattering of the sample.
3. Dry the evaporated sample for at least 1 hour at 103-105°C. Cool in a desiccator and weigh. Repeat the cycle of drying at 103-105°C, cooling, desiccating and weighing until a constant weight is obtained or until loss of weight is less than 4% of the previous weight, or 0.5 mg, whichever is less.

I. CALCULATION

Calculate total Solids as follows:

$$\text{Total Solids, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of sample + dish in mg

B = weight of dish in mg

C = volume of sample in ml

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6.1.A.43.0 DETERMINATION OF TRACE METALS BY INDUCTIVELY COUPLED ARGON PLASMA

A. PURPOSE

To provide an analytical method for the determination of trace metals.

B. SCOPE

This analytical procedure is applicable to water samples, the liquid portion of the extractive toxicity test for hazardous wastes, lubricant, and coolant analysis for wear metals.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemsit to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the analytical procedure was followed without deviation.

D. SAFETY

Safety glasses required. All doors to the instrument shall be closed when in operation. The instrument is equipped with multiple automatic interlock systems which will shut it down if any door is opened, if gas pressure fails, if water pressure fails, or if reflected radio frequency power exceeds a factory set level.

Additionally, an audible alarm will sound if the reflected radio frequency power exceeds a user set level. The alarm also sounds when the instrument is turned on or if a power interruption occurs. The alarm may be reset by pushing the alarm reset button. These automatic shutdown systems shall not be bypassed at any time. In the event of automatic shutdown the instrument shall not be restarted until the cause of shutdown has been determined.

E. QUALITY CONTROL

A spike sample, a duplicate sample, and a blank shall be analyzed with each ten samples in a sample run. If fewer than ten samples are to be analyzed in a sample run, then one spike sample, one duplicate, and one blank shall be analyzed.

F. PROCEDURE

1. Reagents - Anti-freeze,commercial, of the type and dilution used by the mine machinery.
2. Lubricant - commercial, type and weight being used by the mine machinery.
3. Standards, Water - Five separate standards prepared by the Environmental Chemist for calibrating the instrument. Prepared

using reagent grade chemicals and deionized water.

4. Standards, Coolant - 5 Standards prepared by the Environmental Chemist using reagent grade chemicals and commercial anti-freeze. Used for calculating the instrument.

5. Standards, Lubricant - Standards prepared by the Environmental Chemist using reagent grade organometalic compounds and reagent grade xylene. Mixed with a blank of the lubricant of interest for use in calibrating the instrument.

G. INSTRUMENT INITIALIZATION

1. Install, as instructed in the instrument manual, the appropriate torch and nebulizer assembly for the determination being performed.
2. Install the proper type of tubing in the peristaltic pump. Isoversinic tubing for lubricant analysis, and polyvinylchloride tubing for coolant and water analysis.
3. Adjust the pump speed to deliver 1.5 to 1.6 ml/min of the sample.
4. Attach the pump tubing to the nebulizer feed line.
5. Actuate the plasma torch as instructed in the instrument manual. Initialize the sample flow into the nebulizer.
6. Call up the appropriate program matrix for the samples of interest by typing in the four letter matrix code.

Matrix Code	Application
MINE	Water, no dynamic background correction
SSNE	Water, dynamic background correction as required by EPA for NPDES analyses.
LUBE	Lubricant, no dynamic background correction.
HEAT	Coolant, no background correction.

7. Simultaneously depress the shift key and the letter M to allow the computer to function.
8. Simultaneously depress the control key and the letter P to switch the instrument into profile mode. The computer will respond with PCN #. Enter the number 14.
9. Place the sampling tube into the copper profiling standard and profile the response. If the torch has been changed since the previous use profile the flame vertically and horizontally. See instrument manual.
10. Release instrument from profile mode by depressing the ESC (escape) key.
11. Call up background correction if desired by depressing the letter D. After the computer responds enter the number of background cycles with the number 3.

H. DETERMINATION FOR WATER

1. Ensure that the proper matrix, SSNE, is in the computer. Ensure that the background correction is enabled.
2. Ensure that the N + 1 channel is adjusted to the proper wave length as required.
3. Open the computer to calibration by pressing the letter J.
4. Place the sampling tube in the first standard, UNCI. Press the fast forward button on the pump until the standard enters the nebulizer.
5. On the teletype console type QEGIGIGIAIN, then the return key. This will set up the computer to erase any previous standard memory (E), analyze the standard three times (G), print the results in intensities (I), print the average intensity (AI), and ask the name of the standard (N). The computer will now execute this command string every time the letter O is typed until a new Q is typed. Type the letter O. When the computer is through, type the standard name, UNCl.
6. Place the sampling tube in the next standard. Press the fast forward button on the pump until the standard enters the nebulizer. Type the letter O and when the computer is through enter the standard name.
7. Repeat step 6 for all 5 standards.

8. When all standards have been analyzed, type the letter S to enter calibration. The instrument is now calibrated and need not be calibrated until the next use.
9. Type QEGCGCGCAC, then the return key. This will set up the computer to erase any previous sample memory (E), analyze the standard three times (G), print the results in concentration (C), and print the average concentration (AC). The computer will execute this command string every time the letter O is typed until a new Q is typed.
10. Place the sampling tube in the sample. Press the fast forward button on the pump until the sample enters the nebulizer.
11. Type / - and then sample identification and N+1 element if any. Press return key.
12. Type O. When the analysis is complete, the three individual analyses results and the average result will be printed.
13. Return to step 10 for the next sample.

I. DETERMINATION FOR LUBRICANT

1. Ensure that the proper matrix, LUBE, is in the computer.
2. Make standards using the LUB series of organometalics in xylene and a fresh, unused sample of the specific lubricant being analyzed. Mix 25 ml of each LUB series standard with 25 ml of

lubricant. Label as LUB1 through LUB5 and the type and weight of lubricant. These standards may be used in the future if the same type and weight of lubricant, from the the same source, is analyzed again.

3. Standardize the instrument as in steps 4 through 8 for the determination for water except use the Q string QEGIGIAIN.
4. Analyze each sample as in steps 9 through 13 of the determination for water except use the Q string QEGCGCAC.

J. DETERMINATION FOR COOLANT

1. Ensure that the proper matrix, HEAT, is in the computer. Ensure that the HOT series of standards is used.
2. Standardize the insturment as in step 4 through 8 for the determination for water except use the Q string QEGIGIAIN.
3. Analyze each sample as in steps 9 through 13 of the determination for water except use th Q string QEGCGCAC.

K. INSTRUMENT SHUT DOWN

1. Press the RF off button.
2. Turn off gas flow at the tank then at the instrument.
3. Turn off water flow.
4. Turn off teletype console.

L. <u>CALCULATION</u>:

The analysis results are printed directly in mg/liter for each element. Subtract the concentrations of the blank sample from the sample concentrations.

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6.1.A.44.0 DETERMINATION OF AMMONIA NITROGEN IN WATER, USING AN ION SELECTIVE ELECTRODE

A. PURPOSE

To provide an analytical method for ammonia nitrogen in water samples.

B. SCOPE

This analytical procedure applies to the analysis of all routine well water and surface water samples.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that this procedure is followed by the analyst and to evaluate the analytical results.

It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Environmental Chemist. The analyst must sign the laboratory worksheet in the appropriate location to indicate that the procedure has been followed without deviation.

D. SAFETY

Safety glasses required.

E. QUALITY CONTROL

One spike sample, one duplicate, and one blank shall be analyzed with each ten samples in an analysis run. If fewer than ten samples are to be analyzed in a run, then one spike sample, one duplicate and one blank shall be analyzed.

F. INTERFERENCES

1. Volatile amines act as a positive interference.
2. Mercury interferes by forming a strong complex with ammonia. Thus the samples cannot be preserved with mercuric chloride.

G. APPARATUS

1. Electrometer (pH meter) with expanded mV scale or a specific ion meter.
2. Ammonia selective electrode, such as Orion Model 95-10 or equivalent.
3. Magnetic stirrer, thermally insulated, and Teflon-coated stirring bar.

H. REAGENTS

1. Distilled water: Special precautions must be taken to insure that the distilled water is free of ammonia. This is accomplished by passing distilled water through an ion exchange column con-

taining a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin.

2. Sodium hydroxide, 10N: Dissolve 400 g of sodium hydroxide in 800 ml of distilled water. Cool and dilute to 1 liter with distilled water.
3. Ammonium chloride, stock solution: 1.0 ml - 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in water and bring to volume in a 1 liter volumetric flask using distilled water.
4. Ammonium chloride, standard solution: 1.0 ml - 0.01 mg NH_3-N. Dilute 10.0 ml of the stock solution to 1 liter with distilled water in a volumetric flask.

I. DETERMINATION

1. Preparation of standards: Prepare a series of standard solutions covering the concentration range of the samples by diluting either the stock or standard solutions of ammonium chloride.
2. Calibration of electrometer: Place 100 ml of each standard solution in clean 150 ml beakers. Immerse electrode into standard of lowest concentration and add 1 ml of 10N sodium hydroxide solution while mixing. Keep electrode in the solution until a stable reading is obtained.

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NOTE: The pH of the solution after the addition of NaOH must be above 11. Caution: Sodium hydroxide must not be added prior to electrode immersion, for ammonia may be lost from a basic solution.

3. Repeat this procedure with the remaining standards, going from lowest to highest concentration. Using semilogarithmic graph paper, plot the concentration of ammonia in mg NH_3-N/l on the log axis vs. the electrode potential developed in the standard on the linear axis, starting with the lowest concentration at the bottom of the scale.
4. Calibration of a specific ion meter: Follow the directions of the manufacturer for the operation of the instrument.
5. Sample measurement: Follow the procedure for 100 ml of sample in 150 ml beakers. Record the stabilized potential or each unkown sample and convert the potential reading to the ammonia concentration using the standard curve. If a specific ion meter is used, read the ammonia level directly in mg NH_3-N/l.

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6.1.A.45.0 PROCEDURES FOR THE USE OF RADIOACTIVE MATERIALS

A. PURPOSE

To provide procedures for the use and storage of radioactive materials for laboratory use.

B. SCOPE

These procedures are applicable to all radioactive materials used in the laboratory and to all laboratory personnel. Environmental samples collected on the Morton Ranch mine properties are not considered radioactive materials until a radioactive tracer has been added or the sample has been concentrated for the determination of radioactivity.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that the procedure is followed without deviation by laboratory personnel and to ensure that the required documentation of activities involving radioactive materials is maintained. It is the responsibility of the laboratory personnel to follow the procedure without deviation and to report any abnormalties to the Environmental Chemist.

D. PROCEDURE

1. Laboratory Apparel and Safety Equipment

   a. Laboratory apparel and safety equipment requirements are detailed in the Laboratory Safety Manual.

2. Limitations on the Handling of Radioactive Materials

   a. All shipping containers in which radioactive materials have been received by the laboratory will be opened only by the Environmental Chemist and only in the radioisotope fume hood. Any leakage or spillage of the contents must be reported to the Environmental/Radiation Department Manager and the manufacturer immediately. The Environmental/Radiation Department Manager will direct the appropriate containment of the material.

   b. All operations involving the use of concentrated radioactive materials as received from the manufacturer shall be performed only with specific written approval and written instructions from the Environmental Chemist or the Enviornmental/Radiation Manager. See Procedure 6.1.A.15.0 Dilution of Radioactive Standard Solutions.

   c. All operations involving the use of concentrated radioactive materials and dilute standard solutions used in the analyses will be performed in the radioisotope fume hood. The fume hood must be operating properly when radioactive materials are present in it.

d. No fume hood, including the radioisotope fume hood, may be used for the storage of radioactive materials. Fume hoods are only to be used when required in the performance of laboratory work.

e. Concentrated radioactive solutions will be kept until use in the container supplied by the manufacturer with sufficient absorbent packing material surrounding the material container to absorb any leakage. The material will be kept in the radioactive materials storage cabinet which will be locked.

f. Access to the radioisotope storage cabinet will be authorized only by the Environmental/Radiation Department Manager or the Environmental Chemist. Laboratory personnel will not have keys to the radioisotope cabinet.

g. All dilute radioactive standard solutions will be kept in polyethylene or teflon bottles with a maximum size of 1 liter. The contents of each bottle will be clearly labeled. See Procedure 6.1.A.17.0 Storage, Use, and Handling of Radioactive Standard Solutions.

h. Standard solution containers may not be opened except in the radioisotope fume hood.

i. Transfer of standard solution aliquots to samples will be performed only with pipettes specifically assigned to the individual solution and labled accordingly. Pipettes will be stored in the radioisotope cabinet when not in use.

j. Pipetting by mouth is not allowed under any circumstances. Use of a pipetting aid is required. If a pipetting aid is contaminated by over pipetting of a solution, it must be disposed of as radioactive waste. The Environmental Chemist must be notified immediately and an estimate made of the amount lost. The estimate of loss will be entered in the radioisotope log book. The Environmental Chemist will direct any necessary decontamination.

k. Environmental samples to which radioactive tracers have been added may be moved about in the laboratory work area subject to the following limitations;

    1. Any evaporation must be performed in a fume hood and samples must not be removed from the fume hood until cool.
    2. Any volatile fraction from analysis must be kept in a fume hood during analysis.

3. Sample containers shall be covered whenever they are not in a fume hood or whenever the analysis does not require that the container be open.

1. Electroplated and foil covered sources will be stored in the lead shielded box in the radioisotope cabinet when not in use. The use and handling of the radioactive sources is covered in Procedure 6.1.A.13.0 Care and Handling of Calibrated Soruces.

E. RADIATION SURVEY AND MONITORING PROCEDURES

1. Control TLD badges will be placed in the laboratory to monitor background radiation levels. The TLD badges will be returned to the manufacturer for readout on a quarterly basis.
2. Surveys of each laboratory room will be made using a Ludlum Model 19 micro R meter or similar instrument on a quarterly basis.
3. Swipe samples will be taken from laboratory work surfaces, equipment, and the exterior and interior surfaces of the radioisotope cabinet on a monthly basis.
4. Documentation of all survey and monitoring results, including any necessary decontamination, will be maintained.

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F. SAFETY PRECAUTIONS

1. Radioactive solutions, except when added as tracers to environmental samples, will be stored in break resistant screw cap containers in the radioisotope cabinet.
2. Radioactive materials will not be removed from the laboratory area until discarded as laboratory radioactive waste or returned to the manufacturer.
3. General laboratory safety is covered in Procedure 6.1.A.12.0 Laboratory Safety.

G. STORAGE OF RADIOACTIVE MATERIALS

Storage of radioactive materials is covered under procedure B above.

H. POSTING OF AR[illegible]N WHICH RADIOACTIVE MATERIALS ARE USED

1. The entire laboratory is considered to be a radioactive materials use area. Each entrance to the laboratory and the radioisotope cabinet is clearly posted with a sign stating:

Radiation
Radioactive
Materials

The signs are 14" x 10" with the work "Radiation" in 2¼" high yellow letters on red background and the word "Radioactive" and "Materials" in 2" high black letters on yellow background. The standard radiation symbol appears in red on either side of the word "Materials."

2. Because of the presence of radioactive and other materials in the laboratory, access to the entire laboratory is restricted. Access is governed by Procedure 3.1.A.11.0 Laboratory Security and Controlled Access.

I. PERSONNEL MONITORING DEVICES

1. All laboratory personnel will wear Thermoluminescent Dosimetry Badges at all times when in the laboratory. Badges should be attached to the lab coat lapel or breast pocket upon entering the laboratory.
2. When not in use, TLD badges will be stored in the badge box in the laboratory entrance. The badge box will be locked during non-working hours.
3. No radioactive materials may be placed on or in the badge box.

4. TLD badges may not be removed from lab coats and placed on work surfaces or on radioactive sources or materials at any time.
5. TLD badges will be collected, and new badges issued, on a quarterly basis by the Environmental Chemist.
6. TLD badges will be returned to the manufacturer for readout.
7. The manufacturer's TLD readout service will provide a quarterly Radiation Exposure Report on an NRC form 5 or other approved form.

J. BIOASSAY SAMPLES

1. Laboratory personnel will be required to submit a urine sample to the Environmental Chemist on a quarterly basis. The urine will be analyzed for gross alpha and gross beta minus K-40.
2. In the event of suspected contamination, additional urine samples may be requested by the Environmental Chemist.

K. WASTE DISPOSAL PROCEDURES

1. All solid radioactive waste, such as planchettes after analysis, shall be placed in the radioactive storage cabinet. The lid to the disposal container shall be kept closed.

2. Any spillage of radioactive waste shall be reported to the Environmental Chemist immediately. The Environmental Chemist will direct containment and decontamination.
3. Liquid wastes, generated during analysis, may be disposed of through the laboratory sinks into the laboratory sewer system using large volumes of running water.
4. The liquid sample remaining after the determination of Ra 226 by Radon deemanation shall be evaporated to dryness in the radioisotope fume hood and disposed of as solid waste.
5. The solid waste container will be checked for external contamination, decontaminated if necessary, transported to the mill tailings pit, and the contents buried on a monthly basis under the direct supervision of the Environmental/Radiation Department Manager or the Environmental Chemist.

L. EMERGENCY PROCEDURES

Emergency procedures are specified in the Laboratory Safety Manual.

M. RECEIPT OF PACKAGES CONTAINING RADIOACTIVE MATERIALS

Receipt of packages is covered under section D of this manual. All documentation will be maintained by the Environmental Chemist.

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6.1.A.46.0 LABORATORY ANALYTICAL PROCEDURE REFERENCES

A. ALL RADIOCHEMICAL PROCEDURES

Tennessee Valley Authority, Division of Environmental Planning Radiological Health Branch, Radioanalytical Laboratory Procedures.

U. S. Atomic Energy Commission, Health and Safety Laboratory, HASL Procedure Manual, 1972 Edition. HASL-300.

U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory. Radiochemical Analytical Procedures for Analysis of Environmental Samples, March, 1979. EMSL-LV-0539-17.

U. S. Environmental Protection Agency, Technical Support Laboratory, National Environmental Research Center. Handbook of Radiochemical Analytical Methods.

B. TRACE ELEMENT ANALYSIS

Federal Register, Volume 44 No. 233, Monday, December 3, 1979, Appendix IV. Inductively Coupled Plasma Optical Emission Spectrometric Method for Trace Element Analysis of Water and Wastes.

C. ALL OTHER WATER QUALITY ANALYSES

U. S. Environmental Protection Agnecy, Environmental Monitoring and

Support Laboratory. Methods for Chemical Analysis of Water and Wastes. March, 1979. EPA-600/4-79-020.

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Waste Water, 14th Edition, 1975.

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6.1.A.47.0 INSTRUMENT OPERATOR'S MANUALS

A. PURPOSE

To provide documentation of the proper operation of the instruments currently in the laboratory.

B. SCOPE

These operator's manuals are applicable to the specified instruments and to the uses required by the analytical procedures.

C. RESPONSIBILITIES

It is the responsibility of the Environmental Chemist to ensure that the operating procedures contained in the instrument operator's manuals are followed by the analyst and to evaluate the analytical results. It is the responsibility of the analyst to follow the procedures and report any abnormal results to the Environmental Chemist.

D. APPENDICES

The following operation manuals for each applicable instrument are provided in the appendices:

1. Appendix 46.0.0 - Canberra Model 2200 Low Level Alpha/Beta Counting System Operator's Manual
2. Appendix 46.0.1 - Nuclear Measurements Corporation Proportional Counter Operator's Manual
3. Appendix 46.0.2 - Canberra Model 8100 Alpha Spectrometer Operator's Manual
4. Appendix 46.0.3 - Ludlum Measurements, Inc. Model 2000 Portable Scaler Operator's Manual
5. Appendix 46.0.4 - Eberline Instrument Corporation Model MS-3 Single Channel Scaler Operator's Manual
6. Appendix 46.0.5 - Jarrell-Ash Model 850 Atomcomp Inductively Coupled Argon Plasma Operator's Manual
7. Appendix 46.0.6 - Bausch and Lomb Model 2000 Spectrophotometer Operator's Manual
8. Appendix 46.0.7 - Orion Research Corporation Model 701A Digital pH/mv Meter Operator's Manual

APPENDIX 2.3.0

OPERATING MANUAL PASSIVE RADON MONITORS

EDA

OPERATING MANUAL

RDT-310
PASSIVE RADON MONITOR

EDA INSTRUMENTS INC.

3813RDT3R8 Printed in Canada 1 Nov 79

WARRANTY

EDA Instruments Inc., warrants all manufactured equipment against defective material and workmanship for a period of one year from date of shipment. In the event of failure of equipment EDA Inc., at its expense will repair or replace any materials, equipment, work, or parts which prove defective or deficient under normal operating conditions. Except for the express warranty stated above, EDA Inc. disclaims all warranties of merchantability and fitness, and any stated express warranties herein are in lieu of all obligations or liability on the part of EDA Inc. for damages, including but not limited to special, indirect, or consequential damages arising out of, or in connection with, the use or performance of the equipment.

WARRANTY SERVICE

When warranty service is required or when technical consultation is required, contact:

In Canada:

EDA INSTRUMENTS INC.
1 Thorncliffe Park Drive
TORONTO, ONTARIO
CANADA, M4H 1G9
Telephone (416) 425-7800
TELEX 06-23222 EDA TOR

In U.S.A.

EDA INSTRUMENTS INC.
5151 Ward Road
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Colorado 80033
Telephone (303) 422 9112

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EDA PRODUCTS LINE

EDA Instruments Inc., is a leading designer and manufacturer of geophysical instruments and microprocessor-controlled systems. The company specializes in magnetics, exploration and mining, and in environmental control. Current equipment includes:

Magnetic:

- AMOS III Automatic Magnetic Observatory System
- FM-100 series Fluxgate Magnetometers for three-axis measurements
- PPM-105 series Proton Precession Magnetometers for total Magnetic Field Measurement and Analysis
- DIM-100 Measurement standard for declination and inclination readings

Exploration and Mining:

- RD-200 RDU-200 Radon and Radium Detectors
- DFS-300 Uranium Assayers
- GRS-400 Integral Gamma Ray Spectrometer Scintillometer
- GRS-500 Differential Gamma Ray Spectrometer Scintillometer
- BGA-300 Uranium Ore Analyzer
- RFB-300 Rotary Fusion Burner

Environmental:

- RDA-200 RDU-200 Radon/Radon Daughter and Radium Detectors
- RDT-322 Passive Radon Monitor
- RDT-310 Passive Radon Monitor (humidity controlled)
- MOD-225 Integrating Radon Daughter Monitors
- RDM-500 Continuous Microprocessor Controlled Radon Monitor System
- RDR-425 Continuous Radon Monitor

WARNING

The circuitry within this unit involves high voltage at all times. Ensure that battery power is disconnected when servicing internally. EDA suggests that when servicing is required, contact the marketing department at EDA first.

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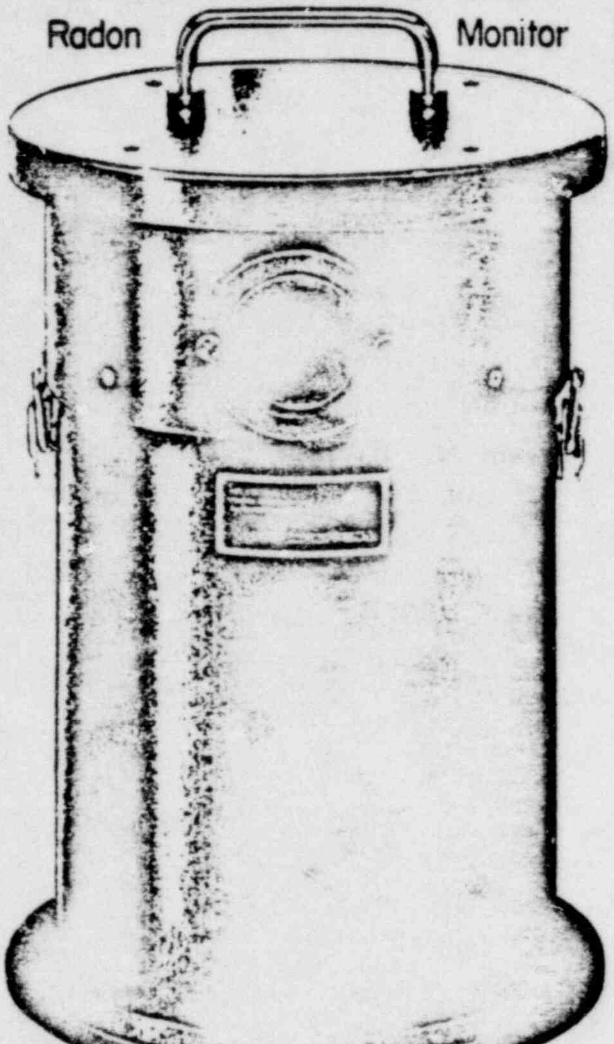
EDA



RDT-310 Passive Environmental Radon Monitor

SECTION 1

GENERAL INFORMATION

1.1 SCOPE OF MANUAL

This manual describes the RDT-310 Passive Radon Monitor manufactured by EDA Instruments Inc., Toronto, Canada. The information is provided for personnel responsible for operation of the unit.

1.2 PURPOSE OF UNIT

Radon gas is a contaminant in ambient air, which decays radioactivity by alpha emission. It occurs particuarly in areas of higher natural radioactivity, near uranium and phosphate mining and processing plants and also in the vicinity of coal-fired power plants.

The RDT-310 monitor provides a simple technique for measuring low radon levels over relatively long periods (ie, up to one month). This period ensures integration of temporal variations. The monitors are used to determine remedial programs or to study long-term biophysical effects. Typical applications include:

(a) Monitoring total radon exposure levels in residential and industrial environments.

(b) Local and regional baseline surveys of both natural and man-made radon concentrations.

(c) Time and space perturbation studies.

The unit design is based on current TLD (Thermoluminescent dosimeter) technology. Units are used extensively to monitor hostile as well as indoor environments which are contaminated by radon to ensure that the radon exposure levels do not exceed those established by government agencies.

Table 1-1 provides a technical sumary for this module.

SPECIFICATIONS TABLE I-1

Technique	Diffusion of radon through a desiccant bed and subsequent Electrostatic collection of short-lived Radon daughters on thermoluminscent chip and subsequent registration of resulting alpha activity.
Detector	LiF, 0.125"x0.125"x0.015"(3.18x3.18x 0.38mm) Harshaw TLD. Other chips including CaF:Dy may be used.
Interior Filter	Whatman #41
Detection Limit	0.03 pCi/l/week
Sensitive Volume	1.68 litres
Detector Response - alpha - gamma	For LiF, 7.8±0.3 counts/pCi hour/l .03$\overline{6}$ counts/ R/h per hour
Electrostatic Field	900 V DC
Power Supply	Three Eveready Mini-Max No. 493. Shock protected.
Battery Life	Normal shelf life in excess of one year
Temperature Range	Below 10°C to above 30°C
Desiccant	1.6kg activated silica gel, depth 67mm (2.6").
Response Time	5 hours with Whatman #41 Filter
Humidity Range	0-80% RH for extended periods
Effective Diffusion Area	$324 cm^2 (50 in^2)$
Weights & Dimensions Monitor	4.5kg (9.9lb) 250mm diameter x 350mm (9¾ x 14½")
Shipping	6.1kg (13.4lb) 355x355x480mm (14"x14"x19")
Standard System	Monitor complete with Batteries, Silica Gel, special shipping carton and Instruction Manual. TLD Chips available on request.

Options & Accessories

RDX-836 Spare TLD Holders complete with electrode
RDX-727 Battery Set (3)
RDX-308 Spare Desiccant Chamber
RDX-826 Battery Tester
- Replacement Silica Gel Desiccant, 1.6 kg
- 20cm Whatman #41 filter

DESCRIPTION

2.1 PHYSICAL DESCRIPTION

The RDT-310 is a portable, self-contained unit which operates from internal batteries. The unit is shown diagrammatically in Figure 2-1. It consists of two functional asemblies: an upper, stocky, cylindrical chamber, commonly referred to as the "top hat" and a lower base cyliner which contains the high voltage batteries and the two TLD (thermoluminescent dosimeter) chips.

2.2 UPPER CHAMBER

This upper compartment basically contains the silica gel desiccant that dries the ambient air to 0% relative humidity. This indicator-quality desiccant is kept in place by two perforated brass screens, in the top and base of this chamber. The bottom brass screen is sealed with a special high temperature asket to facilitate drying of the silica gel.

2.3 TLD CHIP HOLDER

The TLD chip is mounted onto a brass pin that has been inserted inside a black rubber stopper. The well in which this chip is mounted is encircled with a white plastic housing that secures the chip's position.

2.4 CHIP DETECTOR

The TLD chip is a square wafer with the dimensions 3mm x 3mm. The thickness will vary depending upon the type and specific application.

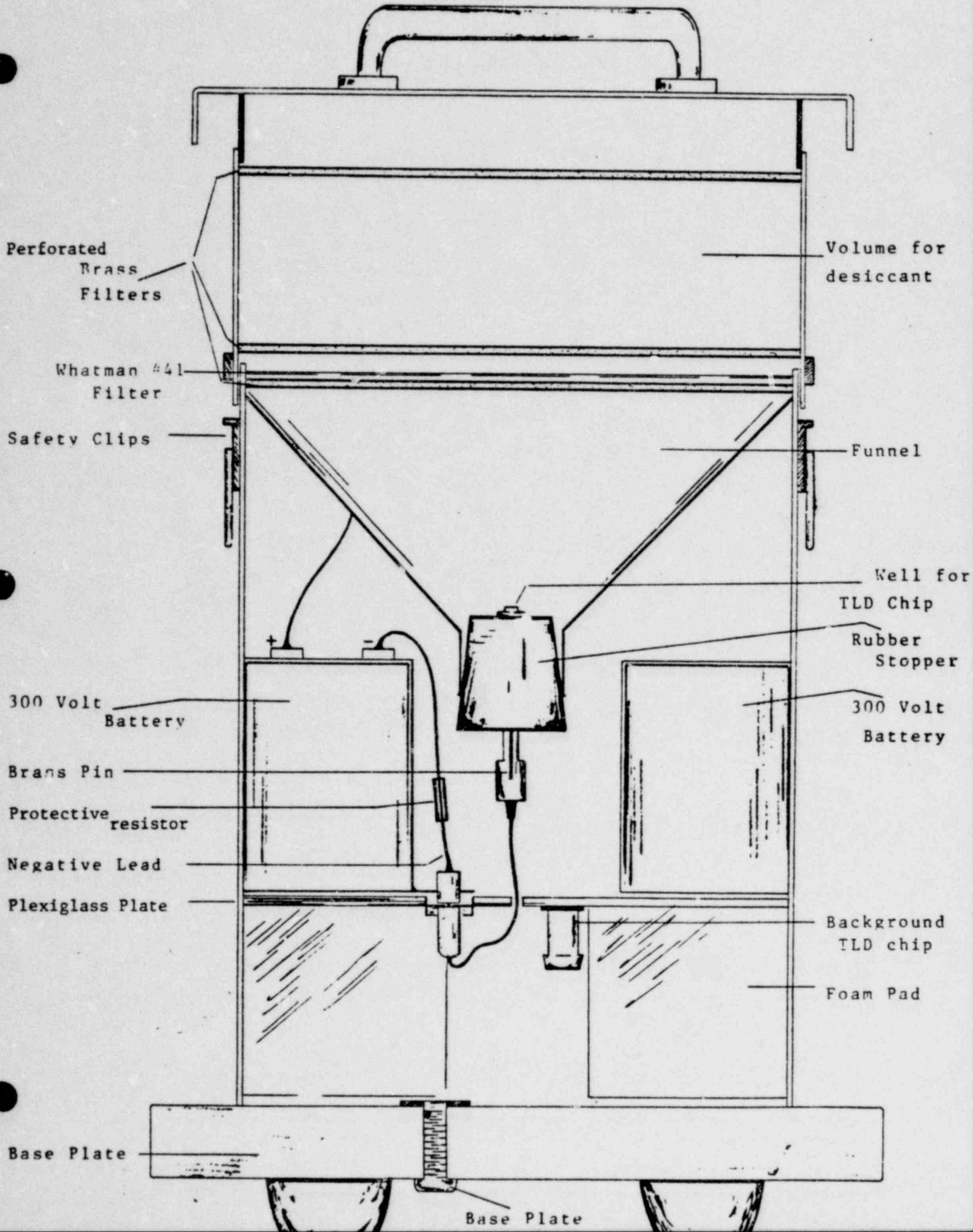
2.5 BACKGROUND CONTROL CHIP

A background control chip is housed inside a small vial that is mounted onto the underside of a triangular plexiglass plate in the base section of the monitor.

2.6 BASE SECTION

The base section contains three 300-volt batteries and a metallic funnel-shaped construction, designed to channel radon and radon daughter isotopes toward the TLD chip (securely mounted onto the rubber stopper).

Figure 2-1 RDT-310, Sectional Diagram



A perforated brass filter plus a Whatman #41 filter tops the base section of the monitor to clear the ambient air of all aerosol and particular matter.

By simply tightening the base screw on the underside of the base plate, insures that all the internal workings are securely in place.

2.7 ELECTRICAL CIRCUIT

The three batteries are connected in series to provide a source of 900 V DC. The positive terminal (right-hand banana clip) of one battery is connected to the negative terminal of the next sequential battery, with a small lead wire.

Each battery has its own bracket mount, designed to prevent accidental polarity reversals and unexpected contact with the battery terminals during installation. These battery holders also eliminate unnecessary handling of the interconnecting battery cables. Safety precautions have been afforded by a safety interlock and an in-series 10 megohm resistor.

As soon as the batteries are installed, the unit is operational.

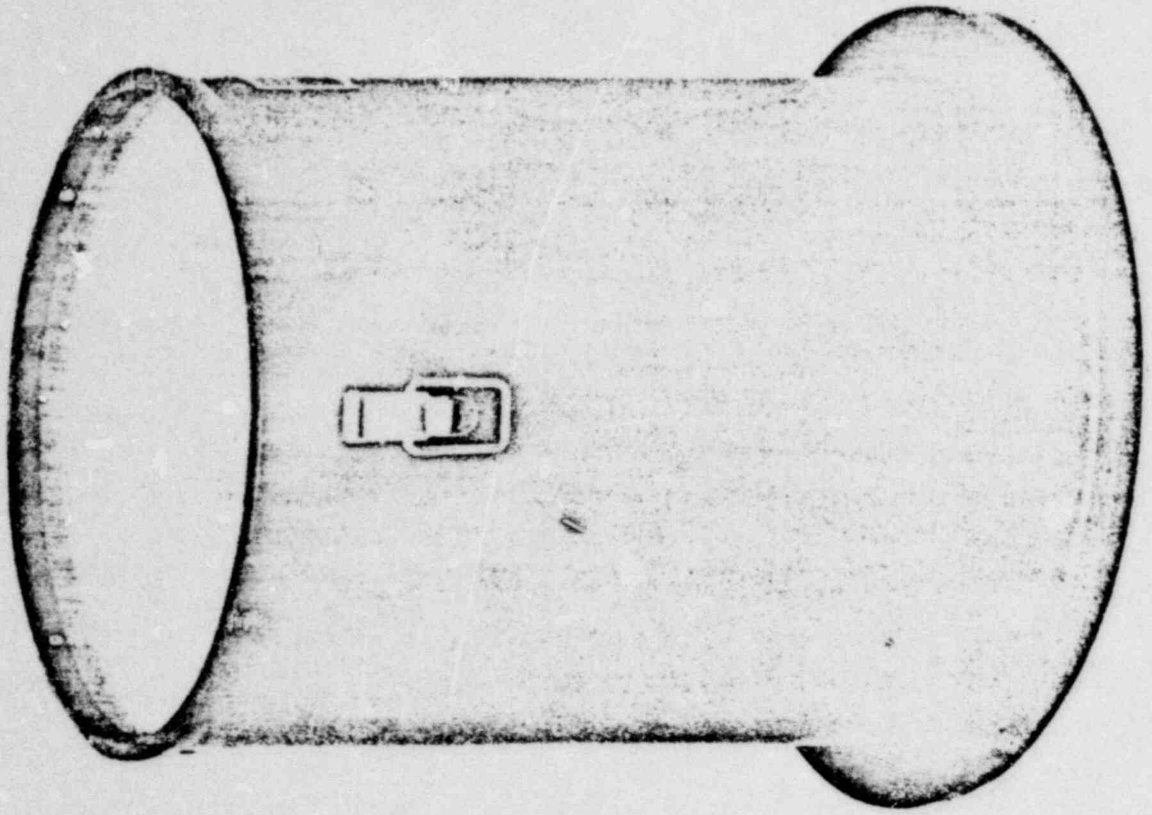
2.8 PRINCIPLES OF OPERATION

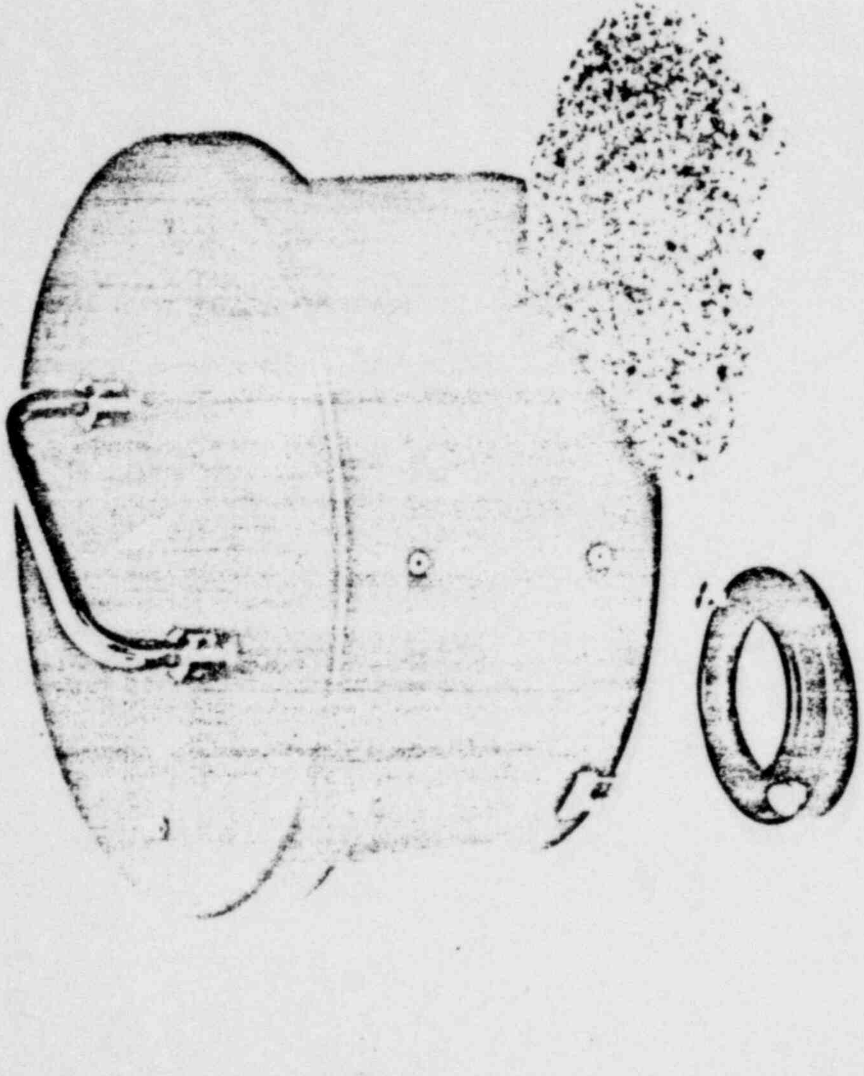
The battery circuit produces an electrostatic field within the lower chamber forming the effective sensitive volume.

When the unit is placed in the area to be monitored, the radon present in the atmospheric surroundings, is passed down (by diffusion) through a bed of silica gel desiccant. Here, the air is dried to 0% relative humidity. Over extended sampling periods, the air can be effectively desiccated in areas up to 85% humidity. The radon gas is then cleaned of all foreign matter as it passed down through the Whatman #41 filter (which separates the upper from the bottom segment of the unit).

Upon entering the sensitive lower chamber, the radon disintegrates producing positive ^{218}Po ions (Radium-A). Due to the strong 900 volt electrostatic field, created by the three 300-volt batteries, the ^{218}Po ions are drawn toward the TLD chip. Since the TLD chip is held at a negative potential (recall the section 2.7 on electrical circuit), each ^{218}Po ion breaks down into an alpha particle. Each of these, in turn, impart their energies (metastable electrons) onto the molecular structure of the chip. These chips can be either a highly-sensitive dysprosium-doped calcium fluoride chip or a less sensitive, but more stable lithium fluoride chip.

In the decay process and from terrestrial and cosmic sources, gamma rays are also detected by the chip. A gamma control chip, mounted in the plexiglass plate, detects only the gamma radiation. Therefore, when the chip contamination has been analyzed, the count from the control chip is subtracted from the count of the alpha chip.





SECTION 3

RADON MONITORING OPERATIONS

3.1 MONITORING CYCLE

A typical monitoring cycle consists of the following stages:

(a) Installation of TLD and control chips in unit.

(b) Exposure.

(c) Removal of chips and shipment.

(d) Processing chips and de-activation.

3.2 CHIP INSTALLATION

The chips are very fragile, transparent and easily contaminated with dirt. When the chips are installed in the holder, certain handling precautions must be observed.

(a) Do not touch the chip. Skin oils can contaminate the surface.

(b) When inserting the chips in the holder, use a suction device for handling.

(c) Do not drop the chip or cause any violent impact.

3.3 EXPOSURE

The monitoring method requires exposure of the chips to the air sample for an extended period so that an integrated value can be obtained. This period should be between one and two weeks. The activated chip should then be processed within one week after removal to obtain a valid value.

3.4 REMOVAL OF CHIPS AND SHIPMENTS

It is important to identify the face of the chip which is adjacent to the mylar (ie, the exposed face). This presents some difficulty because of the minute dimensions. One solution is to use the holder as the transportation device. In this mode, the chips remain undisturbed. However, it is imperative to protect the mylar tip and silver paint conducting surface.

(a) Loosen the base plate screw and lift open the hinged base of the monitor.

(b) Disconnect the red battery lead from its terminal, lift the triangular plexiglass plate, and remove the other half of the lead (black) from the brass pin.

(c) Remove the rubber stopper and obtain the TLD chip from its positioning well.

The TLD and control chips must be shipped together, expecially if air transport is used, so that they are exposed equally to ambient gamma radiation.

Install the new chips using the reverse sequence.

3.5 CHIP PROCESSING AND DE-ACTIVATION

The activated chips are processed in a reader to determine the accumulation of alpha particles. Conventional readers include Harshaw Model 2000 and 3000 Thermoluminescent Dosimetry Systems. The general procedure is as follows:

(a) The chip is cleaned. Then it is placed in the processor and annealed for 45 minutes at low temperature.

(b) The temperature is increased from 130°C to 250°C at a rate of 15°C per second. During this period, the activity is read and integrated to give a total count in nanocoulombs.

(c) The chip is then annealed again, but at a temperature of 450°C. This de-activates the chip to wipe the memory clean.

(d) The activity is calculated by subtracting the background count from the total TLD count.

SECTION 4

MAINTENANCE

4.1 INTRODUCTION

The monitor is passive, therefore, maintenance is minimal.

4.2 BATTERY CONDITION

The condition of the batteries is checked using an RDX-827 battery tester, Figure 4-1.

(a) Remove the chip holder (rubber stopper) from the unit.

(b) Insert the "dipstick" into the orifice until it contacts the base. The positive and negative terminals of the batteries, connected in series, are then connected across the tester.

(c) When the remaining battery power has dropped to 800 V, change the batteries.

4.3 BATTERY REPLACEMENT

Renew all four batteries simultaneously. Proceed as follows:

(a) Invert the unit. Loosen the base retaining screw and open the hinged base plate.

(b) Disconnect negative battery cable that goes to TLD chip holder. Lift off the triangular plexiglass plate. Remove the other end of the negative lead wire attached to brass pin and remove plexiglass assembly entirely.

(c) Simply detach the batteries from their banana clips and lift out.

(d) Replace with new batteries.

4.4 CALIBRATION OF TLD

Thermoluminescent dosimeters are not absolute radiation detectors. There is no way to relate directly to the thermoluminescence released from an irradiated sample to the amount of radiation exposure or to the radiation dose received by the dosimeter material. Consequently all TLD systems must be calibrated by exposing the dosimeter to known amounts of radiation, and the accuracy of the overall result can be no better than the accuracy of the calibration. The procedure used for calibration of a dosimeter depends upon the type of measurement to be made. The chip should be irradiated so that the TD response is related to the exposure.

For greatest calibration accuracy of the chip, the radioactive source used in the laboratory for this purpose should be the same as the source to be monitored in the field. Otherwise, a correction must be made for the possible difference in energy response of the chip between the sources.

4.5 DESICCANT CONDITION

Saturation of the crystals is indicated by a colour change from blue to white or clear. For highest efficiency, either renew the crystals or heat them in a laboratory or residential oven to remove the moisture.

APPENDIX 1

TLD CHIP DATA

A1-1 THERMOLUMINESENCE

Thermoluminescence is the release of light in a crystalline material as the material is heated. Perfect crystals do not exhibit thermoluminescence, because it is dependent upon trace quantities of impurities locked within the lattice structure of the crystals. When such an imperfect crystal is exposed to some form of external radiation, electrons of the atoms forming the lattice capture enough energy to become displaced from their shells. In this higher energy state, the free electrons diffuse through the crystal until they are captured and held more strongly by the atoms of the impurities. This phenomenon is referred to as a trap. The greater the radiation dosage, the more free electrons are produced and greater becomes the number of filled traps. Heating the crystal at a later time releases the extra kinetic energy stored in the trapped electrons, and for certain traps, the excess energy is liberated as light.

The light released is proportional to the previous radiation dosage received by the material. Also, thermoluminescence responds to the total dosage through time. Therefore, thermoluminescence can be a much more sensitive radiation indicator than a scintillation counter. Once the light is produced, the previous radiation history of the material is erased because thermal cycling performs a destructive readout of the past history of the sample. In this way, the thermoluminescent material can be re-used repeatedly with little loss of sensitivity.

There are many materials available commercially which exhibit thermoluminescence. They include: $CaSo_4$:Mn; $Li_2B_40_7$:Mn; and CaF_2:Dy. All of these are used in dosimetry, and generally are referred to as thermoluminescent dosimeters, or TLDs.

A1-2 TLD CHARACTERISTICS

Thermoluminescent sensitivity may be defined as the amount of light released by the TLD per unit of radiation exposure. The lower limit of useful sensitivity depends upon the characteristics of the chip and of the TLD reader. The upper limit of the useful range is generally limited by the TLD alone.

While many radiation detectors show a change in response as a function of dose rate, TLDs in general are linear over the full useful exposure range.

The term 'glow curve' refers to the graph of thermoluminescence as a function of either temperature or time. Usually it refers to the time function.

The glow curve of a TLD as a function of time probably best characterizes the chip. For example, the appearance of glow peaks only at low temperatures implies that the chip loses its stored TL with time, and therefore would be unsuitable for long-term measurements. A glow curve with peaks at very high temperatures indicates a chip which will produce infrared radiation at the temperature necessary to release the TL. This causes special instrumentation problems. A glow curve without well defined peaks makes the selection of the appropriate end point for integration difficult. Ideally then, a glow curve should reveal only a single thermoluminescent peak which occurs at a temperature high enough to insure room temperature stability, but not so high as to present instrument problems.

For routine uses of TLD, it is generally not necessary to record glow curves. It is, however, convenient to produce glow curves for initial screening of the chips obtained from the manufacturer, and also for situations when changes in the TLD are suspected.

The following factors may effect the shape of a glow curve:

(a) The heating rate and its uniformity.

(b) The size, shape and thermal conductivity of the sample.

(c) The recording instrument used.

(d) The level of exposure.

(e) The type of radiation.

(f) The radiation and annealing history of the sample and spurious effects.

A1-3 INSTRUMENTATION

The instrumentation which is needed to observe thermoluminescence is basically very simple: A method to heat the TLD, and an instrument to detect the emitted thermoluminescence. Therefore, all TLD reading instruments consist of the following basic components. These features are incorporated in most TLD readers that are available commercially:

(a) Heating and temperature control unit.

(b) Mechanical arrangement for positioning the phosphor on a heating pan into a dark chamber.

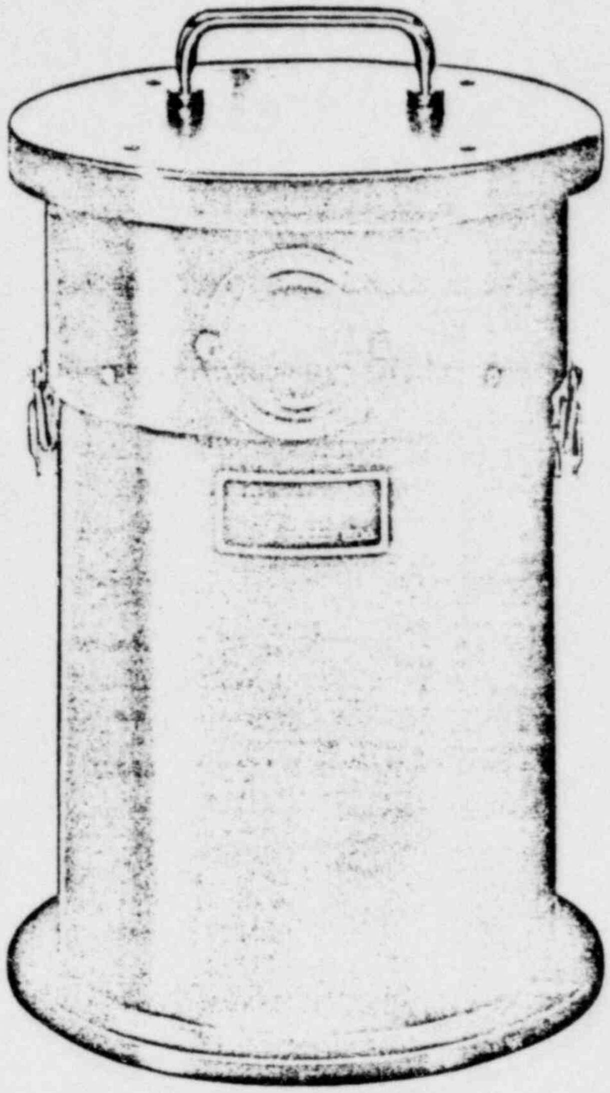
(c) Luminescence detector with optical filters.

(d) Power supply.

(e) Readout.

Spurious luminescence within the TLD reader can produce a significant error in TLD measurement. Most of the spurious luminescence is caused by oxygen in the air. Consequently, most TLD readers are capable of making measurements in a nitrogen atmosphere. This technique is referred to as 'nitrogen quenching'.

RDT-310
Passive Environmental Radon Monitor



Applications

With the rising public and medical concern for the effects of radon and its immediate series of daughter isotopes, a practical means of conducting long term integrated measurements was required. The RDT-310 alone or in conjunction with other integrating devices provides essential data on radon concentrations in a variety of environments. Various monitoring applications are:

☐ In the vicinity of uranium and phosphate tailings dumps.
☐ Pre-remedial studies at abandoned radium and uranium processing plants.
☐ In proximity to uranium, phosphate and coal stockpiles.
☐ Within residential areas including in-home sites.
☐ Around coal-fired power plants, geothermal areas and natural gas processing facilities.
☐ Inside buildings constructed of material such as granites which emanate radon.
☐ Studies of radon build-up resulting from energy conservation measures.
☐ Pre-mining baseline studies.
☐ General population exposure studies.

Description

The RDT-310 is a practical adaptation of proven techniques and designs originating at New York University and improved by the Environmental Measurements Laboratory of the U.S. Department of Energy. Its operation is simple and straight forward. Radon present in the ambient atmosphere passes down by diffusion, through a bed of indicator-quality silica gel and a single filter where it is desiccated to 0% relative humidity and cleared of all aerosol and particulates. It then enters the sensitive chamber where it disintegrates producing positive ^{218}Po ions (Radium-A). Under the strong influence of a 900 Volt electrostatic field, the ^{218}Po ion is immediately drawn toward a TLD (thermoluminescent dosimeter) chip held at a negative potential. There, each ^{218}Po ion resides until it, in turn, disintegrates producing an alpha particle. Each alpha particle striking the TLD chip imparts its energy to the crystalline molecular structure of the chip in the form of metastable elevated electrons.

In order to check the level of background gamma radiation, a control chip is used. This control chip is housed inside a small vial which is mounted onto a triangular plexiglass plate in the base of the unit.

After a period of time, the chip complete with holder is removed, along with the gamma control chip, and sent to a central location for analysis in a standard TLD reader. The resulting data in nanocoulombs, is simply converted using calibration curves to units of radon concentration.

Physically, the RDT-310 is well designed and constructed with dimensions optimized for efficiency, effectiveness and simplicity. Many features have been added to the original design. The entire container is suited for use in wet hostile environments yet it may be placed in homes where it blends inconspicuously and inoffensively with most furnishings. Access to the chip and the power supply is restricted by using special fasteners requiring a standard key. Further security may be added if desired by using padlocks or wired clips on the lock tab which is standard to the RDT-310. Protection against high voltage shock is afforded by a safety interlock and an in-series 10 megohm resistor. All wiring is rated at 5000 V DC. The design of the battery holder prevents accidental polarity reversals and unexpected contact with the battery terminals during installa-

RDT-31
Passive Environmental Radon Monitor

EDA

tion. The battery holders also eliminate handling of the interconnecting battery cables.

The top desiccant chamber containing the silica gel has an integral viewing / filling port. The entire chamber easily detaches and all components are rated to withstand temperatures up to 260°C (500°F) for prolonged periods. This allows the operator to reactivate the desiccant quickly and easily in a home or laboratory oven. Alternatively, the desiccant may easily be removed and fresh silica gel introduced.

The entire assembly is convenience oriented and requires only occasional maintenance, such as checking the battery supply, changing the chips or replacing the desiccant.

Features

☐ Integrates radon concentration for up to one month.
☐ High sensitivity, 0.03 pCi / l / week with LiF.
☐ Uses any of a variety of reuseable TLD materials.
☐ Simple, reliable and inexpensive.
☐ Reuseable Silica Gel desiccant in top canister may be reactivated at temperatures up to 260°C.
☐ Suitable for in-mine and residential installations.
☐ Secure against unauthorized entry.
☐ Built-in high voltage safety interlock.
☐ Backed by EDA's technical and service staff.

Specifications

Technique	Diffusion of radon through a desiccant bed and subsequent Electrostatic collection of short-lived Radon daughters on thermoluminscent chip and subsequent registration of resulting alpha activity.
Detector	LiF, 0.125" × 0.125" × 0.015" (3.18 × 3.18 × 0.38mm) Harshaw TLD. Other chips including CaF: Dy may be used.
Interior Filter	Whatman #41
Detection Limit	0.03 pCi / l / week.
Sensitive Volume	1.68 litres
Detector Response—alpha	For LiF, 7.8 ± 0.3 counts / pCi hour / l
—gamma	.036 counts / µR / h per hour
Electrostatic Field	900 V DC.
Power Supply	Three Eveready Mini-Max No. 493. Shock protected.
Battery Life	Normal shelf life in excess of one year
Temperature Range	Below 10°C to above 30°C.
Desiccant	1.6kg activated silica gel, depth 67mm (2.6").
Response Time	5 hours with Whatman #41 Filter
Humidity Range	0-80% RH for extended periods.
Effective Diffusion Area	324cm² (50in²).
Weights and Dimensions Monitor	4.5kg (9.9lb) 250mm diameter × 350mm (9¾" × 14½")
Shipping	6.1kg (13.4lb) 355 × 355 × 480mm (14" × 14" × 19").
Standard System	Monitor complete with Batteries, Silica Gel, special shipping carton and Instruction Manual. TLD Chips available on request.
Options and Accessories	RDX-836 Spare TLD Holders complete with electrode RDX-727 Battery Set (3) RDT-308 Spare Desiccant Chamber RDX-826 Battery Tester —Replacement Silica Gel Desiccant, 1.6 kg. —20cm Whatman #41 filter

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APPENDIX 2.5.0

OPERATING INSTRUCTIONS

LUDLUM MODEL 19 SCINTILLOMETER

INSTRUCTION MANUAL

MODEL 19 MICRO R METER

I. INTRODUCTION

The Ludlum Model 19 Micro R Meter utilizes an internally mounted 1" x 1" NaI(Tl) scintillator to offer an optimum performance in counting low level gamma radiation. Designed to be moisture and dust resistant, conveniences are not overlooked as the unit features a push button lighted meter.

Five range divisions are provided to select the most desirable range in the 0-5000 micro R/Hr spectrum. The meter face is made up of two scales 0-50 and 0-25, plus two scales for the battery test. The 0-50 scale corresponds to the 50, 500 and 5000 positions on the range selector switch. The 0-25 scale corresponds to the 25 and 250 positions on the range selector switch.

The instrument is capable of using either the standard carbon zinc battery or the nickel cadmium rechargeable battery. However, the Model 19 does not include circuitry for recharging the batteries. The BAT. TEST scales are marked to indicate 0%, 50%, and 70-100% charge (solid green area).

Designed to provide the monitor with the electronic capability generally associated with fixed laboratory devices, this unit incorporates design features such as -- Non overloading linear amplifier -- Wide pulse amplitude discrimination range -- Negative feedback stabilization -- Temperature compensation -- Regulated power supply -- Elimination of marginal circuits, component selection, critical adjustments - and Fully transistorized.

All controls, including a calibration potentiometer for each range, are located on the front panel. Two "D" cell batteries are located in an isolated compartment, and easily changed from the front panel. The meter is housed in a rugged, two piece aluminum bezel with an "O" ring seal.

1.1 SPECIFICATIONS

Linearity: Plus or minus 5% of full scale.

Input Impedance: 0.1 Megohm

High Voltage: Variable from 400 to 1500 volts DC electronically regulated to within -1%

Calibration Stability: Less than 15% variance to battery end point.

Battery: Exceeds 100 hours when using standard flashlight "D" cells. RM-42R Mercury cells or nickel cadmium cells are directly interchangeable.

Audio output: Unit is furnished with built-in unimorph speaker, ON-OFF switch provided on front panel.

1.1 SPECIFICATIONS, cont'd

Battery Complement: Two standard size "D" cell batteries, secured with screws and a gasket for dust and moisture proofing.

Counting Ranges: Two scale (and two color) meter face presenting 0-50 Micro R/Hr in black with full scale range positions of X5000, X500, and X50; and, in red, 0-25 Micro R/Hr with range selections of X250 and X25.

Meter: 50 micro-amp, 2 1/2" diameter, taut-band suspension.

Detector: An RCA 6199 coupled to a 1" x 1" NaI(Tl) mounted inside the instrument housing.

Finish: Instrument housing is of drawn and cast aluminum fabrication with brown epoxy paint and silk screened nomenclature. Switches are rubber booted.

Size: 6.4 x 3.5 x 7.0 inches (H x W x L exclusive of handle).

Weight: 4.5 lbs.

1.2 DESCRIPTION OF CONTROLS AND FUNCTIONS

OFF-RANGE SELECTOR SWITCH: A six position switch for turning the instrument OFF or selecting one of 5 counting ranges.

AUDIO ON-OFF TOGGLE SWITCH: The audio ON-OFF only controls the audio speaker output.

FAST-SLOW TOGGLE SWITCH: Selects reaction time of meter. In "F" position, full scale deflection requires 3 seconds. In "S" position, 11 seconds is required.

BATTERY PUSH BUTTON SWITCH: When range selector is out of the OFF position, depressing the BAT switch indicates battery charge status on the meter. The appropriate BAT test scale should be used depending upon the type of batteries installed.

RESET: Push button switch provides a rapid means to reset meter from an overextending meter deflection. Depressing the "RES" button grounds the meter and causes the needle to return to zero position.

LIGHT PUSH BUTTON SWITCH: Depressing "L" button lights the meter face.

HIGH VOLTAGE SCREW DRIVER ADJUST: Adjusts the high voltage from 400 to 1500 volts.

RANGE CALIBRATION SCREW DRIVER ADJUSTS (5): Provides means to calibrate each range.

LUDLUM MODEL 19 MICRO R METER

2. OPERATING PROCEDURES

The Model 19 is a simple instrument to operate. All controls and adjustments are located on the front panel along with the battery compartment. The 1" x 1" NaI(Tl) Scintillator is mounted internally deleting external cords or cables. The instrument is set at the factory for 60 KEV cut-off. If recalibration is required, refer to calibration procedures.

2.1 PRIOR TO TURN-ON.

1. Check batteries - type installed and no corrosion
2. Audio On-Off switch - as desired
3. Meter response switch- as desired.

2.2 TURN-ON

1. Range Selector Switch - Select 0-5000 range.
2. BAT Test Button - depressed.
   Check BAT test on appropriate scale. Replace batteries if meter pointer is below the solid green area.
3. Light Button - Depressed. Check for light on meter face.
4. Meter Response Switch - Check "F" and "S" positions.
5. Audio On-Off Switch - Check for audio indication.
6. Check instrument for proper scale indication with known source.
   Check all ranges for appropriate scale indication
7. Reset Button - Depressed. Check meter pointer returns to zero position.
8. Instrument ready for monitoring.

3. CALIBRATION

Calibrating the Model 19 requires some knowledge of pulser test equipment. The container must be removed from the rest of the instrument before calibrating. Unsnap the two latches and lift the front panel and the rest of the instrument out of the container. Disconnect the detector high voltage lead (white wire) from the circuit board and connect the pulser in its place. The following calibration procedure is used at the factory to set a 60 KEV cut-off, which results in the same scale presentation for energies above 60 KEV.

The input pulse to the Model 19 from the pulser should be a negative 40 MV pulse, five μsecond in duration with a 1 μsecond rise time or less and a tail of 10-20 μsecond.

3. CALIBRATION, cont'd

The instrument is first calibrated in counts per minute (CPM) on the 0-5000 and 0-500 ranges. This is accomplished by supplying 400,000 CPM and 40,000 CPM respectively from the pulser and calibrating on the 40 mark on the meter face. AM-241 is used to provide the 60 KEV energy. The high voltage is adjusted until the meter no longer increases with an increase of high voltage (just on the plateau). The meter indication is read as CPM and divided by five which provides a calibration point for the 0-500 scale. The appropriate CPM is supplied by the pulser to calibrate the 0-500 scale.

To calibrate the rest of the ranges, the same technique is used. After the 0-500 range is calibrated, the 0-250 range is selected and calibrated to the appropriate mark. The pulser is then switched one decade lower and the 0-25 range is selected and calibrated. The 0-50 scale is then selected and calibrated using the 0-25 CPM value.

Some non-linearity exists in the instrument, therefore, a calibration of actual micro R/Hr vs. meter indication from the calibration range is provided for reference.

Example:

1. Remove container
2. Disconnect selector high voltage lead from circuit board.
3. Connect pulser to circuit board (where detector lead was disconnected).
4. Turn on instrument and pulser.
5. Select 0-5000 range.
6. With 400,000 CPM input, calibrate range at 40 scale mark.
7. Decrease one decade (40,000) on pulser and select 0-500 range. Calibrate range on 40 scale mark.
8. Turn-off instrument and pulser.
9. Disconnect the pulser and reconnect the internal detector.
10. Replace container and turn-on instrument.
11. Place AM-241 source on front of container.
12. Adjust high voltage until meter indication "just" on the plateau. Using the 10 mark as an example corresponds to 100,000 CPM.
13. Remove container and reconnect pulser to instrument.
14. Dividing CPM (100,000) by 5 = 20,000 CPM.
15. Select and calibrate the 0-500 range at the 20 scale mark with a 20,000 CPM input.
16. Select 0-250 range. Calibrate on the 20 scale mark (red scale)
17. Decrease pulser one decade (2000 CPM).
18. Select 0-25 range (red scale). Calibrate on the 20 scale mark.
19. Select 0-50 range (black scale). Calibrate on the 20 scale mark.

This completes the calibration procedure.

4. MAINTENANCE

Instrument maintenance consists of keeping the instrument clean, periodic battery and calibration maintenance.

An instrument calibration check should be performed as required to keep the instrument in proper working order. Normally recalibration should not be required if the batteries are maintained in good condition.

At three month intervals, the batteries should be removed and the battery contacts cleaned of any corrosion. If the instrument has been exposed to very dusty or corrosive atmosphere, more frequent battery servicing should be used.

Use a spanner wrench to unscrew the battery contact insulation, exposing the internal contacts and battery spring. Removing the handle will facilitate access to these contacts.

NOTE: Never store instrument over 30 days without removing batteries. Although the instrument will operate at very high ambient temperatures, battery seal failure can occur at temperatures as low as 100°F. Neglected battery seal failure can cause excessive corrosion and render the instrument inoperative.

APPENDIX 2.8.0

METHODS FOR CALCULATION OF PARTICULATES AND CALIBRATION

SYMBOLS AND NOMENCLATURE

Symbols that are used throughout this paper and their definitions are given here. Those symbols used only in one section of the report are defined when they are used. Subscripts and/or superscripts are employed to differentiate between specific situations.

ΔH = Pressure drop across the secondary calibration device (i.e., orifice calibration unit or reference flow device), in H_2O.

I = Flow rate indication (rotameter reading or pressure transducer recording) arbitrary scale.

P_a = Barometric pressure as read from a barometer, mmHg.

P_{std} = Standard absolute pressure, 760 mmHg (29.92 in. Hg).

Q_r = Flow rate that would occur at standard reference conditions of 298 K (25°C) and 760 mmHg for a specific pressure indication (ΔH or I) observed at any temperature and pressure.

Q_{std} = The rate of flow of a standard volume.

TSP = Total suspended particulates, ug/m^3.

T_a = Ambient temperature, K. (273° + ambient temp. °C)

T_{std} = Standard temperature, 298 K (25°C).

t = Time the hi vol sampler is in operation, minutes.

Orifice unit equation:

$$Q_r = a\,\Delta H^b$$

Constants a and b are determined from EPA Region VIII orifice calibration data.

Hi vol sampler regression equation:

$$Q_r = aI \pm b$$

Constants a and b are determined from hi vol sampler calibration data. Constant a is equivalent to slope and b to the intercept.

II. Calibration of High Volume Sampler[1]

The hi vol sampler calibration procedure requires only the measurement of the following: (1) the pressure drop across the orifice ΔH in inches of water for a fixed flow rate and (2) the flow rate indication (I). This may be determined by use of a rotameter or a pressure transducer-recorder.

Ambient temperature and barometric pressure measurements are not required at the time the hi vol sampler is calibrated.

There are two different procedures that may be used to calibrate a hi vol sampler. The first is the "plates procedure" described in 40 CFR 50 Appendix B - Reference Method for the Determination of Suspended Particulates in the Atmosphere. This is the reference method and can be used on a sampler using a rotameter or a pressure transducer-recorder to indicate flow. The second calibration procedure, which is not universally usable, involves the use of a variable transformer or variac in place of resistance plates. This procedure permits a person calibrating a sampler to generate 5-10 different flow rates. Because the use of the resistance plates is preferred this procedure will reflect this use. In some areas of high altitude, it may be desirable to have additional resistance plates to better cover the operating range of the hi vol sampler.

(Note: The variac calibration procedure must not be used on samplers with a rotameter as a flow indicator. This procedure produces erroneous data when used on rotameter-type samplers.)

A mass flow controlled sampler cannot be calibrated in the same manner as a standard hi vol. This is discussed in detail in Appendix B.

A. Calibration Procedure

Detailed instructions on the mechanical aspects of maintenance and calibration of a high volume sampler will not be provided in this paper. If more information is required, consult the red Quality Assurance Handbook, Vol. II (Reference 3) or the manufacturer's instruction manual provided with the high volume sampler. Sampler motors with new brushes should not be calibrated until after the brushes have been properly seated to the armature. This is accomplished by running the motor with a #18 resistance plate or a clean filter in place for a minimum of 20 minutes.

Calibration and calculations should occur on site. However, both processes can be performed at a central location and the sampler transported to the site. If the sampler is calibrated at a central location, the operator must verify that the calibration has not been altered in transit. There should be a minimum of five

Figure 1

HI VOL SAMPLER CALIBRATION DATA SHEET

Sampler Location________________________

Orifice Unit No.____________ Calibrated by:____________

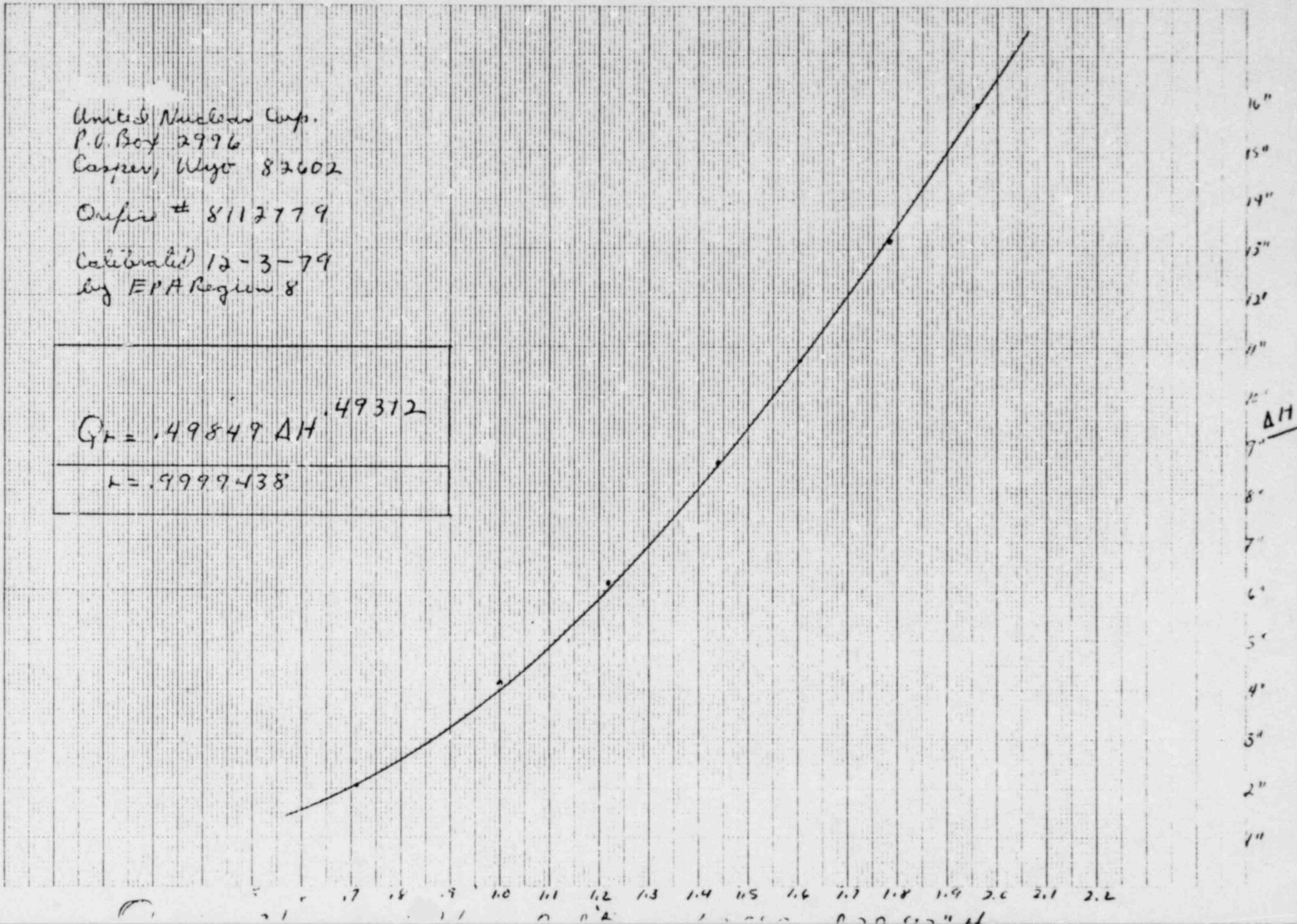
Sampler No.____________ Date____________

Indicator No.____________ $Q_r = aI \pm b$ ____________
(Rotameter/Recorder)

Correlation coefficient of r = ____________ $Q_r =$ ______ I ____________

Run Number	Voltage or Plate Number	ΔH Manometer in. Water		I Indicator Reading	Q_r Flow Rate* cmm
		Left Right	Total		
1					
2					
3					
4					
5					
6					
7					
Dup-1					
Dup-2					

*Flow rate from orifice unit calibration chart or equation.



United Nuclear Corp.
P.O. Box 2996
Casper, Wyo 82602

Orifice # 8117779

Calibrated 12-3-79
by EPA Region 8

$Q_r = .49849\,\Delta H^{.49372}$

$r = .9999438$

Hi Vol Sampler Calibration Curve

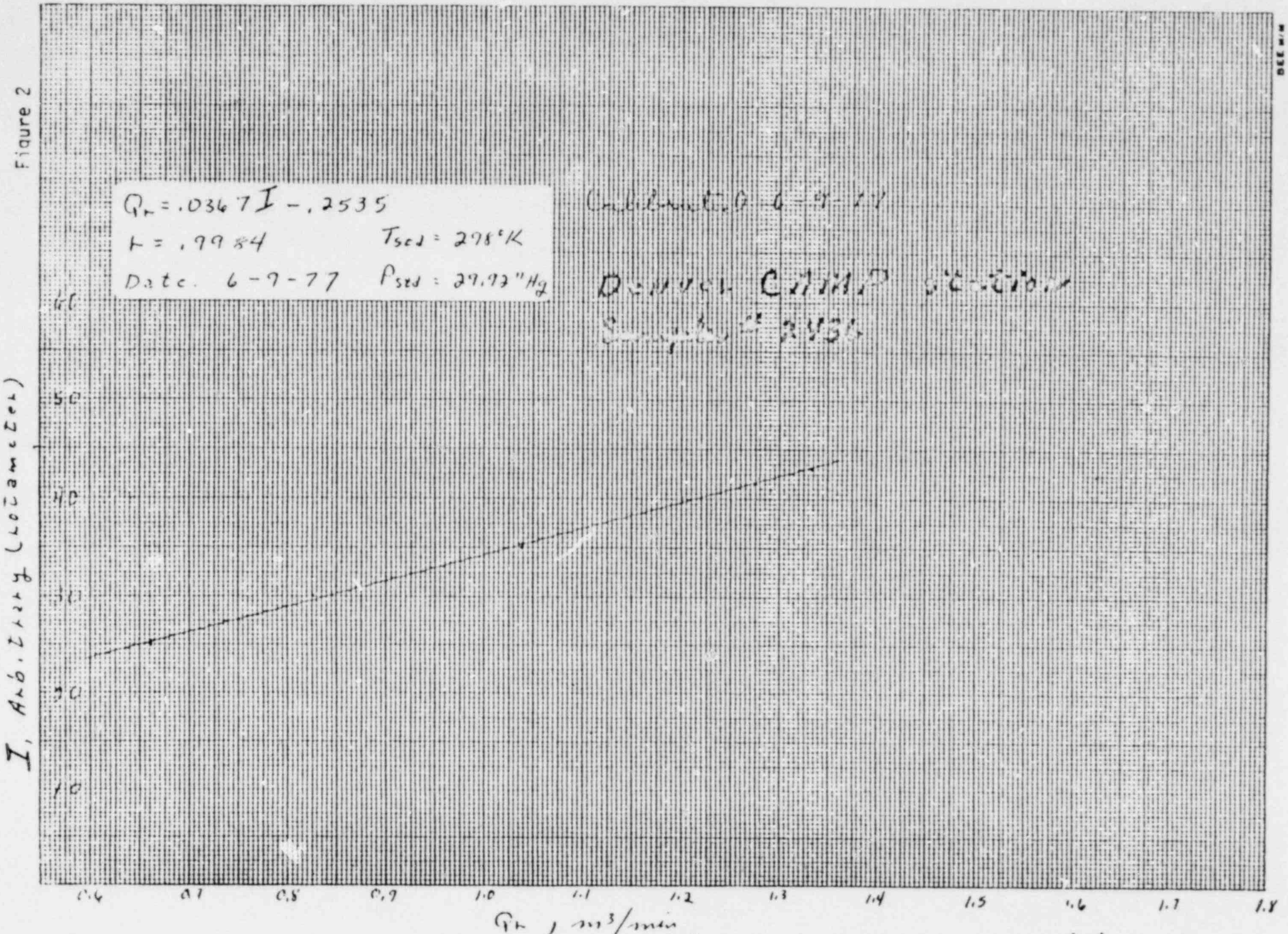


Figure 2

$Q_r = .0367 I - .2535$

$r = .9984$ $T_{std} = 298°K$

Date. 6-9-77 $P_{std} = 29.92''Hg$

Calibrated 6-9-77

Denver CAMP station

Sampler # 2456

points for each calibration curve. For each point, there should be a ΔH reading taken from the orifice manometer in inches of water. The example data sheet (Figure 1) has a blank for the left and right arm of the manometer reading and a blank for the total manometer reading. At the same time a ΔH reading is taken, a flow indicator (I) reading must be taken and recorded on the data sheet. This may be the reading taken from a rotameter (read the middle of the ball) or from the pressure transducer-recorder chart. Determine the flow rate Q_r for each ΔH by reading directly from the orifice calibration curve or calculate from its curve equation. A calibration curve should be prepared on linear graph paper by plotting flow rate Q_r vs. flow indication I. A linear regression equation of Q_r vs I may be calculated using the equation: $Q_r = aI \pm b$

An acceptable hi vol sampler calibration curve should consist of five points and with a correlation coefficient of $r \geq 0.99$, and no point deviating more than $\pm$ 0.02m^3/min from the value calculated by the regression equation.

B. Calibration Calculation

Calculations should be performed on site and during the calibration of the hi vol sampler. Items needed are:

1. Orifice calibration curve or
2. Calculator with y to x power capability and the orifice calibration curve equation.
3. Hi vol sampler calibration data sheet and linear graph paper.

C. Calculation Procedure

1. Calculate the flow rate Q_r for each ΔH using the orifice curve equation and a calculator, or convert each ΔH value to flow rate Q_r by reading the orifice calibration graph. Repeat the above calculation of Q_r for each ΔH value.

2. Prepare a calibration curve of Q_r vs. I on linear graph paper. Include the date and name of the individual that performed the calibration and sampler location and identification.

3. Compute a linear regression of Q_r on I.

See Appendix A for an example of calculation using actual data.

III. Field Use of High Volume Sampler

Four determinations are required during sample collection in order to calculate the volume of air sampled and to correct that volume to reference conditions. These four determinations are:

1. Average flow rate (Q_r) for the sampling period.

2. Sampling period time (t) in minutes.

3. Average ambient temperature (T_a) during the sampling period.

4. Average barometric pressure (P_a) during the sampling period.

The first two measurements, average flow rate and sampling period time in minutes, are the most important and require on-site measurement. Average ambient temperature and barometric pressure can be measured on site or adequately approximated in any of several ways. Recommended procedures for determining or estimating these values are as follows:

Ambient Temperature Determinations. An accurate estimate of the average temperature for a 24-hour sampling period is difficult to obtain without a continuous recorder. Some alternate procedures which could be used to obtain estimates of average temperature are listed below in order of preference:

1. For sites close to or as a part of an air quality monitoring station equipped to monitor ambient temperature, the average temperature for the day recorded by the station's temperature sensor should be used for the average temperature for the sampling period.

2. For high volume sampling sites visited daily, a mini-max thermometer giving the minimum and maximum temperature occurring since the last reset could be located on-site, and the average of the two extremes used for the sampling period average.

3. For sites not too far removed from a weather station and having about the same degree of exposure as the weather station, the average daily temperature as recorded by the weather station would provide an adequate estimate of the average sampling period temperature.

Barometric Pressure Determinations.

1. When requesting temperature information from a weather station or airport as in 1(c), also request the average daily "station pressure."

2. For sites not located near a weather station or airport, the barometric pressure may be regarded as a constant. Barometric pressure seldom varies more than ± five percent from the average. Therefore, if the site elevation is known the average barometric pressure constant can be taken from Table 2.

Field Measurement Procedures. Procedures for a high volume sampler equipped with a rotameter are as follows:

1. Make a rotameter reading at the start I_i and at the end I_f of the sampling period. Operate the sampler motor for five minutes to allow it to warm up and reach equilibrium before taking each rotameter reading. Measure or estimate the average ambient temperature T_a and average barometric pressure P_a for the sampling period.

2. Determine the initial flow rate Q_{ri} and the final flow rate Q_{rf} corresponding to I_i and I_f, respectively, from the hi vol sampler calibration curve.

3. Calculate the average flow rate (Q_r) for the sampling period:

$$Q_r = \frac{Q_{ri} + Q_{rf}}{2}$$

4. Calculate the average flow rate of a standard volume (Q_{std}) in std m^3/min. at T_{std} and P_{std} from Q_r by:

$$Q_{std} = Q_r \left[\frac{P_a}{T_a} \; \frac{T_{std}}{P_{std}}\right]^{\frac{1}{2}}$$

5. Calculate the volume of air sampled at standard conditions as:

$$V(\text{std m}^3) = Q_{std}\ (\text{std m}^3/\text{min}) \times t(\text{min}).$$

6. Calculate TSP using the weight of particulate W_p (grams) on the filter and the volume of air sampled at standard conditions.

$$\text{TSP}\ (\text{ug/std m}^3) = \frac{\text{Wp (g)} \times 10^6(\text{ug/g})}{\text{V(std M}^3)}$$

Table 2.1

BAROMETRIC PRESSURE AT VARIOUS ALTITUDES

[Abridged from the Smithsonian Tables]

Values of $60368\,[1+0.0010195\times 36]\cdot\log.\ \frac{29.90}{B}$

Barometric pressure, B inches	0	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
	Feet	*Feet*	*Feet*	*Feet*	*Feet*	*Feet*	*Feet*	*Feet*	*Feet*	*Feet*
17.00	15,347	15,331	15,315	15,299	15,283	15,267	15,251	15,235	15,219	15,203
.10	15,187	15,172	15,156	15,140	15,124	15,108	15,092	15,076	15,061	15,045
.20	15,029	15,013	14,997	14,982	14,966	14,950	14,934	14,919	14,903	14,887
.30	14,871	14,856	14,840	14,824	14,809	14,793	14,777	14,762	14,746	14,730
.40	14,715	14,699	14,684	14,668	14,652	14,637	14,621	14,606	14,590	14,575
17.50	14,559	14,544	14,528	14,512	14,497	14,481	14,466	14,451	14,435	14,420
.60	14,404	14,389	14,373	14,358	14,342	14,327	14,312	14,296	14,281	14,266
.70	14,250	14,235	14,219	14,204	14,189	14,173	14,158	14,143	14,128	14,112
.80	14,097	14,082	14,067	14,051	14,036	14,021	14,006	13,990	13,975	13,960
.90	13,945	13,930	13,914	13,899	13,884	13,869	13,854	13,839	13,824	13,808
18.00	13,793	13,778	13,763	13,748	13,733	13,718	13,703	13,688	13,673	13,658
.10	13,643	13,628	13,613	13,598	13,583	13,568	13,553	13,538	13,523	13,508
.20	13,493	13,478	13,463	13,448	13,433	13,418	13,404	13,389	13,374	13,359
.30	13,344	13,329	13,314	13,300	13,285	13,270	13,255	13,240	13,226	13,211
.40	13,196	13,181	13,166	13,152	13,137	13,122	13,107	13,093	13,078	13,063
18.50	13,049	13,034	13,019	13,005	12,990	12,975	12,961	12,946	12,931	12,917
.60	12,902	12,888	12,873	12,858	12,844	12,829	12,815	12,800	12,785	12,771
.70	12,756	12,742	12,727	12,713	12,698	12,684	12,669	12,655	12,640	12,626
.80	12,611	12,597	12,583	12,568	12,554	12,539	12,525	12,510	12,496	12,482
.90	12,467	12,453	12,438	12,424	12,410	12,395	12,381	12,367	12,352	12,338
19.00	12,324	12,310	12,295	12,281	12,267	12,252	12,238	12,224	12,210	12,195
.10	12,181	12,167	12,153	12,138	12,124	12,110	12,096	12,082	12,068	12,053
.20	12,039	12,025	12,011	11,997	11,983	11,969	11,954	11,940	11,926	11,912
.30	11,898	11,884	11,870	11,856	11,842	11,828	11,814	11,800	11,786	11,772
.40	11,758	11,744	11,730	11,716	11,702	11,688	11,674	11,660	11,646	11,632
19.50	11,618	11,604	11,590	11,576	11,562	11,548	11,534	11,520	11,507	11,493
.60	11,479	11,465	11,451	11,437	11,423	11,410	11,396	11,382	11,368	11,354
.70	11,340	11,327	11,313	11,299	11,285	11,272	11,258	11,244	11,230	11,217
.80	11,203	11,189	11,175	11,162	11,148	11,134	11,121	11,107	11,093	11,080
.90	11,066	11,052	11,039	11,025	11,011	10,998	10,984	10,970	10,957	10,943
20.00	10,930	10,916	10,903	10,889	10,875	10,862	10,848	10,835	10,821	10,808
.10	10,794	10,781	10,767	10,754	10,740	10,727	10,713	10,700	10,686	10,673
.20	10,659	10,646	10,632	10,619	10,605	10,592	10,579	10,565	10,552	10,538
.30	10,525	10,512	10,498	10,485	10,472	10,458	10,445	10,431	10,418	10,405
.40	10,391	10,378	10,365	10,352	10,338	10,325	10,312	10,298	10,285	10,272
20.50	10,259	10,245	10,232	10,219	10,206	10,192	10,179	10,166	10,153	10,139
.60	10,126	10,113	10,100	10,087	10,074	10,060	10,047	10,034	10,021	10,008
.70	9,995	9,982	9,968	9,955	9,942	9,929	9,916	9,903	9,890	9,877
.80	9,864	9,851	9,838	9,825	9,812	9,799	9,786	9,772	9,759	9,746
.90	9,733	9,720	9,707	9,694	9,681	9,668	9,655	9,642	9,629	9,617
21.00	9,604	9,591	9,578	9,565	9,552	9,539	9,526	9,513	9,500	9,487
.10	9,474	9,462	9,449	9,436	9,423	9,410	9,397	9,384	9,372	9,359
.20	9,346	9,333	9,320	9,307	9,295	9,282	9,269	9,256	9,244	9,231
.30	9,218	9,205	9,193	9,180	9,167	9,154	9,142	9,129	9,116	9,103
.40	9,091	9,078	9,065	9,053	9,040	9,027	9,015	9,002	8,989	8,977
21.50	8,964	8,951	8,939	8,926	8,913	8,901	8,888	8,876	8,863	8,850
.60	8,838	8,825	8,813	8,800	8,788	8,775	8,762	8,750	8,737	8,725
.70	8,712	8,700	8,687	8,675	8,662	8,650	8,637	8,625	8,612	8,600
.80	8,587	8,575	8,562	8,550	8,538	8,525	8,513	8,500	8,488	8,475
.90	8,463	8,451	8,438	8,426	8,413	8,401	8,389	8,376	8,364	8,352
22.00	8,339	8,327	8,314	8,302	8,290	8,277	8,265	8,253	8,240	8,228
.10	8,216	8,204	8,191	8,179	8,167	8,154	8,142	8,130	8,118	8,105
.20	8,093	8,081	8,069	8,056	8,044	8,032	8,020	8,008	7,995	7,983
.30	7,971	7,959	7,947	7,935	7,922	7,910	7,898	7,886	7,874	7,862
.40	7,849	7,837	7,825	7,813	7,801	7,789	7,777	7,765	7,753	7,740
22.50	7,728	7,716	7,704	7,692	7,680	7,668	7,656	7,644	7,632	7,620
.60	7,608	7,596	7,584	7,572	7,560	7,548	7,536	7,524	7,512	7,500
.70	7,488	7,476	7,464	7,452	7,440	7,428	7,416	7,404	7,392	7,380
.80	7,368	7,356	7,345	7,333	7,321	7,309	7,297	7,285	7,273	7,261
.90	7,249	7,238	7,226	7,214	7,202	7,190	7,178	7,166	7,155	7,143
23.00	7,131	7,119	7,107	7,096	7,084	7,072	7,060	7,048	7,037	7,025
.10	7,013	7,001	6,990	6,978	6,966	6,954	6,943	6,931	6,919	6,907
.20	6,896	6,884	6,872	6,861	6,849	6,837	6,825	6,814	6,802	6,790
.30	6,779	6,767	6,755	6,744	6,732	6,721	6,709	6,697	6,686	6,674
.40	6,662	6,651	6,639	6,628	6,616	6,604	6,593	6,581	6,570	6,558

See Reference No. 2

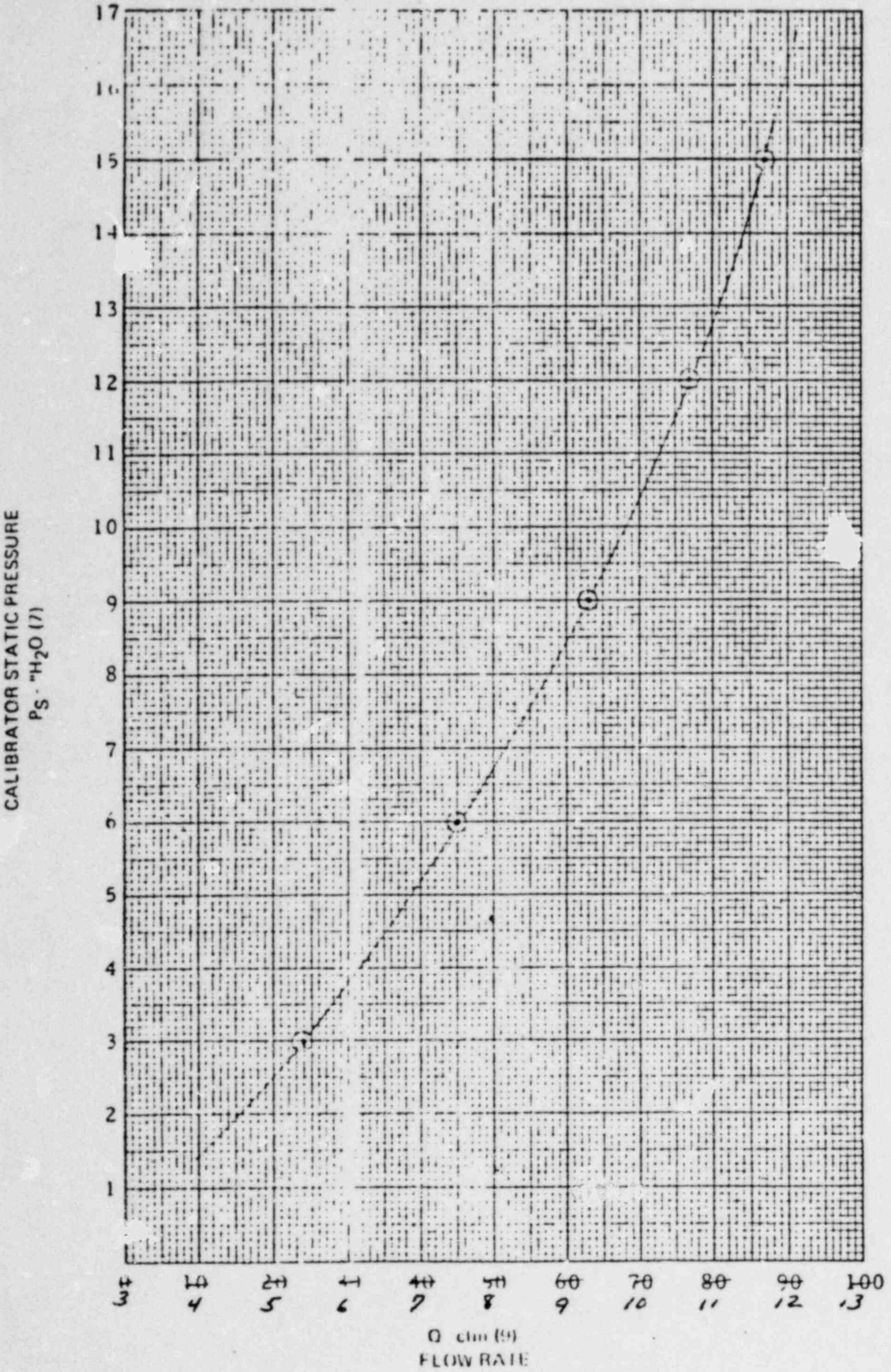
BAROMETRIC PRESSURE AT VARIOUS ALTITUDES

(continued)

Table 2.2

Barometric pressure, inches	0	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
	Feet	Feet	Feet	Feet	Feet	Feet	Feet	Feet	Feet	Feet
23.50	6,546	6,535	6,523	6,512	6,500	6,489	6,477	6,466	6,454	6,443
.60	6,431	6,420	6,408	6,397	6,385	6,374	6,362	6,351	6,339	6,328
.70	6,316	6,305	6,293	6,282	6,270	6,259	6,247	6,236	6,225	6,213
.80	6,202	6,190	6,179	6,167	6,156	6,145	6,133	6,122	6,110	6,099
.90	6,088	6,076	6,065	6,054	6,042	6,031	6,020	6,008	5,997	5,986
24.00	5,974	5,963	5,952	5,940	5,929	5,918	5,906	5,895	5,884	5,872
.10	5,861	5,850	5,839	5,827	5,816	5,805	5,794	5,782	5,771	5,760
.20	5,749	5,737	5,726	5,715	5,704	5,693	5,681	5,670	5,659	5,648
.30	5,637	5,625	5,614	5,603	5,593	5,581	5,570	5,558	5,547	5,536
.40	5,525	5,514	5,503	5,492	5,480	5,469	5,458	5,447	5,436	5,425
24.50	5,414	5,403	5,392	5,381	5,369	5,358	5,347	5,336	5,325	5,314
.60	5,303	5,292	5,251	5,270	5,259	5,248	5,237	5,226	5,215	5,204
.70	5,193	5,182	5,171	5,160	5,149	5,138	5,127	5,116	5,105	5,094
.80	5,083	5,072	5,061	5,050	5,039	5,028	5,017	5,006	4,995	4,985
.90	4,974	4,963	4,952	4,941	4,930	4,919	4,908	4,897	4,886	4,876
25.00	4,865	4,854	4,843	4,832	4,821	4,810	4,800	4,789	4,778	4,767
.10	4,756	4,745	4,735	4,724	4,713	4,702	4,691	4,681	4,070	4,659
.20	4,648	4,637	4,627	4,616	4,605	4,594	4,584	4,573	4,562	4,551
.30	4,540	4,530	4,519	4,508	4,498	4,487	4,476	4,465	4,455	4,444
.40	4,433	4,423	4,412	4,401	4,391	4,380	4,369	4,358	4,348	4,337
25.50	4,326	4,316	4,305	4,295	4,284	4,273	4,263	4,252	4,241	4,231
.60	4,220	4,209	4,199	4,188	4,178	4,167	4,156	4,146	4,135	4,125
.70	4,114	4,104	4,093	4,082	4,072	4,061	4,051	4,040	4,030	4,019
.80	4,009	3,998	3,988	3,977	3,966	3,956	3,945	3,935	3,924	3,914
.90	3,903	3,893	3,882	3,872	3,861	3,851	3,841	3,830	3,820	3,809
26.00	3,799	3,788	3,778	3,767	3,757	3,746	3,736	3,726	3,715	3,705
.10	3,694	3,684	3,674	3,663	3,653	3,642	3,632	3,622	3,611	3,601
.20	3,590	3,580	3,570	3,559	3,549	3,539	3,528	3,518	3,508	3,497
.30	3,487	3,477	3,466	3,456	3,446	3,435	3,425	3,415	3,404	3,394
.40	3,384	3,373	3,363	3,353	3,343	3,332	3,322	3,312	3,301	3,291
26.50	3,281	3,270	3,260	3,250	3,240	3,230	3,219	[illegible]	3,199	3,189
.60	3,179	3,168	3,158	3,148	3,138	3,128	3,117	3,107	3,097	3,087
.70	3,077	3,066	3,056	3,046	3,036	3,026	3,016	3,005	2,995	2,985
.80	2,975	2,965	2,955	2,945	2,934	2,924	2,914	2,904	2,894	2,884
.90	2,874	2,864	2,854	2,843	2,833	2,823	2,813	2,803	2,793	2,783
27.00	2,773	2,763	2,753	2,743	2,733	2,723	2,713	2,703	2,692	2,682
.10	2,672	2,662	2,652	2,642	2,632	2,622	2,612	2,602	2,592	2,582
.20	2,572	2,562	2,552	2,542	2,532	2,572	2,512	2,502	2,493	2,483
.30	2,473	2,463	2,453	2,443	2,433	2,423	2,413	2,403	2,393	2,383
.40	2,373	2,363	2,353	2,343	2,334	2,324	2,314	2,304	2,294	2,284
27.50	2,274	2,264	2,254	2,245	2,235	2,225	2,215	2,205	2,195	2,185
.60	2,176	2,166	2,156	2,146	2,136	2,126	2,116	2,107	2,097	2,087
.70	2,077	2,067	2,058	2,048	2,038	2,028	2,018	2,009	1,999	1,989
.80	1,989	1,970	1,960	1,950	1,940	1,930	1,921	1,911	1,901	1,891
.90	1,872	1,872	1,862	1,852	1,843	1,833	1,823	1,814	1,804	1,794
28.00	1,784	1,775	1,765	1,755	1,746	1,736	1,726	1,717	1,707	1,697
.10	1,688	1,678	1,668	1,659	1,649	1,639	1,630	1,620	1,610	1,601
.20	1,591	1,581	1,572	1,562	1,552	1,543	1,533	1,524	1,514	1,504
.30	1,495	1,485	1,478	1,466	1,456	1,447	1,427	1,428	1,418	1,408
.40	1,396	1,389	1,380	1,370	1,361	1,351	1,342	1,332	1,322	1,313
28.50	1,303	1,294	1,284	1,275	1,265	1,256	1,246	1,237	1,227	1,218
.60	1,208	1,199	1,189	1,180	1,170	1,161	1,151	1,142	1,132	1,123
.70	1,113	1,104	1,094	1,085	1,075	1,066	1,057	1,047	1,038	1,028
.80	1,019	1,009	1,000	990	981	972	962	953	943	934
.90	925	915	906	896	887	878	868	859	849	840
29.00	831	821	812	803	794	784	775	765	756	746
.10	737	728	718	709	700	690	681	672	663	653
.20	644	635	625	616	607	597	588	579	[illegible]	560
.30	551	542	532	523	514	505	495	486	477	468
.40	458	449	440	431	421	412	403	394	384	375
29.50	366	357	348	338	329	320	311	302	292	283
.60	274	265	256	247	237	228	219	210	201	192
.70	182	173	164	155	146	137	128	118	109	100
.80	+91	+82	+73	+64	+55	+45	+36	+27	+18	+9
.90	0	-9	-18	-27	-36	-45	-55	-64	-73	-82
30.00	-91	-100	-109	-118	-127	-136	-145	-154	-163	-172
.10	-181	-190	-199	-208	-217	-226	-235	-244	-253	-262
.20	-271	-280	-289	-298	-307	-316	-325	-334	-343	-352
.30	-361	-370	-379	-388	-397	-406	-415	-424	-433	-442
.40	-451	-460	-469	-478	-487	-495	-504	-513	-522	-531
30.50	-540	-549	-558	-567	-576	-585	-593	-602	-611	-620
.60	-629	-638	-647	-656	-665	-673	-682	-691	-700	-709
.70	-718	-727	-735	-744	-753	-762	-771	-780	-788	-797
.80	-806	-815	-824	-833	-841	-850	-859	-868	-877	-885

For sampler equipped with a pressure-transducer recorder, the average flow indication I is determined from the recorder chart. If the flow indication varies more than 5% during the sampling period, read the chart at two-hour intervals and calculate the average I for the sampling period.


CALIBRATOR STATIC PRESSURE
P_S - "H_2O (7)
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
~~0~~ 3
~~10~~ 4
~~20~~ 5
~~30~~ 6
~~40~~ 7
~~50~~ 8
~~60~~ 9
~~70~~ 10
~~80~~ 11
~~90~~ 12
~~100~~ 13
Q cfm (9)
FLOW RATE

WORK SHEET

	(1) Initial Meter Reading M^3	(2) Final Meter Reading M^3	(3) Elapsed Time - Δt Min.	(4) Initial Volume V_{in} M^3	(5) Meter Static Pressure - P_S "H_2O	(5) Meter Static Pressure - P_S "Hg	(6) True Volume V_T M^3	(7) Calibrator Static Press. "H_2O	(8) Volume Q M^3/min.	(9) Flow Rate Q ft^3/min.
1	4229.40	4229.50	0.29	0.1		1.1	0.0963	15	0.332	11.73
2	4229.80	4229.90	0.32	0.1		0.9	0.0970	12	0.303	10.7
3	4230.10	4230.20	0.37	0.1		0.7	0.0977	9	0.264	9.3
4	4230.40	4230.50	0.46	0.1		0.5	0.0983	6	0.214	7.5
5	4230.70	4230.80	0.65	0.1		0.25	0.992	3	0.153	5.4

EQUATIONS

$V_{in} = (2) - (1)$

$$V_T = V_{in} \frac{P_B - P_S}{P_B}$$

$$= (4)\ \frac{(10) - (5)}{(10)}$$

$$Q = \frac{V_T}{\Delta t} = \frac{(6)}{(3)}$$

$M^3 \times 35.31 = ft^3$

"$H_2O \div 13.55 =$ "Hg

When using this instrument to calibrate a high volume sampler at conditions other than those at which the calibration took place (10) (11), the following relation should be applied.

$$Q_T = Q \left[\frac{T_T}{T} \frac{P_B}{P_T} \right]^{1/2}$$

where:

Q_T = flow at new conditions T_T and P_T.

Roots Meter No.: 7509364

Calibrator:

Model No.: GMW-SPEC-1/2" C.O.F.

Serial No.: CO2

(10) P_B: 30.05 "Hg

(11) T: 72 °F

(12) RH: 29 %

Calibration performed by:

Date of Calibration: 10 MAR 78

Date placed in service:
(To be noted by user)

For additional information consult:

1. The Federal Register, Vol. 36, No. 84, pp. 8191 - 8194, April 30, 1971.
2. Guidelines for Development of a Quality Assurance Program – High Volume Method. EPA - R4 - 73 - 028C.

Notes: Calibrators should be recalibrated after one year of field use. See ref. 2, page 25. Copies of this calibration are not kept on file.

APPENDIX 3.13.0

OPERATING INSTRUCTIONS
GEOFILTER WELL PUMP
AND COMPRESSOR

GEOFILTER®

Filtration Equipment and Supplies

SMALL WELL PUMP
MODEL # 0500

MANUFACTURED BY

LEONARD MOLD AND DIE WORKS INC.

960 W. 48th AVE.
Phone 303-433-9278
DENVER, CO. 80221

PREFACE

The small diameter ground water sampler, explained in the following pages, was designed and engineered in conjunction with the United States Geological Survey, Quality of Water Branch.

The theory behind the unit is based on Pascal's Theory, stating that water in a balloon exerts equal pressure in all directions. Building on this theory, a gallon of water weighs 8.3453 pounds and contains 231 cubic inches, by dividing, the weight per cubic inch equals .036125. This figure times 12 gives us the amount of pressure needed to equalize a foot of water which is .4335. Additional pressure will be required to assure adequate flow, so as a rule of thumb, we will assume, for purposes of instruction, that one-half pound of pressure per foot of well depth will be required for operation of the well sampler.

The unit itself is comprised of two main components, the Pneumatic Logic Unit, and the pressure chamber. Although all areas of the well sampler are covered in this booklet, special emphasis will be placed in these areas.

INDEX

COMPRESSED POWER SUPPLY

The small diameter well sampler is designed to be operated by any inert air supply, the most common of which is in the form of compressed air, air compressor, and compressed nitrogen. Care should be exercised in selecting tanks or an air compressor which comply with compressed gas standards.

REGULATOR

A tank regulator of the design that is single valve, two gauge, capable of reading rated tank pressure and 250 PSI outlet line pressure is required. If the outlet line pressure exceeds 250 PSI, a Pressure Relief Valve set at 250 PSI should be placed in line to insure that the system is not overcharged.

WHIP AND CONNECTORS

A hose whip capable of withstanding 250 PSI should be used where the Pneumatic Logic Unit is not attached directly to the tank regulator or air compressor. Connectors on this whip should comply with compressed gas standards.

PRESSURE LINE

The pressure line provided with the unit is of rubber material and twin line construction rated to 250 PSI. This twin line hose is reinforced to provide tensile strength for placing and removing the unit in the well. Fittings on the twin line hose that come in contact with the water are of stainless steel construction.

SAMPLE LINE

The main prerequisite for the sample line is that it be of food grade material. There are several different brands and materials available. As with the pressure line, any fittings coming in contact with the water must be of stainless steel construction.

FILTERS

Two filters are provided with the Pneumatic Logic Unit, a 100µm and a 5 um. These filters are designed to remove particles and moisture from the power supply. A manual drain is located at the bottom of the bowl, and must be pressed regularly to remove the moisture from the filter bowl. If this is not done, damage will result to the filter element as well as the Pneumatic Logic Unit.

SERVICING:

Disassembly:

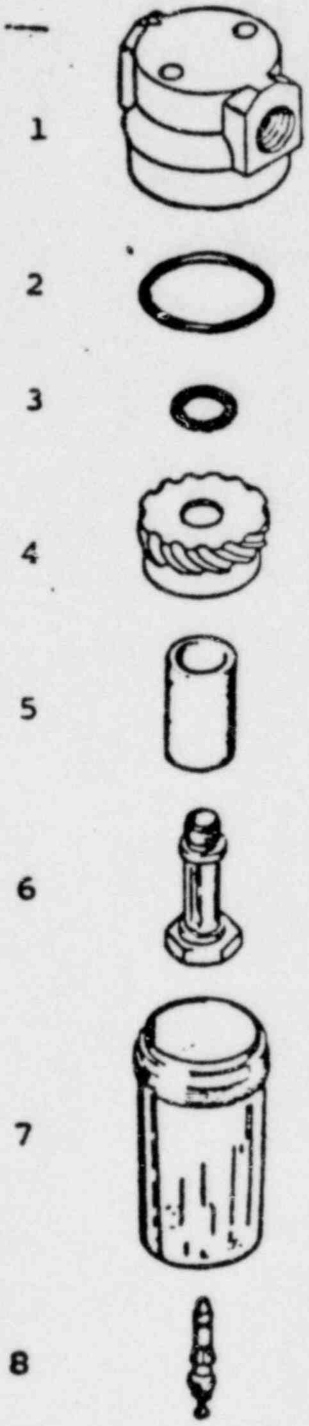
1. Shut off inlet pressure and reduce pressure in bowl to zero. Filter may be disassembled without removal from air line.
2. Unscrew bowl (7) and remove O-ring (2).
3. Unscrew stud (6), then remove gasket (3), louver (4) and element (5).
4. Remove manual drain (8).

Cleaning:

1. Clean parts using warm water and soap. Dry parts and blow out internal passages in body (1) using clean, dry air. Blow air through filter element (5) from inside to outside to dislodge surface contaminants. Replace filter element when plugged.
2. Inspect all parts carefully.
3. Replace damaged parts.

Reassembly:

1. Prior to reassembly, lubricate the O-ring (2) and threads on the bowl (7) with a thin grease.
2. Install element (5) on stud (6) with rounded end facing stud head.
3. Tighten the stud (6) and bowl (7) to between 5 and 10 inch-pounds of torque.



PNEUMATIC LOGIC UNIT

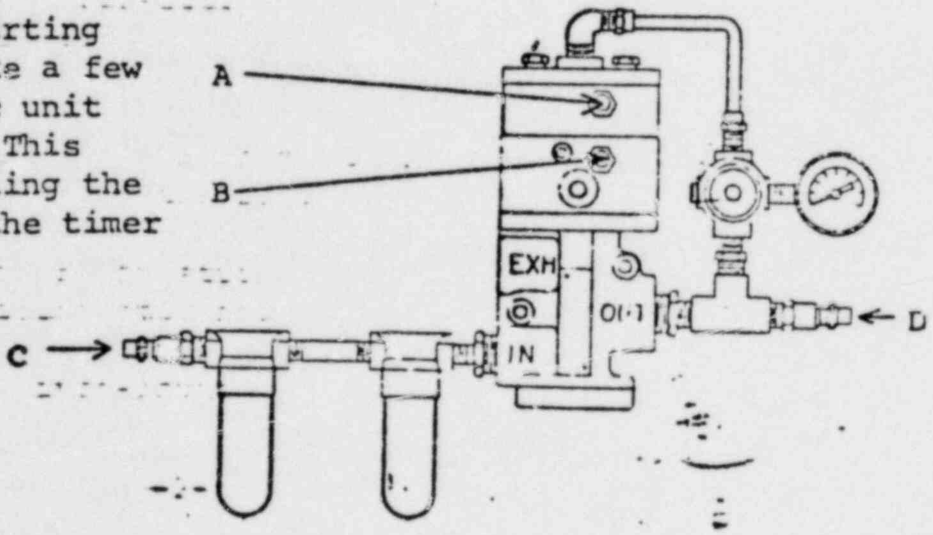
The Pneumatic Logic Unit is comprised of several components: Timer Modules, Timer Regulator, and Valve Body. These components provide the control center that charges and bleeds the pressure chamber of the well sampler. Needle valve (A) controls the length of time air enters the chamber. Needle valve (B) controls the length of time the chamber is exhausted. On both valves, clockwise turns increase delay time; counterclockwise decreases delay time. Port (C) is the intake port from the power supply. Port (D) is the exhaust port to the well sampler.

Due to the varying depth of wells, there is no hard fast rule for setting the unit. An average for the charge cycle is 3 - 5 seconds and for the bleed cycle 10 - 12 seconds. The bleed cycle is distinguished by a "pop" sound when the unit switches to the bleed mode.

The Valve Body is rated to 250 PSI, the Timer Modules are rated to 150 PSI, thus the requirement for the Timer Regulator. Although the Timer Regulator is installed simply to protect the Timer Modules, once the needle valves are adjusted, the Timer Regulator will proportionately increase or decrease the setting by adjusting the regulated pressure if desired. The red ring below the adjust knob is for locking the adjustment knob. When pushed toward the valve body the regulator is locked. When pulled toward the adjustment knob, the regulator is unlocked.

A servicing guide for these components is not included due to their complexity. If problems exist, call our service department for advice.

NOTE: When first starting the unit, it will take a few minutes for the logic unit to cycle regularly. This is caused by air filling the various chambers of the timer module.

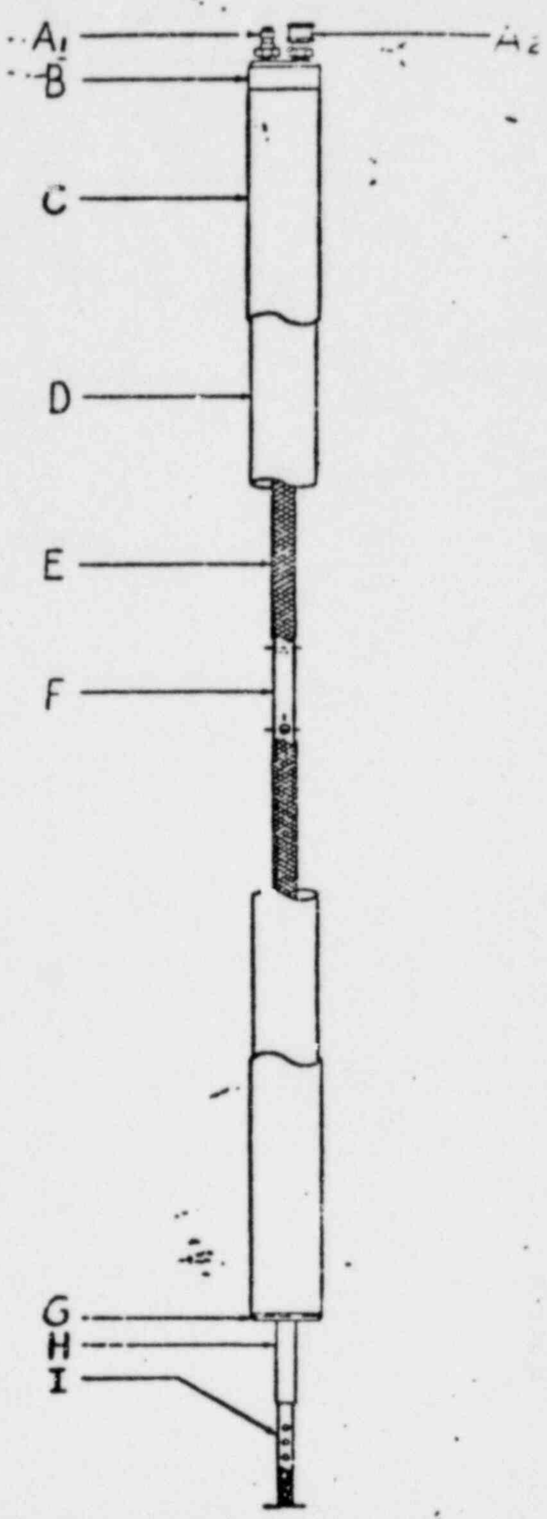


WELL SAMPLER

The well sampler is constructed from stainless steel, unless otherwise specified, which gives it corrosion resistance and durability.

OPERATION:

Water enters through the strainer (I) and passes the checkvalve (H) filling the neoprene bladder (D) by way of the center shaft (E&F). When the bladder is full, air is compressed into the chamber (C) through the top air port (A^2). This pressure closes the checkvalve (H) and compresses the bladder (D) forcing the water back through the center shaft (E&F) and past the checkvalve (B) to the sample port (A^1) and up the sample tubing. When the pressure is bled from the chamber (C) through the air port (A^2), the head pressure closes the checkvalve (B) and the cycle is repeated.



DISASSEMBLY:

To disassemble the unit, remove the checkvalve and strainer (H&I) and the washers and O-ring they secure. Replace the checkvalve and strainer (H&I) leaving one-half inch between the top of the checkvalve (H) and the bottom cylinder cap (G). While holding the top of the unit, tap the bottom of the unit on the ground. This will force the top cylinder cap (B) past its O-ring seal. The top cylinder cap (B), bladder (D) and center shaft (E&F) will come out as one piece when the checkvalve (H) is finally removed. The bladder (D) may be removed from the center shaft (E&F) by loosening the hose clamps located at the top and bottom of the bladder and sliding the bladder off the bottom.

REASSEMBLY:

To reassemble the unit, reverse the process. Do not, however, replace the bottom O-ring until the top cylinder cap (B) has been drawn completely down by the checkvalve (H). This will prevent damage to the O-ring. All O-rings should be given a thin coat of grease prior to reassembly. Replace any worn or damaged parts.

Warning

Read instructions
before installing
Mark II
Load Genie

Warning

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before installing
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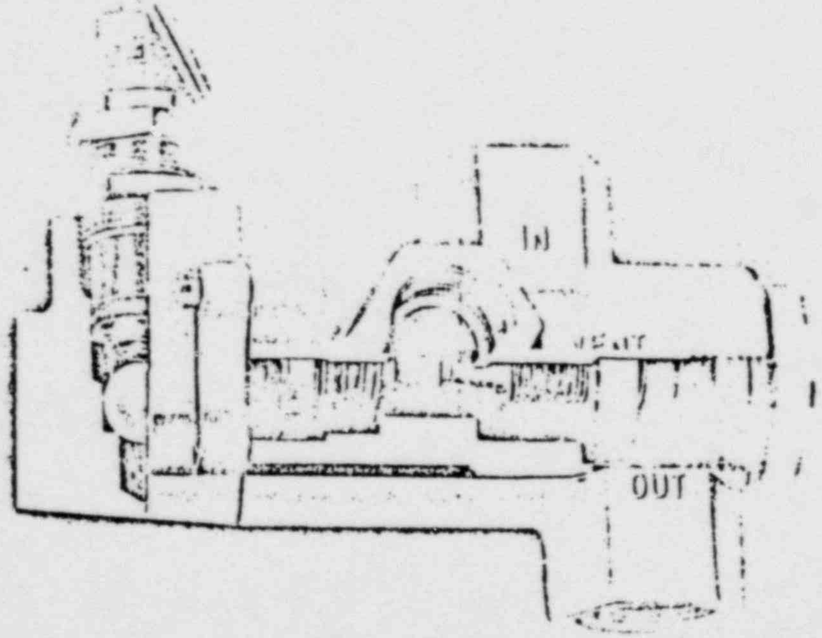


Mark II
Load Genie®
operation and installation data

control
devices inc.

Mark II Load Genie®
U.S. Patent No. 3,587,619.

711 Hanley Ind[illegible] Court
P.O. Box 20021

Oper[illegible]n and Installation Data

The Mark II LOAD GENIE is a self contained, automatic, continuous run unloader and check valve.

HOW IT WORKS

When compressed air in the tank has reached the factory preset pressure, the control will unload the compressor through the vent port. Once air has been used from the Tank, and pressure drops, the Mark II control will permit the compressed air to be pumped into the tank again.

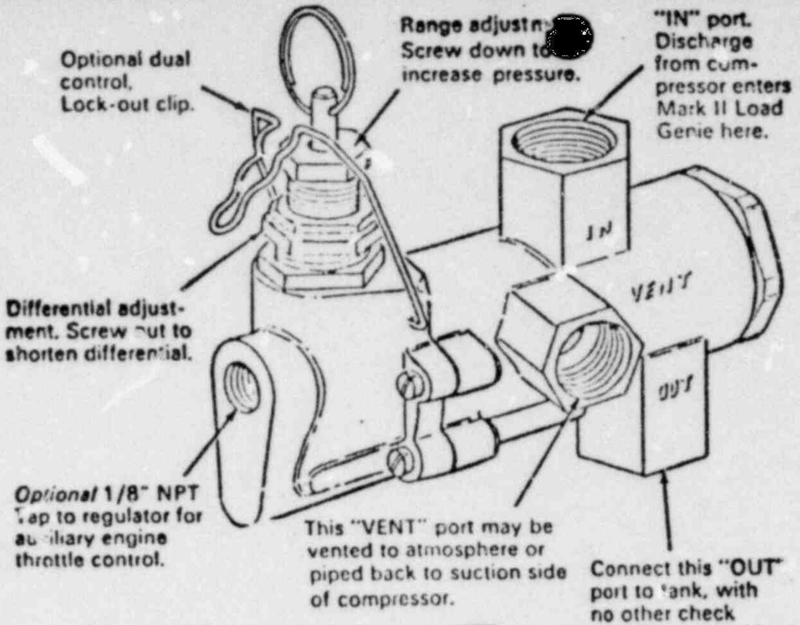
REGULATOR ADJUSTMENTS

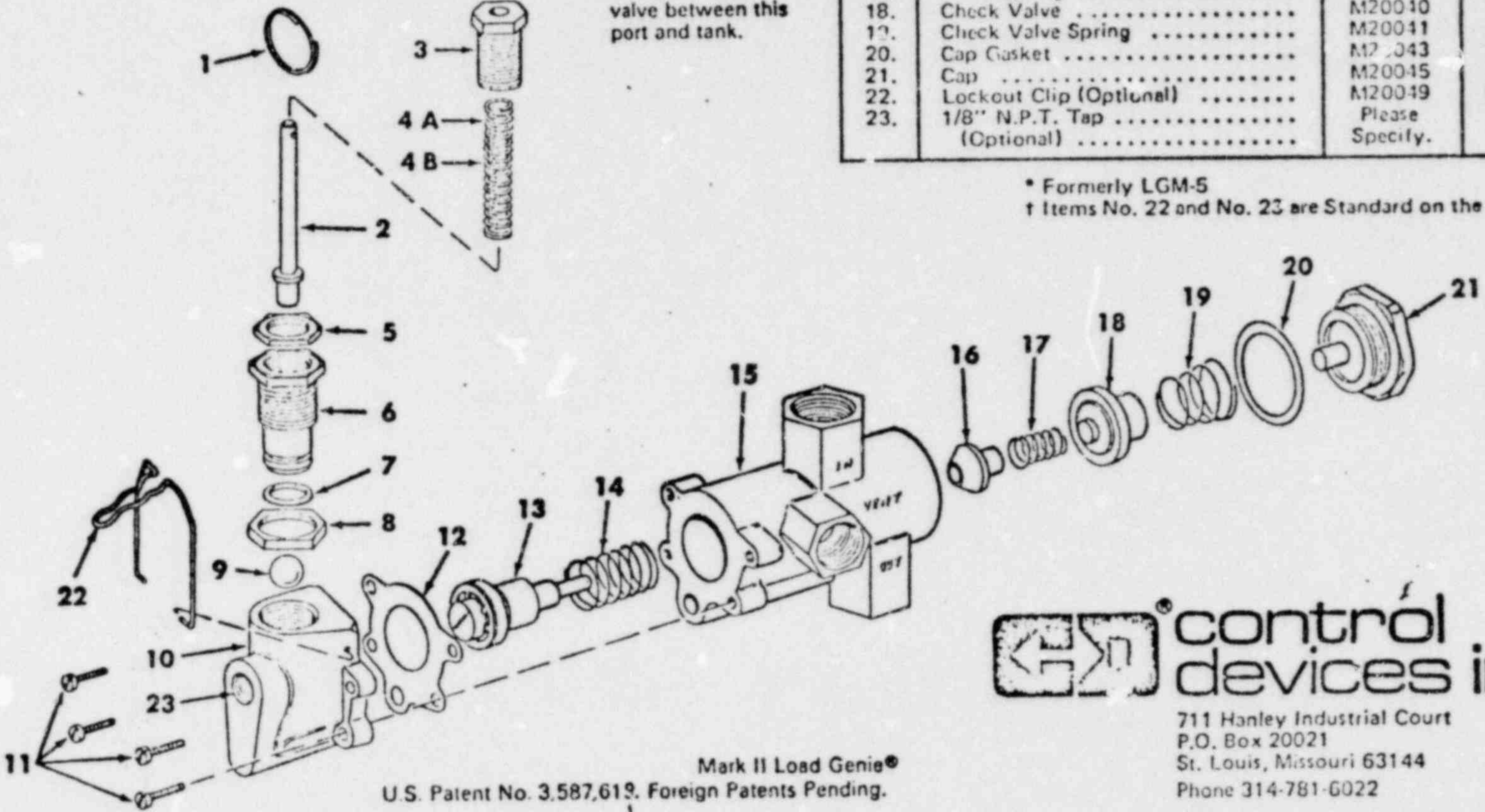
1. Loosen jam nuts #5 & #8.
2. The unloading pressure can be adjusted by screw #3. Turn clockwise to increase and counterclockwise to decrease pressure.
3. The differential (difference between cut-in and cut-out pressure) is obtained by adjusting screw #6. Turning clockwise will widen the differential and counterclockwise will narrow the differential.
4. Repeat items 1 & 2 until desired settings are obtained. It is not advisable to have a differential of less than 10% of the maximum cut-out pressure.
5. After adjustments are made, tighten jam nuts #5 & #8.

Minimum recommended compressor speed 400 R.P.M.

Maximum recommended working pressure 250 P.S.I.

Maximum recommended operating temperature 450 Degrees F.





Mark II Load Genie®
U.S. Patent No. 3,587,619. Foreign Patents Pending.

LGM SERIES PARTS LIST

Please order by Part Number — Not by Key No.

Key No.	Part Name	LGM-20* Part No.	LGM-125 Part No.
1.	Pull Ring	M20001	M20001
2.	Pressure Rod	M20003	M20003
3.	Spring Housing	M2000[illegible]	M20005
4.	(a) Standard Regulator Spring (RED); 50 psi to 150 psi.	M20007	M20007
	(b) Optional High Pressure Regulator Spring (BLUE); 125 psi to 250 psi.	M20008	M20008
5.	Upper Jam Nut	M20011	M20011
6.	Ball Seat	M20013	M20013
7.	"O" Ring	M20015	M20015
8.	Lower Jam Nut	M20017	M20017
9.	Ball	M20019	M20019
10.	Regulator Body	M20021	M12521
11.	Screw (4 required)	[illegible]20023	M12523
12.	Regulator Gasket	M20025	M12525
13.	Piston Assembly (one part)	M20028-1	M12528
14.	Piston Spring	M20033	M12533
15.	Valve Body	M20035	M12535
16.	Valve Stem	M20039	M12537
17.	Valve Spring	35050[illegible]	
18.	Check Valve	M20040	M12539
19.	Check Valve Spring	M20041	M12541
20.	Cap Gasket	M2[illegible]043	M12543
21.	Cap	M20045	M12545
22.	Lockout Clip (Optional)	M20049	M20049†
23.	1/8" N.P.T. Tap (Optional)	Please Specify.	†

* Formerly LGM-5
† Items No. 22 and No. 23 are Standard on the LGM-125.

control devices inc.
711 Hanley Industrial Court
P.O. Box 20021
St. Louis, Missouri 63144
Phone 314-781-6022

MINIATURE FILTER

FOR COMPRESSED AIR SERVICE

OPERATION

A filter is used in a compressed air system to remove most solid particles and liquid from the compressed air. Air entering the filter is guided into a swirling pattern by the louvers (1). Coarse solid particles and liquids are forced to the bowl wall (2) by centrifugal force and drop to the bottom. Air leaving the bowl passes through the filter element (3) where finer solid particles are retained.

These filters have either a manual or automatic drain. Manual drain models must be drained periodically. Automatic drain models expel liquid automatically as flow through the filter is cycled.

SPECIFICATIONS

PORT SIZE: 1/8" or 1/4" PTF
MAXIMUM RATED OPERATING CONDITIONS –
Polycarbonate Bowl: 150 psig (10.3 bar); 125°F (52°C)
✓Metal Bowl: 250 psig (17.2 bar); 175°F (79°C)
ELEMENT: 5 micron or 100 micron
BOWL: 1/2-oz. transparent polycarbonate or aluminum
DRAIN: Manual or automatic

MATERIALS OF CONSTRUCTION

Body	Zinc
Bowl –	
Transparent	Polycarbonate
Metal	Aluminum
Element	High density polyethylene
Elastomers	Neoprene & Buna-N

WARNING

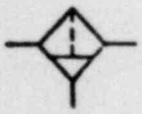
THESE UNITS ARE INTENDED FOR USE IN INDUSTRIAL COMPRESSED AIR SYSTEMS ONLY. THEY MUST NOT BE USED WHERE PRESSURE OR TEMPERATURE MAY EXCEED MAXIMUM RATED OPERATING CONDITIONS. SEE SPECIFICATIONS.

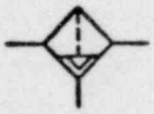
THE POLYCARBONATE PLASTIC BOWLS USED ON THESE UNITS CAN BE DAMAGED AND POSSIBLY BURST IF EXPOSED TO SUCH SUBSTANCES AS CERTAIN SOLVENTS, STRONG ALKALIES, COMPRESSOR OILS CONTAINING ESTER-BASED ADDITIVES, OR SYNTHETIC OILS. FUMES OF THESE SUBSTANCES IN CONTACT WITH THE POLYCARBONATE BOWL, EXTERNALLY OR INTERNALLY, CAN ALSO RESULT IN DAMAGE. CLEAN WITH WARM WATER ONLY.

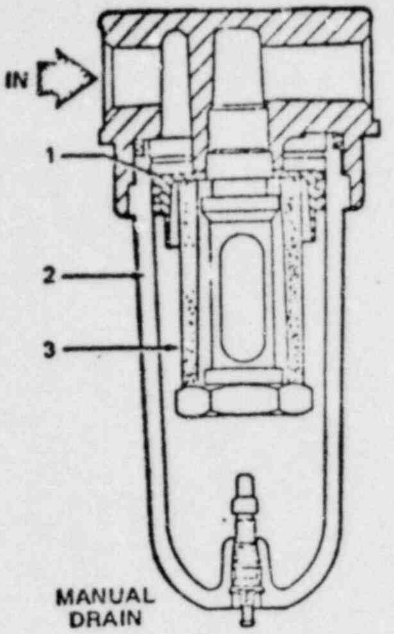
USE METAL BOWL IN APPLICATIONS WHERE A PLASTIC BOWL MIGHT BE EXPOSED TO SUBSTANCES THAT ARE INCOMPATIBLE WITH POLYCARBONATE.

THESE PRODUCTS ARE NOT DESIGNED FOR USE WITH FLUIDS OTHER THAN AIR, FOR NONINDUSTRIAL APPLICATIONS, OR FOR LIFE SUPPORT SYSTEMS.

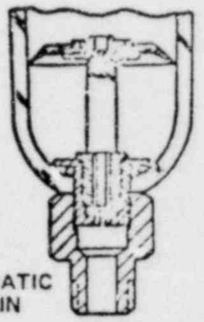
GRAPHIC SYMBOLS


Manual Drain Filter


Automatic Drain Filter



MANUAL DRAIN

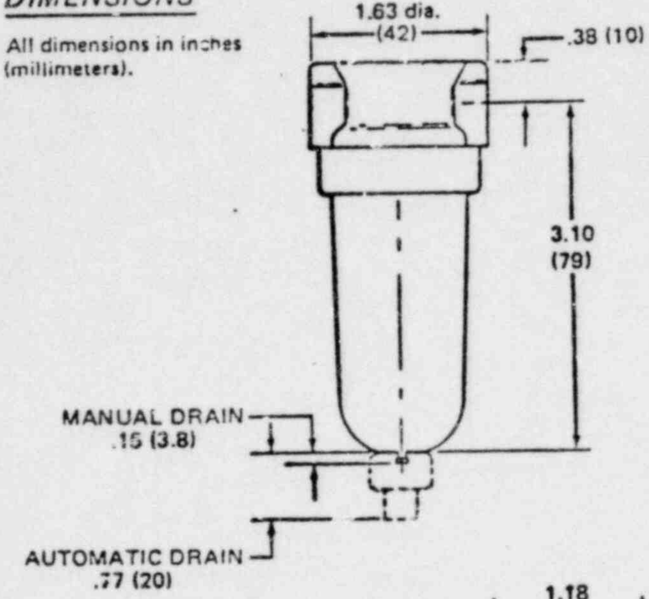

AUTOMATIC DRAIN

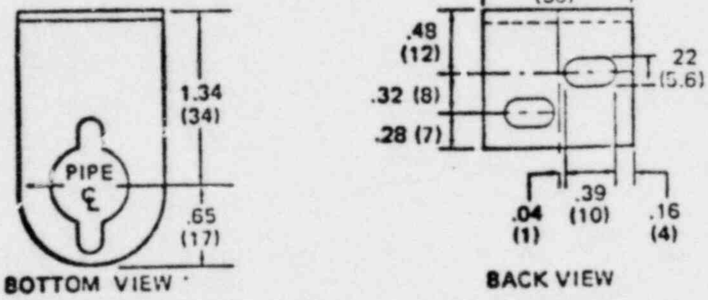
INSTALLATION

1. Air line piping should be same size as filter ports.
2. Install filter in air line upstream of regulators and lubricators as near as possible to the device being served.
3. Air flow must be in direction of arrow on top of body. Connect piping to proper ports using pipe thread sealant on male threads only. Do not allow sealant to enter interior of filter.
4. Automatic drains are ported 1/8" NPTF to allow piping away of expelled liquid. Use flexible tubing or nonrigid piping (1/8" minimum I.D.) for the drain line.

DIMENSIONS

All dimensions in inches (millimeters).





BOTTOM VIEW
BACK VIEW

WALL MOUNTING BRACKET

RVICING

Filter with manual drains must be drained as frequently as necessary keep liquid level in bowl below the bottom of the element mounting d (6). If liquid level rises above the stud, liquid will be carried down-am. Replace filter element when plugged or dirty.

SEMBLY

Shut [illegible] inlet pressure and reduce pressure in bowl to zero. Filter may be disassembled without removal from air line.

Unscrew bowl (12) and remove O-ring (10).

Unscrew stud (6), then remove gasket (14), louver (2) and element (4).

Remove manual drain (11) or automatic drain (7, 8, 9, 13). To remove automatic drain, invert bowl and shake out valve (7), then unscrew nut (13) and remove insert (8) and O-ring (9).

LEANING

Clean plastic bowl using warm water only. Clean other parts using warm water and soap. Dry parts and blow out internal passages in body (1) using clean, dry compressed air. Blow air thru filter element (4) from inside to outside to dislodge surface contaminants. Replace filter element when plugged.

Inspect all parts carefully.

Replace damaged parts. If transparent bowl shows any signs of cloudiness or cracking, replace with a metal bowl.

REASSEMBLY

. Prior to reassembly, lubricate the following items:

ITEM	LUBRICANT
10 (O-ring)	Wipe coat of Dow Corning 44M grease (or equivalent)
12 (threads on metal bowls)	Small, even amount of Dow Corning Molykote Type G lubricant (or equivalent)

2. During reassembly, make sure the valve lip (7) is turned down. Use a soft, blunt tool such as a piece of cardboard to turn lip down. DO NOT SCRATCH THE BOWL SURFACE WHEN TURNING LIP DOWN.

[illegible] element (4) on stud (6) with rounded end facing stud head.

. ighten the following items to the specified torque:

ITEM	TORQUE (INCH-POUNDS)
6 (element stud)	5 to 10
12 (bowl)	5 to 10
13 (auto drain nut)	5 to 8

REPAIR KITS

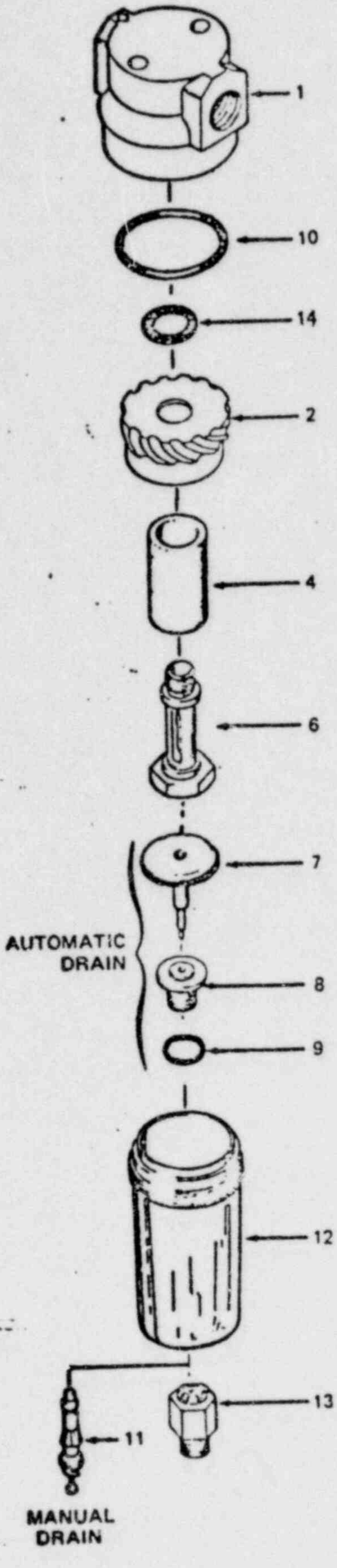
Filter Element (items 4, 10, 14)	
5-micron	3652-11
100-micron	3652-09
Automatic Drain (items 7, 8, 9, 10)	3654-01

REPAIR PARTS

Bowl (item 12)	
Transparent with automatic drain	3635-52
Transparent with manual drain	3635-51
Metal with automatic drain	3809-50
Metal with manual drain	3809-52
Manual Drain Valve (item 11)	773-01

ACCESSORIES

Wall mounting bracket	5939-02

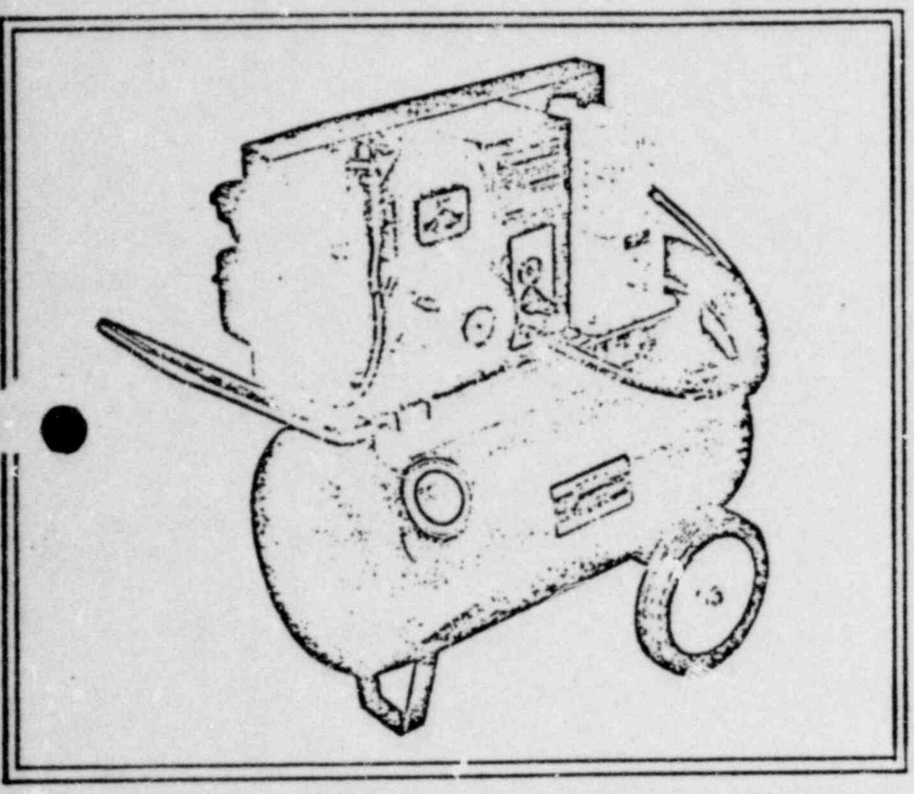


AIR COMPRESSOR

MODEL 112GU-22

CAREFULLY READ INSTRUCTION MANUAL BEFORE ATTEMPTING TO OPERATE THIS UNIT.

All air compressors are precision built of the finest materials available and will perform efficiently for many years with proper care and maintenance. A few of the many uses are; paint spraying, air cleaning, operating air tools, inflating tires, operating air doors and cylinders, greasing equipment, air sanders and drills, staple guns, air nailers, vacuum cleaning, riveting machines, air controls and many more . . .



FEATURES

MOTOR – 5 H.P. Briggs & Stratton w/recoil starter – includes air operated throttle.

COMPRESSOR – Twin cylinder.

TANK – 22 gallon for adequate storage.

GUARD – Totally enclosed to protect operator and machine. It also inhances the appearance of your new piece of equipment.

BUILT-IN DIAL–O–MATIC CONTROL – Eliminates the need for an expensive regulator. Let's you dial the amount of air needed for the job. Refer to Dial–O–Matic instruction decal on your unit.

OPERATING INSTRUCTIONS

Your compressor is equipped with a briggs and stratton engine, to supply adequate power. Consult engine owners manual for instructions, which is included.

Be sure your compressor is full of a good grade 10–30 motor oil. Use a lighter oil in winter if your unit must be started in extreme cold weather.

Be sure all connections are tight. A small air leak in your hose or connections will make a big difference in the performance of your compressor.

Drain moisture from tank daily with the handy combination safety valve and tank drain which is No. 503D on your compressor console. Simply pull the ring which is attached to the plunger.

After a few days of operation; remove guard, adjust belt, tighten pulleys and all fittings BE SURE TO RE-INSTALL GUARD!!!

Always be careful when operating or transporting your machine.

HELPFUL HINTS

1. Never do any work on the air compressor without first, (1) shutting off the power, and (2) bleeding all air from the receiver.
2. Do not re-adjust pressure switch on safety valve.
3. Do not use a long extension cord of undersize wire, see wire diagram on reverse side.
THIS IS VERY IMPORTANT!
4. Fasten your compressor down when transporting in a car, pickup, or truck. They can be easily tipped, and parts are expensive.
5. If compressor fails to start check your pressure, the tank may be full of air.
6. Keep your belt and pulleys tight.
7. Use only factory authorized parts.

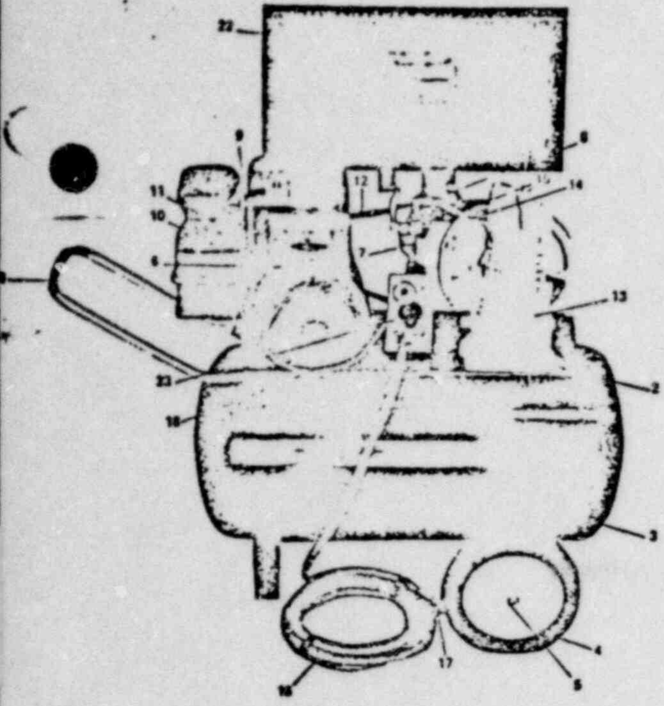

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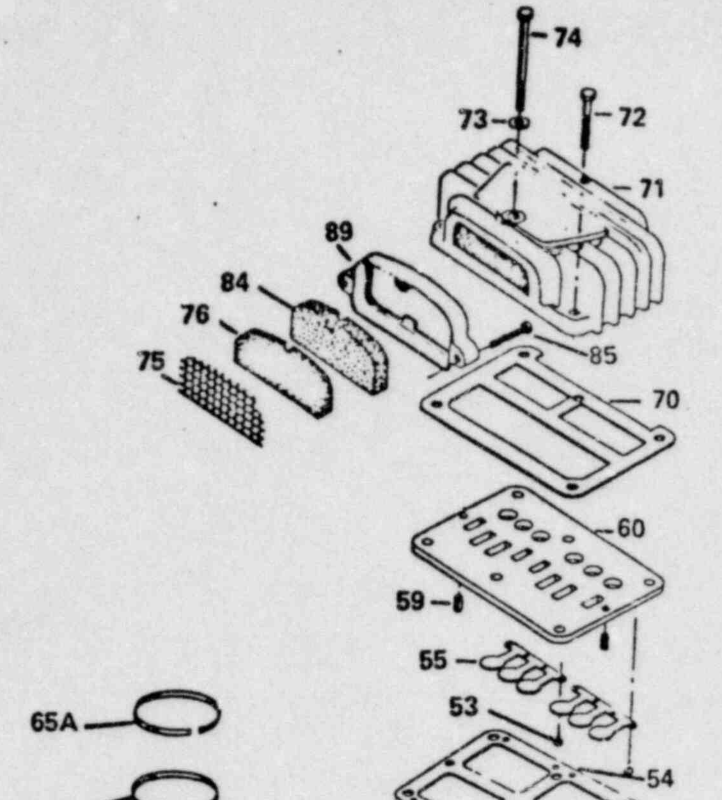
Fig. 1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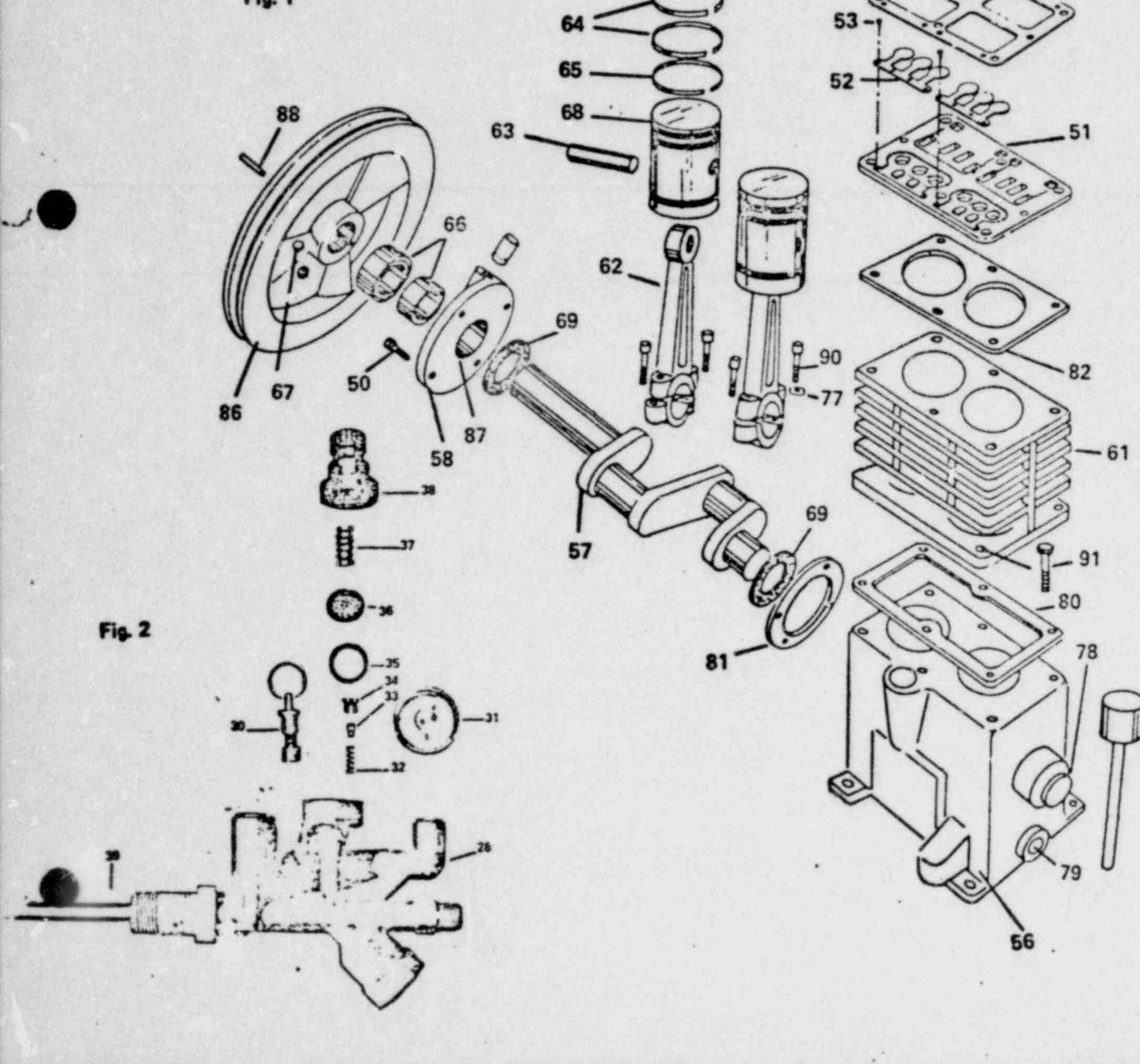
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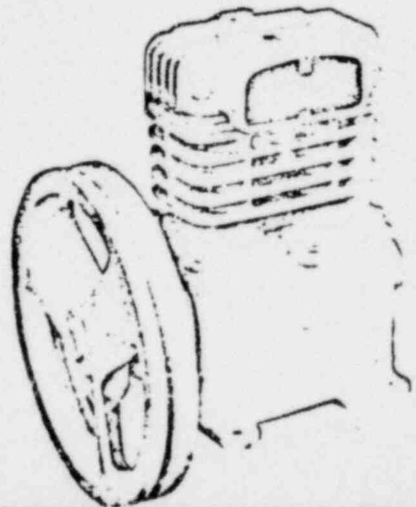
Fig. 2

PARTS LIST

REF. NO.	PART NO.	DESCRIPTION	NO. REQ.
	349D	Handle 18 ga. steel plated	1
[illegible]	516	5/16-18 hex cap screw	16
3	22P3100WC	Air receiver 22 gal. w/base, stand and axel	1
4	10175	10 x 175 wheel	2
5	8176	1/2 inch hub cap	2
6	815	Gas engine	1
7	900	Unloader valve for continuous running	1
8	903	3/8 x 3/8 x 90° compression fitting	1
9	905	Air throttle	1
10 •	2G50	Motor pulley	1
11 •	308	Hollow-head set screw for motor pulley	1
12	4L490	V-belt	1
13	1120	Compressor pump	1
14	68D	3/8 x 1/2 compression fitting w/sleeve and nut	1
15	338G	3/8 inch aluminum exhaust tube	1
16	201	Air hose male fitting both ends 15 to 20 ft.	1
17	399	1/4 inch female air chuck	1
18	339G	1/4 inch aluminum tubing w/fittings	1
[illegible]	345D	Belt guard with attachments	1
	500D	Dial O Matic manifold (See Fig. 2)	1
28	501D	Manifold block	1
29	502D	1/2 x 2-1/2 inch nipple	1
30	503D	Safety valve and tank drain	1
31	504D	Air guage 200 lb.	1
32	505D	Spring	1
33	506D	Seat	1
34	507D	Guide	1
35	508D	O ring	1
36	509D	Plunger	1
37	510D	Spring	1
38	511D	Adjustment knob	1
39	512D	17 inch tube for tank drain	1
50	1134	Bearing Plate Bolt	4
51	2037	Bottom Valve Plate Only (Discharge)	1
52	2036	Reed Valve Only (Discharge)	2
53	2057	Screws For Reed Valves	8
54	2058	Gasket Middle (For Valve Plate)	1
55	2035	Reed Valve Only (Intake)	2
56	1386	Crankcase	1
57	1200	Crank Shaft	1
58	1201	Bearing Plate	1
[illegible]	2023	Spirol Pin For Valve Plate	2
[illegible]	2149	Top Valve Plate Only (Intake)	1
[illegible]	1394	Cylinder	1
62	1208	Connecting Rod	2
63	1319	Piston Pin	2
64	1533	Compression Ring (2 Per Piston)	4
65A	1534	Compression Ring (1 Per Piston)	2
65	1321	Oil Control Ring (2 Per Piston)	4
66	1215	Journal Bearing	2
67	1217	Pulley Clamp Bolt And Nut	1
68	1460	Piston	2
69	1219	Fiber Washer	2
70	2138	Gasket Top (Between 2100 & 2149)	1
71	2100	Head Only	1
72	1174	Head Bolts (Short)	4
73	1244	Gasket Washer For Long Head Bolt	2
74	1528	Head Bolts (Long)	2
75	2130	Filter Screen	1
76	2129	Filter Inner Element	1
77	1227	Connecting Rod Washer	4
78	1334	Bearing Plug	1
79	1229	Oil Drain Plug	1
80	1404	Cylinder Gasket	1
81	1231	Bearing Plate Gasket	1
82	1316	Head Gasket	1
83	1237	Breather Assembly And Oil Level Gauge	1
84	2128	Filter Outer Element	1
85	2131	Screws For Filter	1
86	1240	10 Inch Pulley 1 Groove (1–1½" Belt)	1
87	1242	Bearing Seal	1
88	1243	Pulley Key	1
89	2104	Filter Retainer	
90	1250	Connecting Rod Bolt	4
91	1246	Cylinder Bolt	6
		Assemblies	
92	1548	Piston, Pin And Rings	2
93	1194	Connecting Rod	2
94	2150	Cylinder Head With Filter And Gasket	1
95	2215	Complete Set Gasket	1
96	1363	Complete Set Rings	1

When ordering repairs give exact quantity wanted, model number of unit, number of the part and the name of the part.

• Not Illustrated



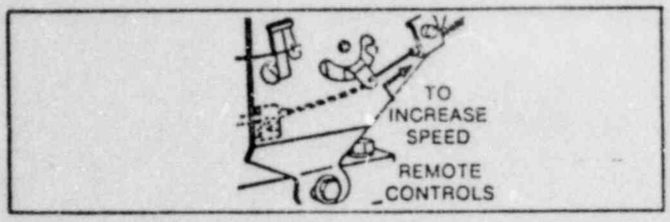
ment on which the engine is used, specifies the top governed no load speed at which the engine may be operated. DO NOT EXCEED this speed.

STANDARD SPEED CONTROL ADJUSTMENT

Speed adjusting thumb nut is located on top of engine. To increase speed turn thumb nut clockwise.

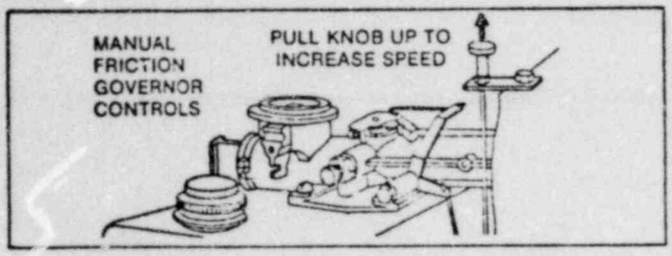
REMOTE SPEED CONTROL

Controls on powered equipment should move speed lever in direction illustrated to increase speed. Remote controls may be connected to engine at points indicated. Wire travel is shown by arrows.



MANUAL SPEED CONTROL

Move knob as shown to change engine speed.



GENERAL INFORMATION

This engine is a single-cylinder, L-head, air-cooled type.

MODEL SERIES 100200 to 100299

Bore .... 2-1/2" (63.5 mm)
Stroke .... 2-1/8" (53.98 mm)
Displacement .... 10.43 cu. in. (170.9 cc)
Horsepower .... 4.0 max. @ 3600 RPM
Torque (Ft. Lbs.) .... 5.93 max. @ 3050 RPM

MODEL SERIES 130200 to 131299

Bore .... 2-9/16" (65.09 mm)
Stroke .... 2-7/16" (61.91 mm)
Displacement .... 12.57 cu. in. (206.0 cc)
Horsepower .... 5.0 max. @ 3600 RPM
Torque (Ft. Lbs.) .... 7.66 max. @ 3000 RPM

The horsepower rating listed is established in accordance with the Society of Automotive Engineers Test Code - J607. For practical operation, the horsepower loading should not exceed 85% of this rating. Engine power will decrease 3½% for each 1,000 feet (304.8 m) above sea level and 1% for each 10° above 60° F (15° C).

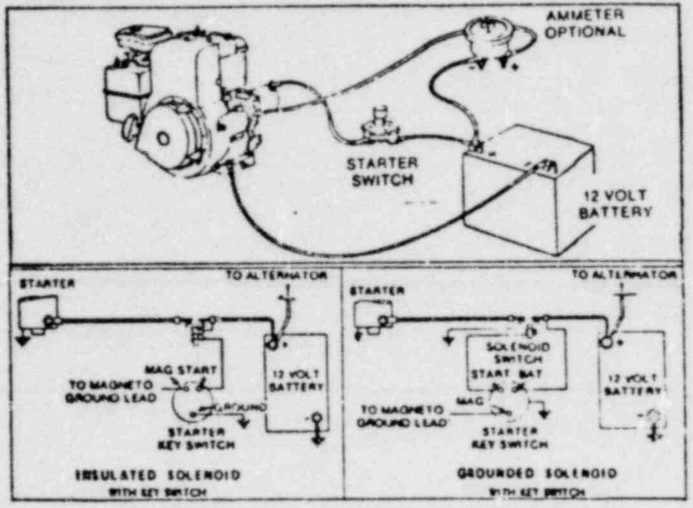
In some areas, local law requires the use of a resistor spark plug so as to suppress ignition signals. If an engine was originally equipped with a resistor spark plug, be sure to use the same type of spark plug for replacement.

TUNE-UP SPECIFICATIONS

Spark Plug Type	Champion	Autolite	Robert Bosch
Short Plug	CJ-8	235	WS9E
Long Plug	J-8	295	—
Resistor Short Plug	RCJ-8	245	WSR9E
Resistor Long Plug	RJ-8	306	—

Spark Plug Gap .... .030" (.76 mm)
Ignition Point Gap .... .020" (.51 mm)
Intake Valve Clearance .... .005" - .007" (.13 - .18 mm)
Exhaust Valve Clearance .... .009" - .011" (.23 - .28 mm)

WARNING: For electrical safety always remove cable from negative (-) side of the battery before attempting any repairs or maintenance.



STORAGE INSTRUCTIONS

Engines to be stored over 30 days should be completely drained of fuel to prevent gum deposits forming on essential carburetor parts, fuel filter and tank.

The use of a fuel additive, such as STA-BIL, or an equivalent, will minimize the formation of fuel gum deposits during storage. Such an additive may be added to the gasoline in the fuel tank of the engine, or to the gasoline in a storage container.

a. All fuel should be removed from the tank. Run the engine until it stops from lack of fuel. The small amount of fuel that remains in the sump of the tank should be removed by absorbing it with a clean, dry cloth.

b. While engine is still warm, drain oil from crankcase. Refill with fresh oil.

c. Remove spark plug, pour one ounce (29.6 cc) of engine oil into cylinder and crank slowly to distribute oil. Replace spark plug.

d. Clean dirt and chaff from cylinder, cylinder head fins, blower housing, rotating screen and muffler areas.

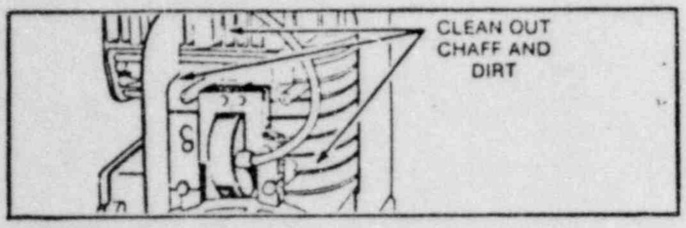
e. Store in a clean and dry area.

BRIGGS & STRATTON ENGINES ARE MADE UNDER ONE OR MORE OF THE FOLLOWING PATENTS

2 999 491
2 999 562
3 114 851
3 118 433
3 149 618
3 134 224
3 236 937
3 242 741
3 252 445
3 276 439
3 305 223
3 457 804
3 465 740
3 526 146
3 572 218
3 572 307
3 625 492
3 625 071
3 650 354
3 745 393
3 738 345
28 960
3 831 268
3 882 336
3 901 199
3 961 724
3 968 854
3 971 353
3 991 152

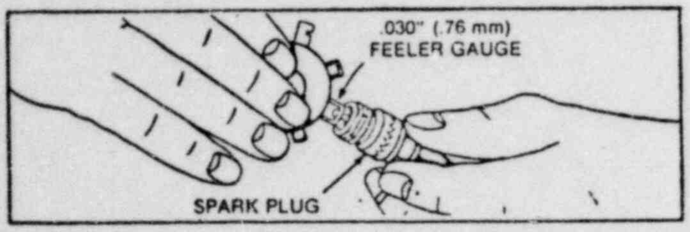
DESIGN
D-213 476 D-215 769 D 224 170 D 247 177
OTHER PATENTS PENDING

CLEAN COOLING SYSTEM — Grass, chaff or dirt may clog the rotating screen and the air cooling system, especially after prolonged service cutting dry grass. Yearly or every 100 hours, whichever occurs first, remove the blower housing and clean the areas shown to avoid overspeeding, overheating and engine damage. Clean more often if necessary.



DANGER: Periodically clean muffler area to remove all grass, dirt and combustible debris.

SPARK PLUG — Clean and reset gap at .030" every 100 hours of operation.



CAUTION: Do not blast clean spark plug. Spark plug should be cleaned by scraping or wire brushing and washing with a commercial solvent.

Sparking can occur if wire terminal does not fit firmly on spark plug, or if stop switch vibrates against spark plug. Reform terminal or repair switch if necessary.

REMOVE COMBUSTION DEPOSITS every 100-300 hours of operation. Remove cylinder head and cylinder head shield. Scrape and wire brush the combustion deposits from cylinder, cylinder head, top of piston and around valves. Use a soft brush to remove deposits. Re-assemble gasket, cylinder head and cylinder head shield. Turn screws down finger tight with the three longer screws around the exhaust valve, if so equipped. Torque cylinder head screws in a staggered sequence to 140 inch pounds (15.82 Nm).

SPARK ARRESTER EQUIPPED MUFFLER — If engine muffler is equipped with spark arrester screen assembly, remove every 50 hours for cleaning and inspection. Replace if damaged.

ADJUSTMENTS

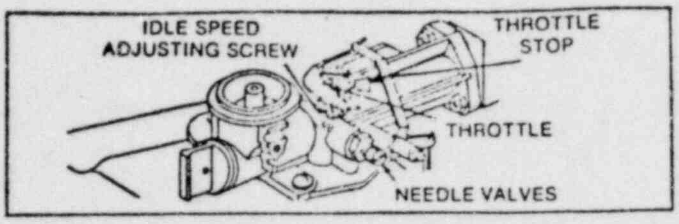
CARBURETOR ADJUSTMENTS

Minor carburetor adjustment may be required to compensate for differences in fuel, temperature, altitude or load.

TO ADJUST CARBURETOR — Turn needle valve clockwise until it just closes.

CAUTION: Valve may be damaged by turning it in too far.

Now open needle valve 1½ turns counterclockwise. This initial adjustment will permit the engine to be started and warmed up prior to final adjustment.



FINAL ADJUSTMENT

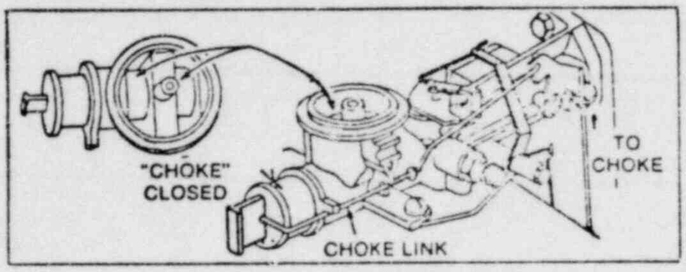
Place governor speed control lever in "FAST" position. Turn needle valve in until engine misses (clockwise — lean mixture) then turn it out past smooth operating point until engine runs unevenly (rich mixture). Now turn needle valve to the midpoint between rich and lean so the engine runs smoothly. Next, adjust idle RPM. Rotate throttle counterclockwise and hold against stop. Adjust idle speed adjusting screw to obtain 1750 RPM. Release throttle — engine should accelerate without hesitation or sputtering. If engine does not accelerate properly, the carburetor should be re-adjusted, usually to a slightly richer mixture.

CONTROL ADJUSTMENTS:

Proper choke and speed control operation is dependent upon proper adjustment of remote controls on the powered equipment.

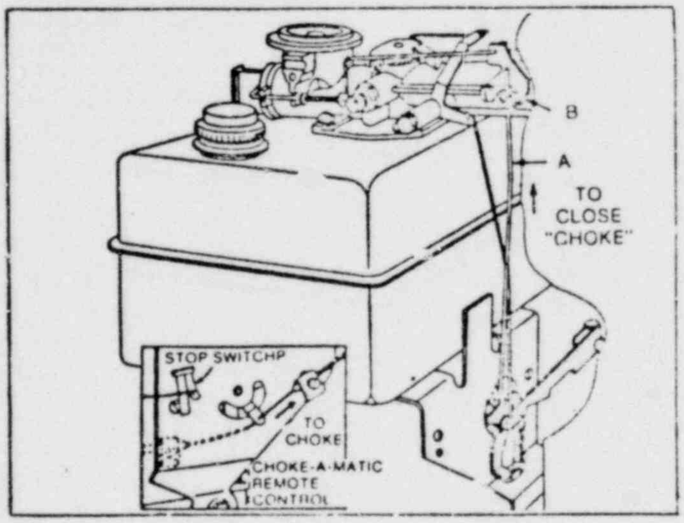
TO CHECK OPERATION OF CHOKE CONTROLS:

Remove air cleaner. Move remote control lever to "CHOKE" or "START" position. Choke should be fully closed as shown. Replace air cleaner.



To Adjust:

Place remote control lever on equipment in FAST position. Choke operating link "A" should be just touching bell crank lever at "B." See illustration.



SPEED CONTROL ADJUSTMENT

The acceptable operating speed range is 1800 to 3600 RPM. Idle speed is 1750 RPM. The manufacturer of the equip-

To avoid misunderstandings which might occur between engine owners and authorized Briggs & Stratton service accounts, we are listing causes of engine failures, where repair or replacement is NOT covered by Briggs & Stratton warranty.

NORMAL WEAR:

Warranty will not cover repair where normal use has exhausted the life of a part or engine. Engines, like all mechanical devices, need periodic parts replacement and service to perform well. It should be remembered that the service life of any engine is dependent on the care it receives and the conditions under which it has to operate. Some applications, such as tillers, trash pumps, rotary mowers, are very often used in dusty or dirty conditions, which can cause what appears to be premature wear. Such wear, when caused by dirt, dust, spark plug cleaning grit or other abrasive material, which has entered the engine because of improper maintenance, is not covered by Warranty.

ABUSE OR NEGLECT:

1. Bent or broken crankshafts: Such damage is normally the result of abuse, such as striking a solid object with the cutter blade on a rotary lawn mower, and is not covered by Warranty.
2. Engine repairs required from the use of old or stale fuel are not covered by Warranty. Such failures would include sticking valves, and carburetors and fuel pipes which are clogged by gum deposits which form through the use of stale fuel. Always use clean fresh regular gasoline.
3. Parts which are scored or broken because an engine was operated without sufficient lubricating oil or the proper grade of lubricating oil are not covered by Warranty. Check oil level at least every five hours and refill when necessary. Change oil at recommended intervals.
4. Damage caused by overheating or overspeeding is not covered by Warranty. Overspeeding or overheating occurs if the cooling fins become plugged with dirt, grass or debris, or if an engine is operated in a confined area without sufficient ventilation. Clean fins on the cylinder, cylinder head and flywheel regularly.
5. Damage or wear caused by dirt which enters the engine because of improper air cleaner maintenance is not covered by Warranty. Clean and re-oil the air cleaner regularly.
6. Damage or wear caused by grit from blast cleaning spark plugs is not covered by Warranty. We do not approve of cleaning spark plugs on an abrasive blast cleaning machine, since grit can remain in the plug, and later enter the engine.
7. Warranty does not cover the tune-up or adjustment of an engine unless the need for such repair is the result of defects in material or workmanship or both. If equipment is assembled and adjusted by the owner, the engine Operating and Maintenance Instructions are sufficiently clear to permit the average owner to make minor adjustments. Such minor adjustments are NOT covered by Warranty.
8. Broken parts, which result from excessive vibration caused by loose engine mounting, loose cutter blades, blade unbalance, improperly attaching equipment to engine crankshaft, overspeeding or abuse in operation, are not covered by Warranty.
9. Repair or adjustment of associated parts or assemblies such as clutches, transmissions, remote controls, etc., which are not of Briggs & Stratton manufacture will not be covered by Briggs & Stratton Warranty.
10. Only ORIGINAL Briggs & Stratton parts, or parts approved by Briggs & Stratton may be covered by Warranty.

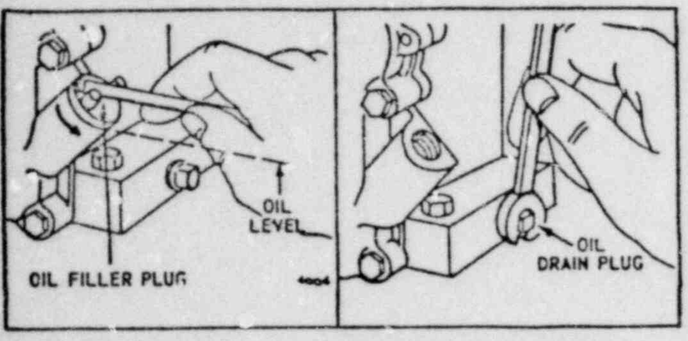
WARRANTY IS AVAILABLE ONLY THROUGH SERVICE CENTERS WHICH HAVE BEEN AUTHORIZED BY THE BRIGGS & STRATTON CORPORATION. YOUR NEAREST SERVICE CENTER IS LISTED IN THE YELLOW PAGES OF YOUR TELEPHONE DIRECTORY, UNDER "ENGINES, GASOLINE" OR "GASOLINE ENGINES."

FORM 271027-2/79

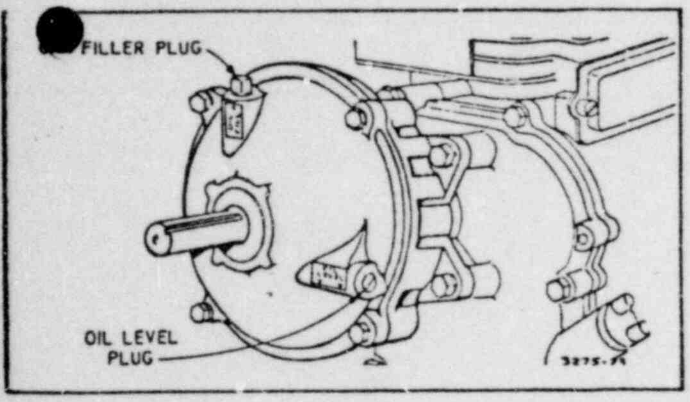
MAINTENANCE

CHECK OIL LEVEL regularly — after each five hours of operation. BE SURE OIL LEVEL IS MAINTAINED.

CHANGE OIL after first five hours of operation. Thereafter change every 25 hours of operation. Remove oil drain plug and drain oil while engine is warm. Replace drain plug. Remove oil fill plug or oil-minder and refill with new oil of proper grade. Replace oil fill plug or oil-minder.



CHANGE OIL (GEAR REDUCTION optional) Remove oil level plug and oil fill plug. Drain oil every 100 hours of operation. To refill, pour 10W-30 oil into filler hole until it runs out level check hole. Replace both plugs. Oil fill plug has a vent hole and must be installed on top of gear case cover.



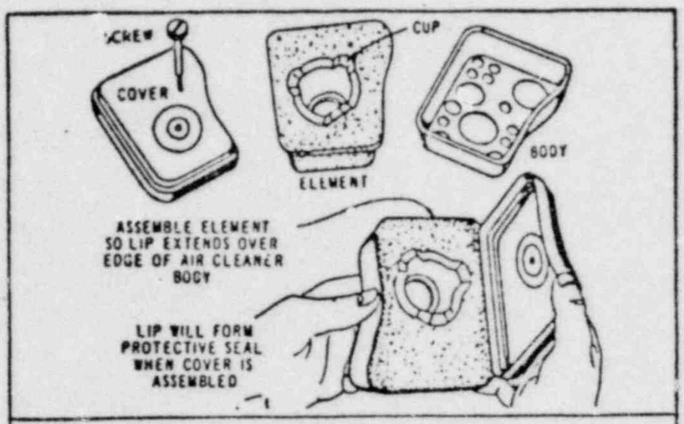
TO SERVICE AIR CLEANER

"OIL FOAM" AIR CLEANER

Clean and re-oil foam element at three month intervals or every 25 hours, whichever occurs first.

NOTE: Service air cleaner more often under dusty conditions.

1. Remove screw.
2. Remove air cleaner carefully to prevent dirt from entering carburetor.
3. Take air cleaner apart and clean.
   a. WASH foam element in a liquid detergent and water to remove dirt.
   b. Wrap foam in cloth and squeeze dry.
   c. Saturate foam with engine oil. Squeeze to remove excess oil.
4. Reassemble parts and fasten to carburetor securely with screw.



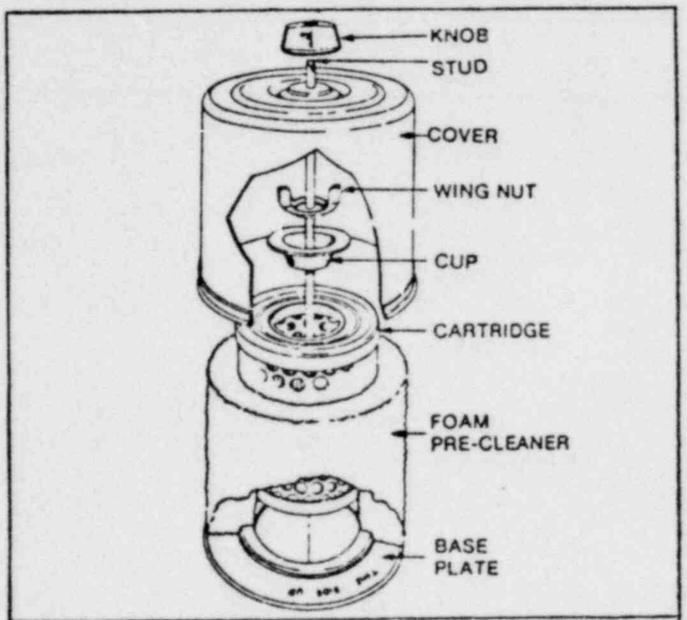
OIL FOAM AIR CLEANER

DUAL ELEMENT AIR CLEANER

Clean and re-oil foam pre-cleaner at three month intervals or every 25 hours, whichever occurs first.

NOTE: Service more often under dusty conditions.

1. Remove knob and cover.
2. Remove foam pre-cleaner by sliding it off of the paper cartridge.
3. a. Wash foam pre-cleaner in liquid detergent and water.
   b. Wrap foam pre-cleaner in cloth and squeeze dry.
   c. Saturate foam pre-cleaner in engine oil. Squeeze to remove excess oil.
4. Install foam pre-cleaner over paper cartridge. Reassemble cover and screw down tight.



DUAL ELEMENT AIR CLEANER

Yearly or every 100 hours, whichever occurs first, remove paper cartridge. (Service more often if necessary.) Clean by tapping gently on flat surface. If very dirty, replace cartridge, or wash in a low or non-sudsing detergent and warm water solution. Rinse thoroughly with flowing water from inside out until water is clear. Cartridge must be air dried thoroughly before using.

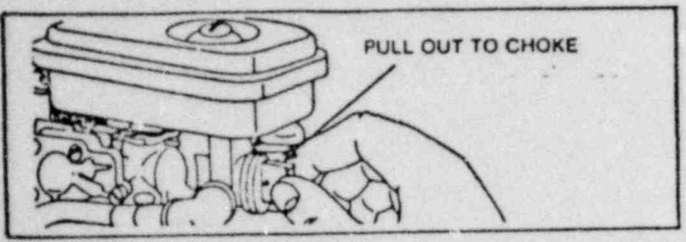
CAUTION: Petroleum solvents are not to be used to clean cartridge. They may cause deterioration of the cartridge. DO NOT OIL CARTRIDGE. DO NOT USE PRESSURIZED AIR.

STARTING

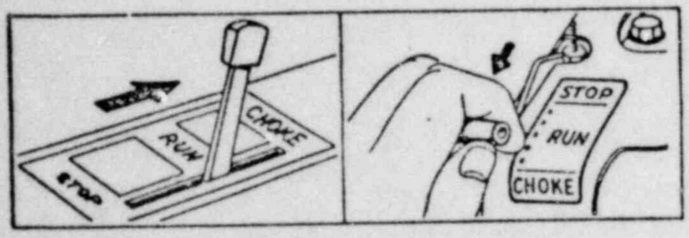
Start, store and fuel engine in a level position.

CHOKE ENGINE — Engine may be equipped with either Manual, Choke-A-Matic or Lever-Trol controls.

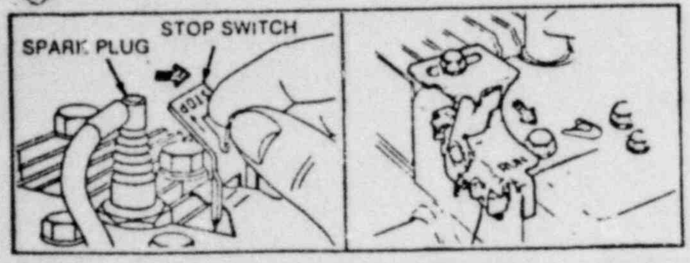
MANUAL CHOKE: Pull choke as illustrated.



CHOKE-A-MATIC and LEVER-TROLL — Move controls as far as possible toward "Choke" or Start."



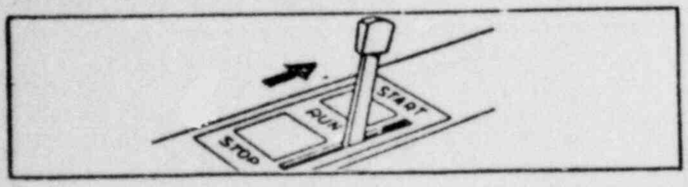
[illegible]OP SWITCH: Move STOP switch away from spark plug [illegible]llustrated, if so equipped.



NOTE: A warm engine requires less choking than a cold engine.

NOTE: Engine may not start if controls on powered equipment do not close choke fully. See ADJUSTMENT section.

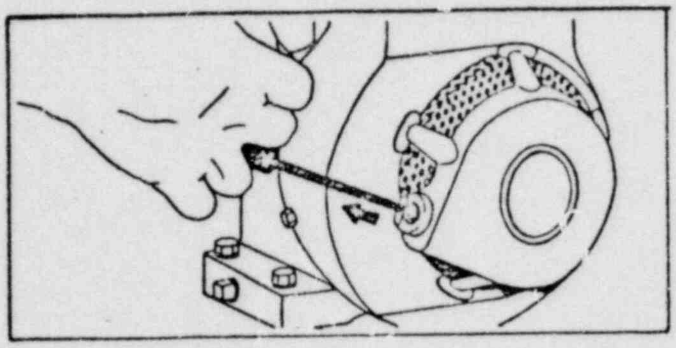
GOVERNOR SPEED CONTROL LEVER: Move governor throttle control lever to "RUN," "FAST" or "START" position if so equipped.



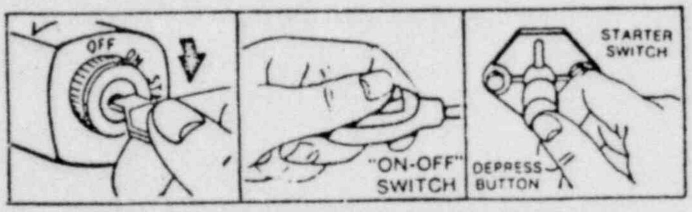
TO START ENGINE

DANGER: ALWAYS KEEP HANDS AND FEET CLEAR OF [illegible]WER BLADE OR OTHER ROTATING MACHINERY.

Rewind Starter. (Can be used to start engine if the battery is run low or if engine cannot be started electrically. Place engine controls in "Start" and key in "On" position.) Grasp starter handle as illustrated and pull out cord rapidly to overcome compression and prevent kickback. Repeat if necessary with choke opened slightly. When engine starts, open choke gradually.



Electric Starter. On engines equipped with 12 volt starting systems, turn key to "START" position or press starter button. On engines equipped with 120 volt starting systems press "On" button of conductor cord's integral "On-Off" switch. Release as soon as engine starts and gradually open choke.



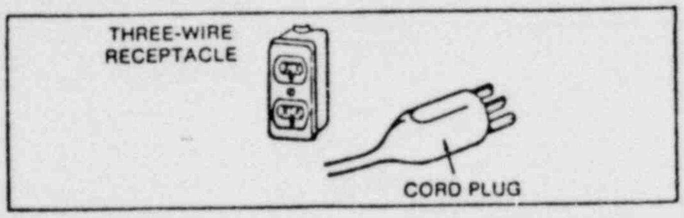
Tips to obtain best electric starter performance

a. Short starting cycles (2 to 3 seconds) provide the longest battery life.

b. Keep the battery fully charged. This assures quick and easy starts.

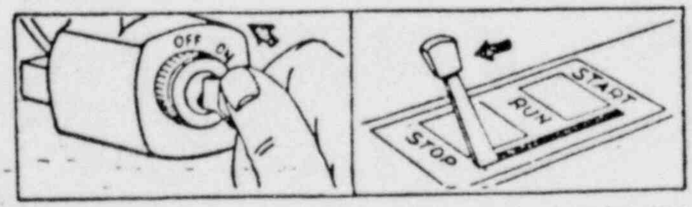
c. Disengage load from engine during start.

CAUTION: The 120 volt electric starter is equipped with a three-prong plug for your safety. The longer prong in this plug is connected to the starter motor housing. When the starter motor is plugged into the three wire cord supplied, and the cord is plugged into a properly grounded receptacle, it will protect the user from shock should the starter motor insulation fail for any reason. If a longer extension cord is used with this starter it should also have three-prong and three-hole plugs.

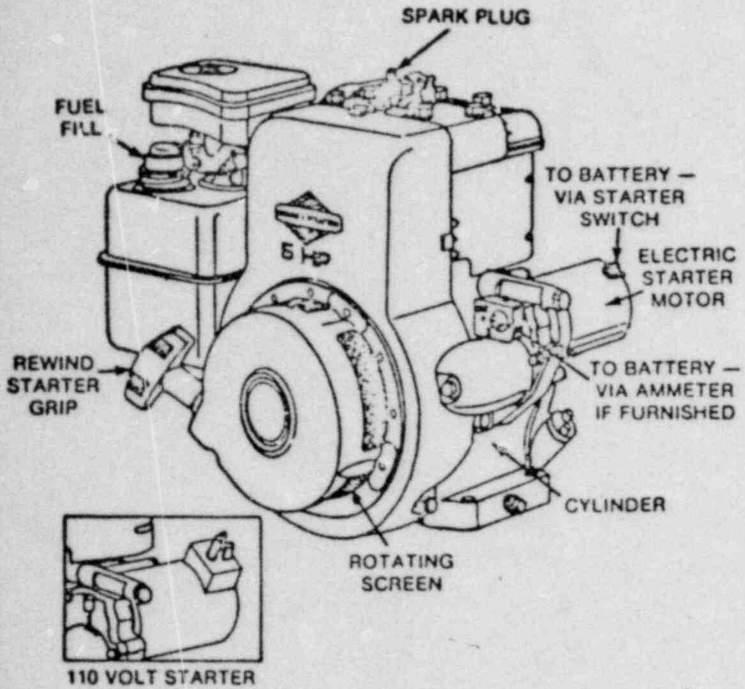


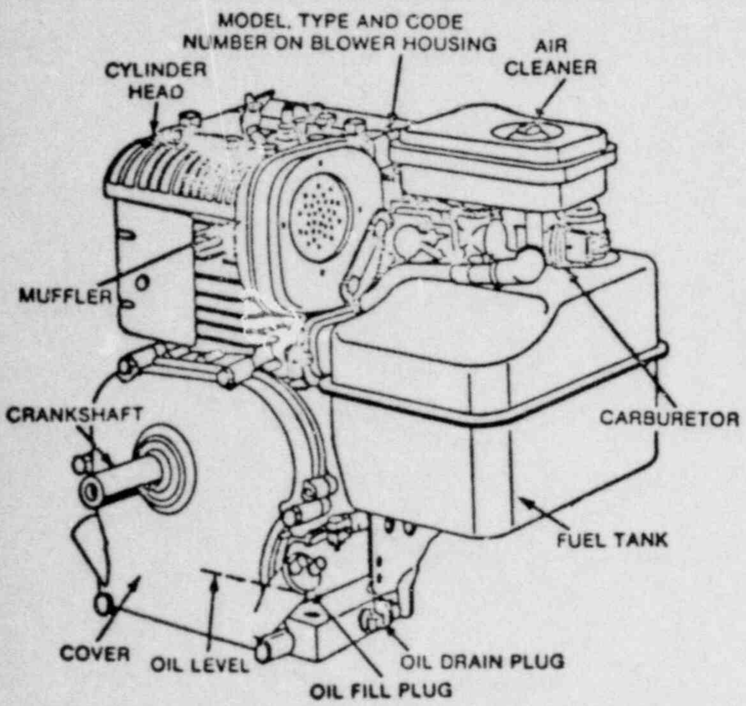
TO STOP ENGINE

Turn key to "OFF" position or move control lever to "STOP" position.



CAUTION: Always remove key from switch when leaving equipment unattended or when equipment is not in use.



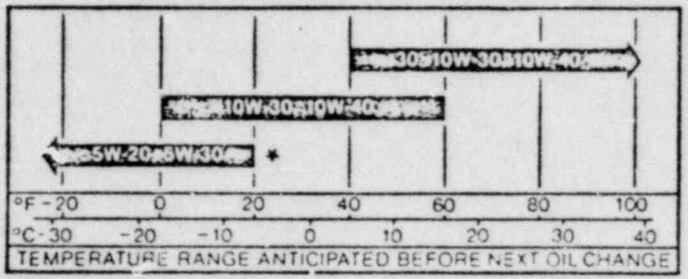


BEFORE STARTING

READ THE OPERATING INSTRUCTIONS OF THE EQUIPMENT THIS ENGINE POWERS

Use a high quality detergent oil classified "For Service SC, SD, SE or MS." Detergent oils keep the engine cleaner and retard the formation of gum and varnish deposits. Nothing should be added to the recommended oil.

RECOMMENDED SAE VISCOSITY GRADES

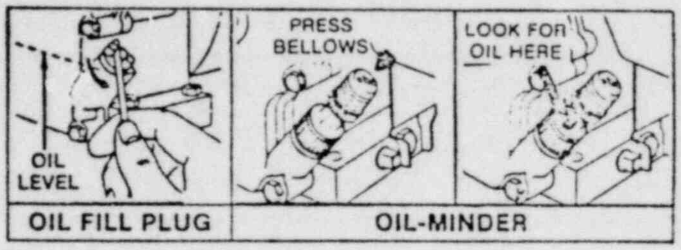


*If not available, a synthetic oil may be used having 5W-20, 5W-30 or 5W-40 viscosity.

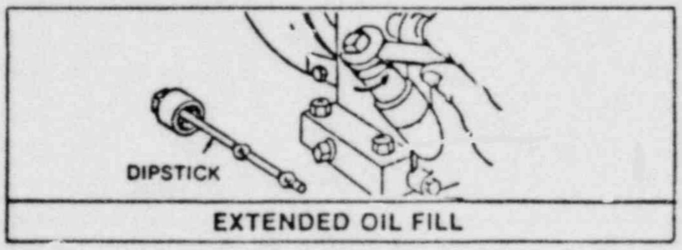
TO FILL CRANKCASE WITH OIL

Place engine level. Clean area around oil fill before removing oil fill plug or oil minder.

OIL FILL PLUG Remove oil fill plug or (optional) oil-minder. Fill crankcase to point of overflowing. POUR SLOWLY. Capacity 1¼ pints (0.6 liters). Replace oil fill plug or oil-minder.



EXTENDED OIL FILL. (Optional) Remove cap and dipstick. FILL TO FULL MARK on dipstick, POUR SLOWLY. Capacity 1¼ pints (0.6 liters). When checking oil level, screw dipstick assembly firmly but slowly until cap bottoms on tube. DO NOT OVERFILL. Dipstick assembly must be securely assembled to tube at all times when engine is operating.

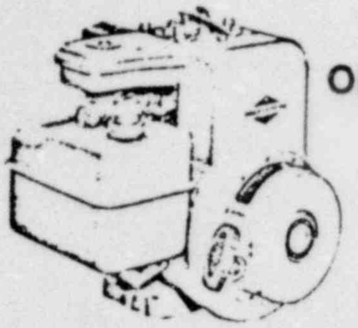


CHARGE BATTERY

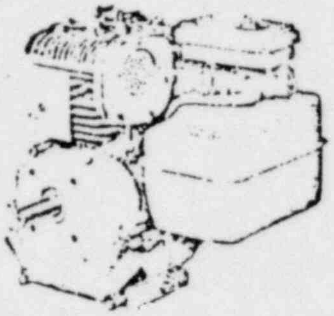
Charge battery before use on engines equipped with (OPTIONAL) 12V electric starter motor. See equipment manufacturers recommendations.

FILL FUEL TANK

Use clean, fresh "regular, low-lead or lead-free" grade gasoline. DO NOT MIX OIL WITH GASOLINE.



Briggs & Stratton
OPERATING AND MAINTENANCE INSTRUCTIONS
MODEL SERIES
100200 to 100299
130200 to 131299



IN THE INTEREST OF SAFETY

DO NOT RUN ENGINE AT EXCESSIVE SPEEDS. Operating an engine at excessive speeds increases the hazard of personal injury. DO NOT TAMPER WITH PARTS WHICH MAY INCREASE THE GOVERNED SPEED.

For rotary lawnmower safety, A.N.S.I. Standard Safety Specifications for Power Lawn Mowers specify a maximum blade tip speed of 19,000 feet per minute (96.5 meters per second), primarily to reduce the hazard from thrown objects.

Rotary lawnmower manufacturers select the governed top speed of the engine based on the length and design of the cutter blade and design of other mower parts.

All rotary lawnmowers should be checked for conformance to the A.N.S.I. Standard Safety Specifications for Power Lawn Mowers on blade tip speed, if the engine is repaired or replaced, or if mower parts are changed.

DANGER: GASOLINE VAPOR IS HIGHLY FLAMMABLE. Refuel outdoors preferably, or only in well ventilated areas.

DO NOT STORE, SPILL OR USE GASOLINE NEAR AN OPEN FLAME or devices such as a stove, furnace, water heater which utilize a pilot light, or devices that can create a spark.

If gasoline is accidentally spilled, move machine away from area of spill and avoid creating any source of ignition until gasoline vapors have dissipated.

DO NOT REFUEL GASOLINE TANK WHILE ENGINE IS RUNNING.

DO NOT RUN THE ENGINE IN AN ENCLOSED AREA. Exhaust gases contain carbon monoxide, an odorless and deadly poison.

TO PREVENT ACCIDENTAL STARTING always remove the spark plug from the engine, before working on the engine or equipment driven by the engine.

Except for adjustment; DO NOT operate engine if air cleaner or cover directly over carburetor air intake is removed. Removal of such part could create a fire hazard.

DO NOT OPERATE WITHOUT A MUFFLER OR TAMPER WITH THE EXHAUST SYSTEM. Damaged mufflers or spark arresters could create a fire hazard. Inspect periodically and replace if necessary.

ALWAYS KEEP HANDS AND FEET CLEAR OF ROTATING PARTS.

IN THE INTEREST OF ENVIRONMENT

A muffler which leaks because of rust or damage can permit an increased exhaust noise level. Therefore, examine the muffler periodically to be sure it is functioning effectively. To purchase a new muffler, see SERVICE AND REPAIR INFORMATION.

WARNING: If this engine is not equipped with a spark arrester and is to be used on any forest covered, brush covered, or grass covered unimproved land, before using on such land a spark arrester must be added to the muffler. The arrester must be maintained in effective working order by the operator. In the State of California the above is required by law (Section 4442 of the California Public Resources Code). Other states may have similar laws. Federal laws apply on federal lands. See your Authorized Briggs & Stratton Service Center for spark arrester muffler options.

SERVICE & REPAIR INFORMATION

If service or repair is needed, contact an Authorized Briggs & Stratton Service Center. To serve you promptly and efficiently, the Service Center will need the model, type and code number on your engine.

Each Authorized Service Center carries a stock of original Briggs & Stratton repair parts and is equipped with special service tools. Trained mechanics assure expert repair service on all Briggs & Stratton engines.

Major engine repairs should not be attempted unless you have the proper tools and a thorough knowledge of internal combustion engine repair procedure.

Your nearest service center is listed in the "Yellow Pages" under "Engines, Gasoline" or "Gasoline Engines". He is one of over 25,000 authorized dealers available to serve you.

yellow pages

This illustrated book includes "Theories of Operation", common specifications, and detailed information covering the adjustment, tune-up and repair procedures for 2 through 16 H.P. single cylinder models. It is available from any Authorized Briggs & Stratton Service Center. Order as Part Number 270962.

BRIGGS & STRATTON

FORM NO. 270096-2/79
PRINTED IN U.S.A.

BRIGGS & STRATTON CORP.
Milwaukee, Wisconsin 53201

Superseding Warranty

BRIGGS & STRATTON ENGINE WARRANTY POLICY

effective October 1, 1976

Replaces all undated previous Warranties and all Warranties dated before October 1, 1976

When warranty repair is justified, Briggs & Stratton Corporation welcomes such repair by any of its Authorized Service Centers.

In most instances, the requests for warranty brought to the attention of our service accounts are handled in a prompt routine manner with no question regarding their validity. However, some requests for warranty are received which are not justified. In these cases, engine owners may not be aware that the premature failure of their engine was the result of abuse or neglect, or that the difficulty which they experience is caused by the equipment rather than the engine.

BRIGGS & STRATTON WARRANTY covers only DEFECTIVE MATERIAL and/or WORKMANSHIP.

ONE YEAR LIMITED WARRANTY

"For one year from date of purchase, Briggs & Stratton Corporation will replace for the original purchasers, free of charge, any part, or parts of the engine, found upon examination by any Factory Authorized Service Center, or by the Factory at Milwaukee, Wisconsin, to be defective in material or workmanship or both; this is the exclusive remedy. All transportation charges on parts submitted for replacement under this warranty must be borne by purchaser. For warranty service contact your nearest Authorized Service Center as listed in the 'Yellow Pages' under 'Engines, Gasoline' or 'Gasoline Engines.' THERE IS NO OTHER EXPRESS WARRANTY. IMPLIED WARRANTIES, INCLUDING THOSE OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE ARE LIMITED TO ONE YEAR FROM PURCHASE AND TO THE EXTENT PERMITTED BY LAW ANY AND ALL IMPLIED WARRANTIES ARE EXCLUDED. LIABILITY FOR CONSEQUENTIAL DAMAGES UNDER ANY AND ALL WARRANTIES ARE EXCLUDED TO THE EXTENT EXCLUSION IS PERMITTED BY LAW. Some states do not allow limitations on how long an implied warranty lasts, and some states do not allow the exclusion or limitation of incidental or consequential damages, so the above limitation and exclusion may not apply to you. This warranty gives you specific legal rights and you may also have other rights which vary from state to state."

Briggs & Stratton Corporation

F.P. Stratton Jr.

F.P. Stratton, Jr.
President and Chief Executive Officer

NO REGISTRATION (WARRANTY) CARD IS NECESSARY TO OBTAIN WARRANTY ON BRIGGS & STRATTON ENGINES. FILL IN THE REQUIRED INFORMATION AND RETAIN FOR YOUR RECORD:
(See Decal on Blower Housing for Model, Type and Code Number)

Engine Model No. ____________________ Type No. ____________________ Code No. ____________________

Dealer Purchased From ____

Type of Equipment ____

Name or Trademark of Equipment Manufacturer ____

If warranty service is needed contact your nearest Authorized Service Center. For prompt attention your center will need to know the engine model, type and code number, the trouble experienced and the total number of hours the engine has run. If you differ with the decision of a Service Center on a warranty claim, ask the Service Center to submit all supporting facts to the Factory for review. If the Factory decides that your claim is justified, you will be fully reimbursed for those items accepted as defective.

(over)

APPENDIX 10.0.0

STAFF QUALIFICATIONS

STAFF QUALIFICATIONS

A. S. Chater - Environmental/Radiation Department Manager

EDUCATION:

M. S. Nuclear Engineering - University of Utah, 1977,
Specialization: Environmental and Radiation Contamination. Research, System Design and Analysis.

Thesis - "Radon-222 Exhalation as a Function of Depth of Tailings." University of Utah, June 1977, Salt Lake City. The subject dealt with Radon-222 exhalation rate vs. various depths of sandy and fine tailings and comparison of experimental values to theoretical values.

B. S. Industrial Engineering - University of Utah, 1975.

EXPERIENCE:

11/78 to Present: UNC MINING AND MILLING SERVICES, INC.
Environmental/Radiation Department Manager. Complete responsibility of establishing and implementing radiation safety and environmental monitoring programs to assure complete compliance with the rules and regulations. Develop appropriate analytical procedures, training programs, emergency and disaster procedures. In charge of developing environmental and work area surveillance procedures and programs. Establishing environmental radiological laboratory to aid and enhance UNC capability for detection of radiation, radioactive contamination, environmental contamination and personnel exposures. Procurement and maintenance of environmental and radiation monitoring and protection activities of the Radiation Technicians. Additional responsibilities are to review and approve the maintenance of environmental and radiation monitoring instruments, operating procedures, to assure complete compliance with rules and regulations. Furthermore, auditing conformance to procedural requirements dealing with health, safety, radiation and environmental protection. Establishing and directing all Quality Control and Quality Assurance programs associated with environmental sampling, sample preservation, and laboratory activities.

1977 to 1978: GEORGIA - PACIFIC CORPORATION
Plant Engineer. Complete responsibility of monitoring boiler stack and reactor stack emmission to comply with State and Federal regulations. Also monitoring dust emmissions from storage bins and recirculating system. Monthly SEC environmental report to G. P. headquarters. Notification of Utah State Division of Health, Bureau of Air Quality in case of excessive boiler, reactor, storage bins or recirculating

system emissions within 24 hours of occurrance. Monthly energy consumption report to the Department of Energy at Washington, D. C. Obtained approvals of Utah State Division of Health, Bureau of Air Quality for any modification, revision, or installation of new equipment. Upgrading plant operation for increased production with proper engineering analysis and design. Installation and application of electronic and pneumatic controls and drive for increased production, reliability and efficiency. General Plant Engineering responsibilities.

1975 to 1977: FORD, BACON & DAVIS UTAH, INC.
Research Assistant. Worked on the stabilization of uranium mill tailings piles. Also had complete responsibility for determination of Radon-222 emanation vs. moisture content, depth of tailings and pressure. Also investigating the effects of different types of cover material, chemical treatment and chemical stabilization on the reduction of Radon-222 emanation.

B. M. Angle - Environmental Chemist

EDUCATION:

B. A. Chemistry - Northwestern University 1970.

EXPERIENCE:

5/80 to Present: UNC MINING AND MILLING SERVICES, INC.
Environmental Chemist. Responsible, under supervision of RSO, for establishing of environmental and radiation laboratory, procurement and maintenance of radiation detection instruments. Responsible for adoption and evaluation of laboratory analytical procedures to assure compliance with State and Federal requirements. Responsible for laboratory certification programs and for Quality Control and Quality Assurance program. Responsible for the training and supervision of all laboratory technicians. Assumes all duties and responsibilities of the RSO in his absence.

7/79 to 5/80: CONTROLS FOR ENVIRONMENTAL POLLUTION, INC.
Operations Manager. Supervise the three analytical divisions of the company. Evaluate, interpret, and report all analytical results. Assist clients in the design and operation of sampling programs to assure compliance with their environmental and bio-assay monitoring requirements. Correspond with clients regarding special requirements or problems with their samples.

9/79 to 5/80: CONTROLS FOR ENVIRONMENTAL POLLUTION INC.
Manager, Nuclear Measurements. Supervise the routine operation and maintenance of all nuclear measurement instruments used by the company. Responsible for the evaluation and selection of new instruments as needed by the company.

9/78 to 7/79: CONTROLS FOR ENVIRONMENTAL POLLUTION, INC.
Operations Coordinator. Responsible for coordinating the use of all laboratory facilities and equipment by the three operational departments and for ensuring that all work performed for a client by the different departments was properly assembled and reported. Informed clients of any difficulties or delays in the analysis of their samples. Arranged personnel and facilities for special analytical and report writing projects.

1976 to 1979: CONTROLS FOR ENVIRONMENTAL POLLUTION, INC.
Manager of Biological Sciences. Supervised the personnel of the radiobioassay department. Supervised the calculation and reporting of results. Supervised the purchase of all reagents and equipment. Developed procedures and analytical techniques when necessary to meet the requirements of the client. Performed numerous special analyses when client needs required results which could not be obtained by routine methods.

1974 to 1976: CONTROLS FOR ENVIRONMENTAL POLLUTION, INC.
Radiobioassay Chemist. Analyzed human biological samples for specific radioactive elements and isotopes using standard analytical procedures when available and designing procedures and techniques when necessary. Gained substantial experience in the analysis of urine and feces for radiometric and fluorometric Uranium and isotopes of Plutonium, Americium, Cobalt, Radium, and other elements.

ADDITIONAL EXPERIENCE:

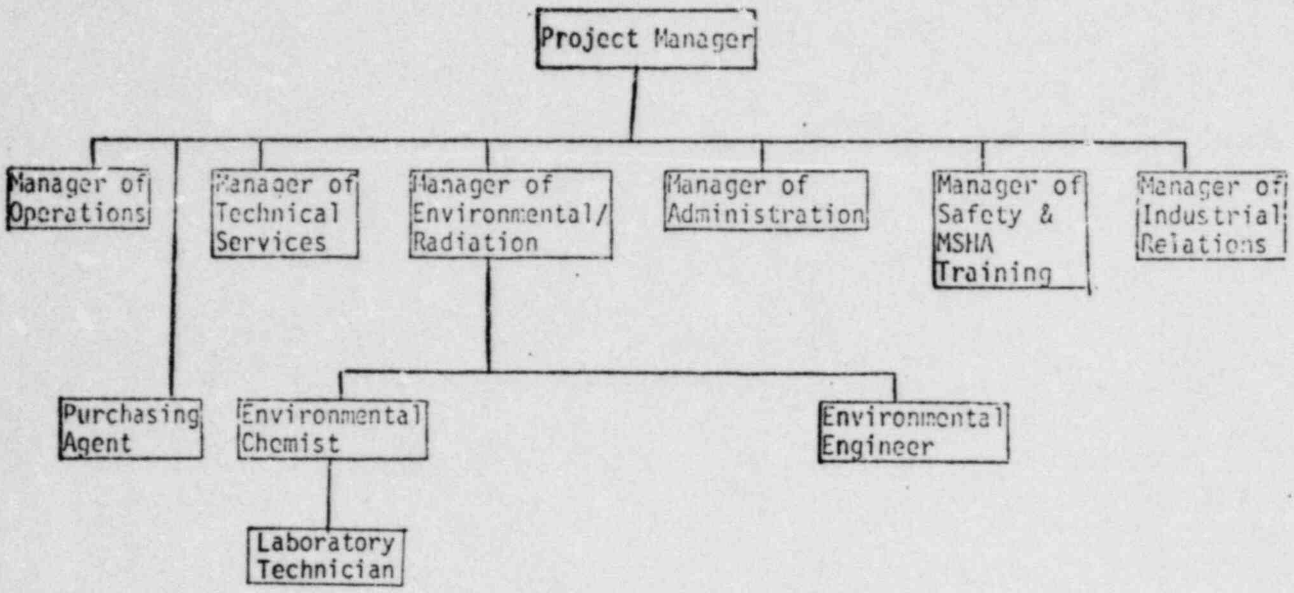
1976 to 1980: CONTROLS FOR ENVIRONMENTAL POLLUTION, INC.
Analyze samples and report results in a program to monitor the I-131 concentration in sewage effluent to be used as secondary coolant for a nuclear reactor. Initial work included the modification of an existing analytical procedure so that analytical results of the required sensitivity could be obtained. These results are used to predict the trends of I-131 concentration in the water in order to design a facility capable of substantially reducing or removing the iodine from the water before the water is used by the reactor. Additionally, assisted in the design and performed analyses in a program to determine the necessary types and concentrations of chemicals to provide for the efficient removal of Radium-226 from a uranium tailings pond.

INSTRUMENTATION USED:

Experienced with the routine operation, maintenance, and repair of:

Alpha Proportional Counters (Nuclear Measurements Corp., Ludlum Instruments)
Alpha Scintillation Counters (Ludlum Instruments)
Alpha/Beta Proportional Counters (Beckman Low Beta II, Beckman Wide Beta)
Alpha Spectrometers (Northern Scientific, Nuclear Data)
Gamma Sepctrometers (Northern Scientific/Data General)
Liquid Scintillation Counters (Beckman)
Computer (Tektronics 4051 Basic Language)
Fluorometers (Jarrel Ash, Turner)

DEPARTMENTAL ORGANIZATION



APPENDIX 10.0.1

STATISTICAL TERMINOLOGY

STATISTICAL TERMINOLOGY

mean: The sum of the test results divided by the number of results taken; that is, $X = \Sigma X_i/n$, where X = mean, X_i = individual result, and n = number of results.

precision: A measure of the reproducibility among replicate observations.

variance: The sum of the squares of deviations of the test results from the mean after division by one less than the total number of results; that is, $\text{VAR} = \sum_{i=1}^{n} (\overline{X} - X_i)^2/(n - 1)$

standard deviation: The square root of the variance; that is

$$\sigma = (\text{VAR})^{\frac{1}{2}} = \left[\sum_{i=1}^{n} (\overline{X} - X_i)^2/(n - 1)\right]^{\frac{1}{2}}$$

range: The difference between the highest test result and the lowest test result in a set of observations.

accuracy: A measure of the agreement observed and accepted values.

systematic error: Errors that may be traced to the personal errors of the experimenter, the instrumental errors of his measuring devices, the errors that repose in the method of analysis he employs, or a combination of these. Accuracy describes this type of variability or error.

random error: The necessity for making estimations is inherent in the process of collecting data for the measurement of any quantity. For this reason, any measurement will be uncertain, in any amount that depends on the relative magnitude of the estimations involved in its evaluation. Careful experimental design can reduce this uncertainty; however, small irreducible variations will remain. Since radioactive decay is a random process, any counting measurement will have a random error associated with it. Precision desribes this type of variability or error.

bias: The difference between the average of a set of test results and the accepted value. Bias usually is indicated only when a consistent difference is observed over time and can be corrected for by the application of appropriate correction factors. Bias is a measure of the systematic error.

APPENDIX 10.0.2

SIGNIFICANT FIGURES

SIGNIFICANT FIGURES

Although calculations, whether performed by hand, calculator, or computer, usually can generate more digits than are actually needed, valid digits often are thrown away. For this reason the proper use of significant figures should be emphasized.[1,2] A brief list of rules can be given for these evaluations.

1. Final zeros in a whole number may or may not be significant. In the measurement 1800 mm, do the zeros signify that the length was measured to 1 mm or do they merely locate the decimal point (i.e., to distinguish 1800 from 18 mm)? To avoid confusion in cases of this type, use a larger unit. If one chooses the meter as the unit, then 1800 mm becomes 1.8, 1.80, or 1.800 m, depending on the accuracy of the measurement.

2. Zeros before a decimal point with other preceding digits are significant. With no preceding digits, a zero before the decimal point is not significant. For example, in the measurement 150.12, the zero is significant, but in the measurement 0.12, the zero is not significant.

3. If there are no digits preceding a decimal point, the zeros following the decimal point but preceding other digits are not significant; these zeros only indicate the position of the decimal point. Thus, in the measurement 0.050015 kg, the first two zeros are not significant and serve only to locate the decimal point; however, the zeros between 5 and 1 are significant.

4. Final zeros after a decimal point are always significant figures. A weight such as 7.530 g indicates that the measurement was made to the nearest milligram.

5. To round off a figure, if the digit following those to be retained is less than 5, the digit is dropped and the retained digits are not changed.

6. To round off a figure, if the digit following those to be retained is greater than 5, the digit is dropped and the last retained digit is raised by 1.

7. To round off a figure, if the digit following those to be retained is 5 and there are no digits following it except zeros, then the last retained digit is raised by 1 if it is odd and kept unchanged if it is even.

Good books concerning quantitative analysis should be consulted for discussion of how to propagate rounding through arithmetic calculation as well as detailed discussion on the rules above. Generally, analytical results should not be reported to more than four significant figures.

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1. Skoog, D. A., and D. M. West. Fundamentals of Analytical Chemistry. 2nd ed. Holt, Rinehart, and Winston, Inc., New York, 1969. 853 pp.

2. Pierce, W. C., D. T. Turner, and E. L. Haenisch. Quantitative Analysis. 4th ed. John Wiley & Sons, Inc., New York, 1958. 647 pp.

APPENDIX 10.0.3

ERRORS CAUSED BY COMPUTATIONAL PROCESSES

ERRORS CAUSED BY COMPUTATIONAL PROCESSES

Two types of errors, rounding and truncation, may occur in any numberical process.

Truncation errors result from the necessary termination of an otherwise naturally infinite (or very lengthy) process. In most computations, it is neither possible nor necessary to carry an infinite number of significant figures. Therefore, certain classes of numbers, funtions, or constants will never be used with exact accuracy. Included are irrational numbers ($\sqrt{2}$). transcendental numbers (π or e) or functions (logarithm, exponential, sine), and fractions that have no terminating representation. With regard to this last category, fractions that have an exact representation in one number base may not possess this property in another. For example, in the decimal system used in manual calculations and by the majority of hand-held and desktop calculators, the fraction 1/10 is accurately expresses as 0.1. In the binary system used by most large scale computers, however, the fraction has no terminating representation and cannot be expressed exactly. Therfore, the sum of 10 numbers, each expressed as binary representation of 0.1, will not necessarily result in 1.0.

Rounding and chopping (truncation) errors are generally considered to be a troublesome problem when using a computer. By rounding, we refer to the symmetrical rounding procedure discussed previously. Rounding may best be compared with the chopping process by an example: As the result of some numerical operation, we obtain the number 0.41877 and wish to express it to four significant digits; by rounding, 0.41877 becomes 0.4188 but by chopping 0.41877 becomes 0.4187. In chopping, the digits beyond those that have been declared significant are ignored and have no effect on the final resulting number.

Because computers generally have a fixed word length and cannot express any number with infinite precision, some decision must be made as to what to do when the limits of precision within the computer are reached. A large number of FORTRAN compilers do, in fact, set up the object program to use chopping, which introduces less error in the calculated result than the familiar rule for rounding. In addition, the use of the familiar symmetrical rounding procedure in every arithemetric operation, including the many places in a program in which it is not really necessary, would waste computer time.

For computers and calculators, rounding and chopping may introduce errors not only in internal calculations, but also in displayed or printed output. While the machine internally may carry out arithemetic operations with precision is generally not necessary for display or printout of final results. For straight-line calculations, rounding or chopping the final answer to four significant figures may introduce a relative error that is greater by several orders of mangitude than any error accumulated during the calculation. Also, chopping of the final displayed results by the computer does introduce more error in the displayed decimal than would rounding.

Recursive or iterative operations suffer more from the effect of rounding or chopping than do straight-line calculations. This problem is typified in the resolution of multicomponent gamma ray spectra, where a set of simultaneous equations must be solved. In general, there are two types of numerical techniques for solving simultaneous equations: direct methods, which are finite. (Gaussian elimination), and indirect methods, which are infinite (iterative techniques). Obviously, no practical technique can actually be infinite; what is meant is that the direct methods will produce, in principle (neglecting rounding errors), an exact soulution, if one exists, in a finite number of artihmetic operations. An indirect method, on the other hand, would in principle require an infinite number of arithmetic operation to produce an exact solution. That is, an direct method has a truncation error, where as a direct method does not.

Rounding errors may not be neglected. In a large, ill-conditioned system the rounding errors in a direct method may make the solution meaningless; the magnitude of the final error may, in severe cases, be larger that the derived result. Therefore, in spite of its truncation error, an indirect method may be much more desirable because the problem of accumulated rounding error is minimized.

APPENDIX 10.0.4

RADIOCHEMICAL STATISTICS AND COUNTING DATA

RADIOCHEMICAL STATISTICS AND COUNTING DATA

Statistics, as a science, deals with probability. Probability is a useful concept in all areas of science becasue no two analyses or measurements give identical answers. Measurements of radioactivity are bound even more closely to statistical evaluation because of the random nature of the disintergration process. The basic laboratory question is how to determine, from a few measurements or a single measurement, the best approximation to a "true" value.

The goal of this manual has been to examine areas and to demonstrate methods that can be used to reduce systematic errors to the point that only the small unavoidable fluctuations of random error are present. Of course, every analytical result still has a degree of uncertainty. These uncertainties arise from all the measurement processes involved in the analysis, such as weighing, volumetric processes, and counting error. An analyst must try to estimate this error (uncertainty) for each single analysis by propagating the random error in all measurements for each indiviual sample. Measuring radioactivity involves a statistical evaluation of counting measurments to estimate these uncertainties and reduce them to as small a figure as possible.

The lower the level of activity in a sample, the more difficult it is to distinguish the activity of the sample measurement from statistically allowable fluctuations in the background. To evaluate sample measurements, one must be able to estimate the sensitivity of the analysis, which is sometimes called lower limit of detection (LLD) or minimum detectable activity (MDA). This section describes how sensitivities can be estimated for some analytical methods.

Counting statistics

Two types of statistical distrubutions are used most often in the radioanalytical laboratory: (1) The Gaussian, or normal, distribution is use to describe continuous variables, and (2) the Poisson distribution applies to descrete variables.

The form of the Gaussian distribution for n measured is

$$f(x) = \frac{1}{\sigma_x\sqrt{2\pi}} \exp \left[- \frac{(x - \mu)^2}{2(\sigma_x)^2} \right] ,$$

where μ = mean of the measurement values (x) ,

σ_x = standard deviation = $[\Sigma(x - \mu)^2/(n - 1)]^{\frac{1}{2}}$

This equation describes a bell-shaped curve, any area under which is related to the probability of a particular result. This area is often divided into ranges of σ_x about the mean, μ. Thus,

Area	Probability
$\mu \pm \sigma_x$	0.683
$\mu \pm 2\sigma_x$	0.955
$\mu \pm 3\sigma_x$	0.997

This division into regions is the source of terms such as 2σ, 2 sigma level, and 95 percent confidence level. Gaussian statistics are used in the radioanalytical laboratory to describe the behavior of multiple measurements of a single value.

Multiple measurements are seldom possible for most work performed in the radioanalytical laboratory. Availability of instruments for repeated countings is too expensive to maintain. For this reason, the use of Poisson statistics can allow an estimate of behavior from a single measurement.

The form of the Poisson distribution is expressed as

$$f(x) = \frac{(e^{-x})(\bar{x})^{x})}{x!} \quad ,$$

where $x = 0, 1, 2, \ldots,$

$\bar{x}$ = estimated mean.

The standard deviation, s, for the estimated mean is $\sqrt{\bar{x}}$.

EXAMPLE 1: A standard check source was counted to determine the counter efficiency. The following one-minute counts were observed: 7747, 7738, 7840, 7785, 7705, 7667, 7812, 7827, 7623, and 7739. The total count was 77483, and the mean was calculated to be $\mu = 77483/10 = 7748$. Thus, for the Gaussian distribution,

$$\sigma = [\Sigma(x - \mu)^2/(n - 1)]^{\frac{1}{2}} = 70,$$

and for the Poisson distribution,

$$s = \sqrt{\bar{x}} = 88.$$

The agreement of the two estimates indicates that the counting proceeded satisfactorily. The Gaussian estimate and the Poisson estimate should be approximately the same. The agreement of the results also shows that the Poisson estimate is a quick but satisfactory measure of counting data.

For situations in which the standard deviation is of the same order of magnitude as the mean, the distribution of data will be markedly non-Gaussian, and Gaussian probability levels and also propagation of errors give grossly biased answers. The only way to eliminate the bias is a very tedious trial-and-error use of the Poisson distribution itself.

The Poisson standard deviation often appears as slightly different expressions from that shown above when converted to a counts-per-minute (counts/min) figure:

$$s_{\text{counts/min}} = \sqrt{\bar{x}}/t$$

$$= \sqrt{R/t} \quad ,$$

where t = counting time,

R = counting rate.

PROPAGATION OF ERRORS

If all radioanalytical analyses involved only one counting measurement, estimating the uncertainty of a final result by applying Poisson statistics to obtain a standard deviation would be quite simple. However, this is seldom, if ever, the case since almost all counting data must be corrected for background contributions. Both the sample and background counts have uncertainties that must be reflected in the final result. The theory of propagation of errors can be applied to estimate the reliability of the final calculated result.[5, 6]

If Q = f(X,Y,...), where X,Y,... are independent, normally distributed variables, the asymptotic uncertainty (variance) in Q resulting from uncertainties in X,Y,... is given by the expression

$$\sigma_Q^2 = (\partial Q/\partial X)^2\sigma_x^2 \quad + \quad (\partial Q/\partial Y)^2\sigma_y^2 \quad + \quad . \; . \; .$$

The standard deviation for Q is found by taking the square root of the expression. For the specific case of the error of difference between two counting determinations (Q = X - Y), the proper equation is

$$\sigma_Q = (\sigma_s^2 \; + \; \sigma_B^2)^{\frac{1}{2}} \quad ,$$

where σ_Q = error of the difference,

σ_s = estimated standard deviation for the sample,

σ_B = estimated standard deviation for the background.

Formulas for other simple functions are shown in Table

TABLE 2 FORMULAE OF PROPAGATION OF ERROR FOR SOME SIMPLE FUNCTIONS

Function	Error formula
$Q = X \pm Y$	$\sigma_Q = (\sigma_x^2 + \sigma_y^2)^{\frac{1}{2}}$
$Q = aX \pm bY$	$\sigma_Q = (a^2\sigma_x^2 + b^2\sigma_y^2)^{\frac{1}{2}}$
$Q = XY$	$\sigma_Q = XY(\sigma_x^2/X^2 + \sigma_y^2/Y^2)^{\frac{1}{2}}$
$Q = X$	$\sigma_Q = X/Y(\sigma_x^2/X^2 + \sigma_y^2/Y^2)^{\frac{1}{2}}$
$Q = X^n$	$\sigma_Q = n(X^{n-1}\sigma_x)$
$Q = \ln X$	$\sigma_Q = \sigma_x/X$
$Q = \log X$	$\sigma_Q = 0.434\sigma_x/X$

Sources: Overman, R. T., and H. M. Clark. Radioisotope Techniques. McGraw-Hill Book Co., New York, 1960. 476 pp.

Ku, H. H. J. Res. Nat. Bur. Stand. Sect. C, 70: 263, 1966.

EXAMPLE 2: A sample counted for 50 minutes gave a gross count of 123 counts; the background for the same period was 68 counts. The error of the net count is shown below:

$$\sigma_s = 123^{\frac{1}{2}} = 11.09 \qquad (\sigma_s^2 = 123) \;;$$

$$\sigma_B = 68^{\frac{1}{2}} = 8.25 \qquad (\sigma_B^2 = 68) \;;$$

$$\sigma_Q \doteq (68 + 123)^{\frac{1}{2}} \doteq 13.82 \quad .$$

Therefore, the net count is 55±14.

A more common expression for this error term, when both a sample measurement and background measurement are involved, is

$$\sigma_Q = (R_s/t_s + R_B/t_B)^{\frac{1}{2}},$$

where R_s = gross sample counting rate,

R_B = background counting rate,

t_s = sample counting time,

t_B = background counting time.

EXAMPLE 3: A sample counted for 50 minutes gave a gross count of 137 counts. The background for a 10-minute count was 15 counts.

$$\sigma_Q = \left(\frac{137/50}{50} + \frac{15/10}{10}\right)^{\frac{1}{2}}$$

$$= (0.055 + 0.15)^{\frac{1}{2}} = (0.205)^{\frac{1}{2}}$$

$$= 0.45 \text{ counts/min.}$$

Therefore, the net counting rate is 1.74±0.45 counts/min (neglecting significant figures). Because the gross counting rate is the sum of the background and sample counting rates, the background will dictate the precision of the final answer if background and sample counting times are equal. Therefore, counting times must be selected to minimize the background contribution to the error term.

Several possibilities exist for reducing the counting error for a particular analysis: (1) increasing the counter efficiency; (2) increasing the sample size; (3) increasing the counting time; and (4) lowering the background Increasing the efficiency or sample size gives the greatest benefit since these terms are applied directly in all final calculations. Possibilities (3) and (4) vary with the square root and therefore yield a lesser reduction in the size of the error term.

When using background measurements in analytical calculations, a figure representing the background over a long period of time, rather than figures from daily measurements, is preferable. For example, background of 1.27±0.10 counts/min averaged over a year is a much better value for calculation purposes than a value of 1.33 counts/min determined for a particular day. Although using a long-term average in calculations will tend to smooth out fluctuations, an average figure should not, of course, be used when a radical change in background has occurred.

A test often used to evaluate instrument background measurements to determine whether the instrument is performing as statistically expected is the Chi-square test, in which Chi-square (χ^2) is calculated by

$$\chi^2 = [\sum_{i=1}^{n} (x_i - \bar{x})^2]/\bar{x},$$

where x_i = individual result,

$\bar{x}$ = average of all the measurements.

Values of Chi-square have been calculated and tabulated for the number of values measured.

Number of measurements	Allowed χ^2 limits
5	0.3 - 13
10	2 - 22
15	4 - 29
20	7 - 36
30	14 - 50

A value outside the limits indicates that the instrument is not performing as expected from statistical considerations.

LIMITS OF DETECTION

An examination of equation (15) shows that the percent error increases as the gross counting rate approaches the background counting rate. Neither the gross counts nor the background counts are hard, fast numbers; instead, they are members of two distributions that begin to overlap when the sample has very low amounts of activity above background. Estimating the reliability of the difference between the two measurements becomes a complex problem when overlap occurs.

Several methods for estimating the LLD or MDA in radioanalytical measurements have been proposed.[6,7] The method most commonly used assumes that, unless the gross counting rate is greater than the background counting rate plus two standard deviations, the amount of activity contained in the sample is less than detectable.

EXAMPLE 4: A low-background beta counter has an average background of 64 counts in 50 minutes measured over a period of a year.

$$s = \sqrt{\bar{x}} = \sqrt{64} = 8,$$

$$2s = 16 \text{ counts.}$$

In this example, the gross counts must exceed 80 counts in 50 minutes to give 95 percent confidence that there is actually any activity present in the sample.

Another approach to this problem is presented in Refs. 8 and 9. This procedure uses a statistical technique known as hypothesis testing, in which two types of error are assumed to be possible: (1) concluding that there is sample activity when there is none (Type I error) and (2) concluding that there is no sample activity when there is some (Type II error). The terms alpha (α) and beta (β) represent Type I and Type II, respectively; alpha is usually allowed to be 5 percent (0.05) and beta is usually set at 5 percent (0.05). The LLD is then approximated as

$$\text{LLD} \simeq (k_\alpha + k_\beta)s_o \quad ,$$

where k_α = the value of the upper percentile of the standardized normal variate corresponding to the preselected α,

k_β = the corresponding value for the predetermined degree of confidence for detecting the presence of activity $(1 - \beta)$,

s_o = the estimated standard error for the net sample counting.

If the values of α and β are set at the same level (0.05) and the sample and background counts are close, as would be the case at the LLD, other approximations also may be made. If

$$s_{net} = \sqrt{s_s^2 + s_B^2} \simeq \sqrt{2}\, s_B,$$

and

$$k = k_\alpha = k_\beta \ ,$$

then

$$\text{LLD} = 2\sqrt{2}\, k s_B \ .$$

α	$1 - \beta$	k
0.01	0.99	2.327
0.02	0.98	2.054
0.05	0.95	1.645
0.10	0.90	1.282
0.20	0.80	0.842

For the previous example, we can calculate the LLD by this method using $\alpha = \beta = 0.05$ to find the background variance:

$$\text{LLD} = 2\sqrt{2}\ ks_B \ ;$$

$$s = \sqrt{\bar{x}} = \sqrt{64} = 8 \ ;$$

$$\text{LLD} = 2\sqrt{2}\ (1.645)(8) \cong 37 \text{ counts.}$$

This value is the number of net counts above background that must be observed before one can report a result above LLD. Note that this is a more conservative estimate of the LLD than that shown in example 4. This method of LLD calculation is becoming the most accepted procedure.

This 95 percent confidence criterion is a very rigorous one. A more liberal criterion might be to allow a 20 percent chance of erroneously reporting activity when none is actually present and to keep the 5 percent requirements for not reporting activity when it is actually present. This change in criteria would lead to the following expression:

$$\begin{aligned} \text{LLD} &= (k + k)\ \sqrt{2}\ s_B \\ &= (1.645 + 0.842)\ \sqrt{2}\ s_B \\ &= (2.487)\ \sqrt{2}\ s_B \\ &= 3.52 s_B \ . \end{aligned}$$

For the previous example, the LLD calculated by this method would be 28 net counts.

The extraction of results from allowable variations can be difficult and always introduces a degree of uncertainty in the final result. This final result must be measured in light of the LLD levels before attaching meaning or significance to the value. Allowable variations and LLD levels also must be considered when formulating quality control analysis of laboratory data.

APPENDIX 10.0.5

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BIBLIOGRAPHY

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