

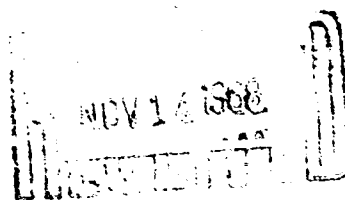
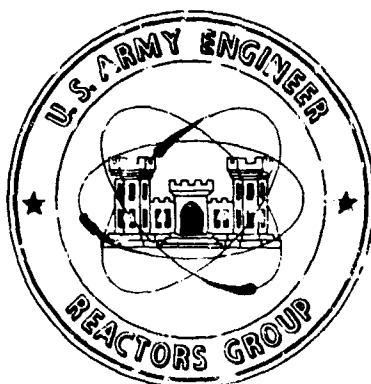
ENGINEERING DEPARTMENT
NUCLEAR POWER FIELD OFFICE
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Volume III

HEALTH PHYSICS—
PROCESS CONTROL
REFERENCE MANUAL



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SECTION 700 - PERSONNEL ACCESS CONTROL

Personnel monitoring is a Health Physics function. It includes controls to minimize and measurements to determine the extent of internal radiation to which personnel are exposed when working in an area where a significant quantity of radiation may be encountered. The main devices used for external personnel monitoring are film badge dosimeters and direct-reading pocket ionization chambers. These devices are used in conjunction with radiation work permits identifying area involved, personnel exposed, time of exposure, and pocket dosimeter readings. All personnel are also surveyed with sensitive portable instruments to assure freedom from contamination upon leaving a radiation area.

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METHOD 711

ESTABLISHMENT AND CONDITIONS FOR ALL
RADIOLOGICALLY CONTROLLED AREAS

1. SCOPE.

The purpose of this method is to describe the establishment of conditions applicable to radiologically controlled areas.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Radiation signs: "Radiation Area," "High Radiation Area," "Radioactive Material Area," "Contaminated Area," and "Airborne Radioactivity Area."

Smears: Whatman No. 41 or H. V. 70, 1- or 2- inch-diameter filter paper.

Portable radiation survey instruments: High-and-low-range beta gamma instruments; alpha instruments, if necessary.

Radiation rope and tape (magenta and yellow).

Film badges.

Dosimeters, high and low range.

Full protective clothing: coveralls, shoe covers, gloves (cotton or rubber) hoods and head covers.

Personnel respiratory protection: Full face masks, self-contained breathing apparatus.

Portable air samplers.

Stanchions

Self-adhering tapes or labels including the following: "contaminated area," "radiation hazards" and "contaminated."

4. PROCEDURES.

4.1 Area Designations for Army Nuclear Power Plants.

4.1.1 General areas.

4.1.1.1 Restricted Areas - All areas within the site boundary or fence enclosure.

4.1.1.2 Unrestricted Areas - All areas outside the site boundary or fence enclosure.

4.1.2 Specific plant areas.

4.1.2.1 Clean Area - Any area free of radioactive materials.

4.1.2.2 Designated area - Any area where personnel access is controlled. These designated areas will be the various radiation exposure areas specified in Methods 712 through 716.

4.2 General Conditions for All Designated Areas

4.2.1 No eating, drinking, or smoking shall be allowed in any area, except in those clean areas designated for this purpose.

4.2.2 The use of radioactive materials or contaminated items in a clean area will be under direct surveillance of a health physicist.

4.2.3 Radioactive materials may be carried through a clean area

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provided (1) the radioactive material is contained so that the loss of any of the material cannot occur while being transported; (2) surfaces of outside containers are free of radioactive material contamination.

4.2.4 Contaminated protective clothing shall not be worn in clean areas.

4.2.5 If radioactive material contamination occurs in a clean area , it must be removed as quickly as possible, using Decontamination Procedures listed in Method 351.

4.2.6 All visitors who enter any of the designated areas shall be required to obtain prior approval to enter the area from the Officer-In-Charge and shall be escorted by plant personnel under a RWP.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 712

RADIATION AREAS

1. SCOPE.

This method specifies the procedures to be followed in classifying and identifying the Radiation Area and the requirements for entering the area.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 711, Paragraph 3.

4. PROCEDURES.

4.1 Classifying and Identifying Radiation Area.

The method for establishing the type of area shall be by appropriate radiation surveys as described in Method 500.

4.2 Posting.

4.2.1 Post the Radiation Area with the appropriate magenta and yellow "Radiation Area" signs.

4.2.2 Post the signs at the barrier of the area which may be a radiation rope or a door. Place the signs at each possible entrance so that they are easy to see.

4.2.3 Mark the signs to indicate the radiation levels at the barrier and the date of the radiation survey. A room or area is not required to be

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posted with a caution sign owing to the presence of a sealed source, provided the radiation level 12 inches from the source container or housing does not exceed 5 millirem per hour.

4.3 Requirements for Entering Radiation Area.

4.3.1 Film badges and pocket dosimeters shall be worn by all individuals entering the area.

4.3.2 Persons shall read, comply with, and sign applicable RWP.

4.3.3 Occupancy time limits shall be set by the health physicist to insure that personnel exposure limits are not exceeded. These limits shall be adhered to under the supervision of the health physicist.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION

For the signs used for Radiation Areas, see Reference (26) in the Reference Section.

METHOD 713

HIGH RADIATION AREAS

1. SCOPE.

This method specifies the procedures to be followed in classifying and identifying a High Radiation Area, and the requirements for entering the Area.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 711, Paragraph 3.

4. PROCEDURES.

4.1 Classifying and Identifying High Radiation Area. See the radiation surveys, as described in Method 500.

4.2 Posting.

Post the High Radiation Area with the appropriate magenta and yellow signs. Follow all general instructions for posting as outlined in Method 712, Paragraph 4.2.

4.3 Requirements for Entering High Radiation Area.

4.3.1 A RWP shall be issued, properly signed, and complied with, in accordance with Method 731.

4.3.2 The shift supervisor on duty shall approve all entries and

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operations in the High Radiation Areas.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The signs used for Radiation Areas are specified in AR 385-30.

METHOD 714

RADIOACTIVE MATERIALS AREAS

1. SCOPE.

This method specifies the procedures to be followed in classifying and identifying a Radioactive Materials Area and the requirements for entering the Area.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 711, Paragraph 3.

4. PROCEDURES.

4.1 General. Radioactive Materials Areas may include any of the areas described in Methods 712 through 716. Specific radiation areas (as described in Methods 712, 713, 715, and 716) that are within the Radioactive Materials Area shall be clearly marked with the signs specific to the individual area.

4.2 Classifying and Identifying Radioactive Materials Areas.

These areas shall be determined by accountability techniques, and including recording the types and amounts of radioactive material entering and leaving the plant and the amounts and storage location of material in inventory. Radioactive Materials Areas may also be established to restrict personnel and equipment traffic in locations where radiation or contamination hazards exist

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4.3 Posting.

Post the appropriate Radioactive Materials Area sign at the entrance to the area. The conditions established by the Health Physicist is the same as those listed in Method 712, Paragraph 4.2. The source and type may also be included on a card attached to the sign or on the sign proper.

4.4 Requirements for Entering Radioactive Materials Area.

4.4.1 Restrict the area to personnel having definite business in the area.

4.4.2 Caution signs are not required to be posted at an area or room containing radioactive materials for periods of less than 8 hours, provided that (1) the materials are constantly attended during such periods by an individual who shall take the precautions necessary to prevent the exposure of any individual to radiation or radioactive materials in excess of the limits established in AR-40-14 and (2) access to such area or room is subject to control by the OIC.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The signs used for Radiation Areas are specified in AR 385-30.

METHOD 715

SURFACE CONTAMINATION AREAS

1. SCOPE.

The purpose of this method is to specify the procedures to be followed in classifying and identifying a Surface Contamination Area and the requirements for entering the Area.

2. SAMPLE.

Not Applicable.

3. APPARATUS.

Same as Method 711, Paragraph 3.

4. PROCEDURES

4.1 Classifying and Identifying Surface Contamination Area. Determine the "Surface Contamination Area" by a thorough smear and instrument survey, as described in Methods 511 and 512.

4.2 Posting.

4.2.1 Post the appropriate Surface Contamination Area sign at the entrances to all surface contamination areas.

4.2.2 The sign shall meet the conditions as specified in Method 712, Paragraph 4.2.

4.2.3 On the sign post the survey date, highest surface contamination level recorded, and the initials of the surveyor.

4.3 Requirements for entering Surface Contamination Areas:

4.3.1 The entrance into all Surface Contamination Areas shall be restricted. Protective clothing shall be required as specified in Paragraph 3.7 of Method 711.

4.3.2 If the contamination is confined to movable equipment, identify the item with a Surface Contamination Area sign or tags (see Method 251). Arrange to have the item removed to the decontamination area. Loose contamination shall be contained during transportation by use of polyethylene bags or sheet, or blotting paper (see Method 211, Paragraph 4.3).

4.3.3 If the contamination is present on fixed equipment, establish a barrier approximately 2 to 4 feet from the equipment, or cover the equipment with polyethylene sheet and tape the edges until the equipment can be decontaminated.

4.3.4 If the contamination is present on the floor and immediate decontamination (within 24 hours) is possible, mark the area off with radiation marker rope and post the Surface Contamination Area signs.

4.3.5 If immediate decontamination of the floor is not possible, mark the area off as described in Paragraph 4.3.4. In addition, place magenta and yellow adhesive tape on the floor under the rope.

4.3.6 If contamination is confined to a small floor that cannot be cleaned immediately, cover the area with blotting paper or polyethylene

sheet and tape the edges. Identify the area with a Surface Contamination Area sign (see AR 385-30) or tag (see Method 251).

4.3.7 Establish a control point for entrance into the area.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The signs used for Radiation Areas are specified in AR 385-30. The specification for limits for a Surface Contamination Area shall be as determined by the plant technical manual.

METHOD 716

AIRBORNE RADIOACTIVITY AREAS

1. SCOPE.

The purpose of this method is to specify the procedures to be followed in classifying and identifying an Airborne Radioactivity Area and the requirements for entering the area.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 711, Paragraph 3.

4. PROCEDURES.

4.1 Classifying and Identifying Airborne Radioactivity Area. Determine the airborne radioactivity by taking air samples as described in Method 513. The method of counting the air sample is described in Method 631.

4.2 Posting.

4.2.1 Post all Airborne Radioactivity Areas with the appropriate Airborne Radioactivity Area sign.

4.2.2 The sign shall be initialed and dated by surveyor and shall contain all conditions mentioned in Method 712, Paragraph 4.2.

4.3 Requirements for Entering Airborne Radioactivity Area.

4.3.1 An RWP is required for work in all Airborne Radioactivity Areas.

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4.3.2 Attempt to find the cause of the high activity if the airborne activity is greater than the limits specified in Section 6.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The signs used for Radiation Areas are specified in AR 385-30. The operational limits for airborne radioactivity shall not exceed 10% of the amount specified in Appendix B, Table I, Column I of 10 CFR 20 or M. P. C. derived therefrom.

METHOD 721

ESTABLISHING TEMPORARY CONTROL POINTS

1. SCOPE.

This method specifies the criteria and procedures for establishing Temporary Control Points.

2. SAMPLE.

Not applicable.

3. APPARATUS.

3.1 Absorbent paper.

3.2 Masking tape.

3.3 Clothes containers.

3.4 Monitoring instrument. Low-level beta-gamma survey meter and (if necessary) alpha survey meter.

3.5 Protective clothing. coveralls, labcoats, and cotton and rubber gloves, shoe covers, head covers, and rubbers.

3.6 Plastic bags.

3.7 Dosimeters.

3.8 Film badges.

3.9 Full face masks.

3.10 Self-contained breathing apparatus.

3.11 Smears.

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3.12 Air Samplers.

3.13 Polyethylene sheeting.

4. PROCEDURES.

4.1 Temporary control points may be established at the entrance to each of the designated areas described in Methods 712 through 716. Temporary control points shall be established primarily to control the spread of contamination. Entry into a Radiation Area, High Radiation Areas, Airborne Radioactivity Area, and Surface Contamination Area shall require an RWP and the establishment of a Permanent Control Point.

4.2 If the area is a Radiation Area only, the individual entering the area shall read his dosimeter before entering, and when leaving, the area. If there is a time limit for the area, the self-reading dosimeter shall be read frequently, depending on the radiation level. If applicable, time limits for exposure to beta radiation will be furnished by the health physicist.

4.3 For entrance into a Surface Contamination Area, a step-off pad of absorbent paper or polyethylene shall be provided at the entrance of the barrier to the control point. The step-off pad may be on both sides of the barrier, however, the controlled side should be clearly marked from the uncontrolled side.

NOTE

The step-off pad shall be made by taping absorbent paper or polyethylene sheeting to the floor. The absorbent paper may be taped over the polyethylene where there is a possibility of wet contamination or where there will be heavy traffic through the control point. The pad shall be replaced whenever it is contaminated or badly worn.

4.3.1 If the general area contamination levels are greater than the gross limits (as specified by the plant technical manual), protective clothing shall be required as specified by the health physicist for all persons entering the area.

4.3.2 Laundry hampers for use (presumably contaminated) clothing shall be provided on the contaminated side of the barrier.

4.3.3 Clothing will be worn only once and then placed in laundry containers unless monitored and released.

4.3.4 A low-level beta-gamma and (if needed) alpha instrument shall be provided for personnel leaving the Surface Contamination Area so that they may monitor themselves to prevent the tracking of contamination into clean areas.

4.3.5 All personnel will check out on hand and foot monitor after cleaning up and before resuming normal duties in clean areas.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION

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The plant technical manual specifies the allowable surface contamination levels at control points before changing the step off pad. The plant technical manual also specifies frequency of survey of control points.

METHOD 722

PROCEDURES FOR PERSONNEL ACCESS CONTROL

1. SCOPE.

The method specifies the criteria and procedures for establishing control points for personnel access to the various designated areas within the nuclear facility.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 721, Paragraph 3.

4. PROCEDURES

4.1 Maintain personnel access control by establishing a series of control points through which people and material pass.

4.2 Establish permanent control points for areas where the radiation and contamination conditions are not subject to rapid change.

4.3 Locate permanent control points at the entrance to any of the controlled areas described in Methods 711 through 716 or at other areas where the Officer In Charge wishes to control personnel access for an extended period of time.

NOTE

The conditions which require the establishment of any particular Permanent Control Points shall

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be reviewed periodically by the Health Physicist and OIC, to insure that the control point criteria are satisfactory and meet the needs of the existing conditions in the controlled areas involved.

4.4 Make a thorough survey of the area before establishing a control point. This survey shall include air, smear, and instrument surveys as indicated in Methods 511, 512, and 513. After obtaining the results, the health physicist shall establish the criteria for passage through the control point. These entry criteria include types of clothing required, respiratory equipment needed, occupancy time limits set, and the personnel monitoring equipment required.

4.5 The control points shall provide only entrance through the barrier into the controlled area, and shall be posted with the appropriate signs (see AR 385-30). The health physicists shall perform a smear test on areas surrounding control points at regular intervals to assure contamination control.

4.6 Issue RWP's to indicate the proper precautions to be observed while work is being performed in a controlled area. The precautions to be observed shall be reviewed, written down, and posted at the control point of the area in question. The RWP's are covered in Method 731.

4.7 Personal clothing may be worn in areas having positively no surface

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or airborne contamination. Personal clothing may not be worn under protective clothing unless specifically authorized by the OIC. Personal clothing and valuables will be taken into controlled areas where protective clothing is worn at the owner's risk.

4.8 Wear protective clothing in all areas where there is a possibility that surface and airborne contamination might exist. The type of protective clothing shall be determined by the health physicist on the basis of the job to be done. The clothing shall be stored either at the clean side of the control point or at a designated change room.

NOTE

It is good practice to tape all openings of protective clothing. Tape coveralls and booties shut, sleeves to gloves, and the collar of coveralls snug around neck. Protective clothing will be worn only once and then placed in laundry hampers unless monitored and released.

4.9 In any area where airborne radioactive material may be present, the health physicist shall take an air sample to determine the airborne radioactivity levels. On the basis of this air sample, the health physicist shall determine the need for protective respiratory equipment before workers go into the area. Even if the activity level of the air sample is within specified limits, the equipment shall be conveniently available, because conditions could change rapidly.

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4.10 Personnel monitoring equipment shall be worn by all people entering a restricted area.

4.10.1 Film badges. Personnel shall have a beta-gamma film badge at all times while inside the restricted area. Neutron badges shall be provided for entry into an area where neutron exposure is possible.

4.10.2 Dosimeters. Personnel shall wear either self-reading dosimeters or non-self-reading dosimeters with the film badges in all radiation areas, or whenever the health physicist declares it. During operations in which large external doses are possible, a high-range dosimeter should be carried by each member of the work party.

4.10.3 Film badges and dosimeters should normally be worn together on the front part of the body between the waist and the shoulders. During special operations, the film badge and dosimeters should be worn together in a position that will record the greatest exposure to the body. In contaminated areas and on jobs that require a great deal of agility, the film badges and dosimeters should be securely attached to the individual and covered lightly to preclude loss and contamination.

4.11 Provide low-level beta gamma survey meters at the exit to the controlled area so that personnel, equipment, and clothing may be monitored before they leave the controlled area.

4.12 Provide airborne radioactivity monitors and samplers to take an

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air sample before operations begin. Air samplers shall be available to take samples periodically during work operations.

4.13 Tools and other equipment may be brought into a controlled area without restriction.

4.14 Provide a step-off pad at the Control Point as specified in Method 721, Paragraph 4.3.

4.15 Provide clothes hampers on the contaminated side of the barrier for the disposal of protective clothing.

4.16 Provide receptacles for contaminated waste.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The plant technical manual specifies the maximum allowable airborne radioactivity within a given area.

METHOD 723

PROCEDURES FOR PERSONNEL LEAVING A CONTROL POINT

1. SCOPE.

This method specifies the criteria and procedures to be observed for personnel leaving a control point for the various designated areas within the nuclear facility.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 721, Paragraph 3.

4. PROCEDURES.

4.1 Removal of Protective Clothing.

Remove Protective Clothing in such a manner as to prevent contamination of the skin or articles of clothing worn underneath. Individual techniques may be developed, but certain basic procedures shall be followed. Any special instructions shall be given by the health physicist. It is generally recommended that clothing be removed and discarded in the proper container in the following sequence:

- a. Outer gloves.
- b. Full faces masks.

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- c. Cap.
- d. Coveralls.
- e. Outer shoe covers.
- f. Inner gloves.
- g. Inner shoe covers.

The monitoring procedure shall begin as soon as the inner shoe cover has been removed and the individual has stepped onto the step-off pad.

4.2 Monitoring Procedures.

4.2.1 Control Point for Radiation Area.

4.2.1.1 All personnel shall read their dosimeters at the control point for a Radiation Area when leaving the area.

4.2.1.2 Personnel who spend extended periods of time in radiation areas should be constantly monitored to insure that Radiation Exposure Limits are not exceeded.

4.2.2 Control Point for High Radiation Area.

4.2.2.1 Personnel working in a High Radiation Area shall be periodically checked by health physicist to insure that Radiation Exposure Limits are not exceeded. The time between checks will depend on the radiation level.

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4.2.2.2 In areas where the radiation levels are extremely high or operations are of a nature that radiation levels could become extremely high, constant monitoring will be provided by a health physicist.

4.2.3 Control point for Surface Contamination Area or Airborne Radioactivity Area.

4.2.3.1 Monitor every item removed from a Surface Contamination or Airborne Radioactivity Area for beta-gamma contamination.

4.2.3.2 When an individual leaves a controlled area, he shall be monitored for beta gamma contamination by the following procedure: Remove protective clothing and step onto the step-off pad at the control point for the Surface Contamination or Airborne Radioactivity Areas. Here, the soles of the feet shall be monitored by a health physicist or an individual trained in personnel monitoring. If his feet are clean, the individual may step off the pad into the clean area and the monitoring shall be completed. If the feet or any other part of the body are found to be contaminated the individual shall put on rubbers or shoe covers and be taken to a personnel decontamination area for decontamination. (See Methods 321 through 324).

5. RESULTS AND COMPUTATIONS.

Not applicable.

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6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 731

ISSUANCE OF RADIATION WORK PERMIT

1. SCOPE.

1.1 This method specifies the procedures to be followed in issuing and terminating Radiation Work Permits (RWP).

1.2 Also included are the personnel monitoring and record-keeping requirements and regulations to be observed in the working area.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Portable high-and-low-range-beta-gamma instruments, alpha and neutron instruments, if needed.

Smears.

RWP Forms.

Protective clothing: coveralls, labcoats, head covers, gloves (cotton and rubber), shoe covers, and rubbers.

Full face masks.

Self-contained breathing apparatus.

Masking tape.

Absorbent paper.

Portable air sampler.

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Dosimeters.

Pencils.

4. PROCEDURES.

4.1 The two types of RWP's used in the ANPP are Special Radiation Work Permits and Routine Radiation Work Permits.

4.1.1 The purpose of the Special Radiation Work Permit is to assure that no work will commence on potentially contaminated equipment or in areas where radiation is present until each job has been properly evaluated from a radiological standpoint and approved by responsible personnel.

4.1.2 The Special Radiation Work Permit shall be initiated by the health physicist to authorized personnel for entry and work in a Radiation Area, High Radiation Area, Surface Contamination Area, and Airborne Radioactivity Area.

4.1.3 A Routine Radiation Work Permit shall be issued to cover all jobs of a repetitive nature where radiological conditions do not change.

4.2 The Health Physics procedures for filling out both Routine and Special Radiation Work Permits shall be as follows:

4.2.1 The RWP shall be requested by the job supervisor or any one of the group that will be performing the work.

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4.2.2 The Health Physicist shall monitor the work area for external and internal radiation hazards and establish time limits, set up protective clothing requirements, and designate the type of area to be set up.

4.2.3 The health physicist shall complete the RWP containing instructional procedures to be followed, clothes to be worn, and time limits to be observed.

4.2.4 A copy of the RWP will be issued and posted at the job site.

4.2.5 There shall be only one Special Radiation Work Permit for a specific job at any one time. The Special Radiation Work Permit will take precedent over the Routine Radiation Work Permit whenever there is a job in the same area and where there is a conflict between the two.

4.3 Special Radiation Work Permit.

4.3.1 The Special Radiation Work Permit shall be approved by the health physicist and the shift supervisor. If any question arises concerning operations in the plant, the OIC shall stipulate under what operating conditions the Special Radiation Work Permit shall be issued.

4.3.2 Every individual performing, observing, supervising or being in any way connected with the work performed under

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the permit shall sign his last name and initials to the permit, and note his film badge number and dosimeter serial number. The reading of the self-reading dosimeter upon entry into and return from a work permit area shall be noted as well as the time in and out. Total time and estimated dosages received shall be noted. A signature on the RWP designates understanding of the permit and compliance with instructions.

4.3.3 Each Special Radiation Work permit shall be prepared for one specific job and shall remain valid for periods not exceeding 24 hours. It shall not be used on a second job, even if the second job is identical to the first job covered on the permit. A new Special Radiation Work Permit may be issued for the same work at the discretion of the health physicist and OIC.

4.3.4 If the conditions under which a Special Radiation Work Permit was issued change considerably for the worse during the work, the RWP shall be terminated by the supervisor or the health physicist.

4.4 Routine Radiation Work Permit.

4.4.1 The Routine Radiation Work Permit is issued for jobs of a repetitive nature. It shall be valid for an extended period of time. It shall require approval by the health physicist, OIC, and the supervisor of the section doing the work. The same conditions as

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specified in Paragraph 4.3.2 shall be met for a Routine Radiation Work Permit.

4.4.2 The Routine Radiation Work Permit shall not be written for any operation in which conditions may suddenly change for the worse, even though the job is one of a repetitive nature.

4.5 Posting Requirements.

4.5.1 Post the RWP's at the job site control point until the completion of the job. The permit shall be placed in a conspicuous place where it may be seen easily.

4.6 Entries made on an RWP involved by personnel.

4.6.1 The only entries made on RWP's with the exception of the time record section, shall be those made by the individual originating the RWP, the health physicist and the authorizing shift supervisor.

4.7 RWP Termination.

4.7.1 Anyone may terminate a work permit by checking with the working party for completion of the job and ascertaining that all participants have signed off on the permit prior to signing this section and dating it. When a permit is terminated, entry will be made in the Station Log. The permit shall not be used for another job.

4.7.2 The Radiation Work Permit will be terminated during the job by the health physicist, supervisor or OIC, if conditions

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under which it was issued change sufficiently.

4.8 The health physicist shall always be present during operations he believes to be radiologically hazardous. Even when his continuous presence is not required, he shall check the job periodically to be certain that conditions of the job have not changed and that no new hazards have developed.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 741

VAPOR CONTAINER ENTRY

1. SCOPE.

This method specifies the procedures to be followed in entering a vapor container. The procedures also specify the equipment to be used and the surveys to be taken. The surveys include Radiation Surveys, Smear Surveys, Airborne Radioactivity Surveys, and Visual Surveys.

2. SAMPLE.

Vapor Container or Primary Building Air.

3. APPARATUS.

Protective clothing: coveralls, headcovers, hoods, shoe covers, (rubber or plastic), gloves (cotton and rubber).

Respiratory Equipment - full face masks, self-contained breathing apparatus.

High-volume air sampler.

High and low-range beta-gamma survey instruments and neutron instruments, if needed.

Clipboard with schematic diagrams of vapor container.

Film badges and beta-gamma and neutron film packets.

Dosimeters - beta-gamma (0-200 mr).

Pencils and pencil crayons.

4. PROCEDURES.

4.1 Using a constant air monitor determine gross level of airborne activity in vapor container. This will decide whether self-contained breathing apparatus or full face masks will be worn for initial entry. If no air monitor is available, self-contained breathing apparatus is required.

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- 4.2 Set up a control point outside the vapor container entrance,
using the procedures as specified in Method 722.
- 4.3 The person making the initial entry shall wear protective clothing
as specified by the health physicist. It is recommended that full
protective clothing be worn for initial entry. Beta-gamma and neutron film
badges and a self-reading dosimeter shall be worn.
- 4.4 Respiratory protective equipment shall be worn until an air
sample is taken and evaluated.
- 4.5 After the reactor has been shut down for a minimum of 15 minutes
and the vapor container has been opened, two health physicists
shall enter the compartment first. Using a radiation survey instrument, they
shall ascertain the radiation level at the entrance to the container.
- 4.6 When the vapor container is too small for more than one person
to make the entry, the second health physicist shall stand by at
the entrance in case he is needed.
- 4.7 Upon ascertaining the general radiation levels, the health physicist
will be able to judge their time limit in the container and dose they
will receive.
- 4.8 One of the health physicists shall then proceed to start an air
sample in the vapor container. The air sampler will be located
in the area where the major work is to be done. The air sample shall be

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taken with a high volume air sampler for approximately 15 minutes.

The samples shall be counted by a health physicist.

4.9 The health physicist shall make a thorough instrument survey with a high and low-range beta-gamma instrument. He shall record all readings on a schematic or data sheet. He shall concentrate on the areas of future major work.

NOTE

Available neutron instruments shall be used to check for neutrons on initial entry.

4.10 The last survey the health physicist shall take will be a general smear survey of the vapor container. If extended maintenance is to be performed on any single piece of equipment, it shall be thoroughly smear surveyed. The smears will give an indication of the contamination level and whether or not protective respiratory equipment may be needed.

4.11 The health physicist shall be thoroughly acquainted with the vapor container, and while making his other surveys, he should be constantly looking for any unusual conditions in the operating equipment of the vapor container.

4.12 When the health physicist has completed his surveys, he shall proceed to the control point, where he shall follow the general procedures for leaving a control point as outlined in Method 723.

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4.13 He shall then post the radiation readings of the vapor container on duplicate schematics or data sheets just outside the entrance to the control point. These schematics or data sheets shall be large and easy to follow.

4.14 A continuous watch shall be posted at the control point when personnel are inside the vapor container.

4.15 If the vapor container is to be open for several days, the container and the area around the container shall be monitored at least 4 hours after the original survey, 30 hours after the original survey and every 24 hours thereafter. In buildings where the vapor container is only a tank in the building, the building shall be surveyed on the same schedule.

4.16 The health physicist shall post instructions for entrance into the vapor container immediately after the initial survey air sample has been counted. These instructions shall include time limits, protective respiratory requirements (if any), and temporary shielding placement (if any). Temporary shielding shall always be installed under the direction of a health physicist using a survey instrument.

4.17 The health physicist shall be present during the performance of radiologically hazardous jobs in the vapor container.

5. RESULTS AND COMPUTATIONS.

Not applicable.

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Process Control

6. TEST METHOD IMPLEMENTATION.

The plant technical manual specifies the shutdown procedures for the reactor.

This should include the following:

- a. The time of entry after shutdown.
- b. Coordination of the entry with the control room to eliminate any possibility of the reactor going critical.
- c. Assure that the ventilation air through the vapor container is started immediately after shutdown.

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Process Control

SECTION 800 - RADIATION AND CONTAMINATION CONTROL

Radiation work must be performed in a safe manner. Safety practices should be observed regarding food handling, check of personnel activity, and waste disposal. The objective is that every worker follow the necessary procedures for his own protection, and for the protection of others. In a radioisotope laboratory, skill in radiation protection is as necessary as skill in chemical or biological manipulations. Safety procedures and special equipment descriptions are presented on the following pages.

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METHOD 811

WORK IN VAPOR CONTAINER AND ON PRIMARY SYSTEMS

1. SCOPE.

The purpose of this method is to describe the health physics requirements for working in the vapor container and on primary systems.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Full protective clothing.

Lab coats.

Full face masks.

Self-contained breathing apparatus.

Polyethylene sheeting.

Absorbent blotting paper.

Air particulate sampler - portable.

Smears.

Beta-gamma survey meters, high- and low-range.

Vacuum cleaner with filter AEC approved 99.9% efficient for 0.3 micron D.O.P. particle, attached to exhaust.

4. PROCEDURES.

4.1 Work in Vapor Container.

4.1.1 Make entry into the vapor container according to procedures described in Method 741.

4.1.2 Follow the same general procedures specified in Paragraph 4.2 for work on systems other than primary systems in the vapor container.

4.2 Work on Primary Systems.

4.2.1 Obtain an RWP (see Method 731).

4.2.2 Set up a Control Point as specified in Method 722.

4.2.3 Change to protective clothing in accordance with Method 821, with appropriate respiratory protective equipment, if required.

4.2.4 Obtain all tools necessary to perform the desired work and to control contamination. Handle all tools and material as though contaminated when brought past the control point into the work area.

4.2.5 Read the dosimeter before entering the controlled area.

4.2.6 A health physicist shall make a careful radiation survey of the working area and recommend any additional safety measures.

4.2.7 A health physicist shall periodically monitor the area for radiological hazards and revise the RWP when necessary.

4.2.8 While in the work area, all persons must observe the radiation and contamination safety measures as follows:

4.2.8.1 Welding, grinding, burning, chipping, and opening of the Reactor Primary Systems require that respiratory protective equipment be worn.

NOTE

The health physicist should be certain that all lines containing radioactive materials have been depressurized and drained before initiating work.

4.2.8.2 Venting of main coolant pumps also requires that respiratory protective equipment be worn.

4.2.8.3 Cover all openings made in the Reactor Coolant System. For example, if the primary pump is dismantled, the pump volute should be blanked off with a blind flange.

4.2.8.4 Avoid the spread of contamination by vacuuming the area, if possible, and by using absorbent paper and polyethylene sheeting.

4.2.8.5 Vacuum, if possible, the parts to be removed and wrap them in polyethylene.

4.2.9 General safety rules for maintenance people while in work area are as follows:

4.2.9.1 Do not smoke or touch hands to face.

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- 4.2.9.2 Consult health physicist, as applicable,
before moving beyond the work area.
- 4.2.9.3 Keep respiratory protective equipment
available at all times.
- 4.2.9.4 Enter the controlled area and perform
operations without undue delay.

NOTE

If any unusual conditions occur, vacate
the area immediately.

- 4.2.10 Leaving the control point shall be by procedures
specified in Method 723.

- 4.2.11 The health physicist shall assist in hazardous
operations where radioactive materials are being
handled.

- 4.2.12 Hazardous jobs should be reviewed with the operating
crew. In many instances, a dry run will enable the
operations chief and the health physicist to gain an idea of the
personnel required in relation to the time and dose rates of the
particular job.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The plant technical manual covers extensive
maintenance work on the primary systems.

METHOD 812

WORK ON RADIOACTIVE WASTE DISPOSAL SYSTEM
AND SPENT FUEL PIT

1. SCOPE.

The purpose of this method is to describe the health physics techniques and procedures necessary to work on the radioactive waste disposal system (RWDS) and the spent fuel pit.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Same as Method 811, Paragraph 3.

4. PROCEDURES.

4.1 Work on RWDS.

4.1.1 Obtain an RWP for maintenance on the radioactive waste disposal system.

4.1.2 Follow applicable procedures as outlined in Method 811.

4.1.3 Notify the shift supervisor before performing maintenance work on the system.

4.2 Transfer of Fuel from Core to Spent Fuel Pit.

4.2.1 Follow applicable procedures of Method 811.

4.2.2 The mechanics of the fuel transfer will be established by the plant technical manual.

4.2.3 Provide polyethylene sheeting and tape for packaging handling tools for movement.

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4.2.4 Decontaminate tools according to Methods 311 through 318.

4.3 Removal of Fuel Cask from Spent Fuel Pit.

4.3.1 Survey the cask with a portable radiation gamma survey instrument as it is being raised from the pit.

4.3.2 Permit excess water to drain from the cask surfaces into the pit, then wipe the cask dry with an absorbent cloth.

4.3.3 Provide a pad of absorbent paper to place cask on.

4.3.4 Thoroughly wipe down cask. Use procedure as outlined in Method 354.

4.3.5 Perform a thorough smear survey on the cask in accordance with Method 512.

4.3.6 If still contaminated above levels as set by the plant technical manual decontaminate until acceptable limits are met.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Surface contamination limits of the spent fuel cask are established by the plant technical manual.

METHOD 813

PRIMARY COOLANT SAMPLING FOR RADIOCHEMICAL ANALYSIS

1. SCOPE.

The purpose of this method is to describe the techniques used in taking primary coolant samples for radiochemical analysis.

2. SAMPLE.

Primary coolant samples for radiochemical analysis.

3. APPARATUS.

Same as Method 811, Paragraph 3.

4. PROCEDURES.

4.1 Notify the shift supervisor before taking a primary coolant sample.

4.2 A routine RWP is required for routine sampling at the sampling station.

NOTE

See Method 731 for detailed instructions concerning RWP Procedures.

4.3 Establish a controlled area around the sampling station if not already in a controlled area.

4.4 Wear protective clothing, including shoe covers, rubber gloves and protective respiratory equipment.

4.5 Provide particulate and gaseous air samplers, as applicable, in accordance with Method 513.

- 4.6 Take primary coolant samples in accordance with normal procedures.
 - 4.7 If the sample is to be removed from the controlled area, place collected sample container in a double polyethylene bag.
 - 4.8 Monitor sample with a portable beta-gamma survey meter to establish radiation levels.
 - 4.9 Place the primary coolant sample in a carrying container.
 - 4.10 Turn off the air sampler and count air samples, using techniques specified in Method 631.2.
 - 4.11 Smear survey controlled area including sampling station.
 - 4.12 If the controlled area count is lower than limits established by the plant technical manual, then remove area controls.
 - 4.13 If the controlled area is contaminated, decontaminate it, using procedures specified in Methods 351 through 355.
5. RESULTS AND COMPUTATIONS.
Not applicable.
 6. TEST METHOD IMPLEMENTATION.
Not applicable.

METHOD 821

PROTECTIVE CLOTHING FOR HANDLING RADIOACTIVE MATERIALS

1. SCOPE.

The purpose of this method is to describe the types, purposes, and criteria for use of the various kinds of protective clothing worn when handling radioactive materials.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Shoe covers.

Gloves.

Head covers.

Lab coats.

Coveralls.

4. PROCEDURES.

4.1 Shoe Covers.

4.1.1 Shoe covers consist of the following types:

- a. Cotton shoe covers that just cover the shoes.
- b. Vinyl shoe covers that just cover the shoes (rubbers).
- c. Cotton shoe covers approximately 13-17 inches high.
- d. Vinyl shoe covers 13-19 inches high that will have elastic or tie tops.

e. Regular rubber boots.

4.1.2 The purpose of shoe covers is to provide protection to the shoes and feet from loose radioactive materials, both wet and dry.

4.1.3 The shoe covers are used in all areas where loose radioactive material is present. Whenever there is a possibility of the surface contamination being wet, vinyl shoe covers shall be worn, or the cotton shall be covered with polyethylene bags. Shoe covers which are torn or ripped shall be discarded.

4.2 Gloves.

4.2.1 Gloves consist of the following types:

- a. Cotton.
- b. Latex (surgeons gloves).
- c. Heavy rubber process gloves.

4.2.2 The purpose of protective gloves is to protect the hands against wet and dry loose radioactive contamination. Surgeon's gloves and the heavy rubber gloves are also used to protect the hands against chemicals that may irritate the skin.

4.2.3 Protective gloves are worn whenever the possibility of contamination from loose radioactive materials exists.

4.3 Headcovers.

4.3.1 Head covers consist of the following:

- a. Surgeon's cloth cap.

b. Hood that is made from either paper, cotton, urethane, or a vinyl plastic.

4.3.2 The purpose of the surgeon's cap is to protect the hair and the top of the head from radioactive materials. The hood gives additional protection to the back of the neck and the ears.

4.3.3 Surgeon's caps are used to prevent airborne transfer from contamination objects. Paper and cotton should never be used where there is a possibility of liquid radioactive contamination. Where a wet airborne contaminant is present, vinyl or rubberized hoods should be worn.

4.4 Lab Coats.

4.4.1 The two types of lab coats generally used are as follows:

- a. Cotton.
- b. Vinyl plastic.

4.4.2 The purpose of the lab coat is to protect personnel clothing against water, chemicals, acids, and radioactive materials.

4.4.3 The lab coat is used primarily in the radiochemistry laboratory, but may be used in other areas where extensive personnel protective clothing is not required. The cotton lab coat may be used for wet operations, provided a latex or plastic apron is worn to cover the front of the lab coat.

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4.5 Coveralls.

4.5.1 The three types of coveralls are as follows:

- a. Paper.
- b. Cotton.
- c. Latex or vinyl ("acid suit").

4.5.2 The purpose of the protective coveralls is to protect the body of personnel against loose radioactive materials.

4.5.3 The treated paper suit of protective clothing is used only for dry operations. The cotton protective coveralls is the one most frequently utilized in nuclear facilities. The latex or vinyl suit should only be used for wet operations where the body may be contaminated extensively with radioactive or other toxic materials.

4.6 General Instruction for Protective Clothing.

4.6.1 Ripped or torn protective clothing should be repaired immediately, or if badly damaged, they should be discarded.

4.6.2 Latex or vinyl suits should only be worn when absolutely necessary. When the ambient temperature goes above 80° F and an individual is wearing a latex or vinyl suit, the health physicist should constantly check the individual for heat prostration, dizziness, and weakness. Water intake should be increased

to compensate for fluid losses. Also, frequent rest periods should be provided. Additional salt tablets need not be taken unless the suit is used frequently for long periods of time.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The plant technical manual specifies the type of protective clothing to be worn when working at various concentrations of airborne radioactivity.

METHOD 831

GENERAL REQUIREMENTS FOR ALL RESPIRATORY EQUIPMENT

1. SCOPE.

The purpose of this method is to describe the general requirements applicable to all respiratory equipment.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Respirators.

Self-contained breathing apparatus.

Goggles.

Full face masks, M17, M9A1, Mark IV.

Air supplied masks.

Compressor.

Gas cylinders.

Air supplied suits.

4. PROCEDURES.

4.1 The best facial fit of respiratory equipment is obtained with the maximum amount of flat rubber surface lying against the skin area.

4.2 A second factor in facial fit is the action of the head harness, which should accomplish the following:

a. It should stay adjusted.

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Process Control

- b. The harness buckle should not cut into the harness.
 - c. It should be so placed that it can be used to adjust the facepiece portion.
 - d. Adjustment should be tight enough to assure a comfortable fit.
- 4.3 Personnel using respiratory equipment should be constantly drilled in its use.
- 4.4 Special fitted glasses should be provided for people who need glasses and have to wear a mask.
- 4.5 Openings in the facepiece require special attention to insure that the seal around these openings are leak-tight.
- 4.6 Full face masks, when used at very low temperatures (-30° F) give poor visibility and exhibit freezing of exhalation valves. Anti-fog compounds and nose cups inserted in the face will reduce these problems.
- 4.7 Wear respiratory protective equipment that is designed specifically for the toxic material to which an individual will be exposed.
- 4.8 Respiratory protective equipment is a temporary measure to be used only until the source of the toxic material can be eliminated.
- 4.9 The necessity for good maintenance and monitoring of equipment is of primary importance in keeping protective respiratory equipment in good condition and available for immediate use.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Use an airline mask or self-contained breathing apparatus when welding brass, bronze, or galvanized iron in confined spaces.

When cutting iron or steel coated with lead or paint containing lead, always wear a suitable airline mask. No operation of this type should be considered free from the effect of lead fumes. Positive protection against breathing lead fumes that occur when lead is heated to vaporization temperature is mandatory.

METHOD 832

THE RESPIRATOR

1. SCOPE.

The purpose of this section is to describe the use of the respirator.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Respirator: facepiece assembly, filter cartridge holder, and filter.

Goggles.

4. PROCEDURES.

4.1 Filters should be specific for the material to be filtered.

A respirator that is specified for radioactive materials should not be used for chlorine gas unless so stated in the Manufacturers' Literature.

4.2 Wear goggles for eye protection when wearing a respirator.

4.3 Wear respirators when working with non-radioactive and non-toxic dusts.

4.4 Adjust respirator so that it fits close, but not extremely tight. The respirator must fit the head and face so that it will be air tight when properly donned.

4.5 Check respirator, using the following procedure:

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4.5.1 To clear, close the outlet valve by cupping the hand
firmly over the opening. Blow hard to clear facepiece.

4.5.2 To test for leaks, shut off air supply by placing
hands over air inlets. Inhale. No air should enter,

and the facepiece should collapse against the face.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 833

FULL FACE MASK

1. SCOPE.

The purpose of this method is to describe the use of the full face mask.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Full face mask.

4. PROCEDURES.

4.1 Use the full face mask in low-level airborne radioactivity areas. The M17, M9A1, and the Navy Mark V are used in the ANPP.

4.2 When using other types of commercial masks, the limitations and specifications of the filter cartridge should be known.

4.3 Put on the full face mask, using procedures given in FM21-41 Department of the Army Field Manual "Soldiers Handbook for Chemical and Biological Operations and Nuclear Warfare".

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 834

THE SELF-CONTAINED BREATHING APPARATUS

1. SCOPE.

The purpose of this method is to describe the use and limitations of self-contained breathing apparatus.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Self-contained breathing apparatus.

Compressed air supply.

Re-charging equipment.

4. PROCEDURES.

4.1 The self-contained breathing apparatus is a full faced, self-contained air supply mask, equipped with a compressed air cylinder. The cylinder contains approximately a 30-minute air supply under normal conditions. The air supply is reduced considerably under heavy working conditions and accelerated breathing.

NOTE

The health physicist should know the limitations of all air cylinders at his installation.

4.2 The self-contained breathing apparatus is used in the following circumstances:

- a. Insufficient oxygen in the air.
 - b. Chlorine or some other toxic gas present.
 - c. High concentration of radioactive dust, vapor, or gases.
- 4.3 The self-contained breathing apparatus should be used under the direction of the health physicist.

NOTE

Sufficient instruction should be given and drills conducted so that all personnel who may be required to use the self-contained breathing apparatus will be familiar with its operation and capable of using it promptly.

- 4.4 Follow the manufacturer's instructions for use of the equipment.
- 4.5 Store all self-contained breathing apparatus with full air cylinders. All air cylinders whose regulators indicate less than capacity shall be recharged to capacity, using the breathing air recharging equipment.
- 4.6 Locate the recharging unit in a clean area. The compressor shall be equipped with adequate pre-filters so that air coming into the storage cylinders will be clean.

NOTE

Use Air Only! Do Not Use Oxygen!

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Process Control

4.7 Decontaminate apparatus, using procedures in Method 341.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 835

THE AIR LINE MASK

1. SCOPE.

The purpose of this method is to describe the techniques employed in the use of the air line mask.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Air line mask.

a. Full face mask.

b. Regulator.

c. Egress cylinder.

d. Connecting line with quick-disconnect fitting to air cylinder or compressor.

4. PROCEDURES.

4.1 When entering or leaving a contaminated area, the wearer uses the egress cylinder containing a 5-minute air supply.

The unit is automatically replenished when the unit is connected to the main air supply. The air supply or compressor is never moved into the contaminated area.

4.2 Air line masks work on the same principle as the self-contained breathing apparatus except that the source of air is not carried with the individual.

NOTE

The advantage of the air linemask is that it allows an individual to work longer in a contaminated area than he could with self-contained breathing apparatus. The disadvantages of the air linemask are as follows:

- a. The range of movement is limited to the length of the supply hose connected to the mask.
- b. The supply hose makes supplied air masks difficult and clumsy to work with in confined spaces.

4.3 Follow the same principles of using, wearing, storing, and cleaning as are followed for self-contained breathing apparatus. (Method 834.)

CAUTION

Any individual having a known physical impairment (asthma, severe head cold) that would be aggravated by using the equipment shall be prohibited from wearing the equipment.

4.4 Periodic drills to familiarize personnel using the air line mask shall be conducted by the health physicist.

4.5 Compressed air from plant compressors, from special breathing air lines, or from bottled tanks and cylinders under high pressure may be used.

NOTE

Air compressors not specifically designed for supplying respirable air should not be used unless they have specifically been approved for the purpose. Also, be sure that bottled gas is pure. The supply lines shall have traps and drains at proper places to assure that the air is clear and free from excessive water and oil. The respirable air bottles shall be distinctly marked.

4.6 If the pressure of the air supply exceeds 30 psi, a pressure regulator must be provided to prevent the pressure to the air line hose from exceeding 30 to 36 psi. The regulator shall be adjusted to give the following pressures to the mask:

<u>Hose length, ft</u>	<u>Pressure at hose, psi</u>
15-50	8-17
50-250	17-30

NOTE

Two distinct types of regulators
are used:

- a. The air demand regulator admits air to the mask when the wearer inhales.
- b. The pressure demand regulator maintains a slight constant positive pressure on the mask to prevent contaminated air from leaking in.

4.7 The air supply to the mask shall be not less than 2 cfm of air at the minimum recommended pressure and maximum length of hose.

4.8 Respirable air supplies shall be from a source of air free from contamination from radioactive materials and other toxic materials.

4.8.1 The air shall contain the normal percentage of oxygen (21%), carbon monoxide (0%), and carbon dioxide (0.03%).

4.8.2 The air shall be pure. This may be insured by inspecting the air around the compressor intake for radioactive materials; gasoline and diesel engine exhausts of all types; smokes and gases from exhaust stacks; and gases, dusts, and vapors from any chemicals that would adulterate the air supply.

Compressed air sometimes carries fumes from the oil necessary to lubricate the compressor valves. Therefore, an air line filter is necessary to remove all these foreign materials from the air supply. Care in the operation and maintenance of the air purifier and the compressor is necessary, and a separate motor driven blower should be used with the device.

NOTE

As a safety precaution, station an individual at the source of air supply when the air supplied masks are being used. Keep the air supply cylinder and compressor out of the contaminated atmosphere.

4.9 Establish simple positive signals between individuals using the supplied air respirators and individuals in attendance at the air source so that a message indicating impending or actual trouble can be conveyed from one end of the air line to the other.

4.10 Decontaminate air line masks, using procedures specified in Method 341.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

METHOD 836

THE OXYGEN BREATHING APPARATUS

1. SCOPE.

The purpose of this method is to describe the techniques employed in the use of the oxygen breathing apparatus (OBA).

2. SAMPLE.

Not applicable.

3. APPARATUS.

Full face mask with chemical canister.

4. PROCEDURES.

4.1 Do not use canisters if the seal has been broken.

4.2 Make sure canisters are capable of generating oxygen before entering the contaminated area.

4.3 If the OBA is equipped with lungs, do not allow the lungs to collapse while in a contaminated atmosphere. If this happens, leave the contaminated area immediately. Short, quick, panting breaths should reinflate the lungs and allow escape from the area.

4.4 The time limitation of the chemical canister shall be known by all personnel using the canister and the time shall be under the control of the health physicist.

4.5 The health physicist shall remove oxygen breathing apparatus 5 minutes before the estimated time of depletion of the chemical canister.

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- 4.6 Dispose of used canisters immediately, in accordance with manufacturer's instructions.
- 4.7 Provide a readily available supply of spare chemical canisters.
- 4.8 Store oxygen breathing apparatus with fresh unused canisters.
- 4.9 Decontaminate the oxygen breathing apparatus, using procedures as outlined in Method 341.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

The operational limits for airborne radioactivity shall not exceed 10% of the amount specified in Appendix B, Table 1, Column 1, of 10 CFR 20 or MPC derived therefrom. Self-contained breathing apparatus shall be worn when the 10% figure is exceeded by a figure of 50.

METHOD 841

TEMPORARY GAMMA SHIELDING

1. SCOPE.

The purpose of this method is to describe temporary gamma shielding. The shielding discussed in this method and in Methods 843 and 844 will be primarily that required for maintenance and other short term operations.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Not applicable.

4. PROCEDURES.

4.1 Gamma rays interact with shielding in three ways; photoelectric effect, Compton recoil, and pair production, depending on the energy level of the photon.

4.2 Gamma radiation will be effectively attenuated by high-density materials. Lead and steel are used predominately; however, large thicknesses of light materials, water or plastic, also augment the attenuation of gammas.

4.3 The interaction of gamma radiation with high-density material will result in a loss of energy to the gamma radiation. The attenuation of the gamma rays is a function of the energy of the radiation and the thickness of the shielding material.

4.4 Methods are given in Results and Computations to find the thickness of different shielding materials, which will reduce the gamma radiation by a factor of one half (half-value layer) and also a factor of ten (tenth-value layer).

4.5 Examples of gamma shielding to be used by the health physicist are lead sheet, lead brick, and lead shot. Steel brick and depleted uranium may also be used in place of lead brick.

4.6 The following general principles for placing shielding will apply to all operations.

4.6.1 Monitor the radiation levels of the gamma radiation by taking a radiation survey described in Method 511.

4.6.2 Attempt to establish the predominate gamma emitting isotope to facilitate shielding computations.

NOTE

When shut down and working on primary systems, assume Cobalt-60 to be the predominate isotope.

4.6.3 When using lead shielding, estimate the area to be shielded. Make allowance for all irregularities (if any).

4.6.4 Pre-cut the sheet to the required configuration and thickness.

NOTE

Thickness may be either computed or found in the graphs which are given in Part 5.

- 4.6.5 Place sheets in designated location.
- 4.6.6 If sheets are to be placed around piping, hold them in place by means of straps or bands.
- 4.6.7 Survey after placing sheets to ascertain if the desired radiation level has been attained.
- 4.6.8 Use lead shot in bags to shield the valves & fittings.
- 4.6.9 The health physicist shall supervise placement of lead shot.

NOTE

The attenuation factor using lead shot will be approximately 60% of that used for lead shielding.

- 4.6.10 Use lead and steel brick to build semi-permanent shields.

NOTE

The brick should be placed on a sturdy base, because the average brick weighs 25 pounds and the use of many bricks



will introduce a stress problem on
a flimsy base.

4.6.11 In some instances, molten lead may have to be
poured into crevices to prevent radiation streaming.
This operation should always be done under the guidance of a health
physicist. Proper respiratory equipment shall be provided as
personnel protection from airborne lead fumes.

5. RESULTS AND COMPUTATIONS.

5.1 Attenuation of Gamma Radiation by Calculation.

5.1.1 Attenuation of a monoenergetic beam of gamma
radiation may be calculated by the following formula:

$$I = I_0 e^{-ux} \quad (841-1)$$

Where I = Intensity of beam after shielding.

e = Base of natural logarithm (2.718).

I_0 = Intensity of beam before shielding.

u = Linear absorption coefficient, cm^{-1} .

x = Thickness of shielding material, cm .

5.1.2 $x_{1/2}$ is defined as the thickness (cm) of shielding
material required to attenuate the intensity of
gamma radiation by a factor of two.

$$I/I_0 = e^{-ux_{1/2}} \quad (841-2)$$

$$\ln I_0/I = -ux_{1/2}$$

When $I_0 = 2I$

$$\ln 2 = 0.693 = \mu x_{1/2}$$

$$x_{1/2} = 0.693/\mu$$

5.1.3 The value of μ , the linear absorption coefficient for various shielding materials as a function of gamma energy ranges is given in Table 841-1. This value of μ is an approximation and does not include the buildup factor.

5.1.4 The formula in Paragraph 5.1.1 may be used to estimate values other than the half value layer if a natural logarithm table is available.

5.2 Graphical Solutions of Attenuation Equation.

5.2.1 Figure 841-1 gives inches of shielding material necessary to attenuate the gamma intensity by a factor of one half. Thus one half-value layer of shielding material would reduce the dose rate to 1/2 of the original dose rate, two half-value layers would reduce the dose rate to 1/4 of the original dose rate, three half-value layers would reduce the dose rate to 1/8, and seven half-value layers would reduce the dose rate to 1/128 of the original dose rate.

5.2.2 Figure 841-2 gives the inches of shielding material necessary to attenuate the gamma intensity by a factor of 10. One tenth-value layer of shielding material would reduce the dose rate to 1/10 of the original dose rate. Two tenth-

Table 841-1. Linear Absorption Coefficients (u) for Various Shielding Materials as a Function of Gamma Energy

Energy, MeV	Lead, u(cm)	Iron, u(cm)	Aluminum, u(cm)	Water, u(cm)	Air $\times 10^{-3}$, u(cm)	Concrete, u(cm)
0.3	4.32	0.864	0.282	0.119	0.135	0.251
0.5	1.75	.652	.236	.092	.111	.204
1.0	0.85	.471	.154	.071	.082	.149
1.3	.69	.415	.148	.063	.073	.132
1.5	.59	.378	.137	.057	.067	.121
2.0	.51	.334	.112	.050	.056	.104
2.5	.48	.302	.102	.044	.050	.095
3.0	.47	.279	.094	.039	.046	.085
3.5	0.46	.268	.087	.036	.042	.080
4.0	*	.251	.083	.034	.039	.074
5.0	--	.243	.072	.030	.034	.067
10.0	--	0.235	0.059	0.023	0.028	0.050

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*Value beyond which the linear absorption coefficient will not decrease regardless of increase in energy.

value layers of shielding material will reduce the original dose rate by a factor of 100. Figure 841-2 will be more applicable for shielding large dose rates.

EXAMPLE

Reduce the dose rate from a 1000 mr/hr
Co-60 source by a factor of 20, using
lead as the shielding material.

Co-60 = 1.3 Mev gamma ray energy.

Solution: Read on Figure 841-2 the
value of the ordinate at the point where
the 1.3-Mev abscissa intersects the curved
line representing lead. It will take
approximately 1.5 inches of lead to
reduce the dose rate by a factor of ten.

To reduce the dose rate by a factor of 20,
find the half value layer on Figure 841-2 .
This is approximately 0.4 inch. Therefore,

1/10-value layer = 1.5 inches

1/2-value layer = 0.4 inch

TOTAL 1.9 inches

It will take approximately 1.9 inches of
lead to reduce a 1000-mr/hr Co-60 source
by a factor of 20 or to 50 mr/hr.

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6. TEST METHOD IMPLEMENTATION.

See Reference 23 in the Reference Section for regulations of
personnel exposure.

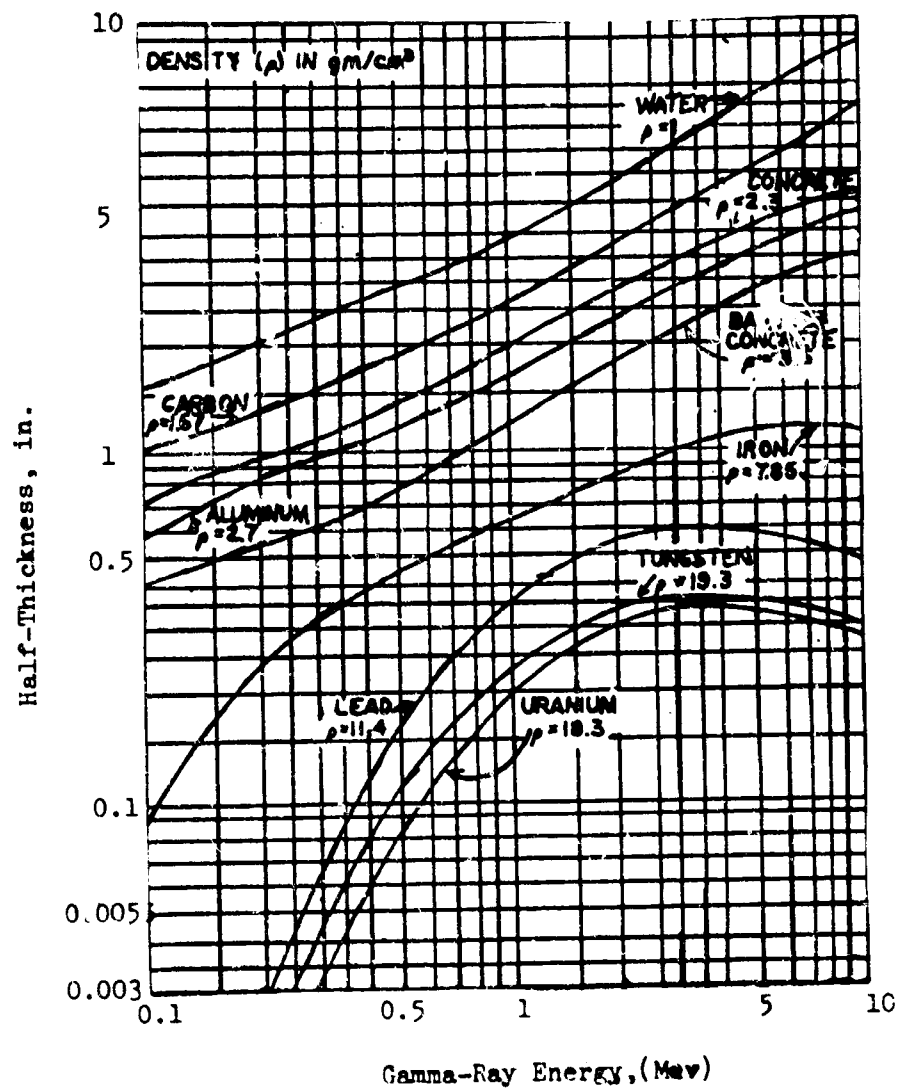


Figure 841-1. Half Thickness of Various Materials Versus Energy

Taken from "Basic Nuclear Physics" NAVPERS 10786 published by
Bureau of Naval Personnel.

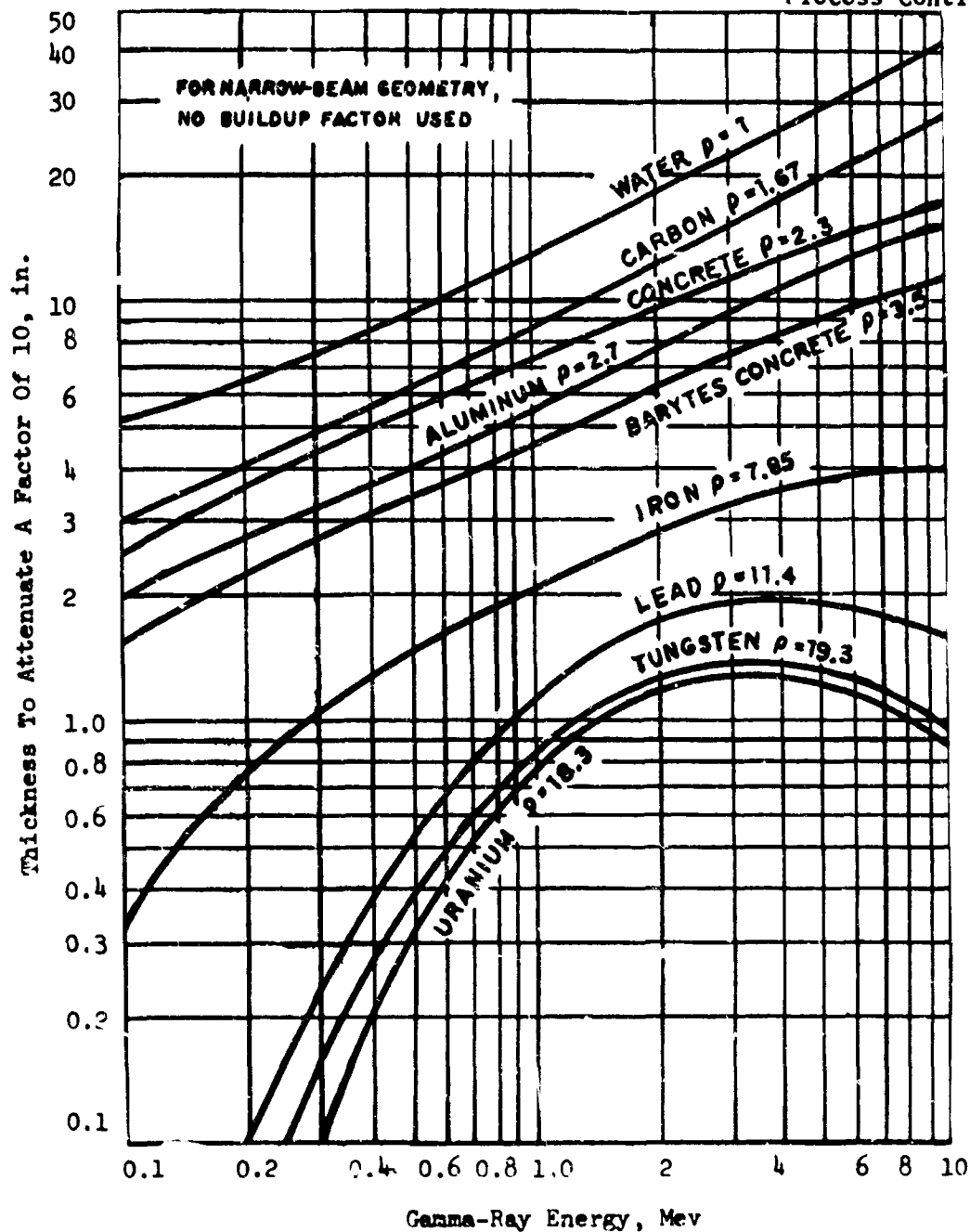


Figure 841-2 . Tenth-Value Thickness for Gamma-Ray Absorption

Taken from "Principles of Radiation and Contamination Control
Volume 2, Procedures and Guidelines Relating to Nuclear Weapons
Effects" NAVSHIPS 250-341-3 Bureau of Ships, Navy Department,
Washington 25, D.C.

METHOD 842

TEMPORARY NEUTRON SHIELDING

1. SCOPE.

The purpose of this method is to describe the need for temporary neutron shielding in the nuclear power plant.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Not applicable.

4. PROCEDURES.

4.1 Temporary shielding for neutrons in a nuclear power plant constitutes less of a problem than similar shielding for gamma rays because of the following reasons:

4.1.1 The original power plant was designed to contain the neutron flux from the core. However, there may be neutron streaming through cracks and fissures in the secondary shielding when the reactor is operating.

4.1.2 At a maximum of 5 minutes after shutdown (this contains an adequate safety factor), no neutrons should be coming from the core.

NOTE

Beryllium components, if present in the core, will continue to emit photo-

neutrons after the reactor is
shut down.

4.1.3 Another accessible source of neutrons in a nuclear power plant would be from a calibration source used for calibrating the various neutron survey meters and possibly startup sources.

4.2 Temporary shielding is required for isolated streaming through secondary shielding and for additional shield penetrations.

4.3 Neutrons are most effectively attenuated by material of low atomic number. Hydrogenous material, such as water and most organic compounds, are useful in this respect. Heavy plastic sheets may be used when liquid neutron shielding is not feasible. Polyethylene borated to about 3% boron is the plastic most commonly used, but other plastics or elastomers, wood, concrete, and paraffin may be used.

NOTE

Low atomic number materials are usually backed up by high cross-sectional materials that, in turn, are followed by high-density materials for complete neutron shielding.

Neutron shielding may also come in other combinations and in block form which provides for easier machining. Rubber (50% lead, 1% boron) and Paraffin (25% lead) are examples.

4.4 Neutron flux streaming through the existing shielding will be measured by neutron surveys.

4.4.1 Survey areas with a fast neutron instrument to determine the fast neutron flux ($n/cm^2/sec$).

4.4.2 Measure the area thoroughly, getting all dimensions.

4.4.3 Fill the streaming area as much as possible with the appropriate shielding material.

4.4.4 Resurvey the shielded area with the shielding temporarily in place.

4.4.5 If more shielding is necessary, add layers of shielding material, such as borated polyethylene, until the required attenuation factor is reached.

NOTE

This survey should be taken at full reactor power; otherwise, extrapolate values to that of full reactor power.

4.4.6 Fasten shielding in place.

4.4.7 After shielding for fast neutrons, survey for thermal neutrons and gammas. Apply high cross-sectional material followed by high-density material. Resurvey as necessary.

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4.4.8 Temporary shielding for the start up sources will be the same as that presented in Paragraph 4.4.1 through 4.4.7.

4.4.9 If the paraffin shielding is lost from a neutron source container, fill the container immediately with water as temporary shielding.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

See Reference 23 in the Reference Section for personnel exposure regulations.

METHOD 843
TEMPORARY BETA SHIELDING

1. SCOPE.

The purpose of this method is to describe the use and need of special temporary beta shielding.

2. SAMPLE.

Radioactive beta emitter to be shielded.

3. APPARATUS.

Not applicable.

4. PROCEDURES.

4.1 Beta radiation is not usually a shielding problem in a nuclear power plant. When large amounts of beta isotopes are present and they are shielded by high-density shielding, there will be a problem from Bremsstrahlung (see Nomenclature). Pure betas are best shielded by shielding materials of low atomic number to eliminate Bremsstrahlung.

4.2 Occasionally, it will be necessary for personnel to do visual maintenance work on an open contaminated system, whereby they will be exposed to high levels of beta radiation.

4.2.1 A bus or automobile windshield (preferably curved) will provide excellent beta shielding. Clear plastic may also be used.

4.2.2 Place windshield between maintenance personnel and



source of radiation with the convex side outward.

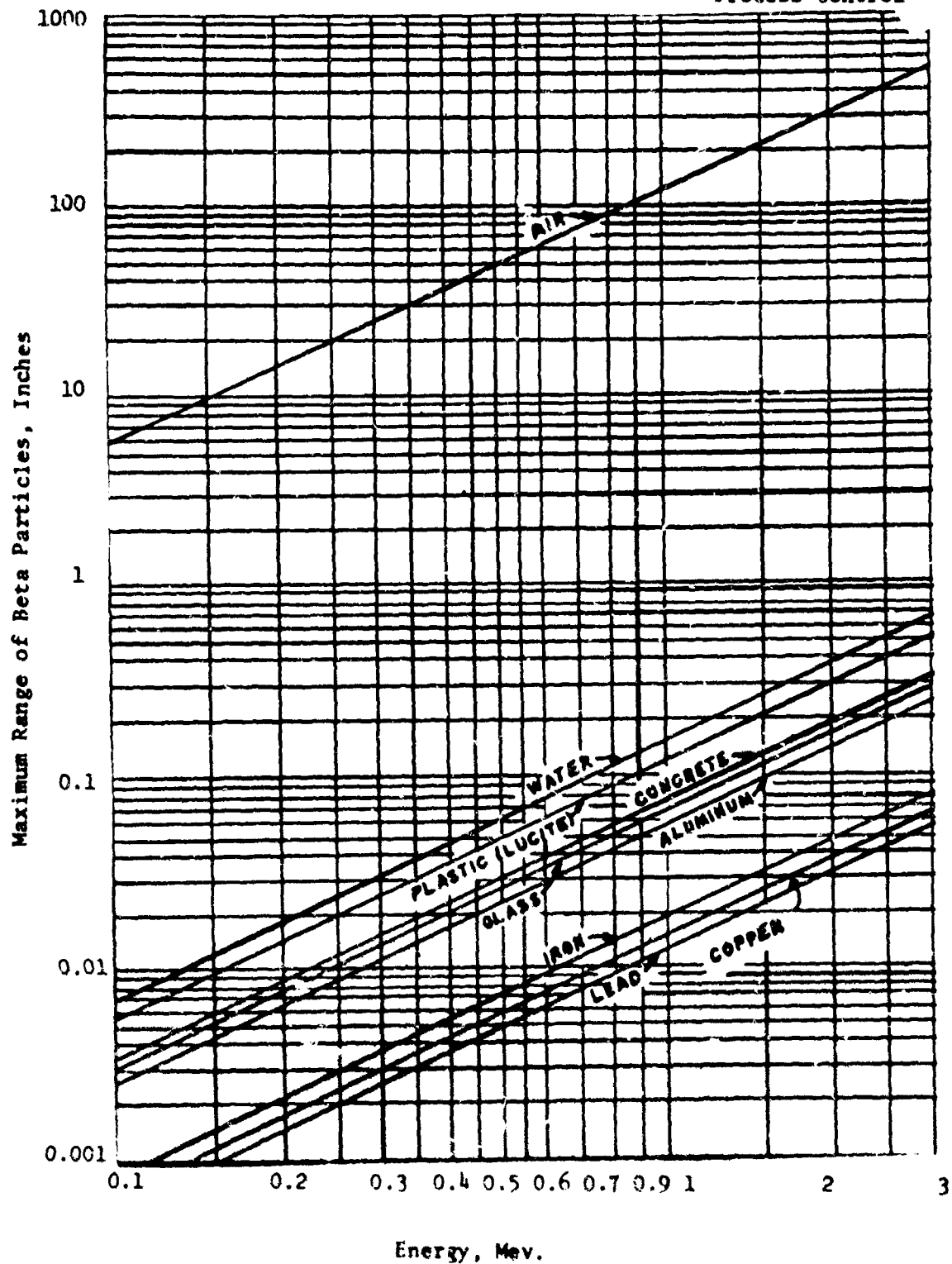
4.2.3 A clear plastic in sheet or moulded form, or material such as aluminum or copper, may be kept and used when beta-active material is handled.

5. RESULTS AND COMPUTATIONS.

Figure 843-1 illustrates the penetration ability of beta particles of energy up to 4 Mev through various medias, such as lead, copper, and glass. This graph may also be considered a shielding graph. This may be done by taking the maximum range of beta particles in various thicknesses of materials and providing a thickness of material sufficient to stop the most energetic beta particle.

6. TEST METHOD IMPLEMENTATION.

See Reference 23 in the Reference Section.



Energy, Mev.
Figure 843-1. Penetration Ability of Beta Radiation

Taken from page 155 of the Radiological Health Handbook by United States Public Health Service.

METHOD 844

SPECIAL HANDLING DEVICES

1. SCOPE.

The purpose of this method is to describe special handling devices, such as various types of tongs, forceps, and remote pipettes. Special handling tools for core changes and refueling are not discussed here, but are included in Plant Operating Manuals.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Not applicable.

4. PROCEDURES.

4.1 The special handling tools will consist of a backup device on a shaft of variable length, managed by an operating handle to pick up or release various types of samples and radioactive sources.

4.1.1 The pickup devices normally used in the nuclear plant are described below. These devices are normally equipped with aluminum or carbon steel handles up to 8 feet long. The handles are usually syringe or pistol grip type.

4.1.1.1 Niptongs - Used to pick up small sources.

They usually only open about 2 inches.

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4.1.1.2 Griptongs - Used to pick up glassware.

They open up to about 4 inches and are for picking up glassware.

4.1.1.3 Scissortongs - Can be made to handle material 6 to 8 inches in diameter.

4.1.1.4 Monkey wrench - Used to pick up material 3 inches in diameter or used to loosen bolts or nuts.

4.1.1.5 Small magnet - May be substituted for the tongs. It may be used for handling a small sealed source.

4.1.1.6 Long-handled forceps - May also be used to handle sources. The radioactivity level of the source that may be handled by the forceps will depend on personnel exposure time and the intensity of the source at the handling distance.

4.2 Remote pipettes eliminate all manual contact with the pipette while handling highly radioactive liquid samples. These are accurate for small quantities of solutions.

4.3 Special handling tools are used in the following applications: Source exposure for calibration, in the chemistry lab, handling highly radioactive samples, and in remote mechanical operations.

4.4 The purpose of special handling devices is to provide as

much distance between personnel and the radiation source as possible.

5. RESULTS AND COMPUTATIONS.

The predicted gamma dose rate for a given point source at various distances may be estimated by the following formula:

$$D = D_0 (1/r)^2 \quad (844-1)$$

Where D = Dose rate in rem/hr. at
distance r

D_0 = Dose rate in rem/hr. at unit
distance

r = Distance from the source

The dose rate is always less at a greater distance.

EXAMPLE

The potential dose rate at a distance of 1 foot from a point source is 50 rem/hr. What will be the average dose rate, using a five foot handling tool?

$$D = 50 (1/5)^2$$

$$D = 50 (1/25)$$

$$D = 2 \text{ rem/hr}$$

6. TEST METHOD IMPLEMENTATION.

See Reference (23) in the Reference Section for personnel exposure regulations.

METHOD 851

GENERAL PROVISIONS FOR CONTAMINATION CONTROL

1. SCOPE.

The purpose of this method is to give the general provisions for contamination control.

2. SAMPLE.

Not applicable.

3. APPARATUS.

Roll of sheet polyethylene.

Absorbent paper.

Masking tape,

Wood 2- x 4-inches for frame.

Anti-contamination clothing.

Roll of light weight canvas.

Film badges.

Dosimeters.

Small cloth bags for film badges and dosimeters.

Blower unit containing fan and filters.

Acetone.

Complete local exhaust system:

- a. Hood or capturing unit.
- b. Exhaust piping.
- c. Air cleaning plant roughing filters, absolute filter

AEC approved 99.97% efficient for 0.3 micron D.O.P.
particle.

- d. Exhaust fan capable of delivering a face velocity at
the hood of 125-150 lineal fpm with the sash completely
open.

Exhaust fan.

4. PROCEDURES.

4.1 The purpose of contamination control is to prevent
radioactive materials from entering the body and to pre-
vent the contamination of clean areas and equipment.

4.2 Radioactive materials enter the body in the following ways:

4.2.1 Inhalation - Breathing in of radioactive aerosols.

4.2.2 Ingestion - Eating, drinking, or smoking in areas
where loose radioactive materials are present.

4.2.3 Absorption - Absorbing directly through the skin or
through cuts and openings in the skin.

4.3 Contamination control will be effected by the following:

4.3.1 Placing the job in a temporary containment area,
hot cell, fume hoods, or glove box.

4.3.2 Requiring working personnel to wear protective
clothing (Method 821) and protective respiratory
equipment (Methods 831 through 836).

4.4 The following general steps shall be considered in
contamination control:

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4.4.1 Practice good housekeeping in all work with radioactive materials to minimize the spread of contamination.

4.4.2 Minimize all exposure to external and internal radiation.

4.4.3 Always wear personnel monitoring equipment as specified in Section 100.

4.4.4 Plan all jobs. For hazardous operations, determine the quantity and type of radioactive materials that will be handled. Set up experimental procedures including a flow plan. Conduct dry runs.

4.4.5 Set up portable air sampling equipment to measure airborne radioactive material. See Method 513.

4.4.6 Measure the surface radioactive material activity, and determine whether the activity is fixed or loose See Method 512.

4.4.7 Mark the work area to delineate the contamination zone. Set up control points. See Methods 711 through 716 and Method 721.

5. RESULTS AND COMPUTATIONS.

Not applicable.

6. TEST METHOD IMPLEMENTATION.

Not applicable.

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Process Control

SECTION 900 - ACCIDENT/INCIDENT CONTROL

This Section will be supplied at a later date.

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SECTION 1000 - BIOASSAYS

An analysis of radioactivity of the urine is a desired procedure.

Normal urine contains radio-potassium in amounts which may mask the added radioisotopes for which tests are made. Either potassium should be separated from the sample and the residual activity measured, or when the possible exposure is restricted to one isotope, this should be chemically separated from the urine. Examination of the feces may be required when the predominant elimination is by feces. Special tests for specific isotopes are in order when they exist (e.g., radio-iodine may be estimated in the thyroid gland in terms of the emitted gamma radiation measured by a Geiger Counter or ionization chamber). Where exposure to radioisotope dust or spray is a possibility, it may be desirable to test the activity of a nasal smear, or of the sputum.

1 July 1966

URINALYSIS

1. SCOPE.

The purpose of this method is to describe the procedure to be followed for urine bioassay for radioactive materials.

2. SPECIMEN.

Urine specimen collected in polyethylene specimen bottle.

3. APPARATUS.

- 3.1 Specimen bottle, six ounce.
- 3.2 Beaker - 250 ml.
- 3.3 Graduate cylinder - 100 ml.
- 3.4 Hot plate.
- 3.5 Muffle furnace.
- 3.6 Volumetric flask - 10 ml.
- 3.7 Pipette - one ml.
- 3.8 Planchet - 2" stainless steel.
- 3.9 Infra-red lamp.
- 3.10 Gas flow proportional counter.
- 3.11 Flame spectrophotometer.
- 3.12 Potassium standards (2, 4, 8, 16 mg/ml).
- 3.13 Linear graph paper.
- 3.14 Nitric acid - concentrated.
- 3.15 Hydrogen peroxide - 30%

4. PROCEDURE.

- 4.1 Set up numbered 250-ml beakers, corresponding to sample numbers.
- 4.2 Measure out 50 ml of specimen and pour into numbered 250-ml beaker.
- 4.3 Add 10 ml of concentrated nitric acid and 10 ml of 30% hydrogen peroxide.
- 4.4 Place on hot plate and evaporate to dryness. This should be performed in a hood.
- 4.5 Add 10 ml of concentrated nitric acid and 10 ml of 30% hydrogen peroxide.
- 4.6 Repeat the nitric acid, peroxide steps until ash is white.
- 4.7 Heat the white ash in muffle furnace for 15 minutes at 1000°F.
- 4.8 Cool and transfer the white ash to a numbered 10 ml volumetric flask, using demineralized water.
- 4.9 Bring to volume with demineralized water.
- 4.10 Pipette one ml of this solution into a weighed stainless steel planchet that has been properly labeled.
- 4.11 Evaporate the sample to dryness under an infra-red lamp.
- 4.12 Beta count the sample as outlined in Method 631.5.
- 4.13 Analyze the remainder of the sample in the volumetric flask for potassium, using a flame photometer.
- 4.14 Calculate the gross foreign beta activity (Method 631.5) and record results.

5. RESULTS AND COMPUTATION.

- 5.1 Enter all samples results on the urinalysis record. (See Attachment No. 1 for forms).

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- 5.2 Correct gross beta count as outlined in Method 631.5. Report the results in uc/cc.
 - 5.3 Calculate K^{40} activity as outlined in Method 631.5. Report results as dpm of K^{40} per total sample.
 - 5.4 Subtract K^{40} activity from the gross beta activity. Report results as dpm per total sample.
6. TEST METHOD IMPLEMENTATION.
- 6.1 A base urinalysis is recommended on all newly assigned personnel before they are assigned work at a nuclear site.
 - 6.2 Routine urinalysis is recommended every 6 months on all personnel working with radioactive material.
 - 6.3 Urinalyses are also made as the need is indicated by working conditions. (Reference AR 40-582). "Where there is reason to believe that an individual has been internally contaminated with radioactive material, a 24-hour urine sample will be initiated as soon as possible."
 - 6.4 A re-sample will be taken if it is determined that the gross beta activity is in excess of 1.98×10^{-6} uc/cc.
 - 6.5 Frequency of determination will be determined by the plant technical manual.

ATTACHMENT NO. 1

METHOD 1051

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Process Control

URINALYSIS RECORD

Name	Film Badge No.	Sample Date	Reason	Results dpm/ml
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Routine, Base Reference, Working Conditions.

1011-4

1 July 1966

METHOD 1012
FECAL ANALYSIS

Health Physics-
Process Control

1. SCOPE.

The purpose of this method is to describe the procedures to be followed for determining radioactivity in feces.

2. SPECIMEN.

2.1 Stool specimens, collected in a standard plastic-lined carton.

3. APPARATUS.

3.1 Carton - standard plastic lined, with uniform internal diameter of 85 mm.

3.2 Tamper assembly including base plate, tamper with scale on handle and slotted disc. (Reference Figure 1012-1 in step 4.4).

3.3 Height correction curve.

3.4 Gamma-ray scintillation detector and counter. (Multi-channel analyzer).

3.5 Waxed paper.

4. PROCEDURE.

4.1 Collect the individual stool specimen in standard carton.

4.2 Place the carton containing the specimen on the base plate.

4.3 Place the tamper in carton with the slotted disc in place on top of the carton.

4.4 Tamper the specimen and read the height of the specimen from the tamper handle.

NOTE

Waxed paper can be used as a separator between tamper and the specimen.

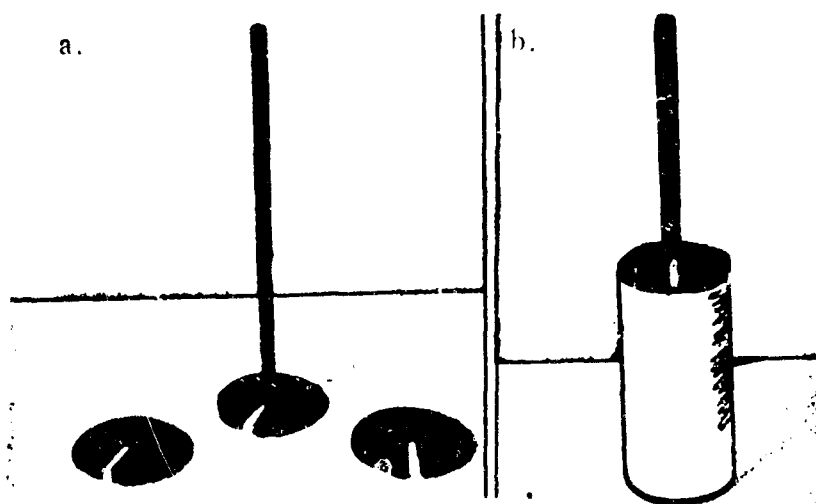


Figure 1012.1 . a. The base plate is at the left, the tamper with scale on handle in the center, and the slotted disc at the right.
b. Standard carton on the base plate. The slotted disc is in place on top of carton so that height of specimen can be read from tamper handle.

4.5 Remove the tamper from the carton and seal the carton.

4.6 Wrap the carton in polyethylene bag.

4.7 Radioassay (gross counts and gamma-ray spectrum) the specimen by placing the carton on top of 3" x 3" Na(I) T1 flat crystal

4.8 Record the results in dpm/total egestion.

5. RESULTS AND COMPUTATIONS.

5.1 Construct a height correction curve using Cs^{137} and the following example as a guide.

EXAMPLE

Height-correction factor for radioactivity: If a standard carton is used, it can be assumed that the only geometric variable of the counting system is the height of the stool specimen.

An isotope solution (one ml) containing radioactivity (one μc of radioiodine, radiochromium, or radioiron) is placed

in the center of the bottom of the carton. The volume of the radioisotope is so small that it is considered as a point source of radiation, and the radioactivity counted is taken as 100 per cent or a correction factor of 1.0.

After the radioisotope is counted at zero volume, the volume in the container is increased by adding known increments of water, and the radioactivity determined. The counts obtained per second are divided by the net counts per second for the respective isotopes at zero volume. Height correction curves are thus obtained (Figure 1012-2). From each curve, the division correction factors are determined. Table 1012-1 shows the height correction factor for radiochromium.

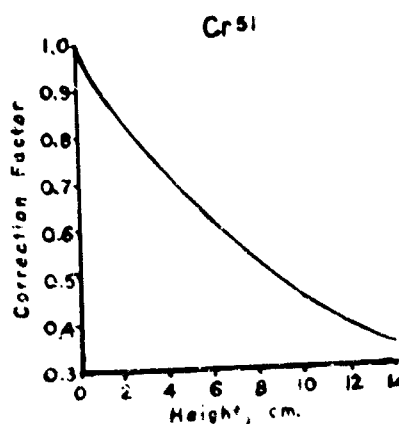


Figure 1012-2 Example of Height-Correction Curve for Determining Radioactivity in Standard Carton

- 5.2 Calculate the total activity egested per day using the following formula:

$$\text{Total activity/day} = \frac{(A) (f)}{\Sigma}$$

Where A = Corrected net cpm for total specimen (corrected for background and decay).

f = Height - multiplication correction factor
(see preceding example for construction of height correction curve).

Σ = Counter efficiency

- 5.3 Identify the photopeaks in gamma-ray spectrum by comparing the spectrum with those in R. L. Heath's "Gamma-ray Spectrum Catalogue."

6. TEST METHOD IMPLEMENTATION.

In an accident situation, the medical officer will prescribe the frequency of collection.

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Table 1012-1. Height-Correction Factor for Determining Radio-Chromium (Cr-51) in Individual Stool Specimens

Height, cm	Division Factor	Multiplication Factor	Height, cm	Division Factor	Multiplication Factor
0.3	0.965	1.036	5.0	0.670	1.493
0.5	.950	1.053	5.2	.660	1.515
0.6	.935	1.070	5.4	.650	1.538
0.8	.920	1.087	5.6	.640	1.563
1.0	.905	1.105	5.8	.630	1.587
1.2	.890	1.124	6.0	.620	1.613
1.4	.876	1.142	6.2	.610	1.639
1.6	.862	1.160	6.4	.600	1.667
1.8	.850	1.176	6.6	.590	1.695
2.0	.838	1.193	6.8	.580	1.724
2.2	.825	1.212	7.0	.570	1.754
2.4	.812	1.232	7.5	.550	1.818
2.6	.800	1.250	8.0	.530	1.887
2.8	.788	1.269	8.5	.510	1.961
3.0	.775	1.290	9.0	.490	2.041
3.2	.762	1.312	9.5	.470	2.128
3.4	.750	1.333	10.0	.455	2.198
3.6	.740	1.351	10.5	.440	2.273
3.8	.730	1.370	11.0	.425	2.353
4.0	.720	1.389	11.5	.410	2.439
4.2	.710	1.408	12.0	.400	2.500
4.4	.700	1.429	13.0	.375	2.667
4.6	.690	1.449	14.0	0.350	2.857
4.8	0.680	1.471			

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SECTION 1100 - RADIOCHEMISTRY FOR HEALTH PHYSICS OPERATIONS

Production of radioactive materials in a nuclear power reactor imposes certain operating limitations and requires additional safeguards. Radiochemical analytical procedures to be performed by the Process Control Specialist are presented in this Section.

1 July 1966

METHOD 1111

30-MINUTE DEGASSED AND 120-HOUR GROSS ACTIVITY

1. SCOPE

The purpose of this method is to describe the procedure for analyzing the primary coolant for gross activity.

2. SAMPLE

Reactor primary coolant water.

3. APPARATUS

Polyethylene bottle, 500-ml capacity.

Support stand - apparatus - rectangular base 6 x 9.

Clamps (2), utility.

Fritted funnel (fine), 60-ml capacity.

Stopcock, glass, 3-way.

Hose - vacuum, 1/4-in. ID.

Rubber stopper - one hole - No. 9.

Vacuum pump

Pipet - 2-ml volumetric.

Vials - soft glass - 4-ml capacity with cap.

Saran wrap or equivalent.

Planchets - 2-in. diameter.

Gamma ray multichannel analyzer with well crystal or a beta counter.

4. PROCEDURE

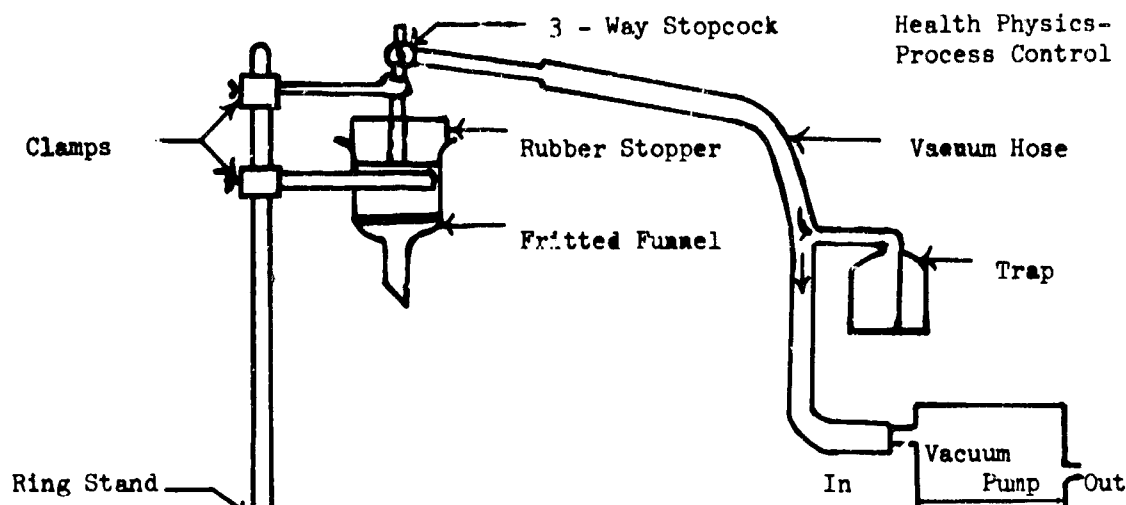


Figure IIII-1. Degassing Apparatus.

- 4.1 Set up the degassing apparatus in a fume hood as shown in Figure IIII-1
- 4.2 Collect a sample before the demineralizer as outlined in Method 515.
- 4.3 Transfer 10 ml of the sample to the fine fritted funnel.

NOTE

The porosity of a fine fritted funnel is such that
water will not drain from the funnel.

- 4.4 Stopper the funnel with a 1-hole rubber stopper. Reference
Figure 1.
- 4.5 Check to ensure that the 3-way stopcock is open to the atmosphere.
- 4.6 Start the vacuum pump.
- 4.7 Regulate the amount of air being pulled through the funnel by
manipulating the 3-way stopcock. Do not permit the water to be
pulled from the funnel.
- 4.8 Degas the sample for 1 minute.
- 4.9 Open the stopcock to the atmosphere, and stop vacuum pump.

- 4.10 Remove the rubber stopper and withdraw a 2-ml sample, using a 2-ml volumetric pipet.
- 4.11 Transfer the sample to a 4-ml soft glass vial or a 2-in. planchet.
- 4.12 Cap the counting vial and wrap in Saran wrap or evaporate the sample in the planchet to dryness with an infrared lamp.
- 4.13 Place the counting vial in a NaI (Tl) well crystal or the planchet in a beta counter.
- 4.14 At exactly 30 minutes from sample time, start the counter and count for 2 minutes.
- 4.15 Record the gross count rate, cpm/ml. See Attachment No. 1.
- 4.16 Schedule the 2-ml sample for a recount at 120 hours.
- 4.17 Recount the sample at 120 hours from sample time.
- 4.18 Record the gross count rate.
- 4.19 Calculate activity, recording results in $\mu\text{C}/\text{cc}$ as outlined in Paragraph 5.

5. RESULTS AND COMPUTATIONS

- 5.1 Record sample time.
- 5.2 Record reactor power level.
- 5.3 Record the period of time at which the reactor is at steady state power (ΔT).
- 5.4 Record gross 30-minute and 120-hour counts (cpm).

Health Physics-
Process Control

5.5 Calculate gross activity at 30 minutes and 120 hours ($\mu\text{c/cc}$)

as outlined in Method 631.5.

5.5.1 Gross activity =

5.5.1.1. For gamma counting $\mu\text{c/cc} =$

$$\frac{\text{cpm Sample} \times \mu\text{c/Cs}^{137} \text{ Standard}}{\text{cpm Cs}^{137} \text{ Standard} \times \text{cc Sample}}$$

5.5.1.2 For beta counting $\mu\text{c/cc} =$

$$\frac{\text{cpm Sample} \times \mu\text{c Tl}^{204} \text{ Standard}}{\text{cpm Tl}^{204} \text{ Standard} \times \text{cc Sample}}$$

5.6 Correct to 100% power if taken at 10% power or higher.

6. TEST METHOD IMPLEMENTATION

6.1 Sample should not be taken unless the reactor is above 10% power
for a minimum of 4 hours.

6.2 The sample must be prepared for counting in less than 30 minutes.

6.3 If the 30-minute gross activity is a factor of 2 higher than previous
analysis at the same reactor power, reanalysis should be per-
formed, using all decontaminated or new glassware. If the high activity
is verified, an attempt should be made to identify and determine the origin
of the nuclide(s) responsible for the high activity.

ATTACHMENT NO 1.

METHOD 1111

PRIMARY COOLANT GROSS ACTIVITY

Sample Date	Sample Time	Percentage Power	Time at Power, °F	Gross 30 min.	cpm/ml 120 hr.	Gross 30 min.	dpm/ml 120 hr.
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METHOD 1112
GROSS IODINE

1. SCOPE

The purpose of this method is to describe the procedure to be followed in the analysis of the primary coolant for gross iodine activity.

2. SAMPLE

2.1 Reactor primary coolant water.

2.2 The sample aliquots are selected on the basis of the estimated gross iodine activity.

3. APPARATUS AND REAGENTS

Apparatus and reagents are the same as those listed in Method 1131 with the exception of the Gamma Ray Multi-Channel Analyzer.

Beta counter.

Filter tower apparatus - stainless steel 10-ml capacity - 15/16 in. filter paper.

4. PROCEDURE

4.1 Follow Steps 4.1 through 4.16 as outlined for Method 1131.

4.2 Mount the sample for beta counting.

4.3 Count the sample exactly 15 minutes after sampling time. Count for 10 minutes or until 10,000 counts are collected. The techniques for beta counting in Method 631.5 shall be used.

4.4 Calculate the specific activity of gross iodine as outlined in

Section 5.

5. RESULTS AND COMPUTATIONS

5.1 Record the sample time and date.

5.2 Record the sample aliquots in cc.

5.3 Record standardization data of iodine carrier solution.

5.4 Record chemical yield of AgI.

5.5 Calculate the specific activity of gross iodine. Record the results

in $\mu\text{c/cc} \pm$ one standard deviation at sample time. See Method

631.5.

5.5.1 Activity of Gross Iodine ($\mu\text{c/cc}$) =

$$\frac{\text{cpm I} \times \mu\text{c Tl}^{204} \text{ Standard}}{\text{cpm Tl}^{204} \text{ Standard} \times \text{cc Sample} \times \text{Fractional Yield}}$$

METHOD 1113
GROSS CESIUM

1. SCOPE

This method is used to determine gross cesium activity in the primary coolant.

The decay of Cs-138 is followed.

2. SAMPLE

2.1 Reactor primary coolant water.

2.2 The sample aliquots are selected on the basis of the estimated gross cesium activity.

3. APPARATUS AND REAGENTS

Apparatus and reagents are the same as those listed in Method 1133.

Beta counter.

4. PROCEDURE

4.1 Follow Steps 4.1 through 4.30 as outlined for Method 1133.

NOTE

Freshly drawn primary coolant water samples contain gaseous xenon activity, which decays to cesium daughters. Since the gross determination is to serve as an indicator of the cesium level, the period of degassing is standardized by performing Step 4.17, which includes centrifuging

and discarding of the supernate exactly 20 minutes after sampling time. All reagents requiring chilling should be chilled before sampling. All carriers should be pipeted prior to sampling. The short half-life (32 min.) for Cs-138 requires the sample to be counted within 2 hrs. after drawing the sample.

4.2 Mount the sample for beta counting.

4.3 Count the sample in a beta counter every 15 minutes to give ten counts. Count twice the next day. The techniques for beta counting in Method 631.5 shall be used.

4.4 Plot the decay curve of the corrected gross counting rate on semi-log paper. The principle corrections will be background, standard factor and coincidence when applicable. A 32-minute half-life determined by the extrapolation method indicates the presence of Cs-138.

4.5 Extrapolate the gross curve back to sampling time.

4.6 Calculate the specific activity of gross cesium as outlined in Paragraph 5.

5. RESULTS AND COMPUTATIONS

5.1 Record the sample time and date.

5.2 Record the sample aliquots in cc.

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5.3 Record standardization data for cesium carrier.

5.4 Record the chemical yield.

5.5 Calculate the specific activity of gross cesium. Record the results in $\mu\text{c/cc} \pm$ one standard deviation at sample time. (See Method 631.5.)

5.5.1 Activity of Gross Cesium ($\mu\text{c/cc}$) =

$$\frac{\text{cpm Cs} \times \mu\text{c Tl}^{204} \text{ Standard}}{\text{cpm Tl}^{204} \text{ Standard} \times \text{cc Sample} \times \text{Fractional Yield}}$$

METHOD 1121
MANGANESE - 54

1. SCOPE

The purpose of this method is to describe the procedures for determining Mn-54 activity. The basic corrosion product separation is one based on anion exchange separation of the carrier equilibrated mixture.

2. SAMPLE

2.1 The sample can be either a liquid or a solid (crud).

2.2 Aliquots for radiochemical analysis are selected on the basis of gross beta, gamma activity and will vary with the sample source.

3. APPARATUS.

Porcelain crucible, size double O.

Beakers - 100 ml, 250 ml, 400 ml, 500 ml, 1 liter, 2 liter.

Furnace, muffle, 0 - 2000°F.

Desiccator with Coors plate - 200 mm ID.

Analytical balance.

Burner (high temperature).

Erlenmeyer flasks - (2) - 125 ml.

Pipets - 2-ml capacity.

Graduate - 100-ml capacity.

Graduates - 25, 10 ml.

Chromatographic tube - 10 mm ID x 300 mm length.

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Hot plate 115 - 220 volt with temperature selector.

Centrifuge for 50-ml tubes - 4-place head.

Centrifuge tubes - 50 ml Pyrex.

Whatman - 542 filter paper, 15/16 in. diameter.

Filter tower apparatus-stainless steel - 10ml. Capacity 15/16 in.

filter paper.

Vacuum pump, 115 volt.

Rubber stopper - one hole, No. 8.

Filtering flask with side-tube, 1000-ml capacity.

Oven, 115 volt, 60 cycles, 40 - 200 °C Range.

Scotch tape, 1 in. wide.

Counting vial with cap, soft glass, 4-ml capacity.

Silica gel 6-16 mesh.

Potassium pyrosulfate, fused, powder, reagent grade.

Hydrochloric acid, 12 N, reagent grade.

Resin, anion, chloride form, 50 - 100 mesh (Dowex-i or equivalent).

Nitric acid, concentrated, reagent grade.

Ammonium hydroxide, concentrated, reagent grade.

Ethyl alcohol, reagent grade.

Iron carrier (10 mg/ml) (Dissolve 48.4 of reagent grade of $\text{FeCl}_3 \cdot$

6 H_2O in 1 N HCl and make up to 1 liter with 1 N HCl).

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Manganese carrier (10 mg/ml) (Dissolve 22.9 gm of reagent grade MnCl_2 in water and make up to 1 liter.)

Chromium carrier (10 mg/ml). (Dissolve 37.3 gm of reagent grade K_2CrO_4 in water and make up to one liter.)

Cobalt carrier (10 mg/ml). (Dissolve 49.3 of reagent grade $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in H_2O , add 1 ml concentrated HNO_3 and dilute to 1 liter with water.)

Copper carrier (10 mg/ml). (Dissolve 1 gm of reagent grade copper metal in 100 ml 1:3 nitric acid.

Antimony carrier (10 mg/ml). (Dissolve 1.9 gm of reagent grade SbCl_3 in 100 ml 6N HCl .)

Zinc carrier (10 mg/ml). (Dissolve 1 gm of reagent grade zinc metal in the minimum quantity of 2M HCl and dilute to 100 ml.)

Potassium chlorate, reagent grade.

Hydrogen peroxide, 30% reagent grade.

Thioacetamide - 4% solution.

Ammonium chloride, reagent grade.

Diammonium hydrogen phosphate, reagent grade.

Sintered glass crucibles, fine porosity.

4. PROCEDURE

4.1 Place the particulate sample in a OO weighed porcelain crucible.

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If the sample is a liquid, add sample to an appropriate size beaker and add 1 ml of concentrated HCl and proceed with Step 4.8.

NOTE

All procedures shall be carried out using
a fume hood.

- 4.2 Place the crucible in muffle furnace and ignite at 800°C for 15 minutes.
- 4.3 Cool crucible and contents in desiccator.
- 4.4 Determine the weight of the sample by weighing the crucible and its contents and subtracting the weight of crucible.
- 4.5 Cover the sample with potassium pyrosulfate (fusing powder) and heat over a burner until the crud is completely fused.
- 4.6 Cool the crucible and contents and dissolve and melt with a minimum of 8 N HCl.
- 4.7 Transfer the dissolved melt to a 100-ml Erlenmeyer flask.
- 4.8 Pipet 2 ml each of standard iron, chromium, cobalt, and manganese carriers (10 mg/ml).
- 4.9 Evaporate the solution just to dryness.

CAUTION

Do not bake .

- 4.10 Pick up the residue in about 15 ml of concentrated HCl.

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- 4.11 Prepare an anion exchange column by adding Dowex-1 or equivalent resin (50-100 mesh) to a chromatographic tube. The height of the resin bed should be approximately 6 in.
- 4.12 Condition the column with about 5 column volumes of concentrated HCl. Discard the effluent. Keep the resin bed covered with acid at all times. If the column is allowed to go dry, channeling will result.
- 4.13 Transfer the solution from Step 4.10 to the resin column using concentrated HCl.
- 4.14 Elute the manganese from the column with 8N HCl (25 ml), collecting the eluate in a 100-ml flask. The collected fraction contains manganese and chromium.
- 4.15 Evaporate the fraction containing the manganese and chromium to dryness.
- 4.16 Add 25 ml of concentrated HNO_3 and about 1 mg of solid potassium bromate and boil to precipitate the MnO_2 .
- 4.17 Centrifuge off the MnO_2 . The supernate contains the chromium. The supernate can be used for chromium analysis if desired.
- 4.18 Add 15 ml of concentrated HNO_3 and several drops of 30% H_2O_2 to the MnO_2 .
- 4.19 Evaporate to dryness and take up in minimum amount of concentrated HCl.

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- 4.20 Dilute to 10 ml with deionized water.
- 4.21 Add 1 ml of Sb, Cu, and Zn hold back carriers.
- 4.22 Add 2 ml of the 4% solution of thioacetamide to precipitate the Cu and Sb.
- 4.23 Add NH_4OH to make the supernate alkaline and precipitate the Mn and Zn sulfides.
- 4.24 Centrifuge off the Mn and Zn sulfides, discarding the supernate.
- 4.25 Dissolve the precipitate in concentrated HNO_3 . The final volume should be 10 ml.
- 4.26 Add about 1 mg of solid potassium chlorate to precipitate the MnO_2 .
- 4.27 Centrifuge and reject the supernate.
- 4.28 Weigh a Whatman No. 542 filter disc on analytical balance.
- 4.29 Place the weighed filter disc in a stainless steel filtering tower.
- 4.30 Slurry the MnO_2 with water and transfer the MnO_2 to the filtering tower. Vacuum filter.
- 4.31 Wash the precipitate with 10 ml of H_2O and 10 ml of ethyl alcohol.
- 4.32 Dry the filter containing the MnO_2 in a drying oven set at 100°C . for approximately 10 minutes. Transfer the filter to a desiccator to cool.
- 4.33. Weigh the filter containing the MnO_2 to constant weight on a analytical balance and determine the chemical yield by subtracting

the weight of the filter paper.

4.34 Mount the sample for gamma counting. (Reference Section 600.)

4.35 Count the sample, using a gamma ray spectrometer.

5. RESULTS AND COMPUTATION

5.1 Record sample time and date.

5.2 Record sample volume or weight.

5.3 Record standardization data of carrier solution.

NOTE

Standardization of the manganese carrier solution is performed as follows: Pipet 5.0 ml of carrier solution into a 400-ml beaker and dilute to 200 ml. Almost neutralize with 1:3 ammonium hydroxide and add 20 gm of NH_4Cl and 2 gm of $(\text{NH}_4)_2\text{HPO}_4$. If a precipitate forms, dissolve in a few drops of 10% HCl heat the solution almost to boiling, and add 1:3 ammonium hydroxide dropwise with stirring until a precipitate begins to form. Do not add any additional ammonium hydroxide. Continue the heating and stirring until the precipitate becomes crystalline, then add one to two drops of ammonium hydroxide. Continue heating and stirring until no additional precipitate is produced and

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the appearance remains unchanged. The solution must be at 90 to 95°C throughout the precipitation and a large excess of ammonia must be avoided. Allow the solution to stand at 0°C for 2 hours. Filter through a tared sintered glass crucible and wash with cold 1% solution until chloride free. Dry at 100-105°C to constant weight. Cool in a desiccator. Weigh as $\text{MnNH}_4\text{PO}_4\text{H}_2\text{O}$. The analysis is performed in quadruplicate. Results should agree to within 1%.

$$\text{Mn (mg/ml)} = \frac{(\text{mg MnNH}_4\text{PO}_4\text{H}_2\text{O}) (0.2955)}{5}$$

5.4 Record chemical yield (Step 4.33).

5.5 Determine the area under 0.835 Mev photopeak (total counts).

5.6 Calculate specific activity of Mn-54 at sample time. Record results in Ac/cc or $\text{Ac/mg} \pm$ one standard deviation. (See

Method 633.3.)

METHOD 1122
COBALT-58 and -60

1. SCOPE

The purpose of this method is to describe the procedures for determining Co-58 and -60 activity in both soluble and particulate (crud) samples. The basic corrosion product separation is one based on ion exchange separation of the carrier equilibrated mixture.

2. SAMPLE

2.1 The specimen can be either a liquid or a solid (crud).

2.2 Aliquots for radiochemical analyses are selected on the basis of gross beta-gamma activity and will vary with the sample source.

3. APPARATUS

Apparatus and reagents are the same as those listed in Method 1121,

3.1 through 3.34

Thioacetamide - 4% solution.

4. PROCEDURE

4.1 Follow Steps 4.1 through 4.14 as outlined for the Mn-54 procedure (Method 1121).

4.2 Wash resin bed with approximately 20 ml of 6M HCl, until the cobalt is nearly down to the bottom of the column. This is seen as a green color on the column.

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- 4.3 Wash with 3M HCl, discarding the HCl (approximately 10 ml) until the cobalt starts to come off (pink color).
- 4.4 Collect the cobalt fraction in a 125-ml Erlenmeyer flask.
- 4.5 Make the solution alkaline with ammonium hydroxide.
- 4.6 Add thioacetamide dropwise while stirring the solution to precipitate CoS.
- 4.7 Centrifuge off the CoS.
- 4.8 Filter the CoS onto a Whatman 542 filter disc.
- 4.9 Transfer the filter paper containing the CoS to an OO porcelain crucible.
- 4.10 Dry under a heat lamp or in an oven set at 100°C.
- 4.11 Cover with a porcelain cover and ignite for 10 minutes at 700°C.
- 4.12 Remove the cover and continue the ignition for 30 minutes to convert the CoS to Co₂O₃.
- 4.13 Cool, add 1 cc ethanol, and slurry with a polished glass stirring rod.
- 4.14 Transfer the Co₂O₃ to a weighed 543 Whatman filter paper with ethanol as required.
- 4.15 Dry at 110°C for 10 minutes, cool in a desiccator, and weigh to determine the chemical yield.
- 4.16 Mount the sample for gamma counting.
- 4.17 Radioassay the sample, using gamma ray spectrometry techniques as outlined in Method 633.3.

5. RESULTS AND COMPUTATION

- 5.1 Record sample time and date .
- 5.2 Record sample volume or weight.
- 5.3 Record standardization data of carrier solutions.

NOTE

Standardization of the cobalt carrier solution is performed as follows: Pipet 5.0 ml of carrier solution to 10 ml H₂O. Add 2 ml of concentrated NH₄OH and thioacetamide drop by drop with stirring to precipitate CoS. Filter the CoS onto a Whatman No. 542 filter paper and wash with 5 ml of 1:3 aqueous ammonia. Transfer the paper and precipitate to a tared No. 0 porcelain crucible. Dry under a heat lamp, cover with a porcelain cover and ignite for 10 minutes at 700°C. Remove the cover and continue the ignition for 30 minutes. Cool in a desiccator. Weigh the crucible to a constant weight.

The analysis is performed in quadruplicate. Results should agree to within 0.5%.

$$\text{Co (mg/ml)} = \frac{(\text{mg Co}_2\text{O}_3) (0.7106)}{5}$$

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- 5.4 Record chemical yield (Step 4.15).
- 5.5 Determine the area under the 0.81 (Co-58) and 1.33 Mev (Co-60) photopeaks.
- 5.6 Calculate the specific activity of Co-58 and 60 at sample time.
- Record results in $\mu\text{C/cc}$ or $\mu\text{C/mg} \pm$ one standard deviation.
- (See Method 633.3).

METHOD 1123

IRON-59

1. SCOPE

This method is used for determining Fe-59 activity in both soluble and particulate (crud) samples. The basic corrosion product separation is one based on anion exchange separation of the carrier equilibrated mixture.

2. SAMPLE

2.1 The sample can be either a liquid or a solid (crud).

2.2 Aliquots for radiochemical analyses are selected on the bases of gross beta-gamma activity and will vary with the same sample source.

3. APPARATUS AND REAGENTS

Apparatus and chemical requirements are the same as those listed in Method 1121, 3.1 through 3.34.

Separatory funnel -- 250 ml.

Isopropyl ether.

4. PROCEDURE

4.1 Follow Steps 4.1 through 4.4 as outlined for the Co-58 and 60 procedure (Method 1122).

4.2 Drain the HCl in the column to the top of the resin bed.

4.3 Fill the column with 0.51M HCl and allow the HCl to drain.

4.4 Discard the HCl until the iron starts off the column (yellow color).

- 4.5 Collect the iron fraction in a 125-ml Erlenmeyer flask.
- 4.6 Make the 0.5M HCl solution obtained from the column basic with NH_4OH to precipitate the $\text{Fe}(\text{OH})_3$.
- 4.7 Centrifuge off the $\text{Fe}(\text{OH})_3$.
- 4.8 Dissolve the $\text{Fe}(\text{OH})_3$ in 10 ml of 8M HCl, and transfer the solution to a 250-ml separatory funnel.
- 4.9 Add 30 ml of isopropyl ether and shake for approximately 1 minute.
- 4.10 Discard the water layer.
- 4.11 Wash the ether layer with 10 ml of 8M HCl and discard the wash.
- 4.12 Remove the iron from the ether by washing it three times with 10 ml of deionized water.
- 4.13 Place the water layer in a centrifuge tube and make basic with NH_4OH to precipitate the $\text{Fe}(\text{OH})_3$.
- 4.14 Centrifuge off the $\text{Fe}(\text{OH})_3$.
- 4.15 Transfer the $\text{Fe}(\text{OH})_3$ with methyl alcohol to an unweighed filter paper.
- 4.16 Place the filter and contents in a porcelain crucible.
- 4.17 Dry the filter and contents under a heat lamp.
- 4.18 Ignite the filter and $\text{Fe}(\text{OH})_3$ at 800°C to convert the Fe_2O_3 .
- 4.19 Cool, add 1 cc ethanol, and slurry with a polished glass stirring rod.
- 4.20 Transfer the Fe_2O_3 to a weighed 542 Whatman filter paper with ethanol as required.

- 4.21 Dry at 110°C for 10 minutes, cool, and weigh to determine the chemical yield.
- 4.22 Mount the sample for gamma counting.
- 4.23 Count the sample using a gamma ray spectrometer.

5. RESULTS AND COMPUTATIONS

- 5.1 Record sample time and date.
- 5.2 Record sample volume or weight.
- 5.3 Record standardization data of carrier solution.

NOTE

Standardization of the iron carrier solution is performed as follows: Pipet 5.0 ml of the carrier solution into a tared No. 0 porcelain crucible. Add concentrated NH_4OH dropwise to precipitate $\text{Fe}(\text{OH})_3$. Evaporate to dryness under an infrared heat lamp. Ignite in a muffle furnace at 700°C for 15 minutes. Cool in a desiccator and weigh to constant weight.

The analysis is performed in quadruplicate.

Results should agree to within 0.5%

$$\text{Fe}(\text{mg/ml}) = \frac{(\text{mg Fe}_2\text{O}_3) (0.6994)}{5}$$

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- 5.4 Record chemical yield (Step 4.21).
- 5.5 Determine the areas under the 1.29-Mev photopeak (total counts).
- 5.6 Calculate the specific activity of Fe-59 at sample time. Record the results in $\mu\text{c/cc}$ or $\mu\text{c/mg}$ $\pm$ one standard deviation. (See Method 633.3)

METHOD 1124
CHROMIUM - 51

1. SCOPE

This method is used for determining Cr-51 activity in both soluble and particulate (crud) samples. The basic corrosion product separation is one based on anion exchange separation of the carrier equilibrated mixture.

2. SAMPLE

2.1 The sample can be either a liquid or a solid (crud).

2.2 Aliquots for radiochemical analyses are selected after an estimate of Cr-51 activity is made. The estimate is made, based on gamma-ray spectrum of the sample to be analyzed.

3. APPARATUS AND CHEMICALS

Apparatus and chemical requirements are the same listed for Mn-54, Steps 3.1 through 3.34.

Separatory funnel - 125-ml volume.

Ice bucket about 10 in. high.

Sodium nitrite (NaNO_2), reagent grade.

Ammonium acetate (NH_4Ac), reagent grade.

Acetic acid glacial, reagent grade.

Barium nitrate $\text{Ba}(\text{NO}_3)_2$, reagent grade

Hydrogen peroxide 30%, reagent grade.

Ethyl ether, reagent grade.

Potassium chlorate KClO_3 , reagent grade.

4. PROCEDURE

4.1 Follow Steps 4.1 through 4.17 as outlined for the Mn-54 procedure (Method 1121).

4.2 Evaporate the supernate liquid to about one half its volume.

4.3 Add 1 M NaNO_2 dropwise until the $\text{Cr}_2\text{O}_7^{-2}$ is reduced to Cr^{+3} .

4.4 Heat to remove the excess NO_2 .

4.5 Add concentrated NH_4OH dropwise until the $\text{Cr}(\text{OH})_3$ precipitates.

If an excess of NH_4OH is added, heat to drive off the excess. Be sure precipitation is complete.

4.6 Centrifuge and discard the supernate liquid.

4.7 Wash with 15 ml of deionized water. Discard the wash water.

4.8 Dissolve precipitate in 6-8 drops of concentrated HCl and dilute to 15 ml.

4.9 Reprecipitate the $\text{Cr}(\text{OH})_3$ with concentrated NH_4OH .

4.10 Centrifuge and discharge the supernate liquid.

4.11 Wash the precipitate with 5 ml deionized water. Discard the wash.

4.12 Dissolve the $\text{Cr}(\text{OH})_3$ in 5 drops of HNO_3 and dilute to 15 ml.

4.13 Add 3 ml of saturated KBrO_3 and heat to oxidize the Cr^{+3} to $\text{Cr}_2\text{O}_7^{-2}$.

4.14 Make just basic with concentrated NH_4OH .

- 4.15 Add 3 drops of 6N HNO_3 , 2 ml 6N NH_4Ac and 1 ml of 6M HAc .
- 4.16 Heat to near boiling and add 3 ml of saturated $\text{Ba}(\text{NO}_3)_2$ to precipitate BaCrO_4 .
- 4.17 Transfer the BaCrO_4 to a weighed 542 Whatman filter paper with water.
- 4.18 Wash with water and ethanol.
- 4.19 Dry at 110°C for 10 minutes, cool, and weigh to determine the chemical yield.
- 4.20 Mount the sample for gamma counting.
- 4.21 Count the sample, using techniques for gamma pulse height analyses (See Method 633.3).

5. RESULTS AND COMPUTATION

- 5.1 Record the sample time and date.
- 5.2 Record the sample volume or weight.
- 5.3 Record the standardization data of the carrier solution.

NOTE

Standardization of the chromium carrier is performed as follows: Dilute 5.0 ml of carrier to 200 ml. Make slightly acid with 6M $\text{HC}_2\text{H}_3\text{O}_2$. Heat near boiling, add a 10% solution of barium acetate dropwise with stirring. Continue heating until the precipitate settles. Test for completeness

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of precipitation by adding 1 to 2 ml of barium acetate solution. Cool, then filter through tared sintered glass crucible and wash with hot water until the excess barium is removed. Dry at 110°C then cool in a desiccator. Weigh as BaCrO_4 . The analysis is performed in quadruplicate. Results should agree to within 1%.

$$\text{Cr(mg/ml)} = \frac{(\text{mg } \text{BaCrO}_4) (0.2053)}{5}$$

- 5.4 Record chemical yield. (Step 4.19).
- 5.5 Determine the area under the 0.32-Mev photopeak (total counts).
- 5.6 Calculate the specific activity of Cr-51 at sample time. Record the results in $\mu\text{c/cc}$ or $\mu\text{c/mg} \pm$ one standard deviation. (See Method 633.3.)

METHOD 1131
IODINE-131 and -133

1. SCOPE

This method is used to determine fission product I-131 and -133 specific activity. Exchange between carrier and radioiodine is assured by the oxidation to IO_3^- with NaClO . Iodine is then effectively separated from other fission products by extraction of molecular iodine into carbon tetrachloride (CCl_4). The separation from other halogens is accomplished by a manipulation of their oxidation states.

2. SAMPLE

Sample aliquots are selected on the basis of the estimated iodine activity. As a general rule, when primary coolant is being analyzed, a 25-ml sample is used. When secondary coolant is being analyzed, a 500-ml sample is used. Samples larger than 500 ml would be difficult to process due to the large separatory funnel required for the initial solvent extraction step.

3. APPARATUS AND REAGENTS

Pipet - 2.5 ml.

Separatory funnels - 500, 250, 125 ml capacity.

Centrifuge tubes - Pyrex - 50 ml capacity.

Graduates - 10, 25, 100, 500 ml capacity.

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Hot plate - 115 volt.

Water bath tray for 50-ml centrifuge tubes.

Filter paper 15/16 in. -Whatman 542.

Desiccator.

Balance - analytical.

Gamma ray multichannel analyzer.

Iodine carrier (10 mg/ml). (Dissolve 13.1 gm of KI in demineralized water and dilute to 1 liter with water).

Bromine carrier (10 mg/ml). (Dissolve 14.9 gm of KBr in demineralized water and dilute to 1 liter with water).

2M Sodium carbonate (Dissolve 213 gm of Na_2CO_3 in demineralized water and dilute to 1 liter).

Sodium hypochlorite solution, 4-6% NaOCl .

Carbon tetrachloride, reagent grade.

Nitric acid (HNO_3), reagent grade.

Hydroxylamine hydrochloride, 10%.

1 M Sodium bisulfite (NaHSO_3), reagent grade. (Dissolve 104 gm of NaHSO_3 in demineralized water and dilute to 1 liter.)

0.1M Silver nitrate (AgNO_3), reagent grade. (Dissolve 16.9 gm of AgNO_3 in demineralized water and dilute to 1 liter.)

Ethyl alcohol, absolute.

Silica gel, 6-16 mesh.

Beaker, 100 ml.

Sintered glass crucibles, fine porosity.

4. PROCEDURE

4.1 Pipet 2 ml of iodine carrier (10 mg/ml) into a separatory funnel.

4.2 Add approximately 2 ml of bromine, hold back carrier.

4.3 Pipet in the sample and make basic with 2M Na_2CO_3 .

4.4 Add 1 ml of NaClO and mix well.

4.5 Add approximately 100 ml of (CCl_4) carbon tetrachloride.

4.6 Acidify with 3 ml of (HNO_3) nitric acid.

4.7 Add 3 ml of 10% ($\text{NH}_2\text{OH} \cdot \text{HCl}$) hydroxylamine hydrochloride and shake the funnel to extract the I_2 into the CCl_4 layer.

4.8 Allow the CCl_4 and water layers to separate and discard the water layer.

4.9 Extract the I_2 from the CCl_4 by shaking with 10 ml H_2O containing three drops of 0.1M NaHSO_3 .

4.10 Allow the CCl_4 and water layers to separate and discard the CCl_4 layer.

4.11 Transfer the water layer to a 50-ml centrifuge tube and add 1 ml of concentrated nitric (HNO_3) acid.

4.12 Add dropwise sufficient 0.1M (AgNO_3) silver nitrate solution to precipitate all the iodine.

- 4.13 Digest the precipitate in a hot water bath for 5 minutes.
- 4.14 Transfer the precipitate to a weighed Whatman 542 filter paper and wash with absolute ethanol.
- 4.15 Dry in a vacuum desiccator.
- 4.16 Weigh filter and AgI to constant weight and determine the chemical yield.
- 4.17 Mount for gamma counting.
- 4.18 Count the sample, using techniques for gamma pulse height analyses given in Method 633.3.

5. RESULTS AND COMPUTATIONS

- 5.1 Record sample time and date.
- 5.2 Record sample aliquot used.
- 5.3 Record standardization data of iodine carrier solution.

NOTE

Standardization of the iodine carrier solution is performed as follows: Pipet a 5.0-ml aliquot into a 100-ml beaker. Acidify with 1 ml concentrated HNO_3 . Add dropwise sufficient 0.1M AgNO_3 solution to precipitate all the I^- . Digest the precipitate on a hot water bath for 10 minutes. Filter the precipitate through a tared fine porosity sintered glass filter crucible. Wash the precipitate with three 5-ml

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portions of H_2O and three 5-ml portions of absolute ethanol. Dry the precipitate for 20 minutes at 100 to 110°C and cool in a desiccator. Weigh to constant weight. The analysis is performed in quadruplicate. Results should agree to within 0.5%.

$$I \text{ (mg/ml)} = \frac{(\text{mg AgI}) (0.5405)}{5}$$

- 5.4 Record chemical yield (Step 4.16).
- 5.5 Determine the area under the 0.530-Mev peak of I-133. (Total counts .)
- 5.6 Determine the area under the 0.364-Mev peak of I-131. (Total counts.)
- 5.7 Calculate the specific activity of I-131 and I-133. Record the results in $\mu\text{C/cc} \pm$ one standard deviation at sample time. (See Method 636.)

METHOD 1132
STRONTIUM - 90

1. SCOPE

This method is used to determine Sr-90 specific activity. The Sr-90 activity is calculated from the count rate of its daughter Y-90.

2. SAMPLE

2.1 The sample can be either a solid or a liquid.

2.2 The sample aliquots are selected on the basis of the estimated Sr-90 activity.

3. APPARATUS AND REAGENTS

Porcelain crucible size OO.

Erlenmeyer flasks - 1 liter, 500 ml, 250 ml, 125 ml.

Furnace, muffle, 0-2000°F.

Meeker burner.

Analytical balance, readable to 0.01 mg.

Pipets - 2-ml capacity.

Hot plate - 115-120 volts with temperature selector.

Ice bath.

Centrifuge for 50-ml tubes - 4-place head.

Centrifuge tubes - 50 ml, Pyrex.

Graduates-500 ml, 250 ml, 100 ml, 10 ml.

Beakers -100 ml.

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Filter tower apparatus - stainless steel -10-ml capacity -

15/16 in. filter paper.

Filter paper - Whatman No. 40 and No. 542 - 15/16-in. diameter.

Vacuum pump-115 volts capable of 14-in. vacuum.

Oven-115 volts 60 cycles, 40-200°C Range.

Lusteroid Tubes 50 ml.

Separatory funnel -125-ml capacity.

Beta counter.

Potassium pyrosulfate, fused, powder, reagent grade.

Hydrochloric acid-4 N. (333 ml of concentrated HCl made up to

1 liter with demineralized water).

Strontium carrier-10 mg/ ml. (Dissolve 32.4 gm of $\text{Sr}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$

in demineralized water and dilute to 1 liter.)

Barium carrier - 10 mg/ml. (Dissolve 19 gm of $\text{Ba}(\text{NO}_3)_2$ in

demineralized water and dilute to 1 liter).

Nitric acid, fuming, reagent grade.

Iron carrier - 10 mg/ml. (Dissolve 48.4 gm of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 1N

HCl and make up to 1 liter with 1N HCl).

Ammonium hydroxide, concentrated, reagent grade.

Nitric acid - 6N (390 ml of concentrated nitric acid made up to 1

liter with demineralized water).

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Ammonium acetate (NH_4Ac) -6M. (Dissolve 231 gm of $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ in 300 ml of demineralized water and dilute to 500 ml.)

Acetic acid - 6M - (344 ml of concentrated acetic acid made up to 1 liter with demineralized water).

Potassium dichromate 1.5M. (Dissolve 110.2 gm $\text{K}_2\text{Cr}_2\text{O}_7$ and dilute to 250 ml.)

Ammonium carbonate (NH_4)₂ CO_3 , saturated.

Ethyl alcohol.

Yttrium carrier 10 mg/ml. (Dissolve 43 gm of $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ in demineralized water, add 5 ml of 6N HNO_3 and dilute to 1 liter.)

Hydrofluoric acid, 48%, reagent grade.

Boric acid (H_3BO_3) saturated solution, reagent grade.

Phenolphthalein (0.5%) indicator.

Tributyl phosphate- benzene - nitric acid solution. (Add 30 ml of tributyl phosphate and 20 ml of benzene to separatory funnel and add 5 ml of nitric acid and shake for 1 minute.)

Hydrochloric acid 6N (495 ml of concentrated HCl made up to 1 liter.)

Ammonium oxalate (NH_4)₂ C_2O_4 , saturated solution, reagent grade.

Sintered glass crucibles, fine porosity.

4. PROCEDURE

- 4.1 Place particulate sample (filter paper or cloth wipe) in a tared porcelain crucible (Cooks OO or No.1). If sample is liquid, accurately

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measure an aliquot and transfer it to Erlenmeyer flask and proceed with Step 4.8.

- 4.2 Ignite over a Meeker burner until completely ashed. (Millipore filter paper is completely ashed in 15 minutes, while cloth wipes generally take about 30 minutes, depending on the size.)
- 4.3 Weigh the crucible containing the ashed sample and determine the total ignited crud weight.
- 4.4 Weigh approximately 2 gm of potassium pyrosulfate ($K_2S_2O_7$) and spread it evenly over the ashed crud.
- 4.5 Heat with a Meeker burner until the flux melts and turns cherry red. Stop fusion at the first sign of SO_3 fumes.
- 4.6 Cool and dissolve the flux with 4N HCl. (Slight heating will be necessary.)
- 4.7 Transfer the dissolved flux to a 125-ml Erlenmeyer flask.
- 4.8 Pipette 2 ml of standardized strontium carrier and add 2 ml barium carrier to the sample.
- 4.9 Evaporate to less than 5 ml.
- 4.10 Cool in an ice bath and add approximately 30 ml of fuming nitric acid.
- 4.11 Cool for approximately 5 minutes.
- 4.12 Transfer the contents to a 50-ml centrifuge tube and centrifuge.

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- 4.13 Discard the supernate.
- 4.14 Dissolve the precipitate in 1-2 ml of demineralized water, heating if necessary.
- 4.15 Cool and slowly add 20 ml of fuming nitric acid.
- 4.16 Cool for approximately 5 minutes.
- 4.17 Centrifuge and discard the supernate liquid.
- 4.18 Dissolve the precipitate in 10 ml of demineralized water and four drops of iron carrier.
- 4.19 Heat nearly to boiling and add concentrated NH_4OH to precipitate the $\text{Fe}(\text{OH})_3$.
- 4.20 Centrifuge and decant the supernate into another 50-ml centrifuge tube.
- 4.21 Record the time. This is (t_0) for Y-90 separation.
- 4.22 Make just acid with 6M HNO_3 .
- 4.23 Add 2 ml of 6M NH_4Ac and 1 ml of 6M HAc .
- 4.24 Heat nearly to boiling and add dropwise 2 ml of 1.5 M $\text{K}_2\text{Cr}_2\text{O}_7$.
- 4.25 Centrifuge and decant the supernate into a 100-ml beaker. Discard the precipitate.
- 4.26 Add 2 ml of concentrated NH_4OH and heat nearly to boiling.
- 4.27 Add 20 ml of saturated $(\text{NH}_4)_2\text{CO}_3$ and allow to cool.
- 4.28 Decant as much of the liquid as possible and use the rest to transfer the SrCO_3 to a weighed Whatman NO. 542 filter paper mounted in a

filter chimney assembly.

- 4.29 Wash the precipitate with about 10 ml of ethyl alcohol.
- 4.30 Dry at 110°C , cool, and weigh to determine the chemical yield for strontium carbonate.
- 4.31 Place the filter disc containing the strontium sample in a 50-ml centrifuge tube, stopper the tube and set aside for 15 days to allow for the growth of Y-90.
- 4.32 At the end of the 15-day growth period, unstopper the tube and add 2 ml of standardized yttrium carrier and 5 ml of fuming nitric acid. Transfer the contents to a 100-ml beaker.
- 4.33 Boil to near dryness, cool, and add 2 ml of fuming nitric acid.
- 4.34 Boil to near dryness and cool.
- 4.35 Repeat the fuming nitric additions until the liquid is clear.
- 4.36 Transfer the sample to a 50-ml lusteroid tube and dilute to 15 ml with water.
- 4.37 Add about 2 ml of HF, centrifuge, and discard the supernate. Record the time of the final Sr-90 separation.
- 4.38 Dissolve the precipitate in 1 ml of H_3BO_3 and 10 ml of water.
- 4.39 Add two drops of phenolphthalein indicator and, using NH_4OH , neutralize to the phenolphthalein end point.
- 4.40 Centrifuge and discard the supernate.
- 4.41 Dissolve the $\text{Y}(\text{OH})_3$ in 2 ml of 6M HCl and dilute to about 10 ml.

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- 4.42 Heat in a water bath and add 20 ml of saturated $(\text{NH}_4)_2\text{C}_2\text{O}_4$.
- 4.43 Heat for 10 minutes and allow to cool to room temperature.
- 4.44 Centrifuge and discard the supernate.
- 4.45 Transfer the precipitate to a filter paper with a minimum amount of water.
- 4.46 Place filter paper and contents in a porcelain crucible.
- 4.47 Heat sample at 800°C in muffle furnace for 1 hour.
- 4.48 Cool, slurry the Y_2O_3 in alcohol, and transfer the Y_2O_3 to a weighed Whatman No. 542 filter paper mounted in a filter chimney assembly.
- 4.49 Dry in a vacuum desiccator and weigh to determine the chemical yield for Y_2O_3 .
- 4.50 Mount for beta counting.
- 4.51 Count the sample in beta counter immediately and once daily for 5 days using techniques for beta counting in Method 633.1.
- 4.52 Plot the decay curve of the corrected gross counting rate on semi-log paper. The principle corrections will be background, standard factor, and coincidence when applicable. A pure sample should give a slope equal to the decay constant of 64.24 hours.
- 4.53 Extrapolate the curve back to the Sr-90 separation time (Step 4.37). This value represents the amount of Y-90 in equilibrium with Sr-90.

4.54 Calculate the specific activity of Sr-90 as outlined in the following section.

5. RESULTS AND COMPUTATIONS

5.1 Record the sample time and date.

5.2 Record the sample aliquots in cc or mg.

5.3 Record standardization data for Y and Sr carriers.

NOTE

a) Standardization of the strontium carrier solution is performed as follows: Pipet a 5.0-ml aliquot into a 150-ml Erlenmeyer flask and add 10 ml 2M Na_2CO_3 solution. Digest on water bath for 15 minutes, cool and filter through a tared fine porosity sintered glass crucible. Wash with water, ethanol, and ether. Dry at 110°C for 10 minutes. Cool in desiccator and weigh to constant weight.

The analysis is performed in quadruplicate. Results should agree to within 0.5%.

$$\text{Sr (mg/ml)} = \frac{(\text{mg SrCO}_3) (0.5941)}{5}$$

b) Standardization of the yttrium carrier solution is performed as follows: Add 20 ml of demineralized water, to 5.0 ml of the carrier solution, heat just to boiling, and

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add 20 ml of saturated $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution with stirring. Heat for 10 minutes on a hot water bath and then cool in an ice bath for 10 minutes. Filter through a Whatman No. 40 filter paper. Wash the precipitate with water, transfer to a tared porcelain crucible, dry under a heat lamp, cover, and ignite at 800°C for 10 minutes. Remove the cover and continue the ignition for 1 hour. Cool in a desiccator. Weigh the crucible to constant weight.

The analysis is performed in quadruplicate. Results should agree to within 1%.

$$Y \text{ (mg/ml)} = \frac{(\text{mg } Y_2O_3) (0.7875)}{5}$$

5.4 Record chemical yield of SrCO_3 (Step 4.3) and Y_2O_3 (Step 4.48)

5.5 Calculate the specific activity of Sr-90 the results in uc/cc or uc/mg $\pm$ one standard deviation at sample time. (Reference Section 600).

$$5.5.1 \text{ Activity of Sr-90 (uc/cc)} = \frac{A_{Y90} \times f(\text{yield}) \times f(\text{growth})}{E \times f(\text{weight}) \times 2.22 \times 10^6 \text{ dpm/uc}}$$

$$\text{or activity of Sr-90 (uc/mg)} = \frac{A_{Y90} \times f(\text{yield}) \times f(\text{growth})}{E \times f(\text{weight}) \times 2.22 \times 10^6 \text{ dpm/uc}}$$

A_{Y90} Activity of Y-90 at end of the Y-90 ingrowth. This value was determined in Step 4.53.

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$$f \text{ (yield)} = \frac{100\% \text{ yield of yttrium carrier}}{\text{Actual yttrium yield}} \times \frac{100\% \text{ yield of strontium carrier}}{\text{Actual strontium yield}}$$

f (volume) = volume of sample analyzed (ml)

f (weight) = weight of sample analyzed (mg)

f (growth) = factor for ingrowth of Y-90. At the end of 15 days, the growth factor approaches one (1) and for all practical purposes is one.

E = Counter efficiency for Y-90.

METHOD 1133
CESIUM-134 and -137

1. SCOPE

This method is used to determine Cs-134 and -137 specific activity in particulate (crud) and liquid samples. Cesium is precipitated with chloroplatinic acid and the specific activity of the individual cesium isotopes is determined by use of their characteristic photopeak (0.97 Mev for Cs-134 and 0.663 Mev for Cs-137).

2. SAMPLE

Sample aliquots are selected on the basis of the total gamma spectrum on which an estimate of the cesium activity is made.

3. APPARATUS AND CHEMICALS

Porcelain crucible-size OO.

Erlenmeyer flasks - 1 liter, 500 ml, 250 ml, and 125 ml.

Graduates-500, 250, 100, 10 ml.

Meeker burner.

Balance - analytical.

Pipets-2 ml and 10 ml capacity.

Centrifuge tubes 50 ml, Pyrex.

Centrifuge for 50-ml tubes - 4-place head.

Ice bath.

Filter tower apparatus-stainless steel - 10-ml capacity -15/16-in filter paper.

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Filter paper - Whatman No. 542 15/16-in. diameter.

Vacuum pump - 115 volts.

Furnace, muffle. 0-2000°F.

Desiccator

Potassium pyrosulfate fused, powder, reagent grade.

Hydrochloric acid - 4N (333 ml of concentrated HCl made up to 1 liter with demineralized water).

Sodium hydroxide - 12N (Dissolve 240 gm of NaOH in 400 ml of demineralized water and dilute to 500 ml.)

Sodium carbonate - 2M (Dissolve 106 gm of Na_2CO_3 in 400 ml of demineralized water and dilute to 500 ml.)

Acetic acid, concentrated, reagent grade.

Hydriodic acid - bismuth triiodide mixture (add 10 gm of BiI_3 to 50 ml of 55% HI).

1:7 mixture of HCl and water (Add 10 ml of 2 M hydrochloric acid to 70 ml of demineralized water.)

Nitric acid, concentrated.

Chloroplatinic acid (Dissolve 2.5 gm of platinum chloride in minimum amount of 2 M HCl and dilute to 25 ml.)

Ethyl alcohol.

Cesium carrier - 10 mg/ml. (Dissolve 12.7 gm of CsCl in demineralized water and make up to 1 liter.)

4. PROCEDURE

- 4.1 Place the particulate sample (filter paper or cloth wipe) in a tared porcelain crucible (Coors OO). If the sample is liquid, accurately measure an aliquot and transfer it to an Erlenmeyer flask and proceed with Step 4.8.

- 4.2 Ignite over a Meeker burner until completely ashed.
- 4.3 Weigh the crucible with ash to determine the total ignited crud.
- 4.4 Weigh out approximately 2 gm of potassium pyrosulfate ($K_2S_2O_7$) and spread it evenly over the ashed crud.
- 4.5 Heat with a Meeker burner until the flux melts and turns cherry red. Stop fusion at the first sign of SO_3 fumes.
- 4.6 Cool and dissolve the flux with 4N HCl (slight heating will be necessary).
- 4.7 Transfer the dissolved flux to a 125-ml Erlenmeyer flask.
- 4.8 Pipet 2 ml of cesium, barium, strontium, and iron carriers and evaporate to near dryness. (See Section 3 of Method 1132 for the preparation of carriers.)
- 4.9 Transfer the sample to 50-ml centrifuge tube with minimum amount of water.
- 4.10 Add 2-3 drops of 12N NaOH. Solution should be basic.
- 4.11 Add approximately 2 ml of 2M Na_2CO_3 .
- 4.12 Centrifuge and discard the precipitate.
- 4.13 Cool all reagents required for Steps 4.14 through 4.18.
- 4.14 Add glacial acetic acid dropwise until the supernate is acid.
(Note evolution of CO_2 .)
- 4.15 Add 1 ml of III- BiI_3 reagent.

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- 4.16 Cool in an ice bath for approximately 5 minutes to precipitate the $\text{Cs}_3\text{Bi}_2\text{I}_7$.
- 4.17 Centrifuge and discard the supernate.
- 4.18 Wash the precipitate with a 1 : 7 cold mixture of 2M HCl and water.
- 4.19 Centrifuge and discard the wash.
- 4.20 Add three drops of 12N NaOH and 5 ml of water to the $\text{Cs}_3\text{Bi}_2\text{I}_7$ precipitate.
- 4.21 Centrifuge and discard the precipitate.
- 4.22 Add concentrated nitric acid until the solution becomes black-brown.
- 4.23 Heat to expel I_2 . The solution should be clear after I_2 is expelled.
- 4.24 Cool the solution in an ice bath and add 6 drops of 10% H_2PtCl_6 .
- 4.25 Add 20 ml of cold ethyl alcohol.
- 4.26 Centrifuge and discard the supernate.
- 4.27 Transfer the precipitate to a weighed filter disc with water.
- 4.28 Wash the precipitate with approximately 10 ml of cold ethanol.
- 4.29 Dry in a vacuum desiccator for about 10 minutes.
- 4.30 Weigh filter and contents and determine the chemical yield.
- 4.31 Mount the sample for gamma ray scintillation counting.
- 4.32 Count the sample using techniques for gamma pulse height analyses given in Method 633.30.

5. RESULTS AND COMPUTATIONS

- 5.1 Record the sample time and date.
- 5.2 Record the sample aliquots in cc or, in the case of a crud sample, the weight, in mg, of crud analyzed.
- 5.3 Record standardization data for cesium carrier. (100% chemical yield.)

NOTE

Standardization of the cesium carrier solution is performed as follows: Pipet a 5.0-ml aliquot into a 50-ml centrifuge tube. Add 5 ml of concentrated HCl. Add 4 ml of 10% chloroplatinic acid. Stir well and then let stand for 10 minutes at 70°C. Filter through a tared fine porosity sintered glass crucible. Wash the precipitate three times with 5-ml portions of 6N HCl and three times with 5-ml portions of absolute ethanol. Dry the precipitate at 100-110°C for 20 minutes. Cool in a desiccator and weigh to constant weight. The analysis is performed in quadruplicate. The results should agree within 0.5%.

$$\text{Cs (mg/ml)} = \frac{(\text{mg Cs}_2\text{PtCl}_6) (0.3945)}{5}$$

- 5.4 Record the actual chemical yield (Reference Step 4.30).

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5.5 Calculate the Cs-134 specific activity from the 0.794-Mev photopeak.

Cs-137 specific activity is calculated from its characteristic 0.663-Mev photopeak. Record the results in uc/cc or uc/mg $\pm$ one standard deviation. (See Method 636.)

METHOD 1141
SUBMITTAL OF SAMPLES FOR OFF-SITE RADIOASSAYS

1. SCOPE

This method specifies the procedure to be followed when samples are shipped off site for radioassay.

2. SAMPLES

The samples can be either solid, (i.e. crud) liquid (i.e. reactor coolant) or gas (i.e. environment air). The samples are collected under the supervision of a health physicist.

3. APPARATUS

Polyethylene collection bottles.

Filter paper - millipore -HA white - 47 mm.

Filter tower assembly.

Vacuum pump.

Oven - 0-100°C.

Balance - analytical.

Desiccator.

Watch glass - ribbed.

Filter paper - Whatman No. 42.

Forceps - 115 mm.

Polyethylene tube - 50 ml.

Rubber stopper - No. 3.

Tape - electrical.

Hydrochloric acid - concentrated.

Hot plate.

Beaker-100, 250, 500, 1000, 2000 ml.

Polyethylene sample bottle - 500 ml.

Paraffin.

Shipping containers, metal - (1-gal. paint cans).

Vermiculite.

Lead solder.

Shipping labels for radioactive material.

Gas cylinder - 500-ml stainless steel - pressure tested for 1000
psi. (Can be obtained from Oak Ridge National Laboratory.)

Data sheet.

4. PROCEDURES

4.1 Liquid samples (Reference Army Test Procedure 203).

4.1.1 Dry several millipore type HA filter (47 mm) in an oven
at 100-105°C for 2 hours. To prevent curling, place the
filter paper on a watch glass and put a ribbed watch glass on top of each
filter.

4.1.2 Remove filters from the oven, and cool in a desiccator to
room temperature.

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- 4.1.3 Weigh the filter to the nearest 0.1 mg.
- 4.1.4 Continue the drying cycle until weight change is less than 0.1 mg.
- 4.1.5 Record the final weight on appropriate data sheet.
- 4.1.6 Collect the liquid sample in a polyethylene bottle.
- 4.1.7 Filter the sample through the pre-weighed filter. All filtering must be performed in a fume hood.
- 4.1.8 Remove the millipore filter with forceps and place it on a Whatman No. 42 filter paper.
- 4.1.9 Roll up the wet millipore filter in the Whatman filter and insert in a polyethylene test tube.
- 4.1.10 Insert rubber stopper, seal with tape and label sample by reactor, sample location, time and date of sample, volume of water filtered, and contact radiation reading.
- 4.1.11 Add to the filtered water sample 20 mg each of standard carriers. The type of carriers will depend on the analyses requested.
- 4.1.12 Record the carriers added, ml, and standardization on the data sheet.
- 4.1.13 Spike the sample with concentrated hydrochloric acid (1 ml of acid per 25 ml of sample).

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- 4.1.14 Evaporate, water samples of more than 500 ml to approximately 400 ml. Record the initial and final volume.
- 4.1.15 Transfer the water sample to a clean polyethylene bottle.
- Insure that an air blanket is maintained over the liquid to allow for expansion during transportation.
- 4.1.16 Seal the cap with paraffin and label the sample for identification and contact radiation readings.
- 4.1.17 Complete the appropriate data sheet in duplicate. (For primary coolant samples use USAERG Form 36). Send one copy with the sample; the other copy is for the plant files.
- 4.1.18 Place the samples and data sheet in an appropriate sized metal can (i. e., paint can) and fill the can with vermiculite.
- 4.1.19 Seal the lid on the can, using lead solder.
- 4.1.20 Monitor the shipping container for compliance with shipping regulations.
- 4.1.21 Verify that the shipment has the prior approval of recipient and that he is informed of all hazards.
- 4.1.22 Verify that the recipient has the appropriate license and can handle the material to be shipped.
- 4.1.23 Apply the appropriate radioactive material and shipping labels.

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- 4.1.24 Obtain a release for shipment from the health physicist and the officer-in-charge.
- 4.2 Solid samples (i.e., soil, biota, smears, or swipes).
 - 4.2.1 Collect the solid sample as directed by the health physicist.
 - 4.2.2 Transfer the sample to an appropriate sized polyethylene container.
 - 4.2.3 Label the sample as to sample location, date and time sampled, and type of sample.
 - 4.2.4 Complete the appropriate data sheet and enclose one copy with the sample.
 - 4.2.5 Follow Step 4.1.17 through 4.1.23 for preparation and handling of shipping container.
- 4.3 Gaseous samples (i.e., environmental gaseous samples).
 - 4.3.1 Set up the air pump in the area to be sampled.
 - 4.3.2 Attach the 500-ml gas cylinder to the discharge of the pump.

NOTE

A vacuum hose attached to the suction side of the pump may be used for sampling in confined areas.

- 4.3.3 Start pump and pressurize to 100 psi.
- 4.3.4 Record the pressure and contact radiation reading on the gas collection cylinder.

- 4.3.5 Check cylinder for surface contamination, using smear sample techniques.
- 4.3.6 Complete the appropriate data sheet.
- 4.3.7 Place gas cylinder and copy of data sheet in a fiberboard box.
- 4.3.8 Pack tightly with paper wadding.
- 4.3.9 Follow Steps 4.1.19 through 4.1.23 for shipping preparation.
- 4.4 Bioassay samples (i.e., urine, feces).
 - 4.4.1 Collect the samples for bioassay as outlined in sub section 1151.
 - 4.4.2 Prepare the samples as directed by the medical laboratory under contract to do the analysis.

5. RESULTS AND COMPUTATIONS

Complete the required data sheets.

6. TEST METHOD IMPLEMENTATION

After radioactive material has been placed in its shipping container, the container is checked for outside radiation to ensure that the package meets Interstate Commerce Commission, Civil Aeronautics Board, and Coast Guard regulations. These regulations specify that there must be no more than 200 mrem/hr of radiation at the surface of the container and no more than 10 mrem/hr at one meter from the surface of the package.

PART II. WATER CHEMISTRY

Safe and reliable operations are prime requirements for a nuclear reactor; radioactivity from the nuclear core and the corrosive nature of water and gases dissolved in the water create significant interrelated problems. Careful design, proper selection of construction materials, definition of exacting water specifications, and water treatment procedures minimize any detrimental effects to plant components.

This Section contains water chemistry procedures necessary for the performance of required test and the protection of the Process Control Specialist during operation and maintenance of a nuclear power plant.

SECTION 1200 - ANALYTICAL PROCEDURES

This Section presents the analytical procedural methods required for routine process control and radiochemical analyses. Sample collection and handling techniques are of prime importance for the accurate and meaningful interpretation of the resulting analytical data. Samples must be representative of the systems from which they are taken; this is assured by either recirculation of the liquid to be sampled or by flushing of the sample lines with a volume of the liquid that is greater than the volume of the sample.

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METHOD 1211

DETERMINATION OF ALUMINUM IN WATER

1. SCOPE

This method is used to determine the aluminum content of various plant system fluids as a means of measuring the amount of corrosion occurring.

2. SUMMARY OF METHOD

This sensitive method for aluminum is based on the formation of adsorption compounds (lakes) between aluminum and certain dyes. The color is stable but should be measured within 1 day. Several elements, particularly iron, give similar colorations. Interfering elements besides iron include: beryllium, zirconium, thallium, scandium, gallium, rare earths, zinc, titanium, and chromium.

Aluminum can be separated from the above interfering elements by loading on an anion exchange resin in the citrate form, followed by elution with 10 M hydrochloric acid. The eluate containing the aluminum is evaporated to dryness to remove hydrochloric acid and the aluminum is determined colorimetrically by use of Eriochrome Cyanine R. The range of the method is 0 -5 μ gm aluminum.

NOTE

Traces of aluminum can be introduced by attack of the glass containers. Also, many reagents contain traces

of aluminum. Therefore, it is necessary to carry a
blank of deionized water through the entire procedure.

3. APPARATUS

3.1 Small ion-exchange columns, (Figure 1211-1 having a resin capacity of 5 ml and a reservoir (liquid) capacity of 12 ml, are filled with Dowex AG-1-X10, 100-200 mesh resin in the chloride form. The column is rinsed with water until the eluate is neutral to pH paper. Pass two reservoir volumes of 50% citric acid through the column. This column is now ready for use.

3.2 A second column is prepared in a similar manner except that 10 M hydrochloric acid is used instead of 50% citric acid.

3.3 Separatory funnels (Figure 1211-1) (125-ml capacity) are fitted with a one-hole stopper to provide an airtight connection between the delivery stem of the separation funnel and the reservoir of the ion-exchange column.

3.4 100-ml beakers

3.5 5-ml pipets

3.6 50-ml volumetric flasks

3.7 Spectrophotometric or colorimeter

4. REAGENTS

4.1 HCL, 10N: Dilute 855 ml of concentrated HCl to a volume of 1 liter

by adding deionized water.

4.2 Citric acid solution, 50%: Dissolve 50 gm citric acid in 100 ml of water.

4.3 Eriochrome Cyanine R, 0.035% solution: Place 0.3500 gm of Eriochrome Cyanine R in a dry 250 ml beaker. Add 2 ml of dilute (1 : 1) HNO_3 and swirl at room temperature until the orange-red dye is completely dissolved. Add 75 ml water, then 0.25 urea. Swirl to dissolve; dilute to 1 liter.

4.4 Buffer solution, pH 7.0, 32%. Dissolve 32 gm of ammonium acetate in 100 ml H_2O and adjust pH to 7.0 using acetic acid or ammonium hydroxide.

5. PROCEDURE

5.1 Attach the separatory funnel to the reservoir of the prepared ion-exchange column (citrate-form resin) and add 100 ml of the water sample to the separatory funnel.

5.2 Allow the water to pass through the resin bed. Rinse the separatory funnel with 20 ml of deionized water and allow same to flow through the ion-exchange column.

5.3 This column (No.1) contains the aluminum. Place it above the column containing the resin in the chloride form No. 2 so that the eluate from Column 1 passes into the reservoir of Column 2.

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5.4 Place a clean 100-ml beaker under Column 2. Fill the reservoir of Column 1 with 10 N hydrochloric acid. When this solution has passed through both Columns, refill the reservoir of Column 1 with 10 N hydrochloric acid and allow to drain through Column 2 into the beaker. Cover the beaker with a Speedevap lid and evaporate to dryness.

5.5 Add 25 ml deionized water.

5.6 Pipet 5 ml of Eriochrome Cyanine R reagent, wait 2 minutes and pipet 5 ml of buffer solution.

5.7 Adjust the pH to 5.8 (use pH-Hydrion paper) with acetic acid or ammonium hydroxide.

5.8 Transfer the sample quantitatively to a 50-ml volumetric flask. Dilute to volume with deionized water and mix. Allow at least 7 minutes for the color to develop.

5.9 Read the absorbance at 535 m μ (or use a green filter). The reference solution consists of 100 ml of deionized water that has been carried through the entire procedure.

6. CALIBRATION

Prepare a graph of absorbance versus micrograms Al per ml (ppm) by carrying standard 0, 1, 3, 5, 10, and 15 μ gm samples (or ml of working standard) of Al in 100 ml (0, 0.01, 0.03, 0.05, 0.10 and 0.15 ppm, respectively) through the entire procedure. The Al standards are prepared by dissolving 0.100 gm of high purity aluminum in a minimum of dilute (1:1)

hydrochloric acid and diluting to 1 liter with water. Pipet 10 ml of this solution and dilute to 1 liter with water to obtain a working standard solution containing 1 ppm (1 ~~mg~~gm per ml).

7. CALCULATIONS

Using the calibration curve prepared in Section 3.1.3 above and the absorbance measured for the sample, obtain the concentration of aluminum in the sample.

Obtain the aluminum concentration of the blank using the calibration curve above and the absorbancy determined for the blank.

Calculate the aluminum concentration of the sample using the following equation:

$$C = A_s - A_b$$

Where C = true concentration of a aluminum in sample, ppm

A_s = concentration of aluminum in sample, ppm

A_b = concentration of aluminum in blank, ppm

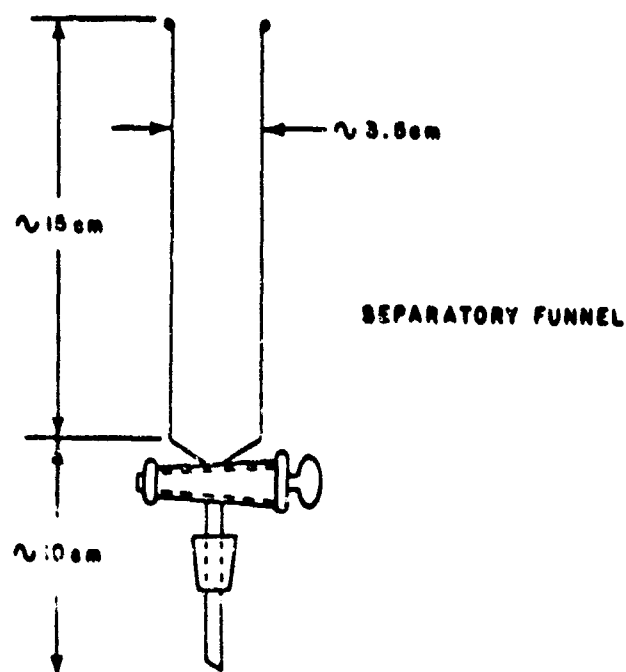
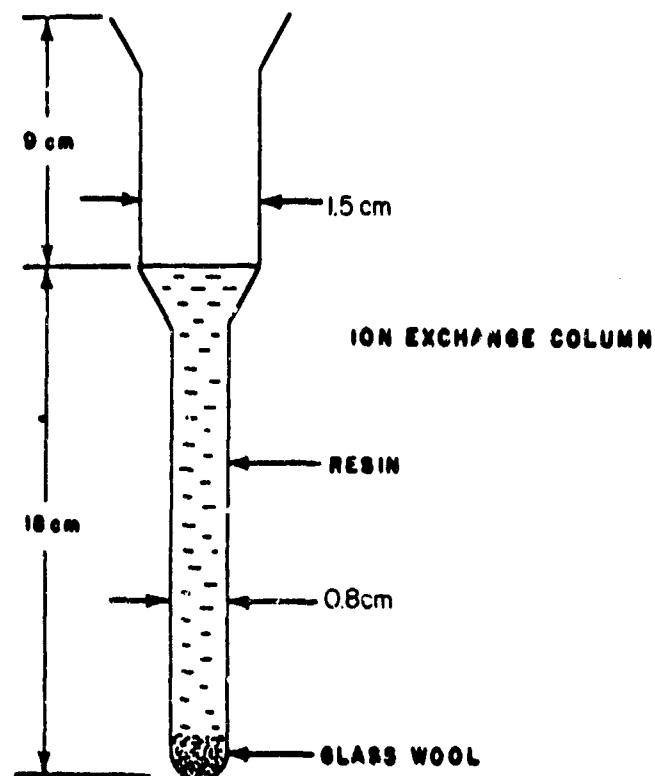


Fig. 1211-1 ION EXCHANGE COLUMN AND SEPARATORY FUNNEL.

METHOD 1212

DETERMINATION OF CHLORIDE CONTENT IN WATER

1. SCOPE

This method is used to determine the chloride content of makeup water and the water present in various plant systems.

2. SUMMARY OF METHOD

Chloride is determined by titration with standard mercuric nitrate to form mercuric chloride complexes. Diphenylcarbazone is used as an indicator to detect excess mercuric ion at the end point. The reaction occurs quantitatively only at a pH of 3.0 to 3.5. If the chloride content is below 1 ppm the analysis becomes inaccurate unless the mercuric nitrate is standardized in the low chloride range. Care must be taken in the washing of glassware, particularly that used with concentrated HCl in other procedures.

3. APPARATUS

1 and 2 ml pipets

2 - 250 ml Erlenmeyer flasks

pH paper

Buret

2-100 ml graduated cylinders

4. REAGENTS

4.1 Standard mercuric nitrate, 0.025 N for 1 ppm chloride and above.

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4.2 Dissolve 4.3 gm $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ in 50 ml warm water containing 1 ml of dilute (1 : 1) HNO_3 . Cool, filter into a volumetric flask, and dilute to 1 liter with H_2O .

4.3 Standard chloride solution, 500 ppm Cl.

4.4 Dissolve 0.8242 gm of NaCl in H_2O and dilute to 1 liter in a volumetric flask.

4.5 Mixed chloride indicator.

4.6 Add ethanol to a mixture of 0.5 gm diphenylcarbazone and 0.05 gm bromphenol blue. Dilute with ethanol to a total volume of 100 ml in a volumetric flask. Store in a brown bottle. Discard after 6 months.

4.7 Dilute nitric acid, 0.5 N.

4.8 Add H_2O to 33 ml HNO_3 to make a final volume of 1 liter.

4.9 Hydrogen peroxide, 30%.

5. STANDARDIZATION OF MERCURIC NITRATE

5.1 Standardization of 0.010 N Mercuric Nitrate:

Prepare a solution containing 5 ppm of chloride by diluting 1 ml of standard solution containing 500 ppm of chloride to 100 ml in a volumetric flask. Pipette a 2-ml portion of this solution into another 100 ml volumetric flask and dilute to mark thus giving a solution containing 0.1 ppm of chloride. Analyze the latter solution, in triplicate for chloride. Analyze a blank of 100 ml demineralized water.

5.2. Standardization of 0.025 N Mercuric Nitrate:

Pipet 5 ml of 500 ppm standard chloride solution into a 100 ml volumetric flask and dilute to exactly 100 ml with demineralized water. This solution will contain 25 ppm chloride. Analyze this solution in triplicate for chloride. Analyze a blank of 100 ml demineralized water.

6. PROCEDURE

6.1 Transfer a 100-ml water sample from a graduate to a 250-ml Erlenmeyer flask.

6.2 Add 3 drops of 35% H_2O_2 to eliminate sulfite.

6.3 Add 10 drops of mixed chloride indicator. Using pH paper, adjust to pH 3.0 - 3.5 with 0.5N HNO_3 added dropwise.

6.4 Titrate the yellow acidified solution with standard mercuric nitrate until a light blue-violet color is obtained. The end-point is first detected as a purple coloration in surface bubbles. (Alternatively, the titration may be carried on the neutral point in which a colorless solution results. Whichever end point is selected, the blank must be run similarly.)

6.5 Determine a blank, by repeating the titration using 100 ml of demineralized water.

7. CALCULATIONS

7.1 $\text{N Mercuric Nitrate} = \frac{(\text{ppm chloride analyzed}) (100 \text{ ml})}{(\text{S-B}) (35, 460)}$

7.2 Chloride content of sample:

$$\text{ppm chloride} = \frac{(S-B) (N) (35,460)}{(V)}$$

Where S = volume of standard mercuric nitrate, required
for sample, ml.

B = volume of standard mercuric nitrate, required
for blank, ml.

N = normality of standard mercuric nitrate.

V = volume of sample analyzed, ml.

8. INTERFERENCES

Nickel, iron, and chromium ions affect the end-point color but do not reduce the accuracy of the method if these ions are present to the extent of less than 100 ppm. Sulfite interferes with the end-point and must be oxidized to sulfate by the addition of hydrogen peroxide.

9. PRECISION AND ACCURACY

The precision of the method is 0.1 ppm or 2% of the chloride content of the sample; the accuracy is approximately equal to the precision in the absence of interferences.

METHOD 1213

DETERMINATION OF IRON IN WATER

1. SCOPE

This method is used to determine the iron content of water in the event corrections are required for the iron present in samples used for other analyses, or if a specific iron determination is required.

2. SUMMARY OF METHOD

Ferrous ions yield a red complex with o-phenanthroline; the color is stable. Many metals, such as Al, Cd, Zn, Hg, Mo, W, Cu, Cr, Ni, and Co, can interfere if present in more than trace quantities. Phosphates should also be absent. All of these impurities, excepting Mo, can be removed by an ion-exchange technique.

3. APPARATUS

Hot plate.

600-ml beaker.

100-ml beakers.

Buret.

Colorimeter equipped with 508 m μ (green filter).

4. REAGENTS

4.1 Concentrated HCl.

4.2 4.5 M HCl.

4.3 Dilute 96 ml of concentrated HCl to 250 ml with deionized water.

4.4 0.5 M HCl.

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- 4.5 Dilute 11 ml of concentrated HCl to 250 ml with deionized water.
 - 4.6 Resin.
 - 4.7 Dowex AG-1-X10, 100-200 mesh.
 - 4.8 O-phenanthroline.
 - 4.9 Dissolve 0.100 gm o-phenanthroline in 100 ml of deionized water.
 - 4.10 Hydroxylamine hydrochloride, ($\text{NH}_2\text{OH} \cdot \text{HCl}$) 10%.
 - 4.11 Dissolve 10 gm $\text{NH}_2\text{OH} \cdot \text{HCl}$ in 100 ml of deionized water.
5. PROCEDURE
- 5.1 Add 5 ml of concentrated HCl to a 100 ml water sample. Evaporate to dryness.
 - 5.2 Prepare an ion exchange column by placing about 5 ml of Dowex AG-1-X10, 100-200 mesh resin in a small buret or ion exchange column. Convert the resin to the chloride form by passing 25 ml of 4.5 M HCl through the resin. The resin column is now ready to be used.
 - 5.3 Dissolve the evaporated sample in several ml of 4.5 M HCl; add the sample to the column. Drain into a beaker.
 - 5.4 Rinse the beaker three times with 5 ml volumes of 4.5 M HCl and add to the resin column.
 - 5.5 Elute the iron from the resin by adding three 10-ml volumes of 0.5 M HCl; collect the product in a clean beaker.
 - 5.6 Cover the beaker with a speed-evap lid and evaporate to dryness.

- 5.7 Add 50 ml of deionized water to dissolve the sample.
- 5.8 Add 2 ml of 10% hydroxylamine hydrochloride solution.
- 5.9 Adjust the solution to pH 3-5 with NaOH or acetic acid.
- 5.10 Pipet 10 ml of the o-phenanthroline solution to the sample; transfer to a 100 ml volumetric flask and dilute to volume with deionized water.
- 5.11 Wait for 5 minutes and read the absorbance at 508 m μ (green filter).

For reference, carry a blank of deionized water through the procedure.

6. CALCULATIONS

Determine the iron content from a standard curve that is prepared by analyzing a series of standard iron solutions, using Steps 5.8 through 5.11.

METHOD 1214

DETERMINATION OF SILICA IN WATER

1. SCOPE

This method is used to determine the silica content of the steam generator blowdown and, in rare instances, that of the makeup water.

2. SUMMARY OF METHOD

The silica content of water is determined by measuring the molybdenum blue color developed by reduction of the silicomolybdate complex with sodium sulfite.

3. APPARATUS

Polyethylene bottles.

Plastic graduated cylinders.

Teflon beakers, 100 ml.

Colorimeter with 650 m μ -A filter.

Plastic funnels for filtering.

4. REAGENTS

4.1 Standard sodium silicate, 100 ppm SiO₂.

4.2 Dissolve 0.4732 gm of sodium silicate (Na₂SiO₃ · 9H₂O) in water and dilute to 1 liter. Plastic ware should be used where possible and polyethylene bottles should be used for storage.

4.3 Dilute HCl.

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- 4.4 Dilute 2 ml of concentrated HCl to 100 ml with deionized water.
- 4.5 Ammonium molybdate reagent, 5%.
- 4.6 Dissolve 50.0 gm of ammonium molybdate $(\text{NH}_4)_2\text{MoO}_4$ in 500 ml of warm water and cool to room temperature. Dilute with water to 1 liter.
- 4.7 Sodium sulfite.
- 4.8 Dissolve 170 gm of sodium sulfite (Na_2SO_3) in 500 ml of deionized water and dilute to 1 liter.

5. CALIBRATION

- 5.1 Prepare a 2 : 10 dilution of the stock silica standard using deionized water. This is the working standard.
- 5.2 Pipet out 1, 2, 4, 6, 8, and 10 ml aliquots of the working standard into teflon beakers. Dilute to 10 ml with deionized water and carry each standard through the procedure described below. These standards contain 2, 4, 8, 12, 16 and 20 ppm respectively.
- 5.3 Prepare a calibration curve of % transmission versus concentration of the silica standards.

6. PROCEDURE

- 6.1 Pipet 10 ml of a filtered sample of secondary water into a 100-ml teflon beaker.
- 6.2 Add 10 ml of dilute HCl to the sample and mix well.

- 6.3 Add 10 ml of ammonium molybdate solution and mix well.
- 6.4 Let stand for 1 minute.
- 6.5 Add 20 ml of sodium sulfite solution and mix well.
- 6.6 Let stand exactly 1 minute after adding the sulfite solution. Determine the transmission using a 650 ~~nm~~ -A filter.
- 6.7 The silica content of the sample in ppm is obtained from the calibration curve prepared above.

7. INTERFERENCES

7.1 Germanium (IV), phosphorus (V) or arsenic (V) give the same reaction and therefore interfere directly. In the presence of oxalic acid (3 ml of a 10% solution) citric or tartaric acids (4 ml of a 10% solution) the coloration due to phosphates is less sensitive and up to 5 parts phosphorus (V) to 1 part silica (IV) may be tolerated. Barium, bismuth, lead, and antimony produce turbidity. Lead, iron (III), and titanium (IV) interfere at concentrations greater than 10 ppm. Other common ions (except germanium, phosphorus and arsenic) may be tolerated up to 50 ppm. High concentrations (0.5 - 1 M) of alkali metal salts prevent maximum color development.

7.2 Aluminum, zinc, and iron may be held in solution as complexes by use of ammonium tartrate. Phosphate ions do not interfere when the pH is adjusted to 4.2 - 6.8.

8. ACCURACY

The accuracy of the results is $\pm 2\%$.

METHOD 1215

DETERMINATION OF SULFITE IN WATER

1. SCOPE

This method is used to determine the sulfite content of water, particularly steam-generator blowdown.

2. SUMMARY OF METHOD

Sulfite is determined by reduction of iodine and titration of the excess iodine with standard thiosulfate solution.

3. APPARATUS

1-liter volumetric flask.

250-ml Erlenmeyer flasks.

50-ml buret.

4. REAGENTS

4.1 Approximately 0.025 N iodine. Dissolve 75 gm potassium iodide,

KI, in 60 ml water, add 1.6 gm iodine, I_2 , dilute to 500 ml with water; store in an amber bottle.

4.2 Standard sodium thiosulfate, 0.0250 N. Dissolve 6.2 gm sodium

thiosulfate, $Na_2S_2O_3 \cdot 5H_2O$, and 0.1 gm sodium carbonate, Na_2CO_3 , in a volumetric flask and dilute to 1-liter (using water previously boiled for at least 30 minutes and cooled to room temperature in a sealed polyethylene bottle). Standardize as follows: Weigh out triplicate samples (approximately 40 mgm each) of potassium iodate (KIO_3) accurately to 0.1

mgm and transfer to 250-ml Erlenmeyer flasks. Dissolve in about 25 ml water and add 2 gm potassium iodide, KI, and 10 ml of 1 N sulfuric acid, H₂SO₄. Titrate with the 0.0250N sodium thiosulfate to be standardized using a 50-ml buret. Add 10 drops of starch indicator when the solution becomes a pale yellow and continue the titration to the disappearance of the blue color. The normality of the thiosulfate (N) is calculated as follows:

$$\underline{N} = \frac{1000W}{35.66 (S)}$$

S = volume of sodium thiosulfate required, ml.

W = weight of KIO₃, gm.

4.3 Sodium thiosulfate solutions may decompose on standing as evidenced by the formation of milky colloidal sulfur. The use of boiled water, free of dissolved O₂, CO₂, and bacteria, provides a stable reagent. If the standard thiosulfate solution becomes cloudy, discard and prepare a new standard solution.

4.4 Starch indicator.

4.5 Grind 1 gm of soluble starch and 1 mg mercuric iodide HgI₂ with water in a mortar to form a paste. Pour the paste into 100 ml of boiling water, boil 2 minutes, cool, and pour off clear liquid into a bottle.

4.6 Glacial acetic acid.

5. PROCEDURE

5.1 Pipet 5 ml of 0.0250N iodine into a 250-ml Erlenmeyer flask.

- 5.2 Add 5 ml glacial acetic acid.
- 5.3 Using a graduated cylinder, measure a 100-ml water sample and place in a flask.
- 5.4 Titrate promptly with 0.0250N sodium thiosulfate until the solution is a pale yellow color. Add 10 drops starch indicator and continue titration to a colorless end point.
- 5.5 Repeat using 100 ml demineralized water as a blank.

6. CALCULATIONS

$$\text{ppm SO}_3 = \frac{(B-S) (1000) (N) (40.03)}{V}$$

Where S = volume of thiosulfate required for sample, ml

B = volume of thiosulfate required for blank, ml

N = normality of thiosulfate

V = volume of the sample, ml

7. INTERFERENCES

Any oxidizable substance such as organic matter, thiosulfates, sulfides, and nitrites produce high results.

8. ACCURACY

The accuracy of the results is $\pm 1\%$.

METHOD 1216

DETERMINATION OF THE PHOSPHATE CONTENT OF WATER

1. SCOPE

This method is used to determine the phosphate content of the secondary system steam-generator blowdown.

2. SUMMARY OF METHOD

This method is based on the yellow phosphovanadomolybdate complex formed when ammonium vanadate is added to phosphomolybdic acid. The color has an absorption maximum at 460 m μ and is stable for several weeks.

3. APPARATUS

Colorimeter and 425-B filter

Cuvettes for colorimeter

Pipets 1, 5, 10, 25 ml

100-ml volumetric flasks

4. REAGENTS

4.1 Standard Phosphate Solution: 1000 ppm = 1000 mgm/liter = 1 mgm/ml.

Add water to 1.433 gm monobasic KH_2PO_4 , or 1.834 gm dibasic potassium phosphate K_2HPO_4 ; dilute to 1 liter.

4.2 Dilute (1 : 1) nitric acid - one part concentrated nitric acid, HNO_3 is mixed with one part by volume of water.

4.3 5% ammonium molybdate solution - add water to 50 gm ammonium

molybdate, $(\text{NH}_4)_2\text{MoO}_4$, to make a total volume of 1 liter.

4.4 0.25% ammonium vanadate solution - dissolve 2.5 gm ammonium vanadate, NH_4VO_3 , in 500 ml of hot water, cool, add 20 ml of nitric acid, and dilute to 1 liter.

5. CALIBRATION

Prepare a set of working phosphate standards by pipeting 1, 5, 10, 25, and 30 ml of standard phosphate solution (1000 ppm PO_4) into 100-ml volumetric flasks and carrying a sample of each diluted solution through the analytical procedure. Thus, the working standards correspond to 10, 50, 100, 250, and 300 ppm of phosphate.

6. PROCEDURE

- 6.1 Pipet 10 ml of filtered water into a 100-ml volumetric flask. A blank analysis using demineralized water should be run simultaneously.
- 6.2 Add demineralized water to make total volume 30 to 40 ml.
- 6.3 Add 1 ml of dilute (1 : 1) nitric acid to flask and mix.
- 6.4 Pipet 10 ml of ammonium molybdate solution into flask and mix.
- 6.5 Pipet 10 ml ammonium vanadate solution into flask and mix.
- 6.6 Dilute to mark with demineralized water and mix.
- 6.7 Allow color to develop for 10 minutes.
- 6.8 Transfer aliquots of the unknown sample and blank to clean cuvettes. Inset the blank into the colorimeter fitted with the 425-B

filter and adjust the instrument to read 100% transmission. Replace the blank with the unknown; read percent transmission.

7. CALCULATION

Construct a calibration curve using the data obtained for the standards. Plot the percent transmission versus ppm of phosphate in the standard using linear graph paper.

Using the calibration curve, obtain the ppm of phosphate in the unknown corresponding to the observed transmission.

8. INTERFERENCES

Silicon gives a yellow silicomolybdate complex; however, 1 part silicon (IV) for 1 part phosphorus (V) may be tolerated. Iron (III) in excess of 200 ppm interferes by producing a yellow color, but may be compensated for by using a properly diluted aliquot of the sample solution in the reference cell. Compounds which precipitate interfere by carrying down phosphorus. These include zinc, niobium, tantalum, titanium, zirconium, tungsten, and vanadium. Copper (II) and nickel ions change the color of the solution, but as much as 1000 ppm of these ions can be tolerated. Sulfide ion (II), thiosulfate and the cyanate, if present in excess of 100 ppm, interfere either by reducing the phosphovanadomolybdate complex or the excess of molybdate to molybdenum blue. Chloride ions inhibit the development of the color; the chloride concentration should not exceed 50 ppm.

9. ACCURACY

The accuracy of the results is $\pm 2\%$.

METHOD 1221

DETERMINATION OF TOTAL SOLIDS IN WATER

1. SCOPE

This method is used to determine the total solids content of water.

2. SUMMARY OF METHOD

The total solids are determined by evaporating a sample of water in a pre-weighed dish and obtaining the weight of the solids plus dish. The total solids content is obtained by difference.

3. APPARATUS

Hot plate.

Balance.

Porcelain evaporating dish.

Drying oven

4. REAGENTS

None required.

5. PROCEDURE

5.1 Clean a porcelain evaporating dish by washing with demineralized water; heat the dish at $105 - 110^{\circ}\text{C}$ for 4 hours and cool in a desiccator to room temperature. (See note.) Handle dish only with tongs to avoid grease from fingerprints. Determine weight of dish to nearest 0.1 mgm; repeat until a constant weight is obtained, i.e. until two consecutive

weighings are within 0.5 mgm of each other.

5.2 Pipet a 100-ml water sample into the dish and evaporate slowly over low heat almost to dryness. Pipet another 100-ml sample into the same dish and evaporate almost to dryness over low heat. Keep dish covered with watch glass during the procedure to protect from dust or particles.

5.3 When the sample is almost dry, transfer to the oven and continue drying for 4 hours at 179° - 181° C to decompose the hydrated material. Cool in a desiccator to room temperature. (See note.)

5.4 Determine weight of dish plus residue to nearest 0.1 mgm.

5.5 Repeat Steps (3) and (4) until two consecutive weighings are within 0.5 mgm of each other (constant weight).

6. CALCULATIONS

$$\text{Total solids, ppm} = \frac{100 (W_2 - W_1)}{V}$$

W_1 = initial weight of dish, mgm

W_2 = final weight of dish, mgm

V = volume of sample, ml

7. INTERFERENCES

There are no interferences.

8. ACCURACY

The accuracy of the results is $\pm 2\%$.

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NOTE

The temperature of the dish and the balance should
be the same to avoid air currents due to temperature
differentials which will result in erratic weighings.

METHOD 1231

DETERMINATION OF THE AMMONIA CONTENT OF WATER

1. SCOPE

This method is used to determine the ammonia content of various water samples.

2. SUMMARY OF METHOD

Ammonia and ammonium salts give an orange color with Nessler's reagent. The transmission of the solution to light with a wave length of 425 microns is measured in a colorimeter and compared with standard solutions containing ammonium ions.

3. APPARATUS

Colorimeter.

Cuvettes for colorimeter.

50 ml volumetric flasks.

Pipets.

4. REAGENTS

4.1 Nessler's reagent: K_2HgI_4

Mix 45.5 gm of mercuric iodide with 35 gm of potassium iodide and a little water; shake until just dissolved. After dissolution, add 112 gm of potassium hydroxide and dilute to 1 liter of water. Store in a polyethylene bottle.

4.2 Standard ammonia solution, 2 ppm NH_4^+ :

Prepare a standardized solution of ammonium chloride by dissolving 5.933 gm of ammonium chloride in 1 liter of water. Pipet exactly 1 ml of this solution into a 1-liter volumetric flask and dilute to the mark with water. (A standardized solution of ammonium hydroxide may be used as the standard stock solution.)

5. PROCEDURE

Ammonia and ammonium salts give an orange color with Nessler's reagent. Colorimetric determinations can be made over a range of 0.1 - 2 ppm NH_4^+ at 425 m μ

5.1 Pipet 40 ml of the sample into a 50-ml volumetric flask.

5.2 In the event a 40 ml sample is too concentrated, a smaller sample is to be taken. Then, dilute to a volume of approximately 40 ml with deionized water.

5.3 Pipet 4 ml of reagent into the flask and cool to 25°C. Dilute to the mark with water and mix.

5.4 Wait 30 minutes, read absorption at 425 m μ and 25°C (or blue filter), using for reference a sample of water carried through the above procedure.

6. CALCULATION

Read NH_4^+ concentration from a curve prepared by carrying standard NH_4^+ solutions through the above procedure.

If analysis indicates ammonia is out of range, dilute sample to bring analysis within range and repeat procedure.

NOTE

If hydrazine is present, high values will be obtained for the ammonia concentration. Samples should not be taken for ammonia analysis shortly after an addition of hydrazine to the primary system.

7. INTERFERENCES

Aliphatic or aromatic amines interfere by producing a yellow color. An interfering turbidity is produced by aldehydes, acetone, and alcohols among other organic compounds. Ions insoluble in alkaline solutions or producing precipitates with iodine or mercury cause turbidity. The principal interfering ions include Mg, Mn, Ca, Fe, and sulfide.

8. ACCURACY

The accuracy of the results is $\pm 2\%$.

METHOD 1232

DETERMINATION OF CARBON DIOXIDE IN WATER

1. SCOPE

This method is used to determine the carbon dioxide content of water.

2. SUMMARY OF METHOD

The carbon dioxide content of water is determined by titration of the carbon dioxide with sodium hydroxide to yield sodium bicarbonate.

The end point (pH 8.35) is determined by the color change of phenolphthalein from colorless to pink.

3. APPARATUS

Buret

250 ml Erlenmeyer flask

4. REAGENTS

4.1 Phenolphthalein

4.2 0.05 gm phenolphthalein in 50-ml ethyl alcohol; dilute to 100 ml with deionized water.

4.3 0.02 N sodium hydroxide

4.4 Dilute 5.0 ml of 1 N sodium hydroxide to 250 ml with deionized water.

5. PROCEDURE

5.1 Add 2 drops of phenolphthalein indicator to a 100 ml water sample .

5.2 Titrate to a pink color with 0.02 N NaOH.

5.3 Repeat procedure with deionized water to obtain a blank determination.

6. CALCULATIONS

$$\text{ppm CO}_2 = \frac{(S-B) (N) (4.4 \times 10^4)}{V}$$

Where S = volume NaOH required for sample, ml

B = volume NaOH required for blank, ml

N = normality of NaOH

V = volume of sample, ml

METHOD 1233

DETERMINATION OF RESIDUAL CHLORINE IN WATER

1. SCOPE

This method is used to determine the residual chlorine in water.

2. SUMMARY OF METHOD

When water containing free chlorine is treated with orthotolidine reagent, a distinct color is obtained. Small amounts of chlorine give a yellow color; larger amounts give an orange color. The quantitative estimation is carried out by comparison with color standards.

3. APPARATUS

Chlorine test kit.

4. REAGENTS

Ortho-tolidine tablets.

5. PROCEDURE

5.1 Rinse three sample tubes of the chlorine test kit with the water to be tested.

5.2 Introduce one ortho-tolidine tablet into one tube and crush with a stirring rod.

5.3 Fill all three tubes to the mark with the sample to be analyzed.

Note: The temperature of the test sample should be approximately 68°F (20° C).

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- 5.4 Mix the ortho-tolidine with the sample by placing a clean dry portion of the hand over the top and inverting a few times.
- 5.5 Compare with known color standards . The reading is made at the point of maximum color development which appears in 5 to 10 minutes after mixing.
- 5.6 Place the tube with the developed color in the center of the comparator block and the two blank samples on either side.
- 5.7 Place various consecutive color standards in the comparator until the color of the sample falls between two known values.
- 5.8 If the color of the sample is too deep, use only 0.5 ml of water and deionized water for the remainder.

6. CALCULATIONS

- 6.1 If an exact match is obtained in Step 5.7 above, the amount of free or residual chlorine is read off directly from the standard with which the match is obtained.
- 6.2 If the color lies between that of two standards, the reading is taken as an average of the two.
- 6.3 If the sample was diluted as in Step 5.8, multiply the result obtained in Step 6.1 or 6.2 by a dilution factor of 20 to obtain the true concentration.

METHOD 1234

DETERMINATION OF OXYGEN CONTENT OF WATER

1. SCOPE

This method is used to determine the oxygen content of primary coolant and various makeup waters.

2. SUMMARY OF METHOD

The following two methods are available for the determination of oxygen in water:

1. Injection of a reduced indigo-carmin solution into an inline sample bulb and visual color comparison. This method is to be used for oxygen concentrations below 50 ppb.

2. A more detailed photometric method, also using an indigo-carmin color-forming solution, for oxygen concentrations higher than 50 ppb.

3. METHOD I

3.1 The determination of dissolved oxygen over the concentration range of 0 to 50 ppb in relatively pure water with indigo-carmin, is based on the oxidation of an exact quantity of reduced indigo-carmin. In this method, the resulting color of the water indigo-carmin mixture is compared with the Oxygen Comparator II (R. L. Johnson Co.). This comparator is a disc device that gives matching colors, one at a time, for six oxygen concentrations: 0, 5, 10, 15, 25, and 50 ppb.

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3.2 Apparatus

Sample flask, 300 ml (rubber diaphragm attached)

Buret, 25 ml

Hypodermic needle, 1-1/2 in.

Hypodermic syringe, 5 ml

Oxygen Comparator II

3.3 Reagents

3.3.1 Indigo-Carmine Stock Solution - Dissolve 0.45 gm of

reagent grade indigo-carmin and 4.5 gm of reagent grade glucose (dextrose) in enough water to make 400 ml of solution. Add 500 ml of reagent grade glycerol and mix. Store in dark bottle and use within 2 weeks.

3.3.2 Potassium Hydroxide, 33.3% - Dissolve 100 gm of reagent grade potassium hydroxide in 200 ml water.

3.3.3 Indigo-Carmine Indicator - In a 100-ml flask, mix 40 ml of the stock indigo-carmin solution with 10 ml of 33.3% potassium hydroxide. Stopper and invert several times to mix. Ascertain a lemon yellow color. Fill a brown bottle with an inert gas (argon) under a slight positive pressure. Transfer reagent solution to bottle, stopper, and invert.

3.4 Procedure

The water is passed through the sample flask (300 ml) for 20 to

25 min. at 200 to 1500 ml/min. The gas flask is valved close. Reagent is removed from its storage bottle by forcing the syringe needle through the rubber stopper. Then, 4.8 ml of the indicator is injected into the sample via the sample flask rubber diaphragm. Mix the contents by repeated shaking. Determine the oxygen concentration immediately by matching the solution color with the Oxygen Comparator II.

3.5 Limitations

The maximum oxygen concentration measurable by this procedure is 50 ppb.

4. METHOD II

4.1 The dissolved oxygen content of a 0.6-ml sample of water is determined by measuring the red color produced by the partial oxidation of reduced indigo-carmines acted upon by the oxygen in the water. Atmospheric oxygen is excluded by carrying out the entire operation, including the reading of the absorbance, in an air-tight syringe. This procedure is applicable to oxygen concentrations greater than 50 ppb. It is more involved than Method I and should be used only when the oxygen concentrations cannot be determined by Method I.

4.2 Apparatus

4.2.1 Fischer Electrophotometer, fitted with a microabsorption assembly and micro cells.

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4.2.2 1-ml Tuberculin syringe, graduated in 0.01 ml, fitted with a No. 22 hypodermic needle.

4.2.3 Special, custom-fitted bushing with a slit window (which may be turned on a lathe from a piece of polyethylene) to adapt the syringe to the sample holder so the geometric positioning of the syringe can be reproduced.

4.2.4 Small stainless steel cylinder, cut from the end of a rod or wire, approximately 3 mm diameter x 3 mm high, to be permanently placed inside the barrel of the syringe to facilitate the mixing of the reagents.

4.2.5 A 16-oz. narrow-mouthed bottle fitted with a rubber stopper that has been shortened to about 5/8 inch long to facilitate insertion and removal of the hypodermic needle. (A gum rubber cap may also be used).

4.2.6 A supply of O₂-free inert gas under pressure.

4.3 Reagents

4.3.1 Indigo-carmin

4.3.2 Glucose

4.3.3 Potassium carbonate

4.4 Preparation of Reagent Solution

2.000 gm each of indigo-carmin, glucose and anhydrous potassium carbonate are placed in a 16-oz. amber glass reagent bottle and 200 ml of

H₂O are added. The rubber stopper is wired in place. The air space within the bottle is flushed with O₂-free nitrogen (also O₂-free argon, helium, or even P-10 gas may be used) for 10 minutes through two No. 22 hypodermic needles, after which the bottle is left under 5 pounds pressure of nitrogen. The reagent is heated an hour in an 80°C water bath until the color changes from blue to straw yellow. The color change is due to the reduction of indigo-carmin by glucose. After use, additional purified nitrogen is introduced through a needle from time-to-time to maintain the internal pressure of about 5 pounds. The reagent is stable indefinitely.

4.5 Procedure

4.5.1 The Fischer Electrophotometer is fitted with the micro-absorption assembly and the red (650 mμ) filter. The first micro-cell is filled with water and used as a reference to zero the instrument. The second micro-cell is fitted with a custom made adapter to position the syringe in an upright position. If possible, the micro-cell and the cell compartment should be modified by drilling holes so that the needle may be left on the syringe at all times, but this is not absolutely necessary as it is possible to remove the needle for each reading without adversely affecting the results. So that the plunger of the syringe in the "out" position may be accommodated, the cell compartment lid is replaced by a specially constructed light-tight box which may be made from cardboard and heavy tape with strips of black cloth stapled around the edges to exclude light.

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4.5.2 The reagent bottle is mounted in an inverted position and the syringe, with the stainless steel mixing cylinder inside, is inserted through the stopper and filled to the 1.00-ml mark. This first rinse is discarded through the needle and the rinsing is repeated for at least one more time until the liquid in the barrel is a clear yellow. Bubbles of dissolved inert gas are removed by pointing the needle upward and discharging into tissue.

NOTE

Air bubbles, differentiated from bubbles of inert gas by the blue layer of oxidized reagent which surrounds them, will spoil the sample. These air bubbles are introduced when the connection between the syringe and hypodermic needle is not air-tight. A layer of stopcock grease aids in the making of an air-tight seal.

4.5.3 After rinsing, the syringe is filled again to the 1.00-ml mark, wiped with a tissue and shaken about 50 times by inverting the syringe and allowing the mixing cylinder to fall to the other end. After mixing, the absorbance of the reagent alone is immediately read. The plunger is advanced to exactly the 0.40-ml mark. The syringe is then lowered into the sample of water to be tested and the barrel filled to exactly the 1.00-ml mark. After wiping and shaking as before, the absorbance of the sample is determined as quickly as possible.

NOTE

The color is unstable and fades quite rapidly on standing.

4.6 Calculations

The calibration factor is first determined by analyzing an air-saturated sample of water by the above procedure. The air-saturated water may be conveniently prepared by bubbling air through water in a large beaker for approximately 24 hours. The temperature of water is measured and the ppm O₂ is read from the tables. (See Table 1234-1) For calculations, the reagent blank absorbence is divided by 2 since the reagent blank was later diluted with clear water.

EXAMPLE 1

Problem: Water known to contain 9.0 ppm O₂ was analyzed by the above procedure. The blank absorbence was 0.102 and the sample absorbence was 0.790. What is the calibration factor?

Solution:

Let X = calibration factor

$$0.790 - \frac{1}{2} (0.102) = 0.790 - 0.051 = 0.739$$

(net absorbence)

$$0.739X = 9.0$$

$$X = 12.18$$

EXAMPLE 2

Problem: An unknown sample was analyzed. The blank absorbence was 0.096 and the sample absorbence was 0.353. Using the above calibration factor, find the O₂ content of the water in ppm.

Solution:

$$0.353 - \frac{1}{2} (0.096) = 0.353 - 0.048 = 0.305$$

(net absorbence)

$$(0.305) (12.18) = 3.7 \text{ ppm}$$

5. INTERFERENCES (METHODS I and II)

All compounds capable of oxidizing or reducing indigo-carmin, such as organic matter, nitrates, chlorates, nitrites, iron salts or sulfites, will interfere.

6. ACCURACY

6.1 Method I: ± 5 ppb over 0-25 ppb range

± 15 ppb over 25-50 ppb range

6.2 Method II: $\pm 2.5\%$

TABLE 1234-1. SOLUBILITY OF OXYGEN IN FRESH WATER

Dissolved Oxygen in Chloride-free Water

<u>C°</u>	<u>ppm</u>
30	7.6
31	7.5
32	7.4
33	7.3
34	7.2
35	7.1
36	7.0
37	6.9
38	6.8
39	6.7
40	6.6
41	6.5
42	6.4
43	6.3
44	6.2
45	6.1
46	6.0
47	5.9

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C°

ppm

48

5.8

49

5.7

50

5.6

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METHOD 1235

DETERMINATION OF PRIMARY WATER HYDROGEN AND TOTAL
GAS CONTENT

1. SCOPE

This method is used to determine the hydrogen and total gas content of primary coolant water.

2. SUMMARY OF METHOD

A sample of coolant is taken in a special apparatus and the hydrogen and other dissolved gases are stripped from the water by depressurization. The evolved gases are sampled and the hydrogen and nitrogen concentrations measured with a Fischer gas partitioner. The total gas is taken as the sum of the nitrogen and hydrogen concentrations.

3. APPARATUS

Vacuum filter flask, 500 ml, provided with a three-hole rubber stopper, two inlet tubes, thermometer, and a serum bottle type cap for the sidearm.

Pressure gauge 30 inches Hg (absolute) to 15 psig.

Syringe (1 ml) and needles.

Vacuum pump.

Fischer gas partitioner with necessary accessories. (Figure 1235-2). The sampling system is shown in Figure 1235-1.

4. PROCEDURE

Since the solubility of hydrogen in water at atmospheric pressure is considerably lower than the desired hydrogen concentration in the pressurized primary water, care must be exercised to avoid any loss of hydrogen during collection of the sample. A sample of primary water is obtained in a manner which permits the quantitative collection of hydrogen and other gases released during depressurization. The hydrogen content of the pressurized primary water is obtained by measuring the amount of hydrogen flashed during depressurization and by calculating the hydrogen concentration of the depressurization water in equilibrium with the gas phase.

- 4.1 Flush the "Coolant Purification Demineralizer Inlet" sampling line thoroughly as per instructions at beginning of Chapter 3.
- 4.2 Remove stopper from flask. Open valves PIF21-VH8, 4, 13, and 11 and flush 250 ml of primary coolant through the line. Close valves. Empty flask and flush thoroughly with air and replace the stopper.
- 4.3 Open valve PIF21-VH12. Turn on vacuum pump and evacuate the flask to exactly 15 inches Hg absolute. Close valve and shut off pump. Check the system for leak tightness by observing the pressure gauge for 2-3 minutes. If there is no change in pressure, continue to next step.
- 4.4 Open valves PIF21-VH8, 4, and 13. Crack valve PIF21-VH11 and

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slowly admit primary coolant to the sampling flask and carefully observe the pressure during the coolant addition. When the pressure equals 0.0 psig, close PIF21-VH11.

4.5 Shake flask vigorously for 2-3 minutes to equilibrate the gas and liquid phases. Allow the sample to be quiescent for 1 minute and then record the gas temperature.

4.6 Withdraw three (3) 1-ml gas samples through the serum cap using a syringe. Each syringe should be flushed several times with the gas in the sampling flask before collecting the sample. Take samples to the gas partitioner for analysis.

4.7 The Fischer Gas Partitioner is turned on 15 minutes prior to use with a flow of 80 cc/min of argon carrier gas. The temperature stabilizer is continuously on and set at 50°C. The current is set for hydrogen detection at 7.2 ma.

4.8 The certified hydrogen standards (2%, 1% and 0.5%) are injected into the sample port and a calibration curve of hydrogen concentration versus peak height readings is obtained. (The calibration curve does not have to be determined each time. However, two standards should be run weekly to establish proper operation of the instrument.) The samples contained in two syringes are injected in turn and the hydrogen concentrations in the gas, (H), are determined. The concentration of hydrogen in the water in ml (STP)/kg (uncorrected) is obtained from Figure 1235-3.

Figure 1235-4 contains a typical gas chromatogram. When hydrogen is present, four distinct peaks are observed.

4.9 One ml of air is injected into the partitioner and the nitrogen peak height is compared with the nitrogen peak height of 1-ml of sample contained in the third syringe. (Use appropriate sensitivity scale factor.)

4.10 The instrument is shut off and the carrier gas flow is stopped.

5. CALCULATIONS

5.1 The uncorrected hydrogen content H_u is obtained from Figure 1235-3.

5.2 The nitrogen content of the gas sample in excess of that present in air (N) is as follows:

$$N = 78 \left[\frac{\text{Sample } N_2 \text{ pk ht.}}{\text{Air } N_2 \text{ pk ht.}} - 1 \right] (\text{vol } \%)$$

The corrected primary water hydrogen content H_c is as follows: *

$$(H_c) = \frac{100 + H + N}{100} \cdot H_u \cdot \frac{\text{cc(STP)}}{\text{kg water}}$$

Where H = hydrogen in gas (Vol %)

N = excess nitrogen in gas (Vol %)

H_u = uncorrected hydrogen content of
primary water $\frac{\text{cc}}{\text{kg}}$

*(The correction may be omitted if it accounts for less than a 10% change in H_u .)

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The primary water nitrogen content N_c is as follows:

$$N_c = 1.22 \frac{N}{H} H_c \frac{\text{cc (STP)}}{\text{kg water}}$$

(The correction factor 1.22 is obtained from the difference in solubility between hydrogen and nitrogen.)

The total gas content G is as follows:

$$G = H_c + N_c \frac{\text{cc (STP)}}{\text{kg water}}$$

6. ACCURACY

The accuracy of the results is $\pm 5\%$.

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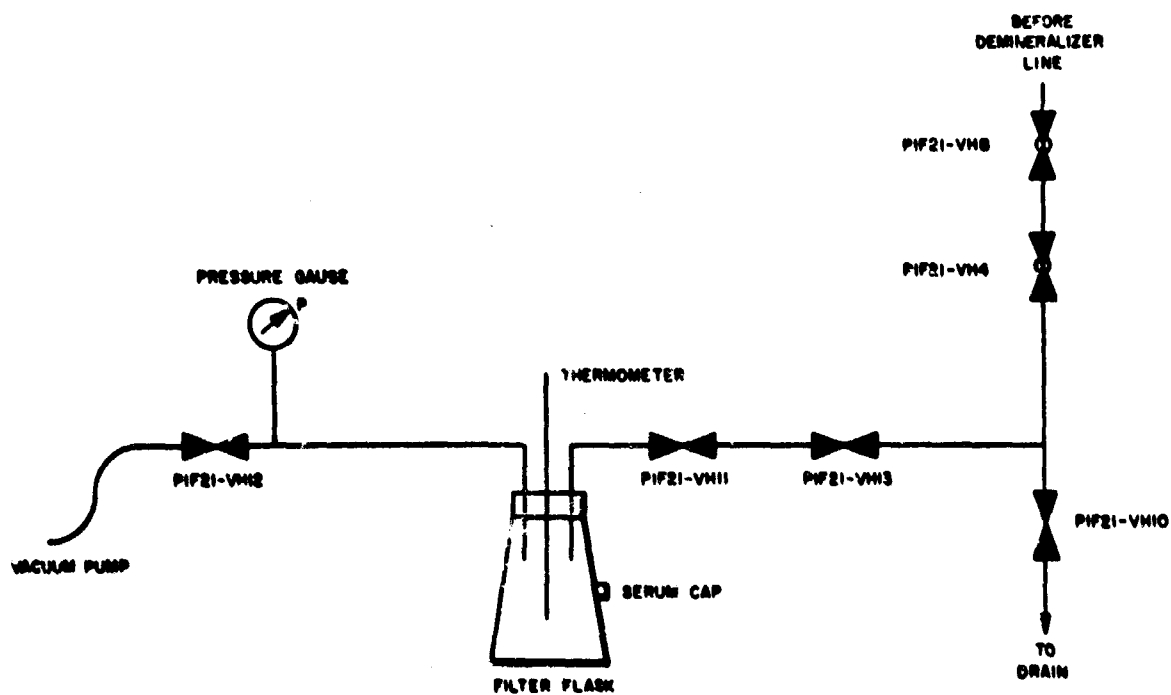


Figure 1235-1. Primary Water Gas Sampling Apparatus.

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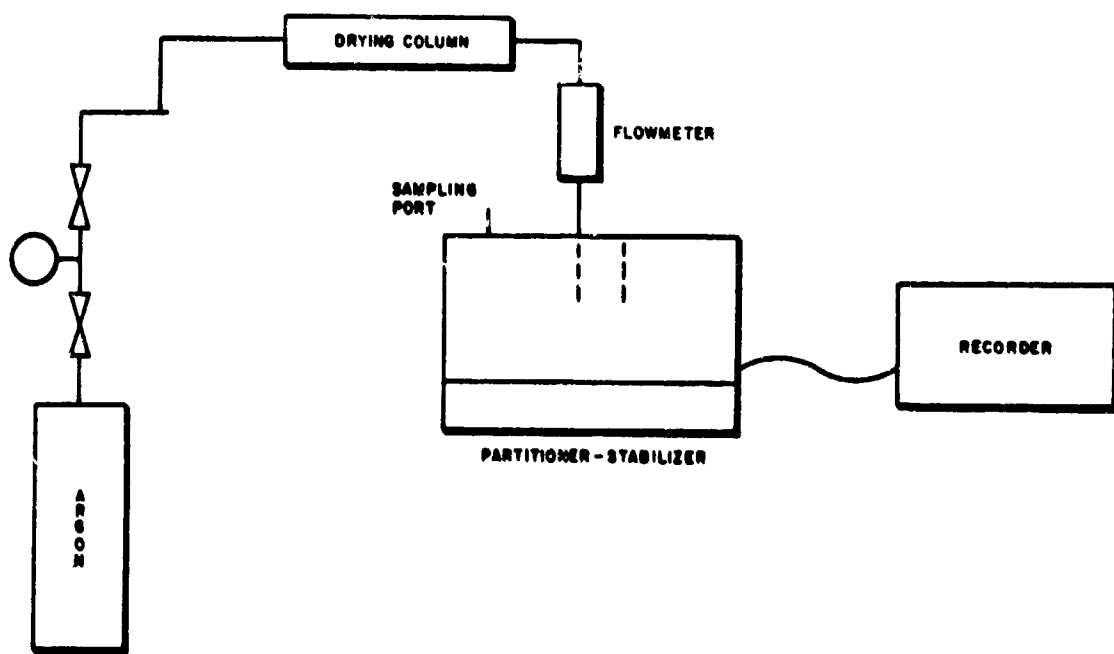


Figure 1235-2. Gas Partitioner System.

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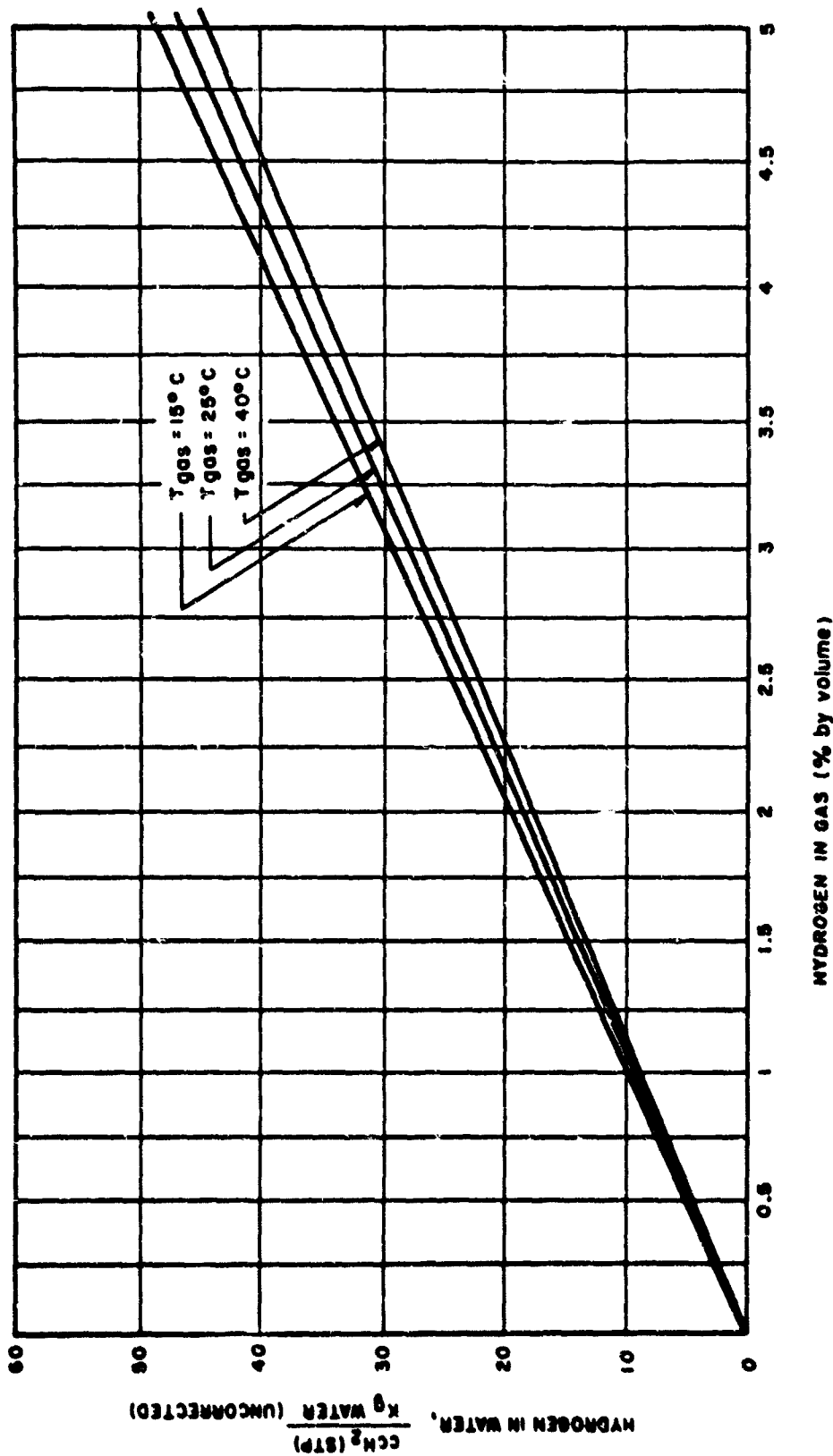


Figure 1235-3. Determination of Hydrogen Concentration in Primary Water.

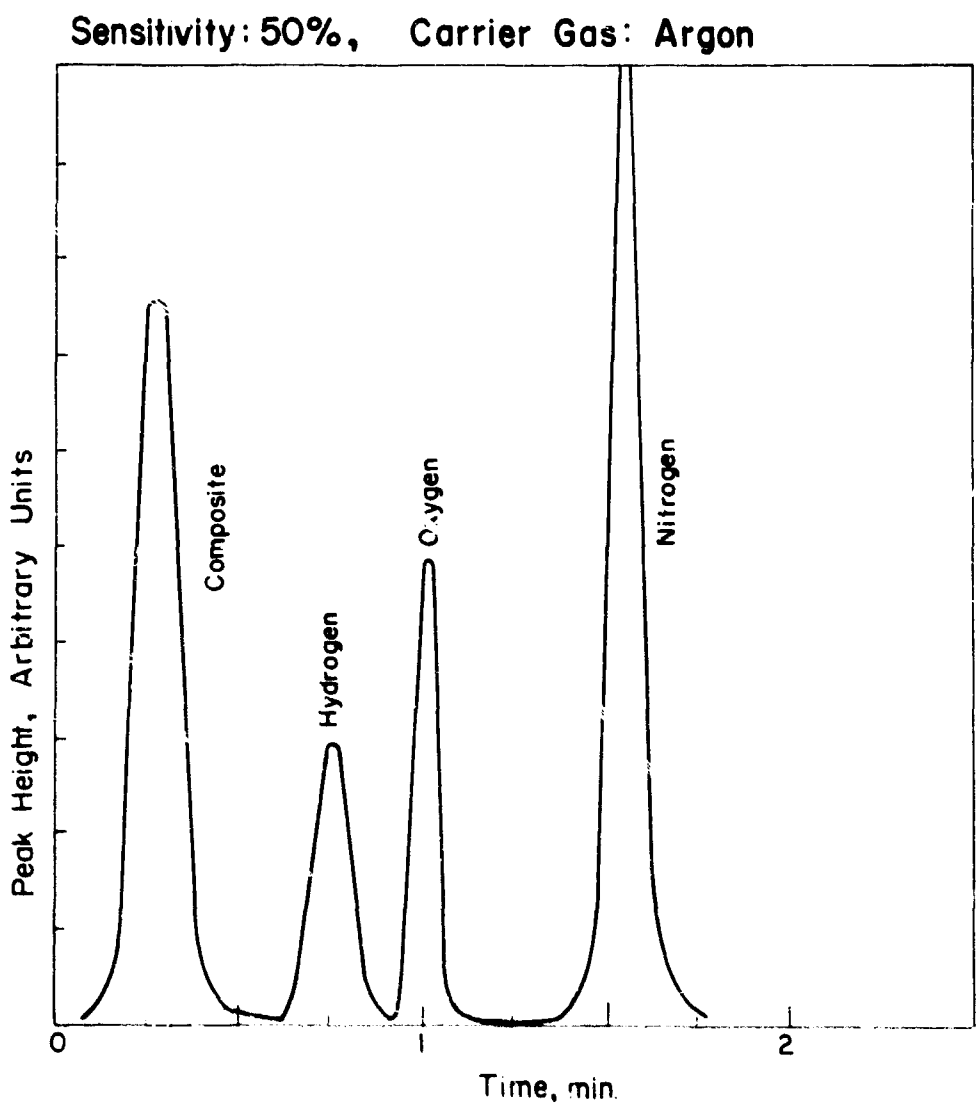


Figure 1235-4. Characteristic Chromatogram for Hydrogen Analysis.

METHOD 1241
DETERMINATION OF pH

1. SCOPE

This method is used to determine the pH of the water from various plant systems.

2. SUMMARY OF METHOD

The pH of a solution is the log of the reciprocal of the hydrogen ion concentration. The higher the pH the more basic the solution; i.e. pH values below 7 indicate an acid solution, pH values greater than 7 mean an alkaline solution, while pH of 7 is neutral. A glass electrode and calomel electrode are immersed in the solution to be tested and a direct current voltage applies. The operating principle is based on a complex ion-exchange diffusion process across the porous glass membrane. The observed voltage drop across the electrodes is a direct measure of pH and the instrument is calibrated with solutions of known pH.

3. APPARATUS

Beckman Zeromatic pH meter (Model 9600) for 120-volt, 60-cycle,

A-c single phase.

Glass electrode for use between 20°C and 60°C.

Calomel electrode.

Lead-wires and necessary connectors.

4. REAGENTS

Buffer solutions: pH 4, 7, and 10.

5. PROCEDURE

5.1 The detailed instructions for measuring pH and for operation of the Beckman Zeromatic pH meter are given in the manufacturer's manual.

5.2 Wash the electrodes thoroughly with demineralized water; wipe off excess water with tissue. Check the standardization with pH 7 and 10 buffers. Rinse electrodes with demineralized water and wipe dry after each standardization.

5.3 Immerse electrodes into solution to be measured.

5.4 Read the pH as soon as possible after sampling.

5.5 Rinse electrodes with demineralized water and replace in beaker of distilled or deionized water.

6. CALCULATIONS

Instrument reads pH directly.

7. PRECAUTION

A new glass electrode must be soaked in deionized water for several hours before using. Be certain that the calomel reference electrode is filled with saturated KCl and an excess of KCl crystal.

8. INTERFERENCES

No interferences when the instrument is calibrated with a standard

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buffer solution of known purity. To insure purity of buffer solutions a fresh portion should be used for each standardization then, discarded after use. For economy and accuracy small packets of concentrated buffer should be used to prepare fresh buffer solutions.

9. ACCURACY

The accuracy of the results is ± 0.1 pH unit.

METHOD 1251
CONDUCTIVITY

1. SCOPE

This method is used to determine the purity of water in use in the power plant.

2. SUMMARY OF METHOD

The method is used to measure the electrical resistance of a water sample when an alternating current is passed through a pair of electrodes inserted in the sample. The electrodes and sample form one leg of a Wheatstone Bridge arrangement and calibrated resistance in another leg of the bridge is varied so that the current flow through the two legs is equal. The instrument is calibrated to read the specific resistance of the unknown directly.

3. APPARATUS

3.1 Solu-Bridge (Type RD-132 Industrial Instruments Inc.) - The instrument is calibrated for use with a conductivity cell having a cell constant of 0.10 and reads specific resistance directly on a calibrated dial.

3.2 Conductivity cell, having a calibrated cell constant of 0.10.

4. PROCEDURE

4.1 The instrument is allowed to operate continuously.

4.2 Pour an aliquot of the sample into a beaker.

- 4.3 Measure the temperature of the water and set the temperature control accordingly.
- 4.4 Insert conductivity cell and move up and down several times to insure equilibrium.
- 4.5 Rotate the dial control until the black segment of the "tuning eye" is at maximum width and the edges between the black and green sections are sharply defined.
- 4.6 Read the specific resistance of the sample in ohm-cm directly from the dial.

5. CALCULATIONS

$$\text{Specific conductance } \mu\text{mho/cm} = \frac{1 \times 10^6}{\text{specific resistance}}$$

6. STANDARDIZATION OF APPARATUS

The operation and maintenance of the conductivity apparatus is described in the manufacturer's instruction manual. Standardization of the conductivity cells, the standard solutions and the resultant calculations are fully described in the ASTM Standard 1961 Part 10, pages 1290 - 1297. A standardization of the apparatus should be done semi-annually or whenever a cell is damaged or behaves in an erratic fashion. If the faulty operation of a cell cannot be corrected, the cell is to be replaced.

7. INTERFERENCES

Any anions other than OH^- will result in a high net conductance.

The pH correction does not compensate for these anions.

8. ACCURACY

The accuracy of the results is $\pm 2\%$.

METHOD 1261

DETERMINATION OF TOTAL ALPHA ACTIVITY IN WATER

1. SCOPE

This method is used to determine the alpha activity of water samples.

2. SUMMARY OF METHOD

A sample of water is evaporated to dryness in a suitable planchet and alpha counted using a proportional counter (NMC).

3. APPARATUS

1.5- or 2-liter beaker

Hot plate

2-inch diameter stainless steel counting planchet

4. REAGENTS

Concentrated nitric acid

5. PROCEDURE

5.1 Measure 1 liter of sample into a 1.5- or 2-liter beaker.

5.2 Evaporate carefully to 50 or 100 ml on a hot plate in a hood.

5.3 Reduce heat and evaporate to 5 ml. Avoid over-drying.

5.4 Add 5 ml of concentrated nitric acid to the beaker and swirl to dissolve or loosen the residue on the sides and bottom of the beaker.

5.5 Continue heating for 2 or 3 minutes.

5.6 Remove from the hot plate and cool.

5.7 Transfer carefully into a clean 2-in. stainless steel planchet.

Heat the planchet slowly on a hot plate to evaporate.

5.8 Rinse the beaker with 5 ml of water and transfer in small portions to the planchet, as evaporation permits.

5.9 Cool planchet to room temperature and count in a proportional counter using the "alpha only" operating voltage for 10 minutes.

5.10 Record total counts and counting interval on counting sheet.

6. CALCULATIONS

The gross alpha activity is calculated as follows:

$$A = \frac{N_s - N_b}{(V)(E)(Y)}$$

WHERE

A = gross alpha activity, dpm/ml

N_s = counting rate of sample plus background, cpm

N_b = counting rate of background, cpm

V = volume of sample, ml

E = efficiency of counter

Y = boil down yield (This factor is determined by adding a known amount of alpha activity to one liter of distilled water and processing as outlined above. Y is the ratio of the recovered activity to that added originally.) (Use 60%)

METHOD 1262

DETERMINATION OF TOTAL BETA ACTIVITY IN WATER

1. SCOPE

This method is used to determine the total non-volatile beta activity of water.

2. SUMMARY OF METHOD

A sample of the water is evaporated to dryness and the residue is counted in a suitable counter.

3. APPARATUS

1.5- or 2-liter beaker

Hot plate

2-in. diameter stainless steel planchet

4. REAGENTS

Concentrated nitric acid

5. PROCEDURE

5.1 Measure 1 liter of sample into a 1.5-liter beaker.

5.2 Evaporate carefully to 50 or 100 ml on a hot plate.

5.3 Reduce heat and evaporate to 5 ml. Avoid over-drying.

5.4 Add 10 ml of concentrated nitric acid to the beaker and swirl to

dissolve or loosen the residue on the sides and bottom of the beaker. (Iodine will be lost during the nitric acid boil down.)

- 5.5 Continue heating for 2 to 3 minutes.
- 5.6 Remove from the hot plate and cool.
- 5.7 Transfer carefully into a clean 2-inch stainless steel planchet.
- 5.8 Rinse the beaker with 5 ml of water and transfer in small portions to the planchet as evaporation permits.
- 5.9 Let the planchet cool to room temperature, and count in a proportional counter using the beta plateau or a Geiger-Mueller counter.

6. CALCULATIONS

The gross beta-gamma activity is calculated as follows:

$$A = \frac{N_s - N_b}{(V)(E)(Y)(2.22 \times 10^6)}$$

WHERE

A = gross beta activity, uc/ml

N_s = counting rate of sample including background, c/m

N_b = counting rate of background, c/m

V = volume of sample, ml

E = efficiency of counter, determined daily

Y = boil down yield factor (This factor is determined by adding a known amount of beta activity to one liter of distilled water and processing as outlined above. Y is the ratio of the recovered activity to that added originally)

METHOD 1271

DETERMINATION OF DEMINERALIZER DF

1. SCOPE

The DF (decontamination factor) is determined to verify the operation of the ion exchange demineralizer.

2. SUMMARY OF METHOD

The DF is determined by counting degassed samples of the water before and after passage through the demineralizer. The decontamination factor is the ratio of the inlet activity to the outlet activity.

3. APPARATUS

Degassing apparatus

2- 2 ml pipettes

2-4 ml vials

Single channel analyzer and well crystal biased to only accept pulses greater than 30 kev.

4. REAGENTS

None required.

5. PROCEDURE

5.1 Obtain samples of water before and after the demineralizer and record the sampling times. The samples should be collected as close together as possible.

5.2 Degas the samples as soon as possible after sampling using a clean apparatus for each sample.

6. COUNTING PROCEDURE

Count the samples as described in Method 3.18 except that the activity should be counted 2.5 hours after sampling.

7. CALCULATIONS

$$DF = \frac{A_B - B}{A_A - B}$$

WHERE A_B = count rate of sample before demineralizer, cpm

B = background count rate, cpm

METHOD 1281

DETERMINATION OF RADIOACTIVE BARIUM

1. SCOPE

This method is used to determine the radioactive Barium concentration of aqueous solution.

2. SUMMARY OF METHOD

The barium and strontium are separated from other fission products by a basic carbonate precipitation. The barium is then separated from the strontium by a chromate precipitation followed by cleanup steps using ether-HCl.

3. APPARATUS

400-ml beaker

Hot plate

Filter paper

Filter tower

Ice bath

Centrifuge

Centrifuge cones

4. REAGENTS

4.1 Barium carrier - standardized

4.2 Strontium carrier - standardized

- 4.3 3M sodium carbonate solution
 - 4.4 6M sodium hydroxide solution
 - 4.5 Methyl orange indicator
 - 4.6 6M acetic acid
 - 4.7 6M ammonium acetate
 - 4.8 1.5M sodium chromate
 - 4.9 6M hydrochloric acid
 - 4.10 Diethyl ether
 - 4.11 6M nitric acid
 - 4.12 3M sulfuric acid
 - 4.13 Ferric nitrate solution approximately 10 mg/ml iron
5. ANALYTICAL PROCEDURE
- 5.1 Place 200-ml sample in 400-ml beaker.
 - 5.2 Add 2.0 ml Ba^{++} standardized carrier.
 - 5.3 Add 2.0 ml Sr^{++} standardized carrier.
 - 5.4 Heat to boiling and add 5 ml 3M Na_2CO_3 .
 - 5.5 Centrifuge in 40-ml conical centrifuge tube and discard supernate.
 - 5.6 Dissolve precipitate in minimum amount 6M HNO_3 and dilute to approximately 15 ml with water.
 - 5.7 Boil solution, add several drops $\text{Fe}(\text{NO}_3)_3$ solution.
 - 5.8 Add 6M NaOH dropwise to precipitate $\text{Fe}(\text{OH})_3$. This is a "scavenging" precipitate.

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- 5.9 Centrifuge and decant supernate into a clean centrifuge tube.
Discard precipitate.
- 5.10 Take supernate and scavenge twice more with $\text{Fe}(\text{OH})_3$; discard precipitates and save supernate.
- 5.11 Add methyl orange indicator to supernate obtained above and add HNO_3 acid until indicator turns from yellow to red.
- 5.12 Add 1-ml 6M acetic acid.
- 5.13 Add 2-ml 6M ammonium acetate.
- 5.14 Heat sample in hot water bath and add 1-ml 1.5M Na_2CrO_4 .
- 5.15 Digest sample in hot water bath for 5 minutes.
- 5.16 Centrifuge out BaCrO_4 precipitate and save supernate for strontium analysis.
- 5.17 Dissolve BaCrO_4 in 1-ml 6M HCl .
- 5.18 Place sample in ice bath and add 1 ml diethyl ether and 25 ml concentrated HCl . Stir solution and allow to cool 5 minutes.
- 5.19 Centrifuge out BaCl_2 and discard supernate.
- 5.20 Dissolve BaCl_2 in 15 ml water.
- 5.21 Add 2 ml 3M H_2SO_4 to solution.
- 5.22 Filter BaSO_4 onto a weighed filter paper.
- 5.23 Wash precipitate with water and then 95% ethanol.
- 5.24 Dry sample by vacuum desiccation or in an oven and weigh BaSO_4 to closest 0.1 mgm.

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5.25 Calculate percent chemical yield.

5.26 Roll filter paper in plastic and place in 4-ml vial.

6. COUNTING PROCEDURE

6.1 Count sample with 3-in. x 3-in. NaI (Tl) crystal gamma spectrometer immediately after sample preparation.

6.2 Plot data on 3-cycle semilog graph paper.

6.3 On graph record following information:

Time and date sample was taken

Time and date sample was counted

Length of count

Gain

Volume of water sample, ml

7. CALCULATIONS

7.1 12.8 d Ba-140.

7.1.1 Use area under 0.540 Mev γ .

7.1.2 Use 25% for number of disintegrations leading to 0.540 Mev.

7.1.3 Calculate decay = $e^{-\lambda t}$

Where $\lambda = \frac{0.693}{12.8}$ (day⁻¹), and t = days decay

7.1.4 Calculate Dis/min/ml as follows:

$$\text{Dis/min/ml} = \frac{1}{t_c} \times \frac{\text{Area}}{E} \times \frac{e^{-\lambda t}}{0.25} \times \frac{1}{\text{Chem. Yield} \times \text{Vol}}$$

E = efficiency - counts per gamma ray

t_c = count time

7.2 85 min Ba-139

7.2.1 Use 0.165 Mev γ . Correct for contribution of 12.8 d Ba-140 to this energy region.

7.2.2 Use 23% for number of disintegrations leading to 0.165 Mev γ .

7.2.3 Calculate decay = $e^{-\lambda t}$

Where $\lambda = \frac{0.693}{85}$ (min⁻¹), and t = minutes decay

7.2.4 Calculate Dis/min/ml as follows:

$$\text{Dis/min/ml} = \frac{1}{t_c} \times \frac{\text{Area}^*}{E} \times \frac{e^{-\lambda t}}{0.23} \times \frac{1}{\text{Chem. Yield} \times \text{Vol.}}$$

E = efficiency - counts per gamma ray

t_c = count time

8. ALTERNATE COUNTING PROCEDURE

The barium activity is primarily composed of 85 min Ba-139 and 12.8 d Ba-140. Both are beta and gamma emitters. A beta proportional counter may be used for sample counting. For the Ba-139 determination, count every 15 minutes for 1-1/2 hours. For the Ba-140 determination, if Ba-139 is present, begin counting 1 day after purification. Then wait for 2 weeks, and count daily thereafter for 5 to 10 days.

9. CALCULATIONS

9.1 Plot a decay curve on semilog paper of the corrected cpm versus

*Area corrected for 12.8 d Ba-140 contribution to 0.165 Mev photopeak of 85 min Ba-139.

time. The gross counting rate corrected for background, standard factor, and coincidence, where applicable, is the "corrected cpm".

9.2 Calculate the slope of this curve. If the sample contains Ba-139, the slope of the curve should be equal to the decay constant of 85 min Ba-139.

9.3 The counting rate of Ba-139 at sampling time is obtained by extrapolating the 85 min portion of the decay curve back to the sampling time.

9.4 To determine the contribution of 12.8 d Ba-140 to the sample, allow the sample to equilibrate with its 40 hr La-140 daughter over a period of 2 weeks. Then count daily for 5 days. The count taken 1 day after separation may be computed for Ba-140 provided a standard curve such as given in Figure 1281-1 has been obtained from a known pure source on the same instrument used for the analysis.

9.5 The Ba-140 count rate at Ba-140 - La-140 separation time is calculated using the following equation:

$$A_1^0 = A_1 e^{+\lambda_1 t}$$

Where A_1^0 = Ba-140 count rate at sampling time, cpm

A_1 = Ba-140 count rate at separation time, cpm

t = elapsed time between sampling and Ba-140
La-140 separation time.

9.7 The disintegration rate of both Ba-129 and Ba-140 is calculated

using the equation:

$$\text{Dis/min/ml} = \text{cpm (corrected)} \times f_v \times f_z \times f_y$$

Where f_v = factor for volume of sample

f_z = counting efficiency factor

f_y = yield factor

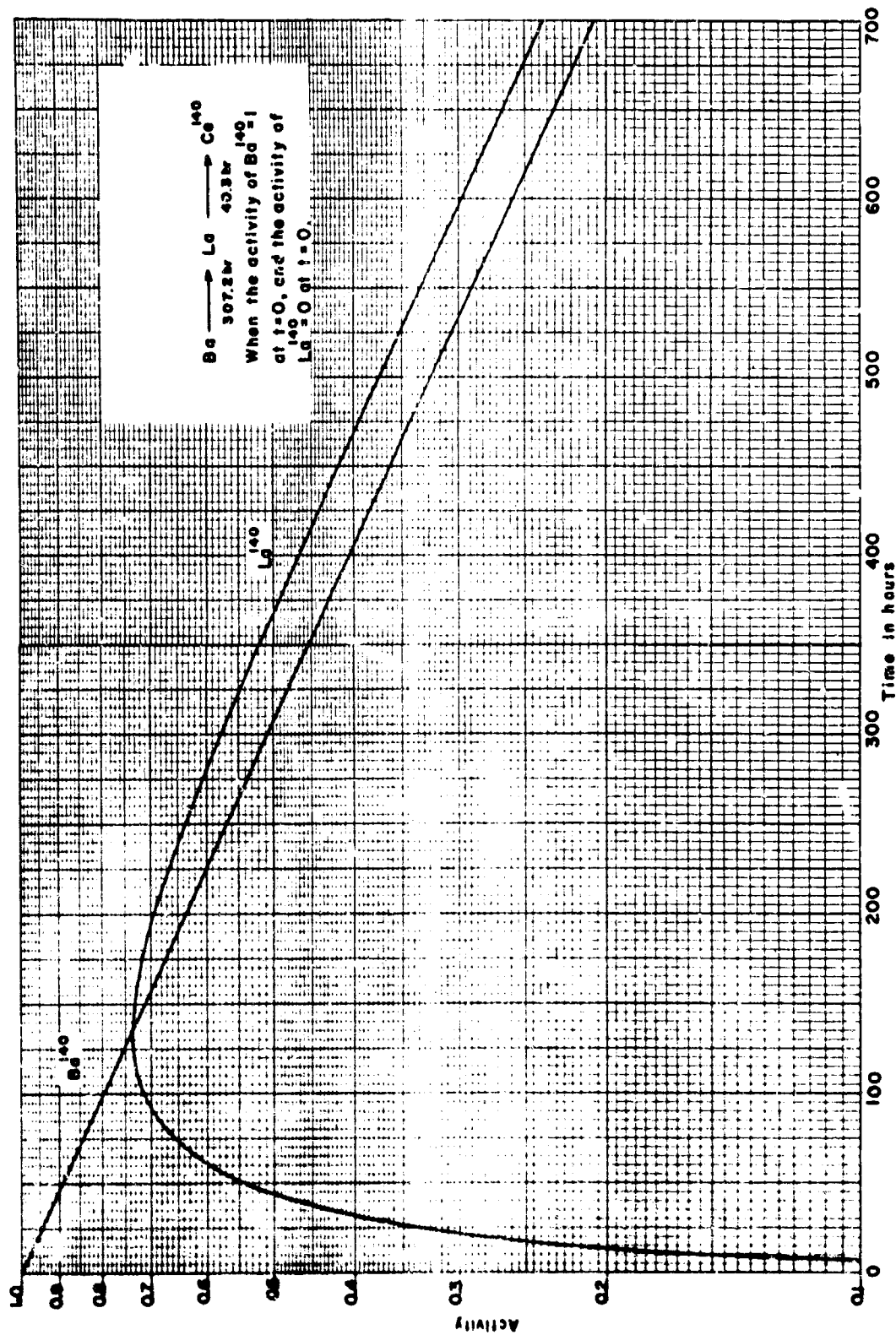


Figure 1281-1. Decay of Ba^{140} and Growth of La^{140} Activity

METHOD 1282

DETERMINATION OF RADIOACTIVE CRUD IN WATER

1. SCOPE

This method is used to determine the crud content of water when measurable amounts of crud are not present in a reasonable volume of water.

2. SUMMARY OF METHOD

The crud content of coolant is normally determined by filtering a known volume of solution and weighing the deposit collected. However, in some instances, the crud concentration is very small and filtering 10 or more liters of coolant is impractical. Therefore, an alternative procedure is to filter a known amount of coolant and measure the radioactivity of the deposit.

3. APPARATUS

2-liter side arm filter flask

47-mm diameter Millipore filter tower or equivalent

47-mm diameter, 0.45-micron pore size, Millipore filter paper

One-hole rubber stopper to fit flask and tower.

Rubber tubing

Vacuum pump

4. REAGENTS

None required

5. PROCEDURE

5.1 Collect at least 1 liter of primary coolant, before demineralizer, in a polyethylene bottle.

5.2 Assemble the filtration apparatus and filter exactly 1 liter of sample through a Millipore filter.

5.3 Rinse the filter with deionized water. If the filtrate is needed for some other analyses, collect the rinse water separately from the original sample. The rinse water may be discarded.

5.4 Remove the filter and place on a small piece of clean plastic film. Fold the sample and plastic into a small square keeping the sample inside the film at all times.

5.5 Using a pair of forceps, insert the folded sample into a 4-ml counting vial and seal the vial with a cap.

5.6 Wrap the vial in clean plastic film and place in the well crystal.

Having the single channel analyzer set for integral counting of all gammas above 30 Kev, count the sample (one hour after collection) for 10 minutes or 10,000 counts, whichever is reached first. Record total counts and counting interval.

5.7 Place the sample on the 3-in. x 3-in. crystal and obtain a gamma spectrum and total gamma count. Plot out the spectra using the x - y plotter. Record counting interval, date and time counting, calibration energy per channel, date and time of sampling, and size sample filtered.

NOTE

- a) In the event the crud content is high enough to weigh, visually determined from previous sample, the filter should be preweighed and filter plus deposit weighed after drying under a heat lamp or in an oven at 110°C for about 10 minutes.
- b) Do not rinse the Millipore filter with acetone as the filter is soluble in many organic solvents.

6. CALCULATION

Calculate the sample activity using the data obtained with the single channel analyzer operated in the integral mode. (30-Kev base line with water switch in out position.)

$$A = \frac{N_s - N_b}{(0.40) (2.2 \times 10^6) (V)}$$

Where A = sample activity, $\mu\text{c/ml}$

N_s = count rate of sample including background
 $\mu\text{c/m}$

N_b = count rate of background, cpm

0.40 = counter efficiency factor, cpm/dpm

2.2×10^6 = dpm/ μc

V = column of sample filtered, ml

The activity of individual nuclides may be obtained from the spectrum if desired. In this case the sample should be counted hourly for

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8 hours then daily for 5 days to obtain a decay curve for each photopeak of interest. The decay curves should be resolved into components and each component extrapolated to 1-hour sampling before completing the calculation.

Report the crud level as $\mu\text{c/ml}$ 1 hour after sampling.

METHOD 1283

DETERMINATION OF RADIOACTIVE EUROPIUM IN WATER

1. SCOPE

This method is used to determine the europium content of primary coolant.

2. SUMMARY OF METHOD

2.1 Europium is present in the coolant as the result of fissioning tramp uranium in the fuel element cladding and may also be present as a result of a defected control rod. The amount of europium produced as a result of fission is very small and will be masked by other rare earth nuclides produced by fission unless a very specific separation is performed.

2.2 The europium released as a result of a control rod defect is composed of several nuclides, stable as well as radioactive. The radioactive nuclides are tabulated on the following page along with their half-lives and photon energies.

2.3 The method utilized is a separation of all the rare earth elements as a group followed by gamma spectrum analysis to identify the europium. Consequently, the fission product concentration in the coolant will influence the sensitivity of the method. Complete separation of corrosion products is also required.

3. APPARATUS

Water bath - 400-ml beaker containing water

Hot plate

Filter tower and paper for mounting samples

4-ml vials

250-ml beaker

Glass centrifuge cones, 40 or 50 ml

Plastic centrifuge cones, 50 ml

Centrifuge

4. REAGENTS

4.1 Europium carrier, 10 mgm/ml: Dissolve 1.160 gm of europium oxide, Eu_2O_3 , in 10 ml of 6M hydrochloric acid and dilute to 100 ml with deionized water.

4.2 Barium carrier, 10 mgm/ml: Dissolve 9.5 gm of barium nitrate, $\text{Ba}(\text{NO}_3)_2$, in water. Add 2 ml of concentrated nitric acid and dilute to 500 ml with deionized water.

4.3 Iron carrier, 10 mgm/ml: Dissolve 4.84 gm of hydrated ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, in deionized water and dilute to 1 liter.

4.4 Saturated ammonium oxalate solution .

4.5 55% Hydrofluoric acid.

4.6 Concentrated hydrochloric acid.

TABLE 1283-1
PHOTON ENERGIES OF EUROPIUM ISOTOPES PRODUCED BY NEUTRON CAPTURE

<u>Nuclide</u>	<u>Half-Life</u>	<u>Photon Energies</u>
Eu-152m	9.3 hr.	0.122(14A), 0.839(10A), 0.961(10A), 0.344(3A), 1.327(3A)
Eu-152	12.5 yr.	0.122(67A), 0.344(27A), 1.416(27A), 0.965(60R), 1.092(50R)
Eu-154	16 yr.	1.277(42A), 0.123(35A), 0.725(21A), 1.007(17A), 0.988(14A)

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- 4.7 Concentrated nitric acid.
- 4.8 1 : 1 dilute ammonium hydroxide (concentrated ammonium hydroxide diluted 1 : 1 with deionized water)
- 4.9 Saturated boric acid solution.
- 4.10 Sulfuric acid, dilute concentrated sulfuric acid 1 : 1 with deionized water.

5. PROCEDURE

- 5.1 Filter a 100-ml sample of primary coolant through a Millipore filter. Dissolve the separated crud as specified in Method 1291.
- 5.2 Transfer the filtered 100 ml of primary coolant to a 250-ml beaker, add the crud solution from preceding Step 5.1 above, and add 1 ml of europium carrier solution to the sample.
- 5.3 Cover beaker with a speed-evap lid and evaporate the sample to near dryness on a hot plate. Cool the beaker and sample.
- 5.4 Add about 6 ml of concentrated hydrochloric acid and 2 ml of concentrated nitric acid slowly to the sample. Heat gently on the hot plate and evaporate to near dryness. Cool beaker.
- 5.5 Repeat Step 5.4
- 5.6 Using deionized water, dissolve the residue and transfer the sample to a glass centrifuge cone.
- 5.7 Add 2 ml of barium carrier to the sample and adjust the volume to about 25 ml using deionized water.

5.8 Heat the sample in a water bath and carefully add dilute sulfuric acid until a white precipitate is formed. Add a few drops of sulfuric acid in excess.

5.9 Centrifuge, transfer the supernate to a plastic centrifuge cone, and discard the precipitate.

5.10 Repeat Steps 5.7 through 5.9.

5.11 Add 1 ml of iron carrier to the sample solution. Adjust volume to 10 ml and carefully add 10 - 20 drops of 55% hydrofluoric acid.

Heat sample in water bath for 5 minutes until a white precipitate forms.

CAUTION

Fluorides are poisonous and can cause severe burns.

5.12 Centrifuge and discard the supernatant solution.

5.13. Wash the precipitate with deionized water, centrifuge and discard supernatant.

5.14 Dissolve the precipitate by adding 5 ml of saturated boric acid to the sample and heat in hot water bath. Add 1 ml of concentrated nitric acid, heat, then dilute to 10 ml with water.

5.15 Add dilute ammonium hydroxide until a precipitate forms. Heat in water bath 3 minutes, cool, and centrifuge. Discard supernatant solution. Wash precipitate with water, centrifuge, and discard supernatant.

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- 5.16 Dissolve the precipitate by adding 5 ml of water and 1 ml of concentrated hydrochloric acid. Heat in a water bath to dissolve Precipitate.
- 5.17 Dilute solution to about 25 ml with water and heat in a water bath.
- 5.18 Add 5 ml of saturated ammonium oxalate and heat sample for 5 minutes in water bath.
- 5.19 Centrifuge and discard the supernatant.
- 5.20 Wash precipitate with deionized water. Centrifuge and discard the supernatant.
- 5.21 Filter the precipitate through a tared (paper plus small piece of plastic film) Whatman No. 542 filter paper disc placed on a small square of plastic film.
- 5.22 Wash the precipitate with a few ml of water and then ethyl alcohol.
- 5.23 Dry the precipitate at 110°C and weigh as europium oxalate, $\text{Eu}_2(\text{C}_2\text{O}_4)_3 \cdot 10\text{H}_2\text{O}$.
- 5.24 Fold the precipitate and filter in a clean piece of plastic film and using a pair of forceps, transfer the sample to a 4 ml counting vial.
- 5.25 Count the sample on a gamma pulse height analyzer and obtain a spectrum.

6. CALCULATIONS

6.1 Obtain the chemical recovery using the following equation:

$$\text{Yield} = \frac{(W_p - W_f) 0.4063}{W_a}$$

Where W_p = weight of precipitate plus filter paper plus plastic film

W_f = weight of filter paper plus plastic film

W_a = weight of europium added

0.4063 = fraction of europium in europium oxalate

6.2 Calculate the europium-152m present in the sample at time of sampling using the following equation:

$$A = \frac{N}{(F) (t) (E) (Y) (V) (e^{-\lambda T}) (2.22 \times 10^6)}$$

Where A = activity of europium-152m, $\mu\text{c/ml}$

N = number of counts in photopeak less background
(use photopeaks specified in Table 1283-1.)

t = counting time in minutes

F = efficiency factor of counter, counts per gamma

Y = chemical yield

V = volume of sample taken, ml

λ = $\frac{0.693}{t_{1/2}}$ 0.075 hr^{-1}

T = elapsed time between sample collection and counting hr

F = abundance of photopeak of interest

METHOD 1284

DETERMINATION OF SODIUM-24 IN WATER

1. SCOPE

This method may be used to obtain confirmatory evidence of the presence of sodium that has been qualitatively identified in a gamma ray spectrum of primary coolant.

2. SUMMARY OF METHOD

This method is based upon a precipitation of iron, strontium, and lanthanum hydroxides to remove some fission contaminants and subsequent precipitation of sodium as sodium chloride.

3. APPARATUS

Centrifuge

Centrifuge cones, 40 ml

Graduated cylinder, 10 ml

Beaker, 250 ml

Hot plate

Ice bath

Glass fiber filter, 24 mm

Filtration apparatus

Balance

Pipet, 2 ml

Gamma counting system

4. REAGENTS

- 4.1 Sodium carrier, 10 mgm Na/ml
- 4.2 Strantium carrier, 10 mgm Sr/ml
- 4.3 Lanthanum carrier, 10 mgm La/ml
- 4.4 Iron carrier, 10 mgm Fe/ml
- 4.5 Ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$, saturated solution
- 4.6 Hydrochloric acid, HCl, concentrated
- 4.7 Diethyl ether

5. PROCEDURE

- 5.1 Pipet 2 ml of sodium carrier into 200 ml of filtered primary coolant in a 250-ml beaker. Evaporate on a hot plate to about 25 ml.
- 5.2 Transfer to a 40-ml centrifuge cone. Add 5 drops each of strantium, lanthanum, and iron carriers.
- 5.3 Add a saturated ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$, solution dropwise until no more brown gelatinous precipitate forms. Centrifuge for 2 minutes.
- 5.4 Transfer the supernatant to 125 ml Erlenmeyer flask. Add 2 ml concentrated hydrochloric acid, HCl. Evaporate to dryness on the hot plate.
- 5.5 Dissolve the residue with 2 ml of water. Transfer to a 40-ml centrifuge cone with 10 ml of concentrated hydrochloric acid, HCl.

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- 5.6 Add 10 ml of ether. Cool in an ice bath and stir vigorously.
- 5.7 Centrifuge for 2 minutes. Discard the supernatant.
- 5.8 Dissolve the white precipitate in 2 ml of water. Add 10 ml of concentrated hydrochloric acid, HCl. Repeat Step 5.6.
- 5.9 Suction filter through a weighed glass fiber filter. Wash with 5 ml of ether.
- 5.10 Remove the filter and place in a weighed vial. Determine the weight of precipitate.
- 5.11 Gamma count integrally for at least 1 minute or at least 10,000 counts or obtain a gamma ray spectrum.

6. CALCULATIONS

- 6.1 If the sample has been integrally counted using a base line of 30 kev, the activity may be calculated as follows:

$$A = \frac{N_s - N_b}{(V) (E) (Y) (2.2 \times 10^6) (t)}$$

Where A = sodium-24 activity, $\mu\text{c/ml}$

N_s = counting rate of sample plus background, cpm

N_b = counting rate of background, cpm

V = volume of sample analyzed, ml

E = counting efficiency for sodium-24, 0.37
cpm/dpm

Y = chemical yield = $\frac{(W_t - W_f - W_p) 0.3933}{W_{Na}}$

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t = counting time, minutes

W_t = weight of sample plus vial

W_f = weight of clean filter paper

W_v = weight of empty vial

0.3933 = conversion weight of NaCl to weight of Na

2.2×10^6 = conversion factor from dpm to μc

METHOD 1285

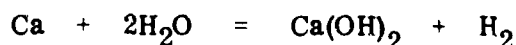
DETERMINATION OF TRITIUM IN WATER

1. SCOPE

This method is used to determine the tritium content of water.

2. SUMMARY OF METHOD

The tritium determination is based upon the measurement of the ionization produced by tritium, as the gas, in an ionization chamber. However, the samples to be analyzed are in liquid form and consequently it is necessary to dissociate the water into its component gases. The hydrogen generator used for dissociation of the water molecules depends upon the following reaction occurring between water and calcium metal.



This reaction, if uncontrolled, results in the evolution of large amounts of heat and hydrogen. Hence, care must be exercised in controlling the reaction.

3. APPARATUS

Hydrogen generation apparatus as shown in Figure 1285-1.

Mechanical vacuum pump.

Vacuum gauge.

Ionization chamber 500- to 1000-ml volume.

Cary Vibrating Reed Electrometer, Model 32.

45- or 90-volt dry cell.

Cylinder of hydrogen with reducing and metering valves.

4. REAGENTS

Calcium metal, shot or turnings.

5. CALIBRATION PROCEDURE

5.1 Carefully open one of the National Bureau of Standards' vials of tritiated water. Take a 1-ml aliquot and place in a 100-ml volumetric flask and dilute to the mark using deionized water (preferably not from the RWDS system). Concentration will be approximately $0.2 \mu\text{C/ml}$.

Denote this Standard "A".

5.2 Dilute 20 ml of Standard "A" to 100 ml using a clean volumetric flask. This is Standard "B".

5.3 Using an aliquot of Standard "A" and following the procedures given below, determine the equilibrium voltage reading for each turret resistor setting.

5.4 Repeat Step 5.3 using an aliquot of Standard "B".

5.5 Repeat Step 5.3 using the water employed to prepare the standards. This is a reagent blank determination.

5.6 The calibration determination should be made at the following times:

- a) When a new system is used for evacuating and filling the chamber.
- b) When a new ionization chamber is used.
- c) When the electrometer has been repaired, altered, or replaced.

d) Periodically to determine if the calibration has changed.

6. ANALYTICAL PROCEDURE

6.1 Assemble the ionization chamber and hydrogen generator as shown

in Figure 1285-1. All glassware must be completely dry. Use care in handling glassware. Gloves should be worn when assembling the apparatus - which must be used in an operating hood. The drying tube must contain dry silica gel (blue in color) and all glass joints should be sealed only with silicone stopcock grease.

6.2 Open the stopcock to the ionization chamber (H) and valve to the vacuum pump (F), close the stopcock on sample buret (B). Handle these valves gently to avoid breaking the seals, damage to the valves, etc.

6.3 Start the vacuum pump and operate with the valve to the vacuum pump (F) open until the gauge (E) reads half scale. Immediately close the valve to the vacuum pump and turn off the pump.

6.4 Observe the vacuum gauge for 1 minute. If the gauge goes down, there is a leak in the system. If there is a leak in the system, carefully release the vacuum by opening valve (B) a little at a time. Do not open this valve wide open. Check all joints in the glassware and connections to rubber hoses for proper seal. Repeat Steps 6.1 through 6.4 until the vacuum holds.

6.5 Release the vacuum carefully by opening valve (B) a little at a time. Allow the gauge reading to return to zero.

- 6.6 Estimate the volume of the apparatus (ionization chamber gas generator, and connecting lines).
- 6.7 Remove the sample buret (B) and using blunt forceps add 0.9 to 1 gm of calcium metal per liter of apparatus volume to the reaction flask.
- 6.8 Replace the sample buret(B), close valve (B), open valve (F) and evacuate the entire system including the ionization chamber until the vacuum gauge reads full scale. Immediately close the valve (F) to the vacuum pump and turn off the vacuum pump. Check the system for leaks. If there is a leak, repeat Steps 6.1 through 6.4 until the vacuum holds.
- 6.9 Add 10 ml of sample to be analyzed to the sample buret (B).
Carefully open stopcock (B) and add the sample in small increments (5 - 10 drops at one time) to the reaction flask. An ice bath around the reaction flask will slow the reaction down permitting a more rapid addition of the water being analyzed.
- 6.10 Continue adding the water sample until sufficient hydrogen has been generated to return the pressure to atmospheric as indicated by the vacuum gauge (E). Excess hydrogen will open the relief valve (G) thereby preventing a pressure above 1 atmosphere from building up in the apparatus. Tap the pop-off valve gently to be sure there is no pressure buildup in the system.

NOTE

When one atmosphere of hydrogen has been evolved, the calcium should be about expended and 1.6 ml of water will have been used.

6.11 When the vacuum gauge (E) reads zero, close the valve on the ionization chamber (H). This should be done only when there is no pressure differential between the system and the outside (or Hood) pressure. The chamber now contains a gaseous sample. The tritium content can now be analyzed as described in the procedure for the vibrating reed electrometer. Record equilibrium voltage and turret setting.

6.12 Disassemble parts (A), (B) and (C) of the hydrogen generator, observing precautionary measures necessary for handling contaminated glassware, i.e., gloves, care in handling to avoid breakage, etc. Empty contents of generator flask (A) into the waste bottle.

6.13 Thoroughly wash glassware pieces (A), (B), and (C) and place in a drying oven preheated to about 110°C. Handle hot glassware with asbestos gloves. Be sure all equipment is dry before using it again.

6.14 After the sample in the ionization chamber has been assayed, reassemble the apparatus with the ion chamber as in Step 6.1. Evacuate the entire system and flush with air five times before using the apparatus for a new assay.

7. PRECAUTIONS

7.1 Tritium gas exchanges with hydrogen atoms in organic compounds and in water. If the hydrogen (tritium) generated in the reaction is permitted to remain in contact with any residual water in the reaction flask, exchange of tritium with protons in the water will occur. However, if the conditions of temperature, pressure, volume of water, and time are reproducible for gaseous tritium samples of uniform composition, the loss by exchange will be comparable. The standard solution, therefore, must be run identically to the standards.

7.2 If water is inadvertently added too rapidly in Step 6.9 a violent reaction results. The ice bath will help to control the reactor.

7.3 A rapid reaction will cause water droplets to carry over to the drying tube and necessitate replacement of the desiccant before using again. It is essential that no moisture be carried beyond the drying tube and into the ionization chamber.

7.4 The tritium-contaminated water is decomposed to release tritium in the gaseous form. The reaction should be carried out only in a hood with an exhaust fan operating. Do not inhale the vapors coming from the disassembled apparatus. Do not generate hydrogen with

an open flame or sparking equipment in operation as hydrogen is a very flammable gas.

8. BACKGROUND

8.1 Background readings may be due to three sources: (a) contamination within the ionization chamber, and (b) external contamination of the ionization chamber, and (c) external radiation from nearby sources as well as cosmic radiation. The background reading should from nearby sources as well as cosmic radiation. The background reading should be low compared to the sample reading but should be determined using non-plant water or another source of water if available; or hydrogen may be obtained from a tank of hydrogen gas.

8.2 The following steps are to be followed if tank hydrogen is used:

8.2.1 Assemble the apparatus as for the hydrogen generation from the water sample. However, in place of the sample buret insert a one hole stopper fitted with a short piece of metal tubing which is connected to a cylinder of hydrogen fitted with a tank pressure gauge and reducing valve with a low range pressure gauge to prevent over-pressurization of the system.

8.2.2 Valve off the hydrogen cylinder and with valve (B) open evacuate the entire system including the ionization chamber.

When the gauge reads full scale, turn off the valve to the vacuum pump

(F). Turn off the vacuum pump.

8.2.3 Check the system for leaks and if the system is tight,
slowly add hydrogen until the gauge reads zero.

8.2.4 When the gauge on the hydrogen generation system reads
zero, turn off the hydrogen tank. Tap the pop-off valve (G)
lightly to make certain the system is at atmospheric pressure. Close the
valve (H) on the ionization chamber.

8.2.5 Connect the ionization chamber to the electrometer and
obtain the background reading.

9. CALCULATIONS

9.1 Since the same amount of radioactivity can produce a current that
will give a voltage reading on more than one turret resistance
setting, a set of standard curves must be determined, one for each turret
setting.

9.2 Calculate the voltage reading for the standard from the data
obtained for each turret setting as follows:

$$\text{Corrected } E_{s1} = E_{s1}$$

$$\text{Corrected } E_{s2} = E_{s2}$$

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Where E_{s_1} , E_{s_2} = readings obtained from 2 standards
at the same turret settings

E_b = reading obtained for the blank (water
used for standard dilution) or tank
hydrogen.

If the water used for dilution has a sufficiently low tritium content,
the correction for E_b may be neglected.

9.3 Using the equilibrium voltage reading obtained for the sample,
determine the tritium concentration from the standard curves.

9.4 Using the equilibrium voltage obtained for the background,
determine the tritium concentration from the standard curves.

10. OPERATION OF VIBRATING REED ELECTROMETER

10.1 Summary of Method

10.1.1 When radiation reacts with air, there is usually one ion pair (electron and positive charged particle) produced per 32.5 electron volts of absorbed energy. For a tritium beta particle some 150 electrons would be produced. These radiation produced electrons, collected in an ionization chamber, produce the current that is detected by the measuring system. Since the number of electrons is directly proportional to the amount of radiation in the chamber, the amount of current produced by the ionization chamber and measured by the electrical measuring system is directly proportional to the amount of radioactivity in the ionization chamber.

10.1.2 The collection of electrons in an ionization chamber is a relatively simple matter. The electrons, which are very mobile, move toward and collect on any positively charged surface. This collection area is usually a small polished rod somewhere near the center of the chamber. In practice, the wall of the chamber is given a negative charge from a battery of between 45 and 300 volts, and the center electrode is kept fairly close to zero or ground potential. The electrons inside the chamber see this only as a voltage differential and are readily collected on the center electrode. The voltage over which electrons are adequately collected is usually very wide. However, too high a voltage

results in acceleration of the electrons when they interact with molecules on their way to the collecting wire. The result is an erroneously high reading.

10.1.3 The electrons moving toward the center wire result in a current that is bled off through a high resistance connected to the center electrode and a feedback system. This resistance draws off the current being produced in the ionization chamber so that an equilibrium voltage is reached at the collection electrode, which is a function of the amount of current being produced and the resistance itself.

10.1.4 The turret switch holder on the electrometer contains four resistors that may be changed readily by moving a lever so that a wide range of sensitivities may be used in the resistance-leak method for ionization chamber current measurement. An open and ground position are also included in the turret switch.

10.1.5 The electrometer is capable of reading from 0 to 10 volts.

The voltage selector functions merely as a scale expander to enable one to read the voltage more accurately. The voltage obtained depends upon the current generated (I), which is proportional to radioactivity present and the resistance (R) added through the turret switch, or voltage = IR . For different values of R different voltages will be obtained.

10.2 Procedure

10.2.1 Fill the ionization chamber with hydrogen-tritium mixture generated as described under Step 6.10.

10.2.2 Connect the chamber to the electrometer.

10.2.3 Connect the negative terminal of 45- or 90-volt battery to the outer shell of the chamber and the positive lead to the ground terminal on the electrometer.

10.2.4 Having the turret switch in the open position, zero the instrument. This zero is crude to compensate for electronic drift or noise.

10.2.5 Having the turret switch in the ground position, again zero the instrument using the zero control.

10.2.6 Set the voltage selector to the highest range (10 volt) and the turret switch to the lowest resistance position. (The resistance of each position can be determined by looking in the turret switch.) Wait until the meter reading has reached a constant value. This is the equilibrium voltage E_g . Record this voltage.

10.2.7 If no reading is obtained, switch the voltage selector to the 1-volt range and wait until the meter reading has reached a constant value.

10.2.8 If no reading is obtained on the 1-volt range, go to the 0.1-volt range and then to the 0.01-volt range if a reading is not obtained.

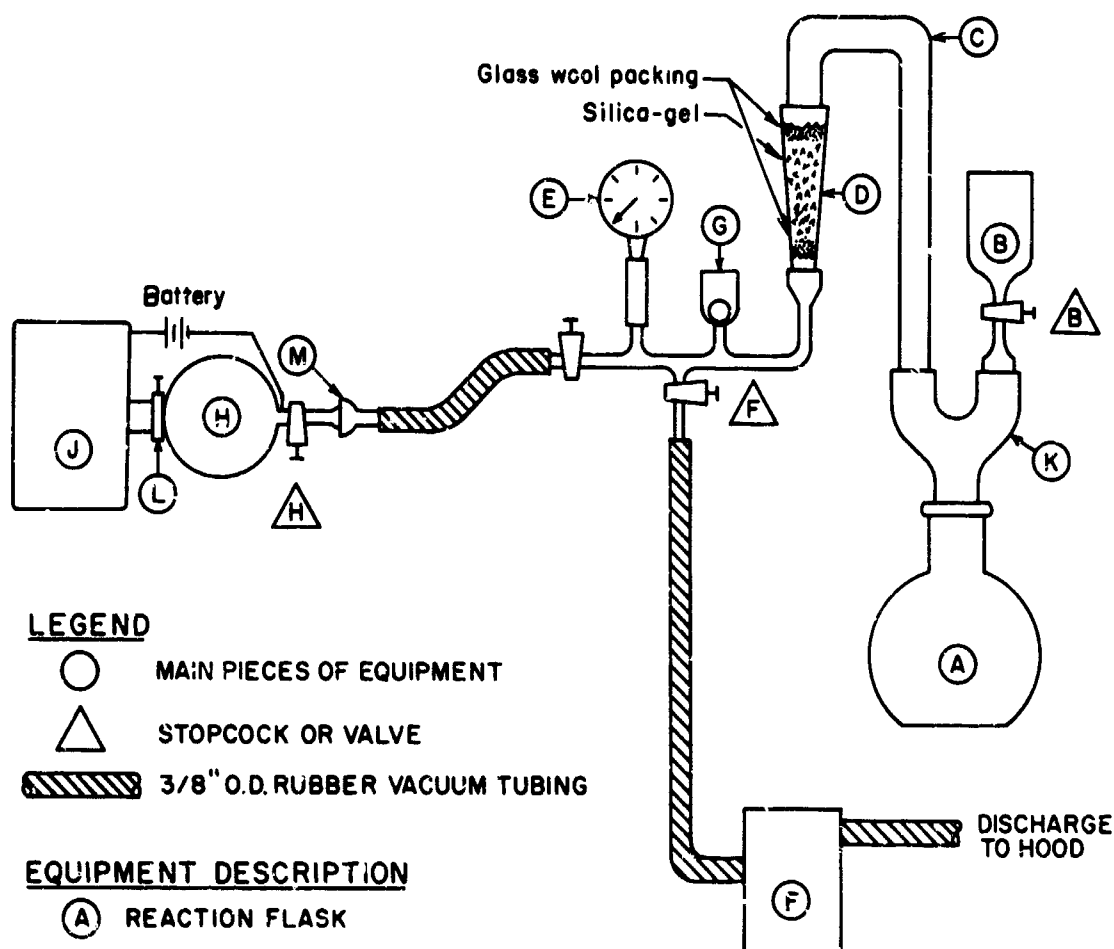
10.2.9 Repeat the above last four steps for each turret setting.

CAUTION

If at any time the instrument is permitted to read off scale, subsequent readings should be considered as questionable until test show that dielectric absorption currents are again negligible.

Calculations are performed as in Step 9.

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LEGEND



MAIN PIECES OF EQUIPMENT



STOPCOCK OR VALVE



3/8" O.D. RUBBER VACUUM TUBING

EQUIPMENT DESCRIPTION

- (A) REACTION FLASK
- (B) SAMPLE BURETTE
- (C) "U" TUBE
- (D) SILICA-GEL MOISTURE TRAP
- (E) VACUUM GAUGE, 0-30" MERCURY
- (F) VACUUM PUMP
- (G) SAFETY VALVE-RELIEVES AT ATMOSPHERIC PRESSURE
- (H) ONE LITER IONIZATION CHAMBER
- (J) VIBRATING REED ELECTROMETER
- (K) "Y" TUBE
- (L) MULTIPLE RESISTOR SWITCH
- (M) GLASS JOINT TO CONNECT RUBBER TUBING TO SPHERICAL JOINT ON IONIZATION CHAMBER

Figure 1245-1. Hydrogen Generator Apparatus.
1285-14

1 July 1966

METHOD 1291

DISSOLUTION OF CRUD

1. SCOPE

This method is used to put primary system crud into solution for analysis.

2. SUMMARY OF METHOD

The filtered crud and filter are ignited and burned. The sample is then fused with pyrosulfate, cooled, and the melt dissolved in acid.

3. APPARATUS

40-ml vycor crucible

Butane torch

150-ml beaker

Crucible tongs

Graduated 50-ml cylinder

Hot plate

4. REAGENTS

4.1 Potassium pyrosulfate, $K_2S_2O_7$, solid.

4.2 Hydrochloric acid, HCl, 6M.

5. PROCEDURE

5.1 Place the filter paper containing the crud in a vycor crucible.

5.2 Ignite over a gas burner for 10 minutes

5.3 Add just enough solid potassium pyrosulfate to cover the sample.

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- 5.4 Heat over a gas burner until a cherry red melt is formed. Cool.
- 5.5 Add 20 ml of 6M hydrochloric acid to the sample and heat gently on a hot plate until the solid is loosened. Transfer to a 150-ml beaker.
- 5.6 Boil until the solid has completely dissolved.
- 5.7 Dilute the solution to 50 ml with water. The crud sample is now in a form in which it is ready for chemical and/or radiochemical analyses.

SECTION 1300 - GENERAL CHEMICAL PROCEDURES
METHOD 1311

CONCENTRATIONS

1. GENERAL.

All solutions that are prepared for use in the laboratory will be labeled with respect to concentration, date of preparation, and the initials of the individual preparing the reagent. Concentrations may be expressed in several ways.

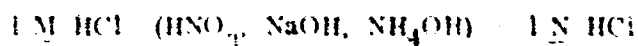
2. SOLUTIONS.

2.1 Molar Solution.

A molar solution consists of a 1-gm molecular weight of the substance dissolved in a solvent to make 1 liter of solution. The formula of the substance must be known before the molecular weight can be calculated. If water of hydration is present, it must be considered. The purchased reagent label normally provides this information.

2.2 Normal Solution.

A normal solution consists of 1 gm. equivalent weight of a substance dissolved in a solvent to make 1 liter of solution. In this case, not only the formula of the substance but also its stoichiometry in a specific reaction must be known. Thus, the normality of a solution may be different for different chemical reactions. When speaking of neutralization reactions, the equivalency is determined by the number of replaceable hydrogen or hydroxyl groups in the compound. Therefore, the following is true:



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2.3 Percentage.

The amount of a substance dissolved in a solution is sometimes expressed as percentage of the total weight (w/w) or total volume (v/v) of the solution. The molecular weight is not required, but the formula should be given to avoid confusion when a substance is available in more than one hydrated state. The ratio of molecular weights may be used to find the weight of a hydrated compound necessary to prepare a standard solution.

For example:

$$\frac{\text{CuSO}_4 \cdot 5\text{H}_2\text{O}}{\text{CuSO}_4} = \frac{(250)}{(160)} = 1.6 \text{ gm}$$

take 1.6 gm of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ to prepare 100 gm of 1% CuSO_4 solution (w/w).

2.4 Grams/Liter (mgm/ml)

A gm/liter solution is defined as 1 gm of the substance dissolved in a solvent to make 1 liter of solution. A similar definition applies to concentrations, such as mgm/ml and gm/ml. This method of expression is most often used with carrier solutions. The formula and molecular weight are required. To prepare 10 mgm/ml of stable I_2 carrier, dissolve

$$10 \text{ mgm} \times \frac{\text{NaI} (150)}{\text{I} (127)} = 11.8 \text{ mgm of sodium iodide}$$

to make 1 ml of solution. A 1-ppm solution contains 1 mgm of the substance per liter of solution.

2.5 Saturated Solutions

A saturated solution is defined as a solution containing the maximum amount of a substance solute capable of being dissolved in the solvent at a given temperature without supersaturation. A saturated solution is best prepared by adding an excess of the substance to the solvent and warming the solution; additional substance is added if necessary. Upon cooling of the solution, the excess solute will crystallize out; the solution is then saturated but not supersaturated. Excess solute must be present. Where extreme temperature fluctuations exist, care must be taken in using these solutions as additional recrystallization will occur at night. In the hot part of the day, some or all of the solute will dissolve and possibly result in a less than saturated solution.

METHOD 1321

ACIDS AND BASES

1. GENERAL

A large number of dilute acids and bases are used routinely in the laboratory. In nearly all cases, it is not necessary that the concentration of each solution be known exactly; but each solution should be prepared in a similar manner by each person.

2. SAFETY PRECAUTIONS

Wear rubber gloves when handling strong acids and bases.

Always pour the strong acid or base into water. The reverse procedure may cause violent splattering due to an exothermic reaction. Pour slowly, stirring to prevent splattering.

3. PROCEDURE

3.1 The following normalities are used for concentrated acids and bases:

Hydrochloric acid, HCl 37% - (12N)

Hydrofluoric acid, HF 48% - (29N)

Nitric acid, HNO_3 70-72% - (16N)

Sulfuric acid, H_2SO_4 98-100% - (36N)

Phosphoric acid H_3PO_4 85% - (36N)

Ammonium hydroxide NH_4OH 28-30% - (7.5N)

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3.2 Determine the total volume of dilute solution needed.

Normally this will be 500 or 1000 ml.

3.3 Calculate the volume of concentrated acid or base required
by the following formula:

$$\text{ml (conc.)} = \frac{\text{ml (dilute)} \times \underline{N} \text{ (dilute)}}{\underline{N} \text{ (conc.)}}$$

where N is corresponding normality.

3.4 Transfer the solution to a reagent bottle and initial and
label the bottle with the type of acid or base, the concentration, and the
date of the preparation.

METHOD 1331

PREPARATION OF CARRIERS

1. GENERAL

Usually, radioactive nuclides are present in solution at very low concentrations. It is common practice to dilute the radioactive material with its non-radioactive isotopes so that the chemistry of the radioactive material proceeds in a normal manner. The stable isotope material added is called isotopic carrier. In cases where there are radioactive nuclides present in addition to the desired constituent, carrier material is added for those nuclides as well, to prevent their being coprecipitated with the desired material. Such carriers are designated holdback carriers. In special cases, carrier-free manipulations are desired and specific procedures for these situations must be devised.

2. PROCEDURE

2.1 Determine the volume and concentration of carrier to be prepared. Normally the concentration is 10 mgm of the carrier element per ml of solution.

2.2 Calculate the amount of carrier compound required and weigh out the material. The weight should be accurate to ± 0.1 mgm.

2.3 Dissolve the compound in a solvent that is compatible with the procedure or application. Normally water is used although acid or

base may be required to maintain solubility.

2.4 While standardization may not be required, it is usually good practice to standardize the carriers so that the exact concentration is known if it is desired for a specific analysis. Standardization is best accomplished by duplicate or triplicate precautions of the carrier in the final form required by the procedure, such as precipitating a known amount of iodide as silver iodide and accurately weighing.

METHOD 1341

LABELING OF SOLUTIONS

1. STANDARD SOLUTIONS

All standard solutions of radioisotopes shall be labeled with the following information:

1.1 Standard: Radionuclides that are contained in the solution, e.g., Sr-90, shall be recorded.

1.2 Date: Date of standardization.

1.3 Concentration: The activity per unit volume shall be recorded.

The diluent shall also be indicated, e.g., 0.1N HCl. Units of volume are normally ml or liters while units of activity are normally dpm or μ c.

2. REAGENT SOLUTIONS

All reagent solutions shall be properly labeled on a conventional white label showing the reagent compound, concentration, diluent, date of preparation, and initials of the person who prepared the solution.

METHOD 1342

CLEANING OF GLASSWARE

1. PURPOSE

It is essential that the pipettes and glassware in the laboratory be thoroughly cleaned before use. In addition, two sets of pipettes are used, one for non-radioactive solutions and one for radioactive solutions. This separation of pipettes further assists in the prevention of contamination of carrier or non-radioactive solutions.

2. CLEANING

2.1 Immediately after use, the pipette will be rinsed in water and stored in 3N HNO_3 until cleaned. For a small number of pipettes, cleaning is best performed by use of a side arm suction flask and funnel.

2.2 Place the pipette tip down into a funnel inserted in a suction flask. Rinse the pipette inside and out with water, 3N HNO_3 , and then water. Final drying will be done in an oven or at room temperature.

2.3 Occasionally pipettes will not drain properly and require cleaning with sulfuric acid - dichromate solution. This cleaning solution is made by carefully diluting 75 ml of saturated sodium dichromate with 9 lb. of conc. sulfuric acid (one large bottle). Caution, considerable heat is generated so use a heavy walled, Pyrex, suction flask cooled with water.

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2.4 Other glassware will be rinsed with water and cleaned thoroughly with detergent such as "All" and water. Rinse thoroughly with tap water, then with distilled or deionized water.

METHOD 1343

LABORATORY SAFETY

1. GENERAL

1.1 Volatile solvents not in use will be stored in an approved safety can.

1.2 Open flames are not permitted in areas where volatile solvents are used or stored.

1.3 The cover of the centrifuge will not be lifted until rotation has stopped.

1.4 Never hold a stopper in your hand when using a cork borer.

1.5 All reagents will be clearly labeled.

1.6 Volatile or flammable materials will not be placed in ovens or furnaces.

1.7 Services (air, water, gas) will be turned off at the service cock when not in use.

1.8 Hot plates should be turned off when not in use; never assume such equipment is cold.

1.9 Every person working in the laboratory will be cognizant of the location and activity level of all radioactive materials in the area.

All material received will be smeared and surveyed.

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1.10 Spills of radioactive material will be cleaned immediately and the area checked for residual contamination.

1.11 Surface smears will be taken after every cleanup operation to check for alpha or beta contamination.

1.12 Work with high level samples will be confined to the hood area wherever possible.

1.13 Any injury in which the skin is lacerated provides direct access to the bloodstream for radioactive material. All injuries except those requiring immediate medical attention should be checked for contamination and decontaminated prior to reporting to the dispensary.

2. HOUSEKEEPING

2.1 Arrangement

2.1.1 A definite storage place will be provided for each item and the item placed therein when not in use. These storage areas will be kept neat, clean, and orderly.

2.1.2 Passageways will be kept clean, dry, and free of obstructions.

2.1.3 Arrangement of laboratory equipment will be planned so transportation of hot objects or solution in glassware is kept to a minimum. Tongs and/or asbestos gloves will be used when necessary.

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2.1.4 When choosing a site for an operation or the storage of materials, consideration will be given to the toxicity, flammability, fragility, and/or other hazardous properties of the materials being handled.

2.2 Equipment

2.2.1 Do not place equipment near the edge of the lab bench.

2.2.2 Dirty glassware should be washed promptly and returned to proper storage.

2.2.3 Equipment and working surfaces shall be cleaned frequently to prevent accumulation of dust.

2.2.4 Chipped and cracked glassware shall be disposed of in the proper manner.

2.2.5 Drawers and cabinet doors will be kept closed when not in use.

2.2.6 Functional equipment of all types will be kept in safe working condition.

2.2.7 Reagents and apparatus, after use, will be returned promptly to their place of storage.

2.2.8 Bench tops will be kept as clean as possible at all times.

2.3 Spills and Leaks

2.3.1 Spilled materials whether liquid or solid shall be cleaned up immediately and completely. All liquid spills will be handled as corrosive unless the material is definitely known to be noncorrosive.

2.3.2 If the spilled material is flammable, toxic, radioactive, or corrosive, the individual involved will take all precautionary measures necessary to guard personnel against injury during cleanup operation. If flammable, extinguish all flames in the vicinity immediately, and call for assistance.

2.3.3 Do not place corrosive or toxic materials in waste cans. Dilute any acidic or basic waste with water.

3. WASTE DISPOSAL

3.1 Non-radioactive waste paper will be disposed of in waste baskets and not allowed to accumulate on benches or tables. Radioactive dry waste will be placed in a special low-level waste contained for disposal.

3.2 Flammable or organic materials will not be discarded in the sink. They will be disposed of in a special container for organic solvents.

3.3 Aqueous radioactive wastes will be disposed of to the RWDS system special container provided for these materials.

3.4 Non-radioactive and all strong acidic or basic solutions will be diluted to less than one molar prior to being discarded in the sinks.

3.5 No solutions containing ammonia or carbon tetrachloride will be dumped into the laboratory sinks. These materials must not be placed in the RWDS system.

4. SAFETY PRACTICES

4.1 All fires and injuries will be reported immediately to the watch supervisor and the injuries logged in the HP and WC section log.

4.2 Warning signs should be used only when necessary and removed when no longer needed.

5. SAFETY EQUIPMENT

5.1 Emergency Equipment

5.1.1 CO₂ fire extinguishers are located near each door and one ANSUL type extinguisher. Best results may be obtained when the CO₂ is blown directly at the base of the burning material from a distance of 3 or 4 feet. Care should be exercised in the use of these extinguishers. Serious burns will result from handling the nozzle with the bare hands. Confined quarters can result in a significant buildup in the CO₂ content of the atmosphere, which presents a breathing hazard. Unused extinguishers will be checked periodically for leakage by the plant fire chief.

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5.1.2 The eye wash fountain in the hot lab will be used for irrigation of the eyes if corrosive liquids are splashed onto the face. The lab faucet with an attached hose will also provide a convenient method for flushing the eyes.

5.1.3 A safety shower is located in the decontamination package and should be used in the event acid is spilled on the clothing or the skin.

5.2 Protective Equipment

5.2.1 Laboratory coats or aprons will be worn by all persons handling chemicals or working in the laboratory. Laboratory coats will not be worn outside the lab.

5.2.2 Face shields will be used when handling large quantities of corrosive liquids.

5.2.3 Gloves will be worn when handling hot or corrosive material.

5.2.4 All acid digestion, fuming, etc., will be done in hoods with adequate air flow.

5.2.5 Hands will be washed after handling reagents and prior to leaving the laboratory.

METHOD 1344

LABORATORY MANIPULATIONS

The following are general guidelines to safe laboratory manipulations. Good judgment and careful assessment of the job before proceeding will assist greatly in providing a safe operation.

(a) Broken or chipped glassware will be disposed of or fire polished before using.

(b) Broken glass in the sink should be removed immediately. Never reach blindly into the sink or a cabinet. Assume the presence of broken glass until you confirm its absence.

(c) Extreme care should be used when inserting glass rod or tubing through stoppers. Fire polish the ends of the glass first, lubricate the glass with water or glycerin and wear leather gloves. Pull the tubing through rather than push it through whenever possible.

(d) Cut the stopper away from thermometers or tubing, particularly if the setup is old or has been subjected to heating.

(e) Beakers, etc., should be grasped around the sides. If too large for the hand to reach at least half way around, use two hands or tongs. Liter or larger beakers should be supported from the bottom when in use.

(f) Never fill bottles completely; leave air space for expansion.

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(g) Frozen stopcocks or ground glass stoppers should not be forced. Use either a stopcock puller or if the nature of the contents permits cool the plug and shell and then heat the shell with hot water momentarily. Pull the plug before it expands with the heat.

METHOD 1345

HANDLING OF CHEMICALS

The following are general safe-handling rules:

- (a) All unknown chemicals, reagents, samples, etc., are to be considered hazardous until assurance to the contrary is obtained. All chemicals are potentially poisonous and should be handled with respect.
- (b) Wash all spills of reagents quickly with large amount of water.
- (c) When opening bottles containing dissolved gases (NH_4OH , HCl etc.) care must be taken to prevent the inhalation of the fumes and avoid spraying of the material into the eyes.
- (d) Any procedure resulting in the evolution of fumes should be performed in a hood.
- (e) When extracting organic solvents, the pressure buildup may be relieved by removing the stopper or inverting the funnel and slowly opening the stopcock.
- (f) Hydrofluoric acid is very toxic and should never be handled without rubber gloves. Rinse the outside of the container carefully to remove any residual acid on the bottle.
- (g) All pipetting will be done with rubber bulbs. Mouth pipetting is forbidden.

SECTION 1400 - SUPPLIES

METHOD 1411

CHEMICALS

The following analytical grade chemicals are required for use in the laboratory:

1. Acetic acid, glacial
2. Acetone
3. Ammonium acetate
4. Ammonium carbonate
5. Ammonium chloride
6. Ammonium hydroxide
7. Ammonium molybdate
8. Ammonium oxalate
9. Ammonium vanadate
10. Barium nitrate
11. Barium hydroxide
12. Benzoic acid
13. Bismuth tri-iodide
14. Boric acid, powder
15. Bromophenol
16. Buffer powder, pH 4, 7, and 10

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17. Calcium metal, pellets
18. Calcium hydroxide
19. Carbon tetrachloride
20. Cesium nitrate
21. Chloroplatinic acid
22. Citric acid
23. Curcumin
24. Diphenyl carbazone
25. Ethyl alcohol, 95%, denatured
26. Ethyl ether, anhydrous (diethyl ether)
27. Eriochrome cyanine R
28. Europium oxide
29. Glucose
30. Glycerol
31. Hydrochloric acid
32. Hydrofluoric acid - 55%
33. Hydrogen peroxide
34. Hydroxylamine hydrochloride
35. Hydriodic acid - 47-55%
36. Indigo carmine
37. Ion exchange resin Dowex Ag 1-X10, 100-200 mesh

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38. Iron, metal, powder
39. Lanthanum nitrate
40. Mercuric iodide
41. Mercuric nitrate
42. Nitric acid, concentrated
43. Oxalic acid
44. Phenolphthalein
45. O-Phenanthroline
46. Potassium carbonate
47. Potassium chloride
48. Potassium dichromate
49. Potassium hydroxide
50. Potassium iodide
51. Potassium iodate, KIO_3
52. Potassium phosphate, dihydrogen
53. Potassium phosphate, monohydrogen
54. Potassium pyrophosphate
55. Rubidium chloride
56. Silver nitrate
57. Starch indicator
58. Sodium bisulfite

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- 59. Sodium carbonate
- 60. Sodium chloride
- 61. Sodium fluoride
- 62. Sodium hydroxide
- 63. Sodium hypochlorite
- 64. Sodium iodide
- 65. Sodium nitrite
- 66. Sodium silicate
- 67. Sodium sulfite
- 68. Sodium thiosulfate
- 69. Sulfuric acid
- 70. Strontium nitrate
- 71. Urea
- 72. Yttrium oxide
- 73. Iodide
- 74. Sodium bicarbonate
- 75. Methyl orange
- 76. Sodium chromate
- 77. Ferric nitrate
- 78. Platinum chloride
- 79. Ferric chloride
- 80. Ortho toluidine tablets

METHOD 1421

GLASSWARE

The following glassware is required for use in the laboratory:

1. Beaker, Griffin, with spout, Pyrex, 100, 150, 250, 400, 800, 1000, and 3000 ml
2. Bottles, polyethylene, narrow-mouth, 8, 16, and 32 oz
3. Bottles, dropping, flat stopper, 60 ml and 32 oz
4. Bottles, reagent, Pyrex, blank, 250 ml
5. Bottles, reagent, Pyrex, amber, 250 ml
6. Bottles, washing, polyethylene, 16 and 32 oz
7. Centrifuge cones, Pyrex glass, heavy duty, 40 ml
8. Centrifuge cones, plastic, 50 ml
9. Cylinders, graduated Exax, 10, 25, 50, 100, 500, and 1000 ml
10. Cylinders, graduated, polystyrene, 10 ml, 100 ml
11. Dishes, evaporating, Pyrex, 90 ml, 100 ml
12. Dishes, evaporating, porcelain, 250, 1000, and 1500 ml
13. Flasks, Erlenmeyer 125 and 250 ml
14. Flasks, filtering with side-tube, Pyrex, 500, 1000, and 2000 ml
15. Funnel, separatory, Exax, Squibb pear shaped with stopper and stopcock, 60, 100, 125, and 250 ml

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16. Glass stirring rods, soft, 200 mm
17. Glass wool, Pyrex
18. Pipettes, volumetric, Pyrex, 1, 2, 5, 10, 20 ml
19. Teflon beakers, 100 ml
20. Buret, 25 and 50 ml
21. Buret, micro
22. Vials, specimen, with black molded plastic screw caps,
4 ml
23. Volumetric flasks, 10, 25, 50, 100, 250, and 1000 ml
24. Watch glasses, beaker covers, speed evap., 3, 4, 5, and
6 inches in diameter
25. Centrifuge cones, graduated, 50 ml

METHOD 1431

MISCELLANEOUS

The following miscellaneous items are required for use in the laboratory:

1. Asbestos board mats
2. Brushes, nylon bristle, assorted sizes
3. Filter paper, Whatman No. 42 for filter tower
4. Filter paper, millipore, 47 mm diameter, 0.45 micron
pore size
5. Forceps
6. Paper, pH
7. Pencils, non-run, black, red
8. Planchets, 2 inches diameter, 1/8-in. deep, stainless steel
9. Planchets, 1 inch diameter, 5/16-in. deep, stainless steel
10. Rings, support
11. Ring stands
12. Rubber bulbs
13. Scoopula, stainless steel
14. Sponge, cellulose
15. Stoppers, rubber, assorted sizes, with cork borer
16. Support, funnel, for four funnels
17. Stopcock grease, silicone

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18. Syringes, gas sampling
19. Syringes, hypodermic, 1, 5 ml
20. Tissues, cleansing, disposable
21. Tubing, glass Pyrex, assorted sizes
22. Tongs, beaker
23. Tubing, rubber, assorted sizes
24. Tubing, plastic assorted sizes
25. Fisher gas sampling flasks with and without side support
26. Centrifuge
27. Stirring Hot Plate

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This Manual is designed for use by Nuclear Power Plant Process Control Specialists. It contains health physics and water chemistry procedures for guidance in plant operation. Although this Manual cannot give detailed recommendations, necessary and sufficient for all conditions, it gives general recommendations suitable for typical plant use. Part 1 presents health physics procedures, as well as radiochemical analyses for health physics operations. Contained in this Section are standards of health and safety necessary in the operation of nuclear reactors, including personnel monitoring and access control, radioactive materials control and waste management, decontamination, radiological monitoring, and health physics and radiochemistry instrumentation. Part 2 contains general chemical procedures for analyzing the water in the Primary, Shield Water, and Secondary Systems. This water impurity control prevents equipment corrosion, thus serving to prolong the life of the equipment, insure maximum operating efficiency, and reduce maintenance time.		

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