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G-000-1104.4

RADIOLOGICAL SCIENCES LABORATORY RADIOANALYTICAL LABORATORY PROCEDURES VOL. 1

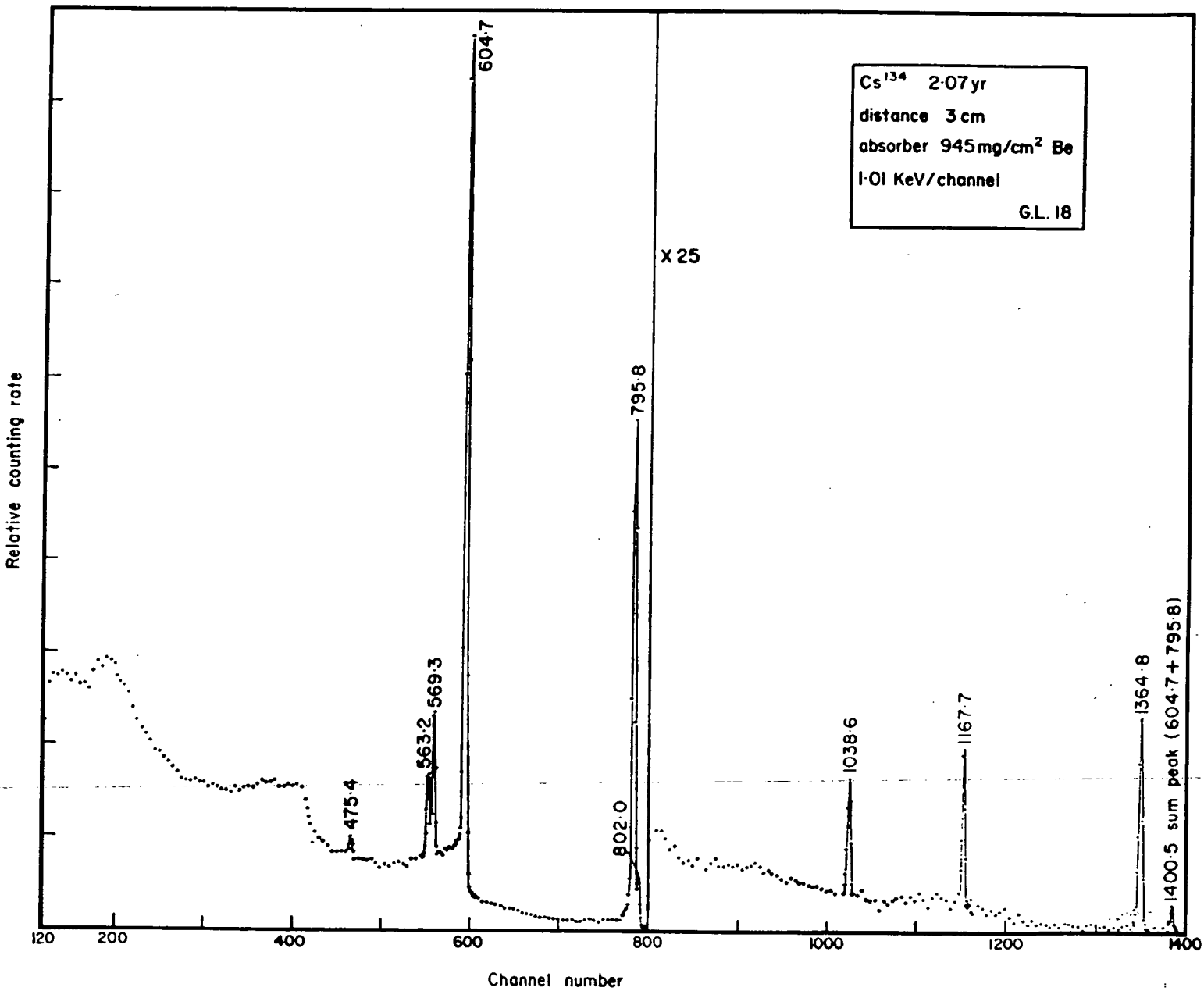
04/06/87

500
MANUAL

INTERNATIONAL TECHNOLOGY CORPORATION

Radiological Sciences Laboratory

Radioanalytical Laboratory Procedures



RESPONSIVE TO THE NEEDS OF ENVIRONMENTAL MANAGEMENT

IT/RSL QUALITY ASSURANCE MANUAL COMPENDIUM

The following list provides an index of Radioanalytical Laboratory Procedures contained in the IT/RSL QA Manual:

Number	Title	Revision	Date Issued
RSL-001	Acquisition and Use of Standard Reference Material	0	5-22-87
RSL-002	Standardization of Carrier Solutions	0	5-22-87
RSL-102	Operation of Alpha Spectrometer Systems	0	5-22-87
RSL-103	Calibration of Liquid Scintillation Counting Systems	0	5-22-87
RSL-104	Operation of Low Background Alpha/Beta Counting Instruments	0	5-22-87
RSL-105	Calibration of Alpha Spectrometer Systems	0	5-22-87
RSL-106	Calibration of Alpha/Beta Counting Instruments	0	5-22-87
RSL-107	Operation of Liquid Scintillation Counting Instruments	0	5-22-87
RSL-108	Operation of Alpha Scintillation Counting Instruments	0	5-22-87
RSL-109	Background Determination of a Germanium Detector System	0	6-23-87
RSL-110	Energy Calibration of Computer-Assisted Germanium Spectroscopy Systems	0	5-22-87
RSL-111	Linearity Check of Germanium Gammaray Spectroscopy Systems	0	5-22-87
RSL-112	Operation of Germanium Gamma Spectroscopy System	0	6-23-87
RSL-113	Determination of Germanium Detector Efficiency Tables	0	8-28-87
RSL-114	Determination of Germanium Detector Counts Reproducibility	0	8-28-87

RSL-201	Samples Preparation	0	8-28-87
RSL-202	Extraction of Iron from Soil or Metallic Samples	0	6-23-87
RSL-301	Determination of Promethium - 147	0	6-23-87
RSL-302	Determination of Tritium in Aqueous Samples by Distillation	0	6-23-87
RSL-303	Determination of Phosphorus - 32 Activity	0	5-22-87
RSL-304	Determination of Selected Alpha Emitting Isotopes	0	6-23-87
RSL-305	Determination of Strontium - 89, 90 in Water Samples	0	6-23-87
RSL-306	Determination of Strontium - 89, 90 in Milk Samples	0	5-22-87
RSL-307	Determination of I-131 Activity in Milk and Water Samples	0	5-22-87
RSL-308	Determination of Gross Alpha and/or Beta Activity	0	5-22-87
RSL-309	Determination of Radium - 226, 228 in Aqueous Samples	0	6-23-87
RSL-310	Determination of Technicium - 99	1	11-12-87
RSL-311	Determination of Calcium - 45 by Chemical Separation	0	6-23-87
RSL-312	Determination of Iron - 55	0	6-23-87
RSL-313	Direct Counting of Tritium, Calcium - 45 by Liquid Scintillation Counting Systems	0	5-22-87
RSL-314	Determination of Polonium - 210	0	8-28-87
RSL-315	Determination of Lead - 210	0	6-23-87
RSL-316	Determination of Ruthenium - 106	0	6-23-87
RSL-501	Data Verification	0	5-22-87

RSL-601	Performing Intralaboratory Quality Control Analysis	0	5-22-87
RSL-602	Performing Interlaboratory Quality Control	0	5-22-87
RSL-603	Internal Surveillances and Audits	0	5-22-87
RSL-604	Software Quality Assurance	0	11-12-87
RSL-701	File Set-up and Inventory for Perry Nuclear Power Plant	0	5-22-87
RSL-702	Perry Nuclear Records Transfer	0	5-22-87
RSL-703	Storage and Maintenance of Records	0	5-22-87
RSL-801	Instructions for Glassware Cleaning Laboratory Housekeeping	0	5-22-87
RSL-802	Checking Laboratory Measuring and Test Equipment	0	5-22-87
RSL-901	Training and Qualifications of Laboratory Technicians	0	6-23-87
RSL-902	Q.A. Orientation of New Employees	0	6-23-87
RSL-903	Material and Equipment Procurement, Receipt, Storage and Control	0	5-22-87
RSL-904	Reporting of Defects and Noncompliances per 10 CFR 21	0	8-28-87
RSL-1001	Sample Receiving	0	5-22-87
RSL-1002	Radiological Screening and Classification of Mixed Waste Samples	0	5-22-87
RSL-1004	Sample and Data Flow	0	8-28-87
RSL-1005	Sample Transfer to the Shipping Department	0	11-12-87
RSL-2103	Instructions for the Calibration of the Duall pH Control System	0	8-28-87

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RADIOLOGICAL SCIENCES LABORATORY

RADIOANALYTICAL LABORATORY PROCEDURES

ISSUED TO _____ UNCONTROLLED COPY COPY NO. _____

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APPENDIX 1

RADIOLOGICAL SCIENCES LABORATORY
RADIOANALYTICAL LABORATORY PROCEDURES INDEX

Number	Title	Revision	Date Issued
RSL-001	Acquisition and Use of Standard Reference Material	0	5-22-87
RSL-002	Standardization of Carrier Solutions	0	5-22-87
RSL-102	Operation of Alpha Spectrometer Systems	0	5-22-87
RSL-103	Calibration of Liquid Scintillation Counting Systems	0	5-22-87
RSL-104	Operation of Low Background Alpha/ Beta Counting Instruments	0	5-22-87
RSL-105	Calibration of Alpha Spectrometer Systems	0	5-22-87
RSL-106	Calibration of Alpha/Beta Counting Instruments	0	5-22-87
RSL-107	Operation of Liquid Scintillation Counting Instruments	0	5-22-87
RSL-108	Operation of Alpha Scintillation Counting Instruments	0	5-22-87
RSL-109	Background Determination of a Germanium Detector System	0	6-23-87
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RSL-111	Linearity Check of Germanium Gamma-ray Spectroscopy Systems	0	5-22-87
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RSL-114	Determination of Germanium Detector Counts Reproducibility	0	8-28-87

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RSL-202	Extraction of Iron from Soil or Metallic Samples	0	6-23-87
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RSL-309	Determination of Radium-226,228 in Aqueous Samples	0	6-23-87
RSL-310	Determination of Technicium-99	1	11-12-87
RSL-311	Determination of Calcium-45 by Chemical Separation	0	6-23-87
RSL-312	Determination of Iron-55	0	6-23-87
RSL-313	Direct Counting of Tritium, Calcium-45 by Liquid Scintillation Counting Systems	0	5-22-87
RSL-314	Determination of Polonium-210	0	8-28-87
RSL-315	Determination of Lead-210	0	6-23-87
RSL-316	Determination of Ruthenium-106	0	6-23-87
RSL-317	Determination of Cerium Isotopes	0	6-23-87
RSL-501	Data Verification	0	5-22-87

RSL-601	Performing Intralaboratory Quality Control Analysis	0	5-22-87
RSL-602	Performing Interlaboratory Quality Control	0	5-22-87
RSL-603	Internal Surveillances and Audits	0	5-22-87
RSL-604	Software Quality Assurance	0	11-12-87
RSL-701	File Set-up and Inventory for Perry Nuclear Power Plant	0	5-22-87
RSL-702	Perry Nuclear Records Transfer	0	5-22-87
RSL-703	Storage and Maintenance of Records	0	5-22-87
RSL-801	Instructions for Glassware Cleaning Laboratory Housekeeping	0	5-22-87
RSL-802	Checking Laboratory Measuring and Test Equipment	0	5-22-87
RSL-901	Training and Qualifications of Laboratory Technicians	0	6-23-87
RSL-902	Q.A. Orientation of New Employees	0	6-23-87
RSL-903	Material and Equipment Procurement, Receipt, Storage and Control	0	5-22-87
RSL-904	Reporting of Defects and Noncompliances per 10 CFR 21	0	8-28-87
RSL-1001	Sample Receiving	0	5-22-87
RSL-1002	Radiological Screening and Classification of Mixed Waste Samples	0	5-22-87
RSL-1004	Sample and Data Flow	0	8-28-87
RSL-1005	Sample Transfer to the Shipping Deparment	0	11-12-87
RSL-2103	Instructions for the Calibration of the Duall pH Control System	0	8-28-87

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INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-001

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Acquisition and Use of Standard Reference Material

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Niess DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cupini DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED ISSUED COPY

REVISION: 0				
DATE: 4-06-87				

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PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (x) No ( )
If yes, enter paragraph number to reference insertion point: 10.1, 10.2, 10.3, 10.4

Procedure Title & Number (If exists) RSL-001, R0

ACQUISITION AND USE OF STANDARD REFERENCE MATERIAL

Description of experiment or method Add running total of activity on working solution and stock solution log. Add Log # and date to the disposal record. Add verified by to calculation form.

Time Period From: 8/28/87 To: Issue of RSL-001, R1 Samples Affected All from 8-28-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

See attachment

Submitted by: [signature]

Approved by: James T. Harvey, Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature], QA/QC/Director, ITAS

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TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 6
	REVISION: 0	DATE: 4-06-87

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Appendix 10.1

IT/RADIOLOGICAL SCIENCES LABORATORY
RADIOACTIVE MATERIAL INVENTORY CONTROL

Date Received: _____ Vendor ID: _____ Log#: _____

Form: Solid Liquid Gas

Isotope(s)	Amount	Concentration

Assay Date: _____

STOCK SOLUTION ID given: _____

Diluted with: _____ Amount: _____

USE:

DATE	AMOUNT TAKEN	FOR USE IN	ACTIVITY	RUNNING TOTAL	INITIALS

430-1-87

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 7
	REVISION: 0	DATE: 4-06-87

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Appendix 10.2

DILUTION CALCULATION

Log#: _____ Date: _____

Calculations made by: _____

Verified by: _____

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 8
	REVISION: 0	DATE: 4-06-87

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Appendix 10.3

IT/RADIOLOGICAL SCIENCES LABORATORY
WORKING SOLUTION LOG

Log# _____

Form: Solid Liquid Gas

Isotope(s)	Amount	Concentration

Assay Date: _____

WORKING SOLUTION ID given: _____

Diluted with: _____ Amount: _____

USE:

DATE	AMOUNT TAKEN	FOR USE IN	ACTIVITY	RUNNING TOTAL	INITIALS

430-1 87

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 9
	REVISION: 0	DATE: 4-06-87

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Appendix 10.4

DISPOSAL RECORD

Log # _____

Total volume used _____ Amount Remaining _____

Reason for disposal: _____

Method of disposal: _____

Final disposal approved by: _____

Removed from inventory and disposed by: _____

Date _____

1.0 PURPOSE

This procedure provides the requirements for the acquisition and receipt of radioactive reference standards; the preparation of dilutions; labeling; storage and control of standards and subsequent dilutions; and the disposal of outdated radioactive reference material.

2.0 SCOPE

This procedure applies to all radioactive reference standards that are acquired by IT/RSL for calibration and procedures testing.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the acquisition and receipt of radioactive reference standards, preparation of dilutions, labeling, and storage and control of standards and subsequent dilutions.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel who are experienced with this procedure and with associated equipment.

3.3 It is the responsibility of those persons performing this procedure to follow the procedure and report any abnormal results to the Laboratory Manager, or his designee.

3.4 It is the responsibility of the Health Physics Services Supervisor, or his designee, to dispose of radioactive materials and make appropriate data entries in the "Disposal" section of the "Radioactive Material Inventory" logbook.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 ITAS Quality Assurance Manual.

4.1.2 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.3 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.1.4 USNRC, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Program (Normal Operatons) - Effluent Streams and the Environment.

4.2 Standards

None

4.3 Procedures

4.3.1 RSL-1001, "Sample Receiving", IT/RSL Laboratory Procedures Manual (latest edition).

4.4 Other Publications

4.4.1 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.2 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0-160.0000 gram capacity)

5.2 Materials

5.2.1 Glass bottles; 50 ml, 100 ml with polyvinyl lined caps

5.3 Reagents

5.3.1 Nitric Acid (HNO_3), ACS reagent grade, concentrated

5.3.2 Hydrochloric Acid (HCl), ACS reagent grade, concentrated

5.3.3 1M Hydrochloric Acid: Add 85 ml of concentrated HCl to 915 ml of demineralized water. Mix thoroughly.

5.3.4 1M Nitric Acid: Add 65 ml of concentrated HNO_3 to 935 ml of demineralized water. Mix thoroughly.

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 3
	REVISION: 0	DATE: 4-06-87

6.0 PROCEDURE

6.1 Acquisition

6.1.1 Radioactive reference standards shall be obtained from the National Bureau of Standards (NBS), Environmental Protection Agency (EPA) and/or a NBS traceable source. The supplier shall provide necessary documentation which should include: radionuclide identity, source identification number, mass and volume, activity, assay date, half-life, and traceability statement where applicable.

6.2 Receipt

6.2.1 When a radioactive reference material is received, the procedure outlined in Procedure RSL-1001 shall be followed.

6.2.2 Specifications accompanying the source shall be entered on a "Radioactive Material Inventory Control" form (see appendix 10.1) in the "Radioactive Material Inventory" logbook.

6.2.3 The source is assigned a number which corresponds to the page number in the logbook (section 6.2.2). The number is marked on the source container. Any subsequent dilutions of this source shall be identified with the same reference number.

6.2.4 If dilutions will not be made immediately, the standard reference material source is placed in the "Radioactive Reference Standard" cabinet.

6.2.5 The original documents received with the radioactive reference material are sent to the QA office for filing.

6.2.6 Copies of all documents received with the radioactive reference material are given to the Laboratory Manager.

6.3 Dilution of Sources

6.3.1 The Laboratory Manager, or his designee, shall make calculations necessary to dilute the source. The calculations shall be entered on the "Calculations for Dilutions" sheet, (see appendix 10.2) in the "Radioactive Material Inventory" logbook.

6.3.2 The dilution of a source shall be carried out per the instructions of the Laboratory Manager, or his designee. Dilutions shall be made on a by weight basis.

6.3.3 When the initial dilution of the source is made, the resulting dilution is denoted stock solution of the original source. The stock solution of the source is used to calibrate instruments or to make up the Quality Control working standards.

6.3.4 Quality Control working standards shall be made up per the Laboratory Manager, or his designee's instructions. Working standards are also made by weight. Use appendix 10.3 and appendix 10.2, as necessary.

6.3.5 Quality Control working standards shall be identified as "Control Standard Dilutions" followed by arabic numerals in sequential order.

6.4 Labeling

6.4.1 After each dilution of the radioactive reference source or the preparation of Quality Control working standards, a label shall be immediately affixed to the bottle identifying the log page number of the original source per Section 6.2. In addition, the vendor ID number, dilution or solution identification, activity, isotopic content, diluent used assay date and expiration date (5 half-lives or 10 years, whichever is shorter) shall be identified on the label.

6.5 Inventory Control

6.5.1 When a quantity of a radioactive reference material stock solution is used for any purpose, appropriate entries shall be made in the "Radioactive Material Inventory Control" logbook. These entries shall include date, amount taken, use, and the initials of the responsible person.

6.6 Disposal

6.6.1 When a radioactive reference material is identified for disposal by the Laboratory Manager, or his designee, the supervisor of Health Physics Services shall be notified.

6.6.2 The Supervisor of Health Physics Services, or his designee, shall dispose of the radioactive material, making appropriate entries in the "Disposal Form" (appendix 10.4) of the "Radioactive Material Inventory Control" form in the "Radioactive Material Inventory" logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Quality Assurance staff shall perform periodic surveillances to determine compliance with this procedure by laboratory personnel.

8.1.2 The Quality Assurance staff shall maintain and control all original documents pertaining to the radioactive reference standards and copies of the "Radiation Material Inventory" logbook.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balance shall be checked to ensure calibration dates have not expired.

8.4.2 Balance shall be in control as determined by routine balance check procedures.

8.5 Certification

None

9.0 CALCULATIONS

9.1 As required and noted on appropriate form (appendix 10.2).

10.0 APPENDICES

Appendix 10.1

IT/RADIOLOGICAL SCIENCES LABORATORY
RADIOACTIVE MATERIAL INVENTORY CONTROL

Date Received: _____ Vendor ID: _____ Log #: _____

Form: Solid Liquid Gas Vendor: _____

Isotope(s)	Amount	Concentration	Assay Date: _____

STOCK SOLUTION ID Given: _____

Diluted with: _____ Amount: _____

Concentration: _____

USE:

DATE	AMOUNT TAKEN	FOR USE IN	ACTIVITY	INITIALS

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 7
	REVISION: 0	DATE: 4-06-87

Appendix 10.2

CALCULATIONS FOR DILUTIONS

Log #: _____ Date: _____

Calculations Made by: _____

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 8
	REVISION: 0	DATE: 4-06-87

Appendix 10.3

IT/RADIOLOGICAL SCIENCES LABORATORY
WORKING SOLUTION LOG

Log #: _____

Form: Solid Liquid Gas

Isotope(s)	Amount	Concentration	Assay Date: _____

WORKING SOLUTION ID Given: _____

Diluted with: _____ Amount: _____

USE:

DATE	AMOUNT TAKEN	FOR USE IN	ACTIVITY	INITIALS

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 9
	REVISION: 0	DATE: 4-06-87

Appendix 10.4

DISPOSAL RECORD

Total volume used: _____ Amount Remaining: _____

Reason for Disposal: _____

Method of Disposal: _____

Final disposal approved by: _____

Removed from inventory and disposed by: _____

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-002

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Standardization of Carrier Solutions

APPROVED: James T. Harney DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Mills DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cupin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-21-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.5, 6.1.5

Procedure Title & Number (If exists) RSL-002, R0

STANDARDIZATION OF CARRIER SOLUTIONS

Description of experiment or method Procedure clarification and modification to allow for use of currently available reagent.

Time Period From: 8/n/87 To: ~~//~~ Issue of RSL-002, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

5.3.5 ~~Ethyl Alcohol, 95 percent,~~ Ethanol (CH_3CH_2OH), 89% ACS reagent grade

6.1.5 Filter with weak vacuum through tared filter paper. Wash with deionized water followed by ~~ethyl alcohol~~ ethanol.

Submitted by: James M. Gillen

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.2.1, 6.1.5, 6.2.5, 6.3.6

Procedure Title & Number (If exists) RSL-002, R0

STANDARDIZATION OF CARRIER SOLUTIONS

Description of experiment or method Procedure clarification and modification to allow for use of currently available equipment

(Note: This change revision incorporates CH-1 (6.1.5)
ISSUE OF RSL-002, R1

Time Period From: 8/28/87 To: ++ Samples Affected All from 8-28-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

5.2.1 ~~Whatman 42 filter paper~~ Nylon Membrane Filter or glass fiber paper, as necessary

6.1.5 Filter with weak vacuum through tared glass fiber filter paper. Wash with deionized water followed by ~~ethyl alcohol~~ ethanol.

6.2.5 Filter through a tared ~~glass fiber~~ membrane filter. Wash with 5 ml deionized water followed by 5 ml of acetone.

6.3.6 Filter the sample through a tared ~~glass fiber~~ membrane filter. Wash with 1 ml of demineralized water and 5 ml of acetone.

Submitted by: James A. Dillard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John A. Lupfer, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Standardization of Carrier Solutions	NO: RSL-002	PAGE: 1
	REVISION: 0	DATE: 4-21-87

1.0 PURPOSE

This procedure provides an analytical method for the standardization of carrier solutions.

2.0 SCOPE

This analytical procedure applies to the standardization of carriers used in radiochemical procedures to determine chemical yield.

3.0 RESPONSIBILITIES

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed by the technician during the standardization of carriers.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Collected Radiochemical Procedures", J. Kleinberg, LA-1721, Third Edition, 1972.

4.4 Other Publications

4.4.1 "The Radiochemistry of Barium, Calcium and Strontium", D.N. Sunderman and C.W. Townley, NAS-NS-3010, National Academy of Science, January 1960.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Filtering apparatus

5.1.2 Centrifuge

5.2 Materials

5.2.1 Whatman 42 filter paper or glass fiber paper, as necessary

5.2.2 Centrifuge cones

5.3 Reagents

5.3.1 Iodine carrier prepared for use in iodine procedure

5.3.2 Nitric Acid (HNO_3), concentrated ACS reagent grade

5.3.3 Silver Nitrate solution prepared for use in iodine procedure

5.3.4 1 M Nitric Acid: add 63 ml of concentrated HNO_3 to 500 ml of demineralized water and dilute to 1 liter. Mix thoroughly.

5.3.5 Ethyl Alcohol, 95 percent, ACS reagent grade

5.3.6 2 M Ammonium Hydroxide (NH_4OH): Add 135 ml of concentrated NH_4OH to 500 ml of demineralized water and dilute to 1 liter. Mix thoroughly.

5.3.7 Strontium carrier solution prepared for use in strontium procedure

5.3.8 Yttrium carrier solution prepared for use in strontium procedure

5.3.9 Sodium sulfate (Na_2SO_4), 0.7 M, prepared for use in strontium procedure

5.3.10 Acetic Acid (CH_3COOH), glacial, ACS reagent grade

5.3.11 Oxalic Acid Solution, 0.4 M, prepared for use in strontium procedure

5.3.12 Sodium Diethylenetriaminepentaacetate (DTPA), 0.2 M, prepared for use in the strontium procedure

5.3.13 Acetone (CH_3COCH_3), ACS reagent grade

5.3.14 Strontium wash solution prepared for use in the strontium procedure

5.3.15 Phenophthalein indicator 1%, prepared for use in the strontium procedure

5.3.16 Metacresol purple indicator, 0.1%, prepared for use in the strontium procedure

5.3.17 Yttrium wash solution prepared for use in the strontium procedure

5.3.18 Calcium carrier solution prepared for use in the calcium procedure

5.3.19 Saturated ammonium oxalate solution prepared for use in the calcium procedure

6.0 PROCEDURE

6.1 Iodine Carrier - precipitated as AgI

6.1.1 Pipette 4 ml of carrier into 20 ml of deionized water in a 100-ml beaker. Run in triplicate.

6.1.2 Place a small magnetic stirring bar in the beaker and stir the solution on a magnetic stirrer. Add 2 ml of concentrated HNO_3.

TITLE: Standardization of Carrier Solutions	NO: RSL-002	PAGE: 4
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6.1.3 Add 5 ml of $AgNO_3$ solution while stirring. Heat solution until silver iodide coagulates.

6.1.4 Transfer to a centrifuge tube and centrifuge until complete separation is achieved. Discard the supernate just prior to filtering. Wash precipitate with 10 ml of 1 M HNO_3. Centrifuge and discard supernate. Slurry precipitate with 5 ml of demineralized water.

6.1.5 Filter with weak vacuum through tared filter paper. Wash with deionized water followed by ethyl alcohol.

6.1.6 Dry in oven at 80° C for 30 minutes, then in desiccator until weight is constant. See 9.1 to calculate carrier concentration.

6.2 Strontium Carrier

6.2.1 Pipette 1 ml of strontium carrier into a centrifuge cone containing 5 ml 0.2 M DTPA solution. Add 1 drop of phenophthalein indicator. Run in triplicate.

6.2.2 Add 2 M NH_4OH dropwise until slight pink color appears.

6.2.3 Add 5 ml of sodium sulfate solution and 3 ml of glacial acetic acid; mix.

6.2.4 Heat solution in hot water bath for 5 minutes; then cool for 10 minutes in an ice bath to ensure complete precipitation.

6.2.5 Filter through a tared glass fiber filter. Wash with 5 ml deionized water followed by 5 ml of acetone.

6.2.6 Dry the precipitate in a desiccator until the weight is constant. See 9.2 to calculate carrier concentration.

6.3 Yttrium Carrier

6.3.1 Pipette 1 ml of yttrium carrier into a centrifuge tube containing 5 ml of 1 M HNO_3. Run in triplicate.

6.3.2 Add 5 ml of 0.4 M oxalic acid, 1 drop of MCP indicator and adjust the pH to 1.6 with concentrated NH_4OH. Add 1 drop additional HH_4OH to where yellow color appears to precipitate yttrium oxalate.

6.3.3 Heat and cool the solution as in 6.2.4 above.

6.3.4 Centrifuge and discard the supernate.

6.3.5 Wash the precipitate with 3 ml of the yttrium wash solution.

430 1 87

6.3.6 Filter the sample through a tared glass fiber filter. Wash with 1 ml of demineralized water and 5 ml of acetone.

6.3.7 Dry the precipitate in a desiccator until the weight is constant. See 9.3 to calculate carrier concentration.

6.4 Calcium Carrier

6.4.1 Pipette 2 ml of calcium carrier into a centrifuge tube containing 5 ml of demineralized water. Run in triplicate.

6.4.2 Add 1 ml of 2 M ammonium hydroxide and heat solution in tube to 80° C in a hot water bath.

6.4.3 Add 3 ml of saturated ammonium oxalate dropwise.

6.4.4 Filter the sample through a tared glass fiber filter. Wash with three 10 ml portions of hot demineralized water and 10 ml of ethanol.

6.4.5 Dry the precipitate in a desiccator until the weight is constant. See 9.4 to calculate carrier concentration.

7.0 PRECISION AND ACCURACY

7.1 For any carriers, triplicate results shall agree within 2%. Repeat as necessary.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall be responsible for the actions of those technicians performing this procedure.

8.1.2 The Laboratory Manager shall periodically observe the technician during the performance of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.3 The Quality Assurance staff shall periodically review the carrier standardization logbook for compliance with this procedure.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria.

7.1 Precision specification

8.3 Material Monitoring

None

8.4 Equipment Montoring

None

8.5 Certification

None

9.0 CALCULATIONS

9.1 Calculation of Iodine Results

Carrier Concentration (as AgI mg/ml) = W/V

W = weight (mg) AgI recovered

V = volume (ml) I^- carrier added

NOTE: Record results in carrier standardization logbook.

9.2 Calculation of Strontium Results

Carrier Concentration (as $SrSO_4$ mg/ml) = W/V

W = weight (mg) $SrSO_4$ recovered

V = volume (ml) Sr^2 + carrier added

NOTE: Record results in the carrier standardization logbook.

9.3 Calculation of Yttrium Results

Carrier Concentration (as $Y_2(C_2O_4)_3$ mg/ml) = W/V

W = weight (mg) of $Y_2(C_2O_4)_3$ recovered

V = volume (ml) of Y^{+3} carrier added

NOTE: Record results in the carrier standardization logbook.

9.4 Calculation of Calcium Results

Carrier Concentration (as CaC_2O_4 mg/ml) = W/V

W = weight (mg) of CaC_2O_4 recovered

V = volume (ml) of Ca^{+2} carrier added

NOTE: Record results in the carrier standardization logbook.

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-102

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Operation of Alpha Spectrometer Systems

APPROVED: James T. Harney DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Reed DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cupian DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

429-1-87

TITLE: Operation of Alpha Spectrometer Systems	NO: RSL-102	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides instructions for the operation of the alpha spectrometer.

2.0 SCOPE

The procedure applies to the routine operation of all alpha spectroscopy systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this operating procedure is followed during the use of all alpha spectroscopy systems.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with associated equipment.

3.3 It is the responsibility of the technician to operate the instrument in accordance with this procedure and to report any abnormal results to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 Instruction Manual, Tennelec TC-256/257 Alpha Spectrometer, Tennelec, Inc. Oak Ridge, TN, 1984.

4.4.2 Operator Manual, ND-66 Multichannel Analyzer, Nuclear Data, Inc., Schawbury, IL, 1982.

4.4.3 Model 7401 Quad Alpha Spectrometer, Canberra Industries, Inc., Meriden, CT, 1983.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Alpha spectroscopy system with multichannel analyzer

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Establish the normal instrument settings for all controls. These settings are tabulated in the individual instrument logbook. Controls should be checked before each use of the instrument.

6.2 Enter all necessary information into the multichannel analyzer system.

6.3 Shut off the vacuum before opening the sample chamber.

6.4 Open each chamber and place the sample on the adjustable platform. Raise the platform so that the sample is next to the detector face.

6.5 Seal the chamber and turn on the vacuum to pump the chamber down.

6.6 Turn on the high voltage.

6.7 Initiate the count by turning on the multichannel analyzer (MCA).

6.8 When the sample has finished counting, record the data on the appropriate data sheet.

6.9 Erase the MCA memory; shut off the vacuum; remove the sample.

6.10 Repeat 6.2 to 6.9 as necessary to count remaining samples.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to issue.

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-103

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Calibration of Liquid Scintillation Counting Systems

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Kelly DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cupjes DATE: 4-28-87
RSL Director

COPY NO.: COPY ISSUED UNCONTROLLED

REVISION: 0				
DATE: 4-06-87				

TITLE: Calibration of Liquid Scintillation Counting systems	NO: RSL-103	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides instructions for determining the count reproducibility and background stability of the liquid scintillation counting systems.

2.0 SCOPE

This procedure applies to all liquid scintillation counting systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the check of the liquid scintillation counting system.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and the liquid scintillation counting system to be checked.

3.3 It is the responsibility of the technician performing this procedure to follow it and report any unusual results or trends in results to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 Instruction Manual, Model 2000 CA/LL Liquid Scintillation Spectrometer System, Packard Instruments, Inc., 1985.

4.4.2 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.3 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Liquid scintillation counting system

5.2 Materials

5.2.1 System tritium standard, unquenched

5.2.2 System blank

5.2.3 Black liquid scintillation vial

5.3 Reagents

None

TITLE: Calibration of Liquid Scintillation Counting systems	NO: RSL-103	PAGE: 3
	REVISION: 0	DATE: 4-06-87

6.0 PROCEDURE

6.1 Count Reproducibility Check

6.1.1 Place the appropriate unquenched tritium source in the counter and lower into counting position.

6.1.2 Count for the specified time as given in the instrument logbook.

6.1.3 Compare the value obtained from the count with the limits on the appropriate control chart.

6.1.4 Unless there was an obvious error in determining the value (i.e., incorrect source, source position, count time, etc.), apply the appropriate step below to the value.

6.1.5 Value $<2\sigma$, plot, record value and proceed to 6.1.10.

6.1.6 Value $>2\sigma$, plot and record the value, and obtain a second count by performing the same steps used in obtaining the first value.

6.1.7 When a second value is obtained in accordance with 6.1.1 and 6.1.2, apply the appropriate step below to the value.

6.1.8 Second value $<3\sigma$, plot and record value. Distinguish between the first and second values plotted on the control chart by using different representations for the points (i.e., use dots for first values and x's for second counts).

6.1.9 Second value $>3\sigma$, plot and record the value. Distinguish between the first and second values as described in 6.1.8. Take corrective action. (See Section 6.3)

6.1.10 Record and initial all necessary data in the logbook.

6.1.11 If all results are satisfactory, unload the source and return it to the proper storage location.

6.2 Background Check

6.2.1 An unquenched background blank is included in each group of samples. Since each group of samples is counted several times, the average background for the counting period should be calculated. The count time for backgrounds and the number of counts to be averaged are given in the instrument logbook.

6.2.2 Record the calculated background in the appropriate section of the logbook. Date and initial the entry. Plot the result on the corresponding background control chart.

6.2.3 Compare the result with the limits on the control chart.

6.2.4 If the result is within ±2σ of the expected value, the analyst may use the system average background for calculation of results. If the result is outside the ±2σ limit, use the determined background value and refer to 6.4 for corrective actions.

6.2.5 The technician should review the background control chart periodically to see if significant trends are developing. Notify the Laboratory Manager, or his designee, as necessary.

6.3 Corrective Actions for Poor Count Reproducibility Check Performance

6.3.1 Clean the standard vial with alcohol to remove dirt and fingerprints.

6.3.2 Check system settings.

6.3.3 Clean the sample compartment.

6.3.4 Repeat count reproducibility check.

6.3.5 Notify Laboratory Manager, or his designee, if the recount value is > ± 3 σ and proceed to section 6.5.

6.4 Corrective Actions for Poor Background Check Performance

6.4.1 Perform steps 6.3.1 through 6.3.3.

6.4.2 Check for electronic noise using a black vial.

6.4.3 Lower the black vial into the proper counting position and perform several 10-minute counts.

6.4.4 A count rate of more than 1-3 cpm is indicative of electronic noise.

6.4.5 Notify Laboratory Manager, or his designee, of problem.

6.5 Instrument Shutdown

6.5.1 If the instrument fails the daily quality control tests for background and counts reproducibility, and the corrective actions in 6.3 and 6.4 do not resolve the problem, the instrument must be declared Out of Service. Note this action in the system logbook. Appropriate IT/RSL forms shall be affixed to the instrument (see appendix 10.1).

6.5.2 The instrument may be placed back in use once the malfunction has been corrected and the above outlined quality control criteria have been passed. Note this action in the system logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 It shall be the responsibility of the technician staff to ensure that all test equipment used is calibrated and that the calibration is traceable to known standards, unless otherwise stated.

8.1.2 The Laboratory Manager shall be responsible for the actions of those technicians during the performance of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.3 The Quality Assurance staff shall periodically perform surveillances to determine compliance to this procedure and review all associated control charts for any long range trends in data.

8.1.4 The technician performing this procedure shall ensure that all equipment used is working properly and all instrument settings are set to the proper value or position.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.3

6.2.4

8.3 Material Monitoring

8.3.1 None

8.4 Equipment Monitoring

8.4.1 None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Calibration of Liquid Scintillation Counting Systems	NO: RSL-103	PAGE: 7
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APPENDIX 10.1

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

_____

_____

_____

_____

____ Work may continue with this item subject to the following limitations:

_____

_____

_____

_____

____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-104

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Operation of Low Background Alpha/Beta Counting Instruments

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Mill DATE: 5/4/87
ITAS QA Director

CONCURRED: John L. Caupin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

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PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 4.4.4; 6.3

Procedure Title & Number (If exists) RSL-104, R0

Operation of Low Background Alpha/Beta Counting Instruments

Description of experiment or method Add procedure for LB-4000

Time Period From: 11/24/87 To: Issue of R1 Samples Affected ALL

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

ADD the following steps:

4.4.4 "LB 4000 Instruction Manual", Tennelec, Inc., Oak Ridge, TN, 1986.

6.3 Tennelec LB 4000

6.3.1 Refer to 4.4.4 for a complete description of all instrument controls.

6.3.2 Normal operational settings for all controls are tabulated in the system logbook. Those controls used infrequently should be checked for proper settings before the instrument is used.

Submitted by: [signature]

Approved by: [signature], Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature], QA/QC/Director, ITAS

6.3.3 Load samples by rotating the position knob to the DOWN position and pulling the sample slide slowly out. Place sample planchets on the sample slide noting on the sample work sheet the counting position number and group designation.

CAUTION : Check the planchet position to be sure the planchet does not extend above the level of the planchet holder. Never allow a planchet to be loaded if it is too high.

Note: Segregate samples of expected high counting rates to counting groups not being used for low level counting.

6.3.4 Slowly insert sample slide into instrument and slowly rotate the positioning knob into the UP position.

6.3.5 Verify that the instrument clock indicates the correct time.

6.3.6 Position cursor in the display group box to be counted. Press F8 to clear previous count display. Verify that group counting times are correct. If counting times are to be changed, position cursor at time to be changed, type new time and press ENTER. Start counting by pressing F1.

6.3.7 Observe counting rate display to verify that counting has started. If the counting rate for a sample is disproportionally high for the group, contact the Laboratory Manager.

6.3.8 When counting is complete, record appropriate sample data on the work sheet and counting sheet. Remove samples from counter by rotating the position knob to the DOWN position and pulling the sample slide slowly out.

6.3.9 Repeat 6.3.3 to 6.3.8 as necessary to count remaining samples.

TITLE: Operation of Low Background Alpha/Beta Counting Instruments	NO: RSL-104	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides instructions for the operation of the low background alpha/beta counting instrumentation.

2.0 SCOPE

This procedure applies to the routine operation of all low-background alpha and/or beta counting instrumentation with gas flow proportional detectors.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the operation of any low-background alpha/beta counting system.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to operate the instrument in accordance with this procedure and to report any abnormalities to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criterion "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Alpha/Beta Systems, Model 2400", Operator's Manual, Canberra Industries, Meriden, CT, 1978.

4.4.2 "LB5100 Operating Instructions," Tennelec, Inc., Oak Ridge, TN, 1979.

4.4.3 "LB5100 Instruction Manual," Tennelec, Inc., Oak Ridge, TN, 1979.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Low Background Alpha/Beta Counting System

5.2 Materials

5.2.1 None

5.3 Reagents

5.3.1 None

6.0 PROCEDURE

6.1 Canberra Model 2401

6.1.1 Refer to 4.4.1 for a complete description of all instrument controls.

6.1.2 Normal operational settings for all controls are tabulated in each of the instrument logbooks. Controls should be checked for proper settings before an instrument is used.

6.1.3 A user program must be set up and running specific to the analysis to be performed. Refer to 4.4.1.

6.1.4 Before each sample is loaded into the slide, be sure the sample planchet does not extend above the level of the planchet holder. Never allow a planchet to be loaded if it is too high.

6.1.5 Load the sample and initiate the counting.

6.1.6 When counting is complete, record the sample results and remove the sample.

6.1.7 Repeat 6.1.4 to 6.1.6 as necessary to count remaining samples.

6.2 Tennelec LB-5100

6.2.1 Refer to 4.4.2 and 4.4.3 for a complete description of all instrument controls.

6.2.2 Normal operational settings for all controls are tabulated in the system logbook. Those controls used infrequently should be checked for proper settings before the instrument is used.

6.2.3 Planchet holder numbered 1 has been reserved for calibration purposes. Regular samples will not be placed in this holder.

6.2.4 Backgrounds will be interspersed with the samples in a fashion so that they will not interfere with samples that must be counted within a specified time.

6.2.5 Samples which have critical decay or ingrowth times (i.e., $^{89,90}Sr$ samples on the first count) must be assigned to the first available counting positions.

6.2.6 Each planchet holder is numbered. Be sure that the sample loaded into the number planchet holder corresponds with what has been recorded on the data sheet.

6.2.7 Before the sample is placed in the stack, be sure the sample planchet does not extend above the level of the planchet holder. Such a situation can cause a serious jam of the sample changer. If a jam occurs, depress the "Halt" button and notify the counting room supervisor.

6.2.8 To set up first and last sample, type "F" on the teletype. Answer the questions as prompted by the teletype. Refer to 4.4.2 for additional instrument capabilities.

6.2.9 Ensure that first sample to be counted is in the sample holder positioned to be counted next.

6.2.10 Push the advance (ADV) or Reverse (REV) button until the desired planchet holder is at the load position. Ensure that the correct group is counting.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to use.

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-105

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Calibration of Alpha Spectrometer Systems

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Mills DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cuffin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

TITLE: Calibration of Alpha Spectrometer Systems	NO: RSL-105	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides instructions for checking the energy calibration of the alpha spectrometer, determining if the counts of a standard source are reproducible and determining the stability of the background.

2.0 SCOPE

This procedure is applicable to all alpha spectrometer systems using a multichannel analyzer.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed when calibrating the alpha spectrometry system.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and the alpha spectroscopy system to be checked.

3.3 It is the responsibility of the technician performing this procedure to follow it and report any unusual results or trends in results to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.2 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Alpha Spectroscopy system

5.2 Materials

5.2.1 Alpha source - ^{230}Th

5.3 Reagents

None

6.0 PROCEDURE

6.1 Instructions for Energy Calilbration

TITLE: Calibration of Alpha Spectrometer Systems	NO: RSL-105	PAGE: 3
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6.1.1 Establish the general instrument settings as listed in the appropriate system logbook.

6.1.2 Mount the ^{230}Th source and acquire a spectrum for the specified time indicated in the logbook.

6.1.3 Check position of ^{230}Th peak to insure it falls in correct region.

6.1.4 Notify Laboratory Manager, or his designee, if peak is outside expected region.

6.2 Count Reproducibility Check

6.2.1 Total the counts in the ^{230}Th region. Calculate counter efficiency factor.

6.2.2 Compare the efficiency factor with the control limits specified on the control chart.

6.2.3 Unless there was an obvious error in determining the value (i.e., incorrect source, source position, count time, etc.) apply the appropriate step below to the value.

6.2.4 Value $<2\sigma$, plot and record value. Proceed to 6.2.9.

6.2.5 Value $>2\sigma$, plot and record the value, and obtain a second count by performing the same steps used in obtaining the first value.

6.2.6 When a second value is obtained in accordance with 6.1.1 through 6.2.1 apply the appropriate step below to the value.

6.2.7 Second value $<3\sigma$, plot and record value. Proceed to 6.2.9. Distinguish between the first and second values plotted on the control chart by using different representations for the points (i.e., use dots for first values and X's for second counts.

6.2.8 Second value $>3\sigma$, plot and record the value, take corrective action (see section 6.4). Distinguish between the first and second values as described in 6.2.7.

6.2.9 Record and initial all necessary data in the logbook.

6.2.10 Return source to the proper storage location if all results are satisfactory.

6.2.11 The technician should review the control chart periodically to see if significant trends are developing. Notify the Laboratory Manager, or his designee, as necesssary.

6.3 Detector Background

6.3.1 Establish the general instrument settings as listed in the appropriate system logbook.

6.3.2 Position the counting platform with a blank planchet under the surface barrier detector and acquire an alpha spectrum for 1000 minutes. Run blank planchets weekly, as a minimum, or more frequently as the counting schedule permits.

6.3.3 Visually inspect the spectrum for peaks or abnormalities. Report an unusual spectrum to the Laboratory Manager, or his designee, immediately.

6.3.4 Total the counts in the region of interest.

6.3.5 Compare the totaled counts with previous values specified in the logbook.

6.3.6 Notify the Laboratory Manager, or his designee, if the values are different by more than a factor of 3. Record values in system logbook.

6.4 Corrective Action

6.4.1 Check instrument settings.

6.4.2 Check cable connections.

6.4.3 Check detector high voltage and system vacuum.

6.4.4 Check source position (if applicable).

6.4.5 Repeat appropriate section of procedure.

6.4.6 Report abnormal results to Laboratory Manager, or his designee.

6.5 Instrument Shutdown

6.5.1 If the alpha spectrometer fails the energy calibration or reproducibility check, and the corrective action described in Section 6.4 does not resolve the problem, the instrument must be declared Out of Service. Note this action in the system logbook. Appropriate IT/RSL forms shall be affixed to the instrument (see appendix 10.1).

6.5.2 The instrument may be placed back in use once the malfunction has been corrected and the above outlined quality control criteria have been passed. Note this action in the system logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 It is the responsibility of the technician to ensure that all test equipment used is calibrated and that the calibration is traceable to known standards, unless otherwise stated.

8.1.2 The Laboratory Manager shall be responsible for the actions of those technicians performing this procedure.

8.1.3 The Laboratory Manager shall periodically observe the technician during the performance of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.4 The Quality Assurance staff shall periodically perform surveillances to determine compliance to this procedure and review all associated control charts for any long range trends in data.

8.1.5 The technician performing this procedure shall ensure that all equipment used is working properly and all instrument settings are set to the proper value or position.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.2.4 Control chart limits

6.2.5 Control chart limits

6.3.6 Control limits

8.3 Material Monitoring

8.3.1 None

8.4 Equipment Monitoring

8.4.1 None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

APPENDIX 10.1

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

_____

_____

_____

_____

____ Work may continue with this item subject to the following limitations:

_____

_____

_____

_____

____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-106

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Calibration of Alpha/Beta Counting Instruments

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Mills DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cuffin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.4.1

Procedure Title & Number (If exists) RSL-106, R0
Calibration of Alpha/Beta Counting Instruments

Description of experiment or method Procedure clarification of Alpha background check plot

Time Period From: 5/22/87 To: ~~11~~ Issue RSL-106, R1 Samples Affected N/A

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

10-30-87

6.4.1 Proceed as in steps 6.3.1 through 6.3.4 to complete this check.
Omit plotting of alpha background values.

Submitted by: James Millard

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. A. Cooper, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS
for 11/15/87

TITLE: Calibration of Alpha/Beta Counting Instruments	NO: RSL-106	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides instructions for the calibration of alpha and/or beta counting instrumentation by periodically checking a reference standard in a reproducible manner and by monitoring the day-to-day background associated with each instrument.

2.0 SCOPE

This procedure applies to all low background systems used in counting alpha and/or beta radiation.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the check of any low background alpha/beta counting system.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and the low background alpha/beta counting system to be checked.

3.3 It is the responsibility of the technician performing this procedure to follow it and report any abnormal results or trends in results to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "LB5100 Operating Instructions," Tennelec, Inc., Oak Ridge, TN 1979.

4.4.2 Instruction Manual, Model 2000 Portable Scaler, Ludlum Measurements, Inc., Sweetwater, TX, 1982.

4.4.3 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

4.4.4 Alpha/Beta Systems, Model 2401, Operators Manual, Canberra Industries, Meniden, CT, 1978.

4.4.5 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Low Background Alpha/Beta Counting System

5.2 Materials

5.2.1 Alpha check source - normally ^{230}Th

5.2.2 Beta check source - normally ^{90}Sr-^{90}Y

5.3 Reagents

None

6.0 PROCEDURE

6.1 Beta Count Reproducibility Check

6.1.1 Load the beta check source into counting position in the manner prescribed by the instrument's operating procedure and count it for the specified time. The geometry must be reproducible. The check source and proper count time are given in the appropriate instrument logbook.

6.1.2 Compare the result with the limits on instrument control chart.

6.1.3 Unless there was an obvious error in determining the value (i.e., incorrect source, source position, count time, etc.), apply the appropriate step below to the value.

6.1.4 If the value falls within $\pm 2\sigma$ of the mean, plot and record the value and proceed to 6.1.10.

6.1.5 If the value falls outside $\pm 2\sigma$, plot and record the value, and obtain a second count by performing the same steps used in obtaining the first value.

6.1.6 When a second value is obtained in accordance with 6.1.5, apply the appropriate step below to the value.

6.1.7 If the second value falls within $\pm 3\sigma$ of the mean, plot and record the value and proceed to 6.1.9. Distinguish between the first and second values plotted on the control chart by using different representations for the points (i.e., use dots for the first values and x's for the second counts).

6.1.8 If the second value falls outside ±3σ, plot and record the value and notify the Laboratory Manager, or his designee, so that corrective action can be taken. Distinguish between the first and second values as described in 6.1.7. Refer to 6.5 for corrective actions.

6.1.9 Record and initial all necessary data in the logbook.

6.1.10 If all results are satisfactory, unload the check source and return it to the proper storage location.

6.2 Alpha Count Reproducibility Check

6.2.1 Load the alpha check source into counting position in the manner prescribed by the instrument's operation procedure and count it for the specified time. The check source and proper count time are given in the appropriate instrument logbook.

6.2.2 Proceed as in steps 6.1.2 through 6.1.10 to complete this check.

6.3 Beta Background Check

6.3.1 Background counts are to be interspersed daily throughout the routine sample load for the instrument. Calculate the average background in total counts. The count time for backgrounds is given in the appropriate instrument logbook.

6.3.2 Record the background in the appropriate section of the instrument logbook. Date and initial the entry. Plot the value on the instrument background control chart.

6.3.3 Compare the background calculated for the day with the mean value on the background control chart. If the daily value is within ±2σ of the mean, use the mean background value for calculations. If the daily figure falls outside the ±2σ variation, use the actual background value for calculating sample results.

6.3.4 If the value of daily background is >3σ above or below the mean average background, take corrective action as described in 6.6.

6.4 Alpha Background Check

6.4.1 Proceed as in steps 6.3.1 through 6.3.4 to complete this check.

6.5 Corrective Actions for Poor Count Reproducibility Check Performance

6.5.1 Check the source positioning.

6.5.2 Check the gas supply for pressure variations (if applicable).

6.5.3 Check the gas by changing to a different cylinder. Bad gas usually is indicated when several instruments operating off the same supply change drastically at the same time.

6.5.4 Check the instrument high voltage setting.

6.5.5 Check the instrument cabling and grounding to see if the high voltage is arcing.

6.5.6 For instruments using pulse height discrimination to separate alpha and beta, check the alpha/beta crossover.

6.5.7 Repeat count reproducibility check.

6.5.8 Notify Laboratory Manager, or his designee, if the recount value is $> \pm 3\sigma$ and proceed to section 6.7.

6.6 Corrective Action for Poor Background Check Performance

6.6.1 Perform checks described in steps 6.5.2 through 6.5.7.

6.6.2 Clean the sample counting compartment and planchet holders.

6.6.3 Thoroughly but carefully clean the detector window with water or ethanol. Notify the Laboratory Manager prior to performing this step.

6.6.4 Check the instrument preamplifiers and other signal processing circuits for malfunction.

6.6.5 Check the instrument guard detector (in a dual detector system).

6.6.6 Repeat appropriate background check.

6.6.7 Notify the Laboratory Manager, or his designee, if the recount value is $> \pm 3\sigma$ and proceed to section 6.7.

6.7 Instrument Shutdown

6.7.1 If the instrument fails the daily quality control tests for background or count reproducibility, and the corrective actions in 6.5 and 6.6 do not resolve the problem, declare the instrument Out of Service. Note this in the logbook. Appropriate IT/RSL forms shall be affixed to the instrument (see appendix 10.1).

6.7.2 The instrument may be placed back in service once the malfunction has been corrected and the above outlined quality control criteria have been passed.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 It shall be the responsibility of the technician to ensure that all test equipment used is calibrated and that the calibration is traceable to known standards, unless otherwise stated.

8.1.2 The Laboratory Manager shall be responsible for the actions of those technicians performing this procedure.

8.1.3 The Laboratory Manager shall periodically observe the technician during the performance of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.4 The Quality Assurance staff shall periodically perform surveillances to determine compliance to this procedure and review all associated control charts for any long range trends in data.

8.1.5 The technician performing this procedure shall ensure that all equipment used is working properly and all instrument settings are set to the proper value or position.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.4 Control chart limits

6.1.5 Control chart limits

6.1.7 Control chart limits

6.1.8 Control chart limits

6.2.2 Control chart limits

6.3.3 Control chart limits

6.3.4 Control chart limits

6.4.1 Control chart limits

6.5.8 Control chart limits

6.6.7 Control chart limits

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Calibration of Alpha/Beta Counting Instruments	NO: RSL-106	PAGE: 8
	REVISION: 0	DATE: 4-06-87

APPENDIX 10.1
Nonconforming Item Tag

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

_____

_____

_____

_____

____ Work may continue with this item subject to the following limitations:

_____

_____

_____

_____

____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-107

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Operation of Liquid Scintillation Counting Instruments

APPROVED: James T. Harney DATE: 4-28-87
Laboratory Manager

APPROVED: Carl Miller DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cupin DATE: 4-28-87
RSL Director

COPY NO.: COPY ISSUED: UNCONTRO

REVISION: 0				
DATE: 4-06-87				

TITLE: Operation of Liquid Scintillation Counting Instruments	NO: RSL-107	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides instructions for the operation of the liquid scintillation instrumentation.

2.0 SCOPE

This procedure applies to the routine operation of all liquid scintillation counters.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the operation of the liquid scintillation counting instruments by the technician and that all necessary personnel are thoroughly familiar with it.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the associated equipment.

3.3 It is the responsibility of the technician to operate the instrument in accordance with this procedure and to report any abnormalities to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 Operation Manual, Packard 2000 CA/LL Liquid Scintillation Counter, Packard Instrument Company, Downers Grove, IL, 1985.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Liquid scintillation counter

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Refer to 4.4.1 for a complete description of all instrument controls.

6.2 Normal operational settings for all controls are tabulated in the instrument logbooks. Those controls used infrequently should be checked for proper settings before each use of the instrument.

6.3 Check to ensure that the instrument is set for loading. The appropriate counting protocol flag shall be inserted in the front carriers to be used. Refer to 4.4.1 to set up a protocol.

6.4 Load each sample in the order noted on the appropriate data analysis sheet.

6.5 Initiate counting as described in 4.4.1.

6.6 When counting has ceased, examine the printout to note any samples with unusually high or low count relative to other similar samples. If a high or low count is noted, contact the Laboratory Manager, or his designee for appropriate action.

6.7 Unload the samples from the instrument and return to the technician who performed the analysis.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.3 Correct counting protocol

8.3 Material Monitoring

8.3.1 None

8.4 Equipment Monitoring

8.4.1 None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to issue.

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-108

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Operation of Alpha Scintillation Counting Instruments

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Wells DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cupfer DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-10-87				

429-1-87

TITLE: Operation of Alpha Scintillation Counting Instruments	NO: RSL-108	PAGE: 1
	REVISION: 0	DATE: 4-10-87

1.0 PURPOSE

This procedure provides instructions for the operation of alpha scintillation counting instrumentation.

2.0 SCOPE

This procedure applies to the routine operation of all alpha scintillation counting instrumentation.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the operation of all alpha scintillation counting instrumentation.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to operate the instrument in accordance with this procedure and to report any abnormalities to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criterion "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 Model 1000 Six Decade Scaler, Instruction Manual, Ludlum Instruments Inc., Sweetwater, Texas 79556

4.4.2 Model 2000 Portable Scaler, Instruction Manual, Ludlum Instruments Inc., Sweetwater, Texas 79556.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Alpha Scintillation Counting System

5.2 Materials

5.2.1 Scintillation discs

5.3 Reagents

5.3.1 None

6.0 PROCEDURE

6.1 Refer to 4.4.1 and 4.4.2, as appropriate, for a complete description of all instrument controls.

6.2 Normal operational settings for all controls are tabulated in each of the instrument logbooks. Controls should be checked for proper settings before an instrument is used.

6.3 Before each sample is loaded into the slide, be sure the sample planchet does not extend above the level of the planchet holder. Never allow a planchet to be loaded if it is too high.

6.4 Load the sample and initiate the counting for the required count time.

6.5 When counting is complete, record the sample results and remove the sample.

6.6 Repeat 6.3 to 6.5 as necessary to count remaining samples.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to use.

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-109

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Background Determination of a Germanium Detector System

APPROVED: James T. Harney DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Nieg DATE: 6-4-87
ITAS QA Director

CONCURRED: John A. Cupin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

3229

Supplement to existing procedure? Yes (x) No ( )
If yes, enter paragraph number to reference insertion point: 6.1.5

Procedure Title & Number (If exists) RSL-109, R0

BACKGROUND DETERMINATION OF A GERMANIUM DETECTOR SYSTEM

Description of experiment or method CHANGE MCA channel markers

Time Period From: 8/28/87 To: Issue of RSL-109, R1 Samples Affected All until revision of RSL-109

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

6.1.5 Using the MCA feature of the ND-66 set the left marker to channel ~~50~~ 100 and the right marker to ~~2620~~ 4096.

Submitted by: James H. Dellard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000085

TITLE: Background Determination of a Germanium Detector System	NO: RSL-109	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides instructions for determining the background of a germanium detector system.

2.0 SCOPE

These instructions are applicable to all germanium detector systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed whenever the background determination of a germanium detector system is performed.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

3.3 It is the responsibility of the person who performs this procedure to follow the instructions and to report any problem to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 ND66-B Multichannel Analyzer/Remote Terminal Operator's Instruction Manual, Nuclear Data Inc. Schamberg, IL, 1983.

4.4.2 Nuclide Indentification Software Instruction Manual, Nuclear Data Inc., Schamberg, IL, 1983.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Germanium Spectroscopy System

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Instructions

6.1.1 Establish the instrument settings for a background determination as listed in the systems logbook.

6.1.2 Remove all radioactive materials (i.e., sources and/or samples) from the detector shield and close the shield lid.

6.1.3 Accumulate a fifteen-hour (54000 sec) background spectrum.

6.1.4 Analyze the spectrum using any MARN-900 geometry file. Record background activities in the logbook and discard the nuclide report printout.

6.1.5 Using the MCA feature of the ND-66 set the left marker to channel 50 and the right marker to 2620.

6.1.6 Read (from the screen) the total number of counts between markers. Record reading of total background counts in logbook; date and initial. Plot on the appropriate control chart. If plotted point is greater than 3 standard deviations from mean, follow steps given in section 6.2.

6.2 Corrective Actions

6.2.1 Ensure all instrument settings are correct. Check all cable connections and ensure cables are in good working order. Check for external radiation sources. Repeat Section 6.1.

6.2.2 If abnormal values are still obtained, notify the Laboratory Manager, or his designee.

6.3 Instrument Shutdown

6.3.1 If the instrument fails the quality control test outlined above in 6.1.6, and the corrective actions in Section 6.2 do not resolve the problem, the instrument must be declared Out of Service. Note this action in the system logbook. Attach appropriate form (see appendix 10.1).

6.3.2 The instrument may be placed back in use once the malfunction has been corrected and the above outlined quality control criteria have been passed. Note this action in the system logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.1.3 The Quality Assurance staff shall periodically perform surveillances to determine compliance to this procedure and review all associated control charts for any long range trends in data.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to issue.

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Background Determination of a Germanium Detector System	NO: RSL-109	PAGE: 5
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

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____ Work may continue with this item subject to the following limitations:

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____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-110

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Energy Calibration of Computer-Assisted Germanium Spectroscopy Systems

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Miller DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Arpin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

429-1-87

TITLE: Energy Calibration of Computer-Assisted Germanium Spectroscopy Systems	NO: RSL-110	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides instructions for the energy calibration of computer-assisted germanium spectroscopy systems.

2.0 SCOPE

These instructions are applicable to all gamma spectroscopy systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed whenever the energy calibration of a germanium spectroscopy system is performed.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

3.3 It is the responsibility of the person who performs this procedure to follow the instructions and to report any problem to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 ND66-B Multichannel Analyzer, Remote Terminal Operator's Instruction Manual, Nuclear Data Inc., Schaumberg, IL, 1983.

4.4.2 Nuclide Identification Software Instruction Manual, Nuclear Data Inc., Schaumberg, IL, 1983.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Nuclear Data germanium gamma spectroscopy system (ND661682)

5.2 Materials

5.2.1 Mixed radioisotope gamma sources in geometry suitable for counting using germanium gamma spectroscopy system. Gamma-ray energies should cover an energy range from 60 keV to 2615 keV.

5.3 Reagents

None

6.0 PROCEDURE

6.1 Instructions for Energy Calibration

6.1.1 Establish the instrument settings and experiment parameters as listed in the system logbook.

6.1.2 Position the gamma source on the top of the germanium detector.

6.1.3 Accumulate a spectrum for the time interval noted in the logbook.

6.1.4 Utilizing the terminal feature of the analyzer, run a peak search on the spectrum and collect the print-out which shows channels and associated gamma energies.

6.1.5 Run the appropriate energy calibration program (R-ENERGY) and enter the channel number (from step 6.1.4) and the corresponding exact gamma energy for all abundant energy lines of the gamma source. (Exact gamma energies are available from Radioactive Decay Data Tables.)

6.1.6 After channel - energy data is entered, choose the fit (Quadratic or Linear) which gives the minimum chi square.

6.1.7 Update the Energy Calibration file currently at the monitor by answering yes (Y) to the last question in the "ENERGY" program.

6.1.8 Run a peak search on the same spectrum and find the difference between exact gamma energies (from tables) and those measured by the spectrometer. If this difference is less than 0.5 keV, omit instructions 6.1.9 through 6.1.12 and continue with instructions 6.1.13 through 6.1.15. If the difference exceeds 0.5 keV, proceed with 6.1.9 through 6.1.15.

6.1.9 Adjust the fine gain on the system's amplifier so that the 1332.5 keV gamma peak of Co-60 appears in channel 1332.5 (for slope of ≈ 1 keV/channel).

6.1.10 Using the zero adjustment of the system's ADC, set the peak channel of the 88.02 keV Cd-109 gamma ray to channel 88 (for slope = 1 keV/channel).

6.1.11 Repeat steps 6.1.9 and 6.1.10 until the peaks of the gamma lines are positioned to within ± 1 channel and then repeat instructions 6.1.3 through 6.1.8.

6.1.12 If proper settings can not be obtained, see section 6.2.

6.1.13 Record the system's settings (i.e., fine gain, coarse gain) and the slope and offset of energy calibration. Date and initial the entries.

6.1.14 Repeat steps 6.1.5 through 6.1.7 for all command files (@ files).

6.1.15 Repeat steps 6.1.1 through 6.1.13 for each ADC connect to the analyzer.

6.2 Corrective Action

6.2.1 Check all instrument settings.

6.2.2 Check all cables for any apparent damage and to ensure that all cables are routed to proper connector.

6.2.3 Notify the Laboratory Manager, or his designee, if the problem still exists.

6.3 Instrument Shutdown

6.3.1 If the instrument fails the quality control check outlined above in sections 6.1.8 and 6.1.11 and corrective actions of section 6.2 did not resolve the problem, the instrument must be declared "Out of Service". Make a note to this effect in the system's logbook. Attach appropriate form (see appendix 10.1).

6.3.2 The instrument may be placed back in use once the malfunction has been corrected and the outlined quality control criteria have been met. Note return to use in the system logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.2 Acceptance Criteria

6.1.8 Tolerance

6.1.11 Tolerance

8.3 Material Monitoring

Check the expiration date of radioactive standard to assure that material is current.

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to issue.

9.0 CALCULATIONS

None

10.0 APPENDICES

APPENDIX 10.1

Nonconforming Item Tag

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

_____

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____ Work may continue with this item subject to the following limitations:

_____

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____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-111

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Linearity Check of Germanium Gamma-ray Spectroscopy Systems

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Miller DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cuyler DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-10-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 4.4.2; 4.4.3; 5.1.1

Procedure Title & Number (If exists) RSL-111, RO

Linearity Check of Germanium Gamma-ray Spectroscopy Systems

Description of experiment or method Update of Germanium System

Issue of RSL-111, R1

Time Period From: 7/09/87 To: -/-/- Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

ADD:

4.4.2 ND9900 VAX/VMS Spectroscopy Applications Package User's Manual (07-0196), Nuclear Data Inc., Schaumberg, IL, August 1986.

4.4.3 ND9900 VAX/VMS Nuclide Identifcation Package (48-0201), Nuclear Data Inc., Schaumberg, IL, August 1986.

CHANGE:

5.1.1 ND9900 Germanium Spectroscopy System

Submitted by: [signature]

Approved by: [signature], Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature], QA/QC/Director, ITAS

TITLE: Linearity Check of Germanium Gamma-ray Spectroscopy Systems	NO: RSL-111	PAGE: 1
	REVISION: 0	DATE: 4-10-87

1.0 PURPOSE

This procedure provides instructions for checking the linearity of the ADC, preamplifier and amplifier of a germanium gamma-ray spectroscopy system.

2.0 SCOPE

These instructions are applicable to all gamma-ray spectroscopy systems utilizing hyperpure germanium or gamma-x detectors.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed whenever a linearity check of the germanium spectroscopy system is performed.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

3.3 It is the responsibility of the person who performs this procedure to follow the instructions and to report any problem to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Germanium Spectroscopy System

5.1.2 Mixed radionuclide gamma source containing Cd-109, Ce-139, Sn-113, Co-60, Cs-137, Co-57 and Y-88 radioisotopes. The source has to be prepared in a geometry suitable for counting on the germanium spectroscopy system.

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Instructions

6.1.1 Establish the instrument parameters for the system linearity check as listed in the system logbook.

6.1.2 Position the gamma source on the detector.

6.1.3 Accumulate spectrum for period noted in the logbook.

6.1.4 Perform peak search on the acquired spectrum.

6.1.5 In the logbook, tabulate the difference between the results tabulated under "ENERGY" on the peak search printout and the actual energy of the 122.06, 165.85, 391.71, 661.60, 898.04, 1173.21, and 1836.13 keV lines (e.g., results - 122.06). Initial and date the entry. Consult Section 6.2 if any difference is more than ± 1.00 keV.

6.1.6 Repeat instructions 6.1.1 through 6.1.5 for each ADC - detector system.

6.2 Corrective Action

6.2.1 Ensure that all instrument settings are correct. Check all cable connections and ensure cables are in good working order. Repeat Section 6.1.

6.2.2 If abnormal values are still obtained, notify the Laboratory Manager, or his designee.

6.3 Instrument Shutdown

6.3.1 If the instrument fails the quality control test outlined above, and the corrective actions in Section 6.2 do not resolve the problem, the instrument must be declared Out of Service. Note this action in the system logbook. Attach appropriate form (see appendix 10.1).

6.3.2 The instrument may be placed back in use once the malfunction has been corrected and the above outlined quality control criteria have been passed. Note this action in the system logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technician performing this procedure shall inspect worksheets for accuracy and completness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy and shall inspect equipment for proper operation.

8.2 Acceptance Criteria

8.2.1 The following sections contain acceptance criteria:

6.1.5 Tolerance

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Linearity Check of Germanium Gamma-ray Spectroscopy Systems	NO: RSL-111	PAGE: 5
	REVISION: 0	DATE: 4-10-87

APPENDIX 10.1

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

_____

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____ Work may continue with this item subject to the following limitations:

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____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-112

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Operation of Germanium Gamma Spectroscopy System (ND66/680)

APPROVED: James T. Harney DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Miller DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Auxier DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 5-21-87				

429-1-87

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (✓) No ( )
If yes, enter paragraph number to reference insertion point: Title page 1.0 4.4.1 to 4.4.3, 5.1.1, 6.1, 6.1.1 to 6.1.2, 6.2, 6.2.1 to 6.2.4, 6.3, 6.3.1 to 6.3.2, 10.0
Procedure Title & Number (If exists) Operation of Germanium Gamma Spectroscopy System (ND 9900), RSL-112 (Rev. 0)

Description of experiment or method Replacement of existing equipment.

Time Period From: 7/29/87 To: Issue of RSL-112, Rev. 1 Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

7-29-87

See attached.

Submitted by: [signature]

Approved by: [signature], Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature] for 11/6/87 QA/QC/Director, ITAS

TITLE: Operation of Germanium Gamma Spectroscopy System [~~ND66/680~~] Delete

1.0 PURPOSE

This procedure provides instructions for the operation of the ~~ND66/680~~ ND9900 Germanium Gamma Spectroscopy System.

Delete old steps 4.4.1 and 4.4.2.
Add new steps 4.4.1 to 4.4.3.

4.4.1 ND9900 VAX/VMS Spectroscopy Applications Package User's Manual (07-0196), Nuclear Data Inc., Schaumberg, Ill., August 1986.

4.4.2 Nuclide Identification Software Instruction Manual, Nuclear Data Inc., Schaumberg, Ill, August 1986.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories," L.G. Kanipe, EPA.

5.1.1 Germanium Gamma Spectroscopy System utilizing ~~ND66/680~~ ND9900 data acquisition system

6.1 Initial Setup

6.1.1 Establish the normal instrument settings for all controls. These settings are tabulated in the individual instrument logbook. Controls should be checked before each use of the instrument.

6.~~2~~ 1.2 Place the sample on the top of the germanium detector. For liquid samples cover the germanium detector with a plastic bag to avoid any possible contamination. Once the sample is properly seated, close the lead shield and secure the door.

Delete old section 6.3
Add new section 6.2

6.2 Initiation of Counting

6.2.1 With the terminal board of the ND9900, clear the old results by depressing the ERASE key followed by the DO key.

6.2.2 Start counting by depressing the ACQ key followed by the DO key.

6.2.3 Change sample parameters by typing "Pars Detect" (1-2 Etc.). Type ED PR (Time), ED SA (Sample), ED Peak Parameters etc. Type EX once completed.

6.2.4 Transfer the efficiency data for the sample geometry to the detector being used for counting by typing, for example: MOVE/PART ~~D~~PETRI-1/EFF DETECT1.

NOTE: This example is for movement of Petri Dish Efficiency for Detector 1 to the Detector 1 configuration. Other established geometries (Appendix 10.1) are moved in a like manner.

Delete old Section 6.4.
Add new Section 6.3.

6.3 Retrieval of Data

6.3.1 Depress PF1 key on ND9900 Terminal for data acquisition on Detector 1. Depress PF2, 3, 4 etc. for other detectors.

NOTE: By depressing PF1, 2, 3, etc., keys prompt an automated analysis command procedure. The procedure performs a peak hypermet search, NID, weighted-mean calculation, and minimum activity calculation with all results listed to the line printer.

6.3.2 Repeat step 6.2 to 6.4 for all subsequent samples.

10.0 APPENDICES

[~~None~~] ← Delete

By _____ Date _____ Subject Geometry Table Sheet No. ____ of ____

Chkd. By ____ Date _____ Proj. No. _____

For 900 mL Marnelli (Detector 1)

move/part space m900-1/eff space Detect 1 ~~or Detector~~

900 mL Marnelli (Detector 2)

move/part space m900-2/eff space Detect 2

500 mL Marnelli (Detector 1)

move/part space m500-1/eff space Detect 1

500 mL Marnelli (Detector 2)

move/part space m500-2/eff space Detect 2

Charcoal Filter (Detector 1)

move/part space chfilter1/eff space Detect 1

Charcoal Filter (Detector 2)

move/part space chfilter2/eff space Detect 2

Single Air Filter

move/part _ SAF-1/eff - Detect 1

move/part _ SAF-2/eff _ Detect 2

Composite Air Filter

move/part 13-cfilter-1/eff - Detect 1

move/part 13-cfilter-2/eff - Detect 2

INTERNATIONAL TECHNOLOGY CORPORATION

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By _____ Date _____ Subject _____ Sheet No. ____ of ____

Chkd. By ____ Date _____ Proj. No. _____

650 gram MARNELLI (DETECTOR 1)

MOVE / PART ___ MSDIL-1 / EFF ___ DETECT 1
SPACE SPACE

650 gram MARNELLI (DETECTOR 2)

MOVE / PART ___ MSDIL-2 / EFF ___ DETECT 2
SPACE SPACE

PETRI DISH (DETECTOR 1)

MOVE / PART ___ PETRI-1 / EFF ___ DETECT 1
SPACE SPACE

PETRI DISH (DETECTOR 2)

MOVE / PART ___ PETRI-2 / EFF ___ DETECT 2
SPACE SPACE

* Remember when units change ~~to~~ from μCi to pCi and vica versa.

Go PARS ___ DETECT 1 OR 2 (which ever detector your on)
SPACE

ED ___ N1D
SPACE

you can then change the units

It will then prompt you to change the ~~mult~~ multiplication factor.

when you need things to be in pCi = 1.0 E+06
" " " " " " " μCi = 1.0

000110

INTERNATIONAL TECHNOLOGY CORPORATION

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Date _____ Subject _____ Sheet No. ____ of ____

Chkd. By ____ Date _____ Proj. No. _____

900 mL MARNELLI (DETECTOR 3)

move / PART _ M900-3 / EFF _ DETECT 3

900 mL MARNELLI (DETECTOR 4)

move / PART _ M900-4 / EFF _ DETECT 4

500 mL MARNELLI (DETECTOR 3)

move / PART _ M500-3 / EFF _ DETECT 3

500 mL MARNELLI (DETECTOR 4)

move / PART _ M500-4 / EFF _ DETECT 4

650 gram MARNELLI (DETECTOR 3)

move / PART _ MSOIL-3 / EFF _ DETECT 3

650 GRAM MARNELLI (DETECTOR 4)

move / PART _ MSOIL-4 / EFF _ DETECT 4

PETRI DISH (DETECTOR 3)

move / PART _ PETRI-3 / EFF _ DETECT 3

PETRI DISH (DETECTOR 4)

move / PART _ PETRI-4 / EFF _ DETECT 4

TITLE: Operation of Germanium Gamma Spectroscopy System (ND66/680)	NO: RSL-112	PAGE: 1
	REVISION: 0	DATE: 5-21-87

1.0 PURPOSE

This procedure provides instructions for the operation of the ND66/680 Germanium Gamma Spectroscopy System.

2.0 SCOPE

This procedure applies to the routine operation of Germanium Gamma Spectroscopy System.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this operating procedure is followed during the use of all gamma spectroscopy systems.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with associated equipment.

3.3 It is the responsibility of the technician to operate the instrument in accordance with this procedure and to report any abnormal results to the Laboratory Manager, or his designee, immediately.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 ND66-B Multichannel Analyzer/Remote Terminal Operator's Instruction Manual, Nuclear Data, Inc., Schaumburg, IL., 1983.

4.4.2 ND680 Series RT-11 Application Software Operator's Manual, Nuclear Data, Inc., Schaumburg, IL., 1985.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Germanium Gamma Spectroscopy System utilizing ND66/680 data acquisition system

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Establish the normal instrument settings for all controls. These settings are tabulated in the individual instrument logbook. Controls should be checked before each use of the instrument.

6.2 Place the sample on the top of the germanium detector. For liquid samples cover the germanium detector with a plastic bag to avoid any possible contamination. Once the sample is properly seated, close the lead shield and secure the door.

6.3 Initiation of Counting

6.3.1 With the ND66 Analyzer in the MCA mode, clear old results by depressing the erase 'ERAS' key followed by 'INIT' key.

6.3.2 Set the counting time by typing in 'LI', depress the space bar, type in the counting time in seconds and depress the 'RETURN' key. The word 'DONE' should appear on the screen indicating that the ND66 has been successfully programmed for the desired counting line time.

6.3.3 Start counting (or stop counting) by depressing the 'ACQ' key followed by 'INIT' key.

6.4 Retrieval of Data

6.4.1 Depress 'MCA' key to put the ND66 in the terminal mode.

6.4.2 While depressing the shift key, type in '@' (and then release the shift key) followed by the proper command file name, e.g., '@' 'petri' for a sample in petri dish geometry on Detector #1.

6.4.3 Depress the 'RETURN' key and wait until "INPUT COMMAND" is displayed on the screen.

6.4.4 Type in 'TI', depress 'RETURN' key; type in customer's name and their code. Depress the 'RETURN' key.

6.4.5 Type in 'ID', depress 'RETURN' key and then type in the assigned RSL identification number of the sample. Depress 'RETURN' key.

6.4.6 Type in 'TY', depress 'RETURN' key and then type in sample matrix. Depress 'RETURN' key.

6.4.7 Type in 'QU', depress 'RETURN' key and then type in sample quantity and depress RETURN. Type in the units of sample sample quantity and then depress 'RETURN' key.

6.4.8 Type in 'SA', depress 'RETURN' key, type in collection date and time of the sample (if no collection date is given, then type in date of receipt of sample). Depress 'RETURN' key.

6.4.9 Type in 'SM', depress 'RETURN' key and computer will display sample perimeters. Check for accuracy. (If mistake is found, correct mistake by typing in appropriate command, depress 'RETURN' key and make the correction. Depress 'RETURN' key).

6.4.10 Type in 'EX' and depress the 'RETURN' key. Computer will now print out stored data.

6.4.11 Repeat 6.2 to 6.4 to count all samples using the proper command file (subsection 6.4.2).

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 The germanium detector system shall have passed all applicable quality control tests at the required frequency prior to use.

8.5 Certification

8.5.1 This procedure shall be tested by running spiked samples through the procedure steps.

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

3223

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-113

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Germanium Detector Efficiency Tables

APPROVED: James T. Harvey DATE: 8-26-87
Laboratory Manager

APPROVED: Paul Miller DATE: 8-27-87
ITAS QA Director

CONCURRED: John C. Cutler DATE: 8-28-87
RSL Director

COPY NO. UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 8-25-87				

429-1-87

TITLE: Determination of Germanium Detector Efficiency Tables	NO: RSL-113	PAGE: 1
	REVISION: 0	DATE: 8-25-87

1.0 PURPOSE

This procedure provides instructions for determining the efficiency of a germanium detector system.

2.0 SCOPE

These instructions are applicable to all germanium detector systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed whenever the efficiency determination of a germanium detector system is performed.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

3.3 It is the responsibility of the person who performs this procedure to follow the instructions and to report any problem to the Laboratory Manger, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

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4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities". ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 ND66-B Multichannel Analyzer/Remote Terminal Operator's Instruction Manual, Nuclear Data Inc. Schaumburg, IL, 1983.

4.4.2 Nuclide Identification Software Instruction Manual, Nuclear Data Inc., Schaumburg, IL, 1983.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Germanium Spectroscopy System

5.2 Materials

5.2.1 Mixed nuclide standard prepared in the proper geometry. When counted, the total spectrum count rate should be less than 1000 cps.

5.3 Reagents

None

6.0 PROCEDURE

6.1 Existing Efficiency Table

6.1.1 Establish the instrument settings for routine counting as listed in the logbook.

6.1.2 Position mixed-nuclide source on the crystal and accumulate a spectrum with at least 10,000 counts (net area) in all peaks to be used in the determination.

6.1.3 Perform a nuclide identification of the spectrum using the current appropriate efficiency table.

6.1.4 Compare the values determined in the nuclide identification to the known values calculated for the mixed-nuclide source. Calculate the percent error associated with each nuclide.

6.1.5 If the percent error associated with 7 of the 9 nuclides present is 5.5 percent or less and the percent error for all nuclides is less than 10 percent, complete the remainder of this section (6.1) and stop. If any of the errors are greater than specified, then go to section 6.2 and continue.

6.1.6 Store the results from this determination in a designated file.

6.1.7 In tabular form in the logbook, record the nuclides, their known activities, the determined activities (from the nuclide report), the difference between the known and the determined activities, and the percent errors where

$$\text{Percent Error} = \frac{\text{Determined} - \text{Known}}{\text{Known}} \times 100$$

6.1.8 Date and initial the entry.

6.1.9 Enter the geometry description and a comment indicating whether the results of the check were within the tolerance stated in this procedure.

6.2 Efficiency Table Determination

6.2.1 Accumulate two additional spectra according to step 6.1.2.

6.2.2 Run the peak search program for each spectrum to obtain the area of each peak of interest. Using the mixed standard solution, the normal peaks of interest (in approximate keV) are 59, 88, 122, 165, 279, 391, 513, 661, 898, 1173, 1332, and 1836. Review the data for accuracy, particularly noting the problem areas. The 59-keV peak is optional and may not be used in all geometries. Other lines may be used to establish points not covered by the above range.

6.2.3 Calculate the detector efficiencies for the energies to be used based on the average data of each peak.

6.2.4 Compare the new efficiency curve to one from a previous year if such exists. Resolve and document any obvious discrepancies between the two curves.

6.2.5 Enter the efficiency values into the appropriate geometry table on the analysis systems.

6.2.6 Record in the logbook all written information and data necessary to reproduce the geometry table.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.1.3 The Quality Assurance staff shall periodically perform surveillances to determine compliance to this procedure and review all associated control charts for any long range trends in data.

8.2 Acceptance Criteria

The following sections of this procedure contain acceptance criteria:

6.1.5 Percent error limits

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to issue.

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-114

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Germanium Detector Counts Reproducibility

APPROVED: James T. Hanley DATE: 8-26-87
Laboratory Manager

APPROVED: Paul Miles DATE: 8-27-87
ITAS QA Director

CONCURRED: John A. Auxier DATE: 8-28-87
RSL Director

COPY NO.: UNCONTROLLED ISSUED COPY

REVISION: 0				
DATE: 8-25-87				

429-1-87

RSL-114, RO, CH-1

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PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 4.4.1; 4.4.2; 5.1.1; 6.1.3

Procedure Title & Number (If exists) RSL-114, RO

Determination of Germanium Detector Counts Reproducibility

Description of experiment or method Updated Germanium System

Issue of RSL-114, R1

Time Period From: 7/29 87 To: --/-/-- Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

CHANGE:

4.4.1 ND9900 VAX/VMS Spectroscopy Applications Package User's Manual (07-0196), Nuclear Data Inc., Schaumberg, IL, August 1986.

4.4.2 ND9900 VAX/VMS Nuclide Identification Package (48--0201), Nuclear Data Inc., Schaumberg, IL, August 1986.

5.1.1 ND9900 Germanium Spectroscopy System

ADD:

6.1.3 Note: System Sensitivity must be set to 18.0, R Sensitivity to 17.0, and CHI Sensitivity to 18.0.

Submitted by: [signature]

Approved by: James T. Harvey, Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature], QA/QC/Director, ITAS

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TITLE: Determination of Germanium Detector Counts Reproducibility	NO: RSL-114	PAGE: 1
	REVISION: 0	DATE: 8-25-87

1.0 PURPOSE

This procedure provides instructions for determining the counts reproducibility of a germanium detector system.

2.0 SCOPE

These instructions are applicable to all germanium detector systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed whenever the counts reproducibility determination of a germanium detector system is performed.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 ND66-B Multichannel Analyzer/Remote Terminal's Operator's Instruction Manual, Nuclear Data Inc. Schaumburg, IL, 1983.

4.4.2 Nuclide Identification Software Instruction Manual, Nuclear Data Inc., Schaumburg, IL, 1983.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Germanium Spectroscopy System

5.2 Materials

Gamma check source: Bi-207

5.3 Reagents

None

6.0 PROCEDURE

6.1 Counts Reproducibility Check

6.1.1 Position the source in a reproducible manner on the end of cap of the germanium detector.

6.1.2 Establish a preset live time sufficient for 10,000 counts in the 1063 Kev peak and initiate acquisition.

6.1.3 Upon completion of acquisition, perform a nuclide identification.

6.1.4 Check nuclide identification to determine if the three main peaks of the Bi-207 source were found.

6.1.5 If the main peaks were located and identified, obtain the sum of the area plus background for the control chart. If the main peaks were not located and identified proceed to section 6.2.

6.1.6 If the value falls within $\pm 2\sigma$ of the mean, plot and record the value and proceed to 6.1.8.

6.1.7 If the value falls outside $\pm 2\sigma$, plot and record the value, and obtain a second count by performing the same steps used in obtaining the first value.

6.1.7.1 When a second value is obtained in accordance with 6.1.7, apply the appropriate step below, 6.1.7.2, to the value.

6.1.7.2 If the second value is $\pm 2\sigma$ of the mean, plot and record the value and proceed to 6.1.8. Distinguish between the first and second values plotted on the control chart by using different representations for the points (i.e., use dots for first values and x's for second values).

6.1.7.3 If the second count falls between the 2σ and 3σ limits on either side of the mean, record and plot the value and consult the Laboratory Manager, or his designee, for approval before using the instrument for sample counting.

6.1.7.4 If the second value falls outside ±3σ, plot and record the value and notify the Laboratory Manager so that corrective action can be taken. Distinguish between the first and second values as described in 6.1.7.2. Refer to 6.2 for corrective actions.

6.1.8 Record the counts reproducibility information in the logbook. Date and initial the entries.

6.2 Corrective Action

6.2.1 Check source positioning and all instrument settings.

6.2.2 Check all instrument cables to ensure all cables are routed to proper connectors and ensure all cables are in good working order.

6.2.3 Perform energy calibration if required by results from 6.1.5.

6.2.4 Notify the Laboratory Manager, or his designee, if the above steps do not correct the problem.

6.3 Instrument Shutdown

6.3.1 If the instrument fails the quality control test outlined above in 6.1, and the corrective actions in Section 6.2 do not resolve the problem, the instrument must be declared Out of Service. Note this action in the system logbook. Attach appropriate form (see appendix 10.1).

6.3.2 The instrument may be placed back in use once the malfunction has been corrected and the above outlined quality control criteria have been passed. Note this action in the system logbook.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager shall periodically observe the technician during the use of this procedure to ensure the results obtained are in accordance with the procedure.

8.1.2 The technician performing this procedure shall ensure that all equipment used is working properly and is calibrated prior to starting this procedure.

8.1.3 The Quality Assurance staff shall periodically perform surveillances to determine compliance to this procedure and review all associated control charts for any long range trends in data.

8.2 Acceptance Criteria

The following sections of this procedure contain acceptance criteria:

6.1.5 Peaks identified

6.1.7 Control limits

6.1.7.2 Control limits

6.1.7.3 Control limits

6.1.7.4 Control limits

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be approved by the Quality Assurance staff for completeness and concurrence with the calibration criterion prior to issue.

9.0 CALCULATIONS

None

10.0 APPENDICES

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-201

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Sample Preparation

APPROVED: James T. Harvey DATE: 8-27-87
Laboratory Manager

APPROVED: Paul Mills DATE: 8-28-87
ITAS QA Director

CONCURRED: John A. Lupfer DATE: 8-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 8-05-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.11

Procedure Title & Number (If exists) RSL-201, R0
Sample Preparation

Description of experiment or method Provide instructions for sample preparation of fecal samples

Time Period From: 9/30/87 To: Issue of RSL-201, R1 Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

9-30-87

6.11 Fecal Samples

6.11.1 Weigh the sample in its sample bag and record as the WET WEIGHT.

6.11.2 Place sample and bag in a container designated for ashing. Muffle at 210°C for 8 hours, then 375°C for 16 hours followed by 24 hours at 525°C.

6.11.3 Dissolve the ash in 50 ml of a 2:1 mixture of concentrated nitric acid and concentrated hydrochloric acid. Add the appropriate yield carriers/tracers as required by the analysis request. Evaporate the sample solution to dryness slowly. Repeat the acid addition and evaporation.

Submitted by: Jane M. Willard

Approved by: Roger T. Harvey, Laboratory Manager

Approved by: D. G. Cupino, Laboratory Director

Approved by: Cheryl D. Grimes, QA/QC/Director, ITAS

6.11.4 Dissolve the residue in 50 ml of concentrated nitric acid. Evaporate to dryness. Repeat twice.

6.11.5 Dissolve residue in 100 ml of 1M HNO_3. Filter through Whatman 42 or equal as necessary. If filtered, wash beaker and filter with small amounts of 1M nitric acid.

6.11.6 Add 5 drops of octyl alcohol and 10 ml of hydrogen peroxide, stir and heat to 85-90°C.

6.11.7 While stirring, add 2 ml 1 M calcium nitrate and 10 ml concentrated phosphoric acid. Adjust the pH to 8.5 - 10.0 using ammonium hydroxide and pH paper as indicator.

6.11.8 Remove the stirrer and cover the beaker with plastic wrap to exclude atmospheric carbon dioxide and let the covered beaker stand for approximately 16 hours to complete the precipitation and settling.

6.11.9 Decant and aspirate the supernatant as completely as possible without disturbing the precipitate and discard it in the pH>2 waste acid container.

6.11.10 Slurry the precipitate with distilled water and transfer it quantitatively to a centrifuge bottle. Centrifuge for 5 minutes and discard the supernatant. Wash the precipitate with 50 ml 0.1 M phosphoric acid (approximately pH8), centrifuge and discard the supernatant.

6.11.11 Add 15 ml concentrated nitric acid to beaker used for precipitation, heat on hot plate, swirl acid to rinse walls and pour contents into centrifuge bottle containing the washed precipitate. Transfer mixture to a clean second beaker.

6.11.12 Rinse the walls of the beaker used for precipitation with water, transfer rinse to centrifuge tube used for collecting the precipitate, rinse the bottle and pool the rinse with the mixture in the second beaker. Evaporate the mixture to dryness on a hot plate and wet ash the residue with concentrated nitric acid as many times as necessary to obtain a clean white residue. This residue is ready to be analyzed.

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.1.5

Procedure Title & Number (If exists) RSL-201, R0
Sample Preparation

Description of experiment or method Add preparation of core Samples

Time Period From: 10/22/87 To: ~~++~~ Issue of RSL-201, R1 Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

6.1.5 Core Samples 10-22-87

6.1.5.1 Cut Shelby tube with a rigid pipe cutter and cut sample with a wire saw at 2 inch intervals.

6.1.5.2 If more than 650g of sample is available, then carefully remove sample from every other sliced section of the Shelby tube and place in a tared 650g marinelli beaker (tare weight includes empty container and lid) If less than 650g of sample is available, all of the sample may be utilized as directed by the laboratory Manager, or his designee.

6.1.5.3 Close the sample container securely. Determine the net weight. Seal with electrical tape. Store unused portions.

6.1.5.4 Write sample information (ID number, net weight of sample and customer's name) on the container, on the appropriate laboratory work sheet, and in sample prep log book (see appendix 10.1)

6.1.5.5 Submit for counting

Submitted by: [signature]

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. L. Austin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS
for 11/6/87

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PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (XX) No ( )
If yes, enter paragraph number to reference insertion point: 4.4.1; 4.4.2; 5.1.10; 6.8.7 - 6.8.27; Appendix 10.2 and 10.3
Procedure Title & Number (If exists) RSL-201, RO, Sample Preparation

Description of experiment or method Add procedure for Microwave digestion and use of programmable ashing furnace.

Time Period From: 11/21/87 To: Issue R1 Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

See attached.

Submitted by: James M. Gillard

Approved by: James T. Harvey, Laboratory Manager

Approved by: D. A. Aspier, Laboratory Director

Approved by: Sheryl D. Kruce, QA/QC/Director, ITAS

000135

ADD

4.4.1 "Use & Care Manual", Sanyo Microwave Oven, Model EM-550, Sanyo Electric Inc., Little Ferry, N.J., 1987.

4.4.2 "Instruction Manual", Fisher Isotemp Programmable Ashing Furnace, Model 497, Instrument Manufacturing Division, Pittsburgh, Pennsylvania, 1986.

ADD

5.1.10 Microwave Oven

DELETE

old 6.8.7 through 6.8.25

ADD following steps:

6.8.7 If chemistry analysis is required and specific procedure(s) assume dissolution, weigh an appropriate (less than 10g) size sample and place in 50 ml beaker. Add appropriate yield tracers and carriers, if required.

6.8.8 Muffle at 525 ^{o}C for 8 hours. Cool.

6.8.9 Transfer sample to Teflon beaker, wash walls of the glass beaker with 25 ml of concentrated HNO_3 and add to the Teflon beaker. Add 10 ml of concentrated HF. Evaporate to dryness, repeat the addition of acids and evaporation two more times.

6.8.10 Add 30 ml of concentrated HF and 5 ml of concentrated HNO_3 to the residue. Loosen insoluble portions with a Teflon policeman and transfer to the Teflon PFA digestion vessel. Rinse beaker walls with up to 5 ml of concentrated HNO_3. Reserve Teflon beaker.

6.8.11 Torque lid of the Teflon PFA vessel to 24 newton-meters.

6.8.12 Place up to six vessels in a circle on the pre-wound microwave carousel.

6.8.13 Touch CLEAR. Touch TIME. Touch numbers for desired heating time (typically 10 minutes 00 seconds). Touch COOK CONTROL. Touch numbers for desired power level (typically 30).

CAUTION: To avoid possible rupture of digestion vessels, check values for heating time and power level by:

1. Touch TIME: verify time setting.
2. Touch COOK CONTROL: verify power level setting.

6.8.14 Touch START. Pull down sash on hood completely. Do not raise hood sash until microwave digestion sequence is complete.

6.8.15 After digestion sequence is complete, allow vessels to cool for 5 minutes before removing from the microwave.

CAUTION: To avoid hazards of hot acid vapors or liquid, air cool vessels for at least 30 minutes additionally in hood.

6.8.16 After the vessels have cooled to room temperature, untorque lid.

6.8.17 Transfer sample back into Teflon beaker reserved in previous step 6.8.10. Rinse digestion vessel walls with up to 5 ml of concentrated HNO_3.

6.8.18 Add 5 ml of concentrated $HClO_4$ and heat over low heat in the perchloric acid hood until perchloric acid fumes are no longer present.

6.8.19 Add 10 ml of concentrated HNO_3 to dissolve and loosen residue. Transfer residue to 100 ml glass beaker and add 10 ml of HCl. Evaporate to dryness.

6.8.20 Dissolve the residue in 20 ml of concentrated HNO_3. Heating may be necessary to effect solution of residue

6.8.21 If strontium analysis is requested, transfer sample to a centrifuge tube, rinsing beaker with a small amount of concentrated HNO_3. If strontium analysis is NOT requested, transfer to step 6.8.26.

6.8.22 Add 10 ml of fuming HNO_3 dropwise to the sample and mix cautiously. Cool in an ice water bath for at least 15 minutes.

6.8.23 Centrifuge sample for three (3) minutes on high speed.

6.8.24 Collect supernatant in a 250 ml beaker for further analysis if required. Otherwise discard.

6.8.25 Transfer the precipitate in the centrifuge tube to RSL-305 "Determination of Strontium-89,90 in Water Samples" at step 6.6.

6.8.26 The supernatant retained in 6.8.24 or the solution from 6.8.20 is evaporated to dryness.

6.8.27 Transfer the sample to RSL-304 "Determination of Selected Alpha Emitting Isotopes".

ADD

Appendix 10.2
Microwave Oven Operation Reference Guide

Appendix 10.3
Muffle Furnace Operation Reference Guide

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SUMMARIZED OPERATING INSTRUCTIONS

Your new solid state control system allows you to use your microwave oven in many ways.

TIME OF DAY	"HI-POWER"/"MULTI-POWER" COOKING	TEMPERATURE CONTROLLED "HI-POWER"/"MULTI-POWER" COOKING
To set: 1. Touch CLEAR. 2. Touch TIME OF DAY. 3. Touch numbers for correct time of day. 4. Touch TIME OF DAY.* * This "locks" in the time and starts the clock running.	To set: 1. Touch CLEAR. 2. Touch TIME. 3. Touch numbers for desired cooking time. 4. Touch COOK CONTROL. 5. Touch numbers for desired cooking power level. 6. Touch START. NOTE: It is not necessary to touch COOK CONTROL when cooking with the highest power ("HI"). To set "HI-POWER" Cooking, skip steps 4 and 5 above. (See Page 12 for more detailed instructions.)	To set: 1. Plug in Temperature Probe. 2. Touch CLEAR. 3. Touch TEMP CONTROL. 4. Touch numbers for desired food temperature. 5. Touch COOK CONTROL. 6. Touch numbers for desired cooking power level. 7. Touch START. NOTE: It is not necessary to touch COOK CONTROL when cooking with the highest power ("HI"). To set "HI-POWER" Cooking, skip steps 5 and 6 above. (See Page 16 for more detailed instructions.)
TIMER		
To set: 1. Touch CLEAR. 2. Touch TIME. 3. Touch correct numbers for time. 4. Touch COOK CONTROL. **5. Touch "0" (power level 0).** 6. Touch START. (See Page 10 for more detailed instructions.)		
MULTI-STAGE COOKING	**DELAY START COOKING**	**AUTOMATIC DEFROST**
To set: 1. Touch CLEAR. 2. Touch TIME or TEMP CONTROL. 3. Touch numbers for cooking time or temperature. 4. Touch COOK CONTROL. 5. Touch numbers for cooking power level. 6. Touch MEMORY/RECALL or PAUSE and repeat steps 2-5 to set 2nd cooking stage. 7. Touch START. (See Pages 22 and 23 for more detailed instructions.)	To set: 1. Touch CLEAR. 2. Touch DELAY START. 3. Touch numbers for Time of Day to finish cooking. *4. Touch TIME. 5. Touch numbers for cooking time. 6. Touch COOK CONTROL. 7. Touch numbers for cooking power level. 8. Touch START. * Multi-Stage cooking can be used with Delay Start. After step 3 above, follow instructions for Multi-Stage cooking beginning with step 2. (See Page 24 for more detailed instructions.)	To set: 1. Touch CLEAR. 2. Touch AUTO DEFROST. 3. Touch numbers for desired defrosting time. 4. Touch START. (See Pages 17 thru 21 for more detailed instructions.)

OVEN CONTROL PANEL

Stage Lights—When cooking stages are set, lights will go on indicating the stage in which your oven is cooking.

Indicator Lights—Time, Cook, Temp, Delay Start and Auto Defrost lights indicate which cooking operation is being used.

Time—Used in setting cooking time.

Auto Defrost—Used in setting Automatic Defrost.

Cook Control—Used to select cooking power levels.

Temp Control—Used in setting the desired food temperature. "Temperature Controlled" cooking can be done only when the temperature probe is plugged into the socket inside the oven.

Memory/Recall—For Multi-Stage cooking, MEMORY/RECALL is used in setting:
- Cooking time.
- Power level.
- Temperature.

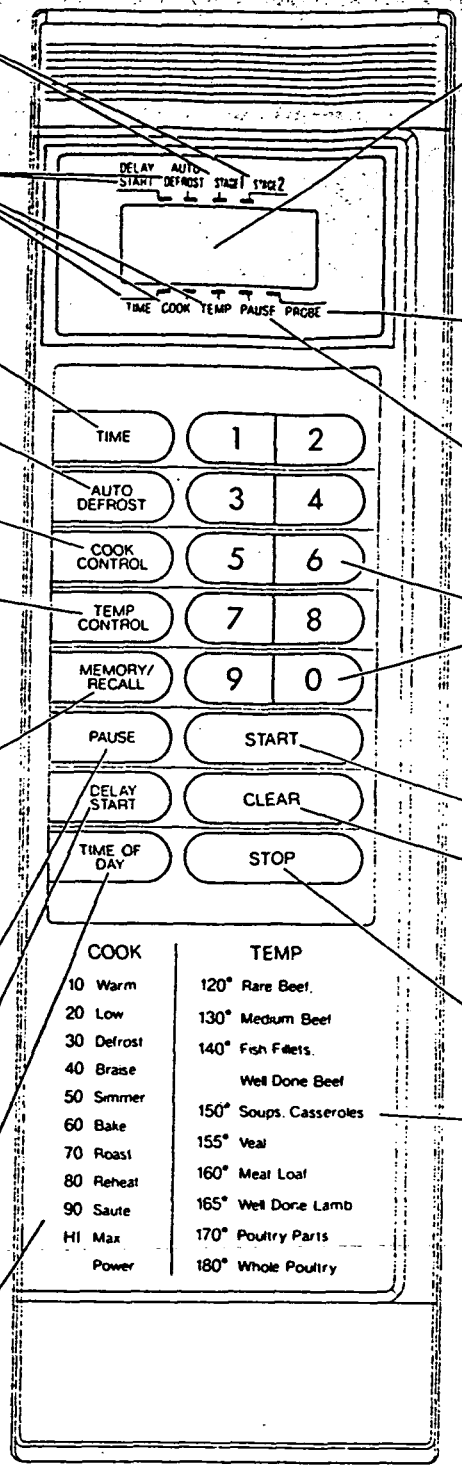
Also, it is used to recall each memory stage in a multi-stage cooking program.

Pause—Used to stop oven between cooking stages.

Delay Start—Used in setting a Delay Start cooking. Cooking can be finished at the preset stop time.

Time of Day—Used in setting Time of day.

Cook Guide—Cooking guide which lists the types of cooking that most often occur at these cooking power levels.



Display Window—Used to show:
- Time of day.
- Cooking time.
- Cooking power level.
- Food temperature.
- Hold, End.

It also functions as countdown timer or temperature indicator when cooking.

Probe Light—Flashes to warn that temperature probe is not plugged into the socket inside the oven for "Temperature Controlled" cooking.

Pause Light—Shows oven has been set to pause between cooking stages.

Numbers—Used to enter the:
- Time of day.
- Delay start times.
- Cooking times.
- Cooking power levels.
- Food temperatures.

Start—Starts the oven.

Clear—Clears all entries except TIME OF DAY. Once cooking has begun, however, CLEAR will function only after STOP has been touched.

Stop—Stops the oven.

Temp Guide—Temperature guide which lists the examples of food that are best cooked to these temperatures.

NOTE: A "beep tone" sounds when a "pad" on the control panel is touched, to indicate a setting has been entered.

[illegible]TION

his section includes a description of the controls and [illegible] ent a sample program for operation of the Cottrell model [illegible] rogrammable ashing furnace based on [illegible] ining ash content of coal and coke. [illegible] uggestions for [illegible] ion of the system [illegible]

[illegible]ser Controls

he descriptions below are to help familiarize the user with the ontrol keys and indicators, located on a single panel on the ront of the unit (see figure 5). The indicators are grouped in he top half of the control panel and include the digital read- ut, furnace operating mode indicators, and furnace parameter ndicators. The control keys, including the numeric and func- ion keys, are arranged on the lower portion of the control anel.

[illegible]isplay Indicators

[illegible]igital Display Indicator. A four-segment LED array that contin- ously displays either actual furnace temperature in degrees elsius or hold time remaining in a program. The display may be witched from furnace temperature mode to hold-time remaining ode and back again by using the TEMP/TIME key. The furnace tem- erature display indicates the actual furnace temperature to the earest 1°C. The hold-time remaining display continuously ounts down the hold time remaining in minutes as soon as the urnace reaches the programmed transition temperature.

[illegible]urnace Indicators

[illegible]eat Control Indicator. An LED indicator which illuminates when ower is supplied to the heaters. Normally, the indicator will e continuously lit until the programmed temperature is reached, nd [illegible] will cycle on and off as power is supplied to maintain [illegible]ure.

[illegible]r[illegible]am Complete Indicator. An LED indicator that illuminates hen the program is completed. A buzzer sounds at this point to lert the operator that the program has finished. The power is ut off from the heater and the heat control indicator is extin- uished until a new program is initiated.

[illegible]ower Indicator. An LED indicator that remains lit as long as he furnace is connected to the power source, indicating that ower is being supplied to the unit.

[illegible]an Indicator. An LED indicator that illuminates when the fur- ace air circulating fan is operating. The fan is switched on nd off with the FAN ON/OFF key.

[illegible]afety Sentinel Indicator. An LED indicator that illuminates hen the safety circuit assumes control in the event that the rimary circuitry fails. The safety circuit controls at a fixed emperature of 1120°C, or shuts the furnace down to prevent amage to the unit.

[illegible]rogram Mode Indicators

[illegible] series of 18 heating and cooling program indicators arranged n an ascending and descending pattern on the control panel. hese indicators guide the operator through programming the urnace, then indicate its progress as it follows the programmed eating/cooling schedule.

[illegible]eating Rate (R1, R2, R3) °C/min. When a temperature run is eing programmed, these indicators light at the appropriate oints to indicate that you should enter a heating rate value. he heating rate is the rate at which the furnace will heat up, n °C/minute, until it reaches the transition temperature elected for the current heating step. During a program run, hese indicators light when the furnace is heating to the next ransition temperature.

[illegible]ransition Temperature (T1, T2, T3) °C. When a temperature run [illegible] programmed, these indicators light at the appropriate [illegible] indicate that you should enter a transition temper- [illegible] the heat-up portion of the run. This is the peak [illegible]ure the furnace will achieve at each heating step. uring a program run, these indicators light when the threshold emperature has been reached.

[illegible]old Time (H1, H2, H3) minutes. When a temperature run is being rogrammed, these indicators light at the appropriate points to ndicate that you should enter a hold time. This is the time hat the furnace will maintain the selected transition tem- erature. During a program run, these indicators light to in- icate that the furnace is holding at the threshold temperature

[illegible]ooling rate (R4, R5, R6) °C/min. When a temperature run is [illegible] being programmed, these indicators light at the appropriate [illegible] points to indicate that you should enter a cooling rate value. [illegible] cooling rate is the rate at which the furnace will cool down, [illegible] °C/minute, until it reaches the threshold temperature [illegible] programmed for the cooling step. During a program run, [illegible] these indicators light to show that the furnace is cooling down to the threshold temperature.

[illegible]ransition Temperature (T4, T5, T6) °C. When a temperature run is [illegible] being programmed, these indicators light at the appropriate [illegible] points to indicate that you should enter a transition temperature [illegible] the processor is ready to [illegible] temperature during the cool [illegible] the target temperature, the [illegible]urnace will cool at each cooling step. During a program run, [illegible] these indicators light when the furnace has reached one of the [illegible] cool down threshold temperatures.

[illegible]old Time (H4, H5, H6) minutes. When a temperature run is [illegible] being programmed, these indicators light at the appropriate [illegible] points to indicate that you should enter a hold time. This is the time the furnace will maintain the transition temperature [illegible] programmed at each cooling step. During a program run, these [illegible] indicators light to show that the furnace is holding at one of the cool down threshold temperatures for the specified time.

Operating Keys

An array of 16 keys that enables the operator to enter numeric data and to establish all necessary operating modes (see figure 5). The function of each key is as follows:

NUMERIC	Ten keys labeled 0 through 9 which enable the operator to enter all numeri data in appropriate units as required (either time in minutes or temperature in °C). Data selected always appears on the digital readout for visual confirmation of accuracy before it is entered into processor memory.

Function Keys

The remaining six keys, each with a specifically labeled function, enable the operator to select and "set" the processor for entry of data, to establish operating conditions, and to cause the digital display to exhibit either furnace temperature or hold time remaining (see figure 5). The function of each key is as follows:

PARAMETER SET	Directs the processor to accept program data. When the program set key is pressed, the initial heating rate indicator (R1) illuminates, and the digital display exhibits the rate previously stored in memory. If the previously stored heating rate is to be revised, the operator may select a new rate using the numeric keys and then pressing the [ENTER] key. If the stored heating rate is to be accepted, the operator simply presses the [ENTER] key to restore the rate and advance the indicator to the next parameter.
FAN ON/OFF	Directs the processor to turn the furnace air-circulating fan on and off. Pressing the key once turns the fan on pressing it a second time turns the fa off. Fan and key operate in conjunction with FURNACE FAN indicator which illuminates when the fan is operating.
PROGRAM START/STOP	Directs the processor to begin or inte rupt a programmed operation of the furnace. The operator presses the key only after establishing the six operat ing parameters as described above. Once the furnace is in progress, it ca be interrupted at any time (to change parameter, for example) by again press ing the PROGRAM START/STOP key. NOTE: Only program events that have not occurred can be reprogrammed. The program will not revert to past events
ENTER	Directs the processor to accept any keyed-in data as shown on the digital display and incorporate the data into the operating program.

TEMP/TIME — Directs the digital display to exhibit either current furnace temperature or the time remaining if program is calling for hold-time mode. Pressing the key once will display temperature, pressing it a second time will display time. The two indicators (FURNACE TEMP °C and HOLD TIME REMAIN (MIN)), located below the display, alert the operator as to which parameter is being displayed.

C (CLEAR) — Clears the digital readout, allowing the operator to correct any input data before entering it into the processor memory. Also utilized to silence alarm buzzer when correcting an error code alert.

Programming the Furnace

It is important to note that the specified 18.4°C/min heating rate is obtainable only with an input line voltage of 230 VAC and with the air-circulating fan off.

Similarly, the 6.3°C/min cooling rate is obtainable only with 230 VAC supplied to the unit and the fan on. When the available supply voltage falls below this level, or if the circulating fan is operated, heating and cooling rates will be proportionately reduced. For example, reducing the line voltage from 230 VAC to 208 VAC results in a reduction in power available to the heating elements.

The procedure for programming the furnace is illustrated below using ASTM D-3174, a standard test method for the analysis of the ash content of coal or coke. Ash content is determined by weighing the residue remaining after burning the coal under rigidly controlled conditions of sample weight, temperature and atmosphere.

To program the furnace to implement the test, perform the following:

Fill the crucibles (Fisherbrand No. 10-490-4, or equivalent) with approximately 1 gram of sample using a porcelain spoon (Fisher No. 14-430B).

2. Weigh and record each sample, together with its crucible, to within 0.1 mg. After weighing, place the samples into the appropriate racks (the Fisher No. 10-466-6 rack is designed for the Fisherbrand crucibles, and the No. 10-490-5 rack is for other types of crucibles). Be certain to record each crucible's location. A variety of markers is listed in the Fisher Catalog for marking ceramic materials.

4. Insert the racks into the oven and latch the door closed.

5. Connect the furnace line cord plug into the appropriate electrical outlet.

   NOTE: When the furnace is initially plugged in, the alarm buzzer will sound. Press the CLEAR key to turn off the alarm. Also, note that the POWER and FURNACE TEMP indicators are lit and that the digital display shows the temperature inside the furnace chamber.

6. To initiate programming, press the PARAMETER SET key. The R1 (initial rate °C/min) indicator will light.

7. Enter the value for R1 as 8°C/min.

8. Verify that the rate showing on the digital display is correct, and place it into memory by depressing the ENTER key. The program will now advance to the transition temperature. Next, indicator T1 will light.

9. Enter 500°C for the transition temperature, and press the ENTER key. The program indicator will now advance to H1 (hold time in minutes).

10. Key in "0" minutes for the hold time and enter it into the processor memory by pressing the ENTER key. The R2 indicator should now be lit.

11. Enter a gradual heating rate of 4°C/min, and then press the ENTER key. The program mode indicator will now advance to T2.

12. Enter the transition temperature of 750°C and then press the ENTER key. The parameter LED will advance to H2.

13. Enter 180 minutes for H2, and press the ENTER key. No other heating steps are required for this ASTM method, so the user must program the furnace to bypass the remaining heating step.

    NOTE: Any heating or cooling step may be bypassed by entering a "0" for the heating or cooling rate requested. This automatically causes the program to advance to the next heating or cooling step; in this case, the program will advance to R4, the first cooling step.

14. Enter cooling rate R4 as 4°C/min. The T4 indicator should now light.

15. Enter 50°C for T4 to cool the furnace down to ambient temperature. The H4 parameter will now light.

16. No other cooling steps are required, so you must program the furnace to bypass the remaining cooling steps.

    NOTE: Any heating or cooling step may be bypassed by entering a "0" for the heating or cooling rate requested. This automatically causes the program to advance to the next heating or cooling step.

17. The programming phase is now completed. None of the program-mode indicators should be illuminated. The furnace temperature and power indicators should be the only indicators lit.

18. Press the FAN ON/OFF key to circulate air in the furnace.

19. To start the program, press the PROGRAM STOP/START key. The furnace control and the initial rate (R1) indicators should be illuminated. As the furnace slowly heats up at 8°C/minute, the HEAT CONTROL light will pulse on and off as power is demanded. A buzzer will sound when the furnace run is complete. The PROGRAM COMPLETE indicator will come on, indicating that the power to the furnace is shut off.

The following table provides the maximum linear heating rates that can be expected between ambient and 1100°C, as a function of line voltage:

NOTE: Values for fan "OFF" mode.

Temperature Range	Maximum Linear Heating Rate	Line Voltage
1100°C - 850°C	14°C/MIN	230 VAC
1100°C - 850°C	8°C/MIN	208 VAC
850°C - 600°C	19°C/MIN	230 VAC
850°C - 600°C	12°C/MIN	208 VAC
600°C - 200°C	20°C/MIN	230 VAC
600°C - 200°C	20°C/MIN	208 VAC

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (XX) No ( )
If yes, enter paragraph number to reference insertion point: 6.12

Procedure Title & Number (If exists) RSL-201, R0

Sample Preparation

Description of experiment or method Add procedure for preparation of milk samples for non-volatile alpha emitter analysis.

Time Period From: 11/24 87 To: Issue of R1 -/-/-- Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

See attached.

Submitted by: James M. Dillard

Approved by James T. Harvey, Laboratory Manager

Approved by: R. A. Cantin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

ADD

6.12 Milk Samples - Non Volatile Alpha Emitters

6.12.1 Measure and record an appropriate volume of sample. Place sample in a drying pan lined with a double layer of plastic wrap or appropriate sized glass beaker. Add yield tracer as necessary. Dry under a heat lamp in a hood until all moisture is removed from sample.

6.12.2 Place sample in furnace and muffle at 210 °C for 2 hours, then 375 °C for 4 hours followed by 6 hours at 525 °C. For samples grater than 10 ml, increase ashing times by a factor of four (4).

6.12.3 Allow sample to cool. Add 50 ml of concentrated HNO_3 and 1 ml of 30% H_2O_2. Slowly wet-ash to dryness. Repeat reagent additions and wet-ashing.

6.12.4 Add 10 ml of concentrated HNO_3 and transfer sample to a centrifuge tube. Rinse walls of beaker with concentrated HNO_3. Police residue if necessary.

6.12.5 Add 10 ml of fuming HNO_3 dropwise to the sample and cautiously mix. Cool in an ice water bath for 15 minutes.

6.12.6 Centrifuge sample for three (3) minutes on high speed.

6.12.7 Collect the supernatant in a 250 ml beaker and evaporate to dryness. Discard the precipitate.

6.12.8 If total uranium by fluorometry is requested, dissolve residue in 1 ml of 4M HNO_3 and transfer to tared glass scintillation vial. Rinse beaker with two 2 ml portion of demineralized water and add rinse to vial. Determine volume of final solution by weight to three (3) significant figures.

6.12.9 Transfer to the appropriate procedure for further analysis.

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.2.7; 6.13

Procedure Title & Number (If exists) RSL-201, RO

Sample Preparation

Description of experiment or method Add procedure for preparation of urine samples for total uranium analysis.

Issue of R1

Time Period From: 12/4/87 To: -/-/- Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

See attached.

Submitted by: James A. Gillard

Approved by: James T. Hunter, Laboratory Manager

Approved by: John R. Fragan, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000145

ADD

5.2.7 Glass liquid scintillation vials.

6.13 Urine Samples - Total Uranium

6.13.1 Thoroughly shake sample to suspend any particulates.

6.13.2 Measure an appropriate volume (up to 5 ml) of sample. Place in a tared glass liquid scintillation vial that has been pre-leached in 4M HNO_3 at 60 + °C for 48 hours followed by boiling in deionized water and final rinse with deionized water. Determine aliquot volume by weight to three (3) significant figures.

6.13.3 Add 3 ml of concentrated HNO_3 and 0.5 ml of 30% H_2O_2.

6.13.4 Place vials on a hot plate at medium heat. Wet-ash to dryness. Repeat reagent additions and wet-ashing two (2) more times.

6.13.5 Muffle vials as 525 °C for 1 hour. Cool.

6.13.6 Dissolve residue in 1 ml of 4M HNO_3. Add 4 ml of deionized water. Determine volume of solution by weight to three (3) significant figures.

6.13.7 Transfer to the appropriate procedure for further analysis.

TITLE: Sample Preparation	NO: RSL-201	PAGE: 1
	REVISION: 0	DATE: 8-05-87

1.0 PURPOSE

This procedure provides instructions for preparing samples for analysis.

2.0 SCOPE

This procedure applies to all samples requiring preparation prior to specific analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the processing of all samples.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experience with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee. The technician shall maintain a bound Laboratory Record Book for recording pertinent information. See Appendix 10.1 for an example format.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

None

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Balance (0-3600.00 grams capacity)

5.1.2 Blender

5.1.3 Drying Oven

5.1.4 Hot Plate

5.1.5 Mortar and Pestle

5.1.6 Muffle Furnace

5.1.7 Pulverizer

5.1.8 Sieves

5.1.9 Food Chopper/Grinder

5.2 Materials

5.2.1 Assorted Corningware Dishes

5.2.2 Assorted Stainless Steel Pans

5.2.3 Assorted Marinelli Beakers

5.2.4 Assorted Petri Dishes

5.2.5 Whatman 42 filter paper, or equal

5.2.6 Assorted tools and routine laboratory equipment (such as knives, beakers, graduated cylinders, etc.)

5.3 Reagent

5.3.1 Ethanol, 95% Reagent Grade

5.3.2 Glycerol, ACS Reagent Grade

5.3.3 40-Percent Formalin, ACS Reagent Grade

5.3.4 1M Calcium Nitrate Solution [$Ca(NO_3)_2$ $4H_2O$] (ACS Reagent Grade). Dissolve 118 grams of calcium nitrate in deionized water and dilute to 500 ml.

5.3.5 Phosphoric Acid (H_3PO_4), Concentrated, ACS Reagent Grade

5.3.6 Ammonium Hydroxide (NH_4OH), Concentrated, ACS Reagent Grade

5.3.7 0.1 M Phosphoric Acid: add 3.4 ml of H_3PO_4 to 300 ml of deionized water and dilute to 1 liter.

5.3.8 Octyl Alcohol

5.3.9 30% Hydrogen Peroxide (H_2O_2), ACS Reagent Grade

5.3.10 Hydroxylamine Hydrochloride ($NH_2OH \cdot HCL$), ACS Reagent Grade

5.3.11 Hydrofluoric Acid (HF), 48%, ACS Reagent Grade

5.3.12 Nitric Acid (HNO_3), Concentrated, ACS Reagent Grade

5.3.13 Fuming Nitric Acid (HNO_3), 90%, ACS Reagent Grade

5.3.14 8M Nitric Acid: Add 500 ml of concentrated HNO_3 to 400 ml of deionized water and dilute to 1 liter.

5.3.15 4M Nitric Acid: Add 250 ml of concentrated HNO_3 to 500 ml of deionized water and dilute to 1 liter.

5.3.16 1M Nitric Acid: Add 62.5 ml of concentrated HNO_3 to 500 ml of deionized water and dilute to 1 liter.

5.3.17 Hydrochloric Acid (HCl): Concentrated, ACS Reagent Grade

5.3.18 8M Hydrochloric Acid: Add 667 ml of concentrated HCl to 300 ml of deionized water and dilute to 1 liter.

5.3.19 Perchloric Acid ($HClO_4$), Concentrated, ACS Reagent Grade

5.3.20 Ethanol-glycerol mixture (10:1 vol/vol.)

6.0 PROCEDURE

6.1 Gamma Counting

6.1.1 Liquid Samples

6.1.1.1 All liquid samples are made up in one of two geometries (500 or 900 ml) in the appropriate polyethylene marinelli beakers. The volume of the sample depends on the radioactivity and the amount of liquid supplied by the customer.

6.1.1.2 Using a clean graduated cylinder, measure the required sample volume (500 or 900 ml) and pour the liquid into the appropriate marinelli beaker.

6.1.1.3 Place lid securely on the marinelli beaker and seal with electrical tape. Check for any leakage before sending the sample for counting.

6.1.1.4 Write sample information (ID number, sample volume and customer name) on the beaker and fill out the appropriate laboratory work sheet.

6.1.1.5 Submit for counting.

6.1.2 Soil Samples

6.1.2.1 All soil samples are made up in one of two geometries (650g marinelli or petri dish) depending on the quantity of the sample supplied by the customer.

6.1.2.2 For the marinelli geometry, approximately 650 grams of sample are needed.

6.1.2.3 For petri dish geometry (approximately 2.75 inches diameter) approximately 45 grams of sample are needed.

6.1.2.4 Dry and pulverize the sample according to directions in 6.8 below.

6.1.2.5 Weigh empty container with lid, (i.e., tare weight) fill with the required amount of sample and close the sample container securely. Determine net weight. Seal with electrical tape.

6.1.2.6 Write sample information (ID number, net weight of sample and customer's name) on the container, on the appropriate laboratory work sheet, and in sample prep logbook (see appendix 10.1).

6.1.2.7 Submit for counting.

6.1.3 Vegetation Samples

6.1.3.1 All vegetation samples are made up in 900 ml marinelli geometry. A sufficient quantity of sample to fill the beaker is needed for this geometry.

6.1.3.2 Weigh empty container with lid (i.e., tare weight).

6.1.3.3 Use the appropriate tool (e.g., shredder, blender, etc.) to cut the vegetation sample into small pieces such that the sample can be placed into the 900 ml marinelli beaker.

6.1.3.4 Cover the container, determine net weight and secure the lid with electrical tape.

6.1.3.5 Write sample information (ID number, net weight of sample, customer's name) on the container, on the appropriate laboratory work sheet, and in sample prep logbook (see appendix 10.1).

6.1.3.6 Submit for counting.

6.1.4 Air Filter Samples

6.1.4.1 All air filter samples are counted in the appropriate air filter geometry (i.e., single filter or composite filters).

6.1.4.2 Load the air filter(s) into the counting planchet and place into a petri dish. Cover dish and secure lid with electrical tape.

6.1.4.3 Write the sample information (ID number, etc.) on the lid of the dish and on the appropriate laboratory work sheet.

6.1.4.4 Submit for counting.

6.2 Water Samples - Beta Emitters

6.2.1 Where required by individual procedures, a measured volume of water is evaporated in a suitably sized glass beaker. Carriers may be added as necessary.

6.2.2 Dissolve residue in a small amount of 8M nitric acid and dilute as necessary for the appropriate procedure to be run.

6.3 Water Samples - Alpha Emitters (except Radium)

6.3.1 Measure the required volume of sample into a suitably sized glass beaker. Record data on appropriate laboratory work sheet.

6.3.2 Add the appropriate yield carriers/tracers as required by the analysis request.

6.3.3 While stirring, add 2 ml 1 M calcium nitrate and 10 ml concentrated phosphoric acid. Adjust the pH to 8.5 - 10.0 using ammonium hydroxide and pH paper as indicator.

6.3.4 Remove the stirrer and cover the beaker with plastic wrap to exclude atmospheric carbon dioxide and let the covered beaker stand for approximately 16 hours to complete the precipitation and settling.

6.3.5 Decant and aspirate the supernatant as completely as possible without disturbing the precipitate and discard it in the pH>2 waste acid container.

6.3.6 Slurry the precipitate with distilled water and transfer it quantitatively to a centrifuge bottle. Save beaker. Centrifuge for 5 minutes and discard the supernatant. Wash the precipitate with 50 ml 0.1 M phosphoric acid (approximately pH8), centrifuge and discard the supernatant.

6.3.7 Add approximately 15 ml concentrated nitric acid to beaker used for precipitation, heat on hot plate, swirl acid to rinse walls and pour contents into centrifuge bottle containing the washed precipitate. Transfer mixture to a second clean beaker.

6.3.8 Rinse the walls of the beaker used for precipitation with water, transfer rinse to centrifuge bottle used for collecting the precipitate, rinse the bottle and pool the rinse with the mixture in the second beaker. Evaporate the mixture to dryness on a hot plate. The residue is then ready for further analysis by the appropriate procedure.

6.4 <u>Urine Samples - Alpha Emitters (except Radium)</u>

6.4.1 Measure and record the volume of the urine specimen and transfer to a beaker. Record data on appropriate laboratory work sheet. Use nitric acid (50 ml/liter urine) to rinse the original container and the measuring cylinder and pour rinse into the beaker. Add the appropriate yield carriers/tracers as required by the analysis request. Add 5 drops of octyl alcohol and 10 ml of hydrogen peroxide to the urine, stir and heat to 85-90°C. (If plutonium analysis is required, add 0.5 g hydroxylamine hydrochloride).

6.4.2 While stirring, add 2 ml 1 M calcium nitrate and 10 ml concentrated phosphoric acid. Adjust the pH to 8.5 - 10.0 using ammonium hydroxide and pH paper as indicator.

6.4.3 Remove the stirrer and cover the beaker with plastic wrap to exclude atmospheric carbon dioxide and let the covered beaker stand for approximately 16 hours to complete the precipitation and settling.

6.4.4 Decant and aspirate the supernatant as completely as possible without disturbing the precipitate and discard it in the pH>2 waste acid container.

6.4.5 Slurry the precipitate with distilled water and transfer it quantitatively to a centrifuge bottle. Centrifuge for 5 minutes and discard the supernatant. Wash the precipitate with 50 ml 0.1 M phosphoric acid (approximately pH8), centrifuge and discard the supernatant.

6.4.6 Add 15 ml concentrated nitric acid to beaker used for precipitation, heat on hot plate, swirl acid to rinse walls and pour contents into centrifuge bottle containing the washed precipitate. Transfer mixture to a clean second beaker.

6.4.7 Rinse the walls of the beaker used for precipitation with water, transfer rinse to centrifuge tube used for collecting the precipitate, rinse the bottle and pool the rinse with the mixture in the second beaker. Evaporate the mixture to dryness on a hot plate and wet ash the residue with concentrated nitric acid as many times as necessary to obtain a clean white residue. This residue is ready to be analyzed.

6.5 <u>Urine - Non-Volatile Beta Emitters</u>

6.5.1 Measure and record the volume of the urine specimen and transfer to a beaker. Record data on appropriate laboratory work sheet. Add nitric acid (50 ml/liter urine) to the beaker. Add 5 drops of octyl alcohol and 10 ml of hydrogen peroxide to the urine, stir and wet ash to dryness.

6.5.2 Muffle the sample in the beaker for 16 hours at 525°C.

6.5.3 Dissolve residue in 50 ml concentrated nitric acid. Add 2 ml of hydrogen peroxide and evaporate to dryness. Repeat as necessary until residue is white.

6.5.4 Dissolve residue in a small amount of 8M nitric acid and dilute as necessary for the appropriate procedure to be run.

6.6 <u>Air Filter Samples - Alpha/Beta Emitters</u>

6.6.1 The air filter sample is placed in a suitably sized glass beaker and the beaker placed in a muffle furnace.

6.6.2 The sample is muffled @ 210°C for 8 hours, then @ 375°C for 16 hours, followed by 24 hours at 525°C.

6.6.3 Allow sample to cool, then transfer to a teflon beaker if glass fiber filters were used. If paper filters were used proceed to 6.6.5.

6.6.4 Add concentrated hydrofluoric acid slowly to the filters until dissolved. Evaporate to dryness. Repeat twice with 10 ml portions of hydrofluoric acid.

6.6.5 To the dry residue, add 25 ml of concentrated nitric acid. Evaporate to dryness. Repeat twice.

6.6.6 The dry residue is ready for analysis. The residue is made to 100 ml in a volumetric flash with 1 M nitric acid. Aliquots are taken from the flask and yield tracers and carriers added according to the analysis requested.

6.7 <u>Vegetation Samples - Non-Volatile Alpha/Beta Emitters</u>

6.7.1 Place the sample in a tared container. Weigh and record the weigh as the WET WEIGHT in grams, if not accomplished in 6.1.3 above. Record the data on appropriate laboratory work sheet and in sample prep logbook (See appendix 10.1).

6.7.2 Place the sample in a drying oven for approximately 16 hours at a temperature of approximately 110°C.

6.7.3 If the sample is dry, remove it from the oven and allow it to cool to room temperature. If the sample is not dry, continue heating until it is dry.

6.7.4 Weigh and record the weight as the DRY WEIGHT in grams.

6.7.5 Place sample in glass dish and place in muffle furnace.

6.7.6 Muffle at 210°C for 8 hours, then 375°C for 16 hours followed by 24 hours at 525°C.

6.7.7 Allow sample to cool prior to removing from furnace.

6.7.8 Obtain ASH WEIGHT of sample, if completely ashed. If sample is not completely ashed, return it to the furnace until it is completely ashed.

6.7.9 An appropriate ashed sample is weighed and dissolved in 50 ml of a 2:1 mixture of concentrated nitric acid and concentrated hydrochloric acid.

6.7.10 Evaporate the sample solution to dryness slowly. Repeat the acid addition and evaporation.

6.7.11 Dissolve the residue in 50 ml of concentrated nitric acid. Evaporate to dryness. Repeat twice.

6.7.12 Dissolve residue in 50-100 ml of 4 M nitric acid. Filter through Whatman 42 or equal as necessary. Dilute to volume in a volumetric flask. The sample is now ready for further analysis.

6.8 <u>Sediment and Soil Samples - Non-Volatile Isotopes</u>

6.8.1 Place the sample in a tared container. Weigh and record the weight as the WET WEIGHT in grams. Record data on appropriate laboratory work-sheet and sample prep logbook.

6.8.2 Place the sample in a drying oven for approximately 16 hours at a temperature of approximately 110°C.

6.8.3 If the sample is dry, remove it from the oven and allow it to cool to room temperature. If the sample is not dry, continue heating until it is dry.

6.8.4 Weigh and record the weight as the DRY WEIGHT in grams.

6.8.5 Grind the sample to at least 40 mesh. Transfer the sample to a container and label with the laboratory sample number. Store for analysis.

6.8.6 If gamma scan is required, prepare sample as described in 6.1.2 above.

6.8.7 If chemistry analysis is required and specific procedure(s) assume dissolution, weigh an appropriate (less than 20g) size sample and place in a 250 ml beaker. Add appropriate yield tracers and carriers, if required.

6.8.8 Place a stirring bar in the beaker. Cautiously add 100 ml of concentrated HNO_3. Cover with a watch glass. Stir slowly. When the reaction ceases, cautiously add 50 ml of concentrated HCl. DO NOT STIR. Replace watch glass and allow the sample to stand at room temperature for 2-4 hours. Slowly begin stirring after initial reaction subsides (approximately 30 min.). Gradually heat the sample on a low temperature hot plate overnight. The temperature should be high enough so that refluxing occurs without stirring, but sample does not go dry.

6.8.9 Allow the mixture to cool and settle, then add 100 ml of water. Filter the soil residue onto a Whatman #42 or equal filter. Wash the precipitate with 20 ml 8M HNO_3, collect the filtrate in a 400 ml beaker and reserve.

6.8.10 Transfer the filter and soil residue to a 100 ml beaker, dry over low heat and carefully muffle at 525°C overnight.

6.8.11 Cool, add 25 ml of concentrated HNO_3, transfer to teflon beaker and add 10 ml of concentrated HF to the soil residue. Evaporate to dryness, repeat the additions of acids and evaporations three more times.

6.8.12 Add 25 ml of concentrated HNO_3. Transfer to glass beakers, then add 5 ml of concentrated $HClO_4$ and heat over low heat in the perchloric acid hood until perchloric acid fumes are no longer present. Repeat twice.

6.8.13 Add 10 ml of concentrated nitric acid and 10 ml of concentrated hydrochloric acid to dissolve the residue. Heat if necessary. Cool, then slowly add 25 ml of demineralized water.

6.8.14 Filter the sample through Whatman 42 or equal filter paper catching the filtrate with the filtrate reserved earlier in 6.8.9.

6.8.15 Evaporate to wet dryness (approximately 5 mls) over low heat.

6.8.16 If strontium analysis is not requested proceed to 6.8.23.

6.8.17 If strontium is requested, dissolve the residue in 20 ml of concentrated nitric acid. Heating may be necessary to effect solution of residue.

6.8.18 Transfer sample to centrifuge tube, rinsing beaker with a small amount of concentrated nitric acid.

6.8.19 Add 10 ml of fuming nitric acid to the sample and mix thoroughly. Cool in ice water bath.

6.8.20 Centrifuge sample for three minutes on high speed.

6.8.21 Collect supernatant in a 250 ml beaker for further analysis if required. Otherwise discard.

6.8.22 Transfer the precipitate in the tube to the strontium in water procedure at step 6.6.

6.8.23 The supernatant retained in 6.8.21 is evaporated to wet dryness on a hot plate on low to medium heat.

6.8.24 Dissolve residue to approximately 20 ml of concentrated hydrochloric acid and again evaporate to dryness. Repeat twice with 10 ml addition of acid.

6.8.25 Transfer residue to iron separation procedure, if necessary.

6.9 <u>Metallic Samples</u>

6.9.1 Weigh an appropriate sample size and place in glass beaker large enough to accommodate acids volumes 10-20 times the weight of the sample. Record data on appropriate laboratory work sheet.

6.9.2 Add alpha yield tracers, as required.

6.9.3 Slowly add concentrated nitric acid to the sample. If sample does not dissolve with low heating, add concentrated hydrochloric acid slowly until a 2:1 acid mixture is obtained.

NOTE: If volatile isotopes are being determined, the dissolution may need to be done more slowly without heat and with diluted acids or in a refluxing apparatus.

6.9.4 When sample is dissolved, slowly evaporate to wet dryness.

6.9.5 Add 25 ml of 8 M hydrochloric acid and evaporate slowly to wet dryness. Repeat twice with 10 ml additions of acid.

6.9.6 Transfer to iron separation procedure, if necessary.

6.10 Meat Samples

6.10.1 Wash the sample and remove the skin, bones, and fat, as necessary.

6.10.2 Cut into small pieces. Grind in food chopper/grinder, if required. Weigh and record the weight as the WET WEIGHT in grams. Record data in appropriate laboratory work-sheet and in sample prep logbook.

6.10.3 If gamma scan is required, prepare sample as described in 6.1.3 above using 500 or 900 ml marinelli, as required.

6.10.4 If wet chemistry is required, proceed as described below.

6.10.5 Transfer the sample to a tared stainless steel pan and place in a drying oven for approximately 32 hours at a temperature of approximately 110°C.

6.10.6 If the sample is dry, remove it from the oven and allow to cool to room temperature. If the sample is not dry, continue heating until it is dry.

6.10.7 Weigh and record the weight as the DRY WEIGHT in grams.

6.10.8 Put the pan on top of a preheated hot plate under a hood and add 400 ml of ethanol-glycerol mixture for each 300 g of dry sample.

6.10.9 Ignite the mixture; stir with a glass rod while burning until the sample oil has completely burned.

6.10.10 Transfer the sample to a Corningware dish and place in a muffle furnace.

6.10.11 Transfer to 6.7.6.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility of Inspection

8.1.1 The technician performing this procedure shall be responsible for inspecting samples after ashing, blending, drying, or grinding to determine if processes are complete. The technician shall be responsible for inspecting labels for correctness and for inspecting quantities of samples. The technician is responsible for inspecting equipment to determine if it is working properly.

8.1.2 The Laboratory Manager, or a designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.3 The Laboratory Manager shall periodically review and initial record books and data entries.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

Reagents shall be ACS Reagent grade or better as indicated in Section 5.3. Lower grade material may be used for items not so indicated. If ACS Reagent grade material is not available, the best grade available shall be used.

8.4 Equipment Monitoring

8.4.1 Drying oven thermometer shall be in place and show correct oven temperature according to oven logbook.

8.4.2 Balances shall be checked to ensure calibration dates have not expired.

8.4.3 Balances shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 Portions of this procedure may be certified by submitting multiple spiked samples through the procedure steps and analyzing for the activity found.

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Sample Preparation	NO: RSL-201	PAGE: 16
	REVISION: 0	DATE: 8-05-87

APPENDIX 10.1
Sample Preparation Record Book Format Example

Collection Date + Time	Sample Number	Sample Type	WET WEIGHT			DRY WEIGHT			GROSS DRY WEIGHT			ASH WEIGHT			Crucible Weight g	Analyst Name	Comments
			Tare g	Pan + Sample g	Net Weight g	Tare g	Pan + Sample g	Net Weight g	Tare g	Pan + Sample g	Net Weight g	Tare g	Pan + Sample g	Net Weight g			
6-21-81 1300	107818-7	Corn	291	3658	3367	1369 A 1402 B	1923 1768	610	—	—	610	1587 1500	1587 1500	36	3.6 kg	MKA	
8-31-81 1110	108063-5	Fish Flesh SB	—	—	—	—	—	273	—	—	273	1310	1530	20	233.15	MKA	Cond top leading balance
10-26-81 1030	109509-6	Vegetation	861	1194	333	861	952	91	861	951	90	1056	1062	6	89.12	MKA	
11-23-81 1145	110-23-6	Soil	852	1892	1040	852	1677	825	852	1666	81	1075	1842	767	600.05	MKA	
																	RAI 12-1-81
12-1-81 0833	110333-9	Sediment	832	1975	1143	832	1956	1122	832	1949	1117	912	2010	1098	599.16	EKA	

430-1-87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-202

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Extraction of Iron from Soil or Metallic Samples

APPROVED: James T. Harney DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Miller DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cuffin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED

REVISION: 0				
DATE: 5/18/87				

429-1-87

RSL-202, R0-CH1
1 of 1

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.4; 6.1

Procedure Title & Number (If exists) RSL-202, R0
Extraction of Iron from Soil or Metallic Samples

Description of experiment or method Procedure enhancement to assure ferric oxidation state. CAUTION on possible explosive mixture

Time Period From: 8/25/87 To: Issue of RSL-202, R1 Samples Affected All until revision of RSL-202

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-25-87

5.3.4 Hydrogen Peroxide (H_2O_2), 30% ACS reagent grade

6.1 Dissolve sample in 30 ml of 8 M HCl. Add 100 microliters (100λ) of hydrogen peroxide (30%) and heat for approximately 10 minutes until the hydrogen peroxide no longer bubbles from solution. Cool to room temperature.

If any solid remains, filter sample, wash beaker with additional 20 ml 8 M HCl and add to filtered sample catching in 100 ml beaker.

CAUTION: Inorder to avoid a possible explosive mixture, Do Not add sample to isopropyl ether unless all of the hydrogen peroxide is removed from the sample and the solution is cooled to room temperature.

Submitted by: James M. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Fraser, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000164

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.4

Procedure Title & Number (if exists): RSL-202, R0
Extraction of Iron from Soil or Metallic Samples

Description of experiment or method: Add disposal instructions for ether waste.

Time Period From: 9/28/87 To: Issue of RSL-202 R1 Samples Affected: All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 9-28-87

5.3.5 0.1 M Sodium Sulfite (Na_2SO_3): Dissolve 1.3 g of Na_2SO_3 in 50 ml of demineralized water and dilute to 100 ml with demineralized water. Make fresh prior to use.

6.4 Drain off sample into 100 ml beaker. Discard ether into a clean waste ether container which contains 25 ml of 0.1 M sodium sulfite (Na_2SO_3). Allow the waste ether to evaporate in a fume hood. Discard remaining acqueous waste into the >pH2 liquid waste container.

Submitted by: James H. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. A. Cuyler, Laboratory Director

Approved by: Cheryl D. Prince QA/QC/Director, ITAS

000165

TITLE: Extraction of Iron from Soil or Metallic Samples	NO: RSL-202	PAGE: 1
	REVISION: 0	DATE: 5/18/87

1.0 PURPOSE

This procedure provides an analytical method for removal of iron from soil or metallic samples when necessary.

2.0 SCOPE

This procedure applies to all samples requiring removal of bulk quantities of iron from samples prior to continuing with other procedures.

3.0 RESPONSIBILITY

3.1 Is is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the removal of iron from samples requiring such removal.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

None

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Separatory funnels

5.1.2 Laboratory equipment as required (beakers, etc.)

5.2 Materials

None

5.3 Reagents

5.3.1 Isopropyl Ether, ACS reagent grade

5.3.2 Hydrochloric Acid, concentrated, ACS reagent grade

5.3.3 8 M Hydrochloric Acid; add 500 ml of concentrated hydrochloric acid to 500 ml of demineralized water. Mix thoroughly.

6.0 PROCEDURE

6.1 Dissolve sample in 30 ml of 8 M HCl. If any solid remains, filter sample. Wash beaker with additional 20 ml 8 M HCl. Add to filtered sample catching in 100 ml beaker.

6.2 Pour into 125 ml separatory funnel.

6.3 Add 50 ml isopropyl ether. Shake 1 minute. Let set 5 min.

6.4 Drain off sample into 100 ml beaker. Discard ether.

6.5 Repeat steps 6.2, 6.3 and 6.4.

6.6 Evaporate to approximately 1-2 ml. Do not let go dry or bake.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

TITLE: Extraction of Iron from Soil or Metallic Samples	NO: RSL-202	PAGE: 4
	REVISION: 0	DATE: 5/18/87

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-301

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Promethium-147

APPROVED: James T. Harvey DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Mill DATE: 6-8-87
ITAS QA Director

CONCURRED: John C. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-20-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-301, RO
Determination of Promethium-147

Description of experiment or method Correct equations to calculate error and Lower Limit of Detection

Time Period From: 4/20/87 To: 8/11/87 Samples Affected All between 8-11-87 and 4-20-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James M. Willard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John G. Griffin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Pm-147 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME}$$

Pm-147 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.005\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME* N}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency

$$= \frac{STC - BKG}{STDPM}$$

SVOL = Sample volume (l)
N = Number of counts averaged
STC = Standards counts, average N counts
STDPM = Standard activity (dpm)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Promethium-147	NO: RSL-301	PAGE: 1
	REVISION: 0	DATE: 4-20-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of promethium-147 in urine and environmental aqueous samples.

2.0 SCOPE

This procedure applies to all liquid samples requiring promethium-147 determination.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring promethium-147 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 E-PM-01, "Radiochemical Determination of Promethium-147", EML Procedures Manual (HASL), 26th Edition, 1983.

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Liquid scintillation counter

5.2 Materials

5.2.1 Polyethylene liquid scintillation vials (7 ml capacity)

5.2.2 pH paper, narrow range around pH 3.4

5.2.3 Routine laboratory equipment

5.3 Reagents

5.3.1 5 M Sodium Hydroxide (NaOH), ACS reagent grade solution commercially prepared

5.3.2 Extractant-Scintillator solution: Dissolve 0.6 g of PPO (2,5-diphenyloxazole), 0.1 g of di-methyl o-POPOP (1.4-bis-2(4-methyl-5-phenyloxazolyl) benzene) and 7 g of di-2-ethyl hexyl phosphoric acid in 100 ml of toluene. (This solution should be stored in the dark, and be prepared fresh every month.)

5.3.3 Liquid scintillation cocktail (commercial)

5.3.4 Nitric acid (HNO_3), concentrated, ACS Reagent grade

6.0 PROCEDURE

6.1 Aqueous Samples

6.1.1 Add 20 ml of concentrated HNO_3 to a suitable aliquot and evaporate to 2-3 ml. Run two spikes and two blanks with each set of samples. Record on appropriate data form (appendix 10.1).

6.1.2 Add 5 ml of water. Transfer to a 25 ml volumetric flask.

6.1.3 Add NaOH solution dropwise, with stirring, and adjust the pH to 3.4. (Use narrow range pH paper)

6.1.4 Add water until the level of the solution is in the neck of the flask.

6.1.5 Add 200 µl of extractant-scintillator solution.

6.1.6 Shake the flask vigorously for two minutes.

6.1.7 Remove the upper organic phase with a micropipet and transfer to a sample vial. Do not attempt to draw off the last bit of organic phase.

6.1.8 Repeat the extraction twice using 200 µl and then 100 µl aliquots of extractant-scintillation solution.

6.1.9 Count for beta activity. Standardize counter against known amounts of Pm-147 carried through the procedure.

6.2 Urine Samples

6.2.1 To 250 ml of urine in an Erlenmeyer flask, add 20 ml of concentrated HNO_3 and boil to near dryness. Run two spikes and two blanks with each set of samples. Record data on appropriate form (appendix 10.2).

6.2.2 Add an additional 5 ml of concentrated HNO_3 and evaporate to near dryness.

6.2.3 Continue 5 ml additions of concentrated HNO_3 until the sample is colorless.

6.2.4 Dissolve the salts with water and transfer to a 25 ml volumetric flask. Continue with step 6.1.3 above.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 15% at 200 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.3 pH adjustment

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Pm-147 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME}$$

Pm-147 Activity Error (pCi/l) = ERR

$$= ACT* \quad \frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*SVOL*CTIME* \quad N}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency

$$= \frac{STC - BKG}{STDPM}$$

SVOL = Sample volume (l)
N = Number of counts averaged
STC = Standards counts, average N counts
STDPM = Standard activity (dpm)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Promethium-147	NO: RSL-301	PAGE: 6
	REVISION: 0	DATE: 4-20-87

Appendix 1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

387-9-86

430-1 87

TITLE: Determination of Promethium-147	NO: RSL-301	PAGE: 7
	REVISION: 0	DATE: 4-20-87

Appendix 2

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367-9-86

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-302

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Tritium in Aqueous Samples by Distillation

APPROVED: James T. Harvey DATE: 5-6-87
Laboratory Manager

APPROVED: Paul Mills DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cuyjet DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-302, RC
Determination of Tritium in Aqueous Samples by Distillation

Description of experiment or method Correct equations to calculate error and lower limit of Detection

Time Period From: 4/14/87 To: 8/11/87 Samples Affected All between 4-14-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James Mallad

Approved by: John R. Frazier, Laboratory Manager

Approved by: John G. Cuffier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000181

TITLE: Determination of Tritium in Aqueous Samples by Distillation	NO: RSL-302	PAGE: 5
	REVISION: 0	DATE: 4-14-87

9.0 CALCULATIONS

Tritium Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME} * \frac{(VNEUT + VNAOH)}{VNEUT}$$

Tritium Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME* N} * \frac{(VNEUT + VNAOH)}{VNEUT}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency

$$= \frac{STC - BKG}{STDPM}$$

SVOL = Sample volume (l)
VNEUT = Sample volume neutralized (ml)
VNAOH = NAOH volume added (ml)
N = Number of counts averaged
STC = Standards counts, average N counts
STDPM = Standard activity (dpm)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

10.0 APPENDICES

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: [illegible]

Procedure Title & Number (if extant) RSL-302 R0, Determination of Tritium in Aqueous Samples by Distillation

Description of experiment or method to conform to EPA 900 Methodology

Issue of RSL-302 R1

Time Period From: 12/9/87 To: ~~/ /~~ Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

See attached.

Submitted by: James T. Harvey

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. A. Cuffin, Laboratory Director

Approved by: Paul Mills, QA/QC/Director, ITAS

000183

CHANGE

3.3.1 Sodium Hydroxide, ACS Reagent Grade

3.3.2 Potassium Permanganate, ACS Reagent Grade

6.1.1 Measure 75 ml of sample and add 0.5 g of sodium hydroxide and 0.1 g of potassium permanganate. Mix. Check pH with pH paper to insure sample is basic. Add sodium hydroxide as necessary. Record on appropriate data form. See Appendix 10.1. Use demineralized water for a blank with each set of samples.

6.1.2 Transfer sample to the distillation flask.

6.1.3 Distill approximately 10 ml and discard. Collect the next 20-40 ml of distillate.

6.1.4 Pipette 1 to 10 ml of distillate into a counting vial. Fill vial with cocktail and mix. Prepare a tritium standard for counter efficiency determination with each set of samples.

9.0 CALCULATIONS

Tritium Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME}$$

Tritium Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.0025 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME* (N)^{1/2}}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency

$$= \frac{STC - BKG}{STDPM}$$

SVOL = Sample volume (l)

N = Number of counts averaged
STC = Standards counts, average N counts
STDPM = Standard activity (dpm)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Tritium in Aqueous Samples by Distillation	NO: RSL-302	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of tritium in urine and environmental aqueous samples by distillation.

2.0 SCOPE

This procedure applies to all samples requiring tritium activity analysis by distillation.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring tritium activity determination by distillation.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

None

5.2 Materials

5.2.1 Polyethylene liquid scintillation vials (20 ml capacity)

5.2.2 Routine laboratory equipment

5.3 Reagents

5.3.1 5 M Sodium Hydroxide (NaOH), ACS reagent grade solution commercially prepared

5.3.2 0.1 M Sodium Hydroxide: Add 1 ml of 5 M NaOH to 50 ml of demineralized water in a plastic centrifuge cone, mix thoroughly.

5.3.3 Liquid scintillation cocktail (commercial)

6.0 PROCEDURE

6.1 Environmental Aqueous Samples - Distillation Method

6.1.1 Measure between 50 - 100 ml of sample and adjust pH, by pH meter, to be between 7.0 and 7.5 using sodium hydroxide. Remeasure adjusted sample volume. Record on appropriate data form. See appendix 10.1. Use demineralized water for a blank with each set of samples.

6.1.2 Transfer an aliquot of the sample to the distillation flask and assemble the apparatus.

6.1.3 Distill approximately 5 ml and discard. Collect the next 15 to 20 ml of distillate.

6.1.4 Pipette 5.0 ml of distillate into counting vial, containing 15 ml of scintillation cocktail and mix. Prepare a tritium standard for counter efficiency determination with each set of samples.

6.1.5 Submit sample(s) to counting room for counting. Obtain required number of counts.

6.1.6 Record the counting data on the data form.

6.1.7 Calculate the results as shown in section 9.0.

6.2 Urine - Distillation Method

6.2.1 Transfer using 6.1.1 (Adjust pH using pH paper)

6.2.2 Follow 6.1.2 through 6.1.7.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 10% at 4000 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.1 pH adjustment and sample volume

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 pH meter shall be checked to ensure calibration.

8.4.2 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Tritium Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME} * \frac{(VNEUT + VNAOH)}{VNEUT}$$

Tritium Activity Error (pCi/l) = ERR

$$= ACT* \quad \frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.0025$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*SVOL*CTIME* \quad N} * \frac{(VNEUT + VNAOH)}{VNEUT}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency

$$= \frac{STC - BKG}{STDPM}$$

SVOL = Sample volume (l)
VNEUT = Sample volume neutralized (ml)
VNAOH = NAOH volume added (ml)
N = Number of counts averaged
STC = Standards counts, average N counts
STDPM = Standard activity (dpm)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Tritium in Aqueous Samples by Distillation	NO: RSL-302	PAGE: 6
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-303

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Phosphorus-32 Activity

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul [illegible] DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Cuyin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
(If yes, enter paragraph number to reference insertion point: 5.2.15 5.2.18, 6.1 = 6.22)
Procedure Title & Number (If exists) RSL 304, R0

Determination of Selected Alpha Emitting Isotopes

Description of experiment or method Reverse order of column to improve isotopic separation

Issue of RSL-304, R1

Time Period From: 9/24/87 To: ~~11~~ Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 9-24-87

I. Change steps

From	To
5.2.15	5.3.15
5.2.16	5.3.16
5.2.17	5.3.17
5.2.18	5.3.18
6.11	6.15
6.12	6.16
6.13	6.17
6.14	6.18
6.15	6.19
6.16	6.20
6.17	6.21
6.18	6.22
6.19	6.23
6.20	6.24
6.21	6.25
6.22	6.26

II. Delete old steps 6.1 through 6.10

III. Add new steps 6.1 through 6.14 see attached

Submitted by: James M. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. C. Cupin, Laboratory Director

Approved by: Vicky L. Hutton for C. Prince, QA/QC/Director, ITAS

000193

6.1 Fill out appropriate data sheets (see appendix no. 1). Evaporate sample to dryness. Add 5ml of concentrated HNO_3 and 100 µl of 30% H_2O_2. Evaporate to dryness. If only uranium analysis is requested, proceed to step 6.8

6.2 Dissolve residue in 20ml of 8M HNO_3. Heat as necessary to dissolve, then cool to room temperature. Centrifuge if necessary to eliminate insoluble material.

6.3 Add sample to DOWEX 1-X8 (100-200 mesh) anion column which has been preconditioned with 10ml of 8M HNO_3. Collect effluent (Am, U) in a 250ml beaker.

6.4 If only Am is required, wash column with 20ml of 8M HNO_3, otherwise, wash column with 60ml of 8M HNO_3, collecting in same beaker as previous step. Once through column, transfer beaker to low heat hotplate, add 1ml of 30% H_2O_2 and evaporate to dryness. For Americium only, proceed to step 6.8.

6.5 Elute Th with 30ml of 8M HCl. Collect in beaker. Once through column, evaporate to dryness on hot plate.

6.6 Elute Pu with 30ml of 8M HCl + 0.1N NH_4I solution. Collect in a 250ml beaker. Once through column, add 1ml of concentrated HNO_3, evaporate to dryness on hot plate.

6.7 Elute Np with 30ml of 6.5M HCl + 0.004M HF solution. Collect in Teflon beaker. Once through column, evaporate to dryness. Add 5ml of concentrated HCl and evaporate to dryness.

6.8 To the dried residue from step 6.4 or 6.1, add 5 ml of concentrated HCl. Evaporate to dryness.

6.9 Dissolve residue in 15 ml of 8M HCl. Heat as necessary to dissolve, then cool to room temperature.

6.10 Add sample to a DOWEX 1-X8 (100-200 mesh) anion column which has been preconditioned with 10 ml of 8M HCl. Wash column with 10 ml of 8M HCl after sample has passed through. Collect effluents in beaker for Am analysis. Evaporate to dryness.

6.11 Wash column with 25 ml of 8M HCl + 0.1 M NH_4I solution, followed by 25 ml of 6.5 M HCl + 0.004 M HF solution, followed by 5 ml of 6.5 M HCl. Discard all effluents in < pH2 acid waste container.

6.12 Elute U with 30 ml of 0.5 M HCl. Collect in beaker for U analysis. Evaporate to dryness.

6.13 For U, Th, Np or Pu analysis, proceed to step 6.21

6.14 Dissolve the dried Am residue in 5 ml of concentrated HNO_3 and evaporate to dryness. Repeat.

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 4, 4.1, 4.2, 4.3, 6, 15, 16, 20, 6.2

Procedure Title & Number (if exists): RSL-304 R0

Determination of Selected Alpha Emitting Isotopes

Description of experiment or method: To improve Am, Cm yield and decontamination factors

Issue of RSL-304, R1

Time Period From: 12/9/87 To: -/-/-- Samples Affected: All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

See attached.

Submitted by: James T. Harvey

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. G. Cuffin, Laboratory Director

Approved by: Paul Nutty, QA/QC/Director, ITAS

000196

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of uranium, thorium, neptunium, plutonium, americium and curium alpha emitting isotopes.

2.0 SCOPE

This procedure applies to all samples requiring uranium, thorium, neptunium, plutonium, americium and curium activity analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring gross alpha and/or gross beta activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Montioring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Ouality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual

4.1.6. ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "Radioisotope Techniques," R.T. Overman and H.M. Clark, McGraw-Hill Book Co., Inc., New York, 1960. (Chapter 6)

4.4.2 "The Radiochemisry of Uranium", J.E. Grindler, NAS-NS-3050, National Academy of Sciences, March 1962.

4.4.3 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water"; H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, August 1980.

4.4.4 "Standard Methods for the Examination of Water and Waste Water," APHA, AWWA, WPCF, American Public Health Association, Washington, D.C, latest edition.

4.4.5 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.6 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

4.4.7 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.8 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

4.4.9 "Radiometric Method for the Determination of Uranium in Water: Single Laboratory Evaluation and Interlaboatory Collaborative Study", C.T. Bishop, V.R. Casella, and A.A. Glosby, EPA-600/7-79-093, U.S. Environmental Protection Agency, April 1979.

4.4.10 "The Radiochemistry of Neptunium", G.A. Burney and R.M. Harbour, NAS-NS-3060, National Academy of Science, December 1974.

4.4.11 "The Radiochemistry of Plutonium", G.H. Coleman, NAS-NS-3058, National Academy of Science, September 1965.

4.4.12 "The Radiochemistry of Thorium", E.K. Hyde, NAS-NS-3004, National Academy of Science, January 1960.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Beaker, assorted sizes

5.1.2 Water bath with magnetic stirrers

5.1.3 100 ml glass centrifuge tubes

5.1.4 Plastic centrifuge tubes

5.1.5 Ion exchange columns

5.1.6 Analytical balance (0-160.0000g)

5.1.7 Assorted tools and routine laboratory equipment

5.2 Materials

5.2.1 Routine laboratory supplies

5.2.2 Anion exchange resin, Bio-Rad AG 1-X8, 100 - 200 mesh

5.2.3 Membrane filters, 0.8 μm pore size

5.3 Reagents

5.3.1 Hydrochloric Acid (HCl), concentrated, ACS reagent grade

5.3.2 Nitric Acid (HNO_3), concentrated, ACS reagent grade

5.3.3 Hydrofluoric Acid (HF), concentrated, ACS reagent grade

5.3.4 8 M Hydrochloric Acid (HCl): Add 667 ml of concentrated HCl to 333 ml of demineralized water. Mix thoroughly.

5.3.5 0.5 M Hydrochloric Acid (HCl): Add 42 ml of concentrated HCl to 958 ml of demineralized water. Mix thoroughly.

5.3.6 8 M Hydrochloric Acid (HCl) + 0.01 M Nitric Acid (HNO_3): To 1 liter of 8 M HCl add 1 ml of concentrated HNO_3. Mix thoroughly.

5.3.7 13 M Hydrofluoric Acid (HF): Add 450 ml of concentrated HF to 550 ml of demineralized water. Mix thoroughly.

5.3.8 1 M Nitric Acid (HNO_3): Add 65 ml of concentrated HNO_3 to 935 ml of demineralized water. Mix thoroughly.

5.3.9 Phosphoric Acid (H_3PO_4), concentrated, ACS reagent grade

5.3.10 8 M Nitric Acid (HNO_3): Add 500 ml of concentrated HNO_3 to 500 ml of demineralized water. Mix thoroughly.

5.3.11 8 M Hydrochloric Acid (HCl) + 0.1 M Ammonium Iodide (NH_4I): Dissolve 3 grams of ammonium iodide in 200 ml of 8 M HCl. Mix thoroughly.

5.3.12 Cerium Nitrate solution (200 µg/ml, Ce): Add 0.3 grams of cerium nitrate to 500 ml of demineralized water. Mix thoroughly.

5.3.13 2% Titanous Chloride ($TiCl_3$) solution: Add 10 ml of 20% titanous chloride solution to 90 ml of demineralized water. Mix thoroughly.

5.3.14 20% Titanous Chloride, ACS reagent grade

5.2.15 Bismuth Nitrate solution (100 mg/ml bismuth): Add 232 grams bismuth nitrate [$Bi(NO_3)_3.5H_2O$] to 1 liter of demineralized water. Mix thoroughly.

5.2.16 6.5 M Hydrochloric Acid (HCl): Add 540 ml of concentrated HCl to 460 ml of demineralized water. Mix thoroughly.

5.2.17 6.5 M Hydrochloric Acid (HCl) + 0.004 M Hydrofluoric Acid (HF): Add 1.5 ml of concentrated HF to 1 liter of 6.5 M HCl. Mix thoroughly.

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 5
	REVISION: 0	DATE: 4-06-87

6.0 PROCEDURE

6.1 Fill out appropriate data sheets (see appendix 10.1). The dry sample is dissolved in 15 ml 8 M hydrochloric acid/0.01 M nitric acid. Apply to column which has been pre-equilibrated with 5 ml of the mixed acid at a flow rate of approximately 0.5 ml/minute. Collect effluent in 100 ml beaker.

6.2 Wash column with 35 ml 8 M hydrochloric acid, pool wash with effluent and evaporate solution to dryness. This fraction contains americium, curium and thorium.

6.3 Plutonium is then eluted from the column with 15 ml of 8 M hydrochloric acid + 0.1 M ammonium iodide. Wash column with 20 ml 8 M hydrochloric acid, pool with eluate, add 0.5 ml of concentrated HNO_3 and evaporate to dryness. Proceed to 6.17.

6.4 Neptunium is eluted from the column with 15 ml of 6.5 M HCl + 0.004 M HF followed by 20 ml of 6.5 HCl. Collect in teflon beaker. Evaporate to dryness. Using concentrated HCl, dissolve residue and transfer to glass beaker. Proceed to 6.17.

6.5 Uranium is eluted from the column with 35 ml 0.5 M hydrochloric acid and the eluant evaporated to dryness. Proceed to 6.17.

6.6 If americium, curium or thorium analysis is required, dissolve residue from 6.2 in 20 ml of 8 M nitric acid.

6.7 Pass over a small anion exchange resin column which has been pre-equilibrated with 5 ml of 8 M HNO_3 at a flow rate of approximately 0.5 ml per minute. Collect effluent for americium and curium analysis.

6.8 Wash the resin column with 30 ml of 8 M nitric acid collecting the effluent in same beaker used in 6.7. Evaporate to dryness slowly.

6.9 Elute the thorium from the column using 30 ml of 8 M hydrochloric acid, collecting the elutant in a 50 ml beaker and evaporate to dryness. Proceed to 6.17.

6.10 If americum and/or curium analysis is requested, the dry residue from 6.8 above is dissolved in 2 - 3 ml reagent grade nitric acid and the acid evaporated. Repeat this step.

6.11 Dissolve residue in 10 ml 1 M nitric acid and transfer with water to centrifuge tube. Total volume about 80 ml.

6.12 Place tube in hot water bath and stir mechnically.

6.13 Add 1 ml bismuth nitrate solution followed by 1 ml phosphoric acid. Stir for 1 hour.

6.14 Centrifuge and discard supernatant.

6.15 Transfer precipitate with water to 50 ml centrifuge tube.

6.16 Centrifuge and discard the supernatant.

6.17 The dried residue (or precipitate) is dissolved in 1 - 2 ml of reagent grade hydrochloric acid. Heat to effect dissolution of sample.

6.18 Transfer solution to tube, rinse out beaker with 10 ml water and pool into tube.

6.19 Add 1 ml cerium nitrate carrier.

6.20 Add 1 ml 2% titanous chloride (not necessary for americium and curium). Allow to stand for 5 minute in hot water bath.

6.21 Add 1 ml hydrofluoric acid, cool in ice water bath, then filter through membrane filter.

6.22 Mount for counting and submit to counting room. The yield determined by tracer shall be greater than 40%.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 15% at 1 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequte quality.

8.1.2 The technician performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.22 Chemical yield

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 The alpha spectroscopy system shall be calibrated before use.

8.4.2 Balances shall be checked to ensure calibration dates have not expired.

8.4.3 Balances shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 8
	REVISION: 0	DATE: 4-06-87

9.0 CALCULATIONS

9.1 Alpha Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG) * SD}{SC * SVOL * 2.22 * IC}$$

Alpha Activity Error (pCi/l) = ERR

$$= ACT * \frac{TSC}{(TSC - BKG)^2} + \frac{SC}{(SC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{(4.65 * BKG + 3) * SD}{SC * 2.22 * SVOL * IC}$$

TSC = Sample counts in region of interest
BKG = Background counts in region of interest
SD = Standard dpm
SC = Standard counts
IC = Alpha branching correction; U-235 = 0.85; Other = 1.0
SVOL = Sample volume (l)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 9
	REVISION: 0	DATE: 2-23-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367 4 86

430 1-87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-305

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Strontium-89,90 in Water Samples

APPROVED: James T. Harvey DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Nill DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Angier DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

RSL-305, RO-CH1

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.2.1; 6.7; 6.10
Procedure Title & Number (If exists) RSL-305, RO
Determination of Strontium-89,90 in Water Samples

Description of experiment or method Retain barium chromate precipitate for radium analysis if required. Modify materials list to include Nylon 66 membrane filter.

Time Period From: 8/8/87 To: Issue of RSL-305, R1 Samples Affected All from 8-8-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-8-87

5.2.1 Nylon 66 membrane filter, 1.2 micron, 4.7 cm ~~Whatman 42 or glass fiber filter paper, 4.7 cm, as necessary~~

6.7 Add 5 ml of 1.5 M sodium chromate and heat the solution for 3 minutes. Add 1 drop of phenolphthalein then add glacial acetic acid dropwise until the color changes from reddish-orange to golden-yellow and add 1 ml of 6 M acetic acid. Heat 2 minutes, centrifuge, add an additional 0.5 ml of barium carrier. Gently swirl the solution to mix while the previous precipitate is disturbed as little as possible. Cool the solution for 2 minutes, centrifuge and ~~discard the precipitate~~ retain the precipitate for further analysis unless otherwise directed.

6.10 Precipitate the yttrium hydroxide from the saved strontium sulfate solution by adding 10 M NaOH to the purple end point of the MCP indicator and 2 drops extra. Add 1.25 ml of strontium carrier and 10 ml of 10 M NaOH while swirling the solution. Heat and cool 5 minutes each then centrifuge the solution. Decant and discard the supernate. Dissolve the yttrium hydroxide precipitate in 2 ml of concentrated nitric acid. Add 5 ml each of water and 0.4 M oxalic acid, 1 drop of MCP indicator and concentrated ammonium hydroxide dropwise to the pH 1.5 endpoint, then 1 drop extra to where the yellow color appears, to precipitate yttrium oxalate. Heat and cool the solution as in 6.9 and filter the yttrium oxalate precipitate on a tared ~~Whatman 42~~ filter. Rinse the centrifuge tube with about 3 ml of yttrium wash solution. Wash the precipitate with 1 ml of water followed by 5 ml of acetone. Dry filter under heat lamp, weigh to determine yttrium yield and count to determine the Y-90 activity from which the Sr-90 activity is calculated. If yield <70%, see Laboratory Manager, or his designee. Record the time at the mid-point of the count as "T4". T4-T2 is the decay of Y-90.

Submitted by: James M. Gillard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cupier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000207

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-305, R0
Determination of Strontium-89,90 in Water Samples

Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/14/87 To: 8/11/87 Samples Affected All between 4-14-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James M. Willard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cuyler, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000208

9.0 CALCULATIONS

Sr-90 Activity (pCi/l) = ACT

$$= \frac{TYC - BKG}{2.22*CTIME*Y\text{-}YIELD*S\text{-}YIELD*Y\text{-}EFF*Y\text{-}GRD*Y\text{-}DEC*VOL}$$

Sr-90 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TYC}{(TYC - BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*CTIME*Y\text{-}YIELD*S\text{-}YIELD*Y\text{-}EFF*Y\text{-}GRD*Y\text{-}DEC*VOL}$$

TYC = Total yttrium counts
BKG = Background counts
CTIME = Couint time (min)
Y-YIELD = Recovery (chemical yield) of yttrium
S-YIELD = Recovery (chemical yield) of strontium
Y-EFF = Counter efficiency for counting yttrium
Y-GRD = Yttrium ingrowth (see appendix 10.2)
Y-DEC = Yttrium decay (see appendix 10.3)
VOL = Sample volume (l)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedures? Yes (X) No ( )

If yes, enter paragraph number to reference insertion point 5.2.15 5.2.18, 6.1 - 6.22

Procedure Title & Number (if exists) RSL 304, R0

Determination of Selected Alpha Emitting Isotopes

Description of experiment or method Reverse order of column to improve isotopic separation

Issue of RSL-304, R1

Time Period From: 9/24/87 To: ++ Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 9-24-87

I. Change steps

From	To
5.2.15	5.3.15
5.2.16	5.3.16
5.2.17	5.3.17
5.2.18	5.3.18
6.11	6.15
6.12	6.16
6.13	6.17
6.14	6.18
6.15	6.19
6.16	6.20
6.17	6.21
6.18	6.22
6.19	6.23
6.20	6.24
6.21	6.25
6.22	6.26

II. Delete old steps 6.1 through 6.10

III. Add new steps 6.1 through 6.14 see attached

Submitted by: James M. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. C. Crispin, Laboratory Director

Approved by: Vicky L. Hutton for C. Prince, QA/QC/Director, ITAS

000210

6.1 Fill out appropriate data sheets (see appendix 10.1). Evaporate sample to dryness. Add 5ml of concentrated HNO_3 and 100 µl of 30% H_2O_2. Evaporate to dryness. If only uranium analysis is requested, proceed to step 6.8

6.2 Dissolve residue in 20ml of 8M HNO_3. Heat as necessary to dissolve, then cool to room temperature. Centrifuge if necessary to eliminate insoluble material.

6.3 Add sample to DOWEX 1-X8 (100-200 mesh) anion column which has been preconditioned with 10ml of 8M HNO_3. Collect effluent (Am, U) in a 250ml beaker.

6.4 If only Am is required, wash column with 20ml of 8M HNO_3, otherwise, wash column with 60ml of 8M HNO_3, collecting in same beaker as previous step. Once through column, transfer beaker to low heat hotplate, add 1ml of 30% H_2O_2 and evaporate to dryness. For Americium only, proceed to step 6.8.

6.5 Elute Th with 30ml of 8M HCl. Collect in beaker. Once through column, evaporate to dryness on hot plate.

6.6 Elute Pu with 30ml of 8M HCl + 0.1N NH_4I solution. Collect in a 250ml beaker. Once through column, add 1ml of concentrated HNO_3, evaporate to dryness on hot plate.

6.7 Elute Np with 30ml of 6.5M HCl + 0.004M HF solution. Collect in Teflon beaker. Once through column, evaporate to dryness. Add 5ml of concentrated HCl and evaporate to dryness.

6.8 To the dried residue from step 6.4 or 6.1, add 5 ml of concentrated HCl. Evaporate to dryness.

6.9 Dissolve residue in 15 ml of 8M HCl. Heat as necessary to dissolve, then cool to room temperature.

6.10 Add sample to a DOWEX 1-X8 (100-200 mesh) anion column which has been preconditioned with 10 ml of 8M HCl. Wash column with 10 ml of 8M HCl after sample has passed through. Collect effluents in beaker for Am analysis. Evaporate to dryness.

6.11 Wash column with 25 ml of 8M HCl + 0.1 M NH_4I solution, followed by 25 ml of 6.5 M HCl + 0.004 M HF solution, followed by 5 ml of 6.5 M HCl. Discard all effluents in < pH2 acid waste container.

6.12 Elute U with 30 ml of 0.5 M HCl. Collect in beaker for U analysis. Evaporate to dryness.

6.13 For U, Th, Np or Pu analysis, proceed to step 6.21

6.14 Dissolve the dried Am residue in 5 ml of concentrated HNO_3 and evaporate to dryness. Repeat.

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (✓) No ( )
(If yes enter paragraph number to reference insertion point: 4.4.13, 5.2.1, 5.2.2, 6.15.6.20, 6.2.)

Procedure Title & Number (If exists) RSL-304, R0

Determination of Selected Alpha Emitting Isotopes

Description of experiment or method To improve Am,Cm yield and decontamination factors

Issue of RSL-304, R1

Time Period From: 12/9/87 To: -/-/-- Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

See attached.

Submitted by: James T. Harvey

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. G. Cuffin, Laboratory Director

Approved by: Paul Nutty, QA/QC/Director, ITAS

000213

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 1
	REVISION: 0	DATE: 4-06-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of uranium, thorium, neptunium, plutonium, americium and curium alpha emitting isotopes.

2.0 SCOPE

This procedure applies to all samples requiring uranium, thorium, neptunium, plutonium, americium and curium activity analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring gross alpha and/or gross beta activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Montioring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Ouality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual

4.1.6. ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "Radioisotope Techniques," R.T. Overman and H.M. Clark, McGraw-Hill Book Co., Inc., New York, 1960. (Chapter 6)

4.4.2 "The Radiochemisry of Uranium", J.E. Grindler, NAS-NS-3050, National Academy of Sciences, March 1962.

4.4.3 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water"; H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, August 1980.

4.4.4 "Standard Methods for the Examination of Water and Waste Water," APHA, AWWA, WPCF, American Public Health Association, Washington, D.C, latest edition.

4.4.5 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.6 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

4.4.7 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.8 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

4.4.9 "Radiometric Method for the Determination of Uranium in Water: Single Laboratory Evaluation and Interlaboatory Collaborative Study", C.T. Bishop, V.R. Casella, and A.A. Glosby, EPA-600/7-79-093, U.S. Environmental Protection Agency, April 1979.

4.4.10 "The Radiochemistry of Neptunium", G.A. Burney and R.M. Harbour, NAS-NS-3060, National Academy of Science, December 1974.

4.4.11 "The Radiochemistry of Plutonium", G.H. Coleman, NAS-NS-3058, National Academy of Science, September 1965.

4.4.12 "The Radiochemistry of Thorium", E.K. Hyde, NAS-NS-3004, National Academy of Science, January 1960.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Beaker, assorted sizes

5.1.2 Water bath with magnetic stirrers

5.1.3 100 ml glass centrifuge tubes

5.1.4 Plastic centrifuge tubes

5.1.5 Ion exchange columns

5.1.6 Analytical balance (0-160.0000g)

5.1.7 Assorted tools and routine laboratory equipment

5.2 Materials

5.2.1 Routine laboratory supplies

5.2.2 Anion exchange resin, Bio-Rad AG 1-X8, 100 - 200 mesh

5.2.3 Membrane filters, 0.8 μm pore size

5.3 Reagents

5.3.1 Hydrochloric Acid (HCl), concentrated, ACS reagent grade

5.3.2 Nitric Acid (HNO_3), concentrated, ACS reagent grade

5.3.3 Hydrofluoric Acid (HF), concentrated, ACS reagent grade

5.3.4 8 M Hydrochloric Acid (HCl): Add 667 ml of concentrated HCl to 333 ml of demineralized water. Mix thoroughly.

5.3.5 0.5 M Hydrochloric Acid (HCl): Add 42 ml of concentrated HCl to 958 ml of demineralized water. Mix thoroughly.

5.3.6 8 M Hydrochloric Acid (HCl) + 0.01 M Nitric Acid (HNO_3): To 1 liter of 8 M HCl add 1 ml of concentrated HNO_3. Mix thoroughly.

5.3.7 13 M Hydrofluoric Acid (HF): Add 450 ml of concentrated HF to 550 ml of demineralized water. Mix thoroughly.

5.3.8 1 M Nitric Acid (HNO_3): Add 65 ml of concentrated HNO_3 to 935 ml of demineralized water. Mix thoroughly.

5.3.9 Phosphoric Acid (H_3PO_4), concentrated, ACS reagent grade

5.3.10 8 M Nitric Acid (HNO_3): Add 500 ml of concentrated HNO_3 to 500 ml of demineralized water. Mix thoroughly.

5.3.11 8 M Hydrochloric Acid (HCl) + 0.1 M Ammonium Iodide (NH_4I): Dissolve 3 grams of ammonium iodide in 200 ml of 8 M HCl. Mix thoroughly.

5.3.12 Cerium Nitrate solution (200 µg/ml, Ce): Add 0.3 grams of cerium nitrate to 500 ml of demineralized water. Mix thoroughly.

5.3.13 2% Titanous Chloride ($TiCl_3$) solution: Add 10 ml of 20% titanous chloride solution to 90 ml of demineralized water. Mix thoroughly.

5.3.14 20% Titanous Chloride, ACS reagent grade

5.2.15 Bismuth Nitrate solution (100 mg/ml bismuth): Add 232 grams bismuth nitrate [$Bi(NO_3)_3.5H_2O$] to 1 liter of demineralized water. Mix thoroughly.

5.2.16 6.5 M Hydrochloric Acid (HCl): Add 540 ml of concentrated HCl to 460 ml of demineralized water. Mix thoroughly.

5.2.17 6.5 M Hydrochloric Acid (HCl) + 0.004 M Hydrofluoric Acid (HF): Add 1.5 ml of concentrated HF to 1 liter of 6.5 M HCl. Mix thoroughly.

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 5
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6.0 PROCEDURE

6.1 Fill out appropriate data sheets (see appendix 10.1). The dry sample is dissolved in 15 ml 8 M hydrochloric acid/0.01 M nitric acid. Apply to column which has been pre-equilibrated with 5 ml of the mixed acid at a flow rate of approximately 0.5 ml/minute. Collect effluent in 100 ml beaker.

6.2 Wash column with 35 ml 8 M hydrochloric acid, pool wash with effluent and evaporate solution to dryness. This fraction contains americium, curium and thorium.

6.3 Plutonium is then eluted from the column with 15 ml of 8 M hydrochloric acid + 0.1 M ammonium iodide. Wash column with 20 ml 8 M hydrochloric acid, pool with eluate, add 0.5 ml of concentrated HNO_3 and evaporate to dryness. Proceed to 6.17.

6.4 Neptunium is eluted from the column with 15 ml of 6.5 M HCl + 0.004 M HF followed by 20 ml of 6.5 HCl. Collect in teflon beaker. Evaporate to dryness. Using concentrated HCl, dissolve residue and transfer to glass beaker. Proceed to 6.17.

6.5 Uranium is eluted from the column with 35 ml 0.5 M hydrochloric acid and the eluant evaporated to dryness. Proceed to 6.17.

6.6 If americium, curium or thorium analysis is required, dissolve residue from 6.2 in 20 ml of 8 M nitric acid.

6.7 Pass over a small anion exchange resin column which has been pre-equilibrated with 5 ml of 8 M HNO_3 at a flow rate of approximately 0.5 ml per minute. Collect effluent for americium and curium analysis.

6.8 Wash the resin column with 30 ml of 8 M nitric acid collecting the effluent in same beaker used in 6.7. Evaporate to dryness slowly.

6.9 Elute the thorium from the column using 30 ml of 8 M hydrochloric acid, collecting the elutant in a 50 ml beaker and evaporate to dryness. Proceed to 6.17.

6.10 If americum and/or curium analysis is requested, the dry residue from 6.8 above is dissolved in 2 - 3 ml reagent grade nitric acid and the acid evaporated. Repeat this step.

6.11 Dissolve residue in 10 ml 1 M nitric acid and transfer with water to centrifuge tube. Total volume about 80 ml.

6.12 Place tube in hot water bath and stir mechnically.

6.13 Add 1 ml bismuth nitrate solution followed by 1 ml phosphoric acid. Stir for 1 hour.

6.14 Centrifuge and discard supernatant.

6.15 Transfer precipitate with water to 50 ml centrifuge tube.

6.16 Centrifuge and discard the supernatant.

6.17 The dried residue (or precipitate) is dissolved in 1 - 2 ml of reagent grade hydrochloric acid. Heat to effect dissolution of sample.

6.18 Transfer solution to tube, rinse out beaker with 10 ml water and pool into tube.

6.19 Add 1 ml cerium nitrate carrier.

6.20 Add 1 ml 2% titanous chloride (not necessary for americium and curium). Allow to stand for 5 minute in hot water bath.

6.21 Add 1 ml hydrofluoric acid, cool in ice water bath, then filter through membrane filter.

6.22 Mount for counting and submit to counting room. The yield determined by tracer shall be greater than 40%.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 15% at 1 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequte quality.

8.1.2 The technician performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.22 Chemical yield

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 The alpha spectroscopy system shall be calibrated before use.

8.4.2 Balances shall be checked to ensure calibration dates have not expired.

8.4.3 Balances shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

9.1 Alpha Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG) * SD}{SC * SVOL * 2.22 * IC}$$

Alpha Activity Error (pCi/l) = ERR

$$= ACT * \frac{TSC}{(TSC - BKG)^2} + \frac{SC}{(SC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{(4.65 * BKG + 3) * SD}{SC * 2.22 * SVOL * IC}$$

TSC = Sample counts in region of interest
BKG = Background counts in region of interest
SD = Standard dpm
SC = Standard counts
IC = Alpha branching correction; U-235 = 0.85; Other = 1.0
SVOL = Sample volume (l)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Selected Alpha Emitting Isotopes	NO: RSL-304	PAGE: 9
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APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

167 4 86

000222

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-305

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Strontium-89,90 in Water Samples

APPROVED: James T. Harvey DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Nielsen DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cuffin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

429-1-87

RSL-305, RO-CH1

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.2.1; 6.7; 6.10
Procedure Title & Number (If exists) RSL-305, RO
Determination of Strontium-89,90 in Water Samples

Description of experiment or method Retain barium chromate precipitate for radium analysis if required. Modify materials list to include Nylon 66 membrane filter.

Time Period From: 8/8/87 To: Issue of RSL-305, R1 Samples Affected All from 8-8-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-8-87

5.2.1 Nylon 66 membrane filter, 1.2 micron, 4.7 cm ~~Whatman 42 or glass fiber filter paper, 4.7 cm, as necessary~~

6.7 Add 5 ml of 1.5 M sodium chromate and heat the solution for 3 minutes. Add 1 drop of phenolphthalein then add glacial acetic acid dropwise until the color changes from reddish-orange to golden-yellow and add 1 ml of 6 M acetic acid. Heat 2 minutes, centrifuge, add an additional 0.5 ml of barium carrier. Gently swirl the solution to mix while the previous precipitate is disturbed as little as possible. Cool the solution for 2 minutes, centrifuge and ~~discard the precipitate~~ retain the precipitate for further analysis unless otherwise directed.

6.10 Precipitate the yttrium hydroxide from the saved strontium sulfate solution by adding 10 M NaOH to the purple end point of the MCP indicator and 2 drops extra. Add 1.25 ml of strontium carrier and 10 ml of 10 M NaOH while swirling the solution. Heat and cool 5 minutes each then centrifuge the solution. Decant and discard the supernate. Dissolve the yttrium hydroxide precipitate in 2 ml of concentrated nitric acid. Add 5 ml each of water and 0.4 M oxalic acid, 1 drop of MCP indicator and concentrated ammonium hydroxide dropwise to the pH 1.5 endpoint, then 1 drop extra to where the yellow color appears, to precipitate yttrium oxalate. Heat and cool the solution as in 6.9 and filter the yttrium oxalate precipitate on a tared ~~Whatman 42~~ filter. Rinse the centrifuge tube with about 3 ml of yttrium wash solution. Wash the precipitate with 1 ml of water followed by 5 ml of acetone. Dry filter under heat lamp, weigh to determine yttrium yield and count to determine the Y-90 activity from which the Sr-90 activity is calculated. If yield <70%, see Laboratory Manager, or his designee. Record the time at the mid-point of the count as "T4". T4-T2 is the decay of Y-90.

Submitted by: James M. Willard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cupier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000224

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-305, R0
Determination of Strontium-89,90 in Water Samples

Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/14/87 To: 8/11/87 Samples Affected All between 4-14-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James M. Dillard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cuffer, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of Strontium-89,90 in Water Samples	NO: RSI-305	PAGE: 8
	REVISION: 0	DATE: 4-14-87

9.0 CALCULATIONS

Sr-90 Activity (pCi/l) = ACT

$$= \frac{TYC - BKG}{2.22*CTIME*Y\text{-}YIELD*S\text{-}YIELD*Y\text{-}EFF*Y\text{-}GRD*Y\text{-}DEC*VOL}$$

Sr-90 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TYC}{(TYC - BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*CTIME*Y\text{-}YIELD*S\text{-}YIELD*Y\text{-}EFF*Y\text{-}GRD*Y\text{-}DEC*VOL}$$

TYC = Total yttrium counts
BKG = Background counts
CTIME = Couint time (min)
Y-YIELD = Recovery (chemical yield) of yttrium
S-YIELD = Recovery (chemical yield) of strontium
Y-EFF = Counter efficiency for counting yttrium
Y-GRD = Yttrium ingrowth (see appendix 10.2)
Y-DEC = Yttrium decay (see appendix 10.3)
VOL = Sample volume (l)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

Sr-89 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*CTIME*S\text{-}YIELD*S\text{-}EFF1*VOL} - \frac{ACT(90)*S\text{-}EFF2*(1+Y\text{-}GRD)}{S\text{-}EFF1} * \frac{1}{S\text{-}DEC}$$

SR-89 Activity Error (pCi/l) = ERR

$$= ACT * \left[\frac{TSC}{(TSC - BKG)^2} + \frac{TYC}{(TYC-BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*CTIME*S\text{-}YIELD*S\text{-}EFF*VOL}$$

TSC = Total strontium counts
BKG = Background counts
CTIME = Count time (min)
S-DEC = Decay correction for Sr-89 (See appendix 10.4)
S-YIELD = Recovery (chemical yield) of strontium
S-EFF1 = Counter efficiency for counting strontium-89
S-EFF2 = Counter efficiency for counting strontium-90
VOL = Sample volume (l)
Y-GRD = Yttrium ingrowth into final Sr fraction (See appendix 10.2)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Strontium-89,90 in Milk Samples	NO: RSL-306	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for Strontium-89,90 in milk samples.

2.0 SCOPE

This analytical procedure applies to all milk samples requiring Strontium-89,90 analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Strontium-89,90 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "Handbook of Radiochemical Analytical Methods," F.B. Johns, Editor, EPA 680/4-75-001, February 1975.

4.4.2 C. Porter, D. Ghill, R. Schneider, P. Robbins, W. Perry, and B. Kahn, Anal. Chem. 33, 1306 (1961).

4.4.3 C.R. Porter, B. Kahn, M. Carter, G.L. Rehnberg, and E.W. Pepper, Environ. Sci. Technol. 1, 745 (1967).

4.4.4 C.R. Porter and B. Kahn, Anal. Chem. 36, 676 (1964).

4.4.5 "The Radiochemistry of Barium, Calcium and Strontium," D.N. Sunderman and D.W. Townley, NAS-NS-3010, National Academy of Science, January 1960.

4.4.6 "Determination of Strontium-89 and -90 in Soil with Total Sample Decomposition," D.B. Martin, Analytical Chemistry, October 1979, 1968-1972.

4.4.7 Analytical Chemistry Branch Procedure Manual," IDO-12096, U.S. Department of Energy, Idaho Falls, Idaho, (1982).

4.4.8 "Nuclear and Radiochemistry", G. Freidlander, J.W. Kennedy, and J.M. Miller, John Wiley and Sons, New York, N.Y. (1964).

4.4.9 "Standard Methods for the Examination of Water and Waste Water," 11th Ed., Alpha, Inc., New York, N.Y., p. 448 (1960).

5.0 REQUIREMENTS

5.1 Equipment

None

5.2 Materials

5.2.1 Glass columns, 3.5 cm diameter containing 80 ml of cation exchange resin, DOWEX 50W-X8, 50 - 100 mesh

5.2.2 Plastic reservoirs

5.2.3 Common laboratory apparatus

5.3 Reagents

5.3.1 Acetic Acid (CH_3COOH)- Concentrated, ACS reagent

5.3.2 Ammonium Acetate Buffer, (pH 5.0) - Dissolve 200 grams Ammonium Acetate ($NH_4C_2H_3O_2$) in 750 ml demineralized water. Add 28.6 ml glacial acetic acid. Dilute to 1 liter with demineralized water and adjust the pH to 5.0.

5.3.3 EDTA Complexing Solution (Disodium Ethylenediaminetetraacetate) - Dissolve 216 grams of EDTA in 2300 ml of demineralized water. Add 15 ml of Sr++ carrier, 20 mg/ml, 20 ml of Ba++ carrier, 20 mg/ml, 200 ml of ammonium acetate buffer, and 70 ml of 6N NH_4OH. Adjust pH to 5.65, dilute to 3 liters with demineralized water, and re-check pH.

5.3.4 Disodium Ethylenediaminetetraacetate (EDTA-3%) - Dissolve 33.3 grams of EDTA in 900 ml of demineralized water. Adjust the pH to 5.2 with concentrated ammonium hydroxide and dilute to 1 liter with demineralized water.

5.3.5 1.5 M Sodium Chloride (NaCl) - Dissolve 88 grams of NaCl in 900 ml of demineralized water and dilute to 1 liter with demineralized water.

5.3.6 4 M Sodium Chloride - Dissolve 234 grams of NaCl in 800 ml of demineralized water and dilute to 1 liter with demineralized water.

6.0 PROCEDURE

6.1 Record the sample information on the appropriate data sheet. See appendix 10.1

6.2 Add 300 ml of EDTA complexing solution to one liter of milk. Adjust the pH to 5.2 with ammonium acetate buffer and mix well.

6.3 Pour the resulting milk solution into the reservoir and allow the milk to flow through the column by gravity with the column wide open. When all the milk has passed through the funnel, add approximately 200 ml of demineralized water to the reservoir and let it flow through the resin columns until just enough water remains to cover the resin.

6.4 Add 800 ml of 3% EDTA to the reservoir and let it flow at approximately 20 ml per minute.

6.5 Add 200 ml of demineralized water to the reservoir and let it flow through the column at the same rate as above.

6.6 Wash absorbed EDTA from the column by passing 200 ml of 1.5 M NaCl through the column at approximately 10 ml per minute.

6.7 Elute strontium with 400 ml 4 M NaCl at a flow rate of approximately 20 ml/min.

6.8 Proceed to section 6.0 of method for the determination of Strontium-89,90 in water, (RSL-305) and follow procedure through, except do not add additional carriers.

7.0 PRECISION AND ACCURACY

7.1 For strontium-89, the standard deviation for a single determination is 5% at 100 pCi/l.

7.2 For strontium-90, the standard deviation for a single determination is 5% at 30 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balance shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Determination of Strontium-89,90 in Milk Samples	NO: RSL-306	PAGE: 6
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-307

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of I-131 Activity in Milk and Water Samples

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Meier DATE: 5/4/87
ITAS QA Director

CONCURRED: John L. Anderson DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-307, RO
Determination of I-131 Activity in Milk and Water Samples

Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/16/87 To: 8/11/87 Samples Affected All between 4-6-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James Hallard

Approved by: John R. Frogin, Laboratory Manager

Approved by: John L. Lupin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 7
	REVISION: 0	DATE: 4-06-87

9.0 CALCULATIONS

131-Iodine Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG)}{CTIME*2.22*YLD*EFF*DF*SVOL}$$

131-Iodine Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{(TSC - BKG)^2} + 0.0025 \right]^{1/2}$$

Lower Limits of Detection (pCi/l) = LLD

$$= \frac{\left[4.65 * (BKG)^{1/2} + 3\right]}{CTIME*2.22*YLD*EFF*DF*SVOL}$$

TSC = Total sample counts
BKG = Background counts
CTIME = Count time (min)
YLD = Chemical yield
EFF = Counting efficiency
DF = Decay factor (see appendix 10.2)
SVOL = Sample volume (L)
2.22 = Conversion from dpm to pCi
4.65,3 = Constants
0.0025 = Estimates of relative error from all other sources.

10.0 APPENDICES

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.1.9

Procedure Title & Number (If exists) RSL-307, R0
Determination of I-131 Activity in Milk and Water Samples

Description of experiment or method Temporary change to utilize available equipment

Time Period From: 8/13/87 To: ~~TT~~ Issue of RSL-307, R1 Samples Affected All until revision of RSL-307

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-13-87

5.1.9 Ion exchange column (~~3~~ 2.3 cm diameter X ~~17~~ 30 cm), 100ml of resin

Submitted by: James Moreillard

Approved by: John R Frazier, Laboratory Manager

Approved by: John A. Cuyler, Laboratory Director

Approved by: Cheryl A. Prince, QA/QC/Director, ITAS

RSL-307, R0, CH-3

page 1 of 1

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3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (x) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.2, 6.26

Procedure Title & Number (If exists) RSL-307, R0

DETERMINATION OF I-131 ACTIVITY IN MILK AND WATER SAMPLES

Description of experiment or method Procedure clarification and modification to allow for use of currently available reagent

Time Period From: 8/19/87 To: Issue of RSL-307, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

5.3.2 ~~Ethyl Alcohol~~ Ethanol (CH_3CH_2OH), ~~95 Percent~~ 89% ACS reagent grade

6.26 Filter with weak vacuum through a tared filter paper (Whatman 42). Wash the centrifuge tube and filtering apparatus with demineralized water followed by ~~ethyl alcohol.~~ ethanol

Submitted by: James A. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Fragier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000238

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.8, 6.3, 6.6, 6.7, 6.15, 6.20

Procedure Title & Number (If exists) RSL-307.RD

DETERMINATION OF I-131 ACTIVITY IN MILK AND WATER SAMPLES

Description of experiment or method PROCEDURE CLARIFICATION AND ADDITION OF CAUTION ABOUT THE NITRIC ACID/SODIUM HYPOCHLORITE REACTION

Issue of RSL-307, R1

Time Period From: 8/28/87 To: ~~/ /~~ Samples Affected All from 8-28-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

5.3.8 1 M ~~Sodium Bisulfite (Na_2HSO_3)~~ Sodium meta-Bisulfite ($Na_2S_2O_5$): Dissolve approximately 2.9 grams of ~~Na_2HSO_3~~ $Na_2S_2O_5$ anydrous powder in demineralized water and dilute to 30 ml. Make fresh daily.

6.4 Wash the resin with ~~three~~ successive 100 ml batches of hot water until solution is clear. Stir manually, allow the resin to settle to the bottom of the beaker. Decant and discard the wash water.

6.6 Add a fresh 125 ml of NaOCl solution to the resin remaining in the beaker and stir for another 10 minutes. Allow the resin to settle and filter the NaOCl solution into the Buchner funnel.
CAUTION: The following step MUST be performed in a fume hood. The nitric acid/sodium hypochlorite reaction is vigorous, therefore, acid additions MUST be made slowly.

6.7 In the fume hood, slowly add 20 ml of concentrated HNO_3 to the NaOCl solution from the previous step. Stir until the bulk of the chlorine gas has been evolved from the solution.

Submitted by: James Mullard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Zaci, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

6.15 Add 25 ml of demineralized water and 1 ml of 1 M sodium ~~bisulfite~~ meta-bisulfite. Shake until the organic layer is colorless.

6.20 Add 20 ml of demineralized water and 1 ml of 1 M sodium ~~bisulfite~~ meta-bisulfite. Shake for approximately 30 seconds. Collect the bottom aqueous layer in a 100-ml beaker containing a small magnetic stirring bar.

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 1
	REVISION: 0	DATE: 4-05-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Iodine-131 activity in milk and water samples.

2.0 SCOPE

The procedure applies to all samples of milk and water requiring Iodine-131 activity analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Iodine-131 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 J.J. Gabay, C.J. Paperiello, S. Goodyear, J.C. Daly, and J.M. Matuszek, Health Physics 26, 89 (1974).

4.4.2 D.E. McCurdy, R.A. Mellen, R.W. Lambdin, and M.E. McLain, "The Use of Cuprous Iodine as a Precipitation Matrix in the Radiochemical Determination of I-131 in Milk," ASTM Conference on Effluent and Environmental Radiation Surveillance, Johnson, Vermont, July 9-14, 1978.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0-160.0000 gram capacity)

5.1.2 Centrifuge

5.1.3 Desiccator

5.1.4 Vortex mixer

5.1.5 Hot plate

5.1.6 Hot water bath

5.1.7 Stirrers

5.1.8 Separatory funnels

PROCEDURE DEVELOPMENT/CHANGE

3223

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.15; 5.3.22; 6.8
Procedure Title & Number (If exists) ASL-305, A0
Determination of Strontium-89,90 in Water Samples
Description of experiment or method Temporary change to utilize present stock of reagents

Time Period From: 8/13/87 To: ~~TT~~ until present stock utilized Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-13-87

5.3.15 1M Sodium Carbonate Solution ($Na_2CO_3 \cdot 10H_2O$) (~~Na_2CO_3~~): Dissolve 286 ~~106~~ grams of sodium carbonate in water and dilute to 1 liter. Mix thoroughly.

5.3.22 Yttrium Nitrate Carrier Solution (5 ~~10~~ mg/ml, Y): Dissolve 21.5 ~~43~~ grams of yttrium nitrate [$Y(NO_3)_3 \cdot 6H_2O$] in a mixture of 2 ml of concentrated nitric acid and 700 ml of water and dilute to 1 liter with water. Mix thoroughly on hot plate on low setting until dissolved.

6.8 Add 10 ml of 0.7 M sodium sulfate and 3 ml of glacial acetic acid to the barium chromate supernate to precipitate strontium sulfate. Record the time "T1" as the start of the growth of Y-90. Heat the solution for 5 minutes and cool. Centrifuge the solution, decant and discard the supernate. (Note: If Sr-90 results are required immediately, mount strontium sulfate as described in 6.9 below and calculate as directed by the Laboratory Manager.) Dissolve the strontium sulfate in 5 ml of 0.2 M DTPA with heating and stirring. Add ~~1~~ 2 ml of yttrium carrier. Add 1 drop of phenolphthalein indicator to make the solution slightly alkaline (light pink in color). Cover the tube, and store the sample for 4 to 14 days.

Submitted by: James [illegible]

Approved by: John R. Fragia, Laboratory Manager

Approved by: John A. Cuyler, Laboratory Director

Approved by: Sheryl A. Prince, QA/QC/Director, ITAS

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (x) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-305, R0

DETERMINATION OF STRONTIUM-89,90 IN WATER SAMPLES

Description of experiment or method CORRECT equation for Lower Limit of Detection. Clarify use of times T_0, T_2, and T_3. (NOTE: This change revision incorporates CH-2, page 3)

Time Period From: 8/17/87 To: Issue of RSL-305, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

See attachment

Submitted by: James [illegible]

Approved by: James T. Hanley, Laboratory Manager

Approved by: John P. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of Strontium-89,90 in Water Samples	NO: RSL-305	PAGE: 9
	REVISION: 0	DATE: 4-14-87

Sr-89 Activity (pCi/l) = ACT

$$= \left[\frac{TSC - BKG}{2.22*CTIME*S\text{-}YIELD*S\text{-}EFF1*VOL} - \frac{ACT(90)*S\text{-}EFF2*(1+Y\text{-}GRD)}{S\text{-}EFF1}\right] * \frac{1}{S\text{-}DEC}$$

SR-89 Activity Error (pCi/l) = ERR

$$= ACT * \left[\frac{TSC}{(TSC - BKG)^2} + \frac{TYC}{(TYC-BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* (BKG)^{1/2} + 3}{2.22*CTIME*S\text{-}YIELD*S\text{-}EFF1*VOL * S\text{-}DEC}$$

TSC = Total strontium counts
BKG = Background counts
CTIME = Count time (min)
S-DEC = Decay correction for Sr-89 (See appendix 10.4) $T_3 - T_0$
S-YIELD = Recovery (chemical yield) of strontium
S-EFF1 = Counter efficiency for counting strontium-89
S-EFF2 = Counter efficiency for counting strontium-90
VOL = Sample volume (l)
Y-GRD = Yttrium ingrowth into final Sr fraction (See appendix 10.2) $T_3 - T_2$
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources
T_0 = sample collection date/time

10.0 APPENDICES

RSL 305 R0
page 1 of 2

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0, 10.2, 10.3, 10.4

Procedure Title & Number (If exists) RSL-305, R0

DETERMINATION OF STRONTIUM-89,90 IN WATER SAMPLES

Description of experiment or method To clarify use of Appendices

(Note: This change revision incorporates CH-4, page 2)

Issue of RSL-305, R1

Time Period From: 8/28/87 To: -/-/- Samples Affected All from 8-28-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

See attachment

Submitted by: James A. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John A. Anfinson, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000246

TITLE: Determination of Strontium-89,90 in Water Samples	NO: RSL-305	PAGE: 9
	REVISION: 0	DATE: 4-14-87

Sr-89 Activity (pCi/l) = ACT

$$= \left[\frac{TSC - BKG}{2.22 \cdot CTIME \cdot S\text{-}YIELD \cdot S\text{-}EFF1 \cdot VOL} - \frac{ACT(90) \cdot S\text{-}EFF2 \cdot (1 + Y\text{-}GRD)}{S\text{-}EFF1}\right] \cdot \frac{1}{S\text{-}DEC}$$

SR-89 Activity Error (pCi/l) = ERR

$$= ACT \cdot \left[\frac{TSC}{(TSC - BKG)^2} + \frac{TYC}{(TYC - BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65 \cdot (BKG)^{1/2} + 3}{2.22 \cdot CTIME \cdot S\text{-}YIELD \cdot S\text{-}EFF \cdot VOL \cdot S\text{-}DEC}$$

TSC = Total strontium counts
BKG = Background counts
CTIME = Count time (min)
S-DEC = Decay correction for Sr-89 (See appendix 10.4) $T_3 - T_0$
S-YIELD = Recovery (chemical yield) of strontium
S-EFF1 = Counter efficiency for counting strontium-89
S-EFF2 = Counter efficiency for counting strontium-90
VOL = Sample volume (l)
Y-GRD = Yttrium ingrowth into final Sr fraction (See appendix 10.2) $T_3 - T_2$
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources
T_0 = sample collection date/time

10.0 APPENDICES

APPENDIX 10.2

Yttrium Ingrowth

t(days)	y-grd 1-e-λt	t(days)	y-grd 1-e-λt	t(days)	y-grd 1-e-λt
0.00	.0000	9.00	.9029	18.00	.9906
0.25	.0627	9.25	.9090	18.25	.9912
0.50	.1215	9.50	.9147	18.50	.9917
0.75	.1766	9.75	.9201	18.75	.9922
1.00	.2283	10.00	.9251	19.00	.9927
1.25	.2767	10.25	.9298	19.25	.9932
1.50	.3221	10.50	.9342	19.50	.9936
1.75	.3646	10.75	.9384	19.75	.9940
2.00	.4045	11.00	.9422	20.00	.9944
2.25	.4418	11.25	.9458	20.25	.9948
2.50	.4768	11.50	.9492	20.50	.9951
2.75	.5097	11.75	.9524	20.75	.9954
3.00	.5404	12.00	.9554	21.00	.9957
3.25	.5692	12.25	.9582	21.25	.9959
3.50	.5963	12.50	.9608	21.50	.9962
3.75	.6216	12.75	.9633	21.75	.9964
4.00	.6453	13.00	.9656	22.00	.9967
4.25	.6676	13.25	.9678	22.25	.9969
4.50	.6884	13.50	.9697	22.50	.9971
4.75	.7080	13.75	.9716	22.75	.9973
5.00	.7263	14.00	.9734	23.00	.9974
5.25	.7435	14.25	.9751	23.25	.9976
5.50	.7596	14.50	.9766	23.50	.9977
5.75	.7746	14.75	.9781	23.75	.9979
6.00	.7888	15.00	.9795	24.00	.9980
6.25	.8020	15.25	.9808	24.25	.9981
6.50	.8145	15.50	.9820	24.50	.9982
6.75	.8261	15.75	.9831	24.75	.9984
7.00	.8370	16.00	.9842	25.00	.9985
7.25	.8472	16.25	.9852	25.25	.9986
7.50	.8568	16.50	.9861	25.50	.9987
7.75	.8658	16.75	.9870	25.75	.9987
8.00	.8742	17.00	.9878	26.00	.9988
8.25	.8820	17.25	.9886	26.25	.9989
8.50	.8896	17.50	.9893	26.50	.9990
8.75	.8864	17.75	.9900	26.75	.9990
				27.00	.9991

APPENDIX 10.3

Yttrium decay and Yttrium Ingrowth

t(hr)	Y-dec $e^{-\lambda t}$	Y-grd $1-e^{-\lambda t}$	t(hr)	Y-dec $e^{-\lambda t}$	Y-grd $1-e^{-\lambda t}$	t(hr)	Y-dec $e^{-\lambda t}$	Y-grd $1-e^{-\lambda t}$
0.0	1.0000	.0000	22.5	.7843	.2157	45.0	.6151	.3849
0.5	.9940	.0054	23.0	.7801	.2199	45.5	.6118	.3882
1.0	.9893	.0107	23.5	.7759	.2241	46.0	.6085	.3915
1.5	.9839	.0161	24.0	.7717	.2283	46.5	.6053	.3947
2.0	.9786	.0214	24.5	.7676	.2324	47.0	.6020	.3980
2.5	.9734	.0266	25.0	.7634	.2366	47.5	.5988	.4012
3.0	.9681	.0319	25.5	.7593	.2407	48.0	.5955	.4045
3.5	.9629	.0371	26.0	.7552	.2448	48.5	.5923	.4077
4.0	.9577	.0423	26.5	.7512	.2488	49.0	.5891	.4109
4.5	.9526	.0474	27.0	.7471	.2529	49.5	.5860	.4140
5.0	.9474	.0526	27.5	.7431	.2569	50.0	.5828	.4172
5.5	.9423	.0577	28.0	.7391	.2609	50.5	.5797	.4203
6.0	.9373	.0627	28.5	.7351	.2549	51.0	.5766	.4234
6.5	.9322	.0678	29.0	.7311	.2689	51.5	.5735	.4265
7.0	.9272	.0728	29.5	.7272	.2728	52.0	.5704	.4296
7.5	.9222	.0778	30.0	.7233	.2767	52.5	.5673	.4327
8.0	.9172	.0828	30.5	.7194	.2806	53.0	.5642	.4358
8.5	.9123	.0877	31.0	.7155	.2845	53.5	.5612	.4388
9.0	.9074	.0926	31.5	.7117	.2883	54.0	.5582	.4418
9.5	.9025	.0975	32.0	.7078	.2922	54.5	.5552	.4448
10.0	.8976	.1024	32.5	.7040	.2960	55.0	.5522	.4478
10.5	.8928	.1072	33.0	.7002	.2998	55.5	.5492	.4508
11.0	.8880	.1120	33.5	.6965	.3035	56.0	.5462	.4538
11.5	.8832	.1168	34.0	.6927	.3073	56.5	.5433	.4567
12.0	.8785	.1215	34.5	.6890	.3110	57.0	.5404	.4596
12.5	.8737	.1263	35.0	.6853	.3147	57.5	.5375	.4625
13.0	.8690	.1310	35.5	.6816	.3184	58.0	.5346	.4654
13.5	.8644	.1356	36.0	.6779	.3221	58.5	.5317	.4683
14.0	.8597	.1403	36.5	.6743	.3257	59.0	.5288	.4712
14.5	.8551	.1449	37.0	.6706	.3294	59.5	.5260	.4740
15.0	.8505	.1495	37.5	.6670	.3330	60.0	.5232	.4768
15.5	.8459	.1541	38.0	.6634	.3366	60.5	.5203	.4797
16.0	.8413	.1587	38.5	.6599	.3401	61.0	.5175	.4825
16.5	.8468	.1632	39.0	.6563	.3437	61.5	.5148	.4852
17.0	.8323	.1677	39.5	.5428	.3472	62.0	.5092	.4880
17.5	.8278	.1722	40.0	.6493	.3507	62.5	.5092	.4908
18.0	.8234	.1766	40.5	.6458	.3542	63.0	.5065	.4935
18.5	.8189	.1811	41.0	.6423	.3577	64.0	.5010	.4990
19.0	.8145	.1855	41.5	.6388	.3612	65.0	.4957	.5043
19.5	.8101	.1899	42.0	.6354	.3646	66.0	.4903	.5097
20.0	.8058	.1942	42.5	.6320	.3680	67.0	.4851	.5149
20.5	.8014	.1986	43.0	.6286	.3714	68.0	.4799	.5201
21.0	.7971	.2029	43.5	.6252	.3748	69.0	.4747	.5253
21.5	.7928	.2072	44.0	.6219	.3781	70.0	.4696	.5304
22.0	.7885	.2115	44.5	.6185	.3815	71.0	.4646	.5354

APPENDIX 1054

Strontium Decay

t(days)	S-dec $e^{-\lambda t}$	t(days)	S-dec $e^{-\lambda t}$	t(days)	S-dec $e^{-\lambda t}$
0.0	1.0000	20.0	.7620	40.0	.5808
0.5	.9932	20.5	.7569	40.5	.5769
1.0	.9865	21.0	.7518	41.0	.5730
1.5	.9798	21.5	.7568	41.5	.5690
2.0	.9732	22.0	.7416	42.0	.5652
2.5	.9668	22.5	.7366	42.5	.5613
3.0	.9601	23.0	.7317	43.0	.5575
3.5	.9536	23.5	.7267	43.5	.5539
4.0	.9471	24.0	.7218	44.0	.5500
4.5	.9407	24.5	.7169	44.5	.5462
5.0	.9344	25.0	.7120	45.0	.5427
5.5	.9280	25.5	.7072	45.5	.5380
6.0	.9217	26.0	.7023	46.0	.5352
6.5	.9155	26.5	.6977	46.5	.5318
7.0	.9093	27.0	.6930	47.0	.5280
7.5	.9031	27.5	.6882	47.5	.5245
8.0	.8970	28.0	.6836	48.0	.5210
8.5	.8909	28.5	.6790	48.5	.5175
9.0	.8849	29.0	.6742	49.0	.5140
9.5	.8789	29.5	.6699	49.5	.5105
10.0	.8729	30.0	.6651	50.0	.5070
10.5	.8670	30.5	.6608	50.5	.5035
11.0	.8612	31.0	.6562	51.0	.5000
11.5	.8553	31.5	.6519	51.5	.4967
12.0	.8495	32.0	.6473	52.0	.4933
12.5	.8438	32.5	.6430	52.5	.4900
13.0	.8381	33.0	.6388	53.0	.4868
13.5	.8324	33.5	.6342	53.5	.4834
14.0	.8268	34.0	.6300	54.0	.4801
14.5	.8212	34.5	.6259	54.5	.4769
15.0	.8156	35.0	.6215	55.0	.4734
15.5	.8101	35.5	.6172	55.5	.4702
16.0	.8046	36.0	.6131	56.0	.4671
16.5	.7991	36.5	.6090	56.5	.4640
17.0	.7938	37.0	.6050	57.0	.4608
17.5	.7883	37.5	.6009	57.5	.4578
18.0	.7881	38.0	.5968	58.0	.4547
18.5	.7778	38.5	.5928	58.5	.4513
19.0	.7725	39.0	.5888	59.0	.4484
19.5	.7672	39.5	.5848	59.5	.4454

RSL 305, R0 CHG
Page 1 of 1

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.4

Procedure Title & Number (If exists): RSL-305, R0

Determination of Strontium-89,90 in Water Samples

Description of experiment or method: Add Caution for addition of fuming nitric acid to sample

Time Period From: 9/28/87 To: Issue of RSL-305, R1 Samples Affected: All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 9-28-87

Add the following between steps 6.4 & 6.5:

Caution: If the precipitate is not a compact fine precipitate and is in excess of normally expected amounts, contact the Laboratory Manager. Proceed only under direction of the Laboratory Manager.

Caution: The following step must be performed in a fume hood designated for strong oxidizing acids. Cautiously add fuming nitric acid by Pasteur pipet.

Submitted by: [signature]

Approved by: [signature], Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature], QA/QC/Director, ITAS

000251

TITLE: Determination of Strontium-89,90 in Water Samples	NO: RSL-305	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Strontium-89,90 in water.

2.0 SCOPE

The procedure applies to all water samples requiring Strontium-89,90 analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all water samples requiring Strontium-89,90 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6. ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "The Radiochemistry of Barium, Calcium and Strontium," D.N. Sunderman and D.W. Townley, NAS-NS-3010, National Academy of Science, January 1960.

4.4.2 Martin, D.B., "Determination of Strontium-89 and -90 in Soil with Total Sample Decomposition," Analytical Chemistry, October 1979. 1968 -1972.

4.4.3 Analytical Chemistry Branch Procedure Manual," IDO-12096, U.S. Department of Energy, Idaho Falls, Idaho, (1982).

4.4.4 "Nuclear and Radiochemistry", G. Freidlander, J.W. Kennedy, and J.M. Miller, John Wiley and Sons, New York, N.Y. (1964).

4.4.5 "Standard Methods for the Examination of Water and Waste Water," 11th Ed., Alpha, Inc., New York, N.Y., p. 448 (1960).

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Beta particle counter, low background proportional counter

5.1.2 Hot plate

5.1.3 Centrifuge

5.1.4 Analytical balance (0-160.0000 gram capacity)

5.2 Materials

5.2.1 Whatman 42 or glass fiber filter paper, 4.7 cm, as necessary

5.2.2 General laboratory supplies

5.3 Reagents

5.3.1 Acetic Acid (CH_3COOH), glacial (Sp. Gr. 1.05)

5.3.2 6 M, Acetic Acid: Mix 1 volume acetic acid, glacial with 1.9 volumes of water. Mix thoroughly.

5.3.3 Acetic Acid + Sodium Sulfate (Na_2SO_4) Wash Solution: Dissolve 50 g of sodium sulfate in 800 ml of demineralized water. Add 100 ml of glacial acetic acid and dilute to 1 liter with demineralized water. Mix thoroughly.

5.3.4 Acetone [$(CH_3)_2CO$], ACS reagent grade

5.3.5 Ammonium Hydroxide (NH_4OH), concentrated

5.3.6 Barium Nitrate Carrier Solution (10 mg/ml, Ba): Dissolve 19 grams of barium nitrate in 500 ml water. Add 5 ml concentrated nitric acid and dilute to 1 liter with demineralized water. Mix thoroughly.

5.3.7 Hydrochloric Acid (HCl), concentrated

5.3.8 6 M Hydrochloric Acid: Mix 1 volume of concentrated hydrochloric acid with 1.04 volumes of water. Mix thoroughly.

5.3.9 0.1% Metacresol purple (MCP) indicator: Dissolve 0.25 grams of m-cresolsulfonephthalein in water and add 2 ml of 5 N NaOH. Dilute to 250 ml with water. Mix thoroughly.

5.3.10 Nitric Acid (HNO_3), 90% fuming

5.3.11 Nitric Acid, concentrated nitric acid

5.3.12 4 M Nitric Acid: Mix 1 volume of concentrated nitric acid with 4.2 volumes of water. Mix thoroughly.

5.3.13 0.4 M Oxalic Acid ($H_2C_2O_4$) Solution: Dissolve 52.02 grams of oxalic acid in water and dilute to 1 liter. Mix thoroughly.

5.3.14 1% Phenolphthalein Indicator: Dissolve 2.5 grams of phenolphthalein in 125 ml of ethyl alcohol and dilute to 250 ml with water. Mix thoroughly.

TITLE: Determination of Strontium-89,90 in Milk Samples	NO: RSL-306	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for Strontium-89,90 in milk samples.

2.0 SCOPE

This analytical procedure applies to all milk samples requiring Strontium-89,90 analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Strontium-89,90 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "Handbook of Radiochemical Analytical Methods," F.B. Johns, Editor, EPA 680/4-75-001, February 1975.

4.4.2 C. Porter, D. Ghill, R. Schneider, P. Robbins, W. Perry, and B. Kahn, Anal. Chem. 33, 1306 (1961).

4.4.3 C.R. Porter, B. Kahn, M. Carter, G.L. Rehnberg, and E.W. Pepper, Environ. Sci. Technol. 1, 745 (1967).

4.4.4 C.R. Porter and B. Kahn, Anal. Chem. 36, 676 (1964).

4.4.5 "The Radiochemistry of Barium, Calcium and Strontium," D.N. Sunderman and D.W. Townley, NAS-NS-3010, National Academy of Science, January 1960.

4.4.6 "Determination of Strontium-89 and -90 in Soil with Total Sample Decomposition," D.B. Martin, Analytical Chemistry, October 1979, 1968-1972.

4.4.7 Analytical Chemistry Branch Procedure Manual," IDO-12096, U.S. Department of Energy, Idaho Falls, Idaho, (1982).

4.4.8 "Nuclear and Radiochemistry", G. Freidlander, J.W. Kennedy, and J.M. Miller, John Wiley and Sons, New York, N.Y. (1964).

4.4.9 "Standard Methods for the Examination of Water and Waste Water," 11th Ed., Alpha, Inc., New York, N.Y., p. 448 (1960).

5.0 REQUIREMENTS

5.1 Equipment

None

5.2 Materials

5.2.1 Glass columns, 3.5 cm diameter containing 80 ml of cation exchange resin, DOWEX 50W-X8, 50 - 100 mesh

5.2.2 Plastic reservoirs

5.2.3 Common laboratory apparatus

5.3 Reagents

5.3.1 Acetic Acid (CH_3COOH)- Concentrated, ACS reagent

5.3.2 Ammonium Acetate Buffer, (pH 5.0) - Dissolve 200 grams Ammonium Acetate ($NH_4C_2H_3O_2$) in 750 ml demineralized water. Add 28.6 ml glacial acetic acid. Dilute to 1 liter with demineralized water and adjust the pH to 5.0.

5.3.3 EDTA Complexing Solution (Disodium Ethylenediaminetetraacetate) - Dissolve 216 grams of EDTA in 2300 ml of demineralized water. Add 15 ml of Sr++ carrier, 20 mg/ml, 20 ml of Ba++ carrier, 20 mg/ml, 200 ml of ammonium acetate buffer, and 70 ml of 6N NH_4OH. Adjust pH to 5.65, dilute to 3 liters with demineralized water, and re-check pH.

5.3.4 Disodium Ethylenediaminetetraacetate (EDTA-3%) - Dissolve 33.3 grams of EDTA in 900 ml of demineralized water. Adjust the pH to 5.2 with concentrated ammonium hydroxide and dilute to 1 liter with demineralized water.

5.3.5 1.5 M Sodium Chloride (NaCl) - Dissolve 88 grams of NaCl in 900 ml of demineralized water and dilute to 1 liter with demineralized water.

5.3.6 4 M Sodium Chloride - Dissolve 234 grams of NaCl in 800 ml of demineralized water and dilute to 1 liter with demineralized water.

6.0 PROCEDURE

6.1 Record the sample information on the appropriate data sheet. See appendix 10.1

6.2 Add 300 ml of EDTA complexing solution to one liter of milk. Adjust the pH to 5.2 with ammonium acetate buffer and mix well.

6.3 Pour the resulting milk solution into the reservoir and allow the milk to flow through the column by gravity with the column wide open. When all the milk has passed through the funnel, add approximately 200 ml of demineralized water to the reservoir and let it flow through the resin columns until just enough water remains to cover the resin.

6.4 Add 800 ml of 3% EDTA to the reservoir and let it flow at approximately 20 ml per minute.

6.5 Add 200 ml of demineralized water to the reservoir and let it flow through the column at the same rate as above.

6.6 Wash absorbed EDTA from the column by passing 200 ml of 1.5 M NaCl through the column at approximately 10 ml per minute.

6.7 Elute strontium with 400 ml 4 M NaCl at a flow rate of approximately 20 ml/min.

6.8 Proceed to section 6.0 of method for the determination of Strontium-89,90 in water, (RSL-305) and follow procedure through, except do not add additional carriers.

7.0 PRECISION AND ACCURACY

7.1 For strontium-89, the standard deviation for a single determination is 5% at 100 pCi/l.

7.2 For strontium-90, the standard deviation for a single determination is 5% at 30 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balance shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Determination of Strontium-89,90 in Milk Samples	NO: RSL-306	PAGE: 6
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-307

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of I-131 Activity in Milk and Water Samples

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Nieto DATE: 5/4/87
ITAS QA Director

CONCURRED: John L. Cuyler DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-307, RO
Determination of I-131 Activity in Milk and Water Samples

Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/16/87 To: 8/11/87 Samples Affected All between 4-6-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

See attached

Submitted by: James Wheelland

Approved by: John R. Frogier, Laboratory Manager

Approved by: John L. Lupin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 7
	REVISION: 0	DATE: 4-06-87

9.0 CALCULATIONS

131-Iodine Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG)}{CTIME*2.22*YLD*EFF*DF*SVOL}$$

131-Iodine Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{(TSC - BKG)^2} + 0.0025 \right]^{1/2}$$

Lower Limits of Detection (pCi/l) = LLD

$$= \frac{\left[4.65 * (BKG)^{1/2} + 3\right]}{CTIME*2.22*YLD*EFF*DF*SVOL}$$

TSC = Total sample counts
BKG = Background counts
CTIME = Count time (min)
YLD = Chemical yield
EFF = Counting efficiency
DF = Decay factor (see appendix 10.2)
SVOL = Sample volume (L)
2.22 = Conversion from dpm to pCi
4.65,3 = Constants
0.0025 = Estimates of relative error from all other sources.

10.0 APPENDICES

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.1.9

Procedure Title & Number (If exists) RSL-307, R0
Determination of I-131 Activity in Milk and Water Samples

Description of experiment or method Temporary change to utilize available equipment

Time Period From: 8/13/87 To: ~~11~~ Issue of RSL-307, R1 Samples Affected All until revision of RSL-307

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-13-87

5.1.9 Ion exchange column (~~3~~ 2.3 cm diameter X ~~17~~ 30 cm), 100ml of resin

Submitted by: James Hoillard

Approved by: John R Frazier, Laboratory Manager

Approved by: John A. Cuyler, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (x) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.2, 6.26

Procedure Title & Number (If exists) RSL-307, R0

DETERMINATION OF I-131 ACTIVITY IN MILK AND WATER SAMPLES

Description of experiment or method Procedure clarification and modification to allow for use of currently available reagent

Time Period From: 8/19/87 To: Issue of RSL-307, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

5.3.2 ~~Ethyl Alcohol~~ Ethanol (CH_3CH_2OH), ~~95 Percent~~ 89% ACS reagent grade

6.26 Filter with weak vacuum through a tared filter paper (Whatman 42). Wash the centrifuge tube and filtering apparatus with demineralized water followed by ~~ethyl alcohol.~~ ethanol

Submitted by: James A. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Fragier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.8, 6.4, 6.6, 6.7, 6.15, 6.20

Procedure Title & Number (If exists) RSL-307, R0

DETERMINATION OF I-131 ACTIVITY IN MILK AND WATER SAMPLES

Description of experiment or method PROCEDURE CLARIFICATION AND ADDITION OF CAUTION ABOUT THE NITRIC ACID/SODIUM HYPOCHLORITE REACTION

Time Period From: 8/28/87 To: Issue of RSL-307, R1 Samples Affected All from 8-28-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

5.3.8 1 M ~~Sodium Bisulfite (Na_2HSO_3)~~ Sodium meta-Bisulfite ($Na_2S_2O_5$): Dissolve approximately 2.9 grams of ~~Na_2HSO_3~~ $Na_2S_2O_5$ anydrous powder in demineralized water and dilute to 30 ml. Make fresh daily.

6.4 Wash the resin with ~~three~~ successive 100 ml batches of hot water until solution is clear. Stir manually, allow the resin to settle to the bottom of the beaker. Decant and discard the wash water.

6.6 Add a fresh 125 ml of NaOCl solution to the resin remaining in the beaker and stir for another 10 minutes. Allow the resin to settle and filter the NaOCl solution into the Buchner funnel.

CAUTION: The following step MUST be performed in a fume hood. The nitric acid/sodium hypochlorite reaction is vigorous, therefore, acid additions MUST be made slowly.

6.7 In the fume hood, slowly add 20 ml of concentrated HNO_3 to the NaOCl solution from the previous step. Stir until the bulk of the chlorine gas has been evolved from the solution.

Submitted by: James Mardlad

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Zaca, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

6.15 Add 25 ml of demineralized water and 1 ml of 1 M sodium ~~bisulfite~~ meta-bisulfite. Shake until the organic layer is colorless.

6.20 Add 20 ml of demineralized water and 1 ml of 1 M sodium ~~bisulfite~~ meta-bisulfite. Shake for approximately 30 seconds. Collect the bottom aqueous layer in a 100-ml beaker containing a small magnetic stirring bar.

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 1
	REVISION: 0	DATE: 4-05-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Iodine-131 activity in milk and water samples.

2.0 SCOPE

The procedure applies to all samples of milk and water requiring Iodine-131 activity analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Iodine-131 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with the implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 J.J. Gabay, C.J. Paperiello, S. Goodyear, J.C. Daly, and J.M. Matuszek, Health Physics 26, 89 (1974).

4.4.2 D.E. McCurdy, R.A. Mellen, R.W. Lambdin, and M.E. McLain, "The Use of Cuprous Iodine as a Precipitation Matrix in the Radiochemical Determination of I-131 in Milk," ASTM Conference on Effluent and Environmental Radiation Surveillance, Johnson, Vermont, July 9-14, 1978.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0-160.0000 gram capacity)

5.1.2 Centrifuge

5.1.3 Desiccator

5.1.4 Vortex mixer

5.1.5 Hot plate

5.1.6 Hot water bath

5.1.7 Stirrers

5.1.8 Separatory funnels

5.1.9 Ion exchange column (3-cm diameter X 17 cm)

5.1.10 Membrane filtration apparatus (15-ml capacity, 25-mm diameter)

5.1.11 Assorted tools and routine laboratory equipment

5.2 Materials

5.2.1 Whatman 42 filter paper, 2.4 cm

5.2.2 Resin, Anion Exchange, Bio Rad AG1-X8 Cl form (50 - 100 mesh)

5.3 Reagents

5.3.1 Silver nitrate ($AgNO_3$) solution (12 mg/ml, Ag): Add 1.7 gram of ACS reagent grade $AgNO_3$ to 100 ml of demineralized water.

5.3.2 Ethyl Alcohol (CH_3CH_2OH), 95 Percent: ACS reagent grade

5.3.3 Hydroxylamine hydrochloride ($NH_2OH \cdot HCL$): Crystals, ACS reagent grade

5.3.4 Iodide Carrier (10 mg/ml): Dissolve 6.54 grams of potassium iodide (KI) in 400 ml of demineralized water and dilute to 500 ml with demineralized water.

5.3.5 Nitric Acid (HNO_3): Concentrated, ACS reagent grade

5.3.6 6 M Nitric Acid: Add 375 ml of concentrated HNO_3 to 500 ml of demineralized water and dilute to 1 liter with demineralized water. Mix thoroughly.

5.3.7 1 M Nitric Acid: Add 63 ml of concentrated HNO_3 to 500 ml of demineralized water and dilute to 1 liter with demineralized water. Mix thoroughly.

5.3.8 1 M Sodium Bisulfite (Na_2HSO_3): Dissolve approximately 2.9 grams of Na_2HSO_3 anydrous powder in demineralized water and dilute to 30 ml. Make fresh daily.

5.3.9 1 M Sodium Nitrite ($NaNO_2$): Dissolve approximately 2 grams of $NaNO_2$ in 10 ml of demineralized water and dilute to 30 ml. Make fresh daily.

5.3.10 Sodium Hypochlorite (6 Percent NaOCl): Commercial strength bleach

5.3.11 Toluene ($C_6H_5CH_3$): ACS reagent grade

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 4
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6.0 PROCEDURE

6.1 Add 4 ml of iodide carrier to 2 liters (or the required amount) of milk or water. Record volume used on appropriate data sheet (see appendix 10.1). Mix thoroughly.

6.2 Pass the sample through the column at maximum flow rate.

6.3 Extrude the anion exchange resin from the column into a 600 ml beaker. Effect complete transfer of the resin by washing the column walls with 5 - 10 ml of demineralized water.

6.4 Wash the resin with three successive 100 ml batches of water. Stir manually, allow the resin to settle to the bottom of the beaker. Decant and discard the wash water.

6.5 Place a magnetic stirring bar in the beaker (in hood) with the washed resin and add 125 ml of NaOCl. Place the beaker on a magnetic stirrer and stir for 10 minutes. Allow the resin to settle. Filter the NaOCl solution by suction on a Buchner-type funnel using a glass fiber filter.

6.6 Add a fresh 125 ml of NaOCl solution to the resin remaining in the beaker and stir for another 10 minutes. Allow the resin to settle and filter the NaOCl solution into the Buchner funnel.

6.7 In the fume hood, slowly add 20 ml of concentrated HNO_3 to the NaOCl solution from the previous step. Stir until the bulk of the chlorine gas has been evolved from the solution.

6.8 Pour the acidified solution into a 500 ml separatory funnel.

6.9 Add 50 ml of toluene and shake vigorously.

6.10 Add approximately 1.5 grams of hydroxylamine hydrochloride and shake for approximately 60 seconds.

6.11 Add another 1.0 gram of hydroxylamine hydrochloride and shake for approximately 60 seconds.

6.12 Add 3 ml of 1 M sodium nitrite and shake for approximately 30 seconds.

6.13 Allow the layers to separate. Drain off the bottom aqueous layer into a clean separatory funnel. Save the organic layer in the original separatory funnel.

6.14 Add 30 ml of toluene to the aqueous layer and shake for approximately 30 seconds. Let the layers separate, then drain off the bottom aqueous layer and discard. Combine the remaining toluene layer with the previous toluene extraction.

6.15 Add 25 ml of demineralized water and 1 ml of 1 M sodium bisulfite. Shake until the organic layer is colorless.

6.16 Allow the layers to separate. Retain the bottom aqueous layer and discard the toluene.

6.17 Add 50 ml of toluene, 1 ml of 6 M nitric acid and 1 ml of 1 M sodium nitrite. Shake for approximately 60 seconds.

6.18 Allow the layers to separate. Discard the bottom aqueous layer.

6.19 Add 25 ml of demineralized water and shake for approximately 30 seconds. Allow the layers to separate. Discard the bottom aqueous layer.

6.20 Add 20 ml of demineralized water and 1 ml of 1 M sodium bisulfite. Shake for approximately 30 seconds. Collect the bottom aqueous layer in a 100-ml beaker containing a small magnetic stirring bar.

6.21 Add 2 ml of concentrated nitric acid, a 1-inch magnetic stirring bar and, with stirring, slowly add 5 ml of silver nitrate solution.

6.22 Heat the solution on a hot plate until the silver iodide coagulates.

6.23 Quantitatively transfer to a centrifuge tube. Centrifuge and discard the supernatant.

6.24 Wash the precipitate with 10 ml of 1 M nitric acid, centrifuge and discard the wash.

6.25 Add 5 ml of distilled water and slurry the precipitate.

6.26 Filter with weak vacuum through a tared filter paper (Whatman 42). Wash the centrifuge tube and filtering apparatus with demineralized water followed by ethyl alcohol.

6.27 Dry the precipitate in a desiccator or in a drying oven at 80°C to constant weight. Record the tare weight and the total weight.

6.28 Mount sample and submit for counting.

7.0 PRECISION AND ACCURACY

The standard deviation for a single determination is 10% at 55 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1 Sample volume

6.7 Reaction time

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balance shall be in control as determined by routine balance check procedure.

8.4.3 Counting instrument shall be calibrated prior to use.

8.5 Certification

8.5.1 Portions of this procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

131-Iodine Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG)}{CTIME*2.22*YLD*EFF*DF*SVOL}$$

131-Iodine Activity Error (pCi/l) = ERR

$$= ACT* \frac{TSC}{(TSC - BKG)^2} + 0.0025$$

Lower Limits of Detection (pCi/l) = LLD

$$= \frac{4.65 * BKG + 3}{CTIME*2.22*YLD*EFF*DF*SVOL}$$

TSC = Total sample counts
BKG = Background counts
CTIME = Count time (min)
YLD = Chemical yield
EFF = Counting efficiency
DF = Decay factor (see appendix 10.2)
SVOL = Sample volume (L)
2.22 = Conversion from dpm to pCi
4.65,3 = Constants
0.0025 = Estimates of relative error from all other sources.

10.0 APPENDICES

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APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-308

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Gross Alpha and/or Beta Activity

APPROVED: James T. Harney DATE: 4-28-87
Laboratory Manager

APPROVED: [illegible] DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Auffier DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-20-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0; 9.1.1; 9.1.2; 9.2.1; 9.2.2; 9.3.1; 9.3.2
Procedure Title & Number (If exists) RSL-308, RO
Determination of Gross Alpha and/or Beta Activity
Description of experiment or method Correct equations to calculate Error and Lower limit of Detection

Time Period From: 4/20/87 To: 8/11/87 Samples Affected All between 4-20-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James [illegible]
Approved by: John R Fraser, Laboratory Manager
Approved by: John A. Cupin, Laboratory Director
Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 7
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9.0 CALCULATIONS

9.1 Air Filter Calculations

9.1.1 Beta

Beta Activity (pCi/m^3) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME}$

Beta Activity Error (pCi/m^3) =

$$ERR = ACT* \left[\frac{TSC}{(TSC - BKG)^2} + 0.01 \right]^{1/2}$$

Lower Limit of Detection (pCi/m^3) = LLD = $\frac{[4.65* (BKG)^{1/2} + 3]}{2.22*EFF*VOL*CTIME}$

TSC	=	Sample counts (Beta)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Total air volume (m^3)
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.01	=	Estimate of relative error from all other sources

9.1.2 Alpha

Alpha Activity (pCi/m^3) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME}$

Alpha Activity Error (pCi/m^3) =

$$ERR = ACT* \left[\frac{TSC}{(TSC - BKG)^2} + 0.01 \right]^{1/2}$$

Lower Limit of Detection (pCi/m^3) = LLD = $\frac{[4.65* (BKG)^{1/2} + 3]}{2.22*EFF*VOL*CTIME}$

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
VOL	=	Total Air Volume (m^3)
EFF	=	Counting efficiency
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.01	=	Estimate of relative error from all other sources

9.2 Solid Sample Calculations

9.2.1 Beta

Beta Activity (pCi/g) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*RF*CTIME}$

Act (pCi/kg) = ACT*1000

Beta Activity Error (pCi/g) =

$$ERR = ACT*\left[\frac{TSC}{(TSC - BKG)^2} + 0.01\right]^{1/2}$$

ERR (pCi/kg) = ERR*1000

Lower Limit of Detection (pCi/g) = LLD = $\frac{\left[4.65*(BKG)^{1/2} + 3\right]}{2.22*EFF*VOL*RF*CTIME}$

LLD (pCi/kg) = LLD*1000

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Sample volume (g)
RF	=	Ratio factor
2.22	=	Conversion from dpm to pCi
1000, 4.65,3	=	Constants
0.01	=	Estimate of relative error from all other sources

Ash to dry	RF = dry weight/ash weight
Ash to wet	RF = wet weight/ash weight
Dry to wet	RF = wet weight/dry weight
Otherwise	RF = 1

9.2.2 Alpha

Alpha Activity (pCi/g) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*RF*CTIME}$

ACT (pCi/kg) = ACT*1000

Alpha Activity Error (pCi/g) =

$$ERR = ACT*\left[\frac{TSC}{(TSC - BKG)^2} + 0.01\right]^{1/2}$$

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 9
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ERR (pCi/kg) = ERR*1000

Lower Limit of Detection (pCi/g) = LLD = $\frac{[4.65*(BKG)^{1/2} + 3]}{2.22*EFF*VOL*RF*CTIME}$

LLD (pCi/kg) = LLD*1000

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Sample volume (g)
RF	=	Ratio factor
2.22	=	Conversion from dpm to pCi
1000, 4.65, 3	=	Constants
0.01	=	estimate of relative error from all other sources

Ash to dry RF = dry weight/ash weight
Ash to wet RF = wet weight/ash weight
Dry to wet RF = wet weight/dry weight
Otherwise RF = 1

9.3 Liquid Samples Calculations

9.3.1 Beta

Beta Activity (pCi/l) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME*R}$

Beta Activity Error (pCi/l) =

$$ERR = ACT*\left[\frac{TSC}{(TSC - BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/g) = LLD = $\frac{[4.65*(BKG)^{1/2} + 3]}{2.22*EFF*VOL*CTIME*R}$

TSC	=	Total sample counts (Beta)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Sample volume (l)
R	=	Self absorption correction
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.0025	=	Estimate of relative error from all other sources

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 10
	REVISION: 0	DATE: 4-20-87

9.3.2 Alpha Activity (pCi/l) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME*R}$

Alpha Activity Error (pCi/l) =

$$ERR = ACT* \left[\frac{TSC}{(TSC - BKG)^2} + 0.0025\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD = $\frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*EFF*VOL*CTIME*R}$

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Sample volume (l)
R	=	Self absorption correction
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.0025	=	Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 1
	REVISION: 0	DATE: 4-20-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of gross alpha and/or gross beta activity.

2.0 SCOPE

The procedure applies to all samples requiring gross alpha and/or gross beta activity analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring gross alpha and/or gross beta activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6. ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples," Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "Radioisotope Techniques," R.T. Overman and H.M. Clark, McGraw-Hill Book Co., Inc., New York, 1960. (Chapter 6)

4.4.2 "Determination of Beta Activity in Water," F.B. Barker and B.P. Robinson, U.S. Geological Survey Water-Supply Paper 1696-A, Washington, D.C., 1963.

4.4.3 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water"; H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, August 1980.

4.4.4 "Standard Methods for the Examination of Water and Waste Water," APHA, AWWA, WPCF, American Public Health Association, Washington, D.C, latest edition.

4.4.5 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.6 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

4.4.7 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.8 "Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", Annual Book of ASTM Standards, Vol. 12, C1009-83.

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 3
	REVISION: 0	DATE: 4-20-87

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0-160.0000 gram capacity)

5.1.2 pH meter

5.1.3 Hot plate

5.1.4 Heat lamp

5.1.5 Desiccator

5.2 Materials

5.2.1 Assorted stainless steel planchets

5.2.2 Assorted tools and routine laboratory equipment (such as spatulas, forceps, beakers, graduated cylinders)

5.3 Reagents

5.3.1 Nitric Acid (HNO_3); ACS reagent grade; concentrated

5.3.2 1M Nitric Acid: Add 65 ml of concentrated HNO_3 to 935 ml of demineralized water. Mix thoroughly.

6.0 PROCEDURE

6.1 Air Filters

6.1.1 Record the sample information on the appropriate data form. See appendix 10.1.

6.1.2 Secure the air filter into a shallow stainless steel planchet. No portion of the filter shall be exposed above the lip of the planchet. Determine counting background for air filters by using a blank (clean) air filter.

6.1.3 Submit the sample to the counting room for counting.

6.1.4 Record the counting data on the appropriate data form.

6.1.5 Calculate the results as shown in the appropriate section of paragraph 9.0.

6.2 Solid Samples

6.2.1 Record the sample information on the appropriate data form. See appendix 10.2.

6.2.2 Refer to Table I for appropriate sample sizes depending on sample type.

Table I

Sample Type	Sample Size (grams)
beef	2
fish	2
food crops	2
grain	2
poultry	2
sediment	3
soil	3
vegetation	2

6.2.3 Distribute the sample or sample ash evenly in a tared deep stainless steel planchet and carefully pack down with a flat weight of the same diameter as the planchet. Record the tare and total weight in grams.

6.2.4 Submit the sample to counting room for counting.

6.2.5 Record the counting data on the appropriate data form.

6.2.6 Calculate the results as shown in the appropriate section of paragraph 9.0.

6.3 Water Samples

6.3.1 Record the sample information on the appropriate data form. See appendix 10.3.

6.3.2 Using a graduated cylinder, measure an appropriate volume of the sample into a beaker. Record the sample volume used.

6.3.3 Add 5 ml of concentrated nitric acid.

6.3.4 Place samples on a hotplate, and evaporate to near dryness. Transfer to smaller tared beaker, as necessary, to determine solids content of the sample. Use concentrated nitric acid to assist transfer.

6.3.5 Evaporate to dryness. Determine the solids content of the sample. If the solids content exceeds 100 mg, return to 6.3.2 and adjust the initial sample volume accordingly.

6.3.6 Dissolve the sample in a small volume of 1 M nitric acid.

6.3.7 Quantitatively transfer the sample to a tared deep stainless steel planchet. Using a rubber policeman, wash down the beaker walls with small portions of 1 M nitric acid.

6.3.8 Evaporate to dryness under a heat lamp positoned not closer than 8 inches above the planchet. If green residue forms, see Laboratory Manager, or his designee.

6.3.9 Weigh the cooled planchet and record the tare and total weight in grams. Do not exceed 100 mg of solids in the planchet.

6.3.10 Store in a dessicator until the sample is counted for gross alpha and/or gross beta analysis.

6.3.11 Submit the sample to counting room for counting.

6.3.12 Record the counting data on the appropriate data form.

6.3.13 Calculate the results as shown in the appropriate section of paragraph 9.0.

7.0 PRECISION AND ACCURACY

7.1 For gross alpha, the standard deviation for a single determination is 25% at 20 pCi/l.

7.2 For gross beta, the standard deviation for a single determination is 5% at 100 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the tehnician performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.2.2 Sample size

6.3.8 Color of residue

6.3.9 Weight limitation

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balance shall be in control as determined by routine balance check procedure.

8.4.3 Counting instruments shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

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9.0 CALCULATIONS

9.1 Air Filter Calculations

9.1.1 Beta

Beta Activity (pCi/m^3) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME}$

Beta Activity Error (pCi/m^3) =

$$ERR = ACT* \frac{TSC}{(TSC - BKG)^2} + 0.01$$

Lower Limit of Detection (pCi/m^3) = LLD = $\frac{4.65* \ BKG + 3}{2.22*EFF*VOL*CTIME}$

TSC	=	Sample counts (Beta)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Total air volume (m^3)
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.01	=	Estimate of relative error from all other sources

9.1.2 Alpha

Alpha Activity (pCi/m^3) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME}$

Alpha Activity Error (pCi/m^3) =

$$ERR = ACT* \frac{TSC}{(TSC - BKG)^2} + 0.01$$

Lower Limit of Detection (pCi/m^3) = LLD = $\frac{4.65* \ BKG + 3}{2.22*EFF*VOL*CTIME}$

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
VOL	=	Total Air Volume (m^3)
EFF	=	Counting efficiency
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.01	=	Estimate of relative error from all other sources

9.2 Solid Sample Calculations

9.2.1 Beta

Beta Activity (pCi/g) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*RF*CTIME}$

Act (pCi/kg) = ACT*1000

Beta Activity Error (pCi/g) =

$$ERR = ACT* \frac{TSC}{(TSC - BKG)^2} + 0.01$$

ERR (pCi/kg) = ERR*1000

Lower Limit of Detection (pCi/g) = LLD = $\frac{4.65*\ BKG + 3}{2.22*EFF*VOL*RF*CTIME}$

LLD (pCi/kg) = LLD*1000

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Sample volume (g)
RF	=	Ratio factor
2.22	=	Conversion from dpm to pCi
1000, 4.65,3	=	Constants
0.01	=	Estimate of relative error from all other sources

Ash to dry	RF = dry weight/ash weight
Ash to wet	RF = wet weight/ash weight
Dry to wet	RF = wet weight/dry weight
Otherwise	RF = 1

9.2.2 Alpha

Alpha Activity (pCi/g) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*RF*CTIME}$

ACT (pCi/kg) = ACT*1000

Alpha Activity Error (pCi/g) =

$$ERR = ACT* \frac{TSC}{(TSC - BKG)^2} + 0.01$$

ERR (pCi/kg) = ERR*1000

Lower Limit of Detection (pCi/g) = LLD = $\frac{4.65* \quad BKG + 3}{2.22*EFF*VOL*RF*CTIME}$

LLD (pCi/kg) = LLD*1000

TSC = Total sample counts (Alpha)
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
VOL = Sample volume (g)
RF = Ratio factor
2.22 = Conversion from dpm to pCi
1000,
4.65, 3 = Constants
0.01 = estimate of relative error from all other sources

Ash to dry RF = dry weight/ash weight
Ash to wet RF = wet weight/ash weight
Dry to wet RF = wet weight/dry weight
Otherwise RF = 1

9.3 Liquid Samples Calculations

9.3.1 Beta

Beta Activity (pCi/l) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME*R}$

Beta Activity Error (pCi/l) =

$$ERR = ACT* \frac{TSC}{(TSC - BKG)^2} + 0.0025$$

Lower Limit of Detection (pCi/g) = LLD = $\frac{4.65* BKG + 3}{2.22*EFF*VOL*CTIME*R}$

TSC = Total sample counts (Beta)
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
VOL = Sample volume (l)
R = Self absorption correction
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.0025 = Estimate of relative error from all other sources

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9.3.2 Alpha Activity (pCi/l) = ACT = $\frac{TSC - BKG}{2.22*EFF*VOL*CTIME*R}$

Alpha Activity Error (pCi/l) =

$$ERR = ACT* \frac{TSC}{(TSC - BKG)^2} + 0.0025$$

Lower Limit of Detection (pCi/l) = LLD = $\frac{4.65* \ BKG + 3}{2.22*EFF*VOL*CTIME*R}$

TSC	=	Total sample counts (Alpha)
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency
VOL	=	Sample volume (l)
R	=	Self absorption correction
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.0025	=	Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 11
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Appendix 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367-9 86

433 1 57

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Appendix 10.2

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367 9 86

430-1 87

TITLE: Determination of Gross Alpha and/or Beta Activity	NO: RSL-308	PAGE: 13
	REVISION: 0	DATE: 2-23-87

Appendix 10.3

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367-9 86

430 1 87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-309

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Radium-226,228 in Aqueous Samples

APPROVED: James T. Harvey DATE: 5-6-87
Laboratory Manager

APPROVED: Paul Miller DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cantin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.6

Procedure Title & Number (If exists) RSL-309, R0
Determination of Radium-226, 228 in Aqueous Samples

Description of experiment or method Prevent crystallization of sodium sulfate during subsequent filtration

Time Period From: 8/6/87 To: Issue of RSL-309, R1 Samples Affected All from 8-6-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-6-87

6.6 Add 5 ml 40% sodium sulfate solution and 2 ml 6 M acetic acid. Swirl to mix. Cool in ice bath 30 minutes. Centrifuge and discard supernatant. Slurry precipitate with 5 ml deionized water.

Submitted by: [signature]

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-309, RO
Determination of Radium-226, 228 in Aqueous Samples

Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/14/87 To: 8/11/87 Samples Affected All between 4-14-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James M. Dillard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John G. Cuspier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

8.4.2 Alpha spectrometer system shall be calibrated before use.

8.4.3 Alpha/beta counter shall be calibrated before use.

8.4.4 Balances shall be checked to ensure calibration dates have not expired.

8.4.5 Balances shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Radium-226 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*R}$$

Radium-228 Activity Error (pCi/l) = ERR

$$= ACT*\left[\frac{TSC}{(TSC - BKG)^2} + 0.005\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65*(BKG)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME*R}$$

TSC = Sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
SVOL = Sample volume (l)
R = Recovery
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

TITLE: Determination of Radium-226,228 in Aqueous Samples	NO: RSL-309	PAGE: 7
	REVISION: 0	DATE: 4-14-87

Radium-228 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*R*DEC}$$

Radium-228 Activity Error (pCi/l) = ERR

$$= ACT*\left[\frac{TSC}{(TSC - BKG)^2} + 0.005\right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65*(BKG)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME*R*DEC}$$

TSC = Sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
DEC = Ac-228 decay factor (see appendix 10.2)
SVOL = Sample volume (l)
R = Recovery
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
(If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If existing): RSL-309-R0

DETERMINATION OF RADIUM-226/228 IN AQUEOUS SAMPLES

Description of experiment or method: Correction of typographical error

(Note: this change revision incorporates CH-2, page 2)

Issue of RSL-309,R1

Time Period From: 8/28/87 To: -/-/- Samples Affected: All from 8-28-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-28-87

9.0 CALCULATIONS

Radium-226 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22 * EFF * SVOL * CTIME * R}$$

Radium-226 Activity Error (pCi/l) = ERR

$$= ACT * \left[\frac{TSC}{(TSC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65 * (BKG)^{1/2} + 3\right]}{2.22 * EFF * SVOL * CTIME * R}$$

TSC = Sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
SVOL = Sample volume (l)
R = Recovery
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

3223

Submitted by: James H. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Fraga, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000300

RSL 309 R1 C1

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.2

Procedure title & number (if existing): Radium 226, 228 RSL 309 R0

Description of experiment or method: Procedure clarification

Time Period From: 4/14/87 To: Issue of RSL 309 R1 Samples Affected: All from 4-14-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

From ~~4-14-87~~ 6-25-87 CLP 9/23/87

6.2 ---- add 2 ml of 1 M $Pb(NO_3)_2$.
If precipitate occurs, add concentrated HNO_3 until it dissolves (but less than 30 ml).
add 0.05 ml $Ba(NO)_3$ ---

Submitted by: MJF

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. A. Cuffin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000301

RSL 309 R1 (2) 5

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure: Yes (X) No ( )
If Yes, enter paragraph number or reference insertion point: 6.5, 6.6

Procedure Title & Number (if exists): RSL 309 R1

Determination of Sulfate, SO4 in Aqueous Samples

Description of experiment or method: Procedure enhancement

Time Period: From: 4/14/87 To: tt Samples Affected: All from 4-14-87

Issue of RSL 309 R1

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

From 4-14-87
6-25-87
CDP 9/25/87

(up to 10ml if precipitate amount is excessive)

6.5 Dissolve precipitate in 5 ml 30% EDTA solution. If the solution does not clear or appears dirty, place in hot water bath for approximately 10 minutes. Spin down and pour off supernatant into another centrifuge tube. Discard precipitate.

Use option volume if 10ml of 30% EDTA was used in step 6.5.

(up to 10ml) (up to 4ml)

6.6 Add 5 ml 40% sodium sulfate solution and 2 ml 6 M acetic acid. Swirl to mix. Cool in ice bath 30 minutes. Centrifuge and discard supernatant. Slurry precipitate with 5ml deionized water.

Submitted by: James Mullard

Approved by: James T. Harvey, Laboratory Manager

Approved by: [illegible], Laboratory Director

Approved by: Cheryl A. Prince, QA/QC/Director, ITAS

PROCEDURE DEVELOPMENT/CHANGE

Supporting evidence [illegible] procedure [illegible]

[illegible]

Procedure [illegible] (if exists) [illegible]

Method [illegible] samples

Description of experiment or method Procedure [illegible]

Time Period From: 9/29/87 To: Issue of SOL 304, R1 Samples Affected All from 9/29/87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 9/29/87

5.3.6 7.5% EDTA Solution: Add 75 grams of (Tetra Sodium Ethylenediamine Tetraacetate) EDTA salt to 1000 ml of demineralized water. Mix thoroughly.

5.3.7 20% Sodium Sulfate (Na_2SO_4): Add 20 grams of sodium sulfate to 100 ml of demineralized water. Mix thoroughly to dissolve. Filter through 0.45 micron filter before use.

5.3.14 30% EDTA Solution: Add 300 grams of (Tetra Sodium Ethylenediamine Tetraacetate) EDTA salt to 1000 ml of demineralized water. Mix thoroughly.

Submitted by: [signature]

Approved by: James T. Harvey, Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000303

TITLE: Determination of Radium-226, 228 in Aqueous Samples	NO: RSL 309	PAGE:
	REVISION: 1	DATE: 9/22/87

Use option volume if 10ml of 30% EDTA was used in step 6.5.

6.6 Add 5 ml ~~40%~~ (up to 10 ml) 20% sodium sulfate solution and 2 ml (up to 4ml) 6 M acetic acid. Swirl to mix. Cool in ice bath 30 minutes.

6.10 Transfer residue to centrifuge tube with 10 ml 7.5% EDTA solution. Record T_1, start of Actinium decay. add 10 ml ~~40%~~ 20% sodium sulfate and 1 ml 6 M acetic acid. Cool in ice bath 30 minutes, centrifuge and transfer supernatant to 60 ml separatory funnel. Discard precipitate.

6.14 Submit for counting. Record midpoint of count as T_2.

000304

TITLE: Determination of Radium-226,228 in Aqueous Samples	NO: RSL-309	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Radium-226,228 in aqueous samples.

2.0 SCOPE

This procedure applies to all water samples requiring Radium-226,228 activity determination.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all water samples requiring Ra-226,228 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.2 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.3 ITAS Quality Assurance Manual.

4.1.4 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment

4.2 Standards

None

4.3 Procedures

4.3.1 "Procedures for Determination of Stable Elements and Radionuclides in Environmental Samples", Public Health Service Publication 999-RH-10, January 1965.

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

4.4.5 "The Radiochemistry of Radium", H.W. Kirby and M.L. Salutsky, NAS-NS-3057, National Academy of Science, December 1964.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Centrifuge, cone type

5.1.2 Sepratory funnels, 60 ml

5.1.3 Mechanical shaker

5.1.4 Automatic pipets

5.1.5 Filtering apparatus

5.1.6 Assorted tools and routine laboratory equipment

5.1.7 Analytical balance (0-160.0000g)

5.2 Materials

5.2.1 Millipore filters, 0.45 and 0.8 micron

5.2.2 Filter paper, P5

5.3 Reagents

5.3.1 Nitric Acid (HNO_3), concentrated, ACS reagent grade

5.3.2 1 M Lead Nitrate [$Pb(NO_3)_2$]: Add 165.6 grams of lead nitrate to a 1 liter beaker and add 500 ml of demineralized water. Stir on hot plate on low until dissolved. Transfer to reagent container.

5.3.3 Barium Nitrate [$Ba(NO_3)_2$] (10mg/ml): Add 1.0 gram of barium nitrate to a 250 ml beaker containing 100 ml of 1 M nitric acid. Mix thoroughly until dissolved and transfer to a reagent container.

5.3.4 Sulfuric Acid (H_2SO_4), concentrated, ACS reagent grade

5.3.5 1 M Nitric Acid (HNO_3): Add 65 ml of concentrated nitric acid to 935 ml of demineralized water. Mix thoroughly.

5.3.6 7.5% EDTA Solution: Add 75 grams of EDTA salt to 1000 ml of demineralized water. Mix thoroughly.

5.3.7 40% Sodium Sulfate (Na_2SO_4): Add 40 grams of sodium sulfate to 100 ml of demineralized water. Mix thoroughly to dissolve. Filter through 0.45 micron filter before use.

5.3.8 6 M Acetic Acid (CH_3COOH): Add 175 ml of glacial acetic acid to 500 ml of demineralized water. Mix thoroughly.

5.3.9 Acetic Acid (CH_3COOH): glacial, ACS reagent grade

5.3.10 Hydrogen Peroxide (H_2O_2), 30%

5.3.11 2 M Monochloracetic Acid ($ClCH_2COOH$): Add 94.5 grams of monochloracetic acid to 500 ml of demineralized water. Mix thoroughly and store in reagent container.

5.3.12 15% Di-2 Ethylhexyl-Phosphoric Acid (DEHPA): Add 150 ml of DEHPA to 850 ml of N-Heptane. Mix thoroughly and store appropriately.

5.3.13 N-Heptane, ACS reagent grade

5.3.14 30% EDTA Solution: Add 300 grams of EDTA salt to 1000 ml of demineralized water. Mix thoroughly.

5.3.15 Actinium Wash Solution: Add 50 grams of monochloracetic acid ($ClCH_3COOH$), 1 gram of EDTA salt and 12.7 grams of sodium hydroxide (NaOH) to 500 ml of demineralized water. Mix thoroughly.

5.3.16 Sodium Hydroxide Pellets, ACS reagent grade

6.0 PROCEDURE

6.1 Filter the sample through P5 filter paper into a 1500 ml beaker. If sample appears to contain organics (oily, sulphur smell, etc.), boil approximately 45 minutes and let cool. Record volume on data sheet (appendix 10.1). Make to 1 liter with demineralized water as necessary. Run a blank and spike with each set of samples.

6.2 While stirring, add 2 ml of 1 M lead nitrate and 0.05 ml of barium nitrate. Add 5 ml of sulfuric acid slowly and continue stirring for approximately 10 minutes.

6.3 Allow precipitate to settle for about 1 - 2 hours.

6.4 Decant supernatant, transfer precipitate to centrifuge tube with demineralized water and centrifuge. Discard supernatant.

6.5 Dissolve precipitate in 5 ml 30% EDTA solution. If the solution does not clear or appears dirty, place in hot water bath for approximately 10 minutes. Spin down and pour off supernatant into another centrifuge tube. Discard precipitate.

6.6 Add 5 ml 40% sodium sulfate solution and 2 ml 6 M acetic acid. Swirl to mix. Cool in ice bath 30 minutes.

6.7 Filter through 0.8 micron millipore filter, mount and analyze for Ra-226 by alpha spectrometry.

6.8 If Ra-228 analysis is required, allow Ac-228 to grow in for three days after step 6.7.

6.9 Ash down millipore filter (step 6.7) with nitric acid and hydrogen peroxide. Add 1 ml barium nitrate (10 mg/ml) and evaporate to dryness. Take down once with concentrated nitric acid and the final time with small amount of demineralized H_2O.

6.10 Transfer residue to centrifuge tube with 10 ml 7.5% EDTA solution, add 10 ml 40% sodium sulfate and 1 ml 6 M acetic acid. Cool in ice bath 30 minutes, centrifuge and transfer supernatant to 60 ml separatory funnel. Discard precipitate.

6.11 To separatory funnel, add 10 ml of demineralized water, 5 ml 2 M monochloracetic acid and 10 ml 15% Di-2-ethylhexyl-phosphoric acid (DEHPA) in n-heptane. Shake for 1 minute. Discard aqueous phase.

6.12 Wash organic phase twice with 10 ml each time of actinium wash solution, shaking for 1 minute each time.

6.13 Back extract with 5 ml of 1 M HNO_3 and filter thru glass wool. Repeat with another 5 ml of 1 M HNO_3. Pipette onto 2 inch stainless steel planchets and evaporate.

7.0 PRECISION AND ACCURACY

7.1 For a single determination, the standard deviation at 5 pCi/liter is 15% for Ra-226.

7.2 For a single determination, the standard deviation at 7 pCi/liter is 15% for Ra-228.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 pH meter shall be checked to ensure calibration.

8.4.2 Alpha spectrometer system shall be calibrated before use.

8.4.3 Alpha/beta counter shall be calibrated before use.

8.4.4 Balances shall be checked to ensure calibration dates have not expired.

8.4.5 Balances shall be in control as determined by routine balance check procedure.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Radium-226 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*R}$$

Radium-228 Activity Error (pCi/l) = ERR

$$= ACT* \frac{TSC}{(TSC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*SVOL*CTIME*R}$$

TSC = Sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
SVOL = Sample volume (l)
R = Recovery
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

Radium-228 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*R*DEC}$$

Radium-228 Activity Error (pCi/l) = ERR

$$= ACT* \frac{TSC}{(TSC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*SVOL*CTIME*R*DEC}$$

TSC = Sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
DEC = Ac-228 decay factor (see appendix 10.2)
SVOL = Sample volume (l)
R = Recovery
2.22 = Conversion from dpm to pCi
4.65,
3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Radium-226,228 in Aqueous Samples	NO: RSL-309	PAGE: 8
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

TITLE: Determination of Ra-226, 228 in Acqueous Samples	NO: RSL-309	PAGE: 9
	REVISION: 0	DATE: 4-14-87

Appendix 10.2

Ac-228 Decay Factor

HRS: 0.00	Ac-228: 1.00000000
HRS: 0.50	Ac-228: 0.94503126
HRS: 1.00	Ac-228: 0.89308408
HRS: 1.50	Ac-228: 0.84399237
HRS: 2.00	Ac-228: 0.79759917
HRS: 2.50	Ac-228: 0.75375614
HRS: 3.00	Ac-228: 0.71232312
HRS: 3.50	Ac-228: 0.67316761
HRS: 4.00	Ac-228: 0.63616443
HRS: 4.50	Ac-228: 0.60119527
HRS: 5.00	Ac-228: 0.56814833
HRS: 5.50	Ac-228: 0.53691793
HRS: 6.00	Ac-228: 0.50740422
HRS: 6.50	Ac-228: 0.47951285
HRS: 7.00	Ac-228: 0.45315463
HRS: 7.50	Ac-228: 0.42824529
HRS: 8.00	Ac-228: 0.40470519
HRS: 8.50	Ac-228: 0.38245905
HRS: 9.00	Ac-228: 0.36143576
HRS: 9.50	Ac-228: 0.34156809
HRS: 10.00	Ac-228: 0.32279252
HRS: 10.50	Ac-228: 0.30504902
HRS: 11.00	Ac-228: 0.28823086
HRS: 11.50	Ac-228: 0.27243442
HRS: 12.00	Ac-228: 0.25745905

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-310

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Technicium-99

APPROVED: James T. Harvey DATE: 10-27-87
Laboratory Manager

APPROVED: Paul Mills DATE: 10-30-87
ITAS QA Director

CONCURRED: J. A. Cupin DATE: 11-9-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0	1			
DATE: 5-22-87	11-12-87			

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? (Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (if exists) RSL-310, R1

Determination of Technetium-99

Description of experiment or method Correct calculation section

Time Period From: 11/12/87 To: Issue of R2 Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 11-12-87

9.0 **CALCULATIONS**

Tc-99 Activity (pCi/L) = ACT

$$ACT = \frac{(TSC - BKG)}{2.22*EFF*SAC*CTIME*VOL*RF}$$

Tc-99 Activity Error (pCi/L) = ERR

$$ERR = ACT*\left[\frac{(TSC+BKG)}{(TSC - BKG)^2} + 0.0025\right]^{0.5}$$

Lower Limit of Detection (pCi/L) = LLD

$$LLD = \frac{[4.65*(BKG)^{0.5} + 3]}{2.22*EFF*SAC*CTIME*VOL*RF}$$

RF	=	Recovery Factor
TSC	=	Total sample counts
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency cpm/dpm
SAC	=	Self absorption correction
VOL	=	Sample volume (L)
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.0025	=	Sum of all other errors

Submitted by: James H. Ballard

Approved by: James T. Harvey, Laboratory Manager

Approved by: W. A. Cupin, Laboratory Director

Approved by: Cheryl R. Pence, QA/QC/Director, ITAS
for 11/20/87

000315

TITLE: Determination of Technicium-99	NO: RSL-310	PAGE: 1 of 8
	REVISION: 1	DATE: 11-12-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Technicium-99 activity.

2.0 SCOPE

The procedure applies to water samples requiring Technicium-99 activity analysis. This procedure provides good decontamination for Tc from fission product mixtures containing several grams of uranium or thorium.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Technicium-99 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in The Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.2 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Curie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

4.4.3 "The Radiochemistry of Technicium", Edward Anders, NAS-NS-3021, National Academy of Sciences, November 1960.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Centrifuge

5.1.2 Separatory funnels

5.1.3 Vacuum Desiccator

5.1.4 Analytical Balance (4-place)

5.2 Materials

5.2.1 Glass Microfiber filter paper, 4.7 cm, 0.6 cm particle retention in liquids.

5.2.2 Assorted tools and routine laboratory equipment

5.3 Reagents

5.3.1 Copper carrier (10 mg/ml, Cu^{2+}): Dissolve 3.7 grams of ACS reagent grade cupric nitrate [$Cu(NO_3)_2 \cdot 2\ 1/2\ H_2O$] in 75 ml of demineralized water. Add 0.1 ml of concentrated HNO_3, mix, and dilute to 100 ml with demineralized water. Store reagent crystals in a desiccator.

5.3.2 Hydrogen sulfide (H_2S), Gas 99.9% pure (lecture bottle size).

5.3.3 Hydrogen Peroxide (30%, H_2O_2), ACS reagent grade.

5.3.4 Sodium Sulfite (Na_2SO_3): Dissolve 1.3 grams of ACS reagent grade sodium sulfite in 50 ml of demineralized water and dilute to 100 ml with demineralized water. Make fresh prior to use.

5.3.5 Petroleum Ether, ACS reagent grade.

5.3.6 Ethanol (89%, CH_3CH_2OH) supplier certified grade.

5.3.7 Bromoform, supplier certified grade.

5.3.8 Tetrapropylammonium Hydroxide [10% in water, $(CH_3CH_2CH_2)_4NOH$], supplier reagent grade.

5.3.9 Tetrapropylammonium hydroxide/Bromoform solution: Add 2 ml of tetrapropylammonium hydroxide to 20 ml of bromoform per sample. Make fresh prior to use.

5.3.10 Hydrochloric Acid (HCl), concentrated, ACS reagent grade.

5.3.11 1N Hydrochloric Acid (HCl): Add 80 ml of concentrated HCl to 500 ml of demineralized water, and dilute to 1 liter with demineralized water.

5.3.12 3N Hydrochloric Acid (HCl): Add 240 ml of concentrated HCl to 500 ml of demineralized water, and dilute to 1 liter with demineralized water.

5.3.13 Nitric Acid (HNO_3) concentrated, ACS reagent grade.

5.3.14 Ammonium Hydroxide (HN_4OH), ACS reagent grade.

5.3.15 Sodium Hydroxide (NaOH), pellets, ACS reagent grade.

5.3.16 4N Sodium Hydroxide (NaOH): Dissolve 80 grams of sodium hydroxide pellets in 400 ml of demineralized water with stirring. Dilute to 500 ml with demineralized water.

6.0 PROCEDURE

6.1 Record all analysis data on a sample work sheet (Appendix 10.1). Reduce volume of an appropriate aliquot to 10-20 ml over low heat. Transfer to centrifuge tube. Analyze an analytical blank, spike and duplicate with each set of samples.

6.2 In a centrifuge tube, add 3 ml of concentrated HCl. Add 2 ml of copper carrier (10 mg Cu^{2+}/ml).

Note: The copper is added as a chemical carrier for the technicium.

CAUTION: The following step must be performed in a fume hood to remove hazardous H_2S fumes.

6.3 Add 100 uL of hydrogen peroxide (30%, H_2O_2). Pass hydrogen sulfide gas (H_2S) through the solution for approximately 3 minutes to precipitate the Cu as CuS.

Note: The H_2O_2 oxidizes all the Tc to the +7 oxidation state. The H_2S coprecipitates the Tc with the CuS.

6.4 Centrifuge and discard the supernate into the <pH2 liquid waste container.

6.5 Dissolve the CuS precipitate in 2 ml of concentrated HNO_3. In an ice bath, carefully add 4 ml of concentrated NH_4OH to complex the Cu^{2+}.

6.6 Transfer the solution with two 5 ml portions of 4N NaOH into a separatory funnel.

CAUTION: The following five steps must be performed in a fume hood to remove hazardous organic vapors. The fume hood must not contain any equipment which may produce heat or sparks. The fume hood must not contain any containers of oxidizing acids.

6.7 Add 20 ml of the tetrapropylammonium hydroxide/ bromoform solution to the separatory funnel. Shake for 30 sec. Allow the phases to separate. Discard the bottom aqueous phase into the >pH2 liquid waste container.

Note: TcO^4 is extracted into the bromoform solution.

6.8 Add 30 ml of 1N HCl and 5 ml of petroleum ether to the separatory funnel. Shake for 1 minute. Allow the phases to separate. Collect the bottom aqueous phase into a centrifuge tube. Discard the ether phase into a clean waste ether container which contains 25 ml of 0.1 M sodium sulfite (Na_2SO_3). Allow the waste ether to evaporate in a fume hood. Discard remaining aqueous waste into the >pH2 liquid waste container.

Note: The Tc is back extracted into the aqueous phase. The bromoform solution is soluble in the ether phase.

6.9 Add 2 ml of copper carrier (10 mg/ml, Cu^{2+}). Add 100 uL of hydrogen peroxide (30% H_2O_2). Pass hydrogen sulfide gas (H_2S) through the solution for approximately 3 minutes to precipitate the Cu as CuS.

6.10 Go to step 6.5 if directed by the Laboratory Manager to further decontaminate samples if necessary. Otherwise proceed to next step.

6.11 Quantitatively filter the CuS precipitate on to a tared glass fiber filter. Utilize demineralized water to transfer the precipitate. Wash with 5 ml portions of water and ethanol. Discard the ethanol into a clean waste ethanol container. Allow the waste ethanol to evaporate in a fume hood.

6.12 Vacuum desiccate the filtered precipitate to constant weight.

6.13 Submit for counting.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 10% at 100 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size , shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

Reagents shall be ACS Reagent grade or better. If ACS Reagent grade is not available, the best grade available shall be used. Lower grade material may be used for items as indicated in Section 5.3.

8.4 Equipment Monitoring

None

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Tc-99 Activity (pCi/L) = ACT

$$ACT = \frac{(TSC - BKG)}{2.22*EFF*SAC*CTIME*VOL*RF}$$

Tc-99 Activity Error (pCi/L) = ERR

$$ERR = ACT* \left(\frac{TSC}{(TSC - BKG)^2} + 0.0025 \right)^{0.5}$$

Lower Limit of Detection (pCi/L) = LLD

$$LLD = \frac{[4.65*(BKG)^{0.5} + 3]}{2.22*EFF*SAC*CTIME*VOL*RF}$$

RF	=	Recovery Factor
TSC	=	Total sample counts
BKG	=	Background counts
CTIME	=	Count time (min)
EFF	=	Counting efficiency cpm/dpm
SAC	=	Self absorption correction
VOL	=	Sample volume (L)
2.22	=	Conversion from dpm to pCi
4.65, 3	=	Constants
0.0025	=	Sum of all other errors

10.0 APPENDICES

TITLE: Determination of Technicium-99	NO: RSL-310	PAGE: 8 of 8
	REVISION: 1	DATE: 11-12-87

Appendix 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367-0-86

430 1 87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-311

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Calcium-45 by Chemical Separation

APPROVED: James T. Harney DATE: 5-6-87
Laboratory Manager

APPROVED: Paul Mills DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-08-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-311, RO
Determination of Calcium-45 by Chemical Separation

Description of experiment or method Correct equations to calculate Error and lower Limit of Detection

Time Period From: 4/8/87 To: 8/11/87 Samples Affected All between 4-8-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James M. Willard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John L. Cupin, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of Calcium-45 by Chemical Separation	NO: RSL-311	PAGE: 7
	REVISION: 0	DATE: 4-08-87

9.0 CALCULATIONS

Ca-45 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*YLD*CTIME*SVOL}$$

Ca-45 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{(TSC - BKG)} + 0.0025 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*EFF*YLD*CTIME*SVOL}$$

TSC = Total sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
YLD = Chemical yield
SVOL = Sample volume
4.65, 3 = Constants
2.22 = Conversion from dpm to pCi
0.0025 = Estimate of relative error from all other sources

10.0 APPENDICES

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.6, 6.3, 6.4

Procedure Title & Number (If exists) RSL-311, R0

DETERMINATION OF CALCIUM-45 BY CHEMICAL SEPARATION

Description of experiment or method Procedure clarification to allow for use of currently available reagent (5.3.6). Solutions saturated with H_2S after 2 minutes; reduced time to conserve reagent

Time Period From: 8/14/87 To: Issue of RSL-311, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

5.3.6 Ethanol (CH_3CH_2OH), ~~95%~~ 89% ACS reagent grade

6.3 Add 0.5 ml of concentrated HCl, 4 drops of Pd carrier and 8 drops of Cu carriers. Heat to boiling and bubble H_2S for ~~4-5~~ approximately 2 minutes through sample. Filter the acid sulfide scavenging precipitate on No. 40 Whatman paper and discard it, catching the filtrate in a centrifuge tube.

6.4 Add 4 drops each of Ni and Co carriers to the filtrate and heat to boiling. Add concentrated NH_4OH until the solution is basic; then add an additional 0.5 ml of NH_4OH. Pass in H_2S for ~~X~~ approximately 2 minutes and filter the ammonium sulfide scavenging precipitate on No. 40 Whatman paper and discard it, catching the filtrate in a 40-ml centrifuge tube.

Submitted by: James M. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of Calcium-45 by Chemical Separation	NO: RSL-311	PAGE: 1
	REVISION: 0	DATE: 4-08-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Calcium-45 in a chemically separated sample.

2.0 SCOPE

This procedure applies to aqueous samples requiring Calcium-45 activity analysis where chemical separation is required.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Calcium-45 activity determination where chemical separation is required.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 ITAS Quality Assurance Manual.

4.1.5. ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.1.6 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Radioisotope Techniques," R.T. Overman and H.M. Clark, McGraw-Hill Book Co., Inc., New York, 1960. (Chapter 6)

4.4.2 "Radiochemical Studies: The Fission Products," E.P. Steinberg, Book 1, McGraw - Hill, NY 1951, p. 482.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0-160.0000 gram capacity)

5.1.2 Filter funnels

5.1.3 Hot plate

5.1.4 Heat lamp

5.1.5 Desiccator

5.2 Materials

5.2.1 Assorted stainless steel planchets

5.2.2 Assorted tools and routine laboratory equipment (such as spatulas, forceps, beakers, graduated cylinders)

5.2.3 Whatman 40 filter paper

5.2.4 47 mm glass fiber filter paper

5.3 Reagents

5.3.1 Nitric Acid (HNO_3): Yellow fuming, ACS reagent grade

5.3.2 Ammonium Hydroxide (NH_4OH): Concentrated ACS reagent grade

5.3.3 Saturated Ammonium Oxalate [$(NH_4)_2C_2O_4 \cdot H_2O$]: Add 35 grams of ammonium oxalate to 1 liter of demineralized water and mix thoroughly.

5.3.4 1 M Sodium Bromate ($NaBrO_3$): Add 151 grams of sodium bromate to 1 liter of demineralized water and mix until dissolved.

5.3.5 Hydrogen Sulfide (H_2S): Gas (lecture bottle size)

5.3.6 Ethanol (CH_3CH_2OH): 95% ACS reagent grade

5.3.7 Calcium carrier, 10 mg/ml: Add 4 grams of calcium nitrate [$Ca(NO_3)_2 \cdot 4H_2O$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.8 Iron carrier, 10 mg/ml: Add 7.2 grams of ferric nitrate [$Fe(NO_3)_3 \cdot 9H_2O$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.9 Palladium carrier, 10 mg/ml: Add 0.8 grams of palladium chloride ($PdCl_2$) to 50 ml of demineralized water and mix.

5.3.10 Copper carrier, 10 mg/ml: Add 3.7 grams of cupric nitrate [$Cu(NO_3)_2 \cdot 2\frac{1}{2}H_2O$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.11 Nickel carrier, 10 mg/ml: Add 5 grams of nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.12 Cobalt carrier, 10 mg/ml: Add 5 grams of cobalt nitrate [$Co(NO_3)_2 \cdot 6H_2O$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.13 Strontium carrier, 10 mg/ml: Add 2.4 grams of strontium nitrate [$Sr(NO_3)_2$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.14 Barium carrier, 10 mg/ml: Add 1.9 grams of barium nitrate [$Ba(NO_3)_2$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.15 Lanthanum carrier, 10 mg/ml: Add 3.1 grams of Lanthanum nitrate [$La(NO_3)_3 \cdot 6H_2O$] to 100 ml of demineralized water. Add 0.1 ml of concentrated HNO_3 and mix.

5.3.16 Hydrochloric Acid (HCl), concentrated ACS reagent grade

5.3.17 Nitric Acid, concentrated ACS reagent grade

6.0 PROCEDURE

6.1 To an appropriate sample aliquot in a centrifuge tube, add 4 ml of Ca carrier. Enter appropriate information on the data sheet (appendix 10.1).

6.2 Acidify the solution with concentrated HNO_3, add 6 drops of Fe carrier, heat to boiling, and precipitate $Fe(OH)_3$ by the dropwise addition of concentrated ammonium hydroxide. Centrifuge and discard precipitate. Transfer sample to clean centrifuge tube.

6.3 Add 0.5 ml of concentrated HCl, 4 drops of Pd carrier and 8 drops of Cu carriers. Heat to boiling and bubble H_2S for 4-5 minutes through sample. Filter the acid sulfide scavenging precipitate on No. 40 Whatman paper and discard it, catching the filtrate in a centrifuge tube.

6.4 Add 4 drops each of Ni and Co carriers to the filtrate and heat to boiling. Add concentrated NH_4OH until the solution is basic; then add an additional 0.5 ml of NH_4OH. Pass in H_2S for 3 minutes and filter the ammonium sulfide scavenging precipitate on No. 40 Whatman paper and discard it, catching the filtrate in a 40-ml centrifuge tube.

6.5 Add 3 ml of saturated ammonium oxalate to the filtrate. Centrifuge and discard the supernate. Wash the precipitate with 30 ml of demineralized water.

6.6 Dissolve the precipitate in 4 ml of demineralized water and 1 ml of concentrated HNO_3. Add 1 ml each of Ba and Sr carrier. Add 30 ml of fuming HNO_3. Cool in an ice bath for several minutes. Centrifuge and discard the precipitate, transferring the supernate to a 125-ml erlenmeyer flask or beaker.

6.7 Boil down the supernate to 2-3 ml, transfer to a centrifuge tube and make to approximately 30 ml with demineralized water. Add NH_4OH to make solution basic and 2 ml of ammonium oxalate solution. Centrifuge and discard supernatant.

6.8 Dissolve the calcium oxalate in 2 ml of concentrated HNO_3. Add 2 ml of 1 M $NaBrO_3$ and transfer to small beaker. Evaporate to about 1 ml. Add 30 ml of H_2O. Make strongly basic with NH_4OH and transfer to centrifuge cone. Add 4 ml of saturated ammonium carbonate solution. Centrifuge the calcium carborate and discard the supernate.

6.9 Dissolve the calcium carborate in 1-2 ml of concentrated HNO_3. Dilute to 30 ml and add 1 ml of La carrier. Precipitate $La(OH)_3$ by the addition of NH_4OH. Centrifuge and transfer the supernate to a 40-ml centrifuge tube. Discard the precipitate.

6.10 Heat the supernate to 80°C and precipitate by the dropwise addition of 3 ml of saturated ammonium oxalate. Filter the calcium oxalate on a tared glass fiber filter with slight vacuum. Wash three times with 10-ml portions of hot demineralized water and then with 10 ml of ethanol. Dry in oven at 110° for 5 minutes or overnight in desiccator.

6.11 Weigh final precipitate and submit for counting. Yield shall be greater than 30%.

6.12 Calculate results in accordance with 9.0 below.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 15% at 10 pCi/L.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.11 Yield size

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balance shall be in control as determined by routine balance check procedure.

8.4.3 The beta counter shall be calibrated prior to use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples thorough the procedure steps.

TITLE: Determination of Calcium-45 by Chemical Separation	NO: RSL-311	PAGE: 7
	REVISION: 0	DATE: 4-08-87

9.0 CALCULATIONS

Ca-45 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*YLD*CTIME*SVOL}$$

Ca-45 Activity Error (pCi/l) = ERR

$$= ACT* \frac{TSC}{(TSC - BKG)} + 0.0025$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*YLD*CTIME*SVOL}$$

TSC = Total sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
YLD = Chemical yield
SVOL = Sample volume
4.65,
3 = Constants
2.22 = Conversion from dpm to pCi
0.0025 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Calcium-45 by Chemical Separation	NO: RSL-311	PAGE: 8
	REVISION: 0	DATE: 4-08-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

367-9 86

430-1-87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-312

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Iron-55

APPROVED: James T. Harvey DATE: 5-6-87
Laboratory Manager

APPROVED: Paul Mill DATE: 6-8-87
ITAS QA Director

CONCURRED: John L. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-10-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.2; 5.3.7; 6.2; 6.3; 6.6.
Procedure Title & Number (If exists) RSL-312, R0

Determination of Iron-55

Description of experiment or method Procedure clarification and modification to allow for use of commercial carrier standard.

Time Period From: 8/8/87 To: Issue of RSL-312, R1 Samples Affected All from 8-8-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-8-87

5.3.2 Fe Carrier, 1 mg/ml, commercial Fe standard (1000 ppm) ~~Dissolve 5.00 grams of metallic iron (ACS reagent grade) in 300 ml of 6 M hydrochloric acid (HCl) and filter. Dilute to 1 liter with demineralized water and calibrate by using an Atomic Absorption Spectrophotometer or purchase commercially.~~

5.3.7 Niobium Carrier, ~~2~~ 1 mg/ml, commercial Nb standard (1000 ppm) ~~Dissolve 2 grams of niobium wire in 90 mls of demineralized water containing 10 ml of concentrated HF in a plastic bottle.~~

6.2 Add ~~1~~ 5 ml ~~(5 mg)~~ 1 mg/ml of the Fe carrier solution and mix well.

6.3 Add 6 M NaOH, ~~mix and heat to precipitate iron hydroxide.~~ dropwise with mixing and heating until precipitation of iron hydroxide is complete. (>pH7)

6.6 Dissolve the precipitate in approximately 20 ml 10 M HCl and add 2 mg each of the appropriate hold back carriers (Co^{+2}, Zn^{+2}, Zr^{+4}, Nb^{+}, Mn^{+2}, and Cs^{+} carriers). Transfer the solution with 10 M HCl washes to a beaker and dilute to approximately 50 ml with 10 M HCl. Pass the solution through the anion exchange column at 5 to 8 ml/min.

Submitted by: James M. Dillard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cuyler, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-312, RO
Determination of Iron-55

Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/10/87 To: 8/11/87 Samples Affected All between 4-10-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

See attached

Submitted by: James M. Dillard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cumpier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

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8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balances shall be in control as determined by routine balance check procedure.

8.4.3 The liquid scintillation counter shall be calibrated prior to use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Iron-55 Activity (nCi/l) = ACT

$$= \frac{TSC5 - BKG - (TSC9*C)}{E*Y*VOL*T*2.22 \times 10^3}$$

Iron-55 Activity Error (nCi/l) = ERR

$$= ACT*\left[\frac{TSC5}{[TSC5 - BKG - (TSC9*C)]^2} + 0.005\right]^{1/2}$$

Iron-55 Lower Limit of Detection (nCi/l) = LLD

$$= \frac{\left[4.65*(BKG)^{1/2} + 3\right]}{E*Y*VOL*T*2.22 \times 10^3}$$

Where:

C	= cross talk factor obtained from iron-59 quench curve,
E	= fractional iron-55 efficiency obtained from the iron-55 quench curve,
Y	= chemical recovery from spike data
VOL	= sample volume (l)
TSC5	= gross counts in iron-55 channel
TSC9	= gross counts in iron-59 channel
BKG	= gross background counts in iron-55 channel
T	= counting time
465, 3	= constants
0.005	= estimate of relative error from all other sources
2.22×10^3	= conversion from dpm to nCi

3223

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 6.2, 6.4

Procedure Title & Number (If exists) RSL-313, RO DIRECT COUNTING OF TRITIUM, CARBON-14, AND CALCIUM-45 BY LIQUID SCINTILLATION COUNTING SYSTEMS

Description of experiment or method Allow for sample volume flexibility to meet detection limit requirements

Time Period From: 8/19/87 To: ~~++~~ Issue of RSL-313, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

6.2 Pipette two ~~3 ml~~ portions of the sample into counting vials containing ~~18 ml of~~ scintillation cocktail. Utilize appropriate sample volume up to a 10mL limit. Adjust scintillation cocktail volume for a total volume of approximately 20mL. To one vial, add the appropriate amount of internal standard solution used for the required analysis. Mix thoroughly. Prepare a spike and blank sample with each set of samples.

6.4 Record the data on the appropriate data sheet and calculate the results as shown in section 9.0.

Submitted by: James M. Dylland

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000340

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )

If yes enter paragraph number to correct/change/add for points 5.2.2, 5.3.12, 5.3.21, 5.3.22, 6.1, 6.7, 6.8

Procedure Title & Number (if existing) RSL-SOP-R01, Chg 3

Determination of Iron-55

Description of experiment or method Procedure clarification and enhancement

Issue of RSL-SOP-R01

Time Period From 9/1/87 To: ~~7-1~~ Samples Affected All from 9-1-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 9-11-87

5.2.2 Resin - AG1-X8, ~~100-200~~ 50-100 mesh; 15 ml equilibrated with 10 M HCl.

5.3.12 DELETED

5.3.21 Nitric Acid (HNO_3), ACS Reagent Grade concentrated

5.3.22 Hydrogen Peroxide (H_2O_2), 30% ACS Reagent grade.

6.1 Add 1ml of concentrated nitric acid and 100µL of hydrogen peroxide to an appropriate aliquot of the sample. Evaporate acidified sample to approximately 20ml. ~~Weigh an acidified and filtered aliquot of the sample into a 50 ml centrifuge tube.~~ Enter appropriate information on data sheet (appendix 10.1). Run a blank and spike with each set of samples.

6.7 After the sample has been passed through the column, wash the column with 50 ml of 10 M HCl, then 50 ml of 6 M HCl - 0.05 M HF followed by ~~100 ml of 4 M HCl~~ 50ml of 6M HCl. Discard the effluents.

6.8 Elute the iron with approximately ~~75 ml~~ 35ml of 0.01 M HCl ~~(discard the first 10 ml)~~ and collect the yellow band indicative of Fe^{+3} in a beaker. Do not allow the column to run dry.

Submitted by: James Hollard

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. C. Cunningham, Laboratory Director

Approved by: Gerald K. Prince, QA/QC/Director, ITAS

000341

6.11 Add 6 drops H_3PO_4 to the solution and heat the solution on a hot plate until it ~~clears~~ becomes colorless. Note: this will drive off any excess HCl and water because the boiling point of HCl and water is lower than that of H_3PO_4. Do not allow the solution to be baked dry or to exceed 0.5mL. Add 1mL of water and swirl in the glass vial. Cool and add 15 ml liquid scintillation cocktail. Submit for counting.

TITLE: Determination of Iron-55	NO: RSL-312	PAGE: 1
	REVISION: 0	DATE: 4-10-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Iron-55 activity.

2.0 SCOPE

The procedure applies to all water samples requiring Iron-55 activity analysis.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring Iron-55 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.2 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.3 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0 - 160.0000g)

5.1.2 pH meter

5.1.3 Hot plate

5.1.4 Anion exchange columns

5.1.5 Assorted tools and routine laboratory equipment

5.2 Materials

5.2.1 Liquid scintillation vials (glass; 20 ml)

5.2.2 Resin - AG1-X8, 100-200 mesh; 15 ml equilibrated with 10 M HCl.

5.3 Reagents

5.3.1 Commercially available liquid scintillation cocktail

5.3.2 Fe Carrier: Dissolve 5.00 grams of metallic iron (ACS reagent grade) in 300 ml of 6 M hydrochloric acid (HCl) and filter. Dilute to 1 liter with demineralized water and calibrate by using an Atomic Absorption Spectrophotometer or purchase commercially.

5.3.3 Manganese Carrier, 2 mg/ml: Dissolve 0.72 grams of ACS reagent grade manganese chloride ($MnCl_2 \cdot 4H_2O$) in 100 ml of 0.5 M hydrochloric acid (HCl).

5.3.4 Cobalt Carrier, 2 mg/ml: Dissolve 0.99 grams of ACS reagent grade cobalt chloride ($CoCl_2 \cdot 6H_2O$) in 100 ml of 0.5 M HCl.

5.3.5 Cesium Carrier, 2 mg/ml: Dissolve 0.25 grams of ACS reagent grade cesium chloride (CsCl) in 100 ml of demineralized water.

5.3.6 Zirconium Carrier, 1 mg/ml, commercial Zr standard (1000 ppm).

5.3.7 Niobium Carrier, 2 mg/ml: Dissolve 2 grams of niobium wire in 90 mls of demineralized water containing 10 ml of concentrated HF in a plastic bottle.

5.3.8 Zinc Carrier, 2 mg/ml: Dissolve 2 grams of zinc metal in 5 ml of 10 M HCl. Add 95 ml of demineralized water.

5.3.9 6 M Sodium Hydroxide (NaOH): Dissolve 24 grams ACS reagent grade NaOH in 90 ml demineralized water, allow to cool and dilute to 100 ml.

5.3.10 10 M Hydrochloric Acid (HCl): Add 833 ml of concentrated HCl to 167 ml of demineralized water in a 1 liter bottle.

5.3.11 6 M Hydrochloric Acid (HCl) - 0.05 M Hydrofluoric Acid (HF): Add 500 ml of concentrated HCl and 2 ml of 49 percent HF to 498 ml of demineralized water in a plastic or teflon container.

5.3.12 4 M Hydrochloric Acid (HCl): Add 333 ml of concentrated HCl to 667 ml of demineralized water in 1 liter bottle.

5.3.13 0.5 M Hydrochloric Acid (HCl): Add 42 ml of concentrated HCl to 958 ml of demineralized water in a 1 liter bottle.

5.3.14 0.01 M Hydrochloric Acid (HCl): Add 20 ml of 0.5 M HCl to 980 ml of demineralized water in a 1 liter bottle.

5.3.15 6 M Hydrochloric Acid (HCl): Add 500 ml of concentrated HCl to 500 ml of demineralized water in a 1 liter bottle. Mix thoroughly.

5.3.16 Ammonium Hydroxide (NH_4OH), ACS reagent grade, concentrated

5.3.17 0.5 M $(NH_4)_2HPO_4$: Dissolve 66 grams of $(NH_4)_2HPO_4$ in 1000 ml of water in a 1 liter bottle.

5.3.18 Phosphoric Acid (H_3PO_4), ACS reagent grade, concentrated

5.3.19 Hydrochloric Acid (HCl), ACS reagent grade, concentrated

5.3.20 Hydrofluoric Acid (HF), ACS reagent grade, concentrated

6.0 PROCEDURE

6.1 Weigh an acidified and filtered aliquot of the sample into a 50 ml centrifuge tube. Enter appropriate information on data sheet (appendix 10.1). Run a blank and spike with each set of samples.

6.2 Add 1 ml (5 mg) of the Fe carrier solution and mix well.

6.3 Add 6 M NaOH, mix and heat to precipitate iron hydroxide.

6.4 Centrifuge and decant the supernate.

6.5 Wash the precipitate with 40 ml of hot water and decant the supernate.

6.6 Dissolve the precipitate in 10 M HCl and add 2 mg each of the appropriate hold back carriers (Co^{+2}, Zn^{+2}, Zr^{+4}, Nb^{+}, Mn^{+2}, and Cs^{+} carriers). Transfer the solution with 10 M HCl washes to a beaker and dilute to approximately 50 ml with 10 M HCl. Pass the solution through the anion exchange column at 5 to 8 ml/min.

6.7 After the sample has been passed through the column, wash the column with 50 ml of 10 M HCl, then 50 ml of 6 M HCl - 0.05 M HF followed by 100 ml of 4 M HCl. Discard the effluents.

6.8 Elute the iron with approximately 75 ml of 0.01 M HCl (discard the first 10 ml) and collect the yellow band indicative of Fe^{+3} in a beaker. Do not allow the column to run dry.

6.9 Add 10 ml of 0.5 M $(NH_4)_2HPO_4$ to the beaker and dropwise add concentrated NH_4OH until the pH = 3.0. Gently heat the sample on a hot plate to completely precipitate the iron as $Fe(PO_4)$. Centrifuge, decant and wash the precipitate with hot demineralized water.

6.10 Dissolve the precipitate with a minimal amount (approximately 2 ml) of 6 M HCl. Transfer the solution with 2 ml of demineralized water to a clean, glass scintillation vial.

6.11 Add 6 drops H_3PO_4 to the solution and heat the solution on a hot plate until it clears. Cool and add 15 ml liquid scintillation cocktail. Submit for counting.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 6% at 100 ηCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technician performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Balances shall be checked to ensure calibration dates have not expired.

8.4.2 Balances shall be in control as determined by routine balance check procedure.

8.4.3 The liquid scintillation counter shall be calibrated prior to use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Iron-55 Activity (nCi/l) = ACT

$$= \frac{TSC5 - BKG - (TSC9*C)}{E*Y*VOL*T*2.22 \times 10^3}$$

Iron-55 Activity Error (nCi/l) = ERR

$$= ACT* \frac{TSC5}{[TSC5 - BKG - (TSC9*C)]^2} + 0.005$$

Iron-55 Lower Limit of Detection (nCi/l) = LLD

$$= \frac{4.65* \ BKG + 3}{E*Y*VOL*T*2.22 \times 10^3}$$

Where:

C	= cross talk factor obtained from iron-59 quench curve,
E	= fractional iron-55 efficiency obtained from the iron-55 quench curve,
Y	= chemical recovery from spike data
VOL	= sample volume (l)
TSC5	= gross counts in iron-55 channel
TSC9	= gross counts in iron-59 channel
BKG	= gross background counts in iron-55 channel
T	= counting time
465, 3	= constants
0.005	= estimate of relative error from all other sources
2.22×10^3	= conversion from dpm to nCi

TITLE: Determination of Iron-55	NO: RSL-312	PAGE: 7
	REVISION: 0	DATE: 4-10-87

10.0 APPENDICES

TITLE: Determination of Iron-55	NO: RSL-312	PAGE: 8
	REVISION: 0	DATE: 4-10-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-313

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Direct Counting of Tritium, Carbon-14 and Calcium-45 by Liquid Scintillation Counting Systems

APPROVED: James T. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul [illegible] DATE: 5/4/87
ITAS QA Director

CONCURRED: John A. Auxier DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED ISSUED: COPY

REVISION: 0				
DATE: 4-10-87				

429-1-87

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-313, R0
Direct Counting of Tritium, Carbon-14 & Calcium-45 by Liquid Scintillation Counting Systems.
Description of experiment or method Correct equations to calculate Error and Lower Limit of Detection

Time Period From: 4/10/87 To: 8/11/87 Samples Affected All between 4-10-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: [signature]

Approved by: [signature], Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature], QA/QC/Director, ITAS

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG) * SD}{(SC - TSC) * SVOL * 2.22}$$

Activity Error (pCi/l) = ERR

$$= ACT * \left[\frac{TSC}{N*(TSC - BKG)^2} + \frac{SC}{N*(SC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{(4.65 * (BKG)^{1/2} + 3) * SD}{(SC - TSC) * 2.22 * SVOL * N}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
SD = Standard dpm
SC = Standard counts, average N counts
SVOL = Sample volume (l)
N = Number of counts averaged
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure Yes X No ( )

If yes enter procedure number to be supplemented or replaced and provide brief description

Procedure Title & Number (if exists) 8.1, 8.2, 8.3 DIRECT COUNTING OF LABORATORY SAMPLES AND SPIKED LIQUID SCINTILLATION COUNTING SAMPLES

Description of experiment or method Allow for sample volume variability to meet detection limit requirements

Issue of 8.1, 8.3, 8.4

Time Period From: 8/19/87 To: ~~/ /~~ Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

6.2 Pipette two ~~3 ml~~ portions of the sample into counting vials containing ~~18 ml of~~ scintillation cocktail. Utilize appropriate sample volume up to a 10mL limit. Adjust scintillation cocktail volume for a total volume of approximately 20mL. To one vial, add the appropriate amount of internal standard solution used for the required analysis. Mix thoroughly. Prepare a spike and blank sample with each set of samples.

6.4 Record the data on the appropriate data sheet and calculate the results as shown in section 9.0.

Submitted by: James A. Willard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000354

PROCEDURE DEVELOPMENT/CHANGE

Is this a new procedure? Yes (X) No ( )

If yes, what is the number to reference insertion point? 9.0

Procedure Title & Number (if exists) RSL-313, R0

Direct Counting of Tritium, Carbon-14 and Cesium-137 by Liquid Scintillation Counting System

Description of experiment or method: Correct calculation section

Time Period From: 5/22/87 To: Issue of R1 Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 11-16-87

9.0 CALCULATIONS

Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG) * SD}{(SC - TSC) * SVOL * 2.22}$$

Activity Error (pCi/l) = ERR

$$= ACT * \left[\frac{(TSC + BKG)}{N*(TSC - BKG)^2} + \frac{(SC + BKG)}{N*(SC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{(4.65 * (BKG)^{1/2} + 3) * SD}{(SC - TSC) * 2.22 * SVOL * (N)^{1/2}}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
SD = Standard dpm
SC = Standard counts, average N counts
SVOL = Sample volume (l)
N = Number of counts averaged
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

Submitted by: James Hilliard

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. A. Auger, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS
Pam 11/20/87

000355

TITLE: Direct Counting of Tritium, Carbon-14 and Calcium-45 by Liquid Scintillation Counting Systems	NO: RSL-313	PAGE: 1
	REVISION: 0	DATE: 4-10-87

1.0 PURPOSE

This procedure provides an analytical method for direct counting of tritium, carbon-14 and calcium-45 by liquid scintillation counting systems.

2.0 SCOPE

This procedure applies to all samples requiring direct counting by liquid scintillation counting systems.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring tritium, carbon-14 and calcium-45 activity determination by liquid scintillation counting systems.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.2 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.3 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

None

5.2 Materials

5.2.1 Polyethylene liquid scintillation vials (20 ml capacity)

5.2.2 Routine laboratory equipment

5.3 Reagents

5.3.1 Internal standards for tritium, carbon-14 and calcium-45.

5.3.2 Liquid scintillation cocktail (commercial)

6.0 PROCEDURE

6.1 Obtain and fill out the appropriate data sheet (see appendix 10.1).

6.2 Pipette two 3 ml portions of the sample into counting vials containing 18 ml of scintillation cocktail. To one vial, add the appropriate amount of internal standard solution used for the required analysis. Mix thoroughly. Prepare a spike and blank sample with each set of samples.

6.3 Submit sample to counting room for counting. Obtain the required number of counts.

6.4 Record the data on the appropraite data sheet and calculate the results as shown in section 9.0.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 10% at 4000 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG) \ *SD}{(SC - TSC)*SVOL*2.22}$$

Activity Error (pCi/l) = ERR

$$= ACT* \quad \frac{TSC}{N*(TSC - BKG)^2} + \frac{SC}{N*(SC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{(4.65* \ BKG + 3) \ *SD}{(SC - TSC) \ *2.22*SVOL* \ N}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
SD = Standard dpm
SC = Standard counts, average N counts
SVOL = Sample volume (l)
N = Number of counts averaged
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Direct Counting of Tritium, Carbon-14 and Calcium-45 by Liquid Scintillation Counting Systems	NO: RSL-313	PAGE: 4
	REVISION: 0	DATE: 4-10-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-314

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Polonium-210

APPROVED: James T. Harvey DATE: 8-27-87
Laboratory Manager

APPROVED: Paul Nill DATE: 8-27-87
ITAS QA Director

CONCURRED: John A. Auxier DATE: 8-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 5-08-87				

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (if exists): RSL-SOP-80

Determination of Polonium 210

Description of experiment or method: Correct calculation of LLD and error

Time Period From: 12/30/87 To: ++ Samples Affected: All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

Po-210 Activity Error (pCi/l) = ERR

$$= ACT * 2 * \left[\frac{TSC + BKG}{(TSC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65 * (BKG)^{1/2} + 3}{2.22 * EFF * SVOL * CTIME * Y}$$

TSC	= Sample counts
BKG	= Background counts
CTIME	= Count time (min)
EFF	= Counting efficiency
SVOL	= Sample volume (l)
Y	= Yield
2.22	= Conversion from dpm to pCi
4.65, 3	= Constants
0.005	= Estimate of relative error from all other sources

Submitted by: James A. Ellis

Approved by: James T. Harvey, Laboratory Manager

Approved by: J. G. Angus, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

000362

TITLE: Determination of Polonium-210	NO: RSL-314	PAGE: 1
	REVISION: 0	DATE: 5-08-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of polonium-210 in urine and environmental aqueous samples by distillation.

2.0 SCOPE

This procedure applies to all samples requiring polonium-210 activity determination.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring polonium-210 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Polonium", Radiochemical Purification Procedures for the Elements, M. Lindner, Ed., UCRL-14258, 1965.

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Alpha spectrometer counting System

5.1.2 Ion exchange columns (0.7 cm I.D. x 15 cm)

5.2 Materials

5.2.1 Stainless Steel planchets

5.2.2 Dowex anion exchange resin, AG 1-x 8, 100 - 200 mesh.

5.2.3 Routine laboratory equipment

5.3 Reagents

5.3.1 Hydrochloric acid (HCl), concentrated, ACS reagent grade

5.3.2 Nitric acid (HNO_3), concentrated, ACS reagent grade

5.3.3 Perchloric acid ($HClO_4$), concentrated, ACS reagent grade

5.3.4 8 M HCl: add 500 ml of concentrated HCl to 500 ml of demineralized water. Mix thoroughly.

5.3.5 0.1 M HCl: add 9 ml of concentrated HCl to 1000 ml of demineralized water. Mix thoroughly.

6.0 PROCEDURE

6.1 To the sample aliquot in an appropriate size beaker, reduce the volume to about 5 ml, or to the point at which "salting out" occurs if large amounts of solids are present. Add concentrated HCl until the concentration of HCl is about 7-8 M. Run two blanks and two spikes with each set of samples. Record data on appropriate data sheet (appendix 10.1).

6.2 Pass the sample solution through a Dowex 1 x 8 anion exchange column previously prepared by being washed sequentially with 30 ml each of concentrated HCl, 0.1 M HCl and finally, concentrated HCl. After the sample solution has passed through, sequentially wash the column with 25 ml concentrated HCl, 25 ml 8 M HCl, 25 ml 0.1 M HCl, 20 ml concentrated HCl and finally, with 10 ml 0.1 M HCl.

6.3 Pass about 30 ml of concentrated HNO_3 through the column over a period of at least 20-30 minutes. Collect eluate.

6.4 To the eluate add 3 ml concentrated $HClO_4$ and fume until about 1 ml of concentrated $HClO_4$ remains. Cool and dilute to 8-10 ml with water to a pH approximately 1.0. Check pH with pH meter.

6.5 With continuous stirring, plate the polonium on a stainless steel planchet for about 2 hours.

6.6 Remove disc, wash twice with water, and rinse with acetone. Dry under a heat lamp at low temperature for several minutes.

6.7 Count by alpha spectroscopy for the necessary count time.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 15% at 1 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Po-210 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*Y}$$

Po-210 Activity Error (pCi/l) = ERR

$$= ACT^* \left(\frac{TSC}{(TSC - BKG)^2} + 0.005 \right)^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65^* \quad BKG^{1/2} + 3}{2.22^*EFF^*SVOL^*CTIME^*Y}$$

TSC = Sample counts
BKG = Background counts
CTIME = Count time (min)
EFF = Counting efficiency
SVOL = Sample volume (l)
Y = Yield
2.22 = Conversion from dpm to pCi
4.65,
3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Polonium-210	NO: RSL-314	PAGE: 6
	REVISION: 0	DATE: 5-08-87

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____ DATE RECEIVED _____

SAMPLE IDENTIFICATION _____ P.O. NUMBER _____

LABORATORY NUMBER _____ SURVEY INSTRUMENT READING _____ mR/hr.

SAMPLE MATRIX _____ WIPE COUNTING RESULT _____ cpm

COLLECTION DATE AND TIME: _____ _____ OF _____ PAGES

START _____ SPECIAL INSTRUCTIONS:

STOP _____

SAMPLE VOLUME _____

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

361-9-86

000368

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-315

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Lead-210

APPROVED: James T. Harvey DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Mills DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 5-08-87				

429-1-87

3222

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-315, RO
Determination of Lead-210

Description of experiment or method Correct equation to calculate Error and Lower Limit of Detection

Time Period From: 5/8/87 To: 8/11/87 Samples Affected All between 5-8-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

see attached

Submitted by: James McMillan

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cumpian, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Pb-210 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME}$$

Pb-210 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* \left(BKG\right)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME* N}$$

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.3, 5.3.10, 6.2, 6.3

Procedure Title & Number (If exists) RSL-315, RO

Determination of Lead-210

Description of experiment or method Correct reagent list concentrations of solutions and aliquots used.

Issue of RSL-315, R1

Time Period From: 10/23/87 To: ++ Samples Affected All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 10-23-87

5.3.3 2 M H_2SO_4: add 55 ml of concentrated sulfuric acid to 500 ml of demineralized water and dilute to 1 ~~beta~~ liter with demineralized water. Mix throughly.

5.3.10 Nitric Acid (HNO_3), concentrated, ACS reagent grade.

6.2 To the solution add 1ml of lead carrier. Add 2 ml concentrated H_2SO_4 and boil until H_2SO_4 fumes, cool. Very cautiously add water with vigorous agitation, transfer to a centrifuge cone, centrifuge, and discard supernate. Wash precipitate once with 15 ml ~~1~~ 2 M H_2SO_4.

6.3 Dissolve the $PbSO_4$ in approximately 2 ml 6 M NH_4Ac. Dilute to 10 ml with water and saturate with H_2S. Centrifuge and wash the PbS once with approximately 20ml of 0.5 M NH_4Ac saturated with H_2S.

Submitted by: [signature]

Approved by: [signature], Laboratory Manager

Approved by: [signature], Laboratory Director

Approved by: [signature] 11/5/87, QA/QC/Director, ITAS

6.4 Dissolve the PbS in about 1 ml concentrated HCl, transfer to a small beaker and fume to dryness. Take up the residue in 0.3 M HCl, heating, 20ml if necessary, to dissolve the residue. Transfer to a centrifuge cone. Saturate the solution with H_2S. Centrifuge and wash the precipitate with approximately 20 ml of 0.3 M HCl saturated with H_2S.

6.5 To the precipitate add a few drops concentrated HNO_3, transfer to a small beaker and boil to near-dryness. Add 10 ml fuming HNO_3. Transfer to a centrifuge cone. Cool in an ice bath for 10 minutes. Centrifuge and wash precipitate with 10 ml fuming HNO_3, discarding supernate and wash.

6.6 To the precipitate, add about 2 ml concentrated HCl, transfer to a small beaker and fume to dryness. To the residue, add 1-2 ml 6 M CH_3COONH_4 and warm, if necessary, to bring about dissolution. Transfer to centrifuge tube with H_2O and dilute to 10 ml. Saturate with H_2S. Centrifuge and wash precipitate once with approximately 0.5 M CH_3COONH_4 saturated with H_2S. 20ml

RSL-315.RO.CH

PROCEDURE DEVELOPMENT CHANGE

Supplement to Existing Procedure? (Yes, No)
If yes, enter procedure number to reference insertion point: 9.0

Procedure Title & Number (if exists): RSL-315 RO

Determination of Lead-210

Description of experiment or method: Correct calculation section

Time Period From: 6/23/87 To: Issue of R1 Samples Affected: All

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 11-16-87

9.0 CALCULATIONS

Pb-210 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME}$$

Pb-210 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{(TSC+BKG)}{N*(TSC - BKG)^2} + \frac{(STC+BKG)}{N*(STC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{[4.65* (BKG)^{1/2}+3]}{2.22*EFF*SVOL*CTIME* (N)^{1/2}}$$

Submitted by: James Hilliard

Approved by: James T. Harvey, Laboratory Manager

Approved by: [illegible], Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS
for 11/20/87

TITLE: Determination of Lead-210	NO: RSL-315	PAGE: 1
	REVISION: 0	DATE: 5-08-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of lead-210 in urine and environmental aqueous samples by distillation.

2.0 SCOPE

This procedure applies to all samples requiring lead-210 activity determination.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring lead-210 activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

4.3.1 "Lead", Radiochemical Purification Procedures for the Elements, M. Lindler, Ed., UCRL-14258, 1965.

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Liquid Scintillation Counter

5.2 Materials

5.2.1 Polyethylene liquid scintillation vials (20 ml capacity)

TITLE: Determination of Lead-210	NO: RSL-315	PAGE: 3
	REVISION: 0	DATE: 5-08-87

5.2.2 Routine laboratory equipment

5.3 Reagents

5.3.1 Lead carrier, 10 mg/ml Pb++: add 16 g of lead nitrate [$Pb(NO_3)_2$] to 100 ml of demineralized water. Mix throughly.

5.3.2 Sulfuric acid (H_2SO_4), concentrated, ACS Reagent grade

5.3.3 2 M H_2SO_4: add 55 ml of concentrated sulfuric acid to 500 ml of demineralized water and dilute to 1 beta with demineralized water. Mix throughly.

5.3.4 6 M ammonium acetate (CH_3COONH_4): add 462 grams of ammonium acetate to 500 ml of demineralized water and dilute to 1 liter. Mix throughly.

5.3.5 0.5 M ammonium acetate: add 83 ml of 6 M ammonium acetate to 917 ml of demineralized water. Mix throughly.

5.3.6 Hydrochloric acid, concentrated, ACS reagent grade.

5.3.7 0.3 M HCl: add 25 ml of concentrated HCl to 975 ml of demineralized water. Mix throughly.

5.3.8 Hydrogen Sulfide gas (H_2S), commercially prepared.

5.3.9 Nitric acid (HNO_3), 90% Fuming, ACS reagent grade.

6.0 PROCEDURE

6.1 Reduce a sample aliquot to a small volume (~25 ml). Record the sample information on the data sheet (appendix 10.1). Run two spikes and two blanks with each set of samples.

6.2 To the solution add lead carrier. Add 2 ml concentrated H_2SO_4 and boil until H_2SO_4 fumes cool. Very cautiously add water with vigorous agitation, centrifuge, and discard supernate. Wash precipitate once with 15 ml 1 M H_2SO_4.

6.3 Dissolve the $PbSO_4$ in approximately 2 ml 6 M NH_4Ac. Dilute to 10 ml with water and saturate with H_2S. Centrifuge and wash the PbS once with approximately 0.5 M NH_4Ac saturated with H_2S.

6.4 Dissolve the PbS in about 1 ml 12 M HCl, and fume to dryness. Take up the residue in 0.3 M HCl, heating, if necessary, to dissolve the residue. Saturate the solution with H_2S. Centrifuge and wash the precipitate with 0.3 M HCl saturated with H_2S.

6.5 To the precipitate add a few drops 16 M HNO_3 and boil to near-dryness. Add 10 ml fuming HNO_3. Cool in an ice bath for 10 minutes. Centrifuge and wash precipitate with 10 ml fuming HNO_3, discarding supernate and wash.

6.6 To the precipitate, add about 2 ml concentrated HCl and fume to dryness. To the residue add 1-2 ml 6 M CH_3COONH_4 and warm, if necessary, to bring about dissolution. Dilute to 10 ml and saturate with H_2S. Centrifuge and wash precipitate once with approximately 0.3 M HCl saturated with H_2S.

6.7 Repeat Steps 6.4 and 6.5.

6.8 To the final $Pb(NO_3)_2$ add about 2 ml demineralized water.

6.9 Transfer the dissolved residue to a scintillation vial with two 2 ml demineralized water rinse.

6.10 Add 15 ml of cocktail. Shake and submit for counting.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 10% at 20 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Liquid scintillation counter shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Pb-210 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME}$$

Pb-210 Activity Error (pCi/l) = ERR

$$= ACT* \quad \frac{TSC}{N*(TSC - BKG)^2} + \frac{STC}{N*(STC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*SVOL*CTIME* \quad N}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency

$$= \frac{STC - BKG}{STDPM}$$

SVOL = Sample volume (l)
N = Number of counts averaged
STC = Standards counts, average N counts
STDPM = Standard activity (dpm)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Lead-210	NO: RSL-315	PAGE: 7
	REVISION: 0	DATE: 5-8-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-316

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Ruthenium-106

APPROVED: James T. Harvey DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Mills DATE: 6-8-87
ITAS QA Director

CONCURRED: John C. Cuxin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 9.0

Procedure Title & Number (If exists) RSL-316, RO
Determination of Ruthenium-106

Description of experiment or method Correct equation to calculate Error and Lower Limit of Detection

Time Period From: 4/14/87 To: 8/11/87 Samples Affected All between 4-14-87 and 8-11-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE

8-11-87

See attached

Submitted by: James Millard

Approved by: John R. Frazier, Laboratory Manager

Approved by: John A. Cuffier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

9.0 CALCULATIONS

Ru-106 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*Y}$$

Ru-106 Activity Error (pCi/l) = ERR

$$= ACT* \left[\frac{TSC}{(TSC - BKG)^2} + 0.005 \right]^{1/2}$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{\left[4.65* (BKG)^{1/2} + 3\right]}{2.22*EFF*SVOL*CTIME*Y}$$

TSC	= Sample counts, average N counts
BKG	= Background counts, average N counts
CTIME	= Count time (min)
EFF	= Counting efficiency from curve
Y	= Yield
SVOL	= Sample volume (l)
2.22	= Conversion from dpm to pCi
4.65, 3	= Constants
0.005	= Estimate of relative error from all other sources

10.0 APPENDICES

RSL-316, R0, CH-2 with

page 1 of 1

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PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.3

Procedure Title & Number (If exists) RSL-316, R0

DETERMINATION OF RUTHENIUM-106

Description of experiment or method Procedure clarification to allow for the use of currently available reagent

Issue of RSL-316, R1

Time Period From: 8/19/81 To: ~~/ /~~ Samples Affected All from 8-19-81

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-81

(CH_3CH_2OH), 89% ACS

5.3.3. Ethanol, ~~95%~~, Reagent grade

Submitted by: James M. Dillard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Frazier, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

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TITLE: Determination of Ruthenium-106	NO: RSL-316	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of ruthenium-106 in urine and environmental aqueous samples by distillation.

2.0 SCOPE

This procedure applies to all liquid samples requiring ruthenium-106 analysis by distillation.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring ruthenium-106 determination by distillation.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Alpha/Beta counting system

5.2 Materials

5.2.1 Stainless steel planchets

5.2.2 Filter paper, 47 mm, glass fiber or Whatman 42, as necessary

5.2.3 Routine laboratory equipment

5.3 Reagents

5.3.1 5 M Sodium Hydroxide (NaOH), ACS reagent grade solution commercially prepared

5.3.2 Cobalt carrier, 5 mg/ml: add 2.5 g of cobalt nitrate [$Co(NO_3)_2 \cdot 6H_2O$] to 100 ml of demineralized water. Mix thoroughly.

5.3.3. Ethanol, 95%, Reagent grade

5.3.4 Hydrochloric acid (HCl), concentrated, ACS reagent grade

5.3.5 Phosphoric acid (H_3PO_4), concentrated, ACS reagent grade

5.3.6 0.5 M potassium permanganate (KMn_2O_4): add 7.9 grams of potassium permanganate to 100 ml of demineralized water. Mix thoroughly.

5.3.7 Ruthenium carrier, 10 ml/ml: add 3.3 g of ruthenium tetrachloride ($RuCl_4 \cdot 5H_2O$) to 100 ml of demineralized water. Standardize.

5.3.8 Nitrogen gas, commercial

6.0 PROCEDURE

6.1 Pre-concentrate a sample aliquot to approximately 50 ml. Record sample information on appropriate data sheet (appendix 10.1).

6.2 Place sample in a special ruthenium distillation flask (appendix 10.2). Add 2.0 ml ruthenium carrier and 1 ml cobalt carrier.

6.3 Connect the ruthenium still to a nitrogen inlet and insert the distilling end into a centrifuge containing 15 ml of 6 M NaOH in an ice bath.

6.4 To distillation flask add 3 ml concentrated H_3PO_4 and 3 ml 0.5 M $KMnO_4$, and immediately close system.

6.5 Pass N_2 gently through system to drive volatile ruthenium into the NaOH solution.

6.6 Gently heat to boiling and continue heating for 10-15 minutes. Turn off N_2, disconnect apparatus and discard contents of still.

6.7 Add 3-5 ml ethanol to distillate, stir, boil to precipitate RuO_2 and cool. Centrifuge and discard supernatant.

6.8 Suspend precipitate in 10 ml water containing 1 ml 5 M NaOH. Boil, cool, centrifuge and discard supernatant.

6.9 Transfer precipitate to a tared glass-fiber filter with 15 ml water. Wash with successive portions of water and ethanol.

6.10 Dry, weigh and submit for counting.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 10% at 40 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Alpha/Beta counting system

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

TITLE: Determination of Ruthenium-106	NO: RSL-316	PAGE: 5
	REVISION: 0	DATE: 4-14-87

9.0 CALCULATIONS

Ru-106 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*SVOL*CTIME*Y}$$

Ru-106 Activity Error (pCi/l) = ERR

$$= ACT* \frac{TSC}{(TSC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*EFF*SVOL*CTIME*Y}$$

TSC = Sample counts, average N counts
BKG = Background counts, average N counts
CTIME = Count time (min)
EFF = Counting efficiency from curve
Y = Yield
SVOL = Sample volume (l)
2.22 = Conversion from dpm to pCi
4.65, 3 = Constants
0.005 = Estimate of relative error from all other sources

10.0 APPENDICES

TITLE: Determination of Ruthenium-106	NO: RSL-316	PAGE: 6
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK SHEET

CUSTOMER _____

SAMPLE IDENTIFICATION _____

LABORATORY NUMBER _____

SAMPLE MATRIX _____

COLLECTION DATE AND TIME: _____

START _____

STOP _____

SAMPLE VOLUME _____

DATE RECEIVED _____

P.O. NUMBER _____

SURVEY INSTRUMENT READING _____ mR/hr.

WIPE COUNTING RESULT _____ cpm

_____ OF _____ PAGES

SPECIAL INSTRUCTIONS:

ALIQUOT						
ANALYSIS						
DATE AND TIME OF ANALYSIS						
% RECOVERY						
COUNTER NO.						
COUNTER BKG c/m						
COUNTER EFF. d/c						
COUNT START DATE AND TIME						
COUNT STOP DATE AND TIME						
COUNT TIME-MIN.						
TOTAL COUNTS						
GROSS c/m						
BKG c/m						
NET c/m						
d/m PER ALIQUOT						

RESULTS REQUESTED IN: _____

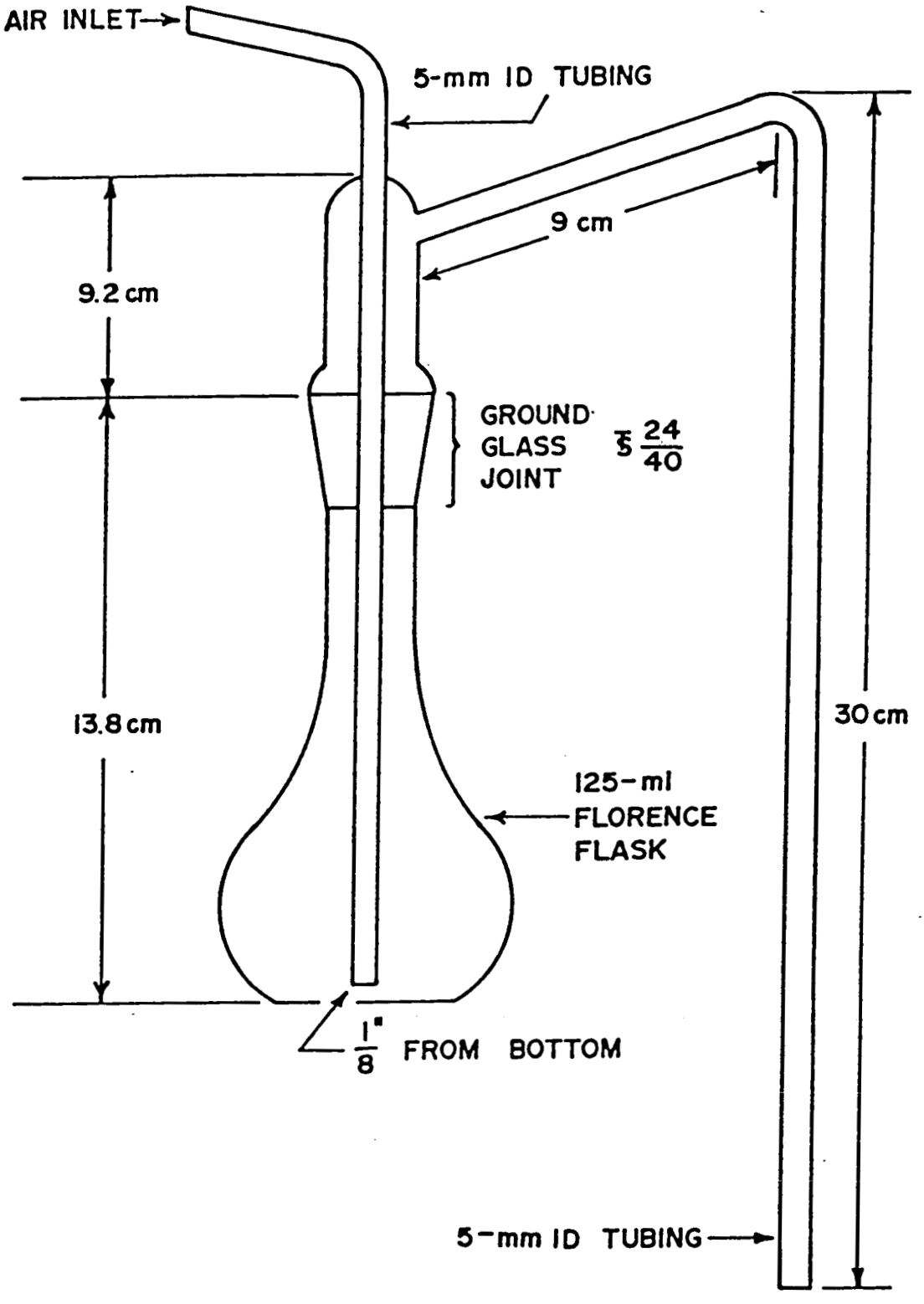
REMARKS AND CALCULATIONS

SAMPLE PREPARATION						
SAMPLE ANALYSIS						
SAMPLE COUNTING						
CALCULATION						
DATA REVIEW						
APPROVAL						

430-1-87

TITLE: Determination of Ruthenium-106	NO: RSL-316	PAGE: 7
	REVISION: 0	DATE: 4-14-87

APPENDIX 10.2



Distillation Apparatus for Ruthenium Analysis

430 1 87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-317

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Cerium Isotopes

APPROVED: James T. Harney DATE: 6-3-87
Laboratory Manager

APPROVED: Paul Miller DATE: 6-8-87
ITAS QA Director

CONCURRED: John A. Cuxier DATE: 6-12-87
RSL Director

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DATE: 5-08-87				

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PROCEDURE DEVELOPMENT/CHANGE

Supplement to existing procedure? Yes (X) No ( )
If yes, enter paragraph number to reference insertion point: 5.3.3

Procedure Title & Number (If exists) RSL-317, R0

DETERMINATION OF CERIUM ISOTOPES

Description of experiment or method Procedure clarification to allow for use of currently available reagent

Time Period From: 8/19/87 To: ~~//~~ Issue of RSL-317, R1 Samples Affected All from 8-19-87

Procedure: (Test Eqpt., Data Recorded, Problems, Steps Used, etc.) DATE 8-19-87

5.3.3 Ethanol (CH_3CH_2OH), ~~95%~~ 99% ACS reagent grade

Submitted by: James M. Tullard

Approved by: James T. Harvey, Laboratory Manager

Approved by: John R. Fraser, Laboratory Director

Approved by: Cheryl D. Prince, QA/QC/Director, ITAS

TITLE: Determination of Cerium Isotopes	NO: RSL-317	PAGE: 1
	REVISION: 0	DATE: 5-08-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of cerium isotopes in urine and environmental aqueous samples.

2.0 SCOPE

This procedure applies to all samples requiring cerium isotope activity determination.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the analysis of all samples requiring cerium isotope activity determination.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of this procedure to personnel that are experienced with this procedure and with the equipment associated with implementation of this procedure.

3.3 It is the responsibility of the technician to follow this procedure and to report any abnormal results to the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Title 10, Code of Federal Regulations, Part 50, Appendix I, Numerical Guides for Design Objectives and Limiting Conditions for Operation to Meet the Criteria "As Low as is Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.1.2 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Programs for Monitoring Radioactivity in the Environs of Nuclear Power Plants.

4.1.3 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.1.4 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

4.1.5 ITAS Quality Assurance Manual.

4.1.6 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", H.L. Krieger and E.L. Whittaker, EPA-600/4-80-032, U.S. Environmental Protection Agency, August 1980.

4.4.2 "Annual Book of ASTM Standards", Section 11.02, Water and Environmental Technology, American Society for Testing and Materials, Philadelphia, PA 19103, latest edition.

4.4.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

4.4.4 "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements", L.A. Currie, NUREG/CR-4007, U.S. Nuclear Regulatory Commission, September 1984.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Germanium Counting System

5.2 Materials

5.2.1 Stainless Steel planchets

5.2.2 Filter paper, 47 mm, glass fiber

5.2.3 Anion Exchange Resin, 20 - 50 mesh and 100 - 200 mesh, AG2 x 8 and AG1 x 8, respectively

5.2.4 Routine laboratory equipment

5.3 Reagents

5.3.1 Cobalt carrier, 5 mg/ml: add 2.5 grams of cobalt nitrate [$Co(NO_3)_2 \cdot 6H_2O$] to 100 ml of demineralized water. Mix thoroughly.

5.3.2 Cerium carrier, 5 mg/ml: add 15 grams of cerous nitrate [$Ce(NO_3)_3 \cdot 6H_2O$] to 100 ml of demineralized water. Mix thoroughly. Standardize.

5.3.3 Ethanol (CH_3CH_2OH), 95%, reagent grade

5.3.4 Hydrochloric acid (HCl), concentrated, ACS reagent grade

5.3.5 6 M HCl: add 500 ml of concentrated hydrochloric acid to 500 ml of demineralized water. Mix thoroughly.

5.3.6 Hydrogen peroxide (H_2O_2), 30%, ACS reagent grade

5.3.7 Manganese carrier, 5 mg/ml: add 2.1 g of manganese chloride ($MnCl_2 \cdot 4H_2O$) to 100 ml of demineralized water. Add 0.1 ml of concentrated HCl and mix thoroughly.

5.3.8 Nitric acid (HNO_3), concentrated, ACS reagent grade

5.3.9 Potassium Bromate (KBrOg), solid

5.3.10 1 M KIO_3: add 22 g of potassium iodate (KIO_2) to 100 ml of demineralized water. Mix thoroughly. Potassium iodate wash solution: add 5 ml of 1 M KIO_3 solution to 100 ml of demineralized water. Mix thoroughly.

6.0 PROCEDURE

6.1 To an aqueous sample, add 1.0 ml cerium carrier, 0.5 ml of 6 M HCl and evaporate to dryness. Run a spike and a blank with each set of samples. Record the sample information on the appropriate data sheet (appendix 10.1).

6.2 Prepare a mixed anion exchange resin column as follows: Individually equilibrate 10 g Dowex 1-X8 anion resin (100 - 200 mesh) and 10 g Dowex 2-X8 anion resin (20 - 50 mesh) with 50 ml of concentrated HCl each. Into a glass column slurry Dowex 2-X8 to a height of 5 cm, then add the Dowex 1-X8 resin up to 10 cm.

6.3 Dissolve sample residue in 10 ml concentrated HCl and add 1 drop H_2O_2 to reduce the cerium to the cerous (+3) state.

6.4 Add 1 ml cobalt carrier to the solution and stir well. Pass over column at a rate of 0.25 ml/min. Collect effluent in a beaker.

6.5 Wash column with 15 ml concentrated HCl and add wash to effluent in the beaker.

6.6 Evaporate effluent almost to dryness to remove chlorides. Do not bake. Dissolve residue in 50 ml water, add 0.5 ml manganese carrier and 2.5 ml concentrated HNO_3.

6.7 Heat to boiling and slowly add 1 g $KBrO_3$. Boil until MnO_2 coagulates. Cool, centrifuge, and transfer into a clean beaker.

6.8 Heat solution to boiling and add a pinch of $KBrO_3$ to assure complete oxidation to the ceric (+4) state.

6.9 Add 20 ml KIO_3 solution, stir and boil until the $Ce(IO_3)_4$ coagulates. Cool, centrifuge and discard supernatant.

6.10 Wash precipitate twice with 15 ml KIO_3 wash solution and discard washings.

6.11 Transfer to a tared glass-fiber filter with KIO_3 wash solution.

6.12 Wash with successive portions of KIO_3 wash solution, water and ethanol.

6.13 Dry in oven at 110°C for 15 minutes, cool, weigh, mount and gamma count.

7.0 PRECISION AND ACCURACY

7.1 The standard deviation for a single determination is 15% at 50 pCi/l.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Laboratory Manager, or his designee, shall inspect the work of the technicians performing this procedure for completeness and adequate quality.

8.1.2 The technicians performing this procedure shall inspect worksheets for accuracy and completeness, shall inspect sample for proper volume and size, shall inspect labels and tags for accuracy, shall inspect equipment for proper operation, and shall inspect balances to ensure that calibration is not out of date.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Germanium detector system shall be calibrated before use.

8.5 Certification

8.5.1 This procedure shall be certified by running spiked samples through the procedure steps.

9.0 CALCULATIONS

Results are reported by the nuclide ID report from the germanium detector system.

10.0 APPENDICES