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RADIOLOGICAL SCIENCES LABORATORY RADIOANALYTICAL LABORATORY PROCEDURES VOL. II

04/21/87

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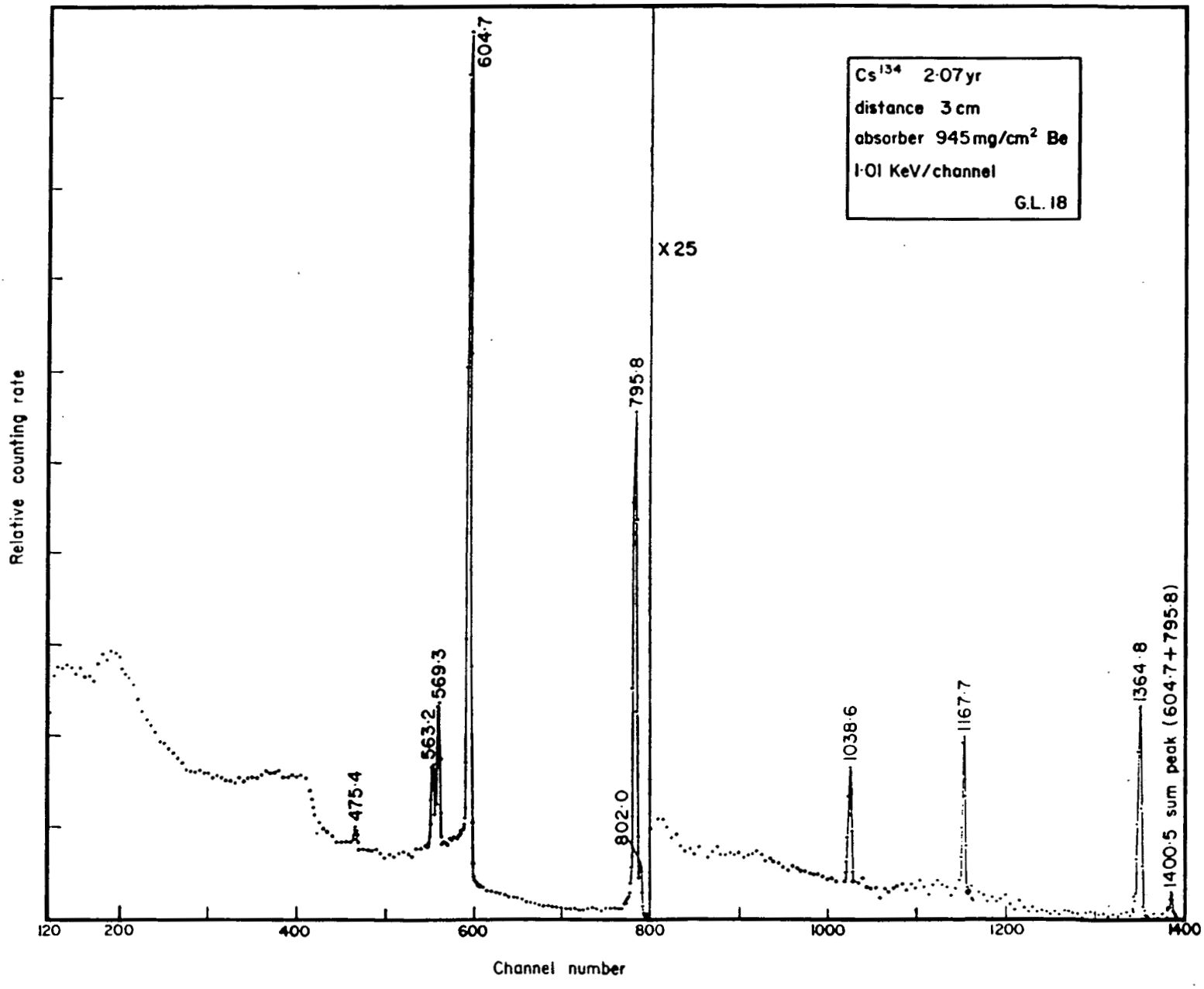
MANUAL

INTERNATIONAL TECHNOLOGY CORPORATION

3224

Radiological Sciences Laboratory

Radioanalytical Laboratory Procedures



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RESPONSIVE TO THE NEEDS OF ENVIRONMENTAL MANAGEMENT

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3224

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

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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
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RSL-307	Determination of I-131 Activity in Milk and Water Samples	0	5-22-87
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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 Systems	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 Gamma-ray Spectroscopy Systems	0	5-22-87
RSL-112	Operation of Germanium Gamma Spectroscopy System (ND 66/680)	0	6-23-87
RSL-202	Extraction of Iron from Soil or Metallic Samples	0	6-23-87
RSL-301	Determination of Promethium-147	0	6-23-87

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-002

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Standardization of Carrier Solutions

APPROVED: James T. Harvey 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

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DATE: 4-21-87				

429-1-87

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.

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.

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. Harvey DATE: 4-28-87
Laboratory Manager

APPROVED: Paul Reed 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: 4-06-87				

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 Wells DATE: 5/4/87
ITAS QA Director

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-06-87				

429-1-87

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

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.

TITLE: Calibration of Liquid Scintillation Counting systems	NO: RSL-103	PAGE: 5
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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

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

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	0	5-22-87
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, Carbon-14 and Calcium-45 by Liquid Scintillation Counting Systems	0	5-22-87
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-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 and 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-1001	Sample Receiving	0	5-22-87
RSL-1002	Radiological Screening and Classification of Mixed Waste Samples	0	5-22-87

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 Nieves DATE: 5/4/87
ITAS QA Director

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

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

429-1-87

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 1
	REVISION: 0	DATE: 4-06-87

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.

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

TITLE: Acquisition and Use of Standard Reference Material	NO: RSL-001	PAGE: 6
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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
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Appendix 10.2

CALCULATIONS FOR DILUTIONS

Log #: _____ Date: _____

Calculations Made by: _____

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

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-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 A. Caspian DATE: 4-28-87
RSL Director

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429-1-87

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. Cuffier DATE: 4-28-87
RSL Director

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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

TITLE: Calibration of Alpha Spectrometer Systems	NO: RSL-105	PAGE: 7
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ARPENDIX 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. Cooper DATE: 4-28-87
RSL Director

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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: Paul Wiley 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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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. Cuffin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

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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-112

RADIOANALYTICAL LABORATORY PROCEDURE

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

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

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

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

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 5-21-87				

429-1-87

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

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 Nielsen DATE: 6-4-87
ITAS QA Director

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

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REVISION: 0				
DATE: 4-14-87			000078	

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:

_____

_____

_____

_____

____ 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-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. Cuepin 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:

_____

_____

_____

_____

____ 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-111

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Linearity Check of Germanium Gamma-ray Spectroscopy Systems

APPROVED: James T. Harney 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

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:

_____

_____

_____

_____

____ 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-202

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Extraction of Iron from Soil or Metallic Samples

APPROVED: James T. Harvey 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

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 A. Cuyjian DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-20-87				

429-1-87

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						

367-9-86

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						

367-8-86

430-1 87

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				

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.

TITLE: Determination of Tritium in Aqueous Samples by Distillation	NO: RSL-302	PAGE: 4
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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
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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						

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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. Cupin DATE: 4-28-87
RSL Director

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DATE: 4-14-87				

TITLE: Determination of Phosphorus-32 Activity	NO: RSL-303	PAGE: 1
	REVISION: 0	DATE: 4-14-87

1.0 PURPOSE

This procedure provides an analytical method for the determination of Phosphorus-32.

2.0 SCOPE

This procedure applies to water samples requiring Phosphorus-32 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 water samples requiring phosphorus-32 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 "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.

4.4.2 "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.

4.4.4 J.D. Eakins, P.J. Gomm, and S. Jackson, Health Physics, 12, 593 (1966).

4.4.5 "The Radiochemistry of Phosphorus", W.T. Mullins and G.W. Leddicotte, NAS-NS-3056, National Academy of Science, March 1962.

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Analytical balance (0-160.0000 gram capacity)

5.1.2 Hot plate (stirring)

5.1.3 Heat lamp

5.1.4 Desiccator

5.1.5 Centrifuge (cone and bottle type)

5.1.6 Laboratory oven

5.1.7 Assorted tools and routine laboratory equipment

5.2 Materials

5.2.1 Whatman 41 filter paper

5.3 Reagents

5.3.1 Ammonium Hydroxide (NH_4OH): Concentrated, ACS reagent grade

5.3.2 1 M Ammonium Hydroxide: Dilute 66.5 ml of concentrated ammonium hydroxide to one liter with demineralized water.

5.3.3 Cobalt Carrier (5 mg/ml): Dissolve 4.94 g of ACS reagent grade cobalt nitrate [$Co(No_3)_2.6H_2O$] in demineralized water and dilute to 200 ml with demineralized water.

5.3.4 Hydrochloric Acid (HCl), Concentrated, ACS reagent grade

5.3.5 6 M Hydrochloric Acid: Add 500 ml of concentrated hydrochloric acid to 1 liter of demineralized water.

5.3.6 Hydrogen Peroxide (H_2O_2), 30 percent, ACS reagent grade

5.3.7 Magnesia Mixture: Dissolve 20.0g of ACS reagent grade ammonium chloride (NH_4Cl) in demineralized water, add 10.0g of magnesium chloride ($MgCl_2 \cdot 6H_2O$) and dilute to 100 ml with demineralized water.

5.3.8 Manganese Carrier, 5 mg/ml: Dissolve 1.80g of ACS reagent grade manganese chloride ($MnCl_2 \cdot 4H_2O$) in demineralized water and dilute to 100 ml with demineralized water.

5.3.9 Methyl Red Indicator, 0.1 percent: Dissolve 0.1g of methyl red indicator, (dimethylaminoazobenzene-carboxylic acid), in 18.6 ml of 0.02N-sodium hydroxide (NaOH) solution and dilute to 250 ml with demineralized water.

5.3.10 Molybdate-Vanadate Reagent: Dissolve 19.5g of ACS reagent grade ammonium molybdate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] in 80 ml of demineralized water. This is solution A. Dissolve 0.50g of reagent grade ammonium metavanadate (NH_4VO_3) in 50 ml of boiling demineralized water and allow solution to cool. This is solution B. Transfer solution B to a two-liter container and add 200 ml of concentrated HNO_3, dilute with demineralized water to approximately one liter and mix. Then add solution A to the two liter container and dilute to 2.0 liters with demineralized water. Mix well.

5.3.11 0.5 M Nitric Acid (HNO_3): Add 31.2 ml of concentrated HNO_3 to one liter of demineralized water.

5.3.12 6 M Nitric Acid: Add 375 ml of concentrated HNO_3 to one liter of demineralized water.

5.3.13 Phosphorus Carrier, 5 mg/ml: Dissolve 2.232g of ACS reagent grade sodium dihydrogen phosphate ($NaH_2PO_4H_2O$) in demineralized water and dilute to 100 ml with demineralized water.

5.3.14 0.1 M Hydrochloric Acid: Add 9 ml of concentrated hydrochloric acid to 1 liter of demineralized water.

5.3.15 Phosphorus 32 internal standard: Solution prepared from NBS traceable material containing 50-100 dpm per aliquot.

5.3.16 6 M Potassium Hydroxide(KOH): Dissolve 33.7g KOH in demineralized water and dilute to 100 ml with demineralized water.

5.3.17 0.02 M Sodium Hydroxide (NaOH): Dissolve 0.80g of NaOH in 100 ml of demineralized water and dilute to 1 liter with demineralized water.

5.3.18 Zirconium Carrier, 10 mg/ml: Dissolve 3.5g of ACS reagent grade zirconyl chloride ($ZrOCl_2 \cdot xH_2O$) in 0.1 M HCl and dilute to 100 ml with 0.1 M HCl.

6.0 PROCEDURE

6.1 Transfer two 100 ml aliquots to two 250 ml beakers for each sample. Spike one aliquot with internal standard solution. Also run a 100 ml demineralized water sample to determine the background count rate. Record the sample volume on the data sheet. See appendix 10.1.

6.2 Add 1 ml of 6 M HNO_3, 1.0 ml of phosphorus carrier and 0.5 ml of cobalt, zirconium, and manganese carriers.

TITLE: Determination of Phosphorus-32 Activity	NO: RSL-303	PAGE: 5
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6.3 Slowly add eight drops of 30 percent hydrogen peroxide followed by the addition of 6M KOH until the precipitation of hydroxides is complete. Do not add more than 15 ml of 6M KOH.

6.4 Heat the sample to near boiling to coagulate the precipitate. Then filter the precipitate while hot through Whatman 41 filter paper. Discard the filter paper and precipitate.

6.5 To the filtrate, add two drops of cobalt and zirconium carriers and four drops of 30 percent hydrogen peroxide. Heat to coagulate and then filter while hot through Whatman 41 filter paper. Discard filter paper and precipitate.

6.6 Add concentrated HCl to the filtrate until it is acidic. Boil the solution a few minutes to remove H_2O_2. Cool the beaker to room temperature then place in an ice bath for 30 minutes.

6.7 While in the ice bath add 3 ml of magnesia mixture, 1 drop of methyl red indicator and neutralize to the methyl red endpoint with concentrated NH_4OH. Let stand two minutes then add another 3 ml of concentrated NH_4OH. Allow the sample to remain in the ice bath for 1 hour then centrifuge and discard the supernatant (the white precipitate is $MgNH_4PO_4$). Since $MgNH_4PO_4$ precipitate is very flocculent, it may be necessary to centrifuge the precipitate in small cone bottom tubes, 40 ml at a time.

6.8 Wash the precipitate with 10 ml of 1 M NH_4OH, and if not done in 6.7 transfer to 50 ml centrifuge cone, centrifuge, and discard the wash solution.

6.9 Dissolve the precipitate with 1 ml of 6 M HCl. Transfer to an ice bath. Add 10 ml of demineralized water and 1 ml of magnesia mixture. Neutralize to the methyl red endpoint with concentrated NH_4OH then let stand for two minutes and add 3 ml more of concentrated NH_4OH. Allow sample to remain in the ice bath for 15 minutes then centrifuge and discard supernatant.

6.10 Wash the precipitate with 10 ml of 1 M NH_4OH, centrifuge and discard the wash solution.

6.11 Dissolve the final precipitate in a minimum amount (approximately 1 ml) of 6 M HCl.

6.12 Transfer the sample to a 20-ml scintillation vial using at least three washings of 0.5 M HNO_3; dilute to 20 ml with 0.5 M HNO_3. Mix well and allow sample to stand for one hour.

6.13 Submit samples, background, and standard to the counting room for counting on the liquid scintillation counter. Obtain required number of counts.

7.0 PRECISION AND ACCURACY

The standard deviation for a single determination is 10% 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

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1 Sample Size

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 The liquid scintillation counter shall be calibrated before use.

8.4.2 Balances shall be checked to ensure calibration dates have not expired.

8.4.3 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.

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9.0 CALCULATIONS

Phosphorus Activity (pCi/l) = ACT

$$= \frac{(TSC - BKG) * SD}{(SC - TSC) * SVOL * 2.22}$$

Phosphorus Activity Error (pCi/l) = ERR

$$= ACT * \frac{TSC}{N*(TSC - BKG)^2} + \frac{SC}{N*(SC - BKG)^2} + 0.005$$

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

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	REVISION: 0	DATE: 4-14-87

APPENDIX 10.1

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						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-304

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Selected Alpha Emitting Isotopes

APPROVED: James T. Harvey DATE: 4-30-87
Laboratory Manager

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

CONCURRED: John G. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

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

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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.

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						

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. Cuffin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

429-1-87

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)

TITLE: Determination of Strontium-89,90 in Water Samples	NO: RSL-305	PAGE: 3
	REVISION: 0	DATE: 4-14-87

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.

5.3.15 Sodium Carbonate Solution (Na_2CO_3): Dissolve 106 grams of sodium carbonate in water and dilute to 1 liter. Mix thoroughly.

5.3.16 1.5 M Sodium Chromate (Na_2CrO_4) Solution: Dissolve 513.2 grams of sodium chromate in water and dilute to 1 liter. Mix thoroughly.

5.3.17 0.2 M Sodium Diethylenetriaminepentaacetate (DTPA): Dissolve 39 grams of diethylenetriaminepentaacetate acid and 25 grams of sodium hydroxide pellets in 400 ml of water. Filter through a glass fiber filter with suction and dilute to 500 ml with demineralized water. Mix thoroughly.

5.3.18 10 M Sodium Hydroxide (NaOH): Dissolve 400 grams of sodium hydroxide pellets in water and dilute to 1 liter. Mix thoroughly.

5.3.19 0.7 M Sodium Sulfate (Na_2SO_4): Dissolve 100 grams of sodium sulfate in water and dilute to 1 liter. Mix thoroughly.

5.3.20 Strontium Nitrate Carrier Solution (50 mg/ml, Sr): Dissolve 120.77 grams of strontium nitrate [$Sr(NO_3)_2$] in water (approximately 250 ml), add 5 ml concentrated nitric acid and dilute to 1 liter with demineralized water. Mix thoroughly.

5.3.21 Strontium wash solution: Add 20 grams of sodium sulfate to 500 ml of water. Add 30 ml of glacial acetic acid and dilute to 1 liter with demineralized water. Mix thoroughly.

5.3.22 Yttrium Nitrate Carrier Solution (10 mg/ml, Y): Dissolve 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.

5.3.23 Yttrium Wash Solution: Dissolve 13 grams of oxalic acid in 500 ml of water, add 2 drops of MPC indicator and concentrated ammonium hydroxide to the yellow endpoint. Dilute to 1000 ml with water and mix thoroughly.

6.0 PROCEDURE

6.1 Record sample information on appropriate data sheet. See appendix 10.1.

6.2 Place the sample in a beaker of suitable size, add 2 drops of MCP indicator and acidify to the pink endpoint with nitric acid. Add 1.0 ml each of barium and strontium carriers and mix. Add 15 ml of 10 M NaOH and heat to near boiling. Add 10 ml of Na_2CO_3 solution, and stir slowly for 15 minutes.

TITLE: Determination of Iron-55	NO: RSL-312	PAGE: 3
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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 nCi/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

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						

TITLE: Determination of Strontium-89,90 in Water Samples	NO: RSL-305	PAGE: 5
	REVISION: 0	DATE: 4-14-87

6.3 Cool, decant and discard most of the clear supernate. Transfer the precipitate to a centrifuge tube with the remaining supernatant solution. Centrifuge and discard the supernate.

6.4 Rinse the beaker with 2 ml of concentrated nitric acid to transfer any precipitate that remains on the walls of the beaker and add it to the centrifuge tube.

6.5 Add 30 ml of fuming nitric acid. Cool in a cold water bath for 10 minutes with periodic stirring to precipitate strontium and barium nitrates. Centrifuge and discard the supernate. Invert the tube and drain off the excess nitric acid.

6.6 Wash the precipitate with 30 ml concentrated nitric acid. Agitate periodically for 15 minutes. Cool in a water bath, centrifuge and discard the supernate. Dissolve in 5 ml of water, 1 drop of phenolphthalein and 10 M sodium hydroxide dropwise with swirling to the red color change and 3 drops extra. Transfer the sample to a 50 ml conical or semi-conical centrifuge tube with 3 ml of 0.2 M DTPA.

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.

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 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.

6.9 After the ingrowth period, add 5 ml of 0.7 M sodium sulfate, and 3 ml of glacial acetic acid to reprecipitate the strontium sulfate. Record the time "T2" as the end of the ingrowth period. Heat the solution for 5 minutes, cool for 5 minutes. Centrifuge, then transfer supernatant to clean cone. Add 5 ml of strontium wash to cone with precipitate. Mix. Filter the precipitate on a tared filter. Use 3 ml of strontium wash solution to rinse the centrifuge tube and wash the precipitate with wash solution then with 2 ml of water. Wash the strontium sulfate precipitate with about 5 ml of acetone. Dry under a heat lamp, then desiccate to constant weight. Weigh to determine the strontium yield, mount and count the Sr-89,90. If yield <50%, see Laboratory Manager, or his designee. Record the counting time to the mid-point of the count and date as "T3".

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.

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

8.2.1 The following sections of this procedure contain acceptance criteria:

6.9 Yield, Sr-90

6.10 Yield, Y-90

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.

8.4.3 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.

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

$$= ACT* \frac{TYC}{(TYC - BKG)^2} + 0.0025$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \ BKG + 3}{2.22*CTIME*Y\text{-}YIELD*S\text{-}YIELD*Y\text{-}EFF*Y\text{-}GRD*Y\text{-}DEC*VOL}$$

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

$$= ACT \quad \frac{TSC}{(TSC - BKG)^2} + \frac{TYC}{(TYC-BKG)^2} + 0.0025$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \quad BKG + 3}{2.22*CTIME*S\text{-}YIELD*S\text{-}EFF*VOL}$$

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 Water Samples	NO: RSL-305	PAGE: 10
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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						

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	REVISION: 0	DATE: 4-14-87

APPENDIX 10.2

t(days)	$1-e^{-\lambda t}$	t(days)	$1-e^{-\lambda t}$	t(days)	$1-e^{-\lambda t}$
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

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APPENDIX 10.3

t(hr)	$e^{-\lambda t}$	$1-e^{-\lambda t}$	t(hr)	$e^{-\lambda t}$	$1-e^{-\lambda t}$	t(hr)	$e^{-\lambda t}$	$1-e^{-\lambda t}$
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

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APPENDIX 10.4

t(days)	$e^{-\lambda t}$	t(days)	$e^{-\lambda t}$	t(days)	$e^{-\lambda t}$
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

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-306

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Strontium-89, 90 in Milk Samples

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

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

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-14-87				

TITLE: Determination of Strontium-89,90 in Milk Samples	NO: RSL-306	PAGE: 1
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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).

TITLE: Determination of Strontium-89,90 in Milk Samples	NO: RSL-306	PAGE: 3
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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

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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-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 Meiss DATE: 5/4/87
ITAS QA Director

CONCURRED: John G. Auxier DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-06-87				

429-1-87

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 1
	REVISION: 0	DATE: 4-06-87

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

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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.

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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.

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 7
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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

TITLE: Determination of I-131 Activity in Milk and Water Samples	NO: RSL-307	PAGE: 8
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APPENDIX 10.1

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						

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-308

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Determination of Gross Alpha and/or Beta Activity

APPROVED: James T. Harvey 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:

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DATE: 4-20-87				

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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.

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

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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

000183

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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						

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430-1-87

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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						

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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 Miles DATE: 6-8-87
ITAS QA Director

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

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DATE: 4-14-87				

429-1-87

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: 4-28-87
Laboratory Manager

APPROVED: Paul Weiss 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				

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TITLE: Determination of Technicium-99	NO: RSL-310	PAGE: 1
	REVISION: 0	DATE: 4-10-87

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.

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 water 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. Currie, 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 Hot plate

5.2 Materials

5.2.1 Plastic liquid scintillation vials (20 ml)

5.2.2 Liquid scintillation cocktail, commercial

5.2.3 Assorted tools and routine laboratory equipment

5.3 Reagents

5.3.1 Sodium hydroxide (NaOH), 40% (w/v): Dissolve 40g of sodium hydroxide in 100 ml of demineralized water.

5.3.2 Sodium persulfate ($Na_2S_2O_8$), 5% (w/v): Dissolve 5g of sodium persulfate in 100 ml of demineralized water.

5.3.3. Ferric nitrate, [$Fe(NO_3)_3 \cdot 9H_2O$] 10% (w/v): Dissolve 10g of ferric nitrate in 100 ml of demineralized water.

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

6.0 PROCEDURE

6.1 Add 0.5 ml concentrated nitric acid to a 250 ml sample aliquot and evaporate to 15 - 20 ml. Run a blank and spike with each set of samples. Record appropriate information on data sheet (appendix 10.1).

6.2 Transfer sample into plastic centrifuge tube, add 1 ml concentrated nitric acid, 2 ml sodium persulfate and warm in water bath at 70°C for 10 minutes.

6.3 Cool sample to room temperature, add 1 ml ferric nitrate and adjust pH of solution to be >7 with sodium hydroxide. Monitor with pH paper.

6.4 Dilute sample to 40 ml with demineralized water, mix well and centrifuge.

6.5 Pipette 5 ml of supernatant into two counting vials containing 15 ml liquid scintillation cocktail. Add Tc-99 internal standard to one vial, mix well and submit sample to counting room 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

None

8.4 Equipment Monitoring

8.4.1 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.

TITLE: Determination of Technicium-99	NO: RSL-310	PAGE: 5
	REVISION: 0	DATE: 4-10-87

9.0 CALCULATIONS

Tc-99 Activity (pCi/l) = ACT

$$= \frac{TSC - BKG}{2.22*EFF*DF*CTIME*VOL}$$

Tc-99 Activity Error (pCi/l) = ERR

$$= ACT* \frac{TSC}{(TSC - BKG)^2} + 0.005$$

Lower Limit of Detection (pCi/l) = LLD

$$= \frac{4.65* \ BKG + 3}{2.22*EFF*DF*CTIME*VOL}$$

TSC = Total sample counts
BKG = Background counts
CTIME = Count time (min)
DF = Dilution factor
EFF = Counting efficiency - interval standard cpm/dpm
VOL = Sample volume (l)
2.22 = Conversion from dpm to pCi
4.65,
3 = Constants
0.005 = Sum of all other errors

10.0 APPENDICES

TITLE: Determination of Technicium-99	NO: RSL-310	PAGE: 6
	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-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 G. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: ISSUED

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REVISION: 0				
DATE: 4-08-87				

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

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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

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COPY NO.: _____ ISSUED: _____

REVISION: 0				
DATE: 4-10-87				

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

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. Aupin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-10-87				

429-1-87

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 _____ 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						

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 L. Cuyler DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED

REVISION: 0				
DATE: 5-08-87				

429-1-87

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)

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 A. Cuspin DATE: 6-12-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-14-87				

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

TITLE: Determination of Ruthenium-106	NO: RSL-316	PAGE: 3
	REVISION: 0	DATE: 4-14-87

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: _____

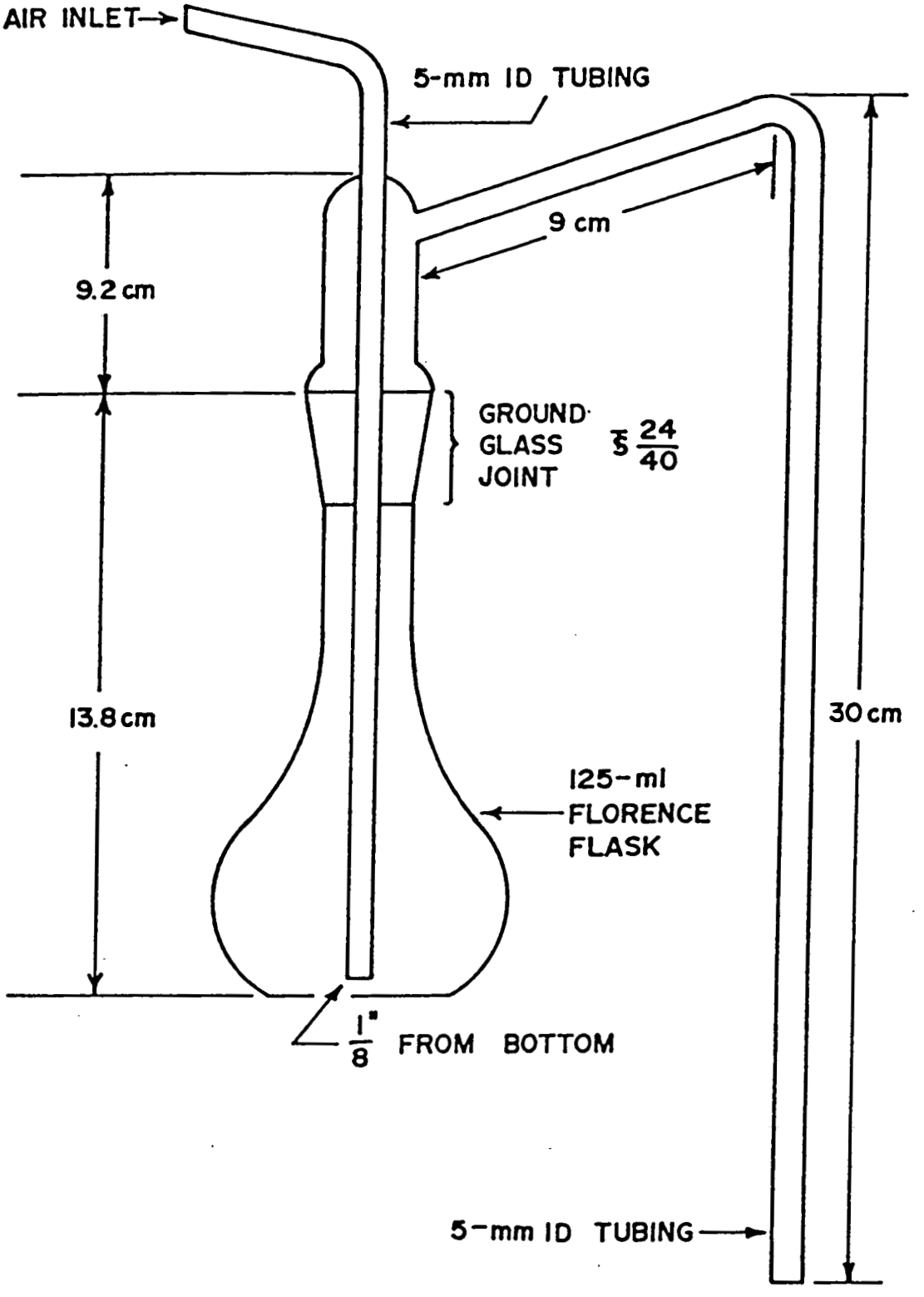
REMARKS AND CALCULATIONS

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

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. Ampier DATE: 6-12-87
RSL Director

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TITLE: Determination of Cerium Isotopes	NO: RSL-317	PAGE: 1
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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

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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-501

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Data Verification

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

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

CONCURRED: John C. Crupin DATE: 4-28-87
RSL Director

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-07-87				

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1.0 PURPOSE

This procedure establishes the requirements and assigns the responsibilities for the recording, review and analysis of data as well as computational checks.

2.0 SCOPE

This procedure applies to all data generated from sample analyses and quality control activities.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that the recording, review, analysis and computational checks of data are performed according to this procedure. (Excluding review by Quality Assurance Coordinator).

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.

3.3 It is the responsibility of those persons performing any activities included in this procedure to follow the procedure and report any suspect results to the Laboratory Manager.

3.4 It is the responsibility of the Quality Assurance Coordinator to ensure that the Quality Assurance report and data reviews are performed according to this procedure.

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 Operations) - Effluent Streams and the Environment.

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

None

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Data Recording

6.1.1 Record all data in a clear, legible manner in appropriate boxes on lab work order (appendix 10.1), lab work sheets (appendix 10.2), and Quality Control sample data sheets, (appendix 10.3, & 10.4) using black indelible ink.

6.1.2 If it is necessary to perform calculations manually, make calculations on IT calculation form (appendix 10.5). Sign (full signature) and date in black, indelible ink each page of calculations.

6.1.3 Enter "N/A" (non-applicable) in analytical sections of lab worksheet which do not apply to that particular analysis.

6.1.4 For each analytical step performed, initial and date the corresponding box under the "remarks and calculations" section of the lab work sheet. (See appendix 10.6 for description of task and date for each section. If more than one technician performs a step of an analysis, both technicians shall initial and date that section.)

6.1.5 To correct a data entry or calculation error, draw a single line through the entry, make correction, initial and date.

6.1.6 Submit completed sample lab work sheet(s), work order (if more than one analysis) and computer printouts (if applicable) to Laboratory Manager, or his designee, promptly after completion of all analyses and data entries for data review.

6.2 Data Review

Review a minimum of 20% of all data (if errors are found in data set, 100 percent review must be performed). Data review must be performed by the Laboratory Manager, or a designee, other than the person performing the analysis or computations. The following method shall be used:

- Review data for:

  - Appropriateness of equations used
  - Correctness of numerical input
  - Numerical correctness of all calculations by re-performing numerical calculations
  - Reasonableness of data results for type of analysis performed.

- Mark all entries and calculations reviewed with a check mark. To make entry or calculation change, mark through the number with a single line and place revised number above it.

- Review changes with the originator. If the originator does not agree with change, the Laboratory Manager shall resolve the problem.

- Sign and date lab work sheet, and/or calculation sheet and associated quality control sample work sheets indicating agreement with calculations.

- Have originator sign and date lab work sheet and/or calculation sheet and associated quality control sample work sheets indicating that he/she agrees with any changes which have been made.

- If data is computer-generated, check each input entry. Indicate agreement with a check mark on each line. To make a change, mark through entry with a single line and place corrected entry above it. Back check all corrections with originator as discussed above.

- If an input error is found, reprocess data. Check second set of data inputs and indicate agreement with changes by signing and dating the computer sheet.

6.3 Data Approval

The Laboratory Manager shall approve 100 percent of sample and quality control lab work sheet data results before a data report can be generated. The following method shall be used:

- If the Laboratory Manager performs the data review, he shall initial and date the approval box on the lab work sheet and quality control sample worksheet.

- If a designee performs the data review, the Laboratory Manager shall:

  - Check the sample and quality control data for completeness and reasonableness for the type of analysis.
  - Check the Data Review box for designee's initials and date.
  - Initial and date Approval box on the lab work sheet as an indication that the data has been reviewed and is approved for report generation.

- If approval is not made, mark "Void" or "rejected data" on lab worksheet. Submit worksheet to Quality Assurance Coordinator for filing in customer file under "Void/Rejected Data" file in Analytical Data section, Category "F".

- Submit approved analytical data to Data Base Manager for generation of data report.

6.4 Review of Data Report

6.4.1 The Data Base Manager shall check draft data report against lab work order and work sheets for transcription errors using the following method:

- Check customer and sample identification information (e.g., customer identification number) on draft data report against lab work sheet information.

- Check data entries on draft data report against lab work sheet entries.

- Check reporting units on draft data report against lab work sheet units.

6.4.2 Indicate correct entries with a black ink check mark next to draft data report entry.

6.4.3 Indicate errors by circling incorrect information on draft data report in red ink and enter correct information beside it.

6.4.4 If no changes are to be made, generate and submit final data report to Laboratory Manager for approval.

6.4.5 If errors were found in draft report, make corrections in computer data base, regenerate draft data report and review report using previous steps.

6.4.6 Generate and submit final data report to Laboratory Manager for approval and signature.

6.5 Quality Assurance Report and Data Review

6.5.1 The Quality Assurance Staff shall review the completed data paperwork for a minimum of twenty percent of all samples analyzed at IT/RSL. Selection will be by random sampling.

6.5.2 Quality Assurance review will be performed using the following method:

- Duplicate the following sample paperwork: request for analysis, analytical data sheets, Quality Control sample data, data reports, requirements and non-conformances as applicable.
- Cross-check all information included for correctness, completeness and correct data calculations.
- Indicate review by making a check-mark in black, indelible ink next to items reviewed.
- Circle any errors found in red, indelible ink.
- Submit sample data package to Laboratory Manager for appropriate action if errors are discovered.
- If errors found cause other associated sample data packages to be suspect, pull paperwork for immediate review by Laboratory Manager.

- Indicate Quality Assurance Review by initialing and dating the copy of the Data Report and placing that in the front of the sample data package.

- Maintain documentation of the Quality Assurance Report and Data Review in the OP/QA files.

6.6 Final Data Report Approval

6.6.1 The Laboratory Manager shall approve all final reports before issuance to the customer.

6.6.2 The Laboratory Manager shall review the final data report for reasonableness and completeness of entries.

6.6.3 He shall indicate final approval by signing "certified by" entry on report.

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 to appropriate sections of this procedure by laboratory personnel.

8.1.2 The ITAS semi-annual systems audit shall determine compliance to applicable sections of this procedure by the Quality Assurance Coordinator.

8.2 Acceptance Criteria

As stated in procedure.

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

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8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

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APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RSL LAB WORK ORDER

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:

ANALYSIS								
COMPLETED								

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INTERNATIONAL TECHNOLOGY CORPORATION

APPENDIX 10.2

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						

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INTERNATIONAL TECHNOLOGY CORPORATION

APPENDIX 10.3

INTRALABORATORY QUALITY CONTROL DATA SHEET

DUPLICATE ANALYSIS DATA

Project _____ Sample Type: Water _____

_____ Soil _____

Analytical Method: _____ Sediment _____

Instrument Used: _____ Other _____

(Analytical results reported in d/m or pCi/unit)

SAMPLE #	INIT'LS	DATE ANALYZED	1st ANALYSIS	DATE ANALYZED	2nd ANALYSIS

COMMENTS:

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INTERNATIONAL TECHNOLOGY CORPORATION

APPENDIX 10.4

INTRALABORATORY QUALITY CONTROL DATA SHEET

SPIKE ANALYSIS DATA

Project _____

_____

Analytical Method: _____

STANDARD NUMBER _____

Sample Type: Water _____

Soil _____

Sediment _____

Other _____

(d/m or pCi/unit)

SAMPLE #	DATE SPIKED	INIT'LS	ADDED	OBSERVED	DEVIATION

COMMENTS:

000255

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APPENDIX 10.5

INTERNATIONAL TECHNOLOGY CORPORATION

IT/RADIOLOGICAL SCIENCES LABORATORY CALCULATION SHEET

Sample number _____ Date _____

Calculations performed by _____

APPENDIX 10.6

Section Title	Description	Date
Sample Preparation	All preparation steps including drying, grinding and ashing.	Start Date
Sample Analysis	Chemical separation activities	Start Date
Sample Counting	Putting sample on counter; entering data after completion of sample counting.	Count Date
Calculations	Calculations using raw data and appropriate formulas to obtain values in units to be reported	Calculation Date
Data Review	Review of all entries on lab data sheet for completeness, reasonableness and correctness as far as can be determined	Date Data Checked
Approval	Data approval for generation of data report	Approval Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-601

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Performing Intralaboratory Quality Control Analysis

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

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

CONCURRED: J. A. Ampier DATE: 4-28-87
RSL Director

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TITLE: Performing Intralaboratory Quality Control Analysis	NO: RSL-601	PAGE: 1
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1.0 PURPOSE

This procedure outlines the use of quality control samples required at IT/RSL. The procedure describes: type of sample, purpose of sample, frequency with which the sample is to be analyzed within the normal sample stream, and person responsible for entering the quality control sample into the sample stream.

2.0 SCOPE

This procedure applies to all quality control samples routinely analyzed at IT/RSL.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that Method Blank, Duplicate and Matrix Spike Quality Control Samples are performed at the required frequency as indicated in this procedure.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of the preparation and analysis of quality control samples to personnel who are experienced with this procedure and with the use of equipment used to perform this procedure.

3.3 It is the responsibility of the Quality Assurance (QA) staff to ensure that all blind quality control samples and verification/reference standards are prepared and entered into the routine sample stream as indicated in this procedure at the required frequency.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 ITAS Quality Assurance Manual.

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

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 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1, Program for Monitoring Radioactivity in the Environ of Nuclear Power Plants.

4.1.6 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 Reasonably Achievable" for Radioactive Material in Light-Water-Cooled Nuclear Power Reactor Effluents.

4.2 Standards

None

4.3 Procedures

None

4.4 Other

4.4.1 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.C. 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

None

5.2 Materials

None

5.3 Reagents

None

TITLE: Performing Intralaboratory Quality Control Analysis	NO: RSL-601	PAGE: 3
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6.0 PROCEDURE

6.1 Method Blank

6.1.1 The technician shall take a volume of demineralized water or an appropriate solid matrix for soil/sediment samples approximately equal to the volume or weight of the sample to be processed and carry it through the entire analytical process using identical conditions used for sample analysis, including the addition of all the reagents in the quantity required by the method.

6.1.2 The technician shall record the results on the appropriate procedure analysis form and identify as "Method Blank".

6.1.3 If blank interference is found it must be taken into account when computations are made.

6.1.4 A method blank shall be performed with each new group of samples, on 1 out of every 20 samples or daily, whichever is more frequent, if that specific analysis is to be performed.

6.2 Blind Replicate

6.2.1 The QA staff shall take an aliquot of sample approximately equal in mass or volume to the original sample and enter it into the routine sample stream.

6.2.2 The QA staff shall complete a "Duplicate Analysis Data" form (appendix 10.1) when the data results are received from the lab.

6.2.3 One blind repeat sample shall be performed for every 50 samples or at least once a week.

6.3 Duplicate

6.3.1 The technician shall take an aliquot of sample approximately equal in mass or volume to the original sample and enter it into the routine sample stream for analysis.

6.3.2 The technician preparing the sample shall initiate a "Duplicate Analysis Data" form (see appendix 10.1) to be completed by the technician performing the analysis.

6.3.3 Data sheets shall be submitted to the QA staff for data compilations and reporting.

6.3.4 Duplicate analysis shall be performed on 1 out of 20 samples or 1 per set of samples.

6.4 Matrix Spike

6.4.1 The technician shall take a separate aliquot of sample and spike the sample with a known concentration of the analyte of interest. (The concentration shall be determined by the Laboratory Manager.) The standard used for spiking must be checked to be sure that the expiration date has not been exceeded.

6.4.2 The technician shall enter necessary data on the "Spike Analysis Data" sheet, (see appendix 10.2).

6.4.3 The sample shall be analyzed and the results computed.

6.4.4 The data sheets shall be submitted to the QA staff for data compilation and reporting.

6.4.5 A matrix spike analysis shall be performed on 1 out of 20 samples or with each new group of samples, which ever is more frequent.

6.5 Verification/Reference Standard

6.5.1 The QA staff shall introduce either a verification sample or a reference standard sample into the routine sample stream. Triplicate analysis is required.

6.5.2 Verification/Reference standard samples shall be analyzed on a quarterly basis or more often as deemed necessary by the QA staff or the Laboratory Manager.

6.5.3 The data sheets should be submitted to the QA staff for data compilation and reporting.

6.6 Blind Replicate Standard

6.6.1 The QA/QC staff shall enter a replicate standard of fixed concentration into the routine sample stream as a blind sample. Triplicate analysis is required.

6.6.2 A blind replicate standard shall be analyzed on a monthly basis or more often as deemed necessary by the QA staff or the Laboratory Manager.

6.6.3 The data sheet shall be reviewed and calculations made by the QA Coordinator to determine accuracy and precision.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Quality Assurance staff shall periodically perform surveillances to ensure compliance to this procedure by laboratory personnel and submit a report to the Laboratory Manager.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

8.3.1 All standards shall be used before expiration date given on the standard's label. After the expiration date, standards shall be disposed of per Section 6.6 of RSL-001, Acquistion and Use of Standard Reference Material.

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

9.1 Duplicate analyses are analyzed by computer program maintained by the Quality Assurance staff.

9.2 Verification standards are analyzed by computer program maintained by the Quality Assurance staff.

10.0 APPENDICES

10.1 Abbreviations and Definitions

10.1.1 Accuracy - A measure of the agreement between observed and known values.

10.1.2 Precision - A measure of the reproducibility among replicate observations.

10.1.3 Method Blank - A volume of demineralized water of similar volume or weight of the sample to be processed, which is carried through the analytical process using identical conditions to the actual sample analysis.

10.1.4 Duplicate Sample - An aliquot of a sample known to the analyst.

10.1.5 Matrix Spike - An aliquot of sample which is spiked with a known concentration of the analyte of interest.

10.1.6 Blind Replicate - A duplicate sample unknown to the analyst which is introduced as a separate sample into the sample stream.

10.1.7 Verification/Reference Standard - A prepared sample of known concentration of a purchased standard reference material.

10.1.8 Standard Reference Materials - Standards prepared by a recognized external agency such as the National Bureau of Standards or the Environmental Protection Agency.

10.1.9 Blind Replicate Standard - A prepared sample of known concentration of a purchased standard reference material submitted as a blind sample to the technician.

TITLE: Performing Intralaboratory Quality Control Analysis	NO: RSL-601	PAGE: 7
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APPENDIX 10.1

INTERNATIONAL TECHNOLOGY CORPORATION

INTRALABORATORY QUALITY CONTROL DATA SHEET

DUPLICATE ANALYSIS DATA

Project _____ Sample Type: Water _____

_____ Soil _____

Analytical Method: _____ Sediment _____

Instrument Used: _____ Other _____

(Analytical results reported in d/m or pCi/unit)

SAMPLE #	INIT'LS	DATE ANALYZED	1st ANALYSIS	DATE ANALYZED	2nd ANALYSIS

COMMENTS:

TITLE: Performing Intralaboratory Quality Control Analysis	NO: RSL-601	PAGE: 8
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APPENDIX 10.2

INTERNATIONAL TECHNOLOGY CORPORATION

INTRALABORATORY QUALITY CONTROL DATA SHEET

SPIKE ANALYSIS DATA

Project _____ Sample Type: Water _____

_____ Soil _____

Analytical Method: _____ Sediment _____

STANDARD NUMBER _____ Other _____

(d/m or pCi/unit)

SAMPLE #	DATE SPIKED	INIT'LS	ADDED	OBSERVED	DEVIATION

COMMENTS:

000266

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-602

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Performing Interlaboratory Quality Control

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

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

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

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DATE: 4-07-87				

000267

TITLE: Performing Interlaboratory Quality Control	NO: RSL-602	PAGE: 1
	REVISION: 0	DATE: 4-07-87

1.0 PURPOSE

This procedure documents IT/RSL participation in available interlaboratory crosscheck programs.

2.0 SCOPE

The procedure applies to all interlaboratory cross-check programs in which IT/RSL participates.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Quality Assurance Coordinator to coordinate IT/RSL participation in interlaboratory cross-checks applicable to the overall work scope performed by the radioanalytical laboratory, as frequently as administered.

3.2 It is the responsibility of the Laboratory Manager to ensure that the radioanalytical laboratory performs the required analysis on the cross-check samples in a timely manner and reports the required results on time.

3.3 It is the responsibility of the laboratory technician to perform the required analyses in a timely manner so the results may be reported when due.

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 "Environmental Radioactivity Laboratory Intercomparison Studies Program" - Fiscal Year 1981 - 1982"; Authur N. Jarvis and Leonard Sin, EPA-600/4-81-004, February, 1981 (or latest edition).

4.4.2 "Handbook for Analytical Duality 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

None

5.2 Materials

None

5.3 Reagents

None

TITLE: Performing Interlaboratory Quality Control	NO: RSL-602	PAGE: 3
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6.0 PROCEDURE

6.1 Interlaboratory cross-check samples are received and processed into the laboratory analysis scheme by routine methods applicable to all samples received. Original paperwork shall be given to Quality Assurance Coordinator.

6.2 Once logged into the data management system, the samples shall be analyzed by the routine laboratory procedures as applicable to the required analyses.

6.3 When the analyses are completed, the results are reviewed through the normal process and reported to the Quality Assurance staff.

6.4 The Quality Assurance staff shall fill out and return the appropriate report forms to the agency submitting the samples to the laboratory. A copy of each report shall be maintained in the Quality Assurance files with associated documents.

6.5 Interlaboratory cross-check results returned to IT/RSL shall be reviewed by the Quality Assurance staff and the Laboratory Manager on a routine basis. If the values reported fall outside the two standard deviation limit set by the EPA, the Laboratory Manager shall investigate the particular analysis in question. If values reported fall outside the three standard deviation limit, an investigation of the particular analysis shall be performed by the Laboratory Manager and a written explanation of the corrective action taken, if necessary, shall be sent to the Quality Assurance Coordinator.

6.6 If the values reported have fallen outside the three standard deviation limits for two consecutive reports for the same analysis of the same matrix, the Quality Assurance staff shall issue a non-conformance report (NCR) per procedure RSL-1005.

6.7 The Laboratory Manager shall instruct the laboratory staff to analyze no further samples of that type until the NCR is satisfied.

6.8 The Quality Assurance staff and the Laboratory Manager shall review the other available quality control data to determine if the out-of-limits results indicate the analysis in question must be evaluated in detail. If evaluation is deemed necessary, any customer results obtained during the time frame covered by the out-of-limits data for the analysis and matrix in question shall be considered suspect and the customer(s) notified.

6.9 If the procedure for the analysis in question is found to be at fault, all samples must be rerun. After corrections to the procedure are made, a set of quality control samples must be run satisfactorily through the procedure. When successfully completed, the effected customers' samples may be rerun.

6.10 If the procedure for the analysis in question is found not to be at fault, the affected customer(s) may be notified.

6.11 The NCR may be closed and the data documenting the actions taken filed appropriately.

7.0 PRECISION AND ACCURACY

7.1 For each analysis performed, the agency issuing the cross-check sample sets precision and accuracy requirements and reports the requirements as the data report.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.2 The Quality Assurance staff shall maintain and control all original documents pertaining to the performance of this procedure.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.5 Control limits

6.6 Control limits

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

TITLE: Performing Interlaboratory Quality Control	NO: RSL-602	PAGE: 5
	REVISION: 0	DATE: 4-07-87

9.0 CALCULATIONS

None

10.0 APPENDICES

None

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INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-603

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Internal Surveillances and Audits

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

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

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

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000273

429-1-87

TITLE: Internal Surveillances and Audits	NO: RSL-603	PAGE: 1
	REVISION: 0	DATE: 4-07-87

1.0 PURPOSE

This procedure provides the requirements for the scheduling, preparation, performance, reporting and documentation of internal surveillances and audits of the IT/RSL Quality Assurance Program to verify compliance with and determine the effectiveness of the program. Descriptions of surveillances and audits are covered to clearly distinguish their differences.

2.0 SCOPE

This procedure applies to all internal surveillance and audit activities at IT/RSL and to project specific audits.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Quality Assurance Staff to follow this procedure when performing any surveillance and audit activities.

3.2 It is the responsibility of the Quality Assurance Staff to maintain and control all surveillance and audit-related documents as quality records.

3.3 It is the responsibility of the Laboratory Manager, or his designee, to attend pre-audit and post-audit meetings and provide necessary personnel, documents and other information as requested by the Auditor.

3.4 It is the responsibility of the Laboratory Manager to respond to audit findings and to recommend and initiate corrective action.

4.0 REQUIREMENTS

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 Operations) - Effluent Streams and the Environment.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

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

5.0 REQUIREMENTS

5.1 Equipment

None

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Surveillance

6.1.1 A surveillance is the act of monitoring or observing to verify whether an item or activity conforms to specified requirements. Notification of the Laboratory Manager is not required. Planning and preparation shall be less extensive than an audit.

6.1.2 Monthly surveillance activities shall be performed using the following steps:

- Identify all areas or activities of the Quality Assurance program that are to be monitored or observed.

- Prepare a monthly schedule (appendix 10.1) to include surveillance of each area or activity at a minimum of twice a year. The schedule shall be prepared in the last quarter of the preceeding year.

- Before performing a surveillance, review all applicable procedures, instructions, and sections of the Quality Assurance Manual.

- Observe or monitor the activity or item of interest.

- Make appropriate entries on the Quality Assurance Surveillance checklist (appendix 10.2).

- Report all deficiencies to the Laboratory Manager upon completion of the surveillance.

- If a non-conformance is found, notify the Laboratory Manager immediately.

- Issue a non-conformance report for any non-conformance identified.

- Follow-up on each corrective action and close-out non-conformance when associated corrective action is verified.

- Maintain a copy of all surveillance records in Quality/Operations audit file.

6.2 Audit

6.2.1 An audit is a planned and documented activity performed to determine by investigation, examination, or evaluation of objective evidence the adequacy of and compliance with established procedures, instructions, drawings and other applicable documents, and the effectiveness of implementation. The audit shall be performed by persons who are independent of any direct responsibility for the performance of audit activities.

6.2.2 The Quality Assurance Coordinator shall perform quality intensive internal audits every two months as a minimum using the following steps:

- Identify all areas or activities of the Quality Assurance program that require auditing.

- Prepare an audit schedule (appendix 10.3) for the calendar year in the last quarter of the preceeding year.

- Distribute copies of the schedule to the Laboratory Manager and the ITAS Quality Assurance Manager.

- Notify the Laboratory Manager in writing two weeks prior to the audit. Make arrangements for availability of necessary personnel for the audit.

- Become familiar with various aspects of the quality program by reviewing audit plans, audit reports, pertinent procedures and requirements.

- Prior to the audit, prepare the audit plan to include:

  - Audit team members

  - Requirements

  - Activities to be audited

  - Applicable documents

  - Schedule

  - Checklists (appendix 10.4)

- During the pre-audit meeting with the Laboratory Manager, other laboratory personnel and audit team members, identify the scope of the audit and the planned audit agenda.

- Conduct the audit according to the audit plan using the ITAS System Audit checklist as applicable and the IT/RSL laboratory specific checklist (latest edition). Make additions or probe areas more intensely as necessary to satisfy the audit objectives.

- Report immediately any serious findings or conditions to the Laboratory Manager or appropriate supervisor.

- Initiate an Audit Action Report (appendix 10.5) for each finding noted during the audit.

- Conduct a post-audit meeting with Laboratory Manager, other laboratory personnel and audit team members. Provide an overview of findings, recommendations, and a date for formal audit report completion.

- Prepare the formal audit report using the format outlined on appendix 10.6. Be factual, orderly and brief (without sacrificing clarity). All members of the audit team shall sign the report.

- Prepare audit report cover letter.

- Issue the audit report with attached Audit Action Reports within thirty days of audit conclusion to the IT/RSL Laboratory Director and Laboratory Manager and to the ITAS Quality Assurance Manager.

6.2.3 A response of corrective action taken or a schedule for corrective action must be issued to the Quality Assurance Coordinator by the Laboratory Manager, or his designee within thirty days of the issuance of the audit report.

6.2.4 The Quality Assurance Coordinator shall follow-up on audit corrective/preventive action by performing the following steps:

- Evaluate each corrective/preventive action report response for adequacy.

- Indicate whether response is satisfactory or un-satisfactory under the evaluation section of appendix 10.4. If response is un-satisfactory, request further corrective action and indicate date for response.

- File all Audit Action Reports in the Quality/Operations audit files.

- Perform a follow-up corrective action audit during the next scheduled audit. Report, in writing, findings to the Laboratory Manager and Director.

- Sign and date "Follow-up and Final Close-out" section of appendix 10.4 when corrective action has been verified.

6.2.5 Maintain and control the following audit documentation as quality records:

- Audit reports

- Audit action reports

- Completed audit plans

- Checklists

- Associated correspondence

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 Compliance to this procedure shall be determined by ITAS semi-annual systems audits.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Internal Surveillances and Audits	NO: RSL-603	PAGE: 7
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APPENDIX 10.1

IT/Radiological Sciences Laboratory

Surveillance Schedule

Calendar Year:_____ Prepared by:_____

Date Issued:_____ Q.A. Coordinator

Date Revised:_____

Surveillance Number	Subject & Scope	Dates		Performed by
		Target	Actual	

000280

APPENDIX 10.2

Reviewer:_____

Date:_____

Page:_____

IT/Radiological Sciences Laboratory

Quality Assurance

Surveillance Checklist

Attribute or Condition Checked	N/A	Satisfactory	
		YES	NO

TITLE: Internal Surveillances and Audits	NO: RSL-603	PAGE: 9
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APPENDIX 10.3

YEARLY AUDIT PLAN AND SCHEDULE

IT/Radiological Sciences Laboratory
Audit Plan and Schedule

Calendar Year:_____

Date Issued:_____

Date Revised:_____

Prepared by:_____
Quality Assurance

Approved by:_____

Audit Number	Subject and Scope	Auditor(s)	Dates		
			Target	Actual	Report

APPENDIX 10.4

INTERNAL AUDIT CHECKLIST

IT/RADIOLOGICAL SCIENCES LABORATORY

SHEET 1 of _____

INTERNAL AUDIT CHECKLIST

1.0 AUDIT SCOPE: _____

_____

2.0 AUDIT PURPOSE: _____

_____

3.0 APPLICABLE DOCUMENTS: _____

_____

4.0 AUDIT CHECKLIST: Prepared By: _____ Date: _____

Approved By: _____ Date: _____

QUESTIONS	AUDITOR'S COMMENTS

APPENDIX 10.5

AUDIT ACTION REPORT

Audit Number:_____
AAR Number:_____

To:_____ Title:_____

Organization:_____

Description of Finding Discovered by Audit Team:

Signature:_____ _____
Lead Auditor - Date Quality Coordinator

Complete Items 1, 2, & 3 Within ____ Days and Return to Lead Auditor.

1) Reason For Discrepancy:

2) Corrective Action Plan (Including Action to Prevent Recurrence):

3) Corrective Action
Will Be Completed By: _____ Signature:_____
Cognizant Mgr. - Date

Evaluation of Proposed Corrective Action:

Signature:_____ _____
Lead Auditor - Date ITAS QA/C Director - Date

Follow-Up and Final Close-Out:

Signature:_____ _____
Lead Auditor - Date ITAS QA/C Director - Date

APPENDIX 10.6

AUDIT REPORT FORMAT
(TYPE OF AUDIT PERFORMED) (AUDIT NUMBER)

1.0 OBJECTIVE

Short paragraphs that outline the objectives of the audit.

2.0 SCOPE

A short paragraph that states when the audit was performed, who performed it, what areas were reviewed, and the names of the key persons contacted.

3.0 CONCLUSIONS

3.1 A sentence or short paragraph, separately numbered for each major conclusion. The conclusions shall directly relate to the objective(s) of the audit as stated in "1.0 OBJECTIVE".

3.2 A sentence or short paragraph detailing the status (if applicable) of corrective/preventive action of previous audits.

3.3 A short sentence detailing how many attributive checks were made and how many were found to be nonconforming.

4.0 OBSERVATIONS

The following statement will be entered in this section of the report:

"An audit report is attached describing each finding"

Audit Performed By:

Name:_____

Title:_____

Lead Auditor:

Name:_____

Approved By:

Name:_____

Title:_____

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-701

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: File Set-Up and Inventory for Perry Nuclear Power Plant

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

APPROVED: [illegible] DATE: 5/4/87
ITAS QA Director

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-20-87				

429-1-87

TITLE: File Set-Up and Inventory for Perry Nuclear Power Plant	NO: RSL-701	PAGE: 1
	REVISION: 0	DATE: 4-20-87

1.0 PURPOSE

This procedure describes the file set up and inventory procedures to be used in conjunction with Section 12 (Records Management) of the ITAS QA Manual.

2.0 SCOPE

This procedure applies to Perry Nuclear Power Plant project files at Radiological Sciences Laboratory (RSL).

3.0 RESPONSIBILITY

The QA Coordinator shall be responsible for the maintenance of Quality records and for supervision of the file inventory.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Memo dated 10-22-86, from W.R. Humphries (Perry Nuclear) to Cheryl Prince (RSL).

4.1.2 ITAS QA Manual, Section 12.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

None

5.0 REQUIREMENTS

None

TITLE: File Set-Up and Inventory for Perry Nuclear Power Plant	NO: RSL-701	PAGE: 2
	REVISION: 0	DATE: 4-20-87

6.0 PROCEDURE

6.1 Set-Up

6.1.1 Each Report Packet (Analytical Data - category "F" in ITAS QA Manual, Section 12) will consist of the Report and the sample data sheets grouped by matrix (soil, water, etc.).

6.1.2 Each Report and sample group will be maintained in an individual manila folder.

6.1.3 The Report Packet (collection of manila folders) will be grouped in an accordian folder.

6.2 Labeling

6.2.1 The "F" category (Analytical Data) will be subnumbered so that each Report Packet (accordian folder) will have a unique number i.e., F1, F2, F3, etc.

6.2.2 The "F" category will be further sub-lettered so that each Report and sample group will have a unique number i.e., F1A-Report, F1B-Soil Samples, F1C-Water Samples, etc.

6.2.3. Each page in a Report or sample group will have a unique number by adding a page number to the above system.

Example: The 1st Quarter, 1985 Report Package has been given the next available Analytical Data number, F20.

The accordian folder would be labeled:

1st Q, 1985	F20
Perry Nuclear	

The manila folder for the Report would be labeled:

Report	F20A
1st Q, 1985	

The Reports six (6) pages would be marked in the upper right hand corner: F20A1, F20A2 .... F20A6.

The manila folder for the sediment sample group would be labeled:

Sediments F20B
1st Q, 1985

The individual data pages would be marked in the upper right hand corner: F20B1, F20B2, etc.

6.3 Inventory

6.3.1 Each page will be marked with its unique page number, as described in paragraph B above.

6.3.2 An inventory sheet (see appendix 10.1) will be stapled to the outside of each accordian folder (Report Packet).

6.3.3 The inventory sheet is in addition to the Project File Index (as per ITAS QA Manual, Section 12).

6.3.4 A copy of the inventory sheet, signed by the QA Coordinator will be shipped with each Report Packet (accordian folder) when files are transferred to Perry Nuclear. Receipt acknowledgement by Perry Nuclear, should be requested. Complete "File Transfer Procedures" are given in Perry Nuclear Standard Operational Procedure number RSL-702.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The QA Coordinator shall review all data packages before signing inventory sheet for transfer.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: File Set-Up and Inventory for Perry Nuclear Power Plant	NO: RSL-701	PAGE: 5
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APPENDIX 10.1

PERRY NUCLEAR POWER PLANT

REPORT PACKET INVENTORY

Report Packet No.:_____

Sampling Event:_____

Transfer Signature:_____
QC Coordinator

FILE SUB-NUMBER	DESCRIPTION	NUMBER OF PAGES

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-702

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Perry Nuclear Records Transfer

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

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

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

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429-1-87

TITLE: Perry Nuclear Records Transfer	NO: RSL-702	PAGE: 1
	REVISION: 0	DATE: 4-27-87

1.0 PURPOSE

This procedure describes the requirements for transfer of analytical records from Radiological Science Laboratory (RSL) to customer.

2.0 SCOPE

This procedure applies to Perry Nuclear Power Plant (PNPP) records generated by RSL.

3.0 RESPONSIBILITY

3.1 The QA coordinator shall be responsible for the identification of records to be transferred, supervision of the preparation of records for transfer, documentation of transfer, and customer notification of transfer.

3.2 The personnel performing these activities are responsible for preparing transfer shipments according to this procedure.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 Memo dated 10-22-85, from W.R. Humphries (PNPP) to Cheryl Prince (RSL).

4.1.2 ITAS Quality Assurance Manual.

4.1.3 ITAS-RSL Quality Assurance Manual, Laboratory.

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

None

5.0 REQUIREMENTS

None

6.0 PROCEDURE

6.1 Records

6.1.1 Transfer the original laboratory data package for each sample and retain a copy of each document.

6.1.2 Group records to include the report submitted and all sample data sheets in the order in which they are included in the report.

6.1.3 Records to be included in a single shipment will be determined by data sheet format. Only those of the same format will be transferred together except where a single report may have a combination of forms.

6.1.4 Include in the data package the laboratory sheet(s), gamma scan print-out, where applicable, and laboratory work order, when applicable.

6.1.5 Check to be sure record packets and the content of each has been numbered sequentially. Put into sturdy file boxes and secure with shipping tape.

6.1.6 Do not transfer correspondence and contractual documents, but retain in client file.

6.2 Frequency of Transfer

6.2.1 Original documents will be transferred in monthly shipments until all records have been shipped. Thereafter, a monthly shipment will be sent which will include only those samples reported in the previous month.

6.2.2 Do not send a shipment until verification of receipt for the previous shipment has been received by RSL.

6.3 Receipt Documentation

6.3.1 Include a cover letter and a copy of each inventory sheet in each transfer shipment. Under separate cover, send a notification letter and another copy of each inventory sheet associated with shipment. Maintain a copy of each in the client's file.

6.3.2 The RSL Q.A. Coordinator will notify the Supervisor of Administrative Records Unit at PNPP the day the shipment is sent.

6.3.3 Upon arrival of the shipment, a PNPP representative will notify the RSL Q.A. Coordinator of receipt.

6.3.4 Upon receipt and verification of transferred documents, one copy of each inventory sheet will be signed by responsible person at PNPP as confirmation of receipt and returned to IT/RSL Q.A. Coordinator within 5 working days.

6.4 Mode of Transfer

6.4.1 Ship records by commercial courier, one day delivery and requirement tracer numbers. Ship records to:

Administrative Records Unit
Perry Nuclear Power Plant
W-160
10 North Center Road
Perry, Ohio 44081

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 The Q.A. Coordinator shall monitor preparation of each shipment to assure the activities are in accordance with this procedure.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

TITLE: Perry Nuclear Records Transfer	NO: RSL-702	PAGE: 4
	REVISION: 0	DATE: 4-27-87

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

None

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-703

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Storage and Maintenance of Records

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

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

CONCURRED: John C. Crupin DATE: 4-28-87
RSL Director

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DATE: 4-21-87				

429-1-87

TITLE: Storage and Maintenance of Records	NO: RSL-703	PAGE: 1
	REVISION: 0	DATE: 4-21-87

1.0 PURPOSE

This procedure establishes storage requirements for the maintenance, preservation and protection of quality assurance records.

2.0 SCOPE

This procedure applies to all records that provide documentary evidence of quality as related to IT/RSL's overall performance and operation. In addition, any project specific records which are contractually designated as quality assurance records shall be stored per this procedure until they are transferred to the customer or destroyed.

3.0 RESPONSIBILITY

It is the responsibility of the Quality Assurance Staff to follow this procedure when storing and controlling all quality assurance records.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 ITAS Quality Assurance Manual.

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

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

4.1.4 "Requirements for Collection, Storage, and Maintenance of Quality Assurance Records for Nuclear Power Plants", ANSI N 45.2.9 (latest edition).

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

4.2 Requirements

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "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

None

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Storage Facility

6.1.1 In general, the records storage facilities shall provide a suitable environment to minimize the risk of damage or destruction from:

- natural disaster such as fires, floods or winds;
- environmental conditions such as temperature and humidity extremes;
- infestation from rodents and insects.

6.1.2 Dual facilities shall be used for record storage at IT/RSL. The facilities shall be sufficiently remote from each other to prevent the posibility of exposure to the same hazard.

6.2 Filing System

6.2.1 See Section No. 12.0 of ITAS-RSL Quality Assurance Manual Laboratory Specific Attachment for a description of the filing system used at IT/RSL.

6.2.2 Duplicate files shall be set up and maintained using the requirements of this procedure.

6.3 Record Receiving and Control

6.3.1 The Quality Assurance Staff shall be responsible for the receiving and control of all Quality Assurance Records. Responsibilities shall include the following:

- Set up new project index and files.
- Add new records to existing files. (Original records shall be placed in master file and copies of original records shall be placed in duplicate file.)
- Withdraw records from master file as requested by laboratory personnel and return records to file.
- Maintain record control with the use of master sign-out sheet to indicate:

  - Project from which file is removed
  - File category
  - Date and person taking file
  - Return date to file system

- Lock all file cabinets when leaving IT/RSL facilities.

6.4 Record Retention

6.4.1 Quality Assurance records shall be classified as "Lifetime" or "Non-Permanent" records for the purpose of retention. (See Section 12.5 of the ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment for the list and complete description of records under the two classifications.)

6.4.2 Mark record index with the classification of each file.

6.4.3 Maintain "Lifetime" records for the lifetime of the project (Lifetime of plant for nuclear power plants).

6.4.4 Maintain "Non-Permanent" records for seven years or until applicable regulatory or customer's contractual requirements are satisfied.

6.5 Record Transfer

6.5.1 The transfer of "Lifetime" records shall take place upon request of the customer or at the end of the contract period.

6.5.2 The Quality Assurance Coordinator shall contact the customer and develop a procedure for the records transfer to include as a minimum:

- Records grouping
- Packaging requirements
- Frequency of transfer
- Receipt documentation
- Mode of transfer
- Customer contact

6.5.3 The Quality Assurance Coordinator shall review the retention period for adequacy before destroying any "Non-Permanent" records.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 Compliance to this procedure by IT/RSL personnel shall be determined by the ITAS semi-annual systems audit.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-801

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Instructions for Glassware Cleaning and Laboratory Housekeeping

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

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

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

COPY NO.: UNCONTROLLED ISSUED: COPY

REVISION: 0				
DATE: 4-07-87				

429-1-87

TITLE: Instructions for Glassware Cleaning and Laboratory Housekeeping	NO: RSL-801	PAGE: 1
	REVISION: 0	DATE: 4-07-87

1.0 PURPOSE

This procedure provides instructions for general glassware cleaning and requirements for the maintenance of laboratory facilities to ensure the performance of analytical work in an efficient, accurate, precise and safe manner.

2.0 SCOPE

2.1 This procedure applies to the cleaning of all washable laboratory apparatus. This procedure should be followed unless instructions for glassware cleaning are provided in a specific laboratory analysis procedure.

2.2 Requirements for housekeeping apply to laboratory areas where analytical work is performed and sample storage and handling areas.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager to ensure that this procedure is followed during the cleaning of all glassware and the cleaning and maintenance of laboratory areas where analytical work is performed and where samples are stored and handled.

3.2 It is the responsibility of the Health Physics Supervisor to ensure that janitorial duties are performed as required.

3.3 It is the responsibility of all laboratory personnel to ensure that lab work areas are kept clean and orderly on a routine basis.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 ITAS Quality Assurance Manual.

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

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 Standard Methods for the Examination of Water and Wastewater, APHA, Washington, D.C. (latest edition).

5.0 REQUIREMENTS

5.1 Equipment

5.1.1 Laboratory sink and/or laboratory dishwasher

5.2 Materials

5.2.1 Brushes, towels, etc.

5.3 Reagents

5.3.1 Commercial detergent and/or liquid cleaner for laboratory use such as but not limited to:

a) Liqui-Nox
b) Sparkleen
c) Radiacwash

6.0 PROCEDURE

6.1 Glassware Cleaning

6.1.1 Rinse glassware thoroughly with tap water immediately after use.

6.1.2 Soak soiled glassware in a solution of hot water and a commercial detergent specifically formulated for laboratory use. Amount of detergent and soak time shall be based on detergent manufacturer's instructions.

6.1.3 Scrub glassware thoroughly with brushes. Never use abrasive materials.

6.1.4 Rinse thoroughly with running tap water followed by thorough rinse with demineralized water. Invert, allow to drain and air dry at room temperature.

6.1.5 Inspect glassware for remaining residue. If glassware is not clean rewash using steps 6.1.2 through 6.1.4. It may be necessary to soak some glassware, etc. in acid solutions. Items falling in this category will be identified as a case-by-case basis and noted in the individual procedure where possible.

6.1.6 If glassware becomes cracked, etched or cannot be cleaned, check with Laboratory Manager, or his designee, before using.

6.2 Housekeeping

6.2.1 The Laboratory Manager, or his designee, shall post a monthly checklist (appendix 10.1) in a conspicuous location in the laboratory.

6.2.2 The Laboratory Manager, or his designee, shall make weekly assignments to selected laboratory technicians for the required routine clean up and maintenance of specified work areas.

6.2.3 Each technician shall be responsible for keeping his/her own work area clean and orderly.

6.2.4 Each technician shall notify the Laboratory Manager of any items or equipment which need repair or routine maintenance performed.

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 to this procedure by laboratory personnel.

8.1.2 The Quality Assurance staff shall maintain Monthly Housekeeping Checklists in the Quality Assurance file as a quality-related document.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.5 Clean glassware

6.1.6 Broken, cracked or dirty glassware

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

ADDENDIX 10.1

HOUSEKEEPING

DATE _____

REVISION 0

PAGE 1 of 1

✓ Work Performed NA Not Applicable

MONTHLY HOUSEKEEPING CHECKLIST

Work Area Description	Week 1	Week 2	Week 3	Week 4	Week 5	Comments
Samples Properly Stored						
Chemicals and Reagents Capped, Labeled, and Properly Stored						
Glassware Cleaned and Stored						
Workbenches Clear and Wiped Down						
Sink Areas Clean						
Eyewash Fountains and Safety Shower Areas Clear						
Cabinets Cleaned						
Hoods Cleaned						
Desk Area Clean, Papers and Books Properly Filed						
Performed By						
Verified By						

Remarks and Action Items:

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-802

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Checking Laboratory Measuring and Test Equipment

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

APPROVED: [illegible] DATE: [illegible]
ITAS QA Director

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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429-1-87

TITLE: Checking Laboratory Measuring and Test Equipment	NO: RSL-802	PAGE: I
	REVISION: 0	DATE: 4-07-87

1.0 PURPOSE

This procedure provides methods for checking the quality of standard laboratory measuring and test equipment.

2.0 SCOPE

This procedure applies to all analytical balances, all top loading balances, and all autopipettes and glass pipettes used in critical steps of laboratory procedures. Critical steps for pipette and autopipette use are defined as those measurements of volume for which an absolute volume of liquid must be measured within two percent of the nominal value of pipette delivery. This procedure does not apply to measuring equipment used for delivery of approximate volumes nor does it apply to adjustable autopipettes.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during quality control checks of standard laboratory equipment.

3.2 It is the responsiblity 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 The Quality Assurance staff shall also set control limits for various balances and pipettes.

3.4 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 is responsible for recording results of measurements in the record books for balance and pipette checks and for adhering to the schedule for these quality control checks.

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 Balances

5.1.2 Pipettes

5.1.3 Autopipettes

5.1.4 Thermometer

5.1.5 Standard Weights protected from dust and fingerprints

5.2 Materials

5.2.1 Stainless steel planchet protected from dust and fingerprints

5.3 Reagents

5.3.1 Deionized water equilibrated to room temperature

6.0 PROCEDURE

6.1 Daily Check for Balances

6.1.1 Zero the balance according to manufacturer's instructions.

6.1.2 Weigh the standard weights designated for the purpose for that individual balance.

6.1.3 Record the weights in the balance logbook and plot the point on the control chart.

6.1.4 Report any value falling outside the control limits as noted in the logbook to the Laboratory Manager, or his designee.

6.2 Monthly Check for Balances

6.2.1 Zero the balance according to manufacturer's instructions.

6.2.2 Weigh standard weights according to the schedule in the logbook. Zero the balance between weighings.

6.2.3 Record the weights in the balance logbook and plot the values on control charts.

6.2.4 Report any values which fall outside the control limits to the Laboratory Manager, or his designee. If maintenance is found to be necessary, appropriate IT/RSL form shall be attached to the balance (see appendix 10.1).

6.3 Annual Check for Glass Pipettes

6.3.1 Wash the pipette and rinse it with deionized water which has been equilibrated to room temperature.

6.3.2 Fill the pipette to the mark with room temperature deionized water and dispense the water into a tared container.

6.3.3 Record the weight of the deionized water and the room temperature in the pipette logbook.

6.3.4 If the value falls outside the control limits recorded in the pipette logbook, report it to the Laboratory Manager, or his designee.

6.3.5 Inspect the pipette for chips or cracks. If any are observed, destroy the pipette and note this in the logbook.

6.4 Monthly Check for Autopipettes

6.4.1 Place an unused tip on the autopipette.

6.4.2 Depress the plunger twice to free the movement.

6.4.3 Fill the pipette with room temperature deionized water.

6.4.4 Pipette the water into a tared container and obtain its weight.

6.4.5 Record the weight and room temperature in the pipette logbook.

6.4.6 If the weight falls outside the control limits recorded in the logbook, contact the Laboratory Manager, or his designee.

7.0 PRECISION AND ACCURACY

7.1 The precision and accuracy of each piece of measuring and test equipment is set by the Quality Assurance staff.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 It is the responsibility of the technician to inspect data to ascertain that it falls within control limits and to inspect balances and pipettes for obvious changes or problems.

8.1.2 It is the responsibility of the Quality Assurance staff to inspect logbooks regularly for trends in data and for completeness.

8.2 Acceptance Criteria

8.2.1 Acceptance criteria for the tests contained herein are specified in the individual logbooks as control limits.

8.2.2 The control limits are twice the calculated standard deviation for a set of ten replicate measurements. If the manufacturer specifies expected standard deviations, twice these values may be used as control limits if they prove to be realistic and suitable after a period of testing.

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

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:

_____

_____

_____

_____

____ 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-901

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Training and Qualifications of Laboratory Technicians

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

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

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

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429-1-87

TITLE: Training and Qualifications of Laboratory Technicians	NO: RSL-901	PAGE: 1
	REVISION: 0	DATE: 4-21-87

1.0 PURPOSE

This procedure provides the training and qualification requirements for radioanalytical laboratory technicians. Additionally, it provides the method for initially, and periodically thereafter, determining the laboratory technician's ability to perform the analytical and counting procedures used by the laboratory and to produce results consistent with the quality assurance program requirements.

2.0 SCOPE

This procedure applies to all IT/RSL laboratory employees performing analyses and using counting equipment.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that the training and qualification program outlined in this procedure is followed and that the training and qualification data are current and documented to certify that the technicians performing quality related work are properly qualified.

3.2 It is the responsibility of the technicians to remain aware of the procedures which they are qualified on, the qualification dates, and perform only those procedures on which they are qualified unless directed by the Laboratory Manager, or his designee.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 ITAS Quality Assurance Manual.

4.1.2 ITAS-RSL Quality Assurance Manual Laboratory Specific Attachment.

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).

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Training and Qualification of Radiochemical Laboratory Analysts", Laboratory Services Administrative Procedure, LSAP-0005, Radiological Health Staff, Tennessee Valley Authority.

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.

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

None

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Training Requirements for Analytical Procedures

6.1.1 The technician trainee (referred to as trainee) shall become familiar with the procedure(s) and observe experienced personnel.

6.1.2 The trainee shall receive one-on-one instruction from the Laboratory Manager, or his designee.

6.1.3 The trainee shall perform the analysis of spike samples under the close supervision of an experienced technician designated by the Laboratory Manager, or his designee.

6.1.4 The trainee may perform the analysis independently when the results for spiked samples are consistently within two standard deviations of the spike known value, however, the trainee's work must be checked frequently by a qualified technician.

6.1.5 The Laboratory Manager may qualify the trainee for independent procedural work when sufficient demonstration of working knowledge of the procedure is achieved through observation and analysis of spike samples.

6.1.6 The Laboratory Manager, or his designee, shall provide retraining when:

A. a technician requests retraining;

B. a technicians's results for spiked samples fall consistently outside three standard deviations of the known value for the spikes;

C. a technician does not perform the analysis for one year.

6.1.7 Retraining shall, at a minimum, include instruction on the procedure and a series of three spike samples analyzed by the technician for which the result must fall within two standard deviations of the known value of the spiked samples.

6.2 Training Requirements for Counting Procedures

6.2.1 The trainee shall observe experienced personnel and become familiar with the procedure.

6.2.2 The trainee shall receive one-on-one instruction from the Laboratory Manager, or his designee.

6.2.3 The trainee shall perform operation of equipment under close supervision of the Laboratory Manager, or his designee.

6.2.4 The trainee may then perform independent counting, but his/her work must be checked frequently by a qualified technician.

6.2.5 The Laboratory Manager may qualify the trainee for independent counting when sufficient demonstration of a working knowledge of the procedure is achieved.

6.2.6 The Laboratory Manager, or his designee, shall provide retraining when:

A. a technician requests retraining;

B. departure from the procedure is identified;

C. a technician does not perform counting activity for one year.

6.2.7 Retraining shall, at a minimum, include instruction on the procedure and operation of equipment under close supervision.

6.3 Documentation of Employee Training

6.3.1 When a trainee has qualified for independent procedural work, the Laboratory Manager shall notify the Quality Assurance staff by completing the "Laboratory Personnel Qualification" form (appendix 10.1).

6.3.2 The Quality Assurance staff shall update the technician qualification record maintained by the Quality Assurance staff. Once updated, copies shall be distributed to the technician and the Laboratory Manager.

6.3.3 Laboratory group training, safety training and QA training shall be documented by attendees signing an attendance record sheet (Appendix 10.2).

6.4 Requirements for Periodic Requalification

6.4.1 Periodic requalification of laboratory technicians is based on the results obtained from running:

A. routine internal known spike samples;

B. internal blind spike samples;

C. interlaboratory cross-check samples.

6.4.2 Requalification of a laboratory technician shall be performed within one year of their qualification date. The Quality Assurance staff shall notify the Laboratory Manager of any laboratory technician whose qualification is within 30 days of expiration on any procedure.

6.5 Records Maintenance

6.5.1 The Quality Assurance staff shall maintain all training records and provide the Laboratory Manager with a current list of qualified technicians.

6.5.2 Copies of all training records shall be maintained in a training file and in each individuals training file.

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 Quality Assurance staff to maintain documentation of employee training and review qualification data quarterly to provide a summary that may be used by the Laboratory Manager to assess technician's performance.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.6 Retraining requirements

6.2.6 Retraining requirements

6.4.1 Requalification requirements

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

TITLE: Training and Qualifications of Laboratory Technicians	NO: RSL-901	PAGE: 6
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APPENDIX 10.1

Lab Personnel Qualification

In accordance with SOP-905, I verify that _____ is qualified to perform the following procedures or activities as described in IT/RSL procedures.

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Verified by: _____ **Date:** _____

TITLE: Training and Qualifications of Laboratory Technicians	NO: RSL-901	PAGE: 7
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APPENDIX 10.2

ATTENDANCE RECORD

PERSONNEL DEVELOPMENT SESSION

Date: _____ Time: _____ Category: _____

Title of Session: _____

_____

Description of Session: _____

_____

_____

Session Conducted by: _____

NAME	SIGNATURE	NAME	SIGNATURE

Approved: _____ Date: _____

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-902

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Q.A. Orientation of New Employees

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

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

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 5-12-87				

429-1-87

TITLE: Q.A. Orientation of New Employees	NO: RSL-902	PAGE: 1
	REVISION: 0	DATE: 5-12-87

1.0 PURPOSE

This procedure provides the requirements for new employee orientation and indoctrination to the IT/RSL Quality Assurance Program as well as associated documentation requirements.

2.0 SCOPE

This procedure applies to the Q.A. Program orientation and indoctrination of all personnel hired at IT/RSL whose jobs are of a technical and/or managerial nature and affect the quality of results produced and reported.

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Quality Assurance Coordinator to follow this procedure when performing new employee Q.A. orientation and training and the documentation of these activities.

3.2 It is the responsibility of the personnel office to inform the Q.A. Coordinator of the hire of any new employee at IT/RSL.

4.0 REFERENCES

4.1 Requirements and Specifications

4.1.1 ITAS Quality Assurance Manual.

4.1.2 ITAS-RSL Quality Assurance Manual Laboratory Specific Attachment.

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).

4.2 Standards

None

4.3 Procedures

None

4.4 Other Publications

4.4.1 "Training and Qualification of Radiochemical Laboratory Analysts", Laboratory Services Administrative Procedure, LSAP-0005, Radiological Health Staff, Tennessee Valley Authority.

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.

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

5.0 REQUIREMENTS

None

6.0 PROCEDURES

6.1 The personnel office shall notify the Q.A. Coordinator within seven days of new employee's report-to-work date. Information shall include:

- Employee's name,
- Job title,
- Date of hire,
- Report-to-work date.

6.2 Q.A. Coordinator shall identify ITAS-RSL & ITAS Quality Assurance Manual sections and/or topics which describe quality-related responsibilities for new employee's job title.

6.3 Q.A. Coordinator shall schedule an orientation session with new employee within fourteen days of his/her report-to-work date.

TITLE: Q.A. Orientation of New Employees	NO: RSL-902	PAGE: 3
	REVISION: 0	DATE: 5-12-87

6.4 During orientation session, the Q.A. Coordinator shall review the following topics with new employee:

- Statement of management position;
- Sections and/or topics describing quality-related responsibilities for job title;
- Nonconformances and corrective action (including 10 CFR 21);
- Data entries and corrections.

6.5 Q.A. Coordinator shall document new employee's orientation session attendance by completing appropriate sections of appendix 10.1, "Quality Assurance Orientation Session Checklist", and have employee sign and date form.

6.6 The Q.A. Coordinator shall check with employee within one month of orientation session date to determine if training was adequate. If training was adequate, indication of this accessment as well as signature and date shall be made on attachment 10.1.

6.7 If further training is needed, Q.A. Coordinator shall perform follow-up training session, document training, sign and date attachment 10.1. New employee shall also sign form.

6.8 Attachment 10.1 shall be maintained as a quality assurance record in employee training file.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

8.1.1 Compliance to this procedure shall be determined by the ITAS semi-annual systems audit.

8.2 Acceptance Criteria

None

8.3 Material Monitoring

None

TITLE: Q.A. Orientation of New Employees	NO: RSI-902	PAGE: 4
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8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.1 APPENDICES

TITLE: Q.A. Orientation of New Employees	NO: RSL-902	PAGE: 5
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ATTACHMENT 10.1

Quality Assurance Orientation Session Checklist

NAME: _____ DATE OF HIRE: _____

JOB TITLE: _____ REPORT-TO-WORK DATE: _____

Quality Assurance Program Section	Reviewed (X)
• Statement of Management Position	_____
• Nonconformances and Corrective Action (Including 10 CFR 21)	_____
• Data Entries and Corrections	_____
• Quality-related Responsibilities for Job Title: (Section and/or Topic)	
_____	_____
_____	_____
_____	_____

I attended the session covering Q.A. sections and/or topics as described above.

_____ Employee Signature _____ Date

_____ Q.A. Coordinator Signature _____ Date

Recheck on _____ (Date) indicates _____ Training was adequate

_____ Further training is needed

_____ Q.A. Coordinator Signature

Follow-up training session covered: _____

_____

_____

_____ Employee Signature _____ Q.A. Coordinator Signature _____ Date

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-903

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Material and Equipment Procurement, Receipt, Storage and Control

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

APPROVED: Paul [illegible] DATE: [illegible]
ITAS QA Director

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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429-1-87

TITLE: Material and Equipment Procurement, Receipt, Storage and Control	NO: RSL-903	PAGE: 1
	REVISION: 0	DATE: 4-21-87

1.0 PURPOSE

This procedure provides the requirements for the procurement, receipt, storage and control of purchased materials and equipment to assure conformance to quality requirements.

2.0 SCOPE

This procedure applies to the following purchased materials and equipment.

- Chemical reagents, solvents and gases
- Analytical instruments and replacement parts
- Analytical glassware
- Laboratory containers
- Miscellaneous quality-related laboratory materials

3.0 RESPONSIBILITY

3.1 It is the responsibility of the Laboratory Manager to follow this procedure in procuring and controlling quality-related materials and equipment.

3.2 It is the responsibiity of the Material & Equipment Receiving person to follow this procedure during the receipt and storage of quality-related materials and equipment.

3.3 It is the responsibility of the Quality Assurance Coordinator to monitor the procurement and control of quality related materials and equipment for adherence to this procedure and maintain records of documentary evidence of compliance.

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.

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

4.2 Standards

None

4.3 Procedures

4.3.1 ITAS/MWL SOP No: MW870129RO-7, "Procurement, Receipt, Storage and Use of Quality Related Purchased Materials".

4.4 Other Publications

4.4.1 "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

None

5.2 Materials

None

5.3 Reagents

None

6.0 PROCEDURE

6.1 Procurement of Quality Related Materials and Equipment

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6.1.1 The quality requirements for the procurement of materials and equipment shall be given by the Laboratory Manager in procurement documents for the specific item or in Standard Operating Procedures.

6.1.2 The Laboratory Manager shall perform the following steps in procuring quality-related materials and equipment:

- Identify the quality requirements for the material or equipment as specified in specific item procurement document or the Standard Operating Procedure.
- Identify supplier or vendor. (The supplier is required to provide written quality requirement documentation with materials or equipment, as appropriate. If commercial grade materials or equipment are purchased, catalog numbers referencing the item description in the supplier's catalog is sufficient for documentation.)
- Complete Purchase Requistion form, appendix 10.1, specifying appropriate grades or other quality requirements, as appropriate.
- Submit Purchase Requisition to Accounting Supervisor for approval and ordering.

6.2 Receipt of Quality Related Materials and Equipment

6.2.1 When materials and equipment are received from supplier, the material and equipment receiving person shall perform the following steps:

- Check the items received against procurement documents (e.g., purchase order) and the receiving documents (e.g., packing list, material certificates).
- If an item which is identified on the procurement or receiving documents is missing notify the Laboratory Manager and Accounting Supervisor.
- Check each item for physical damage.
- Check quality requirements specified on procurement documents against that identified on item label and shipping documents.
- Document any deficiencies on a Materials and Equipment Nonconformance Report, (appendix 10.2); submit original to the Quality Assurance Coordinator and a copy to the Laboratory Manager.

- Initial and date shipping document as evidence of acceptance of materials and equipment. (Note on shipping documents any missing or damaged items or quality related deficiencies.)
- Submit original shipping documents to accounting supervisor.
- Maintain copy of shipping documents in receiving files.
- Submit material certificates to Quality Assurance office for filing.

6.3 Storage and Control of Quality-Related Materials and Equipment

6.3.1 The material and equipment receiving person shall store and control quality related materials and equipment by performing the following steps:

- Check labeling of item for quality level. If not displayed, mark item with information.
- Mark receipt date on item when appropriate.
- Rotate stock to assure use of older items first, when appropriate.
- Store materials and equipment per manufacturer's recommendations.
- Remove materials from storage when shelf life has expired as appropriate.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection

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

8.2 Acceptance Criteria

None

8.3 Material Monitoring

As noted in section 8.1

8.4 Equipment Monitoring

As noted in section 8.1

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

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APPENDIX 10.1

REQUESTOR MUST FILL IN SHADED AREAS ONLY.

INTERNATIONAL TECHNOLOGY CORPORATION

PLEASE PRINT HARD

PURCHASE REQUISITION

NO. 101828

P.O. NO. | VENDOR NO. | VENDOR NAME | VENDOR ADDRESS | CITY AND STATE | ZIP CODE | VENDOR PHONE NO.

SHIP TO | ATTENTION | ADDRESS | CITY & STATE | DATE ORDERED | DATE PROMISED

REQUISITION DATE | REQUESTOR PHONE NO. | CONFIRMING TO | REQUESTED DELIV. DATE

BUYERS I.D. | F.O.B. | TERMS | SHIP VIA | ORIG. SAT. ACCEPTANCE | CONFIRMATION ☐ YES ☐ NO | BILLABLE ITEM ☐ YES ☐ NO

LINE NO.	PART NO	U.M.	DESCRIPTION	QUANTITY	UNIT PRICE	PHASE	TASK	SUB TASK	PRODUCT CODE NO
5									

REMARKS

AUTHORIZED SIGNATURE

SUBTOTAL

TAX (%)

FREIGHT

TOTAL

BUYER SIGNATURE

THIS IS NOT A PURCHASE ORDER

PURCHASING

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APPENDIX 10.2

MATERIALS AND EQUIPMENT NON-CONFORMANCE REPORT

Item:_____ Vendor:_____

Description:_____ Purchase Order No.:_____

Receiving Date:_____

Non-Conformance Description:_____

_____

_____

_____

_____

_____

Corrective Action Taken: _____

_____

_____

_____

_____

_____

_____

Signature/Materials & Equipment Receiving Person

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-1001

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Sample Receiving

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

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

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

COPY NO.: UNCONTROLLED COPY ISSUED:

REVISION: 0				
DATE: 4-21-87				

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TITLE: Sample Receiving	NO: RSL-1001	PAGE: 1
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1.0 PURPOSE

This procedure specifies the required activities for receiving samples at IT/Radiological Sciences Laboratory to ensure compliance with applicable industry standards, licenses, quality requirements, and customers' specifications.

2.0 SCOPE

The procedure applies to all samples received at IT/RSL.

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 receipt of all samples.

3.2 It is the responsibility of the Laboratory Manager, or his designee, to delegate the performance of these activities to personnel experienced with this procedure and with the equipment required for the implementation of this procedure.

3.3 It is the responsibility of the assigned personnel to follow this procedure and report any discrepancies or nonconformances found with in-coming samples or associated paperwork to the laboratory manager or his designee.

4.0 REFERENCES

4.1 ITAS Quality Assurance Manual.

4.2 ITAS-RSL Quality Assurance Manual, Laboratory Specific Attachment

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

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5.0 REQUIREMENTS

5.1 Scintillation survey meter

5.2 Ludlum Scalers, Model 1000 and 2000 or equal, with probe Model 43-10 for alpha particle counting and probe Model 44-40 for beta/gamma counting

6.0 PROCEDURE

6.1 Sample Screening

6.1.1 Receive all sample shipments at the designated receiving area and inspect each shipping container for damage.

6.1.2 Remove samples from shipping container and inspect for spillage, breakage or other damage which might affect sample integrity. If such damage has occurred, place sample into a plastic bag or a clean container. Complete a nonconformance form and notify the Laboratory Manager immediately.

6.1.3 Survey each sample with a scintillation survey meter to determine exposure rate and enter the reading into the sample receiving logbook along with the customer name, sample description, sample condition, date and initial. Only samples with an exposure rate less than or equal to 1 mr/hr shall be accepted into the laboratory unrestricted. Samples with readings higher than 1 mr/hr must be stored in the radioactive waste storage area until disposal or return to customer. Aliquots of these samples may be diluted and analyzed as routine samples if the exposure rate of the aliquot meets the stated requirement.

6.1.4 Smear thoroughly the incoming sample shipment, being sure to include all samples in the shipment, to determine if there is removable surface contamination.

6.1.5 Count each smear for five minutes. (Be sure that the smeared side is facing up.) Activity levels which are acceptable in the laboratory are <5 cpm alpha and <25 cpm for beta/gamma.

6.1.6 If either count is higher than acceptable, decontaminate the sample containers by cleaning with a wet soapy cloth. Dry sample with clean cloth, re-smear and count. If re-smear count continues to be above the acceptable limits, repeat process until acceptable limits are reached. Enter each smear count into the appropriate logbook.

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6.1.7 When screening is completed, place samples in appropriate storage facilities for analyses requested. Hold times and required storage are listed on appendix 10.1 for typical samples and analyses. The Laboratory Manager shall be notified of any sample which is not covered by this table and shall provide storage instructions for such samples.

6.2 Chain of Custody

6.2.1 Compare sample information provided on the chain of custody which is received with the sample shipment to the actual samples received. Samples received from other IT personnel must have a properly completed chain of custody or the samples shall not be accepted. The approved IT chain of custody form is provided in appendix 10.2.

6.2.2 Note on the chain of custody the condition of the samples received. Identify any damaged or missing samples. Sign and date the chain of custody.

6.2.3 If samples are delivered via commercial carrier, attach the waybill to the chain of custody.

6.2.4 Submit the completed chain of custody to the QA office to be filed in the project file. If the samples are to be screened for radiation and then released to another section of ITAS for analysis, enter the IT/RSL laboratory number onto the completed chain of custody, copy and submit the copy for filing in the project file.

6.2.5 If a chain of custody is not received with a sample(s), complete an IT chain of custody form before transferring the sample to another laboratory. Sign and date the designated section of the chain of custody for relinquishing the samples.

6.3 Sample Log-In

6.3.1 Check paperwork received with samples to be sure that the required analyses are clearly defined. Samples received from other sections of IT Corporation must be accompanied by a completed request for analysis form (as shown in appendix 10.3) or samples must be held until proper documentation is received. If any sample is received without proper documentation, tag sample with a "hold" tag and notify the customer services representative. Samples will be processed when appropriate documentation is received.

6.3.2 Log all properly documented samples received for radiological analysis into the computer software for Sample and Analysis Management (SAM). Enter sample information and testing requirements into the appropriate fields.

6.3.3 The computer will assign a work order to each group of samples received from a customer at one time. Each work order will be seven digits long and will be in a xx-xx-xxx format. The first digit will be the letter "R" to indicate RSL. The second digit indicates the last numeral of the year in which the sample is received (i.e., R6 for RSL, 1986). The second set of digits indicate the month in which the sample is received (i.e., 05 for May). The last three digits are assigned in numerical order beginning at the first of each month (i.e., 031 for the thirty first set received during that month). Each sample is then given a "dash" number that identifies each sample uniquely. This number is also assigned in numerical order as the individual sample is logged in (i.e., 02 for the second sample of the group). Use the work order and dash number assigned by the computer program together as the laboratory number to identify each individual sample. Label the sample container with this assigned laboratory number (i.e., R605031-02 for a sample received May, 1986 which was the thirty first group received and the second sample of the group.)

6.3.4 Generate a laboratory work sheet, with all pertinent sample information, for each distinct analysis to be performed. Place the laboratory work sheet in the appropriate work-pending file in the laboratory.

6.3.5 File the paperwork received with samples in the corresponding project file.

6.4 Mixed Waste Samples

6.4.1 When samples are received for the mixed waste laboratory, the MWL Manager, or his designee, shall be notified to determine if samples are expected and should be accepted.

6.4.2 When approval for the acceptance is received from the MWL Manager, or his designee, normal receiving activities shall be performed as described in section 6.1 and 6.2 of this procedure.

6.4.3 A container of each sample should be provided by the customer for the additional sample screening required for mixed waste samples. These samples shall be treated as radiological samples and logged into the sample tracking system to be counted for gross alpha and gross beta.

6.4.4 The unopened container of the sample received shall be transferred to the MWL for storage. The original of the chain of custody and all paperwork associated with samples should be given to the MWL Manager or his designee along with the samples. A copy of the chain of custody and request for analysis shall be retained in RSL project files.

6.4.5 When a sample shipment is received in which some samples are for the MWL, only to be screened by RSL, and others are for analytical work by RSL, the original paperwork shall be filed in the RSL project files. A new chain of custody form shall be completed for only those samples to be transferred to MWL. The new chain of custody form should reference the original form's number. A copy of the request for analysis shall be made and sent with the chain of custody and samples to the MWL.

6.4.6 After the screening has been completed, a designated RSL health physicist shall review the data and classify the sample per procedure RSL-1006. A "Mixed Waste Sample Screening" form shall be completed, signed, and submitted to the MWL Manager, or his designee.. A copy shall be retained in the project file.

7.0 PRECISION AND ACCURACY

None

8.0 QUALITY ASSURANCE

8.1 Responsibility for Inspection

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

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.1.2 Sample condition on receipt

6.1.3 Survey reading limit

6.1.4 Sample smear activity limits

8.3 Material Monitoring

None

8.4 Equipment Monitoring

8.4.1 Counting instruments shall be calibrated prior to use.

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

APPENDIX 10.1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	CONTAINER(a)	VOLUME REQUIRED(mL)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(C)
Inorganic Tests				
Acidity	P,G	50	Cool 4°C	14 days
Alkalinity	P,G	50	Cool 4°C	14 days
Ammonia	P,G	100	Cool 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand	P,G	1,000	Cool 4°C	48 hours
Bromide	P,G	200	None Required	28 days
Chemical Oxygen Demand	P,G	75	Cool 4°C, H_2SO_4 to pH<2	28 days
Chloride	P,G	50	None Required	28 days
Chlorine, Total Residual	P,G	200	None Required	Analyze Immediately
Color	P,G	50	Cool 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P,G	1,500	Cool 4°C, NaOH to pH>12, 0.6g ascorbic acid(d)	14 days
Fluoride	P	300	None Required	28 days
Hardness	P,G	100	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
Hydrogen Ion (pH)	P,G	25	None Required	Analyze Immediately
Kjeldahl and Organic Nitrogen	P,G	500	Cool 4°C, H_2SO_4 to pH<2	28 days

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APPENDIX 10.1
Continued

PARAMETER	CONTAINER(a)	VOLUME REQUIRED(mL)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(C)
Metals				
Chromium VI	P,G	50	Cool 4°C	24 hours
Mercury	P,G	100	HNO_3 to pH<2	28 days
Metals, Except Chromium VI and Mercury	P,G	200	HNO_3 to pH<2	6 months
Nitrate	P,G	100	Cool 4°C	48 hours
Nitrate-Nitrite	P,G	100	Cool 4°C, H_2SO_4 to pH<2	28 days
Nitrite	P,G	50	Cool 4°C	48 hours
Oil and Grease	G	1,000	Cool 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon	P,G	25	Cool 4°C, HCl or H_2SO_4 to pH<2	28 days
Orthophosphate	P,G	50	Filter Immediatley, Cool 4°C	48 hours
Oxygen, Dissolved Probe	G bottle and top	300	None Required	Analyze Immediately
Phenols	G	500	Cool 4°C, H_2SO_4 to pH<2	28 days
Residue, Total	P,G	100	Cool 4°C	7 days
Residue, Nonfilterable	P,G	250	Cool 4°C	7 days

APPENDIX 10.1
Continued

PARAMETER	CONTAINER(a)	VOLUME REQUIRED(mL)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(C)
Metals (continued)				
Residue, Settleable	P,G	1,000	Cool 4°C	48 hours
Residue, Volatile	P,G	100	Cool 4°C	7 days
Silica	P	50	Cool 4°C	28 days
Specific Conductance	P,G	100	Cool 4°C	28 days
Sulfate	P,G	100	Cool 4°C	28 days
Sulfide	P,G	500	Cool 4°C, add zinc acetate plus sodium hydroxide to pH>9	7 days
Sulfite	G	50	None Required	Analyze Immediately
Temperature	P,G	1,000	None Required	Analyze Immediately
Turbidity	P,G	100	Cool 4°C	48 hours
Organic Tests				
Purgeable Halocarbons	G, Teflon-lined septum	40	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	40	Cool 4°C, 0.008% $Na_2S_2O_3$(d) HCl to pH 2	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	40	Cool 4°C, 0.008% $Na_2S_2O_3$(d) adjust pH to 4-5	14 days

APPENDIX 10.1
Continued

PARAMETER	CONTAINER(a)	VOLUME REQUIRED(mL)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(C)
Organic Tests(continued)				
Phenols	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	7 days until extraction 40 days after extraction
Benzidines	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	7 days until extraction 7 days after extraction (oxidant-free atmosphere)
Phthalate Esters	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction 40 days after extraction
Nitrosamines	G, Teflon-lined cap	1,000	Cool 4°C, store in dark, 0.008% $Na_2S_2O_3$(d)	7 days until extraction 40 days after extraction
PCBs	G, Teflon-lined cap	1,000	Cooi 4°C	7 days until extraction 40 days after extraction
Nitroaromatics and isophorone	G, Teflon-lined cap	1,000	Cool 4°C, store in dark, 0.008% $Na_2S_2O_3$(d)	7 days until extraction 40 days after extraction
Polynuclear Aromatic Hydrocarbons	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$	7 days until extraction 40 days after extraction
Haloethers	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$	7 days until extraction 40 days after extraction
Chlorinated Hydrocarbons	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction 40 days after extraction
TCDD	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$	7 days until extraction 40 days after extraction
Pesticides				
Pesticides	G, Teflon-lined cap	1,000	Cool 4°C, pH 5-9	7 days until extraction 40 days after extraction

APPENDIX 10.1
Continued

EPA METHOD Liquids	MINIMUM VOLUME REQUIRED (mL)	CONTAINER	PRESERVATIVE	MAXIMUM HOLDING TIME	COMMENTS
601	40	G	Cool 4°C	14 days	*
602	40	G	Cool 4°C	14 days	*
603	40	G	Cool 4°C Adjust pH to 4-5 Dilute sulfuric acid.	14 days,	*
604	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
605	1,000	G	Cool 4°C	Analyze by 14 days Extract by 7 days	*
606	1,000	G	Cool 4°C	Extract by 7 days Analyze by 40 days	*
607	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
608	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
609	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
610	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
611	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
612	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
613	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*
614	1,000	G	Cool 4°C	Extract within 7 days Analyze within 40 days	*
615	1,000	G	Cool 4°C	Extract within 7 days Analyze within 40 days	*
624	40	G	Cool 4°C	Analyze by 14 days	*
625	1,000	G	Cool 4°C	Analyze by 40 days Extract by 7 days	*

For chlorinated waste, immediately add 35mg sodium thiosulfate per ppm of free chlorine per liter.

APPENDIX 10.1
Continued

METHOD	MINIMUM VOLUME REQUIRED(mL)	CONTAINER	PRESERVATIVE	MAXIMUM HOLDING TIME	COMMENTS
Solids and Liquids					
8010 Halogenated	25	G	4°C	Analyze by 14 days	*
8015 Nonhalogenated	25	G	4°C	Analyze by 14 days	*
8020 Aromatics	25	G	Adjust pH <2 with Hydrochloric acid	Analyze by 14 days	*
8030 Acrolein Acrylonitrile Acetonitrile	25	G	4°C	Analyze by 14 days	*
8040 Phenols	25	G	4°C Adjust pH -2 with H_2SO_4 or NaOH	Extract by 7 days, Analyze by 30 days	*
8060 Phthalate Esters	25	G	4°C do.	Extract by 7 days Analyze within 30 days	*
8080 Organochlorine Pesticides PCB's	25	G	4°C do.	Extract by 7 days Analyze within 30 days	*
8090 Nitroaromatics Cyclic Ketones	25	G	4°C do.	Extract by 7 days Analyze within 30 days	*
8100 Polynuclear Aromatic Hydrocarbons	25	G	4°C do.	Extract by 7 days Analyze by 30 days	*
8120 Chlorinated Hydrocarbons	25	G	4°C do.	Extract by 7 days Analyze by 30 days	*
8140 Organophosphorus Pesticides	25	G	4°C do.	Extract by 48 hours Analyze by 14 days	*

APPENDIX 10.1
Contined

METHOD	MINIMUM VOLUME REQUIRED(mL)	CONTAINER	PRESERVATIVE	MAXIMUM HOLDING TIME	COMMENTS
Solids and Liquids					
8150 Chlorinated Herbicides	25	G	4°C	Extract by 7 days Analyze by 30 days	*
8240 Volatile Organics	25	G, Teflon screw cap	4°C	Extract by 7 days Analyze by 14 days	
8250 Semivolatile Organics	25	G, Teflon screw cap	4°C	Extract by 14 days Analyze by 40 days	
8270 Semivolatile Organics	25	G, Teflon screw cap	4°C	Extract by 14 days Analyze by 30 days	
9020 Total Organic Halides (TOH)	250	Teflon septa protected from light with Teflon, Teflon screw cap	4°C	Not specified	

NOTE: *Do not prewash bottle with samples

REFERENCE: This table includes the requirements of the U.S. Environmental Protection Agency, as published in the Code of Federal Regulations, Volume 49, Number 209, 40CFR 136, dated October 26, 1984, page 43260.

TABLE K5-1 (continued)

(a) Polyethylene (P) or glass (G).

(b) Sample preservation should by performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed.

(c) Samples should be analyzed as soon as possible after collection. The times listed are maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory , is obligated to hold the sample for a shorter period if knowledge exists to show this is necessary to maintain sample stability.

(d) Should only be used in the presence of residual chlorine.

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INTERNATIONAL TECHNOLOGY CORPORATION

CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. 39854

000353

PROJECT NAME/NUMBER _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No.

Special Instructions: _____

Possible Sample Hazards: _____

SIGNATURES: (Name, Company, Date and Time)

1. Relinquished By: _____
   Received By: _____
2. Relinquished By: _____
   Received By: _____
3. Relinquished By: _____
   Received by: _____
4. Relinquished By: _____
   Received By: _____

WHITE - To accompany samples
YELLOW - Field copy

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APPENDIX 10.2
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INSTRUCTIONS FOR COMPLETING CHAIN-OF-CUSTODY RECORD

The Chain-of-Custody record must be accurately completed and the original must accompany the sample(s). When sending samples to the laboratory, a Request for Analysis form must also be submitted.

R/A Control No.: Record the printed control number from th top right corner of the Request for Analysis form, if you are sending these samples to the laboratory.

Project Name/Number: Record the name of the project or client/site location, and the billing number of the project. (Example - 613215; X, Y, Z Chemical Co., WA)

Sample Team Members: List the names of all the members of the team taking these samples: team leader's name first.

Lab Destination: Indicate to which laboratory you are sending the samples. Do not list more than one lab on a single chain-of-custody record. Be certain before sending the samples that the laboratory you are designating is aware of the shipment.

Carrier/Waybill No.: If you are sending these samples by a commercial carrier such as Airborne or Federal Express, record the courier company name and the waybill or airbill number under which these samples will be shipped. (Example - Federal Express/#513631771)

Sample Number: List the complete identifying number of each of the samples you are sending. These numbers must correspond with the identifying labels on the sample containers.

Sample Location and Description: Briefly record where the sample was taken and provide a short physical description of the sample. Draw a sketch on the Sample Collection Log if necessary.

Date and Time Collected: Record date and exact time each sample was collected. Use a 24 hour clock; i.e. 1645 not 4:45 p.m.

Sample Type: Indicate the sample type such as soil., sediment, sludge, water, wipe, air, or bulk.

Container Type: Indicate the volume, color, and type of the sample container used. (Examples - 1 gallon amber glass, 1 liter clear plastic, 40 ml clear glass)

Condition on Receipt: Before a custody transfer, the intended recipient should verify all samples are present and in good condition. This column may be used by the recipient to record any abnormalities found at the time of the transfer. (Examples - jar lid cracked, sample label torn)

Special Instructions: Use this space to record any special instructions to the laboratory regarding the processing of these samples. Leave blank if there are no special instructions.

Possible Sample Hazards: Indicate if the samples are suspected to contain high concentrations of any hazardous materials, or if they may pose other hazards. If you are unaware of any possible hazards, leave this line blank.

Signatures: When releasing custody of these samples, use the "Relinquished By" space to sign your full name, company name, date, and time of release. After verifying that all samples indicated are present, the person receiving the samples must sign in the "Received By" space to take custody of the samples.

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TITLE: Sample Receiving

APPENDIX 10.3

INTERNATIONAL TECHNOLOGY CORPORATION

REQUEST FOR ANALYSIS

R/A Control No. 029813

C/C Control No. _____

PROJECT NAME _____

PROJECT NUMBER _____

PROJECT MANAGER _____

BILL TO _____

PURCHASE ORDER NO. _____

DATE SAMPLES SHIPPED _____

LAB DESTINATION _____

LABORATORY CONTACT _____

SEND LAB REPORT TO _____

DATE REPORT REQUIRED _____

PROJECT CONTACT _____

PROJECT CONTACT PHONE NO. _____

Sample No	Sample Type	Sample Volume	Preservative	Requested Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by the Project Manager.)

Normal _____ Rush _____ (Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances)

Nonhazard _____ Flammable _____ Skin Irritant _____ Highly Toxic _____ Other _____ (Please Specify)

SAMPLE DISPOSAL: (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping, and disposal.)

Return to Client _____ Disposal by Lab _____

FOR LAB USE ONLY

Received By _____ Date/Time _____

WHITE - Original, to accompany samples
YELLOW - Field copy

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APPENDIX 10.3
Continued

INSTRUCTIONS FOR COMPLETING REQUEST FOR ANALYSIS FORM

The original Request for Analysis form must be completed and sent with samples shipped to the laboratory for analysis. All samples must also be accompanied by a Chain-of-Custody record.

C/C Control No.: Record the printed control number from the top right corner of the Chain-of-Custody record you are submitting with these samples.

Project Name: Record the name of the project or client/site location. (Example - X.Y.Z. Chemical Co., WA)

Project Number: Indicate the project number to be charged for the analysis of these samples. (Example: 613215)

Project Manager: Record the project manager's name.

Bill To: Non-IT personnel should indicate the correct billing address and the person to whom the invoice should be sent. IT personnel and IT subcontractors should fill in IT office responsible for project accounting (if known).

Purchase Order No.: Non-IT personnel should use this space to record the purchase order number authorizing the analysis of these samples. IT personnel and IT subcontractors should leave this space blank if a project number has been given for billing.

Date Samples Shipped: Indicate the date these samples are being shipped to the laboratory.

Lab Destination: Indicate the laboratory designated for sample shipment. Do not list more than one lab on this Request for Analysis. Be certain before sending samples that the laboratory you are designating is aware of the shipment, and is capable of accepting high hazard samples if applicable.

Laboratory Contact: Give the name of the laboratory employee you have contacted regarding these samples.

Send Lab Report To: Give the name, address, and phone number of the person to receive the data report for these samples.

Date Report Required: Record the date which you and the laboratory contact have determined the results will be reported.

Project Contact: Indicate the name of the project person to be contacted in case of any questions regarding these samples.

Project Contact Phone No.: Give the phone number where the project contact can be reached on the day these samples should arrive at the laboratory.

Sample Number: List the complete identifying number of each of the samples you are sending, as recorded on the accompanying Chain-of-Custody.

Sample Type: Indicate the sample type such as soil, sediment, sludge, water, wipe, air, or bulk.

Sample Volume: Estimate the amount of sample in the container. For air samples, indicate the volume of air sampled.

Preservative: Indicate what type preservative, if any, has been used for the sample. (Examples - ice, blue ice, nitric acid, sulfuric acid)

Requested Testing Program: List the analyses to be performed on the sample.

Special Instructions: Use this space to record any special instructions to the laboratory regarding the processing of these samples. Leave blank if there are no special instructions.

Turnaround Time Required: Check "normal" or "rush" as determined by the Project Manager and the laboratory contact.

Possible Hazard Identification: Indicate all hazard classes associated with the sample(s).

Sample Disposal: Indicate how the samples should be disposed of following analysis. All samples are held six weeks and then disposed of unless other arrangements for storage have been previously requested.

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: RSL-1002

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: Radiological Screening and Classification of Mixed Waste Samples

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

APPROVED: Paul [illegible] DATE: 5/7/87
ITAS QA Director

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

COPY NO.: UNCONTROLLED COPY ISSUED:

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DATE: 4-21-87				

429-1-87

TITLE: Radiological Screening and Classification of Mixed Waste Samples	NO: RSL-1002	PAGE: 1
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1.0 PURPOSE

This procedure provides the method for radiological screening and classification of mixed waste samples prior to analysis.

2.0 SCOPE

This procedure applies to all samples classified as "mixed waste samples".

3.0 RESPONSIBILITIY

3.1 It is the responsibility of the Laboratory Manager, or his designee, to ensure that this procedure is followed during the screening and classification of mixed waste samples.

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.

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 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.1.5, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.

4.2 Standards

4.2.1 "State Regulations For Protecting Against Radiation", Tennessee Department of Health and Environment (latest edition).

4.3 Procedures

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

4.3.2 RSL-104, "Operation of Low Background Alpha/Beta Instruments", IT/RSL Laboratory Procedures Manual (latest edition).

4.3.3 RSL-308, "Determination of Gross Alpha and/or Beta Activity", IT/RSL Laboratory Procedures Manual (latest edition).

4.4 Other Publications

None

5.0 REQUIREMENTS

5.1 Equipment

None

5.2 Material

None

5.3 Reagent

None

6.0 PROCEDURE

6.1 Receipt

6.1.1 Mixed waste samples shall be received in accordance with procedure RSL-1001.

6.2 Determining Alpha and Beta Activity

6.2.1 Procedures RSL-104 and RSL-308 shall be followed in determining the gross alpha and gross beta activity of the mixed waste sample.

6.2.2 Mixed waste samples that are known to contain or suspected of containing low-energy beta emitters such as tritium (H-3), carbon-14, and iron-55, shall be analyzed for the suspected radionuclide prior to classification.

6.3. Liquid Samples Classification

6.3.1 Once the alpha and beta activity has been determined per step 6.2, classification of the sample shall be determined by following the flow chart given in Appendix 10.1, "Flow chart for classification of liquid samples".

6.4 Soil/Solid Samples Classification

6.4.1 Once the alpha and beta activity has been determined in step 6.2, the classification of the sample shall be determined by following the flow chart given in Appendix 10.2, "Flow chart for classification of soil/solid samples".

6.5 Air Filters Classification

6.5.1 The alpha and beta activity of the filters, as determined in step 6.2, should be divided by the filter weight to determine the activity per gram of sample. The filter shall be classified using the flow chart in Appendix 10.2, "Flow chart for classification of soil/solid samples".

6.6 Documentation

6.6.1 A properly completed "Mixed Waste Sample Screening" form (Appendix 10.4), shall be included with the laboratory worksheet.

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.

8.2 Acceptance Criteria

8.2.1 The following sections of this procedure contain acceptance criteria:

6.3.1 Sample activity below license quantities,

6.4.1 Sample activity below license quantities,

6.5.1 Sample activity below license quantities.

8.3 Material Monitoring

None

8.4 Equipment Monitoring

None

8.5 Certification

None

9.0 CALCULATIONS

None

10.0 APPENDICES

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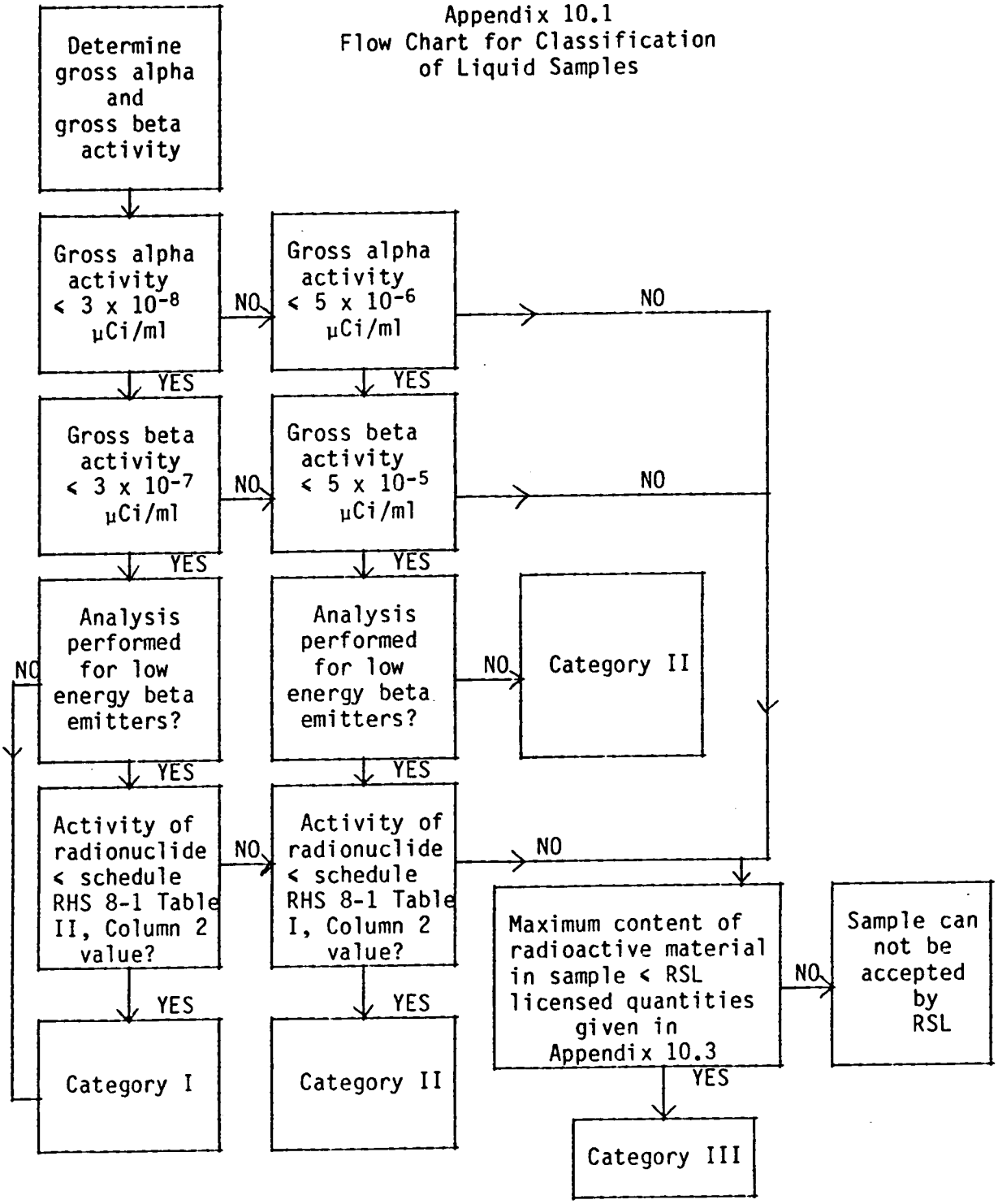
Appendix 10.1
Flow Chart for Classification of Liquid Samples



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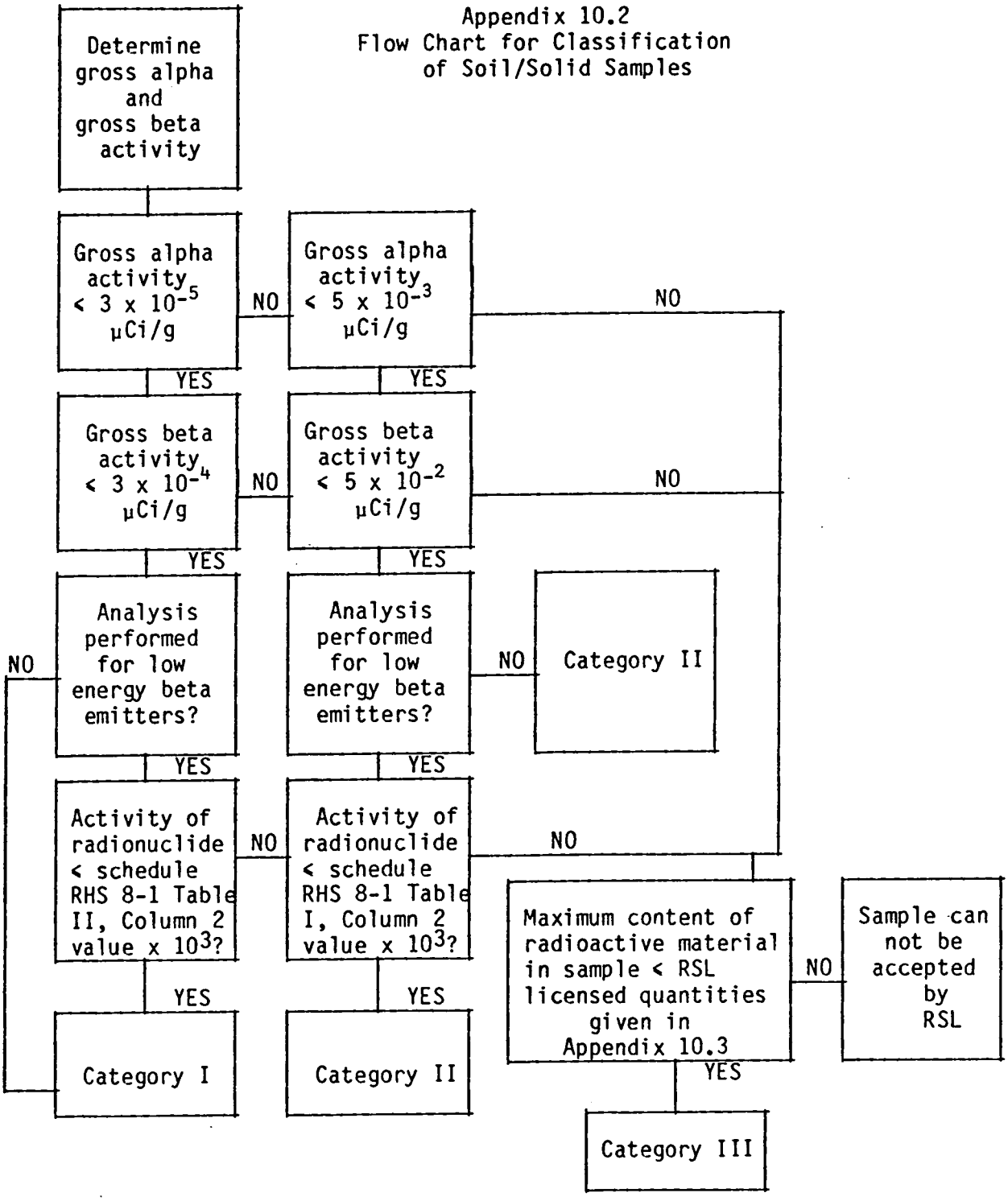
Appendix 10.2
Flow Chart for Classification of Soil/Solid Samples



Appendix 10.3
IT/Radiological Sciences Laboratory
Mixed Waste Sample Screening

The maximum content of radioactive material in any sample delivered to IT/RSL cannot exceed the following:

Radionuclide	Maximum Sample Content
U-233	0.03 gram *
U-235	0.03 gram *
Plutonium	0.03 gram *
Am-241	1 μCi
Ra-226	1 μCi
Ra-228	1 μCi
Natural Uranium	1000 μCi
Natural Thorium	1000 μCi
Mixed Activation/Fission Products	2000 μCi

* Total amount of samples not to exceed 350 grams of U-235, 200 grams of U-233, 200 grams of Pu, or the sum of such ratios of special nuclear material in combination shall not exceed 1.

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Appendix 10.4
IT/Radiological Sciences Laboratory
Mixed Waste Sample Screening

Customer identification: _____

Laboratory number: _____

Sample matrix: Liquid _____ Soil/Solid _____ Air Filter _____

Results (μCi/ml): Gross Alpha _____ Gross Beta _____ Other _____

Date sample counted: _____

Sample category: _____ *

Sample activity below RSL license limits _____ Yes _____ No

Precautions _____

_____

_____

_____

By: _____ Date: _____

* Only Category I samples can be transferred to other IT laboratories.

Category III samples require special handling instructions provided by IT Health Physics personnel.