



*Idaho
National
Engineering
Laboratory*

INEL/EXT-97-00073

January 1997

**APNEA/WIT System
Nondestructive Assay Capability
Evaluation Plan for Select
Accessibly Stored INEL RWMC
Waste Forms**

RECEIVED

JUN 09 1997

OSTI

G. K. Becker

MASTER

LOCKHEED MARTIN



**APNEA/WIT System Nondestructive Assay
Capability Evaluation Plan
for Select Accessibly Stored
INEL RWMC Waste Forms**

G.K. Becker

Published January 1997

**Idaho National Engineering Laboratory
Nuclear Engineering Department
Lockheed Martin Idaho Technologies Company
Idaho Falls, Idaho 833415**

**Prepared for the
U.S. Department of Energy
Assistant Secretary for
Environmental Management
Under DOE Idaho Operations Office
Contract DE-AC07-94ID13223**

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

CONTENTS

ACRONYMS	vii
1. INTRODUCTION	1
1.1 General	1
1.2 Purpose	1
1.3 Test Plan Objectives	2
1.4 Conditions and Limitations of the Test Plan	2
1.4.1 Participant Requirements	3
1.5 General Description of RWMC Accessibly Stored TRU Waste Forms Selected for Use in the Evaluation	3
1.5.1 IDC 001 - Solidified Aqueous Waste, First Stage Sludge: (19.8% RWMC accessible storage inventory)	4
1.5.2 IDC 003 - Solidified Organic Waste, Organic Setups: (7.7% RWMC accessible storage inventory)	4
1.5.3 IDC 007 - Solidified Aqueous Waste, Wet Sludge-Bldg 374: (18% RWMC accessible storage inventory)	4
1.5.4 IDC 300 - Graphite: (4.5% RWMC accessible storage inventory)	4
1.5.5 IDC 330 - Dry Combustibles: (0.7% RWMC accessible storage inventory)	5
1.5.6 IDC 336 - Wet Combustibles: (1% RWMC accessible storage inventory)	5
1.5.7 IDC 376 - Processed Insulation and Filter Media: (6.7% RWMC accessible storage)	5
1.5.8 IDC 440 - Glass, <i>Except Raschig Rings</i> : (1.7% RWMC accessible storage inventory)	5
1.5.9 IDC 442 - Unleached Raschig Rings: (2.7% RWMC accessible storage inventory)	6
1.5.10 IDC 480 - Light Metal: (3% RWMC accessible storage inventory)	6
1.5.11 IDC 481 - Leached Light Metal: (0.4% RWMC accessible storage inventory)	6
2. DEFINITIONS	7
3. TEST PLAN COORDINATION	9
4. SURROGATE/ACTUAL RF TEST SAMPLE PREPARATION AND STAGING	11
4.1 Test Plan Apparatus	11
4.1.1 Surrogate Test Apparatus	11
4.1.2 Actual Rocky Flats Plant Waste Test Samples	13
4.1.3 APNEA/WIT Test Sample Set	14
4.2 Surrogate Test Sample Preparation and Control	18
4.3 Actual Rocky Flats Test Sample Staging and Control	20

5.	ANALYTICAL AND DATA REPORTING REQUIREMENTS	23
5.1	WIT Analysis	23
5.2	APNEA Analysis	23
5.3	Reporting	23
	5.3.1 Standalone Initial Report Requirements/Schedule	23
	5.3.2 Extended Standalone Reporting Requirements/Schedule	24
	5.3.3 Integrated WIT/APNEA Reporting Schedule	24
6.	QUALITY ASSURANCE	25
6.1	Measurement System Quality Control	25
6.2	Test Plan Apparatus Quality Control	25
7.	CAPABILITY EVALUATION CRITERIA AND TECHNIQUE	25
7.1	Evaluation Criteria	26
7.2	Performance Assessment Parameters and Scoring	27
	7.2.1 Precision and Precision Scoring	28
	7.2.2 Total Bias and Total Bias Scoring	30
	7.2.3 Accuracy	32
	7.2.4 Minimum Detectable Concentration	33
	7.2.5 Total Uncertainty	33
	7.2.6 Overall Performance	33
8.	REPORTING OF PERFORMANCE DATA	33
8.1	Summary of Data	34
8.2	Reporting With Argonne-West Radiochemistry Data	34
8.3	Distribution of Data	34
9.	REFERENCES	35

Appendix A—WIT/APNEA Capability Evaluation Project Forms

TABLES

1. Available radioactive standards	12
2. Available surrogate matrix drums	13
3. WIT test sample set	15
4. APNEA test sample set	16
5. Quality assurance objectives for nondestructive assay	26
6. Measured relative precision requirement adjusted for eight replicates	27
7. TRUPACT-II requirements (per container)	27

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

ACRONYMS

APNEA	Active Passive Neutron Examination Assay
BIR	Bio-Imaging Research Inc.
EDL	Economic Discard Limit
FGE	Fissile Gram Equivalent
IDC	Item Description Code
INEL	Idaho National Engineering Laboratory
LMSC	Lockheed Martin Speciality Components
MDC	Minimum Detectable Concentration
NAD	Nuclear Accident Dosimeter
NDA	Non-destructive Assay
NDE	Non-destructive Examination
NIST	National Institute of Standards and Technology
PDP	Performance Demonstration Program
QAO	Quality Assurance Objective
QAPP	Quality Assurance Program Plan
RSD	Relative Standard Deviation
RWMC	Radioactive Waste Management Complex
SWEPP	Stored Waste Examination Pilot Plant
TRU	Transuranic
ZPPR	Zero Power Plutonium Reactor

APNEA/WIT System Nondestructive Assay Capability Evaluation Plan for Select Accessibly Stored INEL RWMC Waste Forms

1. INTRODUCTION

1.1 General

Bio-Imaging Research Inc. (BIR) and Lockheed Martin Speciality Components (LMSC) are engaged in a Program Research and Development Agreement and a Rapid Commercialization Initiative with the Department of Energy, EM-50. The agreement requires BIR and LMSC to develop a data interpretation method that merges nondestructive assay and nondestructive examination (NDA/NDE) data and information sufficient to establish compliance with applicable National TRU Program (Program) waste characterization requirements and associated quality assurance performance criteria. This effort requires an objective demonstration of the BIR and LMSC waste characterization systems in their standalone and integrated configurations. The goal of the test plan is to provide a mechanism from which evidence can be derived to substantiate nondestructive assay capability and utility statements for the BIR and LMSC systems. The plan must provide for the acquisition, compilation, and reporting of performance data thereby allowing external independent agencies a basis for an objective evaluation of the standalone LMSC and BIR measurement systems, APNEA and WIT respectively, as well an expected performance resulting from appropriate integration of the two systems. The evaluation is to be structured such that a statement regarding select INEL RWMC waste forms can be made in terms of compliance with applicable Program requirements and criteria.

1.2 Purpose

The purpose of the test plan is to provide a method and criteria for the evaluation of APNEA/WIT utility relative to nominal configurations of large population fraction waste types in inventory at the Idaho National Engineering Laboratory (INEL) Radioactive Waste Management Complex (RWMC). Utility in this context refers to the ability to acquire and reduce nondestructive characterization data in compliance with the Program Quality Assurance Program Plan¹ (QAPP) requirements and quality assurance objectives. To this extent, the test plan method and evaluation criteria is to be founded in and consider, to the extent practicable, the requirements and quality assurance objectives of the QAPP.

The capability evaluation test sequence is to commence in January of 1997 with a duration of approximately 20 working days. It is recognized that the scope of the test is limited and will not provide all data necessary to completely assess the ability to demonstrate compliance with applicable nondestructive assay requirements and criteria. The test is nevertheless intended to yield capability information regarding key functional parameters which the system/technology must be able to accommodate.

1.3 Test Plan Objectives

The main objective of this test plan is to delineate the methodology which will be used to evaluate the APNEA and WIT system capability. The test plan structure and scope is to be sufficient to allow an objective evaluation of both APNEA and WIT functional capability and performance with respect to select INEL RWMC accessibly stored waste forms. Functional capability refers to the ability to accommodate the various attributes and characteristics known to be associated with certain waste types. The APNEA/WIT technology is to be subject to representative samples of INEL RWMC waste form types/attributes in a manner in which statements can be made regarding the potential utility and performance of these systems at the RWMC, integrated or otherwise. Performance refers to nondestructive assay system quantitation ability in terms of Program requirements and quality assurance criteria. Although the foundation and overall objective of the test plan is to evaluate capability to comply with applicable Program requirements and criteria, constraints on the project do not allow a complete compliance demonstration assessment. To the extent practicable Program requirements and quality assurance objectives are evaluated per the current version of the QAPP. Where this is not feasible, test plan protocols are to be devised in a manner which yields compliance demonstration data and information which can be interpreted relative to the established requirements and criteria. The data acquisition protocols and performance scoring technique are addressed in this plan. The reduced data and scoring results will be detailed in APNEA/WIT Nondestructive Assay Scoring Report. The overall statement of capability is made in a separate report, the APNEA/WIT Capability Evaluation Project Report, prepared through the collective efforts of the RCI Committee. It is not the intent of the test plan to extrapolate APNEA/WIT capability or utility beyond the waste form set comprising the identified test space.

1.4 Conditions and Limitations of the Test Plan

The primary condition of the test plan is that the APNEA and WIT measurement systems, presently in an RWMC waste form data acquisition and development phase, declare prior to commencement of the test plan, readiness for evaluation. At this point the APNEA and WIT measurement acquisition components of the software and the hardware configuration become fixed for the duration of the testing sequence and all evaluated data/information contained in the APNEA/WIT Nondestructive Assay Scoring Report is referenced to the stated configuration. APNEA and WIT personnel will be allowed a second reporting period where the data analysis routines for the APNEA and WIT standalone systems can be modified based on test plan measurement experience. The second reporting period does not allow modification to data acquisition routines or the hardware configuration. A third reporting period is also provided for reporting those results obtained with the an integrated APNEA/WIT system.

Due to the scheduled INEL waste assay system evaluation program² to be conducted in the summer of 1997 some limitations or constraints are placed on the APNEA/WIT test plan. The summer evaluation and demonstration program is being held at the INEL for the purpose of testing and evaluating mobile waste assay systems. Because APNEA and WIT are expected to participate in the INEL test sequence, confidentiality is required for test samples utilized in the APNEA/WIT capability evaluation project to ensure the integrity of the INEL test plan and avoid potential performance evaluation conflicts. Maintaining confidentiality of the current test plan test samples allows them to be utilized in the summer INEL test plan negating the need to specify new surrogate configurations and select another set of actual RF test samples.

Other limitations of the test are related to the preparation of surrogate test samples sufficient to provide for a comprehensive performance evaluation. The surrogate matrix drum and radioactive material standard set presently at the INEL RWMC SWEPP facility is finite. Although a large number of combinations can be realized with the available set of matrix surrogate and radioactive standards, certain parameters of interest cannot be tested. Of particular interest is varying radionuclidic and isotopic

compositions of the radioactive material configurations observed in the waste form inventory cannot be simulated with the present standard set. The radioactive standard set presently at the SWEPP facility available for use in support of the project is comprised of weapons grade plutonium of various denominations and configurations. This will not be an issue in the INEL summer test/demonstration program as additional standards are being procured to address these test parameters.

Another limitation regarding the testing of functional system parameters of interest is the time duration of the testing sequence itself. The number of actual test samples which can be processed through the APNEA and WIT is confined by the allowable test plan duration. The test samples represent the envelop of the capability evaluation and therefore define the space for which statements of utility and capability can be made.

A final limitation of the test is related to the difference in throughput rates of the APNEA and WIT systems. The APNEA throughput rate is greater than that of the WIT system thereby limiting the scope of testing for integrated system capability evaluation to the throughput rate of the WIT system. This being the case, more information regarding the APNEA in a standalone mode can be acquired than the WIT in a standalone mode and in a APNEA/WIT integrated mode. Hence the scope of waste form attributes and characteristics which can be evaluated for the WIT standalone and APNEA/WIT integrated system configurations is reduced relative to the standalone APNEA system.

1.4.1 Participant Requirements

Participants must fully detail, in advance, the configuration of system for the test sequence. The participants are to provide a written notice of their respective hardware configurations, e.g., drawing numbers, and the data acquisition software, e.g., version, to the Project Referee. The Project Referee will maintain the configuration descriptions in the project file for future reference. Software configuration management documentation will also be solicited from the participants to ensure adequate control of software versions and hardware configurations is in place. This requirement allows qualification of performance per the specified system configuration thereby linking all measurement data and subsequent capability statements to a common reference base.

Prior to commencement of the test sequence, a list of all surrogate configurations and actual RF waste containers which have been processed through either/ both the APNEA and WIT systems must be provided to the Project Referee. This requirement allows the Project Referee to ensure that surrogate configurations and RF waste containers are not specified as test samples for the project.

1.5 General Description of RWMC Accessibly Stored TRU Waste Forms Selected for Use in the Evaluation

Included in this section are brief descriptions of RWMC accessibly stored waste forms which have been selected for use in the APNEA/WIT capability evaluation project. The description includes general material composition of the waste type, generation process, typical radioactive emission characteristics, and nominal as packaged configurations.^{3,4} The selected waste type set does not include all accessibly stored waste types at the RWMC but is a reasonable representation of those waste types comprising the bulk of the population. In effect this states that the results of the testing are broadly applicable to waste forms falling within the envelope of the described waste type.

1.5.1 IDC 001 - Solidified Aqueous Waste, First Stage Sludge: (19.8% RWMC accessible storage inventory)

Waste configurations in this category consist of aqueous sludges generated by liquid waste treatment operations in Building 774 at the Rocky Flats Plant. Aqueous waste from numerous buildings and processes at the Plant were received in Building 774 where they were treated to remove radioactive/chemical contaminants and subsequently converted to a solid for disposal. The various generation processes and intermixing of aqueous waste sources leads to the diversity of elemental, chemical, and radionuclidic compositions, which are known to comprise this waste form.

Item description code 001, termed first stage sludge, consists of immobilized materials generated from first-stage treatment operations in Building 774. Aqueous liquids coming into the process originated from Building 771 recovery operations. The liquids were made basic with sodium hydroxide to precipitate iron, magnesium, etc., which also carried down the relatively small quantities of plutonium and americium hydrated oxides. The precipitate was filtered to produce a sludge (IDC 001) which was placed in a drum with Portland cement. Beginning in 1979, sludge waste from second-stage treatment was combined with first-stage sludge. Second-stage sludge (IDC 002) consists of immobilized materials generated from second-stage treatment operations in Building 774. The combined sludges were also designated as IDC 001.

1.5.2 IDC 003 - Solidified Organic Waste, Organic Setups: (7.7% RWMC accessible storage inventory)

The waste configuration typical of this category consists of organic liquid wastes that were solidified in Building 774. Liquid wastes were generated in numerous buildings on the plant site, but originated primarily from Building 707 and 777. The majority of the liquids were oil and chlorinated solvents generated from the machining and degreasing of plutonium metal in Buildings 707 and 777. Organic setups (IDC 003) consist of various organic liquids that were transferred to Building 774 where they were mixed with Microcel-E (a synthetic calcium silicate) to form a grease or past-like material. Small amounts of Oil-Dri were sometimes added to the mixture as well.

1.5.3 IDC 007 - Solidified Aqueous Waste, Wet Sludge-Bldg 374: (18% RWMC accessible storage inventory)

Waste forms categorized in this group consist of sludges generated by liquid waste treatment operations in Building 374 at the Rocky Flats Plant. Aqueous waste from numerous buildings and processes at the plant were received in Building 374 where they were treated to remove radioactive and chemical contaminants. Chemical contaminants were removed using evaporation. Radioactive contaminants were removed using neutralization, precipitation, flocculation, and clarification techniques. The slurry containing the radioactive contaminants was filtered producing a moist sludge. The sludge was either dried or processed to a moist sludge subsequently mixed with Portland cement/water or a diatomite/Portland cement/water mixture.

1.5.4 IDC 300 - Graphite: (4.5% RWMC accessible storage inventory)

Waste in this category consists of graphite generated by production, recovery, laboratory, size reduction, and research and development activities associate with plutonium operations. Graphite waste includes broken molds, furnace liners and spacers, graphite material in configurations ranging from chunks to small pieces, and some laboratory equipment. The waste was generated in Buildings 371, 559, 707, 771, and 776. Graphite items include molds from plutonium casting operations, spacers and liners used in high temperature furnaces and ovens, electrodes, and pieces and chunks generated during mold cleaning. Although the waste is primarily molds from plutonium casting operations in Building 707,

limited amounts of graphite molds were periodically generated by various research and development projects. IDC 300 may also include graphite electrodes from Building 559 laboratory operations. Graphite contaminated with plutonium above the economic discard limit was sent for recovery in Buildings 371 or 771.

1.5.5 IDC 330 - Dry Combustibles: (0.7% RWMC accessible storage inventory)

This waste IDC consists of dry combustible materials generated by the plutonium production, recovery, treatment, laboratory, and maintenance operations in Buildings 371, 374, 559, 707, 771, 774, 776, 777, and 779. Dry combustibles consist of primarily cloth, paper, and wood wastes including items such as wipes, towels, rags, coveralls, booties, gloves, and wood filter frames. Dry combustibles may contain up to 50% plastic and 10% other waste items including metal, glass and leaded gloves. Dry combustibles may be contaminated with any of the solvents, acids, bases, and other reagents used in the processes in which they were generated.

1.5.6 IDC 336 - Wet Combustibles: (1% RWMC accessible storage inventory)

Waste comprising this IDC consists of wet combustibles generated by the plutonium production, recovery, treatment, laboratory, and maintenance operations in Buildings 371, 374, 559, 707, 771, 774, 776, 777, and 779. Wet combustibles consist primarily of cloth, paper, and wood wastes including items such as wipes, towels, rags, coveralls, booties, gloves, and wood filter frames. Wet combustibles contain discernible amounts of process liquids. Wet combustibles may contain up to 50% plastic and 10 percent of other waste items including metal, glass, and leaded gloves. Wet combustibles may be contaminated with any of the solvents, acids, bases, and other reagents used in the processes in which they were generated.

1.5.7 IDC 376 - Processed Insulation and Filter Media: (6.7% RWMC accessible storage inventory)

This waste consists of Ful-Flo incinerator filters, (IDC 328), absolute drybox filters (IDC 335), and insulation and filter media (IDC 338) that were wet or had been exposed to corrosive fumes. IDC 328 consists of Ful-Flo filters from the recovery incinerator Building 771. Ful-Flo filters were in-line cartridge filters designed to remove particulates from liquid streams. The filters were one-piece, molded filters about 10 inches long by 3.5 inches diameter. Filter media consisted of a red fibrous material which filtered particulates greater than 5 microns. Five and one micron fibrous polypropylene filters were also used. Ful-Flo filters may contain caustic free liquids. IDC 335 consists of glovebox air intake and exhaust HEPA filters. Filter sizes include 8x8x6 inches, 8x8x4 inches, and 12x12x6 inches. Filter frames are constructed of either fire-retardant plywood or particle board and cadmium-plated or chromized carbon steel. The filter media is made of Nomex (glass and aromatic polyamide fibers), fiberglass, or asbestos. This waste includes acid, nonacid, and solvent contaminated filters. The waste may also contain limited amounts of combustibles materials. The IDC 338 waste type consists primarily of filter media removed from various types and sizes of filters and includes asbestos or fiberglass pipe and furnace insulation, fire blankets, and asbestos gloves. Filter media that was wet or had been exposed to corrosive fumes was processed in Building 776 as IDC 376.

1.5.8 IDC 440 - Glass, *Except Raschig Rings*: (1.7% RWMC accessible storage inventory)

This waste consists of glass generated by plutonium production, recovery, treatment, laboratory, and maintenance operations in Buildings 371, 374, 559, 707, 771, 774, 776, 777, and 779. The waste consists of items such as bottles, vials, light bulbs, labware, glovebox windows, and process equipment. The materials may be glass, ceramic, leaded glass, or quartz. The waste may also contain limited amounts of metal, plastic rubber, and combustibles.

1.5.9 IDC 442 - Unleached Rachig Rings: (2.7% RWMC accessible storage inventory)

This waste consists of Raschig rings which are borosilicate glass rings used to ensure subcritical conditions in fissile solution storage tanks that were not safe by dimension. When the rings were removed from the tanks they were assayed. If the plutonium content was below the EDL they were assigned IDC 441. Raschig rings that were contaminated above the EDL were reclassified from IDC 441 to IDC 442. The IDC 442 rings were leached in nitric acid to remove the plutonium contaminant adhering to the ring surface.

1.5.10 IDC 480 - Light Metal: (3% RWMC accessible storage inventory)

This waste consists of light metal generated by the plutonium production, recovery, treatment, laboratory, and maintenance operations in Buildings 371, 374, 559, 707, 771, 774, 776, 777, and 779. Light metal includes iron, copper, aluminum, brass, bronze, galvanized metal, stainless steel, carbon steel, and other metal alloys. The metals consist of mechanical and electrical parts, tools, containers, scrap metals, piping, wire, cable, gauges, valves, foil, electric motors, planchets, and a variety of other metal items. The metals may be contaminated with residual amounts of solvents, acids, bases, and other reagents used in the processes where they were generated. Solvent contaminated were not sorted from nonsolvent contaminated metals. Beryllium, pyrophoric metals, and heavy non-SS metal are excluded from IDC 480. The waste may also contain limited amount of combustible wastes.

1.5.11 IDC 481 - Leached Light Metal: (0.4% RWMC accessible storage inventory)

This waste consists of light metal that was washed with hot water in Building 776 to remove radioactive surface contamination. Leached light metal consists of the same metals comprising IDC 480 and originated from the same buildings and processes as IDC 480.

2. DEFINITIONS

Accuracy. The closeness of measured value to the true or to an accepted reference or standard value.

Actual Drums. A test sample selected from the set of pre-specified Rocky Flats Plant generated RF Matrix drums. Actual matrix drums must be evaluated as appropriate by waste NDA experts to determine the most probable radionuclidian composition and mass loading thereof in addition to the overall associated uncertainty before the drum can be considered a test sample for use in the evaluation.

Bias. The systematic error component of the total uncertainty, i.e., a constant positive or negative deviation of the method average from the correct or accepted reference value under specific measurement conditions.

Instrument Bias. The bias of an instrument or measurement system under essentially ideal conditions, i.e., when all sample specific or matrix effects have been minimized relative to the calibration and functional space of the system.

Precision. The standard deviation of a specified number of replicate measurements of an identical test sample under controlled conditions.

Project Administrator. The Project Administrator has overall responsibility for the implementation of the capability evaluation plan. The Project Administrator shall interface with external review agencies and agencies having an interest in the project.

Project Coordinator. The Project coordinator is responsible for assuring the implementation of the WIT/APNEA capability evaluation project. The Project Coordinator reports to the Project Administrator.

Project Referee. The Project Referee is responsible for specifying the test samples employed in the test series, the collection of assay results per project schedule, analysis of assay results per scoring criteria and preparation of the WIT/APNEA Capability Evaluation Scoring report.

RWMC Representative. RWMC single point of contact responsible for the receipt and review of the test drum list, preparing surrogate test samples; staging surrogate and actual RF test samples per project schedule; gathering actual RF drum preliminary data and supplying to the project referee; ensuring confidentiality of test samples; and interfacing with the project coordinator, project referee, and APNEA/WIT Representative.

RWMC Sample Attestant. Responsible for verifying the preparation and documentation of test samples and overseeing test sample chain of custody.

Surrogate Matrix Drum. A surrogate matrix drum is a DOT 17C 208-L (55-gallon) drum fabricated with a matrix configuration containing attributes representative of specific Rocky Flats Plant generated waste types. The term surrogate matrix drum includes matrix drums employed in the National TRU Program NDA PDP Program.

Surrogate Test Sample. A blind sample prepared to referee specified configuration through the use of a surrogate matrix drum and radioactive WRM(s). Test samples also include actual Rocky Flats Plant generated waste forms for which a reasonable estimation of the radioactive material contents is established.

Total Uncertainty. The total measurement error from all bias and precision elements due to waste form attributes such as variable matrix elemental compositions, matrix density distributions, radioactive material chemical compound/radionuclidian composition, radioactive material physical form, etc.

WRM. A radioactive standard configured with surrogate matrix drums to produce a sample for test purposes. The majority of WRMs used in this test evaluation plan are NIST traceable. Those which do not have NIST traceability are noted as such and considered accordingly in the evaluation of results.

3. TEST PLAN COORDINATION

Successful implementation of the test and evaluation plan requires the collective efforts of a number of individuals and groups. The responsibility for implementation is distributed through the RCI working committee, the Project Referee, INEL RWMC operations, APNEA/WIT personnel, identified waste NDA experts, the RWMC fissile material custodian, and the Project Coordinator.

The Project Coordinator shall be responsible for overseeing implementation of the test/evaluation project and ensuring the overall project schedule is adhered to. The Project Coordinator is to manage conduct of the test/evaluation plan from its inception through the delivery of the final APNEA/WIT Capability Evaluation Scoring Report to the Project Administrator. It is a fundamental task of the Project Coordinator to ensure communications necessary to implement the plan are established and maintained through the duration of the test series.

The Project Coordinator is also responsible for resolving conflicts which may impact the test plan objectives and schedule. The Project Coordinator shall notify the Project Administrator, Project Referee, and the RCI committee of events delaying the test sequence. Test plan delays may require that the Project Coordinator, Project Referee, Project Administrator, APNEA/WIT representative, and the RCI committee to adjust the plan to maximize the acquired information. In such an event, it is anticipated that plan objectives can still be maintained through the judicious modification of the test sample replicates, sequence, and associated configurations. Hence the prompt identification of unforeseen delays by the Project Coordinator followed by notification of RCI team members is important to the acquisition of useful capability evaluation data and ensuring test plan objectives are not unduly compromised.

The Project Coordinator shall identify responsible single point-of-contact representatives for each of the primary entities participating in and supporting the test and evaluation project. Each representative is to coordinate individuals and organizations regarding the tasks under their purview per project plan and schedule. There are four single point-of-contact representatives which must be established and be cognizant of the test/evaluation plan details and scope. These representatives are; (1) RWMC Representative, (2) APNEA/WIT Representative, (3) Project Referee, and (4) RWMC Sample Attestant.

The RWMC Representative is responsible for the overall coordination of RWMC operational resources required to support the test/evaluation project. The RWMC Representative shall ensure the retrieval, conditioning, and staging of actual RF test samples per project schedule, preparation, and documentation of surrogate test samples per project schedule, gathering RF generator, and SWEPP characterization test sample data and supplying to the project referee, ensuring confidentiality of test samples, and interfacing with the Project Coordinator, Project Referee, RWMC Sample Attestant, and the APNEA/WIT Representative. The RWMC Representative is to be duly qualified to prepare and handle surrogate test samples.

The RWMC Representative is responsible for the transmittal, tracking, and retrieval of prepared surrogate test samples and actual RF test samples. The RWMC Representative shall maintain the integrity and configuration of a surrogate test samples returned from the APNEA/WIT Representative after the measurement procedure until notified by the Project Referee that disassembly of the surrogate sample is approved. The RWMC Representative is responsible for acquiring APNEA/WIT test measurement data/information and transmittal of such test measurement data/information to the Project Referee per project schedule. Operational constraints, e.g., limitations on fissile material sample loadings per control area, etc., are to be managed by the RWMC Representative in consultation with the Project Referee.

An RWMC Sample Attestant is to be identified to function in the capacity of an independent quality assurance observer. The RWMC Sample Attestant is to ensure test samples are properly identified,

prepared (in the case of surrogate test samples) and staged. The RWMC Sample Attestant shall ensure the mechanics of test sample preparation and distribution are appropriately executed per plan direction. The RWMC Sample Attestant shall maintain confidentiality of surrogate test samples configurations, data/information on actual RF test samples and associated documentation thereof.

The APNEA/WIT Representative is responsible for interfacing with the RWMC Representative to maintain the test/evaluation project schedule, receipt of test samples from the RWMC Representative, coordinating transfer of the WIT sample subset between APNEA and WIT, return of test samples to the RWMC Representative, transmittal of specified APNEA and WIT test sample measurement data to the RWMC Representative per schedule, and reporting measurement system related delays or potential delays to the RWMC Representative and Project Coordinator.

The Project Referee is responsible for specifying the test sample set including surrogate and actual RF drums, acquiring, reviewing and compiling test data, evaluating capability per scoring criteria, and preparation of interim and/or final APNEA/WIT Capability Evaluation Scoring Reports. The Project Referee is responsible for interfacing with the APNEA/WIT Representative regarding the appropriate format for reporting measurement data for evaluation purposes. The Project Referee shall prepare detailed instructions for the assembly of surrogate test samples configured to meet the objectives of the test/evaluation project. The Referee may also direct the sequential delivery of surrogate and actual RF test samples should it be deemed useful to the capability evaluation.

The Project Referee shall acquire APNEA/WIT measurement data from the RWMC Representative. The Project Referee shall review and notify the RWMC Representative of approval to disassemble surrogate test samples. The Referee is responsible for addressing and resolving issues related to technical aspects of the project regardless of source, e.g., schedule delays, fissile material loading restrictions, etc. The Project Referee is responsible for the compilation, reduction, and reporting of the measurement data/results per predetermined scoring criteria delineated in the subject Plan. The Project Referee shall ensure that confidentiality is maintained for all test sample data and configurations employed in the test in addition to all APNEA/WIT supplied measurement data/information through the entire evaluation period. The Project Referee shall not divulge any measurement data raw or reduced until the interim and/or final reports are approved for release by the RCI committee.

4. SURROGATE/ACTUAL RF TEST SAMPLE PREPARATION AND STAGING

4.1 Test Plan Apparatus

The scope of testing and waste form parameter evaluation capability effort is constrained by the waste form characteristics achievable with available test apparatus. For the purposes of APNEA/WIT capability evaluation, the test apparatus consists of surrogate matrix drums, radioactive WRMs, and actual RF drums. It is the objective of the capability evaluation project to configure test samples using an appropriate combination of surrogate matrix drums and WRMs in addition to the selection of actual RF waste containers for which a reasonable estimate of the radionuclidian content is available. Test sample configurations are to reflect nominal attributes and characteristics of the waste types specified for use in the project.

4.1.1 Surrogate Test Apparatus

The surrogate test samples, comprised of surrogate matrix drums and WRMs, are configured such that they present to the measurement system an attribute or combination of attributes characteristic of a given waste type or common to a number of waste types. It is the ability of the measurement system to accommodate such waste form configurations which is of interest in the capability evaluation project. Specified configurations are a function of the selected RF waste drum RWMC accessibly stored inventory and consider such waste form parameters as, matrix density, matrix density distributions, matrix elemental compositions, and distributions thereof, radioactive material radionuclidian/isotopic and chemical compositions, radioactive material physical form, matrix/radioactive material combinations, etc. It is the responsibility of the Project Referee to specify surrogate test samples which target, either singly or in combination, various waste form attributes with magnitudes and characteristics nominally representative of the waste forms of interest.

The apparatus available for the configuration of surrogate test samples is listed in Tables 1 and 2. The configuration of surrogate test samples must be such that given bias and/or precision element(s) representative of a specified waste type can be evaluated. The most desirable set of surrogate test samples would be one in which each bias and precision source is individually presented to the measurement system via the sample and that combinations of such follow. In that the scope of testing is limited by time, a complete test following this logic is not possible. Therefore using the apparatus listed below, it is the objective to judiciously combine surrogate matrix drums and radioactive WRMs to construct a test sample set spanning as much of the capability space of interest.

The preparation of surrogates using the radioactive standard (WRM) set in Table 1 requires some caution to maintain and qualify test and capability evaluation integrity. Some of the WRMs are of a standard denomination and are readily identifiable via radiography and/or computed tomography techniques. To preclude estimation of WRM mass via visual examination alone, the Project Referee shall examine reported data to ensure the specified data acquisition and reduction procedure was utilized per the declared hardware/software configuration to arrive at the mass estimate. Additionally the Project Referee shall endeavor to specify combinations of identifiable and nonidentifiable WRMs for emplacement into the surrogate matrix drum such that the total mass and activity cannot be easily inferred from visual examinations.

Records of WRM traceability shall be maintained in the RCI Capability Evaluation project file. For those standards which do not have traceability data, copies of available characterization data shall be included in the project file.

Table 1. Available radioactive standards.

Radioactive Standards (WRMs)
PuO ₂ Standards (WG Pu) NIST traceable
NDA PDP Set (30mg - 160g) ^c
RWMC QAO Set (8mg - 1.1g) ^b
NAD Foil Standards (1g - 11g) ^{a,b}
ZPPR Fuel Plate Standards ^{a,b}
Nontraceable Radioactive Sources
AmLi Sources (4 @ 4e ⁴ n/sec) ^b
PuO ₂ Source (40mg) ^b

- a. Note: traceability certification exists for a portion of the source set, cross correlation to the balance of set must be completed to link traceability across entire set.
- b. Usable with RWMC surrogate drum set and NDA PDP matrix drums.
- c. Usable with NDA PDP matrix drums only.

Table 2. Available surrogate matrix drums.

Waste Matrix Surrogates
SWEPP Calibration Drums (55 gal)
Base Calibration - zero matrix
IDC 300 - graphite matrix
IDC 480/481 - mixed metals matrix
IDC 440 - glass
IDC 442 - raschig ring
IDC 330 - dry combustibles
IDC 001 - 1 st Stage Sludge (LMSC surrogate)
NDA PDP Matrix Drums
Zero Matrix
Combustibles Matrix
Ethafoam Matrix

4.1.2 Actual Rocky Flats Plant Waste Test Samples

A number of Rocky Flats Plant generated waste containers (55-gallon) have been identified from the RWMC accessibly stored inventory for use in the capability evaluation project. Selection criteria for the waste types in the set is based primarily on large RWMC accessible storage inventory fraction, hence not all IDC waste types are represented in the set. The waste types included in the selected set are; IDCs 001, 003, 007, 300, 330, 336, 371, 376, 440, 442, 480, and 481. Although actual RF waste containers do not have absolute known radioactive material compositions and associated masses, their inclusion in the test sample set is important in that surrogate test samples are limited relative to the approximation of actual waste form configurations. The actual RF waste containers selected must be a reasonable representation of the waste type subpopulation from which they have been selected. For example, the attributes of an IDC 001 actual RF test sample should nominally represent those characteristics of the IDC 001 subpopulation, i.e., density, radioactive material composition, etc., Although it is acknowledged that their are outliers in terms of anomalous waste form configurations within each IDC, time constraints on the capability evaluation project do not allow an APNEA/WIT capability assessment of all configurations in inventory.

In order make APNEA/WIT capability statements relative to actual RF waste forms it is necessary to have a bounded estimate of the contents including both the radioactive and matrix composition and configuration. There exists significant information on actual RF waste containers regarding the matrix and entrained radioactive material to substantiate a APNEA/WIT capability estimate. Such information can be acquired from Rocky Flats Plant Generator data bases, Rocky Flats Plant process generation and

packaging information, evaluation of Rocky Flats Plant NDA assay technique employed per waste type,⁵ data acquired in INEL waste characterization programs, SWEPP NDA and NDE data (for containers which have been processed through SWEPP), INEL NDA uncertainty analysis program, etc. It is the responsibility of the Project Referee to compile available data on each actual RF waste container used as a test sample and determine an appropriate radionuclidic composition/mass and associated uncertainty. The Project Referee is to obtain concurrence on the assigned mass and uncertainty through coordinated reviews supplied by INEL waste NDA experts using best professional judgement. The process for arriving at actual RF test sample radioactive material mass and uncertainty is to be documented in the Capability Evaluation Scoring Report.

Of particular interest for use as actual RF test samples are those drums which have been sampled destructively via various waste characterization programs. Data acquired in destructive assay projects, primarily sludge waste types, will be used as appropriate in the mass determination process for assigning a mass and uncertainty to actual RF test samples.

4.1.3 APNEA/WIT Test Sample Set

The APNEA/WIT test sample set is comprised of surrogate test samples and actual RF waste containers. Tables 3 and 4 illustrate the number of test samples and associate IDCs for the WIT and APNEA systems respectively. The number of test samples provided to the WIT system is substantially less, eight requested by WIT for test sequence, than that of APNEA due to the present configuration and subsequent throughput rate of WIT, approximately two drums per week. The APNEA test sample set necessarily includes the eight WIT samples to allow the APNEA/WIT data integration approach to be evaluated.

Waste type attributes considered in the specification of surrogate test samples and actual RF waste containers include; alpha activity concentration, radioactive material distribution/physical form, radionuclidic composition, chemical composition/matrix compound of radioactive material (reaction products), general radiation emission characteristics of waste type, elemental composition of waste matrix, density of matrix (average and distributed), and packaging configuration of waste matrix. Surrogates are specified to provide

Table 3. WIT test sample set.

Waste Form Designator (content code ID)	Sample Type (surrogate/RF actual)	Sample ID/ Transfer Order ^a
300 ^c	RF actual	1RF/1
336 ^c	RF actual	2RF/2
440	surrogate	1SG/3
442 ^c	RF actual	3RF/4
NDA PDP Combustibles Matrix Drum	surrogate	2SG/5
376	RF actual	4RF/6
480	surrogate	3SG/7
001 ^b	RF actual	5RF/8

a. Specified samples are to be provided to the WIT measurement facility per the transfer order of Table 1.

b. Although not identified as available at the time of test plan writing, those sludge drums which have radiochemistry data derived from multiple coring performed at ANL-W or single sample radiochemistry data from ORNL are desirable for the test sequence.

c. Although not identified as available at the time of writing of the test plan, those waste containers analyzed in support of the INEL total uncertainty assessment are desirable for the test sequence.

Table 4. APNEA test sample set.

Waste Form Designator (content code ID)	Sample Type (surrogate/RF actual)	WIT Sample	Sample ID
300 ^c	RF actual (<i>replicate sample</i>)	yes	1RF
336 ^c	RF actual (<i>replicate sample</i>)	yes	2RF
440 ^c	surrogate (<i>replicate sample</i>)	yes	1SG
442 ^c	RF actual (<i>replicate sample</i>)	yes	3RF
NDA PDP Combustibles Matrix Drum (Serial # 003)	surrogate (<i>replicate sample</i>)	yes	2SG
376 ^c	RF actual (<i>replicate sample</i>)	yes	4RF
480	surrogate (<i>replicate sample</i>)	yes	3SG
001 ^b	RF actual (<i>replicate sample</i>)	yes	5RF
APNEA Only Test Set			
NDA PDP Zero Matrix Drum (Serial # 001)	surrogate (<i>replicate sample</i>)	no	4SG
300	surrogate (<i>replicate sample</i>)	no	5SG
300 ^c	RF actual (<i>replicate sample</i>)	no	6RF
300 ^c	RF actual (<i>replicate sample</i>)	no	7RF
300 ^c	RF actual (<i>optional</i>)	no	8RF
336 ^c	RF actual (<i>replicate sample</i>)	no	9RF
336 ^c	RF actual (<i>replicate sample</i>)	no	10RF
330	surrogate (<i>replicate sample</i>)	no	6SG
440	surrogate (<i>optional sample</i>)	no	7SG

Table 4. (continued)

Waste Form Designator (content code ID)	Sample Type (surrogate/RF actual)	WIT Sample	Sample ID
440 ^c	RF actual (<i>replicate sample</i>)	no	11RF
440 ^c	RF actual (<i>replicate sample</i>)	no	12RF
440 ^c	RF actual (<i>replicate sample</i>)	no	13RF
442	surrogate (<i>replicate sample</i>)	no	8SG
442 ^c	RF actual (<i>replicate sample</i>)	no	14RF
442 ^c	RF actual (<i>replicate sample</i>)	no	15RF
442 ^c	RF actual (<i>optional sample</i>)	no	16RF
480 ^c	RF actual (<i>replicate sample</i>)	no	17RF
480 ^c	RF actual (<i>replicate sample</i>)	no	18RF
481	RF actual (<i>replicate sample</i>)	no	19RF
376	RF actual (<i>replicate sample</i>)	no	20RF
376	RF actual (<i>replicate sample</i>)	no	21RF
376	RF actual (<i>replicate sample</i>)	no	22RF
NDA PDP Combustibles Matrix Drum (Serial # 003)	surrogate (<i>optional sample</i>)	no	9SG
001 ^b	RF actual (<i>surrogate sample</i>)	no	23RF
001 ^b	RF actual (<i>replicate sample</i>)	no	24RF
001 ^b	surrogate (<i>replicate sample</i>)	no	12SG

Table 4. (continued)

Waste Form Designator (content code ID)	Sample Type (surrogate/RF actual)	WIT Sample	Sample ID
001 ^b	RF actual (<i>optional sample</i>)	no	25RF
007 ^b	RF actual (<i>replicate sample</i>)	no	26RF
007 ^b	RF actual (<i>replicate sample</i>)	no	27RF
007 ^b	RF actual (<i>replicate sample</i>)	no	28RF
007 ^b	RF actual (<i>optional sample</i>)	no	29RF
003	RF actual (<i>replicate sample</i>)	no	30RF
003	RF actual (<i>replicate sample</i>)	no	31RF
003	RF actual (<i>replicate sample</i>)	no	32RF

- a. Samples in APNEA Only Test Set are in order of increasing difficulty with respect to the type and number of potential measurement interferences. APNEA personnel may wish to request test drums in tabulated order.
- b. Although not identified as available at the time of test plan writing, those sludge drums which have radiochemistry data obtained from core samples performed at ANL-W or single sample radiochemistry data from ORNL are desirable for the test sequence.
- c. Although not identified as available at the time of writing of the test plan, those waste containers analyzed in support of the INEL total uncertainty assessment are desirable for the test sequence.

data for the evaluation of waste form parameters of interest, e.g., specific bias/precision sources, detection limit, etc. The evaluated variables and associated ranges are to be delineated in the Capability Evaluation Scoring Report.

4.2 Surrogate Test Sample Preparation and Control

This section describes the responsibilities for and the conduct of test sample preparation and control. Considered are confidentiality of surrogate test sample surrogate configurations, transmittal of instructions for surrogate test sample configuration, responsibilities for surrogate test sample preparation, documentation of prepared test samples, transmittal of prepared sample to the APNEA/WIT Representative, control and custody of surrogate test sample during APNEA/WIT measurement sequence, and return of surrogate test sample after measurement sequence complete. The surrogate test samples identified for preparation and testing are noted for both the WIT and APNEA systems in Tables 3 and 4, respectively.

4.2.1 The Project Referee shall specify the surrogate matrix drum fissile material mass loadings, interference source loading and the overall configuration of the surrogate test samples. The surrogate test samples shall be documented as to the contents and configuration and identified for tracking and record keeping purposes. Surrogate test sample configurations and purposely incorporated bias/precision parameters will become an appendix to the Performance Evaluation Scoring Report. It is the responsibility of the Project Referee to ensure the confidentiality of the surrogate test sample configurations through the entire duration of the test/scoring/report preparation process until such time the RCI team deems dissemination appropriate.

4.2.2 The Project Referee shall transmit surrogate test sample preparation instructions to the RWMC Representative. The RWMC Representative is responsible for preparing the surrogate test sample(s) per instruction and initialing on the Project Referee supplied instruction sheet as appropriate to note completion of a particular assembly step.

4.2.3 The RWMC Sample Attestant shall duly oversee and confirm that the emplacement of the specified WRMs is per instruction and in the correct surrogate matrix drum.

4.2.4 The RWMC Representative shall secure the surrogate test sample preparation instructions and maintain confidentiality of the as prepared sample. The balance of the radioactive standard inventory must also be kept confidential during the measurement sequence.

4.2.5 The RWMC Representative shall place a security seal tape over all radioactive standard insertion tubes after assembly. The chain of custody form, see Appendix A, shall be appropriately prepared for transmittal to the APNEA/WIT Representative. The sample information form, Appendix A, shall also be prepared by the RWMC Representative, initialed by the RWMC Sample Attestant and affixed to the external surface of the surrogate matrix drum.

4.2.6 The RWMC Representative shall notify the APNEA/WIT Representative of the prepared surrogate test sample and transfer per schedule. The test sample custody form shall be signed by the APNEA/WIT Representative thereby accepting the test sample for measurement. The RWMC Representative must be cognizant of the location and responsible custodian of the surrogate test sample until its return from the APNEA/WIT Representative.

4.2.7 When the measurement sequence for a given surrogate test sample is complete, the APNEA/WIT Representative shall notify the RWMC Representative to return the sample. The chain of custody form shall be initialed by the RWMC Representative indicating completion of the measurements sequence for the sample.

4.2.8 The RWMC Representative and RWMC Sample Attestant shall inspect the security seal tape integrity, indicate seal condition on the chain of custody form and notify the Project Referee of sample return. The sample is not to be disassembled until indicated by the Project Referee. The RWMC Representative is responsible for maintaining the chain of custody form for each test sample until requested by the Project Referee for inclusion into the project records at completion of the project testing phase.

4.2.9 The RWMC Representative is to obtain the measurement report from the APNEA/WIT Representative per the applicable reporting schedule and transmit to the Project Referee. The RWMC Representative is also responsible for logging the date on which the measurement report was obtained from the APNEA/WIT Representative on the test sample data/report log, Appendix A. The test sample data/report log is used to ensure reporting is performed in accordance with project defined schedules. The Project Referee will perform a preliminary review of the

APNEA/WIT data to ensure the reporting format is correct and no discrepancies are indicated. Pending appropriate measurement sequence and data reporting, the Project Referee will notify the RWMC Representative that the sample is released for disassembly.

(Note 1: Exceptions regarding review of measurement data are indicated until APNEA/WIT system data reduction configurations are finalized. To maintain project schedule, data acquisition can occur prior to data reduction and reporting. In this case the APNEA/WIT Representative is to transmit the acquired measurement data file until data reduction can be performed. The RWMC Representative shall date and initial the appropriate column in the test sample data/report log and transmit the data file to the Project Referee. When the WIT or APNEA data reduction algorithms are finalized and the APNEA/WIT representative indicates readiness to reduce the data and submit a report the Project Referee will return the file to the APNEA/WIT Representative.)

(Note 2: In the event a given surrogate drum or WRM set maintained in a surrogate test sample pending finalization of APNEA/WIT data reduction algorithm(s) is required for the configuration of another surrogate test sample, allowance may be made by the Project Referee to disassemble the test sample prior to transfer of APNEA/WIT reduced data.)

4.2.10 During surrogate sample disassembly, the RWMC Representative and RWMC Sample Attestant shall initial the surrogate test sample instruction form verifying that the WRMs were indeed located per instruction. Discrepancies identified during this stage are to be reported to the Project Referee to ensure proper scoring and evaluation.

4.3 Actual Rocky Flats Test Sample Staging and Control

The number and associated waste form (IDC) distribution of the actual RF test samples for both WIT and APNEA are shown in Tables 3 and 4. The actual RF drum set must be retrieved, conditioned and transmitted to the APNEA/WIT Representative per project requirements. Conditioning of the actual RF test sample set refers to the covering of drum identifiers such as the barcode and Rocky Flats ID number and affixing a temporary project test sample identification number to the drum. Conditioning is required to maintain the blind nature of the testing and to preclude conflicts in subsequent test programs where members of the same actual RF test sample set are employed. In addition, the RWMC Representative is to ensure actual RF test samples retrieved from storage and placed in interim staging are not readily observable by project participants or individuals interfacing with them. This can be accomplished by retrieving and placing in interim staging more actual RF drums than are required. The RF drum set in interim staging will include those selected for a given test phase yet their identity cannot be casually identified simply due to proximity of the participants to the staging area. In a similar manner, the request for data and information on the actual RF test sample set will be in excess the set of those RF drums which are to be used in the project. This prevents individuals associated with the data/information acquisition process from knowing test sample identification and eliminates the need for multiple nondisclosure statements.

It is the responsibility of the Project Referee to identify actual RF test samples for use in the project. The specification of an actual RF test sample radioactive material mass and uncertainty is a detailed process. Information and data collected as input to the process consists of nondestructive assay computational models and data (Monte Carlo Neutron Photon), evaluation of Rocky Flats generator assay data, technique, and measurement configurations, INEL uncertainty analysis information and data⁶, process generation knowledge, SWEPP assay data, SWEPP real-time radiography images and data, waste characterization project information and data, etc. The process consists Project Referee coordinated identification and compilation of all pertinent information and data sources, best professional judgement

weighing of pertinent information and data, appropriate combination of applicable data, specification of best estimate of mass and uncertainty and a review by knowledgeable and competent individuals with expertise in nondestructive waste assay. Details of the process as well as the assigned mass and uncertainty estimates for each actual RF test sample employed in the project are detailed in the Capability Evaluation Scoring Report.

The Project Referee is to perform a preliminary evaluation of available data on a set of actual RF test drum to ensure the attributes of each drum are nominally representative of the IDC subpopulation. The Project Referee is to transmit identified actual RF test sample candidates to the RWMC Representative. The RWMC Representative will ensure appropriate coverings are placed over the RF waste drum and record transmittal of the container to the APNEA/WIT Representative in a timely manner.

- 4.3.1** The Project Referee is responsible for selection of the set of actual RWMC accessibly stored Rocky Flats Plant generated waste drums for testing. The Project Referee is to acquire all pertinent characterization data for each actual RF test sample for the purpose of determining a reasonable and defensible radioactive material mass and uncertainty. The Project Referee shall ensure confidentiality of the actual RF test sample identification numbers, compiled characterization information/data and the radioactive material mass and uncertainty estimate. The actual RF test sample identification and characterization data/information shall be maintained confidential with the RWMC Representative and the Project Referee. The actual RF test sample radioactive material mass/uncertainty estimates will be delineated in the Capability Evaluation Scoring Report using via project specific identification numbers as assigned in Tables 3 and 4.
- 4.3.2** The Project Referee shall transmit the actual RF test sample identifiers to the RWMC Representative. The RWMC Representative is to coordinate the retrieval and staging of the actual RF test samples to accommodate the measurement sequence schedule.
- 4.3.3** The RWMC Representative is to cover actual RF test sample RF ID and barcode numbers in a manner sufficient to maintain confidentiality of the sample for the duration of the APNEA/WIT measurement sequence. The RWMC Representative shall also affix the project specific test sample ID to each staged actual RF test sample. The RWMC Sample Attestant will verify proper completion of this task and duly note on the chain of custody form that conditioning of the test sample is per plan direction.
- 4.3.4** After the actual RF test samples have been prepared and staged with RF ID covers and the project test sample ID, the RWMC Representative is to notify the APNEA/WIT Representative of the prepared sample and transfer per an agreed schedule. The chain of custody form is to be initialed by the RWMC Representative indicating transfer of the sample to the APNEA/WIT Representative.
- 4.3.5** The APNEA/WIT Representative shall appropriately initial the chain of custody form indicating acceptance of the sample for the duration of the measurement series. The RWMC Representative must be cognizant of the location and responsible custodian of the surrogate test sample until its return from the APNEA/WIT Representative.
- 4.3.6** When the measurement sequence for a given test sample is complete, the APNEA/WIT Representative shall notify the RWMC Representative to return the sample. The RWMC Representative is to appropriately initial the chain of custody form indicating the sample has been returned and the measurement series for that sample complete.

- 4.3.7** The RWMC Representative and RWMC Sample Attestant shall inspect the RF ID cover to verify its integrity. The condition of the cover, intact or breached, is to be noted on the chain of custody form. The actual RF test sample covers and temporary project ID shall not be removed until the APNEA/WIT measurement data file and/or report has been transmitted to the Project Referee.
- 4.3.8** The Project Referee will perform a preliminary review of the APNEA/WIT data to ensure the reporting format is correct and no discrepancies are indicated. Pending appropriate measurement sequence and data reporting the Project Referee will notify the RWMC Representative that the sample is released for return to RWMC storage.

(Note: Exceptions regarding review of measurement data are indicated until APNEA/WIT system data reduction configurations are finalized. To maintain project schedule, data acquisition can occur prior to data reduction and reporting. In this case the APNEA/WIT Representative is to transmit the acquired measurement data file until data reduction can be performed. The RWMC Representative shall date and initial the appropriate column in the test sample data/report log and transmit the data file to the Project Referee. When the WIT or APNEA data reduction algorithms are finalized and the APNEA/WIT representative indicates readiness to reduce the data and submit a report the Project Referee will return the file to the APNEA/WIT Representative.)

5. ANALYTICAL AND DATA REPORTING REQUIREMENTS

The procedure and protocols for the measurement of test samples and transmittal of results is addressed in this section. Prior to receipt of the first test sample, both the APNEA and WIT systems shall be declared operational for purposes of executing the test plan. Written notification of the system hardware and software configurations to be employed in the test sequence shall be transmitted to the Project Referee and maintained in the project file. The configuration of the system shall not be modified or changed from this point through the duration of the test sequence and the hardware configuration and software version currently in effect stated. Separate data acquisition protocols are specified for the WIT and APNEA systems due to overall time constraints of the project and system throughput rate differences.

5.1 WIT Analysis

After receipt of a test sample, a single measurement is to be performed per the established system configuration for the test project. Analyses are to be completed and reported per the schedule delineated in Section 5.3. Precision is not evaluated for the WIT system due to throughput limitations. Minimum detectable concentration is evaluated per the configuration of one or more test samples.

5.2 APNEA Analysis

After receipt of a test sample, replicate measurements are to be performed per the established system configuration for the test project. Analyses are to be completed and reported per the schedule delineated in Section 5.3. Data for the evaluation of precision and bias is acquired through eight replicate measurements of the same test sample. The test sample must be completely removed and replaced into the system between replicate measurements. Minimum detectable concentration is evaluated per replicate data reported for of one or more test samples.

5.3 Reporting

There are three stages of reporting for the purpose of evaluating the as implemented capability; (1) standalone mode for both APNEA and WIT, (2) standalone capability after algorithm refinement for both APNEA and WIT, and (3) the APNEA/WIT data integration capability. To maintain project schedule it is acceptable that data be acquired prior to completion of final data reduction routines for either the WIT or APNEA system. After data is acquired, the APNEA/WIT Representative is to transfer a file containing the measurement data to the RWMC Representative. The RWMC Representative will indicate receipt of the file on the test sample data/report form and transfer the file(s) to the Project Referee who will secure and maintain confidentially of the file(s) until the APNEA/WIT Representative indicates they are prepared to reduce the data and report the measurement results. At that time the reporting schedule and requirements addressed below become effective.

5.3.1 Standalone Initial Report Requirements/Schedule

Reporting is to be performed in two stages for the purpose of evaluating the as implemented standalone mode capability and standalone capability after algorithm refinement. The initial reporting period requires that test sample assay report(s) be provided to the RWMC Representative at the end of the day in which the assay was performed. In the event the declared starting version of the data reduction software is not finalized, the APNEA/WIT Representative is to deliver the assay measurement data file to the RWMC Representative at the end of the day in which the assay data was acquired. Such assay measurement data will be logged and transmitted to the Project Referee and maintained confidential until the data reduction routine is finalized. After the APNEA/WIT Representative states the readiness of the data reduction algorithm, the Project Referee will return the measurement data file to the APNEA/WIT

Representative for processing. The initial report is then due one day from the return of the measurement data file to the APNEA/WIT Representative. Reports shall consist of at least the following information for each test sample:

- a. Identification of the measurement system to which the report pertains and an associated software/hardware configuration description
- b. Identification of the test sample for which the data are being reported
- c. Identification of method used to determine the quantity of each isotope, e.g., gamma mass ratio measurement, application of constant ratios, etc.
- d. For eight sample replicate sets, identification of the replicate number corresponding to the analytical data
- e. Identity and activity for each radioisotope and radionuclide identified
- f. Counting uncertainty and estimated total uncertainty for each isotope and radionuclide quantified
- g. Total ^{239}Pu fissile gram equivalent (g) and associated total uncertainty
- h. Total alpha activity and associated total uncertainty estimate
- I. Thermal power and associated uncertainty estimate
- j. Total measurement duration time (for single samples).

In addition to the reporting requirements listed, the technique for estimating total uncertainty for the various reported values is to be delineated for each measurement system. Reports addressing the rudiments of the technique are acceptable but must contain sufficient information to allow a complete evaluation by the Project Referee and waste NDA experts which may be consulted in the preparation of the Capability Evaluation Report.

5.3.2 Extended Standalone Reporting Requirements/Schedule

Reporting intended to allow consideration of measurement data acquired during the test series and potential modifications to algorithms, etc., shall be transmitted to the RWMC Representative within ten working days of the final day of the test series. In the event the final data reduction algorithm is not completed by the end of the test measurement sequence, the extend reporting period report is due 10 working days from the day the Project Referee transmits measurement data held for the duration of algorithm completion to the APNEA/WIT representative. The same requirements as listed in Section 5.3.1 are applicable for the extended standalone report. Reports which have been changed due to the extended time for data consideration are to be indicated on the report with a brief explanation of why adjustments were necessary to change the value.

5.3.3 Integrated WIT/APNEA Reporting Schedule

Reporting for the WIT/APNEA integrated results are to be transmitted to the RWMC Representative within twenty working days of the final day of the test series. The RWMC Representative shall note the receipt date on the test sample data/report form and transmit the report to the Project Referee. The same reporting criteria as listed in Section 5.3.1 is required.

6. QUALITY ASSURANCE

6.1 Measurement System Quality Control

Quality control measures based on QAPP requirements are to be implemented for both the WIT and APNEA systems. Evidence of a APNEA and WIT system Quality Control Plan or equivalent documentation shall be provided to the Project Referee for inclusion into the RCI Capability Evaluation Project file. The quality control plan shall contain provisions for the following:

1. Software configuration management
2. Hardware configuration management
3. Calibration procedures, e.g., energy calibration
4. Periodic performance check procedures, e.g., efficiency checks
5. Documentation of software
6. Software verification and validation data.

Documentation demonstrating the implementation of the above quality control parameters is required.

6.2 Test Plan Apparatus Quality Control

Apparatus used in the execution of the capability evaluation project must have pedigrees and/or documentation sufficient to establish a level of quality commensurate with the objectives of the project. Test plan apparatus consists of surrogate matrix drums, WRMs, and actual RF drums. Establishing the validity of actual RF drum radioactive material mass and uncertainty estimate and determination process is addressed in the Capability Evaluation Scoring Report. Quality control requirements for the surrogate matrix drums and WRMs are discussed in this section.

For the purpose of the project, the WRMs used to configure surrogate test samples, where practicable, are to be traceable to a certificate or other documentation that is issued by a technically competent certifying body such as the New Brunswick Laboratory or the National Institute of Standards and Technology (NIST). Because the project need is for working reference materials, documentation of WRMs is to be sufficiently rigorous to demonstrate traceability at the reference material hierarchy level. Nearly all radioactive standards available for use in the project meet this qualification. Those standards not possessing pedigrees of traceability are to have sufficient characterization data to establish confidence in the use of the standard in the project. Certificates of traceability and/or adequate characterization data for each standard used in the project are to be placed in the project file for reference purposes as appropriate.

Surrogate drums are not per se traceable entities. Detailed documentation on the specification, design, and fabrication is adequate to establish the validity of such devices for use in the project. Documentation of the design and as built configuration for each surrogate matrix drum employed in the project is required for inclusion in the capability evaluation project files.

7. CAPABILITY EVALUATION CRITERIA AND TECHNIQUE

Criteria used to evaluate APNEA and WIT capability in both standalone and integrated modes are derived from the Program QAPP, Section 9.0, Interim Change version and the TRUPACT Transportation Requirements⁷. The QAPP Section 9.0 Table 9-1 is reproduced in Table 5 indicating nondestructive assay performance evaluation parameters and the associated quality assurance objectives (QAOs). Modifications to the QAPP Table 9-1 QAOs for the precision parameter for interfering and non-interfering matrices is presented in Table 6. The precision modifications are based on proposed revisions to the Nondestructive Assay Performance Demonstration Program (NDA PDP)⁸. The proposed revisions are derived from technical discussions of the Department of Energy Program Nondestructive Assay Interface Working Group. Transportation requirements relating to nondestructive assay characterization are reproduced in Table 7. These requirements are used as a part of the evaluation but are not scored with respect to system performance. Evaluation criteria derived from the QAPP for use in the project is delineated in subsequent sections.

7.1 Evaluation Criteria

Table 5. Quality assurance objectives for nondestructive assay.

Waste Activity alpha-Ci range ^a	Nominal ^b Compliance Point, alpha-Ci (g WG Pu)	Precision ^c (%RSD)	Accuracy ^d (%R)	Total Bias ^e (%)
>0.002 - 0.02	0.008 (0.1)	≤ 20	75 - 125	low 25 high 400
>0.02 - 0.2	0.08 (1.0)	≤ 15	50 - 150	low 35 high 300
>0.2 - 2.0	0.8 (10)	≤ 10	75 - 125	low 67 high 150
>2.0	12.5 (160)	≤ 5	75 - 125	low 67 high 150

Minimum Detectable Concentration (nCi/g) ---- 60

- a. Applicable range of TRU activity in a 208-liter (55-gallon) drum to which the QAOs apply, units are Curies of alpha-emitting TRU isotopes with half-lives greater than 20 years.
- b. The nominal activity (or weight of Pu) in the 208-liter (55 gallon) drum used to demonstrate that QAOs can be achieved for the corresponding range in column 1, values in parentheses are the approximate equivalent weights of weapons grade plutonium (WG Pu), fifteen years after purification: for purposes of demonstrating QAOs, "nominal" means \pm 10 percent.
- c. Plus or minus one standard deviation based on fifteen replicate measurements of a noninterfering matrix.
- d. Ratio of measured to known values based on the average of fifteen replicate measurement of a noninterfering matrix.
- e. 95 percent confidence bounds for system bias established by studies to determine contributions to total uncertainty from all significant sources. Units are confidence bounds divided by true value, expressed as a percent. Requirement for the QAO for total uncertainty is to determine and document but no system wide values are established.

Table 6. Measured relative precision requirement adjusted for eight replicates.

Precision, noninterfering (%RSD)	Precision, interfering (%RSD)
≤ 30.3	≤ 45.4
≤ 22.7	≤ 34.0
≤ 15.1	≤ 18.5
≤ 7.5	≤ 11.3

Table 7. TRUPACT-II requirements (per container).

Parameter	Requirement
Pu-239 Fissile Gram Equivalent (FGE)	Pu-239 FGE + 2(error) < 200g
Decay Heat	Decay Heat + error < category limit

7.2 Performance Assessment Parameters and Scoring

Performance scoring and the associated statements of compliance are directly dependent upon and necessarily qualified relative to the attributes of the various test sample configurations. Surrogate test samples are derived from apparatus which can be configured to represent a given subset of waste form variables and combinations thereof. Such test samples are realized through an appropriate combination of NDA PDP, QAO, ZPPR, and NAD radioactive standards with simulated waste matrices of known composition/configuration contained in a 55 gallon DOT 17C type drum container, i.e., NDA PDP matrix and SWEPP calibration drums. The objective of the test sequence is to prepare surrogate standards in a manner simulating actual waste form configurations. The surrogate test sample permits an evaluation of system capability with respect to certain waste form attributes known to represent significant bias and precision error sources. Surrogates can be configured to test such bias and precision sources individually or in a variety of combinations known to be present in actual waste forms.

In addition to surrogate test samples, it is the intent to utilize a subset of actual RF waste containers representing a cross section of IDCs, waste types, of the accessibly stored RWMC waste inventory. Scoring of performance with actual waste containers requires that reasonable and defensible estimations of the entrained radioactive material mass and composition be established. As the estimates of radioactive material mass in actual RF waste containers will never be as exact as the knowledge of the radioactive material content of surrogates, scoring can not be as rigorous, but nonetheless will provide worthwhile capability information.

Due to time limitations, only those actual RF waste types comprising a significant fraction of the RWMC accessibly stored inventory are considered in the project. Waste types comprising small relative fractions of the RWMC accessibly stored inventory will be addressed in other testing programs as appropriate.

The evaluation and scoring of reported data for surrogate and actual RF test samples is based in the requirements and quality assurance objectives delineated in the National TRU Program QAPP. As it is not practicable to precisely duplicate the test prescriptions as called out in the QAPP or the NDA PDP, the quantification and expression of precision and bias are, where practicable, a variate of standard QAPP/NDA PDP techniques or follow other standard means of acquiring measures of bias and precision. Regardless, the intent of the scoring system is to derive information from test sample measurement data sufficient to establish an interpretation relative to the QAPP QAOs.

The QAPP QAO parameters address precision, accuracy, and total bias. QAPP precision and accuracy parameters are determined from replicate sample processing for test sample configurations characterized as noninterfering. A noninterfering test sample is characterized as a combination of matrix and radioactive standard(s) which represent minimal sources of bias and precision error for the purpose of establishing a baseline response and capability of a nondestructive assay system. The QAPP total bias QAO applies to test samples representing actual waste form configurations and the associated spectrum of precision and bias elements either singly or in combination. Similar performance objectives are found in the NDA PDP test plan required by the QAPP.

From a capability evaluation standpoint, the QAPP prescribed precision and accuracy QAOs for noninterfering samples are of less interest than the total bias QAO which is a more direct indicator of performance for actual waste form characteristics. Hence consideration of capability for the purposes of the project puts a much greater emphasis on the determination of WIT and APNEA system performance with respect to a quantifier of bias which can be related to the QAPP bias QAOs.

A standard technique for the evaluation of bias is the acquisition of a replicate measurement data set for a given test sample. Using replicate data an estimation of the mean bias and associated 95% confidence interval bounds can be obtained. Note that bias confidence interval bounds determined in this way will reflect sources of precision present in the measurement. Therefore an approximation of precision for a given test sample can be inferred from the spread around the mean for the replicate set. In general the larger the number of replicates the more refined the bias and precision estimates are. Unfortunately a limitation of the Capability Evaluation Project is time which restricts the number of replicates that can be had per test sample subsequently yielding lesser confidence regarding actual precision and bias. Regardless an approximation of precision and bias can be obtained from a diminished number of replicates sufficient for the purpose of capability estimation. Where the acquisition of replicate data per sample is not practicable, as with the WIT system, scoring must be performed on single sample data. This precludes determining a measure of precision on a per sample basis and prevents a rigorous estimation of bias.

7.2.1 Precision and Precision Scoring

Precision is a measure of the random error component of the total error or total uncertainty sometimes called the repeatability or repeatability error. As defined for the project, precision is expressed as the ratio of the standard deviation of the alpha activity derived from eight replicate measurements of a test sample to the mean alpha activity value for the replicate set. Precision is determined for a variety of surrogate and actual RF test samples for the APNEA system only.

To assess capability to comply with QAPP QAOs for precision, assay results for replicate measurements of test samples will be used. One of the test sample configurations corresponds to the nominal non-interfering matrix qualification as defined in the QAPP allowing direct comparison to the appropriate Table 5 Column 3 QAO range. The remaining test samples consist of those surrogates and actual RF samples identified in Table 4 as replicate samples.

The performance of the APNEA system for test sample (4SG) will be evaluated against the non-interfering precision QAO as a reference point. The deviation, or lack of, relative to the precision obtained with the balance of the test samples, interfering type, will be scored relative to a multiple of the non-interfering QAO, a factor of 1.5 to allow for additional expected variance associated with test samples containing inferences. The expansion of the non-interfering precision QAO to 1.5 for interfering type samples is a reasonable relaxation of the criteria and is based on projected changes to the NDA PDP Plan performance demonstration criteria.

7.2.1.1 Precision Scoring. The analytical results from eight replicate measurements of a given sample are used to calculate the relative standard deviation. Because the precision QAO criteria in Table 5 is based on 15 replicates, additional variance associated with the smaller eight replicate set required for the test project must be accounted for. This is accomplished setting the 95% upper confidence bound of the one-sided chi-square distribution equal to the QAO requirements in Table 5, Column 3. This is expressed mathematically as:

$$RSD \leq \sqrt{\frac{(R_p)^2 \chi^2_{0.05, n-1}}{n-1}} \quad (1)$$

where

- R_p = relative precision QA objective, Table 5, Column 3
- n = number of replicate samples (set to eight)
- $\chi^2_{0.05, n-1}$ = value for the 95th percentile of the chi-square distribution with $n-1$ degrees of freedom
- RSD = measured relative standard deviation for the sample, determined by

where

$$RSD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad (2)$$

x_i is a sample value and \bar{x} is the average sample value, defined by

Using these formulae, the values for the eight replicate measured precision QAOs are obtained and are

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} \quad (3)$$

tabulated in Table 6 for non-interfering and interfering types samples, Columns 1 and 2 respectively. Scoring is based on the evaluation of computed precision values relative to the criteria in Table 6. The measured precision must be equal to or less than the values listed in Table 6 for a pass score.

7.2.2 Total Bias and Total Bias Scoring

As defined for the project total bias is the fixed, systematic or constant component of the total error or total uncertainty. Bias is determined from a replicate set of measurements for the purpose of defining the mean of the bias distribution. Bias is evaluated per test sample replicate set and per combination of all test sample replicate measurements within a given IDC category. Estimates of dispersion about the bias mean are considered per the precision criteria for single sample replicate sets and multiple sample (within IDC) replicate sets per standard precision quantification techniques. The units on the Table 5, Column 5, activity range specify low and high total bias QAOs are measured or system reported low and high 95% confidence bound divided by true activity or reference value, expressed as a percent.

Another parameter is defined for purposes of the project, instrument bias. Instrument bias is the bias of a given instrument under essentially ideal conditions, i.e., no significant sources of bias outside the base calibration parameters of the instrument. The instrument bias parameter is used to evaluate control of the instrument itself, independent of known the measurement interferences and is a measure of fundamental instrument capability. Instrument bias is estimated for the first single replicate acquired on the non-interfering sample (project ID 4SG). Instrument bias is evaluated per the accuracy criteria of Table 5.

Bias for single test sample measurement data as obtained from the WIT system is evaluated and scored qualitatively as a simple ratio of the measured to known reference value as addressed in Section 7.2.2.1.1.

Bias estimation and scoring is required for both surrogate and actual RF test sample types. Bias scoring for surrogate type test samples is relatively straightforward in that the reference value is known and has little uncertainty associated with it. The reference value associated with an actual RF test sample is an estimate derived from the process described in Section 4.3 and as delineated in the Capability Evaluation Scoring Report. Because the actual RF test sample scoring technique will require an accounting of the uncertainty in the assigned reference value, the form of the equation used will vary from that used for surrogate type test samples. For this reason surrogate and actual RF test sample bias scoring is treated in two separate sections.

7.2.2.1 Surrogate Test Sample Bias Scoring. Surrogate test samples are configured with a known total alpha activity and measurement system interference source(s). This allows the presentation of bias elements in various combinations to the measurement system for evaluation purposes. Scoring of measurement system performance with respect to the Table 5, Column 5, total bias QAOs is also straightforward. The system mean total alpha activity value and 95% confidence interval low and high endpoints determined from the replicate set are ratioed to the true total alpha activity value emplaced in the as configured surrogate test sample. Scoring is pass/fail where a pass is reported if the low and high 95 percent confidence bound endpoints ratioed to the true surrogate test sample value are within the applicable total bias QAO range. A fail is reported if either the low or high replicate based 95% confidence interval endpoints ratioed to the true value are outside of the low/high total bias QAOs for the appropriate total alpha activity test sample range of Table 5.

7.2.2.1.1 Surrogate Bias Scoring: Single Test Sample Without Replicate Data - (WIT System). WIT system reported 95 percent confidence for system bias is to account for and propagate all significant sources of bias, experimentally determined or otherwise as well as error associated with

counting statistics. The confidence bounds will be ratioed to the known reference value for purposes of evaluating performance per the Table 5 total bias QAOs. It is to be noted that scoring in this manner is stricter for the WIT system in that it is effectively the total uncertainty which is being requested of the WIT system. Using the total bias QAOs for evaluation of the reported measurement data, which for the WIT system is effectively total uncertainty, is a more demanding and stringent criteria than is actually required.

7.2.2.1.2 Surrogate Bias Scoring: Single Test Sample Without Replicate Data - (Integrated WIT/APNEA System). Scoring as per Section 7.2.2.1.1.

7.2.2.1.3 Surrogate Bias Scoring: Single Test Sample With Replicate Data - (APNEA System). Bias is assessed through the acquisition of replicate test sample processing for the APNEA system. This yields an average bias with confidence interval endpoints computed for a 95% confidence. The upper and lower 95% confidence limits of the average bias are expressed using the Student's t distribution. This condition is expressed mathematically as:

$$(\bar{x} - t_{1-\alpha/2,n-1} \frac{s}{\sqrt{n}}) \leq \bar{x} \leq (\bar{x} + t_{1-\alpha/2,n-1} \frac{s}{\sqrt{n}}) \quad (4)$$

where all values are the same as specified above and

$$\begin{aligned} s &= \text{standard deviation of replicate sample set,} \\ t_{1-\alpha/2,n-1} &= \text{appropriate limit of the Student's t distribution.} \end{aligned}$$

To express the 95% confidence interval endpoints as a percentage for comparison to the Table 5, Column 5, total bias QAOs, is accomplished by dividing by the radioactive material reference value emplaced in the surrogate sample. Scoring is performed by comparing the 95% confidence interval endpoints divided by the reference value, expressed as a percent, to the limits of Table 5, Column 5.

7.2.2.1.4 Surrogate Bias Scoring: Multiple Test Sample With Replicate Data - (APNEA System). There are numerous methods to express an indicator of bias. In addition to the 95% confidence bound assessment represented in the Table 5 QAOs per test sample, it is possible to evaluate bias over a number of test samples and extract additional information on system performance. For an assessment of this nature the relative bias is expressed as a function of mass within an IDC code. This is the reason that four test sample configurations have been specified for each IDC type included in the project. The four samples can be configured to span a mass range appropriate for the IDC and the average bias computed for each of the four replicates expressed as a function of mass. The 95% confidence values of this function can be extracted from the thirty-two data points comprising the set and a comparison to the Table 5, Column 5, QAOs performed. Although not per the prescription of the QAPP QAOs additional information on system capability can be realized from this expression. For this reason the acquired data will also be analyzed and reported in this fashion. Scoring will be per Section 7.2.2.1.2 with adjustments made to the determination of the 95% confidence interval endpoints as appropriate.

7.2.2.2 Actual RF Test Sample Bias Scoring. Actual RF test sample scoring is somewhat complicated due to the uncertainty in the precise total alpha activity loading and matrix/source interference configuration. Because there is uncertainty about the true total alpha activity value an adjustment to the replicate based total alpha activity value and 95% confidence endpoints is required to

evaluate per the Table 5, Column 5, total bias QAOs. A defensible uncertainty in for the actual RF total alpha activity and a reasonable estimate of the nature and magnitude of interference source can be acquired via analysis of available RF waste form data and information. The method of accounting for the additional uncertainty in the actual RF sample in the bias scoring will be detailed in an Appendix to the Capability Evaluation Scoring Report.

7.2.2.2.1 Actual RF Bias Scoring: Single Test Sample Without Replicate Data - (WIT System). The WIT system is to report 95 percent confidence endpoints which account for and propagate all significant sources of bias, experimentally determined or otherwise, as well as error associated with counting statistics. The confidence bounds will be ratioed to the actual RF test sample determined reference value for purposes of evaluating performance per the Table 5 total bias QAOs. It is to be noted that scoring in this manner is stricter for the WIT system in that it is effectively the total uncertainty which is being requested of the WIT system. Using the total bias QAOs to the reported measurement data, which for the WIT system is effectively the total uncertainty, is a more demanding and stringent criteria for scoring than is actually required.

7.2.2.2.2 Actual RF Bias Scoring: Single Test Sample Without Replicate Data - (Integrated WIT/APNEA System). Scoring as per Section 7.2.2.2.1.

7.2.2.2.3 Actual RF Bias Scoring: Single Test Sample With Replicate Data - (APNEA System). Bias is assessed through the acquisition of replicate test sample processing for the APNEA system. This yields an average bias with confidence interval endpoints computed for a 95% confidence. The upper and lower 95% confidence limits of the average bias are expressed using the Student's t distribution.

To express the 95% confidence interval endpoints as a percentage for comparison to the Table 5, Column 5, total bias QAOs, is accomplished by dividing by the radioactive material actual RF test sample determined reference value. Scoring is performed by comparing the 95% confidence interval endpoints divided by the reference value, expressed as a percent, to the limits of Table 5, Column 5.

7.2.2.2.4 Actual RF Bias Scoring: Multiple Test Sample With Replicate Data - (APNEA System). There are numerous methods to express an indicator of bias. In addition to the 95% confidence bound assessment represented in the Table 5 QAOs per test sample, it is possible to evaluate bias over a number of test samples and extract additional information on system performance. For an assessment of this nature the relative bias is expressed as a function of mass within an IDC code. This is the reason that four test sample configurations have been specified for each IDC type included in the project. The four samples can be configured to span a mass range appropriate for the IDC and the average bias computed for each of the four replicates expressed as a function of mass. The 95% confidence values of this function can be extracted from the thirty-two data points comprising the set and a comparison to the Table 5, Column 5, QAOs performed. Although not per the prescription of the QAPP QAOs additional information on system capability can be realized from this expression. For this reason the acquired data will also be analyzed and reported in this fashion. Scoring will be per Section 7.2.2.2.3 with adjustments made to the determination of the 95% confidence interval endpoints as appropriate.

7.2.3 Accuracy

As defined for the project, accuracy is a measure of the closeness of the alpha activity mean obtained from a replicate system measurements to the known or accepted reference or standard value. Accuracy is only evaluated for the non-interfering test sample (project ID 4SG). Due to project time constraints and the present WIT throughput rate accuracy is only evaluated for the APNEA system.

7.2.3.1 Accuracy Scoring: Single Noninterfering Test Sample Replicate Data - (APNEA)

NDA results for replicate analyses for test samples of known alpha activity will be used to determine the accuracy with which a measurement facility can quantitate the total alpha activity. The accuracy of quantitation shall be computed by measuring eight replicate samples and calculating the mean total alpha activity value, \bar{x} , Equation (2). The mean total alpha activity is divided by the reference value, μ_0 (true sample value) to obtain %R. The accuracy %R result for total alpha activity shall not deviate from the reference value, μ_0 (true sample value), by more than the appropriate QAO range in Table 5, Column 4. The Table 5, Column 4, accuracy QAOs apply only to the non-interfering sample (project ID 4SG). The accuracy measurement will pass this QAO parameter if the computed %R is within the Table 5, Column 4, %R range for the appropriate test sample activity range. If the %R for the non-interfering test sample is outside the established Table 5, Column 4, accuracy limits the measurement system will be judged as unable to quantitate for that specific activity range.

Instrument bias will also be determined from the replicate data, scored per Section 7.2.2.1.3 prescription for the noninterfering type surrogate for the APNEA system only. Instrument bias will be evaluated against the Table 5, Column 4, accuracy QAOs even though the prescription for bias is used to evaluate the measured data.

7.2.4 Minimum Detectable Concentration

Minimum detectable concentration (MDC) will be evaluated per the configuration of select test samples. The MDC will be determined using the prescription delineated in Section 9.0 of the QAPP. The Capability Evaluation Scoring Report will tabulate computed MDCs for the configurations for which it was evaluated.

7.2.5 Total Uncertainty

Total uncertainty is the total measurement error from all variance sources, including the precision, the instrument bias, and interference effects such as variable matrices, isotopic compositions, spatial distributions, contaminating radionuclides, and any other interfering effect. The QAPP contains requirements to demonstrate that total uncertainty has been determined and documented at the 95% confidence level but does not specify limits for this parameter. The total uncertainty is of significance with respect to the transportation requirements in that excessive uncertainties can constrain shipping strategies and preclude transportation of certain waste types. The APNEA and WIT method of determining total uncertainty must be supplied to the Project Referee at the time the assay results for the test set are turned in.

7.2.6 Overall Performance

Measurement system performance on the entire set of test samples will be used to assess general problems that may affect WIT and/or measurement system ability to analyze total alpha activity within a 55-gallon waste drum. The criteria used for the evaluation of overall measurement facility performance is specified, as follows: Measurement systems must pass all performance criteria for an activity range demonstrated by this program to be considered qualified to perform NDA on WIPP samples for that activity range tested. The NDA results for the project test samples must meet all applicable criteria identified in Sections 7.2.1, 7.2.2.1, 7.2.2.2, 7.2.3, and 7.2.4 of this Project Plan.

8. REPORTING OF PERFORMANCE DATA

8.1 Summary of Data

The report summary, Capability Evaluation Scoring Report, shall include the values reported by both the APNEA and WIT measurement systems, the reference radionuclide activity values, the acceptance ranges, and the pass or fail status for each test parameter for each test sample per measurement system. These data will be reduced to and used to substantiate general capability statements for the systems with respect to accessibly stored RWMC waste forms included in the test project with qualifications as applicable. The Project Referee, will prepare the Capability Evaluation Scoring Report. The RCI Committee will consider the Capability Evaluation Scoring Report in conjunction with other applicable information to derive statement of capability and limitation to be documented in a APNEA/WIT Capability Evaluation Project Report. Addendums to the Capability Evaluation Scoring Report and the APNEA/WIT Capability Evaluation Project Report will be provided to update the evaluation based on data and information pertinent to the evaluation which for various reasons is not available to the Project Referee and the RCI Committee at the time the initial reports are prepared.

8.2 Reporting With Argonne-West Radiochemistry Data

Destructive sampling is scheduled for several IDC 001 sludge drums at the Argonne-West facility. Radiochemistry data acquired in this process of much interest to the RCI project in that it aids in the determination of the entrained radionuclidic composition, mass, and uncertainty for each of these test samples. Because the radiochemistry data may not be available until after completion of the Capability Evaluation Scoring Report, an addendum to the scoring report will be issued which considers the radiochemistry data in the scoring process.

8.3 Distribution of Data

Copies of the Capability Evaluation Scoring Report and the APNEA/WIT Capability Evaluation Project Report will be distributed to APNEA and WIT representatives and members of the RCI committee. Distributed beyond this contingent to other individuals and organizations shall be as deemed appropriate by the RCI Committee.

9. REFERENCES

1. *National TRU Program Quality Assurance Program Plan*, Section 9.0, Revision: Interim Change, November 11, 1996.
2. J.W. Mandler, G.K. Becker, Y.D. Harker, D.E. Menkhaus, T.L. Clements, Jr., *INEL Test Plan for Evaluating Mobile NDE/NDA Systems (Draft)*, Idaho National Engineering Laboratory, Lockheed Martin Idaho Technologies Company, January 1997.
3. *Acceptable Knowledge Document for INEL Stored Transuranic Waste - Rocky Flats Plant Waste*, Idaho National Engineering Laboratory, INEL-96/0280, October 1996.
4. *Idaho National Engineering Laboratory Code Assessment of the Rocky Flats Transuranic Waste*, Idaho National Engineering Laboratory, INEL-95/0281, July 1995.
5. B. Ulbricht, *Handbook of the Rocky Flats Plant Production Non-Destructive Assay Systems*, comp., PCCO-NDA, Rocky Flats Plant Energy Systems Group, Rockwell International, June 1984.
6. Y.D. Harker, L.G. Blackwood, T.R. Meachum, *Uncertainty Analysis of The SWEPP Drum Assay System for Graphite Content Code 300*, Idaho National Engineering Laboratory, INEL-95/0475, September 1995.
7. *NuPaC TRUPACT-II SAR*, DOE/WIPP 89-004, Revision 1, May 1989.
8. *Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program*, CAO-94-1045, Revision 0, U.S. Department of Energy, Carlsbad Area Office, National TRU Program Office, March 1995.

APPENDIX A

WIT/APNEA Capability Evaluation Project Forms

APPENDIX A

WIT/APNEA Capability Evaluation Project Forms

WIT/APNEA CEP Test Sample Chain of Custody Form

Test Sample ID: _____

Test Sample Conditioning/Preparation

Test Sample Preparation/Stage Date: _____

Actual RF Test Sample

Actual RF Test Sample ID Concealed: _____ / _____ RWMC Rep & RWMC Attestant

Test Sample per Plan: _____ Date: _____
RWMC Representative

Surrogate Test Sample

WRMs Properly Placed: _____ / _____ RWMC Rep & RWMC Attestant

Sample Info Form Attached/Sealed: _____ / _____ RWMC Rep & RWMC Attestant

Test Sample per Specification: _____ Date: _____
RWMC Sample Attestant

Chain of Custody Between RWMC and APNEA/WIT Representative

Relinquished by:	Date/Time	Received by:	Date/Time

Final Disposition	Date/Time	Disposition
RWMC Rep	/	

Comments:

WIT/APNEA CEP Test Sample Information Form

(surrogate test samples only)

Test Sample ID: _____

RWMC Representative

Date

RWMC Sample Attestant

Date

APNEA/WIT CAPABILITY EVALUATION TEST SAMPLE DATA/REPORT LOG

Report Period 1

(maintained by RWMC Representative)

(continued)

**APNEA/WIT CAPABILITY EVALUATION
TEST SAMPLE DATA/REPORT LOG
Report Period 1**

(maintained by RWMC Representative)

(continued)

**APNEA/WIT CAPABILITY EVALUATION
TEST SAMPLE DATA/REPORT LOG
Report Period 1**

(maintained by RWMC Representative)