UNCONTROLLED

Analytical and Process Chemistry

West Valley Nuclear Services Company

Laboratory Quality Assurance

Program Manual

LED0116:ENG-327

8910260175 891025
PDR PROJ
M-32 PDC

LABORATORY QUALITY ASSURANCE PROGRAM MANUAL

INTRODUCTION

This Manual represents the Quality Assurance Program that governs the operation of the Laboratories at West Valley Nuclear Services Company. The Program prescribes appropriate controls to provide effective operations and to assure that the analytical measurement data, generated and reported by the laboratories, are valid. The Program complies with the requirements of designated Quality Assurance standards and conforms to the Quality Assurance Program of the West Valley Demonstration Project.

The Manual is divided into two sections. The first section contains PRDs (Program Requirements/Description), which prescribe general requirements from the applicable Quality Assurance standards and briefly describe the implementation of those requirements. When detailed implementing instructions are required, they are contained in ACPs (Administrative Control Procedure). ACPs are found in the second section of this Manual.

This Quality Assurance Program is being broadened to accomodate the evolving needs of the West Valley Nuclear Services Laboratories. The PRDs are becoming more generic, the ACPs are more explicit and the ACMs or VLTMs are more specific.

In addition, certain Quality Assurance requirements are implemented through instructions found in technical procedures. Those ACMs (Analytical Chemistry Methods) and VLTMs (Vitrification Laboratory Test Methods) are contained in other manuals. The PRDs, ACPs, ACMs and VLTMs provide for complete implementation of program requirements and together these documents provide a comprehensive laboratory Quality Assurance program. Implementation of the program means that laboratory operations are carried out in a controlled manner, giving assurance that the measurement data reported will have been obtained by qualified analysts using approved methods and that the data will be supported by complete and retrievable records. Traceability of reported data from sample receipt, through laboratory operations, to reporting will be possible. The validity of measurement data will be verifiable.

UNCONTROLLED

LABORATORY QUALITY ASSURANCE PROGRAM MANUAL

TABLE OF CONTENTS

I. Program Requirements/Description (PRDs)

	Rev. No.	Effective Date
1.0 Organization	1	07/24/89
2.0 Quality Assurance Program	1	07/24/89
3.0 Training and Qualification	1	07/24/89
4.0 Procedures	2	07/24/89
5.0 Laboratory Records	1	07/24/89
6.0 Control of Records	1	07/24/89
7.0 Control of Equipment and Materials	1	07/24/89
8.0 Control of Measurements	1	07/24/89
9.0 Deficiencies and Corrective Actions	1	07/24/89

Organization

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

> The organization of WVNS laboratories and their position in the WVNS group is documented and described in this PRD. The organization is described in terms of structure, functional responsibilities, levels of authority, and interfaces with other organizations.

2.0 REQUIREMENTS

The location of each WVNS laboratory within the company (WVNS) organizational structure shall be identified and the internal structure of each laboratory shall be defined. The responsibilities assigned to various functional positions within each laboratory shall be documented, along with the authorities granted to carry out the responsibilities. Interfaces between each laboratory and other groups and organizations within and outside of WVNS shall be identified and described.

(Reference: Section 5., ASTM C 1009 - Standard Guild For Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry.)

3.0 PROGRAM IMPLEMENTATION

> 3.1 Organization

Within WVNS, several divisions have laboratories. The manager of each specific laboratory reports to his division manager who, according to individual divisions ultimately reports to Vice Presidents and the President. The total organizational structure of WVNS is documented by a company organization chart which is periodically updated.

> 3.2 Functional Responsibilities

The manager of each WVNS laboratory has the normal responsibilities as assigned to managers at the same management level. Other responsibilities are designated in various PRDs and ACPs.

The functional responsibilities of other WVNS laboratory personnel are outlined in Attachment A & B.

> 3.3 Levels of Authority

The manager has the normal authorities granted to managers at the same management level. The manager has the authority to approve procedures and methods used in the work of their specific laboratory.

Additional authority is granted to other laboratory personnel as necessary in the ACPs and ACMs or VLTMs.

3.4 Interfaces

> Each WVNS laboratory receives samples from various operations within WVNS. Suppliers of samples are responsible for the sampling process, taking samples, identifying samples, designating required analysis, and providing adequate sample information to each WVNS laboratory. This information must be legible and neat, written in black indelible ink; no scratch outs, white outs or erasures. One line should be drawn through mistakes, initialed or signed with the correct response written next to it. Corrections initialed or signed and dated and marked out entirely should still be legible. The reliability and tracibility of samples, from generation to submittal to the laboratory, are established and maintained by the supplier. Upon receipt, each WVNS laboratory is individually responsible for maintaining reliability and tracibility through proper handling and storage practices. When requested, individual laboratories provide guidance on sampling to help assure the reliability of samples.

> Each WVNS laboratory interfaces with various support organizations within WVNS, such as Radiation and Environmental Safety, Procurement and Quality Assurance. The support organizations provide assistance and guidance to each laboratory to help assure safety and effective lab operations.

The WVNS Quality Assurance Department is responsible for verification of compliance and implementation of this program. This responsibility is administered through the program approval cycle, and auditing.

ATTACHMENT A

FUNCTIONAL RESPONSIBILITIES

Analytical and Process Chemistry

RTS Chemist

STS Support
LWTS Support
CSS Support
Hot Cells
Gas Chromatograph
Method Development
Special Projects
Method Writing
Review and Approve
Analytical Results

VF Chemist

VF Support
ICP Spectrometer
IC
Carbon Analyzer
AA Spectrometer
Method Development
Special Projects
Method Writing
Review and Approve
Analytical Results

Radio Chemist

Radio Chemistry
Waste Classification
Gamma Spectrometer
Alpha Spectrometer
Alphabeta Counters
Method Development
Special Projects
Method Writing
Review and Approve
Analytical Results

Quality Assurance and Training Coordinator

Quality Assurance
Quality Control
Training Program
Reagents
Standards
Method Development
Special Projects
Method Writing
Review and Approve
Analytical Results

Supervisor Laboratory Operation

Oversee laboratory operation.
Oversee Radiochemical Technician
Maintain the laboratories in a clean and safe fashion.

Radiochemistry Technician C

Perform Routine Laboratory Operations
Perform Analysis within limits of qualification

Radiochemistry Technician B

Tech. C plus
Setup and Dismantle equipment

Radiochemistry Technician A

Tech. B plus
Review and approve data on routine sample analysis. Train C and B technicians. Prepare Reagents and standards.
Review and approve routine Analytical results.

Senior Radiochemistry Technician

Tech. A Plus
Perform Nonroutine analysis where standard methods are not available
Trouble shoot methods and equipment
Review and approve routine analytical results
Review and update laboratory methods.

Radiochemistry Technician Specialist

Senior Technician plus
Method Development
Method Writing
Review and Approve
Analytical Results

Waste Assay Engineer

Waste Classification
Programming Waste Assay

ATTACHMENT B

FUNCTIONAL RESPONSIBILITIES

Vitrification Laboratory

Cognizant Engineer	Technician	Co-op Students
Method Development Method Update Method Writing Review and Approve Results Approval Issue Reports Training Program, Quality Control, Quality Assurance Approve exceptions in Test Methods. Desiginate Authority to Technician.	Perform routine laboratory operation Perform analysis within limits of qualification. Approve laboratory data on lab notebook. Train co-op students. Maintain filing system. Coordinate Quality Control, Quality Assurance in lab.	Perform routine laboratory operation. Perform analysis within limits of qualification. Approve laboratory data on lab notebooks.

Quality Assurance Program

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

> The Laboratory Quality Assurance Program established for WVNS laboratories is documented and described in this PRD. The basis of the program is presented and provisions are described for documenting, modifying, and administering the Quality Assurance program and for performing management assessments.

2.0 REQUIREMENTS

A Quality Assurance program shall be established by selecting applicable Quality Assurance requirements. The program shall be documented and described through an approved Laboratory Quality Assurance Program Manual. Appropriate procedures shall be prepared and approved to establish how the selected requirements shall be implemented in laboratory operations. Orientation in the Quality Assurance program shall be given to laboratory personnel and they shall be trained in the use of the implementing procedures. Management shall periodically assess the adequacy of the program and shall document the assessments.

(Reference: Section 6., ASTM C 1009) - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry.

3.0 PROGRAM IMPLEMENTATION

3.1 Basis for Quality Assurance Program

This Laboratory Quality Assurance Program is based on the ASTM C 1009 Standard, "Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry." The ASTM standard is related to the ANSI/ASME NQA-1 standard, "Quality Assurance Program Requirements for Nuclear Facilities." The Quality Assurance program of the West Valley Demonstration Project (WVDP) is based on the NQA-1 standard, which signifies that this Laboratory Quality Assurance program complies with the WVDP Quality Assurance Program.

> All nine elements of quality assurance contained in the ASTM standard were selected to establish this Laboratory Quality Assurance program. The nine elements are based on 14 of the 18 requirements in the NQA-1 standard. Requirements 3 (Design Control), 4 (Procurement Document Control), 7 (Control of Purchased Items and Services), and 11 (Test Control) are not addressed in ASTM C 1009. Activities related to those four requirements, for WVNS laboratories will be carried out primarily by other West Valley Nuclear Services (WVNS) organizations such as Purchasing and Quality Assurance. The activities will be controlled and verified through the WVDP Quality Assurance Program.

3.2 Quality Assurance Manual

This Quality Assurance program is documented through a Quality Assurance Manual: "Laboratory Quality Assurance Program Manual." The Manual contains Program Requirements/Descriptions (PRD) and Administrative Control Procedures (ACP). PRDs prescribe requirements and describe implementation of requirements. ACPs contain instructions for implementing specific requirements. The Laboratory Quality Assurance Program is approved through the approval of the PRDs and ACPs.

3.3 Quality Assurance Planning

> The Quality Assurance Program described in this PRD is the basic program used to support the WVDP. A Quality Assurance Plan/Index (QAPI) for this basic program is attached to this PRD as Attachment A and B. The QAPI is another way to document the Quality Assurance Program by showing applicable Quality Assurance requirements from ASTM C 1009, related NQA-1 requirements and implementing documents.

> Should a modified Quality Assurance Program be required for another project or special types of samples, the modified program is established and documented by preparing a QAPI using Attachment A as a guide. A modified Quality Assurance Program may have fewer QA requirements, or it may have additional requirements imposed from another QA standard or other requirements documents. If the modified program requires new ACPs or the revision of existing ACPs, they will be prepared according to ACP 4.1 and will be referenced in the QAPI. The new QAPI will become an attachment to this PRD for the lifetime of the QAPI.

3.4 Quality Assurance Administration

> Each WVNS laboratory under this Quality Assurance Program will desiginate someone to provide administrative control for the Laboratory Quality Assurance Program. The position of Quality Assurance and Training Coordinator is established in Analytical and Process Chemistry to provide an administrative function for the Laboratory Quality Assurance Program. The person assigned to the position maintains the program and provides liaison with Quality

Assurance. Specific responsibilities and authorities assigned to the position are described in PRD 1.0 under Quality Assurance and Training Coordinator.

3.5 Management Assessments

> At least annually, the Manager of each WVNS Laboratory will assess the adequacy of the Laboratory Quality Assurance Program. The assessment may involve reviewing past internal and external audit reports, interviewing laboratory and Quality Assurance personnel, and reviewing records and other documents. The assessment will be documented by an assessment report that includes as a minimum a description of how the assessment was made, recommendations for improvements (if any), and a statement about the overall adequacy of the program. The Manager may do the assessment or have it done by someone outside of the organization.

3.6 Indoctrination and Training

Indoctrination and training for the Laboratory Quality Assurance Program is described and implemented through PRD 3.0.

ATTACHMENT A

QAPI: BASIC LABORATORY QUALITY ASSURANCE PROGRAM FOR ANALYTICAL & PROCESS CHEMISTRY

ASTM C 1009 SECTION	APPLICABLE NQA-1 REQUIREMENTS	IMPLEMENTATION		
		PRD	ACP	ACM*
5.0	1	1.0		
6.0	2	2.0		
7.0	2, 9	3.0	3.1	
8.0	5, 6, 9	4.0	4.1, 4.2	
9.0	6, 9, 17	5.0	5.1	X
10.0	17	6.0		
11.0	8, 10, 12, 13, 14, 15	7.0	7.1, 7.2, 7.3, 7.4	X
12.0	12, 14	8.0	8.1, 8.2	X
13.0	16, 18	9.0	9.1	

* The ACMs (technical methods) contain implementing instructions for the three Quality Assurance Requirements represented by PRD's 5.0, 7.0 and 8.0.

ATTACHMENT B

> QAPI: BASIC LABORATORY QUALITY ASSURANCE PROGRAM FOR VITRIFICATION LABORATORY

ASTM C 1009 SECTION	APPLICABLE NQA-1 REQUIREMENTS	IMPLEMENTATION		
		PRD	ACP	VLTMS+
5.0	1	1.0		
6.0	2	2.0	2.1	
7.0	2, 9	3.0	3.1	
8.0	5, 6, 9	4.0	4.1, 4.2	
9.0	6, 9, 17	5.0	5.2	X
10.0	17	6.0		
11.0	8, 10, 12, 13, 14, 15	7.0	7.1, 7.2, 7.3, 7.4	X
12.0	12, 14	8.0	8.1, 8.2	X
13.0	16, 18	9.0	9.1	

+ The VLTMs (Technical Methods) are used by Vitrification to supplement the ACMs to develop an effective Quality Assurance Program.

TRAINING AND QUALIFICATION

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

> The practices used by each WVNS laboratory to assure that laboratory personnel are qualified to do their work are described in this PRD. Indoctrination in the quality assurance program and periodic evaluations of personnel proficiency are included in this program area. Specific requirements and instructions for training and qualifying laboratory technicians are contained in ACP 3.1, "Training and Testing". While ACP 2.1 clarifies requirements for vitrification laboratory personnel.

2.0 REQUIREMENTS

A system for training and qualifying laboratory personnel shall be established and documented. Training requirements and qualification criteria shall be prescribed. Training may be selectively applied based on job function, but the qualification of all laboratory personnel shall be certified by management. Indoctrination in the quality assurance program shall be included. Periodic evaluations of personnel proficiency shall be made and retraining shall be done if necessary. The qualification of all personnel shall be reviewed and updated at least annually. Training and qualification shall be documented through the record system and individual training and/or qualification records shall be maintained for each person.

(Reference: Section 7., ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry.)

3.0 PROGRAM IMPLEMENTATION

3.1 Indoctrination

> The quality assurance program is reviewed with each person assigned to each WVNS laboratory. The review and orientation consists of a briefing on each PRD and applicable ACP by the manager, which is documented by a memorandum signed and dated by the manager. Each

person is required to read each PRD and applicable ACP, which is documented by a memo signed and dated by the reader. The readers are instructed to raise questions with the manager if parts of the material are not understood. Changes to PRDs and ACPs are reviewed with laboratory personnel and the review is documented. The memos documenting indoctrination of an individual are retained in that person's training and qualification file (see Section 3.5).

3.2 Training

Laboratory technicians are trained in the understanding and use of the technical methods (ACMs or VLTMs) that they will use. Upon completion of training for a method, a technician is qualified to use that method independent of direct supervision. Specific training requirements and directions for training are contained in ACP 3.1 and/or ACP 2.1. Chemists do not require this training.

The reading of ACP's stated in Section 3.1 above constitutes training for these procedures.

3.3 Qualification

As stated in Section 3.2 above, technicians are qualified to use a method upon completion of testing and training on the method. Qualification is also based on education and experience in laboratory operations. Chemists are qualified to use methods, develop and validate new methods, revise methods, and troubleshoot technical problems in their areas of assigned responsibility by reason of their education and experience.

Qualification is documented by the qualification form attached to this PRD as Attachment A. Qualification is certified by the manager signing and dating the form. Qualification is reviewed and updated at least annually. Completed qualification forms are retained in the training and qualification files (see Section 3.5).

3.4 Proficiency Evaluation

The proficiency of laboratory personnel to do their work is evaluated at least annually based on annual performance appraisals. Proficiency evaluations are documented by memos signed and dated by the manager. As a minimum, each memo includes the date of the evaluation and a statement summarizing the status of proficiency. Loss of proficiency in an area of work is documented by updating the current qualification form. When proficiency is regained, the qualification form is again updated and includes the documentation of any retraining done. Memos documenting proficiency evaluations are retained in the training and qualification files (see Section 3.5).

3.5 Records

> A training and qualification file is maintained for each person assigned to each respective laboratory. These files are stored in the manager's office. They include all records associated with training and qualification, except for confidential, company personnel records. Original records are kept when possible. If copies are kept, the copies are easily readable. Training and qualification records are retained for a minimum of six (6) years.

ATTACHMENT A

QUALIFICATION

Name: _____ Date Prepared: _____

1. Education Completed

2. Experience

   a. Total years experience in laboratory work:
   b. Type of experience:

3. Training Received During Past Year

4. Course Work

   a. Company Courses:
   b. Off-site Courses:

5. Methods Qualified to Use

6. Remarks

7. Qualification Certified:

Manager Analytical & Process Chemistry	Date	Qualification Terminated manager date

ATTACHMENT B

QUALIFICATION

Name: _____ Date Prepared: _____

1. Education Completed

2. Experience

   a. Total years experience in laboratory work:
   b. Type of experience:

3. Training Received During Past Year

4. Course Work

   a. Company Courses:
   b. Off-site Courses:

5. Methods Qualified to Use

6. Remarks

7. Qualification Certified:

Manager Vitrification Laboratory	Date	Qualification Terminated _____ _____ manager date

PROCEDURES

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

> The preparation and control of procedures and methods used by each WVNS laboratory to do its work is described in this PRD. The types of procedures and methods are identified. The control of changes to those documents and the control of their distribution is included in this program area. Specific requirements and implementing instructions are contained in ACP 4.1; "Preparation, Approval, and Distribution of PRDs and ACPs"; ACP 4.2; "Preparation, Approval, and Distribution of ACMs" and ACP 2.1, "Vitrification Laboratory Test Methods".

2.0 REQUIREMENTS

Procedures and methods shall be prepared, reviewed, and approved for use in performing laboratory operations and analyzing samples. They shall be prepared by following established formats and shall be reviewed for clarity, completeness, and technical accuracy. Before procedures and methods are issued, they shall be approved by designated management. The distribution of procedures and methods shall be controlled to assure that all controlled copies are updated when revisions are made. Copies of procedures and methods shall be located in work areas where they are used. Only the latest revisions shall be used at work places. Changes to procedures and methods shall be reviewed and approved.

(Reference: Section 8., ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry.)

3.0 PROGRAM IMPLEMENTATION

3.1 Types

> There are three types of documents prepared, approved, and controlled through the Laboratory Quality Assurance Program. Program Requirements/Descriptions (PRDs) prescribe quality assurance requirements based on quality assurance standards and describe how those requirements are implemented. Administrative Control Procedures (ACPs) provide specific requirements and detailed instructions for implementing program requirements. Analytical Chemistry Methods (ACMs) or Vitrification Laboratory Test Methods (VLTMS) are the methods used to analyze samples.

3.2 Preparation

> Procedures and methods are prepared according to established formats. They are reviewed and approved by designated management. The review and approval is documented through the Procedure/Document Review Form. The preparation and approval of PRDs and ACPs is done according to ACP 4.1. ACMs are prepared and approved according to ACP 4.2. VLTMs are prepared and approved in accordance with ACP 2.1.

3.3 Change Control

Changes (revisions) to procedures and methods are reviewed and approved before they are issued. Changes are classified as minor and major. Minor changes are editorial or simple technical changes that affect only a minor portion of a procedure or method and that have no significant affect on the result of the work. For example, a change that improves the efficiency of a method, but which does not affect the result would be a minor change (the result would be the same with or without the change). All other changes are major changes. A major change may also be a collection of minor changes. Generally, when five minor changes are accumulated for one procedure or method, a major change is made.

Major changes are reviewed, approved, and distributed as is done with original procedures and methods according to ACPs 4.1 and 4.2.

Minor changes are made by making handwritten entries in workplace copies by authorized persons (see PRD 1.0). Entries are made in indelible ink and are signed and dated by the person making the change. The signature designates the approval of the change. At this point, the authorizing person leaves a copy of the procedure/method with the Quality Assurance Training Coordinator (QATC). The minor change is forwarded to Records Management, where it is redistributed to all procedure and manual holders.

3.4 Distribution

> PRDs and ACPs are collected in the Laboratory Quality Assurance Program Manual. Their distribution is controlled through control of the manual according to ACP 4.1. ACMs are collected in a manual that is controlled according to ACP 4.2. VLTMs are collected in a manual that is controlled in accordance with ACP 2.1. The latest revisions of procedures and methods are located at appropriate work places, either as individual documents or in their respective manual. Obsolete revisions are removed.

LABORATORY RECORDS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

> The record system used in the laboratories of WVNS provide traceability of analytical results back to raw data, provide control of samples as they are processed through the laboratories, and establish who did the work and when it was done is described in this PRD. The forms and documents used to carry out the five functions of a laboratory record system are described (See ASTM C 1009, referenced in 3.1 of PRD 2.0.). Detailed instructions on using the system, including distribution and retention times of the various records generated in the laboratories, are contained according to department in ACP 5.1, or ACP 5.2,"Laboratory Record System".

2.0 REQUIREMENTS

A laboratory record system shall be established that provides for complete documentation of the work done in the laboratories, from receipt of samples to reporting of analytical results. The system may be a manual system using forms and other documents, a computerized system, or a combination of both. Whatever is used, the system shall be described and instructions for its use shall be available for laboratory personnel. The following five functions shall be included in the system: receipt of sample information from sample submitters, provisions for sample identification, transmission of information and data through the laboratories, provisions for recording data and information generated during analysis of samples, and reporting of analytical results.

> Manual entries in the record system shall be either typed or made in black indelible ink, and should be legible and neat. There should be no erasures, scratch outs or white outs. Incorrect entries should be marked out with a single line through the entry. Marked out entries should still be legible. Corrected entries should be dated and signed or initialed. All analytical (measurement) data entries shall be signed and dated by the analyst doing the work. A signature sheet will be retained in the laboratory manager's office files. This sheet will contain all personnel in the department's legal name; typed, printed and their signature and initials. One copy of this signature sheet will be placed on each laboratory wall. At the end of the calendar year and/or when

soneone leaves the department a copy of this sheet will be placed in the permanent records file. Data entries into a computer system shall be identifiable as to who made the entries and the dates of entries. A computer system shall have provisions for making corrections. However, controls should be established to prevent alteration of data by unauthorized personnel.

> The basic information that the record system shall contain relating to each sample is the following: sample submitter, date received in the laboratory, analyses required, laboratory sample identification (laboratory serial number), date analyses completed (date results reported), and date sample returned or discarded. The record system shall document the following basic information generated during analyses: measurement (sample) data, calibration and control standards used, unusual or unexpected occurrencies, calculations made, results reported, and who did the work and when it was done. Distribution and retention time of each type of record in the system shall be established and documented. Analytical results shall be reviewed (evaluated) and approved before being reported (see PRDs 1.0 and 8.0).

(References: Section 9., ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry.

3.0 PROGRAM IMPLEMENTATION

> The implementation of the five functions of a Laboratory Records System defined in ASTM C 1009 is briefly described below. The specific details for implementation and the description of the records system used by specific WVNS laboratories are found in either ACP 5.1 or ACP 5.2 depending on department.

3.1 Analysis Request

Receipt of samples and sample information are obtained through the request for analysis form. The completed form initiates the analysis process for the sample received. Requirements for receipt and handling of samples are found in PRD 7.0 and implementation of the requirements is described in 3.0 of PRD 7.0 and ACP 7.3.

3.2 Log Book

Sample identification and a record of sample information are recorded through the log book. The log book for each sample includes: log number, requester, requester's sample identification, date sample received, charge number, date analysis completed, and sample disposition and date. Requirements for receipt and handling of samples are found in PRD 7.0 and implementation of the requirements is described in 3.0 of PRD 7.0 and ACP 7.3.

3.3 Worksheets (Traveler)

Information and data are transmitted through the laboratory by the worksheets. Analysis is initiated by the analyst through receipt of the worksheets. Analytical data and results are recorded on the worksheet.

3.4 Data Record

All data and information generated through implementation of Analytical Methods is recorded in that Methods Data Record. The data and information included is that generated during analysis of samples and standards, activities related to measurement control and unusual or unexpected occurences reported. Requirements for measurement control are found in PRD 8.0.

3.5 Analytical Report

Analytical results are forwarded to the requester through an approved Analytical Report. The Analytical Report includes the log number, requesters sample identification, and results with error limits assigned when available. Identification of persons qualified to review and approve analytical data are found in PRD 1.0. Controls used to validate measurements are addressed in PRD 8.0.

CONTROL OF RECORDS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

The record management practices are described in this PRD. The records generated are described in other PRDs and in various ACPs. Once records are generated (identified), records management concerns their distribution, storage, retrievability, and retention times.

2.0 REQUIREMENTS

A records management system shall be established to control records generated by laboratory activities. The records generated and controlled shall be identified. The storage location of records and types of storage facilities shall be documented. To the degree possible, records in storage shall be protected from damage and loss and they shall be easily retrievable from storage locations. A retention time shall be established and documented for each type of record.

(Reference: Section 1.0, ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry.

3.0 PROGRAM IMPLEMENTATION

3.1 Identification

> Records generated from laboratory activities are identified through the PRDs and ACPs. In addition, a master records index for each WVNS laboratory identifies all records generated and retained by said laboratory. Specific records generated from the receipt and analysis of samples are identified in PRD 5.0, ACP 5.1, and ACP 5.2. Training and qualification records are identified in PRD 3.0, ACP 2.1 and ACP 3.1. Records relating to calibration, control, and methods validation are identified in PRD 8.0 and ACPs 8.1 and 8.2. Internal reviews and corrective action records are identified in PRD 9.0 and ACP 9.1.

3.2 Distribution

Whenever possible, original records are retained by Master Records Control (MCR) and copies are distributed when a record must be distributed to other organizations. In some cases, no distribution of a record is made. Distribution requirements for a record are documented in the PRD and/or ACP that provides instructions for generating and handling the record (see 3.1 above).

3.3 Storage

Records are stored at the manager's office, chemists office, or in the laboratories. They are stored in file cabinets or in laboratory drawers that are designated "for record storage only". In the laboratories, the storage areas are located away from direct contact with water, chemicals, and fumes to minimize damage. The storage location for each type of record is designated in the master record index, which is retained in the manager's record files.

3.4 Retrieval

> Completed records are filed in labeled file folders when possible, which are kept in the labeled file drawers (laboratory drawers or Document Controls locked fireproof cabinets). The labels designate the specific records found inside. Records are filled by consecutive serial numbers, dates, or other indicators that allow easy retrievability from a file folder or drawer. Records are not placed into storage in an indiscriminate manner. The master record index is the primary locater of records. The index indicates the storage location of each type of record.

3.5 Retention Time

The retention time for each type of record is documented in the PRDs and/or ACPs that provide the instructions for generating and handling each type.

3.6 Final Disposition

The permanent retention of laboratory records is the responsibility of Master Records Center (MRC) as prescribed in the WVNS Policy Manual. The laboratory will make periodic transfers of records to the MRC per the requirements of the Policy Manual.

CONTROL OF EQUIPMENT AND MATERIALS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

The practices used to control equipment and materials are described in this PRD. Materials include samples received for analysis. Specific requirements and instructions for implementing program requirements are contained in several ACPs. ACPs relating to laboratory safety and handling radioactive materials are included under this PRD.

2.0 REQUIREMENTS

Equipment and materials shall be controlled to the extent necessary to assure that their integrity is maintained while used and stored. Equipment requiring calibration shall be calibrated individually by an analyst or maintained on an established calibration program. There shall be a system for indicating calibration status for each instrument or item of equipment requiring calibration. Out-of-calibration equipment shall not be used to obtain analytical data. The standards used for calibration shall be specified. The basis for calibrating and verifying calibrations of volumetric glassware shall be documented.

The required quality of Reagents and water used shall be specified. Reagents and standards shall be labeled, as appropriate, with name, concentration, matrix, date prepared, and special limitations on storage, use, and age. Reagents and standards found to be unlabeled shall not be used. When required for a reagent or standard, an expiration date shall be included on the label, after which the reagent or standard shall not be used. Reagents and standards shall be stored to protect against deterioration. When required, special storage conditions shall be specified.

Samples shall be inspected when received to assure that they are properly labeled and identified and that they meet criteria established for receipt. If a deficiency is found with a sample, that sample shall not be analyzed until the problem is resolved. Samples shall be uniquely identified and labeled in a manner to prevent loss of identification. Samples shall be handled and stored to preserve their integrity and any

sample whose integrity is suspect shall not be used and it shall be controlled to prevent inadvertent use. Retention times shall be established for samples and their disposition shall also be established. Disposition actions shall be recorded and dated in the record system.

(Reference: Section 11., ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboaratories within the Nuclear Industry.)

3.0 PROGRAM IMPLEMENTATION

3.1 Equipment

> Equipment and Reagents are controlled according to ACP 7.1, "Control of Equipment and Reagents". Besides being addressed in ACP 7.1, calibration requirements are also addressed in PRD 8.0 and in technical methods (ACMs AND VLTMs). Instructions for calibrating volumetric glassware and pipettes are given in ACP 7.1 when special calibration is required. Normally, the manufacturer's calibration is accepted when procuring volumetric glassware and pipettes. Verification of calibration is established by vender certificates of calibration.

3.2 Reagents and Standards

Requirements for the quality of Reagents and water are specified in ACP 7.1. Instructions for labeling Reagents and standards are contained in ACP 7.1. Also included are instructions for handling, using, and storing Reagents and standards.

3.3 Samples

> Samples are received and controlled according to ACP 7.3, "Control of Samples". As stated in PRD 1.0, sampling is the responsibility of sample submitters. The laboratories do not accept samples and will not analyze them unless they meet acceptance criteria as specified in ACP 7.3. When received, each sample is uniquely identified by a laboratory serial number as specified in ACP 7.3. Samples are retained by a WVNS laboratory for a maximum of two months after all analyses have been completed and the results reported, unless otherwise specified by the requisitioner.

3.4 Safety

Activities performed are controlled and consistent with WVNS site policies and procedures governing safety. Requirements for safety are specified in ACP 7.2, "Laboratory Safety", and ACP 7.4 "Handling Radioactive Materials". Procedures governing safety are subject to approval by Radiation and Safety.

CONTROL OF MEASUREMENTS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

The practices used to control measurements to assure the validity of analytical data are described in this PRD. This program area includes calibration, method control, standards, and statistical practices. Also included in this PRD are provisions for validating analytical chemistry methods when first established for use in the laboratories and for approving analytical results before being reported.

2.0 REQUIREMENTS

Practices shall be established to control the measurement process used to generate analytical data (measurement data) and report analytical results. Those practices shall address as a minimum calibration, method control, standards, and statistical practices. Provisions for recording measurement control data and other information obtained from measurement control activities shall be specified and included in the laboratory record system.

Instructions for performing calibrations and for methods control shall be included in each analytical chemistry method as applicable or shall be prepared as separate procedures. Instructions shall specify the standards to be used, the frequency of their use, any special instructions necessary for obtaining reliable calibration and control data, and required treatment of data. Criteria shall be prescribed for indicating when an instrument or analytical set up is in calibration and when a method is in or out of control. Instructions shall be provided for actions to take when out-of-calibration or out-of-control situations occur. Calibration and control standards required for analyses shall be specified in analytical chemistry methods. Instructions for their preparation shall be included as appropriate. When possible, standards shall be traceable to nationally recognized standards. Their source and history (if appropriate) shall be documented.

Statistical practices shall be established for the measurement control system. Those practices shall address requirements for reporting significant numbers, treating outlying data, and prescribing tolerances of measurements made during analyses. Instructions shall be provided for preparing and using control charts when they are to be used.

(Reference: Section 12., ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Chemistry Laboratories Within the Nuclear Industry.

3.0 PROGRAM IMPLEMENTATION

3.1 Calibration

> Instructions for calibration are included in the Analytical Chemistry Methods (ACMs) and Vitrification Laboratory Test Methods (VLTMs) as presented in ACP 4.2, "Preparation, Approval, and Distribution of ACMs". The instructions include the frequency of calibration, criteria that establish when calibration is achieved, the required treatment of calibration data, and actions to take if calibration is lost. The calibration standards to be used are specified in the ACMs and VLTMs (see 3.3 below). The provisions in this PRD and in ACP 4.2 apply to calibrations done by analysts during the measurement process. Other aspects of calibration, such as the indication of calibration status, are described in PRD 7.0 and implemented through ACP 7.1, "Control of Equipment and Reagents".

3.2 Method Control

As used here, method control refers to the use of control charts or some other technique to provide assurance that the responses being obtained from analytical methods over time are falling within prescribed limits. Those prescribed limits are a form of acceptance criteria used to evaluate the acceptance of analytical data.

> Instructions for method control are included in the ACMs and VLTMs as prescribed in ACP 4.2. The instructions include the frequency of analyzing the control standards, criteria that establish when methods are in control, required treatment of control data, and actions to take when out-of-control situations occur. The control standards to be used are specified in the ACMs and VLTMs (see 3.3 below).

3.3 Standards

> Calibration and control standards used in the measurement control system are specified in the ACMs and VLTMs as prescribed by ACP 4.2 and ACP 2.1 respectively. Specific preparation instructions are included in the ACMs and VLTMs when required. General instructions for preparing standard solutions are contained in ACP 8.1, "Preparation of Standard Solutions". The sources of standards and their history (when available) are documented in the ACMs and

VLTMs. When possible, standards are traceable to nationally recognized standards, such as those provided by the National Bureau of Standards. Additional instructions for handling, labeling, using, and storing standards are contained in ACP 7.1 (see PRD 7.0).

3.4 Statistical Practices

Instructions for the statistical practices used are contained in ACP 8.2, "Statistical Practices". The preparation and use of control charts is included, as well as instructions for reporting significant numbers, treating outlying data, and prescribing tolerances of measurements made during analyses. Retention times and distribution (if appropriate) of control charts are addressed in ACP 8.2. Documentation of corrective actions taken because of out-of-control situations is addressed in PRD 9.0.

3.5 Approval of Results

> As prescribed in the Requirements of PRD 5.0, analytical results are reviewed and approved before being reported. Authority for approvals is specified in PRD 1.0. Results are reviewed for acceptability based on such criteria as checking calculations, assuring that a method was in control when a result was obtained, a result is within an expected range based on the sample from which it was obtained, and statistical evaluations when sufficient data were available for such evaluations. Unacceptable results are corrected as appropriate (see PRD 9.0). Approval is documented on the analytical reports (see PRD 5.0 and ACP 5.1 or ACP 5.2).

3.6 Method Validation

Validation of a method for a specific application in the laboratories is done during the developement and setting up of the method. Instructions for validation and documentation of validation are contained in ACP 8.3, "Method Validation". New methods introduced after the effective date of ACP 8.3 are validated. Methods in active use prior to that effective date are not formally validated, but their applicability is documented as prescribed by ACP 8.3.

3.7 Records

Data and information obtained from measurement control activities are recorded in the laboratory record system, primarily in the Data Record part of the system. Requirements and instructions are found in PRD 5.0 and ACP 5.1, "Laboratory Record System". Other records are also generated and they are prescribed in various PRDs and ACPs referenced in the PRD. Retention times and distribution are addressed for the records in their respective PRDs and ACPs.

DEFICIENCIES AND CORRECTIVE ACTIONS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

Practices related to the identification of deficiencies and corrective actions are described in this PRD. One of the principal approaches used in this program area is an internal review system as prescribed by ACP 9.1, "Internal Review".

2.0 REQUIREMENTS

Deficiencies identified through various means shall be addressed through an established corrective action system. Deficiencies shall be evaluated to determine their cause and to identify appropriate actions required to correct them, including actions that will minimize recurrence. Responsibility for taking corrective actions shall be assigned and documented and schedules for completion of these actions shall be established. Responsibility for following up on specific corrective actions shall be assigned to assure the completion of corrective actions. Completion shall be documented and reported to appropriate management.

(Reference: Section 13., ASTM C 1009 - Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories within the Nuclear Industry.

3.0 PROGRAM IMPLEMENTATION

3.1 Identification

> Deficiencies are identified through external audits and internal reviews. The measurement control system is another mechanism for identifying deficiencies through method control and evaluation (review) of analytical results (see PRD 8.0). Normal surveillance carried out by the manager and chemists of each WVNS laboratory may result in the identification of deficiencies.

3.2 Corrective Action

Deficiencies identified though external audits and internal reviews are evaluated, corrected, documented, and reported based on the

Significant deficiencies found through measurement control and surveillance are evaluated, corrected, and documented through the Request for Corrective Action (RCA); see Quality Assurance Procedure Manual (QAP) 16-1. Significant deficiencies are defined as those that would lead to incorrect measurement data. Examples are: method out-of-control, a trend developing that will probably lead to an out-of-control condition, and an unexpected loss of calibration.

A single incorrect analytical result is not normally reported through the RCA (see PRD 8.0). A correction is made by rerunning the sample, recalculating the result, or taking whatever action is appropriate. The correction is documented via the laboratory record system as with regularly generated data and information (PRD 5.0 and ACP 5.1). The RCA is used for significant situations involving incorrect results, such as a series of incorrect results from a single methods over a relatively short time period.

The RCA is used to document and report corrective actions. The form documents the persons responsible for correcting actions, evaluations, actions taken, and completion of corrective actions. It is used also for internal reviews (ACP 9.1). Copies are filed with the Data Record for the analytical chemistry methods involved or with the associated internal review reports. Originals are forwarded to the Quality Assurance Manager for final review and retention.

> The RCA is processed per the instructions in WVNS's Quality Assurance Procedure (QAP) 16-1. The Manager of each WVNS laboratory, or his designee, acts as Quality Assurance representative for the purposes of identifying, assigning responsibilities, and verifications as associated with the procedure. The Quality Assurance Department maintains administrative control of the RCA, with the Quality Assurance Manager determining final review and closure.

3.3 Follow-Up

> Follow-up on corrective actions resulting from external audits is accomplished through the procedures governing the audits. The manager of each WVNS laboratory is responsible for follow-up on nonaudit corrective actions involving RCAs. Follow-up is documented by the RCA through the close out of RCAs.

UNCONTROLLED

TABLE OF CONTENTS

II. Administrative Control Procedures (ACPs)

		Effective Rev. No.	Date
2.1	Vitrification Laboratory Test Methods	0	06/09/89
3.1	Training and Testing	0	06/15/87
4.1	Preparation, Approval and Distribution of PRDs and ACPs	3	07/24/89
4.2	Preparing, Approving and Distribution of ACMs	3	07/24/89
5.1	Laboratory Record System	2	07/24/89
5.2	Vitrification Laboratory Record System	0	06/09/89
7.1	Control of Equipment and Reagents	1	07/24/89
7.2	Laboratory Safety	2	08/29/89
7.3	Control of Samples	1	07/24/89
7.4	Handling Radioactive Materials	0	06/15/87
8.1	Preparation of Standard Solutions	1	07/24/89
8.2	Statistical Practices	1	07/24/89
8.3	Method Validation	1	07/24/89
9.1	Internal Reviews	1	07/24/89

WEST VALLEY NUCLEAR SERVICES CO., INC.

ADMINISTRATIVE CONTROL PROCEDURES
VITRIFICATION LABORATORY, PROCESS DEVELOPMENT

ACP 2.1, Rev. 0
Effective Date: 06/09/89

VITRIFICATION LABORATORY TEST METHODS

Approved by:

S. M. Barnes, Manager
Vitrification Process Development

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

This procedure outlines the required modifications and/or additions to the ACPs which are necessary for application of the Laboratory Quality Assurance Program to the Vitrification Laboratory. This procedure will allow for implementation and control of the Vitrification Laboratory Test Methods (VLTMs).

2.0 APPLICATION

This procedure is applicable to Vitrification Laboratory activities that require the use of VLTMs. ACPs 3.1 to 9.1 apply to the use of the VLTMs with the modifications and additions as described below. This procedure should be referenced whenever an ACP is used by the Vitrification Laboratory.

3.0 DEFINITIONS

3.1 VLTMs: These are the Vitrification Laboratory Test Methods used in the Vitrification Laboratory for the operation and calibration of instruments and property measurements.

4.0 RESPONSIBILITIES

4.1 The personnel of the Vitrification Laboratory are responsible for following the provisions of this procedure as they perform laboratory analyses.

4.2 The Vitrification Laboratory Cognizant Engineer is responsible for assuring that all Vitrification Laboratory personnel are following this procedure and associated ACPs.

4.3 The manager of Vitrification Process Development is responsible for approving exceptions to the provisions of this ACP.

5.0 PROCEDURE

Listed below, as attachments, are the modifications to the ACPs which are necessary for the application of the Laboratory Quality Assurance Program to the Vitrification Laboratory.

Attachment	Modifications in the ACP	
A	ACP 3.1,	"Training and Testing"
B	ACP 4.1,	"Preparation, Approval, and Distribution of PRDs and ACPs"
C	ACP 4.2,	"Preparing, Approving, and Distribution of Laboratory Methods"
D	ACP 5.1,	"Laboratory Record System"
E	ACP 7.1,	"Control of Equipment and Reagents"
F	ACP 7.2,	"Laboratory Safety"
G	ACP 7.3,	"Control of Samples"
H	ACP 7.4,	"Handling of Radioactive Materials"
I	ACP 8.1,	"Preparation of Standard Solutions"
J	ACP 8.2,	"Statistical Practices"
K	ACP 8.3,	"Method Validation"
L	ACP 9.1,	"Internal Reviews"

6.0 REFERENCES

6.1 Laboratory Quality Assurance Program Manual

6.2 Analytical Chemistry Methods

6.3 Vitrification Laboratory Test Methods

ATTACHMENT A

ACP 3.1, "Training and Testing"

1.0 PURPOSE

To provide specific instructions for qualification, training, and testing of vitrification laboratory technicians and co-op technicians.

2.0 APPLICATION

Implement section 2.0 without change.

3.0 DEFINITIONS

Implement section 3.0 without change.

4.0 RESPONSIBILITIES

4.1 The Manager, Vitrification Process Development, is responsible for attesting to a Vitrification Laboratory technician's/Co-op's qualification by completing the certification forms (see attachment A of PRD 3.0).

4.2 The Cognizant Engineer or his designee, Vitrification Laboratory, is responsible for implementing the training and testing procedure.

5.0 PROCEDURE

5.1 Minimum Education and Experience Requirements

5.1.1 Vitrification Laboratory Technician: Job entry; no experience required. AA in chemistry or equivalent in ceramics or science is required.

5.1.2 Vitrification Laboratory Co-op: Job entry; no experience required. Sophomore or junior in BS program in Ceramics or equivalent required.

5.2 Training and Testing

5.2.1 Initial training on Vitrification Laboratory Test Methods (VLTMs) requires the Vitrification Laboratory technician/co-op read the method and discuss its application, potential problem areas, safety, and instrument operation with the Cognizant Engineer or his designate.

5.2.2 Training on the applicable VLTMs will include running of two standard samples on the described equipment and obtaining results within the prescribed limits on the quality control charts or within acceptable standard deviation for that method.

5.3 Implement this section without change.

5.4 Implement this section without change.

6.0 REFERENCES

Implement this section without change.

ATTACHMENT B

ACP 4.1, "Preparation, Approval, and Distribution of PRDs and ACPs"

1.0 PURPOSE

Implement this section without change.

2.0 APPLICATION

This procedure applies to the issuing of and revision of Program Requirements/Descriptions (PRDs) and Administrative Control Procedures (ACPs) used by Analytical and Process Chemistry and the Vitrification Laboratory.

3.0 DEFINITIONS

Implement this section without change.

4.0 RESPONSIBILITIES

4.1 The Manager, Vitrification Process Development, and Manager, Quality Assurance are responsible for reviewing and approving procedures used by Vitrification Laboratory Personnel.

4.2 Implement this section without change. The Manager, Vitrification Process Development is responsible for approving ACPs, applicable to Vitrification Laboratory.

5.0 PROCEDURE

5.1 Preparation

Implement this sub-section without change.

5.2 Review and Approval

Implement this sub-section without change.

5.3 Distribution

5.3.1 Implement without change.

5.3.2 Implement without change.

5.3.3 A. One copy each to the Manager, Analytical and Process Chemistry and the Manager, Vitrification Process Development.

B. One copy to each scientist and technician in the Analytical and Process Chemistry group and one copy to each scientist, engineer, co-op students, and technicians involved in Vitrification Laboratory activities.

C. Implement without change.

D. Implement without change.

E. Implement without change.

5.3.4 Implement without change.

5.3.5 Implement without change.

5.4 Revisions

Implement this section without change.

6.0 REFERENCES

Implement this section without change.

ATTACHMENT C

ACP 4.2, "Preparing, Approving, and Distribution of Analytical Chemistry Methods

1.0 PURPOSE

This procedure establishes the method to be used in preparing, approving and distributing methods covering measurements performed by the Vitrification Laboratory.

2.0 APPLICATION

This procedure applies to the issuing and revision of Vitrification Laboratory Test Methods used by Vitrification Laboratory developed after the implementation of this ACP.

3.0 DEFINITIONS

3.1 VLTMS - These are the Vitrification Laboratory Test Methods used in the Vitrification Laboratory for the operation, calibration of instruments, and physical property measurements.

4.0 RESPONSIBILITIES

4.1 The manager, and the cognizant engineer (other than the author) of the Vitrification Laboratory, and the technical specialist are responsible for reviewing the VLTMs.

4.2 Change "ACM" to "VLTM".

4.3 The manager, Process Development is responsible for approving and issuing the VLTMs.

4.4 The cognizant engineer who does the development work leading to a new VLTM is responsible for validating the method according to ACP 8.3.

5.0 PROCEDURE

5.1 Preparation

5.1.1 When a need for a new VLTM is identified, the Manager, Vitrification Process Development, assigns an author for preparation.

5.1.2 The author follows the format and instructions given in attachments A and B with the following exceptions:

A. Vitrification Laboratory Test Methods shall be written in the following sequence.

1. Purpose of Procedure
2. Safety Procedures
3. Equipment
4. Procedure (Calibration/Operation)
5. Records and Calculations
6. Calibration Verification
7. Calibration Frequency
8. Attachments

5.1.3 An alpha numeric identification code is assigned to each VLTM as follows: VLTM-XXX, where XXX is a three letter numeric code. Examples: VLTM-002, VLTM-025.

5.1.4 The revision number of a new VLTM (first issue) is assigned 0 (original). Revisions are numbered sequentially starting from 1.

5.1.5 Implement this section without change.

5.1.6 Implement this section without change.

5.1.7 The manager, Vitrification Process Development or his designee, maintains a log of VLTM numbers, current revisions, and effective dates. The table of contents of the manual containing the VLTMs may serve as a log (see 5.3.5).

5.2 Review and Approval

5.2.1 When a VLTM is ready for review and approval, the author prepares a WVNS Document/Design Review Transmittal Form (WV-1840) and signs the form.

5.2.2 The form, with a copy of the VLTM, is circulated for review and approval as indicated below:

A. Review only: technical specialist, manager, Vitrification Process Technology and Testing and manager, Quality Assurance; Manager, Radiation and Safety.

B. Approval: Manager, Vitrification Process Development.

5.2.3 Implement without any change.

5.2.4 When all the comments have been resolved and the review completed, the manager, Vitrification Process Development, approves the VLTM by signing the form and the first page of the VLTM.

5.2.6 Release of VLTMs will be done by an Engineering Release, Form WV-1802.

5.3 Distribution

5.3.1 VLTMs are assembled together in a methods manual.

5.3.2 Distribution of VLTMs is controlled through the controlled distribution of the manual.

5.3.3 The manual is controlled through the WVNS Document Control System. Its distribution is as follows:

A. One copy to Manger, Vitrification Process Development.

B. One copy to each Vitrification Laboratory engineer and technician.

C. One copy to Vitrification Laboratory (laboratory desk copy).

D. One copy to Quality Assurance.

E. One copy to Master Record Center per WV-103.

F. One copy to the Manager, Analytical and Process Chemistry (A&PC).

5.3.4 The original VLTMs and their respective, completed Procedures/Document Review Forms are filed in the Document Control Files.

5.3.5 The Table of Contents of the manual documents the current revisions of the ACMs and their effective dates.

5.4 Revisions

Implement this section without change.

6.0 REFERENCES

Implement without any change.

Attachment A can be implemented with the changes listed in section 5.1.2.

Attachment B can be implemented without change except for the title and approval signature. The Title should read as "Vitrification Laboratory Test Method" and should be approved by Manager, Vitrification Process Development.

ATTACHMENT D

ACP 5.1, "Laboratory Record System"

This procedure will be replaced by ACP 5.2, "Vitrification Laboratory Record System."

ATTACHMENT E

ACP 7.1, "Control of Equipment and Reagents"

1.0 PURPOSE

Implement without change.

2.0 APPLICATION

This procedure is applicable for the general control of equipment and for the calibration of equipment that is generally used throughout the laboratory. This also applies to the control of reagents used in the Vitrification Laboratory.

3.0 DEFINITIONS

Implement without change.

4.0 RESPONSIBILITIES

4.1 The personnel of Vitrification Laboratory are responsible for following the provisions of this procedure as they use equipment and reagents in their work.

4.2 The manager, Vitrification Process Development, is responsible for approving exceptions to the provisions of this ACP. Any changes or exceptions will be transmitted in writing to the Manager, Analytical and Process Chemistry.

5.0 PROCEDURE

Implement this section without change.

6.0 REFERENCES

Implement this section without change.

ATTACHMENT F

ACP 7.2, "Laboratory Safety"

1.0 PURPOSE

Implement without any change

2.0 APPLICATION

2.1 Implement without change.

2.2 Implement without change.

2.3 This procedure is applicable for safety hazards encountered in the Vitrification Laboratory while working with hot glasses and sharp glass pieces. These safety guidelines are covered in the VLTM-025.

3.0 DEFINITIONS

3.1 VLTM - Vitrification Laboratory Test Method.

4.0 RESPONSIBILITIES

4.1 The personnel of Vitrification Laboratory are responsible for following the provisions of this procedure to assure a safe work environment.

4.2 The Manager, Vitrification Process Development, is responsible for promoting safe operations and assuring that the staff observes a good safety practices. The manager is also responsible for approving deviations from provisions in this ACP if required.

5.0 PROCEDURE

Implement this section without change.

6.0 REFERENCES

6.1 Implement without change.

6.2 Implement without change.

6.3 Implement without change.

6.4 Implement without change.

6.5 VLTM 025, Vitrification Laboratory Safety Practices.

ATTACHMENT G

ACP 7.3, "Control of Samples"

1.0 PURPOSE

To provide instructions for the control of samples as they are received by Vitrification Laboratory and as they are processed in the laboratories.

2.0 APPLICATION

This procedure applies from the time the samples are received by the Vitrification Laboratory until their final disposition. This includes samples received by automatic transfer and those received by delivery to the laboratory.

3.0 DEFINITIONS

3.1 Submitter - The person or organization transferring samples to laboratory.

4.0 RESPONSIBILITIES

4.1 The vitrification laboratory staff receiving samples are responsible for assuring that the acceptance criteria for samples as prescribed in this ACP are met before accepting the samples.

4.2 The Vitrification Laboratory staff using and handling samples are responsible for assuring that samples are handled according to the provisions of this ACP.

4.3 The Manager, Vitrification Process Development is responsible for approving exceptions to the provision of this ACP.

5.0 PROCEDURE

Implement this section without change. Substitute "Vitrification Laboratory" for "A&PC".

6.0 REFERENCES

Implement this section without change.

ATTACHMENT H

ACP 7.4, "Handling Radioactive Materials"

This procedure does not apply to the Vitrification Laboratory.

ATTACHMENT I

ACP 8.1, "Preparation of Standard Solutions"

This procedure does not apply to the Vitrification Laboratory.

ATTACHMENT J

ACP 8.2 "Statistical Practices"

1.0 PURPOSE

To provide instructions for the use of statistical practices by the staff of Vitrification Laboratory.

2.0 APPLICATION

Implement this section without change.

3.0 DEFINITIONS

3.1 VLTM - Vitrification Laboratory Test Method.

4.0 RESPONSIBILITIES

4.1 The staff of Vitrification Laboratory is responsible for following the provisions of this procedure in their work as appropriate.

4.2 The Manager, Vitrification Process Development, is responsible for approving specified actions in this ACP.

5.0 PROCEDURE

Implement this section without change. Substitute "Vitrification Laboratory" for "A&PC".

6.0 REFERENCES

Implement this section without change.

ATTACHMENT K

ACP 8.3, "Method Validation"

1.0 PURPOSE

Implement this section without change.

2.0 APPLICATION

Implement this section without any changes.

3.0 DEFINITIONS

Not applicable for this procedure.

4.0 RESPONSIBILITIES

4.1 Validation is the responsibility of the Vitrification Laboratory Cognizant Engineer developing and setting up a method. The documentation of applicability of methods in active use prior to the effective date of this procedure is the responsibility of the cognizant engineer responsible for maintaining the methods.

4.2 The manager concurs with each validation.

5.0 PROCEDURE

Implement this section without change. Substitute "Technician/Co-op" for "Chemist".

6.0 REFERENCES

Implement this section without change.

ATTACHMENT L

ACP 9.1, "Internal Reviews"

1.0 PURPOSE

To provide instructions for performing internal reviews of Vitrification Laboratory, giving assurance that program requirements are being met.

2.0 APPLICATION

Implement this section without change.

3.0 DEFINITIONS

Implement this section without change.

4.0 RESPONSIBILITIES

Implement this section without change.

5.0 PROCEDURE

Implement this section without change.

6.0 REFERENCES

Implement this section without change.

UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC.

ADMINISTRATIVE CONTROL PROCEDURE
ANALYTICAL AND PROCESS CHEMISTRY

ACP 3.1 Rev. 0
Effective Date: 06/15/87

TRAINING AND TESTING

Approved By:

J. R. Stimmel, Manager
Analytical and Process Chemistry

Concur:

, Manager
Quality Engineering

1.0 PURPOSE

To provide specific instructions for qualification, training, and testing of laboratory technicians.

2.0 APPLICATION

This procedure is applicable to those activities required for certification of laboratory technicians. This procedure is supplemental to PRD 3.0 (Training and Qualification).

3.0 DEFINITIONS

3.1 Certification: The action of determining, verifying, or attesting to, in writing, the qualifications of personnel.

3.2 Qualifications: The characteristics or abilities gained through education, training, or experience that enable an individual to perform a required function.

4.0 RESPONSIBILITIES

4.1 The Manager, Analytical and Process Chemistry, is responsible for attesting to a technician's qualifications by completing the certification forms. See Reference 6.1.

4.2 Quality Assurance and Training Coordinator (QATC), Analytical and Process Chemistry, is responsible for implementing this procedure.

5.0 PROCEDURE

5.1 Minimum Education and Experience Requirements:

5.1.1 Radiochemistry Technician C. Job entry; no experience required. AA in chemistry or equivalent in science is required.

5.1.2 Radiochemistry Technician B. Meets the requirements of Radiochemistry Technician C plus 6 months experience.

5.1.3 Radiochemistry Technician A. Meets the requirements of Radiochemistry Technician C plus one year experience.

5.1.4 Senior Radiochemistry Technician. Meets the requirements of Radiochemistry Technician C plus two years experience as a Radiochemistry Technician A.

5.1.5 Radiochemistry Technician Specialist. Meets the requirements of Radiochemistry Technician C plus one year experience as a Senior Radiochemistry Technician.

5.1.6 In addition to the above listed requirements, a written and/or an oral examination will be required. A passing grade is required for promotion to a higher technician grade and/or step increases.

5.2 Training and Testing

5.2.1 Technicians will be trained on proper procedures and techniques for working on samples that contain alpha or beta/gamma radiation. No formal testing samples will be received until this training is given and documented. A written test will be given on radioactive materials handling and waste disposal.

5.2.2 Initial training on Analytical Chemistry Methods (ACMs) involves reading the method and discussing its application, chemistry, potential problem areas, safety, and instrument operation with the Supervisor or his designate.

5.2.3 Training on applicable ACMs will include the analysis of a total six (6) bench standards at three or more levels of concentration, when possible. Successful completion of the training will be indicated when the results of the six (6) bench standards satisfy the statistical tests described in Paragraph 5.2.5.

5.2.4 Proficiency testing on applicable ACMs will include the analysis of three unknown control samples. The unknown control samples will span the concentration range of the method but will not be outside of the concentration range carried by the method's quality control program. The unknown control samples will be processed at a rate of one/day for a minimum of three days. The performance testing will be done only after the successful completion of Paragraphs 5.2.2 and 5.2.3. Evaluation of test results will be per Paragraph 5.2.5.

5.2.5 The QATC Chemist will evaluate the unknown control results using bias and precision data generated by all qualified laboratory personnel for the same method through the quality control program. The statistical evaluation of the unknown control results shall be that the results of the three unknowns are within the two (2) sigma limits on the Quality Control Chart for that method. If the technician fails, retesting is required. If the technician passes, then the technician is considered to be qualified to process samples using the ACM.

5.3 A technician will be allowed to analyze samples using only methods on which he has been qualified. An exception to this will be conditions under which supervision deems it necessary to have a technician use a method on which he has not trained. In such cases the technician will perform the analysis under the direct supervision of his supervisor. The supervisor will take steps to assure that the technician performs the method correctly. This will include, but is not limited to, the analysis of bench standards and controls.

5.4 Documentation shall be per the requirements of PRD 3.0.

6.0 REFERENCES

6.1 PRD 3.0, Training and Qualification.

PREPARATION, APPROVAL, AND DISTRIBUTION OF PRDs AND ACPs

UNCONTROLLED

Approved By:

C. W. McVay, Manager,
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> To provide instructions for the preparation, review, approval, revision, and distribution of procedures used to implement program requirements from the Laboratory Quality Assurance Program. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

This procedure applies to the issuing and revision of Program Requirements/Descriptions (PRD) and Administrative Control Procedures (ACP) used by Analytical and Process Chemistry.

3.0 DEFINITIONS

3.1 Program Requirements/Description (PRD): A procedure that prescribes quality assurance requirements based on a specified quality assurance standard which describes how those program requirements are implemented.

3.2 Administrative Control Procedure (ACP): A procedure that provides specific requirements and detailed instructions for implementing program requirements. ACPs supplement PRDs.

4.0 RESPONSIBILITIES

4.1 The Manager, Analytical and Process Chemistry; and Quality Assurance Manager, are responsible for reviewing and approving procedures used by Analytical and Process Chemistry personnel.

4.2 The Manager, Analytical and Process Chemistry, is responsible for issuing PRD's and ACP's.

5.0 PROCEDURE

5.1 Preparation

5.1.1 When the need for a PRD or ACP is identified, the manager assigns an author for preparation.

5.1.2 The author follows the appropriate format and instructions given in Attachments A and B.

5.1.3 An alpha numeric identification code is assigned to each PRD and ACP as specified below.

A. PRD 1.0, PRD 2.0, etc. The numbers represent the basic quality assurance requirements contained in the specified quality assurance standard. The requirements are numbered in order presented in the standard, beginning with 1.0.

B. ACP 3.1, ACP 4.1, ACP 4.2, etc. The first number represents the quality assurance requirement (PRD) supported by the ACP. The number after the decimal point is the sequential number in order of preparation for the same quality assurance requirement.

5.1.4 The revision number of a new procedure (first issue) is assigned 0 (original). Revisions are numbered sequentially starting with 1.

5.1.5 The effective date is the date the original procedure or subsequent revision is authorized for use, which is the date the manager approves it by signing the first page.

5.1.6 The pages are numbered sequentially as follows: Page 1 of 4, Page 2 of 4, etc. The second number is the total number of pages. Pages of attachments are included in the sequence

5.1.7 Attachments are used as necessary to provide supplemental information or to provide a form for recording information associated with the procedure.

5.1.8 The manager maintains a list or log of PRDs and ACPs that documents the current revisions and effective dates. The Table of Contents in the Manual may serve as the log (see 5.3.5).

5.2 Review and Approval

5.2.1 When a procedure is ready for review and approval, the author prepares a WVNS Document/Design Review Transmittal Form (WV-1840) and signs the form.

5.2.2 The form, with a copy of the procedure, is circulated for review and approval as follows:

A. Quality Assurance
B. Manager, Analytical and Process Chemistry
C. Procedures dealing with handling radioactive materials in the laboratory or laboratory safety will also be approved by Radiation and Safety.

5.2.3 The author resolves comments as required. This is an iterative process.

5.2.4 When all of the approvals have been obtained on the Procedures/Document Review Form, the manager approves the procedure for use by signing the first page. The manager of Quality Engineering signs the first page of the procedure denoting approval.

5.2.5 Release of ACPs and PRDs will be done by an Engineering Release, Form WV-1802.

5.3 Distribution

5.3.1 PRDs and ACPs are assembled together in the Laboratory Quality Assurance Program Manual.

5.3.2 Distribution of PRDs and ACPs is controlled through the controlled distribution of the Manual.

5.3.3 The Manual is controlled through the WVNS Engineering Document Control System. Its distribution is as follows:

A. One copy to the Manager, Analytical and Process Chemistry.
B. One copy to each Scientist and Technician in the Analytical and Process Chemistry group.
C. One copy to Quality Assurance.
D. One copy to Master Records Center per WV-103.
E. One copy to Engineering Document Control.
F. Other copies may be issued as required upon approval of Manager, Analytical and Process Chemistry.

5.3.4 The original procedures and their respective, completed Procedures/Document Review Forms are filed in the Engineering Document Control files.

5.3.5 The Table of Contents of the Manual documents the current revisions in the Manual and their effective dates.

5.4 Revisions

5.4.1 When a revision (major change) is required, the manager assigns an author.

5.4.2 The author prepares the revision and obtains its approval by following Sections 5.1 and 5.2.

5.4.3 All changes, edits, and revisions in an ACP or a PRD will be denoted by the use of arrows (>) to indicate where the changes have taken place. Sections that are removed from the text will be replaced by the word "deleted". The outline numbers for these sections will remain unchanged.

5.4.4 Changes made in an ACP or a PRD will be done by Engineering Change Notice, Form WV-1839.

5.4.5 The approved revision is distributed according to Section 5.3. The Table of Contents of the Manual is updated and distributed with the revision.

6.0 REFERENCES

6.1 PRD 2.0, Quality Assurance Program.

6.2 PRD 4.0, Procedures.

ATTACHMENT A

Format For Program Requirements/Descriptions (PRD)

The following three-part format is used to prepare PRDs. The first page heading and the heading for subsequent pages are given.

a. Format

Each PRD relates to one specific quality assurance requirement from the designated quality assurance standard. This basic requirement from the standard is referred to as a program area.

1.0 SCOPE

This section is used to introduce and briefly summarize the program area of concern. Reference may be made to applicable ACPs.

2.0 REQUIREMENTS

The quality assurance requirement of concern is presented in detail as a group of requirements. The imperative verb form is used in writing the requirements. The source of the quality assurance requirement is referenced at the end of the section.

3.0 PROGRAM IMPLEMENTATION

This section describes how the requirements given in Section 2.0. are implemented. The present tense is used in writing the descriptions. The section is divided into appropriate subdivisions of the requirements in Section 2.0. The subsections are numbered sequentially starting with 3.1. In some cases, the description in a subsection will provide for complete implementation of the respective requirement. In other cases, additional and more detailed instructions are required in the form of one or more ACPs. Applicable ACPs are referenced.

ATTACHMENT A (Continued)

b. First Page Heading

The following heading is used on the first page.

PRD _.0 Rev. __
Effective Date: _____

TITLE

Approved By:

_____
Manager,
Analytical and Process Chemistry

Concur:

_____
Manager,
Quality Engineering

---

c. Subsequent Page Heading

The following heading is used on subsequent pages.

PRD _.0 Rev. __
Effective Date: _____

ATTACHMENT B

Format For Administrative Control Procedures (ACP)

The following six-part format is used to prepare ACPs. Page headings used are as used for this ACP. All sections are used in each ACP. If there are no definitions required in Section 3.0, a statement to that effect is made.

a. Format

1.0 PURPOSE

Briefly describes the purpose of the procedure.

2.0 APPLICATION

Presents the area, function, or circumstances for which the procedure is applicable.

3.0 DEFINITIONS

Defines terminology specific to the procedure that may be unfamiliar.

4.0 RESPONSIBILITIES

Identifies the responsibilities of personnel involved in using the procedure or participating in actions associated with the procedure.

5.0 PROCEDURE

Provides instructions for doing the work involved. The instructions are presented in a stepwise format and are divided into appropriate subsections that group the instructions into closely related activities. The subsections are numbered sequentially starting with 5.1.

6.0 REFERENCES

Identifies related PRDs and sources of information related to the purpose of the ASP.

b. First Page Heading

The following heading is used on the first page:

ACP _.1 Rev. __
Effective Date: _____

TITLE

Approved By: Concur:

_____ _____
Manager, Manager,
Analytical and Process Chemistry Quality Engineering

---

c. Subsequent Page Heading

The following heading is used on subsequent pages:

ACP _.1 Rev. __
Effective Date: _____

PREPARING, APPROVING, AND DISTRIBUTION OF ANALYTICAL CHEMISTRY METHODS

UNCONTROLLED

Approved By:

C. W. McVay, Manager,
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> This procedure establishes the method to be used in preparing, approving, and distributing methods covering analyses performed by the Analytical and Process Chemistry group. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

This procedure applies to the issuing and revision of Analytical Chemistry Methods used by Analytical and Process Chemistry.

3.0 DEFINITIONS

3.1 Analytical Chemistry Methods (ACM): Technical procedures (methods) used for the analysis of samples and for other laboratory work such as the preparation of reagents and the operation of instruments.

4.0 Responsibilities

4.1 The manager, scientists (other than the author), and senior technicians of Analytical and Process Chemistry are responsible for reviewing the ACM.

4.2 Quality Assurance is responsible for reviewing the Quality Assurance aspects of the ACM.

4.3 The Manager of Analytical and Process Chemistry is responsible for approving and issuing ACMs.

4.4 The chemist who does the development work leading to a new ACM is responsible for validating the method according to Reference 6.3.

5.0 PROCEDURE

5.1 Preparation

5.1.1 When the need for an ACM is identified, the manager assigns an author for preparation. If the ACM is a new method based on development work, the author is usually the chemist who did the development.

5.1.2 The author follows the format and instructions given in Attachment A and B.

5.1.3 An alpha numeric identification code is assigned to each ACM as follows: ACM XXX-#, where the Xs identify the analyte and/or instrument used, and the # is the sequential number assigned to that analyte or instrument. Examples: ACM Sr90-1, ACM ICP-1, ACM Fe-2.

5.1.4 The revision number of a new ACM (first issue) is assigned 0 (original). Revisions are numbered sequentially starting with 1.

5.1.5 The effective date is the date the original or subsequent revision is authorized for use, which is the date the manager approves it by signing the first page.

5.1.6 The pages are numbered sequentially as follows: Page 1 of 8, Page 2 of 8, etc. The second number is the total number of pages. Pages of attachments are included in the sequence.

5.1.7 The manager maintains a log of ACM numbers, current revisions, and effective dates. The Table of Contents of the manual containing the ACMs may serve as the log (see 5.3.5).

5.2 Review and Approval

5.2.1 When an ACM is ready for review and approval, the author prepares a WVNS Document/Design Review Transmittal Form (WV-1840) and signs the form.

5.2.2 The form, with a copy of the ACM, is circulated for review and approval as indicated below.

A. Review only: chemists and senior technicians in Analytical and Process Chemistry; Quality Engineering.

B. Approval: Manager, Analytical and Process Chemistry.

5.2.3 The author resolves comments from the reviewers. This is an interactive process.

5.2.4 When all of the comments have been resolved and the review completed, the manager approves the ACM by signing the form and the first page of the ACM.

5.2.5 Release of ACMs will be done by an Engineering Release, Form WV-1802.

5.3 Distribution

5.3.1 ACMs are assembled together in a methods manual.

5.3.2 Distribution of ACMs is controlled through the controlled distribution of the manual.

5.3.3 The manual is controlled through the WVNS Engineering Document Control System. Its distribution is as follows:

A. One copy to the Manager, Analytical and Process Chemistry (A&PC).

B. One copy to each chemist and technician in A&PC.

C. One copy to each A&PC laboratory (laboratory desk copy).

D. One copy to Quality Assurance.

E. One copy to Master Records Center per WV-103.

> F. Other copies may be issued as required upon approval of Manager, Analytical and Process Chemistry.

5.3.4 The original ACMs and their respective, completed Procedures/Document Review Forms are filed in the Engineering Document Control files.

5.3.5 The Table of Contents of the manual documents the current revisions of ACMs and their effective dates.

5.4 Revisions

5.4.1 When a revision is required, the manager assigns an author. Revisions are major changes as defined in Reference 6.2.

5.4.2 The author prepares the revision and obtains its review and approval by following Sections 5.1 and 5.2.

5.4.3 All changes, edits, and revisions in an ACM will be denoted by the use of arrows (>) to indicate where the changes have taken place. Sections that are removed from the text will be replaced by the word "deleted". The outline numbers for these sections will remain unchanged.

5.4.4 Changes made in an ACM will be done by Engineering Change Notice, Form WV-1839.

5.4.5 The approved revision is distributed according to Section 5.3. The Table of Contents of the manual is updated and distributed with the revision.

6.0 REFERENCES

6.1 PRD 2.0, Quality Assurance Program

6.2 PRD 4.0, Procedures

6.3 ACP 8.3, Method Validation

ATTACHMENT A

Format for Analytical Chemistry Methods

ACMs are prepared using the following format. The sections are presented in the order given and all sections are used. Should a section not be applicable for a particular method, the heading is used and a statement is made regarding its nonapplicability. The ACMs are divided into two parts. Part I provides general information about the method and Part II provides the instructions and information required to use the method.

Part I

1.0 PURPOSE

Briefly state the purpose of the method.

2.0 APPLICATION

Present information regarding the type of samples for which the method can be used and other information if appropriate, such as interferences, detection limit, applicable concentration range, expected precision, and known bias.

3.0 DISCUSSION

Describe the method in terms of the chemistry involved and include discussion of items presented in Section 2.0 if appropriate.

4.0 REFERENCES

Include references that support the technical basis for the method and provide additional useful information for understanding the method and its application.

Part II

5.0 EQUIPMENT

List the required equipment, including applicable information for each item such as type, quantity required, make and model, and special conditions for use. Reference ACP 7.1.

6.0 REAGENTS AND STANDARDS

List the reagents and standards required. Include with each item (as applicable) concentration, matrix, special storage conditions, limits on use, and instructions for preparation. State the grade or quality of a reagent required if other than reagent grade. When water of a quality other than distilled or deionized is required, state the special quality required and provide instructions for its preparation if appropriate. Reference ACPs 7.1 and 8.1.

7.0 SAFETY PRECAUTIONS

State known or potential safety hazards associated with the use of the equipment and reagents. Give applicable instructions and precautions for controlling hazards. Reference ACPs 7.2 and 7.4 and other documents as applicable.

8.0 RECORDS

State where information and measurement data generated during an analysis and calculations are to be recorded. Reference ACP 5.1.

9.0 CALIBRATION AND CONTROL

Provide step-by-step instructions for calibration of equipment and for controlling the method, as appropriate. Include the standards used, frequency of calibration, frequency of analyzing the control standard, criteria for establishing calibration/control, and actions to take if calibration/control is lost. Criteria governing out-of-control situations are given in ACP 8.2, which should be referenced. When an analytical balance is required, criteria shall be included for checking the balance before and during use. Reference ACPs 7.1 and 8.2 as appropriate.

10.0 PROCEDURE

Provide step-by-step instructions for performing the analysis. Divide this section into appropriate subsections. Tolerances for all measurements required in the procedure shall be specified using the system given in ACP 8.2 (reference ACP 8.2). Include, if appropriate, a subsection on pretreatment or handling of samples (other than the handling of samples as prescribed in ACP 7.3).

11.0 CALCULATIONS

Give any equations necessary for treating the measurement data to arrive at a reportable result, including any instructions required to make the calculations. State the units that are given with the result and significant numbers reported (Reference ACP 8.2).

12.0 ATTACHMENTS

Forms used for data recording and reporting. Worksheets will include: log number, method identification, raw data, calculated result, analyst initial and date, and approval initial and date.

ATTACHMENT B

Page Headings for Analytical Chemistry Methods

FIRST PAGE

WEST VALLEY NUCLEAR SERVICES CO. INC.

ACM XXX-# Rev. X
Effective Date:

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

TITLE

Approved By:

_____
Manager,
Analytical and Process Chemistry

SUBSEQUENT PAGES

ACM XXX-#, Rev. X
Effective Date:

LABORATORY RECORD SYSTEM

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 SCOPE

To provide instructions for the use of the forms and practices that establish the laboratory record system.

2.0 APPLICATION

The application of this procedure provides for complete documentation of the work done in the laboratories of Analytical and Process Chemistry (A&PC), from receipt of samples to reporting of analytical results. This record system provides for traceability of analytical results back to raw data, control of samples as they are processed through the laboratories, and documentation of who did the work and when it was done. See Reference 6.1.

3.0 DEFINITIONS

3.1 Analytical Request Form: Used to receive sample information, submit samples to A&PC, and request analyses. A record used to report analytical results to sample submitters and other appropriate persons. Analytical reports are reviewed for adequacy and approved before reporting (see References 6.4 and 6.5). The completed Analytical Request form serves as the Analytical Report.

3.2 Log Number: A unique number assigned to each sample or group of up to four samples received by A&PC. The first two digits represent the year and the remaining part of the number is the sequential number starting at one for each year; for example: 87-1, 87-2, etc.

> 3.3 Log Book: A bound record book in which sample information is logged. This book is synonymus with sample Tracking Log.

3.4 Work Sheet: Record used to record analytical data and calculations. The Work Sheet functions as a Traveler (see Reference 6.1).

3.5 Environmental Analysis Check Sheet: A form used as a checklist to record the quality of the environmental sample submitted for analysis.

> 3.6 Sample Disposal: Currently being done on Realm Computer Program. See Reference 6.3.

4.0 RESPONSIBILITIES

> 4.1 The personnel of A&PC are responsible for following the provisions of this procedure as they process samples.

4.1.1 Each analytical number reported must be traceable back to raw data.

4.1.2 All work sheets will be included, signed by analyst and approved by qualifed second party. Only the current revision of each worksheet will be used.

4.1.3 No tampering with any analytical results after the Analytical Report has been approved unless an obvious mistake is found. At this point the analyst who approved the report and/or the manager or designee may correct the problem, sign and date the correction and include a brief explanation. Data in the Realm computer program will be changed if analytical results are changed after input.

4.1.4 Analytical worksheets should include which test and measuring equipment were used to obtain the analytical results.

4.1.5 Each Analytical Report file will have each page numbered (ie. 1 of 1) in chronological order (including worksheets, printouts, etc.)

4.1.6 Sample Tracking Logs will be maintained by designee. For sample receipt: log number, sample name, and date. Competed Analytical Report files: date and initials when files are stored with Document Control in locked fire proof cabinets.

> 4.2 Chemists, Radiological Technicians A, senior technicians, and specialists are responsible for reviewing and approving analytical results from their areas of work responsibility. See Reference 6.5.

> 4.3 All primary documentation should be reported in dark indelible ink, legible and neat, no erasures, no whiteouts, no scratch outs, only one line through mistake then dated and initialed or signed and correct response next to mistake. Marked out entries should still be legible. Computer printouts and instrument tapes are acceptable primary documentation.

> 4.4 The manager of A&PC is responsible for approving exceptions to the provisions of this ACP. See 5.1.2.

5.0 PROCEDURE

> 5.1 Analysis Request

5.1.1 The Analytical Request Form shall be filled out by the sample submitter (requester) as follows: (See Attachment 6.7)

(1)	Enter the name of the sample.
(2)	Enter the charge number of the organization requesting the analysis.
(3)	Log number entered by A&PC.
(4)	Name of person(s) completed request sheet is to be sent to.
(5)	Requestors phone number.
(6)	Name of person who sampled.
(7)	Date sampled.
(8)	Time sampled.
(9)	Location sampled.
10, 11, 12, 13	To be filled in stating requirement number requesting analyses
(14), (15)	To be initialed by the deliverer, sampler, supervisor or R&S Representative.
(16), (17) (18)	To be completed by R&S representative if necessary.
(19)	Check the appropriate answer.
(20)	List Hazards.
(21)	Circle or list any requirements needed.
(22)	State the required minimum detection level.
(23)	Enter any additional comments.
(24, 25)	Signature of Supervisor or Manager and date
(26)	Initials of deliverer.
(27)	Time delivered.
(28)	Date delivered.
(29, 30, 31)	To be completed by A&PC.
(32)	Enter the sample identification.
(33, 34)	Enter analysis requested.

5.1.2 All samples shall be submitted with a completed Analysis Request Form, unless prior arrangements have been made and approved by the manager of A&PC.

> 5.1.3 A&PC staff receiving samples shall check the Analytical Requests for completeness in dark indelible ink, legible and neat, no erasures, no whiteouts, no scratch outs, only one line through mistake then dated and initialed or signed and correct response next to mistake. Marked out entries should

still be legible, and if satisfactory, shall enter time and date and sign (or initial) the form indicating acceptance of the samples.

5.1.4 Instructions for the receipt of samples in general and for radioactive samples in particular are given in ACPs 7.3 and 7.4 respectively (References 6.3 and 6.6).

5.2 Sample Log

> 5.2.1 Once samples are accepted, Log Numbers shall be assigned from the Log Book. The numbers shall be assigned from the Log Book sequentially. The computer generates the Log Number using the Realm Program.

5.2.2 The Log Number for each sample shall be recorded on the corresponding Analysis Request form.

> 5.2.3 Sample information shall be entered onto the Computer using the Realm Program.

5.2.4 Each sample shall be labeled with its assigned Log Number.

> 5.2.5 When sample analysis is completed, the date of completion will be recorded into the Realm Computer Program.

> 5.2.6 Date of sample disposal will be logged into the Realm Computer Program.

5.3 Work Sheet

5.3.1 A Work Sheet shall be initiated for each analysis.

5.3.2 The analysts shall enter sample data and information on the Work Sheets as the analyses are performed. Calculations shall also be made on the Work Sheets as appropriate.

> 5.3.3 The results shall be reviewed and approved by a seperate responsible chemist or technician other than the one who performed the analyses.

5.4 Analytical Report

5.4.1 Approved results shall be entered on the corresponding Analytical Request form.

5.4.2 When all of the results have been entered on an Analytical Request form, the completed form shall be approved by the responsible chemist or technician.

> 5.4.3 The data from the approved Analytical Request Form will be entered by a technician or clerk on the Realm Computer Program.

> 5.4.4 Copies of the approved report shall be sent to the appropriate persons and one copy to MRC.

> 5.4.5 The completed Analytical Request form with the associated Work Sheets attached shall be filed in the completed work file.

5.5 Sample Disposal

> 5.5.1 When sample analyses for applicable samples are completed, the Log Numbers of the corresponding samples shall be entered into the Realm Computer Program.

> 5.5.2 When the radioactive sample has been disposed of, the disposal information shall be entered into the Realm Computer Program. Instructions for the disposal of samples are contained in ACPs 7.3 and 7.4 (References 6.3 and 6.6).

> 5.5.3 Sample disposal shall be tracked and recorded using the Realm Computer Program.

5.6 Sample Receipt Inspection Sheet

5.6.1 When EPA samples are submitted, a Sample Receipt Inspection sheet shall be filled out to assure proper sampling and handling of the samples.

5.6.2 The Sample Receipt Inspection sheet shall include a checklist for the following:

5.6.2.1 The use of appropriate containers and closure,

5.6.2.2 Proper filling of the container,

5.6.2.3 Adequate cooling, if required,

5.6.2.4 Adequate pH adjustment, if required,

5.6.2.5 Date and time of sampling so that analysis can be started within the prescribed holding time.

6.0 REFERENCES

6.1 PRD 5.0, Laboratory Records.

6.2 PRD 7.0, Control of Equipment and Materials.

6.3 ACP 7.3, Control of Samples.

6.4 PRD 8.0, Control of Measurements.

6.5 PRD 1.0, Organization.

6.6 ACP 7.4, Handling Radioactive Materials.

6.7 Attachment A, Analytical Request Form

6.8 Attachment B, Sample Receipt Inspection Sheet

ANALYTICAL REQUEST

Sample Name:_____1_____ Charge No.:_____2_____ Log No.:_____3_____

Results Reported To:_____4_____ Phone:_____5_____

Sampled by:_____6_____ Date:_____7_____ Time:____8___ Location:_____9_____

WO#:____10_____ SOP#:____11____ OPDR#:_____12_____ Other:_____13_____

A. Does this sample contain radioactive material? 14 ( ) yes ( ) no
B. Is the gross activity <5E-3 uCi/mL 15 ( ) yes ( ) no
C. Dose rate for samples with activity greater than 5E-03 uCi/mL at 2 inches:

_____16_____ mR/Hr WC R/S Rep.:_____17_____ Date:_____18_____

D. Specific Hazards (ie. HF, Strong Base, Cd, etc.) 19( ) yes ( ) no

If yes, list_____20_____

E. Specific requirements (circle): EPA NRC DOE N/A SPDES Other_____21_____
Process Control EPTOX Waste Classification

F. Discard after two (2) weeks:22( ) yes ( ) no (state reason in comments)

G. Comments:_____23_____

Supervisor or Manager Signature_____24_____ Date:_____25_____

To be filled in when sample delivered to laboratory personnel:

Delivered:__26__ Time:__27__ Date:__28___ Received:__29__ Time:__30__ Date:_31__

SAMPLE IDENTIFICATION

Analysis Requested	Units	32			
33	34	☐	☐	☐	☐
		☐	☐	☐	☐
		☐	☐	☐	☐
		☐	☐	☐	☐
		☐	☐	☐	☐

Data Approved:_____ Date:_____ Time:_____

ANALYTICAL REQUEST

Sample Name:_____ Charge No.:_____ Log No.:_____

SAMPLE IDENTIFICATION

Analysis Requested	Units				

Data Approved:_____ Date:_____ Time:_____

ATTACHMENT B

SAMPLE RECEIPT INSPECTION SHEET

LOG NUMBER	PROPER CONTAINER	PROPER CLOSURE	PROPER FILLING	ADEQUATE COOLING	pH	DATE AND TIME SAMPLED

UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC.

ADMINISTRATIVE CONTROL PROCEDURE
VITRIFICATION LABORATORY, PROCESS DEVELOPMENT

ACP 5.2, Rev. 0
Effective Date: 06/09/89

VITRIFICATION LABORATORY RECORD SYSTEM

Approved by:

S. M. Barnes, Manager
Vitrification Process Development

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

To provide instructions for the use of forms and practices that establish the laboratory record system for the Vitrification Laboratory. This procedure replaces ACP 5.1, Laboratory Record System, for the Analytical and Process Chemistry.

2.0 APPLICATION

The application of this procedure provides for complete documentation of the work done in the Vitrification Laboratory, from the receipt of work request to the reporting of results. This record system provides for traceability of the results back to raw data, control of samples as they are tested, and documentation of who did the work and when it was done. See reference 6.1.

3.0 DEFINITIONS

3.1 Vitrification Laboratory Work Request Form: used to receive test information, submit samples, and request tests. A record used to report results to the sample submitter and other appropriate persons. Vitrification Laboratory Request Forms are reviewed for adequacy and approved before reporting by the Vitrification Laboratory Cognizant Engineer (see references 6.4 and 6.5). The completed Vitrification Laboratory Request form serves as the Vitrification Laboratory Report.

3.2 Log Number: a unique number is assigned to each work request received by the Vitrification Laboratory. The first two digits represent the year and the remaining part of the number is the sequential number starting at one for each year; for example: 89-1, 89-2, etc.

3.3 Log Book: a bound record book in which sample information is logged and from which consecutive Log Numbers are obtained for samples.

3.4 Work sheets: the work sheets are assigned for each experiment by the Cognizant Engineer or his designate. These sheets are used to record observed data and results during experiments.

3.5 Laboratory Note Book: a Laboratory Data Note Book used to record Vitrification Laboratory data and calculations. A unique number is assigned to each Engineering Note Book. The first two digits represent the year and the next two digit represent a sequential number starting at one for each year; for example: 89-01, 89-02, etc. and the remaining part of the number is the initials (first and the last name) of the Vitrification Laboratory technician.

3.6 Analytical Request Record Book: a bound note book recording, in a sequential order, information on the requests sent to Analytical Laboratory for chemical analysis.

3.7 Sample Disposal Record Book: a bound note book recording, in a sequential order, information on the disposition or storage of the samples after the completion of tests.

3.8 VIP: is a vitrification filing system.

4.0 RESPONSIBILITIES

4.1 The personnel of Vitrification Laboratory are responsible for following the provisions of this procedure as they process samples.

4.2 The Cognizant Engineer or his designate are responsible for reviewing and approving Vitrification Laboratory results.

All primary documentation should be reported in dark ink, no erasures, no white outs, no scratchouts, only one line through mistakes then dated and signed and write correct response next to mistake.

4.3 The manager, Process Development, is responsible for approving exceptions to the provisions of this ACP.

5.0 PROCEDURE

5.1 Vitrification Laboratory Work Request

5.1.1 The Vitrification Laboratory request form shall be filled out by the requester as follows (see attachment A):

0) Log number entered by Vitrification Laboratory

1) Enter name of the sample

2) Enter the charge number of the organization requesting the analyses

3) Name of person(s) completed requested sheet is to be sent to

4) Requesters phone number

5), 6), 7) Date, Time and Location of sampling

8) Signature of the supervisor or manager

9), 10), 11) Initialed by deliverer, sampler, or supervisor with time and date

12) Purpose of the analyses

13) Specific requirements or conditions for analyses

14) Circle the appropriate answer and initial

15) Comments regarding samples

16) List and conditions for specific tests requested

17) Format in which results are requested

18), 19), 20) Signature of the Cognizant Engineer with date and time of approval

5.2 Sample Log

5.2.1 Once the work requests are accepted, Log Numbers shall be assigned from the Log Book. The numbers shall be assigned from the Log Book sequentially.

5.2.2 The log number for each sample shall be recorded on the corresponding Vitrification Laboratory Work Request form.

5.2.3 Sample information shall be entered into the Log Book.

5.2.4 Each sample shall be labeled with the assigned Log Number.

5.2.5 When the experiment is completed, the date of completion and the location of the data in the Engineering Note Book will be recorded in the Log Book. Information will include log number assigned to the Laboratory Note Book and the page numbers on which data was recorded.

5.2.6 An example of the sample Log Record Book Format is shown as attachment B.

5.3 Work Sheet

5.3.1 A work sheet shall be initiated for each experiment.

5.3.2 The Vitrification Laboratory Technician shall enter sample data and the information on the work sheets as the experiment is performed.

5.3.3 The results shall be reviewed and approved by a separate responsible technician, other than the one performing the analysis.

5.4 Laboratory Note Book

5.4.1 The assigned Vitrification Laboratory Technician for the Work request shall enter the sample information and the sample data collected in the Note Book assigned to him by the Cognizant Engineer.

5.4.2 When data and results are entered in the Note Book, the completed Note Book shall be reviewed and approved by responsible technician.

5.5 Analytical Request Record Book

5.5.1 An analytical request form (WV-1113, Rev. 1) shall be filled for the samples requiring Analytical Process Chemistry Support.

5.5.2 The information shall be entered in the "Vitrification Laboratory Analytical Request Record Book." Information will include log number, analysis requested, data entered, initials, date analysis received, initials of the person filling the book.

5.5.3 An example of the Analytical Request Record Book Format is attached as an Attachment C.

5.6 Vitrification Laboratory Report

5.6.1 Approved results shall be attached to the corresponding Vitrification Laboratory Request form.

5.6.2 When all the results have been entered on a Vitrification Laboratory Request form, the completed form shall be approved by the Cognizant Engineer, or his designate.

5.6.3 Copies of the approved report shall be sent to the appropriate persons and one copy to MRC by the Cognizant Engineer, or his designate as soon as possible.

5.6.4 The completed Vitrification Laboratory Request form with the associated work sheets and the carbon copies of Engineering Note Book attached shall be filed in the VIP.

5.7 Sample Disposal

5.7.1 When sample analyses/experiments for the applicable samples are completed, the Log Numbers of the corresponding samples shall be entered on the sample disposal record book. Information will include log numbers, description of the action taken, date entered, and initials of the person filling the book.

5.7.2 An example of the sample disposal record format is attached as an Attachment D.

6.0 REFERENCES

6.1 PRD 5.0, Laboratory Records

6.2 PRD 7.0, Control of Equipment and Materials

6.3 ACP 7.3, Control of Samples

6.4 PRD 8.0, Control of Measurements

6.5 PRD 1.0, Organization

6.6 Attachment A, Vitrification Laboratory Work Request form.

6.7 Attachment B, Vitrification Laboratory Log Record Book Format

6.8 Attachment C, Vitrification Laboratory Analytical Request Record Book Format.

6.9 Attachment D, Vitrification Laboratory Sample Disposal/Storage Record Book Format.

ATTACHMENT A
VITRIFICATION LABORATORY WORK REQUEST FORM
(NONRADIOACTIVE ANALYSES/EXPERIMENTS ONLY)

LOG NO. _____(0)_____
SAMPLE NAME _____(1)_____ CHARGE NO. _____(2)_____

RESULTS REPORTED TO _____(3)_____ PHONE _____(4)_____

DATE ____(5)____ TIME ___(6)___ LOCATION _____(7)_____

SUPERVISOR/MANAGER'S SIGNATURE _____(8)_____

DELIVERER _____(9)_____ TIME ___(10)___ DATE ____(11)____

PURPOSE OF ANALYSIS_____(12)_____
_____
_____

SPECIFIC REQUIREMENTS_____(13)_____
_____

SPECIFIC HAZARDS (e.g., STRONG ACID, BASE ETC.) [ ] YES [ ] NO
IF YES, LIST _____(14)_____

COMMENTS_____(15)_____

TESTS/EXPERIMENTS REQUESTED: (BRIEFLY SUMMARIZE THE TYPE, CONDITIONS (LIKE TEMPERATURE, PRESSURE), NUMBER OF TESTS REQUIRED ON THE REQUESTED SAMPLES)
(16)

FORMAT IN WHICH RESULTS ARE REQUESTED: (17)

(SEE THE ATTACHED SHEETS FOR RESULTS)

DATA APPROVED _____(18)_____ DATE ___(19)___ TIME ___(20)___

ATTACHMENT B
VITRIFICATION LABORATORY LOG RECORD BOOK FORMAT

S. NO.	DATE	SAMPLE NAME	ASSIGNED LOG NO.	INITIALS	DATE COMPLETED	NOTEBOOK LOG NO.	NOTEBOOK PG. NOS.	INITIALS

ATTACHMENT C
VITRIFICATION LABORATORY ANALYTICAL REQUESTS RECORD BOOK FORMAT

S. NO.	SAMPLE LOG NO.	ANALYSIS REQUESTED	DATE ENTERED	INITIALS	DATE ANAL. REC'D.	INITIALS
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						

ATTACHMENT D
VITRIFICATION LABORATORY SAMPLE DISPOSAL/STORAGE
RECORD BOOK FORMAT

S. NO.	SAMPLE LOG NO.	ACTION TAKEN	DATE	INITIALS
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				

CONTROL OF EQUIPMENT AND REAGENTS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concern:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> To provide instructions for the control of equipment and reagents as they are handled and used in the laboratory. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

This procedure is applicable for the general control of equipment and for the calibration of equipment that is generally used throughout the laboratories. This procedure also applies to the control of reagents used in A&PC laboratories.

3.0 DEFINITIONS

3.1 ACM: Analytical Chemistry Method.

3.2 Analytical Data: The highest quality measurement data required from a given analysis and used to calculate the result.

3.3 Non-Data: Calibration not required for an instrument or device used only to make approximate or relative measurements.

3.4 Outside Calibration: Calibration performed as a service on a regular basis by an organization external to Analytical and Process Chemistry (A&PC).

3.5 User-to-Calibrate: Calibration made by the analyst during an analysis based on a specified calibration procedure.

3.6 Periodic Calibration: Calibration made on an "as needed" basis.

4.0 RESPONSIBILITIES

4.1 The personnel of A&PC are responsible for following the provisions of this procedure as they use equipment and reagents in their work.

4.2 The manager of A&PC is responsible for approving exceptions to the provisions of this ACP. See 5.3.3.

5.0 PROCEDURE

The proper control of equipment and reagents is important in assuring the reliability of the measurement data produced from their use.

5.1 Calibration Status

5.1.1 The calibration status of instruments used to make measurements shall be indicated by tags or labels or through records retained near the instruments.

5.1.2 Each instrument shall be classified as User-to-Calibrate, Non-Data, Periodic Calibration, or Outside Calibration.

5.1.3 Each User-to-Calibrate label or record shall reference the applicable calibration procedure or ACM (Reference 6.3).

5.1.4 Each Outside Calibration label or record shall indicate the effective calibration period, such as stating the dates when calibration was made and when calibration expires.

5.1.5 When calibration has expired, an instrument shall be labeled or tagged as Out-of-Calibration if it is not to be recalibrated shortly.

5.1.6 Out-of Calibration and Non-Data instruments shall not be used to obtain analytical data.

5.2 Volumetric Ware

5.2.1 The manufacturer's calibration shall normally be accepted for volumetric flasks, burets, and pipettes. Verification of calibration shall be manufacturers' certificates of calibration when available. Certificates shall be retained as records for the life of the equipment to which they belong.

5.2.2 When a piece of volumetric ware requires special calibration, standard practices shall be followed. Each such calibration shall be documented in a laboratory notebook by recording steps taken, measurements obtained, and calculations made. Each piece calibrated shall be uniquely identified and traceable to the record made. Records shall be retained with their corresponding pieces of equipment for the life of the equipment.

5.2.3 If the calibration of a piece of volumetric ware must be routinely checked, a procedure shall be written, which shall include frequency and tolerance limits. A record of each check calibration shall be made and retained with the piece.

5.2.4 Pipetters that require periodic calibration shall be calibrated according to 5.2.3.

5.3 Analytical Balances

5.3.1 Analytical balances are calibrated by an outside organization. A specified calibration frequency shall be established and a calibration certificate or other type of record shall be provided by the calibrating organization for each calibration. The certificate or record shall be retained by A&PC for a minimum of two years.

5.3.2 The calibration status of each balance shall be indicated by an Outside Calibration label as prescribed by 5.1.4.

5.3.3 A balance whose calibration has expired shall not be used without approval of the manager of A&PC. The approval and reasons for taking that action shall be documented.

> 5.3.4 Balances shall be calibrated and checked with Class P Weights or better, daily using one weight selected by manufacturer for each electronic balance.

5.3.5 Class P Weights shall be checked monthly against Class S Weights to assure that calibration is being maintained.

5.4 Thermometers

5.4.1 The manufactures' calibration shall normally be accepted for thermometers. Verification of calibration shall be manufacturers' certificates of calibration when available. Certificates shall be retained as records for the life of the thermometers to which they belong.

> 5.4.2 Each certified laboratory should have access to an NBS traceable, factory certified thermometer. Certification should be at points of interest to the laboratory. After the first year of service and annually thereafter, the certified thermometer should be checked at the ice-point and the correction factors adjusted accordingly.

Record: Date, ice-point reading, adjustment to be made to the correction factors, new correction factors and analyst's initials in a tabular format in a bound notebook.

Each certified laboratory should have a sufficient number of working thermometers so that each may have a dedicated use. Each working thermometer should be uniquely identified by number and calibrated at the temperature(s) of interest prior to being placed into service and annually thereafter for liquid in glass models, or quarterly thereafter for dial models.

Record: Date, thermometer number, calibration temperatures, correction factors, and analyst's signature in a bound notebook.

5.4.3 Thermometers with mercury columns that have separated shall not be used.

5.5 pH Meters

5.5.1 pH meters shall be calibrated daily or with each use, whichever is less frequent. Calibration shall be made using standard buffers.

5.5.2 When a range of pH measurements are required, a two-point calibration shall be used. The slope shall be established using two buffers, one at each end of the required range. The slope shall then be checked using a buffer at the midpoint of the range.

5.6 Grade of Reagents

5.6.1 Normally, the quality of reagents used shall be Reagent Grade (see Reference 6.4). If other grades are required, the requirements shall be stated in the appropriate ACM (see Reference 6.3).

5.6.2 Normally, the quality of water used as a reagent shall be distilled or deionized. If a special quality is required, that quality shall be stated in the appropriate ACM (see Reference 6.3).

5.7 Expiration Dates of Reagents

5.7.1 Manufacturer expiration dates for reagents shall be observed. No reagent shall be used after an expiration date, unless rechecked by use of a standard. If the reagent is found to be good, the expiration date of the material shall be extended for a maximum of one year.

5.7.2 If an expiration date is not given for a specific reagent by a manufacturer, that reagent can be used indefinitely, but must be checked against a known standard yearly to assure that the reagent has maintained the specified value.

5.7.3 When reagents are prepared in the laboratory, such as solutions, expiration dates shall be established as appropriate.

5.8 Reagent Storage

5.8.1 All storage containers shall protect reagents from contamination by impurities and from changes in concentration.

5.8.2 The conditions associated with storage areas or bench storage shall be controlled to prevent unnecessary deterioration of reagents.

5.8.3 When special storage conditions are required, the requirements shall be stated in the appropriate ACM (see Reference 6.3). Those special conditions shall be followed in the laboratory.

5.9 Reagent Labeling

5.9.1 All reagents shall be labeled either by a manufacturer's label, a laboratory label or any combination of the two. As long as all the required information (outlined below) with easy access, is found on each container.

5.9.2 A laboratory label shall contain as a minimum: name of reagent, concentration, matrix if other than distilled water, date received initial and date opened, initial and date emptied, date prepared, expiration date if one is established, and limitations in use if appropriate.

5.9.3 Improperly labeled reagents shall be either discarded or relabeled properly, depending on the certainty of identification when found.

5.9.4 Laboratory labels shall be applied in a manner to prevent their deterioration under normal laboratory conditions.

5.9.5 Never use Reagents that are already opened and do not have any documentation of when Reagent opened.

5.9.6 All Reagents necessary for NYSPDES Analyses must be stored seperate from normal Analytical and Process Reagents.

5.9.7 All Reagents necessary for NYSPDES Analyses must be consumed within three years or less (if manufacture specifies shorter term).

6.0 REFERENCES

6.1 PRD 7.0, Control of Equipment and Reagents.

6.2 PRD 8.0, Control of Measurements.

6.3 ACP 4.2, Preparation, Approval, and Distribution of ACMs.

6.4 "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, Washington D.C.

LABORATORY SAFETY

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

To provide general information relating to safe practices in laboratory operations. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

2.1 This procedure is applicable for normal operations encountered in an analytical chemistry laboratory. Special safety hazards are covered in the ACMs (Reference 7.1). Provisions for handling radioactive materials are found in Reference 7.2.

2.2 Chemical Hazards

2.2.1 General

A. Every work place is exposed to hazardous chemicals. It is the responsibility of all personnel to know what chemicals are present, what hazards are associated, and what precautions are required.

B. A hazardous chemical is a substance (liquid, solid, or gas) which may pose any of the risks listed in 2.2.2, A. of this subpart.

2.2.2 Information and Training

A. Chemicals can be hazardous for any one or more of the following reasons:

1. Toxicity, acute or chronic
2. Mutual reactivity
3. Flammability
4. Pyrophoricity
5. Radioactivity, fissile reactivity
6. Irritation, corrosivity
7. Decomposition, explosively or to form toxic products
8. Carcinogenicity, mutagenicity, and teratogenicity
9. Asphyxiation
10. Pathogenicity
11. High temperature/pressure
12. Age

B. Supervision shall ascertain the hazardous properties, alone and in combination, of each chemical present in the work environment. Adequate protection shall be determined and provided. Employees shall be taught the hazards of all chemicals present and the protective measures to be taken. Compliance with the designated procedures shall be enforced. Personnel training, chemical labeling, and Material Safety Data Sheets (MSDS) required by the OSHA Hazard Communication Standard must be complied with. A Spill Prevention Control and Countermeasures (SPCC) plan as required by ID Order 5480.1, Chapter XII, "Prevention, Control, and Abatement of Environmental Pollution," shall be developed and implemented.

2.2.3 Containers and Storage for Laboratory and Shop

A. The quantities of chemicals kept in laboratories and shops shall be the minimum reasonable quantities. Long term storage of chemicals shall not be permitted in use areas. Chemicals shall be marked or labeled with expiration date for those chemicals that may degrade or have a specified "shelf life." Old or outdated chemicals shall be properly disposed of on a routine basis.

B. Particular attention shall be paid to chemicals which may deteriorate to a more hazardous form with time. Storage conditions which may contribute to deterioration shall be avoided. These conditions may include freezing, high temperature, radiation, other chemical vapors, etc. Inventory listing shall be reviewed and updated periodically.

C. Chemicals shall be segregated by type and compatibility and shall be provided with adequate barriers to prevent hazardous interaction.

D. Chemicals shall be stored so that the possibilities of spillage or brokerage is minimized.

E. All reagents necessary for NYSPDES analyses must be consumed in three years or less (if manufacturer specifies shorter term).

2.2.4 Use of Chemicals

A. Laboratory use of chemicals of high risk, such as the perchlorate and cyanide radicals, shall be used only with approved written procedures, or under the supervision of a Senior Technician or a Chemist. Laboratory work which may involve hazardous reactions or conditions shall also be performed according to written procedures, or under the supervision of a Senior Technician or a Chemist. Other laboratory operations will be performed with standard laboratory precautions.

B. Hazardous chemicals or operations which may generate or release hazardous gases, vapors, or dusts shall require adequate ventilation. In most cases, such work should be performed in hoods or glove boxes.

C. Laboratories using chemicals shall be kept clean and free from spillage. Auxiliary equipment such as pumps, blowers, ducts, protective gear, etc., shall be regularly inspected, tested, and maintained by laboratory personnel. Required hood face velocities shall be maintained.

D. Situations in which the risk of explosion is significant shall be evaluated and effective barriers (shields, distance, etc.), shall be imposed.

E. Special attention shall be given the procedures used with perchlorates. Hoods, ducts, blowers, etc., shall be designed to accommodate regular wash downs, and such wash downs shall be regularly performed. The Handbook of Laboratory Safety, published by the Chemical Rubber Co., is suggested as a reference on the safe handling of perchloric acid, perchlorates, and other hazardous chemicals.

2.2.5 Personnel Protection

Personnel working with hazardous chemicals shall be provided with, and required to use, the appropriate protective devices as listed in the appropriate Analytical Chemistry Method.

2.2.6 Disposal

A. Personnel in charge of work places storing and/or using chemicals shall routinely inventory such chemicals for the purpose of eliminating those which are no longer used, those which are excess to needs, and those which may have deteriorated.

B. Chemicals identified in a., immediately above, shall be disposed of by laboratory personnel according to procedures approved by the responsible safety authority. The method of disposal shall be determined by the economic value, the risks to humans and the environment, the availability of disposal facilities, and the requirements of applicable regulations.

3.0 DEFINITIONS

3.1 ACM: Analytical Chemistry Method.

4.0 RESPONSIBILITIES

4.1 The personnel of Analytical and Process Chemistry (A&PC) are responsible for following the provisions of this procedure to assure a safe work environment.

> 4.2 The manager of A&PC is responsible for promoting safe operations and assuring that the staff observes good safety practices. The manager is also responsible for approving deviations from provisions in this ACP if required and for appointing A&PC Safety Inspectors.

> 4.3 A&PC Safety Inspectors are responsible for performing periodic laboratory inspections in accordance with Attachment A.

5.0 PROCEDURE

5.1 Chemical

5.1.1 Proper protective equipment shall be worn and used when handling corrosive, toxic, and other hazardous chemicals. Emergency actions shall be established for skin contact, inhalation, or ingestion of hazardous chemicals. See 5.6 below.

5.1.2 When diluting concentrated acids with water, the acid should be added to the water slowly with stirring.

5.1.3 Chemicals shall be properly labeled based on industrial practices for indicating hazardous materials. Unlabeled chemicals or those with unreadable labels shall be disposed of as if they were hazardous materials (See 5.1.5).

5.1.4 Spilled chemicals shall be cleaned up immediately. Leaky or corroded containers shall be disposed of (See 5.1.5).

5.1.5 Chemicals shall be disposed of according to established practices based on the nature of each. Disposal shall be in accordance with approved company practices. Hazardous nonradioactive waste must be disposed of in such a manner that neither the environment nor personnel may be adversely affected. Procedure for disposal of this type of nonradioactive waste must be obtained on a case by case basis from Plant Systems Operations. Disposal of mixed hazardous and radioactive waste must be handled by written procedure approved by Facility Management and the Safety Department.

5.1.6 Incompatible chemicals shall not be stored together. Chemicals shall be stored in locations and under conditions to prevent exposure to conditions that could create hazardous situations.

Incompatible chemicals are substances which may react with possible hazardous consequences. The following is a partial listing of chemicals which are incompatible.

Chemicals listed in the left-hand column shall be stored so that they do not come in contact with the corresponding chemicals listed in the right-hand column (see Section 2). In addition incompatible chemicals must not come in contact at any time except under the cognizance of the Analytical and Process Chemistry Department or as permitted by approved operating procedures.

LISTING OF INCOMPATIBLE CHEMICALS

Do No Contact These	With These
Acetic acid (see also Acids)	Chromic acid, ethylene glycol, sodium hydroxide, nitric acid, ammonium hydroxide (aqueous ammonia), perchloric acid, permanganates, peroxides
Acetone (see also Ketones)	Hydrogen peroxide, concentrated nitric-sulfuric acid mixtures, chromic acid
Acetylene	Copper, halogens, mercury, silver, tin, potassium
Acids, concentrated	Lithium, sodium, potassium, calcium, sulfides, carbides, phosphides, cyanides, metals (finely divided), arsenic compounds, sulfites, fluorides, ammonium nitrate, hydrocyanic acid, alkalies
Alcohol	Acetaldehyde, chromic acid, nitric acid, perchloric acid
Alkalies (caustics), concentrated	Aluminum (finely divided), acetaldehyde, cyanides, hydrocyanic acid, zinc (finely divided), acids
Alkali and alkaline earth metals such as: barium, calcium, cesium, lithium, magnesium, potassium, sodium	Water, acids, carbon dioxide, carbon tetrachloride, chlorinated hydrocarbons, flammables, combustibles, oxidizers
Ammonium hydroxide (aqueous) ammonia (see also Ammonia)	Acetic acid
Ammonium nitrate (see also Oxidizers)	Acids, chlorates, finely divided combustibles, flammable liquids, metal powders, sulfur

Do No Contact These	With These
Arsenic compounds	Acids
Barium (see Alkali and Alkaline earths)	
Barium nitrate	Combustibles, organics
Calcium (see Alkali and Alkaline earths)	
Carbon, activated (see also Combustibles)	Calcium hypochlorite, nitric acid
Caustic solutions (see also Alkali and Alkaline earths)	Metals (finely divided)
Cesium (see Alkali and Alkaline earths)	
Chlorides	Acids
Chromates (see Oxidizers)	
Chromic acid (see also Acids)	Acetic acid, alcohol, acetone, flammable liquids, glycerine, naphthalene, turpentine
Combustibles	Alkali and alkaline earth metals, oxidizers, dicumene chromium, hydrazine, bromine
Copper	Acetylene, hydrogen peroxide
Cyanides	Acids, alkalies, carbon dioxide (air)
Dichromates (see Oxidizers)	
Ferricyanides (see Cyanide)	
Ferrocyanides (see Cyanide)	
Flammables	Alkali and alkaline earth metals ammonium nitrate, chromic acid, nitric acid and other oxidizers

Do No Contact These	With These
Fluorides	Acids
Hydrazine	Hydrogen peroxide, nitric acid, asbestos, cloth, wood, metallic oxides
Hydrides	Water, acids
Hydrocarbons	Bromine, chlorine, chromic acid, fluorine, hydrogen peroxide, sodium peroxide
Hydrochloric acid (see also Acids)	Oxidizers, formaldehyde
Hydrofluoric acid (see also Acids)	Ammonia
Hydrogen	Halogens, mercury, oxidizers
Hydrogen peroxide (see also Peroxides)	Acetone, aniline, flammable liquids, combustibles, copper, hydrazine metals (finely divided)
Hydrogen sulfide	Acetaldehyde, nitric acid
Iodine (see also Halogens)	Acetaldehyde, acetylene, ammonia, hydrogen
Lead nitrate (see Nitrates)	
Lithium (see Alkali and Alkaline earths)	
Magnesium (see Alkali and Alkaline earths)	
Magnesium nitrate (see Nitrates)	
Mercury	Acetylene, ammonia, fulminic acid, hydrogen, oxalic acid
Metals (finely divided)	Halogens, oxiders, picric acid

Do No Contact These	With These
Methyl methacrylate	Oxidizers
Nitrates (see also Oxidizers)	Combustibles, organics
Nitric acid (see also Acids)	Acetic acid, ammonia, aniline, chromic acid, carbon, flammable liquids, hydrocyanic acid, hydrogen sulfide, glycerine, hydrazine, metals (finely divided)
Oxalic acid (see also Acids)	Mercury, silver, and other heavy metals
Oxidizers	Acetaldehyde, alkaline and alkaline earth metals, beryllium, dicumene chromium, flammables, hydrochloric acid, metals (finely divided), combustibles
Oxygen	Flammable or combustible substances
Perchloric acid (see also Acids)	Acetic acid, acetic anhydride, aniline, alcohol, organics
Permanganates (see also Oxidizers)	Acetic acid
Peroxides (see also Oxidizers)	Acetic acid
Potassium (see Alkali and Alkaline earths)	Store under kerosene
Potassium chlorate (see Chlorates)	
Potassium nitrate (see also Nitrates)	Trichloroethylene
Potassium permanganate	(see Permanganates)

Do No Contact These	With These
Silver	Acetylene, ammonium compounds, fulminic acid, hydrogen peroxide, oxalic acid
Silver nitrate (see Nitrates)	
Sodium (see Alkali and Alkaline earths)	Store under kerosene
Sodium hydroxide (see also Alkali and Alkaline earths)	Acetic acid, trichloroethylene
Sodium nitrate (see also Nitrates)	Ammonium compounds
Sodium peroxide (see also Oxidizers)	Acetic anhydride, aniline, acetic acid, benzaldehyde, carbon disulfide, ethyl acetate, ethylene glycol, glycerine, methyl acetate, organics, combustibles
Sulfuric acid (see also Acids)	Chlorates, fulmanates, nitrates, perchlorates, permanganates, picrates, combustibles
Zinc (powder or dust)	Acids, chlorinated hydrocarbons, strong alkali hydroxides, carbon disulfide

5.1.7 Mercury should be handled in a fume hood. Spilled mercury shall be cleaned up immediately, using a commercially available mercury clean-up kit, with careful attention being given to removing it from cracks and out-of-the-way places. Mercury should not be left exposed to the room atmosphere.

5.2 Electrical

5.2.1 Electrical cords with worn or cracked insulation shall not be used.

5.2.2 Electrical cords shall be protected from chemical and physical damage.

5.2.3 All electrical equipment shall be grounded.

5.2.4 Corroded electrical equipment shall be inspected for hazardous conditions and should be repaired or replaced.

5.2.5 Electrical equipment with loose electrical connections shall not be used until repaired.

5.3 Fire

5.3.1 Fuel loadings of flammable materials in laboratories shall be minimized.

5.3.2 All flammable liquids shall be stored in appropriate U.L. or FM approved cabinets. Limitations on quantities are as follows:

A. The classifications of flammable and combustible liquids, identified in Table 3-1, shall apply to this section.

B. Containers for flammable and combustible liquids shall conform to Table 3-2, except as provided in Item c below.

C. Class IA and Class IB flammable liquids may be stored in glass containers of not more than 1-gallon capacity if the required liquid purity (such as ACS analytical reagent grade or higher) would be affected by storage in a metal container or if ;the liquid would cause excessive corrosion of a metal container.

D. Not more than 120 gallons of Class I, Class II, and Class IIIA liquids may be stored in a storage cabinet. Of this total, not more than 60 gallons may be of Class I and Class II liquid (source: NFPA 30, 14-3.1).

E. Class I and Class II liquids shall be kept in covered containers when not actually in use.

F. Class I liquids may be used only where there are no open flames or other sources of ignition within the possible path of vapor travel.

TABLE 3-1 (12.4): CLASSIFICATION OF FLAMMABLE AND COMBUSTIBLE LIQUIDS

Class of Liquids		Properties: Flash Point	Properties: Boiling Point	Properties: Common Examples
Flammable Liquids	Class IA	73F (22.8C)	100F (37.8C)	Ethyl ether
	Class IB	73F (22.8C)	100F (37.8C)	Acetone, alcohols, and gasoline
	Class IC	73F (22.8C)	100F (37.8C)	Isoamyl acetate (banana oil)
Combustible Liquids	Class II	100F (37.8C)	140F (60C)	Glacial acetic acid and kerosene
	Class IIIA	140F (60C)	200F (93.4C)	Cresote Oil
	Class IIIB	200F (93.4C)	—	Mineral Oil

TABLE 3-2 (12.4) MAXIMUM ALLOWABLE SIZE OF CONTAINERS FOR FLAMMABLE AND COMBUSTIBLE LIQUIDS++

	Flammable Liquids		Combustible Liquids		
Container Type	Class IA	Class IB	Class IC	Class II	Class III
Glass	1 pint	1 gallon	1 gallon	1 gallon	5 gallons
Metal (other than DOT drums) or approved plastic*	1 gallon	5 gallons	5 gallons	5 gallons	5 gallons
Safety cans+	2 gallons	5 gallons	5 gallons	5 gallons	5 gallons
Metal drum (DOT Spec.)	60 gallons	60 gallons	60 gallons	60 gallons	60 gallons

*Containers shall be provided with lids, caps, or other closures to prevent the escape of either liquid or vapor.

+Safety cans containing flammable liquids (Class IA, IB, or IC) shall be painted red and identified with a black and yellow band around the can.

++Source of sizes is Table 4-1, NFPA 30.

5.3.3 Cylinders of flammable gases and oxygen shall be stored according to the following specifications:

Compressed Gas Cylinders

Compressed gas cylinders can be extremely hazardous if mishandled. The following guidelines have been prepared to ensure that safe practices are followed in the handling, storage, and use of cylinders containing compressed gases.

A. Cylinders shall always be considered to be full unless labeled as empty, and shall be handled and used with corresponding caution.

B. Cylinders (regardless of size whether in use, in storage, or in transit shall be fastened securely by a chain or a rigid retaining bar or structure to prevent cylinders from falling or being knocked over.

C. Protective valve caps shall be in place on all cylinders in storage or transit, except for cylinders that are not supplied with caps. The cap shall be kept on the cylinder until the gas is ready for use and shall be replaced when the cylinder is empty. A broken valve can turn a compressed gas cylinder into a rocket.

D. A regulator, a gauge, or a regulating manifold shall be used on cylinders. Regulators, gauges, and manifolds are to be matched to the specific type of gas and the service for which the cylinders are being used. Adapters to connect cylinders of one type of gas to piping, manifolds, gages or valves intended for other types of gases are not to be used. Do not use lubricants on valves or regulators or modify them in any way.

E. If a cylinder leaks and the leak cannot be remedied by tightening a valve gland or packing nut, close the valve and attach an "UNSAFE" Tag. Move the leaking cylinder out of doors to a well ventilated location, secure it and notify Safety. If the gas is flammable or toxic, rope the area off, and post appropriate signs.

F. Cylinder contents shall be identified by means of a legible label or stencil or by identifying markings embossed on the cylinder by the supplier. Embossing by WVNS is not permitted.

G. Cylinders should not be subjected to a temperature above 125F. A flame or arc should never be permitted to come in contact with any part of a compressed gas cylinder.

H. Compressed gas cylinders should not be dropped, bumped, or handled roughly. Cylinders are never to be used as rollers to move heavy equipment or material. Caution shall be exercised at all times to protect cylinders from sources that could cut or damage the metal surface.

I. Cylinder valves should be closed when not in use. This point is especially important at the end of a day's work or on "empty" cylinders.

J. It is WVNS practice to store gas cylinders in racks and to provide weather protection for the cylinders and racks. Manifolded cylinders stored in outdoors locations shall be protected from the weather by lean-to roofs, manifold cabinets, or other means. Compressed Gas Association pamphlet P-1 states, "Cylinders may be stored in the open, but should be protected from the ground beneath to prevent rusting." Cylinders shall not be stored at temperatures above 125F, and supplier's recommendation for shading shall be observed.

K. Cylinder valves not provided with fixed hand wheels shall have keys or handles on valve stems while cylinders are in service to permit immediate emergency shutdown.

L. Storage areas and manifold installations for flammable gas cylinders shall have conspicuously posted signs warning against smoking, open flames, or open lights.

M. Empty cylinders should be tagged with a completed (signature and date) "Empty Cylinder" tag, be segregated from full cylinders, and returned to the warehouse as soon as possible.

N. Cylinders that are permitted inside buildings shall be stored in a safe location that is at least 20 feet from highly combustible materials, and is not to be exposed to excessive rise in temperature or physical damage.

O. Oxygen cylinders in storage shall be separated from fuel gas cylinders or combustible materials by a minimum distance of 20 feet or by a noncombustible barrier at least 5 feet high having a fire-resistant rating of at least 1/2 hour, as defined by the NFPA Codes.

P. Oxy-acetylene rigs for burning, cutting, or welding (comprised of one fuel gas cylinder and one oxygen cylinder) are exempt from the requirements of Item O. However, oxy-acetylene rigs are required to be separated from each other by a 20-foot minimum distance.

Q. Hydrogen cylinders may not be taken inside of buildings unless authorization is obtained from the Safety Department.

R. For transporting and unloading gas cylinders, use a suitable hand truck, fork truck, roll platform, or similar device with the cylinder firmly secured.

S. Personnel handling and using poisonous gases shall do so only with a properly authorized IWP.

T. WVNS shall not accept vendor supplied gas cylinders which are dented, have valve covers missing, or have rust over one quarter of the bottle surface.

U. Hydrogen cylinder valves shall not be cracked to blow out dirt since hydrogen can self-ignite.

V. Acetylene cylinders shall be stored and used upright at all times. In addition, acetylene shall not be used at pressures greater than 15 pounds per square inch gauge (psig).

W. Because oxygen under pressure may react violently with oil or grease, every possible precaution shall be taken to prevent oxygen from coming in contact with oil or grease. Oxygen cylinders, valves, regulators, hose, and other apparatus shall be kept free from oil or grease and shall not be handled with oily hands, oily gloves, or with greasy equipment.

X. Requirements for protective devices such as flash arresters and grounding wires when installing flammable gases shall be followed.

5.3.5 When using volatile liquids that are flammable and flammable gases, ignition sources such as open flames and high temperature furnaces shall not be operated in the vicinity unless they are a part of a planned and approved laboratory setup.

5.3.6 Emergency phone number for the site shall be posted at all laboratory phones.

5.4 Pressure

5.4.1 Installed gas cylinders shall be chained or otherwise secured against falling by noncombustible means.

5.4.2 Gas cylinders shall not be transported without the cylinder cap being in place.

5.4.3 Installed gas cylinders shall not be exposed to heat sources.

5.4.4 The proper valve, regulator, and gas lines shall be used for each type of gas.

5.4.5 The content of each gas cylinder shall be clearly indicated.

5.4.6 Grease and oil shall be avoided when using gas valves and regulators, especially with oxygen.

5.5 Housekeeping

5.5.1 Exits and aisles shall not be blocked.

5.5.2 Waste receptacles shall be labeled as to the types of materials that can be placed into them.

5.5.3 Fume hoods shall have gravity sash stop latches installed after proper balancing of hood flow is assured. The latches will allow the sashes to be raised to a predetermined height, thereby maintaining adequate hood flow in all fume hoods.

5.5.4 Broken glass shall be discarded only in approved waste receptacles. Chipped or cracked glassware shall not be used; it shall be discarded.

5.5.5 Gross accumulation of waste in laboratories is prohibited.

5.5.6 Broken equipment shall be discarded, repaired, or otherwise removed from the laboratories.

5.5.7 Clutter on bench tops and in fume hoods is prohibited.

5.5.8 Nonradioactive water or other nonradioactive slippery material spilled on the floor in a nonradioactive area shall be cleaned up immediately.

5.6 Protective Equipment

5.6.1 Required eye protection and protective clothing shall be posted in each laboratory. That protection shall be worn when required.

5.6.2 Rubber gloves, a rubber apron, chemical splash goggles, a face shield, or equivalent protection shall be worn when handling fuming nitric acid.

5.6.3 Fume hoods shall be used when handling volatile chemicals that are toxic and when heating or boiling solutions that produce toxic fumes.

5.6.4 Safety showers, eye washers, and other emergency equipment in and near laboratories shall not be blocked; they shall remain clear for quick and easy access.

5.7 General

5.7.1 Food or drink shall not be consumed or taken into laboratories.

5.7.2 Smoking is not permitted in laboratories.

5.7.3 Warning signs shall be heeded and instructions regarding locks and tags shall be followed.

5.7.4 Pipetting by mouth is strictly prohibited.

5.7.5 Heavy objects shall not be stored above five feet.

> 5.7.6 ACRODISK filters potentially break upon pressure. The syringe end filters in use for Analytical and Process Chemistry (A&PC) should only be used in the suction mode to prevent rupture of the disk. Any deviation of the above practice must be approved by the Manager, A&PC. Additional safety precautions will be discussed in accordance with each specific deviation.

5.8 Injury

5.8.1 Injuries shall be reported to supervision and first aid in accordance with company policy.

5.8.2 All skin injuries and abrasions must be covered prior to working in the laboratory.

5.8.3 Emergency phone numbers for aid for serious injuries shall be posted at each laboratory phone. The 812 All Page should be used for emergencies such as fire or serious injury.

5.8.4 Inhalation or ingestion of toxic materials or the possibility of such happening shall be reported to supervision.

> 5.9 Safety Checklist

> 5.9.1 On a periodic basis the A&PC Safety Inspector will follow the checklist (Attachment A) to assure compliance.

> 5.9.2 The completed checklist must be filed in a log.

> 5.9.3 If any noncompliance is found, the A&PC Safety Inspector must immediately notify the Quality Assurance Training Coordinator and the Laboratory Supervisor to assure corrective action.

>6.0 CHEMICAL INVENTORY

> 6.1 Each chemical container will be given unique stock identification letters and numbers.

> 6.2 Each chemical container will be identified by storage location.

> 6.3 The complete container information will be found on IBM PC.

> 6.4 Each container will have expiration date, date opened, date received, and lot number.

> 6.5 The chemical inventory will have automatic reorder capability.

> 6.6 The chemical inventory program will be user friendly.

7.0 REFERENCES

7.1 ACP 4.2, Preparation, Approval and Distribution of ACMs.

7.2 ACP 7.4, Handling Radioactive Materials.

7.3 WV-011, Industrial Hygiene and Safety Manual.

7.4 DOE-1D, Appendix 0550 Subpart IIIJ, Chemical Hazards.

SAFETY CHECKLIST

LEGEND

OK - Everything is in good condition LOW - Sources depleted
N/A - This item does not apply NONEXISTENT - Nonexistent or bad

	ICP & CTS Lab Lab	Vit Lab	Cells Hallway	Cold Lab	Hot Lab	MS Lab
Safety Showers						
Eye Washes						
Fire Extinguishers						
Fire Blankets						
First Aid Kits						
Gas Cylinders						
Mercury Spill Kits						
Chemical Spill Kits (i.e., Spill Pillow, HF,Acid,Solvent,Base)						
Rubber Aprons						
Gloves						
Face Shields						
Bottle Carriers						
Hood Flows						
Everyone Wearing Safety Glasses						
Safety Shoes						
House Keeping						
Hood Stops In Place						

Note: If either choice is low or nonexistent, a plan of action must be addressed in the comment section.

Comments:_____

_____

_____

_____

_____

A & PC Safety Inspector_____ Date:_____
A & PC Safety Assistant_____ Date:_____

CONTROL OF SAMPLES

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> To provide instructions for the control of samples as they are received by Analytical and Process Chemistry (A&PC) and as they are processed in the laboratories. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

This procedure applies from the time samples are received by A&PC until their final disposition. This includes samples received by automatic transfer and those received by delivery to the laboratories.

3.0 DEFINITIONS

3.1 Submitter: The person or organization transferring samples to A&PC.

4.0 RESPONSIBILITIES

4.1 The A&PC staff receiving samples are responsible for assuring that the acceptance criteria for samples as prescribed in this ACP are met before accepting the samples.

4.2 The A&PC staff using and handling samples are responsible for assuring that samples are handled according to the provisions of this ACP.

4.3 The manager of Analytical and Process Chemistry (A&PC) is responsible for approving exceptions to the provisions of this ACP.

4.4 The responsibility for developing sampling plans and for taking samples rests with the submitters. This includes designating the analyses required for each sample submitted. Consultation with A&PC personnel is desirable to help assure that samples are properly taken, that results sought can be achieved with the instrumentation available, and that the desired precision and accuracy can be attained.

5.0 PROCEDURE

The proper control of samples is important in assuring the reliability of the measurement data produced from the analysis of samples. The provisions of this ACP shall be followed in the receipt, use, handling, and disposition of samples.

5.1 Receipt of Samples

5.1.1 No sample shall be received by A&PC without a properly filled out Analysis Request form, unless a sample is from a series that has been preestablished with A&PC as a routine submission. Required sample information for such a series shall have been submitted to A&PC prior to submitting the first samples. See Reference 6.2.

5.1.2 Upon receipt, an inspection shall be made of each sample for any unusual or unexpected characteristics. If such is observed, the sample submitter shall be contacted to determine if the sample is valid.

5.1.3 Each sample received shall be clearly identified in a manner to prevent loss of identification while in custody of A&PC. An exception to this requirement is in the receipt of samples by automated sample transfer. In these cases, however, communications between the submitter and A&PC shall clearly establish the identity of each sample when transmitted.

5.1.4 Samples shall not be accepted nor work started if they are not properly identified, required sample information is not provided, or there is a question about sample validity.

> 5.1.5 Samples labeled by A&PC for sample identification shall be assigned unique laboratory sample identification numbers from the Log Book (See Reference 6.2). These numbers are generated by the REALM computer program. Labeling shall be done clearly and in a manner to prevent loss of identification.

> 5.1.6 Each sample received shall be logged into a Log Book and on the REALM computer program. See Reference 6.2.

5.2 Sample Handling

5.2.1 Samples shall be handled in a manner to prevent contamination by impurities and changes in concentration from evaporation or dilution.

5.2.2 Sample containers shall be opened only as necessary and shall not be left open longer than necessary.

5.2.3 Samples whose integrity is questionable shall not be used and they shall be controlled to prevent their inadvertent use prior to disposition.

5.2.4 Subsamples taken from a primary sample shall be immediately labeled for sample identification. Subsamples shall be uniquely identified in a way that is traceable to the primary sample.

5.3 Storage of Samples

5.3.1 Specific storage areas shall be specified for samples. See 5.5.2 of Reference 6.3.

5.3.2 Samples shall be resturned to their assigned storage areas when not in use.

5.3.3 Storage areas shall be maintained in a manner to preserve the integrity of the samples and the identification labeling on the outside of the sample containers.

5.4 Disposition of Samples

5.4.1 The laboratory will not be responsible for retaining samples for more than two months after all analyses have been completed, unless otherwise requested by the submitter and approved by the manager of A&PC. Radioactive samples shall be retained for a maximum of one month (See 5.5.4 of Reference 6.3).

5.4.2 Storage for archived samples is the responsibility of the submitter.

5.4.3 Disposition of samples shall be documented, and the date of disposition shall be recorded.

5.4.4 Samples discarded to waste shall be properly discarded according to regulations governing the type of materials involved. For example, see Reference 6.4.

6.0 REFERENCES

6.1 PRD 7.0, Control of Equipment and Materials

6.2 ACP 5.1, Laboratory Record System

6.3 ACP 7.4, Handling Radioactive Materials

6.4 Radiological Controls Manual, WVDP-010, West Valley Demonstration Project

UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC.

ADMINISTRATIVE CONTROL PROCEDURE
ANALYTICAL AND PROCESS CHEMISTRY

ACP 7.4 Rev. 0
Effective Date: 06/15/87

HANDLING RADIOACTIVE MATERIALS

Approved By:

B. R. Stimmel, Manager
Analytical and Process Chemistry

Concur:

, Manager
Quality Engineering

1.0 PURPOSE

To provide established guidelines for handling radioactive materials.

2.0 APPLICATION

This procedure applies to the handling of radioactive materials in the laboratories of Analytical and Process Chemistry.

3.0 DEFINITIONS

3.1 R&S: Radiation and Safety, which is the WVNS organization responsible for radiation protection and monitoring.

3.2 A&PC: Analytical and Process Chemistry.

3.3 ACM: Analytical Chemistry Method.

4.0 RESPONSIBILITIES

4.1 Personnel in Analytical and Process Chemistry (A&PC) are responsible for following this procedure when receiving and handling radioactive materials.

4.2 Personnel delivering radioactive materials to A&PC are responsible for assuring that those materials meet the requirements of this ACP, and the Radiological Controls Manual, WVDP-010.

5.0 PROCEDURE

5.1 Receipt of Routine Water Samples

5.1.1 Routine water samples shall be received by A&PC only during normal work hours of A&PC. Routine water samples requiring Gross Alpha and Gross Beta analysis during off-hours are analyzed by R&S.

5.1.2 Routine water samples shall be contained in a primary bottle and bagged in a clean, yellow plastic bag and placed in the sample storage cabinet in the Analytical Aisle if 5.1.1 requirements have been met first. All routine water samples shall be doubly contained as described in 5.1.2.

5.1.3 Primary containers shall not be leaking. Should a primary container be leaking or begin to leak after receipt, the sample shall be disposed of immediately. No further work shall be done on that sample.

5.1.4 Each sample shall be accompanied by a completed Analytical Request Form, which shall be placed in the holder on the outside of the sample cabinet. The activity section of the form is not applicable for routine water samples. See Reference 6.1.

5.1.5 Samples shall be clearly identified by an appropriate label. See Reference 6.2.

5.2 Receipt of Other Radioactive Samples

5.2.1 All radioactive samples (other than routine water samples) shall not be received by A&PC unless they are tagged with a Radioactive Materials Tag by R&S.

5.2.2 The dose rate of each sample submitted shall not exceed 10 mR/hr at two inches (window closed), unless prior arrangements are made with the Manager of A&PC. Any sample suspected of containing more than 0.1 µCi of an alpha emitter shall be so identified (see 5.2.4).

5.2.3 All samples received shall be doubly contained as described in the Radiological Control Manual WVDP-010, in a manner to prevent material leakage should a package be dropped.

5.2.4 Each sample shall be accompanied by a completed Analytical Request Form, unless prior arrangements are made with the Manager of A&PC. For nonroutine samples, a precise

description of the sample (e.g., type of material, physical form, source, precautions required, etc.) shall be included in the comment section of the form. See Reference 6.1.

5.2.5 Samples shall be clearly identified by an appropriate label. See Reference 6.2.

5.2.6 Storage of radioactive samples shall be as prescribed in 5.5.

5.3 Handling Radioactive Samples in the Laboratories

5.3.1 The following protective clothing and equipment shall be used as described when working in controlled portions of the laboratories (posted as Surface Contamination Area.)

A. A disposable lab coat or coveralls, shoe covers, one pair of skindeks, and one pair of anti-c gloves shall be worn.

B. A step-off pad shall be placed at the entrance to controlled areas. Lab coats or coveralls, Anti-C gloves, skindeks, and shoe covers shall be removed before stepping onto the pad when leaving a controlled area.

C. Impermeable Anti-C gloves shall be worn and taped to the sleeves of the lab coat or coveralls. Cotton glove liners are optional and may be worn beneath the impermeable Anti-C gloves.

D. When working inside a fume hood, disposable gloves shall be worn over the Anti-C gloves. The outside gloves shall be changed often enough to minimize the spread of contamination.

E. All observers (visitors) entering a controlled area shall wear as a minimum shoe covers and disposable gloves.

5.3.2 Fume hoods shall be lined with teflon-sealed, impermeable self-adhering paper or equivalent. Stainless steel trays may also be used to minimize contamination of the hood lining.

5.3.3 The amounts of radionuclides used in a fume hood should be limited to the amounts recommended in Section 7.6 of Reference 6.3. For typical WVNS samples, those limits are not exceeded if individual samples do not exceed 10 mR/hr (5.2.2).

5.3.4 The following containment practices shall be used:

A. Generally, radioactive materials shall be doubly contained, except when in a fume hood, glovebox, or glove bag.

B. Materials evaporated, taped, or glued onto counting discs or dishes are not considered singly contained, and they shall be doubly contained, using petri dishes and outer bags, when samples are being transported.

C. Single containment is acceptable for weighing when specified by the ACM (Analytical Chemistry Method) being used or when authorized by a senior technician or chemist. Single containment is used in the counting room while counting prepared samples (double containment is not possible).

5.3.5 Waste materials from fume hoods shall be doubly bagged and shall be disposed of as prescribed by Reference 6.4.

5.4 Handling Radioactive Materials in Counting Room

5.4.1 No radioactive materials may be transported to the Radiochemical Counting Room without the permission of Analytical and Process Chemistry personnel.

5.4.2 Samples in the counting room shall be doubly contained except when being counted (5.3.4 B and C).

5.4.3 Samples prepared for Gamma Scan shall be sealed with plastic electrical tape around lid seal of all primary bottle containers.

5.4.4 Samples shall be removed from the counting room as soon as practicable after being counted. See 5.5.

5.4.5 Calibration and check sources (standards) shall be stored when not in use as specified in 5.6.

5.5 Storage of Radioactive Samples

5.5.1 Samples shall be doubly contained while in storage.

5.5.2 Samples shall be stored in the sample storage cabinets located in the Radiochemical Laboratory.

5.5.3 Disposable gloves shall be worn when removing samples from storage or otherwise handling them in storage.

5.5.4 Samples shall not be stored for more than 30 days after analyses have been completed unless a written notice is received by A&PC from a sample submitter. The notice shall include reason for continued storage and length of time required. The notice shall be approved by the Manager of A&PC.

5.6 Storage of Calibration and Check Sources

5.6.1 Radioactive sources (standards) shall be stored in the standards storage safes or locked storage cabinets located in the laboratories.

5.6.2 Sealed sources shall be included in the R&S sealed source log, and they shall be leak tested according to Reference 6.4.

5.6.3 All radioactive sources (standards) shall be recorded in an A&PC log book for accountability purposes.

5.6.4 Radioactive sources shall be handled as prescribed in 5.3.

6.0 REFERENCES

6.1 ACP 5.1, Laboratory Record System.

6.2 ACP 7.3, Control of Samples.

6.3 Handbook of Laboratory Safety, 2nd Edition, N.V. Steare, Editor, 1971.

6.4 Radiological Controls Manual, WVDP-010, West Valley Demonstration Project.

PREPARATION OF STANDARD SOLUTIONS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

To provide instructions for the control of standard solutions during their preparation and use.

2.0 APPLICATION

This procedure is applied for the standards used in the calibration of laboratory instruments. Specific instructions for the preparation of individual standard solutions are given in ACMs or a laboratory notebook.

3.0 DEFINITIONS

3.1 ACM: Analytical and Chemistry Method.

3.2 Shelf Life: The time during which a standard solution can normally be used before it may deteriorate.

3.3 Stock Solution: A highly concentrated standard solution from which more dilute working standards are prepared. Stock solutions are more stable and have longer shelf lives than working standards.

3.4 Working Standard: A standard used for calibration, usually prepared from a stock solution. Since working standards have short shelf lives, they are often prepared in small quantities each day that they are required. When a working standard is to be used over a period of time, a significantly larger volume than needed is prepared to maximize shelf life.

3.5 Well Characterized Materials: Materials used to prepare standard solutions. They may be certified, high purity reagents or reference materials purchased from the National Bureau of Standards (NBS) or from a commercial source. Purchased reference materials should be certified.

4.0 RESPONSIBILITIES

> 4.1 The senior technician, A technician or B technician who prepares standard solutions or under whose supervision they are prepared is responsible for assuring that only well characterized materials are used and that those materials are certified.

> 4.2 Senior technicians, A technician and B technicians are responsible for assuring that standard solutions are properly labeled according to the provisions of this ACP.

4.3 The manager of Analytical and Process Chemistry (A&PC) is responsible for approving exceptions to the provisions of this ACP. See 5.3.3.

5.0 PROCEDURE

The proper preparation and control of standard solutions is important in assuring the reliability of the measurement data produced from the use of these solutions in calibration. The provisions of this ACP shall be followed in the preparation and control of these solutions.

5.1 Stock Solutions

5.1.1 Stock solutions are prepared with concentration levels and matrices that are stable. They shall be prepared from well characterized materials. Material certificates shall be retained as records.

5.1.2 Each stock solution shall be prepared using an approved set of instructions, contained either in an associated ACM or in a laboratory notebook.

5.1.3 During preparation, materials shall be weighed using calibrated balances, and solutions shall be made to volume using calibrated or certified volumetric flasks. See Reference 6.2.

5.1.4 Stock solutions shall be stored in containers and under conditions that preserve their integrity. See Reference 6.2.

5.1.5 Solutions shall be clearly labeled in such a way that labeling will not be lost through normal use and handling of the containers. As a minimum, labeling shall include name of the solution, concentrations of the analytes, matrix, date prepared, expiration date, and initials of technician making up the solution.

> 5.1.6 The maximum expiration date is normally one year after preparation. No stock solution should have an expiration date past the expiration date of any of its constituents.

5.1.7 A record shall be made for each preparation in a laboratory notebook designated for standard solutions. The following information is recorded:

- o Identification of stock solution
- o Procedure used for preparation (referencing is acceptable)
- o Sources of materials used (manufacturer and lot number)
- o Weights and volumes used
- o Calculations
- o Concentrations of analytes
- o Expiration date
- o Preparer, and
- o Date of preparation

5.1.8 Purchased stock solutions should be obtained with certificates of analysis. These may be incorporated into the labels of the container. Provisions of 5.1.4 and 5.1.5 are followed as appropriate. The maximum expiration date for purchased solutions is normally one year after receipt.

5.2 Working Standards

5.2.1 Instructions for preparing working standards shall be contained in associated ACMs.

5.2.2 Working standards are prepared by diluting one or more stock solutions. Dilutions shall be made using calibrated or certified pipets and volumetric flasks. See Reference 6.2.

> 5.2.3 Frequency of preparation for a specific standard shall be specified in the associated ACM. Since the shelf life of working standards is normally short, expiration dates shall be established for those that will be used more than once. No standard should have an expiration date past the expiration dat of any of its constituents.

5.2.4 The provisions of 5.1.4 and 5.1.5 shall be followed when working standards are retained for use more than once.

5.2.5 A record shall be made of the preparation in the notebook or work sheet used to record the analysis data associated with the standard. The information recorded shall be that listed in 5.1.7, as appropriate.

5.3 Control of Standards

5.3.1 Before being used, a newly prepared stock solution shall be checked against another standard and this verification step shall be documented. Verification is usually done by preparing a series of working standards that are checked against a control standard or against another series of working standards prepared from an already existing stock solution.

5.3.2 The verification working standards is a function of the quality control (method control) placed on the methods for which the standards are used. See Reference 6.1.

5.3.3 No standard shall be used after its expiration date without approval. An exception for a stock solution can be made if the solution is checked against another standard and approved by the manager of A&PC.

5.3.4 Standard solutions shall be thoroughly mixed prior to use, particularly if moisture has condensed around the upper part of a container.

5.3.5 Any stock solution having dried salts around the top of the container shall be discarded or checked against another standard after the salts have been removed.

6.0 PROCEDURE

6.1 PRD 8.0, Control of Measurements

6.2 ACP 7.1, Control of Equipment and Reagents

6.3 ACP 8.2, Statistical Practices

STATISTICAL PRACTICES

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> To provide instructions for the use of statistical practices by the staff of Analytical and Process Chemistry (A&PC). Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

The statistical practices given in this procedure are used primarily in measurement control, in the recording of data, and in calculating and reporting results.

3.0 DEFINITIONS

3.1 ACM: Analytical Chemistry Method.

3.2 ASTM: American Society for Testing and Materials; 1916 Race St., Philadelphia, PA.

4.0 RESPONSIBILITIES

4.1 The staff of A&PC is responsible for following the provisions of this procedure in their work as appropriate.

4.2 The manager of Analytical and Process Chemistry (A&PC) is responsible for approving specified actions in this ACP.

5.0 PROCEDURE

The use of prescribed statistical practices is important to assure the consistent application of uniform practices across A&PC.

5.1 Rounding Off Numbers

When rounding off numbers, the following practice shall be used. See Reference 6.2.

5.1.1 When the next figure beyond the last one to be retained is less than 5, retain the last figure unchanged.

5.1.2 When the next figure beyond the last one to be retained is greater than 5, increase the last figure by 1.

5.1.3 When the next figure to the last one to be retained is 5 and there are no figures or only zeros beyond this 5, increase the last figure by 1 if it is odd. Leave the last figure unchanged if it is even. If there are figures beyond the 5, increase the last figure by 1.

5.2 Outlying Observations

When evaluating a series of results and one (or more) value appears to deviate significantly from the other values, a decision must be made about how to treat the outlier(s). The outlier can be discarded if it can be substantiated that the outlier resulted from a specific error. If an error cannot be substantiated, the outlier must be included in the series of results. However, an outlier still can be discarded if a statistical evaluation shows that the outlier significantly deviates from the other values. See Refereence 6.3 for instructions on how to test an outlier.

5.2.1 All actions taken to discard an outlier shall be recorded in the laboratory record system. See Reference 6.4.

5.2.2 The decision to discard an outlier shall be approved by the manager or chemist of A&PC.

5.3 Tolerances

5.3.1 When necessary a tolerance limit can be stated with a measurement value given in an ACM; for example, 15+/-0.1 mL.

5.4 Calibration Curves

5.4.1 Instructions for the preparation of calibration curves shall be contained in the appropriate ACMs. Normally, a curve shall bracket the concentration range expected for the analyte. See Reference 6.5.

5.4.2 Calibration curves shall be labeled with the applicable ACM, date prepared, analyst making preparation, analyte, and amounts of analyte taken with the corresponding instrument readings.

5.4.3 The procedure shall state the frequency that the curve is checked and when the curve is redone. A new curve shall be prepared whenever a new batch of a reagent is introduced.

5.4.4 When a curve is checked, at least one point on the curve shall be checked, preferably at the middle of the curve. Limits for the check values and actions to take if the limits are exceeded shall be stated in the ACM.

5.5 Control Charts

5.5.1 Obtain a group of repeated results from the analysis of the control standard for which a control chart (Attachment A) is to be prepared. Unless otherwise stated in the associated ACM, a group of at least five results should be obtained over a period of at least three working days.

5.5.2 Calculate the mean ($\bar{x}$) for the group.

5.5.3 Calculate the standard deviation (s) using the following equation:

$$s = \frac{\Sigma(x-\bar{x})^2}{n-1}$$

5.5.4 Calculate the upper control limit (UCL) and the lower control limit (LCL) as follows:

$$UCL = \bar{x} + 3s$$
$$LCL = \bar{x} - 3s$$

5.5.5 Prepare a control chart from the calculated mean and control limits.

5.5.6 Plot each result from the control standard in the order obtained.

> 5.5.7 When a new control chart is prepared, retain the old one for the life of the project. File control charts with MRC.

5.6 Method Out-of-Control

5.6.1 The following criteria are established when a method is out-of-control:

a) If a point falls outside of a control limit, immediately run another standard. If the second result falls outside, the method is out-of-control.

b) If the second result falls within the control limits, run a third standard. The third result determines whether the method is in- or out-of-control.

5.6.2 When a method goes out-of-control, the problem shall be investigated. Steps taken to bring the method back into control shall be documented in the laboratory record system (see Reference 6.4). Out of control events may require documenting on a Request for Corrective Action (RCA) (see Reference 6.7).

5.6.3 Samples shall not be analyzed using a method that is out-of-control.

5.6.4 If consecutive standard results plotted on the chart begin to drift toward a control limit, the method may be going out-of-control and the situation should be investigated.

6.0 REFERENCES

6.1 PRD 8.0, Control of Measurements.

6.2 ASTM E 29, Designating Significant Places in Specified Limiting Values.

6.3 ASTM E 178, Dealing with Outlying Observations

6.4 ACP 5.1, Laboratory Record System.

6.5 ACP 4.2, Preparation, Approval, and Distribution of ACMs.

6.6 C. A. Bennett and N. L. Franklin, Statistical Analysis in Chemistry and the Chemical Industry, p. 22, John Wiley & Sons, N.Y., 1954.

6.7 PRD 9.0, Deficiencies and Corrective Actions.

QUALITY CONTROL CHART

:THOD _____ STANDARD NAME _____ TRUE MEAN _____

ME PERIOD _____ SPECIFIC LIMIT _____ UNITS _____

DATE	VALUE	-3σ	-2σ	x	$+2\sigma$	$+3\sigma$	TECHNICIAN	REVIEW	COMMENTS

LIMITS _____ _____ _____ _____ _____

NEW MEAN _____ 1σ _____ TECH _____

ATTACHMENT A
ALP 8.4, Rev. 1
Effective Date: 07/24/89

METHOD VALIDATION

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> To provide instructions for validating technical methods to assure that they are correctly set up and applied. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

This procedure applies to new methods developed and set up after the effective date of this procedure. Methods in active use before the effective date do not require validation by this procedure, but their applicability is documented using this procedure.

3.0 DEFINITIONS

Not applicable to this procedure.

4.0 RESPONSIBILITIES

4.1 Validation is the responsibility of the chemist developing and setting up a method. The documentation of applicability of methods in active use prior to the effective date of this procedure is the responsibility of the chemists responsible for maintaining the methods.

4.2 The manager concurs with each validation.

5.0 PROCEDURE

5.1 Validation

5.1.1 During development and set up, the chemist responsible investigates the capability of a method and determines its applicability for its intended use.

5.1.2 As a minimum, validation involves investigating and obtaining information on the following parameters of a method.

A. Technical Basis. The method must be based on sound technology, involving the use of proven laboratory and instrumental techniques in ways recognized and accepted by the community of users.

B. Interferences. The method must not be affected adversely by components in the matrix of the material to be analyzed. Interferences, including potential environmental conditions, must be identified and provisions must be developed to eliminate their effect.

C. Range. The method must be capable of responding adequately across the range of concentration levels that will be encountered for the constituent to be determined in the samples. The lowest concentration level that can be determined reliably must be established for methods used to determine impurities in samples.

D. Reliability. The method must be capable of producing data under the expected conditions of use that will meet the bias and precision requirements established for the required analysis.

5.1.3 Validation is established by documenting data and information obtained relating to the four parameters given in 5.1.2.

5.1.4 Documentation is done in a laboratory notebook or in a brief technical report. The entries in the notebook or report are signed and dated by the responsible chemist. The manager also signs and dates the record to signify concurrence with validation.

5.1.5 The record of validation is made traceable to the method and is retained for the life of the method.

5.2 Documentation of Applicability

5.2.1 The applicability of each method in active use prior to the effective date of this ACP is documented using Attachment A.

5.2.2 This record of applicability is retained for the life of the method.

6.0 REFERENCES

6.1 PRD 2.0, Quality Assurance Program.

6.2 PRD 6.0, Control of Records.

6.3 PRD 8.0, Control of Measurements.

6.4 ASTM·C 1068-86, Standard Guide for Qualification of Measurement Methods by a Laboratory within the Nuclear Industry.

ATTACHMENT A

Applicability of Method

Method Identification Code: _____

Method Title: _____

a. Purpose of Method:

b. Length of Time in Use:

c. Performance Experience (Bias and Precision):

Verified:

_____ Date: _____
Responsible Chemist

Concurs:

_____ Date: _____
Manager

INTERNAL REVIEWS

UNCONTROLLED

Approved By:

C. W. McVay, Manager
Analytical and Process Chemistry

Concur:

R. P. Christensen, Manager
Quality Engineering

1.0 PURPOSE

> To provide instructions for performing internal reviews of Analytical and Process Chemistry, giving assurance that program requirements are being met. Specific clarifications, modifications and/or exceptions to this instruction relative to the Vitrification Laboratory are contained in ACP 2.1.

2.0 APPLICATION

This procedure is used to survey laboratory operations based on the approved Laboratory Quality Assurance Program.

3.0 DEFINITIONS

> 3.1 Finding. A finding is a significant deficiency in operations that is a noncompliance with a program requirement.

> 3.2 Observation. An observation is a recommendation from the reviewer (s) for improving operations. An observation may be a situation that is not a finding, but borders on becoming one.

3.3 Internal Review. A survey performed by member(s) of the organization reviewed, who are intimately familiar with operations of the organization.

> 3.4 Significant Deficiency. Deficiencies that would lead to incorrect measurement data. Examples are: method out-of-control, a trend developing that will probably lead to an out-of-control condition, and an unexpected loss of calibration.

4.0 RESPONSIBILITIES

4.1 The Quality Assurance and Training Coordinator (QATC) is responsible for performing internal reviews. That person is authorized access to all laboratory records and facilities and may interview laboratory personnel as required.

4.2 The manager approves the review reports and review schedules. The manager may participate in a review. The manager assigns responsibilities for taking corrective actions and verifies the completion of corrective actions.

5.0 PROCEDURE

5.1 Review Schedule

5.1.1 The QATC prepares an annual schedule of internal reviews by a memorandum to the manager. The frequency depends on the status of the quality assurance program, but a minimum of two are performed annually. Each program area (PRD) is included at least in one review during the year.

5.1.2 The manager approves the schedule by signing and dating the memorandum. The memorandum is retained for a minimum of one year. A copy is sent to Quality Engineering.

5.1.3 The schedule may be revised during the year. The revision is approved by the manager, attached to the original, and a copy is sent to Quality Engineering.

5.2 Planning a Review

5.2.1 Each review is planned by the QATC by selecting the program areas to be reviewed.

5.2.2 A checklist is prepared for each program area selected for review. The checklists are prepared by reviewing program requirements (PRDs), past review reports, and Requests for Corrective Actions (RCAs).

5.3 Performing the Review

5.3.1 The QATC notifies the manager when the review is started.

5.3.2 Records, equipment, and materials are examined based on the checklists. Deficiencies are recorded on the checklists, including information relevant to deficiencies.

5.3.3 When the review is completed, the QATC prepares a review report using Attachment A. The report is issued to and approved by the manager. A copy is sent to Quality Engineering.

5.4 Corrective Action

5.4.1 After approving the review report, the manager assigns responsibilities for taking corrective action on findings and observations. The manager decides which observations, if any, will have corrective action.

5.4.2 Correction action is initiated using a Request for Corrective Action (RCA) - see PRD 9.0.

5.4.3 When a correction action has been completed, the manager verifies completion and signs the RCA.

5.4.4 Copies of completed RCAs are attached to the audit report.

5.4.5 The original RCA is forwarded to the Quality Assurance Manager for validation and retention.

5.5 Closeout of Reviews

5.5.1 When all RCAs have been completed and completion of corrective actions has been verified, the review is closed out by a memorandum issued and signed by the manager. This memorandum states that all corrective actions have been satisfactorily completed, and the review is closed.

5.5.2 A memorandum is attached to the review report and a copy is sent to Quality Engineering.

5.5.3 The review report, including attachments, is retained for a minimum of three years. The completed checklists may be included with the report.

6.0 REFERENCES

6.1 PRD 2.0, Quality Assurance Program.

6.2 PRD 6.0, Control of Records.

6.3 PRD 9.0, Deficiencies and Corrective Actions.

6.4 QAP 16-1, Request for Corrective Action. (Quality Assurance Department Procedure)

ATTACHMENT A

Internal Review Report

Date of Review: (1) Review No.: (2)

Reviewer(s): (3)

Report Approved: (4) Date:

Program Areas Reviewed: (5)

Summary
(6)

Findings
(7)

Observations
(8)

Instructions

(1) Enter the date the review was started.
(2) Review No. is made up with the year and sequential numbers during the year. Example: 87-1
(3) Identify the reviewer(s).
(4) Report is approved by the manager of Analytical and Process Chemistry.
(5) List the program areas (PRDs) included in the review.
(6) Summarize the overall results in a brief paragraph. Emphasize those program areas that were in compliance with requirements.
(7) Number each finding and state each finding in one or two sentences. Provide information with each finding that supports the validity of the finding.
(8) Number each observation and follow the directions in (7).