As of 02/26/93

WASTE ISOLATION PILOT PLANT
ANALYTICAL LABORATORY PROCEDURES MANUAL

WP 12-AL

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01961

WIPP ANALYTICAL LABORATORY
QUALITY ASSURANCE PLAN

WP 12-13, Rev. 0

Change History

Approval page, pages v, vi, 1-1, 4-2, 6-1, 6-2, 7-1, 7-3, 7-4, 7-5, 8-1, 9-2, and 9-3 - 09/09/91; pages v, 4-3, 4-4 - 09/04/92; pages v, vi, 4-1, 4-2, 4-3, 4-4, 5-1, 5-2, 6-1, 6-2, 7-1, 7-5, 8-1, 9-1, 9-2, 9-3, 10-1, 10-2 (deleted page), 11-1, 12-1, and 12-2 - 05/07/93

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List of Acronyms

ACS - Access Control System
DOE - Department of Energy
EPA - Environmental Protection Agency
ES&H - Environmental, Safety and Health
GC - Gas Chromatography
IC - Ion Chromatography
LCS - Laboratory Control Sample
MS - Mass Spectrometry
NIST - National Institute of Science and Technology
PRS - Project Records Services
QA - Quality Assurance
QAO - Quality Assurance Officer
QC - Quality Control
RIDS - Records Inventory and Disposition Schedule
RPD - Relative Percent Difference
SNL - Sandia National Laboratory
SOP - Standard Operating Procedures
TIC - Tentatively Identified Compound
VCR - Vacuum Coupling Retainer
VOC - Volatile Organic Compound
WIPP - Waste Isolation Pilot Plant

5/7/93

1.0 INTRODUCTION AND OBJECTIVES

1.1 Laboratory Purpose

The WIPP Analytical Laboratory has been established to perform selected gas analyses on samples collected from bin-scale CH TRU wastes in support of the WIPP Bin-Scale Test Plan. The gaseous components to be analyzed shall ensure the immediate and long term safety of waste isolation operations and storage. The chemical analysis data will enable the assurance of the "no migration" compliance requirements imposed on waste disposal operations regulated by the EPA under the RCRA, and shall ensure the integrity of the WIPP Bin-Scale Test data.

1.2 Quality Assurance/Quality Control Policy

The management and staff of the Analytical Laboratory are committed to the production of reliable analytical data of high quality which can be measured, documented, or otherwise assured through the periodic performance and recording of general or method specific analytical quality control and support operations.

1.3 Quality Assurance Objectives and the Bin-Scale Test Plan

Management and staff of the Analytical Laboratory support the objectives of the WIPP Bin-Scale Test Plan and the Data Quality Objectives associated with the Test Plan as expressed in the DOE/EM/48063-1, Quality Assurance Program Plan for the Waste Isolation Pilot Plant Experimental - Waste Characterization Program and official correspondence from SNL.

1.4 Purpose of the Quality Assurance Plan

1.4.1 This document meets the requirement specified in Section 1.10 of the QUALITY ASSURANCE PROGRAM PLAN FOR THE WIPP EXPERIMENTAL WASTE CHARACTERIZATION PROGRAM. The requirement specifies that each participating laboratory must have in place a quality assurance plan that describes general quality assurance procedures specific to that laboratory's normal operations.

1.4.2 This document shall serve as a day-to-day operational manual specific to QA/QC activities in the Analytical Laboratory as a supplement to the SOPs.

2.0 REFERENCES

40 CFR 268, Land Disposal Restrictions

ASME NQA-1-1989, Quality Assurance Program Requirements For Nuclear Facilities

Chemistry Quality Control Program; Institute of Nuclear Power Operations; INPO 83-016 (Revision 02), CY-701, August 1989

DOE/EM/48063-1, Quality Assurance Program Plan for the Waste Isolation Pilot Plant Experimental - Waste Characterization Program, Rev. 0, April 1991

DOE/WIPP 91-016, Performance Demonstration Program Plan for the WIPP Experimental Waste Characterization Program; Revision 0, February, 1991

Handbook of Quality Assurance for the Analytical Chemistry Laboratory; Dux, J.P.; Van Nostrand-Reinhold, New York NY, 1986

Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans; QAMS 005/80; U.S. Environmental Protection Agency; December 29, 1980

Quality Assurance and Quality Control; EPA/530-SW-90-121; August 1990

Quality Assurance Project Plan for Bin-Scale Gas Sample Analysis; Prepared for Westinghouse Electric Corporation, Waste Isolation Division, Carlsbad, NM; Prepared by IT Corporation, Cincinnati, OH; March 29, 1991

Standard Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data; ASTM D4210-89

Test Plan: WIPP Bin-Scale CH TRU Waste Tests; Martin A. Molecke; Sandia Report SAND90-1974 UC-721; Printed August 1990

Test Plan Addendum # 1: Waste Isolation Pilot Plant Bin-Scale CH TRU Waste Tests; Martin A. Molecke, Allen R. Lappin; Sandia Report SAND90-2082 UC-721; Printed December 1990

WP 02-6, WIPP Non-Radioactive Hazardous Waste Management Plan

WP 15-701, Review and Approval of WID Operations Procedures

3.0 STRUCTURE OF THE QUALITY ASSURANCE PLAN

This document is structured to provide information, guidance and directives pertinent to the quality assurance policy and programs of the WIPP Analytical Laboratory.

3.1 Section 2 lists the major references used in developing this quality assurance plan.

3.2 Section 4 describes the organizational structure of the Analytical Laboratory, its personnel qualifications and responsibilities, and training. A subsection describes the laboratory facilities and provides a listing of the major analytical instrumentation.

3.3 Section 5 describes the analytical support systems used to generate high quality data at the Analytical Laboratory.

3.4 Section 6 specifies requirements for sample handling, including the preparation of sampling devices, sample custody control, sample receipt and transfer procedures, and sample storage.

3.5 Section 7 describes the analytical methodology and the need for using documented analytical procedures.

3.6 Section 8 describes instrument maintenance requirements and standards preparation necessary to obtain proper quality assurance and quality control of analytical equipment.

3.7 Section 9 describes calibration requirements and verification procedures that calibrations are valid prior to, during, and at the end of analytical runs.

3.8 Section 10 describes data recording, reduction, and reporting requirements of the Analytical Laboratory.

3.9 Section 11 describes the internal and external technical audit procedures which shall periodically be performed on laboratory operations to ensure that all quality control systems are being applied in daily activities.

3.10 Section 12 describes the management of analytical subcontracts used to supplement the WIPP Analytical Laboratory's capabilities in VOCs and inorganic gas analyses.

3.11 Section 13 describes the reports and records management systems to be used to ensure that all validated data are properly reported and that analytical records, including quality assurance records, are maintained as prescribed.

4.0 ORGANIZATION OF THE ANALYTICAL LABORATORY

4.1 Personnel Staffing and Responsibilities

The Analytical Laboratory is a part of the D&AT section under the ES&H department. Currently there are positions for a Team Leader, three Senior Chemists, one Chemist and one Technician. An organization chart is shown in Figure 4-1. 5/7/9

The D&AT Manager is responsible for the administrative and technical functions of the D&AT section. The Manager's responsibilities to Analytical Laboratory functions include staff recruitment, budget control and planning, equipment acquisition, and laboratory safety.

The Team Leader directs the technical activities of the Analytical Laboratory including work assignments, analytical scheduling, and the validation and reporting of data. The Team Leader also ensures that safety measures are taken by laboratory staff when performing analytical activities. 6/7/9

The Senior Chemist's analytical assignments include flammable VOCs, VOCs, flammable fixed and reactive gases. The Senior Chemists are responsible for the development and validation of new methods and procedures. A Senior Chemist will serve as the QAO for the Analytical Laboratory. 5/7/9

The Team Leader may serve as Alternate QAO for the Analytical Laboratory. The QAO shall be responsible for the review and validation of quality control and sample data and for conduct of quality control activities. 5/7/9

The chemists in the Analytical Laboratory shall perform their analytical assignments under the direction of the lead and senior chemists or QAO. Analytical assignments include IC, GC, MS and GC-MS. Chemists shall serve as the Analytical Laboratory Sample Custodians. The Sample Custodian is responsible for receipt, storage, and shipment of samples and sampling equipment. 5/7/9

Technicians shall perform routine sample analyses, equipment maintenance, and records management under the guidance of a Chemist. Technicians shall assist in the procurement of supplies and service contracts.

4.2 Personnel Qualifications and Training

All personnel in the Analytical Laboratory shall have educational and experience qualifications appropriate to their assignment. Generally, one year of education towards a B.S. or advanced degree can be substituted for two years of work experience in a related area.

The D&AT Manager shall have a minimum of a Master's degree, or equivalent, in a scientific discipline and five years of progressively responsible experience in scientific areas.

The Team Leader position shall require a minimum of a Bachelor's degree in a scientific discipline and five years of progressively responsible experience in laboratory activities. 5/7/9

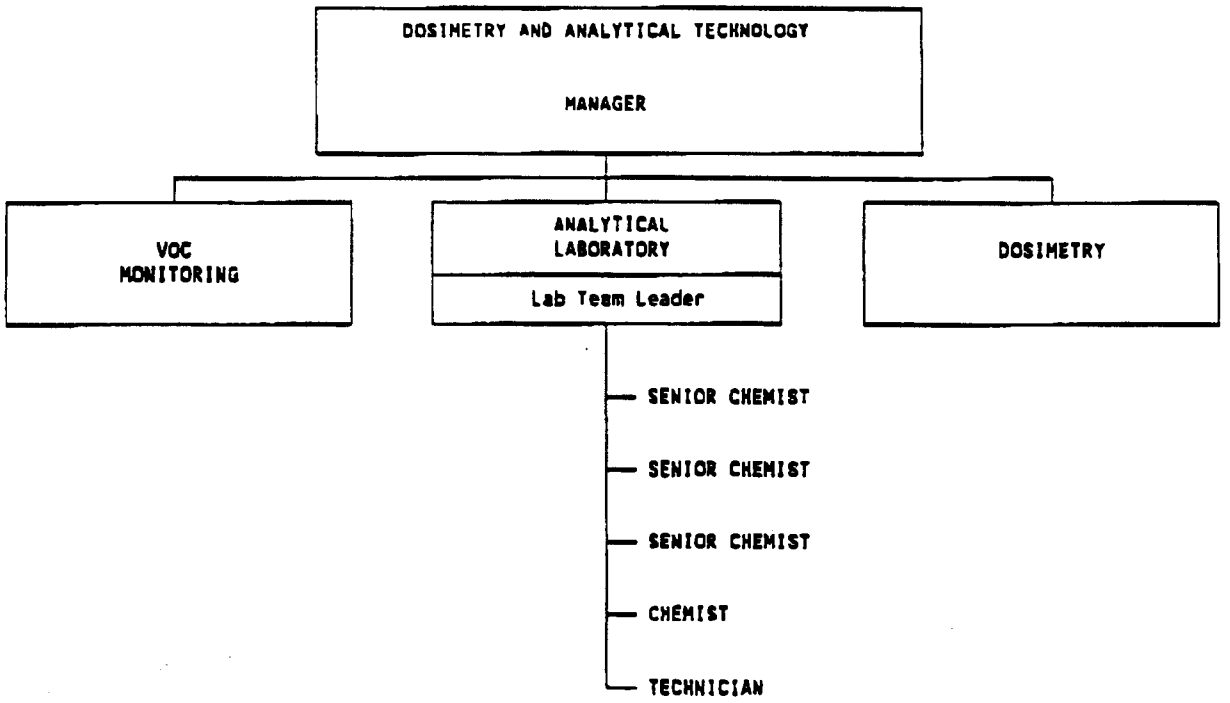

FIGURE 4.1. Organizational Chart

The Senior Chemist shall have a minimum of a Bachelor's degree in a scientific discipline and three years of progressively responsible technical experience in laboratory activities.

The Chemist positions shall require a minimum of a Bachelor's degree in a scientific discipline.

The technician positions require a minimum of a high school diploma and one year of post-high school education or training in a related field.

All Analytical Laboratory personnel shall be provided safety and chemical hygiene training and shall be provided educational opportunities to enhance career development.

All Analytical Laboratory personnel shall be trained by an experienced staff member in accordance with WIPP training procedures. Personnel shall demonstrate competence in their analytical position prior to analyses of any samples. A record of all training accomplishments shall be maintained in an employee's personnel folder, which will be maintained in the Training Department. 9/4/93

4.3 Laboratory Facilities

The Analytical Laboratory occupies approximately 1,500 square feet in the Support Building. The space is subdivided into four instrumentation rooms and three office areas. The major analytical instrumentation currently located in the Analytical Laboratory is shown in Table 4-1. 5/7/93

Table 4-1

ANALYTICAL LABORATORY INSTRUMENTATION

INSTRUMENT TYPE	MANUFACTURER	MODEL NUMBER	INSTALLED
MASS SPECTROMETER	AEROVAC	1000	1991
GAS CHROMATOGRAPH	PERKIN-ELMER	SIGMA 2000	1988
GAS CHROMATOGRAPH	PERKIN-ELMER	SIGMA 2000	1988
ION CHROMATOGRAPH	DIONEX	45001	1991
GC/MASS SPECTROMETER	FINNIGAN	SSQ-710	1992
MASS SPECTROMETER	FISON	VG 30-38	1992
GC/MASS SPECTROMETER	FINNIGAN	MAGNUM	1992

5.0 QUALITY CONTROL OF ANALYTICAL SUPPORT SYSTEMS

In the preparation and processing of samples, laboratories depend on commonly used analytical support systems such as an analytical balance, reagent grade water, thermal devices (ovens/refrigerators, etc.), contaminant free chemicals, and glassware washing facilities. Where the quality of data is dependent on the proper functioning of support systems, the systems shall be monitored at a specified frequency to ensure their proper functioning. Where applicable, control limits shall be specified for such monitoring results and corrective actions taken when control limits are exceeded.

5.1 Electronic Balance

The operation of the electronic balance, including performance checks, is described in the SOP for balance operations.

The proper operation of the balance shall be checked by the weighing of selected Class S or S1 weights, traceable to the NIST.

A balance log book shall be maintained with the electronic balance. The logbook shall contain records of periodic calibration checks, maintenance, and annual service. A balance not meeting the criteria of the SOP shall be removed from service until corrective action is taken and documented in the balance logbook.

5.2 Reagent Grade Water

The Analytical Laboratory uses commercially purchased distilled water which is processed through a water purification system. The water quality is monitored by an internal resistivity meter on the outlet of the purification system which reads in megohms-cm. The resistivity of the water shall be monitored prior to each use. All measurements of water resistivity shall be recorded in a maintenance logbook. The resistivity of the water shall exceed the allowed minimum resistivity for the production of reagent grade water. If the resistivity measurement fails this criteria, a corrective action shall be initiated and logged.

5.3 Thermometers and Thermal Devices

Analytical Laboratory thermometers shall be calibrated by the Calibration Laboratory to standards traceable to NIST.

The operating temperature of thermal devices, such as ovens or refrigerators, shall be measured with calibrated thermometers or thermocouples. The measurements shall be recorded in the appropriate logbook. If temperatures are not within allowable limits, minor adjustments may be made to temperature controls and documented in the appropriate logbook.

A corrective action shall be initiated and logged for any device found to be malfunctioning.

5.4 Glassware or Sampling Equipment Cleaning

Requirements for glassware and other sampling equipment cleaning are normally specified by a SOP. The cleanliness of glassware used for ion chromatographic analyses shall be confirmed by the use of a reagent blank.

Canisters for sampling of head space gases shall be cleaned and certified in accordance with a SOP.

6.0 QUALITY CONTROL FOR SAMPLE HANDLING

The Analytical Laboratory shall receive and analyze, or forward for subcontract analysis, samples collected by Waste Handling Operations. The laboratory shall be responsible for preparation of sampling canisters and have primary custody control for canisters. Waste Handling personnel shall have intermediate custody while sampling vessels are under their control. This section provides guidance on quality control activities intended for the assurance of scientific and legal integrity of the samples.

6.1 Preparation of Sampling Devices

Bin-scale gas samples taken from underground or surface locations shall use a manifold designed to accommodate one or more canisters and a filter pack or single sample canisters used for TRUPACT headspace sampling. Individual SOPs detailing manifold cleaning and filter pack preparation are listed in the applicable procedures manual.

6.2 Sample Custody Control

The general procedures for sample custody control applicable to manifolds, canisters, and filter packs are contained in individual SOPs.

Sub-contractors are required to maintain sample custody and control of all samples received and transferred as directed by the WIPP Analytical Laboratory Shipping and Receiving SOP. The Shipping and Receiving SOP describes shipment to sub-contract facilities with resultant custody transfer. This SOP also provides directives for the initiation of custody on new canisters.

Sample custody in the Analytical Laboratory shall be maintained through the use of custody lockers and intralaboratory custody forms.

6.3 Sample Receipt and Log-In

A logbook shall be maintained of all samples received for analysis or samples transferred to a sub-contractor. A system shall be maintained for assigning unique identifying numbers to each sample received. Upon receipt of the sample, this number will be recorded in the log book, and the sample tagged with the identifying number. The date and time of receipt, sample information, type of analysis to be performed, sub-contractor assignments, and the initials of the person receiving the sample, and any other pertinent information shall be recorded in the logbook.

6.4 Analytical Assignment/Sample Storage/and Holding Time

Upon completion of sample receipt, the Sample Custodian shall notify the analyst(s) of sample receipt. If the analyst is unavailable the sample will be placed in the custody locker. The sample shall be signed out by the analyst from the Sample Custodian or custody locker when the analyst is able to proceed with the analysis. Upon completion of the analysis, the analyst shall return the sampling device to the custody locker.

The Analytical Laboratory shall analyze samples as soon as practical after sample receipt. Flammable gas samples shall be analyzed and reported within 24 hours of sample receipt. Samples to be shipped to a sub-contract facility shall be

shipped within four days of sample receipt. The subcontractor shall analyze samples within 10 working days of verified time of sample receipt. 9

6.5 All samples shall have the prior approval of the Transportation and Hazardous Materials Handling group before shipping offsite.

6.6 The holding time for VOC analysis from a Summa canister is 30 days. 5

The holding time for fixed gas analysis from a Summa canister is 6 months.

The holding time for reactive gas filter pak filter papers is 7 days.

7.0 USE OF DOCUMENTED METHODOLOGY

The use of methodology approved through a regulatory process or thoroughly documented through interlaboratory testing by Standards organizations (i.e., ASTM or EPA) is a critical and necessary component of quality assurance. The Analytical Laboratory shall adhere to its own SOPs to ensure proper QA/QC as adapted from scientific advisory organizations. As requirements for other analytes are demanded, applicable SOPs will be developed in accordance with WIPP policy.

7.1 Volatile Organic Compounds (VOCs)

The SOP used for the analysis of VOCs is adapted from the US EPA Method TO-14. The flammable VOCs are a subset of VOCs. A listing of the target VOCs is provided in Table 7-1, including the designation of those VOCs which are flammable. Non VOC target analytes are listed in Table 7-2.

VOCs not on the target compound list (Table 7-1) are TICs. A compound designated as a TIC shall be reported when its estimated concentration is greater than five times the method detection limit of the nearest target compound, or one ppm, whichever is less. Five target VOCs are used as an LCS for control charting to ensure the quality of analytical data.

7.2 Flammable Gases

The target analytes for flammable gas analysis are Hydrogen (H_2) and Methane (CH_4). The procedure utilizes a magnetic deflection mass spectrometer. A flammable gas LCS with all target analytes shall be control charted to ensure the quality of analytical data.

7.3 Reactive Gases

The target analytes for reactive gas analysis are Hydrogen Chloride (HCl), Hydrogen Sulfide (H_2S) and Sulfur Dioxide (SO_2) reported as Hydrogen Sulfide, Nitrogen Oxides (NO_x) except for Nitric Oxide (NO), and Ammonia (NH_3). The procedure utilizes an ion chromatograph to analyze the anions and cations formed after reaction in the filter pack. An LCS containing all target analytes shall be control charted to ensure the quality of analytical data.

7.4 Nitric Oxide

Nitric Oxide analysis shall be performed on samples collected in canisters as it is not susceptible to the filter pack chemical trapping process. The analysis shall be performed by mass spectrometry. An LCS shall be control charted to ensure the quality of analytical data.

7.5 Prohibition Against Deviation from SOPS

Analysts shall NOT deviate from the SOPs in the performance of their assigned analytical activities. All laboratory procedures shall be written, approved, revised, and canceled in accordance with WP 15-7. When a procedure requires modification, the analyst will notify the QAO. The QAO shall review the required modifications and forward recommendations to the Team Leader. Modifications shall be initiated only after concurrence has been obtained from the Team Leader. A revised or modified procedure shall be subject to validation and documentation

and a copy of the original procedure (prior to revision) shall be retained for records.

Table 7-1

FLAMMABLE VOCs QUANTITATION LIMITS

Volatiles	Flammable VOC	CAS Number	PRQL (ppmv)
1. Acetone	Yes	67-64-1	100
2. Benzene	Yes	71-43-2	1
3. Bromoform		75-25-2	1
4. 1-Butanol	Yes	71-36-3	100
5. 2-Butanone	Yes	78-93-3	100
6. Carbon Tetrachloride		56-23-5	1
7. Chlorobenzene	Yes	108-90-7	1
8. Chloroform		67-66-3	1
9. Cyclohexane	Yes	110-82-7	1
10. 1,1-Dichloroethane	Yes	75-35-3	1
11. 1,2-Dichloroethane	Yes	107-06-2	1
12. 1,1-Dichloroethene	Yes	75-35-4	1
13. cis-1.2.-Dichloroethene	Yes	156-59-2	1
14. Ethyl Benzene	Yes	100-41-4	1
15. Ethyl Ether	Yes	60-29-7	1
16. Methanol	Yes	67-56-1	100
17. Methylene Chloride		75-09-2	1
18. 4-Methyl-2-Pentanone	Yes	108-10-1	100
19. 1,1,2,2-Tetrachloroethane		79-34-5	1
20. Tetrachloroethene		127-18-4	1
21. Toluene	Yes	108-88-3	1
22. 1,1,1-Trichloroethane		71-55-6	1
23. Trichloroethene		79-01-6	1
24. 1,1,2-Trichloro-1.2.2-trifluoroethane		76-13-1	1
25. 1,3,5-Trimethylbenzene	Yes	108-67-8	1

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26. 1,2,4-Trimethylbenzene	Yes	95-63-6	1
27. m-Xylene*	Yes	108-38-3	1
28. o-Xylene	Yes	95-47-6	1
29. p-Xylene*	Yes	106-42-3	1

* These xylene isomers cannot be resolved by the analytical methods employed by this program and will be reported as total m and p xylene

Table 7-2

REACTIVE, FLAMMABLE OR OTHER INORGANIC GASES

GAS	TYPE	CAS NUMBER	PRQL (ppmv)
Hydrogen	Flammable	1333-74-0	≥ 1
Oxygen		7782-44-7	≥ 1
Methane	Flammable	74-82-8	≥ 1
Ammonia	Reactive	7664-41-7	≥ 1
Hydrogen Chloride	Reactive	7647-01-0	≥ 1
Hydrogen Sulfide/ Sulfur Dioxide Reported as Hydrogen Sulfide	Reactive	7783-06-4 7783-09-5	≥ 1
Nitrogen Oxides (NO_x)	Reactive		≥ 1
Nitric Oxide		10102-43-9	≥ 1
Nitrogen		7727-37-9	≥ 1
Carbon Dioxide		124-38-9	≥ 1
Carbon Monoxide		630-08-0	≥ 1
Argon		7440-37-1	≥ 1
Krypton	Tracer	7439-90-9	≥ 1
Xenon	Tracer	7440-63-3	≥ 1

8.0 QUALITY CONTROL OF MEASUREMENT SYSTEMS

8.1 Instrument or Equipment Maintenance

Only properly qualified personnel shall be permitted to repair and maintain analytical instrumentation. An instrument maintenance logbook shall contain all information concerning the service performed by an analyst or authorized manufacturer's service representative. A companion file containing the records of the service personnel's visit will be maintained.

An adequate supply of spare parts shall be kept in the Analytical Laboratory stock for all instruments with replacement parts that can be installed by laboratory personnel. A parts inventory for each major piece of equipment shall be kept with a minimum stocking quantity established for critical and high usage parts. VCR gaskets will be procured from the warehouse stock system. 5/

Instruments shall be assigned a principle operator who will be responsible for maintaining the instrument and the spare parts for that instrument. Spare parts will be kept in locked storage and inventoried on a monthly basis. Shelf lives for parts which deteriorate shall be established and shown on the inventory list and part packaging. Service and repair of instrumentation shall be covered under a manufacturer's service contract consistent with WIPP policy.

8.2 Reagent Chemicals and Standards

All chemicals and solvents used in analytical operations shall meet ACS Reagent Grade quality or be of documented purity or as specified by the applicable SOP. Chemicals and standards shall be received and inventoried in accordance with the Chemical Receipt, Classification, and Inventory SOP.

Chemical testing or monitoring for purity will not routinely be required. The lot numbers of any chemical used in the preparation of reagent blanks, standards, or sample analyses shall be recorded in the appropriate logbook. Logbook documentation shall include the date of preparation of the stock standard and any dilutions performed to obtain a working standard. The documentation shall also include the name of the chemical(s), purity, grade, or stated concentration, and the suppliers name.

To ensure that standards are of proper quality, they shall be certified by any of the following methods: (1) a manufacturer's certificate; (2) standardization against a previously used and traced standard; or (3) standardization against a certified reference material containing known values of the analytes. Records of traceability shall be maintained by the Laboratory for three years or for the duration of the Bin-Scale Test Phase, whichever is longer. Chemicals found to be unsatisfactory for use shall be removed from inventory and discarded through proper disposal procedures. 9/9

9.0 QUALITY CONTROL OF ANALYTICAL OPERATIONS

9.1 Method Calibration

Method calibrations are included in the SOPs. The method shall be calibrated at intervals specified by the SOP. Calibrations shall be verified through the use of an LCS. The analyst shall document all method calibrations in the instrument logbook.

9.2 Reagent Blanks

A reagent blank is used to establish a baseline for the calibration curve and to demonstrate freedom from contamination. The reagent blank shall be processed through the analytical procedure as though it were a sample.

In an aqueous matrix, calibration blanks shall normally consist of reagent water from the same source as that used to prepare standards. Gaseous matrix calibration blanks will use zero air, nitrogen, argon, or helium, as appropriate to the method. The analyst shall be responsible for documenting the calibration blank was run and results were satisfactory prior to the analysis of authentic samples.

Unless specified by the SOP, the concentration of analyte in a reagent blank is not subtracted from the concentration of the analyte in the sample. When a reagent blank exceeds one half the PRDL, the Team Leader and QAO shall be notified and corrective action initiated.

9.3 Laboratory Control Samples (LCS)

A measurement of analytical accuracy shall be obtained by repetitive analyses of LCSs. Following method calibration and immediately before analyzing samples, the calibration shall be checked with an LCS. The LCS shall be prepared independently from the method calibration standards. Documentation of preparation, composition, and/or traceability shall be maintained in the appropriate logbook.

At the end of an analytical run and at intervals of every ten samples during the analytical run, an LCS shall be analyzed to ensure that the method is in calibration. The values for LCSs shall fall within values specified in Table 9-1. If specified values are exceeded, the QAO will be notified and corrective action initiated.

9.4 Precision Measurement/Duplicate Analyses

Analytical precision in the laboratory shall be determined using a laboratory duplicate sample. When sufficient sample exists, a duplicate sample shall be analyzed with each batch of samples or after every tenth sample within an analytical batch.

A duplicate sample will consist of equal aliquots of one sample analyzed within the same analytical batch. The first aliquot will be designated the sample whose value is to be reported. The second will be designated as the sample duplicate. Results are not averaged when reported.

Table 9-1

GAS ANALYSIS QUALITY ASSURANCE OBJECTIVES

Compounds	Precision (%RSD or RPD)	Accuracy (%R)
VOCs	± 25 %	70-130%
Argon Carbon Monoxide Carbon Dioxide Hydrogen Nitrogen Oxygen Methane Other Inorganic Gases Reactive Gases	± 10 %	90-110%
NITROGEN OXIDES	± 20 %	50-150%

% RSD - Percent Relative Standard Deviation

% RPD - Relative Percent Difference

% R - Percent Recovery

Calculation of Relative Percent Difference: The unit for measurement of precision between duplicate samples will be RPD. RPD shall be calculated by the following formula:

$$RPD\ (\%) = [\ S - D\ /\ \{(S + D)/2\}\ ]\ X\ 100\%$$

Where:

S = concentration of analyte in the sample.

D = concentration of analyte in the duplicate.

The RPD shall be compared to acceptability criteria. If the RPD for the duplicate set falls outside the criteria, the QAO shall be notified and corrective action initiated.

RPDs of samples that have concentrations less than 10 times their detection limits do not normally require corrective action unless specified by the Team Leader.

9.5 Accuracy Measurement/Laboratory Control Sample

Calculation of Analyte Recovery: Recovery (%R) of each analyte in the LCS is calculated by the following formula:

$$\%R = \{ FV/TV \} \times 100\%$$

Where:

%R = % Recovery
FV = Found Value in LCS
TV = True (or certified) Value in LCS

When a minimum of 21 %R values have been obtained for a particular analyte, the mean %R and standard deviation will be calculated for that analyte. A Control Chart shall be maintained for %R for each LCS. The Upper and Lower Control Limits shall be established at ±3sd about the Target Line. The Upper and Lower Warning Limit shall be established at ±2sd about the Target Line. After 30 LCSs have been completed, the mean %R for the most recent 30 or greater samples shall be the Target Line.

If the %R for an LCS analyte falls outside its Upper or Lower Control Limit, the QAO shall be notified and corrective action initiated.

All analytes for reactive gas, fixed gas, and flammable gas analysis shall be control charted. Only five selected analytes for VOCs and flammable VOCs shall be control charted.

Results of LCS samples that are outside of 3sd will be control charted unless they have an assignable cause or they are outside of five standard deviations.

9.6 Field Collected Quality Control Samples

A percentage of samples collected in the field shall be field duplicates and/or other control samples. These samples shall be processed as any other sample, and results reported accordingly. If two samples are identified as field duplicates, the RPD shall be calculated and reported. Evidence of any contamination in the manifold blanks shall be reported to the QAO and corrective action initiated. Other field blanks, if separate and distinct from manifold blanks, will simply be reported as analyzed with no necessity for laboratory corrective action.

9.7 Completeness

Completeness is the measure of acceptable sample analyses as expressed by percentage of acceptable out of the number received. The requirement for completeness for the Analytical Laboratory is 90%. For flammability samples the requirement for completeness is 100% expressed as the number of bin headspace samples requiring analysis divided by the number of bin headspace samples with valid results.

10.0 QUALITY CONTROL OF DATA RECORDING/REPORTING

10.1 Software Validation

All software employed in data collection and reduction activities shall be validated in accordance with WIPP software validation policy. Two independent verifications of all mathematical calculation formulas shall be performed. A record of software and formula validation activities shall be maintained in Analytical Laboratory files.

10.2 Data Recording and Data Reduction

The analyst shall have primary responsibility for data reduction and recording in appropriate Laboratory notebooks or data packages.

Entries shall be made in black ink. Corrections shall be made with a single strike-through of the incorrect entry, followed by entry of the corrected value. The strike-through and corrected entry shall be dated and initialed. No "white-out" type correction fluid is permitted in correcting entries.

All notebook pages shall be dated and signed or initialled by the analyst. Computer generated data shall be traceable to the analytical run and analyst recorded in the logbook. There shall be no unaccounted-for gaps in sequentially numbered records and logbooks shall be inventoried and kept in a secure location.

At the time of reporting, 100% of the analysts' hand calculations shall be reviewed by the QAO to ensure that calculations have been properly performed and results correctly reported. The QAO shall sign and date the records to signify that they have been reviewed and found to be acceptable or that appropriate corrections have been made.

A representative sampling of computer calculations shall be reviewed by the QAO to ensure that calculations are valid and results correctly reported. The QAO shall sign and date the computer records to signify they have been reviewed.

The QAO or Team Leader shall, on a monthly basis (or more frequently where the volume of analyses necessitates) review the analyst's logbooks, and raw data records. The QAO or Team Leader shall sign and date the records to signify that they have been reviewed and found to be acceptable or that appropriate corrections have been made.

All raw data and calculated results shall be maintained for a minimum of three years in an orderly record that allows for retrieval. Where regulatory requirements specify a longer retention time for data files, the longer record retention time shall be honored.

10.3 Data Validation and Reporting

Data reporting will be the responsibility of the Team Leader, who may delegate the function to one or more designated persons. The Team Leader and QAO, shall ensure data referred for reporting has been reviewed and data qualifications or exceptions noted. In addition to sample analytical results, the final report shall include sample identification information and QA reports applicable to the analyses performed. The D&AT Manager, or designee, shall validate all Analytical Laboratory reports.

analyses performed. The D&AT Manager, or designee, shall validate all Analytical Laboratory reports.

11.0 LABORATORY SYSTEM AUDITS

11.1 Internal System Audits

Annually, or as required, the D&AT Manager or higher level Westinghouse management, shall arrange for an internal systems review of Analytical Laboratory operations. All laboratory personnel shall cooperate with auditing personnel. Audits may be conducted by corporate or by outside, third party personnel.

Audits may include, but not necessarily be limited to, the examination of any and all records associated with sample receipt, tracking, storage, safety, calibration, analysis, reporting, disposal and records maintenance. The results of audit reviews shall be made known to the Team Leader and the D&AT Manager within 15 calendar days following the review. Upon receipt of the report, the Team Leader will initiate an investigation into the noted deficiencies, shall take the necessary corrective actions, and shall respond within 20 calendar days of receipt of the audit.

11.2 External System Audits

The Analytical Laboratory will be subject to periodic system audits or inspections by representatives of Federal or State agencies. The policy of the Analytical Laboratory shall be to extend full cooperation to the authorized representatives of such agencies. Upon receipt of a report of audit findings, the Team Leader shall initiate an investigation into the noted deficiencies, shall take the necessary corrective actions, and shall appropriately respond to the report within the time constraints specified in the audit.

11.3 Performance Demonstration Program

The Analytical Laboratory shall participate in any Performance Demonstration Program conducted by the DOE or its authorized representatives.

12.0 MANAGEMENT OF ANALYTICAL SUBCONTRACTS

A portion of the analytical work assigned to the Analytical Laboratory may be directed to one or more subcontractor laboratories. Subcontractors shall be solicited, evaluated, and contracts awarded in accordance with applicable Federal Acquisition Regulations and DOE Regulations. WIPP QA personnel shall approve the QA programs of contract laboratories.

Contracts shall be monitored by the D&AT Manager and the Team Leader. Contracts shall specify the types of analyses required, QA provisions, report formats, and the allowable turnaround time.

Subcontractors shall be subject to annual, or more frequent, technical and QA audits by WIPP personnel as determined by the D&AT Manager. Subcontractors shall also be required to participate in Performance Demonstration Programs conducted by the DOE or its authorized representatives, applicable to the types of analyses specified under their contract. Subcontractors shall be expected to respond to any identified analytical deficiencies. In the event of subcontractor nonperformance or deficient performance, the Team Leader shall recommend to the Westinghouse contracts officer the action to be taken relative to the nature of the deficiency in accordance with applicable regulations.

13.0 REPORTS AND RECORDS MANAGEMENT

The Team Leader shall be responsible for the preparation of any periodic progress or summary reports which may be required by the DOE, Westinghouse Electric Corporation, or their authorized representatives.

QA Records shall be corrected, maintained, and stored in accordance with ASME NQA-1-1989 and the WID QA Manual. QA Records originating from directives specified in this manual or applicable SOPs shall be identified on the Analytical Laboratory RIDS.

The D&AT Manager or designee is responsible for the validation of Laboratory records to be transmitted to the PRS.

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Department of Energy
Albuquerque Field Office
P.O. Box 5400
Albuquerque, New Mexico 87185-5400

JUL 6 1 1993

Ms. Judith Espinosa, Secretary
New Mexico Environment Department
P.O. Box 3149
1190 St. Francis Drive
Santa Fe, NM 87504-0968

Dear Secretary Espinosa:

Enclosed please find three additional documents which should be added to the Environmental Protection Agency supplemental detailed documentation delivered to your office on June 22, 1993.

The additional documentation consists of the following:

1. Quality Assurance Program Plan for the Waste Isolation Pilot Plant Experimental Waste Characterization Program, Revision 2 (Draft), October 12, 1992;

2. Waste Isolation Pilot Plant Analytical Laboratory Procedures Manual (Table of Contents only), WP 12-AL;

3. WIPP Analytical Laboratory Quality Assurance Plan, WP 12-13.

If you have any questions or require further information, please contact Patrick J. Higgins, of my staff at (505) 845-5914.

Mark L. Matthews for

W. John Arthur, III
Project Director
WIPP Project Integration Office

3 Enclosures