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Minimum Analytical Chemistry Requirements for Pit Manufacturing at Los Alamos National Laboratory

Ming M. Moy
Craig S. Leasure

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

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Ming M. Moy and Craig S. Leasure

ABSTRACT

Analytical chemistry is one of several capabilities necessary for executing the Stockpile Stewardship and Management Program at Los Alamos National Laboratory (LANL). Analytical chemistry capabilities reside in the Chemistry Metallurgy Research (CMR) Facility and Plutonium Facility (TA-55). These analytical capabilities support plutonium recovery operations, plutonium metallurgy, and waste management. Analytical chemistry capabilities at both nuclear facilities are currently being configured to support pit manufacturing. This document summarizes the minimum analytical chemistry capabilities required to sustain pit manufacturing at LANL.

By the year 2004, approximately $16 million will be required to procure analytical instrumentation to support pit manufacturing. In addition, $8.5 million will be required to procure glovebox enclosures. An estimated 50% increase in costs has been included for installation of analytical instruments and glovebox enclosures. However, no general and administrative (G&A) taxes have been included. If an additional 42.5% G&A tax were to be incurred, approximately $35 million would be required over the next five years to prepare analytical chemistry to support a 50-pit-per-year manufacturing capability by the year 2004.

1.0 INTRODUCTION

Analytical chemistry is one of several capabilities necessary for executing the Stockpile Stewardship and Management Program at Los Alamos National Laboratory (LANL). Analytical chemistry capabilities reside in the Chemistry Metallurgy Research (CMR) Facility and Plutonium Facility (TA-55). These analytical capabilities support plutonium recovery operations, plutonium metallurgy, and waste management. Analytical chemistry capabilities at both nuclear facilities are currently being configured to support pit manufacturing. This document summarizes the minimum analytical chemistry capabilities required to sustain pit manufacturing at LANL. These analytical chemistry capabilities are based on the projected levels of pit production outlined in the document titled the "New Pit Production Strategy and Associated Facility Modifications at Los Alamos National Laboratory"[1]. The analytical chemistry requirements are also based on the pit-manufacturing model generated by the Technology Modeling and Analysis Group (TSA-7)[2], which resides in the Technology and Safety Assessment (TSA) Division.

2.0 ASSUMPTIONS FOR ANALYTICAL CHEMISTRY CAPABILITIES

The approach to determining analytical chemistry capabilities required for pit manufacturing is based on several assumptions:

- Analytical chemistry capabilities are based on the predicted number of samples generated from the TSA-7 model for plutonium processing, pit fabrication, waste management, and other plutonium operations necessary to support pit manufacturing.

- The analytical chemistry capabilities are based on a "one analytical instrument" scenario in most cases, with no backup instrumentation.

- The analytical chemistry capabilities discussed here support only pit manufacturing and are not intended to directly support other projects or programs.

- Additional analytical chemistry capabilities needed to support other projects will be funded by those other projects. However, if there is surplus capacity that can be used to meet the needs of those other projects without compromising pit manufacturing, those projects will be supported as well.

- The expected data delivery time for an analytical chemistry analysis is 30 days from the time the sample is physically received at either the TA-55 or CMR facilities (which does not include samples transport from TA-55 to CMR).

- The assumed level of pits produced at LANL is 50 per year.

- Plutonium-processing solutions generated at the TA-55 facility will be analyzed at TA-55 instead of being shipped to the CMR facility.

- Solid and liquid samples generated from waste management activities at TA-55 will be processed at the CMR facility.

- Analytical chemistry capabilities will not be duplicated because of differences in geography (TA-55 vs. CMR) unless the pit-manufacturing schedule, programmatic requirements, or cost savings drive the need for duplicate capabilities.

- Analytical chemistry equipment will be duplicated only if separate capability is required for nuclear and nonnuclear activities and only to the extent necessary.

- Infrastructure requirements that are not called out in this document will be supported by the operating organizations (division, group, or project) that will support pit manufacturing.

- Laboratory taxes for procuring analytical chemistry equipment and glovebox enclosures have not been included in the costs, because they may change in the future.

- Introductory hoods into glovebox lines and gloveboxes required for bagouts are not included in this document. Because various configurations can affect the number of introductory hoods and gloveboxes required for introduction and bagouts, these additional glovebox enclosures must be added at a later date.

3.0 ANALYTICAL REQUIREMENTS FOR PIT MANUFACTURING

At least seven major pit-manufacturing activities require analytical chemistry, including (1) disassembly and metal purification, (2) plutonium fabrication and assembly, (3) plutonium recovery, (4) solid and liquid waste management, (5) nonnuclear fabrication and assembly, (6) materials characterization and failure analysis, and (7) war reserve (WR) process materials testing. Analytical chemistry capabilities are categorized in these seven areas to relate analytical chemistry requirements to the specific pit-manufacturing unit operations. However, cross-cutting infrastructure activities are also required for sustaining all analytical chemistry analysis techniques. These infrastructure requirements are discussed in Section 4 of this document.

The analytical chemistry capabilities required for pit manufacturing are organized in tabular format. This format is intended to facilitate understanding the relationships between the analytical chemistry techniques and their end use in pit-manufacturing-unit operations. Glovebox enclosures used for chemical analysis have been organized in four categories: standard gloveboxes, specialty gloveboxes, open-front gloveboxes, and fume hoods. Standard and specialty gloveboxes are approximately 10 x 12 ft in area. Standard gloveboxes are totally enclosed gloveboxes, and specialty gloveboxes are typically custom-built to enclose analytical instrumentation. Open-front gloveboxes are approximately 4 x 12 ft in area, and fume hoods are approximately 4 x 5 ft in area. Open-front gloveboxes are typically used to conduct analytical analyses and to contain low levels of radioactive materials. Fume hoods are chemical hoods used to contain chemicals and support chemical preparation and are not intended to contain radioactive materials.

3.1 Disassembly and Metal Purification

Disassembly and metal purification are required to provide plutonium metal feed for fabrication and assembly. Feed materials originating from disassembly and metal purification may include pure plutonium oxide, which is converted to metal by the direct oxide reduction (DOR) process, pure plutonium metal generated from the electrorefining (ER) process, and pure metal requiring removal of americium by the molten salt extraction (MSE) process. The various analytical chemistry analyses required to support disassembly and metal purification are shown in Table 1. Also shown in Table 1 is the precision required for the analytical analysis. The expected numbers of samples per year are shown in Table 2. The numbers of analytical instruments and glovebox enclosures required to support this activity are shown in Table 3.

3.2 Plutonium Fabrication and Assembly

Plutonium fabrication and assembly involve casting plutonium metal feed ingots after adding gallium to the plutonium metal and shape-casting the feed ingots into hemishells. Another analytical chemistry activity is analysis of gases used in plutonium fabrication and assembly. Mass spectrometry is typically used to analyze gases. The various analytical chemistry analyses required to support plutonium fabrication and assembly are shown in Table 4. Also shown in Table 4 is the precision required for the analytical analysis. The expected numbers of samples are shown in Table 5. The number of analytical instruments and glovebox enclosures required to support this activity are shown in Table 6.

Table 1. Analytical Chemistry Requirements for Disassembly and Metal Purification

Required Analysis	Analytical Technique	Analytical Instrument	Required Precision (% Rel. Std. Dev.)
Dissolution	Acid Dissolution	Hot Plate	NA[a]
Plutonium Assay	Coulometry	Coulometer	± 0.1
Plutonium Assay	Cerium Titration	Autotitrator	± 0.05
Plutonium Isotopics	Mass Spectrometry	TIMS[b]	± 0.02–0.5
Trace Elements	Mass Spectrometry	ICP-MS[c]	± 15
Trace Elements	Emission Spectrometry	ICP-AES[d]	± 15
Gallium Analysis	X-Ray	XRF[e]	± 10
Iron Analysis	Colorimetry	Colorimeter	± 1
Americium Analysis	Radiochemistry	Gamma Spectrometer	± 10
Neptunium Analysis	Radiochemistry	Proportional Counter	± 5
Neptunium Analysis	Radiochemistry	Alpha Spectrometer	± 5

[a]NA—not applicable.
[b]TIMS—thermal ionization mass spectrometry.
[c]ICP-MS—inductively coupled plasma mass spectrometer.
[d]ICP-AES— inductively coupled plasma atomic emission spectrometer.
[e]XRF—X-ray fluorescence.

Table 2. Expected Number of Samples from Disassembly and Metal Purification

Sample Location	Number of Samples
ER	125
MSE	176

Table 3. Instrument and Glovebox Enclosure Requirements for Disassembly and Metal Purification

Analysis	# Samples	Instrument	# Instruments	# Std. GBXs[a]	# Spec. GBXs	# Open Fronts	# Fume Hoods
Acid Dissolution	201	Hot Plate	12	3	0	1	1
Plutonium Assay	201	Coulometer	1	4	1	5	1
Plutonium Assay[b]	201	Autotitrator	1	4	5	4	1
Plutonium Isotopics	125	TIMS	1	4	0	1	1
Trace Elements	201	ICP-MS	1	1	1	2	1
Trace Elements	201	ICP-AES	1	1	1	2	1
Gallium Analysis	125	XRF	1	0	0	2	1
Iron Analysis	125	Colorimeter	1	0	1	6	1
Americium Analysis	76	Gamma Spectrometer	2	2	0	1	1
Neptunium Analysis	201	Proportional Counter	1	2	0	1	0
Neptunium Analysis	201	Alpha Spectrometer	1	2	0	0	0

[a]GBX—glovebox.
[b]We currently use coulometry for plutonium assay because of an insufficient quantity of certified reference material plutonium metal standards. A supply of these standards should be available in 2003.

Table 4. Analytical Chemistry Requirements for Fabrication and Assembly			
Required Analysis	**Analytical Technique**	**Analytical Instrument**	**Required Precision (% Rel. Std. Dev.)**
Dissolution	Acid Dissolution	NA	NA
Plutonium Assay	Coulometry	Coulometer	± 0.1
Plutonium Assay	Cerium Titration	Autotitrator	± 0.05
Plutonium Isotopics	Mass Spectrometry	TIMS	± 0.02–0.5
Trace Elements	Mass Spectrometry	ICP-MS	± 15
Trace Elements	Emission Spectrometry	ICP-AES	± 15
Gallium Analysis	X-Ray	XRF	± 2
Iron Analysis	Colorimetry	Colorimeter	± 1
Americium Analysis	Radiochemistry	Gamma Spectrometer	± 10
Interstitial Analysis	Combustion	Thermal Conductivity Analyzer	± 10
Gas Analysis	Mass Spectrometry	Mass Spectrometer	± 1
Neptunium Analysis	Radiochemistry	Proportional Counter	± 5
Neptunium Analysis	Radiochemistry	Alpha Spectrometer	± 5

Table 5. Expected Number of Samples from Fabrication and Assembly	
Location	**Number of Samples**
Feed Casting	125
Shape Casting	125
Gas Sampling	50

Table 6. Instrument and Glovebox Enclosure Requirements for Fabrication and Assembly							
Analysis	**# Samples**	**Instrument**	**# Instru-ments**	**# Std. GBXs**	**# Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Acid Dissolution	125	Hot Plate	12	2	0	1	1
Plutonium Assay	125	Coulometer	1	4	1	5	1
Plutonium Assay	Cerium Titration	Autotitrator	1	4	5	4	1
Plutonium Isotopics	125	TIMS	1	4	0	1	1
Trace Elements	125	ICP-MS	1	1	1	2	1
Trace Elements	125	ICP-AES	1	1	1	2	1
Gallium Analysis	125	XRF	1	0	0	2	1
Iron Analysis	125	Colorimeter	1	0	1	6	1
Interstitial Analysis	125	Thermal Conductivity Analyzer	3	3	3	1	1
Americium Analysis	125	Gamma Spectrometer	1	2	0	1	0
Gas Analysis	50	Gas Mass Spectrometer	1	2	1	1	1
Neptunium Analysis	125	Proportional Counter	1	2	0	1	0
Neptunium Analysis	125	Alpha Spectrometer	1	2	0	0	0

3.3 Plutonium Recovery

Plutonium recovery involves purifying plutonium from solid and liquid feed sources. Acid dissolution, nitrate anion exchange, and chloride anion exchange operations are used in plutonium purification. Plutonium product solutions are precipitated after purification and are calcined to pure plutonium oxide. These activities provide a pure plutonium oxide feed source for disassembly and metal purification. The various analytical chemistry

analyses required to support plutonium recovery are shown in Table 7. Also shown in Table 7 is the precision required for the analytical analysis. The expected numbers of samples are shown in Table 8. The number of analytical instruments and glovebox enclosures required to support this activity is shown in Table 9.

Table 7. Analytical Chemistry Requirements for Plutonium Recovery

Required Analysis	Analytical Technique	Analytical Instrument	Required Precision (% Rel. Std. Dev.)
Dissolution	Acid Dissolution	Microwave	NA
Americium Analysis	Radiochemistry	Gamma Spectrometer	± 5
Plutonium Assay	Radiochemistry	Proportional Counter	± 5
Plutonium Assay	Radiochemistry	Liquid Scintillation	± 5
Plutonium Isotopics	Radiochemistry	Germanium Detector	± 5–10
Trace Elements	Mass Spectrometry	ICP-MS	± 15
Trace Elements	Emission Spectrometry	ICP-AES	± 15
Plutonium III Analysis	Spectrophotometry	Spectrophotometer	± 15
Trace Uranium Analysis	X-Ray	XRF	± 10
Trace Analysis	Atomic Emission	Direct-Current Plasma	± 10
Anion Analysis	Ion Chromatography	Ion Chromatograph	± 5
Loss on Ignition	Combustion	Furnace	± 2
Iron Analysis	Spectrophotometry	Visible Region Spectrophotometer	± 5

Table 8. Expected Number of Samples from Plutonium Recovery	
Location	**# Samples**
Nitrate Ion Exchange	84
Nitrate Precipitation	84
Nitrate Oxide Roasting	84
Nitrate Distillate	84
Acid Recycle Distillate	0
Evaporator Bottoms	28
Chloride Ion Exchange	39
Chloride Precipitation	39
Chloride Oxide Roasting	39
Chloride Effluents	31
Chloride Organic Recycle	0
Blended Oxide for DOR	25

Table 9. Instrument and Glovebox Enclosure Requirements for Plutonium Recovery

Analysis	# Samples	Instrument	# Instru-ments	# Std. GBXs	# Spec. GBXs	# Open Fronts	# Fume Hoods
Dissolution	25	Microwave	3	6	0	1	1
Americium Analysis	28	Gamma Spectrometer	2	2	0	1	1
Plutonium Assay	25	Proportional Counter	1	2	0	1	0
Plutonium Assay	25	Liquid Scintillator	1	2	0	1	1
Plutonium Isotopics	25	Germanium Detector	2	2	0	1	1
Trace Elements	25	ICP-MS	1	1	1	2	2
Plutonium III Analysis	28	Visible-Region Spectrophoto-meter	1	6	1	3	1
Trace Elements	25	ICP-AES	1	1	1	2	1
Trace Analysis	28	Direct-Current Plasma	1	2	0	1	1
Iron Analysis	28	Visible-Region Spectrophoto-meter	1	0	1	6	1
Loss on Ignition	25	Furnace	1	5	0	2	0

3.4 Solid and Liquid Waste Management

Solid and liquid waste management involves final disposal of wastes generated by TA-55 and the CMR facility at the TA-50 Waste Treatment Facility. Solid and liquid wastes are analyzed for actinides before final disposal. The various analytical chemistry analyses required to support solid and liquid waste management are shown in Table 10. Also shown in Table 10 is the precision required for the analytical analysis. The expected numbers of samples are shown in Table 11. The numbers of analytical instruments and glovebox enclosures required to support this activity are shown in Table 12.

Table 10. Analytical Chemistry Requirements for Solid and Liquid Waste Management

Required Analysis	Analytical Technique	Analytical Instrument	Required Precision (% Rel. Std. Dev.)
Dissolution	Acid Dissolution	Microwave	NA
Americium Analysis	Radiochemistry	Gamma Spectrometer	± 5–20
Plutonium Assay	Radiochemistry	Alpha Spectrometer	± 10
Plutonium Isotopics	Radiochemistry	Germanium Detector	± 10
Trace Elements	Mass Spectrometry	ICP-MS	± 15
Trace Elements	Emission Spectrometry	ICP-AES	± 15
Trace Uranium Analysis	X-Ray	XRF	± 10
VOAs[a]	Organic	GC-MS[b]	± 10
SVOAs[c]	Organic	GC-MS	± 10

[a]VOA—volatile organic analysis.
[b]GC-MS—gas chromatography-mass spectrometer.
[c]SVOA—semivolatile organic analysis.

Table 11. Expected Number of Samples from Solid and Liquid Waste Management

Location	# Samples
Cementation	11
Organics to Packaging	12
Neutralization and Carrier Precipitation	45
Industrial Waste Liquid	30
Discharge to Canyon	30
Transuranic Sludge	1
Solid Waste	1

Table 12. Instrument and Glovebox Enclosure Requirements for Solid and Liquid Waste Management							
Analysis	**# Samples**	**Instrument**	**# Instru-ments**	**# Std. GBXs**	**# Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Dissolution	2	Microwave	3	6	0	1	1
Americium Analysis	30	Gamma Spectrometer	2	2	0	1	1
Plutonium Assay	30	Alpha Spectrometer	1	2	0	0	0
Plutonium Isotopics	45	Germanium Detector	1	2	0	1	1
Trace Elements	32	ICP-MS	1	1	1	2	1
Trace Elements	32	ICP-AES	1	1	1	2	1
Trace Uranium Analysis	32	XRF	1	1	0	2	1
VOAs	14	GC-MS	2	2	0	2	2
SVOAs	14	GC-MS	1	1	0	2	1

3.5 Nonnuclear Fabrication and Assembly

Nonnuclear fabrication and assembly provide nonfissile components for pit manufacturing, which requires analyzing nonfissile metals and in-line or at-line analytical chemistry. The various analytical chemistry analyses required to support nonnuclear fabrication and assembly are shown in Table 13. Also shown in Table 13 is the precision required for the analytical analysis. The expected numbers of samples are shown in Table 14. The number of analytical instruments and glovebox enclosures required to support this activity are shown in Table 15.

Table 13. Analytical Chemistry Requirements for Nonnuclear Fabrication and Assembly			
Required Analysis	**Analytical Technique**	**Analytical Instrument**	**Required Precision (% Rel. Std. Dev.)**
Dissolution	Acid Dissolution	NA	NA
Trace Elements	Mass Spectrometry	ICP-MS	± 15
Trace Elements	Emission Spectrometry	ICP-AES	± 15
Surface Residues	Infrared	FT-IR[a]	± 10
Surface Residues	Organic	GC-FID[b]	± 10

[a]FT-IR—Fourier transform-infrared.
[b]GC-FID—Gas chromatography-flame ionization detector

Table 14. Expected Number of Samples from Nonnuclear Fabrication and Assembly	
Location	**# Samples**
Nonnuclear Fabrication	50
Assembly	50

Table 15. Instrument and Glovebox Enclosure Requirements for Nonnuclear Fabrication and Assembly							
Analysis	**# Samples**	**Instru-ment**	**# Instru-ments**	**# Std. GBXs**	**# Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Dissolution	50	NA	NA	0	0	0	2
Trace Elements	100	ICP-MS	1	0	0	0	2
Trace Elements	100	ICP-AES	1	0	0	0	2
Surface Residues	75	FT-IR	1	0	0	0	2
Surface Residues	50	GC-FID	1	0	0	0	2

3.6 Materials Characterization and Failure Analysis

Materials characterization and failure analysis involves providing optical metallography and surface analysis of fissile and nonfissile components. Various materials science and surface science techniques are required to evaluate failure analysis of components and pit-manufacturing-unit operations. The various analytical chemistry analyses required for materials characterization and failure analysis are shown in Table 16. Also shown in Table 16 is the precision required for the analytical analysis. The expected numbers of samples are shown in Table 17. The numbers of analytical instruments and glovebox enclosures required are shown in Table 18. In some cases, duplicate instruments are required for materials characterization and failure analysis area. Duplicate instruments are needed to analyze both fissile and nonfissile components.

Table 16. Analytical Chemistry Requirements for Materials Characterization and Failure Analysis

Required Analysis	Analytical Technique	Analytical Instrument	Required Precision (% Rel. Std. Dev.)
Sample Preparation	Etching	Etching	NA
Sample Preparation	Grinding	Grinder	NA
Sample Preparation	Polishing	Polisher	NA
Surface Analysis	Metallography	Metallograph	NA
Surface Analysis	Microscopy	SEM[a]	NA
Surface Analysis	Microhardness	Microhardness Tester	NA
Surface Analysis	X-Ray Diffraction	XRD[b]	NA
Surface Analysis	X-Ray Spectroscopy	XPS[c]	NA
Surface Analysis	Microscopy	TEM[d]	NA
Surface Analysis	Auger Spectroscopy	Auger	NA

[a]SEM—scanning electron microscope.
[b]XRD—X-ray diffractometer.
[c]XPS—X-ray photoelectron spectroscope.
[d]TEM—transmission electron microscope.

Table 17. Expected Number of Samples from Materials Characterization and Failure Analysis

Location	# Samples
Nuclear	125
Nonnuclear	75

Table 18. Instrument and Glovebox Enclosure Requirements for Materials Characterization and Failure Analysis							
Analysis	**# Samples**	**Instrument**	**# Instru-ments**	**# Std. GBXs**	**# Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Sample Preparation	125	Etcher	2	1	1	0	0
Sample Preparation	125	Polisher	3	2	2	0	0
Sample Preparation	125	Grinder	4	2	2	0	0
Surface Analysis	125	Metallo-graph	3	2	3	0	1
Surface Analysis	25	SEM	2	1	0	0	1
Surface Analysis	25	Microprobe	1	1	0	0	1
Surface Analysis	75	Microhard-ness Tester	2	1	0	0	1
Surface Analysis	25	XRD	2	1	0	0	1
Surface Analysis	25	XRF	2	0	0	0	1
Surface Analysis	10	TEM	2	2	0	0	1
Surface Analysis	10	Auger	2	2	0	0	0

3.7 War Reserve Process Materials Testing

WR process material testing involves accepting and certifying process materials used in pit-manufacturing operations, which includes analyzing the interaction of process materials with fissile and nonfissile components. This activity is a subset of activities called "materials compatibility." The various analytical chemistry analyses required for WR process materials testing are shown in Table 19. Also shown in Table 19 is the precision required for the analysis. The expected numbers of samples are shown in Table 20. The required number of analytical instruments and glovebox enclosures is shown in Table 21. In some cases, duplicate instruments are required for WR materials testing. Duplicate instruments are needed for analyzing both fissile and nonfissile components.

Table 19. Analytical Chemistry Requirements for War Reserve Process Materials Testing			
Required Analysis	**Analytical Technique**	**Analytical Instrument**	**Required Precision (% Rel. Std. Dev.)**
Surface Analysis	Infrared	FT-IR	± 10
Organic Residues	Organic	GC-MS	± 15
Surface Cleanliness	Emission	OSEE*	± 15
Surface Analysis	Microscopy	SEM	NA
Surface Analysis	X-Ray Diffraction	XRD	NA
Surface Analysis	X-Ray Spectroscopy	XPS	NA
Corrosion	Potentiometry	Potentiostat	± 10
*OSEE—Optically stimulated electronic emission.			

Table 20. Expected Number of Samples from War Reserve Process Materials Testing	
Location	**# Samples**
Fissile	100
Nonfissile	100

Table 21. Instrument and Glovebox Enclosure Requirements for War Reserve Process Materials Testing							
Analysis	**# Samples**	**Instrument**	**# Instru-ments**	**# Std. GBXs**	**# Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Surface Analysis	100	FT-IR	2	2	0	1	0
Organic Residues	50	GC-MS	2	2	0	4	2
Surface Clean-liness	75	OSEE	2	1	0	1	2
Surface Analysis	20	SEM	2	1	0	0	1
Surface Analysis	20	XRD	2	1	0	0	1
Surface Analysis	20	XRF	1	0	0	0	1
Acceler-ated Corrosion	75	Poten-tiostat	1	0	0	1	1

4.0 ANALYTICAL CHEMISTRY INFRASTRUCTURE REQUIREMENTS

Five unique infrastructure activities are essential for executing analytical chemistry for pit manufacturing. The five areas are (1) a laboratory information management system, (2) sample management, (3) data reporting, (4) report archiving, and (5) data quality assurance. These areas require equipment and staffing in order to successfully execute analytical chemistry to support pit manufacturing.

4.1 Laboratory Information Management System

A laboratory information management system (LIMS) is a capability required to track the analytical chemistry samples generated from pit manufacturing. Table 22 shows the necessary equipment required for a LIMS.

Table 22. Equipment Required for Laboratory Information Management System

Equipment	# Units
Unclassified LIMS Server	1
Classified LIMS Server and Distribution Network	1
Network Personal Computers	75
Barcode Printers	5
Barcode Readers	10
Network Terminals	75

4.2 Sample Management

Sample management involves managing the paperwork associated with analytical chemistry samples and physically moving samples to various analysis areas. This activity mainly requires adequate staffing to manage the flow of samples originating from the various pit-manufacturing-unit operations. Some classified and unclassified personal computers would be required for the staff who manage this activity.

Gloveboxes are also required for sample management and distribution. Glovebox requirements for sample management are shown in Table 23.

Table 23. Summary of Glovebox Enclosures Required for Sample Management	
Type	**Number**
Standard GBXs	5
Specialty GBXs	2
Open Fronts	9
Fume Hoods	3

4.3 Data Reporting

Analytical chemistry data generated by analytical instruments and analysts must be prepared as a data package for the customer. This activity mainly requires adequate staffing to perform these functions. Some classified and unclassified personal computers would be required for the staff who manage this activity.

4.4 Data Archiving

Data archiving is required for long-term storage of the data packages for pit manufacturing. This activity mainly requires adequate staffing to archive data as requested by the pit-manufacturing customer. Some classified and unclassified personal computers would be required for the staff who manage this activity. Adequate space for physical storage of the reports would be required as well.

4.5 Quality Assurance

Quality assurance involves ensuring that the analysis and data packages meet the quality and reporting requirements for pit manufacturing. This activity mainly requires adequate independent staff to provide quality assurance of the analytical chemistry measurement techniques and the quality of the reported data. Some classified and unclassified personal computers would be required for the staff who manage this activity.

5.0 SUMMARY OF INSTRUMENTS, GLOVEBOX ENCLOSURES, AND ASSOCIATED COSTS FOR PIT MANUFACTURING

Summaries of analytical instrumentation and associated glovebox enclosures are provided in this section. A summary of the analytical chemistry instrumentation and glovebox enclosures required to support pit manufacturing is shown in Table 24. A summary of estimated costs of analytical instruments is shown in Table 25. A summary of the estimated costs for glovebox enclosures is shown in Table 26.

Table 24. Summary of Analytical Instruments and Glovebox Enclosures Required for Pit Manufacturing					
Analytical Instruments	**# Instruments**	**# Std. GBXs**	**#Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Hot Plate	12	3	0	1	1
Coulometer	1	4	1	5	1
Autotitrator	1	4	5	4	1
TIMS	1	4	0	1	1
ICP-MS	1	1	1	2	2
ICP-AES	1	1	1	2	2
XRF	1	0	0	2	2
Colorimeter	1	0	1	6	1
Gamma Spectrometer	2	2	0	1	0
Proportional Counter	1	2	0	1	0
Alpha Spectrometer	1	2	0	0	0
Thermal Conductivity Analyzer	3	3	3	1	1
Gas Mass Spectrometer	1	2	1	1	1
Microwave	3	6	0	1	1
Liquid Scintillation Counter	1	2	0	1	1
Germanium Detector	2	2	0	1	1
Plutonium III Spectrophotometer	1	6	1	3	1
Direct-Current Plasma	1	2	0	1	1
Ion Chromatograph	1	0	0	2	1
Visible Region Spectrometer	1	1	0	6	1
Furnace	1	5	0	2	0
GC-MS (VOA)	2	2	0	2	2
GC-MS (SVOA)	3	1	0	2	1
GC-MS (Materials)	2	2	0	4	0
FT-IR	2	2	0	1	2
GC-FID	1	0	0	0	2
Etcher	2	1	1	0	0
Grinder	4	2	2	0	0
Polisher	3	2	2	0	0
Metallograph	3	2	3	0	1
SEM	2	1	0	0	1

Table 24. Summary of Analytical Instruments and Glovebox Enclosures Required for Pit Manufacturing (cont)					
Analytical Instruments	**# Instruments**	**# Std. GBXs**	**#Spec. GBXs**	**# Open Fronts**	**# Fume Hoods**
Microhardness Tester	2	1	0	0	1
XRD	2	1	0	0	1
TEM	2	2	0	0	0
Auger	2	2	0	0	0
OSEE	2	1	0	1	2
Potentiostat	2	0	0	1	1
Classified LIMS Server and Distribution Network	1	NA	NA	NA	NA
Network PCs	75	NA	NA	NA	NA
Barcode Printers	5	NA	NA	NA	NA
Barcode Readers	10	NA	NA	NA	NA
Network Terminals	75	NA	NA	NA	NA
GRAND TOTAL	240	75	22	55	35

Table 25. Summary of Estimated Costs for Analytical Instruments			
Analytical Instruments	**# Instruments**	**Unit Cost (Thousands)**	**Total Cost (Thousands)**
Hot Plate	12	0.1	1.2
Coulometer	1	50	50
Autotitrator	1	50	50
TIMS	1	500	500
ICP-MS	1	350	350
ICP-AES	1	250	350
XRF	1	350	350
Colorimeter	1	25	25
Gamma Spectrometer	2	40	80
Proportional Counter	1	30	30
Alpha Spectrometer	1	50	50
Thermal Conductivity Analyzer	3	100	300
Gas Mass Spectrometer	1	500	500
Microwave	3	40	120
Liquid Scintillation Counter	1	50	50
Germanium Detector	2	110	220
Plutonium III Spectrophotometer	1	50	50

Table 25. Summary of Estimated Costs for Analytical Instruments (cont)			
Analytical Instruments	**# Instruments**	**Unit Cost (Thousands)**	**Total Cost (Thousands)**
Direct-Current Plasma	1	200	200
Ion Chromatograph	1	50	50
Visible-Region Spectrometer	1	75	75
Furnace	1	50	50
GC-MS (VOA)	2	100	200
GC-MS (SVOA)	3	100	300
GC-MS (Materials)	2	100	200
FT-IR	2	30	60
GC-FID	1	50	50
Etcher	2	50	100
Grinder	4	40	160
Polisher	3	20	60
Metallograph	3	60	180
SEM	2	400	800
Microprobe	1	750	750
Microhardness Tester	2	50	100
XRD	2	150	300
TEM	2	500	1,000
Auger	2	500	1,000
OSEE	2	25	50
Potentiostat	2	50	100
Classified LIMS Server and Distribution Network	1	400	400
Network PCs	75	2	150
Barcode Printers	5	2	10
Barcode Readers	10	2	20
Network Terminals	75	1	75
		Subtotal	10,715
	Installation @ 50%		5,378
		Total	16,093

Table 26. Summary of Estimated Costs for Glovebox Enclosures			
Type of Enclosures	**# Enclosures**	**Unit Cost (Thousands)**	**Total (Thousands)**
Standard GBXs	80	25	2,000
Specialty GBXs	24	50	1,200
Open Fronts	64	40	2,560
Fume Hoods	38	10	380
		Subtotal	6,140
	Installation @ 50%		2,456
		Total	8,596

6.0 ANALYTICAL CHEMISTRY TECHNOLOGY DEVELOPMENT

This section identifies development activities that are being developed or should be developed to benefit pit manufacturing. Pursuing these analytical chemistry technologies will benefit pit manufacturing by improving the following: analytical precision, data delivery, and limits of detection of analytical chemistry techniques.

6.1 Laser Ablation ICP-MS

Benefits of the Technique

Laser ablation ICP-MS would eliminate the current practice of using acid for dissolving samples analyzed for fissile and nonfissile metals and oxides, which, in turn, would eliminate the acid waste streams that result from currently used sample preparation techniques. This technique also allows samples to be analyzed more rapidly.

Drawbacks of the Technique

This technique may not be able to detect trace element concentrations at levels as low as those achieved by conventional acid dissolution and ICP-MS analysis. In addition, sample heterogeneity may reduce the precision of laser ablation ICP-MS.

6.2 Glow Discharge Mass Spectrometry

Benefits of the Technique

Glow discharge mass spectrometry (GDMS) eliminates the need to dissolve fissile and nonfissile oxides. This technique may not be able to detect certain trace elements at levels as low as those achieved by conventional acid dissolution and ICP-MS analysis. This technique also allows samples to be analyzed more rapidly.

Drawbacks of the Technique

The GDMS technique is mainly used for analyzing oxides and powders and would have to be adapted to analyze metals as well.

6.3 Gradient Ion Chromatography

Benefits of the Technique

Gradient ion chromatography will measure trace ionic impurities in complex matrices, including those in radioactive samples. The technique will also provide more efficient analysis of both anionic and cationic species in samples.

Drawbacks of the Technique

The technique has no drawbacks.

6.4 Advanced Process Analytical Chemistry Sensors

Benefits of the Technique

Advanced process analytical chemistry sensors provide the ability to analyze, monitor, and control actinide-processing operations.

Drawbacks of the Technique

The technique has no drawbacks.

6.5 Direct-Current Arc Spectroscopy

Benefits of the Technique

This technique permits development of a more efficient trace analysis capability for analyzing transuranic wastes and avoids using acids to dissolve samples.

Drawbacks of the Technique

This technique for analyzing plutonium samples requires the use of silver, which generates a waste managed under the Resource Conservation and Recovery Act.

6.6 Advanced Etching

Benefits of the Technique

Advanced etching involves developing techniques used in metallography that can eliminate the use of hazardous etching chemicals.

Drawbacks of the Technique

The technique has no drawbacks.

6.7 Metallographic Sample Preparation Using a Diamond Lathe

Benefits of the Technique

This technique will minimize the need to polish metallographic samples.

Drawbacks of the Technique

Use of this technique will necessitate generating diamond bits.

6.8 Supercritical CO_2 Cleaning

Benefits of the Technique

The technique eliminates the need to use conventional solvents for cleaning radioactive and nonradioactive parts. As a result, this technique will also minimize waste from cleaning activities.

Drawbacks of the Technique

A potential hazard is associated with the high pressures used in the technique.

6.9 Development of Radiochemistry Capability at TA-55

Benefits of the Technique

We plan to increase the radiochemistry capability at TA-55 by developing a high-resolution gamma ray analysis that uses germanium detection and a nontypical analysis that uses liquid scintillation. These techniques will provide isotopic information to TA-55 at a lower cost and with faster turnaround time. They will improve the flexibility of analyzing difficult samples, such as plutonium mixed with oils, and will use liquid scintillation to facilitate analyzing samples having a high salt concentration.

Drawbacks of the Technique

The technique requires obtaining space in PF-4 at TA-55 and obtaining development funding.

6.10 Development of Nondestructive Assay Capability for Accountability at CMR

Benefits of the Technique

In collaboration with NMT-4, we propose to develop improved nondestructive assay capabilities at CMR for verification and conformation measurements. This technique would minimize at its origin the volume of waste produced at the CMR building.

Drawbacks of the Technique

Cross-cutting issues with the Facilities Engineering Division and waste management groups need to be resolved. We also need to be certain that we can meet Waste Isolation Pilot Plant acceptance criteria.

6.11 GC-MS SVOC Apex Technology Development

Benefits of the Technique

The technique would reduce waste and improve the sensitivity of analyses.

Drawbacks of the Technique

Use of larger quantities of samples would increase personnel exposure to radioactive materials.

6.12 GC-MS SVOC Accelerated Solvent Extraction

Benefits of the Technique

The technique would improve automation, waste reduction, and sample analysis for SVOC analysis.

Drawbacks of the Technique

Safety issues pertaining to pressurized solvents have yet to be resolved.

6.13 Automated Davies-Grey Titration

Benefits of the Technique

Automating the technique will improve analysis time.

Drawbacks of the Technique

The technique has no drawbacks.

6.14 Hybrid Densitometry Development

Benefits of the Technique

This instrument is a combination X-ray fluorescence and absorption spectrometer that provides semiquantitative to quantitative plutonium and uranium assay measurements. The main advantage is rapid analysis.

Drawbacks of the Technique

This technique does not have the precision and detection limits provided by conventional plutonium and uranium assay techniques. However, many of the needs of actinide-processing operations can be met by using this technique.

6.15 Precision Cutting

Benefits of the Technique

Improving precision-cutting techniques will increase the precision in cutting samples, will reduce sample-cutting time, and will reduce personnel exposure.

Drawbacks of the Technique

The technique has no drawbacks.

6.16 Micro X-Ray Fluorescence

Benefits of the Technique

This technique will allow trace elements in actinides to be analyzed at part-per-billion levels. The technique also uses small quantities of samples.

Drawbacks of the Technique

This technique may not be required for pit-manufacturing operations; however, we may be able to use it for analyzing waste streams generated by actinide-processing operations and waste management activities.

6.17 ICP-AES/ICP-MS Method Development

Benefits of the Technique

This technique will allow integration of concentric nebulizers and direct-injection nebulizers in mass spectrometers to segregate wastes generated from sample analysis.

Drawbacks of the Technique

The technique has no drawbacks.

6.18 Interstitial Analysis Laboratory

Benefits

This new laboratory will provide the ability to test for carbon, nitrogen, oxygen, and sulfur in plutonium metal.

Drawbacks

There are no drawbacks.

6.19 Plutonium Standards Development

Benefits of the Technique

We propose to fabricate certified reference material for plutonium metal standards to analyze plutonium metal for pit manufacturing.

Drawbacks of the Technique

The technique has no drawbacks.

6.20 Laser-Coupled Time-of-Flight Mass Spectrometry

Benefits of the Technique

The technique has the advantages of rapid sample throughput, low detection limits (<1 ppm for most elements), and a large dynamic range. The technique also has the ability of depth-profiling analysis and has a mass resolution of up to 2,000, which is better than that of the quadrupoles used in ICP-MS. This technique could also be adapted to provide actinide analysis on line or at line in a glovebox environment.

Drawbacks of the Technique

The drawback of the technique is that the heterogeneity of the samples interferes with the precision of the analysis, which could render the analytical sensitivity and detection limits inferior to those of ICP-MS. The instrument is currently being developed, and some interfacing with a computer system would be required to make it an operator friendly instrument.

6.21 Thermal Ionization Cavity-Coupled Mass Spectrometry

Benefits of the Technique

This cavity-type ion source is an improvement on the ribbon- or filament-type ion source commonly used in thermal ionization mass spectrometry. The instrument is more compact and has an improved ionization source compared with a commercial TIMS instrument. An improvement in ionization efficiency permits better characterization of small samples and an improvement in detection limits, depending on what type of mass spectrometer is coupled to the ionization source.

Drawbacks of the Technique

The instrumentation is in the development phase. No plans have been made to couple the ionization source to a time-of-flight mass spectrometer or single-focusing or double-focusing magnetic sector instrument. The detection limits will vary, depending on the type of mass spectrometer coupled to the source. More fundamental work on ion transport and generation probably needs to be done to turn this technique into a operator friendly instrument.

7.0 SUMMARY

Approximately $16 million is required to procure analytical instrumentation by the year 2004 to support a 50-pit-per-year pit-manufacturing mission. In addition, $8.5 million is required to procure glovebox enclosures. An estimated 50% increase in installation costs has been included for installation of analytical instruments and glovebox enclosures. However, no G&A taxes have been included. If an additional 42.5% G&A tax were included, approximately $35 million would be required over the next 5 years to prepare analytical chemistry for support of a 50-pit-per-year manufacturing mission by the year 2004. Additional funding would also be required for introduction hoods and bagout locations on the glovebox lines. The number of additional glovebox enclosures depends on the layout of the analytical instruments and their respective gloveboxes.

Based on the estimated sample types and predicted sample numbers for pit manufacturing, most of the samples can be analyzed within 30 days. However, this estimated analysis time is applicable only for the analysis of samples originating from pit manufacturing. The 30-day turnaround analysis time is based on the TSA-7 model of analytical chemistry process efficiencies that uses a "single analytical instrument" scenario. In addition, several samples fall outside a 30- day time frame. However, these samples are isolated cases and would probably have minimal impact on the pit production schedule.

Another outstanding issue is hiring qualified analytical chemistry personnel over the next decade to support pit manufacturing. Part of this effort should be maintaining and transferring the corporate memory of retiring senior scientists to new personnel, which means hiring personnel early and allowing sufficient lead times for training them. From a programmatic standpoint, this approach would require a considerable amount of planning and infusion of funding into the infrastructure of analytical chemistry. Maintenance of skilled personnel is essential to analytical chemistry and its success in supporting the manufacturing mission.

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