Idaho National Laboratory

AUTOMATED AND MANUAL GAS PRESSURIZED EXTRACTION CHROMATOGRAPHY	Identifier: AL-4270-OI-001 Revision: 4 Effective Date: 06/06/17 Page: 1 of **17**

Materials and Fuels Complex	Laboratory Instruction	**USE TYPE 4**	eCR Number: 649165

Manual: MFC Analytical Laboratory Manual

PROCEDURE REVIEW REQUIREMENTS PER SP-20.1.4

DISCIPLINE	REVISION	DISCIPLINE	REVISION
ANALYTICAL RESEARCH LABORATORIES (AL, RCL)	X	MAINTENANCE	N/A
CHARACTERIZATION & ADVANCED PIE (EML, IMCL, SPL)	N/A	NUCLEAR SAFETY REVIEW	N/A
FUEL FABRICATION & NUCLEAR MATERIAL MANAGEMENT (EFF, FASB, FMF, ZPPR)	N/A	OUTSIDE REVIEW	N/A
POST-IRRADIATION EXAMINATION (HFEF, NRAD, MOCK-UP)	N/A	PACKAGING & TRANSPORTATION	N/A
PRODUCTION FACILITIES & INFRASTRUCTURE (BOP, FCF, RDF, INTEC, RSWF/TSDF, TREAT-WH)	N/A	QUALITY ASSURANCE	N/A
SPACE NUCLEAR POWER & ISOTOPE TECHNOLOGIES (SSPSF, 751, 772, 796, INTEC [RSTSF & 1634])	N/A	RADIOLOGICAL CONTROLS	*
CUI REVIEW	N/A	REMOTE-HANDLED LOW LEVEL WASTE (RH-LLW)	N/A
ENGINEERING	N/A	SAFEGUARDS & SECURITY	N/A
ENVIRONMENTAL	*	SAFETY & HEALTH (IH, IS)	*
FIRE PROTECTION	*	TRAINING	*
HOISTING AND RIGGING	N/A	WASTE GENERATOR SERVICES	*
INTER-FACILITY TRANSFERS	N/A		

*DOCUMENT OWNER OR QUALIFIED REVIEWER SHALL DETERMINE THE NEED FOR THESE REVIEWS BASED UPON THE SCOPE OF THE CHANGE AND THE HAZARDS IDENTIFIED

REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	02/13/08	All	See eCR 556684. New issue.
1	11/09/10	All	See eCR 585399. Revision.
2	03/28/11	All	See eCR 589834. Revision and periodic review.
3	09/13/11	All	See eCR 595137. PFC.
4	06/06/17	All	See eCR 649165. Revision and periodic review.

1. PURPOSE/SCOPE/APPLICABILITY

This document supplies instructions for operation of the automated and manual gas-pressurized-extraction chromatography (GPEC) equipment located in the Analytical Laboratory. Several GPEC systems have been set up for operation in gloveboxes, hoods, and on the laboratory bench tops (for non-radiological samples only). The system separates elemental components contained in solutions to facilitate and improve measurements by eliminating interfering components.

Typically, liquid samples for separation are <10 mL in volume. Samples for separation are prepared under different work control.

The GPEC equipment utilizes pressurized inert (N_2, Ar, He) gas (<100 psig) to force small volumes of solution that are typically <1 mL (0.033 oz) through small diameter tubing (typically 1/16-in. outside diameter and 0.02-in. inside diameter) to a piece of tubing that traps a resin material between filters. Typical gas flow rates are <10 mL/min. The system includes a peristaltic pump (low pressure) and miniature multi-port valves.

Separation is a cyclic operation that is repeated for each sample. There are four basic phases: load, rinse, strip, and recondition. During the load phase, a fixed volume of tubing is filled with a sample solution that is forced through another fixed volume of tubing (by compressed gas) containing a resin material with filters at each end. Certain constituents in the sample are separated and collected in the resin. The rinse phase involves filling the sample tubing with clean acid in the same matrix as the sample. This process cleans any remaining constituents from the tubing and forces it through the column. During the strip phase, a different acid, or a different concentration of acid is used to fill the tubing. This acid is forced through the column, stripping off the constituents of the sample that were held up in the resin. After completion of the strip phase, the column is reconditioned for the next sample by refilling the loop with clean acid (in the matrix of the sample) and forcing the acid through the column.

Sample solutions may be corrosive and/or contain a variety of elements (radioactive, non-radioactive, or Resource Conservation and Recovery Act [RCRA] metals). Specific separations, such as cesium from barium, uranium from impurities, etc., are delineated by a collection of special instructions that were created using FRM-900, “Analytical Laboratory Special Instructions.” The special instructions are contained in a binder located in AL administrative office.

Sample solutions may be prepared using, but are not limited to, materials containing salts, metallic or ceramic waste forms, sealed source materials, waste-related materials, RCRA metals, smears, and a variety of irradiated or unirradiated nuclear fuels as well as samples from processes that treat nuclear fuels. This procedure does not cover work with samples containing any insoluble beryllium, beryllium compound, or any alloy containing $\geq$0.1% beryllium that may be released as an airborne particulate.

This procedure is performed by personnel qualified to work in the Analytical Laboratory per the training listed in Subsection 2.1.

The activities directed by this procedure have been designated Quality Level 3 per Quality Level Determination MFC-000503.

2. RISK AND CONTROLS

Sequence of Basic Activities	Potential Hazard	Hazard Control
1. Handling and storage of compressed gas cylinders. • Loading and transporting cylinders • Connecting • Storage.	1a. Back injury/strained muscles, injury to feet/hands.	1a. 1) Lift more than 50 lb or 1/3 of body weight, whichever is less, is not permitted. 2) Movement of heavy cylinders is not to be done by a single performer. 3) Gas cylinder cart used for transport of cylinders. 4) Upright spin-roll technique to move cylinders by hand when necessary.
	1b. Falling cylinders.	1b. 1) Safety shoes. 2) Valve cap in place. 3) Transport device or suitable vehicle to transport cylinders. 4) Cylinders secured to transport device.
	1c. Exploding or flying parts under pressure, damage to cylinder.	1c. 1) Oil is not used on threads. 2) Correct regulators. 3) Personnel will stand to side and open valve slowly. 4) Safety glasses. 5) Cylinders secured to prevent falling over.
2. Operating GPEC with solutions containing chemicals.	2a. Exposure to corrosive chemicals.	2a. 1) GPEC operations are performed in a hood, glovebox, or in the hot cells. 2) When working with chemicals, an operable and readily available emergency shower and eyewash station is required. 3) Read and understand the SDS information provided for each chemical. 4) Do not use cellulose containing materials with nitric acid.

Sequence of Basic Activities	Potential Hazard	Hazard Control
		5) In the laboratory hood, use nitrile, or neoprene gloves unless other glove material is required by IH. 6) Wear safety glasses with side shields, lab coat and substantial shoes. Wear additional PPE as required by the RWP.
	2b. Exposure to oxidizing chemicals.	2b. 1) Oxidizing chemicals are segregated from other incompatible chemicals and materials. 2) Cellulose materials may not be used with oxidizing chemicals. 3) Handling should be completed in a fume hood or glovebox unless an IH-completed exposure assessment identifies other suitable locations. 4) SDS information provided for each chemical. 5) Safety glasses with side shields, lab coat, and substantial shoes and additional PPE as required by the RWP.
	2c. Exposure to flammable or combustible chemicals.	2c. 1) SDS information provided for each chemical. 2) Nitrile or neoprene gloves unless other glove material is required by IH. 3) Safety glasses with side shields, lab coat, and substantial shoes and additional PPE as required by the RWP.
3. Working in a known or suspected CA, HCA, or ARA.	3. Exposure to contamination and/or airborne radioactivity.	3. 1) Containment or use of localized ventilation to mitigate the potential for contamination spread to the adjacent area or airborne exposure to personnel. 2) Decontamination to reduce work area contamination levels, when directed by HPT. 3) RWP.

Sequence of Basic Activities	Potential Hazard	Hazard Control
4. Working in a known or suspected RA or HRA.	4. Radiation exposure.	4. 1) ALARA shielding or temporary shielding to maintain personnel dose ALARA when needed. 2) RWP. 3) All tools, parts, materials, etc., staged prior to entering a RA (or HRA, if applicable) to reduce time personnel are in the area. 4) When possible, use of tools (tongs, pliers, etc.) to increase distance to reduce extremity exposure.
5. Handling hydrofluoric acid.	5. Personnel exposure to a toxic/corrosive material.	5. 1) Neoprene or butyl rubber gloves; safety glasses with secondary barrier (for example, hood sash, face shield, or goggles), safety shoes, and lab coat. 2) Calcium gluconate gel will be available in working area when working with HF. 3) In a hot cell, no PPE is required. 4) Chemical preparations will be completed in a fume hood unless an IH-completed exposure assessment identifies other suitable locations. 5) Handling shall only be performed in areas where an emergency eyewash and shower station is operational and accessible.

2.1 Certification/Qualification/Training Required

2.1.1 AL Lab Worker

- Analytical Laboratory Job Worker Job Code, RDLYMFNP01
- Analytical Laboratory General Core, OJT Checklist No. AFALOOO1
  AND/OR
  Analytical Laboratory Hot Cell Core, OJT checklist ID No. AFAL0002
- GPEC OJT Checklist, AFAL0059
- LST-493.

2.2 Precautions and Limitations

NOTE: *This section applies to risks not necessarily involving hazards to personnel.*

Table 2. Risks

Applies	Activity/Task	Risk	Engineering Control	Administrative Control
YES	1. Sample Management	Conditions adverse to quality, incorrect categorization of waste. Violation of a written requirement.	Sample containers/ cabinets/locations.	MCP-3364
YES	2. Generating Waste	Damage to the environment. Violation of a written requirement.	Waste containers/ cabinets/locations.	MCP-3365
YES	3. Generating Air Emissions	Air emissions above acceptable limits. Violation of a written requirement.	Facility ventilation system.	FRM-1048 SD-40.1.5 Environmental Review on procedures creating air emissions (per LWP-21220)

Applies	Activity/Task	Risk	Engineering Control	Administrative Control
YES	4. Activities involving Quality	Inaccurate/unacceptable results, re-work, loss of unique samples. Violation of a written requirement.	Facility systems, SSCs, instruments, labware, chemicals, consumables	PDD-13000
YES	5. Performing Work	Incomplete or inadequate planning and execution of work, unforeseen hazards and risks. Violation of a written requirement.	NA	LWP-13850
YES	6. Housekeeping	Fire hazard, contamination control, chemical control, radiological control.	Storage containers/ cabinets/locations,	LWP-14620 LWP-9600
YES	7. Radiological Work	Inadequate surveys or postings. Violation of a written requirement.	Radiological instruments	FRM-441.49 FRM-441.49A MCP-139 MCP-187
NO	8. Work that impacts a SAR/TSR-401 requirement	SAR/TSR violation	Safety-Significant SSCs, engineered systems.	TSR-, SAR-Procedures, facility access training. QFMH Training SP-20.1.4

2.2.1 Replacement of columns, fixed volume loops, or other tubing should be followed by a leak test before performing separations containing hazardous liquid or radioactive nuclides.

3. PREREQUISITES

3.1 Notifications

3.1.1 Notify affected organizations (Waste Generator Services [WGS], Environmental, Quality, Life Safety, etc.) as applicable.

3.1.1.1 IF waste will be generated,
THEN ensure waste stream profiles have been established and satellite accumulation areas established as necessary with WGS for the type of waste being generated, per MCP-3365, "Waste Management at the Analytical Laboratory."

3.2 Radiological Conditions

3.2.1 IF a radiological work permit (RWP) is required,
THEN ensure the appropriate RWP has been completed and is available prior to start of work per MCP-15009, "Radiological Work Permit Preparation."

3.3 Briefing

3.3.1 Conduct briefing per LWP-9201, "Briefings."

3.3.2 IF working with new radiological material,
THEN discuss expected radiological conditions (for example; expected neutron exposure, high radiological exposure, total activity of the sample) with the AL radiological engineer.

3.4 Staging Activities

3.4.1 Ensure that the gas cylinder (nitrogen, argon, or helium) has >300 psig of pressure remaining.

3.4.2 Ensure the regulated pressure supplied to the system to actuate valves and pressurize the liquid is ≤100 psig.

3.4.3 Ensure FRM-900, "Analytical Laboratory Special Instructions," are available.

4. FACILITY CONDITIONS

4.1 The applicable radiological monitoring equipment and ventilation systems are operational.

5. INSTRUCTIONS

NOTE: *The steps in Section 5 can be altered at the discretion of the analyst to logically complete various separation schemes. The special instructions form, FRM-900, must be referred to for specific instructions regarding each separation performed.*

5.1 Routing Tables

5.1.1 IF any of the following sections need to be performed,
GO TO that section and perform as directed,
THEN return to this step.

Description	Subsection
Automated GPEC System	5.2
Sample Placement and Separation	5.2.1
Set-up of Computer Controlled Separations via Labview Program	5.2.2
Sample Removal and System Clean-up/Storage	5.2.3
Manual Mini-GPEC System—Separation Performance	5.3
Separation Performance	5.3.1

5.2 Automated GPEC System

5.2.1 Sample Placement and Separation

5.2.1.1 Remove the uptake rack from autosampler No. 1.

5.2.1.2 Remove the caps from the sample tubes, remembering to put them in the same order as the tubes are aligned.

5.2.1.3 Arrange samples in the autosampler rack.

5.2.1.4 Ensure that appropriate acids from rinses, strips, and reconditions are in the correct position.

5.2.1.5 Return loaded rack to the autosampler and close the door.

5.2.1.6 Place the collection tubes in the collection autosampler, ensuring that the positions of the collection tubes are appropriate.

5.2.1.7 Verify that the waste cups in the collection autosampler are empty.

5.2.1.8 Verify that the drain bottles located behind the uptake autosampler have enough room to hold the liquid waste from the rinsing of the loop.

5.2.1.9 Verify that the auto-sampler flowing rinse bottle containing 5% HNO_3 is ≥1/4 full.

5.2.2 Set-up of Computer Controlled Separations via Labview Program

5.2.2.1 Set up program and run separations.

5.2.3 Sample Removal and System Clean-up/Storage

5.2.3.1 AFTER the separation program is complete,
THEN place the appropriate caps on the corresponding sample tubes.

NOTE: *Step 5.2.3.2 is performed to minimize corrosion of the autosampler parts.*

5.2.3.2 Either empty or cap the rinse and strip solutions from the uptake autosampler.

5.2.3.3 WHEN appropriate,
THEN set-up a cleaning procedure to ensure the system is ready for the next separations.

5.3 Manual Mini-GPEC System—Separation Performance

NOTE 1: *Refer to Appendix* A*, Flow Schematics for the Manual Mini-GPEC System.*

NOTE 2: *Compressed nitrogen, argon, or helium may be used.*

5.3.1 Separation Performance

5.3.1.1 Set the gas regulator (GR2) to 20–60 psig.

5.3.1.2 Adjust the gas flow rate using GMV1 to approximately 10 ml/min.

5.3.1.3 Position GSV1 to OFF.

5.3.1.4 Position IV1 to LOAD.

5.3.1.5 Position DV1 to NORMAL position.

5.3.1.6 Start the pump.

5.3.1.7 Place a collection tube at the discharge of DV1.

5.3.1.8 Place the uptake tube into the liquid to fill the loop. (A liquid flow rate of about 1 mL/min is adequate.)

5.3.1.9 Position GSV1 to ON.

5.3.1.10 Position IV1 to INJECT.

5.3.1.11 Remove the uptake tube from the liquid and place into an empty tube.

5.3.1.12 Observe the flow rate. (Liquid should be moving from the loop through the column and into the fraction collection tube positioned at the discharge of DV1.)

5.3.1.13 Collect the liquid into the fraction collection tube until approximately 30 seconds have passed since the liquid cleared the column.

5.3.1.14 Position GSV1 to OFF.

5.3.1.15 Position IV1 to LOAD.

5.3.1.16 Repeat Steps 5.3.1.7 through 5.3.1.15, changing the liquid at the uptake position and the collection tubes as needed, to complete the separation scheme.

5.3.1.17 AFTER completing work with the Manual GPEC system, THEN stop the pump and shut off the gas flow.

6. POST-PERFORMANCE ACTIVITIES

6.1 Perform housekeeping and properly store chemicals and equipment not in use.

6.1.1 Clean up and wipe down the area where the work was performed (hood/glovebox/hot cells).

6.1.2 HPT: Perform a post-job survey.

6.2 Dispose of or store waste per MCP-3365.

6.2.1 Dispose of materials used into the proper waste stream, as directed by WGS.

6.3 Manage samples per with MCP-3364, "Sample Management at the Analytical Laboratory."

6.3.1 Return dilutions/separations to origination point of parent solution.

6.3.2 Contact WGS and the MBA custodian, as necessary, for determination of final disposition of samples.

6.4 IF the activity required the use of forms and/or logs,
THEN ensure the forms and/or logs are updated appropriately.

7. ABNORMAL OPERATIONS

7.1 IF abnormal conditions arise during the performance of this procedure,
THEN stop work, place the system in a safe configuration, and immediately notify the AL shift supervisor (SS).

7.2 If necessary, respond to abnormal facility conditions in accordance with:

MFC-ONRI-0001, "All Facilities Response to MFC Anomalies"

MFC-AOP-0001, "MFC All Facilities Response to Abnormal Operations"

AL-ONRI-0001, "AL Response to Anomalies."

8. RECORDS

Laboratory notebooks are kept per PLN-2224, "Records Management Plan for Nuclear Science and Technology."

Executed copies of laboratory instructions or operational forms.

NOTE: *LWP-1202, "Records Management," the INL Records Schedule Matrix, and associated record types list(s) provide current information on the retention, quality assurance, and/or destruction moratorium requirements for these records. Contact a Records Coordinator for assistance if needed.*

9. REFERENCES

AL-ONRI-0001, "AL Response to Anomalies"

Forms:

Form 441.49, "Radiological Work Permit (Single Radiological Hazard Task)"

Form 441.49A, "Radiological Work Permit (Multiple Radiological Hazard Tasks)"

FRM-1048, "Research Facilities NESHAP Data Tracking Sheet for Radiological Materials Heated to Greater than 100C"

FRM-900, "Analytical Laboratory Special Instructions"

LRD-14118, "Personal Protective Equipment"

LWP-1202, "Records Management"

LWP-13850, "Processing Lessons Learned and Operating Experience"

LWP-14620, "Chemical Hygiene Plan"

LWP-21220, "Work Management"

LWP-9201, "Briefings"

LWP-9600, "Conduct of Operations for the INL"

MCP-139, "Radiological Surveys"

MCP-15009, "Radiological Work Permit Preparation"

MCP-187, "Radiological Control Posting and Labeling"

MCP-3364, "Sample Management at the Analytical Laboratory"

MCP-3365, "Waste Management at the Analytical Laboratory"

MFC-AOP-0001, "MFC All Facilities Response to Abnormal Operations"

MFC-ONRI-0001, "All Facilities Response to MFC Anomalies"

PDD-13000, "Quality Assurance Program Description"

PLN-2224, "Records Management Plan for Nuclear Science and Technology"

SD-40.1.5, "MFC AL NESHAP Periodic Confirmatory Measurements for Radionuclides"

SP-20.1.4, "MFC Document Management Supplement to LWP-1201 and LWP-21220"

10. APPENDIX

Appendix A, Flow Schematics for the Manual Mini-GPEC System.

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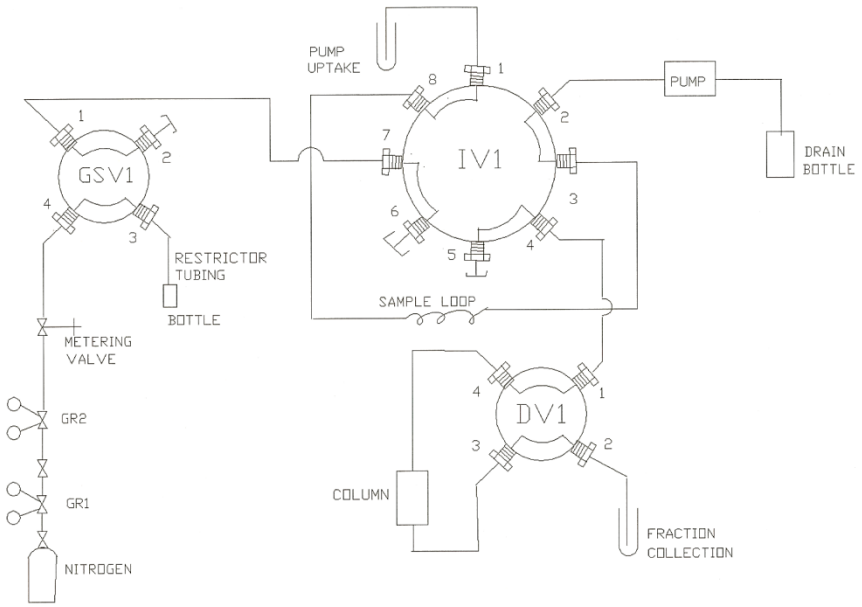
Appendix A

Flow Schematics for the Manual Mini-GPEC System

MINI-GPEC SYSTEM

FIGURE 1 - LOAD LOOP CONFIGURATION



MINI-GPEC SYSTEM

FIGURE 2 - INJECT LOOP CONFIGURATION