PLANT OPERATIONS MANUAL

Volume 08

Section 04

08-S-04-200

Revision 0

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CHEMISTRY INSTRUCTION

OPERATION OF Ge(Li) COUNTING SYSTEM

SAFETY RELATED

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List of Effective Pages:

Page:

Revision

1-11

Rev. 0

Title:		of Ge(Li)	No.:	08-S-04-200	Revision:	0	Page:	1
	Counting	Зузсеш			1			

1.0 INSTRUMENT DESCRIPTION

The Ge(Li) Counting System is primarily a gamma isotopic analysis system. The system uses high resolution lithium-drifted germanium detectors (Ge(Li)). The Ge(Li) Counting System also performs several utility functions such as quality control and data management.

There are two Ge(Li) Counting Systems, one located in the Counting Room (CR) and one located in the Hot Lab (HL). The two systems shall be considered identical, except as noted below. Those components identified as located in the Clean Lab (CL) may be connected to either system.

The individual components that comprise the Ge(Li) Counting System are:

- 1.1 H-P 9845T Desktop Computer
 - 1.1.1 H-P 9845B, Option 312, I/O ROM
 - 1.1.2 H-P 9845B, Option 313, Mass Storage ROM
- 1.2 H-P 7906MR, Option 102, Disk Drive
- 1.3 H-P 13037C, Disk Controller
- 1.4 H-P 98041A, Disk Interface
- 1.5 Canberra Model 7500 Ge(Li) Detector (CR-2, HL-1)
- 1.6 APT Model SPG-16, Lead Shield (CR-2, HL-1)
- 1.7 ND 66, MCA (CR-16K, HL-8K)
- 1.8 ND 575, 80 MHz ADC (CR-3, HL-1)
- 1.9 Ortec 401A, NIM Bin.
- 1.10 Ortec 572, Amplifier
- 1.11 Ortec 459, High Voltage Supply
- 1.12 APT NDAV-100, LN2 Monitor
- 1.13 H-P 98035A, Real Time Clock

Title:	Operation	of	Ge(Li)	No.:	08-S-04-200	Revision:	0	Page:	2	
1	Counting	Sy	stem							. 1

- 1.14 H-P 7225A, X-Y Plotter (CR only)
- 1.15 TI 820 RO, Impact Printer
- 1.16 H-P 2621P, CRT/Terminal (CL only)
- 1.17 Sykes Comm-Stor II (CL only)
- 1 18 Anderson-Jacobsen AD342D Modem and Acoustic Coupler (1 set)

2.0 REFERENCES

- 2.1 Chemistry Procedure 08-S-03-1, Qualifications of Chemistry Program
- 2.2 Chemistry Procedure 08-S-03-7, Control of Chemistry Support Equipment

3.0 DEFINITIONS

- 3.1 Daily Once per day or prior to use, whichever is less frequent.
- 3.2 Weekly Once per week or prior to use, whichever is less frequent.

NOTE

Computer commands will be enclosed in brackets, e.g., [CONT].

4.0 PREREQUISITES

- 4.1 Standard Mixed Gamma Sources
- 4.2 Eu-152 Source

5.0 PRECAUTIONS

- 5.1 Power to the instrument should be left on to provide system stability.
- 5.2 Handle radioactive samples in accordance with radiological safety procedures.
- 5.3 Exercise extreme care to keep the detector and shield free from radioactive contamination.
- 5.4 Prior to use, ensure required functional checks and calibrations are current.

Title:	Operation Counting	of Ge(Li) System	No.:	08-S-04-200	Revision:	0	Page:	3
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- 5.5 The Ge(Li) crystals must be maintained at liquid nitrogen temperatures at all times. Liquid nitrogen must be maintained in the dewars at all times with the gross weight of the dewars and nitrogen greater than 45 pounds.
- 5.6 The Ge(Li) crystal cold finger should never be removed from the liquid nitrogen for more than 45 seconds.
- 5.7 The Ge(Li) Counting System has a manual reset on loss of electrical power. The high voltage to the Ge(Li) crystals <u>must</u> be turned off and adjusted to zero prior to re-energizing the system.
- 5.8 On loss of power, the disk drive should be turned off prior to re-energization.
- 5.9 Disk fault lights should be reported immediately to the Lead Laboratory Chemist.
- 5.10 When shutting down the disk drive, the RUN/STOP switch is placed in the STOP position and the DOOR OPEN light <u>must</u> illuminate prior to shutting off the disk drive power.
- 5.11 Immediately set the RUN/STOP switch to STOP whenever a loud grinding noise is detected in the disk drive.

6.0 INSTRUCTIONS

- 6.1 Instrument Startup
 - 6.1.1 Adjust high voltage power supply to ZERO.
 - 6.1.2 Turn Nim Bin ON.
 - 6.1.3 Turn high voltage power supply power ON.
 - 6.1.4 Slowly increase the high voltage to the operating voltage listed in the Instrument Log Book.

NOTE

Voltage should be increased at a maximum rate of 1000 volts per minute.

6.1.5 Turn environmental cabinet air conditioner ON.

Title:	Operation	of Ge(Li)	No.:	08-s-04-200	Revision:	0	Page:	4
1	Counting	System	1					

6.1.6 Turn disk drive power ON.

NOTE

Verify DOOR OPEN light is illuminated. Report any other disk drive fault lights to the Lead Laboratory Chemist.

- 6.1.7 Turn disk controller ON.
- 6.1.8 Turn disk interface ON.
- 6.1.9 Turn I/O Expander ON.
- 6.1.10 Insert the system operating cartridge in the disk drive.
- 6.1.11 Place RUN/STOP switch to RUN.

NOTE

The DRIVE READY light should illuminate after approximately 60 seconds. If the DRIVE FAULT illuminates, do not set the RUN/STOP switch to STOP. Report any abnormal conditions to the Lead Laboratory Chemist.

- 6.1.12 Depress the AUTO LOAD key on the HP 9845 computer.
- 6.1.13 Place a "Ge(Li) Counting" tape in the right hand tape drive (":T15") of the HP 9845 computer.
- 6.1.14 Turn HP 9845 computer ON.
- 6.1.15 For the CR system, turn the TI 820 printer ON.

NOTE

Verify printer display is 001. Report any other displays to the Lead Laboratory Chemist.

- 6.1.16 Turn the ND66 MCA power ON.
- 6.1.17 The remaining switch settings should be verified per the Instrument Log Book.

	No.:	08-S-04-200	Revision:	0	Page:	5
1	Counting System					11460

- 6.1.18 The interconnection of the equipment should be verified per the Instrument Log Book.
- 6.1.19 The instrument may be placed in the standby mode by any or all of the following steps:
 - a. Disk drive RUN/STOP switch to OFF
 - b. HP 9845 computer power OFF
 - c. TI 820 printer power OFF
 - d. ND 66 MCA power OFF

6.2 Instrument Operation

- 6.2.1 Verify instrument startup in accordance with section 6.1.
- 6.2.2 Verify instrument is within prescribed calibration due dates.
- 6.2.3 Verify required functional checks have been performed.
- 6.2.4 Select the program to perform the desired task from the software package described in the following Chemistry Instructions:
 - a. Chemistry Instruction 08-3-04-212, Germanium Spectroscopy Software.
 - b. Chemistry Instruction 08-S-04-213, Quality Control Software.
 - c. Chemistry Instruction 08-S-04-214, Liquid Radwaste Software.
 - d. Chemistry Instruction 08-S-04-215, Gaseous Radwaste Software.
 - e. Chemistry Instruction 08-S-04-216, Utility Software.
 - f. Chemistry Instruction 08-S-04-217, Data Base Software.
 - g. Chemistry Instruction 08-S-04-218, Sampling Schedule Software.
 - h. Chemistry Instruction 08-S-04-219, Remote Terminal Handling Software.
- 6.2.5 Load the selected program into the HP 9845 computer by either:

6

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Title: Operation of Ge(Li) No.: 08-S-04-200 | Revision: 0 | Page: 6 | Counting System

- a. [LOAD "FINAME: C12"], [EXECUTE] where FINAME is the program file specifier.
- b. Press appropriate special function key.
- 6.2.6 Start the program by pressing the [RUN] key.
- 6.2.7 Set the desired I/O devices using the following select codes:
 - a. Thermal printer 0
 - b. External printer 5
 - c. Plotter 7, 5
 - d. Real time clock 9
 - e. Multichannel analyzer 11
 - f. Disk drive 12
 - g. Graphics 13
 - h. Left tape drive 14
 - i. Right tape drive 15
 - j. CRT 16
- 6.2.8 Respond to the program prompts and instructions. Refer to appropriate software instructions for additional information on each program.
- 6.2.9 When the instrument will not be used for the following eight hours, it may be placed in the standby mode.
- 6.3 Ge(Li) Counting
 - 6.3.1 Verify instrument startup in accordance with step 6.1.
 - 6.3.2 Verify instrument is within prescribed calibration due dates.
 - 6.3.3 Verify required functional checks have been performed.

Title: Operation of Ge(Li) No.: 08-S-04-200 Revision: 0 Page: 7
Counting System

- 6.3.4 Prepare the sample per Chemistry Instruction 08-S-04-14, Sample Preparation for Counting.
- 6.3.5 Samples should be centered on shelf 2 or higher except for marinelli beakers and 1 liter bottles.
- 6.3.6 Close and latch the shield lid.
- 6.3.7 Load and run the GSMCA program in accordance with section 6.2 to acquire a spectrum and transfer the data to the HP 9845 computer.
- 6.3.8 Use the GSRAP program to analyze the spectrum.
- 6.3.9 Refer to Chemistry Instruction 08-S-04-212, Germanium Spectroscopy Software, for additional information.

6.4 Functional Check

- 6.4.1 Daily Source Count
 - a. Verify instrument startup in accordance with section 6.1.
 - b. Place an Eu-152 source of known count rate on shelf 2 and close shield.
 - c. Load and run the GSMCA program in accordance with section 6.2 to acquire a spectrum for 600 seconds and transfer data to the HP 9845 computer.
 - d. Use the QCNDLY program to analyze the spectrum.
 - (1) The results of the QCNDLY program will be stored in file QFILEn. Where n is the detector number.
 - (2) The source number and fractional amount of the source (N.F) will be on the Eu-152 source.
 - (3) The assay date and time will be on the Eu-152 source.
 - e. If the current date is wrong or the time varies from correct time by + 1 minute, the GSCLCK program must be run to correct the internal clock.

Title: Operation of Ge(Li) No.: 08-S-04-200 Revision: 0 Page: 8
Counting System

- f. Report any test failure, i.e., flag = 0, to the Lead Laboratory Chemist.
- g. Refer to Chemistry Instruction 08-S-04-213, Quality Control Software, for additional information.

6.4.2 Weekly Background Count

- a. Verify instrument startup in accordance with section 6.1.
- b. Load and run the GSMCA program in accordance with section 6.2.
 - (1) Verify that there are no sources inside or in the general area of the shield.
 - (2) Collect a background spectrum for at least 4 hours with the sample stand in the shelf 2 position.
 - (3) Transfer the spectrum to the HP 9845 computer.
- c. Use the QCBKG program to analyze the background spectrum.
 - The results of the QCBKG program will be stored in file QCBKGn. Where n is the detector number.
- d. Report any test failure, i.e., flag = 0, to the Lead Laboratory Chemist.
- e. Refer to Chemistry Instruction 08-S-04-213, Quality Control Software, for additional information.

6.4.3 Weekly System Check

- a. Verify instrument startup in accordance with section 6.1.
- b. Load and run the GSGETM program in accordance with section 6.2.
 - Transfer spectrum WTEST from the fixed disk (:D12) to the HP 9845 computer.
- c. Load the GSRAP program to analyze the spectrum.

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Title:	Operation	of Ge(Li)	No.:	08-S-04-200	Revision:	0	Page:	9
	Counting	System			1			

- (1) Use parameters as stored with spectrum.
- d. Examine printout for consistency with previous results.
- e. Refer to Chemistry Instruction 08-S-04-212, Gamma Spectroscopy Software, for additional information.

6.5 Calibration

- 6.5.1 Verify instrument startup in accordance with section 6.1.
- 6.5.2 Obtain calibration sources meeting requirements of Reference 2.2.
- 6.5.3 Load and run the GSEDAT program in accordance with section 6.2 to enter the calibration source data.
- 6.5.4 The standards should be prepared per Chemistry Instruction 08-S-04-14, Sample Preparation for Counting.
- 6.5.5 Samples should be centered on the shelf and the lid closed when counting.
- 6.5.6 Load and run the GSMCA program in accordance with section 6.2.
- 6.5.7 Acquire a spectrum for sufficient time to accumulate greater than 10,000 net counts in each peak used in the efficiency curve determination.
- 6.5.8 Transfer the spectrum to the HP 9845 computer using the GSMCA program.
- 6.5.9 Use the GSEECL program to calculate the efficiency curves for each geometry code and shelf number.
 - a. Geometry codes for each geometry are:
 - (1) lLMB 1-liter marinelli beaker
 - (2) 4LMB 4-liter marinelli beaker
 - (3) 1LBT 1-liter poly bottle
 - (4) OGVL 14 ml off gas vial

Title: Operation of Ge(Li) No.: 08-S-04-200 Revision: 0 Page: 10
Counting System

- (5) CART charcoal cartridge
- (6) 1DVL 1 dram Vial
- (7) WPCH planchet
- (8) FILT filter paper 47 mm
- (9) WHAT Whatman 542 filter
- b. Acceptable shelf numbers are:
 - (1) 0 directly on top of crystal
 - (2) 1 first shelf of sample holder approximately 1 cm from crystal
 - (3) 2 second shelf of sample holder approximately 3 cm from crystal
 - (4) 3 third shelf of sample holder approximately 10 cm from crystal
 - (5) 4 fourth shelf of sample holder approximately 15 cm from crystal.
- 6.5.10 The efficiency curve % difference shall be less than + 5% for each peak used in the calibration.
- 6.5.11 Sources for Functional Checks
 - a. Center the check source on shelf 2 and close shield.
 - b. Count the sample at least 10 times for 600 seconds and calculate the average count rate.
 - c. Calculate the fractional amount of the source in relation to the calibration data stored by the GSEDAT program for the appropriate source.
 - d. Label the check source with the correct source number and fractional amount (NF) and assay date and time.

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Title: Operation	of Ge(L1)	No.:	08-S-04-200	Revision:	0	Page:	11
. Counting	System					1	

6.5.12 Refer to Chemistry Instruction 08-S-04-12, Gamma Spectroscopy Software, for additional information.

7.0 DOCUMENTATION/CORRECTIVE ACTION

7.1 Document results on proper reporting forms per Chemistry Procedure 08-S-03-3, Document Control.