

CP4

LIQUID SCINTILLATION ANALYZER (LSA)

PART A

INTRODUCTION

The liquid scintillation instrument consists of opposing photomultiplier tubes, a sample elevator, computer, software, and a printer.

The specific liquid scintillation analyzer (LSA) used in this laboratory is a Packard Tri-Carb 3100.

Initial setup of new equipment should be done by the vendor. New operators should be trained by experienced laboratory personnel. After learning the fundamentals, reference 1 should be consulted for additional detail. Particularly difficult operating problems should be discussed with a system analyst or the vendor.

PRINCIPLE

Samples suspected of containing beta emitters are placed in a counting vial and a liquid scintillation counting cocktail is added to the sample. When a beta particle is emitted and passes through the cocktail, light is emitted by the cocktail. This light emission is detected by the photomultiplier tubes and is registered as a count.

REFERENCE

1. Instruction Manual for the Packard Liquid Scintillation Analyzer.
2. ANSI N42.15 - 1997 *American National Standard* Check Sources for and verification of Liquid Scintillation Systems.

CERTIFICATION RECORD FOR

CP4

LIQUID SCINTILLATION ANALYZER

CHECKPOINTS

- | | | |
|-------------------------------------|-------|-------|
| 1. JOB HAZARD ANALYSIS (JHA) | _____ | _____ |
| 2. MSDS/HAZARDS DISCUSSION | _____ | _____ |
| 3. SAMPLE SET-UP | _____ | _____ |
| 4. QC CHECK | _____ | _____ |
| 5. DATA ACQUISITION | _____ | _____ |

ANALYST'S SIGNATURE: _____

CERTIFIED BY: _____

DATE : _____

COMMENTS: _____

PART B

1.0 PURPOSE AND SCOPE

This section provides an outline of the procedure for acquiring and analyzing beta radiation using a LSA. This should be combined with the manual from the vendor and hands-on training for greater detail.

2.0 APPARATUS

NIST traceable standards
Packard Tri-Carb 3100 LSA
Sample Cassettes

3.0 MASTER COUNTING ASSAYS

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the QA Manual. See page two for a copy of the certification record. Review the JHA for ESSAP Count Room Operations.

- 3.1 Master counting assays for the 3100 are defined for the isotope of interest. Master counting assays are edited and saved as the sample batch name then associated with counting flags.
- 3.2 The individual assays have parameters, counting options, and data options that are defined upon creation. The Laboratory Manager or designee configures the protocols to meet the Data Quality Objectives (DQO) of each associated analytical procedure or a client specific request. Reference the Instruction Manual for protocol setup details.
- 3.3 The master counting assay for tritium is named 3H.lsa. The regions of interest (ROI) for tritium (H-3) are 0.1 keV to 6.0 keV, Region A; 6.0 keV to 18.6 keV, Region B; and 0.1 keV to 2000 keV, Region C. Region A is used to calculate the concentration of tritium. Regions B and C are used to look for unexpected contaminants in the sample.

Note: The ROI information is stored in the defined assays. After completion of sample counting, the ROI information is electronically saved with the batch data and is printed as header information with the batch data.

- 3.4 The master counting assay for C-14 is named 14C.lsa. The ROI for carbon-14 (C-14) are 2.0 keV to 50.0 keV, Region A; 50.0 keV to 160.0 keV, Region B; and 160.0 keV to 2000 keV, Region C. Region A is used to calculate the concentration of C-14. Regions B and C are used to look for unexpected contaminants in the sample.
- 3.5 The master counting assay for H-3/C-14 in smears is named 3H_14C_Smears.lsa. The ROI for H-3/C-14 in smears are 0.1 keV to 6.0 keV, Region A; 6.0 keV to 50.0 keV, Region B; and 50.0 keV to 2000 keV, Region C. Region A is used to calculate the concentration of tritium. Region B is used to calculate the concentration of C-14. Region C is used to look for unexpected contaminants in the sample.
- 3.6 The master counting assay for Tc-99 is named 99Tc.lsa. The ROI for Tc-99 are 20.0 keV to 150.0 keV, Region A; 0.0 keV to 20.0 keV, Region B; and 5.0 keV to 2000 keV, Region C. Region A is used to calculate the concentration of Tc-99. Regions B and C are used to look for unexpected contaminants in the sample.
- 3.7 Protocols with ROI can be established for other beta emitting isotopes that are not listed in the above steps. When ROIs are established for other beta emitting isotopes, the ROIs must be documented in the task folder.

4.0 DAILY QUALITY CONTROL (QC)

- 4.1 The QC samples (a blank and a C-14 standard) are maintained in a sample cassette in the instrument associated with a "Flag" and all parameters previously defined and stored. To begin the daily QC count, open the instrument cover and push the movable insert to the left to activate the flag. Wait at least five minutes before proceeding.
- 4.2 The 3100 software is a PC based Windows driven program. An ICON has been placed on the desktop of this system. To start the program click on the QuantaSmart ICON.
- 4.3 On the program toolbar, click on the green flag to start samples counting. The data will automatically printout when the count routine is completed.
- 4.4 In the "ALPHA" window of the Canberra system, type QAD <cr>. This will take the operator to the LSA QC data file.
- 4.5 Type LSC31 to access the QC data file
- 4.6 When the QC file menu appears, type "6" and then "5". The data entry screen will appear.

- 4.7 Enter the data requested on the screen. After data entry is complete, press the PF3 key to save the data. Exit the QC file.
- 4.8 Type "Q31" to generate the daily QC review sheet. Type "P" to print the review sheet.

NOTE: The parameters are set-up initially based on 30 counts of each QC sample. The Canberra software automatically calculates the limits that establish the acceptance criteria. Investigation limits are between ± 2 and ± 3 sigma. The action limit is greater than ± 3 sigma.

- 4.9 When an "ACTION" flag is generated, the daily QC sample must be run two more times and all parameters must pass both times for the detector to be used to count samples.
- 4.10 If an "ACTION" flag is generated for either of the two QC parameters on the next two QC counts, the instrument is taken out of service until the problem is resolved.
- 4.11 The operator will sign and date the daily QC review sheet whether or not samples are to be counted on the detector.

5.0 SAMPLE COUNTING

- 5.1 Load the samples into the counting cassette(s), place a numbered "Flag" in the first counting cassette, push the movable insert to the left to activate the flag, and put the cassettes into the instrument.
- 5.2 A "dark adapt" routine ensures that all samples have been properly dark adapted before counting. The "dark adapt" counting assay flag must be activated at the same time and must be placed in front of the samples.
- 5.3 The 3100 software is a PC based Windows driven program. An ICON has been placed on the desktop of this system. To start the program click on the QuantaSmart ICON.
- 5.4 On the program toolbar, click on "File" to open the pull down menu. Click on "Open Assay" and a window with the defined counting assays will appear. Double click on the desired master counting assay, e.g., 3H.lsa.
- 5.5 A window with the counting assay information will open. Input the batch name, e.g., H1800, in the assay description window.

- 5.6 Click on “Save As”. Save the file as the batch name, e.g., H1800.lsa. The folder location for the file “Save As” is predetermined and the user should not change this location.
- 5.7 Click on the “Report Output” tab. In the “Data Report” window, there is an editing box for the File Name. Type the batch, e.g., H1800, in that box. Select the Excel output format as csv (comma separated value). Each of the master counting assays should have the “Run Application” box checked. The name of the application is LSC.exe. This application automatically transfers the appropriate batch information to the Excel data calculation sheet associated with each analysis.
- 5.8 Click on the “WorkList” tab. Input the sample ID information on the line corresponding with its position in the counting cassette.
- 5.9 To start the sample counting process a counting assay must be associated with a counting “Flag”. All the usable “Flags” are located in the left window of the screen. Click on the “Flag” number that was placed in the first sample cassette to highlight it in order to associate the counting assay.
- 5.10 On the program toolbar, click on “File” to open the pull down menu. Click on “Associate Assay” and a window with the defined counting assays will appear. Double click on the desired counting assay, e.g., H1800.lsa. The user will be prompted to select the data transfer folders. The internal data transfer folder will have the same name as the isotope of interest. The external data transfer folder is named “LSC” on the J: drive. Once these data transfer folders have been selected the “Associate Assay” process is complete.
- 5.11 On the program toolbar, click on the green flag to start samples counting.
- 5.12 After the samples have counted, remove the cassettes from the instrument. The samples are then taken to the storage area for samples that are to be disposed.

6.0 CALCULATIONS

- 6.1 Critical data values will be documented on standard forms maintained as critical records. The equations for the analyses counted on this instrument are defined in the analytical procedures for each specific analysis type . The calculations for each type of analysis are performed in validated Excel spreadsheets.
- 6.2 An executable program (LSC.exe) is used to automatically transfer the appropriate data from the LSA once a batch count has been completed. The data transfer is validated for each type of analysis.

7.0 CALIBRATION

- 7.1 An efficiency standard of the appropriate isotopes is counted with every sample batch to establish a counting efficiency for that batch.
- 7.2 A comparison of the quench indicating parameter (QIP) of all samples is made for each batch. The acceptable variation for no quench correction is ± 20 percent. For DOE samples the acceptable variation for no quench correction is ± 5 percent.

8.0 RECORDS

- 8.1 Reference QC manual for general records requirements.
- 8.2 Weekly backups
 - 8.2.1 To reduce the chance of loss of recently acquired QC and sample data, the entire system is backed up a minimum of once weekly.
 - 8.2.2 On the Desktop click on the "Backup Directory" folder icon.
 - 8.2.3 The program will backup the entire QuantaSmart database for the LSA to the "Y" drive on the network. This backup overwrites the previous backup.
- 8.3 Data calculation sheets and data transfer programs are validated for each type of analysis associated with the LSA. These validations are saved electronically and in hard copy, if necessary.
- 8.4 Hard copy records associated with this procedure are defined in the analytical procedures requiring the use of this instrument for counting. Calibration for this instrument uses the same hard copy records.