

CP2

ALPHA SPECTROSCOPY

PART A

INTRODUCTION

The basic alpha spectroscopy system consists of an alpha spectrometer (vacuum chamber and amplifier electronics), alpha detector(s), analog to digital converter(s), multi-channel analyzer (MCA), computer software, and a print device.

The specific equipment used in this laboratory consists of a Canberra Genie-VMS Personal Workstation with software, Canberra and Tennelec alpha spectrometers, Canberra and Tennelec alpha detectors, and Fisher Scientific vacuum pumps.

Initial setup of new equipment should be done by the vendor. New operators should be trained by experienced laboratory personnel. After learning the fundamentals, references 1-3 should be consulted for additional detail. Particularly difficult operating problems should be discussed with a system analyst or the vendor.

PRINCIPLE

Alpha emitting samples are placed on the sample holder in the alpha spectrometer and a vacuum is pulled on the spectrometer vacuum chamber. When alpha particles are emitted by the sample, the alpha detectors produce electronic signals that are analyzed by the MCA to generate a digital spectrum that is equivalent to the energy of the alpha particles. The spectrum is then analyzed by computer software that identifies and quantifies the alpha energies.

REFERENCES

1. Instruction Manual for the Genie-VMS Personal Workstation.
2. Instruction Manual for the Canberra 7401 VR Alpha Spectrometer.
3. Instruction Manual for the Alpha Management Software (AMS).
4. ANSI N42.23 American National Standard Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories

Certification Record for
CP2
ALPHA SPECTROSCOPY
CHECKPOINTS

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

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 alpha spectra. This should be combined with the standard operating procedures and hands-on training for greater detail.

2.0 APPARATUS

Canberra Genie-VMS Personal Workstation
Canberra and Tennelec Alpha Spectrometers
NIST traceable standards
Solid state alpha detectors
Vacuum pump(s)

3.0 PROCEDURE

Before proceeding, you must be certified as indicated in Section 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 Daily Pulser Check

- 3.1.1 Turn on the bias supply and the pulser on the spectrometers of the operating detectors. (For Canberra units flip “bias” to on position and flip “counter/cal” switch toward start until “cal” light is flashing. For the Tennelec units pull “bias/marker” switch out and up to marker position.)
- 3.1.2 Move the AMS Procedure screen to the front.
- 3.1.3 From the main menu select “Count”.
- 3.1.4 From the Count pull down menu select “Pulsers”.
- 3.1.5 Input the number of detector banks for which the pulsers are to be counted (“1-32,” or “A” for all banks). Select “Okay” to make selection.
- 3.1.6 A prompt will appear asking if everything is ready to count. Select “Okay”. The selected detectors will count the pulsers for the preset time.
- 3.1.7 To process the pulsers after counting is complete, select “process” from the main pull down menu.
- 3.1.8 From the process pull down menu select “Create Names.dat”.
- 3.1.9 From the side menu select “Pulsers”.

- 3.1.10 The Processing Information page will appear. Pulsers should be in the item box and the detector numbers entered in the detector box. Select “Okay”.
- 3.1.11 The prompt to process Names.dat will appear. Select “Okay” to start the processing of the pulsers. A pulser report will be printed when the processing is complete.
- 3.1.12 If the daily pulser check passes, turn off the pulser on each detector. For the Tennelec spectrometers, the bias must also be turned off before the unit can be vented. The Canberra units have an automated bias cutoff that is activated when the unit is vented.

3.2 Importing Batch Information

Note: The batch information needed for importing to the ALPHA workstation is created in the ESSAP database (DB) by the analyst when the analytical batch is created.

- 3.2.1 Load the samples into the counting chambers and pull the vacuum.
- 3.2.2 From the ESSAP data base select the “Lab Analysis” option.
- 3.2.3 Scroll to the batch number being counted. Click on the “PROCESS” button on the screen to convert the data to a comma separated value (CSV) file and to transmit the file to the ALPHA system.
Note: The DB will generate a message indicating the successful transfer of the batch information.

3.3 Automated Sample Counting

- 3.3.1 Move the AMS Procedure screen to the front.
- 3.3.2 From the main pull down menu select “Count.”
- 3.3.3 From the count pull down menu select “Samples”.
- 3.3.4 Input the number of detector banks for which the samples are to be counted (“1-32” or “A” for all banks). Select “Okay” to make selection.
- 3.3.5 The sample set up page will come to the front. Enter the batch number in the appropriate box preceded by an actinide abbreviation (e.g. Am1234).

Note: Other sample information will be imported through the batch import file (bif) once the batch number is entered.

- 3.3.6 The bif/results (res) file page will come to the front. “Sequential mode” should be selected, and then “Okay” to continue.
- 3.3.7 The sample information page for the first sample will come to the front. Review data for the sample and select “Okay”. A prompt will appear asking if everything is ready to count. If so, then select “Yes”. The remaining sample information pages will come to the front for review of each sample being counted. The banks will start up individually and count for the preset time.

3.4 Spectrum Processing

- 3.4.1 On the main menu for the AMS procedure screen select “Process”.
- 3.4.2 From the Process pull down menu select “Create Names.dat”.
- 3.4.3 From the side menu select “Samples”.
- 3.4.4 Enter the batch name and actinide in the appropriate boxes. For the Sample Actinide enter the software abbreviation for the isotope of interest, e.g., “Am” for americium, “Pu” for plutonium, “Th” for thorium, and “UU” for uranium. Select “Okay”.
- 3.4.5 A prompt to process Names.dat will appear. Select “Okay” to start the processing of the samples.
- 3.4.6 Each spectrum associated with a batch will be moved into Alpha0 for review and approval by the analyst.
- 3.4.7 After the batch has been processed, the software generates an Excel compatible file titled N1000.xls; 1. To rename the “N” file, go to the DECterm window with alpha prompt, (GAMMA: :ALPHA>). Type “TOP” to go the main default.
- 3.4.8 Type “XFER (XFER = Set Def [dataxfer.alfdata])”. This command accesses the directory in which the Excel file is stored.
- 3.4.9 Type “Dir/since=today *.xls;*” to display a directory of the files that have been created the day of processing.
- 3.4.10 The Excel file needs to be named by the actinide analyzed and the batch number. Type “Rename N1000.xls;1 U1000.xls;1”. If there is more than

one actinide analyzed for a batch, the “N” file must be renamed after processing each actinide sample set.

3.5 Exporting Processed Data

Note: After the batch spectra have been processed and stored in the [dataxfer.alfdata] directory, the processed data is exported to the DB and to the Y:ESSAPLAB/SITEDATA/GAMDAT folder using the import tool in the database.

3.5.1 In the DB find “TOOLS” on the main toolbar and click to display a dropdown menu.

3.5.2 Select the “Import Alpha Spec Result” option. From the listing of .xls files, select the .xls files to be exported and click on the “IMPORT” button to transfer the file(s) to the DB and the Y:ESSAPLAB/SITEDATA/GAMDAT folder.

4.0 CALCULATIONS

4.1 Alpha spectra are analyzed by the Alpha Management Software supplied by Canberra. The results are given for peak search, peak identification, quantities of nuclides, counting errors, and minimum detectable activities.

4.2 The system is capable of generating a hardcopy printout and/or performing electronic data transfer for final data review by the Laboratory manager or designee.

4.3 Critical data values are documented in the electronic configuration file of each sample. The following equations define the critical data values.

$$\text{Concentration} = \frac{G - B}{E \cdot Q \cdot Y \cdot AB \cdot T} = pCi / unit$$

$$2\sigma \text{ Error} = \frac{1.96\sqrt{(G + B) \cdot T}}{E \cdot Q \cdot Y \cdot AB \cdot T} = pCi / unit$$

$$2\sigma \text{ TPU} = C \cdot 1.96 \sqrt{\frac{(G + B) \cdot T}{((G - B) \cdot T)^2} + (RE)^2 + (RY)^2 + (RAB)^2 + (RQ)^2} = pCi / unit$$

$$MDC = \frac{3 + 4.65\sqrt{B}}{E \cdot Q \cdot T \cdot AB \cdot 2.22dpm / pCi} = pCi / unit$$

where:

AB	=	alphas/disintegration
B	=	background counts
C	=	concentration in pCi/unit
E	=	counting efficiency counts/gamma
G	=	sample gross counts
MDC	=	minimum detectable concentration
Q	=	quantity
RAB	=	relative per cent error of the gamma abundance
RE	=	relative per cent error of the counting efficiency
RQ	=	relative per cent error of the quantity
RY	=	relative per cent error of the yield
T	=	time in minutes
TPU	=	total propagated uncertainty
Y	=	radiometric recovery

5.0 CALIBRATION

- 5.1 The alpha system is efficiency calibrated using a NIST traceable mixed alpha source.
- 5.2 The operator follows the COUNT MENU as described in steps 3.1.1 through 3.1.11, except select “Primes” instead of “Pulsers”.
- 5.3 The region of interest for each alpha isotope should contain at least 2000 counts in order for the counting statistics of the efficiency to be considered acceptable.

6.0 RECORDS

- 6.1 Reference QA Manual for general record requirements.
- 6.2 A system backup to DAT tape or equivalent is performed weekly to protect spectra and data generated during the previous week.
- 6.3 A full system backup to DAT tape or equivalent is performed monthly. This backup includes system operating files and all files located on the hard drive.
- 6.4 Hard copy records associated with this procedure are defined in AP11.
- 6.5 Calibration records are maintained electronically.