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) (ADC), multi-channel analyzer (MCA), computer software, and a print device.

The specific equipment used in this laboratory consists of a Personal Computer (PC) Workstation running Canberra Apex Alpha software, Canberra and Tennelec alpha spectrometers, Canberra and Tennelec alpha detectors, and Fisher Scientific vacuum pumps.

Initial setup of new equipment and software training 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 Canberra Apex Alpha Software.

2.   Instruction Manual for the Canberra 7401 VR Alpha Spectrometer.

3.   ANSI N42.23 American National Standard Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories.

4.   National Nuclear Data Center at Brookhaven National Laboratory www.nndc.bnl.gov/

5.   The Berkley Laboratory Isotopes Project's, Richard B. Firestone http://ie.lbl.gov/education/isotopes.htm

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 provided by the manufacturer (References 1 and 2) and hands-on training for greater detail.

2.0   APPARATUS

Dell PC Workstations or equivalent
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 QCP1 of this manual and Section 3 of the Quality Program (QP) Manual. Quality control requirements for this procedure are defined in QCP5. These requirements may be augmented by requirements in a client's statement of work. See page three for a copy of the certification record. Review the JHA for IEAV 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   Go to the PC screen on the monitor. If the monitor displays the gamma spectroscopy system, hit the following keys in quick succession: scroll, scroll, up arrow.

3.1.3   If the Apex Alpha software is not on the screen, double click on the software's icon to open the window. If the software is in operation, go to the step 3.1.4.

3.1.4   Click on the "Sample Assigner" tab. Select detectors to use for pulsers.

3.1.5   Click on the "Pulser Check" button. (All the available detectors will be grayed out.)

3.1.6   Click on the "Load Samples" button. A screen with the list of detectors will appear. Click on "OK". The pulsers count will start for each available detector. To view the pulsers, click on the "Main" tab. An individual detector pulser may be viewed by double clicking on that spectrum.

3.1.7   After the pulsers have finished counting, click on the "Reports" tab.

3.1.8   In the "Reports" window, click on the "QA Summary Report" tab.

3.1.9   Select the Pulser button then click on the "Check All" button.

3.1.10   Click on the "View" button to get the pulser report on the screen.

3.1.11   Print the pulser report and complete the required approval.

3.2   Importing Batch Information

Note: The batch information needed for importing to the ALPHA software is created in the IEAV 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 IEAV database select the "Lab Analysis" option.

3.2.3   Scroll to the batch number being counted. Click on the "Analyze Samples". The select isotope screen will appear.

3.2.4   Click on the isotope of interest, e.g. uranium, click on "Select All" or the individual samples for processing, and then click on "Next".

3.2.5   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 software.

Note: The DB will generate a message indicating the successful transfer of the batch information.

3.3   Manual Batch Creation

3.3.1   Go to the PC screen on the monitor. If the monitor displays the gamma spectroscopy system, hit the following keys in quick succession: scroll, scroll, up arrow.

3.3.2   If the Apex Alpha software is not on the screen, double click on the software's icon to open the window. If the software is in operation, go to the step 3.3.3.

3.3.3   Click on the "Batches" tab.

3.3.4   In the "Create Batches" window, fill in the required information, then click "Next".

3.3.5   Double click on the "Unknown" sample to manually edit and input the correct information for each sample then click on "Update Sample". Once all the sample information is correct, click on "Save".

3.3.6   The batch is ready to count.

3.4   Sample Counting

3.4.1   Click on the "Sample Assigner" tab.

3.4.2   Click on the "Queued Batches" button.

3.4.3   Click and drag the batch to the desired detectors icon or expand the batch to assign each sample ID individually to the desired detector icon.

3.4.4   Click on "Load Samples". A screen with the list of detectors and sample information will appear. Click on "OK". The sample counts will start on the selected detectors. To view the sample spectra, click on the "Main" tab. An individual sample spectrum may be viewed by double clicking on that spectrum.

3.5   Data Review

3.5.1   Click on the "Data Review" tab.

3.5.2   Click "Batches" on the search type box.

3.5.3   Type the batch number for samples to be reviewed. The batch number should be prefixed with isotope code. Example: Uranium batch will be U7600, Plutonium batch will be Pu7600, or Americium batch will be Am 7600.

3.5.4   Check the batch number in the search results window. Click the "Next" button located on the bottom right for the sample review page.

3.5.5   Select a sample to be reviewed in the sample box. The list of samples is displayed in the "Sample" box located to the right of the spectrum. The sample being displayed on the spectrum is "grayed out" in the "Sample" box when selected for analysis.

3.5.6   Review the regions of interest (ROI). If the ROIs are acceptable, go to step 3.5.10. If the ROIs require adjusting, go to step 3.5.7.

3.5.7   The ROIs associated for each isotope may be adjusted at one time using the "Shift All ROIs" slide bar or the left marker may be adjusted using the "Shift only Left Markers" slide bar. If the ROIs are acceptable, go to step 3.5.10.

3.5.8   If the ROIs created in step 3.5.7 are not acceptable, the analyst may adjust the ROIs manually in the review box. If this approach is taken, each ROI that requires adjustment must be deleted by clicking on the "delete ROI" button. The new ROI is set by adjusting the left and right markers and clicking on the "Add ROI" button. The analyst must update the ROIs by clicking on the "Update ROI" button.

3.5.9   Once the ROIs have been updated, click on the "Reanalyze" button.

3.5.10   Review the report on screen. Click on the "Approved" button.

3.5.11   Click on the "Print Report" button. Return to step 3.5.5 to analyze the next sample. Repeat this process until the analysis of the batch is completed.

3.5.12   A print option screen will appear. Click on the button next to the print spectrum option. A hard copy of the report will be printed.

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.

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

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

In the event of G=0 and B=0, 1 will be substituted for G+B to prevent a division by or into zero error in this uncertainty calculation.

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

In the event of G=0 and B=0, 1 will be substituted for G+B/(G-B)² to prevent a division by or into zero error in this uncertainty calculation.

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

where:

AB	=	alphas/disintegration
B	=	background counts
C	=	concentration in pCi/unit
E	=	counting efficiency counts/alpha
G	=	sample gross counts
MDC	=	minimum detectable concentration
Q	=	quantity
RAB	=	relative percent error of the alpha abundance
RE	=	relative percent error of the counting efficiency
RQ	=	relative percent error of the quantity
RY	=	relative percent 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 QP 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.