USTUR 660: ALPHA SPECTROMETRY ACQUISITION PROCEDURE FOR ABSOLUTE ANALYSIS

Purpose	Absolute analysis for α-spectrometry	**Method Number**	USTUR 660
Original Date	10/10/95	**Author**	USTUR Radiochemistry Staff
Revision Number	1	**Approved By**	Jim Elliston
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This procedure is no longer in use – see previous version of the Procedures Manual if you wish to review this procedure.

WARNING: Personnel must be aware of proper handling procedures for radioactive sources and proper usage of the alpha spectrometry equipment. Misuse can result in costly damage to equipment.

SOFTWARE: MAESTRO™ for Windows™ 3.X, Version 2.10, June 3, 1994 and ALPHAMAT™ Version 1.30, February 9, 1995.

1. Principle of Method

1.1 Absolute analysis is used for the spectrometric evaluation of alpha emitting radionuclides.

1.2 Absolute analysis requires that samples be counted in a geometry with a known efficiency.

1.3 Absolute analysis is used primarily to evaluate secondary alpha spectroscopy sources for energy and efficiency calibration purposes as well as for background counts.

1.4 Most radiochemical analyses of USTUR samples require correction for losses during chemical separation and are therefore analyzed by USTUR 650.

1.5 Ultra detectors are not as sensitive to rapid changes in vacuum as standard surface barrier detectors. However, when using standard surface barrier detectors, operation above 500 mtorr will result in destruction of that detector.

2. Procedures Conducted Prior to ABSOLUTE Analysis

2.1 Each chamber to be used for analysis of samples must first have a valid energy, efficiency calibration, and background count. The efficiency calibration must have been performed for the specific counting geometry to be used for absolute analysis.

2.2 The energy calibration, background count, and efficiency calibration must have been conducted two weeks prior to analysis.

2.3 See USTUR 610 for further details on system setup.

2.4 See USTUR 620 for energy calibration, efficiency calibration, and background counting methods.

2.5 See USTUR 600 for further descriptions of alpha spectrometric analysis.

3. Direct Analysis Procedures

3.1 Enter ALPHAMAT™ (Figure 1) and note all chambers which are actively counting, Exit ALPHAMAT™.

3.2 Enter MAESTRO for Windows™, and physically switch the vacuum settings on **all active** chambers from PUMP to HOLD. Alternatively, the entire MCB can be placed on hold by closing the vacuum control unit located at the back of the unit.

3.3 Use <Ctrl> and <F1-4> to toggle to the appropriate MCB(s). Select "Acquire --> Adjust Controls" and turn the bias **"OFF"** on the chamber(s) to be opened. Switch the chamber(s) to be opened from HOLD to VENT, or if not yet on HOLD, then from PUMP to VENT.

3.4 Open the appropriate chamber(s), remove and/or insert sample(s) on the appropriate shelf and close the chamber door(s). If the time period between sample insertion and vacuum application is expected to be greater than 5 minutes return the active chamber(s) vacuum from HOLD to PUMP, to insure that the active chambers do not exceed 100 mtorr. The active chamber(s) **must** be returned to the HOLD position prior to applying vacuum to **any** chambers which had been opened to atmospheric pressure.

3.5 To restart vacuum make sure **ALL** active chamber(s) vacuum are on HOLD, switch the appropriate, inactive, chamber(s) from VENT to PUMP. When chamber pressures are < 100 mtorr turn on the Bias Voltage and then switch **all active chambers** from HOLD to PUMP. Reopen vacuum control unit if necessary.

3.6 Escape "OCTETE™ Control"

Note: Initiating an acquisition in ALPHAMAT™ will automatically clear ROI's.

3.7 Quit MAESTRO™.

3.8 Enter ALPHAMAT program by launching a DOS window and typing "ALPHAMAT™" at the DOS prompt.

Note: Be sure MAESTRO™ is quit before runnng ALPHAMAT.

3.9 Use the F-1 key to select "Menu" (Figure 2).

3.10 Select the Procedure File by using the up and down arrow keys.

3.10.1 Default is USTUR.PRE FILE, <ENTER>

3.11 Select the Detector List File and enter the chamber numbers which will be used for analysis, then <ENTER> (see Figure 3.)

3.11.1 Detectors are numbered from 1 to 32, e.g., a detector listmight read " 1, 2-8, 10".

Note: If using 1-8 for plutonium and 9-16 for americium then the analyses must be conducted in two separate runs or startups of ALPHAMAT™. Different analyses must be started in separate steps.

3.12 Type "Operator Name", then <ENTER>

3.13 Select appropriate count type. For plutonium analysis select "C.A.P. Plutonium" (Count, Analyze, Plot) or other appropriate analysis for other nuclide methods, <ENTER>.

3.14 Set Live-Time (counting time) in seconds appropriate for the analysis.

3.14.1 1800 seconds is used for energy and efficiency calibration.

3.14.2 75,000 seconds is standard for USTUR analyses.

3.14.3 225,000 seconds is used for background counts.

Note: When counting two different samples with two different live times, two separate analyses must be started or the acquisition changed usings MAESTRO.

3.15 Use **F-10** to accept settings, screen changes and identifies the detector for acquisition, then <ENTER>.

3.16 A series of screens is then displayed for each analysis. Enter information as follows (see Figure 4):

3.16.1 Type your name, then <ENTER>.

3.16.2 Enter spectrum name.

e.g. Test-01, <ENTER>.

Note: See USTUR010 for nomenclature.

3.16.3 Enter appropriate sample description.

e.g. "Calibration Sample #23, Shelf # 2" <ARROW DOWN>.

3.16.4 Select the Background File

Note: ALPHAMAT™ always displays the most recent background for use, but this may be changed or eliminated as needed. The F-2 key displays other backgrounds. Backgrounds should always be run without background files.

3.16.5 Select Report Template

"USTURRPT" is the standard report template. <ARROW DOWN>.

3.16.6 Set analysis type to "Absolute" (A).

3.16.7 Set "ROI PEAK TYPE" (Region Of Interest) to R for TEST files.

The ROI's are defined in USTUR 600.

3.16.8 Check analytes are correct. Add or modify as necessary.

3.16.9 Use <F-10> to accept settings.

3.17 Repeat steps 3.16.1 through 3.16.9 for each analysis.

3.18 Enter file names and information in the alpha spectrometry log which is located next to the "Acquisition Computer".

3.19 Exit ALPHAMAT™ when done and LOCK the screen.

3.20 Re-enter ALPHAMAT™ when the count is expected to be completed. ALPHAMAT™ will automatically generate a report and plot of the spectra upon completion via the laser printer. Make sure the printer is on line.

3.21 Exit ALPHAMAT™.

3.22 Enter MAESTRO for Windows™ and print a report of the number of counts in each channel for each analysis if needed.

Note: This is not necessary for energy or efficiency calibrations or backgrounds.

3.23 Upon completion of counting, analyze the plot of the spectra for correct ROI's, counting information, etc. Some adjustments may be required for broad peaks.

3.24 File report as required.

3.25 Examine the plot of each spectra and ensure no modifications are needed to the ROI's.

3.26 If no modifications are needed, then the analysis is complete.
EXIT MAESTRO™.

3.27 If modifications are needed to the ROI's, re-enter ALPHAMAT™.

3.28 Select "F-1" menu to re-analyze existing spectrum.

3.29 Enter the spectrum name to be re-analyzed, <ENTER>.

3.30 Modify the ROI's as needed.

3.31 Select "F-10" when complete to print the modified report.

3.32 Select "F-1" to enter the menu and select "Plot Spectrum" (see Figure 2).

3.33 Enter the spectrum name to be plotted. Select "F-10" to plot the spectrum with the new ROI's.

3.34 If further modifications are required to the ROI's, return to 3.27 - 3.32. If no other modifications are required the analysis is complete.

3.35 EXIT ALPHAMAT™.

FIGURE 1
ALPHAMAT MAIN SCREEN

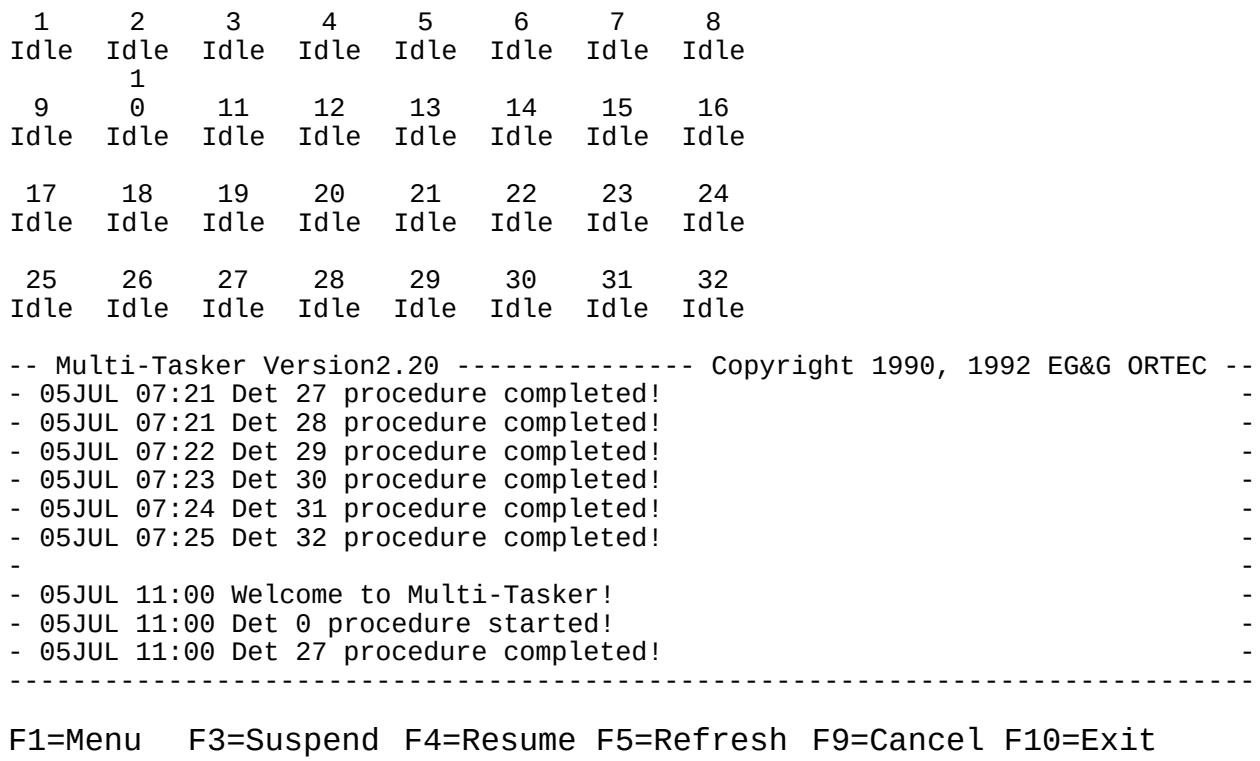

FIGURE 2
ALPHAMAT MENU SCREEN

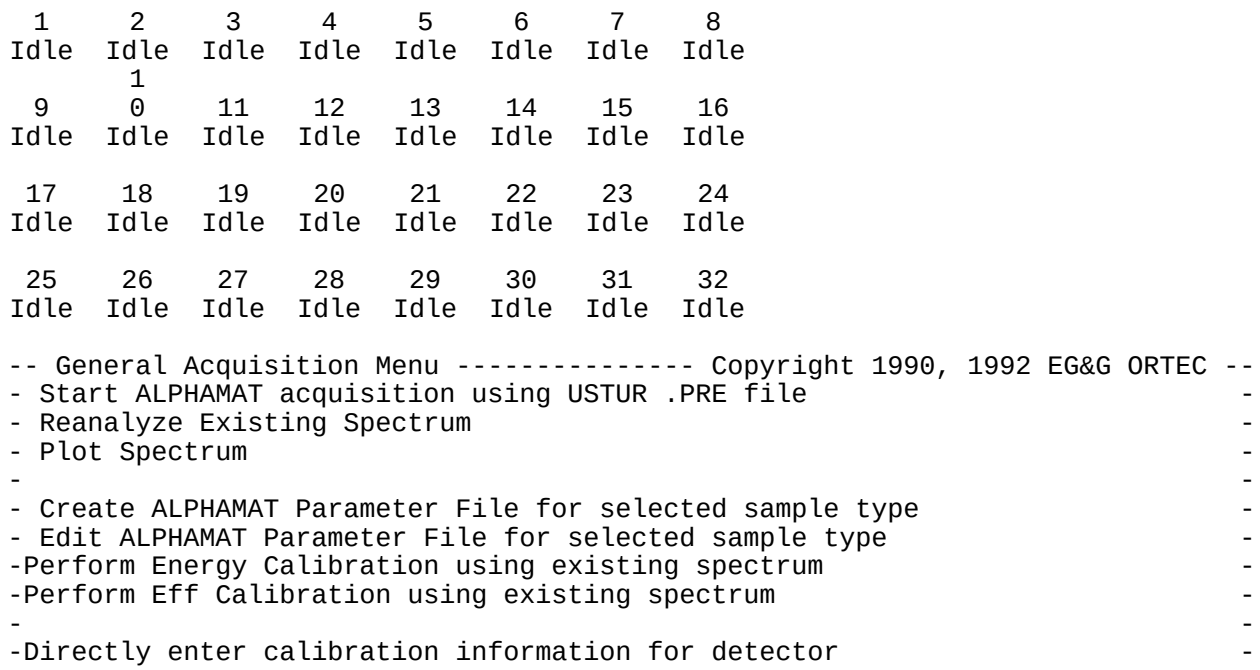

FIGURE 3
ALPHAMAT START ACQUISITION SCREEN

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Detector list..... 1,3-7,32
Operator Name..... Sam Glover

Count Type..... C.A.P. Uranium

Live-Time Preset..... 75000
Real-TIme Preset..... 0
ROI Integral Preset... 0
ROI Peak Preset..... 0
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FIGURE 4
ALPHAMAT ANALYSIS SCREENS

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                              Detector Number 1

Operator Name.........................Sam Glover

Spectrum Name.........................

Sample Description:


Volume Total..........................1.000000
Volume Aliquot........................1.000000

Background Spectrum Filename...........B0195184
   Template...........................USTURRPT

Method of Analysis:
   Relative or Absolute................(R,A)....R........Relative
     Tracer Added to Total or Aliquot? .(T,A)....T........Total
     Tracer DPM Added ..........................12.600000
     Contaminated Tracer? ......................N........No

   ROI, PeakFit, or Library-directed...(R,P.L)..R........Region of Interest
     Peak Matching Restriction .........(in keV).50.000000

   ROI Type............................(A,C)....0........Offset from Centroid

                 Filename  Nuclide   Energy      ROI      Chan
                                     (keV)
Tracer. . . . .  U232      U-232     5320.0000   40   8   364.0000
Analyte 2 . .    U235      U-235     4398.0000   31   36  180.0000
Analyte 3 . .    U238      U-238     4198.0000   36   8   140.0000
Analyte 4 . .    PU242     Pu-242    4901.0000   5    5   280.0000
Analyte 5 . .    AM243     Am-243    5276.0000   5    5   355.0000
Analyte 6 . .    TH228     Th-228    5423.0000   5    5   385.0000
Analyte 7 . .                        0.000000    0    0   0.000000
Analyte 8 . .                        0.000000    0    0   0.000000
Analyte 9 . .                        0.000000    0    0   0.000000


            F1 - Help,  F10 - Accept,           ESC - Abort


Entering analysis information for detector 1 (now gathering spectrum)
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