

ROUTING SHEET

Document Title:	GAMMA SPECTROSCOPY ANALYSIS PROCEDURE
RP#:	SLAC-I-760-2A39C-009
Revision #:	002
Revision Date:	October 16, 2006
Document Author:	Henry Brogonia

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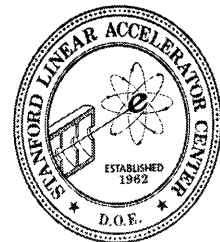
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ES&H DIVISION
RADIATION PROTECTION (RP) DEPT.
SLAC-I-760-2A39C-009-R-002

GAMMA SPECTROSCOPY ANALYSIS PROCEDURE

RP Dept Document, RE # 010

*Stanford
Linear
Accelerator Center*



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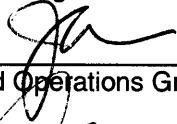
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James Liu, Radiological Environmental Protection Group

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TABLE OF REVISIONS

REVISION #	DATE	SECTION(S)	REASON FOR REVISION
001	March 19, 2003	2,4,5,6,7	Clarify Process
002	October 16, 2006	All	Rewrite of procedure to incorporate Gamma Spec Source Check procedure. Gamma Spec checklist added to appendix.

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1 PURPOSE

This procedure provides general guidance for characterizing samples using gamma spectroscopy. This document applies to anyone characterizing samples in the Radioanalysis Laboratory (Rad Lab). This procedure outlines the necessary steps for gamma spectroscopy counting. A Routine Gamma Spec Counting Checklist (Attachment 1) is attached to provide detailed instructions for routine gamma spectroscopy counting using current Rad Lab equipment.

2 REFERENCES

- 2.1 ORTEC GammaVision-32 Software User's Manual
- 2.2 ORTEC Solid State Photon Detector Operators Manual
- 2.3 SLAC Rad Lab Manual, General Sample Analysis Procedure

3 RESPONSIBILITIES

3.1 Rad Lab Operator

The Rad Lab Operator assigned to perform activities described in this chapter is responsible for:

- ensuring that prerequisites are met as required,
- maintenance of the laboratory equipment and performing daily quality control checks,
- reporting any problems or unexpected events to the Radiological Environmental Protection (REP) Manager,
- routine sample analysis counting,
- providing the data to the REP Manager for analysis and approval, and
- Performing other duties assigned by the REP Manager.

3.2 REP Manager

The REP Manager is responsible for:

- ensuring that the activities in this chapter are conducted in accordance with this written procedure,
- assigning the tasks in this procedure to a qualified operator, and
- providing final approval or disapproval of all data and submittals.

4 PREREQUISITES

- 4.1 Must be familiar with Windows operating menus, Microsoft Access and Notepad.
- 4.2 Must be familiar with Rad Lab general sample analysis procedures
- 4.3 Must have a basic understanding of Gamma Spectroscopy theory
- 4.4 Must be familiar with general laboratory equipment (pipettes, scales, etc.)

5 PROCEDURES

5.1 Perform a QA Check

As part of the instrument quality control process, a QA check should be performed daily when the Gamma Spectroscopy system is in use. This is accomplished by running the source check macro. The macro will first count with an empty cave to verify that detector system has not been contaminated. A source standard is then placed into the detector and counted for a preset time. Upon completion of the macro the data is logged into the QA database of the system software. The macro will verify that the regions of interest and their peaks correspond to the correct gamma energies and activities of the source standard listed on its certificate. If there are any major discrepancies between the count and the source standard certificate the user will be notified before the next count. The software will list satisfactory parameters as an "o.k." in the QA status menu. Major discrepancies will be noted as a "violation" under the same menu. If the system fails any of the QA parameters, the Gamma spectroscopy unit should be adjusted/repared as needed.

5.2 Count the Sample

There is no preparation for Gamma spectroscopy samples. It should be noted that most samples should be counted in the same container/geometry as the source standard used to calibrate the profile. For typical counting 500 ml of sample should be used for liquid profiles and 250 ml should be used for soil profiles. Samples should be placed in a 500 ml Nalgene bottle. There may be

a need for geometry corrections if the sample volumes change. The sample is placed in the shielded volume and ready for counting.

Routine gamma energy analysis with the germanium detector is accomplished using a set of macros. Each macro calls the appropriate calibrations and libraries for a particular sample type. These macros will bring up several system settings that are appropriate for counting a particular sample. These settings include the calibration, count time, library, peak sensitivity, sample volume, decay correction, background subtraction, and geometry corrections. A reference table (Attachment 2) lists the settings for each type of sample being counted. The analysis report (Attachment 3) should be checked against the reference settings to ensure correct analysis data.

5.3 Determine the Results

When the analysis is complete a report will automatically be generated. You may choose to print the report at this time.

The analysis results should be thoroughly scrutinized by a person experienced in gamma spectra analysis for:

- Settings for the sample being counted (calibration, library, background subtraction, etc.) match the reference table
- Extreme departure from the expected result of the analysis
- Isotopes that are not usually found at SLAC
- Isotopes having half lives which should preclude their presence in the sample

A sample may need to be re-analyzed under several conditions to determine its radioactive constituents. For instance, a soil spectrum may need to be analyzed with a separate library to account for naturally occurring isotopes, or background subtraction may be needed to refine the spectrum. A sample may need to be counted longer for better statistics.

Caution: Rerunning a macro can cause the original measurement files to be overwritten.

Under no circumstances may results be released as official unless, they are reviewed by a qualified member of the Radiological Environmental Protection Group.

5.4 Sample Disposal

Upon completion of analysis the sample, container, and release documentation are returned to the customer. A copy of the documentation is filed in the Rad Lab archives. If the sample has been released to the Rad

Lab, then the Hazardous Waste group and/or the Radioactive Waste group should be contacted to determine the appropriate disposal method.

6 RECORDS

- 6.1 All analysis results generated from this procedure must be entered into the REP Rad Lab database on the network drive to ensure data access, security control, and backup.
- 6.2 All hardcopies of the Analysis reports generated from this procedure should also be provided and stored in the Rad Lab archives.

7 ATTACHMENTS/FORMS

- 7.1 Attachment 1: Routine Gamma Spec Counting Checklist
- 7.2 Attachment 2: Reference Table for Gamma Spectroscopy samples
- 7.3 Attachment 3: Sample Report

7.1 Attachment 1: Routine Gamma Spec Counting Checklist

Done?	Step No.	Item
[]	1.	Complete Steps 1 – 7 of “Sample Analysis Checklist” (SAC, See General Sample Analysis procedures in the Rad Lab Manual).
[]	2.	Open GammaVision software; assure there is no job in process (see side bar – if counting, you will see updating of “Real” & “Live” time). Note: If the Gammavision software cannot be found on the desktop or the start menu, shortcuts to the programs and the macros can be found on the V-drive under the following path: V:\ESH\OHP\DREP\RAD LAB\HPGe\Gammavision Shortcuts
[]	3.	If a QA check is not required (see Step 7 of SAC), proceed to Step 13 of this procedure. Otherwise, retrieve the H ₂ O-equivalent (epoxy) calibration standard with an assay date of 5/04.
Daily QA Check		
[]	4.	From the drop down menu on the main page, select the detector you wish to run the Daily QA check.
[]	5.	Use top menu bar to select [Services] and [Job Control]. In the “User” folder, select “DailyQACheck” & [Open].
[]	6.	Enter your operator name, e.g. “HBROGONIA”. Click [OK].
[]	7.	Make sure the shielding cave is empty and click [OK] to run the contamination check. The system will count for a preset time of 60 seconds. Wait until further instructions.
[]	8.	When prompted, place the epoxy standard (S/N # 1046-67-1) in the counter, centered on the blue dot & latch the shielding closed Click [OK]. This starts the 30-minute (1800 second) spectrum accumulation (“count”). Gammavision will notify you when the QA is complete. Click [OK] and check the status page.
[]	9.	When the count is complete check the QA Status page by selecting [Acquire] and [QA] then [Status]. A window should pop up displaying the results. Verify that the Background CPS, Total Activity, Peak Shift, and Av. FWHM Ratio parameters are all satisfactory. This is indicated by an “o.k.” next to each value.
[]	10.	If there is a “warning” or “violation” next to any QA parameter, verify that the QA was performed correctly. Confirm that the correct source was used, the cave was empty during the contamination check, etc. Rerun the DailyQACheck macro once more. If the warning/violation persists, notify the REP program mgr. The system may need an adjustment before any samples can be counted.

Gamma Spectroscopy Analysis Procedure

Done?	Step No.	Item
[]	11.	Date & initial the Gamma Spec Source Check log in the Rad Lab System info binder if the source check was successful. Note any corrections made to the system.
[]	12.	Remove the standard from the counter & store it in cabinet in Room 162. Assure both cabinet & room door are locked.
Count the Sample		
[]	13.	<p>From the drop down menu on the main page, select the detector you wish to count the sample. Use top menu bar to select [Services] & [Job Control]. In the “User” folder, select the applicable macro & [Open].</p> <p>The most commonly used 1000-sec macros are:</p> <ul style="list-style-type: none"> • LIQBOTTLE.JOB: ~500 mL of a unit density liquid sample in 500 mL plastic bottle set directly on the detector. Library file used: SLACstd1 (activated nuclides). Calibration file used: the current epoxy calibration. No background subtraction or geometry correction. • SANDBOTTLE.JOB: ~250 mL / ~425 g soil in 500 mL plastic bottle set directly on the detector. Library file used: SLACstd1 (activated nuclides). Calibration file used: current sand calibration. No background subtraction or geometry correction. <p>Note that the ability of the s/w to correctly determine the radionuclides present & to accurately assess activities depends on a good match between the geometry assumed in the macro & the actual analysis geometry. In cases where results are critical (especially for activity), consult the REP mgr if the sample doesn’t match either of the listed macros.</p>
[]	14.	For “Sample Description”, enter the sample ID from the sample label & any other info you wish to add. Click [OK].
[]	15.	Enter your operator name, e.g. “HBROGONIA”. Click [OK].
[]	16.	Verify that the sample bottle (or other container) is sealed closed. If exterior of sample container could be contaminated, wipe it with a disposable towel; discard towel in rad waste bin. (If the sample is not contained in a sealed container, put it in a plastic bag – avoid contamination of the GS system.) Place the sample in the counter, centered on the blue dot, & latch the shielding closed.
[]	17.	For “Spectrum”, enter the same sample ID as in Step 14. Click [OK].
[]	18.	Click [OK] when the “Sample Description” is shown – this starts the 1000 second spectrum accumulation (“count”).
Determine the Results		

Gamma Spectroscopy Analysis Procedure

Done?	Step No.	Item
[]	19.	After the count, there may be an error message if no peaks are identified. If so, click [OK]. Click on [File] then [Save] to save the spectrum. If there are no error messages, proceed to Step 20.
[]	20.	Check the dead time recorded for the count – it should be less than 10%. (If not, notify the REP program mgr.)
[]	21.	Generally, the s/w will automatically display a report. Verify that the report lists the most recent calibration appropriate for the sample & the appropriate library (usually “SLACstd”) as well as any other counting parameters. Check the report to verify that the system parameters match the settings as listed in the reference table in the Rad Lab System Info binder.
	22.	If the report lists the correct system settings, proceed to Step 27. Otherwise close the window. On top menu bar, select “File”, “Recall” & type in the sample ID. Select the file with the .SPC extension.
[]	23.	Use top menu bar to select [Calibrate] & [Recall Calibration]. In the “User” folder, select the most recent calibration for the type of sample counted & then [Open]. Likewise, select the appropriate library file under [Library] and [Select File]. To change any other settings, consult the Rad Lab Administrator or the Gammavision Manual.
[]	24.	Use top menu bar to select [Analyze] & [Peak Search]. If there is no error message, continue with Step 26.
[]	25.	If an error message appears that no peaks are identified, click [OK].
[]	26.	Select [Analyze] & [Entire spectrum in memory], then return to Step 22.
[]	27.	Use the arrows at “Peak” on the sidebar, move to the various peaks of the spectrum & check the identified peaks against the “library match”. Check the displayed spectrum and report. If any nuclides are reported above the MDA, verify the results by checking to see if key and secondary gammas appear, the peaks have a low uncertainty or overlap with any naturally occurring nuclides. Some cases may require a background subtraction or geometry correction. Note: The report shall be reviewed by the REP, DREP or FO manager prior to releasing the results. If the results are as expected, proceed to step 29.
[]	28.	If the results are unexpected (e.g. radioactivity detected in a location or well where it was not expected or none where it was expected), alert the REP, DREP, or FO mgr immediately.
[]	29.	Print the report.

Gamma Spectroscopy Analysis Procedure

Done?	Step No.	Item
[]	30.	On the <i>Radioactivity Analysis Form</i> (or separate sheet), list any nuclides detected & their activities. In most cases, positive GS results should be converted to pCi & be listed on paperwork with units “pCi – GS”.
[]	31.	Remove the sample from the GS & latch the shielding closed.
[]	32.	Complete Steps 9 – end of “Sample Analysis Checklist”.

Gamma Spectroscopy Analysis Procedure

7.2 Attachment 2: Reference Table for Gamma Spectroscopy samples

Last
Updated: 11/13/2006

Sample Type	Ideal Volume	Macro	Count Time	Ideal Container	Library	Calibration	Comments
Water	500 ml	Liquid	1000 seconds	500 ml Nalgene	Slacstd1	NimCalEpoxy2006F.Clb	
Oil	500ml	Liquid	1000 seconds	500 ml Nalgene	Slacstd1	NimCalEpoxy2006F.Clb	
Soil	250 ml	Soil	1000 seconds	500 ml Nalgene	Slacstd1	NimCalSoil2006B.Clb	Use natchain library to find naturally occuring nuclides
Sand	250 ml	Soil	1000 seconds	500 ml Nalgene	Slacstd1	NimCalSoil2006B.Clb	Use natchain library to find naturally occuring nuclides
Concrete	250 ml	Soil	1000 seconds	500 ml Nalgene	Slacstd1	NimCalSoil2006B.Clb	Use natchain library to find naturally occuring nuclides
Resin	500 ml	Liquid	1000 seconds	500 ml Nalgene	Slacstd1	NimCalEpoxy2006F.Clb	
Slurry	500 ml	Liquid	1000 seconds	500 ml Nalgene	Slacstd1	NimCalEpoxy2006F.Clb	If mostly solid, use soil calibration
Swipe	N/A	Swipe	1000 seconds	N/A	Slacstd1	NimCalSwipe.Clb	Calibration to be Created 12-2006
Filter	N/A	Swipe	1000 seconds	N/A	Slacstd1	NimCalSwipe.Clb	Calibration to be Created 12-2006

Notes: Geometry corrections may be needed if volumes differ (consult with REP manager for appropriate actions)

7.3 Attachment 3: Sample Report

ORTEC g v - i (63) wan32 G53W2.06 21-APR-2005 11:42:55 Page 1
Stanford Linear Accelerator Ce Spectrum name: BKGH2OTap.An1

Sample description
Background file for Tap Water

Spectrum Filename: C:\User\BKGH2OTap.An1

Acquisition information

Start time: 21-Apr-2005 11:26:13
Live time: 1000
Real time: 1000
Dead time: 0.01 %
Detector ID: 1

Detector system
MATC-122

Calibration

Filename: CALLBD060404.Clb
LBD060404 NEW LIQ 10 HR DIRECTLY ON DETECTOR E COMBO

Energy Calibration

Created: 04-Jun-2004 07:55:53
Zero offset: 9.550 keV
Gain: 0.994 keV/channel
Quadratic: 6.117E-08 keV/channel^2

Efficiency Calibration

Created: 04-Jun-2004 09:27:20
Type: Polynomial
Uncertainty: 4.076 %
Coefficients: -0.303824 -5.535106 0.494736
-0.077074 0.005428 -0.000133

Library Files

Main analysis library: SLACstd.lib
Library Match Width: 0.500

Analysis parameters

Analysis engine: wan32 G53W2.06
Start channel: 40 (49.30keV)
Stop channel: 2000 (1997.14keV)
Peak rejection level: 50.000%
Peak search sensitivity: 4
Sample Size: 1.0000E+00
Activity scaling factor: 1.0000E+00/(1.0000E+00* 1.0000E+00) =
1.0000E+00
Detection limit method: Currie limit
Random error: 1.0000000E+00

Gamma Spectroscopy Analysis Procedure

Systematic error: 1.0000000E+00
 Fraction Limit: 0.000%
 Background width: average of five points.
 Half lives decay limit: 12.000

ORTEC g v - i (63) wan32 G53W2.06 21-APR-2005 11:42:55 Page 2
 Stanford Linear Accelerator Ce Spectrum name: BKGH20Tap.An1

Activity range factor: 2.000
 Min. step backg. energy 0.000

Corrections	Status	Comments
Decay correct to date:	NO	
Decay during acquisition:	NO	
Decay during collection:	NO	
True coincidence correction:	NO	
Peaked background correction:	NO	
Absorption (Internal):	NO	
Geometry correction:	NO	
Random summing:	NO	

Energy Calibration
 Normalized diff: 1.0000

***** U N I D E N T I F I E D				P E A K		S U M M A R Y *****		
Channel	Peak Centroid Energy	Background Counts	Net Area Counts	Intensity Cts/Sec	Uncert 2 Sigma %	FWHM keV	Suspected Nuclide	
54.74	63.94	90.	34.	0.034	84.22	2.886	AM-241	
1459.77	1460.21	5.	38.	0.038	37.27	3.334	K-40	M

s - Peak fails shape tests.
 D - Peak area deconvoluted.
 M - Peak is close to a library peak.

***** I D E N T I F I E D				P E A K		S U M M A R Y *****		
Nuclide	Peak Channel	Centroid Energy	Background Counts	Net Area Counts	Intensity Cts/Sec	Uncert 2 Sigma %	FWHM keV	
TA-182	58.56	67.74	104.	0.	0.000	0.00	2.621	
CO-57	113.00	121.84	22.	0.	0.000	0.00	0.000s	
CO-57	128.00	136.74	36.	0.	0.000	0.00	0.000s	
CR-51	311.00	318.59	10.	0.	0.000	0.00	0.398s	
SB-125	423.00	429.88	23.	9.	0.009	164.80	0.000s	
SB-125	456.84	463.51	26.	5.	0.005	289.76	2.979D	
BE-7	470.98	477.56	18.	6.	0.006	217.07	2.991D	
SB-125	595.08	600.88	14.	0.	0.000	0.00	3.093	
SB-124	592.00	597.83	5.	2.	0.002	264.11	0.845s	
SB-125	632.21	637.78	13.	4.	0.004	230.97	1.104s	
AG-110M	650.55	656.00	9.	5.	0.005	187.16	0.927s	
Cs-137	656.24	661.66	14.	0.	0.000	0.00	3.142	
AG-110M	672.29	677.61	14.	0.	0.000	0.00	3.155	
AG-110M	701.53	706.67	9.	8.	0.008	121.43	3.178D	
SB-124	717.74	722.78	10.	12.	0.012	95.13	3.190D	
AG-110M	759.15	763.93	5.	0.	0.000	0.00	0.000s	

Gamma Spectroscopy Analysis Procedure

CO-58 807.07 811.56 2. 2. 0.002 305.51 0.812s

ORTEC g v - i (63) wan32 G53W2.06 21-APR-2005 11:42:55 Page 3
Stanford Linear Accelerator Ce Spectrum name: BKGH2OTap.An1

Nuclide	Channel	Energy	Background	Net area	Cnts/sec	Uncert	FWHM
MN-54	830.47	834.81	7.	2.	0.002	428.76	3.277D
CO-56	842.49	846.75	10.	1.	0.001	1382.38	3.286D
AG-110M	880.65	884.67	1.	0.	0.000	0.00	0.000D
SC-46	883.57	887.57	5.	5.	0.005	167.60	1.747s
AG-110M	935.35	939.03	9.	5.	0.005	207.44	0.924s
CO-56	1035.00	1038.07	1.	0.	0.000	0.00	0.000s
FE-59	1096.54	1099.22	3.	0.	0.000	1309.14	3.466D
ZN-65	1112.94	1115.52	5.	7.	0.007	116.73	3.477D
SC-46	1117.97	1120.52	12.	0.	0.000	0.00	3.481D
CO-60	1171.01	1173.23	2.	0.	0.000	0.00	0.928D
TA-182	1185.36	1187.49	2.	5.	0.005	127.88	0.895s
TA-182	1219.50	1221.42	8.	2.	0.002	389.00	3.548D
CO-56	1236.46	1238.28	1.	3.	0.003	156.18	3.559D
NA-22	1272.95	1274.54	3.	4.	0.004	174.74	3.582D
FE-59	1290.07	1291.56	4.	0.	0.000	1531.49	3.593D
CO-60	1329.33	1330.58	1.	1.	0.001	314.96	0.732s
AG-110M	1381.50	1382.42	0.	5.	0.005	89.44	1.988s
AG-110M	1503.00	1503.18	0.	0.	0.000	0.00	0.000s
SB-124	1692.02	1691.04	0.	0.	0.000	0.00	0.000s
CO-56	1771.00	1769.54	0.	0.	0.000	0.00	0.000s

s - Peak fails shape tests.

D - Peak area deconvoluted.

***** S U M M A R Y O F L I B R A R Y P E A K U S A G E *****
- Nuclide - Average ----- Peak -----
Name Code Activity Energy Activity Code MDA Value
microCi keV microCi microCi Comments

BE-7	0.0000E+00	477.56	0.000E+00	%	2.941E-04	G
NA-22	0.0000E+00	1274.54	0.000E+00	%	3.117E-05	G
SC-46	0.0000E+00	1120.52	0.000E+00	%	3.738E-05	G
		889.26	0.000E+00	&	2.929E-05	G
CR-51	0.0000E+00	320.07	0.000E+00	&	1.641E-04	G
MN-54	0.0000E+00	834.81	0.000E+00	%	2.666E-05	G
FE-59	0.0000E+00	1099.22	0.000E+00	%	3.265E-05	G
		1291.56	0.000E+00	%	5.501E-05	G
CO-56	0.0000E+00	846.75	0.000E+00	%	2.975E-05	G
		1238.28	0.000E+00	%	3.572E-05	G
		1771.49	0.000E+00	&	9.520E-05	G
		1037.83	0.000E+00	%	8.664E-05	G

ORTEC g v - i (63) wan32 G53W2.06 21-APR-2005 11:42:55 Page 4
Stanford Linear Accelerator Ce Spectrum name: BKGH2OTap.An1

Gamma Spectroscopy Analysis Procedure

Nuclide	Ave activity	Energy	Activity	Code	Peak MDA	Comments
CO-57	0.0000E+00	122.07	0.000E+00 %		1.812E-05	G
		136.43	0.000E+00 %		1.976E-04	G
CO-58	0.0000E+00	810.75	0.000E+00 %		1.753E-05	G
CO-60	0.0000E+00	1332.51	0.000E+00 &		1.717E-05	G
		1173.23	0.000E+00 &		1.576E-05	G
ZN-65	0.0000E+00	1115.52	0.000E+00 %		7.307E-05	G
AG-110M	0.0000E+00	657.75	0.000E+00 &		3.005E-05	G
		884.67	0.000E+00 %		1.388E-05	G
		937.48	0.000E+00 %		1.045E-04	G
		1384.27	1.557E-04 &		1.145E-04	G
		763.93	0.000E+00 %		9.781E-05	G
		706.67	0.000E+00 %		1.964E-04	G
		1505.00	0.000E+00 ?		1.010E-04	G
		677.61	0.000E+00		2.708E-04	G
SB-124	0.0000E+00	602.71	0.000E+00 &		1.939E-05	G
		1691.04	0.000E+00 %		2.885E-05	G
		722.78	4.964E-04		3.216E-04	G
SB-125	0.0000E+00	427.95	0.000E+00 &		1.101E-04	G
		600.77	0.000E+00 %		1.459E-04	G
		636.15	0.000E+00 &		2.741E-04	G
		463.51	0.000E+00 %		3.377E-04	G
TA-182	0.0000E+00	67.75	0.000E+00		5.325E-05	G
		1221.42	0.000E+00 %		1.320E-04	G
		1189.05	0.000E+00 %		1.806E-04	G
Cs-137	0.0000E+00	661.66	0.000E+00		3.354E-05	G

(- This peak used in the nuclide activity average.

* - Peak is too wide, but only one peak in library.

! - Peak is part of a multiplet and this area went negative during deconvolution.

? - Peak is too narrow.

@ - Peak is too wide at FW25M, but ok at FWHM.

% - Peak fails sensitivity test.

\$ - Peak identified, but first peak of this nuclide failed one or more qualification tests.

+ - Peak activity higher than counting uncertainty range.

- - Peak activity lower than counting uncertainty range.

= - Peak outside analysis energy range.

& - Calculated peak centroid is not close enough to the

