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| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 1 of 9 |
| Document Control Center: (208) 533-4448 | Document Owner: Analytical Laboratories Department Manager | Effective Date: 10/30/2001 |

Manual: Analytical Chemistry Laboratory
Procedures Section 10**USE TYPE 2**Change Number: 10-008

1. PURPOSE

The purpose of this procedure is to identify and discuss the criteria and methods routinely used by the Radiation Measurements Laboratory (RML) analysts to:

- Verify the presence of valid photopeaks in the gamma-ray spectrum for a given spectral analysis
- Verify that the correct radionuclides associated with the photopeaks have been selected.
- Verify that the radioactivity reported for a selected radionuclide is correct.
- This method is used when it is necessary to report results, which are true (real) detections, at the one-sigma (68%) level. It may also be used to confirm that radionuclides are not observable in the spectrum.

2. SCOPE

Analysts using this procedure have the knowledge of the RML computerized gamma-ray spectral analysis routines being used and the meaning of the terminology associated with the analytical process and to have demonstrated the ability to correctly apply the process.

The RML utilizes computerized gamma-ray spectral analyses programs, written specifically for these samples to calculate the radionuclide activity concentrations used in these reports. The results from these programs are examined, evaluated and verified by technically trained and experienced RML personnel.

3. DEFINITIONS

Goodness-of-Fit. How well the shape of a photopeak matches the shape of a Gaussian function.

Intensity Code. The number or numbers assigned to a radionuclide photopeak or photopeaks emission probability, the energy location of the photopeak (efficiency) and the importance (statistical weight) of the photopeak of the same radionuclide. The most important photopeak is designated by the number 1 and others with 2, 3, 4, etc.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 2 of 9 |
|---|--|---|

Multiplets. Photopeaks with energies so close together that the resolution of the spectrometer system does not allow the spectrum continuum to be observed (sensed) between adjacent photopeaks in a given region of the spectrum.

4. PROCEDURES

4.1 Criteria and Methods Evaluation

4.1.1 Uncertainty (%ERR) Criteria

4.1.1.1 Check that the measured uncertainty (statistic + “goodness-of-fit”) on photopeak area calculations is less than 50% (flagged as true positive) (“+”).

NOTE 1: *Evaluations have shown that photopeaks with uncertainties >(30%-35%) can generally be rejected, however, the other criteria in this procedure must be considered before making this conclusion.*

NOTE 2: *The RML has determined that at these uncertainty levels (see NOTE 1), the photopeak(s) is generally not observable (visually) in the (-ray spectrum.*

4.1.1.2 Examine carefully photopeaks with measured uncertainties near 30%-35% with the graphical display programs before they are accepted or rejected.

4.2 Area Counts Criteria

4.2.1 Check area counts; there should be 20 counts or more for photopeaks in a spectrum counted for 20 minutes, which is typically the shortest count time.

NOTE: *This criterion is not valid for samples counted for longer periods of time or for samples with radionuclide radioactivity well above the detection limits (background).*

4.2.2 Examine carefully photopeak areas with less than 20 counts with the graphical display program to determine the condition of the data and the fit.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 3 of 9 |
|---|--|---|

4.3 Photopeak Intensity (I-Code) Criteria

NOTE: *The intensity code “1” photopeak must be present in order to obtain a computer-generated final result for a radionuclide.*

- 4.3.1 Check that the radionuclide intensity code “1” (IC-1) photopeak is present.
- 4.3.2 Investigate other photopeaks of the radionuclide in order to provide additional confidence and data for the radionuclide being selected. Check the isotope summary multi-photopeak analysis results for reasonable agreement between all the fit photopeaks of the radionuclide being investigated. (Check the FIT FLAGS that are associated with each photopeak.)
- 4.3.3 Determine if a directed fit of secondary photopeaks by the computer is necessary to obtain additional support data. The RML computer can also be used to perform interactive fitting of any supporting photopeaks. Compare the results of these secondary photopeaks to the original analysis result of the IC-1 photopeak.

NOTE: *Intensity code “1” photopeaks that do not have “supporting” photopeaks can be selected; however, the selection is dependent on the particular radionuclide and the ability to satisfy the other criteria in this procedure.*

4.4 Energy Calibration Criteria

- 4.4.1 Check the correctness of the energy calibration by verifying the correct location and radionuclide tag of the measured 2614 keV gamma ray (ThTl-232). Other photopeak energies may be used if known and observable. (Check the consistency of energy-bias over the entire spectrum).
- 4.4.2 Check that the peak widths follow the energy equation defining the energy calibration.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 4 of 9 |
|---|--|---|

NOTE: *The observed photopeak energy must be within 1.0 keV of the known energy to receive the radionuclide tag.*

4.4.3 Check carefully observed photopeaks that are from 0.75 keV to 1 keV of the known energy for correct radionuclide tagging and photopeak fitting (using the graphical display programs). Check the closeness of the known peak energy (library) with the analysis energy (line).

4.4.3.1 Evaluate the gamma ray and X-ray peaks with similar energies to assure they are not false tags.

4.5 Fit Flag (Flags) Criteria

4.5.1 Examine fit flags to determine the type of spectral analysis operation performed on the photopeaks.

NOTE: *The printed fit flag value can be a combination of values listed in Appendix B.*

4.6 “Other-Tagged” Radionuclide Criteria

4.6.1 Evaluate “other tags” on isotope summary page(s) to determine whether or not other radionuclides could be present at the photopeak energies being evaluated.

4.6.2 Determine whether the other tagged radionuclide(s) are causing interferences that need to be corrected. (See 4.3.2, 4.4.2, and 4.8.)

4.7 Reduced Chi Square (R-CHI) Criteria

4.7.1 Examine the R-CHI value that indicates how well a photpeak was fit.

NOTE: *The R-CHI value is dependent on statistics (area counts) and peak shape; that is, large peaks and non-Gaussian peaks produce large R-CHI values. The R-CHI value should be considered along with the uncertainty and with respect to the shape of the peak. Also close-lying multiplets can produce large R-CHI values (see VAXGAP report, EGG-2533, May 1988).*

4.7.2 Evaluate photopeaks with an R-CHI greater than 5.0 with the graphical display program.

NOTE: *A R-CHI of 1.0 is considered to be a near perfect fit.*

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 5 of 9 |
|---|--|---|

4.8 Peak Width Criteria

4.8.1 Check peak width.

NOTE 1: *The analysis code will flag (**) measured peak widths when the difference between the measured and the calculated (from the energy calibration) widths are greater than 20%.*

NOTE 2: *Peak widths (FWHM) must be reasonable (see Appendix C).*

4.8.1.1 Examine unusually wide peaks for “multiplet” photopeaks. Any multiplets should be checked with a graphical display and interactive analysis program to see if they are properly resolved.

4.8.1.2 Check unusually wide peaks for possible compton and/or backscatter effects.

4.8.1.3 Check the peak shape relative to the Gaussian fit and verify that the shape is normal for the spectrometer system being used.

NOTE: *In most cases, it will be necessary to make a decision based on the fitting results displayed using the graphical display program (PHA or PANDA).*

4.9 Activity Criteria

4.9.1 Check the detection limits.

NOTE 1: *The activity/concentration measured may be at or above the quoted or estimated RML detection limits (minimum detectable activity) depending upon the radionuclide content of the sample.*

NOTE 2: *Activities determined by “computer spectral” analyses (e.g., effluents and environments) can produce true positive values, positive values and negative values (see Appendix D).*

4.10 Half-Life Criteria

4.10.1 Check the decay time.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 6 of 9 |
|---|--|---|

NOTE: *The “age” of the sample and the decay time before counting must be considered before reporting some radionuclides. The “10 half-life” rule-of-thumb must be used with caution, especially with higher-intensity radionuclide constituents or when certain parent-daughter production relationships exist.*

4.10.2 Check the half-life of the radionuclide being considered. If 10 or more half-lives have elapsed prior to counting a sample or if the decay correction is > 1000, the radionuclide may not be detectable but all other selection criteria should be considered.

NOTE: *Some RML analysis programs reject a radionuclide if the decay correction is greater than 1000.*

4.11 Entry-Error Check

4.11.1 Check the input analysis control parameters shown on the analysis output (efficiencies, flowrates, dates, times, etc.).

4.11.2 Verify and cross-check all input parameter values used in the analysis that are questionable with the official RML Sample and Counting Information form or other information sources.

4.12 Graphical Display (XTP, PHA or GINA) Criteria

NOTE: *The graphical display programs are used only as a tool to aid in the photopeak fitting evaluations and should not be used as the only criteria. All other evaluations must be considered in the final selection or decision.*

4.12.1 Check questionable radionuclide photopeaks with the graphical display or interactive analysis program to determine whether to reject or accept certain radionuclides and/or the results from a photopeak.

4.12.2 IF a radionuclide selection is based heavily on the graphical display results,
THEN print the graphical display results and save the lots with the RML analysis printouts.

4.13 Untagged Photopeak Check

4.13.1 Check all detected photopeaks that did not receive a computer generated radionuclide tag.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 7 of 9 |
|---|--|---|

NOTE: *The number of untagged peaks is shown at the end of the analysis (just prior to the Isotope summary section).*

4.13.2 Investigate and identify the radionuclide(s) associated with any untagged photopeaks that appear to be true positive.

4.13.3 Determine the activity of the radionuclide(s) by hand calculations or by reanalyzing the spectrum using a radionuclide library that will provide the appropriate identification and quantification. Consult with senior technical staff.

4.14 Naturally Occurring K-40, Be-7, and Radon/Thoron Activity Criteria

NOTE: *With environmental air and water samples, the sample origin, collection dates, collection methods and the state of equilibrium must be considered before reporting natural Radon/Thoron daughter activities.*

4.14.1 Check to verify the natural radionuclide activities are either background normal or sample normal.

4.14.2 If no Radon/Thoron parent activity is observed (i.e., Ra-226/Th-232), the state of equilibrium with their daughter is unknown, and the radionuclides cannot be correctly quantified by the computer/spectral analysis program, report natural radionuclides upon specific request of customer or by the advisement of the RML Technical Leader.

4.15 Sample Knowledge Criteria

4.15.1 Acquire as much information as possible about the sample and any history associated with it. This will be very helpful to make final judgments on acceptance or rejection. Consult senior personnel to assist in questionable choices.

4.16 RML Rejection Criteria Codes

4.16.1 Use the rejection codes in Appendix E when rejecting radionuclide results that do not meet the criteria in this procedure or that of the analyst.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 8 of 9 |
|---|--|---|

4.17 Final Selection

4.17.1 Perform final selection of questionable results (e.g., those that do not satisfy the Appendix E criteria) or sensitive results in collaboration with the RML Technical Leader or the RML Data Analysis Work Leader.

4.18 Final Approval

4.18.1 Obtain final approval and signature from one of the above listed personnel or a qualified, designated alternate.

5. RECORDS



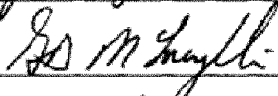



| Records Description | Uniform File Code | Disposition Authority | Retention Period |
|---------------------|-------------------|-----------------------|------------------|
| None | | | |

6. REFERENCES

- 6.1 An Operator Guide to "VAXGAP," ST-CS-027-88, EG&G Idaho, September 1988.
- 6.2 "VAXGAP": A code for the Routine Analysis of Gamma-Ray Pulse-Height Spectra on a VAX Computer, EGG-2533, May 1988.
- 6.3 E. W. Killian and J. K. Hartwell, VAXGAP: A Code for the Routine Analysis of Gamma-Ray Pulse-Height Spectra on a VAX Computer, EGG-2533, May 1988.
- 6.4 E. W. Killian and D. A. Femec, Operator's Guide for VAXGAP, a Gamma-Ray Spectrum Analysis Package, EGG-2672, August 1992.
- 6.5 E. W. Killian and D. A. Femec, Operator's Guide for Using INEL-Specific VAXGAP Programs, EGG-PHY-10338, August 1992.
- 6.6 E. Wayne Killian, PCGAP: Users Guide and Algorithm Description, INEEL/EXT-2000-00908, September 2000.

| | | |
|---|---|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: 9 of 9 |
|---|---|---|

7. APPROVALS

| POSITION TITLE | SIGNATURE | DATE |
|----------------------------------|--|----------|
| Procedure Author |  | 10-29-01 |
| Responsible ALD Technical Leader |  | 10-29-01 |
| Responsible ALD Supervisor |  | 10/30/01 |
| ALD QA Officer |  | 10/30/01 |
| ALD Manager |  | 10/30/01 |
| ALD Facility Manager |  | 10/29/01 |

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|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: A1 of A1 |
|---|--|---|

APPENDIX A

Procedure Basis

| Step(s) | Basis/Summary | Source |
|---------|--|--------|
| All | Verify valid photopeaks in gamma-ray spectrum, and verify correct radionuclides associated with photopeaks have been selected. This procedure is also used to confirm that radionuclides are not observable in a spectrum. | N/A |

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|---|--|--|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: B1 of B1 |
|---|--|--|

APPENDIX B

Fit Flag Value

| Fit Flag Value | Operation |
|----------------|---|
| 0 | -Peak fit by non-linear fitting process |
| 1 | -Peak fit by linear fitting process |
| 2 | -Peak fit by non-linear fitting as part of multiplet |
| 3 | -Peak fit by linear fitting as part of multiplet |
| 5 | -Peak fit by linear fitting-"upper limit" result |
| 7 | -Peak fit by linear fitting as part of a multiplet-"upper limit" result |
| 8 | -Peak background corrected |
| 16 | -Peak background corrected-"upper limit" result |
| 32 | -Peak is interference decontaminated |
| 64 | -Peak interference decontaminated-"upper limit" |
| 128 | -Peak listed as clean in interference library |
| 256 | -Results are converted to "upper limit" in cleanup |
| 512 | -Results are an "outlier"-not used in weighted average |

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: C1 of C1 |
|---|--|---|

APPENDIX C

FWHM Guidelines

| <u>Suggested Guidelines</u> | |
|-----------------------------|-------------|
| <u>Energy (keV)</u> | <u>FWHM</u> |
| 40-300 | 2.0-3.5 |
| 300-600 | 3.0-4.5 |
| 600-1000 | 3.5-5.5 |
| 1000-2000 | 4.5-7.0 |
| 2000-3000 | 6.0-9.0 |

These energy/width guidelines are based on an average of 7 RML detectors. Widths may vary from one detector to another.

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: D1 of D1 |
|---|--|---|

APPENDIX D

GNUL Values

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- True Positive (+) = Activity ≥ 2 std. dev. generally $\leq 50\%$ uncert.
 Positive () = Activity < 2 std. dev. generally $> 50\%$ uncert.
 Negative (-) = Gaussian fit inverted (statistically forced) or ambient background was higher than sample.

EXAMPLES: True Positive = $+(1.0 \pm 0.2)E-08$ $1.0 > 2(0.2)$
 Positive = $(1.0 \pm 0.6)E-08$ $1.0 < 2(0.6)$

| | | |
|---|--|---|
| Laboratory Procedure Analytical Laboratories Department | EVALUATION AND VERIFICATION OF DATA FOR RADIONUCLIDE IDENTIFICATION/SELECTION | Identifier: ACLP-10.31 Revision: 0 Page: E1 of E1 |
|---|--|---|

APPENDIX E

Rejection Codes

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1. Uncertainty too high to be accepted by the analyst.
 2. Area counts too low to be accepted by the analyst.
 3. Radionuclide had no supporting photopeaks to make a judgement.
 4. Energy calibration change and/or gain shift.
 5. Analyst determined that spectral characteristics could not be adequately interpreted by routine analysis.
 6. Reduced CHI-square (photopeak fit) unacceptable by the analyst.
 7. Peak width unacceptable by the analyst.
 8. Radionuclide results below detection limits.
 9. Measured activity was artificially increased by the concurrent background correction.
 10. Other radionuclide gamma-ray interferences.
 11. Graphical display of analyzed photopeaks showed unacceptable photopeak fitting results.
 12. No parent activity, therefore the state of equilibrium is unknown and the radionuclide cannot be quantified.
 13. Naturally occurring radionuclide with expected activity.
 14. Other _____
-