

Analytical Chemistry Procedure

Babcock & Wilcox Technical Services Y-12, LLC Oak Ridge, Tennessee Analytical Chemistry Organization		Y50-AC-65-7230 Revision 0.0 Supersedes: Y/P65-7230, Rev. 0 Page 1 of 8
Level of Use Category		Reference Use
TITLE: PREPARATION OF SAMPLES FOR TOTAL ACTIVITY SCREENING		

Approvals:

J. W. Wade /s/
Manager, Radiochemistry and Bioassay

01-20-2011
Approval Date

01-26-2011
Effective Date

Verified to be latest revision

Initials

Concurrence by the following is documented in the Procedure History File:

Subject Matter Expert
ACO Environmental Compliance
ACO Health & Safety
ACO Quality Assurance

This procedure has been reviewed by a Derivative Classifier and an UCNI Reviewing Official and has been determined to be UNCLASSIFIED and contains no UCNI. This review does not constitute clearance for public release.

Reviewed by: C. R. Horton /s/

Date: 01-19-2011

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REVISION NUMBER	MODIFICATION DESCRIPTION	AFFECTED PAGE(S)
Y/P65-7230, Rev. O	On record	
Y50-AC-65-7230, Rev. 0.0	10-PMR-1020 Reformatted as per Y15-232, <i>Technical Procedure Process</i> Added Level of Use category Modified Title 1.0: Modified Scope 2.0: Updated reference numbers and titles and removed reference to logbooks 4.0: Terminology, added MDA and removed tSIE 12.0: Modified Section 13.0: Modified Section 15.1: Modified Note 2 15.1.2: Modified Step, removed logbook 15.2.3: Modified Step, removed logbook 15.3.2: Modified Step, removed logbook 15.4: Modified Step and deleted Steps 15.4.1 through 15.4.7 15.5: Deleted Section 15.5 and renumbered section 15.6 to 15.5 17.0: Modified Section 19.0: Modified Section 20.0: Added Source Requirements Section Supersedes Y/P65-7230, Rev. O	ALL ALL ALL 3 3 3 5 5 6 6 6 7 7 7 8 8 8

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J. W. Wade is responsible for the accuracy, compliance, and usability of this procedure.

THE USER OF THIS PROCEDURE IS RESPONSIBLE FOR VERIFYING THAT THIS IS THE CURRENT REVISION.

1.0 SCOPE

To provide a method for preparing a variety of samples for total activity screening by liquid scintillation counting.

Batch processing of samples is allowed unless specifically noted. Batch processing may require individual steps, sections, or sequences of the procedure to be repeated.

Identified steps in this procedure do not require step-by-step performance.

This procedure supersedes Y/P65-7230, Rev. O.

2.0 REFERENCED DOCUMENTS

- 2.1 Y15-65-9059, entitled *Analytical Chemistry Organization Procedures*.
- 2.2 Y50-AC-65-7048, entitled *Preparation of Samples for Radiochemical Analysis*.
- 2.3 Y50-AC-65-7068, entitled *Liquid Scintillation Counting on the Packard Tri-Carb 2550 and 3100 TR Series Liquid Scintillation Analyzers*.
- 2.4 Y60-65-0031, entitled *Analytical Balance Performance Verification Procedure*.
- 2.5 Y60-65-9006, entitled *Quality Assurance Plan for the Analytical Chemistry Organization*.
- 2.6 Y60-65-9204, entitled *Control Charts in the Analytical Chemistry Organization*.
- 2.7 Y60-65-9209, entitled *Verification of Pipette Calibration*.
- 2.8 Y73-65-9087, entitled *Chemical Hygiene Plan for the Analytical Chemistry Organization*.

3.0 RESPONSIBILITIES

Refer to Y15-65-9059, entitled *Analytical Chemistry Organization Procedures*.

4.0 TERMINOLOGY

- 4.1 Leachate: A solution obtained by dissolving soluble components of a solid or semi-solid material.
- 4.2 MDA: Minimum detectable activity.
- 4.3 MSDS: Material Safety Data Sheet.
- 4.4 Reflux: To heat so that the formed vapors condense and return to be reheated.

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4.5 TAS: Total activity screen.

5.0 SUMMARY OF TEST METHOD

Aqueous sample aliquots that require no preparation are added directly to the scintillation cocktail. Solid and semi-solid sample aliquots are digested in nitric acid on a hot plate, cooled, filtered, and diluted to a specified volume. Oil sample aliquots are weighed directly into a tared counting vial. A specified volume of liquid scintillation cocktail is added to each vial and mixed with the sample aliquot. The samples are then counted for total activity by Y50-AC-65-7068, *Liquid Scintillation Counting on the Packard Tri-Carb 2550 and 3100 TR Series Liquid Scintillation Analyzers*.

6.0 SIGNIFICANCE AND USE

Department of Transportation regulations require that a total activity screen (TAS) be performed and documented before samples can be transported on public roads (unless there is already sufficient documentation of a sample's radionuclide content).

7.0 INTERFERENCES

Samples known or suspected to contain ⁹⁹Tc should not be heated over 80°C during digestion.

8.0 APPARATUS

- 8.1 Analytical balance.
- 8.2 Pipettor, approximate range 0.25 to 2.0 mL.
- 8.3 Positive displacement pipette.
- 8.4 Disposable scintillation vials.
- 8.5 Disposable transfer pipettes.
- 8.6 Tri-Carb liquid scintillation analyzer.

9.0 REAGENTS AND MATERIALS

- 9.1 Purity of Water: References to water shall be understood to mean water devoid of contaminants that would give rise to uncontrolled interferences in detection of the analytes of interest. Organic free water is required and defined to be when an interferent is not observed at the minimum detection limit (MDL) of the compounds of interest.
- 9.2 Purity of Reagents: Reagent grade chemicals shall be used in all analyses. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades of chemicals may be used, provided it is first documented that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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- 9.3 Liquid Scintillation Cocktail: Packard Ultima GoldLLT or equivalent biodegradable cocktail purchased commercially.

10.0 HAZARDS

- 10.1 Manufacturers' Material Safety Data Sheets containing information on chemical incompatibilities, specific hazards, or spill cleanup steps for any hazardous materials used in this procedure, are accessible on the Y-12 Web Page.
- 10.2 General chemical laboratory hazards are encountered in the performance of this procedure. Important safety information on chemicals, laboratory hoods and personal protective equipment is outlined in Y73-65-9087, *Chemical Hygiene Plan for the Analytical Chemistry Organization*.
- 10.6 Waste shall be disposed of in accordance with the APPROVED Area-Specific Waste Disposal Guide.

11.0 SAMPLING, SUBSAMPLES, AND TEST SAMPLES

Not applicable.

12.0 PREPARATION OF APPARATUS

Refer to Y50-AC-65-7068, *Liquid Scintillation Counting on the Packard Tri-Carb 2550 and 3100 TR Series Liquid Scintillation Analyzers*, for preparation of the liquid scintillation counter.

13.0 CALIBRATION AND STANDARDIZATION

An analytical balance calibration check is performed according to Y60-65-0031, *Analytical Balance Performance Verification Procedure*, and automatic pipettes are verified according to Y60-65-9209, *Verification of Pipette Calibration*. Both are recorded on a daily basis or upon use.

14.0 CONDITIONING

Not applicable.

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15.0 PROCEDURE

NOTE: Each step may be repeated for processing a batch of samples prior to continuing to the next step unless otherwise noted.

15.1 Solid and Semi-Solid Samples

NOTE 1: A reagent blank, a laboratory control sample, a duplicate, and a matrix spike must be prepared and analyzed with each sample batch. Prepare the blank using water as specified in Step 9.1. Refer to Step 19.2 for quality control sample preparation.

NOTE 2: Samples should already be digested according to Y50-AC-65-7048, Preparation of Samples for Radiochemical Analysis.

15.1.1 Pipette an aliquot of the digested sample leachate into a scintillation vial.

NOTE: If possible, a 2.0 ml aliquot should be used; however, based on the color of the sample or sample constituents, a smaller aliquot may be necessary. Contact technical support or supervision if unsure of the aliquot size.

15.1.2 Record the final sample dilution on the appropriate form-

15.1.3 Add 15 mL of scintillation cocktail to the vial.

15.1.4 Secure a labeled cap on the vial.

15.2 Oil Sample Preparation for TAS

NOTE: A reagent blank, a laboratory control sample, a duplicate, and a matrix spike must be prepared and analyzed with each sample batch. Prepare the blank using water as specified in Step 9.1. Refer to Step 19.2 for quality control sample preparation.

15.2.1 Place a scintillation vial on an analytical balance, and tare (zero) the balance.

15.2.2 Transfer an appropriate sample aliquot into the scintillation vial using either a positive displacement pipette or a transfer pipette.

NOTE: If possible, a ~0.25 gram aliquot should be used; however, the amount of sample to use will vary depending upon the color of the sample or sample constituents. Contact supervision or technical support if unsure of the aliquot size.

15.2.3 Record the weight of the aliquot on the appropriate form.

15.2.4 Add 15 mL of scintillation cocktail.

15.2.5 Secure a labeled cap on the vial, and mix well.

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15.3 Aqueous Samples

NOTE: A reagent blank, a laboratory control sample, a duplicate, and matrix spike must be prepared and analyzed with each sample batch. Prepare the blank using water as specified in Step 9.1. Refer to Step 19.2 for quality control preparation.

15.3.1 Pipette an aliquot into a scintillation vial.

NOTE: If possible, a 2.0 mL aliquot should be used; however, the amount of sample to use will vary depending upon the color of the sample or sample constituents, as smaller aliquot may be used. Contact supervision or technical support if unsure of the aliquot size.

15.3.2 Record the aliquot amount on the appropriate form.

15.3.3 Add 15 mL of scintillation cocktail to the vial.

15.3.4 Secure a labeled cap on the vial, and mix well.

15.4 The prepared samples are now ready for counting by Y50-AC-65-7068, *Liquid Scintillation Counting on the Packard Tri-Carb 2550 and 3100 TR Series Liquid Scintillation Analyzers.*

15.5 Retain the scintillation vials until the results have been calculated and approved. Dispose of the scintillation samples according to the APPROVED Area-Specific Waste Disposal Guide.

16.0 CALCULATIONS OR INTERPRETATION OF RESULTS

Not applicable.

17.0 REPORT

All records generated as a result of this procedure are maintained in accordance with Y15-101, *Records and Controlled Documents.*

The Analytical Chemistry Organization is the record copy owner of all records generated by this procedure. Radiochem Access database data sheets are generated by this procedure.

The laboratory analyst documents the following information on the appropriate form.

- Date prepared
- Sample identification number
- Sample aliquot used in procedure
- Final dilution [e.g. (5 g/50 mL x 2 mL) or (2 mL direct)]
- Analyst initials

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18.0 PRECISION AND BIAS

This is a sample preparation procedure. Precision and bias are calculated by the group that performs the analysis. Both the accuracy and the precision of the determinations resulting from this procedure are dependent on how carefully and consistently the preparation is carried out, as well as the analytical instrument and the manner in which instrument calibration is performed.

19.0 QUALITY ASSURANCE/QUALITY CONTROL

- 19.1 The quality control measures defined in Y60-65-9006, *Quality Assurance Plan for the Analytical Chemistry, Organization* shall be adhered to in the performance of this procedure.
- 19.2 A blank, laboratory control sample, duplicate, and matrix spike sample will be analyzed with each sample batch.
- 19.3 Data review consists of 100% peer review by an experienced peer or qualified technical person and 10% supervisory review as per Y60-65-9122, *Data Review and Verification*.
- 19.4 Radiochem Access database software used for this method has been validated and is maintained in the Software Application Manager.

20.0 SOURCE REQUIREMENTS

- Y50-AC-65-7048, *Preparation of Samples for Radiochemical Analysis*
- Y50-AC-65-7068, *Liquid Scintillation Counting on the Packard Tri-Carb 2550 and 3100 TR Series Liquid Scintillation Analyzers*.

APPENDIXES

None.

End of procedure