RESL TECHNICAL PROCEDURE

CHEM-TP-H3.1

THE DETERMINATION OF TRITIUM IN WATER, WASTE WATER, MILK, AND URINE BY LIQUID SCINTILLATION COUNTING

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TITLE: CHEM-TP- H3.1, THE DETERMINATION OF TRITIUM IN WATER, WASTE WATER, MILK, AND URINE BY LIQUID SCINTILLATION COUNTING

PURPOSE

The purpose of this procedure is to describe the method used to determine tritium in water, waste water, milk, and urine samples by liquid scintillation counting. This procedure incorporates and supersedes ACB-TP-H3.01 (Rev 3).

APPLICABILITY

This procedure is applicable to the measurement of tritium in many liquid samples by liquid scintillation counting.

RESPONSIBILITIES

RESL staff responsible for implementing this procedure are:
Radiochemist(s)

DEFINITIONS

HTO - Tritiated water

Dead water - Low-tritium background water

PROCEDURE

1 **ABSTRACT** A commercially available blend of solvent, scintillator, and nonionic surfactant is added to an aliquot of the sample or its distillate. The mixture is shaken thoroughly and the sample is counted in a liquid scintillation spectrometer system.

2 **LIMITATIONS AND INTERFERENCES** Water and waste water samples that are less than pH 5 must be neutralized and distilled; those that are colored, turbid, or that contain high quantities of dissolved solids or high levels of activity other than tritium must be distilled. Milk and urine samples must be distilled. Only the tritium present as HTO will be detected in milk and urine. The spectral capability of the liquid scintillation counter provides greater reliability than older, window-type liquid scintillation counters.

3 **QUALITY ASSURANCE REQUIREMENTS**

3.1 A blank and a calibration standard must be analyzed with each set of samples. Blanks are acceptable if they contain only background quantities of natural radioactivity.

3.2 Correct performance of the liquid scintillation counter must be verified by an Instrument Performance Analysis (IPA) at the end of counting each set of samples. The IPA can be initiated automatically by the instrument.

4 SAFETY REQUIREMENTS

4.1 Follow laboratory safety requirements in CHEM-ACB-AP-002.

4.2 Wear an appropriate lab coat, gloves, and eye protection as addressed in RESL-TP-IH.7.

4.3 Dispose of all wastes in accordance with RESL-AP-10.

5 APPARATUS

5.1 Cocktail dispenser with adjustable volume

5.2 Condensers, water-cooled, with ground-glass fitting at top

5.3 Connecting tubes with ground-glass fittings at both ends

5.4 Eppendorf pipette or equivalent with 10-mL tips

5.5 Kjeldahl flasks, 100-mL, with ground-glass fitting in neck

5.6 Micro Kjeldahl digesting unit with individual heater controls

5.7 Vials, liquid scintillation, 22-mL glass with caps

6 REAGENTS

6.1 Dead water used for background. Obtained from offsite and distilled or demineralized to remove dissolved salts and particles.

6.2 Sodium carbonate, reagent grade, anhydrous, granular.

6.3 ^{3}H standard, E+04 to E+05 dpm/mL.

6.4 Scintillator, ULTIMA GOLD LLT: A commercially available liquid scintillation cocktail which has been demonstrated to be critical for this application.

NOTE: ULTIMA GOLD LLT IS THE ONLY COCKTAIL THAT IS DRAIN DISPOSABLE AND ALSO HAS ADEQUATE SAMPLE CAPACITY.

7 INSTRUMENTATION

INSTRUMENTATION Liquid scintillation, PC-controlled Packard Model 2770 TR/SL Liquid Scintillation Spectrometer.

8 PREPARATION OF CALIBRATION STANDARD AND REAGENT BLANK

8.1 Pipet 1.0 mL of ^{3}H standard solution (E+04 to E+05 dpm) into a glass counting vial containing 9 mL of dead water. Add 12 mL of scintillation cocktail (ULTIMA

GOLD LLT), cap and shake until the mixture clears. This standard will be counted to determine the ^{3}H counting efficiency for the group of samples being analyzed.

8.2 Pipet 10 mL of distilled dead water into a glass counting vial. Add 12 mL of scintillation cocktail (ULTIMA GOLD LLT), cap and shake until the mixture clears. This will be counted as the reagent blank with each group of samples.

9 PREPARATION OF SAMPLES FOR COUNTING

9.1 Begin at Step 9.4 with all production well and monitor well samples submitted by the U.S. Geological Survey (USGS) except those USGS samples listed in EXHIBIT 1. Begin at Step 9.4 with any other samples which, from past experience, have been shown to be suitable for direct counting. Begin at Step 9.2 with distillation of all other samples.

9.2 Pour 40 mL of sample into a 100-mL Kjeldahl flask. Add several boiling chips and about 2 g (more if necessary) of anhydrous, granular Na_2CO_3 slowly to neutralize any acid present. Distill the sample on the Kjeldahl digesting unit. Foaming may be controlled by spraying the sample in the flask with a silicone antifoam spray. Urine and milk samples are particularly susceptible to foaming problems.

9.3 Collect about 25 mL of the distillate in a clean, dry, plastic screw-cap bottle.

9.4 Pipette 10 mL of the distillate from Step 9.3 (or original sample) into a 22-mL glass counting vial. The 10 mL called for in Step 9.4 is standard. The volume is entered in the VAX RESULT program which calculates the result and uncertainties. Add 12 mL of ULTIMA GOLD LLT scintillator.

9.5 Cap the counting vial, shake until the solution clears (about 15 sec), and label the vial cap appropriately for that particular sample.

9.6 Place the samples, the calibration standard, and the reagent blank in the automatic changer of the Packard liquid scintillation counter.

10 **COUNTING** Count the samples, calibration standard, and reagent blank in the Packard Tri-Carb 2770 TR/SL liquid scintillation spectrometer, which is operated by the dedicated instrument computer. Before attempting to count samples in this counter, the analyst should read and become familiar with the Packard Tri-Carb Reference Manual. The Region settings in keV for counting ^{3}H are as follows:

	Lower Limit	Upper Limit	
Region A:	0.0	4.0	^{3}H counting region
Region B:	0.0	10.0	Wider ^{3}H region
Region C:	10.0	2000.0	Monitor higher energy activity

10.1 Count the samples, calibration standard, and reagent blank three times for 20 min. Once the samples, standard, and blank have been counted three (or more) times, mark the printout as to which sample was counted in which position.

10.2 Calculate the ^{3}H counting efficiency (CE) based on the known, decay-corrected dpm added to the calibration standard in Step 8.1. Use the calculated CE, which should be in the range of 0.215 to 0.220, to calculate ^{3}H results for the set of samples.

10.3 Examine Region C cpm values for each sample. They should be statistically the same for all samples whether or not some of the samples contain ^{3}H. Repeat the analysis, using distillation, on samples that were counted directly and found to exhibit elevated cpm values in Region C.

11 **CALCULATION OF RESULTS AND UNCERTAINTIES** Calculate the results and uncertainties using the VAX computer, menu-prompted RESULT program.

REFERENCES

Tri-Carb Liquid Scintillation Analyzers, Model 2770 TR Series Reference Manual Packard Instrument Co., Inc. 1995.

QUALITY RECORDS

Computer hard copy printout of data and results.
The liquid scintillation counter printout
The Sample Record Sheet.

USGS SAMPLES THAT REQUIRE DISTILLATION

USGS #53
USGS #54
USGS #55
USGS #60
USGS #61
USGS #62
USGS #63
USGS #70
TRA #A-13
TRA #A-77