AP6

DETERMINATION OF CARBON-14 AND TRITIUM USING THE BIOLOGICAL MATERIAL OXIDIZER

PART A

PRINCIPLE

The Biological Material Oxidizer (BMO) combusts biological, wet, or semi-liquid, or other non-liquid material in a stream of oxygen gas, passes the products through a series of catalysts, then traps the carbon-14 and tritiated water into the prescribed trapping scintillation cocktail.

REFERENCE

1. Biological Material Oxidizer Manual, OX-500, R. J. Harvey Corp.

2. ANSI N42.15 - 1997 *American National Standard* Check Sources for and verification of Liquid Scintillation Systems.

Certification Record for

PROCEDURE AP6

DETERMINATION OF CARBON-14 AND TRITIUM USING THE BIOLOGICAL MATERIAL OXIDIZER

CHECKPOINTS

1. **JOB HAZARD ANALYSIS (JHA)** _____
2. **MSDS/HAZARDS DISCUSSED** _____
3. **EQUIPMENT SET-UP/POWER ON** _____
4. **BACKGROUND AND SPIKES** _____
5. **SAMPLE PROCESSING** _____
6. **COUNTING SET-UP OF LSC** _____
7. **FINAL CALCULATION(S)** _____

ANALYST'S SIGNATURE: _____

CERTIFIED BY: _____

DATE: _____

ANALYSIS VALUE: _____

KNOWN VALUE: _____

MEASURED/KNOWN: _____

See Task _____, Batch_____ for the original data.

Comments:_____
_____
_____
_____

PART B

1.0 PURPOSE AND SCOPE

This procedure provides the analytical method for determination of carbon-14 and tritium in biological material, wet or semi-liquid, or other non-liquid material.

2.0 REAGENTS

Carbon-14 (C-14) calibration standard, NIST traceable

Carbon trapping scintillation cocktail: Available from RJ Harvey Instrument Company

Methanol, CH_3OH, ACS reagent

Mannitol, $C_6H_{14}O_6$, ACS reagent

Tritium (H-3) calibration standard, NIST traceable

Tritium trapping scintillation cocktail: In a 1L beaker, add 856mL mixed Xylenes, 44mL methoxy 2-propanol, 66mL isopropyl alcohol and 33mL Igepal and mixed with stirring. Add 62g AOT, 0.54g Bis-MSB and mixed to dissolve. Finally add 6.5g White Fluor and dissolve. Filter through a glass fiber filter. Transfer to a dark glass bottle and purge with nitrogen at a low flow rate. Cap tightly.

3.0 APPARATUS

Biological Material Oxidizer
Boats
Cool-down setup for ladles and boats
Forceps
Ladles
Scintillation Vials

4.0 PROCEDURE

4.1 General Requirements

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the Quality Program (QP) Manual. See page two for a copy of the certification record.

A batch yield sample must be run with each batch to determine chemical recovery for the batch (see below for calculations). This is not a QC sample; two QC samples must be run with each batch.

4.2 Instrument Preparation

4.2.1 Insert a ladle in the combustion chamber and secure it with the ladle holder.

4.2.2 Place a dispensing tip and a scintillation vial for the tritium and C-14 delivery spouts.

Note: Check each dispensing tip to ensure there is an opening.

Note: The purge and combustion gases will not flow through the system unless both scintillation vials are in place.

4.2.3 Turn the unit on.

4.2.4 The LED display will show "WAIT/NITROGEN".

4.2.5 Open nitrogen flow valve fully at tank. Adjust flow to 350 cc/min, which is displayed on the LED, using the nitrogen adjustment knob below the LED panel. **See steps 4.2.5 - 4.2.6 of AP6 JHA.**

4.2.6 Open oxygen flow valve fully at tank. Push the "OXYGEN FLOW" button and hold. Adjust flow to 350 cc/min, which is displayed on the LED, using the oxygen adjustment knob below the LED panel. **See steps 4.2.5 - 4.2.6 of AP6 JHA.**

4.2.7 Push "SYSTEM TEST" button. Initially eights, stars and zeros will be displayed to demonstrate that all LED segments are working. The oven temperature readings, ~675 °C and ~900 °C, will be displayed. The display will return to the normal operating mode.

4.3 Preparation for Sample Processing

4.3.1 Press the appropriate button(s) for the isotope of interest, either tritium or C-14. If performing dual analysis, both buttons should be selected.

Note: An isotope button must be selected for the instrument to operate.

4.3.2 Ensure that the scintillation cocktails have been connected to the instrument using Teflon tubing and that each valve is turned to the open position. If the scintillation cocktails are not connected, refer to page 7 of the operating manual. **See steps 4.3.2 - 4.3.3 of AP6 JHA.**

Note: These scintillation cocktails must be located in a fume hood.

CAUTION: CHECK THAT THE NITROGEN FLOW RATE IS AT APPROXIMATELY 350 cc/min BEFORE GOING TO THE NEXT STEP.

4.3.3 Push the "PRIME" button and hold until the vial(s) are half full of scintillation cocktail and make sure that the nitrogen is bubbling through each trapping cocktail. Discard the priming cocktails into the organic waste container for xylene. Replace the scintillation vials. **See steps 4.3.2 - 4.3.3 of AP6 JHA.**

4.3.4 Select the cycle time of 1.5 minutes.

Note: If the function display shows "WAIT", do not proceed to the next step until "READY" appears on the display. "READY" indicates the ovens have reached operating temperatures.

4.3.5 Once "READY" is displayed, push the "START" button. After the cycle has completed, check the volume in the vials. Each vial should have 15 mL of cocktail. Discard the cocktail(s) in the organic waste. If the volumes are not 15 mL, repeat this step. **See steps 4.3.2 - 4.3.3 of AP6 JHA.**

4.4 Sample Processing

4.4.1 Select the cycle time: 2, 3, or 4 minutes. This will depend upon the combustibility of the material.

4.4.2 Select C-14, H-3, or both.

Note: If analyzing C-14 only, cut off about one-eighth of an inch from the tritium dispensing tip to prevent clogging.

4.4.3 Insert new tips and scintillation vials for each blank, sample, or Laboratory Control Standard (LCS) to be processed.

Note: Check each dispensing tip to ensure there is an opening.

4.4.4 Insert an empty ladle into the combustion chamber.

4.4.5 Place blank, sample, or LCS into a clean combustion boat. Follow the weight information below for each sample type:

4.4.5.1 For C-14 blanks - weigh approximately 25 mg of mannitol.
For H-3 blanks - add 0.1 mL of reagent water.

4.4.5.2 For C-14 LCS - weigh approximately 25 mg of mannitol and add the appropriate C-14 standard just before processing.
For H-3 LCS - add the appropriate H-3 standard just before processing. **See step 4.4.5.2 of AP6 JHA.**

4.4.5.3 Samples - weigh 300 mg for fecal, weigh 1-2 g for soil, and 1 g or less for plant material. Refer to page 13 of the operating manual for weights of other material. **See step 4.4.5.3 of AP6 JHA.**

4.4.6 Place the combustion boat containing the material to be analyzed in a ladle. **See step 4.4.6 of AP6 JHA.**

4.4.7 Press "START".

4.4.8 After 22 seconds, an alarm will sound and "INSERT/SAMPLE" will appear on the display. Remove the blank ladle, place on the cool-down holder, and insert the ladle with the material to be analyzed. The ladle containing the sample in the combustion boat must be inserted before the next alarm sounds. The operator has approximately 20 seconds to complete the operation. **See step 4.4.6 of AP6 JHA.**

Note: The ladle that is removed from the combustion chamber will be very hot. Care must be taken when removing the ladle from the combustion chamber and placing it on the cool-down holder. The ladle must be placed so that the hot end cannot be accidentally touched.

4.4.9 When the cycle has ended, a third alarm will sound and "CYCLE/OVER" is displayed. Wait for "READY" to appear on the display. The vial may now be removed.

4.4.10 Remove the ladle containing the combustion boat from the combustion chamber and carefully place it on the cool-down holder. Refer to the note between 4.4.8 and 4.4.9. **See step 4.4.6 of AP6 JHA.**

4.4.11 Repeat steps 4.4.3 through 4.4.10 until the batch has been completed.

4.4.12 Clean the outside of each sample vial, place in a sample tray, and take the samples to the counting room. Refer to CP4 of the laboratory manual for sample counting instructions.

Note: Section 4.5 must be completed before the analyst leaves for the day. If the methanol rinse is not performed at the end of the analyses, the scintillation cocktails will crystallize and clog the dispensing mechanism.

4.5 Instrument Shut Down

4.5.1 After all samples have been processed, turn the cocktail flow controls to the off position. Refer to page 7 of the operating manual.

4.5.2 Place a ladle in the combustion chamber.

4.5.3 Put tips and scintillation vials on the dispensing taps.

4.5.4 Press "PRIME" and hold until the scintillation vials are almost completely full of the methanol rinse. **See steps 4.3.2 - 4.3.3 of AP6 JHA.**

4.5.5 Remove the vials with the methanol rinse from the instrument and discard in the organic waste container. **See steps 4.3.2 - 4.3.3 of AP6 JHA.**

4.5.6 Turn off the nitrogen and oxygen gas supplies at the tank valves. **See steps 4.2.5 - 4.2.6 of AP6 JHA.**

4.5.7 Remove the ladle from the combustion chamber and place on the cool-down holder. Refer to the note between steps 4.4.8 and 4.4.9. **See step 4.4.6 of AP6 JHA.**

4.5.8 Turn off the instrument.

4.5.9 Purge both C-14 and H-3 scintillation cocktails with nitrogen for at least 10 minutes each to retard the oxidation of the cocktails.

5.0 CALIBRATIONS

5.1 Tritium Calibration

5.1.1 Select a NIST traceable tritium standard and prepare the instrument starting at section 4.2

5.1.2 To a combustion boat, add approximately 200 pCi of the tritium standard, proceed to step 4.4.7. **See step 4.4.5.2 of AP6 JHA.**

5.1.3 Once the standard has been processed and a labeled cap placed on the scintillation vial, repeat the process two more times to have a total of three standards for determining the counting efficiency for tritium.

5.1.4 Submit the standards for counting. The counting statistics for the standard should be 1 percent or less.

5.1.5 The Laboratory Manager or designee must review and approve the counting efficiency.

5.2 C-14 Calibration

5.2.1 Select a NIST traceable C-14 standard and prepare the instrument starting at section 4.2

5.2.2 To a combustion boat, add approximately 200 pCi of the C-14 standard, proceed to step 4.4.7. **See step 4.4.5.2 of AP6 JHA.**

5.2.3 Once the standard has been processed and a labeled cap placed on the scintillation vial, repeat the process two more times to have a total of three standards for determining the counting efficiency for C-14.

5.2.4 Submit the standards for counting. The counting statistics for the standard should be 1 percent or less.

5.2.5 The Laboratory Manager or designee must review and approve the counting efficiency.

6.0 CALCULATIONS

The following data will be recorded and reduced according to the following equations:

$$Concentration = \frac{G - B}{E \cdot Y \cdot Q} = pCi / unit$$

$$2\sigma\ Error = \frac{1.96\sqrt{(G + B) \cdot T}}{T \cdot E \cdot Y \cdot Q} = pCi / unit$$

$$2\sigma\ TPU = C \cdot 1.96\sqrt{(\frac{G + B}{(G - B)^2}) \cdot T + RE^2 + RY^2 + RQ^2} = pCi / unit$$

$$MDC = \frac{3 + 4.65\sqrt{B \cdot T}}{T \cdot E \cdot Y \cdot Q} = pCi / unit$$

$$E = \frac{G_E - B}{E_{ACT}} = cpm / pCi$$

$$Y = \frac{(G_{BY} - B) - [(G - B) * \frac{BSQ}{Q}]}{E \cdot Y_{ACT}} = no\ units$$

Where:

B	=	background counts/minute
BSQ	=	batch yield sample quantity
C	=	concentration in pCi/unit
E	=	counting efficiency in cpm/pCi (H-3 or C-14)
E_{ACT}	=	efficiency activity in pCi (H-3 or C-14)
G	=	sample gross counts/minute
G_E	=	efficiency sample gross counts/minute
G_{BY}	=	batch yield sample gross counts/minute

MDC	=	minimum detectable concentration
Q	=	sample quantity
RE	=	1σ relative uncertainty of the efficiency
RQ	=	1σ relative uncertainty of the quantity
RY	=	1σ relative uncertainty of the yield
T	=	count time in minutes
TPU	=	total propagated uncertainty
Y	=	H-3 or C-14 batch yield
Y_{ACT}	=	yield activity in pCi (H-3 or C-14)

7.0 RECORDS

7.1 Reference QP Manual for record requirements.

7.2 The raw count data is saved during the weekly backup of the Liquid Scintillation counter to the ORISE network disks.

7.3 Hard copies of assignment and calculation sheets are maintained in the archived task file. Electronic copies of assignment and calculation sheets are saved during the daily incremental backup of the network system. The following data sheets should be completed and retained:

- H-3 or C-14 Analysis Assignment Form
- H-3 or C-14 Lab Data Sheet
- H-3 or C-14 Concentration and Uncertainty Report (This report may be generated using approved Excel spreadsheets or from the database, if available.)

AP6(Rev 17) - C-14 BY BMO ANALYSIS ASSIGNMENT FORM

Assigned To: _____ **Date:** _____ **Batch:** _____

Task #: _____ **LWR #:** _____ **Activity Level*:** _____

Sample #s: _____

_____

_____

Analysis Required:

Batch Yield ☐ **Sample #** _____
Initial below sample

C-14 STD # _____ **Quantity:** _____
Units: _____

Eff. Spike ☐ **C-14 STD #** _____ **Quantity:** _____
(see Special Instructions, if any) **Units:** _____

QC Required:

Blank ☐

LCS ☐ **C-14 STD #** _____ **Quantity:** _____ **Initials** ☐
Units: _____

Pipette # _____ Volume (mL) _____ Weight (g) _____

Replicate ☐ **Sample #** _____ **# Replicates:** _____

Matrix Spike ☐ **Sample #** _____ **Initials**

C-14 STD # _____ **Quantity:** _____ ☐
Units: _____

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

* If Activity Level is indicated as Moderate or High, perform area survey

COMMENTS: _____

_____

_____

_____

AP6(Rev 17) - C-14 BY BMO LAB DATA SHEET

			BATCH YIELD	SAMPLE			
Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #			
Quantity			
Units			

AP6(Rev 17) - H-3 BY BMO ANALYSIS ASSIGNMENT FORM

Assigned To: _____ **Date:** _____ **Batch:** _____

Task #: _____ **LWR #:** _____ **Activity Level*:** _____

Sample #s: _____

_____

_____

Analysis Required:

Batch Yield ☐ **Sample #** _____
Initial below sample

H-3 STD # _____ **Quantity:** _____
Units: _____

Eff. Spike ☐ **H-3 STD #** _____ **Quantity:** _____
(see Special Instructions, if any) **Units:** _____

QC Required:

Blank ☐

LCS ☐ **H-3 STD #** _____ **Quantity:** _____ **Initials** ☐
Units: _____

Pipette # _____ Volume (mL) _____ Weight (g) _____

Replicate ☐ **Sample #** _____ **# Replicates:** _____

Matrix Spike ☐ **Sample #** _____ **Initials** ☐

H-3 STD # _____ **Quantity:** _____
Units: _____

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

* If Activity Level is indicated as Moderate or High, perform area survey

COMMENTS: _____

_____

AP6(Rev 17) - H-3 BY BMO LAB DATA SHEET

			BATCH YIELD	SAMPLE			
Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #			
Quantity			
Units			

AP6 (Rev 17) - Carbon-14 by BMO Concentration and Uncertainty Report

INPUT BY:

DATE:

TASK#

BATCH#

Batch Yield (BY) Calculation	
BY sample cpm	
BY sample quantity	
BY sample quantity error	
Sample cpm	
Sample quantity	
Sample quantity error	
BY pCi added	
BY pCi added error	
BY	
BY Error	
BY Relative Error	

Efficiency (Eff) Calculation	
Eff spike cpm	
Background cpm	
pCi added	
pCi added error	
Eff (cpm/pCi)	
Eff Error (cpm/pCi)	
Eff Relative Error	

Counting time for efficiency and yield calculations (min)	60

Position #	SAMPLE ID	GROSS cpm	QUANTITY	QUANTITY ERROR	UNITS	TIME (min)	CONCENTRATION	TPU	4.65 sigma MDC
1									
2									
3									
4									
BY									
BY Sample									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									

C-14 Known	Unc.	Meas/ Known	Unc

BLANK CORRECT? YES[ ] NO[ ] INIT_____

LCS CORRECT? YES[ ] NO[ ] INIT_____

BATCH YIELD CORRECT? YES[ ] NO[ ] INIT_____

IF NO, SPECIFY REASON:

ANALYST REVIEW: _____ DATE: _____

REVIEWED BY: _____ DATE: _____

GIVEN TO: _____ DATE: _____

AP6(Rev 17) - Tritium by BMO Concentration and Uncertainty Report

INPUT BY:

DATE:

TASK#

BATCH#

Batch Yield (BY) Calculation	
BY sample cpm	
BY sample quantity	
BY sample quantity error	
Sample cpm	
Sample quantity	
Sample quantity error	
BY pCi added	
BY pCi added error	
BY	
BY Error	
BY Relative Error	

Efficiency (Eff) Calculation	
Eff spike cpm	
Background cpm	
pCi added	
pCi added error	
Eff (cpm/pCi)	
Eff Error (cpm/pCi)	
Eff Relative Error	

Counting time for efficiency and yield calculations (min)	60

Position #	SAMPLE ID	GROSS cpm	QUANTITY	QUANTITY ERROR	UNITS	TIME (min)	CONCENTRATION	TPU	4.65 sigma MDC
1									
2									
3									
4									
BY									
BY Sample									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									

H-3 Known	Unc.	Meas/ Known	Unc

BLANK CORRECT? YES[ ] NO[ ] INIT_____

LCS CORRECT? YES[ ] NO[ ] INIT_____

BATCH YIELD CORRECT? YES[ ] NO[ ] INIT_____

IF NO, SPECIFY REASON:

ANALYST REVIEW: _____ DATE: _____

REVIEWED BY: _____ DATE: _____

GIVEN TO: _____ DATE: _____