QAM-RI-106

Operation and Calibration of the Tri-Carb Liquid Scintillation Counter

Revision: 3

Approval:

______________________ 12-2-20

Laboratory Manager Date

______________________ 12/2/20

RSO Date

Effective Date: 12-11-20

Renewal Date: ______________________ Initials: ______________________

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1.0 Applicability and Purpose

A. The purpose of this procedure is to provide a method for the proper operation and calibration of the Packard Tri-Carb Liquid Scintillation Counter. By utilizing associated sample preparation SOPs, safety and quality methods, analysts can produce accurate and reproducible data in an efficient manner with reduced hazards to themselves and equipment.
B. This method applies to the TIAER Laboratory at Tarleton State University, Stephenville, Texas.
C. Liquid Scintillation Counting (LSC) is a technique used for the detection and quantification of reactivity. This measurement technique is applicable to all forms of nuclear emissions (Alpha and Beta particle, electron capture, and gamma ray emitting radionuclides). Scintillation is proportional to the activity of the radioisotope. The Tri-Carb 1000 liquid scintillation analyzer is designed for quantitative detection of beta radiation, other forms of nuclear radiation and various types of luminescence.

2.0 Definitions

A. Counts per minute (CPM) - number of radiation events detected by a monitoring instrument.
B. Background- natural radioactivity in the environment and/or laboratory equipment that is of a high enough energy to be detected by instrumentation (e.g. LSC)
C. Dead Time- Occurs when radiation events occur very close in time and are therefore not detected due to counting circuitry or other interference.
D. Quenching-interference in counting energy resulting in lower efficiency, often attributed to highly colored samples, chlorine compounds, etc.
E. Scintillation-the release of light photons from a radioactive material when mixed with certain chemical compound mixtures (scintillation cocktails)
F. Compton scattering- Elastic scattering of photons (x or gamma-rays) by electrons. In each such process the electron gains energy and recoils and the photon loses energy.

G. Refer to QAM-R-100, "TIAER Laboratory Radiochemistry Program" for general definitions.

3.0 Equipment and Reagents

A. Tritium standard, ^{3}H, Perkin Elmer part #160-TOL or equivalent (also Refer to SOP-RC-103 "Determination of Tritium by Liquid Scintillation")
B. Carbon 14 standard, ^{14}C, Perkin Elmer part #188-TOL or equivalent.
C. Glass liquid scintillation analyzer vials with caps

4.0 Procedure

A. The efficiency of the Tri-Carb 100 liquid scintillation analyzer will be calculated during the initial calibration procedure. If the efficiency is not above the acceptable minimum (58% specifically for tritium) then a Self-Normalization and Calibration Procedure is done and the efficiency is then retested to determine if it is within acceptable limits. The background tritium activity is tested and samples are prepared according to the Sample Set-Up procedure. Only after all these steps have been adhered to can the samples be counted according to the Sample Count Procedure.
B. Calibration Procedure: (using tritium, ^{3}H)
   1. Ensure the RS-232 line is connected to the computer.
   2. Initiate conversion by pressing the CONV key (Refer to Conversation Flow Diagram in Attachment 1). Once the CONV key is pressed the system asks for the conversation mode. Select EDIT(1).
   3. Select the protocol number for ^{3}H (previously established) and enter that number via the keypad. Press the ENTER key. The LCD will display a series of protocol questions.
   4. Use the system assigned values, with the following exception:

      # COUNTS/VIAL ? Enter 3

   5. Press the CONV key.
   6. Load an unquenched ^{3}H standard and initiate counting.
   7. Calculate the efficiency as follows (Refer to the Radioactive Decay Tables in Attachment 2 to determine the corrected ^{3}H DPM value).

$$\% \ EFF = \frac{CPM \ of \ Region \ A - Background \ Activity \ (CPM)}{Corrected \ \text{3H DPM Value}} x \ 100$$

8. The minimum acceptable efficiency for ^{3}H is 58%
9. If the ^{3}H efficiency does not meet the minimum, perform a system Self-Normalization and Calibration and repeat the test. If the results are still not within the defined limits, contact the laboratory manager.

C. Self-Normalization and Calibration Procedure (daily when used)
1. Load the unquenched ^{14}C standard into the sample holder.
2. Initiate counting by pressing the SNC key on the system's keypad.
3. After the system has completed self-normalization and calibration, remove the standard until needed again.

D. Background Check Procedure
1. Initiate conversion by pressing the CONV key. Once the CONV key is pressed the system asks for the conversation mode. Select EDIT(1).
2. Select the protocol number for BKGD (previously defined) and enter that number via the keypad. Press the ENTER key. The LCD will display a series of protocol questions.
3. Use the system assigned values, with the following exception:

   # COUNTS TIME ? Enter 10
   # RADIONUCLIDE ? Select Manual
4. Press the CONV key.
5. Load the unquenched background standard (blank cocktail) and initiate counting.
6. If the background displayed in region A exceeds 30 CPM for any of the 3 measurements, inform the laboratory manager.

E. Sample Set Up
1. In the event of one or more missing replicates, leave a matching number of empty positions in a replicate group.
2. The number of standards and samples must not exceed the replicates defined in the protocol.
3. For standards and samples counted more than once, provide the number of replicates to be averaged. Repeat counts will provide a single average count.

4. “Skipped” positions are treated and reported as missing vials and are not averaged. The SKIP key is pressed for each missing vial
5. Standards and samples should be sequenced in the following order.
   a. First vial count background
   b. Quench standards of low energy isotope
   c. Quench standards of high energy isotope
   d. % of reference
   e. Samples and QC samples (per individual SOPs).
6. If any of the standards are not called for in the protocol, all other standards and samples move up the sequence order.

F. Counting Samples
1. Ensure that the power is applied to the system.
2. Ensure that the printer has adequate paper for the run, and that the printer is in the ON-LINE mode.
3. Load first vial in sequence as described in “Sample Set Up”
4. The system is not ready for conversion. Conversation is used to program and edit the appropriate protocol parameters and select the desired output format. If the protocol has already been programmed, there is no need for conversion.
5. Press the COUNT key on the keypad to begin the counting process.
6. If the protocol other than the active protocol is desired terminate the current protocol and select the new active protocol for counting.

G. Changing the Active Counting Protocol
1. To select a new protocol for counting, the current active protocol is first terminated. This is done by entering conversation (Press the CONV key), selecting EDIT(1) and entering the active protocol number. The system will ask if the protocol is to be terminated. Answer yes, and end conversation.
2. Now enter conversation with the protocol which is to become the new active protocol.

$$\% \, EFF = \frac{CPM\ of\ Region\ A}{Corrected\ \mathrm{3H\ DPM\ Value}} x\ 100$$

3. The corrected ^{3}H DPM Value can be found by using Attachment 2 in the Radioactive Decay Tables as a fraction of the original amount.
4. The minimum acceptable efficiency for ^{3}H is 58%

H. Data Handling and Reporting
1. Data is transferred to an Excel E-log using an RS-232 connection to the computer system.
2. Record all times, dates, calculations and recoveries on the E-log for transfer to the ESDMS LIMS.

5.0 Quality Control and Safety Aspects

A. All aspects of this procedure comply with QAM-R-100, “TIAER Laboratory Radiochemistry Program”, QAM-Q-101, “Laboratory Quality Control”, QAM-W-101, “Disposal of Laboratory Waste”, and QAM-S-101, “Laboratory Safety”.

B. All data are documented and maintained in accordance with, QAM-A-102, “Document and Data Control”. All raw data are recorded in the appropriate logbook or E-log.

C. Current activities for source checks are found in the Calibration Source Log, Q-102-4. Document source ID and source dates.

D. Check the calibration label before use to ensure that the instrument has been calibrated. Counting equipment is typically calibrated every 12 months, or more often as needed.

E. Always take into account background radiation levels and record the most recent with all data. Measure and record background radiation levels in each E-log analytical batch run. Background values must be within +/- 20% of the initial background average used for calibration or notify the LM.

F. The buildup of static electricity can cause erratic results especially when plastic tubes are used, use glass vials to reduce static build up, unless HF is used in sample prep.

G. Control chart all standards, blanks and spikes in accordance with QAM-Q-100, “Laboratory Quality Control”.

6.0 References

A. Tri-Carb 1000 LCS Operation Manual, Packard Instruments, (1986), Zurich, Switzerland

B. USEPA Method 906, Tritium in Drinking Water.

C. 2016 TNI Accreditation Standards

7.0 Attachments

A. Attachment 1, Conversation Flow Diagram
B. Attachment 2, Radioactive Decay Tables

Attachment 1: Conversation Flow Diagram

Conversation	
Edit (1)	Set Time (2)
protocol # ? Integer: (1-10)	Time of Day ? Integer: Month (1-12)
Time ? Range: (.10-999.99) minutes	Time of Day ? Integer: Day (1-31)
# Counts/Vials? Integer: (1-99)	Time of Day ? Integer: year (0-99)
#Vials/Samples? Integer: (1-9)	Time of Day ? Integer: Hour (0-23)
Vials/Standards? Integer: (1-9)	Time of Day ? Integer: Minute (0-59)
% Referance No (0) Yes (1)	
Data mode CPM (1) SPC (2)	

Attachment 2

RADIOACTIVITY DECAY TABLES

GENERAL DECAY TABLE

The General Decay Table is used to determine the fractions of activity remaining for any radionuclide. Divide the time elapsed (t) by the half-life (T) of the radionuclide to calculate the number of half-lives expired (t/T). Using the General Decay Table, find the fraction of activity remaining.

FRACTION OF ACTIVITY REMAINING

HALF LIVES (t/T)	.00	.01	.02	.03	.04	.05	.06	.07	.08	.09
0.0	-	.993	.986	.979	.973	.966	.959	.953	.946	.940
0.1	.933	.927	.920	.914	.908	.901	.895	.889	.883	.876
0.2	.871	.865	.859	.853	.847	.841	.835	.829	.824	.818
0.3	.812	.807	.801	.796	.790	.785	.779	.774	.768	.763
0.4	.758	.753	.747	.742	.737	.732	.727	.722	.717	.712
0.5	.707	.702	.697	.693	.688	.683	.678	.674	.669	.664
0.6	.660	.655	.651	.646	.642	.637	.633	.629	.624	.620
0.7	.616	.611	.607	.603	.599	.595	.591	.586	.582	.578
0.8	.574	.570	.567	.563	.559	.555	.551	.547	.543	.540
0.9	.536	.532	.529	.525	.521	.518	.514	.511	.507	.504
1.0	.500	.497	.493	.490	.486	.483	.480	.476	.473	.470
1.1	.467	.463	.460	.457	.454	.451	.448	.444	.441	.438
1.2	.435	.432	.429	.426	.423	.421	.418	.415	.412	.409
1.3	.406	.403	.401	.398	.395	.392	.390	.387	.384	.382
1.4	.379	.376	.374	.371	.369	.366	.364	.361	.359	.356
1.5	.354	.351	.349	.346	.344	.342	.339	.337	.335	.332
1.6	.330	.328	.325	.323	.321	.319	.316	.314	.312	.310
1.7	.308	.306	.304	.301	.299	.297	.295	.293	.291	.289
1.8	.287	.285	.283	.281	.279	.277	.276	.274	.272	.270
1.9	.268	.266	.264	.263	.261	.259	.257	.255	.254	.252
2.0	.250	.248	.247	.245	.243	.241	.240	.238	.237	.235
2.1	.233	.232	.230	.229	.227	.225	.224	.222	.221	.219
2.2	.217	.216	.215	.213	.212	.210	.209	.207	.206	.205
2.3	.203	.202	.200	.199	.198	.196	.195	.193	.192	.191
2.4	.190	.188	.187	.186	.184	.183	.182	.181	.179	.178
2.5	.177	.176	.174	.173	.172	.171	.170	.168	.167	.166
2.6	.165	.164	.163	.162	.160	.159	.158	.157	.156	.155
2.7	.154	.153	.152	.151	.150	.149	.148	.147	.146	.145
2.8	.144	.143	.142	.141	.140	.139	.138	.137	.136	.135
2.9	.134	.133	.132	.131	.130	.129	.129	.128	.127	.126
3.0	.125	.124	.123	.122	.122	.121	.120	.119	.118	.117

HALF LIVES (t/T)