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Liquid Scintillation Counting - Packard Triple-Label Calibration

P. A. Torretto

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	FUNCTIONAL AREA IMPLEMENTING PROCEDURE **Liquid Scintillation Counting – Packard Triple-Label Calibration**			
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Procedure Use Level:

☐ **Continuous**

☒ **General**

☐ **Informational**

Preparer: Philip C. Torretto, RML Group Leader (AS&I)

Reviewer: Lori Collins, Quality Assurance Officer (ES&H)

Approver: V.A. Michele Sundsmo, Manager (AS&I)

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LIQUID SCINTILLATION COUNTING – PACKARD TRIPLE-LABEL CALIBRATION

1. PURPOSE

1.1. Purpose.

1.1.1. The Radiological Measurements Laboratory (RML) maintains and operates nine Packard Liquid Scintillation Counters (LSCs). These counters were obtained through various sources and were generally purchased as 2500, 2700 or 3100 series counters. In 2004/2005 the software and firmware on the counters were upgraded. The counters are now designated as 3100 series counters running the Quantasmart software package. Thus, a single procedure can be used to calibrate and operate the Packard LSCs.

1.1.2. The LSCs are used to analyze a variety of samples including swipes, air filters, bioassay samples and environmental samples. Analyses include tritium, C-14, P-32, I-125, I-131, gross alpha and gross beta. Samples are submitted by the Health & Safety Technicians, Bioassay Laboratory Analyst or Analytical Laboratory Analysts.

1.1.3. In order to provide accurate results for Triple-Label, each of the counters must be calibrated for Tingle-Label (tritium,C-14,P-32) nuclides using a NIST traceable standard. For the P-32, calibrations the RML creates standard sets consisting of ten glass scintillation vials containing known quantities of NIST traceable standards mixed with scintillation cocktail. A quenching agent is added to each of the standard vials to provide a range of quench from low to highly quenched. The tritium and C-14 standard sets are NIST traceable standards purchased through Perkin-Elmer Life Sciences for this purpose. The purchased standard sets consists of ten sealed glass ampoules containing a known concentration of Tritium or Carbon 14 mixed with liquid scintillation cocktail. A quenching agent is added to each of the standard vials in order to provide a range of quench from unquenched to highly quenched.

1.1.4. Calibration of each instrument provides a quench versus efficiency curve. This curve is used to determine the counting efficiency for each sample analyzed based on the measured quench of the sample using the transformed Spectra of the Internal Standard (tSIE) mode.

2. APPLICABILITY

2.1. Scope and Application.

2.1.1. This procedure provides detailed procedures for performing the Triple-Label calibrations on the Packard LSCs running the Quantasmart software package. The resultant calibration curve (efficiency versus quench) is used for the analysis

of Triple-Label nuclides (H3, C-14, P-32,) in swipes, air samples, bioassay samples and environmental samples.

2.1.2. This procedure also establishes the control limits for the Triple-Label Resident QC Sealed Glass Vial Set (QC-31 to QC-37). The Triple-Label Resident QC Set consists of four blank spike samples: tritium; carbon-14; Sr/Y-90; and a mixture of tritium, carbon-14 and Sr/Y-90; along with a blank and two background QCs for a total of seven resident QCs. The Triple-Label Resident QC Set is run at the beginning of the Triple-Label assay and processed as samples.

2.2. **Summary of Method.**

2.2.1. The process of system calibration involves multiple steps which must be completed before an instrument can be used for analysis. These steps include: 1) creation of computer data files to record the calibration information and establishing a counting protocol for the calibration; 2) analyzing each of the calibration standards; 3) verification of the results for the calibration standards; 4) establishing the control limits for the QC samples; 5) entering the control limits into RML Calibration Editor; and 6) review of the calibration information. Detailed instructions for each of these steps are given in Section 6.

2.2.2. Examples of the documentation for a completed calibration are included in the LSC Triple-Label and GAB-H3 Calibrations Binder. With experience, the operators will find that the order of the processes as given in this procedure does not have to be followed exactly, and can be reordered somewhat to speed up the act of placing a specific analyzer back in operation.

3. EXCEPTIONS

3.1. **Exceptions.**

3.1.1. None.

3.2. **Limitations.**

3.2.1. None.

3.3. **Interferences.**

3.3.1. It is critical that the amount of activity in the standard vials is well known. Any biases in the known activity of the standard will lead to biases in the analytical results for samples. Standards used for the calibration shall be traceable to a National Standards Laboratory such as NIST.

3.3.2. The energy of the beta-emitting radionuclide affects the instrument response for both efficiency and quench. Analytical results for samples containing radionuclides other than tritium, carbon-14 or phosphorus-32 may be biased.

Generally, results from the Triple-Label analysis produces results which are conservatively biased high for radionuclides other than tritium or carbon-14.

3.3.3. Use of a purchased sealed glass quench set for calibration assumes that the effect of quench on the efficiency is the same for the samples as for the quench standards. Theoretically this is true. However, any differences in the effects of quench on efficiency will lead to a bias in the analytical results.

3.3.4. Excitation of the scintillation cocktail by natural light will create artificially high counts. Once started the sample preparation should be performed in its entirety in order to preserve the integrity of the preparation conditions and to ensure the overall integrity of the sample. The samples shall be dark-adapted for a minimum of one hour prior to counting using the SNC samples at the beginning of all calibration runs. This will minimize sudden excitation of the scintillation cocktail that can create artificially high counts.

4. PREREQUISITES

4.1. Precautions.

4.1.1. The *ES&H Manual* and Radiological Measurements Laboratory IWS describe hazards and controls applicable to this procedure.

4.1.2. Prior to beginning this procedure, the analyst must be familiar with the hazards associated with the chemicals listed in Section 4.3 of this procedure. This information may be found in the Safety Data Sheets for the chemicals.

4.1.3. Standards used for instrument calibration are radioactive and should be handled and disposed of according to policies described in the *ES&H Manual*.

4.1.4. All RML personnel who perform instrument calibration shall be current on the required safety training which is included in LTRAIN.

4.2. Equipment.

4.2.1. Packard Series 3100 Liquid Scintillation Counter running Quantasmart software.

4.3. Reagents and Supplies.

4.3.1. Carbon-14 standard set - NIST traceable, purchased from Perkin-Elmer Life Sciences, approximately 125,000 DPM Carbon-14 in Ultima Gold scintillation cocktail. (Note: The Carbon-14 standard set has an expiration date and must be replaced periodically.)

4.3.2. Ultima Gold liquid scintillation cocktail, purchased from Perkin-Elmer Life Sciences.

4.3.3. Tritium standard set – NIST traceable, purchased from Perkin-Elmer Life Sciences, approximately 250,000 DPM tritium in Ultima Gold scintillation cocktail. (Note: This standard set has an expiration date and must be replaced periodically)

4.3.4. Tritium standard solution – NIST traceable.

4.3.5. Super Polyethylene Vials, 20mL.

4.3.6. Liftaway concentrated decontaminant.

4.3.7. Glass Vials with polyseal caps, 20mL.

4.3.8. Sr/Y-90 standard solution – NIST traceable.

4.3.9. P-32 standard set – Prepared from NIST traceable solution, 100,000 – 900,000 DPM P-32 in Ultimagold scintillation cocktail.

4.4. Quality Control.

4.4.1. After determining the quench versus efficiency curve for the calibration standards the QC Coordinator or the Group Leader reviews the plotted curve for any outlying points. If outliers are found either the standards are re-measured or the outliers are dropped from the curve and the curve re-determined. This curve is then stored by the instrument. The final curve is verified by measuring each of the standards as a sample and determining the deviation of the measured DPM to the known DPM.

5. ROLES AND RESPONSIBILITIES

5.1. RML Group responsibilities.

5.1.1. The RML QC Coordinator is responsible for maintaining this procedure.

5.1.2. Calibrations may be performed by the RML analyst. However, the calibrations shall be reviewed and approved by the RML QC Coordinator or the RML Group Leader prior to use.

5.1.3. Responsibilities for the RML Group members are described in the Quality Assurance Plan.

6. PROCEDURE

6.1. Calibration Preparation.

6.1.1. Server File Generation

A. Locate the following directory on the RML-SERVER X: drive: X:\RML_CALIBRATIONS_IN_PROGRESS

B. Create sub-folders for each of the LSC counters to be calibrated. Label each of the folders CTR_X_CALIB, X signifying the analyzer to be calibrated.

C. Create a NEW DATE sub-folder to correspond to the date of the start of the latest system calibration for the designated counter, EXAMPLE: MMM_DD_YYYY.

D. Create the following four sub-folders within the NEW DATE sub-folder:

WORKING_DATA_FILES
CALIB_UPDATE_HISTORY
FINAL_QC_FILES
RAW_DATA_RUN_FILES

E. All data files and documents pertaining to the analyzer calibration will be stored in the four sub-folders.

F. Start a temporary manila calibration folder for the LSC counter being calibrated. The folder will contain the documents that will be produced throughout the calibration process. The finished documents will be transferred to the calibration binder for the specific analyzer when the calibration is complete.

6.1.2. Certificate of Radioactivity

A. Place a copy of the NIST traceable standards certificates in the manila calibration folder, EXAMPLES: Carbon-14 Master Calibration Standards Set, Tritium Master Calibration Standards Set, and P-32 Standards Solution certificate.

B. Go to the Tools Tab on the QuantaSmart main window. Click TOOLS than Nuclide Decay. Enter the information from the Quench Standards or Solution Set Certificate and then Click Start Decay button. Write down the Current DPM Activity which will be used later in the Quench Calibration.

6.1.3. Preparation of the Triple-Label Resident QC Glass Vial Set (QC31 – QC37)

A. Acquire radioactive standard solutions: tritium, C-14, Sr/Y-90 for producing the Triple-Label Resident QC Glass Vial Set.

B. Calculate the volume required to obtain approximately 1000 DPM for each of the standards. (If this volume exceeds 1.00 mL use a more concentrated standard solution.) Calculate today's decay corrected activity and document your calculations.

C. Get 7 glass 20mL vials with polyseal lids. With a black permanent marker label the lids:

Counter ID = X
Name = QC-XX (QC31 to QC-37)
Analysis = Triple-Label (TL)
Todays Date = MM-DD-YY

D. Prepare the resident QC vials by putting 10mL of UltimaGold scintillation cocktail and a swipe paper in each vial.

E. Working in a chemical fume hood, aliquot the amounts of each of the standards along with de-ionized water into each of the vials as indicated in Table 1 below. A excel workbook is located in the X:\RML_CALIBRATION_IN_PROGRESS\TEMPLATES_H3_C14_P32 folder to assist with determining the volumes of standards and water to use.

		H-3 Std.	C-14 Std.	Sr/Y-90 Std.	H_2O
QC-31	Blank	-----	-----	-----	1.00 mL
QC-32	Background	-----	-----	-----	1.00 mL
QC-33	H-3	~1000 DPM	-----	-----	[1]
QC-34	C-14	-----	~1000 DPM	-----	[1]
QC-35	Sr/Y-90	-----	-----	~1000 DPM	[1]
QC-36	H3/C-14/Sr/Y-90	~1000 DPM	~1000 DPM	~1000 DPM	[1]
QC-37	Background	-----	-----	-----	1.00 mL

Table 1 – Triple-Label Resident QC Glass Vial Set (QC31 to QC37)
1. Add de-ionized water to bring aqueous volume to 1mL

F. Seal the vial with RTV Translucent Adhesive and shake the vial.

G. Count the Triple-Label Resident QC Glass Set on a LSC using Assay 15. Confirm that the measured values agree with the calculated values.

6.2. **Determining Quench Calibration.**

6.2.1. Protocol 50 3H_Quench, Protocol 51 14C_Quench, Protocol 52 32P_Quench,

A. Go to the Assays “3H_Quench" “14C_Quench” and “32P_Quench”. If the assays have not been created, you will need to create the assays starting from Setup Assay for Quench Calibration (6.2.2). If the assays have been created Click on each "(3H_Quench” “14C_Quench” “32P_Quench” and go to the Count Conditions Tab. Follow the instructions in 6.2.4.A and 6.2.4.B to enter the current DPM Activity.

6.2.2. Setup Assay for Quench Calibration

A. Before defining a Quench Standards assay, you must create a new assay by selecting New Assay from the File menu. The Select Assay Type window is displayed.

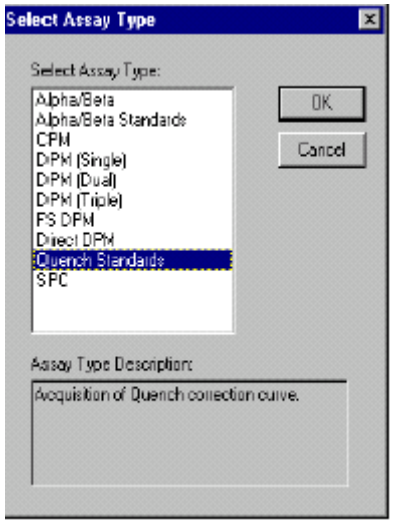

Figure 1 - Select Assay Type Window

B. Select Quench Standards from the Select Assay Type window and click OK.

6.2.3. Assay Parameters Tab

A. Designate your name as author and provide descriptive information for the assay, EXAMPLE: Perkin Elmer Glass (3H,or C-14,or P-32) Quench Set in UG.

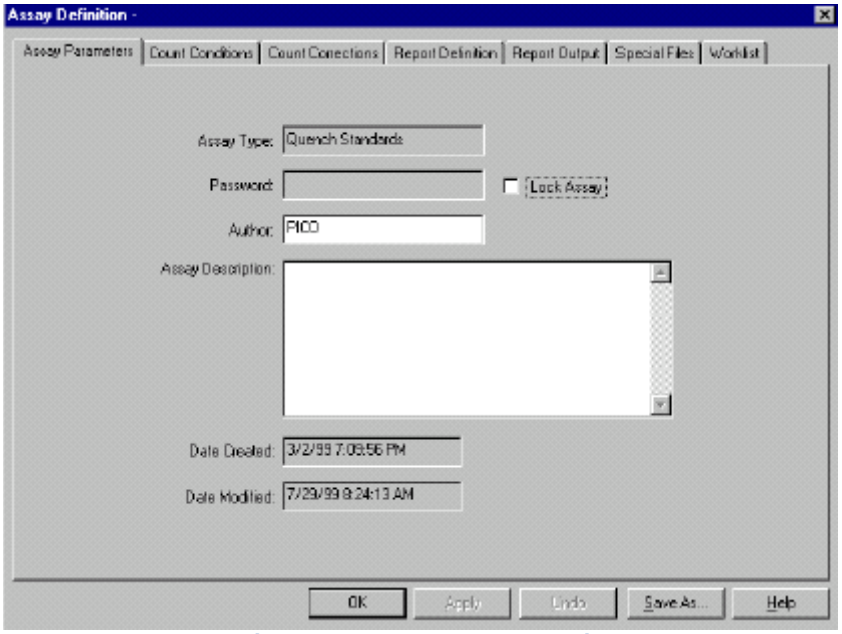

Figure 2 - Assay Parameter Tab

6.2.4. Count Conditions Tab

A. The Count Conditions tab will allow you to define specific counting parameters for the assay.

EXAMPLE: (TL-3H-UG-X)

NAME:	(TL-3H-UG-X) (TL-14C-UG-X) (TL-32P-UG-X) (X is counter name)
Count Mode:	Normal
Quench Indicator:	tSIE/AEC
External STD Terminator:	0.5 2s%
Pre-Count Delay:	0.00
Count Time (min):	30.00
2Sigma%Terminator:	ON (0.50)

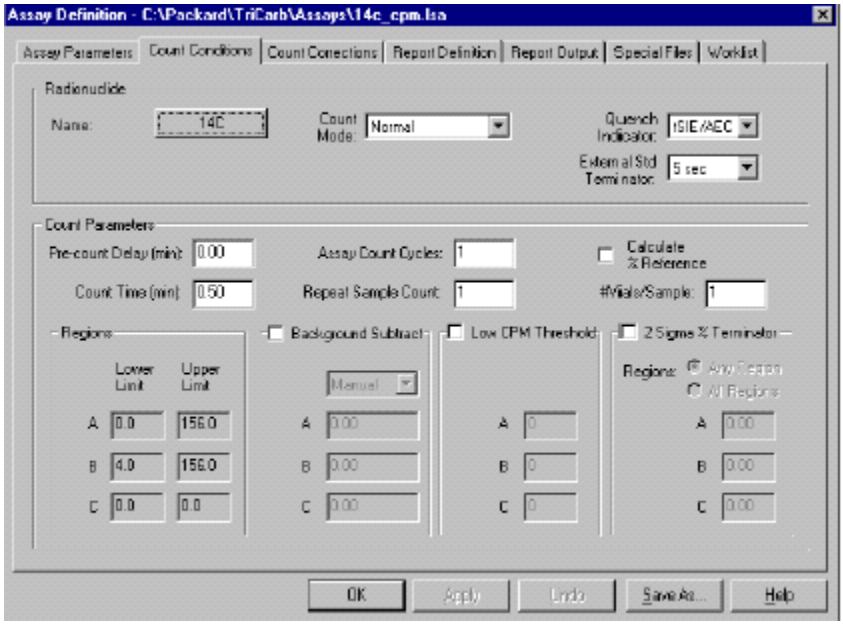

Figure 3 - Count Conditions Tab

B. Click the NAME button to display the Quench Standards Library window. Enter today's DPM value from the Quench Standard Certificate calculated earlier.

EXAMPLE: (TL-3H-UG-X)

NAME:	TL-3H-UG_X TL-14C-UG-X TL-32P-UG-X (X is counter name)
Max KeV:	(3H=18.6, C-14=156, P-32=1710,)
DPM:	XXX,XXX

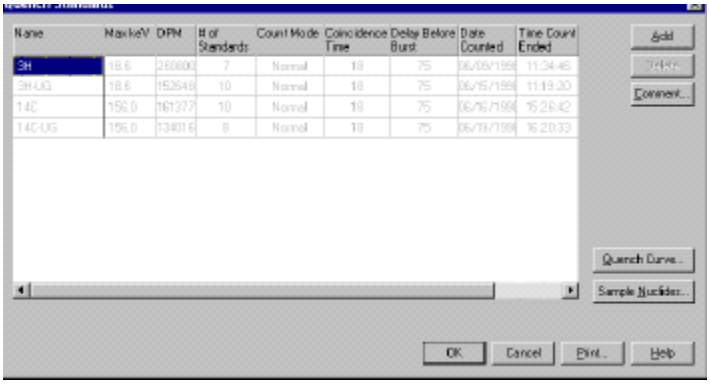

Figure 4 - Quench Standards Library Window

NOTE
If you only needed to enter the current DPM Activity in the Quench Standards Library than proceed to 6.2.8. Print a copy of Assays (3H,14C,32P) Quench.

6.2.5. Count Corrections Tab

A. The Count Corrections tab will allow you to activate certain instrument devices for the assay.

Static Controller:	ON
Coincidence Time:	(nsec)18
Delay Before Burst:	(nsec)75
Half Life Correction:	OFF

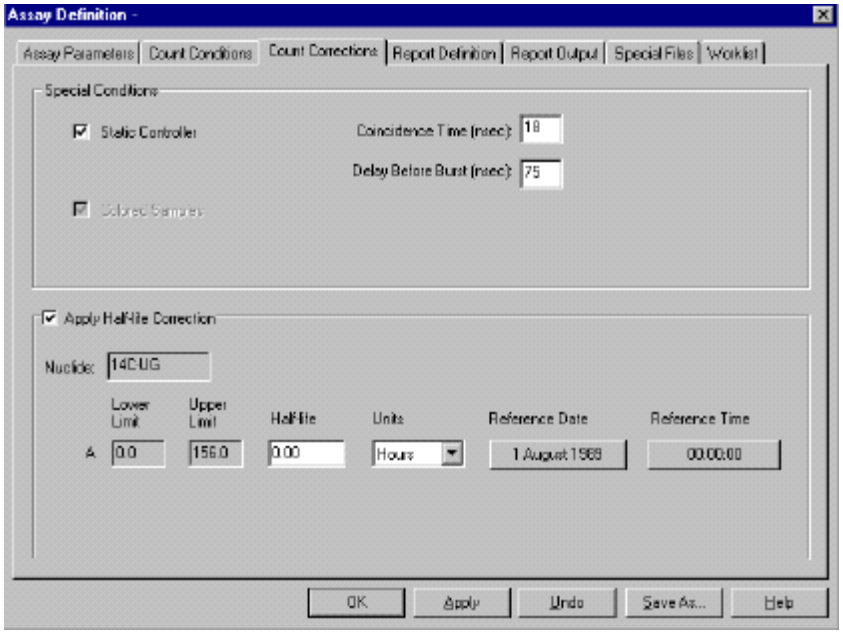

Figure 5 - Count Corrections Tab

6.2.6. Report Definition Tab

A. The Report Definition tab will allow you to specify the data items that are reported and the format for reporting.

EXAMPLE: (3H_Quench)

Report Name:	3H_Quench
	14C_Quench
	32P_Quench
Report Field Order:	S#
	Count Time
	CPMA
	EffA(%)
	SIS
	tSIE
	Flag
Report Block Data:	
Assay Definition:	ON
Quench Curve:	ON

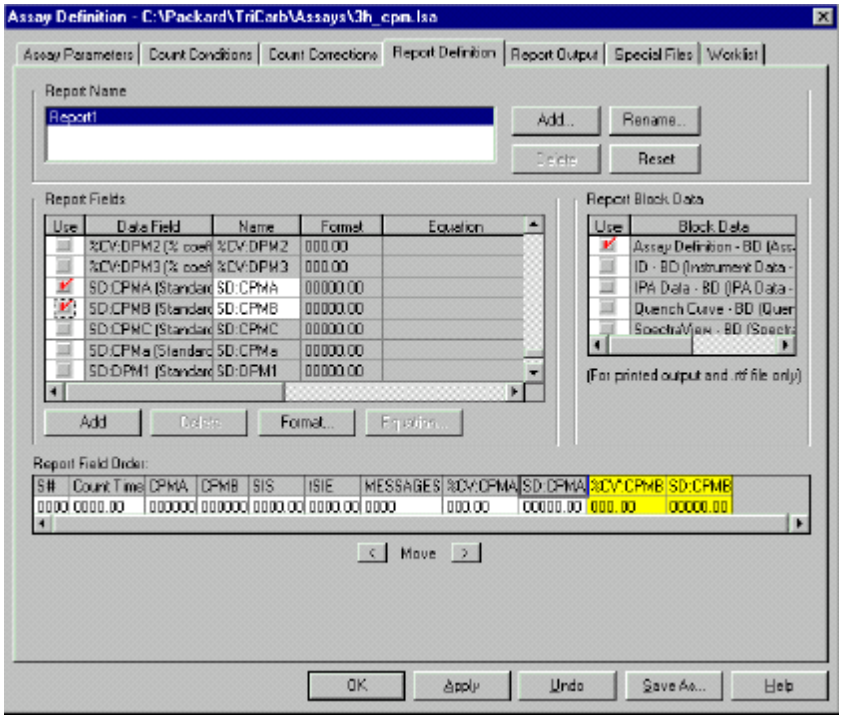

Figure 6 - Report Definition Tab

6.2.7. Report Output Tab

A. The Report Output will allow you to specify the output reports.

EXAMPLES: (3H_Quench)

Report Name:	3H_Quench 14C_Quench 32P_Quench
Output to Printer:	ON
Data File:	ON
File Type:	Delimited Text
File Name:	3H_Quench 14C_Quench 32P_Quench
Output Per:	Sample
Include Column Header:	ON
Rich Text File:	ON
File Name:	3H_Quench 14C_Quench 32P_Quench
Run Application:	OFF

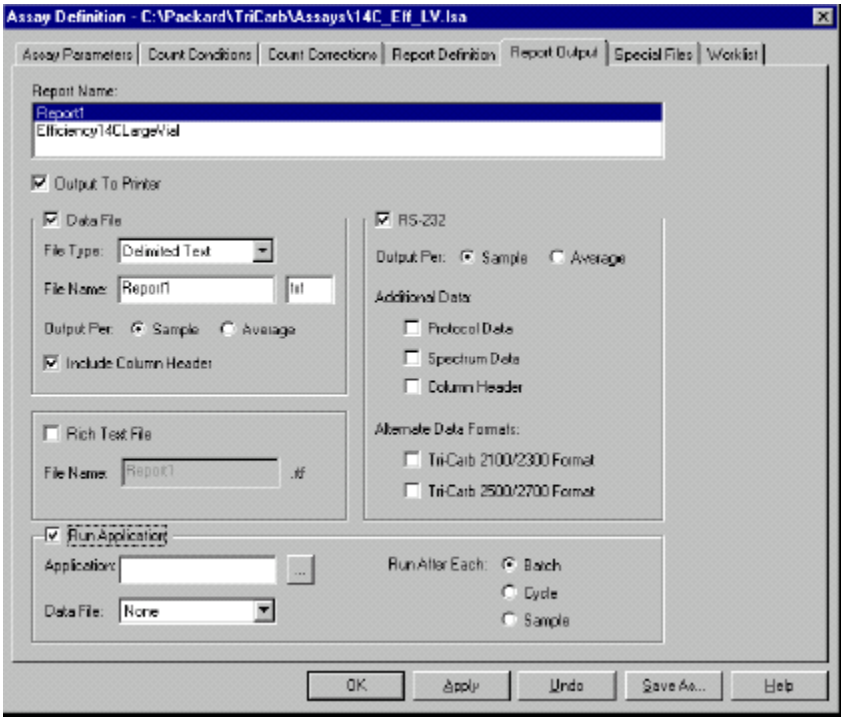

Figure 7 - Report Output Tab

6.2.8. Print a copy of Assays “3H_Quench” “14C_Quench” and “32P_Quench”.

A. Click FILE than Print Assays “3H_Quench” “14C_Quench” and “32P_Quench”. Put the copies in the manila calibration folder.

6.2.9. Load Counter for Calibration Run with the assay tab out.

SNC Rack
Assay (50,) Rack (3H Master Quench Standards 1 to 10)
Assay (51) Rack (C-14 Master Quench Standards 1 to 10)
Assay (52) Rack (P-32 Master Quench Standards 1 to 10)
Assay 59 Halt Rack

6.2.10. Starting the Calibration Run

A. Click the GO button. When the run has finished put the printed copies of the Calibrations in the manila calibration folder.

6.2.11. Processing of the Calibration Run

A. Transfer the run using the RML & WBC Swiss Army Knife Transfer Program. Copy the files (XJJJYYSS.dat, XJJJYYSS.txt) from X:\DATA\YYYY\X_LSC to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY\RAW_DATA_RUN_FILES.

B. From the printer get the copies of H3_Quench, 14C_Quench, and 32P_Quench plot data.

C. Copy file TEMPLATE_CTR_X_U15_H3_C14_P32_RECALIB_PLOT.XLS from X:\RML_CALIBRATIONS_IN_PROGRESS\TEMPLATES_H3_C14_P32 to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY\WORKING_DATA_FILES.

D. Open the plot template file and "save as" CTR_X_U15_H3_C14_P32_RECALIB_PLOT.XLS. (where X is the counter name)

E. Edit the header with the proper counter name, date, and quench set information. Fill QUENCH# (tSIE/AEC) and EFF (%) table block with the information on the count standard printouts found in the manila calibration folder. Populate the equation co-efficient box with the data on the quench versus efficiency plot.

F. Edit "SETUP" Header/Footer tab to put the file name in the header. Set default printer to network color printer and print plot page. Click on the plot and print plot only. Put copies of the plots in the manila calibration folder. Delete the file TEMPLATE_CTR_X_U15_H3_C14_P32_RECALIB_PLOT.XLS from the WORKING_DATA_FILES folder.

G. Click on each Assay "Protocol 50, 3H_Quench" "Protocol 51, 14C_Quench" "Protocol 52, 32P_Quench" and go to the Count Conditions Tab.

H. The Count Conditions tab will allow you to define specific counting parameters for the assay. Return the Nuclide NAME to its original name.

NAME:	(3H-UG-X) (14C-UG-X) (32P-UG-X) (X is counter name)

6.3. **Quench Calibration Verification.**

6.3.1. Assay 15 “3H_14C_32P_Triple_Label”.

A. Go to the Assay 15 “3H_14C_32P_Triple_Label”. If the assay has not been created, you will need to create the assay starting from Setup Assay 15 for Quench Calibration Verification in Step 6.3.2. If the assay has been created Click on 3H_14C_32P_Triple_Label and go to the Count Conditions Tab. Edit the Count Time for 30 minutes and Click OK. Follow the instructions in 6.3.7 to print a copy of assay 3H_14C_32P_Triple_Label.

6.3.2. Setup Assay 15 for Quench Calibration Verification

A. Before defining a Triple-Label assay, you must create a new assay by selecting “New Assay” from the File menu. The Select Assay Type window is displayed.

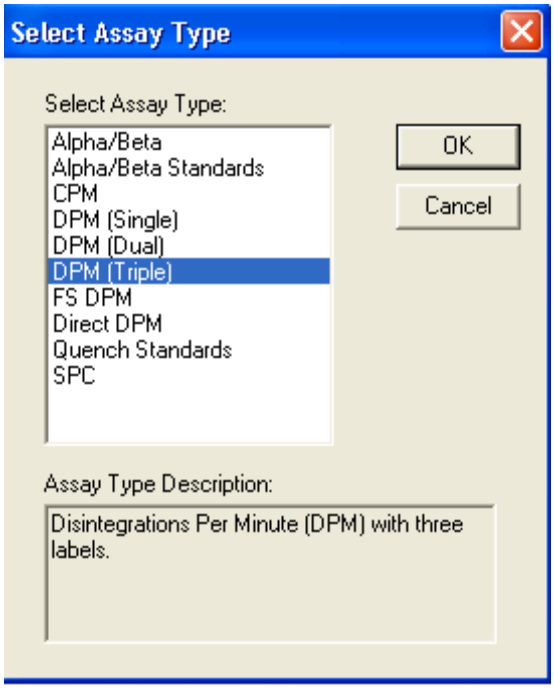

Figure 8 – Select Assay Type Window

B. Select DPM (Triple) from the Select Assay Type window and Click OK.

6.3.3. Assay Parameters Tab

A. Designate your name as author and provide descriptive information for the assay, Example: Triple-Label H3, C14, P32 for Swipes, and Liquids.

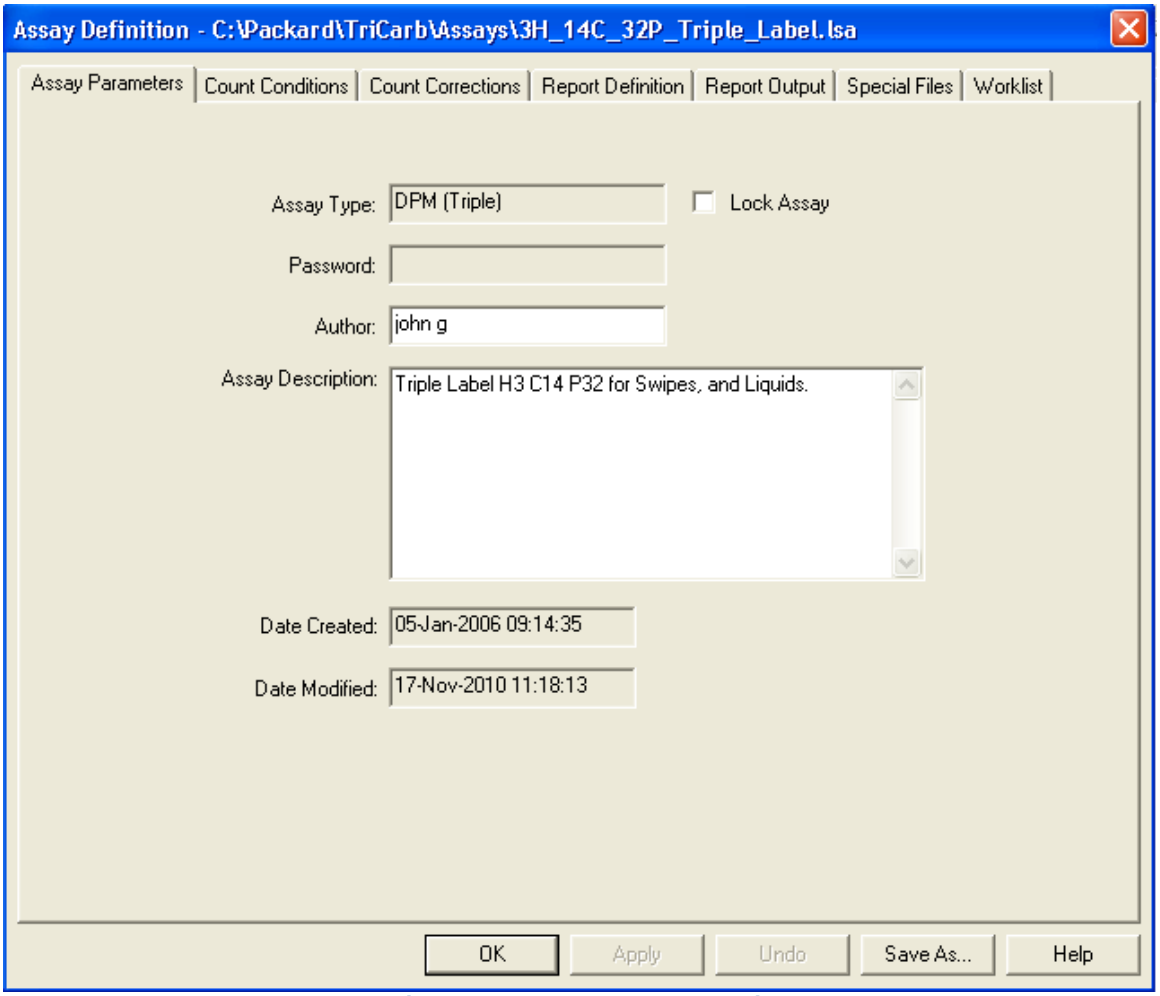

Figure 9 – Assay Parameters Tab

B. The Count Conditions tab will allow you to define specific counting parameters for the assay.

Triple-Label EXAMPLE:

NAME:	3H-14C-32P-RMLUG
Quench Indicator:	tSIE/AEC
Quench Set	TL-3H-UG-X TL-14C-UG-X TL-32P-UG-X (X is counter name)
External STD Terminator:	0.5 2s%
Pre-Count Delay (min):	0.00
Count Time (min):	30.00
Assay Count Cycles:	1
Repeat Sample Count:	1

#Vials/Sample:	1
Calculate % Reference	OFF
Background Subtract	OFF
Low CPM Threshold	OFF
2 Sigma % Terminator	OFF

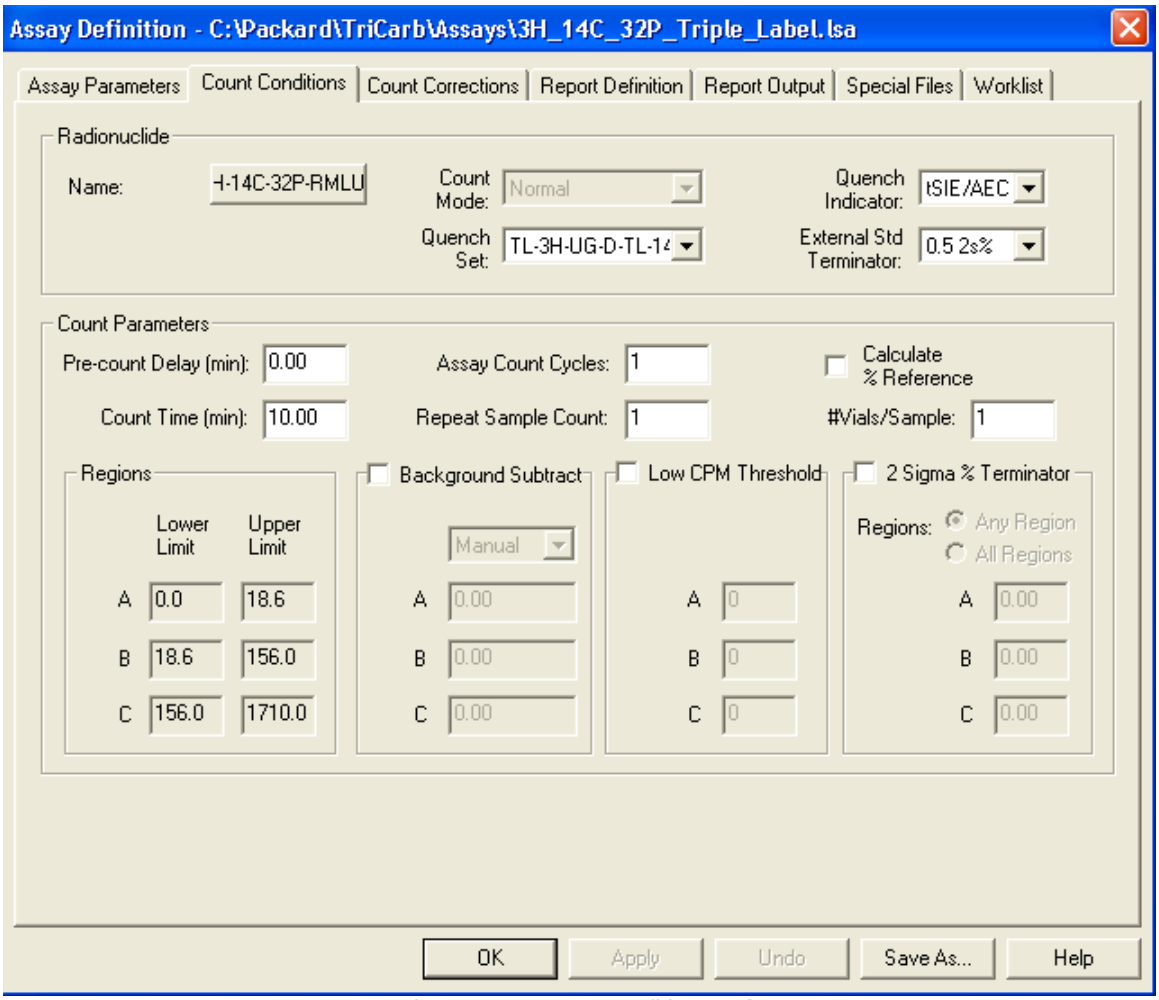

Figure 10 – Count Conditions **Tab**

6.3.4. Count Corrections Tab

A. The Count Corrections tab will allow you to activate certain instrument devices for the assay.

Triple-Label EXAMPLE:

Static Controller:	ON
Luminesence Correction	ON
Coincidence Time:	(nsec)18

Delay Before Burst: (nsec)75
Half Life Correction: OFF

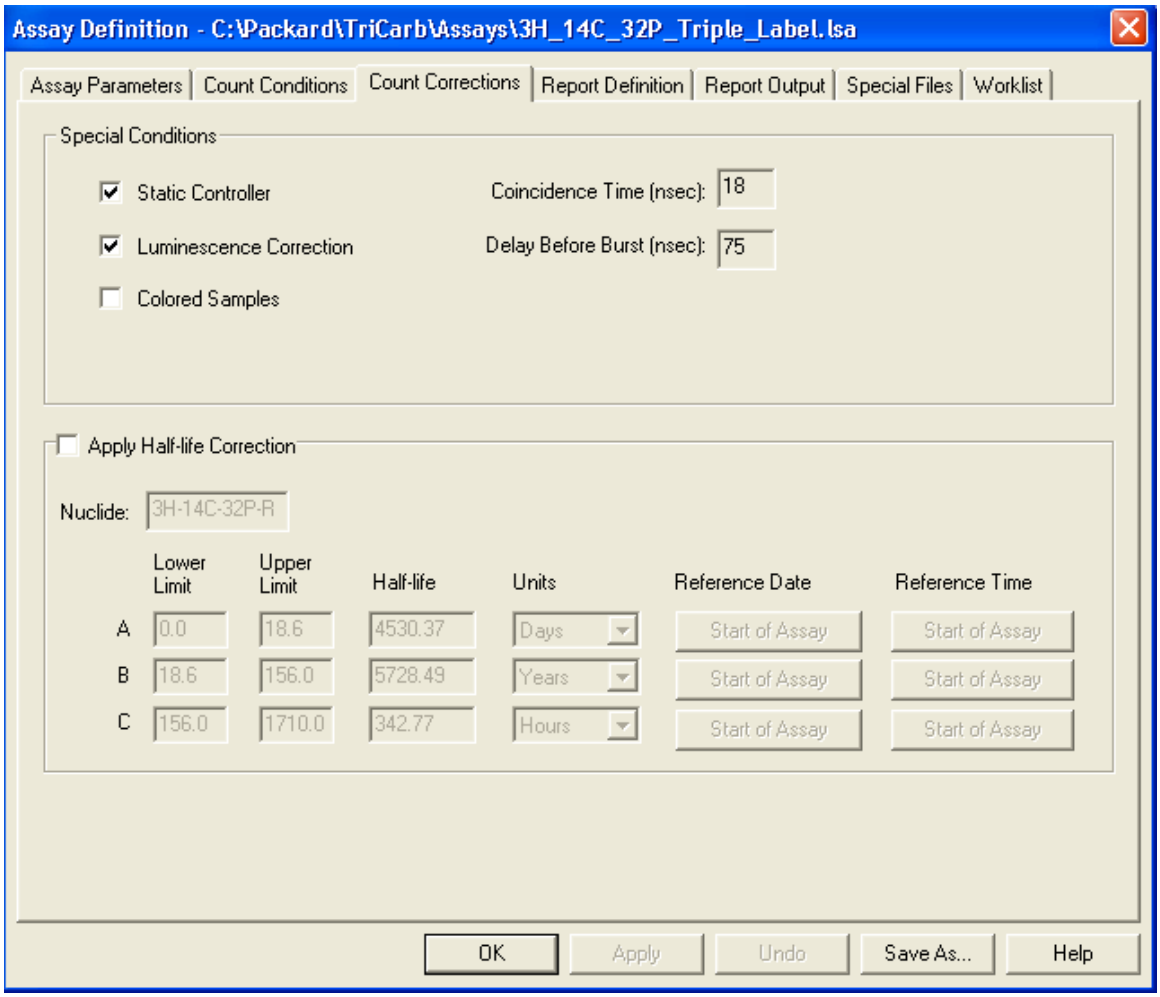

Figure 11 – Count Corrections Tab

6.3.5. Report Definition Tab

A. The Report Definition tab will allow you to specify the data items that are reported and the format for reporting.

Triple-Label EXAMPLE:
Report Name 3H_14C_32P_Triple_Label
Report Field Order: P#
PID
S#
SMPL_ID
Time
3H-CPMA

A:2S%
14C-CPMB
B:2S%
32P-CPMC
C:2S%
3H-DPMA
14C-DPMB
32P-DPMC
SIS
tSIE
LUM
DATE
TOD
FLAG

Report Block Data:
Assay Definition: ON

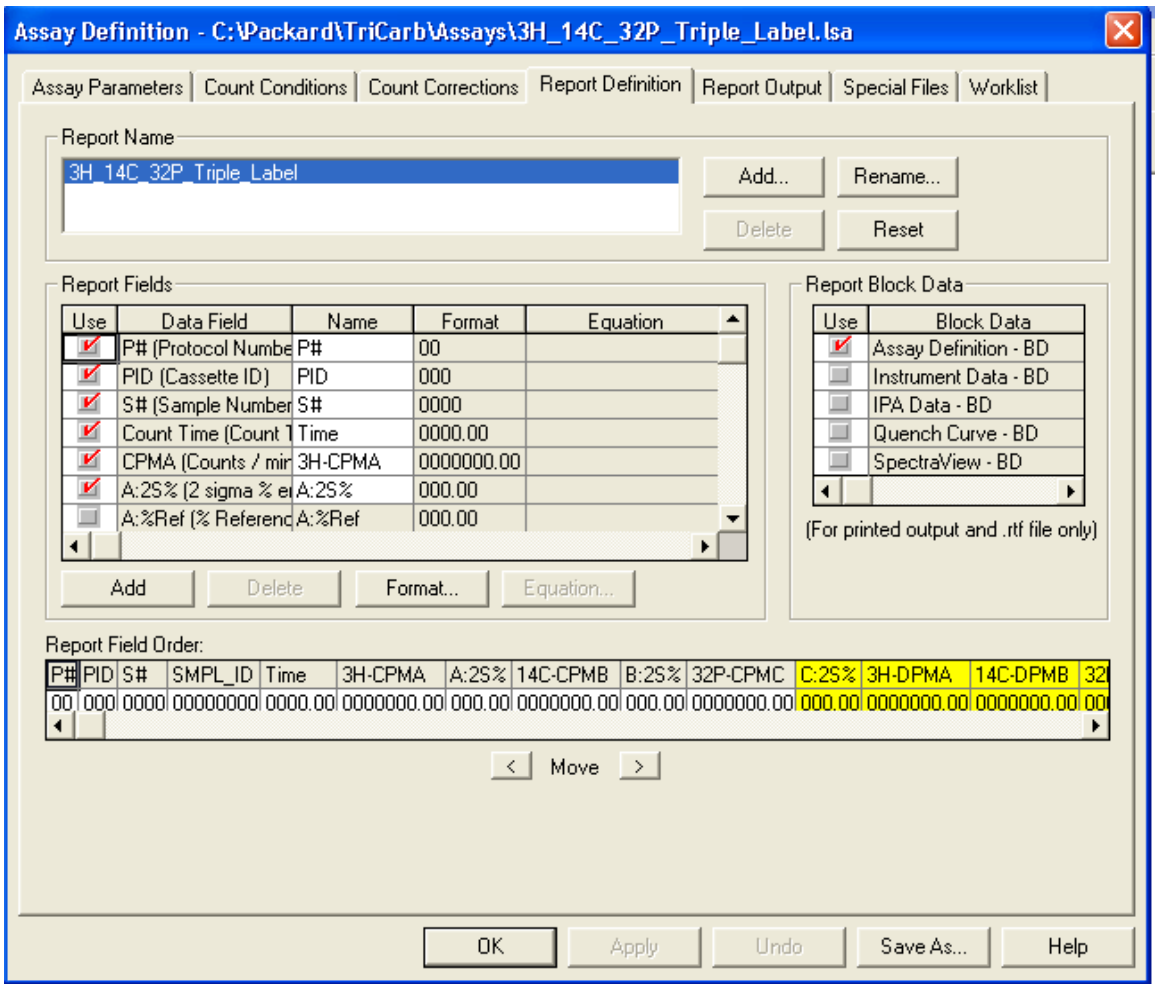

Figure 12 – Report Definition Tab

6.3.6. Report Output Tab

A. The Report Output will allow you to specify the output reports.

Triple-Label EXAMPLE:

Report Name: 3H_14C_32P_Triple_Label
Output to Printer: OFF
Data File: ON
File Type: Delimited Text
File Name:3H_14C_32P_Triple_Label
Output Per: Sample
Include Column Header ON
Rich Text File: ON
File Name: 3H_14C_32P_Triple_Label
Run Application: OFF

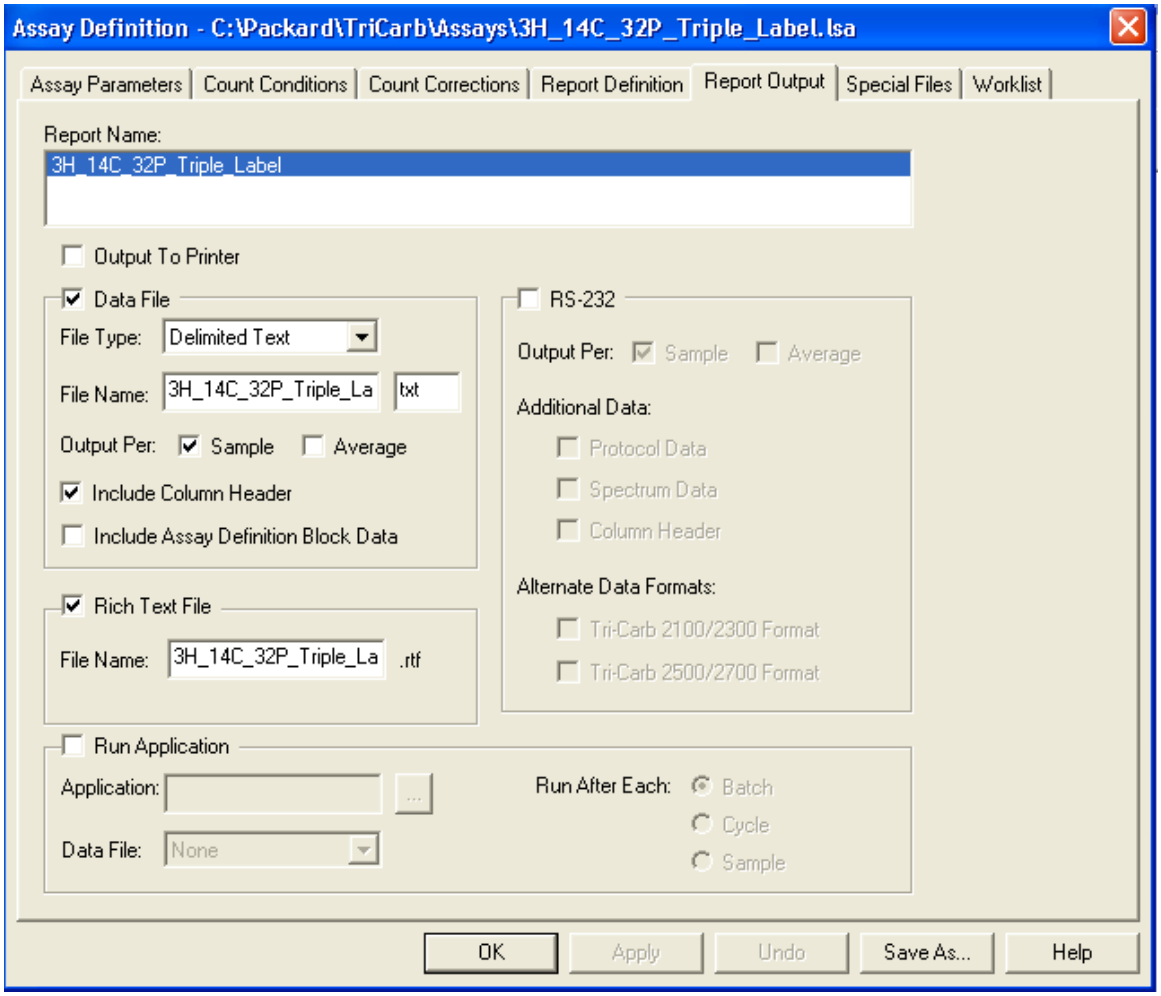

Figure 13 – Report Output Tab

6.3.7. Print copy of Assay 3H_14C_32P_Triple_Label

A. Select “FILE” then Print Assay “3H_14C_32P_Triple_Label”. Put the copy in the manila calibration folder.

6.3.8. Load Counter for Verification Run with the assay tab out.

SNC Rack
Assay (15) Rack (3H Master Quench Standards 1 to 10)
Assay Rack (C-14 Master Quench Standards 1 to 10)
Assay Rack (P-32 Master Quench Standards 1 to 10)
Assay 59 Halt Rack

6.3.9. Starting the Verification Run

A. Click the GO button.

6.3.10. Processing of the Verification Run

A. Transfer the run using the RML & WBC Swiss Army Knife Transfer Program. Copy the files (XJJJYYSS.dat, XJJJYYSS.txt) from X:\DATA\YYYY\X_LSC to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY \RAW_DATA_RUN_FILES.

B. From the RAW_DATA_RUN_FILES folder OPEN XJJJYYSS.txt file with Excel. Save As DATA_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls. Copy the file to WORKING_DATA_FILES folder. Copy the template files TEMPLATE_CTR_X_ U15_H3_C14_P32_RECALIB_VFY_RUN.xls and TEMPLATE_CTR_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls from X:\RML_CALIBRATIONS_IN_PROGRESS\TEMPLATES_H3_C14_P32 folder to the WORKING_DATA_FILES folder.

C. Open three files DATA_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls and TEMPLATE_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls and TEMPLATE_CTR_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls.

D. Edit the DATA_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls file. Delete SMPLID row and from the Time row to the LUM row format cells (Number, 2 decimal places).

E. Edit the TEMPLATE_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls and TEMPLATE_CTR_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls files. Edit the Header Information with the proper counter name and standards. Also the Expected Activity for the Quench Standard today.

F. Copy the data from the DATA_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls to TEMPLATE_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls. Save As CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN (Change X to counter name).

G. Copy the data from the DATA_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls to TEMPLATE_CTR_X_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls. Save As CTR_X_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls (Change X to counter name).

H. Delete the DATA_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls, TEMPLATE_CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls and TEMPLATE_CTR_X_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls files from the WORKING_DATA_FILES folder.

I. Print a copy of the CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls and CTR_X_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls for the manila calibration folder.

6.3.11. Evaluation and Acceptance of Activity Verification Values

A. Check the CTR_X_U15_H3_C14_P32_RECALIB_VFY_RUN.xls report. Ensure that the Deviations meet the following criteria. Initials are acceptable for approvals

Acceptable Deviations:
+/- 2%
+/- 5% with QC/QA Coordinator Approval
+/- 10% with RML Group Leader Approval
Greater than +/- 10% recalibration needed

B. Check the CTR_X_U15_H3_C14_P32_Quench_Sets_Crosstalk.xls report. Ensure that the DPM Crosstalk Deviations meet the following criteria. Initials are acceptable for approvals.

Acceptable Deviations:
+/- 2% P32 to H3
+/- 5% with RML Group Leader Approval
Greater than +/- 5% recalibration needed

+/- 5% P32 to C14
+/- 10% with RML Group Leader Approval
Greater than +/- 10% recalibration needed

Acceptable Deviations:
+/- 5% C14 to H3
+/- 10% with RML Group Leader Approval
Greater than +/- 10% recalibration needed
Or
+/- 50% with tSIE <100, RML Group Leader Approval
Greater than +/- 50% with tSIE <100 recalibration needed

6.4. **Determining Calibration Final QC and Background.**

6.4.1. Preparation of Samples for Final QC Run

A. Prepare 10 Poly Vial background samples: Aliquot 1 mL water and 10 mL Ultima Gold. Label the vials cap 1 to 10.

6.4.2. Edit Assay 15 “3H_14C_32P_Triple_Label”

A. Click on the assay name for Assay 15 then go to the Count Conditions Tab. Edit the Count Time for 10 minutes and Repeat Sample Count for 5 repeats, Click OK.

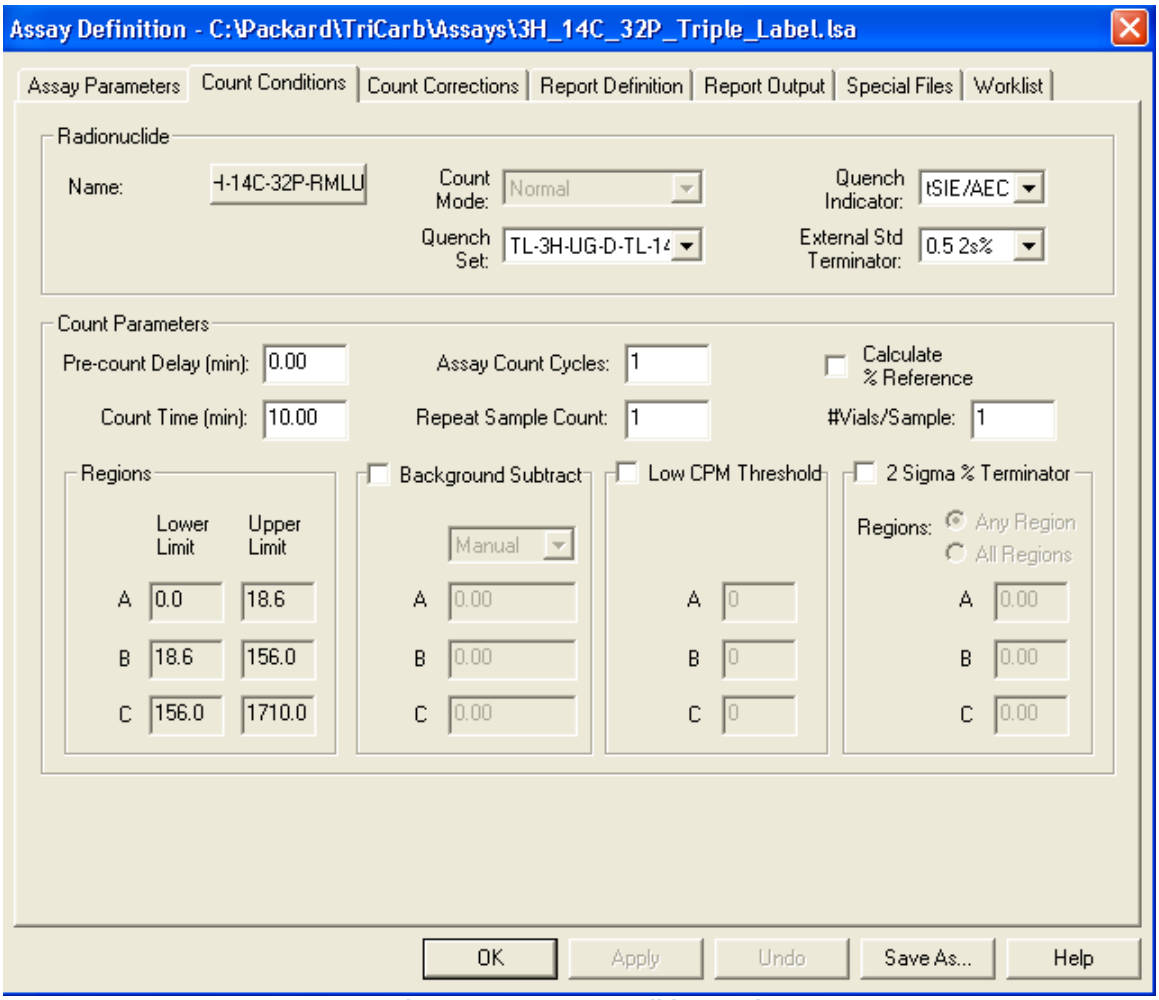

Figure 14 - Count Conditions Tab

B. Click FILE than Print Assay: 3H_14C_32P_Triple_Label

C. Put the copies in the manila calibration folder.

6.4.3. Loading of Samples and Racks for final QC Run

A. Samples to be loaded into racks.

(10)	Poly Vial Backgrounds
QC-31	Glass Background
QC-32	Blank – Glass Background
QC-33	QC Standard H3
QC-34	QC Standard C14
QC-35	QC Standard Sr/Y90
QC-36	QC Standard H3, C14, Sr/Y90
QC-37	Glass Background

B. Load samples into rack with assay flag.

Assay 15	QC-31,QC-32,QC-33,QC-34,QC-35,QC-36,QC-37
Assay Rack	(10) Poly Vial Backgrounds

C. Load sample racks into counter with the assay flag tab out.

SNC Rack	
Assay 15 Rack	QC-31 to QC-37
Assay Rack	(10) Poly Vial Backgrounds
Assay 59 Rack	Halt Rack

6.4.4. Starting the final QC Run

A. Click the GO button.

6.4.5. Processing the Final QC and Background Run

A. Transfer the run using the RML & WBC Swiss Army Knife Transfer Program. Copy the files (XJJJYYSS.dat, XJJJYYSS.txt) from X:\DATA\YYYY\X_LSC to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY \RAW_DATA_RUN_FILES.

B. From the RAW_DATA_RUN_FILES folder OPEN XJJJJYYSS.txt file with Excel. Save As DATA_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls. Copy the file to WORKING_DATA_FILES folder. Copy the template file TEMPLATE_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls from X:\RML_CALIBRATIONS_IN_PROGRESS\TEMPLATES_H3_C14_P32 folder to the WORKING_DATA_FILES folder.

C. Open both files DATA_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls and TEMPLATE_CTR_X_ U15_H3_C14_P32_ RECALIB_FINAL_QC_RUN.xls.

D. Edit the DATA_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls file. The sample numbers #1 to #10 in the SMPLID row need to be changed to QC-13(1). From the Time row to the LUM row format cells (Number,2 decimal places) "SAVE".

E. Edit the TEMPLATE_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls Header Information with the proper counter name and QC Standards Set information. "SAVE"

F. Copy the data from DATA_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls to TEMPLATE_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls for each sample's 10 counts. The template will calculate the final QC Operating Background Parameters. Save As CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls (Change X to counter name).

G. Delete the DATA_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls and TEMPLATE_CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls files from the WORKING_DATA_FILES folder.

H. Print a copy of the CTR_X_ U15_H3_C14_P32_RECALIB_FINAL_QC_RUN.xls for the manila calibration folder.

6.5. **Updating Calibration and QC Parameters.**

6.5.1. Updating RML Counter Calibrations

A. "ACCESS" the Counter Calibrations application (RML & WBC Swiss Army Knife – Managerial Programs – RML Calibration Editor). This will bring up the RML Counter Selection list.

B. "SELECT" the LSC Counter from the RML Counter Selection List. “Click” the Edit Selected button to access the Counter Calibration Editor. This will open the Counter Calibration Editor used to edit the three calibration files X_QCDATA.DOC, COUNTERX.doc, and COUNTERX.EFF.

C. “Click” on the Title and the Header Information page will appear. “Select” the Counter Calibration Date and enter the new date in the New Value box at the bottom of the page. “Click” Update Value.

D. The DOE Serial Number, LLNL Serial Number (Vendor) and the next text line Counter Use Description fields should already be populated with the appropriate information. If update is needed use the New Value box and Update Value at the bottom of the page.

E. “Select” the second text line Calibration Comments field and enter New Value as: “Triple-Label Glass Vial Quench Calibration using Perkin Elmer Ultima Gold Quench Sets #XX (XXX,XXX DPM/VIAL MM,DD,YYYY). Where “XX” is the serial number of the specific quench set used taken from the Certificate of Radioactivity in the calibration folder, XXX,XXX is the activity stated on the certificate and MM,DD,YYYY is the calibration reference date from the certificate. Additional pertinent comments can be added if desired.

F. “Click” on the Triple Sources and the Triple Sources QC Standards screen will display. This screen contains the rows relating to the QC-33, QC-34, QC-35, and QC-36 standard set.

G. “Review” the “calibration date and Original Activity” fields for QC-33 to QC-36. If you have used the same Triple-Label Resident QC Glass Vial Set, the data will not be changed. If a new Triple-Label Resident QC Glass Vial Set has been used, get the printout of CTR_X_U15_H3-C14-P32_CALIB_FINAL_QC.xls from the manila folder. The data needed in the following steps is on pages 1 & 2 of this report.

H. QC-33 – From the Triple-Label Resident QC Solutions get the Tritium calibration date and original activity (DPM) value. From the Final QC Parameters Verification get the +/- QC Control Limits (%) for QC-33 Tritium.

I. “Select” QC-33 Half Life, enter New Value 12.33 (years), Select QC-33 Cal Date, enter New Value mm-dd-yyyy, Select Original Activity (Tritium), enter New Value (DPM), Select QC-33 Plus, enter New Value (+) Control Limit, Select QC-33 Minus, enter New Value (-) Control Limit.

J. QC-34 – From the Triple-Label Resident QC Solutions get the Carbon-14 calibration date and original activity (DPM) value. From the Final QC Parameters Verification get the +/- QC Control Limits (%) for QC-34 Carbon-14.

K. "Select" QC-34 Half Life, enter New Value 5730.00 (years), Select QC-34 Cal Date, enter New Value mm-dd-yyyy, Select Original Activity (C-14), enter New Value (DPM), Select QC-34 Plus, enter New Value (+) Control Limit, Select QC-34 Minus, enter New Value (-) Control Limit.

L. QC-35 – From the Triple-Label Resident QC Solutions get the SR/Y-90 calibration date and original activity (DPM) value. From the Final QC Parameters Verification get the +/- QC Control Limits (%) for QC-35 SR/Y-90.

M. "Select" QC-35 Half Life, enter New Value 28.50 (years), Select QC-35 Cal Date, enter New Value mm-dd-yyyy, Select Original Activity (SR-Y90), enter New Value (DPM), Select QC-35 Plus, enter New Value (+) Control Limit, Select QC-35 Minus, enter New Value (-) Control Limit.

N. QC-36 – From QC-33 get the Original Activity (Tritium). From the Final QC Parameters Verification get the +/- QC Control Limits (%) for QC-36 Tritium.

O. "Select" QC-36 H3 Original Activity, enter New Value (DPM), Select QC-36 H3 Plus, enter New Value (+) Control Limit, Select QC-36 H3 Minus, enter New Value (-) Control Limit.

P. QC-36 – From QC-34 get the Original Activity (C-14). From the Final QC Parameters Verification get the +/- QC Control Limits (%) for QC-36 Carbon-14.

Q. "Select" QC-36 C14 Original Activity, enter New Value (DPM), Select QC-36 C14 Plus, enter New Value (+) Control Limit, Select QC-36 C14 Minus, enter New Value (-) Control Limit.

R. QC-36 – From QC-35 get the Original Activity (SR-Y90). From the Final QC Parameters Verification get the +/- QC Control Limits (%) for QC-36 SR-Y90.

S. "Select" QC-36 P32 Original Activity (QC SR-Y90), enter New Value (DPM), Select QC-36 P32 Plus (SR-Y90), enter New Value (+) Control Limit, Select QC-36 P32 Minus (SR-Y-90), enter New Value (-) Control Limit.

T. "Click" on the Triple Backgrounds and the Triple Backgrounds QC Backgrounds screen will display. This screen contains the rows relating to QC-38, QC-31, QC-32, and QC-37 background set.

U. QC-38 – From the CTR_X_U15_H3-C14-P32_CALIB_FINAL_QC.xls, Io FINAL QC PARAMETERS get the (Tritium, Carbon-14, P-32) Poly Vial BKG values (DPM). The +/- QC Control Limits (%) for QC-38 (Tritium, C-14, and P-32) is +/- 100 (%). The +/- QC Control Limits (%) for QC-38 Backgrounds (Tritium, C-14, and P-32) can be adjusted with RML Group Leader approval.

V. "Select" QC-38 H3 Background Rate (Tritium DPM), enter New Value (DPM). Select QC-38 C14 Background Rate (C-14 DPM), enter New Value (DPM). Select QC-38 P32 Background Rate (P-32 DPM), enter New Value (DPM).

W. “Select” QC-38 H3 Plus, enter New Value (100) Control Limit. Select QC-38 H3 Minus, enter New Value (100) Control Limit. Select QC-38 C14 Plus, enter New Value (100) Control Limit. Select QC-38 C14 Minus, enter New Value (100) Control Limit. Select QC-38 P32 Plus, enter New Value (100) Control Limit. Select QC-38 P32 Minus, enter New Value (100) Control Limit.

X. QC-31, QC-32, QC-37 - From the CTR_X_U15_H3-C14-P32_CALIB_FINAL_QC.xls, Io FINAL QC PARAMETERS get the (Tritium, Carbon-14, P-32) Glass QC Set BKG values (DPM). The +/- QC Control Limits (%) for QC-31, QC-32, QC-37 (Tritium, C-14, and P-32) is +/- 100 (%). The +/- QC Control Limits (%) for QC-31, QC-32, and QC-37 Backgrounds (Tritium, C-14, and P-32) can be adjusted with RML Group Leader approval.

Y. “Select” QC-31-32-37 H3 Background Rate (Tritium DPM), enter New Value (DPM). Select QC-31-32-37 C14 Background Rate (C-14 DPM), enter New Value (DPM). Select QC-31-32-37 P32 Background Rate (P-32 DPM), enter New Value (DPM).

Z. “Select” QC-31-32-37 H3 Plus, enter New Value (100) Control Limit. Select QC-31-32-37 H3 Minus, enter New Value (100) Control Limit. Select QC-31-32-37 C14 Plus, enter New Value (100) Control Limit. Select QC-31-32-37 C14 Minus, enter New Value (100) Control Limit. Select QC-31-32-37 P32 Plus, enter New Value (100) Control Limit. Select QC-31-32-37 P32 Minus, enter New Value (100) Control Limit.

AA. “Select” the Summarize All Changes button to list all changes made to the calibration files.

BB. “Select” Print Summary Report button and put a copy in the manila calibration folder.

CC. “Select” Print Entire Record button at the top of the page and put a copy in the manila calibration folder.

DD. “Select” Update Files and Exit button. This will save the editing just performed and update the three calibration files: (1) “COUNTER.EFF file which contains all of the calibration operating parameters for all of the calibrations performed on CTR X, (2) “X_QCDATA.DOC” file containing just the latest calibration operating parameters for CTR X, and (3) “counterX.doc file which contains a summary of all of the calibrations performed on CTR X.

6.5.2. Final QC Operating Parameters

A. "ACCESS" the RML Server X-Drive, X:\qcdata. "LOCATE" the desired analyzer file "X_QCDATA.DOC" where X is the Counter (D, E, J, K, R, S, T, W or V).

B. "COPY" the file X_QCDATA.DOC from X:\qcdata to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY\FINAL_QC_FILES.

C. From the "FINAL_QC_FILES" folder, "OPEN" the file in Microsoft Word and "Save As" CTR_X_QCAL_FINAL_QC_OPERATING_DATA.doc.

D. "SET" page formatting to: "Courier New 10 Point", "Portrait mode", "Top Margin 0.7 inch", "Bottom Margin 0.5 inch", "Left Margin 0.5 inch", and "Right Margin 0.5 inch" "Save".

E. "LABEL" the top of the first page with the header "CTR X USER 15 TRIPLE-LABEL GLASS QUENCH RECALIBRATION FINAL QC PARAMETERS". "SAVE" and "PRINT" one copy for the manila calibration folder. "DELETE" the original file from the "FINAL_QC_FILES" folder.

6.5.3. Calibration History

A. "ACCESS" the RML Server X-Drive, X:\qcdata\LSC_History. "LOCATE" the desired analyzer file "counterX.doc" where X is the Counter (D, E, J, K, R, S, T, W or V).

B. "COPY" the file counterX.doc from X:\qcdata\LSC_History to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY \CALIB_UPDATE_HISTORY.

C. From the "CALIB_UPDATE_HISTORY" folder, "OPEN" the file in Microsoft Word and "Save As" CTR_X_CALIB_HISTORY_SUMMARY_MM-DD-YYYY.doc. Where MM DD, YYYY represents the day of the calibration parameters update.

D. "SET" page formatting to: "Courier New 10 Point", "Portrait mode", "Top Margin 0.7 inch", "Bottom Margin 0.5 inch", "Left Margin 0.5 inch", and "Right Margin 0.5 inch" "Save".

E. "LABEL" the top of the first page with the header "CTR X CALIBRATION HISTORY SUMMARY AS OF MONTH DAY, YEAR". Where MONTH DAY, YEAR represents the day of the calibration parameters update. "SAVE" and "PRINT" one copy for the manila calibration folder. "DELETE" the original file from the "CALIB_UPDATE_HISTORY" folder.

6.5.4. Detailed Calibration History

A. "ACCESS" the RML Server X-Drive, X:\qcdata\counters. "LOCATE" the desired analyzer file "COUNTERX.EFF" where X is the Counter (D, E, J, K, R, S, T, W or V).

B. "COPY" the file COUNTERX.EFF from X:\qcdata\counters to X:\RML_CALIBRATIONS_IN_PROGRESS\CTR_X_CALIB\MMM_DD_YYYY \CALIB_UPDATE_HISTORY.

WARNING

Do not move or alter this file or any files in the “counters” folder on the X-Drive as they are critical for the Data Reduction Process used in Client Sample Analyses.

C. From the "CALIB_UPDATE_HISTORY" folder, "OPEN" the file in Microsoft Word and "Save As" CTR_X_FULL_DETAILS_CALIB_HISTORY_MM-DD-YYYY.doc. Where MM DD, YYYY represents the day of the calibration parameters update.

D. "SET" page formatting to: "Courier New 10 Point", "Portrait mode", "Top Margin 0.7 inch", "Bottom Margin 0.5 inch", "Left Margin 0.5 inch", and "Right Margin 0.5 inch" "Save".

E. "LABEL" the top of the first page with the header "CTR X CALIBRATION HISTORY FULL DETAILS AS OF MONTH DAY, YEAR". Where MONTH DAY, YEAR represents the day of the calibration parameters update. "SAVE" "DELETE" the original file from the "CALIB_UPDATE_HISTORY" folder.

6.6. QC Review of Calibration.

6.6.1. Resetting Analyzer for Normal Operation

A. Edit Assay 15 “3H_C14_P32_Triple_Label”. Click on the assay name on the QuantaSmart main window for Assay 15 than go to the Count Conditions Tab. Edit the Count Time for 10 minutes. Set Repeat Sample Count for 1 repeat, Click OK.

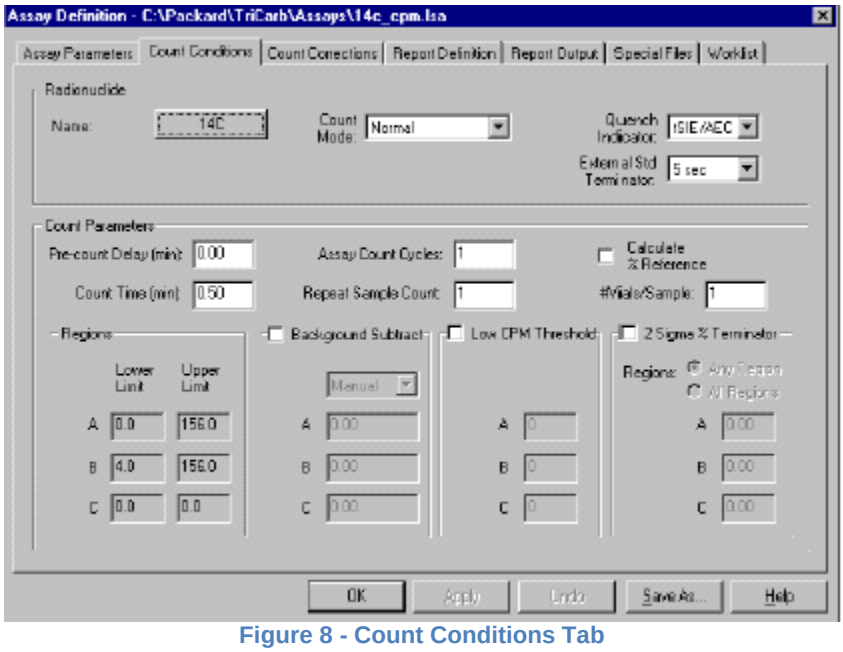

Figure 8 - Count Conditions Tab

B. Click FILE than Print Assay ”3H_C14_P32_Triple_Label”. Put the copies in the manila calibration folder.

C. Set the printer to landscape. Go to the LIBRARIES Tab on the QuantaSmart main window Click SAMPLE NUCLIDES than Print Box. Go to the LIBRARIES Tab on the QuantaSmart main window Click QUENCH STANDARDS than Print Box. Put copies in the manila calibration folder.

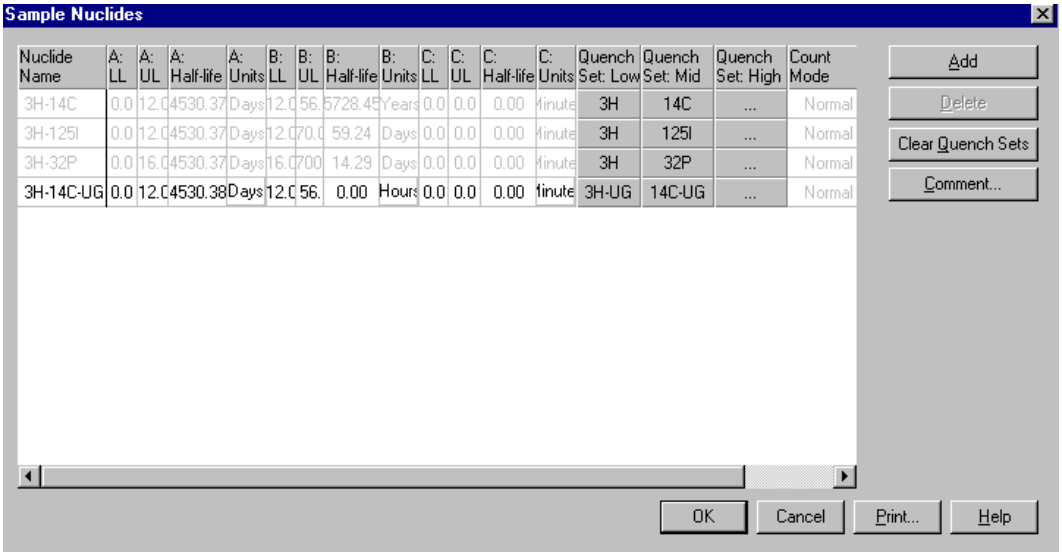

Figure 9 - Sample Nuclides Library Window

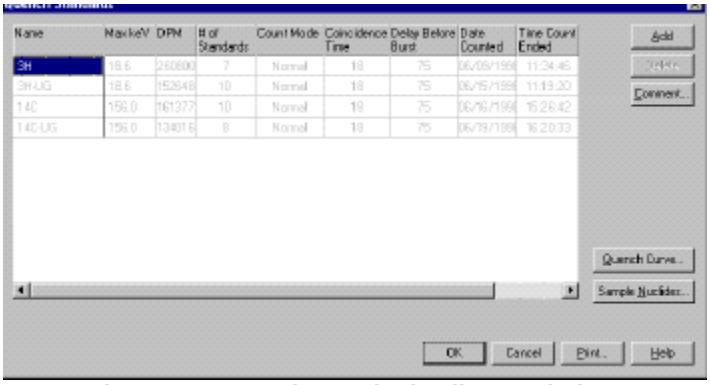

Figure 10 - Quench Standards Library Window

6.6.2. Final Review and Update of Calibration Record

A. Locate the TRIPLE-LABEL/GAB-H3 Calibration Record Binder for the specific counter in the RML QC Coordinator's office. Remove the old calibration and place it in the Historical Calibrations Records file for the specific LSC located in B253 R1903.

B. The new calibration documents are in the manila calibration folder and each document shall be reviewed as instructed in the following steps. The

documents will be dated and initialed with the date the calibration was entered in the RML Calibration Editor. The documents shall than be placed in the RMLTRIPLE-LABEL/GAB-H3 Calibration Record Binder for that specific LSC.

C. The multiple page Calibration History Summary with the header CTR X CALIBRATION HISTORY SUMMARY AS OF MONTH DAY, YEAR. Check the last calibration which should be the one just performed. The parameters should match the parameters in the RML Calibration Editor.

D. Verify that the certificates match the Tritium and C14 Master Calibration Standards Sets and the P-32 Standards Solution.

E. Verify the (H3,C14,P32)_Quench Assay Parameters documents are correct for the Triple-Label Quench Calibration performed.

F. Check the (H3,C14,P32)_Quench Calibration Output Curves are correct for the specific LSC and the analyzer produced quench curve data.

G. Verify the CTR_X_ (U15)_ _H3_C14_P32_CALIB_PLOT.xls QNCH # and EFF (%) match the values from the analyzer produced quench curve data in the previous step.

H. Check the 3H_14C_32P_Triple_Label Assay Parameters are correct for the verification run.

I. Verify the CTR_X_ H3-C14-P32 _RECALIB_VFY_RUN.xls verification run results % deviation is acceptable and has the proper approvals.

J. Check the “3H_14C_32P_Triple_Label” Assay Parameters are correct for the final QC run.

K. Verify the CTR_X_U15_H3-C14-P32_RECALIB_FINAL_QC.xls FINAL QC PARAMETERS match the parameters in the RML Counter Calibration Summary Report showing all changes made to the calibration files.

L. Verify the parameters changed in the Summary Report match the parameters in the printout Entire Record Report from the COUNTERX.eff calibration file.

M. Check the CTR X USER15 Triple-Label CALIBRATION FINAL QC PARAMETERS match the parameters and dates in the Entire Record Report printout from the previous step.

N. Check the Sample Nuclide 3H-14C-32P-RMLUG and Quench Standards TL-3H-UG-X, TL-3H-UG-X, and TL-3H-UG-X printout parameters. Check the “3H_14C_32P_Triple_Label” Assay Parameters are correct for production sample counting.

6.6.3. Releasing of Analyzer for Client Sample Processing

A. Archive the electronic folder by coping the date folder MMM_DD_YYYY FROM X:\RML\CALIBRATIONS_IN_PROGRESS_CTR_X_CALIB to X:\RML_CALIBRATIONS\LSC_OPERATIONS\LSC_Triple-

Label_CALIBRATIONS_\CTR_X_Triple-Label _CALIBRATIONS. Delete CTR_X_CALIB from X:\RML\CALIBRATIONS_IN_PROGRESS.

B. Update the Log Book for the specific counter calibrated with the date and the calibration performed.

C. Update the Excel workbook for next calibration schedule X:\RMLCALIBRATIONS\RML_SYSTEM_CALIBRATION_SCHEDULE.xls.

6.7. **Waste Disposal.**

6.7.1. Collect scintillation vials in a waste container and dispose of them according to the requirements of the *ES&H Manual* and the Waste Acceptance Criteria (WAC). Additional information on waste disposal is included in the EP006 series of training classes.

6.8. **Calculations.**

6.8.1. Efficiency calculations are performed by the instrument operating software. The measured CPM of each standard is divided by the decay corrected DPM to determine the counting efficiency. After each the efficiency for each standard is determined, a best fit curve is determined by the instrument software. A summary of the efficiencies for each standard is printed using the instrument operating software.

6.8.2. The Excel workbooks used in this procedure are used to summarize and display the calibration curve. No additional calculations of efficiency are performed by these workbooks.

6.9. **Data Review and Reporting.**

6.9.1. Prior to approval of a new calibration curve the contents of the temporary manila folder are reviewed by the RML QC Coordinator or the RML Group Leader. Calibration curves are not placed into use until approval by the reviewer.

7. DEFINITIONS

7.1. Procedure-specific terms and definitions.

Word	Definition
RML Master Program Shell (Swiss Army Knife)	A consolidated interface used to access applications used to process analytical results
QuantaSmart	The software system used to operate the Packard LSCs
	Common definitions may be found in the QAP.

7.2. Acronyms.

Acronym	Meaning
IPA	Instrument Performance Assessment
SNC	Self-normalization and calibration
	Common acronyms may be found in the QAP.

8. TRAINING

8.1. Required Qualifications.

8.1.1. Training requirements for the RML Group members are described in the Quality Assurance Plan.

8.1.2. Safety related training requirements are specified in the RML IWS (10463).

8.2. Other required training.

A. None.

9. RELATED DOCUMENTS

9.1. Related implementing documents.

9.1.1. The following documents are used in association with this procedure.

Document number	Document Name
1. 6007600	Certificate of Radioactivity/Traceability.
2. ASI-DES-001	Radioanalytical Laboratories Quality Assurance Plan

10. REQUIREMENT SOURCE DOCUMENTS

10.1. LLNL source documents.

10.1.1. The following *ES&H Manual* Documents and Institutional Procedures contain the source requirements for this procedure.

Document number	Document Name
None.	

10.2. Other source documents.

10.2.1. The following documents contain additional source requirements for this procedure.

Document number	Document Name	Revision or Approval date
1. Publication No. 1694215	Perkin Elmer QuantaSmart for the Tricarb Liquid Scintillation analyzer, Reference Manual	Rev. C

11. RECORDS

11.1. Official records generated by this procedure.

11.1.1. The following records generated in association with this procedure are considered official radiological records and must be retained until DOE authorizes final disposition.

Record	Storage location
1. Triple-Label Calibration Record	Calibrations Records file for the specific LSC located in B253 R1903.

11.2. **Other records generated by this procedure.**

11.2.1. The following documents generated in association with this procedure are not considered official radiological records and must be retained as follows.

Record	Retention period
None.	

12. CHANGE HISTORY

12.1. **Changes in current revision.**

Type of change	Summary of change	Sections with major changes
Editorial	Procedure reformatted into new procedure format.	

12.2. **Document revision history.**

Approval Date	Revision	Revision Description
3/01/2017	0	Renamed to RML-406
4/4/2014	0	Reformatted into new template
12/22/2010	CALH3 201012	Biennial Review
8/19/2005	3H CAL082005	New Procedure

13. CONTACTS

See signatures on title page.

14. ATTACHMENTS AND APPENDICES

None.