Standard Operating Procedure for Liquid Scintillation Counting Using the Packard (PerkinElmer) TriCarb 2900TR Counter

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1.0 Identification of Test Method

1.1 The procedure described in this SOP has been developed by the LDEQ Environmental Radiation Laboratory in accordance with the requirements of the Louisiana Department of Environmental Quality. This SOP describes in detail the essential elements of the procedure for performing Tritium analysis using the Packard TriCarb 2900TR Liquid Scintillation Counter.

2.0 Applicable Matrix

2.1 This SOP is applicable to testing for tritium in non-potable water.

3.0 Detection and Quantitation Limits

3.1 The Limit of Detection (LOD) for Tritium is 484 pCi/L. The LOD was calculated at the $95^{\%}$ confidence interval based on the t-distribution of five background measurements (t_5=2.776; s^2=107; mean value=187).

3.2 The Limit of Quantitation (LOQ) is defined as 1000 pCi/Liter.

4.0 Scope and Application

4.1 This SOP will explain the process and requirements for this standard in measurements using the Packard TriCarb 2900TR Liquid Scintillation Counter

5.0 Summary of Method

5.1 This is a method for Tritium analysis using the Packard TriCarb 2900TR Liquid Scintillation Counter. Water samples are placed in vials along with an equal amount of liquid scintillation cocktail, and are loaded into the liquid scintillation counter. Beta particles emitted from the sample cause certain compounds in the scintillation cocktail to fluoresce. The resulting light pulses are counted by photomultiplier tubes in the instrument.

5.2 One aliquot of each water sample is taken and analyzed for tritium. Each aliquot is counted for three 30-minute counts, so that the total counting time is 90 minutes for each sample.

5.3 A quench curve is used to calculate the rate of decay from the count rate.

5.4 Half-life correction is not necessary for tritium samples collected less than six months prior to analysis.

5.5 Sample activity is calculated based upon the average of three counts for the sample aliquot, with background subtraction.

6.0 Definition of Terms

6.1 AEC – Automatic Efficiency Correction. This is a feature by which the instrument adjusts the counting region settings to compensate for the effect of quenching in a sample.

6.2 Assay – A comprehensive set of parameters that the Packard TriCarb 2900TR instrument uses for the purpose of sample analysis. Assays are defined using seven Assay Definition tabs, each of which allows the user to enter information specific to one aspect of the assay. See Appendix D, "Packard TriCarb 2900TR Assay Definition" for more information. Once an assay has been defined, it can be saved, used or edited.

6.2.1 **NOTE: All of the assays required for this SOP have already been defined and locked.**

6.3 Baselines – Baseline values for the background count and count efficiencies are calculated by the instrument as part of the SNC/IPA calibration procedure. The number of IPA runs (data points) required for the calculation is defined by the user as one of the IPA parameters. Mean and limit values for the baselines are displayed in the bottom section of the IPA Definition window.

6.3.1 Background Mean and Limit – During the calibration procedure, the instrument calculates the mean values of the background for Carbon-14 and Tritium. The limits are calculated as the baseline + 4 standard deviations. When a limit is reached, a message is displayed in the main window.

6.3.2 Efficiency Mean and Limit – During the calibration procedure, the instrument calculates mean values of the count efficiency for the Carbon-14 and Tritium. The limits are calculated as 3% below the baseline. When a limit is exceeded, a message is displayed in the main window.

6.4 Counts per minute (CPM) – The total number of counts detected by the scintillation counter in each counting region, divided by the count time in minutes.

6.5 Disintegrations per minute (DPM) – An expression of radionuclide activity, also referred to as Decays per Minute. 1 pCi = 2.22 DPM. DPM cannot be measured directly, but is calculated from the CPM and the Quench Efficiency.

6.6 Duplicate Measurement – A second measurement made on the same (or identical) sample of material to assist in the evaluation of measurement variance.

6.7 Efficiency – The ratio of measured counts to the number of decay events that occur during a measurement interval.

6.8 ERL – Environmental Radiation Laboratory (analyzes radiation samples).

6.9 External Standard – An external standard is a radioactive source that is placed adjacent to a sample to produce scintillation in the sample for the purpose of monitoring the sample's quench level. This takes place automatically during sample analysis. The Packard TriCarb 2900TR is equipped with a Ba-133 source for this purpose.

6.10 IPA (Instrument Performance Assessment) – A process by which the TriCarb 2900TR instrument measures background, counting efficiency, sensitivity (Figure-of-Merit) and the reproducibility of sample counting (Chi-Square Test).

6.11 Quench – A reduction in the scintillation intensity seen by the photomultiplier tube of a liquid scintillation counter. The result is fewer photons per keV of beta particle energy, and usually a reduction in counting efficiency.

6.12 Quench Curve – A mathematical graph that correlates the counting efficiency to a numerical value used to express the level of quench in a sample. A plot of %Efficiency vs. the level of quench is used to calculate the DPM from the CPM.

6.13 Quench Standards Set – A Quench Standards Set is composed of a series of vials, each containing the same amount of nuclide with varying amounts of quenching agent.

6.14 SNC – Self-Normalization and Calibration. This is the protocol for calibration of the TriCarb 2900TR Instrument.

6.15 SIS – Spectral Index of the Sample. This is a number that the instrument obtains from the spectral distribution of the sample. The number is used as an index of the level of quench in a sample.

6.16 Tritium Efficiency Mean and Limit – The instrument calculates the mean value for Tritium efficiency from the number of IPA runs (data points) specified. The limit is calculated as 3% below the baseline, or less than 58% efficiency. When the limit is exceeded, a message is displayed in the main window.

6.17 tSIE – Transformed Spectral Index of the External Standard. This is a number that the instrument obtains from the spectral distribution of the external standard. The number is used as an index of the level of quench in a sample.

6.18 UG LLT – Ultima Gold Low Level Tritium: High flash point LSC cocktail for counting low level tritium samples for all types of water including sea water.

6.19 See the PerkinElmer "QuantaSmart for the TriCarb Liquid Scintillation Reference Manual" or the DEQ LSD QA Manual, Appendix B for additional definitions.

7.0 Interferences and Pretreatments

7.1 Refer to Section 14.5 for sample preparation.

8.0 Safety

8.1 Always wear lab coat and safety glasses, goggles or lab face shield when working in the lab.

8.2 When opening the lid to the Packard TriCarb 2900TR, be certain the monitor is out of the way of the lid, to prevent damage to the lid and monitor.

8.3 The system uses standards in glass vials. Handle with care so that none of the glass vials fall and shatter.

8.4 Material safety data sheets (MSDS) of all chemicals used in the DEQ Environmental Radiation Laboratory are kept in a yellow binder in Room R-02. Additionally, MSDS are available at a web site maintained by Cornell University. The link is: http://hazard.com\msds\index.php. NOTE: Radioactive materials are not included in the notification requirements of the OSHA Hazard Communication Standard (29CFR1910.1200).

9.0 Equipment and Supplies

9.1 Packard TriCarb 2900TR Liquid Scintillation Counter

9.2 Packard IPA and Quench Standard's

9.3 Burrell Wrist Action Shaker

9.4 Liquid Scintillation counting vials

9.5 10 mL Cole Parmer Macro Pipettor with disposable pipettes

9.6 Disposable laboratory gloves

9.7 Kimwipe lab towels

9.8 Stainless steel tray covered with absorbent paper

10.0 Reagents and Standards

10.1 Information on reagents and standards shall be entered into the designated logbook. Such information shall include the manufacturer, lot number, date of receipt, quantity, and expiration date.

10.2 Reagents

10.2.1 PerkinElmer Ultima Gold LLT Liquid Scintillation counting cocktail (UGLLT).

10.2.2 Deionized water for method blanks.

10.3 Standards

10.3.1 PerkinElmer Tritium (^{3}H) Calibration Standard: Calibrated at 285000 DPM on May 11, 2007.

10.3.2 PerkinElmer Carbon-14 (^{14}C) Calibration Standard: Calibrated at 142300 DPM on May 11, 2007.

10.3.3 PerkinElmer Background Calibration Standard: Dated May 2007.

10.3.4 PerkinElmer Quench Standards Set: 10 standards (A-J), calibrated at 284000 DPM on Oct. 01, 2007.

10.3.5 All standards have been calibrated with traceability to National Institute of Standards and Technology (NIST) Reference Materials.

11.0 Sample Collection, Preservation, Shipment, Storage, and Sample Rejection Policy

11.1 Water samples are collected monthly from three drinking water stations and one surface water location near Waterford-3. Quarterly split surface water samples are collected from upstream and downstream of the Grand Gulf Nuclear Station and River Bend Station. Four liters of water are collected at each sampling point, in 4L collapsible plastic containers. Water samples are stored in the refrigerator until ready for sample preparation. The bulk of the sample is used for gamma analysis, but an aliquot is removed from each sample for tritium analysis, as follows:

11.1.1 A filtered 60-mL aliquot of water is taken from each of the quarterly samples from Grand Gulf and River Bend.

11.1.2 A filtered 20-mL aliquot of water is taken from each of the monthly water samples from Waterford-3, and the aliquots from each sampling point are combined over a three-month period to make quarterly composites. The quarterly composite for each site is logged into the LIMS with a new LIMS number. The collection date is defined as the collection date of the last sample for each quarterly composite. Each composite sample must be recorded in the “Tritium Samples” logbook (RAD-002), with the sample LIMS number, collection date and time, and volume for each individual sample in each quarterly composite, along with the LIMS number for the composite.

11.2 Samples received by the Environmental Quality Laboratory must be entered into LIMS. After samples have been properly logged in, they must be stored in the designated location. The procedure for sample login is described in Appendix A2 of the current official ERL SOP 1023, “Preparation of Samples for Gamma Spectroscopy and Gross Beta Analysis.”

11.3 Water samples from River Bend and Grand Gulf are assigned unique LIMS Sample IDs when they are logged in for gamma analysis, and the same sample IDs are used for tritium analysis. Composite samples for tritium analysis from Waterford-3 must be logged in again with new LIMS Sample IDs, because each composite is taken from three separate samples.

11.4 In case of samples with incomplete chain of custody, contact the person(s) submitting the sample to obtain the missing information. Document the contact and how the information was obtained in the comment section of the chain of custody form.

11.5 As per 40CFR136.3, Table II, the hold time for tritium samples is six months.

11.6 For policy on chain of custody usage, see Section 11.5 of QAM.

12.0 Quality Control

12.1 A quality control program is required for this method. The requirements of this program consist of demonstration of laboratory capability, and the continuing analysis of NIST-traceable standards of known activity as ongoing demonstration of performance. All Demonstrations of Capability (DOCs) must be documented using the appropriate Certification Statement Form. The analyst shall maintain performance records that define the quality of the data that are generated.

12.2 Demonstration of Capability

12.2.1 An initial Demonstration of Capability (DOC) requires the analyst to perform the following procedures, four times on four different days:

12.2.1.1 Running the SNC Protocol as described in Section 14.3.

12.2.1.2 Analysis of the ten Quench Standards using the DOC-Tritium Assay procedure, in which the standards are analyzed as samples, with decay correction to the original calibration date. See Section 16.2 for details on how the DOC procedure is performed.

12.2.2 Continuing Demonstrations of Capability shall be done annually. A continuing DOC may be performed by successful analysis of a Proficiency Test sample, or by repeating the requirements for the initial DOC.

12.3 Assessing Laboratory Performance

12.3.1 The IPA/SNC calibration procedure measures the background, counting efficiency, sensitivity (Figure-of-Merit), and reproducibility (Chi-Square Test) of sample counting.

12.3.2 A Method Blank is analyzed before and after each batch of Tritium samples. The method blank consists of 15 megohm deionized water which has been prepared in the same manner, at the same time and using the same reagents as the other samples in the batch.

12.3.3 Quality Control Samples – A mid-range quench standard is analyzed immediately before the first test sample in the batch. A second mid-range quench standard is analyzed immediately after the last test sample in the batch. The Relative Percent Difference (RPD%) and %Recovery of the standards are calculated and recorded on a controlled spreadsheet.

12.3.4 Replicate analysis – Each sample is counted three times, and the standard deviation of the sample counts is calculated and displayed on the instrument printout.

12.3.5 Acceptance criteria for Quality Control measures are listed in Section 18.0, "Data Assessment and Acceptance Criteria."

13.0 Calibration

13.1 Self-Calibration and Normalization/ Instrument Performance Assessment

13.1.1 The Packard TriCarb 2900TR must be calibrated on each day of operation prior to the analysis of samples. The calibration protocol for the Packard TriCarb 2900TR is called Self-Calibration and Normalization/ Instrument Performance Assessment (SNC/IPA). The SNC/IPA procedure is described in Section 14.3 of this SOP.

13.1.2 The IPA definition specifies the parameters used by the instrument in the SNC/IPA calibration process. The current IPA parameters have already been defined, and need not be changed unless a new set of calibration standards are put in use, or when maintenance is performed on the photomultiplier assembly or other electronic components. The procedure for defining the IPA parameters is described in Appendix C, "Packard TriCarb 2900TR IPA Parameter Definition."

13.1.3 IPA data shall be included in the instrument printout for each sample batch.

13.2 Quench Standards Assay

13.2.1 The Quench Standards Assay shall be run annually and/or whenever a new set of quench standards is acquired. Using data from the quench standards, a quench curve is generated to determine the counting efficiency of samples, and to calculate the DPM from the CPM. The instrument stores the spectrum of each standard in the quench standards set.

13.2.2 Quench curve data shall be included on the instrument printout for each sample batch.

13.2.3 The procedure for generating a quench curve is found in Appendix I, "Running a Quench Standards Assay."

14.0 Procedure

14.1 Samples must be batched in the LIMS prior to analyses.

14.2 **Preparation of Water Samples for Tritium Analysis**

14.2.1 Sample preparation shall be performed inside the fume hood on plastic backed covered lab bench top paper to prevent the possible spill of sample or scintillation cocktail in the lab. Disposable laboratory gloves are used during sample preparation work to avoid contamination.

NOTE: To avoid potential spread of contamination while working with samples, change gloves if any liquid appears on your gloves.

14.2.2 Using the Cole-Parmer 10 mL MacroPipettor, transfer 10 mL of a sample to a sample counting vial.

14.2.3 Next, dispense 10 mL of Ultima Gold Low Level Tritium Liquid Scintillation counting cocktail (UGLLT) into the sample vial. The Ultima Gold bottle-top dispenser has been set to deliver a 10-mL aliquot.

14.2.3 After the sample and scintillation cocktail have been put in the vial, place a cap on the sample vial and tighten carefully. Check for leaks by cleaning the outside of the vial with a Kim wipe towel.

14.2.4 Using a Fine Tip Sharpie pen, label the cap of the sample vial with the LIMS sample ID. **NOTE: Writing shall be done on the cap of the vial ONLY!** NO marking or writing on the sides or bottom of the vial. Any markings on the side or bottom can adversely affect the analysis.

14.2.5 Place the sample vial in the white plastic sample rack. This rack is used to carry the sample vials with a minimum of handling.

14.2.6 Repeat steps 14.2.1 – 14.2.5 until all water samples for Tritium analysis have been prepared.

14.2.7 Prepare **two** method blanks in the same manner as the samples, using 10 mL of 15 megohm deionized water in place of sample.

14.2.8 Carry the white sample rack with the sample vials over to the bench where the Burrell Wrist Action shaker is located.

14.2.9 Transfer the scintillation vials to the Burrell Wrist Action shaker. The Burrell Wrist Action shaker can accommodate up to 16 sample vials per load. Samples must be shaken for a minimum of 20 minutes to thoroughly mix the sample with the scintillation cocktail.

Burrell Wrist Action shaker

14.2.10 Operation of the Burrell Wrist-Action Shaker

14.2.10.1 Place liquid scintillation sample vials into the finger-grip clamp and secure with knurled nut. **Do not over-tighten the knurled nut.**

14.2.10.2 The lever arm on the right side of the shaker controls the amplitude of agitation.

14.2.10.3 The lever moves from zero (least agitation) to 10 (greatest agitation). Adjust the lever to 10 for greatest agitation.

14.2.10.4 On the face of the shaker is the timer knob. Set timer knob to 20 minutes to agitate the samples.

14.2.10.5 After the timer has stopped remove sample vials from shaker.

14.2.10.6 Check vials again for leakage and using Kim wipe towels thoroughly wipe the outside of each vial to remove any fingerprints from the sides and bottom before loading into liquid scintillation counter cassettes.

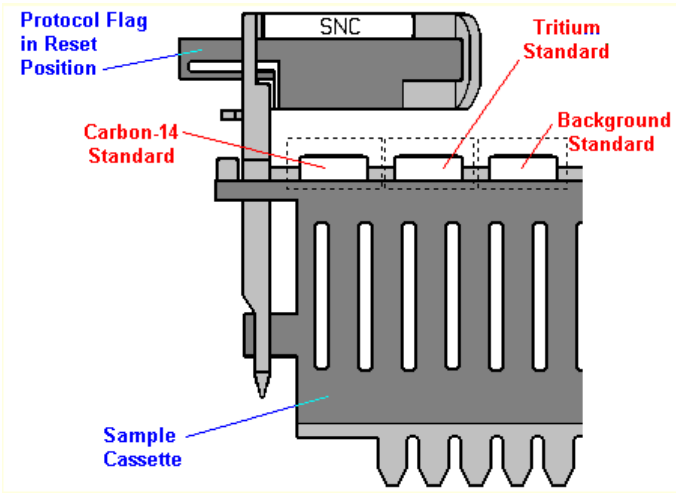
14.2.10.7 Use the white liquid scintillation vial carrier to transport sample vials from the Burrell Wrist Action

Shaker to the Packard TriCarb 2900TR for sample loading.

14.2.10.8 Once all samples are removed, return the lever arm to zero.

14.3 SNC/IPA Procedure

14.3.1 To run the SNC/IPA protocol, you must reset the SNC protocol flag to the "reset" position (the flag is all the way to the left when the flag is on the left end of the cassette). If the protocol flag is not "reset", the SNC/IPA protocol will only run if 23 or more hours have elapsed since the last time that the protocol was run.



Cassette for SNC/IPA Calibration

14.3.2 Confirm that the cassette is loaded with the calibration standards the following order:

14.3.2.1 First position: Carbon-14 standard (this is at the same end of the cassette as the protocol flag).

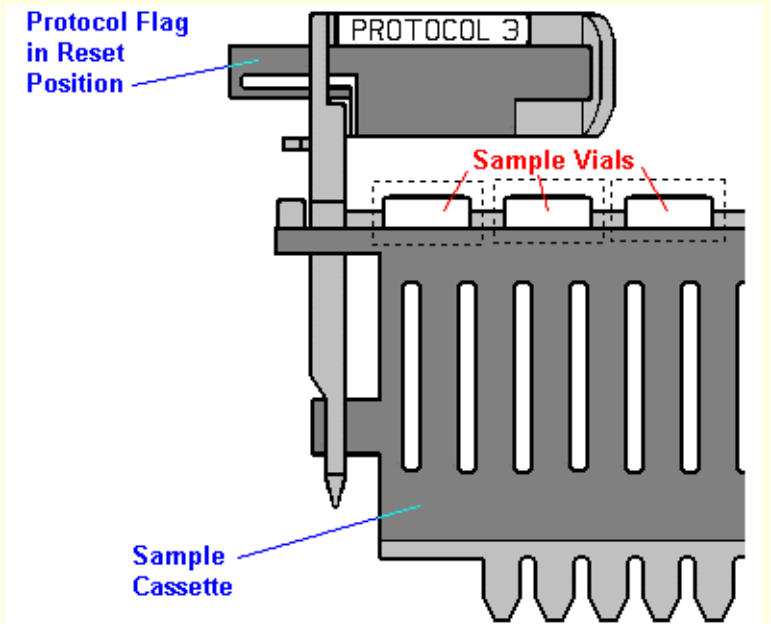
14.3.2.2 Second position: Tritium standard (3H).

14.3.2.3 Third position: Background standard.

14.3.3 Load the calibration cassette on the right-hand side of the sample changer deck such that you can read the protocol flag.

NOTE: This cassette will always remain in the system.

14.3.1 On the header of computer monitor, Press the Start button (green flag) to begin counting.



Sample Cassette

14.4 Loading Samples into Cassettes

14.4.1 Cassettes are the plastic racks which hold sample vials and allow them to be moved on the sample changer deck. Samples are placed into cassettes that accommodate standard 20 ml vials. A numbered protocol flag, which you attach to the cassette, is used to identify the protocol and assay conditions that you have defined for use with your samples. Each cassette is identified by a unique number (PID) located at the end of the cassette. The protocol flag and PID are automatically read by the instrument.

14.4.2 The maximum number of vials per cassette is 12 vials. Each batch includes four Q.C. samples – two at the beginning of the sample run, and two at the end. If the total number of sample vials including Q.C. is greater than 12, it will be necessary to use a second cassette. In that case, you must remove the protocol flag from the second cassette.

14.4.3 Load samples into cassettes in the following sequence, going left to right from the protocol flag:

14.4.3.1 First position: Method Blank.

14.4.3.2 Second position: Mid-range Quench Standard (Quench Standard E).

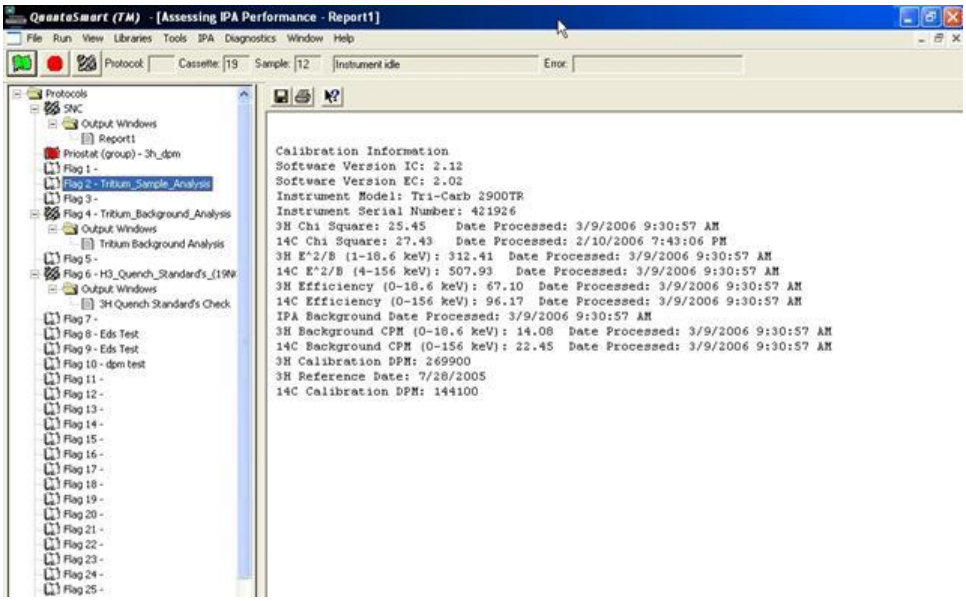
14.4.3.3 Load samples, in order by LIMS ID.

14.4.3.4 After the last sample, load another mid-range Quench Standard (Quench Standard F), followed by the second Method Blank.

14.4.4 Record the sample vial run sequence information and other information as specified on a blank copy of the Packard TriCarb 2900TR Liquid Scintillation Batch Log. See Appendix B. File the log in the designated binder.

14.5 Associating an Assay with a Protocol Flag

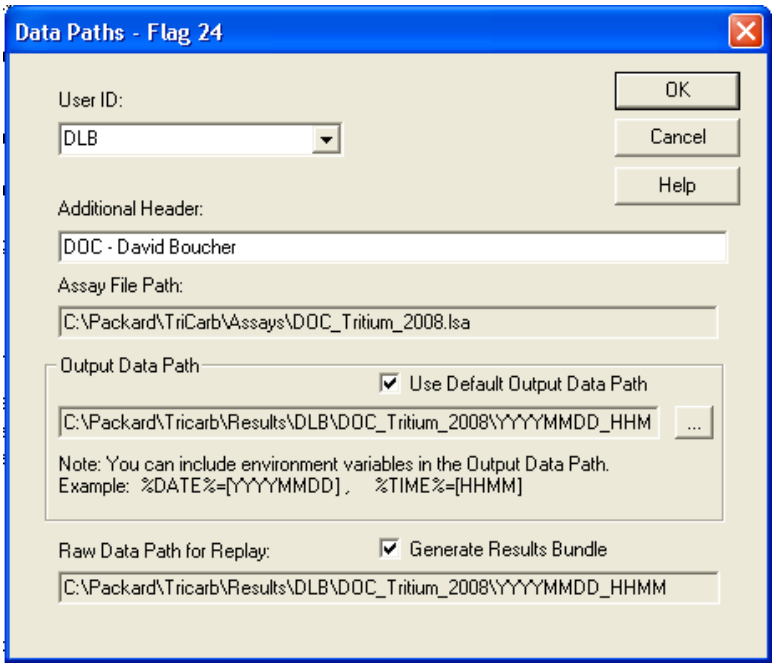
14.5.1 In the left-hand panel of the QuantaSmart window, right-click on the flag with the same number as the protocol flag on your cassette. A menu will appear. Select “Dissociate Assay” on the menu, and click it.



14.5.2 Right-click on the same flag again, and select “Associate Assay.” A window will open with a list of files. Select “Tritium_Sample_Analysis_2008.lsa”, and click on Open.

14.5.3 A menu will appear, with the header “Data Paths – Flag ##” – “##” being the number of the flag you are using. This menu determines the location on the instrument hard drive where the Assay results will be stored.

14.5.4 Select the User ID that corresponds to your own name. Any descriptive information that you want to appear in the header of the instrument printout can be entered in the “Additional Header” field. Click **OK** to save the information and continue.



14.5.5 **NOTE: DO NOT MAKE ANY CHANGES OF ANY KIND TO AN EXISTING ASSAY.** If changes are needed, save the assay under a different name.

14.6 **Loading Cassettes into the Instrument.**

14.6.1 Slowly and carefully raise the lid to the Packard TriCarb 2900TR. **Use caution when raising the lid so that it doesn’t slam into the monitor above the system.**

14.6.2 Place the sample cassettes in the right-hand side of the system, facing so that the protocol flags are on the left. The cassette sequence will be run in reverse order, starting with the one farthest towards the back and ending with the one closet to the front. Place the cassettes in order as follows:

14.6.3 First Cassette (farthest toward the back): SNC/IPA Cassette.

14.6.4 Next Cassette: Sample cassette with protocol flag.

14.6.5 If there is more than one cassette with samples, make sure that only the first cassette in the sequence has a protocol flag.

14.6.6 Make sure that the all of the protocol flags have been pushed to the left-hand position.

14.7 **Running Samples.**

14.7.1 After all samples have been loaded into the instrument and the sample names and position in the cassette have been recorded, close the lid of the instrument.

14.7.2 Wait a minimum of one hour to allow the samples to dark-adapt before starting the sample run. Fluorescent lights can cause luminescence to occur in the scintillation cocktail. Dark-adaption allows any induced luminescence to subside.

14.7.3 After the samples have had time to dark-adapt, the instrument is ready for sample counting.

14.7.4 Check to be certain that the EPSON 880 printer (to the left of the system) is loaded with paper.

14.7.5 On the header of computer monitor, Press the Start button (green flag) to begin counting.

14.7.6 Once sample counting is completed, remove samples from system and dispose of properly (See Section 21.0).

15.0 Evaluation of Data, Reporting Results and Calculations

15.1 All quality control measures must be evaluated. Samples associated with failed Quality Control shall be recounted if possible. Acceptance Criteria for quality control measures are listed in Section 18.0, "Data Assessment and Acceptance Criteria."

15.2 Results are calculated with background subtraction.

15.3 Sample results and uncertainties shall be entered into the LIMS. Quality control data shall be linked to sample results in the LIMS. The procedure for data entry into the LIMS is described in Appendix A.3.

15.4 Chi-square is a measure of the reproducibility of the instrument count. Chi-square values are calculated for ^{3}H and ^{14}C during the SNC/IPA calibration procedure, according to the following formula:

$$\chi^2 = \sum_{i=1}^{n} \frac{(X_i - \bar{X})^2}{X_i}$$

Where: χ^2= Ch-square value

$\bar{\bar{X}}$ = mean value of *n* measurements

X_i= value of ith measurement

15.5 Figure of Merit (E^2/B) is a measure of the sensitivity of the instrument. Figure-of-Merit values are calculated for ^{3}H and ^{14}C during the SNC/IPA calibration procedure, according to the following formula:

$$\text{Figure of Merit} = \frac{(\text{Efficiency})^2}{\text{Background}}$$

15.6 Decay corrections for the activity of the calibration standards and quench standards are calculated according to the following formula:

$$A_t = A_0 e^{-\lambda t}$$

Where: A_t = Decay corrected activity

A_0 = Activity at the time of calibration

$\lambda = \frac{\ln 2}{t_{1/2}}$

$t_{1/2}$ = 4530.37 days

t = time from the calibration date to the date of activity calculation in days

15.7 Decay corrections are not used for tritium test samples. For samples analyzed within the required six-month hold time, the effect of decay correction would be negligible (less than 2.8%).

15.8 The relative percent difference (RPD%) between the two quality control standards is calculated by the following formula:

$$RPD\% = \left| \frac{2 \times (A_1 - A_2)}{A_1 + A_2} \right| \times 100\%$$

Where: A_1 = Activity of 1st Q.C. Standard (DPM)

A_2 = Activity of 2nd Q.C. Standard. DPM

15.9 Percent Recovery for the Quality Control standards is calculated by the following formula:

$$\%Recovery = \frac{A_m}{A_c} \times 100\%$$

Where: A_m = Measured Activity of Q.C. Standard (DPM)

A_c = Calculated Activity of Q.C. Standard (DPM) decayed to date of analysis.

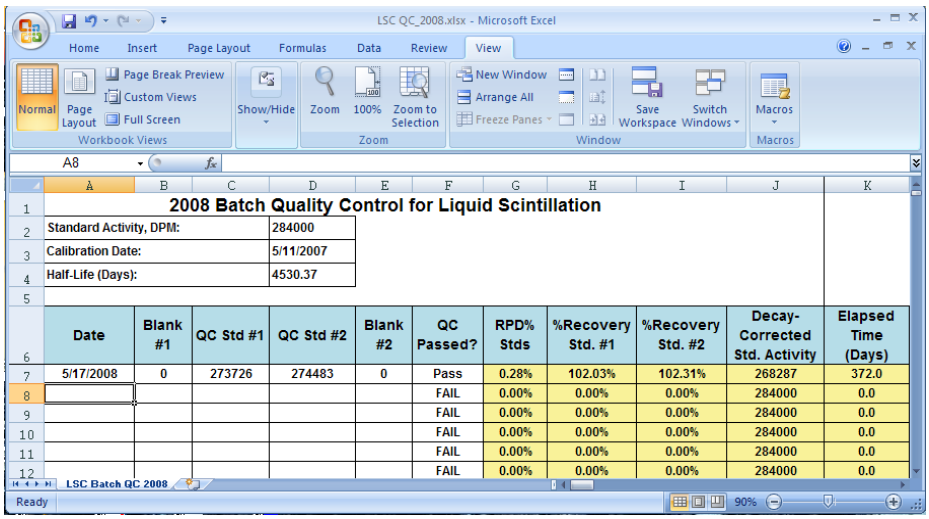
15.10 Recording Batch Quality Control Data on Spreadsheet

15.10.1 Right-click on the “Start” button and select “Explore.”

15.10.2 Open the folder L:\Radfiles\2008_LS_data.

15.10.3 Open the spreadsheet “LSC_Batch_QC_2008.xlsx”

15.10.4 There are five columns with fields for Q.C. entry. All the other fields are locked. Scroll down to the first open row below the last entry.



15.10.5 Enter the following information from the sample results printout in the lowest open row, left to right:

15.10.5.1 In the “Date” column, enter the starting date for the sample run.

15.10.5.2 In the column labeled “Blank #1”, enter the average DPM for the first method blank (1st position on the cassette).

15.10.5.3 In the column labeled “QC Std #1”, enter the average DPM for the first Q.C. sample (2nd position in the cassette).

15.10.5.4 In the column labeled “QC Std #2”, enter the average DPM for the second Q.C. sample (next to last sample position).

15.10.5.5 In the column labeled “Blank #2”, enter the average DPM for the second method blank (last sample position).

15.10.6 The spreadsheet will automatically calculate the RPD% and the %Recovery for the QC Standards. The column labeled “QC Passed?” will automatically indicate whether the Q.C. has passed or failed.

15.10.7 Save the spreadsheet and exit. This spreadsheet will be linked to the sample results in the LIMS. See Section 3 of Appendix A for details.

16.0 Method Performance

16.1 Method Performance is demonstrated by successful analysis of quality control samples of known and documented activity and composition.

16.1.1 Determination of Accuracy and Precision

16.1.1.1 Accuracy is evaluated by the percent recovery (%recovery) obtained in the analysis of Quality Control Standards.

16.1.1.2 Precision is evaluated by the relative percent difference (RPD%) obtained in the analysis of two Quality Control standards with the same level of activity.

16.2 How to Perform a Demonstration of Capability

16.2.1 Load the ten Quench Standards into Cassette #40.

16.2.2 Place the SNC Cassette in the instrument, followed by Cassette #40 with the Quench Standards.

16.2.3 Confirm that the protocol flags are pushed to the left.

16.2.4 Associate the Assay "DOC_Tritium_2008.lsa" with the Protocol flag for Cassette #40, as described in Section 14.5.

16.2.5 Close the lid of the instrument and press the Start button (green flag) to begin counting.

16.2.6 After the counting is complete, review the instrument printout.

16.2.7 Open the folder "L:\Radfiles\RADDOCS" and open the Excel spreadsheet "Liquid_Scintillation_DOC_blank_2008.xlsx." Use "Save As" to save a copy of the spreadsheet, substituting your name for the word "blank" in the file name.

16.2.8 Enter your name, the date, and the average DPM for each standard in the designated spaces on the renamed spreadsheet. The spreadsheet will automatically calculate the %recovery and will indicate whether the result is a pass or fail. See Appendix J for an image of the spreadsheet.

16.2.9 Save and exit the spreadsheet.

16.2.10 After four demonstrations of capability have been performed and recorded, print a copy of the spreadsheet and give it to the Supervisor along with a completed copy of the Demonstration of Capability Certification Statement form.

17.0 Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

17.2 The quantity of chemicals purchased shall be based on expected usage and shelf life in order to minimize the disposal of unused material.

17.3 Because environmental water samples generally have very low tritium activity, matrix spikes are not performed in order not to create an additional waste stream. It is considered that quench measurements provide a sufficient correction for potential matrix effects.

17.4 See also Section 21.0, "Waste Management."

18.0 Data Assessment and Acceptance Criteria

18.1 All data collected in the Packard TriCarb 2900TR Quality Control procedure are assessed for accuracy and acceptance criteria. Values outside of these ranges would indicate problems with the system and actions must be taken to correct any problem(s) before continuing with sample analysis.

18.2 The following acceptance criteria apply to the SNC/IPA calibration procedure:

18.2.1 The ^{3}H Efficiency must be greater than or equal to 60%.

18.2.2 The ^{3}H Background must be less than 17.3 CPM.

18.2.3 The ^{14}C Efficiency must be greater than or equal to 94%.

18.2.4 The ^{14}C Background count must be less than 24.3 CPM.

18.2.5 Chi-square values must be in the range of 7.63-36.19. The upper value represents the 99% confidence limit for 20 counts of 0.5 minutes duration.

18.2.6 Figure-of-Merit values must be greater than 300 for ^{3}H and 430 for ^{14}C.

18.3 The following acceptance criteria apply to sample batch Q.C.

18.3.1 Method Blank count must be below 10 CPM.

18.3.2 The %Recovery of the Quality Control Standards must fall in the range of 95-105%.

18.3.3 The Relative Percent Difference of the Quality Control Standards must be less than or equal to 5%.

18.3.4 For samples with an average sample count greater than 100 DPM, the Standard Deviation of the three sample counts must be less than 5% of the average value.

19.0 Corrective Action for Out of Control Data

19.1 If the SNC/IPA calibration procedure does not successfully complete or fails to meet all acceptance criteria, the SNC/IPA procedure will be repeated once.

19.2 If a second failure occurs, the instrument shall not be used until diagnostics have been completed and the problem resolved. The assistance of PerkinElmer (Packard) Service Engineer(s) shall be utilized to determine the cause of out of control data.

20.0 Contingencies for Handling Unacceptable Data

20.1 Unacceptable Q.C. data will result in the instrument being unavailable for use until corrective action has been taken and the data indicates that the corrective action taken has effectively resolved the problem.

21.0 Waste Management

21.1 Samples with an exposure rate less than or equal to 200 µR/h, or that upon analysis are shown to have activity equivalent to background or control samples can be disposed of in the same manner as ordinary waste. After a sample has been analyzed, if it is determined that the total sample activity is lower than 200 pCi, the sample+ can be handled and disposed of as non-radioactive material.

21.1.1 Liquid samples must be drained into the double black sink in R01 with plenty of running water.

21.2 Samples that are determined to have radioactive levels unsuitable for regular disposal will be disposed in a safe way and by an NRC approved method.

22.0 Data and Records Management

22.1 Procedures for data and records management must adhere to the Quality Manual and subordinate documents covering record keeping and the document control plan. Records documenting all laboratory activities shall be retained permanently. All records shall be stored in such a way as to be secure and accessible.

22.2 Data generated during Liquid Scintillation analysis shall be reviewed by laboratory staff. All printed records must be initialed and dated by the analyst.

22.3 Certificates of analysis for standards used shall be kept in the designated binder by the analyst.

22.4 All ongoing maintenance and preventive maintenance must be documented in the bound maintenance log book for the instrument

(RAD-014, “Packard (PerkinElmer) TriCarb 2900 TR Maintenance Log”). The log book is kept on the bench top to the right of the instrument.

22.5 Electronic records are kept stored on the computer hard drive and are backed up on compact disks.

22.6 Instrument printouts shall be initialed and dated, and kept in reverse chronological order in the designated locations.

22.6.1 All sample analysis report printouts shall be kept in the designated binder in reverse chronological order.

22.6.2 All SNC Protocol printouts shall be kept in the designated binder.

22.6.3 Instrument printouts for Demonstrations of Capability shall be attached to the designated form and kept in the employee training file.

22.7 Older documents shall be moved to storage folders, and kept in document storage boxes by year. Boxed documents shall be placed in the data storage room.

22.8 Tritium analysis data for all samples shall be entered into the LIMS system.

23.0 Tables, Diagrams, Flowcharts, and Validation Data

23.1 N/A

24.0 References

24.1 PerkinElmer “QuantaSmart for the TriCarb Liquid Scintillation Analyzer Getting Started Manual”

24.2 PerkinElmer “QuantaSmart for the TriCarb Liquid Scintillation Analyzer Reference Manual”

Appendix A. Batching Samples into LIMS and Entering Data

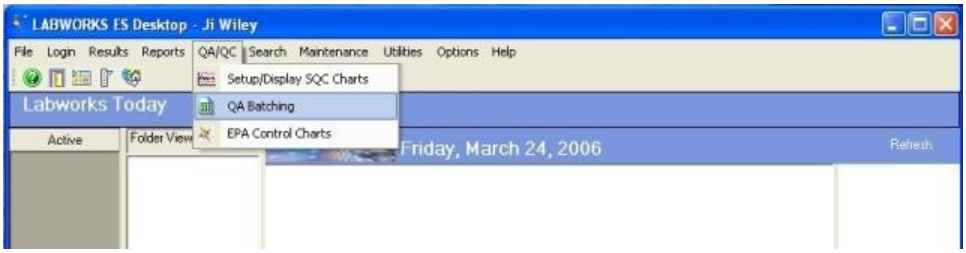
A.1 Logging on to the System

A.1.1 Click on **Labworks** icon on the desktop

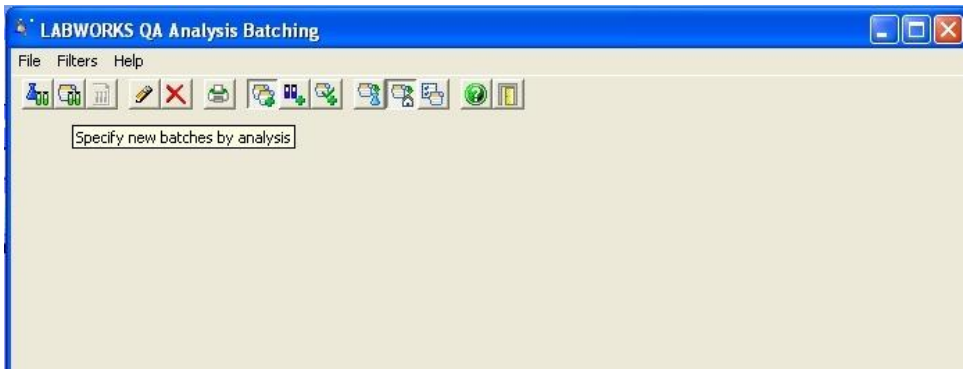
A.1.2 Enter user name and password

A.2 Batching Samples into LIMS

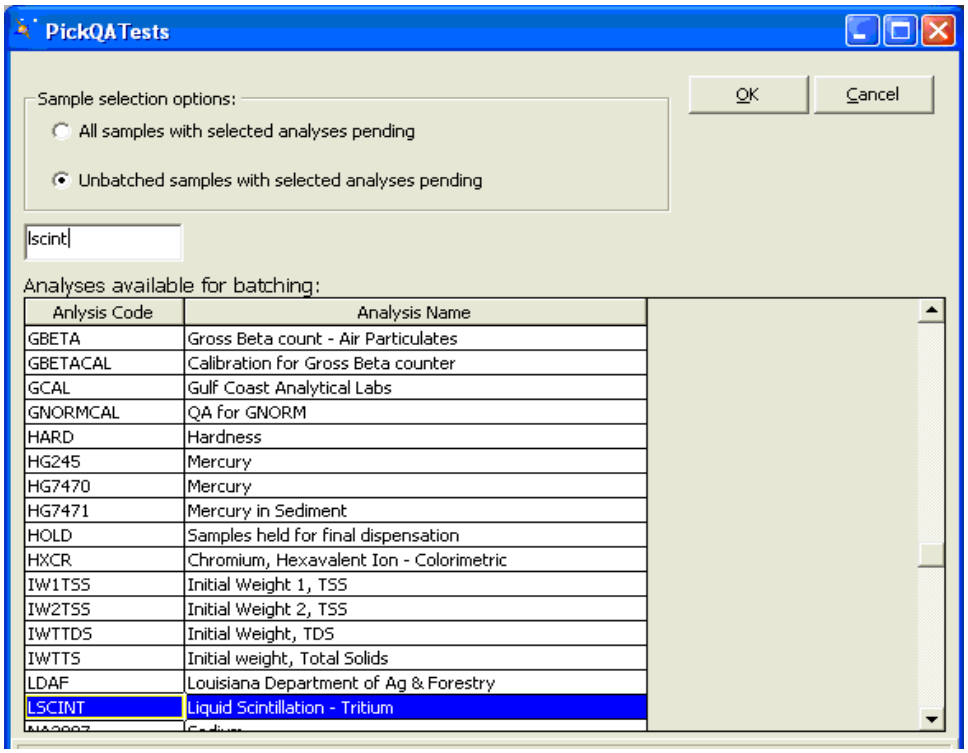
A.2.1 Click **QA\QC** icon and select **QA Batching**.



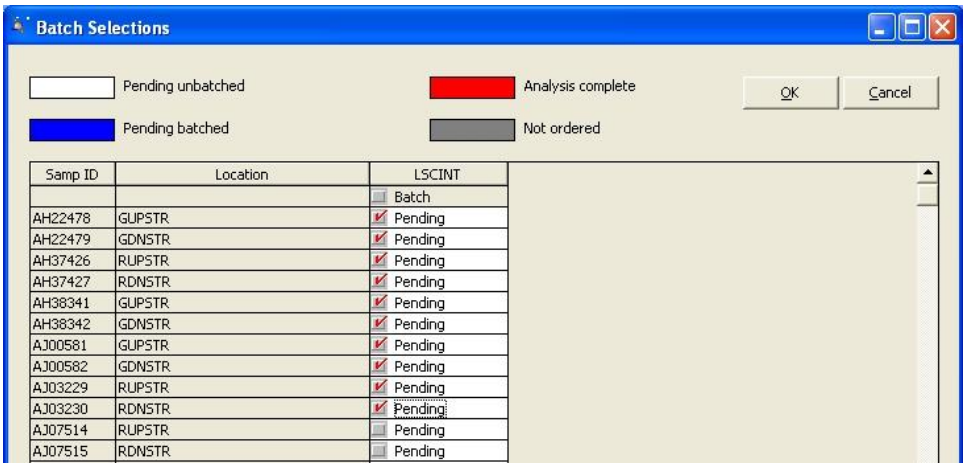
A.2.2 Click on 2nd icon which is selecting new batches by analysis.



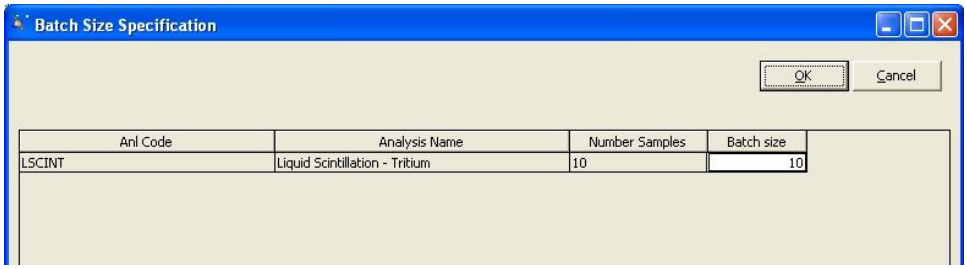
A.2.3 By default, the option **Unbatched samples with selected analysis pending** is selected. Type LSCINT test code in the blank field to select **LSCINT** analyses and click **OK**.



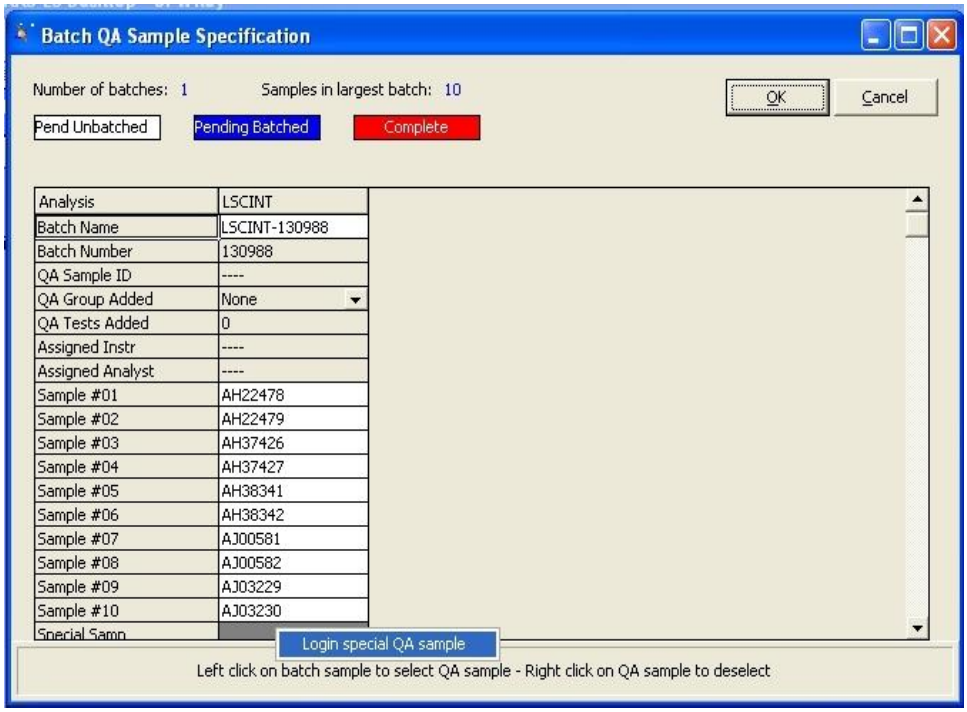
A.2.4 If all samples are to be batched, click **OK** to continue. If not unclick the arrow in the top cell next to Batch which will then unselect all samples. Click the individual samples to select the samples to be batched.



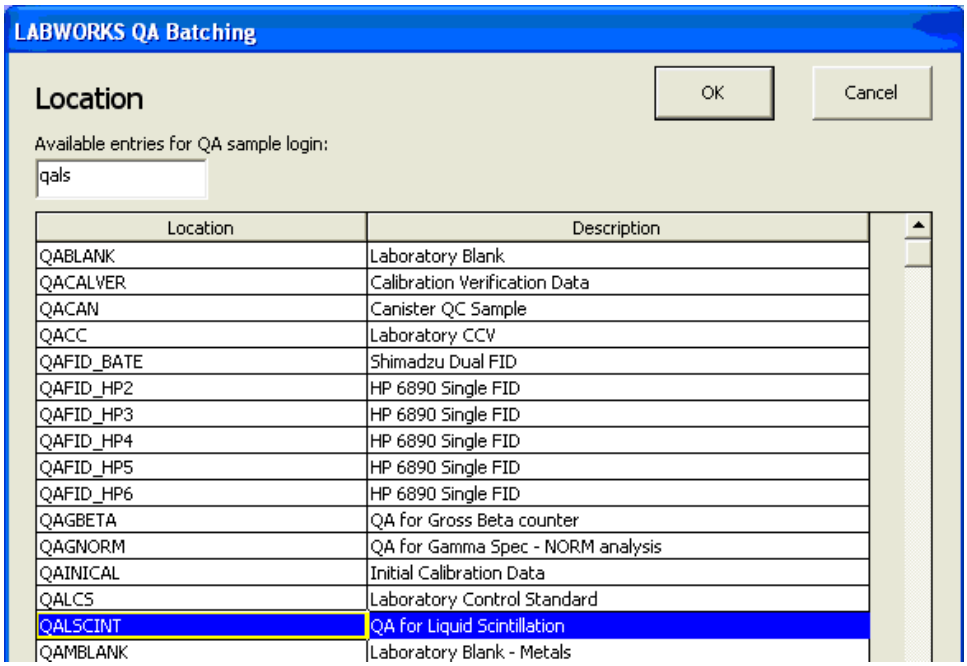
A.2.5 At the next screen, click OK to accept the batch size and continue. By default, LIMS is setup to have a maximum batch size of ten. If more samples are in the batch, type the number of actual samples into the cell under batch size to keep all samples together as one analytical batch.



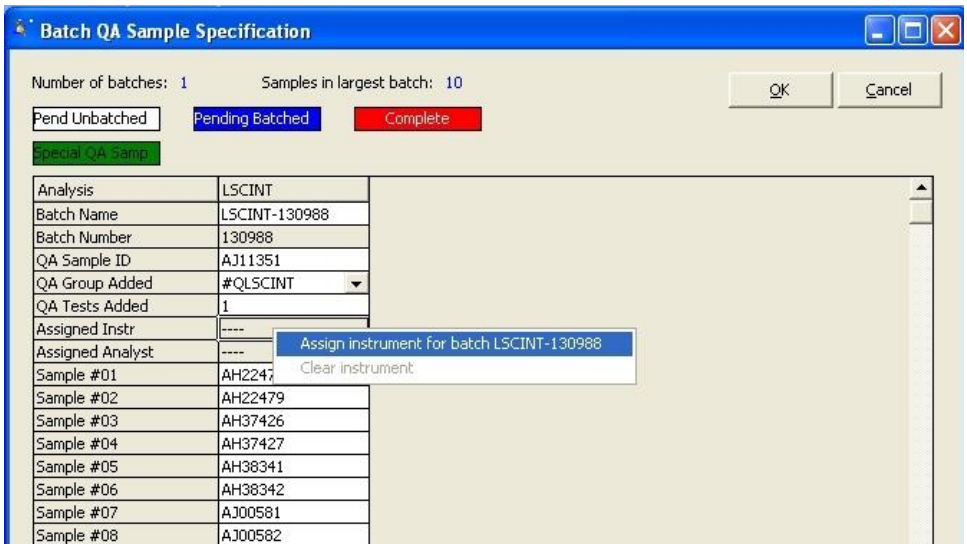
A.2.6 Right click on the empty cell designated **Special Sample** and choose **Login special QA sample**.



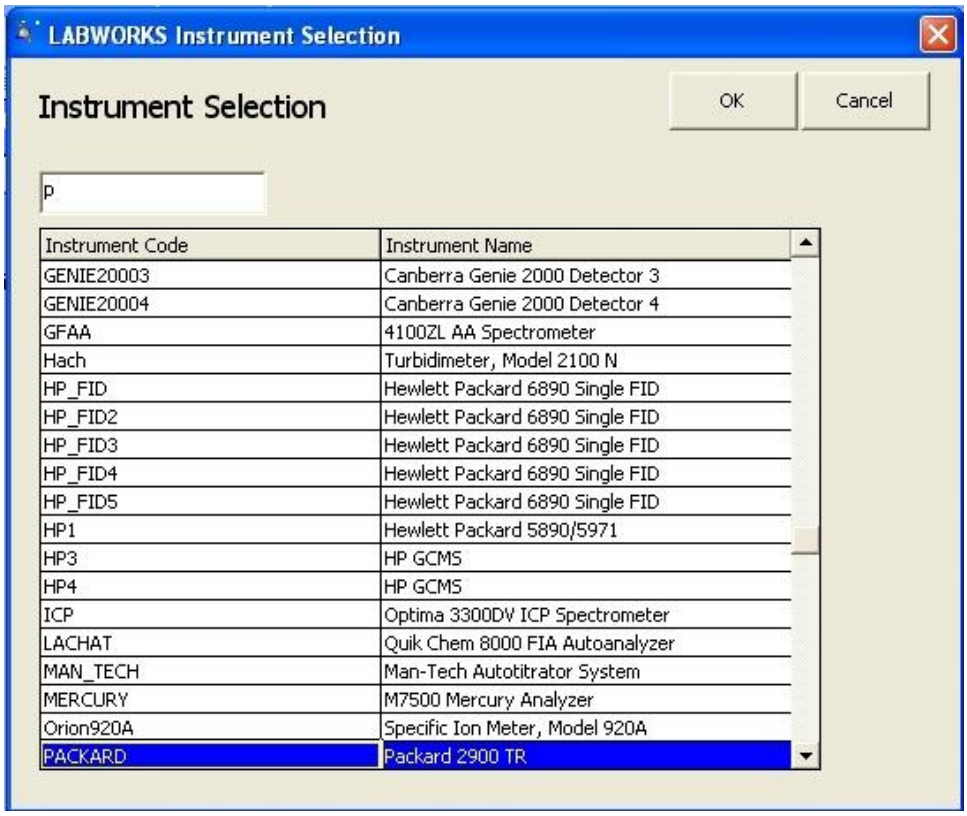
A.2.7 **LABWORKS QA Batching** screen will show up with available location codes. Type QALSCINT in the blank and highlight and click **OK**.



A.2.8 At the **QA Batch Sample specification** screen, select the instrument on which the tests will be run by right click next to the cell **Assigned Instrument**. Click on Assign instrument for batch **LSCINT-#####.**

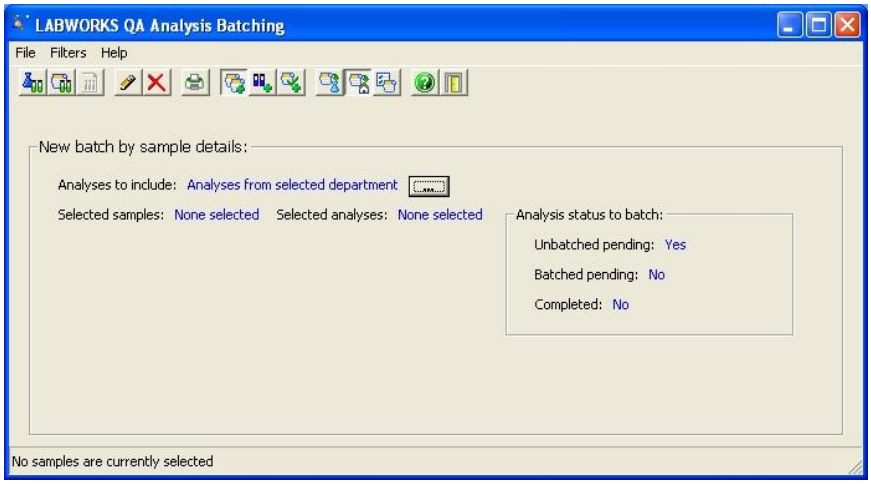


A.2.9 Type Packard into the blank. Select **PACKARD** and click **OK**.



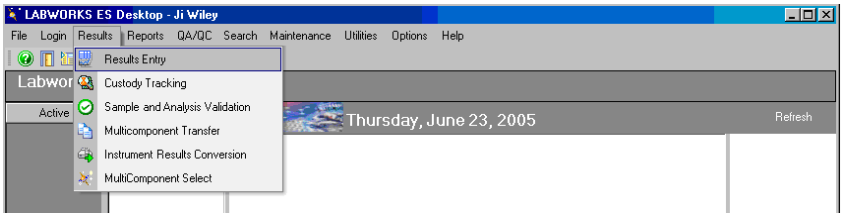
A.2.10 After adding **Special Sample** and assign the instrument to run, **Batch QA Sample Specification** screen will show all the information. Click **OK**. A batching status window will appear. Click **OK A.3.1**

A.2.11 LIMS will go back to **LABWORKS QA Analysis Batching** Screen.

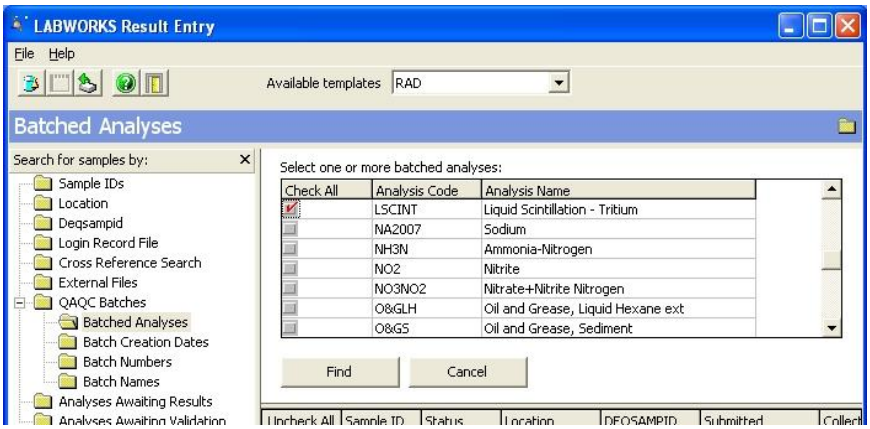


A.3 Entering Data into LIMS

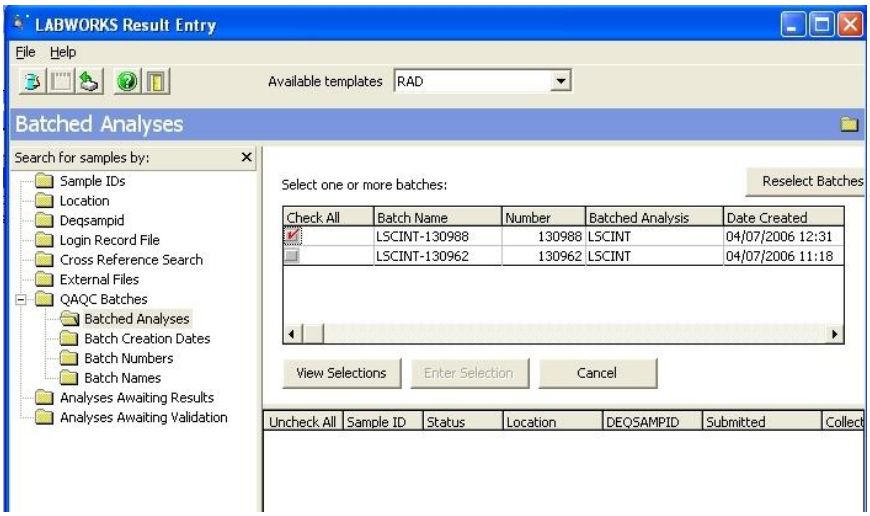
A.3.1 Select **Result Entry** in the **Results** menu.



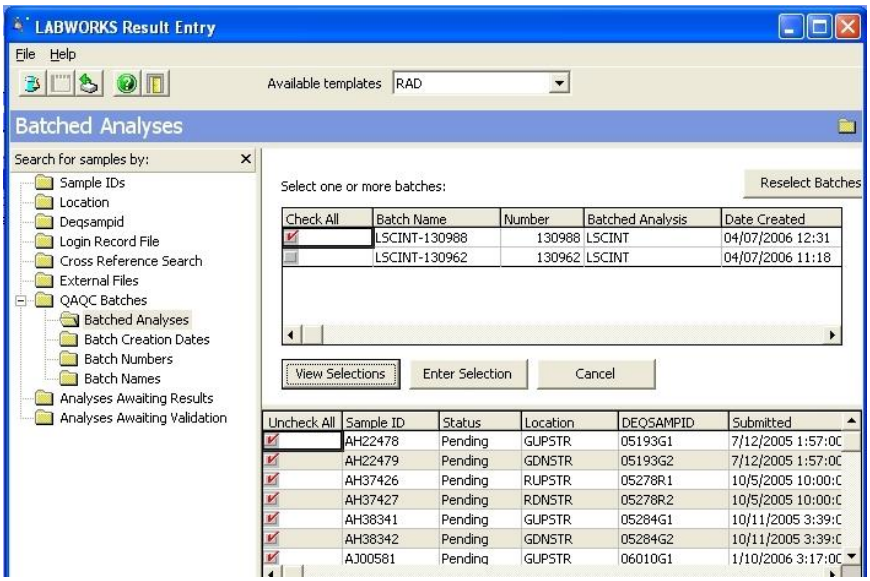
A.3.2 At the Result entry screen, choose the Batched Analyses under QA/QC Batches folder under search for samples by:, check LSCINT under select one or more batched analyses:. Click Find button.



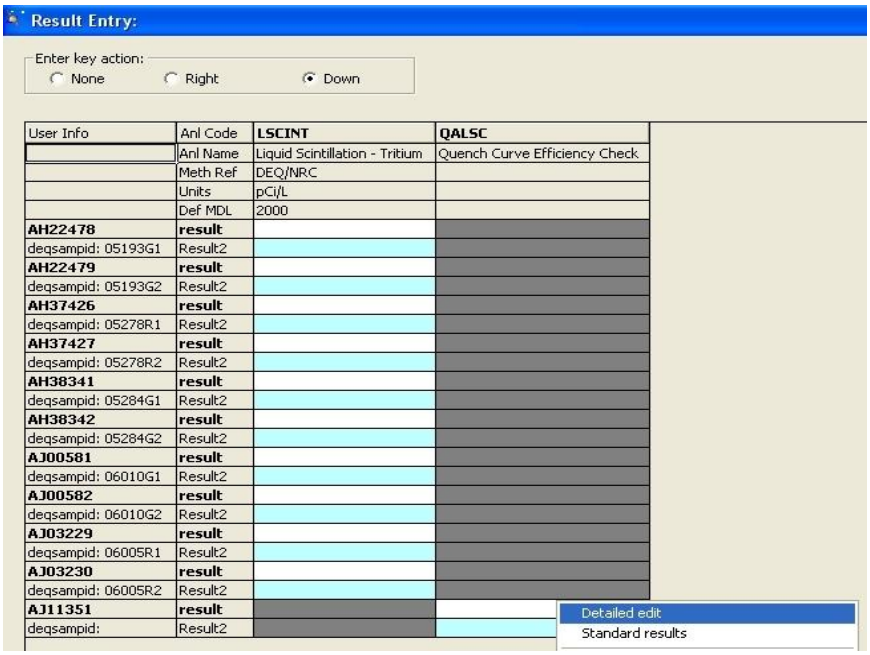
A.3.3 Check the **Batch Name** to enter the data into LIMS and click **View Selections**.



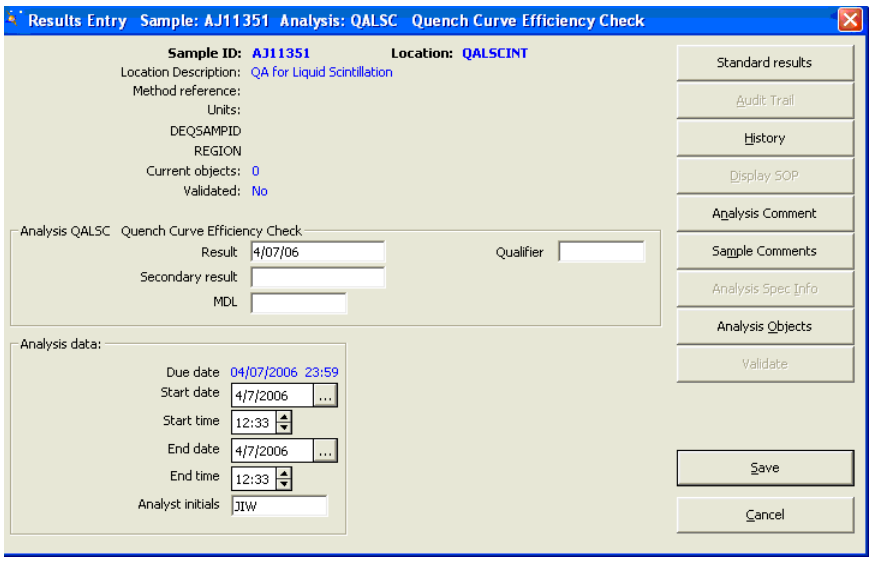
A.3.4 A list of samples will show up. Confirm that the LIMS Sample ID numbers match the entries on the Batch Log, as described in Appendix B. Click **Enter Selection** button.



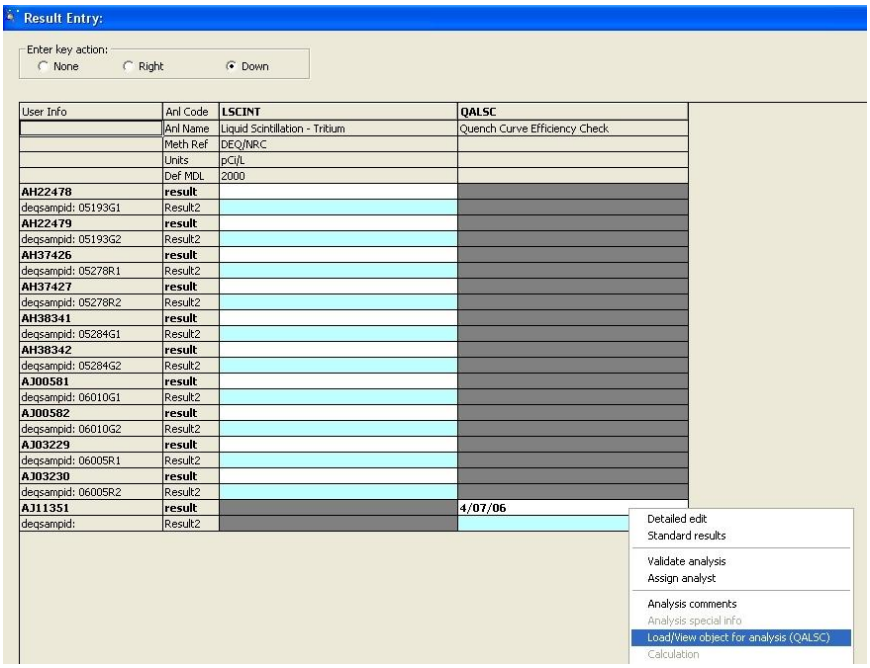
A.3.5 At the resulting screen, right click on the cell for QALSC to bring up the menu. Choose **Detailed Edit**.



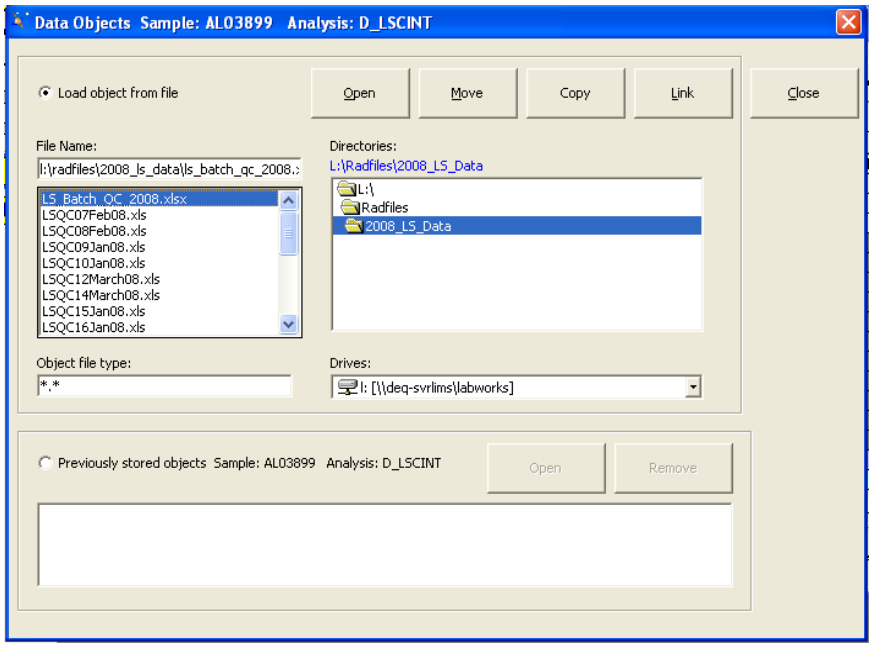
A.3.6 Change the Start Date/Time to match the “Date Processed:” for the Chi-Square values, immediately below the instrument serial number on the second page of the results printout. Change the End Date/Time to match the date/time in the header of the printout. Copy the Start Date into the “Result” field. When all the information is correct, click **Save**.



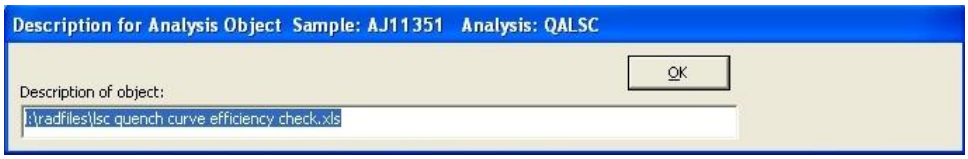
A.3.7 At the resulting screen, right-click on the cell for **QALSC** to bring up the menu again. Choose **Load\View analysis objects**.



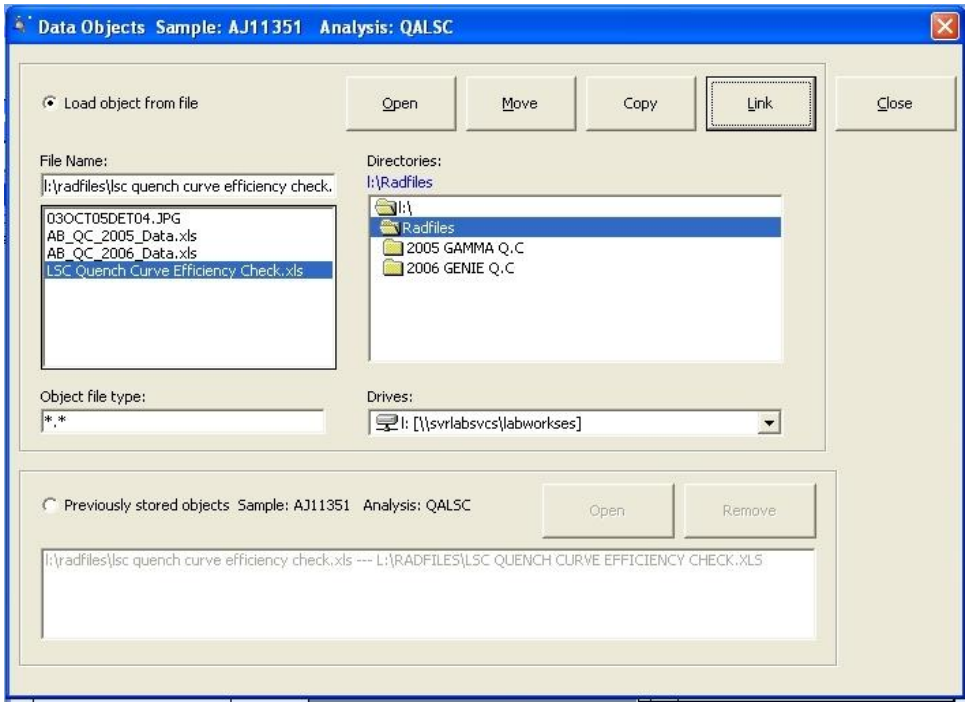
A.3.8 The menu below will appear. Double click the L:\ drive from the drop down menu and open the **Radfiles** folder. Open the sub-folder **2008_LS_Data.** Highlight the Excel spreadsheet "LSC_Batch_QC_2008.xlsx" and click **Link**.



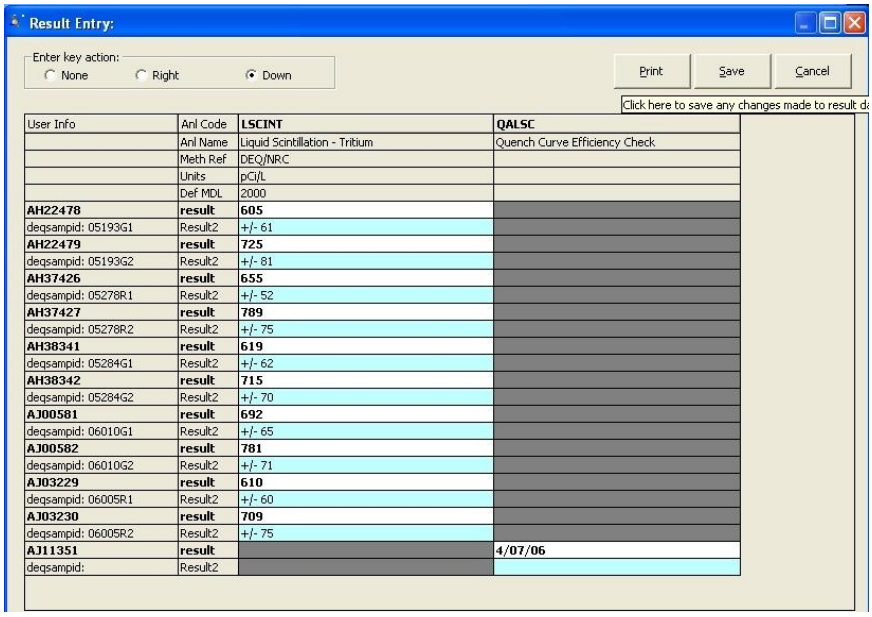
A.3.9 Click **OK** to accept the file description.



A.3.10 Once file has been linked, the name will appear in the previously stored objects box. Click **Close** to finish

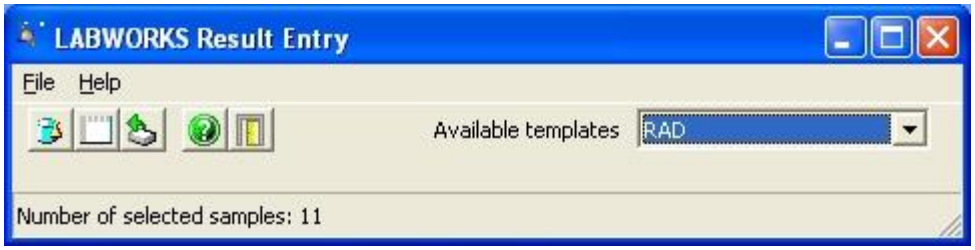


A.3.11 Enter the test results into the column labeled **LSCINT**. The primary Result is the average sample activity from the three sample counts, in pCi/L. "Result2" is the standard deviation of the average activity, also in pCi/L, and is entered with "+/-" as a prefix. After all of the results have been entered and checked for accuracy, click **Print** to get a paper copy of the results. After the copy has printed, click **Save.**



User Info	Anl Code	LSCINT	QALSC
	Anl Name	Liquid Scintillation - Tritium	Quench Curve Efficiency Check
	Meth Ref	DEQ/NRC	
	Units	pCi/L	
	Def MDL	2000	
AH22478	**result**	**605**	
deqsampid: 05193G1	Result2	+/- 61	
AH22479	**result**	**725**	
deqsampid: 05193G2	Result2	+/- 81	
AH37426	**result**	**655**	
deqsampid: 05278R1	Result2	+/- 52	
AH37427	**result**	**789**	
deqsampid: 05278R2	Result2	+/- 75	
AH38341	**result**	**619**	
deqsampid: 05284G1	Result2	+/- 62	
AH38342	**result**	**715**	
deqsampid: 05284G2	Result2	+/- 70	
AJ00581	**result**	**692**	
deqsampid: 06010G1	Result2	+/- 65	
AJ00582	**result**	**781**	
deqsampid: 06010G2	Result2	+/- 71	
AJ03229	**result**	**610**	
deqsampid: 06005R1	Result2	+/- 60	
AJ03230	**result**	**709**	
deqsampid: 06005R2	Result2	+/- 75	
AJ11351	**result**		**4/07/06**
deqsampid:	Result2		

A.3.12 Exit the LABWORKS Spreadsheet Result Entry screen by clicking the **X** button.



A.3.13 Close the **LABWORKS** program.

Appendix B: Packard TriCarb 2900TR Liquid Scintillation Batch Log

State of Louisiana
Department of Environmental Quality
LABORATORY SERVICES DIVISION
Environmental Radiation Laboratory

Packard TriCarb 2900 TR Liquid Scintillation Batch Log

Analyst: _____ Prep. Date: _____

Assay Type: _____ Run Date: _____

Cassette No. (PID):

Sample Position#	Sample ID /LIMS No.	Comments
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		

Cassette No. (PID):

Sample Position#	Sample ID /LIMS No.	Comments
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		

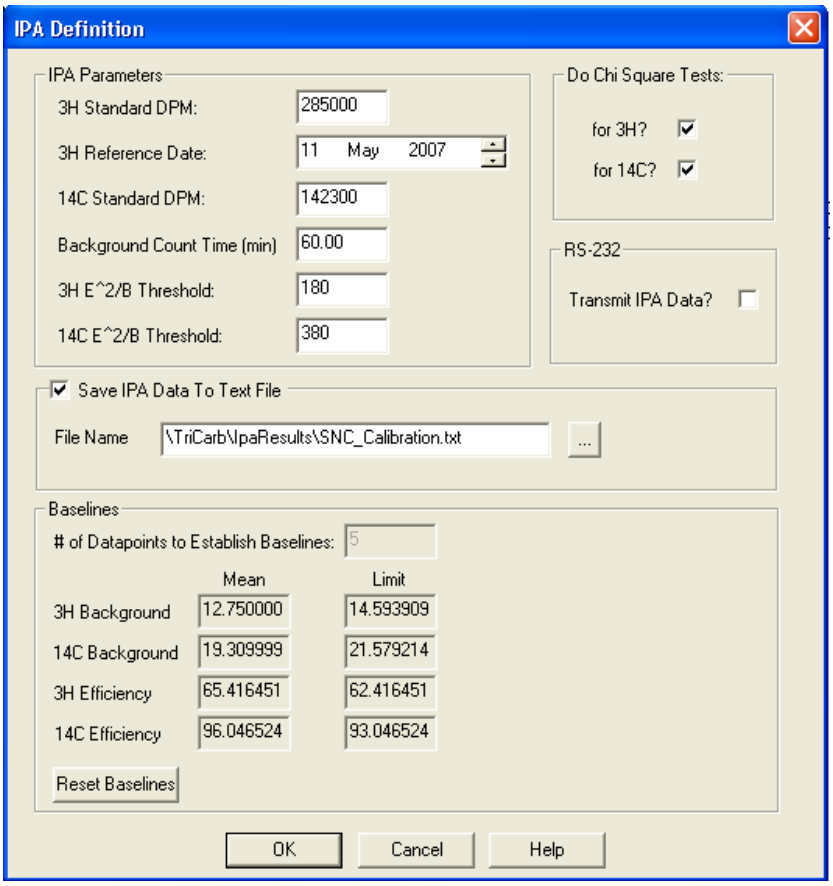
Reviewed by: _____ Date: _____

ENVIRONMENTAL RADIATION LABORATORY 1209 LEESVILLE AVENUE / P.O. BOX 4314 BATON ROUGE, LOUISIANA 70892/70821-4314
Packard TriCarb 2900 TR Liquid Scintillation Batch Log

Appendix C: Packard TriCarb 2900TR IPA Parameter Definition

C.1 The IPA Parameters are only to be changed when a new set of calibration standards are put in use, or when maintenance is performed on the photomultiplier assembly or other electronic components.

C.2 To define the IPA parameters, select “IPA Definition” from the IPA menu. The IPA Definition window is displayed:



C.3 It is not necessary to change the name of the text file in the IPA Definition. The instrument stores each new text file in a separate folder, with the folder name format YYYYMMDD-HHMM (YYYY=Year, MM=Month, DD=Day, HH=Hour, MM=Minute). Additionally, the IPA data are included in all sample test report printouts.

C.4 Do not change the Background Count Time or the E^2/B Thresholds. Do not deselect the Chi-square tests.

C.5 If you are entering a new set of calibration standards, enter the ^{3}H Standard DPM, ^{3}H Reference Date, and ^{14}C Standard DPM values in the designated fields. The standard DPM values are printed on the caps of the calibration standard vials. The ^{3}H Reference Date is printed on

the cap of the Tritium calibration standard vial. Because of the long half-life of Carbon-14, a reference date is not needed for ^{14}C.

C.5.1 After the calibration data for the new standards has been entered, click the "Reset Baselines" button. The baselines will be recalculated after the SNC protocol has been run for the number of times specified in the field labeled "# of Datapoints to Establish Baselines," currently set at 5 data points. Run the SNC protocol consecutively until new baselines have been established before resuming normal operation.

C.6 Data generated from the IPA runs can be accessed by selecting "IPA Charts & Tables" from the IPA menu.

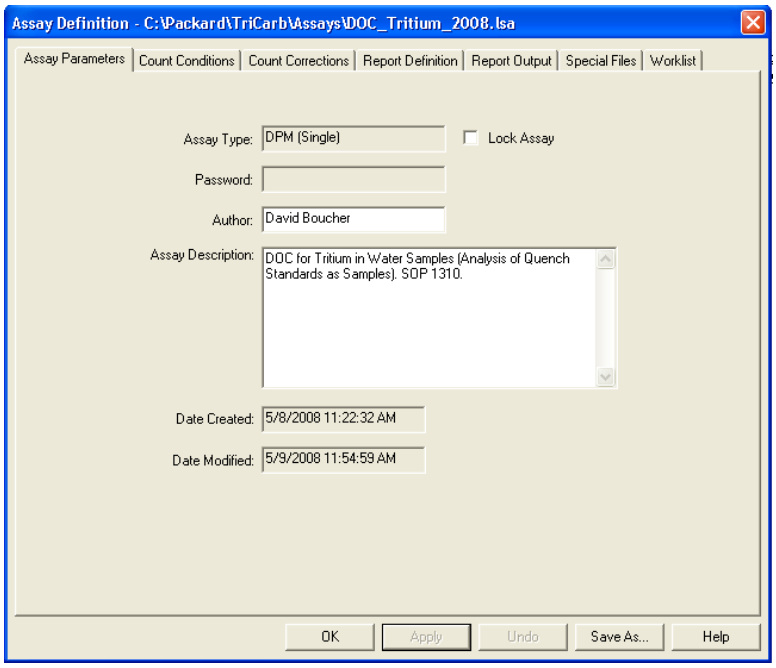
C.7 Current IPA parameters are listed in the table below:

Current IPA Parameters		
3H Standard DPM:		284000
3H Reference Date		1 October 2007
14C Standard DPM		142300
Background Count Time (min:		60.00
3H E^2/B Threshold:		180
14C E^2/B Threshold:		380
Do Chi-Square Tests:		
	For 3H:	Yes
	For 14C	Yes
Save IPA Data To Text File		Yes
File Name:		C:\Packard\TriCarb\IpaResults\SNC_Calibration.txt
# of Datapoints to Establish Baselines:		5

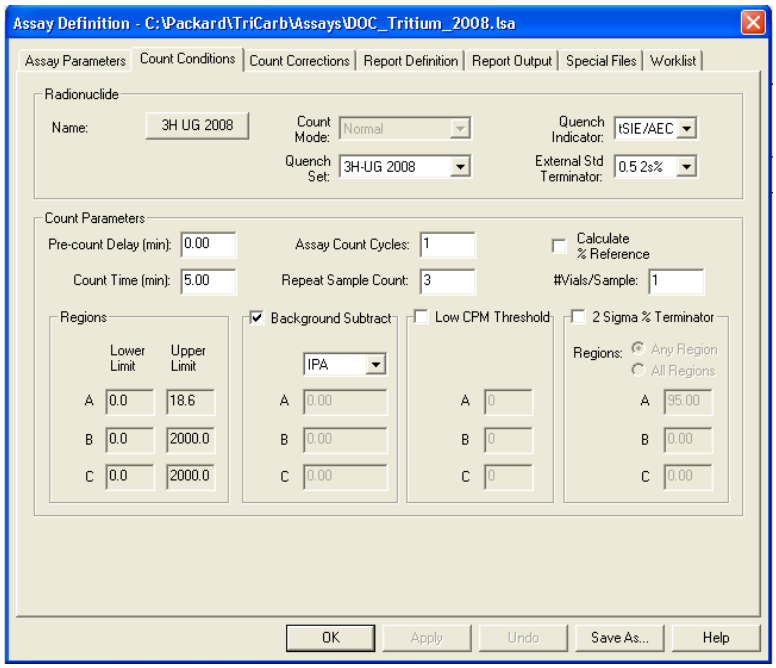
C.8 For more information, refer to Chapter 7 of the PerkinElmer "QuantaSmart for the TriCarb Liquid Scintillation Reference Manual."

Appendix D: Packard TriCarb 2900TR Assay Definition

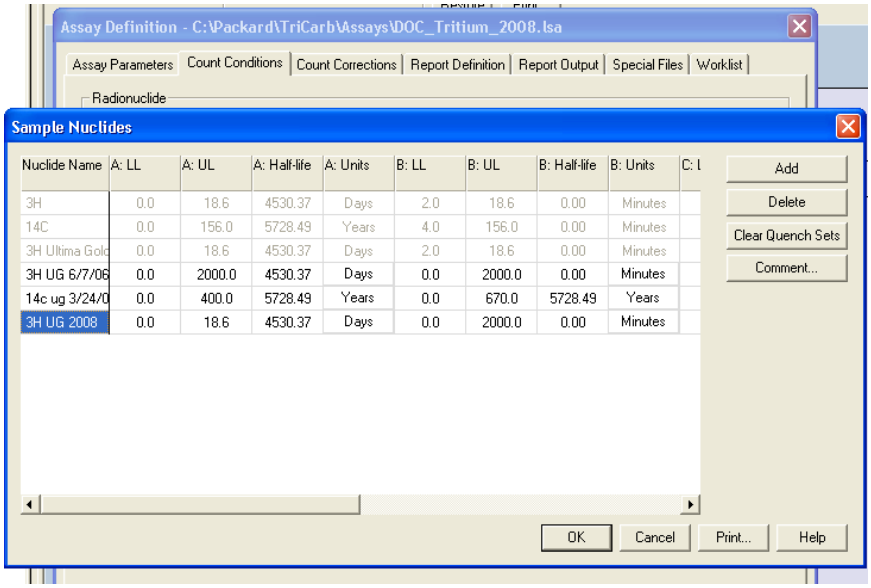
D.1 The Assay Parameters tab in the Assay Definition window allows the analyst to provide descriptive information for an assay. Assays used in the DEQ Environmental Radiation Laboratory have been defined, and can be used without changes.



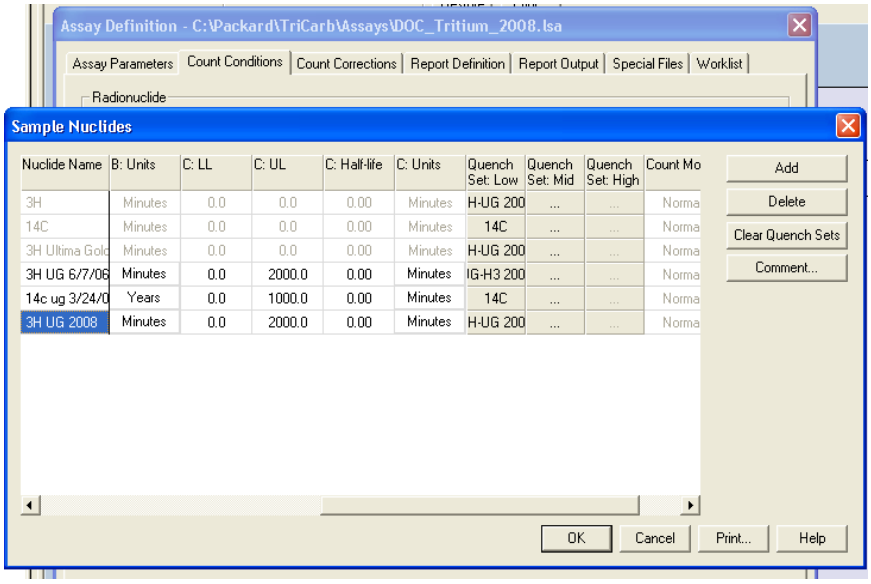
D.2 The Count Conditions tab in the Assay Definition window allows you to define specific counting parameters for an assay.



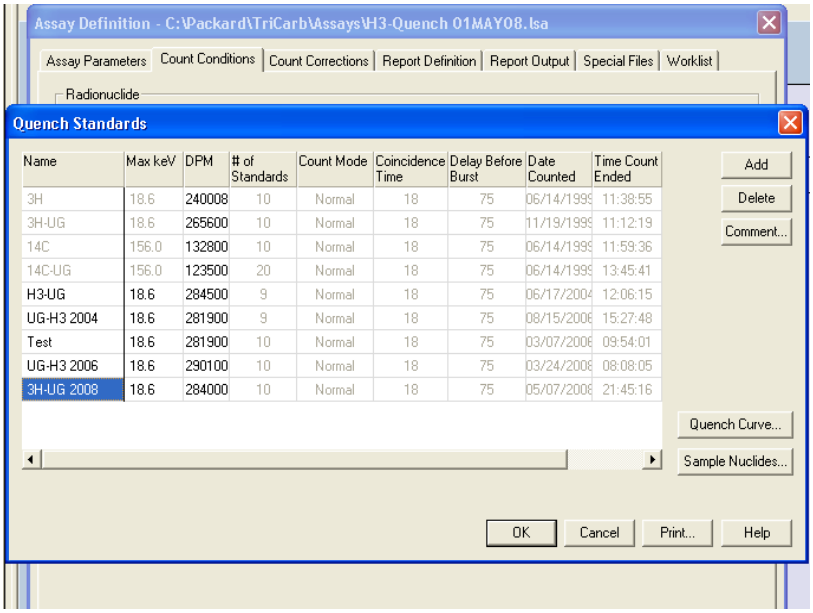
D.3 Clicking on the button for Radionuclide Name on the Count Conditions tab gives the “Sample Nuclides” menu, which allows you to define the nuclide name, half-life, and energy region for the nuclide of interest.



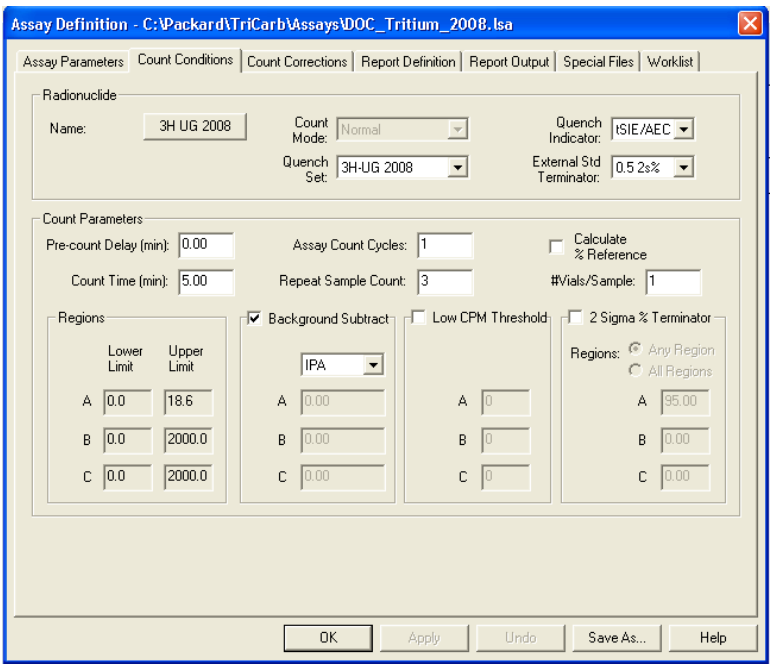
D.4 Slide to the right on the Sample Nuclides menu to select the Quench Curve that will be used in the sample analysis.



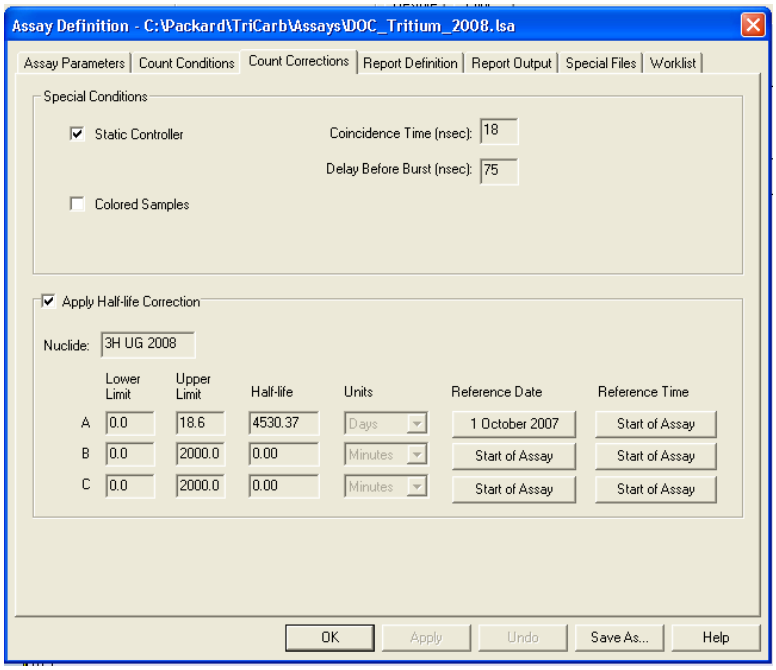
D.5 Click on the cell at the intersection of the “Quench Set Low” column with the row with your selected Radionuclide Name. The Quench Standards window will appear.



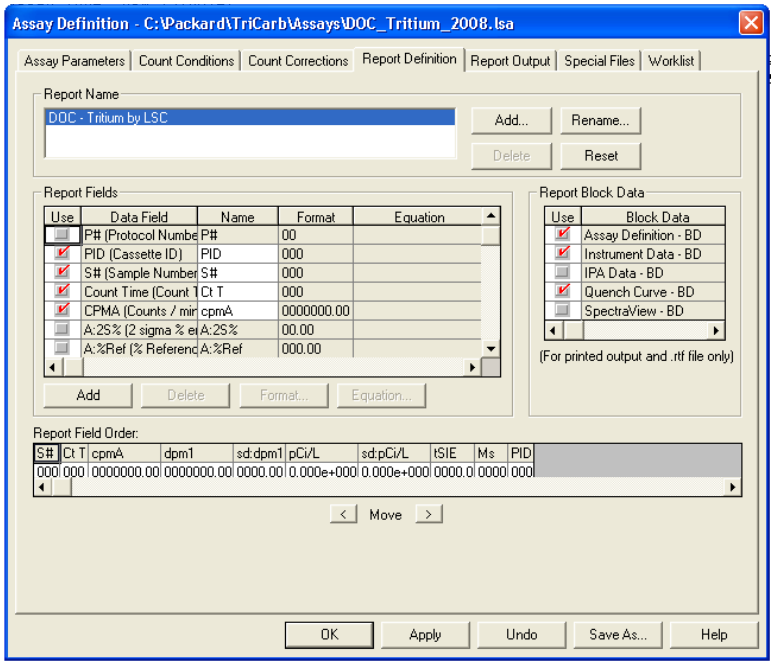
D.6 Select the new Quench Standard set from the list by clicking on it to highlight the selection, then click OK. The Assay Definition window will reappear.



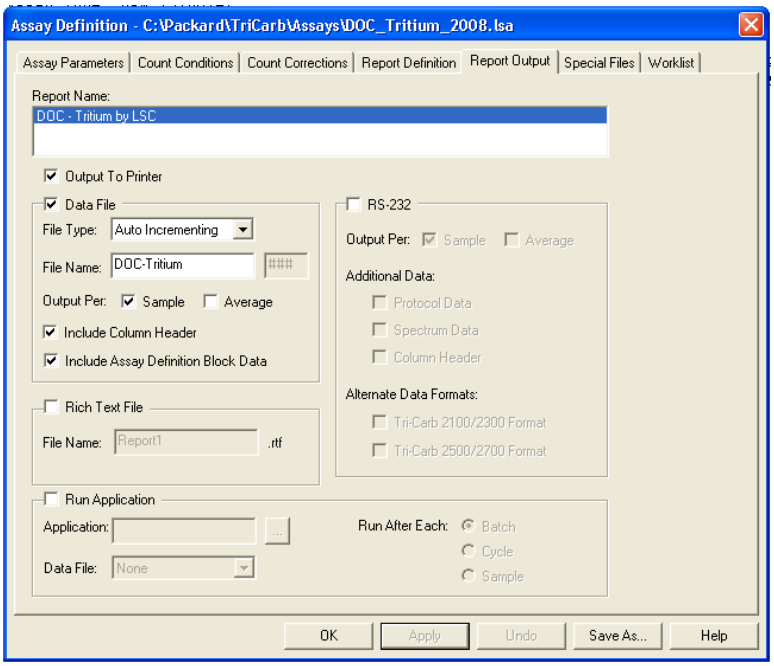
D.7 The Count Corrections tab in the Assay Definition window allows you to apply a half-life correction and to change the reference date.



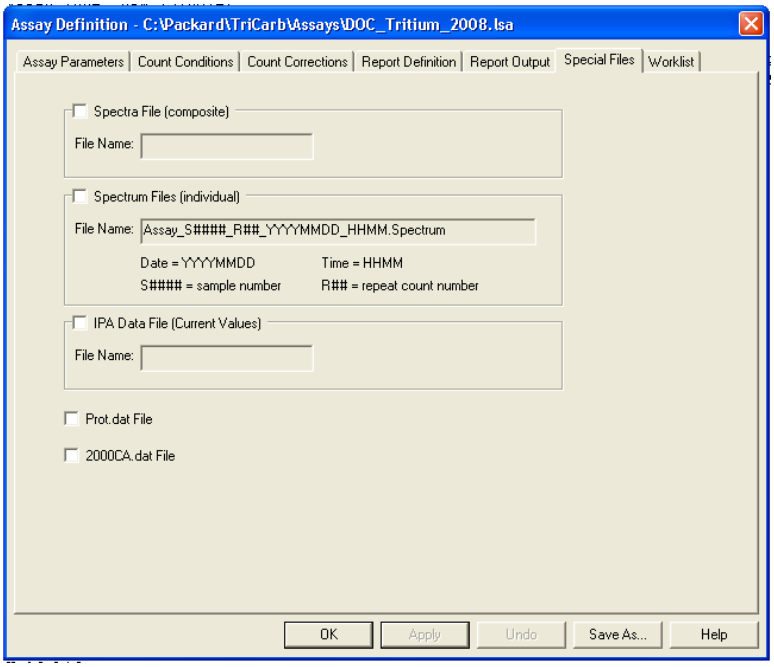
D.8 The Report Definition tab allows you to custom design a report.



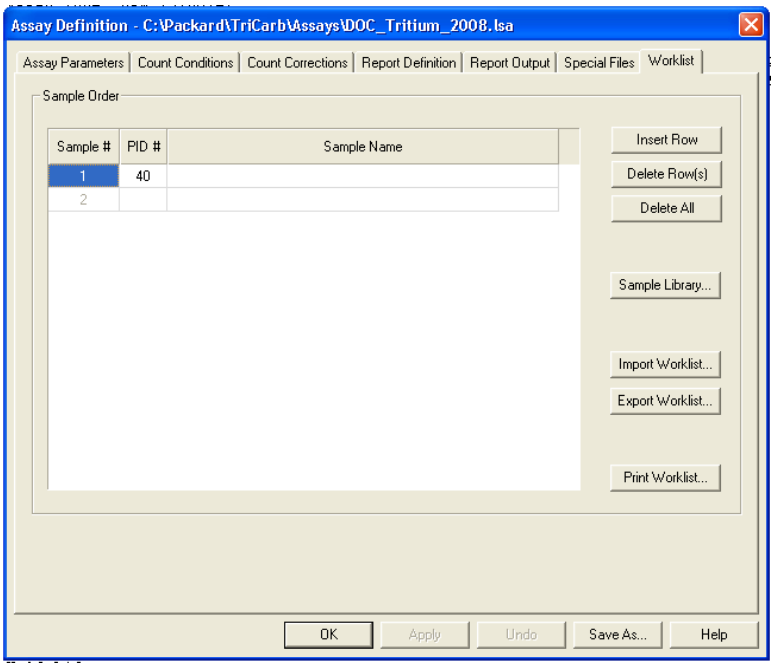
D.9 The Report Output tab offers options for the output of the report.



D.10 The Special Files window allows you to select and configure information regarding files pertinent to the assays.



D.11 The Worklist tab in the Assay Definition window allows you to designate Positive Identification numbers and sample names that correspond to sample numbers on a printout or electronic report. You may also create a Sample List Library of frequently used sample names.



D.12 Once you have defined all the assay parameters described in the seven tabs within the Assay Definition window, click Apply and OK. Save the file to an appropriate folder, giving the file a descriptive name. (See Header of Assay Definition screenshot above for an example).

D.13 For more information, refer to Chapter 5 of the PerkinElmer "QuantaSmart for the TriCarb Liquid Scintillation Reference Manual."

Appendix E: Packard TriCarb 2900TR Assay Parameters: Tritium Sample Analysis

Assay Definition – Tritium Sample Analysis			
Parameter File Name: C:\Packard\TriCarb\Assays\Tritium_Sample_Analysis_2008.lsa			
Assay Parameters Tab			
Assay Type:	DPM (single)		
Assay Description:	Tritium in Water Samples (SOP 1310_r08)		
Count Conditions Tab			
Name:	3H-UG 2008	**Assay Count Cycles:**	1
Quench Set:	3H-UG 2008	**Repeat Sample Count:**	3
Quench Indicator:	tSIE/AEC	**#Vials/Sample:**	1
External Std. Terminator:	0.5 2s%	**Background Subtract**	On - IPA
Pre-count Delay(min):	0.00	**Low CPM Threshold**	Off
Count Time (min):	30.00	**2 Sigma % Terminator**	Off
Regions:			

	Lower Limit	**Upper Limit**	Bkg Subtract
A	0.0	18.6	IPA
B	0.0	2000.0	IPA
C	0.0	2000.0	IPA

Count Corrections Tab	
Static Controller:	Yes
Coincidence Time (nsec):	18
Delay Before Burst (nsec):	75
Colored Samples:	Off
Half-Life Correction	Off

Report Definition Tab		
Report Name:	Tritium Analysis SOP 1310_r08	
Report Fields:		
Data Field	**Name**	**Format**
PID (Cassette ID)	PID	000
S# (Sample Number)	S#	00
Count Time (Count Time)	Ct T	000
CPMA (CPM - Region A)	cpmA	00000000
DPM1 (DPM nuclide 1)	dpm1	00000000
MESSAGES (Warnings or Messages)	Ms	0000
tSIE (Transformed Special Index of the External Std)	tSIE	0000.0
SD:DPM1 (Standard Deviation - nuclide 1)	sd:dpm1	00000.0
Custom 1	pCi/L	0.0000E+000 = dpm1÷(2.22*0.010 L)
Custom 2	sd:pCi/L	0.0000E+000 = sd:dpm1÷(2.22*0.010 L)

Assay Definition – Tritium Sample Analysis		
Parameter File Name: C:\Packard\TriCarb\Assays\Tritium_Sample_Analysis_2008.lsa		
Report Block Data		
Assay Definition - BD		
Instrument Data - BD		
IPA Data - BD		
Quench Curve - BD		
Report Field Order: S#; Ct T; cpmA; dpm1; sd:dpm1; pCi/L; sd:pCi/L; tSIE; Ms; PID		
Report Output Tab		
Report Name:	Tritium Analysis SOP 1310_r08	
Output to Printer	Yes	
Data File	Yes	
File Type:	Auto Incrementing	
File Name:	TritiumSampleAnalysis	.###
Output Per:	Sample	
Include Column Header:	On	
Include Assay Definition Block Data	On	
Rich Text File	n/a	
Special Files Tab		
no selections		
Worklist Tab		
Sample Order		
Sample #	**PID #**	**Sample Name**
1		

Appendix F: Packard TriCarb 2900TR Assay Parameters: Quench Curve

Assay Definition - Quench Curve					
Parameter File Name: C:\Packard\TriCarb\Assays\H3-Quench 01MAY08.lsa					
Assay Parameters Tab					
Assay Type:	Quench Standards				
Assay Description:	Ultima Gold Quench Standards Oct. 01, 2007				
Count Conditions Tab					
Name:	3H-UG 2008	**Assay Count Cycles:**	1		
Quench Set:	n/a	**Repeat Sample Count:**	n/a		
Quench Indicator:	tSIE/AEC	**#Vials/Sample:**	n/a		
External Std. Terminator:	0.5 2s%	**Background Subtract**	Off		
Pre-count Delay(min):	1.00	**Low CPM Threshold**	Off		
Count Time (min):	30.00	**2 Sigma % Terminator**	Off		
Regions:					
	Lower Limit	**Upper Limit**			
A	0.0	18.6			
Count Corrections Tab					
Static Controller:	On				
Coincidence Time (nsec):	18				
Delay Before Burst (nsec):	75				
Colored Samples:	On				
Apply Half-Life Correction	On				
Regions:	**Lower Limit**	**Upper Limit**	**Half-Life**	**Reference Date**	**Reference Time**
A	0.0	18.6	4530.37	01 October 2007	Start of Assay
Report Definition Tab					
Report Name:	Quench Curve				
Report Fields:					
Data Field	**Name**	**Format**			
S# (Sample Number)	S#	0000			
Count Time (Count Time)	Count Time	0000.00			
CPMA (Counts/minute - Region A	CPMA	000000			
SIS (Spectral Index of Sample)	SIS	0000.00			
tSIE (Transformed Special Index of the External Standard)	tSIE	0000.00			
MESSAGES (Warnings or Messages)	MESSAGES	0000			
Report Block Data					
Assay Definition - BD					
Instrument Data - BD					
IPA Data - BD					
Quench Curve - BD					
Report Field Order:	S#; Count Time; CPMA; SIS; tSIE; MESSAGES				

Assay Definition - Quench Curve		
Parameter File Name: C:\Packard\TriCarb\Assays\H3-Quench 01MAY08.lsa		
Report Output Tab		
Report Name:	Quench Curve	
Output to Printer	Yes	
Data File	Yes	
File Type:	Auto-Incrementing	
File Name:	Quench Standards	.###
Output Per:	Sample	
Include Column Header:	On	
Include Assay Definition Block Data	On	
Rich Text File	n/a	
Special Files Tab		
no selections		
Worklist Tab		
Sample Order		

Sample #	**PID #**	**Sample Name**
1	40	UG Tritium Quench Std. A
2	40	UG Tritium Quench Std. B
3	40	UG Tritium Quench Std. C
4	40	UG Tritium Quench Std. D
5	40	UG Tritium Quench Std. E
6	40	UG Tritium Quench Std. F
7	40	UG Tritium Quench Std. G
8	40	UG Tritium Quench Std. H
9	40	UG Tritium Quench Std. I
10	40	UG Tritium Quench Std. J

Appendix G: Packard TriCarb 2900TR Assay Parameters: Demonstration of Capability – Tritium

Assay Definition – Demonstration of Capability - Tritium			
Parameter File Name: C:\Packard\TriCarb\Assays\DOC_Tritium_2008.lsa			
Assay Parameters Tab			
Assay Type:	DPM (single)		
Assay Description:	DOC for Tritium in Water Samples (Analysis of Quench Standards as Samples) SOP 1310_r08		
Count Conditions Tab			
Name:	3H-UG 2008	**Assay Count Cycles:**	1
Quench Set:	3H-UG 2008	**Repeat Sample Count:**	3
Quench Indicator:	tSIE/AEC	**#Vials/Sample:**	1
External Std. Terminator:	0.5 2s%	**Background Subtract**	On - IPA
Pre-count Delay(min):	0.00	**Low CPM Threshold**	Off
Count Time (min):	5.00	**2 Sigma % Terminator**	Off
Regions:			

	Lower Limit	**Upper Limit**	Bkg Subtract
A	0.0	18.6	IPA
B	0.0	2000.0	IPA
C	0.0	2000.0	IPA

Count Corrections Tab	
Static Controller:	Yes
Coincidence Time (nsec):	18
Delay Before Burst (nsec):	75
Colored Samples:	Off
Half-Life Correction	On

Regions:		**Lower Limit**	**Upper Limit**	**Half-Life**	**Reference Date**	**Reference Time**
	A	0.0	18.6	4530.37	01 October 2007	Start of Assay
	B	2.0	2000.0	0.00	Start of Assay	Start of Assay
	C	0.0	2000.0	0.00	Start of Assay	Start of Assay

Report Definition Tab		
Report Name: DOC – Tritium by LSC		
Report Fields:		
Data Field	**Name**	**Format**
PID (Cassette ID)	PID	000
S# (Sample Number)	S#	00
Count Time (Count Time)	Ct T	000
CPMA (CPM - Region A)	cpmA	00000000
DPM1 (DPM nuclide 1)	dpm1	00000000
MESSAGES (Warnings or Messages)	Ms	0000
tSIE (Transformed Special Index of the External Std)	tSIE	0000.0

Assay Definition – Demonstration of Capability - Tritium			
Parameter File Name: C:\Packard\TriCarb\Assays\DOC_Tritium_2008.lsa			
SD:DPM1 (Standard Deviation - nuclide 1)	sd:dpm1	00000.0	
Custom 1	pCi/L	0.0000E+000	= dpm1÷(2.22*0.010 L)
Custom 2	sd:pCi/L	0.0000E+000	= sd:dpm1÷(2.22*0.010 L)
Report Block Data			
Assay Definition - BD			
Instrument Data - BD			
IPA Data - BD			
Quench Curve - BD			
Report Field Order: S#; Ct T; cpmA; dpm1; sd:dpm1; pCi/L; sd:pCi/L; tSIE; Ms; PID			
Report Output Tab			
Report Name:	DOC – Tritium by LSC		
Output to Printer	Yes		
Data File	Yes		
File Type:	Auto Incrementing		
File Name:	DOC - Tritium	.###	
Output Per:	Sample		
Include Column Header:	On		
Include Assay Definition Block Data	On		
Rich Text File	n/a		
Special Files Tab			
no selections			
Worklist Tab			
Sample Order			
Sample #	**PID #**	**Sample Name**	
1	40		

Appendix H: Packard TriCarb 2900TR Report Message Codes

Message Codes	
Code	Meaning
A	Represents an average value for replicate sample vials.
B	Represents data about a background subtraction vial.
C	Color quench correction for this sample is suspect (tSIE < 100)
E	Quench indicating parameter (SIS or tSIE) value was extrapolated from a portion of the quench curve beyond one of the end data points.
I	The DPM is indeterminate for the sample, i.e., the system could not reliably determine the DPM.
R	Represents data about a vial designated as a reference sample.
S	Represents data about a quench standards vial.
W	Indicates that the Positive ID number (PID) entered in the worklist does not match the PID read from the cassette.

Appendix I. Running a Quench Standards Assay.

I.1 If a new set of Quench Standards is being put in use, you must redefine the Quench Curve Assay parameters to conform to the new standards. If you are continuing to use the previous quench standard set, skip to Section I.2.

I.2 On the File menu, select “Open Assay.”

I.3 Open the Assay “C:\Packard\TriCarb\Assays\H3-Quench 01MAY08.lsa.”

I.4 Click the “Save As” button and save the Assay in the same folder with a different file name that incorporates the creation date of the new Assay.

I.5 On the “Assay Parameters” tab, change the Assay Description to incorporate the reference date for the new set of standards.

I.6 On the “Count Conditions” tab, click the “Name” button.

- I.6.1 The Quench Standards library window will open. Click the “Add” button to create an entry for the new Quench Standards set.
- I.6.2 Enter the Name of the new Quench Standards, the Maximum keV (18.6) and the Activity of the new Quench standard set.
- I.6.3 On the “Count Corrections” tab, change the reference date to match the new standards.
- I.6.4 Click the **OK** button to save and exit the new Assay.

I.7 Load the Quench Standard Set into Cassette #40.

I.8 Associate the Protocol Flag with the new Quench Standards Assay, as described in Section 14.5.

I.9 Load the SNC/IPA Cassette and Cassette #40 with the Quench Standards into the instrument, as described in Section 14.6.

I.10 After all samples have been loaded into the instrument, close the lid of the instrument. Wait a minimum of one hour to allow the standards to dark-adapt before starting the run.

I.11 Check to be certain that the EPSON 880 printer (to the left of the system) is loaded with paper.

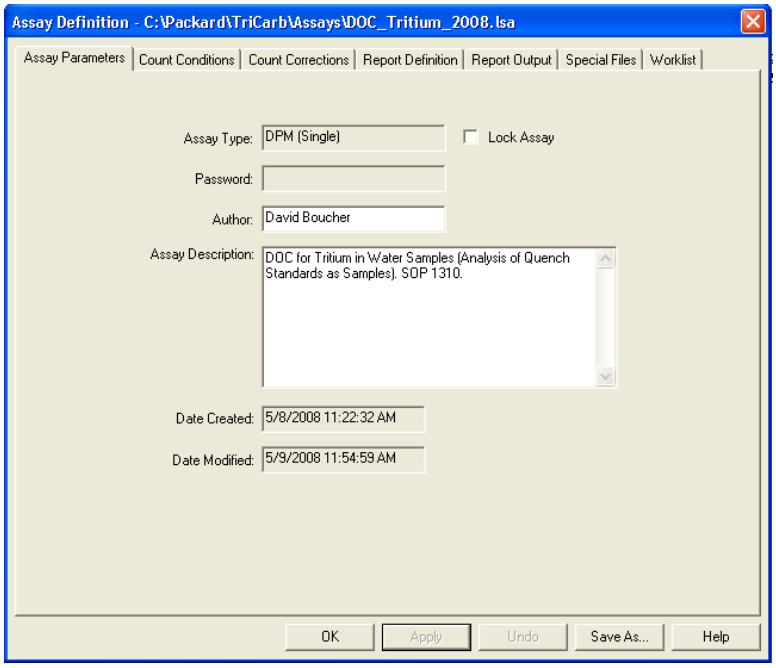
I.12 On the header of computer monitor, Press the Start button (green flag) to begin counting.

I.13 Remove the Cassette with the Quench Standards from the instrument and store it.

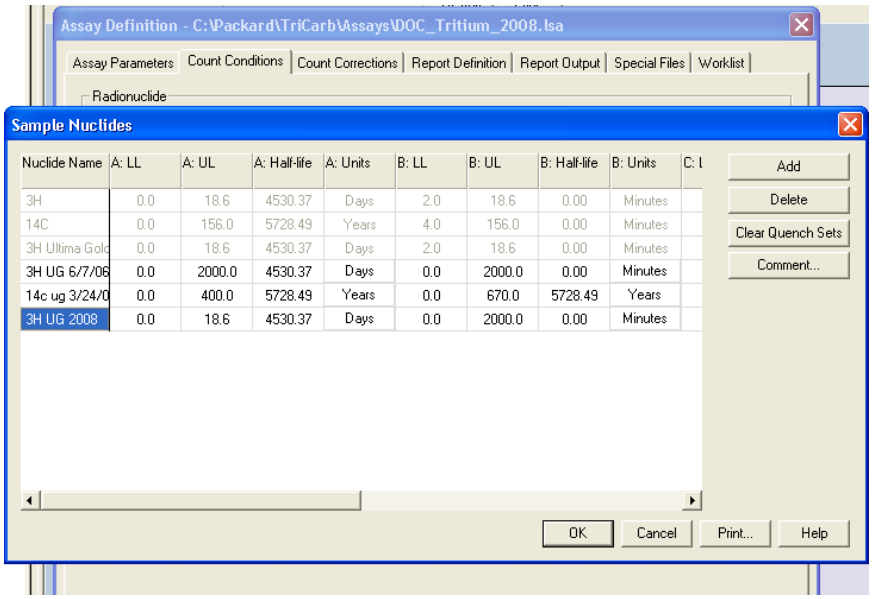
I.14 You will have to update the Sample Analysis and the DOC Assays to associate them with the new Quench Standard Set. If you do not do this, your Sample Analyses will ignore the new Quench Curve and use

the previously stored Quench Curve instead. Use the following procedure to update the Sample Analysis and the DOC Assays:

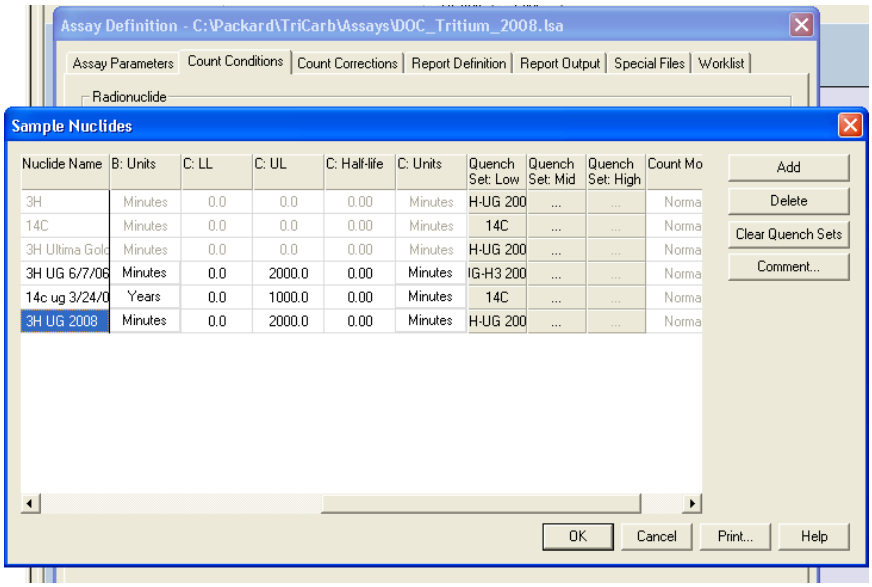
I.14.1 On the File menu, select “Open Assay.” Select the Assay that you wish to change.



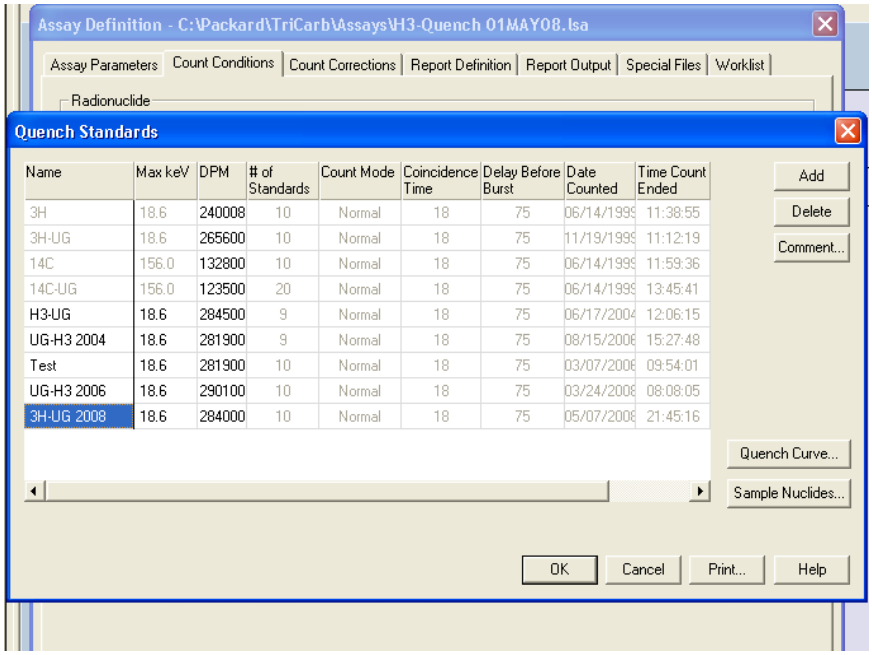
I.14.2 On the “Count Conditions” tab, click the “Name” button. The radionuclide Library window will be displayed.



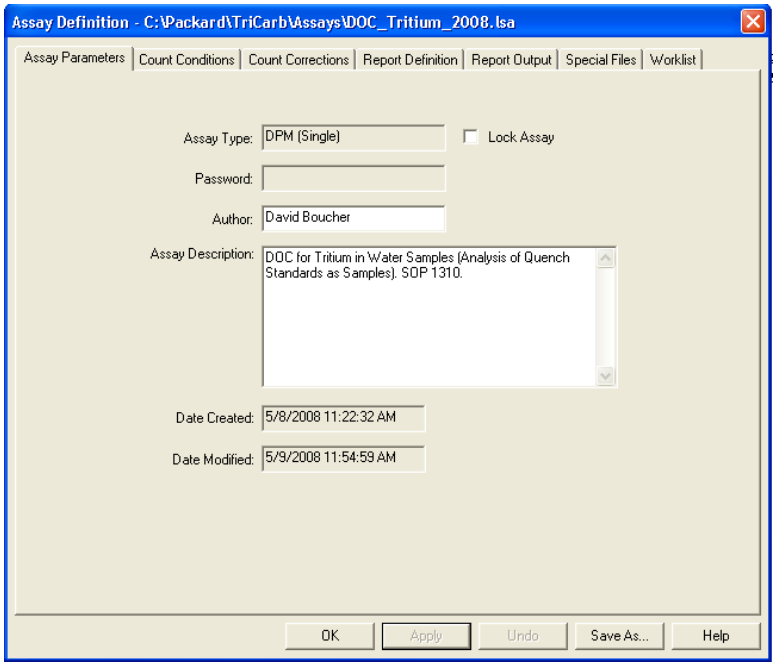
I.14.3 Slide to the right in the window to show the column labeled "Quench Set Low."



I.14.4 Click on the cell at the intersection of the "Quench Set Low" column with the row with your selected Radionuclide Name. The Quench Standards window will appear.



I.14.5 Select the new Quench Standard set from the list by clicking on it to highlight the selection, then click **OK**. The Assay Definition window will reappear.



I.14.6 Click “Save As” to save the modified assay with a new name. After the new assay has been saved, click **OK** to exit.

Appendix J. Demonstration of Capability Form

Demonstration of Capability						
Analyst:	David Boucher			Date:		
Reviewed by:				Date:		
METHOD : Liquid Scintillation - Tritium Analysis						
LDEQ SOP # 1310_r07						
INSTRUMENT: Packard (PerkinElmer) TriCarb 2900TR Scintillation Counter						
Standards Set	Std. No.	DATE	Reference Value (DPM)	VALUE FOUND (DPM)	% Recovery	Result
UG 3H 2008	A	06/12/08	284000	284161	100.05%	Pass
UG 3H 2008	B	06/12/08	284000	284636	100.22%	Pass
UG 3H 2008	C	06/12/08	284000	283401	99.79%	Pass
UG 3H 2008	D	06/12/08	284000	283781	99.82%	Pass
UG 3H 2008	E	06/12/08	284000	284011	100.00%	Pass
UG 3H 2008	F	06/12/08	284000	285096	100.39%	Pass
UG 3H 2008	G	06/12/08	284000	285016	100.36%	Pass
UG 3H 2008	H	06/12/08	284000	283007	99.65%	Pass
UG 3H 2008	I	06/12/08	284000	285824	100.64%	Pass
UG 3H 2008	J	06/12/08	284000	286147	100.76%	Pass
UG 3H 2008	A		284000		0.00%	
UG 3H 2008	B	01/00/00	284000		0.00%	
UG 3H 2008	C	01/00/00	284000		0.00%	
UG 3H 2008	D	01/00/00	284000		0.00%	
UG 3H 2008	E	01/00/00	284000		0.00%	
UG 3H 2008	F	01/00/00	284000		0.00%	
UG 3H 2008	G	01/00/00	284000		0.00%	
UG 3H 2008	H	01/00/00	284000		0.00%	
UG 3H 2008	I	01/00/00	284000		0.00%	
UG 3H 2008	J	01/00/00	284000	/	0.00%	
UG 3H 2008	A		284000		0.00%	
UG 3H 2008	B	01/00/00	284000		0.00%	
UG 3H 2008	C	01/00/00	284000		0.00%	
UG 3H 2008	D	01/00/00	284000		0.00%	
UG 3H 2008	E	01/00/00	284000	/	0.00%	
UG 3H 2008	F	01/00/00	284000		0.00%	
UG 3H 2008	G	01/00/00	284000		0.00%	
UG 3H 2008	H	01/00/00	284000		0.00%	
UG 3H 2008	I	01/00/00	284000		0.00%	
UG 3H 2008	J	01/00/00	284000	/	0.00%	
UG 3H 2008	A		284000		0.00%	
UG 3H 2008	B	01/00/00	284000		0.00%	
UG 3H 2008	C	01/00/00	284000		0.00%	
UG 3H 2008	D	01/00/00	284000		0.00%	
UG 3H 2008	E	01/00/00	284000		0.00%	
UG 3H 2008	F	01/00/00	284000		0.00%	
UG 3H 2008	G	01/00/00	284000		0.00%	
UG 3H 2008	H	01/00/00	284000		0.00%	
UG 3H 2008	I	01/00/00	284000		0.00%	
UG 3H 2008	J	01/00/00	284000		0.00%	