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Liquid Scintillation

TR-LSC APPLICATION NOTE

## TR-LSC™ Application Note

### Time Resolved Liquid Scintillation Counting: (TR-LSC™) for Environmental $^3\text{H}$ Analysis

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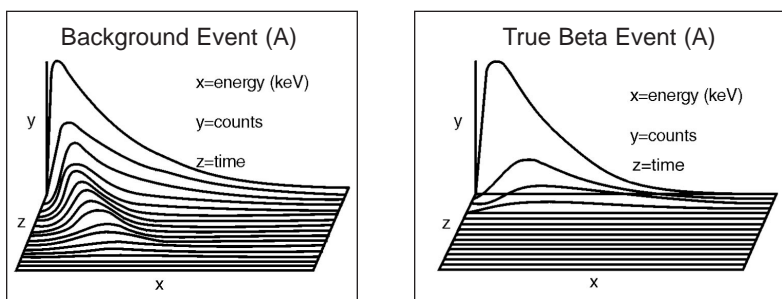
- TR-LSC is a unique background discrimination feature found only on Tri-Carb® LSCs
- Extremely low backgrounds are achievable with standard glass vials under normal laboratory conditions
- TR-LSC is effective for most environmental beta counting applications, especially  $^3\text{H}$

## Introduction

Environmental analysis of many naturally occurring and man-made, beta emitting radionuclides, including  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{90}\text{Sr}/^{90}\text{Y}$ ,  $^{241}\text{Pu}$ , and  $^{234}\text{Th}$  is a significant analytical challenge due to the extremely low activity levels encountered. Under these circumstances, reducing instrument background is essential for improving analytical sensitivity.

Time-Resolved Liquid Scintillation Counting (TR-LSC), a feature available on PerkinElmer Tri-Carb Liquid Scintillation Analyzers, is a form of pulse shape analysis that has proven to be an effective tool in such analyses. TR-LSC increases counting sensitivity by reducing the non-quenchable component of instrument background that is caused by the interaction of high energy cosmic radiation with the sample vial and photomultiplier (PMT) glass face. Other non-quenchable background interference including PMT after-pulsing, and natural radiation in the PMT and vial construction materials are also reduced by TR-LSC. Approximately 68% of the observed total background is non-quenchable and therefore can be greatly reduced by this technique<sup>1</sup>.

Non-quenchable background events can be distinguished from true beta events since they are characterized by a series of low amplitude after-pulses that follow the initial prompt pulse<sup>1</sup>. True scintillation pulses have fewer after-pulses associated with them. Figure 1 shows a 3-D graphical representation of a background pulse (A) and  $^3\text{H}$  beta pulse (B). Note that the background pulse has more prominent after-pulse characteristics that extend over time (z axis). TR-LSC is designed to reduce background by evaluating each event for the presence of these after-pulses, and when a series of after-pulses is detected, TR-LSC characterizes the event as background and rejects it. The efficacy of this rejection can be increased when a bismuth germanium oxide (BGO) detector guard is used in conjunction with TR-LSC electronics. The BGO guard completely surrounds the sample vial and acts as a cosmic guard to further increase after-pulse rejection and thus reduce background.



**Figure 1.** Three Dimensional plots of pulse height spectra. The pulse index is the third dimension (z). TR-LSC uses the pulse index of each scintillation event as one criterion to reduce background

TR-LSC can be optimized for the latest generation of safer cocktail formulations and for higher energy beta emitters. This is easily accomplished through the instrument software by modifying the Delay Before Burst value in the Count Corrections parameters screen on recent Tri-Carb models or the Special Conditions screen in older models. The Delay Before Burst feature functions by implementing a time delay before implementing after-pulse discrimination<sup>2</sup>. This is necessary because: 1. Scintillation cocktails that contain solvents based on di-isopropyl naphthalene (DIN) and 1-phenylxylylethane (PXE) have long scintillation decay constants, *i.e.* produce light pulses of longer duration, and 2. Higher energy beta emitters produce longer duration pulses.

These phenomena must be considered in order to minimize the misclassification of beta and background events since the longer duration pulses arising from either of these conditions mimic background pulses and may be eliminated by TR-LSC. By increasing the time delay before after-pulse discrimination begins, counting efficiency losses are minimized and sensitivity is increased.

$^3\text{H}$  is the most common radionuclide found in the environment that is assayed by liquid scintillation methods. An overview of TR-LSC performance data and methods are presented for environmental analysis of  $^3\text{H}$ .

## $^3\text{H}$ analysis

### Sample Preparation

$^3\text{H}$  is produced naturally in the upper atmosphere and as a result of nuclear power production, therefore,  $^3\text{H}$  monitoring is performed for a variety of hydrological and environmental applications. In addition,  $^3\text{H}$  measurements are common during cleanup of sites formerly used for the production of nuclear weapons. The most common sample matrix is water and generally, three general methods of sample preparation are employed for its analysis. These are direct addition, electrolytic enrichment, and benzene synthesis<sup>3,4</sup>. Of the three methods, direct addition of water to an appropriate cocktail is the most appealing since it is less labor intensive. However, even though it is often possible to add water directly to a scintillation cocktail, sample purification or extraction is often required since low-level  $^3\text{H}$  counting requires reasonably clean samples, free from interference. Various cleanup techniques are used depending on the source of the water, *i.e.* river water, well water, ground water, etc while extraction is necessary when the source is biological material or soil<sup>1</sup>.

### Instrument Considerations

Performing low-level  $^3\text{H}$ -water measurements on a PerkinElmer Tri-Carb LSC is a relatively simple process. Once an acceptable sample is obtained, it is mixed with an aqueous-accepting cocktail. The safer PerkinElmer cocktails, Ultima Gold LLT and Ultima Gold XR, are recommended since they have a high capacity for a variety of different types of water samples and can accept mineral acids. If a classical cocktail is desired, Insta-Gel Plus is recommended. All of these cocktails are designed to work well with TR-LSC.

Typically, an optimum counting region is chosen for environmental measurements. An optimum counting region will provide the highest sensitivity or Figure of Merit (FOM) as determined by either the Efficiency<sup>2</sup>/Background ( $E^2/B$ ) or  $E^2/V^2/B$  value where the V factor represents the volume of sample. The optimum FOM can be determined manually by repeat counting of both a representative background sample and a standard in various regions of interest or by using the region optimization feature found on Tri-Carb LSCs. This feature will automatically find an optimum region for a background /sample pair that can be used in a counting protocol. As an alternative, sample spectra can be electronically stored and analyzed with commercial software or Packard spectral analysis software, SpectraWorks™ to determine the optimum region.

The use of TR-LSC electronic background reduction is controlled by software. The user decides whether to use it or not by a Yes or No response. Depending on the model, various degrees of background discrimination exist. Several TR-LSC count modes have been defined for PerkinElmer Tri-Carb LSCs.

These count modes differ in the degree of electronic background discrimination used. Normal Count Mode (NCM) has the least amount of discrimination. High Sensitivity (Low Activity) Count Mode (HSCM) has an intermediate amount and the Low Level Count Mode (LLCM) has the most amount. NCM is standard on all Tri-Carb LSCs while HSCM or LLCM are available depending on the model.

# Results

## <sup>3</sup>H Performance

The following results are representative of the type of performance that can be obtained for <sup>3</sup>H analysis in InstaGel and Ultima Gold cocktails. The results in Table 1 were compiled from studies using Tri-Carb LSCs operated in several locations. It should be noted that results will vary depending on geographical location, altitude and other local conditions.

The “None” condition was performed by removing TR-LSC electronics from an instrument and is similar to the type of performance from a conventional LSC. Even NCM shows some background reduction over conventional LSC since some TR-LSC discrimination is applied to NCM.

Table 1 contains historical <sup>3</sup>H results acquired with Ultima Gold LLT cocktail in a normal laboratory. Note that these results were obtained with a non-optimized region of interest. Backgrounds count rates were from analysis of “dead water” acquired for 300 minutes. Even in a non-optimized region (0-18.6 keV), sensitivity can be increased nearly 5 times over no TR-LSC.

| <sup>3</sup> H performance, 0-18.6 keV region, 8mL H <sub>2</sub> O and 12mL Ultima Gold LLT in a 20 mL glass vial |                               |                  |                   |
|--|-------------------------------|------------------|-------------------|
| TR-LSC count mode  | <sup>3</sup> H Efficiency (%) | Background (CPM) | E <sup>2</sup> /B |
| None   | 34.2                          | 22.3             | 52                |
| NCM  | 33.8                          | 17.8             | 64                |
| HSCM   | 33.9                          | 12.8             | 90                |
| LLCM   | 29.4                          | 6.2              | 139               |
| LLCM w/BGO detector guard  | 26.7                          | 2.8              | 255               |

**Table 1.** TR-LSC Performance for <sup>3</sup>H with Ultima Gold LLT cocktail

The results in Table 2 have been generated in a normal laboratory at the Scottish Universities Research and Reactor Centre in East Kilbride, Scotland. The data represent performance using varying water:cocktail ratios in an optimum region of interest. FOM is represented by E<sup>2</sup>/V<sup>2</sup>/B to normalize for different sample volumes (V). Background count rates were acquired using 500 minutes count time.

| Tritium performance, optimum region in a 20 mL glass vial; Ultima Gold LLT cocktail |                       |                               |                  |                                  |                                     |
|---|-----------------------|-------------------------------|------------------|----------------------------------|-------------------------------------|
| TR-LSC count mode   | Water:cocktail volume | <sup>3</sup> H Efficiency (%) | Background (CPM) | E <sup>2</sup> V <sup>2</sup> /B | Detection Limit (Bq/L) <sup>1</sup> |
| LLCM  | 8:12                  | 28.0                          | 3.4              | 14758                            | 2.91                                |
| LLCM w/BGO detector guard   | 8:12                  | 26.9                          | 1.9              | 24374                            | 2.25                                |
| LLCM  | 10:10                 | 25.0                          | 3.4              | 18382                            | 2.57                                |
| LLCM w/BGO detector guard   | 10:10                 | 23.2                          | 1.7              | 31661                            | 1.99                                |

**Table 2.** Summary TR-LSC performance for <sup>3</sup>H in a normal laboratory at two water:cocktail ratios

The results in Table 3 represent <sup>3</sup>H performance in plastic vials; same conditions as in Table 2.

| <sup>3</sup> H performance, optimum region in a 20 mL plastic vial, Ultima Gold LLT cocktail |                       |                               |                  |                                  |                                     |
|--|-----------------------|-------------------------------|------------------|----------------------------------|-------------------------------------|
| TR-LSC count mode  | Water:cocktail volume | <sup>3</sup> H Efficiency (%) | Background (CPM) | E <sup>2</sup> V <sup>2</sup> /B | Detection Limit (Bq/L) <sup>1</sup> |
| LLCM   | 8:12                  | 26.8                          | 2.9              | 15850                            | 2.79                                |
| LLCM w/BGO detector guard  | 8:12                  | 24.6                          | 1.2              | 32275                            | 1.93                                |
| LLCM   | 10:10                 | 22.8                          | 2.5              | 20793                            | 2.46                                |
| LLCM w/BGO detector guard  | 10:10                 | 21.2                          | 1.1              | 40858                            | 1.77                                |

**Table 3.** TR-LSC performance with plastic vials at two different water:cocktail ratios

<sup>1</sup> A priori calculated detection limit

Additional results from Ontario Power, Whitby, Ontario are shown in Table 4. Performance data in small and large plastic vials are shown for LLCM with the BGO detector guard. The background sample is dead water obtained from a deep well. The  $^3\text{H}$  counting region was 0-4 keV.

| TR-LSC mode               | Water: cocktail volume (mL) | $^3\text{H}$ Efficiency (%) | Background (CPM) | Count time (minutes) | Target Detection Limit (Bq/L) <sup>1</sup> |
|---------------------------|-----------------------------|-----------------------------|------------------|----------------------|--|
| LLCM w/BGO detector guard | 3:3                         | 19.50                       | 0.64             | 470                  | 5.0  |
| LLCM w/BGO detector guard | 10:10                       | 19.89                       | 0.87             | 60                   | 5.0  |

**Table 4.** LLCM w/BGO detector guard performance – large vs. small samples

The experimental results in Table 4 show that low detection limits are possible even with small volume samples. Thus, it is possible to conserve sample volume and reduce cocktail consumption in many applications and still meet a target detection limit with TR-LSC.

## Discussion and Conclusions

TR-LSC background discrimination is unique in that superior performance is achieved without the need for additional instrument or room shielding, special counting vials, or laborious optimization procedure. Optimum background reduction does not require extensive instrument set up or education to achieve maximum performance for most applications under almost any situation.

For environmental  $^3\text{H}$  analysis, TR-LSC background reduction offers several benefits over other LSCs:

- Very low detection limits can be achieved using direct water samples
- The desired detection limit can be reached with statistical accuracy in a relatively short counting time
- Small volume samples can be used because of high counting sensitivity

Although performance can vary with geography as well as other factors, the results presented are representative of typical performance in a normal laboratory.

## References

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<sup>1</sup> A priori calculated detection limit





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