

# IMPLEMENTING METHODS FOR $^3\text{H}$ AND $^{14}\text{C}$ MEASUREMENT IN ORGANIC WASTE SAMPLES

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## ABSTRACT

The paper presents the results obtained from the implementation of the  $^3\text{H}$  and  $^{14}\text{C}$  measurement methods on the TriCarb 3110TR using Normal Count Mode (typical count rate > 500CPM). Two types of organic solutions were used:  $^3\text{H}$  in toluene, with activity of 44886.51dpm/ml and  $^{14}\text{C}$  SPEC-CHEC solution with activity of 929000dpm/ml. To determinate counting efficiency and calculate DPM (Disintegrations Per Minute), quench curves must be performed for each radionuclide. These curves are obtained using a quench standards set, each containing the same amount of nuclide with varying amounts of quenching agent. The high values obtained by both methods for efficiency recovery of  $^3\text{H}$  and  $^{14}\text{C}$  indicate very good measurement efficiency (for tritium in the range 0-18.6keV, the minimum acceptable efficiency is 60% while for carbon-14 in the range 0-156keV, the minimum acceptable efficiency is 95%).

**Key words: waste, liquid scintillation, quench curve**

## Introduction

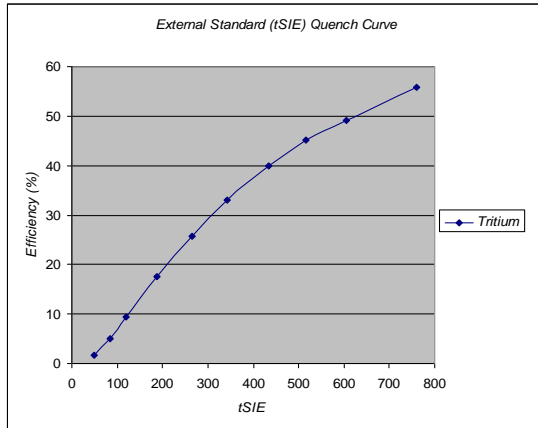
Liquid Scintillation Counting (LSC) is the most sensitive and widely used technique for the detection and quantification of alpha and beta radioactivity. Probably, one of the most important steps in the scintillation counting process is methods implementation for radionuclides measurement in waste sample. The sample must be prepared so that the liquid scintillation analyzer is quantitating a homogeneous mixture of sample and scintillation solution. The scintillation solution (the cocktail) consists of a mixture of a solvent and a solute (scintillator). The counting efficiency of the solvent-solute system can be affected by many different factors which may reduce the detection efficiency [1]. Chemical quench that occurs during the transfer of energy from the solvent to the scintillator and the color quench that is the attenuation of light photons in the solution represent the most important factors affecting the measurement efficiency [2].

Quench curves must be performed for each radionuclide in order to determinate the counting efficiency and calculate DPM. A quench standard curve is a series of standards, each containing the same amount of nuclide with varying amounts of quenching agent. A quench curve uses the relationship between counting efficiency and Quench Indicating Parameters (QIP) to correct the measured Counts Per Minute (CPM) to Disintegrations Per Minute (DPM) [3].

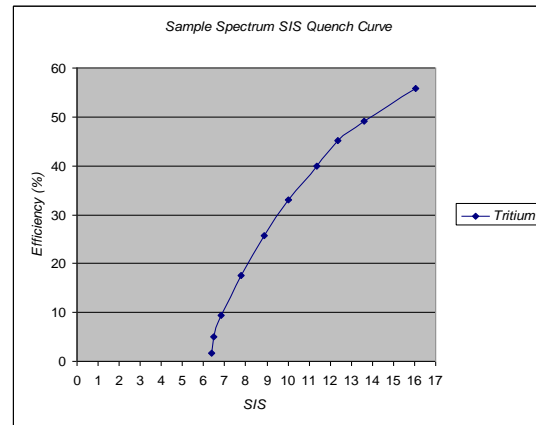
When a quench curve is performed, the DPM value in each standard is known. The counting efficiency is calculated using the following relationship:

$$\text{Efficiency} = \frac{\text{CountsPerMinute}}{\text{DisintegrationPerMinute}} \quad (1)$$

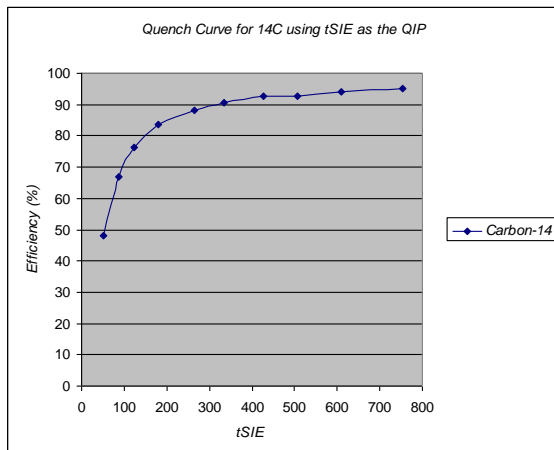
At the same time, the QIP is measured for each standard. Examples of quench curves using transformed Spectral Index of the External Standard (tSIE) and Spectral Index of the Sample (SIS) as QIP are presented in **Figures 1 and 2** for  $^3\text{H}$  and in **Figure 3 and 4** for  $^{14}\text{C}$ .



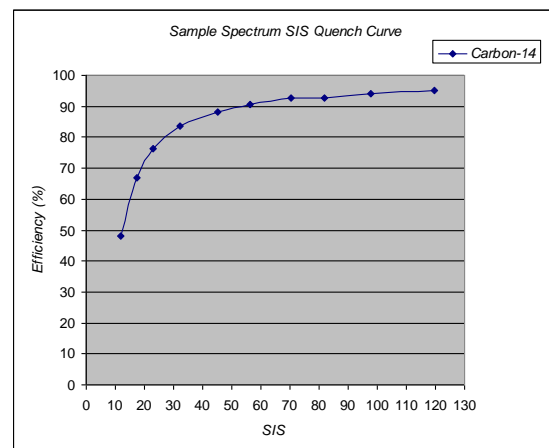
**Figure 1** Quench Curve for  $^3\text{H}$  using tSIE as the QIP



**Figure 2** Quench Curve for  $^3\text{H}$  using SIS as the QIP



**Figure 3** Quench Curve for  $^{14}\text{C}$  using tSIE as the QIP



**Figure 4** Quench Curve for  $^{14}\text{C}$  using SIS as the QIP

Two methods can be used to assess the radionuclides activity in different liquid samples: tSIE and SIS. Using an external Barium-133 standard source, tSIS method assigns a numeric value to the quench associated with a sample. This determination is independent of the quantity of radioactivity in the sample and its counting rate [4]. The source is positioned under the sample vial, causing a Compton spectrum to

be produced in the cocktail solution. The tSIE value is determined using a mathematical transformation of this spectrum [3].

The SIS method is determined from the spectral shape of the sample and is based on actual sample counts [4].

## Experimental and results

Experiments with simulated organic samples labelled with known  $^3\text{H}$  and  $^{14}\text{C}$  quantities were performed in order to implement two measurement methods (tSIE and SIS) on the TRICARB 3110TR spectrometer. A prerequisite condition to get optimal measurement conditions by LSC is to obtain a stable and homogeneous solution between the waste sample and the scintillation cocktail. To attain it the waste sample has to be mixed with adequate scintillation cocktail. For organic samples the adequate scintillation cocktail are: UltimaGoldF, Opti-Fluor O and Insta-Fluor Plus. The mixture is usually performed directly in the measurement vials (glass or plastic) that are immediately shaken and kept away from light prior their measurement.

To simulate an organic waste sample a solution of  $^3\text{H}$  in toluene, with activity of 44886.51dpm/ml was used. The liquid scintillation cocktail used for tritium measurement in the organic waste sample was UltimaGoldF. Three samples were prepared with the composition presented in **Table 1**.

These samples were measured the TriCarb 3110TR spectrometer using both the tSIE method and the SIS one. To assess the performance of the two methods, the efficiency recovery and standard deviation were computed. The efficiency recovery is defined as the ratio between the measured value of the contaminant concentration and the reference one.

The values obtained for the efficiency recovery and standard deviation for  $^3\text{H}$  activity using tSIE method are presented in **Table 2** while those obtained by the SIS method are presented in **Table 3**.

**Table 1** Samples used in experiments

Nr. sample	Liquid scintillation cocktail UltimaGoldF [ml]	Solution of $^3\text{H}$ [ml]
1	17	3
2	16	4
3	15	5

**Table 2** Values obtained for  $^3\text{H}$  using tSIE method

Sample no.	CPM [cpm]	$^3\text{H}$ Theoretical Activity [dpm]	$^3\text{H}$ Measurement Activity [dpm]	Efficiency recovery [%]	Standard Deviation [%]
1	81876	134659.53	134219.0	99.6	0.32
2	107639	179546.04	178688.1	99.5	0.48
3	131109	224432.55	220302.0	98.1	1.8

**Table 3** Values obtained for  $^3\text{H}$  using SIS method

Sample no.	CPM [cpm]	$^3\text{H}$ Theoretical Activity [dpm]	$^3\text{H}$ Measurement Activity [dpm]	Efficiency recovery [%]	Standard Deviation [%]
1	81876	134659.53	135326	99.9	0.49
2	107639	179546.04	182972	99.9	1.87
3	131109	224432.55	229759	98.9	2.32

Similar experiments were also performed for  $^{14}\text{C}$  measurement in organic waste samples. To simulate organic waste samples  $^{14}\text{C}$  SPEC-CHEC solution (from PerkinElmer Company) with activity of 929000dpm/ml was used and dilutions were obtained using toluene.

All samples were prepared and measured in 20 ml glass vials and UltimaGoldF liquid scintillation cocktail was used.

In **Table 4**, the composition of the  $^{14}\text{C}$  labeled samples is presented. The efficiency recovery and the corresponding standard deviation obtained for  $^{14}\text{C}$  activity measurement are shown in **Table 5** for tSIE method and in **Table 6** for SIS method.

**Table 4** Samples used in experiments

Sample no.	Liquid scintillation cocktail UltimaGoldF [ml]	Solution of $^{14}\text{C}$ [ml]	Solution of toluene [ml]
1	15	0.1	4.9
2	15	0.2	4.8
3	15	0.3	4.7
4	15	0.4	4.6
5	15	0.5	4.5

**Table 5** Values obtained for  $^{14}\text{C}$  using tSIE method

Sample no.	CPM [cpm]	$^{14}\text{C}$ Theoretical Activity [dpm]	$^{14}\text{C}$ Measurement Activity [dpm]	Efficiency recovery [%]	Standard Deviation [%]
1	87099	92900	90915	97.8	2.13
2	175251	185800	182914	98.4	1.55
3	266033	278700	277159	99.4	0.55
4	347447	371600	362366	97.5	2.48
5	432516	464500	450883	97.0	2.93

**Table 6** Values obtained for  $^{14}\text{C}$  using SIS method

Sample no.	CPM [cpm]	$^{14}\text{C}$ Theoretical Activity [dpm]	$^{14}\text{C}$ Measurement Activity [dpm]	Efficiency recovery [%]	Standard Deviation [%]
1	87244	92900	91376	98.3	1.64
2	175265	185800	183377	98.6	1.30
3	264225	278700	275952	99.0	0.98
4	343062	371600	358586	96.4	3.5
5	431342	464500	450396	97.0	3.03

## Conclusions

The experiments performed to implement the two methods (tSIE and SIS) on the TriCarb 3110TR spectrometer show that the both methods may be used for accurate measurement of  $^3\text{H}$  and  $^{14}\text{C}$  activities in the organic waste samples.

The experimental results obtained for  $^3\text{H}$  measurement by SIS method are slightly higher than the reference values while those obtained by tSIE method are a little bit lower than the reference ones, but for both measurement methods the standard deviation values range between 0.49% and 2.32% that are acceptable for LSC measurement.

For the experiments performed for  $^{14}\text{C}$  measurement the experimental values were slightly higher than the reference ones both methods (tSIE and SIS). The standard deviation computed for experimental values

were a little bit lower for the tSIE method (between 0.55 and 2.93) than those obtained by SIS method (0.98 – 3.5) but all of them are acceptable for tritium and  $^{14}\text{C}$  measurement by LSC. The high values obtained by both methods for efficiency recovery of  $^3\text{H}$  and  $^{14}\text{C}$  indicate very good measurement efficiency (for tritium in the range 0-18.6keV, the minimum acceptable efficiency is 60% while for carbon-14 in the range 0-156keV, the minimum acceptable efficiency is 95%).

## References

- [1] Liquid Scintillation Analysis – Science and Technology, Publication No.169-3052 Packard Instrument Company, USA 1989
- [2] PerkinElmer and Analytical Sciences, QuantaSmart™ for the Tri-Carb® Liquid Scintillation Analyzer, USA, 2009
- [3] Jock Thomson, Use and Preparation of Quench Curves in Liquid Scintillation Counting, Packard BioScience Company, Meriden, Connecticut, 2001
- [4] Laboratory Manual for Liquid Scintillation Counting, Publication No. 169-4019 Packard Instrument Company, USA, 1988