

Characterization of Low-Level Tritium Measurement Performance of the HIDEX 300SLL Liquid Scintillation Counter

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***Keywords:** tritium, liquid scintillation counting, HIDEX 300SLL, measurement optimization, uncertainty evaluation

1. Introduction

Tritium (^3H) is a low-energy beta emitter with a half-life of 12.32 y and an endpoint energy of 18.6 keV.[1] Because most environmental tritium occurs as tritiated water, reliable low-level measurement is required for radiological environmental surveillance. Liquid scintillation counting (LSC) is widely used for this purpose, but analytical sensitivity is strongly affected by counting efficiency, background count rate, quenching, and instrument-specific settings [2,3].

For low-level LSC, the figure of merit (FOM) is defined as E^2/B , where E is counting efficiency and B is the background count rate. A high FOM generally corresponds to a low minimum detectable activity (MDA) [3,4]. The coincidence time (CT) and region of interest (ROI) control the balance between signal acceptance and background rejection. For the HIDEX 300SLL, the manufacturer provides a predefined tritium ROI of 5–350 channels, whereas no specific CT value is prescribed. Therefore, the manufacturer-provided ROI cannot necessarily be assumed to provide optimal performance for low-level tritium measurement, and instrument-specific optimization of CT and ROI is required. This study systematically optimized CT and ROI based on the FOM and quantitatively evaluated the performance improvement of the optimized ROI relative to the manufacturer-provided tritium ROI at the same CT. The selected conditions were subsequently validated using certified reference material (CRM) at environmental tritium levels. The target was reliable quantification at or below 5 Bq L⁻¹, satisfying the analytical sensitivity required for environmental monitoring [6].

2. Materials and Methods

2.1. Sample preparation and measurement system

A HIDEX 300SLL liquid scintillation counter and Ultima Gold LLT cocktail were used. A KRISS tritium CRM (205-01-201; certified activity, 2.11 kBq g⁻¹ at the reference date) was gravimetrically diluted to prepare eight verification levels: 2.5, 3.5, 5, 10, 15, 20, 30, and 50 Bq L⁻¹. Each 20-mL polyethylene vial contained 8 g of aqueous sample and 12 g of cocktail. A commercial tritium quench standard set was used to establish the efficiency–quench relationship.

2.2. Optimization and validation procedure

CT was varied from 10 to 28 ns at 1-ns intervals. ROI lower and upper boundaries were systematically combined within channels 0–350, and the FOM was calculated for each condition. The final condition was selected from the region providing high FOM and stable response. Each verification sample was then counted for 60 min in ten replicate measurements. Activity was calculated from net count rate, counting efficiency, and sample volume; MDA was determined from background counts, efficiency, counting time, and sample quantity [4].

Measurement uncertainty was evaluated in accordance with the Guide to the Expression of Uncertainty in Measurement [5]. Type A standard uncertainty was determined separately from the ten replicate measurements at each concentration. Type B components included the balance, CRM certificate, and quench-standard certificate. The combined standard uncertainty was expanded using $k = 2$. Validation was accepted when the absolute deviation between the measured and reference values was smaller than the corresponding expanded uncertainty.

$$FOM = E^2/B \quad (1)$$

$$MDA = (2.71 + 4.65\sqrt{N_B})/(E \cdot T \cdot W \cdot Y) \quad (2)$$

3. Results and Discussion

3.1. Optimization of CT and ROI

The FOM map showed a broad optimum at CT values of 24–26 ns and ROI upper boundaries of approximately 170–190 channels (Fig. 1). Shorter CT settings reduced the acceptance of delayed scintillation components, whereas longer CT settings increased accidental coincidences and background. Similarly, an excessively wide ROI admitted high-channel background events, while a narrow ROI excluded part of the tritium beta spectrum. CT 26 ns and ROI 20–180 channels were selected as the final conditions because they provided a high FOM with a stable response near the optimum. At CT 26 ns, the FOM increased from 121.60 for the manufacturer-provided tritium ROI of 5–350 channels to 284.16 for ROI 20–180 channels, corresponding to a 2.34-fold increase (133.7%). This improvement was primarily attributable to the reduction in the background count rate from 14.77 to 4.82 cpm (67.4%), which more than compensated for the decrease in counting efficiency from 42.37% to 37.00%.

The lower ROI boundary also affected performance. Inclusion of the lowest channels increased contributions from electronic and photomultiplier-tube noise, whereas raising the boundary excessively reduced the low-energy portion of the ^3H spectrum. A lower boundary of 20 channels provided an effective compromise between background suppression and retention of tritium counting efficiency. The broad maximum, rather than a single isolated peak, is advantageous for routine analysis because small variations in background or quench do not immediately move the system away from the optimal operating region.

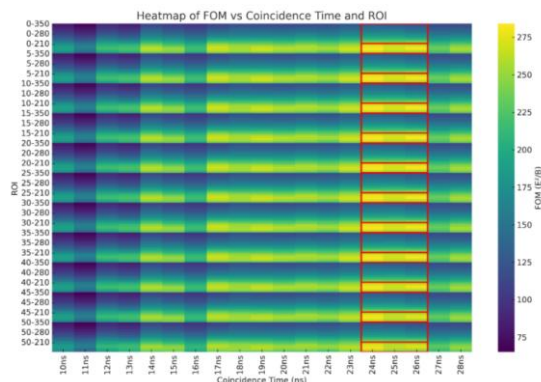


Fig. 1. FOM variation as a function of coincidence time and ROI.

3.2. Validation at environmental activity levels

Under the optimized conditions, the 2.5 Bq L^{-1} sample yielded a mean value of 2.59 Bq L^{-1} . Across all eight levels, relative standard uncertainties from repeatability were 0.77–2.26%, absolute deviations were no greater than 4.7%, and expanded uncertainties were 6.03–7.38% (Table I). The criterion $|\text{deviation}| < U$ was satisfied at every concentration, demonstrating agreement with the CRM-derived reference values at an approximately 95% confidence level. No concentration-dependent bias was evident over $2.5\text{--}50 \text{ Bq L}^{-1}$.

The largest Type A component occurred at 2.5 Bq L^{-1} , as expected from counting statistics at the lowest activity, whereas repeatability improved at higher activities. Among the common Type B components, the CRM certificate (2.5%) and quench-standard certificate (1.5%) dominated; the relative contribution from weighing was negligible (0.00049%). Consequently, the combined standard uncertainty remained in a narrow range of approximately 3.01–3.69% across the validation series. These results indicate that further reduction in overall measurement uncertainty would require consideration of both counting statistics and the uncertainties associated with the reference and quench standards.

Table I. Summary of validation results at CT 26 ns and ROI 20–180 channels.

Validation parameter	Result
Reference activity range	$2.5\text{--}50 \text{ Bq L}^{-1}$
Replicate measurements	10 per level
Relative standard uncertainty	0.77–2.26%
Absolute deviation	0.2–4.7%
Expanded uncertainty, $k=2$	6.03–7.38%

All eight concentration levels satisfied $|\text{deviation}| < \text{expanded uncertainty}$.

The optimized method achieved an MDA at approximately the 2.5 Bq L^{-1} level with a 60-min count, thereby satisfying the 5 Bq L^{-1} environmental-monitoring requirement. These results indicate that instrument-specific adjustment of CT and ROI can compensate for elevated background and substantially improve low-level tritium performance without changing the detector hardware.

3.3. Analytical applicability and limitations

The optimized conditions provide a practical basis for routine environmental monitoring because they use the existing detector and standard 20-mL vial geometry without additional hardware. The method maintained acceptable accuracy below the 5 Bq L^{-1} monitoring criterion and therefore provides sufficient sensitivity for screening and quantitative assessment of environmental water samples. Because MDA decreases with counting time and background reduction, longer counts or additional passive shielding could provide further improvement when lower detection levels are required.

This validation was performed with CRM-derived aqueous standards and a fixed sample-to-cocktail ratio. Actual environmental samples may show different chemical compositions and quench behavior; therefore, routine application should include quench monitoring, background control, blank measurement, and periodic verification with reference materials. Future work should also evaluate long-term background stability and spectrum-based analysis, including spectral deconvolution, to use the full pulse-height distribution without relying solely on fixed ROI boundaries.

4. Conclusion

The HIDEX 300SLL counting conditions were optimized by evaluating FOM over systematic combinations of CT and ROI. The optimum region was CT 24–26 ns with ROI upper boundaries near 170–190 channels, and CT 26 ns/ROI 20–180 channels was adopted for routine measurement. Compared with the manufacturer-provided tritium ROI of 5–350 channels at the same CT of 26 ns, the selected ROI of 20–180 channels increased the FOM from 121.60 to 284.16 (133.7%), demonstrating that improved background rejection substantially enhanced low-level counting performance despite a moderate reduction in counting efficiency. CRM-based validation from 2.5 to 50 Bq L^{-1} showed repeatability below 2.3%, absolute deviation below 4.7%, and acceptance within expanded

uncertainty for all samples. The method also met the environmental MDA requirement within 60 min. The optimized conditions therefore provide a technically valid basis for routine environmental tritium analysis using the HIDEX 300SLL.

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