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**BETA-PARTICLE EMITTING RADIONUCLIDES**

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**Purpose**

The purpose of this procedure is to describe the measurement and reporting procedures for beta- and alpha-particle emitting radionuclides by liquid scintillation (LS) counting.

**Scope**

The procedures covered here include: (i) preparation of LS counting sources; (ii) comparative LS calibrations of submitted radionuclide solutions against NIST standards; (iii) efficiency tracing of beta emitters using the CIEMAT/NIST methodology with tritium ( $^3\text{H}$ ) standards as the LS efficiency monitor; (iv)  $4\pi\alpha\beta$  LS calibrations for alpha emitters or alpha-beta decay chains; and (v) Triple-to-Double Coincidence Ratio (TDCR) Counting.

Scheduled calibration services for submitted samples of radionuclides that are to be measured by LS counting are offered as a complement to Standard Reference Materials. Customers must initially contact NIST Radioactivity Group as to whether the radionuclide or sample composition is suitable for measurement by LS counting.

Many radionuclides can be calibrated by more than one measurement service, e.g., by high-resolution gamma ray spectrometry, or re-entrant ionization chamber measurements, or LS counting. Calibrations by LS counting are very labor intensive. As a result, in general, if it is possible to perform the calibration by ion chamber measurements the calibration services 43010c (half-lives longer than 15 days) or 43020c (half-lives shorter than 15 days) will be used rather than an LS calibration service. Radionuclides most suitably calibrated by LS calibration services are pure beta emitters with no significant gamma ray emissions, e.g.,  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{32}\text{P}$ ,  $^{36}\text{Cl}$ ,  $^{63}\text{Ni}$ ,  $^{89}\text{Sr}$ ,  $^{90}\text{Y}$ ,  $^{204}\text{Tl}$ .

There are stringent limitations on the physical and chemical form and activity range of sources that can be accepted. To ensure that these specifications are understood, it is essential that there be good communication between the technical user and the technical contact at NIST.

Two general requirements apply to all sources submitted for calibration: (1) all shipments must conform to applicable Nuclear Regulatory Commission (NRC) and Department of Transportation (DOT) packaging and transport; and (2) **source descriptions, including approximate activity, must be provided in advance**. The NIST Radiation Safety Division (RSD) must approve the receipt of radioactive material, and sources may be refused if the necessary information is not available. Copies of regulations may be obtained from Operations Division, Office of Hazardous Materials, Department of Transportation, Washington, DC 20950. Postal regulations prohibit the mailing of radioactive materials that require a caution label under DOT regulations.

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All sources arriving at NIST are checked by the RSD for radiation level and source integrity. Sources should be shipped to the attention of the technical contact at NIST.

**Definitions***Liquid Scintillation (LS) Counting*

LS counting is the measurement of radioactivity of a sample which utilizes the technique of intimately mixing the active material with a liquid scintillator to form a LS cocktail and counting the resultant photon emissions. It allows more efficient counting because of the  $4\pi$  detection geometry.

*LS Cocktail*

An LS cocktail is the counting source used for LS measurements. It is typically contained in a 20 mL glass or plastic, screw-capped LS vial and consists of an organic-based commercial scintillation fluid (composed of an organic solvent with solutes of a scintillant and surfactant materials), a small aliquot of a radioactive solution (commonly a dilute acid with carrier ions, though occasionally an alkaline solution), and a carrier solution or water to increase the aqueous fraction in the cocktail.

*CIEMAT/NIST*

CIEMAT/NIST is an acronym for Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas and the National Institute of Standards and Technology, which are the respective national standards laboratory of Spain and the United States. Researchers from the two institutions collaboratively developed the efficiency tracing method which bears the names of those laboratories.

**Equipment and Supplies**

Certain commercial equipment, instruments, or materials are identified in this document to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

The LS counters available for the measurements are: (1) PerkinElmer Tri-Carb 4910TR; (2) Beckman Coulter LS6500; (3) Hitachi Aloka AccuFlex LSC-8000; (4) Perkin Elmer Quantulus Ultra Low-Level LS Spectrometer 1220; (5) NIST Triple-to-Double Coincidence Ratio Counter.

LS vials used to prepare the counting sources are screw-capped glass or plastic.

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Commercially prepared scintillation fluids used to prepare LS counting sources are environmentally safe. They include UltimaGold, UltimaGold AB, InstaGel, OptiFluor (PerkinElmer) amongst others.

Information about safe packaging, labeling and disposing of liquid scintillation vials and fluid can be found here. [https://www.ehs.washington.edu/system/files/resources/Liquid\\_Scintillation\\_Fluid\\_Focus\\_Sheet.pdf](https://www.ehs.washington.edu/system/files/resources/Liquid_Scintillation_Fluid_Focus_Sheet.pdf)

Chemical laboratory equipment needed to prepare the LS counting sources includes scintillation fluid dispensing bottles, aspirating pycnometers, an analytical balance (capacity 200g and readability down to 0.0001 g), a microbalance (capacity 40 g and readability down to 0.000001 g), and suitable carrier solutions to adjust aqueous fraction.

**Radiation Safety**

Radiation safety training and assessment services are provided by the RSD. The laboratories used for preparation of LS counting sources and the room used for LS counting are designated as a Radiation Facility.

LS sources to be measured must be checked and be found to be free of any external radioactive contamination before being loaded into the LS counters.

**Procedures***Preparation of LS Counting Sources*

In general, standardization measurements utilize more than one scintillation fluid, and typically a series of 6 or more counting sources are made for each scintillation fluid. For comparative measurements against a known standard the composition of the cocktails for both the sample solution and standard must be closely matched, and all sources shall be essentially identical in composition. For counting sources with variable chemical quenching as used for  $^3\text{H}$  standard efficiency tracing, the  $^3\text{H}$  standard counting sources are closely matched in terms of chemical composition to the sample counting sources. The two series of sources (sample series and  $^3\text{H}$  series) are identical except for the amount of imposed quenching agent used to vary the quenching. This is done to ensure that there is only one quenching variable (the imposed quench agent). In general, all sources are prepared gravimetrically, however, mass determinations of bulk components are not always required. When very close matching is critical and required, an analytical balance is used for weighing the bulk components. Radioactive solutions are weighed with a microbalance using the aspirating pycnometer method.

The preparation steps are as follows:

1. Each of the series of LS vials are filled with approximately 10 mL of a suitable scintillation fluid from a dispensing bottle.

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2. The aqueous fraction in each vial is adjusted to about 0.06 or greater by adding carrier solution with a pycnometer or syringe.
3. For a quench-varied series, varying amounts of the imposed quench agent (typically a 1:3 ratio of nitromethane in ethanol) is added dropwise with a pycnometer to the series of vials.
4. The radioactive sample vessel (either a flame-sealed glass ampoule or a crimp-sealed glass vial) is opened and the solution is aspirated into a pycnometer.
5. The pycnometer is placed on the microbalance and weighed.
6. A quantity, generally in the range of 15 mg to 100 mg, is dispensed into one of the LS vials, and the pycnometer is reweighed to determine the dispensed mass of radioactive solution. The pycnometer mass is recorded at every step. Buoyancy correction due to air displacement is applied to all mass measurements done.
7. The procedure is repeated until all LS vials are filled. Any remaining radioactive solution is then transferred to an empty ampoule. The empty pycnometer is weighed one last time and disposed in radioactive waste.
8. The ampoule with the remainder of the radioactive sample is flame sealed, the outside checked for external contamination by taking and counting smears and stored.
9. Identical steps are performed for the  $^3\text{H}$  vials and ampoule used for  $^3\text{H}$ -standard efficiency tracing.
10. The LS vials are shaken or swirled to ensure complete mixing of the components. In addition, the solutions in the LS vials are visually examined for apparent non-homogeneity or phase separation after mixing
11. Each filled LS vial is checked to ensure that the LS vial cap is tightly closed, and the outside of each vial is checked for external contamination by taking and counting smears.

*Comparative LS Counting*

Each LS instrument has its own method and protocol for loading and counting samples as well as for data collection and is used in accordance with the respective manufacturer's instructions and user manuals located in Bldg. 245, room H215.

Solutions for calibration will be measured by comparative measurements against NIST standards (e.g., certified SRMs), if available.

A typical experimental design for such comparative measurements consists of: (1) prepare a set of 6 or more LS cocktails of the standard; (2) prepare a set of 6 or more matched cocktails of the solution to calibrate; (3) prepare at least two matched blanks for background subtraction; (4) these sets of cocktails are to be as closely matched as possible in terms of chemical composition; (5) at least two different series of cocktails should be prepared using two different scintillation fluids; (6) All cocktails should be measured in at least two different LS counters, and should be measured for a minimum of 3 to 5 measurement cycles for each trial.

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The data collected contains: (1) start time and date of measurements; (2) elapsed time; (3) counting rate (usually in units of counts per minute) in a given window; and (4) quench indicating parameter (QIP). Options for other instrument quantities, such as luminescence detection and correction, homogeneity monitoring, etc. are available, but are generally not used. Collection and storage of the LS spectra may or may not be employed.

All calculations are performed manually with spreadsheets and extensive graphical analyses. The LS counters' software is not used for calculations.

Massic counting rates for each vial for each measurement are calculated by subtracting that measurement cycle's background (or average background for the entire measurement trial), correcting for radioactive decay to the reference time and normalizing by the mass of the radioactive substance in the vial. The two massic counting rates for the standard  $R_S$  and for the test solution  $R_X$  are related to the massic activity  $A_X$  and standard's massic activity  $A_S$  through  $A_X = A_S ( R_X / R_S )$ .

The mean massic activity is obtained as an unweighted or weighted average of all  $A_X$  values, with an appropriate statistical precision estimator, e.g., a standard deviation of the mean on considering all within- and between-measurement components of variance.

#### *LS Efficiency Tracing*

Calibrations by LS efficiency tracing are often considered to be more "absolute" or primary compared to comparative LS counting. The procedure has been used somewhat routinely in the NIST Radioactivity Group since the late 1980s.

The underlying model for LS efficiency tracing has been described in detail by Zimmerman and Collé [*J. Res. Natl. Inst. Stds Tech.* **102**, 455-477, 1997] and references therein for the standardization of  $^{63}\text{Ni}$ . The following description of the method is an extract from this publication. It must be emphasized that many revised and improved efficiency calculation programs have been developed since the use of EFFY4 in 1996. EFFY4 is a revised and updated version of EFFY2. See E. Garcia-Torano and A. Grau Malonda, *Comp. Phys. Comm.* **36**, 307 (1985); and A. Grau Malonda, E. Garcia-Torano, and J. M. Los Arcos, *Int. J. Appl. Radiat. Isot.* **2**, 157 (1985). For MICELLE and MICELLE2, see C.A. Grau, *Comput. Phys. Comm.* **176**, 305 (2007); and K. Kosset, A. Grau-Carles, *Appl. Radiat. Isot.* **68**, 1482-1488 (2010). Also there have been many computer modernizations that have enhanced computational capability.

The CIEMAT/NIST  $^3\text{H}$  efficiency tracing method is a protocol by which the LS counting efficiency for a cocktail of interest under known, varying quenching conditions is obtained by following the efficiency of a closely matched (in terms of cocktail composition) standard. Tritium is a good candidate for this standard because of its low  $\beta$ -energy, which makes it very sensitive to quenching effects. In order to describe the overall efficiency of the counting system, a "figure of merit"  $M$  is employed. This parameter is meant to describe the energy (in keV) required to produce one photoelectron at the first dynode of the PMT. For a decay event with energy  $E$

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from the  $\beta$ -spectral distribution, the fraction of energy lost due to heat absorption outside the solution (i.e., the container walls) is given by  $1-W(E)$ , while the fractional energy loss due to secondary interactions within the solution (“quenching”) is given by  $1-Q(E)$ . The energy remaining, and thus available to produce photoelectrons, is then  $EQ(E)W(E)$  and represents the unquenched energy of the decay event. The average number of photoelectrons produced at the first dynode of the PMT is then

$$\bar{n} = M^{-1} EQ(E) W(E). \quad (1)$$

For low energies (< 100 keV),  $\beta$ -particles with the distribution of these photoelectrons are assumed to follow Poisson statistics (at higher  $\beta$ -energies the distribution is normal). Thus, the probability of detecting  $x$  photoelectrons from a mean number of photoelectrons  $\bar{n}$  can be calculated by

$$P(x, n) = \frac{\bar{n}^{-x}}{x!} \exp(-\bar{n}), \quad (2)$$

and the probability of detecting zero electrons (the “nondetection probability”) is just

$$P(0, \bar{n}) = \exp(-\bar{n}), \quad (3)$$

where the probability distributions are normalized to 1. The detection probability for a single PMT is then  $(1 - \exp(-\bar{n}))$ . Hence, for a two-PMT system in coincidence, the expected efficiency above the detection threshold is given by

$$\begin{aligned} \hat{\epsilon}(E) &= \{1 - \exp[-\bar{n}]\}^2 \\ &= \{1 - \exp[-M^{-1}EQ(E)W(E)]\}^2. \end{aligned} \quad (4)$$

This efficiency, however, still only applies to events *above* the detection threshold. In order to include events below the threshold, an extrapolation to zero detection threshold must be made.

This is achieved in the CIEMAT/NIST method by relating this probabilistic efficiency  $\hat{\epsilon}$  to one that can be determined experimentally. The calculation model for this transformation is embodied in some efficiency calculational program (e.g., EFFY4 or as used more recently MICELLE2). These programs calculate the efficiency for a given  $M$  by first calculating the shape of the  $\beta$ -spectrum for a particular nuclide through the Fermi distribution function  $P(Z, E) dE$  (not to be confused with the Poisson probability distribution  $P(x, n)$ , given above). By comparing the number of particles emitted to the number of those that would most likely be detected, the efficiency can be calculated by the relationship

$$\hat{\epsilon} = \frac{\int_0^{E_{max}} \{1 - \exp[-M^{-1}EQ(E)W(E)]\}^2 P(Z, E) dE}{\int_0^{E_{max}} P(Z, E) dE} \quad (5)$$

where the integrals represent the total number of counts (numerator) or  $\beta$ -particles (denominator) over the energy from 0 keV to the  $\beta$ -endpoint. In practice, the procedure consists of running the program to obtain a

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table of  $M$  vs  $\hat{\epsilon}$  [(accomplished by numerical integration of Eq. (5)] for each of the nuclides involved in the study, including the tracing standard. The efficiency of the standard is then experimentally determined, and a corresponding value of  $M$  is read from the table. The precision in the determinations is based on the step size between successive  $M$ -values used when running the program. A fit of  $M$  vs  $q$ , an experimentally determined quench indicating parameter (QIP), is obtained for the standard. Since the relationship between  $q$  and  $M$  is assumed to be independent of the radionuclide, the same equation can be used to obtain  $M$  for each of the other nuclides based on the measured  $q$  for that nuclide as long as the measurements are carried out using a set of standards with cocktail compositions identical to those of the radionuclide being analyzed. By performing this fit of  $M$  vs  $q$ , it is possible to make the necessary (small) adjustments to the figure of merit between cocktails which are slightly mismatched in composition, volume, or any other condition that leads to slightly different quenching. Based on the  $M$  calculated from this equation, the efficiency for that particular nuclide can be obtained for those quenching conditions using the tables generated by the program.

The general data analysis scheme for any given experiment is as follows:

1. The count rates of all background blanks are averaged.
2. The count rate for each cocktail is corrected for background by subtracting the average integral background rate from the observed gross integral count rate of the cocktail.
3. The counting rates are then decay corrected to a common reference time.
4. The corrected count rate for each cocktail was divided by the mass of the added radioactive solution to obtain a massic rate  $R_x$  with unit  $s^{-1} g^{-1}$ .
5. An average (across all cycles in the measurement trial) massic count rate,  $\bar{R}_x$  across all cycles in the measurement trial, and an average QIP,  $q_m$  is obtained for each cocktail.
6. An average efficiency for each  $^3H$  cocktail is calculated from the known massic activity of the  $^3H$  standard and the experimental massic count rate for each  $^3H$  cocktail.
7. Based on each average efficiency, an average figure of merit,  $M_{3H}$ , is obtained for that cocktail by looking up the corresponding  $M$  value for that average efficiency in the tables generated by the program for  $^3H$ .
8. A third-degree polynomial fit of  $M_{3H}$  vs  $q_m$  is obtained.
9. From this fit, a figure of merit,  $M_s$ , for each test sample cocktail is calculated from its  $q_m$ .
10. Using the tables generated by the program, an average efficiency for each test sample cocktail is obtained. Dividing this efficiency into the average massic count rate for that cocktail, the massic activity,  $A_s$  is calculated.

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As for comparative LS counting, all calculations are performed using commercially-available spreadsheet programs and do not rely on LS counter internal software calculations. Similarly, the measurement results for the traced activity are thoroughly examined by graphical and statistical analyses, looking for possible systematic effects, including cocktail instability, cocktail composition variations, and between counter and between composition variations. Zimmerman and Collé [op. cit.] for the 1996  $^{63}\text{Ni}$  standardization examined the effect of 10 different experimental conditions.

*LS Counting of  $\alpha$ -Particle Emitting Nuclides and  $\alpha\beta$  Decay Chains*

Alpha particles in LS cocktails are detected with essentially 100 % efficiency. R. Fitzgerald and A.M. Forney, [LSC 2010, “Determination of the liquid scintillation counting inefficiency (wall effect) for alpha emitters using the alpha-gamma anticoincidence method”, *LSC2010 Advances in Liquid Scintillation Spectrometry*, pp. 331-339 (2010)] demonstrated that there is no counting inefficiency due to a “wall effect” for alpha emitters. As a result, LS calibrations of pure alpha emitters are rather straightforward.

Source preparation and counting schema are essentially similar to that for comparative LS counting. A few (3 to 6) LS vials are prepared with cocktails of a scintillation fluid, an aliquot of the radioactive material, and carrier solution to achieve an aqueous fraction of at least 0.06. The massic counting rate  $R_x$  is equivalent to the massic activity  $A_x$ .

For  $4\pi\alpha\beta$  LS counting of decay chains with multiple alpha and beta emitting nuclides, the LS counting rate at time  $t$  with respect to the reference time is given by the sum of the individual counting rates of all members of the decay chain, i.e.,

$$R = \sum_i A_{\alpha(i)} \varepsilon_{\alpha(i)} \exp(-\lambda_i t) + \sum_j A_{\beta(j)} \varepsilon_{\beta(j)} \exp(-\lambda_j t), \quad (6)$$

where  $A_{\alpha(i)}$  and  $A_{\beta(j)}$  are the activities of the respective  $\alpha$  and  $\beta$  emitting nuclides,  $\varepsilon_{\alpha(i)}$  and  $\varepsilon_{\beta(j)}$  are the respective efficiencies for the  $\alpha$  and  $\beta$  nuclides, and  $\lambda_i$  and  $\lambda_j$  are the respective decay constants for the nuclides. The efficiencies for  $\varepsilon_{\alpha(i)}$  are taken as equal to 1, and the  $\varepsilon_{\beta(j)}$  efficiencies are obtained through efficiency tracing using a series of matched  $^3\text{H}$  standard vials as the efficiency monitor. The individual  $A_{\alpha(i)}$  and  $A_{\beta(j)}$  are related to the parent nuclide through use of Bateman equations. The activity of the parent is obtained through use of these relationships and the measured corrected LS rate  $R$ .

Treatment of the chains in the decay of  $^{229}\text{Th}$  and for  $^{234}\text{U}$ ,  $^{235}\text{U}$  and  $^{238}\text{U}$  in natural uranium has been described by R. Fitzgerald, R. Collé, et al. [*Appl. Radiat. Isot* **68**. 1303-1308 (2010)] and R. Collé and L. Laureano-Perez, et al. [J.Res. Natl. Inst Stds Tech. 122, #44 (2017)], respectively.

*Triple-to-double Coincidence Ratio (TDCR) Counting*

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Calibrations by TDCR counting are considered “absolute” or primary because the absolute counting efficiency is determined without reference to another activity measurement. The procedure has been used routinely in the NIST Radioactivity Group since the early 2000s.

TDCR counting is a figure-of-merit technique, relying on the same underlying models as efficiency tracing and the principles have been described in detail in several reviews [e.g., Broda et al., *Metrologia* **44**, S36-S52; Kossert et al., *Metrologia* **52**, S172-S190]. Very briefly, in a system with three photomultiplier tubes having equal quantum detection efficiencies, the counting efficiency for triple coincidences can be calculated from the energy spectrum,  $S(E)$ , and the energy-dependent quench function,  $Q(E)$ , according to

$$\varepsilon_T = \int_0^{E_{\max}} S(E) \left(1 - e^{-\frac{E \cdot Q(E)}{3M}}\right)^3 dE, \quad (7)$$

and the counting efficiency for the logical sum of double coincidences is given by

$$\varepsilon_D = \int_0^{E_{\max}} S(E) \left(3 \left(1 - e^{-EQ(E)/3M}\right)^2 - 2 \left(1 - e^{-EQ(E)/3M}\right)^3\right) dE \quad (8)$$

As in the efficiency tracing method, the counting efficiencies depend on the free parameter  $M$ . The free parameter of a given LS sample is defined by the condition

$$\frac{\varepsilon_T(M)}{\varepsilon_D(M)} = \frac{R_T}{R_D}, \quad (9)$$

where  $R_T$  and  $R_D$  are the experimentally determined net counting rates in the triple and the double coincidence channels, respectively. In practice, the “logical sum of doubles” is used for  $R_D$ . For a counting system with three photomultiplier tubes, A, B, and C, where the double coincidence event rates are designated  $R_{AB}$ ,  $R_{BC}$ , and  $R_{AC}$ , the logical sum of doubles rate is calculated as  $R_D = (R_{AB} + R_{BC} + R_{AC}) - 2R_T$ . After determination of the free parameter, the counting efficiency  $\varepsilon_D$  can be computed and subsequently the sample activity is obtained ( $A = R_{\text{net}}/\varepsilon$ ) with  $\varepsilon = \varepsilon_D$  and  $R_{\text{net}} = R_D$ . In the same manner, the activity can be calculated using  $\varepsilon_T$  and  $R_T$ .

The same calculational programs used in efficiency tracing are used to calculate the relationship between the triple-to-double coincidence ratio and the absolute  $\varepsilon_D$  and  $\varepsilon_T$ . To test the validity of the efficiency model, a range of efficiencies is sampled either via chemical quenching (as in efficiency tracing) or via application of neutral density (“gray”) filters to the LS vials. Additional corrections for, e.g., photomultiplier tube asymmetries and random coincidences are applied in spreadsheets as part of the data analysis.

The general data analysis scheme for any given experiment is as follows:

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1. The count rates of background blanks are corrected for accidental coincidences and averaged. If multiple gray filters are used to vary efficiency, then filter-specific averages are used.
2. Cocktail- and gray filter-specific double and triple count rates for each photomultiplier tube are corrected for accidental coincidences and then the appropriate blank counting rates are used to calculate background-corrected double and triple count rates.
3. The corrected double and triple count rates are then used to calculate the logical sum of doubles and the triple-to-double coincidence ratio.
4. The logical sum of doubles counting rates are then decay corrected to a common reference time.
5. The corrected count rates for each source are divided by the mass of the added radioactive solution to obtain a massic rate  $R_x$  with unit  $s^{-1} g^{-1}$ .
6. The efficiency curve relating the triple-to-double coincidence ratio to  $\epsilon_D$  (constrained to the range of experimentally observed values of the triple-to-double coincidence ratio) is fit to an appropriate polynomial or spline function. That function is then used to calculate a value of  $\epsilon_D$  from the measured triple-to-double coincidence ratio associated with each  $R_x$ .
7. Dividing each  $R_x$  by its associated  $\epsilon_D$  then gives the massic activity. The data are examined for any trending in the calculated massic activities with time or efficiency.

As for comparative LS counting, all calculations are performed using commercially-available spreadsheet programs. Similarly, the measurement results for the activity are thoroughly examined by graphical and statistical analyses, looking for possible systematic effects, including cocktail instability, cocktail composition variations, and between counter and between composition variations.

**Uncertainty Analysis***Comparative LS Counting*

The quality of the LS measurement results is evaluated by graphical and statistical analyses of the  $A_x$  data, evaluating temporal cocktail stability by examining  $A_x$  as a function of measurement time and evaluating the within-measurement variance for replicates on a given vial and the between-vial variance for a given set of cocktails, as well as the between -composition and between-counters components of variance. Acceptable between-component differences should be less than 2 % in worst cases.

Typical uncertainty components are shown in many of the dozens of papers listed in the References Section. The uncertainty components are combined in quadrature to obtain a standard uncertainty, which is expanded

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by a factor of  $k = 2$  to give an expanded uncertainty. Depending on nuclide and other measurement conditions, typical  $k = 2$  relative expanded uncertainties by comparative LS counting range from about 0.3 % to 1.5 %.

*LS Efficiency Tracing*

Uncertainty analyses and evaluations of measurement results for efficiency tracing are similar to those for comparative LS counting.

Zimmerman and Collé [op. cit.] developed a complete measurement and uncertainty model for LS efficiency tracing for the standardization of  $^{63}\text{Ni}$ . The model identified 28 separate uncertainty components (with estimates of their magnitudes), some of which are partially or wholly embodied in computed precision estimators. The model is illustrated schematically in Figure 1.

In addition, a special issue of *Metrologia* [vol. 52 (2015)] on uncertainty treatments for radioactivity measurements includes an article by K. Kossert, R. Broda, P. Cassette, G. Ratel and B. Zimmerman on “Uncertainty determination for activity measurements by means of the TDCR method and the CIEMAT/NIST efficiency tracing technique.”

*TDCR counting*

Uncertainty analyses and evaluations of measurement results for TDCR counting are similar to those for comparative LS counting and efficiency tracing.

The above-mentioned article by Kossert et al. in the special issue of *Metrologia* [vol. 52 (2015)] on uncertainty treatments for radioactivity measurements gives an excellent overview of the major uncertainty components and important considerations.

**Acceptance Criteria**

The data are examined to see if the results show the expected activity, and the standard deviation of repeated measurement on the source is checked to see if it is in the range expected for the radionuclide (normally less than 0.1 percent at 1 sigma). If there is a discrepancy in activity, a new set of sources are prepared and measured. If the standard deviation is abnormally high, the data is examined for possible trends. The customer is contacted if a discrepancy cannot be resolved.

**Validation of Method**

The measurement method is validated using systematic assessment of the factors influencing the result with extensive graphical analyses. The method robustness is tested by using more than one LS cocktail and

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evaluating the measurement results uncertainty based on an understanding of the theoretical principles and practical experience of the performance of the method.

**Calibration Report**

Reports of Test or Calibration, at a minimum should contain or address the following, unless there are valid reasons for not doing so. Additional information may be provided

- a) a title (e.g., "Report of Test", "Calibration Report" or "Report of Special Test").
- b) the first page shall be on NIST letterhead or be equivalently identified as originating from NIST.
- c) the name and address of the laboratory.
- d) the NIST Calibration Services test or calibration number, and service ID number.
- e) page numbers, with the last page designated as the end of the report.
- f) the name and address of the customer (refer to USPS Publication 28, Postal Addressing Standards).
- g) a description, unambiguous identification, and, when necessary, the condition of the item(s) tested or calibrated.
- h) the principal radionuclide
- i) reference time and date
- j) equipment used and method of calibration (traceability to primary standard); a description of the calibration method; additions to, deviations, or exclusions from the method, if applicable.
- k) certified values for activity with the units of measurement
- l) decay-scheme assumptions, if applicable
- m) assessment of radionuclidic purity
- n) overall uncertainty determinations for calibration
- o) the date of receipt of the test or calibration item(s), where this is critical to the validity and application of the results.
- p) the conditions (e.g., environmental) under which the calibrations were made that have an influence on the measurement results, if applicable
- q) the date(s) of performance of the laboratory activity.
- r) the date of issue of the report.
- s) a statement to the effect that the results relate only to the items tested, calibrated or sampled; evidence or a statement that the measurement results are traceable.
- t) the name of the person(s) performing the measurements and analyses; the name, function, and signature of the person(s) authorizing the report.
- u) an explanation of the proper use and interpretation of the reported results as necessary (this can be in the form of an addendum).

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Reported values are checked against the data on the spreadsheet and this check is noted by initialing. Drafts of reports are identified as such and assigned a draft number. Results of draft proofreading are noted and initialed on the draft. Signatures are obtained at the appropriate level and, if paper, the original report is embossed with a Department of Commerce seal. A copy marked file copy and two marked copies are maintained with the calibration printouts, stored in folders identified by company, radionuclide, date, and service number. If requested, the original paper report is sent to the customer. Electronically signed reports are stored in the calibration record of the E-commerce system.

**Records**

Calibration printouts and report copies are stored in folders identified by company, radionuclide, date, and service number.

Ring binders of calibration records, including measurement data, are maintained by the Radioactivity Group in Building 245 for at least five years.

**Documentation**

User manuals for the LS counters are in Bldg 245, Room H215.

SOP 682.04-0071 (March 2011), Sample Preparation and Liquid Scintillation Counting – located in SOP shared drive.

SOP 846.04-0031 (October, 2019), Counting of Liquid Scintillation (LS) Sources – located in SOP shared drive.

NIST website, Activity Calibrations, Special Tests of Beta-Particle-emitting solution Sources, [https://shop.nist.gov/ccrz\\_ProductDetails?sku=43060S&cclcl=en\\_US](https://shop.nist.gov/ccrz_ProductDetails?sku=43060S&cclcl=en_US)

**Filing and Retention**

Filing and Retention Calibration printouts are stored in folders identified by company, radionuclide, date, and service number. For customer calibration, the calibration report is prepared, and the required signatures obtained. A copy is made for customer file and the original sent. Reports created and signed digitally are downloaded to the calibration record in the E-commerce system. The RPD Quality Manager shall maintain the original and all past versions of this RPD Procedure

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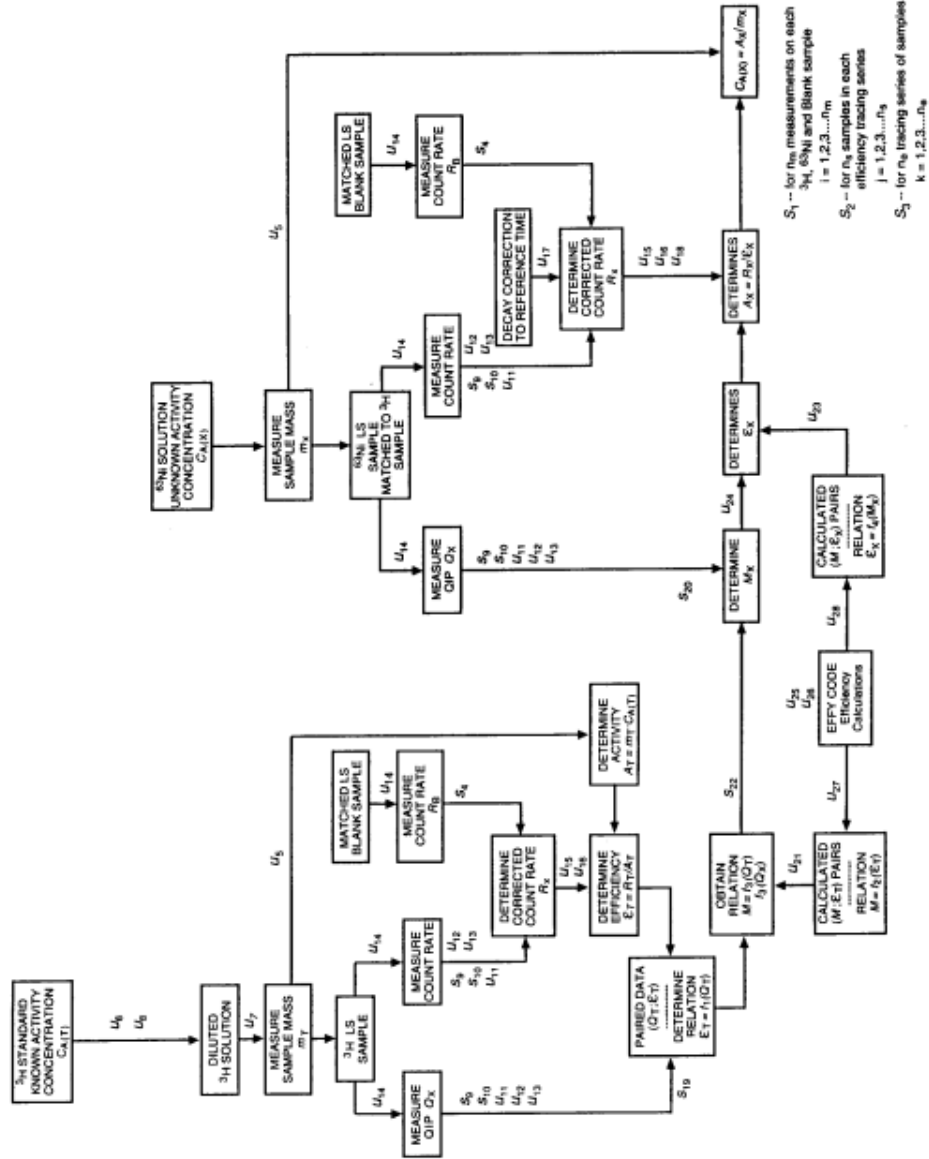


Figure 1. Measurement model for  $4\pi\beta$  LS spectrometry of  $^{63}\text{Ni}$  (as an example) using  $^3\text{H}$ -standard efficiency tracing as given by the CIEMAT/NIST method. The component uncertainties are identified as either  $u_i$  or  $s_i$  as identified by Zimmerman and Collé [op. cit.]

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