American National Standard Check Sources for and Verification of Liquid-Scintillation Counting Systems

Sponsor

National Committee on Radiation Instrumentation, N42

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American National Standards Institute

Secretariat

Institute of Electrical and Electronics Engineers, Inc.

Approved 29 September 1997
Reaffirmed 2 December 2004

American National Standards Institute

Abstract: Tests and procedures to ensure that a liquid-scintillation counting system is producing reliable data are provided for designers and users. This standard does not cover the calculation of sample activity for quenched unknown samples, sample preparation, efficiency correlation (quench correction) procedures, or identification of unknown radionuclides.
Keywords: calculation of sample activity, liquid-scintillation counting systems, quenched unknown samples, tests and procedures for liquid-scintillation counting systems

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345 East 47th Street, New York, NY 10017-2394, USA

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ISBN 1-55937-970-7

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Introduction

(This introduction is not part of ANSI N42.15–1997, American National Standard Check Sources for and Verification of Liquid Scintillation Counting Systems.)

This standard is the responsibility of the Accredited Standards Committee N42 on Radiation Instrumentation. Committee N42 delegated the development of this standard to its subcommittee N42.RM. Drafts were reviewed by Committee N42, Subcommittee N42.RM, and other interested parties, and the comments received were utilized in producing the standard as finally approved.

This standard combines ANSI N42.15-1990, American National Standard Verification of Liquid-Scintillation Counting Systems, and ANSI N42.16-1986, American National Standard Specifications for Sealed Radioactive Check Sources Used in Liquid-Scintillation Counters, with modifications. This standard was approved by N42 letter ballot on 28 January 1997.

At the time it approved this standard, the Accredited Standards Committee on Radiation Instrumentation, N42, had the following members:

Louis Costrell, *Chair*

Organization Represented	*Name of Representative*
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	Jack M. Selby
	Kenneth L. Swinth
	Al N. Tschaeche
	Edward J. Vallario
	Lee J. Wagner

At the time this standard was approved, Subcommittee N42.RM had the following members:

Frank X. Masse, *Chair*

J. M. R. Hutchinson, *Secretary*

Robert Ayres
Joseph G. Bellian
John D. Buchannan
R. F. Coley
Bert M. Coursey
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The working group for this standard had the following members:

Yutaka Kobayashi, *Project Leader*

Al Brunsting	Bert Coursey	Steve Wunderly
	Robert Jones	

The following persons were on the balloting committee that approved this standard for submission to the American National Standards Institute:

Joseph C. Bellian	Edward Groeber	Carl R. Siebentritt
Charles L. Britton	James K. Hesch	Anthony J. Spurgin
George W. Campbell	Edward J. Lampo	Joseph R. Stencel
Ernesto A. Corte	Jesse Lieberman	Kenneth L. Swinth
Louis Costrell	Frank X. Masse	Al N. Tschaeche
Morgan Cox	Joseph C. McDonald	Michael P. Unterweger
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Gerald Goldstein		Sanford Wagner

Valerie E. Zelenty
IEEE Standards Project Editor

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American National Standard Check Sources for and Verification of Liquid-Scintillation Counting Systems

1. Overview

Liquid-scintillation counting systems are widely used for radionuclide assay in nuclear medicine, life science research, and industrial applications. A typical commercial system is a combination of a sample-changing device and a liquid-scintillation spectrometer such that a number of samples may be counted automatically. The liquid-scintillation spectrometer usually consists of a sample chamber and two horizontally opposed photomultiplier tubes viewing the sample chamber, with the associated electronics to detect and record the presence of radioactivity dissolved in the sample. The sample compartment may be either air-cooled or refrigerated (controlled temperature). The theory of the operation of this type of radioactivity detection system is summarized in NCRP Report 58-1985 [B10][1]. Additional references on theoretical and applied aspects of liquid-scintillation counting are given in Birks [B1], Bransome [B2], Horrocks [B5], Knoll [B7], Kobayashi and Maudsley [B8], Noakes et al. [B11], Peng et al. [B12], Ross et al. [B14] and Stanley and Scoggins [B16]. The liquid-scintillation counter is so named because the usual radioactive sample assayed is either dissolved or dispersed in a solution containing one or more organic scintillators.

1.1 Scope

The specifications for check sources to be used in the evaluation of liquid-scintillation counter performance are included in this standard.

Measures of performance considered in this standard are

a) Counting system efficiency
b) Reproducibility of sample and background count rates
c) Preparation of check sources for performance evaluation

Definitions of some terms used here are given in Clause 3.

This standard does not cover the calculation of sample activity for *quenched* unknown samples. Accordingly, this standard does not deal with sample preparation, efficiency correlation (quench correction) procedures, or identification of unknown radionuclides.

[1]The numbers in brackets correspond to those of the bibliography in Annex H.

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1.2 Purpose

The purpose of this standard is to provide the user with the means of verifying the performance of typical liquid-scintillation counting systems.

1.3 Word usage

In the wording of this standard, two verbs have been used to indicate the degree of rigor indicated by a specific criterion. *Shall* indicates minimum criteria that must be met, while *should* indicates criteria that are recommended as good practice and are to be applied when practical.

2. References

This standard shall be used in conjunction with the following publications:

ICRU Report 22-1972, Measurement of Low Level Radioactivity.[2]

IEC 60582 (1977-01), Dimensions of vials for liquid scintillation counting.[3]

IEEE Std 398-1972 (Reaff 1991), IEEE Test Procedures for Photomultipliers for Scintillation Counting and Glossary for Scintillation Counting Field (ANSI).[4]

3. Definitions

3.1 background check source: A sealed vial of liquid-scintillation solution containing no added radioactive material.

3.2 background count rate (in radioactivity counters): Count rate recorded by the instrument when measuring a background check source in counts per minute.

3.3 check source: A radioactive source, not necessarily calibrated, that is used to confirm the continuing satisfactory operation of an instrument.

3.4 counting channel: A region of the pulse-height spectrum that is defined by upper and lower boundaries set by discriminators.

3.5 counting efficiency: The ratio of the count rate to the disintegration rate, usually expressed as a percentage: $E = (R/A) \times 100$. (See 4.4 for nomenclature.)

3.6 counting region: A region that identifies the first and last memory location of a contiguous series to be summed in a multichannel analyzer.

3.7 gain, photomultiplier tube: The ratio of the signal output current to the photoelectric signal current from the photocathode. (See IEEE Std 398-1972.)[5]

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[2]ICRU publications are available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Suite 800, Bethesda, MD 20814, USA.

[3]IEC publications are available from IEC Sales Department, Case Postale 131, 3, rue de Varembé, CH-1211, Genève 20, Switzerland/Suisse. IEC publications are also available in the United States from the Sales Department, American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036, USA.

[4]IEEE publications are available from the Institute of Electrical and Electronics Engineers, 445 Hoes Lane, P.O. Box 1331, Piscataway, NJ 08855-1331, USA.

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3.8 liquid-scintillation solution: A solution consisting of an organic solvent (or mixture of solvents) and one or more organic scintillator solutes.

3.9 organic-scintillator solute material: An organic compound that can absorb radiant energy and immediately (typically within 10^{-9} s) re-emit this energy as photons in the ultraviolet range.

3.10 pulse height analyzer: A circuit that produces an output signal if it receives an input pulse whose amplitude falls between upper and lower assigned values.

3.11 pulse height analyzer, multichannel: A circuit that accepts all input pulses and assigns each pulse to a memory location corresponding to its amplitude.

3.12 quenched sample: A counting sample (material of interest plus liquid-scintillation solution) that contains chemical impurities that reduce the photon output to the photomultiplier tubes.

3.13 resolving time: The minimum time that must exist between successive events if they are to be counted as separate events.

3.14 unquenched sample: A counting sample (material of interest plus liquid-scintillation solution) that contains a minimum of colored species and chemical impurities that would reduce the light output to the photomultiplier tubes.

3.15 vial: A glass or plastic sample container that meets the dimensional specifications of IEC 60582 (1977-01).

4. Standards and check sources

4.1 Radioactivity standard material

A radioactivity standard material is a material having a known radioactivity concentration of a specified radionuclide (see [B17]). Such material shall be either of the following when such standards are available (see [B15]):

a) A radioactivity standard reference material that has been certified by a laboratory recognized as a country's national standardizing laboratory for radioactive measurements.
b) A radioactivity standard material that has been obtained from a supplier who participates in measurement assurance activities with the national standardizing laboratory.

In such measurement assurance activities, the supplier's calibration value should agree with the national standardizing laboratory's value within the overall uncertainty stated by the supplier in its certification of similar sources.

4.2 Check sources

The following four types of check sources that are of the vial type may be used:

a) Flame-sealed glass (activity known)
b) Flame-sealed glass (activity unknown)
c) Screw-capped glass or plastic (activity known)
d) Screw-capped glass or plastic (activity unknown)

[5]Information on references can be found in Clause 2.

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Type a) check sources can be used for all measurements described in this standard. Such sources are available from instrument manufacturers and suppliers of radiochemicals. They are often designated as *unquenched standards.*

The use of types b) through d) is limited to specific tests. Type b) can be used whenever relative data are sufficient and no computation of the disintegration rate is required. Types c) and d) shall be used only for short-term performance tests. The scintillation characteristics of a source in screw-capped vials can change rapidly for several reasons, one of which is the evaporation of volatile components. When polyethylene vials are used, the solvents and solutes can diffuse into the walls, causing swelling of the vial as well as changes in the scintillation properties of the wall (see [B6]).

4.3 Background sources (unquenched blank)

Flame-sealed background sources are equivalent to type a) check sources (see 4.2) with no added activity, and are used to monitor the reproducibility of the counter background. Such sources are sold as *unquenched background* by commercial suppliers.

The glass vials used for background check sources shall be fabricated from low-potassium borosilicate glass, and the dimensions shall conform to the specifications in IEC 60582 (1977-01).

4.4 Symbols

A Activity of the radionuclide contained in the check source. In this standard, activity is expressed in disintegrations per minute (dpm), although the recommended unit for activity is the becquerel (Bq), where 1 Bq equals one transition per second (see NCRP Report 58-1985 [B10]).

B Background count rate in counts per minute.

c Subscript used in t_c to represent elapsed counting time.

E Counting system efficiency: $E = (R/A) \times 100$.

i Any individual measurement in a series of measurements; used as subscript notation (e.g., n_i).

n Number of measurements in a series.

N Total number of counts accumulated in an individual measurement.

R Net count rate in an individual measurement in counts per minute.

t Counting time in minutes.

t_c Elapsed counting time for a short-lived radionuclide expressed in the same units of time as the half-life.

λ Radioactivity decay constant.

S Computed standard deviation of a measured distribution.

$\hat{\sigma}$ An estimate of σ.

σ *True* standard deviation for the measured distribution.

$\bar{N}$ Average of n measurements of N as defined by Equation (1).

χ^2 Statistic used to compare the observed variance of a distribution with a hypothetical variance; $\chi^2 = S^2/\sigma^2$.

X^2 Computed value of χ^2 for a real distribution; see Equation (4).

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5. Materials

The recommendations for the various sample components are based on the availability of the material and established criteria of purity as they relate to assay for radioactivity by liquid-scintillation counting.

5.1 Solvent

Analytical-grade toluene shall be used as the solvent for all check sources. Toluene shall meet or exceed the specifications of [B13] for purity and shall be of fossil origin. (As of this writing, all commercially available toluene is of fossil origin.) Requirements for toluene acceptance are listed in Annex E.

5.2 Organic-scintillator solutes

A primary scintillator, 2,5-diphenyloxazole (PPO), shall be used in preparing check sources. The physical properties of this compound are listed in Annex A. One of the following secondary scintillators may be included in preparing check sources:

a) 1,4-bis-2-(5-phenyloxazolyl)-benzene
b) 1,4-bis-2-(4-methyl-5-phenyloxazolyl)-benzene
c) p-bis-(O-methylstyryl)-benzene

Their physical properties are listed in Annexes B, C, and D, respectively.

5.3 Radioactive material

Tritium-labeled and carbon-14-labeled toluene shall be used as radioactivity standard material.

5.4 Sample container

The sample container shall conform to IEC 60582 (1977-01) for Type II glass vials, except for the overall height, which shall not exceed the specified maximum. For small-vial counting systems, see 8.3. The glass shall be low-potassium, borosilicate glass. The sample shall be contained within the cylindrical portion of the vial.

5.5 Purging gas

The purging gas shall be any inert gas such as argon or nitrogen of at least 99.995% purity, containing no more than 2 parts per million (ppm) oxygen and no more than 1 ppm water.

6. Description of check sources

A given set of check sources (e.g., tritium, carbon-14, background) should be made from the same solvent and organic-scintillator solutes in the same concentration.

6.1 Check sources

6.1.1 Tritium check source

The tritium check source shall contain 15 mL ± 0.2 mL of toluene with a concentration of 5 g ± 1 g PPO per liter of toluene at 20 °C. If a secondary organic scintillator is included, it shall be at a concentration of 0.01–0.5 g/L. The check source vial should be purged with inert gas before sealing. Tritiated toluene with an

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activity of 1×10^5 dpm to 3×10^5 dpm (2–5 kBq) shall be contained in a Type II glass vial [see IEC 60582 (1977-01)].

6.1.2 Carbon-14 check source

The carbon-14 check source shall contain 15 mL ± 0.2 mL of toluene with a concentration of 5 g ± 1 g PPO per liter of toluene at 20 °C. If a secondary scintillator is included, it shall be at a concentration of 0.01–0.5 g/L. The check source vial should be purged with inert gas before sealing. Carbon-14 toluene with an activity of 30 000 dpm to 150 000 dpm (0.5–3 kBq) shall be contained in a Type II glass vial [see IEC 60582 (1977-01)].

6.1.3 Background check source

The background check source shall contain 15 mL ± 0.2 mL of toluene with a concentration of 5 g ± 1 g PPO per liter of toluene at 20 °C. If a secondary scintillator is included, it shall be at a concentration of 0.01–0.5 g/L. The background check source shall be contained in a Type II glass vial and should be purged with inert gas before sealing [see IEC 60582 (1977-01)].

6.2 Expiration date of check source

All flame-sealed check sources shall be dated when made and should be used no longer than 5 years after sealing. (Check sources shall not be stored in direct sunlight or under fluorescent lights.)

7. Operations and tests

7.1 General

All instruments shall be operated in conformance with the manufacturer’s recommendations.

7.2 Test procedures

7.2.1 Frequency of testing

The user shall monitor instrument performance following installation, service, replacement of sealed check sources, or any other circumstance that may affect the accuracy of the data obtained using the counter. Details of these performance tests are given in 7.2.2. In addition, a quality-control program shall be established to monitor the day-to-day performance of the instrument. These routine performance tests are given in 7.2.3.

7.2.2 Initial performance tests

The following tests shall be performed by the user upon installation of the counting system and following any of the events set forth in 7.2.1.

a) Determination of the counting system efficiency (E) of the type a) check source (see 4.2)
b) Determination of the counting rate (B) of a background check source in each counting channel that is used under normal conditions
c) Estimation of dispersion in the counting data by:
    1) The estimated standard deviation of the counting data, assuming it to be Poisson distributed
    2) Determination of the observed standard deviation S

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3) Completion of a chi-squared (χ^2) test, or other test, to determine the reproducibility of the measured sample count rate

7.2.2.1 Counting system efficiency *E* for the tritium check source

Use a tritium check source of known activity *A*. Set the gain and discriminator levels (i.e., the tritium counting channel/region) according to the manufacturer's recommendations. Accumulate approximately 10^5 counts and compute the net sample count rate (*R*). Compute the counting efficiency as $E = (R/A) \times 100$.

For a typically newer system, the maximum tritium efficiency for an unquenched check source shall be greater than 63% when the tritium counting channel/region is set to encompass most of the tritium pulse-height spectrum. A slightly lower value would not be grounds for rejecting the instrument, since the efficiency also depends on the check source. (Failures of the check source are discussed in 8.7.)

7.2.2.2 Counting system background in the tritium channel

Using the instrument settings described in 7.2.2.1, measure the background count rate (*B*). The background check source is described in 3.2.3. Compute the background from these 10 min measurements. If *B* exceeds 40 counts per minute (0.67 Bq), the major sources of the background counts should be identified. Some of the factors that may cause a high background are discussed in 8.8. If the cause of the high background cannot be identified, consult with the manufacturer.

7.2.2.3 Counting system efficiency and background counting rate for other radionuclides

Repeat the procedures in 7.2.2.1 and 7.2.2.2 with counting channels/regions and check sources appropriate for the radionuclide selected. If tritium is not routinely used, another radionuclide can be substituted in 7.2.2.1 and 7.2.2.2.

7.2.2.4 Counting system reproducibility

7.2.2.4.1 Standard deviation

A major measure of performance is that the counting system gives reproducible results. The statistical concepts presented in 7.2.2.4 are necessarily limited to those required to perform a basic test of the counter reproducibility. More detailed statistical treatments are given in NCRP Report 58-1985 [B10], Evans [B4] and NBS Handbook 91 [B9].

If a check source is counted *n* times, for equal counting times, the total number of counts N_i accumulated in the *i*th interval will be distributed about some average value given by:

$$\overline{N} = \frac{1}{n}\sum_{i=1}^{n} N_i \qquad (1)$$

where

N_i is any individual measurement of a series of measurements.

The scatter in these measured values, or, more precisely, the estimated standard deviation of this distribution, $\hat{\sigma}$, is a measure of the counting system reproducibility.

Since the probability of radioactive decay events is distributed according to Poisson statistics, an estimate of the standard deviation for any given single observation is given by:

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$$\hat{\sigma} = \sqrt{N} \tag{2}$$

The standard deviation may also be predicted from the *n* replicate measurements as:

$$S = \left[\frac{1}{n-1} \sum_{i=1}^{n} (N_i - \bar{N})^2 \right]^{1/2} \tag{3}$$

If the measured standard deviation (*S*) of the data as computed from Equation (3) is much greater than that predicted by Poisson statistics, [$\hat{\sigma}$ of Equation (2)], the user shall find the cause of the additional error in the experiment. Perhaps, for example, the sample changer is not positioning the vial reproducibly in the counting chamber.

7.2.2.4.2 Chi-squared (χ^2) test

Statistical tests, such as the χ^2 test (see [B4]) are used to determine whether the system is operating within allow-able limits. The value of χ^2 computed from a sample distribution is given by:

$$X^2 = \frac{1}{\bar{N}} \sum_{i=1}^{n} (N_i - \bar{N})^2 \tag{4}$$

If the X^2 value is within certain limits, the counter is said to be operating satisfactorily at some prescribed confidence. An example of the use of the χ^2 test is given in Annex F. To conform to this standard, a test, such as the one shown there, shall be performed. At least ten replicate measurements shall be made, with *N* lying between 50 000 counts and 500 000 counts. The test shall be performed with a type a) check source (see 4.2), using a counting channel/region set according to the manufacturer's recommendations.

The computed χ^2 value shall fall within the limits corresponding to probabilities of 0.05 and 0.95, respectively. If the system fails to meet this criterion in three consecutive trials, consult with the manufacturer.

7.2.3 Routine performance tests

7.2.3.1 General

The performance of the counting system shall be monitored on a daily basis during periods in which the counter is in use. The tests shall be performed using flame-sealed check sources [see 4.2, type a)] and flame-sealed background check sources (see 4.3). The information that should be recorded is shown on the sample quality control record in Annex G. The data should also be recorded on control charts. If any instrument operating parameter is changed, a new control chart should be started.

7.2.3.2 Check source

The flame-sealed check source shall be used throughout the useful life of the source, where *useful life* is a period not to exceed 5 years after sealing. The check source shall be counted at least once each day while the counting system is in use, for a counting time sufficient to accumulate a minimum of 20 000 counts and a minimum counting time of 30 s. The same counting time shall be used for each measurement to ensure comparable counting statistics.

7.2.3.3 Background check source

The same flame-sealed background check source shall be used at least twice daily for background measurements. It is recognized that the amount of time spent measuring background samples will depend on the rel-

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ative count rates of the routine samples. For the daily background performance check, however, the counting time per measurement shall be constant so as to achieve a statistically comparable number of counts.

7.2.3.4 Control chart limits

After about one month of operation, there will be enough observations of the check source count rate (*R*) and the background count rate (*B*) to allow estimates for the standard deviations for the distributions using Equation (3). The 2S limits should be used for the control charts (see [B9]). For a normal distribution, 95% of the daily observations will lie within 2S of the calculated mean value. This means that only about one measurement per month (20 working days) of the active check source and about two measurements of the background check source will fall outside the control limits.

7.2.3.5 Results outside control limits

If a daily measurement of *R* or *B* falls outside the control limits, the measurement shall be repeated immediately. If the repeat measurement is also outside the limits, check instrument settings and then refer to the precautions listed in 8.7 and 8.8. Some real changes in the mean value for *R* or *B* may occur. For example, the background may drop by a factor of two without affecting the performance of the counter. The purpose of the control chart is to alert the user to possible changes in the instrument surroundings before valuable time is lost.

8. Precautions

8.1 Organic-scintillator solute purity

Commercially available organic scintillators may require recrystallization to meet the purity requirements listed in Annexes A through D.

8.2 Background activity from low-potassium glass

For different vials, a range of values can be expected for the background count rate in the tritium channel due to varying concentrations of radium, thorium, uranium, and potassium in the low-potassium, borosilicate glass.

8.3 Small-vial check sources

For the small-vial counting systems that do not accept the Type II vial [see IEC 60582 (1977-01)], sealed borosilicate-glass vials should be used. To determine appropriate vial dimensions and the optimum volume, consult the operating manual or the instrument manufacturer. The chemical composition and preparation of the check source shall be the same as that specified in 6.1.1 and 6.1.2 with the appropriate volume reduction.

8.4 Sample preparation

When high-viscosity liquid-scintillation solutions such as mineral oil are used, proper mixing is essential to ensure uniform dispersion/solution of solutes within the sample. Failure to observe this precaution will result in nonreproducible results.

8.5 Instrument check sources

Each instrument should have its own assigned set of check sources which includes a background source.

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8.6 Measurement of radioactivity

8.6.1 Nuclear decay parameters

When measuring check sources of short half-life radionuclides such as phosphorus-32 and iodine-25, it shall be necessary to correct the observed data to a common date for comparison purposes.

Count rates observed at time t may be related to those at a reference date t_r by the radioactive decay law:

$$R_{t_r} = R_t e^{-\lambda(t - t_r)} \tag{5}$$

where

λ is the radioactivity decay constant.

8.6.2 Short half-life with respect to counting time

Occasionally measurements will be made on a short half-life radionuclide in which the counting time is an appreciable fraction of half-life. The count rate at the beginning of the measurement, $R_{t=0}$, may be computed from the observed count rate, R_{obs}, by the equation:

$$R_{t=0} = R_{obs}\left[\frac{\lambda t_c}{(1 - e^{-\lambda t_c})}\right] \tag{6}$$

where

t_c is the counting time expressed in the same units as the half-life.

8.6.3 Resolving time of the counting system

During the time period in which the sprectrometer is recording an incoming event, the counting system is said to be *dead,* i.e., pulses arriving at the amplifier during this time interval (typically 0.5–2 μs for commercial liquid-scintillation counters) will not be counted. Methods for measuring and correcting the dead time are given in NCRP Report 58-1985 [B10] and are beyond the scope of this standard. However, in order to minimize dead time errors, the activity of check sources should not exceed 5×10^5 dpm (8.33 kBq).

8.7 Abnormal observations of check source count rate

8.7.1 Integrity of the check source

Abnormal results may indicate deterioration of the check source. Flame-sealed check sources shall be stored in a cool, dark area to prevent thermal and photochemical decomposition of the scintillator(s).

When check sources of types c) or d) (see 4.2) are used, care shall be exercised in the measurement of radionuclides that have a tendency to precipitate or to plate out on the walls of the container (see [B6] and [B7]). Such phenomena will greatly alter the counting efficiency due to the change in geometry and self-absorption of the source.

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8.7.2 Photomultiplier tube and amplifier performance

The photomultiplier tube converts photons emitted by scintillator(s) into photoelectrons proportionate in number to the number of photons collected. Performance of the counting system is dependent on the maintenance of this relationship. Systematic errors of measurement can occur when this relationship is violated. Performance and design characteristics of photomultiplier tubes and associated electronics of liquid-scintillation counters can be found in IEEE Std 398-1972, Engstrom [B3], Knoll [B7], Vaninbroukx [B18] and ICRU Report 22-1972.

Excessive sample count rates and electronic drift appear to be major causes of nonlinear performance of photomultiplier tubes that leads to nonreproducible counting results. The user is advised to verify the sample activity limits of the counting system with the manufacturer.

8.7.2.1 Linearity

The dynode potential or voltage of the photomultiplier tube is basically at equilibrium under normal counting conditions. Nonlinearity in photomultiplier tube performance can arise from counting a highly active sample [in excess of 1×10^6 dpm (17 kBq)]. Pulses can be lost in the amplification process that would normally be collected at a lower count rate. Nonlinearity can also be produced by changes in dynode voltage from their equilibrium value due to persistent high amplitude pulses.

8.7.2.2 Gain shift at high count rates

The counting of a highly active sample [in excess of 1×10^6 dpm (17 kBq)] can cause a significant variation in dynode potential from its equilibrium value, leading to a gain shift. In some situations, a hysteresis effect can be observed. The photomultiplier tube can require a few hours to stabilize and reach its normal equilibrium dynode potential. Some photomultiplier tube structures may not fully recover and the hysteresis effect can remain, resulting in increased background noise.

8.7.2.3 Photomultiplier tube fatigue

Photomultiplier tube structures not designed to handle the high current sustained during counting of highly active samples can become fatigued. Photomultiplier tube fatigue is characterized by the variation in gain that accompanies a large change in photomultiplier tube current as a result of counting a highly active sample.

8.8 Abnormal observations of background check source count rate

8.8.1 Contamination

The most serious cause of an increased background is contamination. Good laboratory practices shall be employed to avoid contaminating the counter.

8.8.2 High-activity sources

Fluctuations in the background count rate can be caused by movement of high-activity sources (such as cobalt-60 irradiation sources) in a nearby room or by the presence of gamma-ray-emitting samples. Other less obvious sources of background counts are discussed in NCRP Report 58-1985 [B10].

8.8.3 Chemiluminescence, phosphorescence, and static electricity

Many methods can be found in the open literature for eliminating these troublesome phenomena (see [B5] and [B8]). It is important that the possibility be recognized that spurious counts may arise from these effects.

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8.9 Automated performance checks

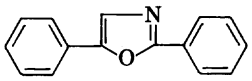
Instrument manufacturers have devised automated systems to perform and record performance checks such as counter efficiency, background, and the chi-square test of their instruments. The user is strongly advised to ensure the reliability of such systems.

Annex A

(informative)

Physical properties of 2,5-diphenyloxazole (PPO)

CAS Registry Number 92-71-7
Scintillation Grade



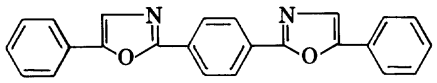
Formula weight: 221.26
Melting point range: 2 °C range including 72 °C
Fluorescence emission (λ max, in toluene): 360 nm
Absorbance (10% in toluene, 10 cm cell at 400 nm versus toluene): 0.25 max. No increase with addition of base.

Annex B

(informative)

Physical properties of 1,4-bis-2-(5-phenyloxazolyl)-benzene (POPOP)

CAS Registry Number 1806-34-4
Scintillation Grade



Formula weight: 364.40
Melting point range: 2 °C range including 245 °C
Fluorescence emission (λ max, in toluene): 415 nm

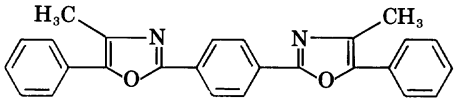
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Annex C

(informative)

Physical properties of 1,4-bis-2-(4-methyl-5-phenyloxazolyl)-benzene (dimethyl POPOP)

CAS Registry Number 3073-87-8
Scintillation Grade



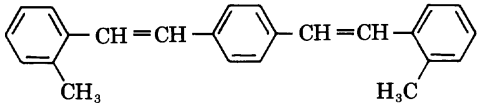
Formula weight: 392.46
Melting point range: 2 °C range including 232 °C
Fluorescence emission (λ max, in toluene): 430 nm
Purity (by TLC): one spot

Annex D

(informative)

Physical properties of p-bis-(O-methylstyryl)-benzene (bis MSB)

CAS Registry Number 13280-61-0
Scintillation Grade



Formula weight: 310.44
Melting point range: 2 °C range including 180 °C
Fluorescence emission (λ max, in toluene): 415 nm

Annex E

(informative)

Requirements for toluene acceptance (see [B13])

Absorbance: to pass test
Color (APHA): not more than 10
Boiling range: entirely within 2.0 °C range including 110.6 °C ± 0.1 °C
Residue after evaporation: not more than 0.001%
Substances darkened by sulfuric acid: to pass test
Sulfur compounds (as S): no more than 0.003%
Water (H_2O): not more than 0.03%

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Annex F

(informative)

Statistical tests of reproducibility—the χ^2 test and other criteria

F.1 Check source

A flame-sealed ^{3}H check source was chosen with an activity of about 10^5 dpm. The net count rate (R) was about 50 000 counts per minute. Thus $R \gg B$ and $R + B \cong R$.

F.2 Measurements

The counter was set according to the manufacturer's recommendations for ^{3}H, and ten 1 min counts were taken. After each measurement, the sample was removed from the sample chamber and then replaced (to test the reproducibility of positioning the sample relative to the photomultiplier tubes).

F.3 Results and calculations

Let Ni be the number of counts accumulated in a 1 min count and let $n = 10$ be the number of determinations. The results for the ten determinations and the deviations from the mean are shown in Table F.l.

F.3.1 Calculation of the mean value, $\overline{N}$

$$\overline{N} = \frac{1}{n}\sum_{i=1}^{n} N_i = \frac{503\ 480}{10} = 50\ 348 \qquad \text{(F.1)}$$

F.3.2 Calculation of the estimated standard deviation for a Poisson distribution

$$\hat{\sigma} = \sqrt{\overline{N}} = \sqrt{50\ 348} = 224 \text{ counts} \qquad \text{(F.2)}$$

From $\overline{N}$ and $\hat{\sigma}$ the relative standard deviation (RSD) is calculated:

$$RSD = \frac{(\hat{\sigma})}{\overline{N}}100 = \frac{224}{50\ 348}100 = 0.44\%$$

F.3.3 Calculation of the observed standard deviation for the measured distribution

$$S = \left[\frac{1}{n-1}\sum_{i=1}^{n}(N_i - \overline{N})^2\right]^{1/2} = \sqrt{\frac{621\ 686}{9}} = 263 \text{ counts} \qquad \text{(F.3)}$$

$$RSD_{\text{obs}} = \frac{S}{\overline{N}}100 = \frac{263}{50\ 348}100 = 0.52\%$$

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F.3.4 The χ^2 value

$$X^2 = \frac{1}{\bar{N}} \sum_{i=1}^{n} (N_i - \bar{N})^2 = \frac{621\ 686}{50\ 348} = 12.3 \quad \text{(F.4)}$$

F.4 Decision on counting system performance based upon statistical observations: the χ^2 test

The χ^2 test is a useful method for the detection of nonstatistical behavior. The distribution of χ^2 is known, and its values have been tabulated. Table F.2, listing the χ^2 values, shows the rejection values for various levels of significance as determined by the number of measurements. Probabilities between 0.05 and 0.95 are considered to indicate that the counting system is probably performing satisfactorily. The observed X^2 is 12.3, which is within the percentage point values of 16.9 and 3.3 indicated in Table F.2 for n =10.

Table F.1—Results of ten determinations and deviations from the mean

Determination	N_i	$(N_i - \bar{N})$	$(N_i \times \bar{N})^2$
1	50 386	38	1 444
2	50 819	471	221 841
3	50 564	216	46 656
4	50 482	134	17 956
5	50 277	–71	5 041
6	50 233	–115	13 225
7	50 365	17	289
8	50 375	27	729
9	49 820	–528	278 784
10	50 159 $\sum_{i=1}^{n} N_i = 503\ 480$	–189	35 721 $\sum_{i=1}^{n} = (N_i - \bar{N})^2 = 621\ 686$
NOTE—The statistics of this distribution are calculated using Equations (1) through (4).			

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Table F.2—χ^2 Values

Number of determinations	Probability						
	0.99	**0.95**	**0.90**	**0.50**	**0.10**	**0.05**	**0.01**
3	0.020	0.103	0.211	1.386	4.605	5.991	9.210
4	0.115	0.352	0.584	2.366	6.251	7.815	11.345
5	0.297	0.711	1.064	3.357	7.778	9.488	13.277
6	0.554	1.145	1.610	4.351	9.236	11.070	15.086
7	0.872	1.635	2.204	5.348	10.645	12.592	16.812
8	1.239	2.167	2.833	6.346	12.017	14.067	18.475
9	1.616	2.733	3.490	7.344	13.362	15.507	20.090
10	2.088	3.325	4.168	8.343	14.684	16.919	21.666
11	2.558	3.940	4.865	9.342	15.987	18.307	23.209
12	3.053	4.565	5.578	10.341	17.275	19.675	24.725
13	3.571	5.226	6.304	11.340	18.549	21.026	26.217
14	4.107	5.892	7.042	12.340	19.812	22.362	27.688
15	4.660	6.571	7.790	13.339	21.064	23.685	29.141
16	5.229	7.261	8.547	14.339	22.307	24.996	30.578
17	5.812	7.962	9.312	15.338	23.542	26.296	32.000
18	6.408	8.672	10.085	16.338	24.769	27.587	33.409
19	7.015	9.390	10.875	17.338	25.989	28.869	34.805
20	7.633	10.117	11.651	18.338	27.204	30.144	36.191

NOTE—In the example given in Annex F, there is only a 5% chance that the measured X^2 value would be less than 3.325. Similarly, there is only a 5% chance that it would be greater than 16.919. Since the measured value was 12.3, a conclusion was reached that the system is in control.

Annex G

(informative)

Performance monitoring log for liquid-scintillation counting

A performance monitoring log for liquid-scintillation counting is given in Figure G.1.

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Month _____ Year_____

Sample Identification

Source	No	Activity (dpm)	Date
Background			
Tritium			
Carbon-14			

Instrument Settings

		Region or Discriminator: Settings	
Region/Channel	Gain	Low Level	High Level
1			
2			
3			

Date	Count Time (min)	Count Rates for Indicated Source and Channel/Region (counts per minute)					Other					Comments	Operator Initials

Figure G.1—Performance monitoring log for liquid-scintillation counting

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Annex H

(informative)

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[B18] Vaninbroukx, R., "Present Status in the Field of Precision Liquid Scintillation Counting," *Nuclear Instrumentation and Methods,* vol. 122, p. 111, 1973, and other references therein.

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[6]NBS publications are available from the Superintendent of Documents, U.S. Government Printing Office, P.O. Box 37082, Washington, DC 20013-7082, USA.

[7]NCRP publications are available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Suite 800, Bethesda, MD 20814, USA.

[8]U.S. Regulatory Guides are available from the Superintendent of Documents, U.S. Government Printing Office, P.O. Box 37082, Washington, DC 20013-7082, USA.

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