



Designation: D 3085 - 75 (Reapproved 1983)

Standard Practice for MEASUREMENT OF LOW-LEVEL ACTIVITY IN WATER¹

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1. Scope

1.1 This practice describes the process necessary to obtain meaningful data from water samples that contain low levels (<10 pCi) of radioactivity.

2. Summary of Practice

2.1 The measurement of low level (up to 10 pCi) fission product or natural activities in environmental samples usually requires separation of the radioelement sought from the water sample. Normally, a measurement of the gross activity of a sample is non-specific and, therefore, of little value. Also, the sample size required to detect a given nuclide quantitatively makes gross counting impractical. An adequate separation of the nuclide sought from the bulk matrix can usually be effected using either carrier or carrier-free techniques.

2.2 It is convenient although not absolutely necessary to add a radioactive tracer to the sample prior to the chemical separation. The tracer is usually another isotope of the element sought and one that has a different mode of decay. For example, strontium-85, a gamma emitter, is commonly used to trace strontium-90, a beta emitter. The chemical recovery of each sample may then be easily and accurately determined by gamma counting the final product of the analysis and comparing this count with a known aliquot of the original tracer solution.

2.3 If the tracer interferes with the measurement of the nuclide sought, then other methods for determining recovery are necessary. One very common method is the gravimetric recovery of the carrier.

2.4 When investigating low levels of fission

products or natural activities in environmental samples, contamination of the sample may occur due to the presence of the nuclide of interest in reagents used in sample processing. Careful analyses of these reagents must be performed to determine the extent of this contamination, so that it may be subtracted from the final sample value. In some instances the extent of contamination of the reagents may dictate the chemical procedure selected for a particular analysis.

3. Significance and Use

3.1 This practice is suited to the gross measurement of alpha, beta and gamma radioactivity in environmental samples with activity levels less than 10 pCi.

4. Instrumentation

4.1 The counters used should be standardized periodically with sources prepared in a manner identical to that used for samples, but with a known amount of the nuclide present. In this way, sample count rates can be converted to disintegration rates. It is, however, impractical to prepare and count such sources each time samples are to be measured. A check source of a permanent nature must be counted whenever samples are measured, and daily, if at all possible, to ensure that the instrument is functioning properly. Control charts carefully maintained on the check source and instrument

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background will almost always detect serious instrument problems. From the control charts it can be determined if the instrument is behaving statistically and in a manner predictable by a Poisson distribution. If no large fluctuations occur, it is always better to use long-term average background and standardization data in the actual calculation of results.

4.2 When counting a low-activity sample, it is always desirable to count for as long a time as possible. Sample workload is usually the determining factor in counting times, but it should also be mentioned that the counting time should be compatible with instrument stability. Overnight counts are usually considered to be a reasonable maximum counting time.

5. Measurement of Alpha Emitters

5.1 The most suitable sample form for the measurement of low-level alpha activity is the electrodeposited source (1).² Since alpha particles are strongly absorbed, even in small sample masses, the counting efficiency of a precipitated source will be much less than that of an electrodeposited source.

5.2 The alpha background of most detectors presently available is low enough so that anti-coincidence circuitry is not necessary. Commonly used alpha counters employing a zinc sulfide (ZnS) scintillator or solid-state detector have backgrounds ranging from less than 1 cpm to a few cph and efficiencies of as high as 52 percent for electrodeposited sources (2).

5.3 Caution should be exercised when counting precipitated alpha emitting sources in an internal proportional flow counter. Filter paper and the precipitate itself are not good conductors and reproducible data are difficult to obtain due to build-up of electrostatic charges.

5.4 In the case where more than one isotope of the same element has been isolated (this is particularly common among the natural elements), gross counting will not always be adequate. High-resolution solid-state alpha spectrometers are extremely useful in such cases and even very low activities of a few dpm may be resolved into several components.

6. Measurement of Beta Activity

6.1 Beta particles are not as strongly affected by sample mass as are alphas and for this reason a sample thickness of less than 15 mg/cm² is suitable for most measurements.

6.2 A low-level beta counter will usually have a background count of less than 1 cpm. To obtain this background level most low-level beta counters require some shielding and anti-coincidence circuitry. The presently available low-level anticoincidence beta counters are gas flow counters and scintillation counters that use a thin plastic phosphor mounted directly on the precipitate (3). Both types of detectors have backgrounds of about 0.2 cpm and efficiencies of approximately 40 percent. In general, the efficiencies obtained by scintillation counting with thin plastic phosphors are higher than those with Geiger-Müller detectors.

6.3 A special case in the measurement of beta activity arises in the determination of tritium and carbon-14. The beta particles from tritium ($E_{\max} = 18.6$ keV) are so weak that a conventional beta measurement is impossible. Carbon-14 ($E_{\max} = 156$ keV) is a borderline case, but is usually classed with tritium with respect to difficulty of measurement. For these two emitters, the measurement is usually carried out with a liquid-scintillation spectrometer. The sample is introduced directly into an appropriate phosphor that is often toluene-based for samples in organic solutions and dioxane-based for samples in essentially water solutions; other systems have been used. Filter paper swipes and finely dispersed particulate material are merely placed in the phosphor solution since the efficiency is not much less than that obtained for a liquid sample.

6.3.1 The several scalers of the spectrometer are each calibrated to count in a particular energy region. In this way, a sample containing a mixture of emitters may be resolved directly. For example, tritium counts will appear in one scaler and carbon-14 counts in another. There is usually some small percentage of overlapping of the counts from the carbon-14 channel into the tritium channel because a spectrum is associated with each end point energy. This introduces no serious problems, however, if the counter is calibrated for the individual nuclide in its own as well as adjacent channels. The sample value may be corrected for the effects of other betas contributing to a particular channel. Several techniques have been published that reduce these calculations to a routine (4, 5).

² The boldface numbers in parentheses refer to the list of references at the end of this practice.