RAPID TECHNIQUE FOR LIQUID SCINTILLATION COUNTING OF CARBON-14-LABELLED BARIUM CARBONATE

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Liquid scintillation counting has become the method of choice for the measurement of low-energy β-activity although some difficulty is still encountered when the radioactive sample is insoluble in the counting solution. For solid samples, one method has been to suspend the finely ground material in the counting solution and by some means correct for the fact that the suspended material will settle out, reducing counting efficiency[1]. Another method that has been used is to reduce the rate of settling of suspensions with gels[2,3]. Although the addition of gels to the counting solution reduces counting efficiency, ranging from a relatively minor decrease[3] to major effects[2], the major disadvantage to both of these methods is the extensive time required for sample preparation. The method reported here utilizes sonication to rapidly reduce particle size, thus decreasing

preparation time and settling rate, and eliminating the need to agitate the sample between successive counts or correct for the settling rate.

Experimental

Reagents. Barium carbonate of low specific activity was prepared by adding acid to $BaC^{14}O_3$ commercially available and trapping evolved $C^{14}O_2$ in 1.0 N sodium hydroxide which contained carrier sodium carbonate. Barium carbonate was then precipitated from this solution. A sample of this barium carbonate was assayed commercially (New England Nuclear Assay Corporation, Boston, Mass.) to determine specific activity. The counting solution, shown by preliminary experiments to give the highest counting efficiency and minimal settling rate, was based upon a dioxane-napthalene system[4] and contained 15 grams of reagent grade naphthalene, 1.05 grams of 2,5-diphenyloxazole (Packard Instrument Co., Downer's Grove, Ill.), 0.045 grams of 4-bis-2-(5-phenyloxazolyl)-benzene (Packard Instrument Co., Downer's Grove, Ill.) dissolved in 15 ml of absolute ethanol and 150 ml of p-dioxane (Spectoquality Reagent, Matheson, Coleman and Bell, East Rutherford, N.J.).

Procedure. $BaC^{14}O_3$ was weighed into glass counting vials, in amounts from 10 to 40 mg and 10 ml of counting solution was added. Each sample was subjected to sonication for 20 to 30 seconds using a Branson Sonifier (Branson Instruments, Inc., Stamford, Conn.) tuned to maximum overtones. The probe of the sonifier was then rinsed with an additional 5 ml of counting solution. After cooling to 2°C, each vial was vigorously agitated just prior to counting. Each sample was counted for 3 minutes three successive times without agitation between counts using a Packard Tri-Carb Liquid Scintillation Counter (Packard Instrument Co., Downer's Grove, Ill.). In studies on settling rates, counts were recorded for each 3-minute interval for a period up to 30 minutes, with no agitation between counts.

Results and Discussion

As shown in Table I, this method gave reproducible results with high counting efficiency. The rate of settling was very slow initially (Table II) so that corrections for the decrease in efficiency over the 9-minute counting period was unnecessary. If a longer counting time was

needed because of low level of activity, the sample could be resuspended by agitation and the normal counting procedure repeated. Once sonicated, samples could be resuspended for counting at any subsequent time up to several months.

The most important aspect of this modification of previous methods is the reduced time needed for sample preparation. The sonication procedure requires less than 1 minute compared to the 3 to 5 minutes needed to grind each sample for suspension counting, and avoids quantitative transfers of solid from grinding vessel to counting vial[1].

Literature Cited

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Table I. Observed specific activity and counting efficiency of various sized samples of sonicated $BaC^{14}O_3$

Sample weight (mg)	Specific Activity* (cpm/mg)	Efficiency* %
10.0	931 ± 14	66.0 ± 0.3
15.0	943 ± 6	66.9 ± 0.1
20.0	941 ± 7	66.7 ± 0.1
25.0	937 ± 11	66.5 ± 0.2
30.0	933 ± 1	66.2 ± 0.1
40.0	914 ± 3	64.8 ± 0.1

*values are given with standard deviation.

Table II. Counting efficiency at various time intervals after agitation relative to the initial three minute counting period.

Time Interval	Relative Efficiency	
(min)	10 mg sample	20 mg sample
0-3	1.000	1.000
9-12	0.999	0.992
18-21	0.985	0.970
27-30	0.970	0.912