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A MINIVIAL FOR SMALL-SAMPLE ^{14}C DATING

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ABSTRACT. We have designed a 0.3-ml Teflon minivial for ^{14}C dating of small samples in a liquid scintillation counter. We use a special adapter of standard vial size to optimize the position of the vial with respect to the phototubes and to intercept the light path between them, thus reducing optical cross-talk. Better performance can be achieved by using customized vials than by diluting small samples for counting in large vials. We have achieved counting efficiencies up to 80% in 0.3-ml vials typically with 0.05 cpm background.

INTRODUCTION

Non-radioactive vials with good transparency for UV light, of homogeneous quality and impermeable to benzene are needed in liquid scintillation counting (LSC) of benzene. Plastic vials are not impermeable to benzene. Glass vials show excellent stability and are easy to clean, but have to be selected for homogeneous quality. Another problem with glass vials is the high background caused by radioactive ^{40}K and some U and Th impurities. In the most advanced counters, the contribution of the slow fluorescence excited by external and internal radiation can now be removed from the beta particles spectrum by using pulse-shape analysis. The contribution from high-energy ^{40}K beta particles (E_{max} = 1.3 MeV) reaching the sample still remains a problem. The search for new materials with lower radioactivity continues. Hogg *et al.* (1991) and Hogg (1992) have shown that silica gives good results.

Wallac introduced specially designed Teflon vials of 3- and 7-ml volumes to measure ^{14}C in benzene and 15- and 20-ml volume vials for other environmental applications, such as ^{3}H in water (Polach *et al.* 1983). The design was based on previous research (Calf & Polach 1974; Kuc & Rozanski 1978). The objectives were to limit the optical size of the vial to the volume needed physically for the sample, and to introduce additional passive shielding against environmental radiation by masking the unused volume of the total 20 ml with a copper top and base. In this way, the background count rate could be minimized.

The background of these vials is almost directly proportional to the volume in the Wallac low background LS spectrometer, Quantulus™[1] (Fig. 1).

NEW DESIGN OF A MINIVIAL

In ^{14}C dating, we continually try to measure ever smaller samples (Haas 1979; Haas & Trigg 1991). This problem has been solved in biological and medical applications where tracers are used in large quantities, and waste has to be minimized, but where background can be neglected. Many commercial counters can measure these vials either as inserts in larger vials or in special trays. New counters for small sample counting have been introduced recently, such as Wallac Betaplate™ and MicroBeta™. The former measures 6 flat-filter paper samples or 400-μl microtitration plate wells, and the latter measures 6 or 4 samples in 200-μl or 1-ml microtitration plate wells, simultaneously.

[1]Quantulus, Betaplate and MicroBeta are trademarks of Wallac Oy, Finland.

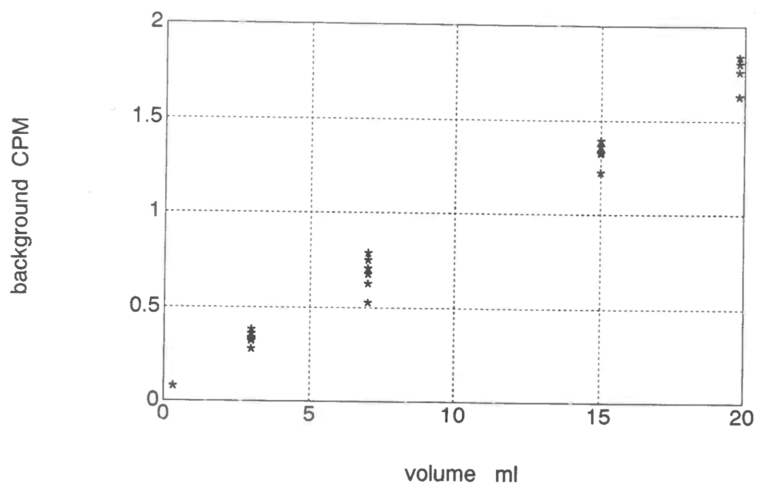

Fig. 1. ^{14}C background count rate of benzene in 3-, 7-, 15- and 20-ml Wallac Teflon vials in Quantulus as a function of volume. Data are recorded at the time of instrument installation in several laboratories with variable environmental gamma flux, which introduces some scatter in the background figures.

In ^{14}C dating, the maximum measurable age (for 2-σ detection criterion, Gupta & Polach 1985) is given by

$$t_{max} = 8033 \ln (A_{ON}/\sqrt{(8B/T)}) \qquad (1)$$

where

A_{ON} = modern sample count rate (cpm)
B = background count rate (cpm) and
T = counting time (min)

In counting low-activity samples, it is therefore desirable for any given volume that the counting efficiency is maximized and background minimized.

The radiocarbon factor of merit is given by Calf and Polach (1974)

$$fM = A_{ON}/\sqrt{B} \qquad (2)$$

which can be expressed as

$$fM = aVE/\sqrt{B} \qquad (3)$$

where

a = 11 dpm ml^{-1} benzene,
V = sample volume (ml),
E = counting efficiency (cpm dpm^{-1}).

In ultra-low-background LSC, B = kV′, where k is a constant and V′ is the vial (optical) volume that determines background.

Let us consider two examples:

Case 1. A 0.3-ml sample in a 3-ml vial: sample V = 0.3 ml, and background V′ = 3 ml.
Case 2. A 0.3-ml sample in a 0.3-ml vial: sample V = 0.3 ml, and background V′ = 0.3 ml.

Theoretically, the fM should be about three times higher for the 0.3-ml vial. The maximum measurable age would then be 9500 years less for the larger vial.

Thus, it is desirable to measure a sample in the minimum optical volume. Dilution of the sample into a larger volume has no advantage, because background will increase accordingly and counting efficiency will not improve.

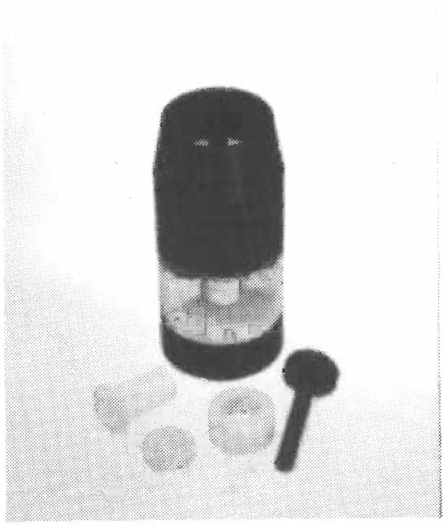

Fig. 2. Wallac 0.3-ml Teflon minivial

Our new 0.3-ml minivial for small-sample dating is made of Teflon and has a Tufbond Teflon/silicone seal. It is contained in a special adapter made of black Delrin with reflectors of white Delrin (Fig. 2). The inherent radioactivity of the vial and adapter is nil. The adapter centers the vials with respect to phototubes for maximum quantum yield, and exposes only the active sample volume to them for minimum background. Table 1 shows typical ^{14}C performance figures for the vial. The background of the 0.3-ml minivial correlates well with the projected background (Fig. 1). Hogg (1992) has also tested these vials recently.

TABLE 1. Performance figures for 300 μl of benzene in the minivial and 3-ml Teflon-copper vial (Polach *et al.* 1988)

V′ (ml)	B (cpm)	E%	fM	t_{max} (yr BP)
0.3	0.05	80.8	11.9	43230
3.0	0.21	76.8	5.5	37290

The Quantulus sample lift mechanism does not rotate, and it does not have a complicated light shutter system to prevent variable geometries. If the counting period contains numerous cycles and thus sample lifts, we recommend modification of the lift piston to prevent rotation of the sample. This can be done by cutting a V-shaped groove into it with a ridge on the bottom of the adapter to fit in the groove. This method has been well tested with quartz vials (Haas & Trigg 1991; Hogg & Noakes 1992).

Conclusion

Adaptation of a vial to the sample size is a straightforward way to achieve best results in liquid scintillation radiometry of low activities of ^{14}C. The minivial concept allows easy adaptation of almost any small vial shape. The same adapter principle can be used for other vial materials such as glass or silica.

Acknowledgment

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References

Calf, G. E. and Polach, H. A. 1974 Teflon vials for liquid scintillation counting of tritium samples. *In* Stanley, P. E. and Scoggins, B. A., eds., *Liquid Scintillation Counting: Recent Developments.* New York, Academic Press: 223–234.

Gupta, S. K. and Polach, H. A. 1985 *Radiocarbon Dating Practices at ANU.* Canberra, The Australian National University.

Haas, H. 1979 Specific problems with liquid scintillation counting of small benzene volumes and background count rates estimation. *In* Berger, R. and Suess, H. E., eds., Proceedings of the 9th International ^{14}C Conference. *Radiocarbon Dating.* Berkeley, University of California Press: 246–255.

Haas, H. and Trigg, V. 1991 Low-level scintillation counting with a LKB Quantulus counter; establishing optimal parameter settings. *In* Ross, H., Noakes, J. E. and Spaulding, J. D., eds., *Liquid Scintillation Counting and Organic Scintillators.* Chelsea, Michigan, Lewis Publishers, Inc.: 669–675.

Hogg, A. G. 1992 Assessment of 0.3-ml minivials for liquid scintillation counting of benzene. *In* Long, A. and Kra, R. S., eds., Proceedings of the 14th International ^{14}C Conference. *Radiocarbon*, this volume.

Hogg, A. G. and Noakes, J. E. 1992 Evaluation of high-purity synthetic silica vials in active and passive vial holders for liquid scintillation counting of benzene. *Radiocarbon*, this issue.

Hogg, A., Polach, H., Robertson, S. and Noakes, J. 1991 Application of high purity synthetic quartz vials to liquid scintillation low-level ^{14}C counting of benzene. *In* Ross, H., Noakes, J. E. and Spaulding, J. D., eds., *Liquid Scintillation Counting and Organic Scintillators.* Chelsea, Michigan, Lewis Publishers, Inc.: 123–131.

Kuc, T. and Rozanski, K. 1978 A small volume Teflon-copper vial for ^{14}C low-level liquid scintillation counting. *International Journal of Applied Radiation and Isotopes* 30: 452–454.

Polach, H., Gover, J., Kojola, H. and Heinonen, A. 1983 An ideal vial and cocktail for low-level scintillation counting: Copper-shielded PTFE (Teflon) and butyl-PBD. *In* McQuarrie, S. A., Ediss, C. and Wiebe, L. I., eds., *Advances in Scintillation Counting.* Edmonton, Alberta, University of Alberta: 508–525.

Polach, H., Kaihola, L., Robertson, S. and Haas, H. 1988 Small sample ^{14}C dating by liquid scintillation spectrometry. *Radiocarbon* 30(2): 153–155.