

NEWLY DESIGNED 0.8-ML TEFLON® VIAL FOR MICROVOLUME RADIOCARBON DATING

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ABSTRACT. We have tested two versions of an 0.8-ml volume Teflon® vial, designed specifically for radiocarbon dating in the microvolume range, using a modern Quantulus 1220™ liquid scintillation (LS) spectrometer. We determined the counting performance of each vial type in conjunction with different designs of copper holder, *viz.*, with and without the incorporation of a “Teflon® light coupler”. We also compared the losses of sample benzene during a typical 28-day counting period. Results show that neither vial design nor the type of vial holder used in the intercomparison had a significant influence on counting performance. We recorded an absolute ¹⁴C detection efficiency of 82% against a background count rate of 0.1 cpm, *i.e.*, a “figure of merit (FM) value” = 67,240. This compares favorably with the operational parameters anticipated for microvolume ¹⁴C dating by modern LS spectrometry. However, variations in the design of the sealing method used between the vial types was reflected in an apparent approximate tenfold difference in the amount of benzene lost during routine counting. In the better case, the evaporation loss was equivalent to 0.032 mg of benzene per day.

INTRODUCTION

Teflon® is often preferred for the construction of liquid scintillation counting (LSC) vials used for low-level detection of ¹⁴C due to its properties, *viz.*, high light transmission, good energy resolution and low inherent background. Commercially available Teflon® vials are designed to accommodate 3-, 7-, 15- and 20-ml sample sizes (Gupta and Polach 1985). The need to count sample sizes significantly smaller than 3 ml (microvolume dating) has encouraged the development of specialized vials constructed in either Teflon® or silica glass to contain 0.3 ml (Hogg 1992; Hogg and Noakes 1992; Kaihola, Kojola and Heinonen 1992). We consider that the restricted size range of vials now commercially available for microvolume dating is a major impediment in routine work. The scale of the reduction, *i.e.*, from 3 to 0.3 ml, is too large; it is five times greater than size stages available for the macrovolume range, *i.e.*, 20 ml, 15 ml, 7 ml and 3 ml. In practical applications, the 3-ml vial size is also open to criticism. In particular, the small capacity can give rise to problems in the preparation of ultra-small counting cocktails due to the unavoidable loss of relatively significant amounts of benzene by leakage and/or evaporation. In this situation, we considered that there was a requirement for a microvolume vial in the intermediate size range, *viz.*, in the region, 0.8- to 1.0-ml capacity.

METHODS

LS counters are generally designed to accommodate vials with a maximum volume of 20 ml. When vials designed for lesser volumes are used, it is necessary to ensure an optimum counting geometry. For microvolume counting, this requirement invariably involves the use of an appropriately designed vial holder (Kaihola, Kojola and Heinonen 1992). Vial and holder dimensions, particularly diameter and height, must be matched to provide an optimum output transmission of light to the photomultiplier tubes of the spectrometer. We designed, constructed and tested two types of microvial and

holder (Figs. 1 and 2). In essence, the difference between vials is in the sealing mechanism, whereas the effect of a "light coupler" was investigated in comparing holder types.

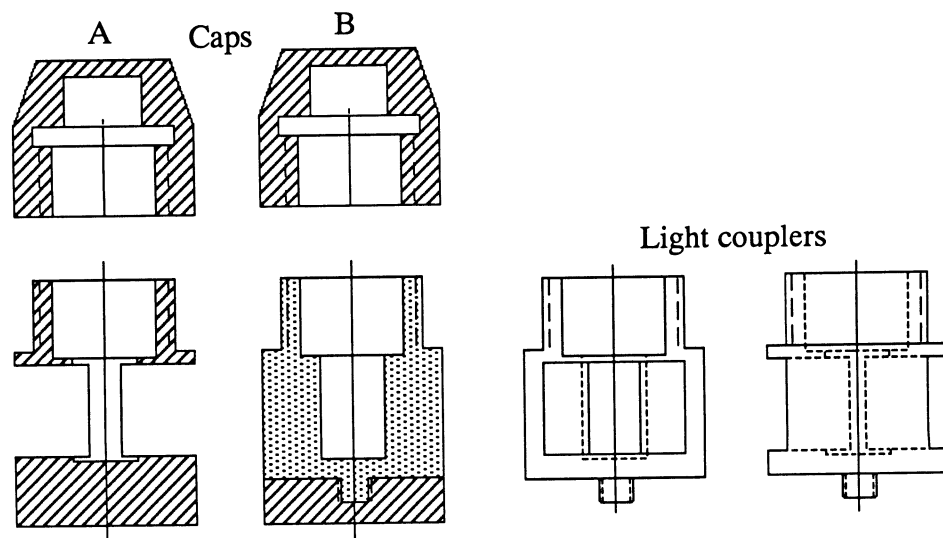


Fig. 1. Vial holder sections. Type A: Copper holder and cap (aluminium can also be used); Type B: Copper holder and cap + Teflon® light couplers.

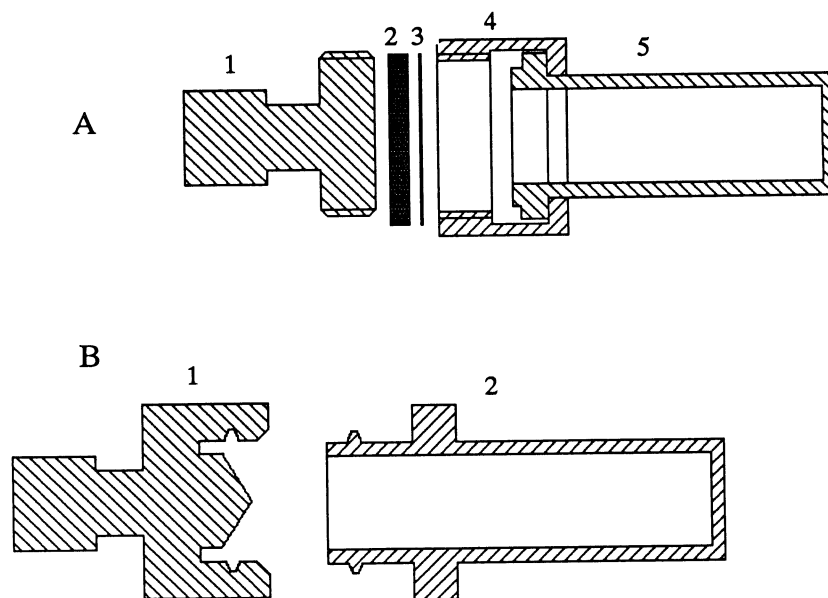


Fig. 2. Vial sections. Type A: Aluminium screw (1), rubber layer (2), Teflon® film (3), aluminium holder (4), Teflon® vial (5); Type B: Teflon® vial and cap: cap (1), vial (2)

We made intercomparisons of vial/holder performance using calibration samples. These were benzene-based scintillation cocktails containing 4 g liter $^{-1}$ PPO and 0.1 g liter $^{-1}$ POPOP. Samples were monitored on a Wallac Quantulus 1220TM LS counter to record a high-coincidence bias.

Throughout the counting programs, mass determinations were made to determine the rates of benzene loss. In the case of vial type B, the mass loss determination was extended to include 100 assembly/disassembly cycles prior to monitoring the benzene loss during the subsequent 20 days.

RESULTS

Counting Performances

We found no significant differences in the direct comparison of both background and ^{14}C detection efficiency among the various combinations of vial and holder types as described above. The mean values are reproduced in Table 1, and this typical performance from the 0.8-ml vials is compared, in Figure 3, with values quoted for the commonly used Teflon[®] vials of varying geometry (Hogg 1993). The only variation noted as a function of vial holder design was with the recorded spectrum quench parameter (SQPE), which depends on the metal used in its construction and whether a Teflon[®] light coupler is employed (see Fig. 4). Clearly, this phenomenon necessitates a specific set of quench-correction values dependent on the vial holder used for microvolume counting.

TABLE 1. Performances for 0.8-ml Teflon[®] Vials Using a Quantulus 1220TM Spectrometer

Vial* volume (ml)	B† (cpm)	N ₀ ‡ (cpm)	^{14}C effi- ciency (%)	fM§ (E ² /BG)	FM# (E ² /BG)	tmax**	tmin††
0.8	0.10	8.96	82	23.2	67240	49072	77

*Benzene weight used: 0.75 g

†Background

‡Derived net cpm for ^{14}C reference standard, 0.95 oxalic acid

§Factor of merit ($N_0 B^{1/2}$)

#Figure of merit (E^2/B)

**Maximum determinable age (using 3000-min count time and 2 σ criterion)

††Minimum determinable age (using 3000-min count time and 1 σ criterion)

Benzene Loss

Table 2 summarizes the recorded loss of benzene from each vial type. In both instances, the rates of sample loss by evaporation are considered acceptable for precise ^{14}C dating by microvolume counting. It is noteworthy, however, that the sealing arrangement on vial type B appears to be about ten times more effective in sample retention. This is also more practical in routine usage.

TABLE 2. Benzene Loss for Vial Version

Vial type	Benzene loss in 28 days (%)	Benzene loss per day (mg)
A	1.3	0.35
B	0.12	0.032

Vial Set Selection

According to the data described above, as well as continuous sample measurement, the better version of each microvolume counting set includes a vial holder of type B and 1 to 3 vials of type B.

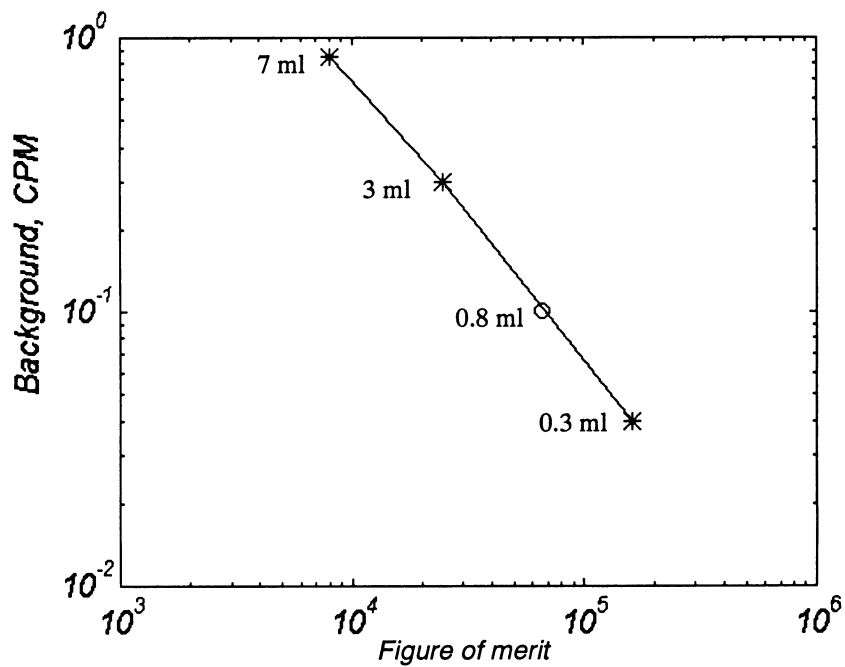


Fig. 3. Performance comparison for Teflon® vials

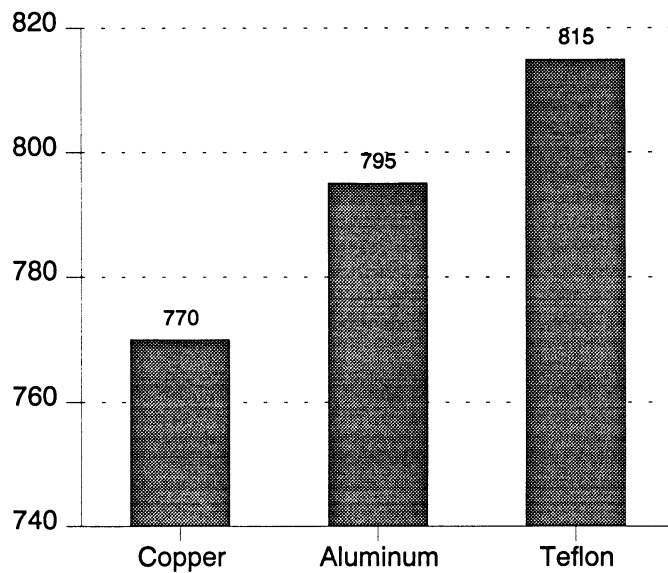


Fig. 4. SQP(E) variation due to holder material

CONCLUSION

The counting performance achieved with new 0.8-ml Teflon® counting vial matched that anticipated from an independent comparison of Teflon® counting vials in general. The low rate of benzene loss during counting makes the type B vial particularly well suited for precise microvolume ^{14}C dating.

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