

AN IDEAL VIAL AND COCKTAIL FOR LOW-LEVEL SCINTILLATION COUNTING

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ABSTRACT

Conventional cylindrical borosilicate glass (^{40}K and ^{226}Ra free), silica (quartz) spherical and polytetrafluoro ethylene (PTFE, Teflon) cylindrical vials were tested for photon transmission efficiency and background counts for ^3H and ^{14}C . For small sample counting (< 20 ml) the vial volume not occupied by a sample was shielded by a mask from direct view by the multiplier phototubes. The masking had no undesirable effects on efficiency of ^{14}C ; for ^3H masking introduced undesirable changes. A new vial design is proposed. It uses a black copper mask cum shield. The vials are made of Teflon and come in four sizes: 3, 7, 15 and 20 ml. Scintillators studied were: PPO + POPOP dissolved in toluene added to sample benzene in a 1:4 ratio; BIBUQ and butyl-PBD if dissolved directly in sample benzene increased the isotope countrate by 25% for same solute + solvent total volumes. The effect of quenching impurities on stability of ^{14}C count rates was determined for various scintillators. Butyl-PBD was the best. It tolerated up to 3% acetone impurities. Conclusions are that the black copper-shielded and masked Teflon vials, with sample benzene as solvent and butyl-PBD as solute, give superior ^{14}C results to any system tested. However, the performance of the vials for ^3H was not good. The application of Teflon-copper vials and butyl-PBD to radiocarbon dating and environmental ^{14}C studies is recommended as the efficiency of the system is significantly increased whilst the background is significantly reduced. The Teflon-copper vials are available commercially.

INTRODUCTION

The function of a vial is to contain the sample solvent and scintillation solute within the space provided for it between the multiplier phototubes (MPTs). It is expected that the vial will have a high photon transmission efficiency, a low self-induced radiation component and exhibit no memory. The most common vials are made of borosilicate glass (^{40}K and ^{226}Ra free) or high density polyethylene or polypropylene. Special vials are made of fused silica (quartz), polytetrafluorethylene (PTFE, Teflon), Nylon or Delrin. Commercially available vials are generally cylindrical of the same height, and have volumes of 22 or 7 mL.

The function of a scintillator is to transfer the energy of an ionizing event (α , β^- , β^+ , γ , neutron or muon) into photons, whose number is proportional to the energy of the ionising event. The scintillator should contain no radionuclides, be pure, soluble in benzene, stable and resistant to quenching.

We shall examine how well certain vials and scintillators perform for low-level tritium (^3H) and radiocarbon (^{14}C) counting and experimentally determine factors which may lead to reduction of background, greater efficiency and improved stability.

VIALS

Variation in performance. The International Electrotechnical Commission recommends standard dimensions of test bottles for liquid scintillation counting: height = 60.5 ± 2.5 mm, outside diameter (OD) = 28 ± 2 mm. The other specifications relate to size of caps and radii of body and caps¹. There are no known specifications relating to wall thickness, uniformity, weight or purity of materials.

Many workers have confirmed that vials made of borosilicate glass will vary in both efficiency and background count rates, not only based on source of supply but also from lot to lot, as reviewed by Painter^{1,2}. Our experience is that if a selection of glass vials is made based in size and weight, the observed variations in background count rates will be minimized. However, we cannot confirm the report by Laney³ that the background count rate of various containers is a function of mass rather than composition of materials. We are certain

that this contradiction is entirely due to our need for high precision of background determinations and that it is valid only in the context of low-level counting. Because we select counting vials carefully we cannot confirm the reported variation in efficiency^{1,2}. Table 1 summarizes our findings.

TABLE I. Variation in background and efficiency of borosilicate, 22 ml vials, due to variation in OD (mm) and weight of glass (g)

	Selection of Vials	
	Uniform (± 0.1 g and mm)	Extremes (± 2 g and mm)
Benzene + Toluene + PPO + POPOP	10 ml	10 ml
Efficiency*, ¹⁴ C	80 \pm 0.5%	80 \pm 0.5%
Poisson error of E	\pm 0.5%	\pm 0.5%
Background*, ¹⁴ C	20 \pm 0.7 cpm	20 \pm 2.5 cpm
Poisson error of BKG	\pm 0.1 cpm	\pm 0.1 cpm

* Limited energy window \pm observed range of mean values

+ 2000 Minute count \pm error of determination

Thus for low-level counting of ¹⁴C, such as radiocarbon dating and ³H or ¹⁴C environmental studies, it is possible to consider the efficiency of all vials, made of the same material and uniform wall thickness, to be the same but it is desirable to evaluate their background individually. The background of 22 ml borosilicate glass vials, even in equipment set up to minimize it, is high. A reduction in background is desirable.

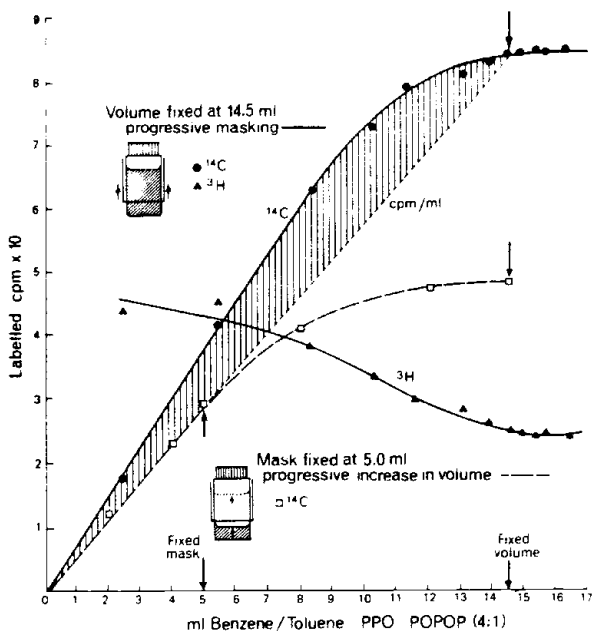
Reduction of background. Average sample volumes for ¹⁴C dating and environmental studies range from 3 to 15 ml of sample solvent and only exceptionally is a vial filled to its nominal capacity⁴. For ³H, on the other hand, 22 ml sample sizes are normal and larger volumes are often desired^{5,6}. It was shown that masking of vials above sample liquid will reduce background^{7,8} and that if so masked, the background

of vials can be, under certain conditions, directly and linearly related to the sample volume⁹. Here we describe new masking experiments carried out at ANU.

The counter, a Beckman LS-200 (1966), was optimized for each isotope, using limited energy windows at balance point⁸. The approximate counting efficiency for ³H was 35% and that for ¹⁴C 65%. An isotope labelled standard was used to determine effect of overmasking (volume of sample is larger than visible volume (area) through which photons can escape (Fig 1)). Background solutions were used to determine the same effect on background radiations (Fig 2).

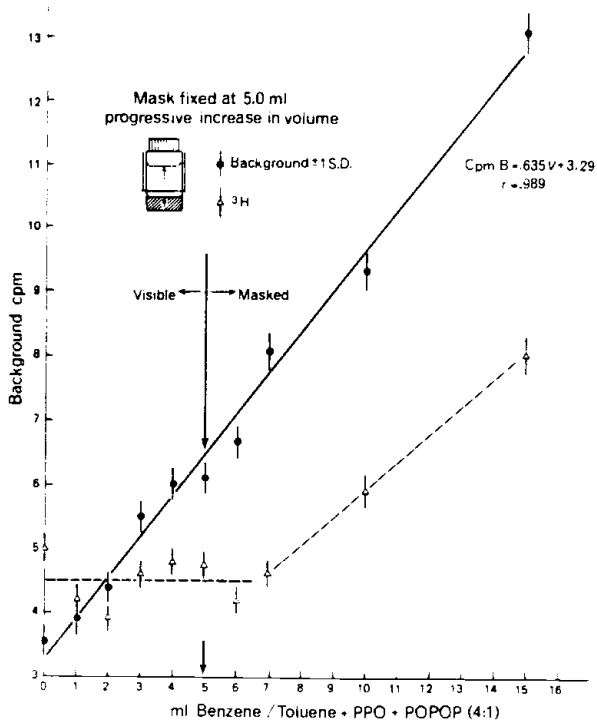
First consider ¹⁴C activity (Fig 1). When the movable mask is at zero volume, whilst sample volume remains fixed at 14.5 ml, observed ¹⁴C count rate is zero. As the mask is moved upwards, exposing different volumes of solution to the MPTs the count rate increases and remains proportionally greater than the calculated cpm/ml line (-----). This demonstrates that a number of photons from the solution above the mask (i.e. without direct access to the MPTs) scatter out giving the increased ¹⁴C count rate (solid line). When the mask

Figure 1.: Effect of masking glass vials on ³H and ¹⁴C count rate when isotope labelled sample volume is fixed (14.5 ml) and mask is moved (upper left) and when mask is fixed (at 5 ml) and sample volume is increased (lower right) from 0 to 14.5 ml).



reaches the meniscus of the sample the calculated and observed ratio becomes unity. Leaving the mask fixed at 5 ml volume and increasing the volume of sample (Fig 1, bottom inset) gives the same result for ^{14}C , i.e. not all photons generated within the masked area can escape and therefore the sample above the mask, whilst contributing some photons, cannot contribute all generated photons. The effect on ^3H efficiency at fixed volume and movable mask conditions are also given. The ^3H count rate was highest when only a portion of the sample was exposed to MPTs. This odd effect is most likely due to pulse height energy spectra shift across our narrow ^3H efficiency window. Since reduction of vial volume is never desired for ^3H low-level counting^{5,6} no attempt was made to improve these odd results; we only conclude that masking may, under certain conditions, have critical effects on observed ^3H efficiencies. On the other hand, should volume of ^{14}C sample be partially masked (the edge of the meniscus hidden) the ratio of observed and calculated cpm/ml will remain unity. The count rate and volume determinations were quite precise. Error, $\pm 1\sigma$, corresponds to the size of markers.

Figure 2: Effect of masking glass vials on background count rate, when isotope free sample volume is increased from 0 to 14.5 ml while mask is fixed at 5 ml.



The effect of background count based on the relationship visible volume to sample volume was assessed by fixing the mask at 5 ml and progressively increasing the volume of an isotope free sample (Fig 2). Surprisingly, the ^{14}C window background (B) remains proportionately and linearly related to volume (V) of sample, irrespective of mask position; i.e. $\text{cpm B} = .635\text{V} + 3.29$. This means that all photons generated by background ionising events are apparently able to scatter out. The ^3H window background was not related directly to sample volume. It remained constant until liquid = exposed by mask + 2 ml. Then background ionization scattering once more increased the observed background count rate.

For purposes of vial design we conclude that masking for ^{14}C is of merit and that sample V to masked V relationship need not be maintained over critically. The masking experiments are not relevant to ^3H low-level counting as preferred sample size is 20 ml or more^{5,6}.

We note that under certain conditions it is possible to get the same ^{14}C background for an empty unmasked vial as it is for a full one. Horrocks¹⁰ documents such a case and J. Stipp and M. Tamers (personal communication) inform us that the ^{14}C background in none of their twelve Beckman LS-150, is sensitive to sample volume changes within the tested range of 5 ± 2 ml. Haas reports a decrease in background with an increase of sample volume¹¹. We believe that these observed variations in background to sample volume relationship are due to differences in empty chamber count - based on magnitude of MPT cross-talk, shape of background spectra - based on HV applied to MPTs, the gain setting of amplifiers, and differences in isotope energy window-width selection and window position across the spectrum^{4,12}.

Vial Shape. Photons originating from within the vial would either escape directly or undergo a series of internal reflections, due to unfavourable escape angles. It is thus accepted that the order of merit for increased probability of photon escape from the vial is: cylindrical \rightarrow square \rightarrow spherical¹³.

Square vials are available commercially. They would suffer from orientation and alignment problems. Nevertheless, Haas uses them with some success¹¹.

We have built a spherical vial out of quartz (fused silica) (Fig 3) and tested it early in 1976 at ANU. Results are given in Table III (see further in the text). No increase in ^{14}C efficiency was observed and, coupled with their alignment problems in automatic changers, this does not warrant the adoption of the spherical design for routine purposes. Our data does not confirm the merit of such a vial attributed to it, based on theoretical computer modelling¹⁴. Admittedly only ^{14}C was tested by us and conventional optical reflectors were used.

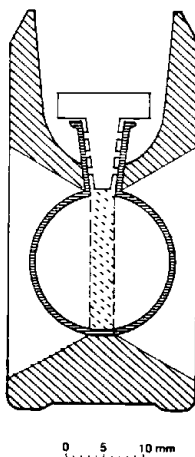


Figure 3: Experimental ANU spherical quartz vial held in inert CaCO_3 moulded to increase photon reflection towards MPTs.

We conclude that the cylindrical design has its functional merit and propose to retain it for low-level counting.

Construction Materials. Borosilicate glass and fused quartz have great merit due to their non-reactive nature. Variations in size can be overcome by careful selection from a large batch. Borosilicate glass is cheap, but has a high background. Silica is expensive, fragile, hard to work and hard to manufacture to precise specifications. For low-level counting, silica has merit¹¹ (cf. Table III in the text).

Other materials available are polyethylene, and polypropylene, both of which are useful for non-aromatic solvents, hence are very practical and cheap for low-level ^3H counting. Nylon, because it is

hygroscopic although impermeable to aromatic solvents, is not useful for low-level counting because the sample weight after prolonged counting needs to be checked and adsorption or desorption of water on vial walls would make precise judgment of sample weight impossible. Incidentally, plastic caps so common in all vials, are also bad for this reason. A large volume vial made of Delrin was used with good results in a Swiss underground laboratory¹⁵.

Calf¹⁶ was first to demonstrate the merit of Teflon for ³H counting. Calf and Polach¹⁷ then demonstrated their usefulness also for ¹⁴C. Since then Teflon vials have become very popular and are routinely used by J. Noakes, J. Stipp, M. Tamers (personal communications) and many others¹⁸⁻²⁰. We had no doubt of their merit, but were concerned about their design, selection of materials for their inherently low activity, selection and location of a source of high density pure and primary Teflon, precision of manufacture, reproducibility of count rate (efficiency and background) and memory effects. In their design we were guided by a progression of experimental vials made at ANU (Fig. 4). The masked glass vials are not illustrated in Fig 4. Vials are of 5 ml and 10 ml volume, justifiably preferred for low-level ¹⁴C work^{4,9}. 'A' represents a borosilicate glass or quartz ampoule shielded by a low activity aluminum sleeve into which one can, with good background attenuation results, insert an aged lead (Pb) plug. 'B' and 'C' represent the Calf-Polach described Teflon vials¹⁷. The 5 ml is for routine work and 10 ml is for radiocarbon dating with increased precision^{9,21,22}.

The Wallac vial series which evolved from these concepts, retain the shielding and the minimal Teflon exposure features. They are presented in Fig 5 in schematic form. They come in 4 standard sizes: 3 ml for ever increasing need to count with undiminished precision small carbon samples (0.1 to 2.4 g)²³; 7 ml to extend the routine dating range up to 5.7 g C; 15 ml for high precision low-level counting of 6 to 12.2 g C^{9,21,22}; and 20 ml for ³H work. A blank for equipment testing is also available. The Teflon-copper vials main feature is that (i) the centre of their active volume always lies on the optical axis of the 180° opposed MPTs and (ii) all the volume not used for counting is shielded by high purity low activity copper of

Finnish origin. Further, (iii) the copper is chemically etched to be a metallic black with low UV reflection characteristic. (iv) The vial is sealed with a silicon rubber backed Teflon disk, (TUFBOND, trademark) thus solvent contact is Teflon to Teflon and vapour loss over one week is not measurable (i.e. < 0.2 mg). (v) The vials are

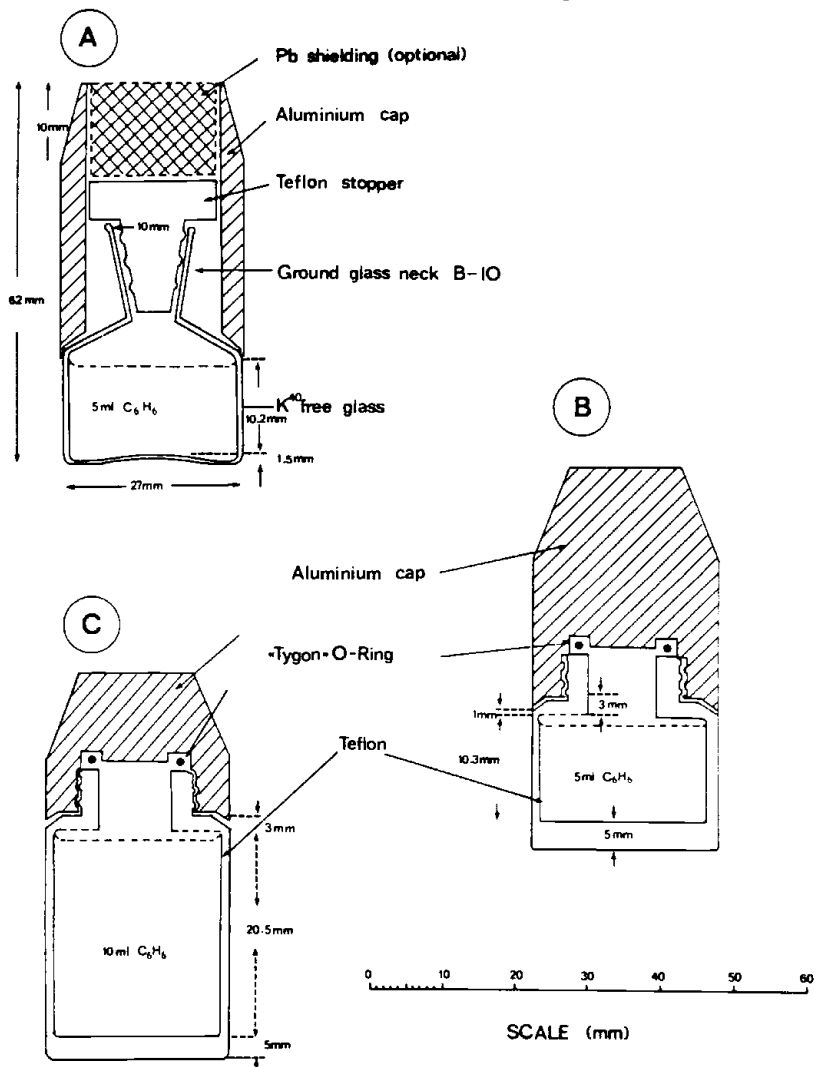


Figure 4: Experimental ANU cylindrical vials. 'A' = borosilicate glass or quartz ampoule with aluminum mask and lead shield. 'B' = 5 ml and 'C' = 10 ml Teflon-aluminum vials.

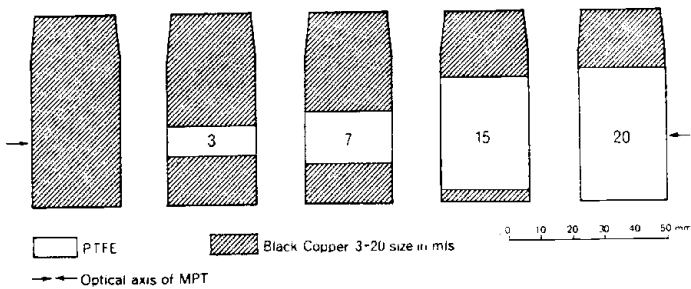


Figure 5: Schematic diagram of Wallac Teflon-copper vial system whose variable counting volume 3, 7, 15 and 20 ml is centred along the optical axis of the MPTs.

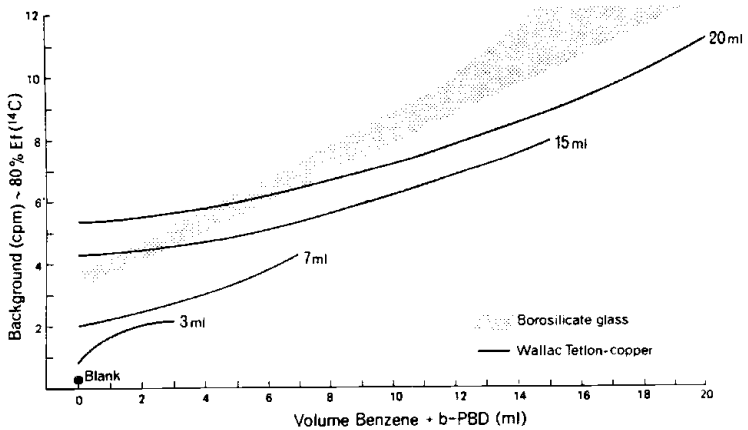


Figure 6: Background, under ¹⁴C window of ~80% efficiency, as a function of sample volume in Wallac Teflon-copper vials of 3, 7, 15 and 20 ml volume (---). Masked borosilicate glass background is given by shaded area. Note higher Teflon/copper vial empty count rate.

made on an automatic high precision lathe and therefore are uniform to very close tolerances and (vi) tests have shown that Teflon chosen for their manufacture does not exhibit memory when re-using vials.

Their characteristic performance is given in Fig 6, from which the merit of the graded vial size system to accommodate sample size variation can be deduced. We believe that the Wallac Teflon-copper vial is ideal for low-level ^{14}C counting.

SCINTILLATORS

Benzene is the most favoured solvent for radiocarbon dating as it can be produced quantitatively by combustion of sample carbon to carbon dioxide, its conversion into acetylene and its catalytic conversion to pure benzene²⁴⁻²⁷. The most commonly used scintillation cocktail was PPO + POPOP dissolved in Toluene. This was added to the benzene in 1 part toluene to 4 parts benzene mixture⁷.

Following the lead given to us by Birks and using his own works for reference^{28,29}, we have constructed Table II which lists the merit

TABLE II. Using benzene as a solvent the scintillation characteristics of various solutes are given in terms of relative pulse height in the presence (Vq), and absence (Vo), of 0.1 M carbon tetrachloride. Based on Birks^{25,26}, sequence of merit is from our data.

Merit	Solute	Vq	Vo
1	10 g/l b-PDB	84	80
2	8 g/l b-PBD	81	77
3	10 g/l PBD	95	77
4	8 g/l BBOT	60	61
5	15 g/l BIBUQ	49	79
6	6 g/l PPO	44	58
7	5 g/l TP	30	52

of scintillators, as we perceive it, using benzene as a solvent. This, of course, is the major feature of the concept. Since background as well as sample signal is related to volume of sample, using the proven 4 benzene:1 toluene recipe⁷, we obtained a signal based on 3.5 g C but a noise based on 4.4 g C. Therefore a 25% improvement in signal could be made using sample benzene alone with a suitable solute.

The basis of our merit listing in Table II is (i) absence of ³H and ¹⁴C isotopes in solute (ii) availability of solute in pure chemical form (iii) high solubility in benzene (iv) long term stability (v) high scintillation yield evaluated in terms of relative pulse height (RPH) (vi) high tolerance to quenching agents determined in terms of changes to RPH.

Our tests confirm the superiority of benzene with butyl-PBD at 15 g/l concentration. Tested were PPO + POPOP (6 and 0.2 g/l respectively) BIBUQ (15 g/l) and b-PBDA in 8-20 g/l concentration range. Using balance point windows at optimal high voltage and gain settings (best E²/B) for ¹⁴C up to 3% of acetone could be added to benzene with butyl-PBD before detecting a change in count rate³⁰ whilst benzene with PPO and POPOP, apart from the solubility problems was the worst with loss of count becoming apparent at 0.05% acetone concentration.

We have also noted that external standardization (ES) quench index based on channel ratio, end-point, slope or inflection point variation of the ES induced Compton spectrum indicated that some quench did occur in all cases. Thus had we used a means of inbuilt automatic quench corrections, our observed and 'true' results would have been falsely corrected³⁰. Here we conclude that for low-level counting of ¹⁴C samples dry powder butyl-PBD dissolved in sample benzene (15 g/l) gave superior count rate stability when balance point settings were used.

TEST RESULTS AND SUMMARY

Advantages. Performance of vial types and solutes described in previous chapters is summarised in Table III. Vial holders were cylindrical of the nominal 22 ml external dimensions. Only the volume

Table III. Relative counting efficiencies of vials and scintillation solutes.

VIAL TYPE	SIZE ml	SOLUTE	g SAMPLE C_6H_6	BKG(B)	ANU SUCROSE net cpm	NET 95% OX No.	No./ B	ANU SUCROSE cpm/(g C_6H_6)
TEFLON-ALUMINUM	5.5	PPO + POPOP*	3.5160	5.2 ± .1	47.4 ± .7	30.8 ± .5	13.5 ± .3	13.5 ± .2
	5.5	b-PDB†	4.3950	5.1 ± .1	60.4 ± .9	39.2 ± .6	17.3 ± .4	13.7 ± .2
GLASS CYLINDER SAND BLASTED	5.5	b-PDB	4.3950	5.8 ± .08	56.5 ± .25	36.7 ± .2	15.2 ± .2	12.8 ± .06
GLASS CYLINDER CLEAR	5.5	PPO + POPOP	3.5160	5.5 ± 0.5	47.4 ± .09	30.8 ± .06	13.1 ± .1	13.5 ± .03
	5.5	b-PDB	4.3950	6.0 ± .1	58.0 ± .9	37.7 ± .6	15.4 ± .4	13.2 ± .2
QUARTZ SPHERE CLEAR	3.9	PPO + POPOP	2.7069	5.1 ± .2	36.4 ± 1.0	23.6 ± .6	10.5 ± .5	13.4 ± .2
	3.9	b-PDB	3.3450	4.9 ± .2	45.5 ± .7	29.5 ± .5	13.3 ± .5	13.6 ± .2
QUARTZ SPHERE	3.9	BIBUQ	3.2450	5.1 ± .1	43.9 ± .3	28.5 ± .2	12.6 ± .3	13.5 ± .1

* 4 ml sample benzene (i.e. 3.5160 g) + 1 ml toluene with scintillators (i.e. 0.8670 g) to give final concentration of 0.4% PPO and 0.01% dm-POPPOP
(Total vol = 5 ml; total weight of solvent + solute = 4.3830 g)

† 5 ml sample benzene (i.e. 4.3950 g) + butyl PBD weighed in to give final concentration of 1.7% (Total weight of solvent + solute = 4.4450 g)

of sample given under SIZE was directly exposed to the MPT. The results confirm the conclusions reached at the end of each chapter. Teflon vial with butyl-PBD gives demonstrably the best results for ^{14}C . Please note that these experiments were carried out using the Teflon vial of ANU design 'B' (Fig 4). The new Wallac vials have not been tested under similar conditions. However, their performance, relative to each other is illustrated in Fig 6. Their absolute performance, relative to ANU Teflon aluminum has been checked using the newly developed LKB-Wallac Rack-Beta, low level *Kangaroo* counter⁴. The detection efficiency of PTFE vials remains the same, i.e. an increase at least of 4% and up to 10% over glass. However, the ^{14}C window background of the Wallac 7 ml vial was significantly reduced in comparison to the background of the ANU 5 ml vial. This is due to better shielding and better cross-talk suppression of the black copper caps. It is proper to say that the Wallac vials are better performers for low-level counting of ^{14}C because they offer highest efficiency with lowest background and a largest volume flexibility than hitherto achieved by any other counting vial system.

Dry butyl-PBD, dissolved directly in sample benzene, increases efficiency based on signal to background ratio by 25% for any vial. The use of Wallac Teflon-copper vials further improves the overall ^{14}C detection sensitivity by ca. 30%. Selection of proper vials and cocktail for low-level counting must be the easiest way a researcher can significantly improve the performance of his equipment⁴. Pure butyl-PBD is available from Fluka Chemical Company, Switzerland, and the Teflon-copper vials are available from Wallac Oy, Turku, Finland.

Disadvantages. Our aim was to design a vial equally suitable for ^3H and ^{14}C low-level work. To achieve this dual isotope counting role the wall thickness had to be kept $< 1 \text{ mm}^{16}$. We have established that the 0.9 mm wall thickness chosen for the Wallac Teflon-copper vials renders the small volume vials dimensionally unstable. That is, they buckle (bell out) under the weight of the copper shield, an effect especially noticeable with the 7 ml volume. This did not affect their good performance, but it could cause problems in some automatic sample changers.

The empty vial count is also unusually and unexpectedly high. It is higher than any vial tested by us (cf. Fig 6). This reduces the merit of the 20 ml vial for low-level ^3H work (C. Taylor, personal communication), hence also undermines the concept of dual isotope counting advantage.

To achieve dimensional stability, the wall thickness of 3,7 and 15 ml vials needs to be increased. Calf has tested this concept and has shown that an increase up to 7.5 mm wall thickness can be tolerated. He has documented that an increase in wall thickness always resulted in a further improvement of the ^{14}C figure (or factor) of merit³¹.

To improve the performance of the 20 ml vial for ^3H , further investigations are necessary. If, as suspected, the Teflon chosen is the cause of the initial high empty count rate, then the remedy might be easy. In the meantime, the Wallac Teflon-copper 20 ml vial does not serve any useful low-level counting purpose.

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