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EVALUATION AND STATUS OF LIQUID SCINTILLATION COUNTING FOR RADIOCARBON DATING*

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ABSTRACT. An international review of liquid scintillation low-level counting procedures and instrumentation made it possible to assess in detail those elements which lead to high-precision liquid scintillation radiocarbon dating with a Figure of Merit of 32,000. Current research is documented and future possibilities are alluded to.

INTRODUCTION

Liquid scintillation (LS) counters are extremely versatile tools which grew, over the last 33 years, out of a need to detect and measure efficiently low energy β -radioactivity. Most commonly, samples containing ^3H and ^{14}C are placed in a sealed counting vial located between two photomultiplier tubes (PMTs) on a common axis. This assembly is surrounded by a minimal lead shield ($\leq 5\text{cm Pb}$). Sample count rates are those which are coincident between the two PMTs and which fall within a selected pulse height (energy) window. Generally, there are up to 5 selectable windows spanning the energy of 0–2MeV. Pulse height analyses are enabled as the output of the PMTs is proportional to the energy of the ionizing event. The counts falling within each energy window are recorded. Because of interest in and application of β -emitting isotopes to biomedical research, LS counters are produced commercially, in great numbers (hence economically) and by many manufacturers, mainly American and European. The detection efficiency for ^3H and ^{14}C is high with acceptable backgrounds for most uses (Table 1).

For low-level counting of ^{14}C (radiocarbon dating) and ^3H (hydrology), at and below natural equilibria abundance levels, the radioisotope signal to background noise ratio of these multi-purpose multi-user LS counters was just acceptable. To improve their performance for low-level counting, attention was being focused progressively and systematically on sample conversion to counting medium, scintillation cocktails, counting vials, shielding and electronics.

This paper traces, briefly, the development of ^{14}C dating by LS counting from “give me a better counting liquid” (Arnold, 1958, p 129) to “let’s have better commercial counters” (Noakes, 1977, p 189), and leads to the recent development of commercially available LS spectrometers designed specially to cater to ultra low-level, high-precision β -counting.

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TABLE 1
Performance of conventional* LS counters with a ≤ 5 cm Pb shield surrounding the counting vial and the PMTs

Isotope	Efficiency (E) %	Background (B) cpm	Figure of merit E^2/B	Sample C_6H_6 ml
3H	67	~25	~180	20
^{14}C	98	~20	~480	15

* Commercially available, set up to manufacturer specifications with 20ml, ^{40}K free, glass counting vials

THE LIQUID SCINTILLATION COUNTING SYSTEM

The LS counting system consists of a number of discrete components, each one of which needs to be optimized for low-level counting. The components are counting liquid (solvent), scintillation solute (fluorescent molecules), vial, shielding and electronics of counters, data processing, data evaluation, and validation.

Sample Preparation

Scintillation solvent. From the 3H and ^{14}C β -detection point of view, the best scintillation solvent is benzene (C_6H_6) or a mixture of benzene and toluene ($C_6H_5CH_3$). For ^{14}C dating, the benzene synthesis has been almost universally adopted as it enables quantitative transfer from sample C to solvent C and the benzene exhibits almost ideal energy transfer properties (eg, Birks, 1974, p 27).

The sample carbon is first converted to CO_2 , then to acetylene (C_2H_2) which in turn is trimerized catalytically to C_6H_6 . Two acetylene syntheses exist:

1) CO_2 is precipitated as strontium carbonate which is ignited with magnesium to form strontium carbide and C_2H_2 by subsequent hydrolysis (Suess, 1954). In the minority of cases, when this technique is now used, consistent yields are obtained by following the procedures established by McDowell and Ryan (1965).

2a) By far the most common and preferred method is the reaction of CO_2 at elevated temperatures with lithium (Li) forming lithium carbide which upon hydrolysis yields C_2H_2 (Barker, 1953). The reaction was well studied and the procedures improved (Tamers, 1965a; Noakes, Kim & Stipp, 1965; Polach & Stipp, 1967; Tamers, 1975; Burleigh, Hewson & Matthews, 1977). Routinely, 2 to 20g of elemental carbon can be converted with reproducibly high yields ($96 \pm 1\%$) and, using CO_2 gas dilution prior to synthesis, the procedure can be applied with equal effectiveness to 0.1g sample C (Polach, 1969; Polach, Gower & Frazer, 1972).

2b) When samples are large and relatively rich in carbon, a variant of the Barker method is often used. It consists of reacting an organic sample directly with hot Li (Starik, Arslanov & Klener, 1963; Swart, 1964).

Benzene is formed from the C_2H_2 by low temperature catalysis

(Noakes *et al*, 1963). When $\text{CO}_2 \rightarrow \text{C}_6\text{H}_6$ conversion yields are high ($\geq 80\%$) the isotopic fractionation is minimal; a depletion of $\delta^{13}\text{C}$ in benzene by $1.7 \pm 1\%$ was observed (Panarello, Albero & Angiolini, 1983, p 531).

Comment: The most often used Li is supplied by Lithium Corporation of America, Bessemer City, North Carolina; the Vanadium V-0701 catalyst by Harshaw Chemical Co, Beechwood, Ohio. A review of various catalysts suitable for benzene synthesis was presented by Kim (1971, p 967). Tritium measurement by benzene was first reported by Tamers and Bibron (1963). The comparative merit of counting aqueous ^3H suspensions (TRITON-XTM or DIOXANETM) are reported by Williams and Florkowski (1967). Direct LS counting of CO_2 absorbed onto an alkaline scintillator (eg, LUMASORBTM, CARBO-SORBTM) was reported by Eichinger *et al* (1980).

Scintillation solute. The solute acts as an efficient source of photons after accepting energy from the excited solvent molecules due to ionizing events. The most commonly used solute in ^{14}C LS dating is PPO + POPOP dissolved in toluene (1 part) and added to the sample benzene (4 parts), thus forming a scintillation cocktail (Tamers, 1965b). Following a suggestion from Birks, and based on his research (Birks & Poullis, 1972; Birks, 1974), Polach *et al* (1983a, p 518) made a comparative study of various suitable scintillators. They concluded that for low-level ^3H and ^{14}C dry powder butyl-PBD dissolved in sample benzene (15g/l) gave superior and stable performance even under extreme quench conditions.

Counting Vials

The function of a vial is to contain the sample solvent and scintillation solute within the space provided between the two PMTs. It is expected that the vial will have a high photon transmission efficiency, a low self-induced radiation component, and will exhibit no memory. The most common vials are made from borosilicate glass (^{40}K and ^{226}Ra free). Special vials are made from fused silica (quartz), polytetrafluoroethylene (PTFE, TeflonTM) or DelrinTM. Commercially available vials are generally cylindrical and have volumes of 7ml or 22ml.

The International Electrotechnical Commission (IEC) recommends standard dimensions for LS vials (height = $60.5 \pm 2.5\text{mm}$, outside diameter = $27 \pm 1\text{mm}$). There are no known specifications relating to wall thickness, uniformity, weight, or purity of materials. Thus, the most common borosilicate glass vials vary both in efficiency and background count rates, not only based on the source of supply but also within a given batch (Garfinkel *et al*, 1965; Painter, 1974).

Average sample sizes for ^{14}C dating range from 1 to 15ml of sample benzene. To reduce the background of a 20ml vial when only partially filled with a sample, the glass vial is either reduced in size and suitably mounted to retain the overall dimensions required by LS counters or a full size vial is masked, exposing only the actual sample volume to the PMTs (Tamers, 1965b; McDowell & Ryan, 1965; Polach, 1969). Calf (1969) established that Teflon, which is not affected by aromatic solvents gave the best results for ^3H counting and Calf and Polach (1974) investigated Teflon vials, Viton

'O' ring seals and shielding caps for ^{14}C dating. Since then, Teflon vials of improved designs (eg, Noakes, 1977, p 191; Kuc & Rozanski, 1978; Polach *et al* (1983a, p 517) have become available commercially and gained popularity as they are made to specific size requirements with their active volumes placed in the center of the optical axis between PMTs. Quartz vials are used for very small sample counting by Haas (1979) and very large sample counting by Eichinger *et al* (1980). Delrin was successfully used by Schotterer and Oeschger (1980). In terms of optimal performance, Quartz, Teflon, and Delrin are of equal merit, with Teflon-copper vials rapidly displacing the original borosilicate glass vials.

Background Reduction

Background is the observed count rate when counting a sample not containing the nuclide to be assayed and preferably no other radionuclide. Sources of background in LS counting have been comprehensively assessed and reviewed by Horroks (1974, p 198–207) and Gupta and Polach (1985, p 56–85). Based on the work of these authors, we can now summarize the sources of LS background and, additionally, review the actions taken to remedy them (Table 2).

The sources and remedies group themselves into logical units: 1) care in sample preparation, 2) selection of materials, 3) improved shielding (passive and active), 4) electronic optimization, and 5) other physical parame-

TABLE 2
Sources of background in LS counting and recommended remedial actions

Source	Remedy
Natural radioactivity in the materials of scintillation solute, vial and caps, PMTs and shield	Selection of materials
Sample contamination by radionuclide to be assayed or other radioactive material	Care in sample preparation; energy discrimination, pulse shape analysis
Environmental gamma and cosmic radiation	Passive and active shielding; simultaneous β - γ counting
Introduced radioactive sources used for external standardization, automatic quench correction, etc	Thicker shield in counters and directional shielding of source
Radiation-induced X-rays, Cerenkov and secondary electron emissions, fast and thermal neutrons	Graded shielding; energy discrimination
Chemiluminescence and phosphorescence	Selection of solvents and solutes; elimination of UV activation; delayed coincidence monitor or counting of singles (non-coincident mode)
Thermionic and secondary electron emissions (afterpulse) from photocathode and dynodes	Shorter coincidence resolution times, cooling of PMTs, high voltage reduction; dead time of $4\mu\text{s}$, selection of PMTs
PMT crosstalk due to electric discharge and/or incident ionizing events	Better shielding (passive and active), better optical geometry and elimination of light piping. Lesser pulse analysis or pulse height comparison; opaque counting vials
Static electricity	Temperature and humidity control, grounding and positive/negative ionizer
Line noise and transients; RF interference	'High Isolation Transformers'; RF pickup and anticoincidence gating

ters. Here we shall concern ourselves with progress made in the last three units.

Passive shielding. LS counters manufactured in high volume for biomedical research have no need to consider reductions in background beyond those already achieved (Table 1). Good results are obtained with 5cm (or less) of lead surrounding the vials and PMTs; and results comparable to the average gas counters are obtained readily for ^{14}C dating (Table 3, and Polach, 1974, p 167; Polach *et al*, 1983b, p 422). Physical constraints imposed by compact designs generally preclude additions of lead by low-level users, although, when this was done the background was significantly reduced. Commercially available counters using massive (>10cm Pb) and incorporating graded attenuation concepts were first designed for ^3H applications using large volume vials (>25ml) (Noakes, Neary & Spaulding, 1973; Iwakura *et al*, 1979). An assymetric graded shield, claiming significant reduction in weight without loss of attenuation, was proposed by Kojola *et al* (1984, p 494).

Active shielding. Notwithstanding the attenuation of cosmic and environmental radiations by massive graded shielding, some radiation components, such as muons, reach the counting vial. Anderson, Arnold, and Libby (1951) were the first to apply to ^{14}C dating the concept of an anticoincidence counter (guard) located within the shield. This became standard practice in gas proportional counting (eg, review by Mook, 1983). It was used in LS counting for the first time by Pietig and Scharpenseel (1964) and forgotten until it emerged some 12 years later. Then, a number of experimental approaches were tested. Studies involved 1) placing guard counter(s) externally to the shield (Alessio *et al*, 1976; Noakes, 1977, p 197), 2) the use of NaI(Tl) guard crystals within the shield (Noakes, 1977, p

TABLE 3
Factors leading to best performance in ^{14}C dating by LS spectrometry

Function	Best performance is achieved by
C_2H_2 synthesis	Lithium carbide hydrolysis
C_6H_6 synthesis	C_2H_2 trimerization over Vanadium catalyst CO_2 gas dilution prior to synthesis of small samples
Fluor	Butyl-PBD (15g/l) dissolved in sample C_6H_6 No UV activation prior to counting
Counting vial	Teflon, quartz, Delrin, graded in size to suit sample volume, and shielded with copper or lead caps
Counting system shielding	1) Passive, assymetric, graded, Pb, Cd, Cu 2) Active, LS guard, assymetric, covering vials as well as PMT assembly
Electronics	Optimized, high voltage reduction or HI-LO coincidence bias, spectral stabilization, cross talk suppression (lesser pulse or amplitude disparity), RF pickup into A/cos, static noise suppression, cooled counting chamber
Environment	Shielded room (1.5m concrete), air conditioned and temp 17°C and RH 40% controlled, power line transients isolation, clean 'electrical earth' with no other users, ventilated counting chamber
Data analysis	'Random Access' sample, BKG and STD cycle Multiparameter multichannel 'Windowless' software
Data acquisition	Online dedicated personal computer, floppies and hard disk

198; Iwakura *et al.*, 1979), and 3) the use of gas, plastic, or LS guards within the shield (Pietig & Scharpenseel, 1964; Punning & Rajamae, 1975; Broda & Radoszewski, 1982; Jiang *et al.*, 1983; Kojola *et al.*, 1984). Systems 2) and 3) achieve the most significant background reductions; the best of these 72–84%, with anticoincidence on. Some of these are commercially available and, because of their very high merit, find favor with newly established laboratories or as replacements of aging gas or liquid counters.

Electronic optimization. Electronic optimization, other than the anticoincidence guard, aims to reduce noise (hence background) or increase the long-term stability of LS counters. Involved are high voltage or spectral stabilization (Soini, 1975a; Berthold, 1980), high voltage reduction or variable (HI-LO) coincidence bias (Polach *et al.*, 1983b, p 425–434), chemiluminescence monitor (Soini, 1977), lesser pulse analysis (Laney, 1971), amplitude disparity discrimination (Soini, 1975b), RF pickup and channeling the amplified signal into the anticoincidence inhibit line.

Other physical parameters. Performance of LS counters is also enhanced (increased stability and/or reduction of background) when consideration is given to other physical parameters. Thus, balance point counting, selection of PMTs, considerations of light reflecting properties, and physical shape of sample chamber (Butterfield & Polach, 1983; Erikson, Winn & Horrocks, 1983), suppression of line noise (transients) by usage of High Isolation Transformers, elimination of static charge by positive and negative ionizers (Kananen *et al.*, 1984), cooling of PMTs, and temperature and humidity control of rooms in which LS counters are placed. Choice of counting vials and scintillation cocktail is already dealt with in preceding chapters. A detailed review of optimization of electronic and physical parameters affecting modifications to old counters and design parameters of new LS counters is given by Gupta and Polach (1985, p 47–98).

Data Processing Evaluation and Validation

Until the mid-1980s LS and gas counters were just that; they counted the number of events occurring in the region of isotope interest. In gas counting of ^3H and ^{14}C , this includes all events occurring within a region (a window) defined by a lower limit discriminator (LLD), set just above electronic noise, and infinity. In LS counting, due to good energy resolution of these isotopes, the window can be restricted to cover a selected percentage of the specified isotope(s) energy range; for ^{14}C dating, this is typically a 60–80% efficiency window. Such procedures resulted in total gross counts, which could be displayed on scalers and output on lister/printers or other data storage devices. Due to excellent energy resolution of LS counters, these, almost from the onset, included some data evaluation parameters such as channel ratio, external standardization, etc. Even so, once a pulse fell within the predefined LS energy window, its energy information was lost as only its presence was recorded as a gross count. The loss of energy information is significant, in terms of data validation, particularly in low-level counting systems, be they based on gas or liquid β -detection. Whilst the power of energy resolution in LS counting is inherent to the method,

irrespective of sample size, the need for it, hence development of suitable technology, did not surface until the mid-1980s. Then external multichannel analyzers (MCAs) were interfaced to existing counters with some success (eg, Gordon *et al*, 1976). The first interactive and inbuilt MCA-supported LS spectrometer (note shift from word 'counter') was reported by Ring, Nguyen and Everett (1980). The MCA-based energy analysis, coupled with microprocessor control and captive online minicomputers (PCs) with data evaluation software for biomedical research, is now available in the upper range models of LS spectrometers, from every major nuclear instrument manufacturer.

The usefulness of MCA analysis for radiocarbon was first evaluated by Polach *et al* (1984b). Using an external MCA interfaced to existing LS counters, the authors proposed 'Windowless' ^{14}C age determinations and data validation based on software programs they had developed for this purpose. The first spectrometer specially built for low-level counting, incorporating not one but two 2000-channel resolution MCAs ('multiparameter LS spectrometry'), as well as the ^{14}C dating data evaluation software, was built by Polach *et al* (1984a). It seems that their counter represents, today, state-of-the-art ^{14}C dating technology.

RESULTS

An international survey, initiated in 1984 by Lauri Kaihola, University of Turku, Finland, reviews the field of low-level LS counting and spectrometry especially for ^{14}C dating. In this area the response was almost total. ^3H dating was less well covered; the field was not so well known to us and our

TABLE 4
Performance comparison of gas proportional and LS counting systems

Sample	Grams C	S/B*	Special features	Ref**
CO ₂	1.5	19	Surface lab, graded shield, A/cos	1
		28	As above but triple A/cos guard	
		33	U/ground, triple A/cos guard	
		37	Extrapolated best U/G, triple A/Cos	
CO ₂	2.1	40	Underground, γ -free materials	2
		59	As above	
CH ₄	1.5	43	Underground, γ -free materials	3
CO ₂	2 to 6	4 to 10	Worst to average systems	4
C ₆ H ₆	2 to 4	4 to 8	Masked glass, set to manufacturer's specs	5
		1 to 5.7	Teflon-copper vials, electronics optimum	
	4.3	12	EHT low, PMT sel, heavy shield >10cm	6
	4.3	68	As above but deep underground	
	0.08	5	3ml T/Cu vial, anti/cos, asymmetric shield	7
	0.4	24	3ml T/Cu	
	0.8	47	3ml T/Cu	
	2.4	141	3ml T/Cu	
	5.7	116	7ml T/Cu	
	12.2	141	15ml T/Cu	
	16.2	141	20ml T/Cu	

* S/B = signal, 0.95 NBS Ox/background

** 1) Gulliksen & Nydal, 1979, p 181; 2) Stuiver, Robinson & Yang, 1979, p 213; 3) Oeschger *et al*, 1979, p 157; 4) Mook, 1983; 5) Polach *et al*, 1983b, p 422; 6) Calf & Airey, 1982, p 355; 7) Kojola *et al*, in press.

inquiry sheets did not necessarily reach the right audience. Hence, the comments that follow are restricted to ^{14}C LS dating. When comparisons to gas counting are being made, as they must be, they are based on published and cited references.

Based on the survey it can be stated that ^{14}C LS dating is a fast growing field. Since 1975 the majority (some 94%) of newly established dating laboratories have opted for an LS system. Within these laboratories changes are taking place even today, as the LS β -detection methodology and instrumentation are being continuously improved. Thus, it was not considered relevant to tabulate 'who uses what and how' (although this was the initial intention). Such statistical analyses are no longer relevant; instead, a list of steps involved in LS counting, which in the hands of users were most successful, is given in Table 3, and a comparison of results of average and best gas and liquid systems is given in Table 4. Literature reference at this point shall be cited only if they were not already cited in the text above, or if required as proof.

The very high S/B of the best of LS systems, in an above ground environment, with a flexibility to count variable size samples from 80mg to 16g is unique in the history of radiocarbon dating. It has been achieved by minimizing every known factor contributing to background, a concept of total optimization.

PROSPECTS AND CONCLUSIONS

A window to see into the future is given but to a few; hence, predictions as to future trends will be made on promising research in hand rather than anticipated research development. Even so, it must be stated that the present progress which has achieved a background reduction of 84% in LS counting and raised its Figure of Merit from 500 to 32,000 for ^{14}C can no longer be duplicated. Improvements will follow; they will be based on user and peer group evaluation and interaction with manufacturers and on the commercial viability of the suggestions.

Promising are simultaneous α - β - γ LS spectrometry, pulse shape, and rise time analyses to distinguish origin, not only energy, of ionizing events. This may involve NaI(Tl) guards. Already background reductions, using NaI(Tl) guards alone have been achieved equivalent to 5cm of Pb (Noakes, Neary & Spaulding, 1974). Does this lead to small portable, typewriter-size field counters, using CO_2 dissolved in a fluor for first order of magnitude field dates? Perhaps more in demand shall be counters using Microchannel Plate PMTs, the small size, inherent low noise, and high gain of which are ideal for smaller, lighter shielding geometry, hence, leading to very cost-effective systems (Spaulding & Noakes, 1983). Certainly, there shall be developments of liquid scintillators with increased sensitivity to γ -radiation in the low energy range to act as anticoincidence guards; but then we are nibbling at a few percent of background reduction, worthwhile only for the sake of excellence.

Multiparameter multichannel analysis with assymmetric shielding leads to high-precision low-level LS counting. This system is with us now. It opens a new and unprecedented dimension in ^{14}C dating. It promises to open new

applications in other fields. I believe that the rules of low-level counting, as applied to ^{14}C dating and related fields, have already been rewritten.

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