RADIOMETRIC ^{14}C DATING: NEW BACKGROUND ANALYSIS, BASIS OF IMPROVED SYSTEMS

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ABSTRACT. A recent broad study of the background of all types of low-level beta and gamma detectors has now made it possible to analyze its components more reliably and with greater detail. This general analysis is developed further here for gas proportional and liquid scintillation counters used in radiocarbon dating. The background of gas counters, which is dominated by secondary cosmic gamma radiation, is now well understood and can be described quantitatively. The background of liquid counters is less well understood and can only be described semiquantitatively, its analysis resting partly on estimates. Methods to reduce the background of both types of systems are described and their effectiveness discussed. This analysis may help in evaluating the quality of existing systems as well as in designing better ones.

INTRODUCTION

For decades, low-level beta and gamma counting has been a conservative technique, characterized by empirical solutions. It has been more of an art than a science, relying on many recipes that are not supported well enough by experimental evidence. During the development of ultralow-level germanium spectrometers, their background components and methods to reduce them were studied intensively. Over the past 15 years these studies have provided us with invaluable information, which additionally helps us to use with greater confidence the large mass of data from earlier background studies on various types of detectors. This has made a deep impact on the whole field of low-level beta and gamma counting, gradually transforming it to a more exact science (Theodórsson 1996b). The new and reevaluated older information gives us now the opportunity to analyze in greater detail the background components of gas proportional counters (GPC) and liquid scintillation counters (LSC) used for ^{14}C dating.

BACKGROUND COMPONENTS AND EFFECTIVE MASS

Primordial radioactivity, close to the sample detector or in its material, and cosmic rays are the source of the background count rate B of beta and gamma detectors (Oeschger and Wahlen 1975). The background can be divided into the following components:

$$B = \{B_\gamma(\mathrm{Ex}) + B_\gamma(\mathrm{Ct}) + B_\gamma(\mathrm{Rn})\} + \{B_\beta(\mathrm{Rn}) + B_\beta\} + \{B_\gamma(\mathrm{Sr}) + B_N + B_\mu\} \tag{1}$$

where their source is:

B_γ(Ex):	External natural radioactivity in material outside main shield
B_γ(Ct):	Gamma radiation from contamination in the shield and materials inside it
B_γ(Rn):	Radon, which diffuses into cavities in the shield, and its progeny
B_β(Rn):	Radon contamination in sample
B_β:	Beta-active contamination in counter material and glass of the photomultiplier tube (PMT)
B_γ(Sr):	Secondary photons, induced by muons and protons in the shield
B_N:	Neutrons, induced by muons and protons, predominantly in the shielding material
B_μ:	Muons, or muon leakage in systems with anticosmic counters.

Here only components that can contribute significantly will be discussed.

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Reducing the background calls for time-consuming studies and often increases the price of the systems. It is therefore a matter of compromise how far we should extend our effort. It is generally difficult to reduce the background beyond some limit, B_L, which is usually determined by one dominating component, while other components are suppressed to a negligible level or below fixed a tolerance level, for example 10% of B_L.

Before dealing with background components of gas proportional and liquid scintillation counting systems, we need to explain the *wall effect*. Sample detector pulses due to gamma radiation are triggered by primary ionization not only in the detecting medium, which in the present case is either gas or liquid, but also when it occurs in the enclosing wall and the released energetic electron escapes into the detecting medium. The background can therefore be split into a gas component B_g and a wall component B_w, where the primary ionization occurs in the gas and in the wall, respectively. Because the wall effect is small in the vials of LSCs, I focus on GPCs. As both the gas (CO_2) and the wall of the counter (copper) are of materials of relatively low atomic number, the density i of the primary ionization (events per minute and gram of matter) is nearly the same in both materials, and we can write:

$$B_g = i\,M_g \quad \text{and} \quad B_w = i\,t\,A \tag{2}$$

where M_g is the mass of the gas; t is a proportionality factor with a dimension of thickness, measured here in mass area density (g cm^{-2}); and A is the cathode area (cm^2). The inner mass layer tA can be considered as an *effective sensitive wall layer*, as the total number of gamma interactions there has the same value as B_w. The thickness t depends on the energy spectrum of the gamma radiation, and increases with its energy. For the gamma radiation inside a typical shield, t has a value of *ca.* 0.025 g cm^{-2} (Theodórsson 1996b: 213). The total effective sensitive mass of the detector, M_{eff}, is the sum of the mass of the gas and the effective wall layer:

$$M_{eff} = M_g + i\,A \ . \tag{3}$$

A 1.0-liter counter has an inner cathode area of *ca.* 800 cm^2 and an effective wall layer of *ca.* 20 g. When filled to 3 atm of CO_2, the mass of the gas is 4 g and $M_{eff} = 24$ g. This clearly demonstrates the disadvantage of the GPC compared to a LSC, as the sample is <20% of the total mass sensitive to background gamma radiation.

SECONDARY RADIATION

The secondary gamma radiation is generally the source of the dominating background component. This gamma radiation is induced by muons, and to a lesser degree by protons, in the shield. As all low-level beta and gamma detectors are operated inside a similar shield, they are exposed to secondary gamma radiation of practically the same intensity and with the same energy distribution. We have detailed information about this component from studies with ultralow-level germanium spectrometers. The secondary gamma radiation interacts through the same processes with the detecting materials of GPCs, LSCs and germanium diodes, the Compton effect being dominant. The interaction probability per mass unit is nearly the same in all these detectors as they are made of materials of relatively low atomic number. We can therefore expect the background in a broad energy window to be similar per mass unit for detectors of all these three types.

The secondary gamma radiation is predominantly produced by the penetrating muons. Their flux is reduced somewhat by the overlying mass in building materials and eventually also in a layer of earth/rocks. This mass, the *overburden* (M_{ob}), is measured in g cm^{-2} or, more frequently, in meters water equivalent (1 mwe = 100 g cm^{-2}). The attenuation factor of the muon flux, $A_\mu(M_{ob})$, is described by the following empirical equation (Skoro *et al.* 1992):

$$A_\mu(M_{ob}) = 10^{(-1.47 \log(1 + M_{ob}/10))} \quad (4)$$

This equation describes the muon flux attenuation with an accuracy of *ca.* 5% to a depth of 40 mwe. The attenuation of muons and protons (which induce most of the neutrons) is shown *vs.* overburden in Figure 1.

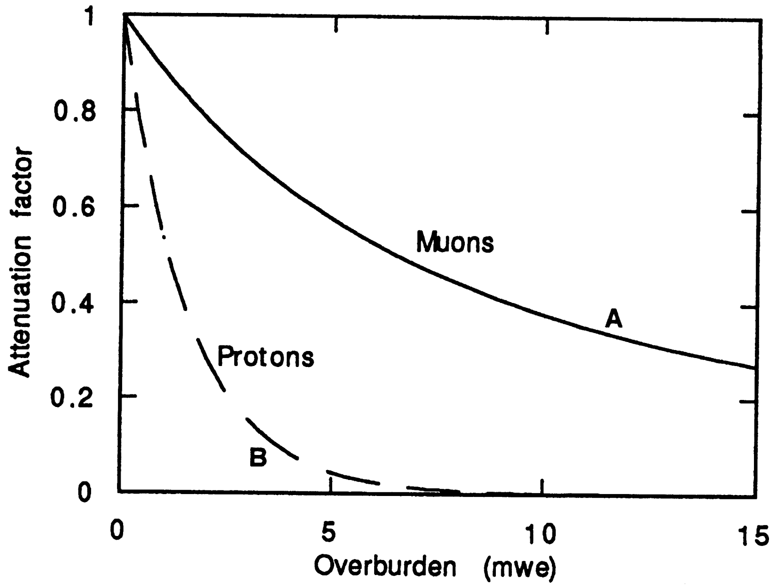

Fig. 1. The relative attenuation of the muon (A) and proton (B) flux *vs.* overburden

BACKGROUND OF GPCS AND ITS REDUCTION

B_γ(Sr): The Component of Secondary Radiation

It has been shown from background studies with a number of good low-level GPC systems without an inner shield and in surface laboratories that this component gives 0.16 cpm per gram of effective mass (Theodórsson 1996b: 221). This component can be reduced by inserting a shielding layer between the guard and sample counter (inner shield). When the main shield is 10 cm of lead, an additional inner lead layer hardly affects the secondary gamma radiation to which the sample detector is exposed. The secondary gamma flux induced in the main shield, which passes the sample detector, is reduced exponentially by an inner lead shield with an attenuation thickness of 2.0 cm (Vojtyla 1995). The total secondary gamma flux to which the sample counter is exposed remains practically unchanged, as this absorption is fully compensated for by the new secondary radiation formed in the inner layer.

Most of the sample detector background pulses coming from gamma photons induced in the inner shield are excluded from the ^{14}C counting channel by the veto signal of the guard counter, *i.e.*, those in coincidence with pulses from the guard counters (prompt pulses) due to the primary event. A fraction, f_d, of these pulses is delayed, however, and they are therefore registered in the background channel. The size of the delayed fraction has a value of *ca.* 0.17 in surface laboratories and seems to decrease with depth, being *ca.* 0.08 in a laboratory with an overburden of 15 mwe (Heusser 1993). This delayed fraction has not been explained. The residual background $B(M_{ob})$ of a low-level GPC at overburden M_{ob} and with an inner shielding layer of thickness s cm, can now be described by the following equation (Theodórsson 1996b: 221):

$$B_\gamma(\mathrm{Sr}) = 0.16\, M_{eff}\, A_\mu\, (f_d + (1 - f_d)\, \mathrm{e}^{-s/2}) \quad \mathrm{cpm}\ . \tag{5}$$

The relative reduction of B_γ(Sr) at varying thickness of the inner shield is shown in Figure 2. According to Equation (5), maximum reduction of B_γ(Sr), 83%, is obtained when all the shielding material is placed inside the guard counter system (Theodórsson and Heusser 1991). This arrangement, where large flat external guard counters cover all sides of the shield except the bottom, has been used for 15 years in ultralow-level germanium spectrometers, where its effectiveness has been well demonstrated (Brodzinski *et al.* 1985). When this arrangement is used, the factor $\mathrm{e}^{-s/2}$ is nearly zero and Equation (5) is reduced to

$$B_\gamma(\mathrm{Sr}) = 0.16\, M_{eff}\, A_\mu\, f_d \quad \mathrm{cpm}\ . \tag{6}$$

No low-level system of this type with either a GPC or a LSC has yet been made.

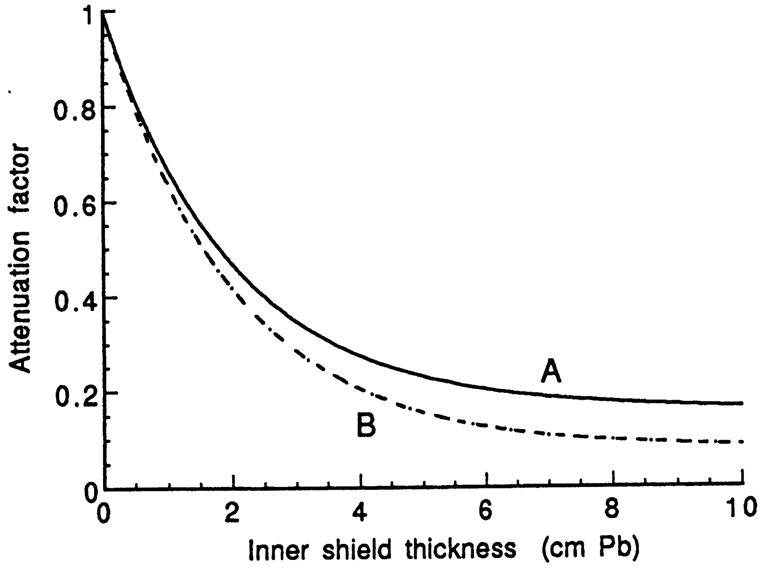

Fig. 2. The relative attenuation of the background component due to secondary gamma radiation at varying thickness of the inner shield in a surface laboratory (A) and at an overburden of 15 mwe (B)

B_γ(Ex): Component Due to External Radioactivity

It has been shown that inside 5 cm of lead this component gives 0.08 cpm per gram of effective mass in detecting materials of low atomic number (Theodórsson 1996b: 156). Its attenuation thickness is equal to that of the most penetrating gamma rays, or 2.0 cm in lead. We therefore have

$$B_\gamma(\mathrm{Ex}) = 1.0\, \mathrm{e}^{-d/2} M_{eff} \quad \mathrm{cpm}\ . \tag{7}$$

In conventional low-level GPC systems, a total thickness of 10 cm of lead is sufficient. When an external shield is used, or if there is a significant overburden, a 12 cm lead shield is necessary to suppress this component to an acceptable value.

It should be noted that B_γ(Ex) is proportional to the concentration of primordial radioactivity in surrounding building materials. The variation of this concentration from one laboratory to another may be estimated from the scattering of values of absorbed terrestrial gamma air dose rate as measured in several countries (UNSCEAR 1982). 80% of the measurements lie in the rather narrow range from 35 to 65 nGy h^{-1}. Significant local deviation from this most common range of intensity may in rare cases affect the selection of the optimum thickness of the shield.

B_γ(Ct) and B_β: Components Due to Gamma and Beta Active Contamination

Extensive studies of the radiopurity of a large variety of materials, made during the development of ultralow-level germanium spectrometers, can now be used to select practically radiopure materials. Electrolytically refined copper (OFHC copper), which is the best material for the counter, is practically free of radiocontamination. Lead is only contaminated with ^{210}Pb (half-life 22 yr), which is a decay product of radium always found in the ore. Bremsstrahlung from the energetic beta particles of ^{210}Bi may contribute significantly to the background. If its concentration is <20 Bq kg^{-1} of lead, its contribution to the background of low-level GPC is negligible (Theodórsson 1996b: 159).

B_γ(Rn): Component Due to Radon in Air

This can be made negligible by minimizing the free space around the sample detector and by ventilating the laboratory well with outside air. The tolerance level of radon is *ca.* 150 Bq m^{-3} (Hedberg and Theodórsson 1995). The concentration in a basement laboratory can easily rise above 1000 Bq m^{-3} if it is not ventilated.

B_β(Rn): Component Due to Radon in the Sample

This can be reduced to a negligible level by storing all samples 3–4 weeks before measurement. Because this calls for a considerable number of gas sample vessels, it would be simpler to omit this storing and instead to correct for an eventual small radon contamination by simultaneously measuring its concentration. This can be done very selectively by delayed coincidence counting (Theodórsson 1996a), as 98% of the alpha pulses from ^{214}Po arrive within 1.0 ms after the beta pulse from ^{214}Bi. The background count rate in this delayed channel will be only a few pulses per day and the ^{214}Po detection efficiency is close to 50%, as the decay products of radon are plated out on the wall of the counter, corresponding to a radon detection efficiency of 25%.

B_N: Neutron Component

The neutrons are mainly produced by protons in the shield. Based on meager data, the neutron component in systems with no paraffin layer in the shield and at zero overburden has been estimated to be 0.5 cpm per liter of CO_2 (Theodórsson 1991). The protons, which are much less penetrating than muons, are attenuated exponentially:

$$I(M_{ob}) = I(0)e^{-M_{ob}/1.6} \tag{8}$$

where $I(M_{ob})$ and $I(0)$ are the proton fluxes at an overburden M_{ob} (mwe) and at zero overburden, respectively (Heusser 1994: 82). The neutron attenuation factor is shown in Figure 1. A typical concrete floor plate has a thickness of 0.5 mwe. Three floor plates, typical for a basement laboratory, therefore reduces the neutron flux by a factor of two.

The elastic cross-section of neutrons with energy from a fraction of an eV to 5 MeV is 5–10 barn in lead, corresponding to a mean free path of 3–6 cm. The neutrons lose only *ca.* 1% of their kinetic energy in each collision. They therefore collide infrequently with lead nuclei and have lost little energy when they diffuse out of the shield. The neutron background component of the sample counter is due to neutron elastic collisions with the nuclei of the carbon and oxygen atoms of the counting gas. The energy spectrum of the neutrons at the instant of formation, as well as the recoil energy of the nuclei they collide with, can be deduced from the background spectra of low-level germanium spectrometers (Theodórsson 1996b: 214). The recoil energy spectrum for ^{12}C and ^{1}H is shown in Figure 3.

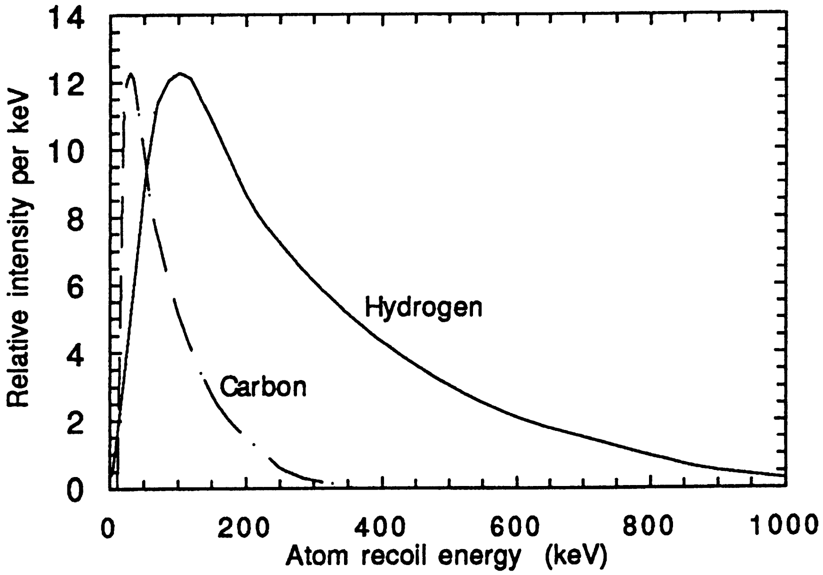

Fig. 3. The recoil energy spectrum for ^{12}C and ^{1}H, hit by energetic cosmic ray neutrons inside a lead shield

The majority of the pulses due to these collisions in CO_2 give a pulse size in the ^{14}C counting window of GPCs (Fig. 3). A layer of paraffin with boric acid is usually inserted inside the main shield to thermalize and absorb the neutrons, following the suggestion of de Vries, who found that a 10 cm paraffin layer reduced the background of his CO_2 counter from 5.3 to 3.0 cpm (de Vries 1957).

As the neutrons lose on average half of their energy in collisions with hydrogen atoms, they only have to make two or three collisions in the paraffin for their energy to fall below the lower level discriminator of the ^{14}C window, which is usually set at a few keV. We can therefore expect that a thinner layer of paraffin would be sufficient. This layer, which inevitably increases greatly the total mass of the shield, is not needed when an external guard is used, which eliminates *ca.* 80% of the neutron pulses.

BACKGROUND COMPONENTS OF LSCs

Almost all LSC systems used earlier for ^{14}C dating were standard commercial units, designed and produced mainly for biomedical tracer work where low background is of secondary importance. Two photomultiplier tubes face the vial and only coincidence pulses are accepted as a valid signal. This significantly reduces the background due to small pulses. The firms that developed these systems naturally published only a part of their studies. Furthermore, the possibility for users to carry out fundamental background studies with these systems is restricted. Reported information about work of this kind is therefore limited and often cannot be interpreted reliably, as important parameters are frequently not given or are insufficiently described, such as the counting window, radioactive contamination in vial and PMTs, overburden, or whether pulse shape or disparity ratio analysis has been applied. Our information and understanding of the background therefore leaves much to be desired.

In the following discussion of the background count rate of LSCs, I assume a sample of 3 mL of benzene (2.4 g) and a ^{14}C detection efficiency of 70%. The effective wall mass of a vial with 3 mL of benzene is estimated to be 0.4 g, and the total effective mass, M_{eff}, is then 2.8 g.

B_γ(Sr)

This component is assumed to have the same value as for a GPC, *i.e.*, that it gives 0.16 cpm per gram of effective mass, as previously stated. In a laboratory with negligible overburden this gives 0.44 cpm for 3 mL of benzene. This seems to be in reasonable agreement with various measured background values. An external guard counter system can reduce this component by a factor of 6 in surface laboratories as in GPC systems. No such system has been made, however, although in 1984 Wallac introduced a new LSC system, the Quantulus™, specially designed for low-level counting work. Its very low background demonstrates the efficiency of the guard counter in reducing the component due to secondary gamma radiation.

B_γ(Ex)

This is given by Equation (7). For a 4-cm-thick shield of lead, which is the most common thickness in conventional LSCs, it is 0.13 cpm g^{-1} or 0.36 cpm for 3.0 mL of benzene.

B_β

This is due to Cerenkov radiation induced by energetic beta particles of primordial radioactivity in the glass of the vial and the photomultiplier tube. This background component, which is usually large in LSCs, can presently be estimated only from the total background and the size of other components. B_β can be reduced by three methods.

1. By selecting glass with low contamination. This is normally in the hands of the manufacturer. Quantitative information about the radiocontamination is scarce. Glass with low potassium concentration has been available for a long time. It is difficult, however, to find raw materials for glass with significantly lower concentrations of Th and U. Some years ago a large project was initiated to produce glass with a very low concentration of primordial radiocontamination for the production of large PMTs to be used in large systems for neutrino detection. This effort has brought us glass with a contamination level 5–10% of that in the glass of in earlier phototubes. This glass is still only used in large special tubes, but it is to be expected that tubes for general LSC will also become available in the near future, but at a price that may be 2–4 times higher than that of present types. Later, vials made of this glass may also become available.

Vials of quartz, practically free of radiocontamination, can be hand-made. Because they are reused in ^{14}C dating as long as they last, their high price is not prohibitive.

2. B_{β} can be reduced by measuring the ratio of the size of the pulses from the two tubes. The Cerenkov pulses will generally be considerably larger in the PMT in which it is induced, giving a high ratio of the larger pulse size to the smaller. Most of the scintillations produced in the benzene sample give a ratio nor far from 1.0. By discarding all events where this ratio, *the disparity ratio*, is larger than, *e.g.*, 2.0, the background is reduced significantly, without seriously affecting the ^{14}C detection efficiency.
3. Cerenkov background pulses due to contamination in the glass can be identified and suppressed through pulse shape analysis. The true signal, the scintillation due to energetic electrons in the scintillator, is very fast; it decays to an insignificant level in a few nanoseconds. The Cerenkov scintillation signal is still faster, but it is accompanied by a slowly decaying fluorescence due to ionization and excitation of glass atoms by the beta particle, having a decay time of a few hundred nanoseconds, adding a tail (or a shower of single electron afterpulses) to the Cerenkov pulse. By discarding all pulses with a tail of this type, B_{β} can be reduced significantly.

The disparity ratio and pulse shape analysis is used both in the low-level LSCs of Packard and in the Quantulus™. Their combined suppression efficiency is convincingly demonstrated in an experiment where the background of a varying volume of benzene was measured in a Quantulus™ (Kaihola *et al.* 1992). When the background count rate is extrapolated to zero volume, the remaining background was <0.05 cpm. As the background due to Cerenkov radiation should be nearly independent of volume, this shows that B_{β} has been reduced to a negligible level.

B_N

Little attention has been given to the neutron background component of LSCs. The energy transfer of the fast cosmic ray neutrons to hydrogen and carbon nuclei in elastic collisions is shown in Figure 3. As the scintillation yield depends not only on the deposited energy, but also on the ionization density, the light output of the scintillation induced by the protons is only 10–20% of that of electrons of the same energy. This light quenching was not taken into account in my earlier analysis (Theodórsson 1996b: 240). Most of the pulses of neutron collisions with carbon atoms therefore fall below the ^{14}C counting channel, but pulses due to recoiling hydrogen atoms lie mostly inside it. Because of the high ionization density of these pulses they have a component with a long decay time, producing a pulse with a tail, which can be eliminated through pulse shape analysis in the same way as alpha pulses are discriminated.

Based on rather meager experimental evidence from systems with small overburden, the neutron component in GPCs with CO_2 has been estimated at 0.27 cpm g^{-1} behind 20 cm of iron. Taking into account that the neutron flux inside a 4-cm-thick shield is *ca.* 40% of that value and that the cross-section of elastic collision with ^{1}H, ^{12}C and ^{18}O are all similar (about 4 barn), we could expect a neutron background component in 3 mL of benzene to be *ca.* 0.2 cpm in a surface laboratory. This estimate needs experimental support.

B_{μ}

Ca. 3 muons traverse the benzene sample per minute. As the energy loss of the muons in benzene is about 150 keV mm^{-1}, only a very small fraction of the muons give a pulse in the ^{14}C window. In addition to this, muons hitting the glass envelope of the PMT give Cerenkov scintillations that may give rise to background pulses.

A LSC in a basement laboratory with three concrete floor plates above, a 4-cm-thick lead shield and with no precaution taken to suppress B_β, *i. e.*, a typical LSC from the 1960s, will have a background of *ca.* 4.0 cpm. My estimate of its background components, based on the discussion above, is the following:

$$B = B_\gamma(\mathrm{Ex}) + B_\beta + B_\gamma(\mathrm{Sr}) + B_N$$
$$B = 0.36 + 3.18 + 0.36 + 0.1 = 4.0\,.$$

All these components can be reduced significantly. If both disparity ratio and pulse shape analysis is used to suppress background pulses, as done in the Quantulus™ and the low-level systems of Packard, we can expect

$$B = 0.38 + 0 + 0.36 + 0 = 0.74\,.$$

If an 8-cm-thick lead shield is used and a liquid scintillation guard counter (Quantulus™), which probably suppresses 80% of the pulses due to secondary radiation, we can expect

$$B = 0.05 + 0 + 0.08 + 0.0 = 0.13\,.$$

This is in reasonable agreement with the measured background of the Quantulus™, which is *ca.* 0.15 cpm in an ordinary laboratory. This analysis should be considered only as a first attempt at a quantitative analysis.

Single PMT Systems

Although single PMT systems are rare in ^{14}C dating, their background will be discussed briefly here because of their future potentialities (Theodórsson 1997). The background components are of the same type as for the conventional systems, but their relative contribution is different.

The disparity ratio naturally cannot be used for background reduction in these systems. It helps, however, that only one tube is used, which can have a smaller diameter, *viz.,* 28 mm, compared to 50 mm in conventional systems (Einarsson 1992). The frequency of Cerenkov pulses due to contamination in the glass can therefore be be expected to be reduced by a factor of *ca.* 5. Einarsson (personal communication) has measured a background value of 1.07 cpm in a system of this type behind 5 cm of lead in a room with three floor plates above. Its background components are estimated as:

$$B = B_\gamma(\mathrm{Ex}) + B_\beta + B_\gamma(\mathrm{Sr}) + B_\mathrm{N}$$
$$B = 0.23 + 0.40 + 0.36 + 0.08 = 1.07\ \mathrm{cpm}\,.$$

This is an attractive background value for this very simple system. All four components can be reduced. That of secondary radiation and neutrons can be reduced by a factor of 6 with an external guard counter system and that due to contamination by a factor of *ca.* 2 through pulse shape analysis. This would give a background of *ca.* 0.4 cpm behind 8 cm of lead. Phototubes with less contamination may also become available in the near future.

CONCLUSION

A coherent analysis of the background components of low-level gas proportional and liquid scintillation counters has been presented. The analysis of the background of GPCs is nearly quantitative, but that for LSCs is semi-quantitative. This analysis can be used to improve existing systems and to design better ones. It also shows where better information is still needed.

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