

NEW POSSIBILITIES FOR ^{14}C MEASUREMENTS BY LIQUID SCINTILLATION COUNTING

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ABSTRACT. Results of intercomparison tests are presented on samples analyzed using gas and liquid scintillation techniques to study the capability of the LKB Quantulus to count an organic solution used for direct absorption of CO_2 and samples with low carbon content. Good agreement was obtained for small samples compared to standard sample size and for the direct absorption compared with the traditional techniques.

INTRODUCTION

During the last decade, new technological contributions such as mini-gas counters and tandem accelerator mass spectrometers for microsamples have opened new horizons for the application of ^{14}C in different areas of research (Sayre *et al*, 1981; Polach *et al*, 1982; Otlet *et al*, 1983; Farwell *et al*, 1983; Donahue *et al*, 1983).

Furthermore, the technology related to ^{14}C counting by liquid scintillation counting has also been improved notably with the appearance of a new generation of liquid scintillation counters (LSC) (Kojola *et al*, 1985; Polach, 1987; Noakes & Valenta, 1989).

This paper presents data that has been gathered to study the capability of LSC, the Quantulus, to count organic solutions used for direct absorption of CO_2 and small samples using the benzene method.

MATERIALS AND METHODS

Samples ranging from close to modern to ca 11,000 BP were used in this study. Most of the samples were run previously by gas counting as CO_2 and aliquots of the same sample gas were used for the tests by liquid scintillation counting.

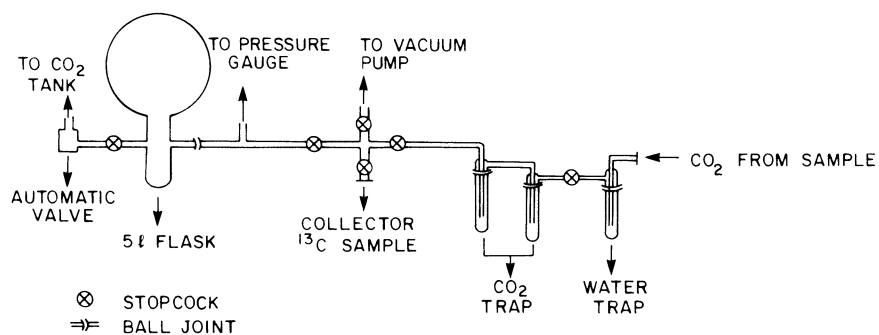
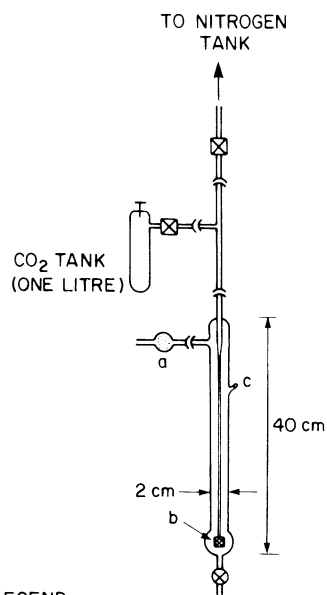
A dilution line was used to add dead CO_2 to the carbon sample to complete the amount of CO_2 required for analyses (2.4g of carbon for benzene synthesis) or 2.21 of CO_2 at STP, that correspond to 1.2g of carbon for the absorption technique (Fig 1).

Samples ranging from 250–350mg of carbon were used to test the Quantulus for small samples using the benzene method. The organic cocktail used for the direct absorption of CO_2 was a mixture of 65% Carbosorb and 35% Permafluor (Canberra Packard). The basic equipment for the CO_2 absorption is presented in Figure 2. A detailed description of this method can be found in Qureshi *et al* (1989).

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Fig 1. CO₂ dilution line**LEGEND :**

- a ASCARITE & DRIERITE TRAP
- b POROUS PLUG
- c SILICONE SEPTUM
- ⊗ NEEDLE VALVE
- ⊗ TEFLON VALVE
- ⇨⇩ ROTULEX BALL JOINT

Fig 2. Preparation line for the absorption of CO₂ in an organic solution

The ^{14}C data is reported in years BP using 95% of the activity of oxalic acid in 1950 as a modern standard. ANU sucrose was also used as a secondary standard specifically for the direct absorption method.

RESULTS AND DISCUSSION

The ^{14}C results for routine and small sample sizes are presented in Table 1. Good agreement is observed in the set of data generated by gas and LS counting. A general consensus about counting precision is that the gas counters are more precise than LS counters; however, with the appearance of the new low-level LS counters, their performance is similar to gas counters. This can also be seen in Table 1.

The data generated using small samples also agree well with data obtained analyzing routine size samples. The performance of the Quantulus for samples with different amounts of carbon is shown in Table 2. Samples with carbon content as low as 100mg can be counted with a detection limit of 22,600 yr for a counting time of 1000 min. It should be noted that these samples were counted in 22ml nylon vials. A better performance should be expected if the 3ml low background teflon vials available for the Quantulus are used.

TABLE 1
Intercomparison of ^{14}C results generated by LSC in the Quantulus, gas counting and AMS for standard and small-size samples

Material*	Lab no.	Date	Sample size (g)
Wood	GSC** - 4155	2440 ± 50	Ca 5
	WAT [†] - 1592	2510 ± 70	2.0 -2.4
	WAT - 1602	2340 ± 150	0.25-0.35
Organics	GSC - 4194	4410 ± 70	Ca 3.5
	WAT - 1594	4480 ± 80	2.0 -2.4
	WAT - 1604	4190 ± 170	0.25-0.35
Shells	GSC - 4201	11,100 ± 90	Ca 5
	WAT - 1593	10,900 ± 100	2.0 -2.4
	WAT - 1609	11,100 ± 400	0.25-0.35
Wood	WAT - 1706	11,850 ± 100	2.0 -2.4
	WAT - 1919	11,700 ± 400	0.25-0.35
DOC, humics	TO* - 817	680 ± 60	Ca 0.005
	WAT - 1792	610 ± 250	0.25-0.35

*The CO_2 that was prepared and counted previously in the GSC Laboratory by proportional gas counting was split for benzene synthesis

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TABLE 2
Quantulus performance for samples with different amounts of carbon, benzene and direct absorption techniques

Vial	1	2	3	4	5	Age limit (yr BP)		Precision (%M)	
	Vol ml	WTC mg	B cpm	E %	FM Aon/ \sqrt{B}	Counting time 1 Kmin	Counting time 4 Kmin	Counting time 1 Kmin	Counting time 4 Kmin
Nylon	3	100	0.4	70	1.49	22,600	28,160	4.6	2.3
22ml benzene		200	0.4	70	2.97	28,140	33,700	3.3	1.6
		400	0.4	70	5.96	33,730	39,300	2.3	1.2
		800	0.4	70	11.92	39,300	44,870	1.6	0.8
		1000	0.4	70	14.89	41,100	46,660	1.5	0.7
		2430	0.4	70	36.20	48,200	53,800	0.9	0.5
Glass	20								
22ml 65% Permafluor + 35% Carbosorb		460	1.4	60	3.19	28,700	34,300	2.3	1.2

1. Volume of sample
2. Weight of elemental carbon
3. Background cpm
4. Counting efficiency
5. ^{14}C dating "Factor of Merit" (Aon/\sqrt{B}), where $Aon = 0.950 \times \text{ref std}$

Precision: defined as the minimum detectable percentile difference which can be distinguished from the Modern Reference Std at 1σ level. It is calculated using: $100 \sqrt{(2/AoT)}$, where T is total counting time.

The intercomparison of ^{14}C results in samples run using the benzene method, gas counting and the CO_2 absorption technique (Table 3) show that the absorption technique has an acceptable accuracy when compared with traditional ones. The only difference is in the analytical error. It should be mentioned that the total counting time used for these samples was on the order of 1000 min. Longer counting times should lower the analytical errors of this method. The detection limit for 1000 min counting time is on the order of 29,000 yr. This technique is attractive because of the simplicity and low cost involved in the analysis (1/3 of the benzene synthesis price). This technique is in use routinely at Waterloo for hydrogeological investigations where high precision is not important, or in cases where fast output is necessary to program future sampling activities.

TABLE 3
Intercomparison of ^{14}C results in samples analyzed using benzene synthesis, gas counting and the CO_2 absorption technique

Material	Lab no.	Date	Technique
Wood	WAT-1182	4050 \pm 70	Benzene
	WAT-1293	3900 \pm 350	Carbosorb
Wood	WAT-1198	9870 \pm 140	Benzene
	WAT-1294	10,650 \pm 620	Carbosorb
Coral	WAT-1193	3780 \pm 120	Benzene
	WAT-1330	3850 \pm 280	Carbosorb
Coral	WAT-1192	6320 \pm 120	Benzene
	WAT-1331	5910 \pm 350	Carbosorb
Organics	GSC-4194	4410 \pm 70	CO_2 gas
	WAT-1471	4490 \pm 310	Carbosorb
Wood	GSC-4352	5720 \pm 70	CO_2 gas
	WAT-1502	5820 \pm 370	Carbosorb
Shells	GSC-4201	11,100 \pm 90	CO_2 gas
	WAT-1486	11,250 \pm 640	Carbosorb

CONCLUSION

The Quantulus expands the possibilities of ^{14}C measurements by LS counting to samples as small as 100mg of carbon. It also makes possible the use of the direct CO_2 absorption technique in a wider age range than previously available, with an accuracy similar to the traditional techniques.

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