[RADIOCARBON, VOL 22, No. 2, 1980, P 442-447]

A MULTI-COUNTER SYSTEM FOR HIGH PRECISION CARBON-14 MEASUREMENTS

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ABSTRACT. A new ^{14}C detector system containing nine, independently working, CO_2 proportional counters is described. The system is designed for a sufficient measuring capacity at a precision level better than $\sigma = \pm 2‰$, which requires a counting time of about one week per sample. The size of the installation requires a simple and economic design of counters and electronics. A single anticoincidence shield for all counters consists of five newly developed flat counters. The modern counting rate (52cpm) is sensitively checked by running Heidelberg sodium carbonate standard samples wth a counting rate of about 10 times modern. A microcomputer (DEC PDP-11/03) is used for data acquisition. Recent developments in laboratory techniques (preparation and gaschromatographic purification of samples) are also reported.

INTRODUCTION

High precision (STD better than about ±3‰) ^{14}C measurements needed for oceanographic applications (Roether and others, 1980) or for tree-ring work (Bruns, Münnich, and Becker, 1980) depend primarily on the good reproducibility of counting efficiency, and not so much on low background. To reach a counting precision of, eg, ±1‰, a total of 10^6 counts is needed. About 14 days are needed to reach this with a counting rate of 50cpm. If a reasonable sample measuring capacity is to be maintained, a multicounter arrangement like the one described below should be used. Because of the many detectors running parallel it is advisable to use a microcomputer for data acquisition.

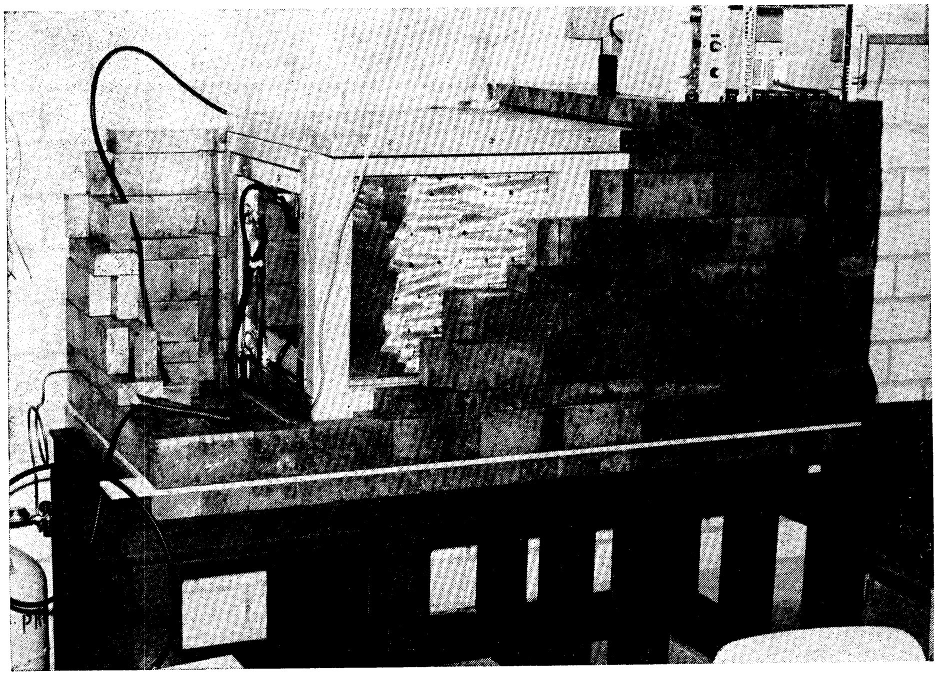
Anticoincidence shield

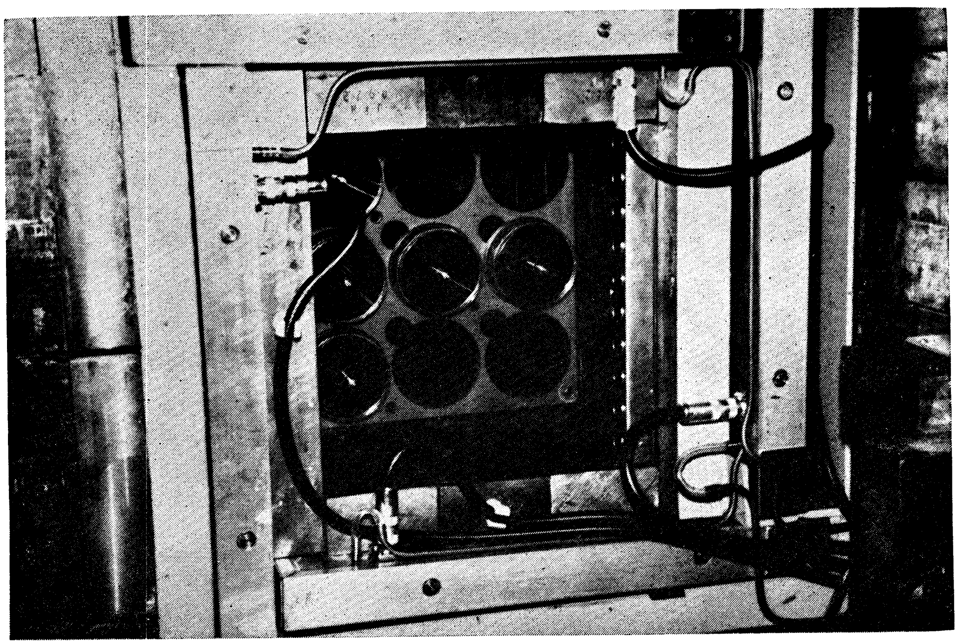
One anticoincidence shield is used for the 9 CO_2 counters. It differs from the traditional cylindrical geometry in that it consists of 5 standardized flat guard counters flushed with camping gas (propane). To maximize its efficiency for cosmic ray muons, being incident under a large angle, the anticoincidence shield is open only on one side, where the pumping leads leave the system (open area $40 \times 40 cm^2$). The shield counter that we designed (pl 1) shows reasonably good counting characteristics with a slope of 1.2%/100V at the muon plateau.

CO_2 proportional counter

Because of the extreme sensitivity of CO_2 counters for electronegative gas impurities the counter construction components should only be metal, quartz, or a similar clean material. We have had success with a very simple counter design (fig 1): tubes made of electrolytic copper, with walls, 2mm thick, 80mm in diameter, and 800mm long, with quartz disks at the ends. All joints between metal and quartz are sealed with pure epoxyresin (Araldit heißhärtend, Ciba AG, Basel). Despite the large difference in expansion coefficients, it is possible to get a nearly strainless junction if the metal thickness is reduced to below 0.3mm. The gas supply tube is brazed to the mantle of the counter. The vacuum/

PLATE 1





The detector system.

https://doi.org/10.1017/S0033822200009747 Published online by Cambridge University Press

pressure valves are all-metal (Nupro, Cleveland, Ohio), as are all other parts of the filling line. In order to reduce field disturbance close to the counter ends, outer metal shielding caps that also contain the preamplifier and a drying agent are used. Wire supports are designed in such a way that the active volume of the counter is nearly equal to its total volume of 4.0L.

All counters are run with a common high voltage (4.3KV, slope of the ^{14}C and muon plateau 1.2%/100V,length 900V). Stability is checked with a weak Ra-226 γ-source inserted to a fixed position in the lead shield. The mean energy loss of the compton electrons produced in the counter wall is constant due to the fixed geometry and gas pressure. (This procedure also serves for checking the gas purity). The sensitivity of this calibration procedure has proven to be sufficient for high precision ^{14}C measurements.

The counter background is between 3cpm and 3.6cpm, depending on the counter position within the shield, and shows reproducible dependence on atmospheric pressure. The background, therefore, can be estimated from the total counting rate with sufficient accuracy. (Slope of the correlation fit around 1).

The modern counting rate (52cpm) is sensitively checked by running samples of Heidelberg sodium carbonate standard. This standard, available since 1956, gives a count rate of about 10 times modern. Our experience shows that extremely high-precision work is greatly facilitated

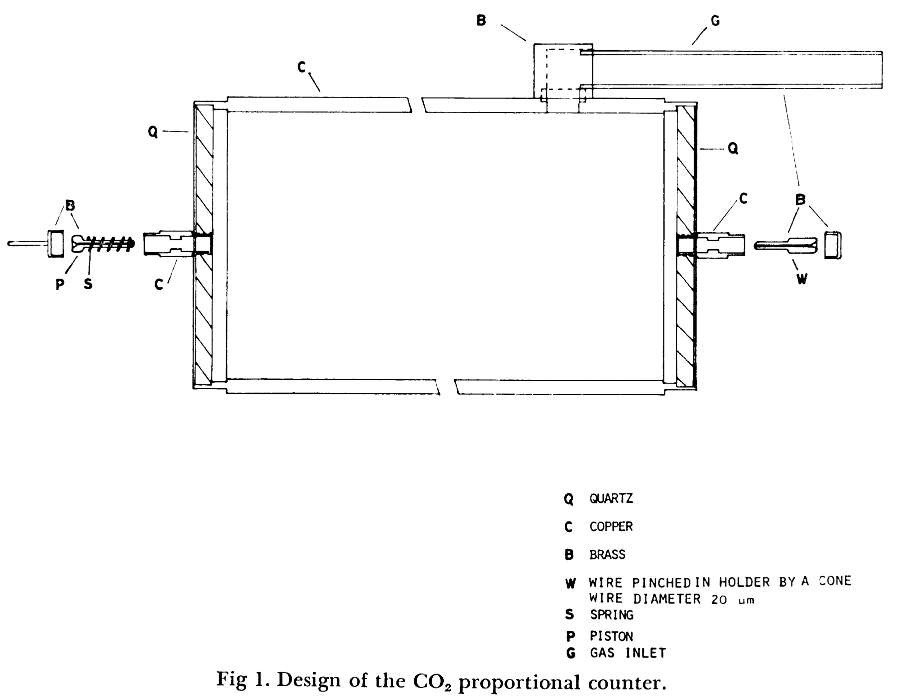


Fig 1. Design of the CO_2 proportional counter.

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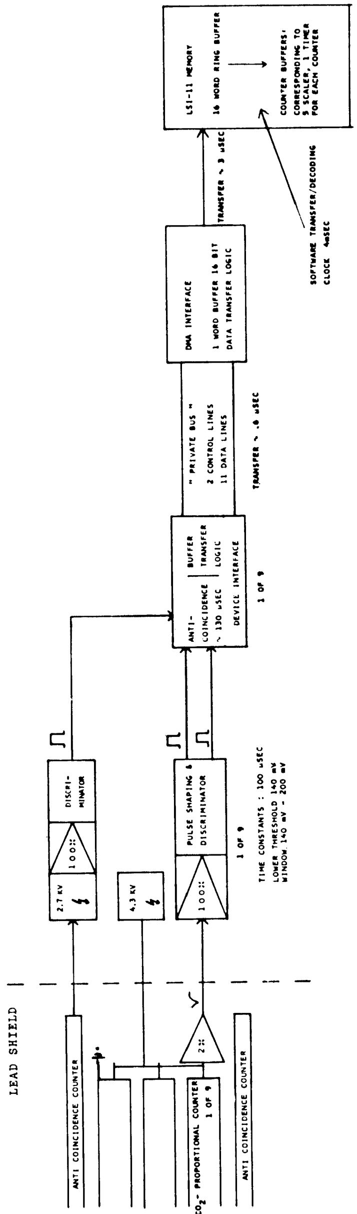

Fig 2. Block diagram of electronics and data acquisition.

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by routinely running sodium carbonate standard samples. These check the whole procedure by going through most of the chemical sample preparation steps.

Electronics and data acquisition

Figure 2 shows our electronics and data acquisition system. The pre-amplifiers are contained in the brass end caps and are directly connected to the wire. Further pulse amplification, shaping and signal discrimination is done with a combined amplifier/discriminator (Berthold, D-7547 Wildbad). The lower discriminator is set at 5 percent of the energy loss median of the compton electrons produced by the γ-source corresponding to about 50KeV energy loss.

Due to the large number of counter channels, the use of conventional scalers would not be economical. Therefore, data is acquired with a microcomputer (PDP-11/03). Digital counter signals and the anticoincidence signal are fed to buffering interfaces, which contain the anticoincidence and the data transfer logic. Information from the counter (address and signals) is transferred to a DMA-interface via a "private bus". Two control lines organize the transfer, priorities being established by the sequence of the counter interfaces along the "private bus". The DMA-interface stores the information in the memory in a 16-word ring-

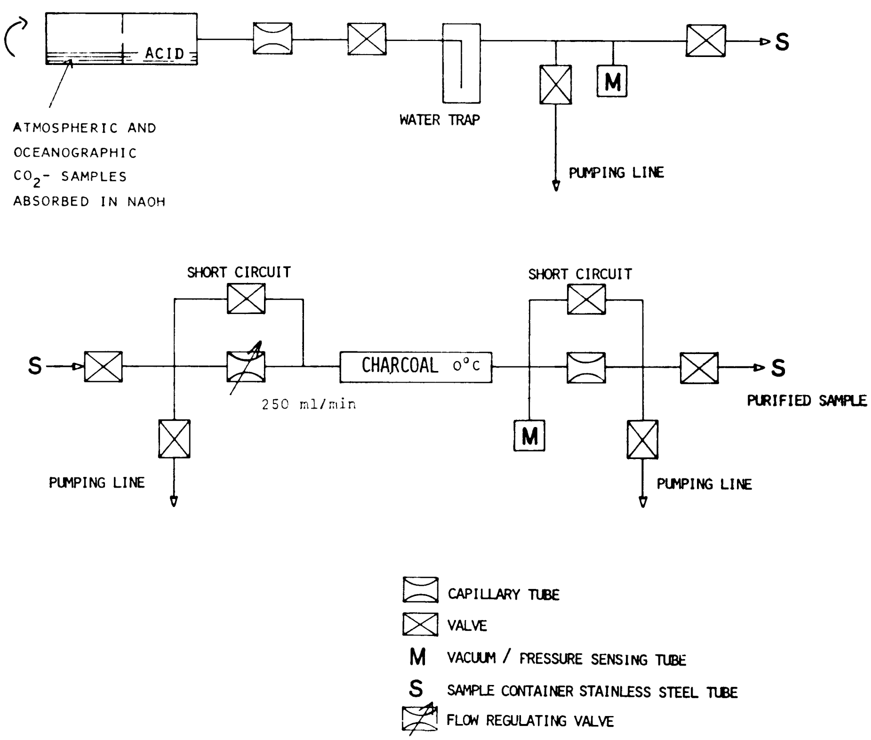

Fig 3. Flow charts of preparation and gaschromatographic purification of samples.

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buffer, which is read out by software every 4msec. The operating system consists of timer, counter, protocol, and system check (4K words, written in Macro 11).

Sample preparation and purification

The amount of CO_2 used is 380 millimoles, purified chromatographically with charcoal (DESOREX F12 Degussa, Ffm, 14g charcoal, diameter of the column, 9mm, flow rate, 250 ml/min, temp 0°C) (fig 3). During this process, water vapor and electronegative contamination is removed. Sample loss is negligible (below 5‰) and no detectable isotope fraction occurs. This cleaning procedure also removes more than 99 percent of the content of the sample (Bruns, 1976). With this procedure, samples can be counted directly after preparation instead of waiting several half-lives of Rn-222 before measurements. Due to the very clean counting and gas storage system (stainless steel tubes with metal valves) sample quality deterioration is normally not observed.

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