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A MULTIPLE PROPORTIONAL ¹⁴C COUNTER SYSTEM FOR MILLIGRAM-SIZED SAMPLES

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ABSTRACT. A system of 10 proportional ¹⁴C counters has been designed. The counters have a volume of 35ml and are operated with CO_2 at a pressure of 1 bar and a voltage of 3300V. The amplified signals of the counters are fed through an analog multiplexer to the inputs of three discriminators. The Geiger signal is fed to a separate discriminator. A logic circuit selects from these discriminators the alpha, beta, muon, and purity counts which are then stored in a microcomputer (ITT 2020 with 48K RAM), where the necessary calculations are also performed. The purity of the gas sample is monitored by counting a part of the muon spectrum. The alpha and beta pulses are stored during 95% of the measuring period; the remaining 5% is used for registration of the muon and purity pulses. The overall accuracy of the system for modern carbon samples is 2% after a counting period of 6 days.

INTRODUCTION

The determination of ¹⁴C/¹²C ratios in very small quantities of carbon would yield results of scientific and/or practical value to such research areas as archaeology, oceanography, environmental studies, climatology, and authentication of museum objects.

Parallel to the development of the mass-spectrometric separation of ¹⁴C and ¹²C ions followed by counting of the ¹⁴C with electrostatic accelerators, conventional proportional counter operation using CO_2 as a counter gas has been extended to very small counters and samples. Although the latter method is slow (counting times up to several weeks may be required for 20mg of carbon) no operator intervention is necessary and many samples may be counted simultaneously.

New installations with small counters are under construction or in the planning stage, eg, at the National Bureau of Standards (Currie, 1978), Brookhaven Laboratory (Harbottle, Sayre, and Stoenner, 1979), Harwell (Sayre et al, 1981) and the Isotope Physics Laboratory of the University of Groningen.

In existing ¹⁴C counting set-ups (Tans and Mook, 1978), the electronic system of one ¹⁴C counter consists of an amplifier, a set of 3 adjustable discriminators, a selection circuit and 4 counting channels, each with a 6-decade counter and display. For a system with several counters, this data acquisition system would become quite complicated. Also, the calibrating procedure of the discriminators would be timeconsuming.

In our application, we developed a relatively small data acquisition system with a microcomputer for 10 small ¹⁴C counters. After the 10 amplifiers for ¹⁴C counter pulses, an analog multiplexer is used with 1 set of 3 adjustable discriminators. After address encoding, a ¹⁴C counter pulse is selected as an alpha, beta, muon, or purity count, which, together with the ¹⁴C counter address is then fed to the peripheral interface adapter (PIA) of the microcomputer where the count is stored in the memory. The dead time is ca 0.2% of the total measuring time. An internal timer selects the alpha-beta counts and the muon-purity counts at a ratio of 95:5.

COUNTERS

Two types of proportional counters were designed (pl l). One type is made from high-purity quartz and uses a gold layer of ca 0.01μ as a cathode. The insulating end pieces are obtained by a shadow technique during vacuum deposition of the gold layer. The small amount of gold which still may be deposited on the end pieces is then removed by careful immersion in aqua regia. The wire support is similar to the one in the counters described by Tans and Nook (1978). The length and diameter of the gold layer are 120 and 19mm,

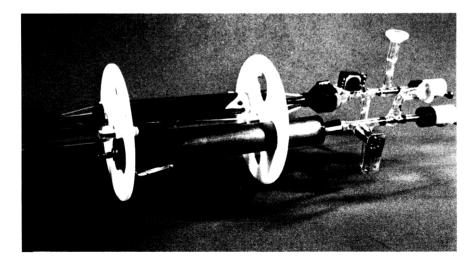


PLATE 1. The two types of proportional counters. The top one is made out of quartz and has a vacuum-deposited gold layer as a cathode. The bottom one is made out of oxygen-free copper.

548

respectively. The other type is made from oxygen-free copper¹. The end pieces are manufactured from high-purity quartz. The anode wire is connected at one side to an Amphenol, hermetically sealed mini-coax receptacle, the outside of which is used as a guard ring. The length and diameter of the cathode are 135 and 20mm respectively. The pressure in both counters is measured by a monolithic pressure transducer with a range of 2 bar and an overall accuracy of 0.5%. Both types have a stainless-steel anode wire with a diameter of $50\mu m$. Both types have a small cold finger at the gas inlet side. The counters remain in their position during sample changing.

The inlet valves were specially designed at the Isotope Physics Laboratory. A stainless-steel plunger with two viton "O"-rings can be moved in a quartz tube with an inner diameter of 4 mm. Novement is made possible by a self-centering brass disk which can rotate in a housing of "Delrin". The counters are surrounded by a cylinder of old lead, 35mm thick, surrounded by 34 Geiger-Müller counters, each 500mm long. Perpendicular to the 34 counters at one end of the cylinder are 7 Geiger-Müller counters 200mm long. The shielding is similar to the one used by Tans and Mook (1978) and also consists of 100mm of lead and a layer of 200mm boron-paraffine around the counters. At the upper part of the shielding is a layer of 200mm of iron. The system is located in the basement of a three-story building. The counters are accessible by front and reardoors made of lead, 100mm thick. These doors move on a rail along the shielding.

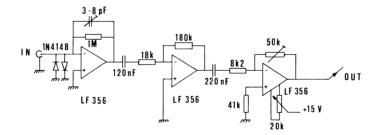
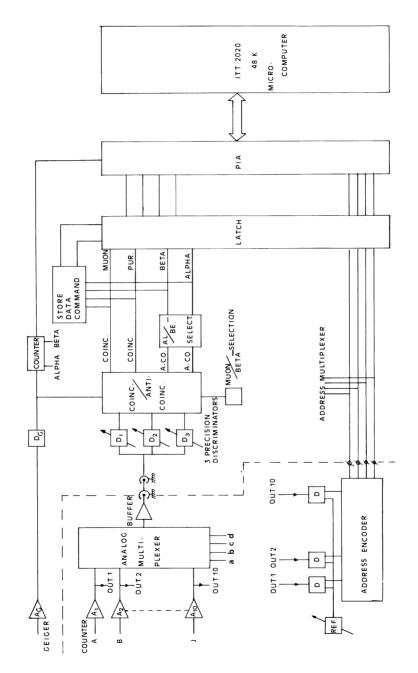


Fig 1. One of the amplifiers for the current pulses from the $^{1\,4}\mathrm{C}$ counters

¹Kindly provided by R Nydal, Radiological Dating Laboratory, Trondheim





DATA ACQUISITION SYSTEM WITH AMPLIFIERS AND ADJUSTABLE DISCRIMINATORS

Figure 1 shows 1 of the 10 amplifiers for the current pulses from the ¹⁴C counters. It consists of a current-tovoltage converter followed by 2 inverting voltage amplifiers. The output voltage is 5.107.iV, where i is the current pulse from the ¹⁴C counter. The slew rate and output noise of the amplifier are $10V/\mu$ sec and 10mV RMS, respectively. Figure 2 shows the total data acquisition system. The output of each amplifier is connected to 1 of 10 rather simple discriminators and an input of the analog multiplexer. The discriminators (D) have a common reference; their outputs generate the ¹⁴C counter address for the multiplexer through an encoder. The output of the multiplexer is fed through an analog buffer to 3 adjustable precision discriminators where the alpha, beta and purity energy levels are set. One of the 3 adjustable discriminators is shown in figure 3. The reference level can be adjusted with R. The hysteresis and response time are 5mV and 110nsec, respectively. The maximum temperature drift of the circuit with amplifier, multiplexer, buffer and adjustable discriminator is $25\mu V/^{\circ}C$. The Geiger pulse is fed to a separate discriminator. The muon and purity pulses are selected from the discriminators D_1 and D_2 . The alpha and beta pulses are in anti-coincidence with the Geiger pulse and are separated in the alpha/beta select circuit. After a pulse appears at one of the alpha, beta, muon or purity lines, the latch will get a delayed strobe pulse and hold the signal with the ¹⁴C counter address information available at the input of the PIA (CCS type 7720A) for 9ms; during this time the data are stored in the memory of the microcomputer. A separate muon/beta selection circuit controls the time ratio between the alpha-beta and muon-purity measurements, after

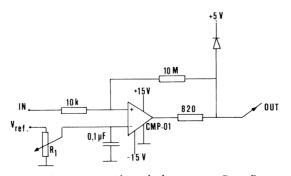


Fig 3. 1 of the 3 adjustable discriminators, D_1 , D_2 , and D_3 . The comparator CMP-01 is from PMI

every 950s of alpha-beta counting follows 50s of muon-purity counting. The alpha, beta, and Geiger pulses are totalized in a preset counter which generates an interrupt in the data acquisition after reaching its preset value, which is then stored in the microcomputer. The circuit left of the dotted line in the diagram of figure 2 is mounted inside the shielding around the Geiger tubes and the ¹⁴C counters. The remaining part of the circuit is mounted on a standard eurocard.

RESULTS

Testing both types of counters for one month yielded the following results:

	Quartz counter	Copper counter
Length	1 20mm	135mm
Cathode diameter	19mm	20mm
Anode diameter	50µm	50µm
Operating pressure	l bar	l bar
Working voltage	3300V	3300V
Muon counting rate (cpm)	14.43 +0.12	15.44 <u>+</u> 0.38
Purity counting rate (cpm)	8.51 +0.09	9.35 +0.06
Net beta counting rate (cpm)	0.219+0.023	0.198+0.075
Background (cpm)	0.273+0.007	0.380+0.010

The alpha and beta pulses were counted separately for correction of the dead time which is ca 0.2% for an alpha-beta count rate of 0.5cpm and a Geiger count rate of 1400cpm.

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REFERENCES

- Currie, LA, 1978, Environmental radiocarbon measurements, in Gove, HE, ed, Conf on radiocarbon dating with accelerators, 1st, Proc: Univ Rochester, p 372-390.
- Harbottle, G, Sayre, EV, and Stoenner, RW, 1979, Dating of small samples by proportional counting: Science, v 206, p 683-685.
- Sayre, EV, Harbottle, G, Stoenner, RW, Otlet, RL, and Evans, GV, 1981, Small gas proportional counter for the ¹⁴C measurement of very small samples, in Methods of low level counting and spectrometry, Proc: Berlin, IAEA Symposium, p 391-407.
- Tans, PP and Mook, WG, 1979, Design, construction and calibration of a high accuracy carbon-14 counting set up: Radiocarbon, v 21, p 22-40.

552