

TABLE 1
COINCIDENCE COUNTING OF C¹⁴-LABELED CAPROIC ACID
DISSOLVED IN A LIQUID SCINTILLATOR

Sample	C^{14} (μc)	Counts/min		Noise background as % of signal
		Uncorrected	Corrected for background	
I Tube voltages 700 and 900				
(1) C^{14} -caproic acid in 0.5% terphenyl in xylene	.031	10,816	10,560	0.3
(2) As (1) + .45 mg cholesterol	.031	9,280	9,024	0.4
(3) As (1) + .45 mg dehydroisoandro- sterone	.031	8,960	8,704	0.4
(4) 0.5% Terphenyl in xylene (back- ground)	—	256	—	
(5) Emptied cell (noise back- ground)	—	32	—	
II Tube voltages 750 and 940				
(1) Same as in I	.031	27,264	25,664	3.2
(2) " " " "	.031	24,128	22,528	3.7
(4) " " " "	—	1,600	—	
(5) " " " "	—	832	—	

emitted energy by the scintillator would solve the problems of geometry and self-absorption that make the usual counting methods technically difficult.

A narrow cell was improvised from flattened glass tubing and placed between the windows of two photomultiplier tubes (RCA 5819). A good optical contact between the cell and the windows was established with Canada balsam. The cell was 4 mm wide internally and 11 mm $\times$ 18 mm on its face sides, with 1-mm walls. A light-tight shield was put around the two photomultipliers and the cell. Fluid could be injected or removed through a small opening covered by black tape. The output pulses of the two photomultipliers were fed into linear amplifiers and discriminator circuits of conventional design. These were followed by a coincidence circuit with a resolving time of about 0.4 μ sec, and the coincident pulses were counted by a 64-scaler, followed by a mechanical register.

Approximately $\frac{1}{2}$ mg of C¹⁴-labeled sodium caproate (7.28 μ c/mg) in 0.5 ml of water was extracted with 2 ml of xylene after the addition of 2 drops of concentrated HCl. An aliquot of the radioactive solution was diluted 1:20 with a liquid scintillator, 0.5% terphenyl in xylene, and 0.45 ml of the diluted solution, containing 0.031 μ c C¹⁴, was injected into the cell to be counted.¹ Similar samples were counted that contained, in addition, nonradioactive steroids in a concentration of 1.0 mg/ml. The background was

¹ The amount of C¹⁴ in the sample was determined by two standard methods, through the kindness of David Feller, Tufts College Medical School.

measured with the liquid scintillator in the cell, and the noise background with the cell emptied.

The results (Table 1) indicate that at the lower voltage 15% of the C¹⁴ disintegrations were counted with a noise background of 0.3% of the count. When the gain of the photomultipliers was raised by increasing the voltages by about 50 v, 37% of the disintegrations were recorded, but the noise background was increased to 3.2% of the count.

The addition of cholesterol to one sample and dehydroisoandrosterone to another caused only a small decrease in the count, suggesting that C¹⁴-labeled steroids can be determined in this way.

The use of two photomultiplier tubes with a coincidence circuit resolved the difficulty in discriminating between the pulses caused by low-energy particles and the background pulses resulting from the dark current of the tube. The noise background, listed as (5) in Table 1, was caused by accidental coincidence of independent dark-current pulses from the two tubes. The more favorable signal-to-noise ratio was obtained with relatively low voltages on the tubes, but at a sacrifice in counting efficiency. To keep the counting efficiency constant, the requirements on the stability of photomultiplier voltage and gain of the linear amplifiers are rather stringent. The unduly high background count when the cell was filled with the liquid scintillator [(4), Table 1] may have been due to radioactive contamination of the apparatus or area.

It would appear from the results that the method is applicable to μ c amounts of C¹⁴. The use of the method requires that the counting efficiency of a sample be determined by adding a radioactive standard to the sample after counting it, or by adding it to a non-radioactive duplicate of the sample.

It is possible that with a less restrictive solvent than xylene, such as dioxane, a larger number of compounds may be counted in this manner.

References

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2. KALLMAN, H. *Ibid.*, 621.

A Recording Warburg Apparatus¹

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During the past 20 years the Warburg manometer and vessel have become standard instruments of the biologist and chemist. As two fine monographs (1,2) on the subject exist, we shall not discuss the directions for use, nor the method of calculating the results. We shall describe a simple and relatively inexpensive modification of the method that gives a graphical recording of the data.

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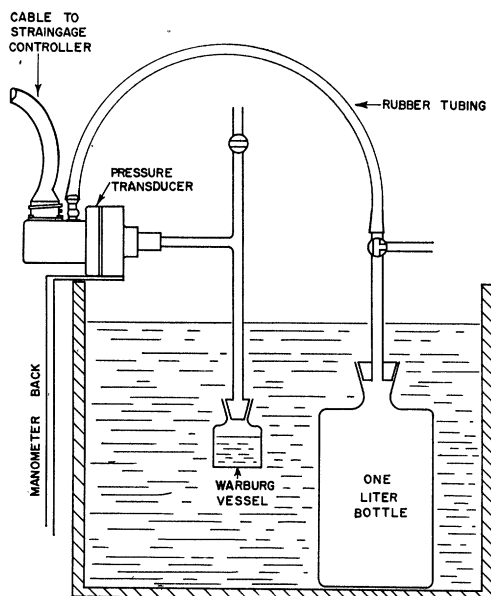


FIG. 1. Schematic diagram of transducer, vessel, and thermostat.

We are using the rectangular Warburg tank made by the American Instrument Company, substituting a mercury thermoregulator in order to have a constant temperature to about $\pm 0.02^\circ \text{C}$. We also use the standard vessels, round, rectangular, conical, with and without wells, etc. The shaking mechanism is used unchanged. The manometers have been replaced by pressure transducers (Statham Laboratories Model P5-0.2 D-125, ± 0.2 psi; cost, approximately \$135). These are mounted on the tops of the old manometer backs and are provided with glass connections to the standard taper joints that hold the vessels and the

stopcocks, just as are the regular manometers (Fig. 1). The pressure transducers can be shaken in a plane normal to their long axis without any effect on their response.

As the ventilating system in our building causes large and irregular changes in air pressure, we do not leave the backs of the pressure transducers open to the atmosphere, as is customarily done with regular manometers, but connect them with rubber tubing to a 1-liter bottle of dry air immersed in the thermostat. This procedure eliminates the need for the "thermo-barometer" (Fig. 1).

The pressure range ± 0.2 psi covered by the transducer corresponds to 133 mm of Brodie's solution. The accuracy and linearity of about 1% of full scale are about the same as when using the regular manometer. The span switch in the controller circuit described below and in Fig. 2 makes it possible to change the range of the instrument at any time.

The strain gauge bridge circuit in the transducer is coupled into a transmitter and controller (Fig. 2) designed by C. A. Mossman, of Oak Ridge National Laboratory. The transmitter and controller contain a source of accurate adjustable voltage for the transducer, a "K" factor setting switch and helipot, a span adjustment, a zero adjustment, and calibration terminals for a calibration resistor. Accompanying each transducer is "F" (calibration factor of the individual transducer—i.e., the output voltage in mv due to a unit input of the variable with 1 v applied to the input terminals), which is multiplied by the pressure rating of the transducer to obtain the "K" factor to be set into the transmitter. The span switch allows the recorder scale to read 100% and 50% of the gauge rating, and the zero adjustment enables the recorder indication to be conveniently positioned on scale. A calibration resistor may be used with the transducer

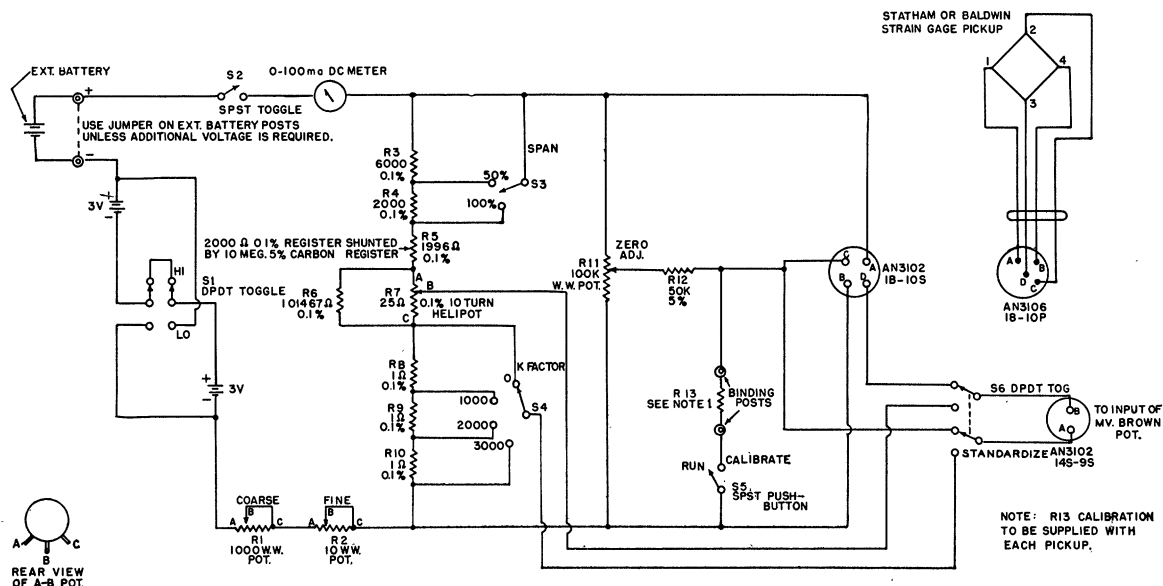


FIG. 2. Wiring diagram of the strain gauge controller.

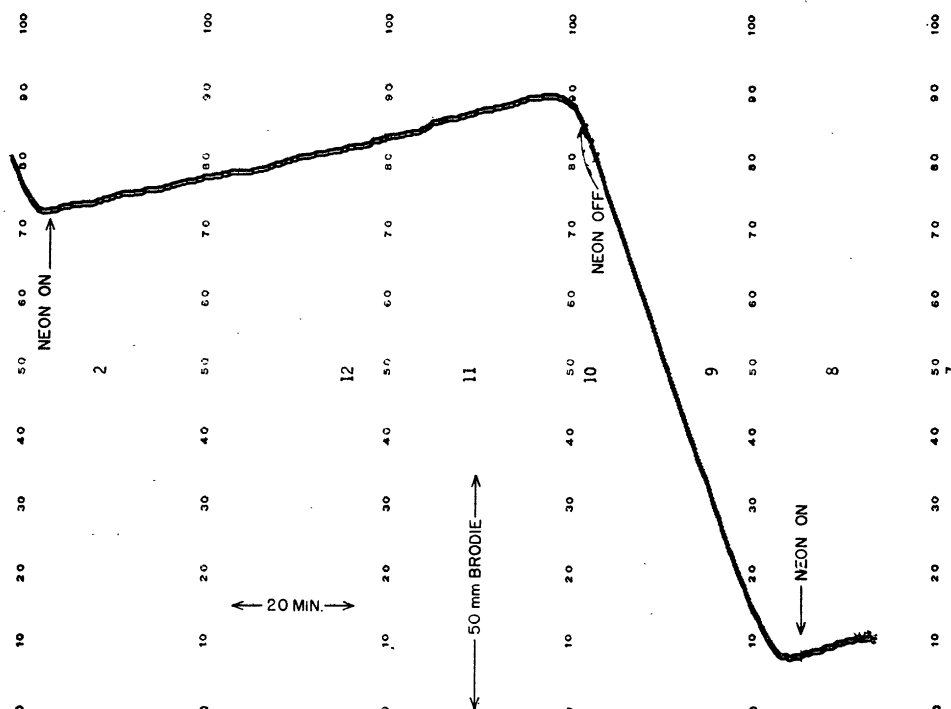


FIG. 3. Example of the performance of the recording Warburg apparatus (photograph of the Brown chart of an experiment in photosynthesis). Time runs to the left.

for the purpose of making an electrical check, which is equivalent to a pressure being exerted on the gauge. This is a means for periodically checking the accuracy of the gauge.

The large gas volume, approximately 6.5 ml, of the transducer head presented two difficulties. First, the gas space must be held to a constant temperature with the same accuracy as the vessel. This can be done by putting the transducer in a small copper box to which is soldered copper tubing. Through the tubing some of the water from the thermostat is pumped; the box is lagged with cellulose sponge. Or the same result can be obtained by putting the transducer much closer to the vessel and running it immersed in the water of the thermostat. In the latter case the cable and the Cannon connection must be made watertight.

The second difficulty comes from the pressure changes associated with varying amounts of water vapor in the gas space of the transducer. A hypothetical experiment will make this clear. Imagine the vessel and transducer filled with dry air and the liquid in the vessel covered with an infinitely thin membrane opaque to water vapor. We shake the vessel and head until they have reached temperature equilibrium. Then we close the stopcock and begin to record the pressure. The pressure in the vessel will be P_0 , atmospheric pressure at the instant the stopcocks are closed. Now imagine that the opaque membrane is removed. Within a few seconds the gas space in the vessel will be saturated with water vapor, and the pressure reading will increase from P_0 to $P_1 = P_0 + \left(\frac{V_g}{V_g + V_h} \right) P_w$,

where V_g = the volume of gas space in the vessel, V_h = the volume of gas space in the transducer, and P_w = vapor pressure of water, as one can show by making use of the gas laws. Water vapor will now slowly diffuse down the glass tube connecting the vessel and the transducer and the pressure will rise until the final pressure given by $P \propto P_0 + P_w$ is reached.

We can calculate the approximate time dependence of this pressure change in the following way: Let C = the concentration of water vapor in the transducer, C_0 = constant concentration of water vapor in the gas space of the vessel, $D = .239 \text{ cm}^2/\text{sec}$ = the diffusion constant of water vapor in air, A = area of tube connecting the vessel and the transducer in cm^2 , and L = length of tube connecting the vessel and the transducer in cm. If we assume that the concentration of water vapor in the tube is a linear function of distance, we have from the definition of the diffusion constant that the flow is given by

$$\text{Flow} = \frac{DA}{L} (C_0 - C).$$

Neglecting the water vapor needed to saturate the tube, the flow is also given by

$$\text{Flow} = V_h \frac{dC}{dt}.$$

Setting these two expressions for the flow equal, we have the differential equation

$$\frac{dC}{dt} + \frac{DA}{V_h L} C = \frac{DA}{V_h L} C_0,$$

which has the solution

$$C = C_0 \left(1 - \text{Exp} \left(- \frac{DA}{V_h L} t \right) \right).$$

Of course, the change in pressure is proportional to C , so that the pressure change will be given by

$$\Delta P = \frac{V_h P_w}{V_g + V_h} \left(1 - \exp \left(- \frac{DA}{V_h L} t \right) \right),$$

where ΔP is the additional pressure due to the diffusion of water vapor into a transducer originally filled with dry air. If we had started with the transducer partially saturated with water, the pressure change would not have been so large, but it would have had the same time dependence. When using the carbonate mixtures introduced by Warburg to hold the carbon-dioxide pressure constant, we have pressure changes connected with the diffusion of carbon dioxide that can be handled in the same way.

The quantity $\frac{V_h L}{DA}$ is a "relaxation time." It is the time that it takes to make all but $\frac{1}{e}$ of the total change.

By making the tube connecting the vessel and the transducer a capillary, the area A can be made so small that the relaxation time will become 1 or 2 days, and thus an experiment can be completed before the pressure has changed very much. This is the method we used with the copper box described above and shown in Fig. 1.

We have also connected the vessel and transducer

by a very short and a very large tube so as to make the relaxation time as short as a minute or so. We used this scheme when the transducer was immersed in the water of the thermostat. Either tube is satisfactory.

The Electro Products Laboratories make a transducer in which the gas space is so small that the diffusion effects are negligible, as they are in the ordinary liquid manometer; but, since the pressure in this instrument is measured by changes in capacity of a small condenser, one side of which is a thin diaphragm, radiofrequency methods must be used. The apparatus necessary to give a signal that can be recorded therefore becomes very expensive indeed.

Fig. 3 is a photograph of the Brown chart of an actual experiment. The vessel contained a thick suspension of *Chlorella* cells in carbonate buffer, so that only changes in oxygen pressure were measured. The light used was neon sign tubing. We see the oxygen production in the light due to photosynthesis and the use of oxygen by respiration in the dark. The time runs to the left on the chart.

References

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National Instrument Conference and Exhibit

From September 10 to 14, Houston, Texas, played host to nearly 7,000 scientists and technicians who attended the conference and exhibit sponsored by the **Instrument Society of America**. Other participating organizations were the Industrial Instruments and Regulators Division, ASME; Instruments and Measurements Committee and the Geophysical Technical Group, AIEE; American Institute of Physics; Institute of Radio Engineers; Scientific Apparatus Makers Association; National Association of Corrosion Engineers; Gulf Coast Spectroscopic Group; The Pipe Liners Club; A & M College of Texas; Research and Development Board; and the National Bureau of Standards.

In 27 technical sessions spread over the five days of the conference, the subjects considered ranged from general surveys of electron diffraction methods (W. O. Milligan) and infrared instrumentation (Van Zandt Williams) to descriptions of a new precision barometer (C. A. Heiland), instruments used in laboratory studies of corrosion (M. A. Judah), an electrical speedometer (J. J. Groncki), and the inevitable computers with their expanding uses (J. H. Curtiss, F. E. Brooks, Jr., Gregory Tobin).

As might be expected at a meeting deep in the heart of Texas, there was some preoccupation with problems peculiar to the petroleum industry. The Pipe Liners Club was concerned with the transmission of

oil and gas; but other organizations less obviously interested in petroleum and its production sponsored programs, or panel discussions, or individual papers on "Physics and Exploration for Petroleum" (M. M. Slotnick, for the American Institute of Physics); on magnetometers (Walter Ruska, for ASME; L. J. Neuman, for AIEE); on refinery control problems (E. D. Mattix, for Texas A & M). Indeed, the titles of papers presented reveal a very lively and practical interest in oil from the time it is little more than a hope in the oil geologist's mind until it leaves the refinery, fractionated and ready for the most highly specialized uses.

To a considerable degree the flavor of the conference was reflected in the exhibit, which occupied 26,000 square feet of floor space in the Sam Houston Coliseum. More than 150 manufacturers displayed equipment that contributed much to the theme of the meeting—"Instrumentation and Our Security."

Developments in Europe were not overlooked, for A. O. Beckman, who was elected president of the ISA at the Houston meeting, addressed the members of the society on "The Instrument Industry of Europe." Other officers elected to vice presidencies at the meeting are D. Boyd, Universal Oil Products Co., and R. Sheen, Milton Roy Co. Continuing officers are W. A. Wildhack, National Bureau of Standards, vice president; N. L. Isenhour, Oak Ridge National Laboratories, vice president; G. R. Feeley, Trinity Equipment Co., treasurer; R. J. Rimbach, Instruments Publishing Co., executive secretary.