IN THE LABORATORY

On the Use of the Campbell-Pressman Lyophilizing Apparatus for Urinary Extractives

HERBERT S. STRICKLER¹ and C. BOYD SHAFFER

Department of Physiological Chemistry, School of Medicine, and Mellon Institute, University of Pittsburgh

In a recent publication, Friedgood, et al. (2) described the use of the Campbell-Pressman (1) apparatus for the concentration and preservation of urinary substances by lyophilization. We have used this apparatus with an adaptor and a special flask (Fig. 1) for the evaporation,



FIG. 1. Diagram of the Claisen-type flask used with the lyophil apparatus. A variety of sizes, ranging in capacity from 250 ml to 1 liter, have been found suitable. This flask is attached to one of the joints of the apparatus by a short, angular adaptor bearing the corresponding ground joint at either end. The adaptor maintains the vertical position of the flask.

at room temperature or below, of urinary extracts, e.g. n-butyl alcohol extracts as encountered in the method of Talbot (3). This flask is filled from the neck. Bumping is effectively controlled by admitting a small amount of air (or other gas) through the sealed-in capillary tube, the flow being regulated by means of thermometer tubing

¹Present address: Westinghouse Research Laboratories, East Pittsburgh.

SCIENCE, January 16, 1948

or other fine capillary attached with rubber tubing to the flask capillary. Further control may be provided by a screw clamp on the rubber tubing. During such evaporation a receiver for distillate is placed on the bottom joint of the apparatus, since liquid may drop down as solid accumulates on the condenser cone.

In the use of the lyophil apparatus with small flasks or tubes to remove organic solvents from mixtures such as those resulting from the acetylation of steroids, we noted a tendency for some light, flaky material to be carried up into the condenser section. To catch this material, we used glass wool, inserted into the mouth of the flask. It is possible that coarse glass cloth on a platinum wire ring would be preferable, since the tuft of glass wool sometimes loosens and is carried over.

We have also found the apparatus useful for the removal of water from the mixture remaining after enzymic hydrolysis of small amounts of steroid glucuronides, preparatory to extraction with organic solvents.

Finally, it should be noted that care must be exercised to remove lubricating grease from the joints before further processing of the sample.

References

- 1. CAMPBELL, D. H., and PRESSMAN, D. Science, 1944, 99, 285.
- FRIEDGOOD, H. B., HAAGEN-SMIT, A. J., GARST, J. B., and STEINITZ, L. Science, 1947, 105, 99.
- TALBOT, N. B., and EITINGON, I. V. J. biol. Chem., 1944, 154, 605.

In Vivo Geiger-Müller Gamma-Ray Counter for Radioisotope Distribution Studies

NELLO PACE,¹ ROBERT LOEVINGER, and ENRIQUE STRAJMAN²

Division of Medical Physics, University of California, Berkeley

One of the important applications of radioisotopes to biological research is the *in vivo* detection of the distribution of atomic species within an organism with respect to both space and time. This has usually been done by placing an ordinary Geiger-Müller tube on the surface of the organism and observing the counting rate following the administration of a radioisotope. The technic of *in vivo* radioisotope study was apparently first used by Blumgart and Yens (1), and there has followed the development of special counter tubes for the purpose. For

 $^{\rm 1}$ Under Contract N7 ONR-295 with the Office of Naval Research.

²Aided by a grant from the International Cancer Research Foundation. example, Curtiss (3) has described a very small counter suitable for insertion into tissues; Brown, Good, and Evans (2), a counter for continuous recording of radioactive gas concentrations in expired air; and Strajman (5), a small beta counter for tissue surface measurements.

A compact gamma counting tube and lead shield assembly has been developed in this laboratory which has proved convenient for use with man and the larger experimental animals. The entire unit is shown schematically in



FIG. 1. Schematic assembly of counter tube and shield, with cross section showing tube construction.

Fig. 1, and details of the tube construction are given in the inset. The tube body is made of copper or brass with a $\frac{1}{16}$ wall which serves as the cathode. The inner diameter of the tube is $\frac{1}{3}$ ", and the inner length is $\frac{3}{4}$ ". The outer length is 1". The central wire, of 3-mil tungsten with a glass bead fused at one end, serves as the anode. A thin, 3-4 mg./cm² sheet of mica is bonded with apiezon wax to the inner surface of the copper disc forming the lower end of the tube, to insulate the disc from the cathode and anode, and the whole is sealed to the tube body with de Khotinsky wax. The copper disc, $\frac{1}{16}$ " thick and 1" in diameter, acts as the counting surface of the tube. The upper end of the tube is drilled in the center to accommodate a short length of glass capillary tubing which is waxed in place and serves to insulate the anode wire. The upper end of the tube is also drilled near the periphery for a piece of ordinary 6-mm pyrex tubing through which the counter is evacuated and filled. The tube, after being pumped down at least overnight at a pressure of $5-10 \mu$, is tested for leaks and then filled with 1 cm Hg of ethyl alcohol vapor and 9 cm Hg of argon. The tubing is then sealed off as shown in Fig. 1.

Tubes prepared in this fashion have a counting voltage of 1,000-1,100 v, and a plateau 200-400 v long with a slope of 2-3%/100 v. The relative sensitivity of various tubes to a standard source is within $\pm 10\%$. The background, in the lead shield, varies from 5 to 50 counts/min, but is constant for any given tube. The reason for this variation is not clear. It may be due to either a discharge phenomenon or contamination somewhere in the construction. Most of the tubes have a low rather than high background. The life of the tubes has not been determined, but appears to be at least of the order of 106 total counts. The resolving time has been determined to be $1.0-1.5 \times 10^{-5}$ min.

Generally more satisfactory performance of the tubes has been obtained with the use of a Neher-Harper preamplifier stage rather than with the Neher-Pickering circuit. When the Neher-Harper preamplifier is used, it is necessary to insulate the tube from the lead shield and to ground the latter. Rubber sheeting is wrapped around the counter body, and the tube rests on a fairly thin sheet



FIG. 2. Cross section of geometrical field of counter tube and shield assembly showing counting efficiency within the field for 0.5 Mev gamma radiation from a homogeneous tissue source.

of mica at the top of the collimator opening in the lead shield. The remainder of the tube space in the lead shield is filled with cotton to prevent motion of the tube inside the shield.

The lead shield was designed so that the counter tube would be surrounded by 1" of lead, except for a collimator opening through which the counting is done. The collimator tube is $\frac{1}{4}$ " in diameter and 2" long. The entire shield is $3\frac{1}{3}$ " in diameter and $5\frac{1}{2}$ " long. As may be seen from Fig. 2, the counting field comprises a cone with a vertex angle of approximately 47°, and organs such as the heart and liver can be well isolated from surrounding structures by this geometry. On the basis of a theoreti-

cal treatment by Strajman (6) it is possible to calculate the characteristics of the tube geometry with respect to the in vivo counting behavior. It may be shown that, as the thickness of a homogeneous source of radioactivity is increased, the counting rate approaches a limiting value. The relationship may be resolved into such factors as the geometry of the counting assembly and the energy and self-absorption, including scattering, of the source. An empirical curve comprising these factors may be constructed which expresses the counting rate at any source thickness as a percentage of the limiting maximal counting rate at infinite thickness. Such a curve was derived for a gamma emitter of 0.5 Mev homogeneously distributed in a medium of specific gravity 1.05, and several values from the curve have been placed at corresponding distances from the tube in Fig. 2. Because of lack of complete data at present, the effect of scattering has been approximated in this case.

The curve has been verified experimentally at several points by counting the activity of various thicknesses of a solution of $K_2C^{11}O_3$. C¹¹ is a positron emitter (4) which therefore exhibits an annihilation gamma energy of 0.51 Mev. It is this energy which may be utilized for *in vivo* measurement of C¹¹.

As many as four of the counting assemblies have been used simultaneously to record distribution of a radioisotope in various parts of the body. This has been done

conveniently by placing four mechanical registers in juxtaposition with an electrical timer reading to 0.01 min on a panel and photographing them with an ordinary 35-mm camera at the desired time intervals. Good results have been obtained with CO tagged with C¹¹ and with gold colloid tagged with Au¹⁹⁸, and it is to be presumed that the performance would be satisfactory with any gamma emitter of relatively low energy. The thickness of the lead shield and the dimensions of the collimator may, of course, be varied to meet individual requirements. It has been found convenient, for measurement of experiment background, to insert a lead plug into the collimator with the counter in position on the body surface, thus taking into account radiation which may penetrate the shield. This has been found to be insignificant with gamma energies of 0.5 Mev, but becomes appreciable with energies greater than 1 Mev.

References

- 1. BLUMGART, H. L., and YENS, O. C. J. clin. Invest., 1927, 4, 1.
- BROWN, S. C., GOOD, W. M., and EVANS, R. D. Rev. sci. Instr., 1945, 16, 125.
- 3. CURTISS, L. F. J. Res. nat. Bur. Stand., 1943, 30, 157.
- DELSASSO, L. A., WHITE, M. G., BARKAS, W., and CREUTZ, E. C. Phys. Rev., 1940, 58, 586.
- 5. STRAJMAN, E. Rev. sci. Instr., 1946, 17, 232.
- 6. STRAJMAN, E. Unpublished observations.

Book Reviews

College physics: mechanics, heat and sound. (Pt. 1.) Francis Weston Sears and Mark W. Zemansky. Cambridge, Mass.: Addison-Wesley, 1947. Pp. 383. (Illustrated.) \$3.50.

This text is essentially an abridged version of Sears' *Principles of physics I.* It is the first volume of two, and the purposes of the abridgement are to reduce a two-year course to one year and to eliminate the use of the calculus in the course. Therefore, a number of chapters have been removed, and a number of topics included in the parent volume have either been neglected or treated more briefly; in addition, all elements of the text requiring a knowledge of the calculus have been removed or rewritten using limit or averaging concepts.

The replacement of the calculus by other methods has been done very skillfully and the revision therefore suffers very little in comparison with its model. The presentation of the principles of physics is indeed, for some topics, somewhat improved by the substitution used.

The general scope of the text has not been further restricted, but for the purposes of a review it is important to list the specific, major topics that were either bodily removed or severely curtailed. These are: the concept of the resultant of a set of concurrent forces; nonuniform acceleration; center of mass and the theorems and problems on the motion of the center of mass; the chapter on work (rewritten; however, it now includes a section on simple machines); the principle of virtual work; the chapter on gravitation (reduced to several paragraphs in the chapter on weight and mass); discussion of the Poisson ratio and the Saint-Venant formula for circular cylinders; Lissajous figures, damped harmonic motion, and resonance phenomena; two-dimensional impacts; the chapter on hydrostatics (somewhat rearranged to give a smoother presentation); surface tension and the Poiseuille formula; discussion of entropy; the chapter on kinetic theory.

Special mention is now made that the reduction of the section on heat has resulted in an improvement of the treatment of the first law. There is now a chapter on the first law and this includes several applications and illustrations previously separated into different chapters. The result is more satisfactory.

The textbook may be classified as excellent and as complete as can be expected for application to a one-year course in college physics.

Columbia University

WILLIAM A. NIERENBERG