On the basis of these studies and observations, we conclude that there is no evidence to support the existence of a beta form of ice.

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The Preparation of Gas for Assay of Tritium in Organic Compounds[1]

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A one-step method has been developed for the quantitative conversion of tritium in organic compounds to a mixture of methane and hydrogen by heating with zinc,[2] nickelic oxide, and water in a sealed tube at 640° C. This mixture has been used for ion current measurements and should also be satisfactory for use in internal gas counters (*1*). Results obtained by this method are equal in accuracy and reproducibility to those obtained by conventional procedures (*2–6*) based on combustion. Furthermore, tritium is retained (to the extent of 1.5%) only in the reaction tube, which is discarded after a single analysis. The absence of "memory" (*6, 7*) and the simplicity of the procedure reduce considerably the time per determination, especially in the analysis of samples having widely different concentrations of tritium.

Reaction tubes 17 cm long with break-tips are made from 1.1-cm OD Pyrex 1720 glass tubing.[3] In each tube are placed 1 g of zinc (Baker and Adamson, 30 mesh), 100 mg of nickelic oxide (Baker's Analyzed, gray-black powder), 5–6 mg of water, and a 5- to 10-mg sample of the tritium compound. Water and liquid samples are introduced in small sealed ampules with bent tips, solid samples in porcelain boats. The reaction tubes are constricted, evacuated to a pressure of 1 mm, sealed off, and agitated until the ampules are broken and the contents are evenly distributed. With asbestos-covered tongs, the tubes are placed horizontally in a furnace at 640 ± 10° C and removed after 3 hr.

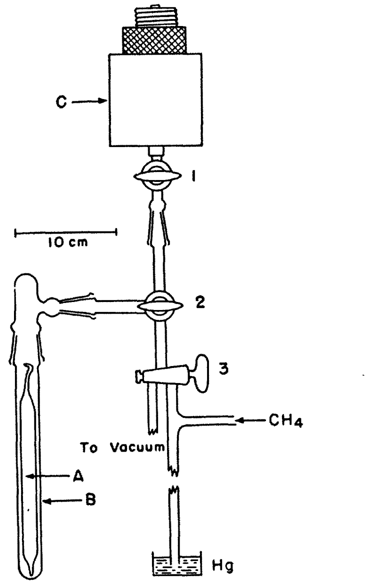

Fig. 1. Apparatus for analysis of gas samples.

The apparatus shown in Fig. 1 has proved convenient in analysis of the gas samples. A reaction tube, A, is placed in tube B, which is then evacuated, closed off, and inverted to break the tip of tube A. The gas is expanded into an evacuated ionization chamber C, of the Borkowski type (*5, 8, 9*); the fraction introduced is calculated from the known volumes, corrected for the displacement of gas by tube A. Alternatively, all the gas may be transferred from tube B to the chamber by means of a Toepler pump. The chamber is filled to atmospheric pressure by the addition of methane and the ion current collected at 360 v is measured with a vibrating reed electrometer.[4]

The results of analyses on representative tritium compounds by the zinc fusion technique are shown in

TABLE 1

Compound*	Tritium, μc/mM	
	Zinc fusion†	Combustion
HOT	325.9 ± 1.1	327.0
CH_3CHTOH	11.65 ± 0.06	11.59
$CH_3CHTOOCC_6H_4NO_2$	11.52 ± 0.05	11.59
$CH_3CT(OH)CH_3$	19.74 ± 0.08	19.42
C_6H_5CHTOH	10.69 ± 0.05	10.53
$C_6H_5CHTOOCC_6H_3(NO_2)_2$	10.72 ± 0.04	10.53
$C_6H_4TCH_3$—(o)	4.90 ± 0.01	4.87
$C_6H_5CT{=}NNHC_6H_3(NO_2)_2$	9.69 ± 0.01	—
$BrCH_2CHTC_6H_4NO_2$	19.89 ± 0.15	19.63
$C(CH_3)_2(NO_2)CHTC_6H_4NO_2$	11.09 ± 0.03	10.98
Cholesterol	15.54 ± 0.08	15.31
Estradiol	28.39 ± 0.23	27.52
Dihydroxypregnanedione	5.35 ± 0.01	5.23
$(C_6H_5)_3SiT$	182.4 ± 0.3	184.0‡

* We are indebted to T. F. Gallagher and D. K. Fukushima for the samples of tritium-labeled steroids.

† Values include a correction of 1.5% for the tritium retained by the reaction tube. The error is the standard deviation of several analyses per compound.

‡ Hydrogen evolved by alkaline hydrolysis.

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[1] A more detailed presentation of this material can be obtained in an Argonne National Laboratory Document, ANL-5056, June, 1953.

[2] This investigation was prompted by the conclusion of T. S. Lee, University of Chicago, that in the determination of oxygen in organic compounds by heating with zinc, the hydrogen appears as methane or hydrogen.

[3] Tubing of Pyrex 1720 glass is available on special order from Corning Glass Works, Corning, N. Y. Quartz and Vycor are unsatisfactory because of their permeability to hydrogen; ordinary Pyrex 7740 is not sufficiently heat resistant.

[4] Applied Physics Corporation, Pasadena, Calif., Model 30.

Table 1 to be 101.0 ± 1.1% of those obtained by a procedure (*5*) based on combustion and conversion of the water to hydrogen. The standard error of an analysis is 0.6% for the zinc fusion method and 0.7% for the combustion method.

Additional information on the scope of the method has been obtained by experiments in which the reaction tubes contained tritiated water and unlabeled organic compounds, including pyridine, thiophene, urea, cholesterol, and various aromatic and aliphatic amines, hydrocarbons, and halides. In such experiments, the recovery of tritium as noncondensible gas is a measure of the completeness of decomposition of the compound, since there is extensive exchange of hydrogen during fusion.[5] With all compounds, except urea and aromatic halides, more than 99% of the expected tritum was found in gas which contained less than 1% of condensible material. With urea and the aromatic halides, the gas, which included 1–3% of ammonia and benzene, respectively, still contained more than 95% of the tritium.

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[5] Incomplete decomposition of Benzyl-*a-t* alcohol by fusion with zinc in the absence of nickelic oxide yielded benzene and a nonvolatile product, both of which contained tritium.

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Book Reviews

Phosphorus Metabolism: A Symposium on the Role of Phosphorus in the Metabolism of Plants and Animals, Vol. II. Sponsored by the McCollum-Pratt Institute of The Johns Hopkins University. William D. McElroy and Bentley Glass, Eds. Baltimore: Johns Hopkins Press, 1952. 930 pp. Illus. $11.00.

The McCollum-Pratt Institute has performed an outstanding service to biochemists in the organization of its annual symposia and in the prompt publication of the symposium papers. This new volume covers a number of topics omitted from the 1951 symposium, which was published as Volume I.

The general subjects included in Volume II are: mechanisms of phosphate assimilation in animals and plants, the role of phosphate in amino acid and protein metabolism, the role of phosphorus in the metabolism of lipids, the chemistry and metabolism of nucleic acids, the role of phosphate in the metabolism of photosynthetic and chemoautotrophic organisms, the influence of hormones on phosphate metabolism, and phosphate metabolism in specialized tissues. These topics are covered in a total of 41 papers together with a large amount of informative material contributed under the general heading "Discussion." A valuable feature of the book is the last chapter, a summary of the symposium by Bentley Glass, in which the major points are reviewed in some 90 pages. The volume closes with author and subject indexes.

Phosphorus Metabolism, II, presents a summary of recent advances in a number of very active fields of research and, as such, is of particular value to graduate students and research workers in the biological sciences. The chemical element phosphorus forms a very tenuous linkage between some of the topics discussed, but this very heterogeneity of subject is in itself a valuable educational aspect of such a symposium.

Because of the many important roles played by phosphorus compounds in metabolism, Volumes I and II of *Phosphorus Metabolism* present a rather comprehensive view of biochemical knowledge at the mid point of the twentieth century.

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Sampling Technique. William G. Cochran. New York: Wiley; London: Chapman & Hall, 1953. 330 pp. Illus. $6.50.

Sample surveys have played an important part in government operations for the past 20 years. They have served as invaluable research tools whenever accurate information is needed about a population, without entailing the comparatively large expense of a complete enumeration. Even if a complete enumeration were possible, it might not be as accurate as a good sample survey owing to the necessarily longer time for a complete enumeration in which time the population might change. Within recent years sample survey techniques have become increasingly more important in many of the social sciences, business, and technical fields. This book, written by a prominent statistician, gives a comprehensive outline of modern