

One animal injected at 10 days of age and sacrificed 2 days later showed variations from the above mentioned pattern. The reaction over the cupula was most intense at the outer border of the apparatus and also in the vicinity of the hair cells. Over the membrana tectoria, the autoradiographic picture indicated a concentration of S^{35} in the distal portion of the structure, away from the limbus spiralis.

When stained with toluidine blue, the tectorial membrane, the otolithic membrane, and the cupula exhibited metachromasia. The metachromatic reaction of the tectorial and otolithic membranes was persistent like that of mucus and mast cells after incubation with hyaluronidase for 6 hr at pH 5.8 and 37° C. No picture of cupula was available from sections treated in this fashion. The Chèvremont and Frédéricq reaction (10) for the sulfhydryl groups of keratin was negative with all three types of membranes.

In conclusion, it seems that all three membranes synthesize organic sulfur compounds. The cupula and the membrana tectoria show a larger concentration of S^{35} than the otolithic membrane. The sulfur compounds formed do not have a rapid turnover. The histochemical tests do not reveal the presence of keratin in the inner ear but point strongly to the existence of sulfo-mucopolysaccharides in all three types of membranes. In the tectorial and otolithic membranes at least part of these were found to be hyaluronidase resistant.

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The Existence of "Beta" Ice

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Seljakov (1-3) has reported the existence of a low-temperature modification of ordinary ice (ice I) prepared at atmospheric pressure, which he has termed "beta" ice to distinguish it from normal hexagonal or "alpha" ice. He reported that beta ice gave a Laue pattern with rhombohedral symmetry different from the sixfold symmetry of the hexagonal Laue pattern of ordinary ice. The beta ice pattern was observed with crystals grown from water at temperatures (am-

bient air) in the range -5 to -16° C. Seljakov further assumed that a strong (0002) reflection characterized the beta structure and that beta ice was transformed into the alpha structure on grinding.

The existence of this beta ice form has not been confirmed by subsequent workers. It is, however, frequently referred to in the literature as a distinct crystal form.¹ During the course of an ice physics investigation conducted for the U.S. Air Force,² we were led to consider the validity of Seljakov's conclusions as part of a laboratory investigation of nucleation and crystallization of supercooled water.

It was difficult to follow Seljakov's experimental procedure in detail as it was not explicitly described. Following his procedures as closely as possible, however, it was found that ice crystals appeared at water temperatures of approximately -2.5 to -3° C, when the ambient air temperature varied between -5 and -16° C. The water was kept in a metal pan and temperature measurements were made with a thermocouple immersed in the supercooled water.

These experiments cast considerable doubt on the assumption implicit in Seljakov's reports that the ambient air temperature is equivalent to, or an indication of, the temperature to which the supercooled water was cooled before freezing occurred. Further, Seljakov has not observed a critical temperature above which beta ice is never found. Thus beta ice was observed (3) (once out of 3 trials) when water was frozen at an ambient air temperature of only -1° C. Seljakov also reported the presence of beta ice on the surface of lake water.

In a review article (4) Owston has noted that Seljakov did not make efforts to test for preferred orientation in his powder specimens, the probable cause of the absence of the (0002) reflection in some photographs, and that he took only one rotation photograph. Further, in an earlier paper Owston and Lonsdale (5) noted that "It is possible to get an apparently pseudotrigonal Laue picture from ordinary hexagonal ice when it is misset by a few degrees from the hexagonal axis."

At our request Dr. Barbara W. Low (6) recently examined ice crystals grown at an ambient air temperature of -15° C. Her x-ray crystallographic studies confirm and elaborate those of Owston and Lonsdale; a single crystal gave a pseudotrigonal Laue pattern when tilted so that the sixfold axis made an angle of approximately 15° with the x-ray beam. Ordinary ice I (alpha) gives a pseudotrigonal pattern in this position because of the numerical relationship between the lengths of the *a* and *c* axes. The Laue pattern arises from two equivalent and one nonequivalent zones. The Seljakov pattern (1) itself shows this same feature—two identical and one dissimilar ellipses. The characteristic beta Laue pattern of ice, therefore, may be obtained from ordinary alpha ice when it is misset in the manner described.

¹ See, for example, *Handbook of Chemistry and Physics*, 32nd ed., p. 2225. New York: Chemical Rubber Pub., 1950-51.

² U.S. Air Force Contract, AF-33(308)-18687.

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 Compounds¹

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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.

The apparatus shown in Fig. 1 has proved con-

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.

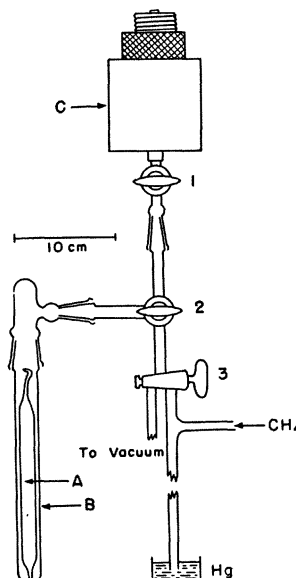


FIG. 1. Apparatus for analysis of gas samples.

venient 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, $\mu\text{c}/\text{mM}$	
	Zinc fusion†	Combustion
HOT	325.9 ± 1.1	327.0
CH ₃ CHTOH	11.65 ± 0.06	11.59
CH ₃ CHTOOCC ₆ H ₄ NO ₂	11.52 ± 0.05	11.59
CH ₃ CT(OH)CH ₃	19.74 ± 0.08	19.42
C ₆ H ₅ CHTOH	10.69 ± 0.05	10.53
C ₆ H ₅ CHTOOCC ₆ H ₃ (NO ₂) ₂	10.72 ± 0.04	10.53
C ₆ H ₄ TCH ₂ —(o)	4.90 ± 0.01	4.87
C ₆ H ₅ CT=NNHC ₆ H ₃ (NO ₂) ₂	9.69 ± 0.01	—
BrCH ₂ CHTC ₆ H ₄ NO ₂	19.89 ± 0.15	19.63
C(CH ₃) ₂ (NO ₂)CHTC ₆ H ₄ NO 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 ₆ H ₅) ₃ SiT	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.

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