

TABLE II

Sample	O ¹⁸ , %
1. Original H ₂ O	4.0
2. Water in equilibrium with algae: light run	3.01
3. Water in equilibrium with algae: dark run	3.06
4. CO sample from xanthophyll fraction of photo-synthesized (5 hr.) algae (50% carotenoid)	0.245
5. CO sample from xanthophyll fraction of algae kept in the dark (40% carotenoid)	0.233
6. Normal H ₂ O	0.204 ^a

^a Thode and Smith, National Research Council of Canada, Atomic Energy Project Report MC-57 (revised), May, 1944.

or from the steroid material. It apparently could not result from exchange with hydroxyl groups already present, however, since such exchanges for ordinary alcohols have been shown to be extremely slow.⁸ The mass spectrographic analysis was of sufficient accuracy that the additional enhancement of the light run is real for this one experiment, but the result would have to be obtained several times before the enhancement could be regarded as general. The experiment is thus inconclusive; it suggests only that it would be worthwhile to repeat the work with water of high oxygen 18 content (30–50%) so that the various carotenoid pigments could be isolated in pure form in sufficient quantities for mass analysis, and so that more than one experiment could be run. Such experiments might provide a real clue as to the path of oxygen in photosynthesis.

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The Preparation of 2-Methyl-2,3-dihydrobenzofuran

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Alkylated dihydrobenzofurans can be readily produced by refluxing allylic bromides with phenols in the presence of potassium carbonate, rearranging the resulting allyl phenyl ethers to the 2-allylphenols and then closing the ring in the latter with hydrobromic acid.¹ In large scale reactions substitution of the allylic chlorides for the corresponding bromides is economically desirable; such attempts, however, usually result in substantially decreased yields. The use of large quantities of sodium iodide along with the allylic chloride¹ to increase the yields is objectionable due to the cost of the iodide. Reagents cheaper than hydrobromic acid for the ring closure are also desirable.

In the present work a Williamson reaction using allylic chlorides gave yields of allylphenols comparable to those previously obtained with the expensive bromides and a mixture of zinc chloride with hydrochloric acid was successfully substituted for the hydrobromic acid in the ring closure.

Experimental

To 2.5 liters of absolute methanol in a 5-liter 3-necked flask fitted with a reflux condenser and mercury-sealed stirrer was slowly added 235 g. (10.2 moles) of sodium in

small pieces. Then 940 g. of phenol (10 moles) in 1 liter of absolute methanol was added slowly and followed by 890 ml. of allyl chloride. After refluxing for 30 hours with stirring, the methanol was distilled off and the organic material was filtered free of precipitated sodium chloride. After washing with 10% potassium hydroxide and drying over anhydrous potassium carbonate, the allyl phenyl ether was fractionated *in vacuo*; yield of the ether 1080 g. (80.6% yield) with b.p. 85 (19 mm.) and *n*_D²⁰ 1.5185.¹

After rearranging the ether by heating as described by Tarbell,¹ 300 g. of the 2-allylphenol was dissolved in 1200 ml. of glacial acetic acid and mixed with 180 g. of anhydrous zinc chloride in 600 ml. of concentrated hydrochloric acid. After refluxing for 30 minutes the mixture was cooled and poured into 2.5 liters of cold water. The organic layer was separated, washed with 10% aqueous potassium hydroxide, extracted with ether and fractionated through a 60-plate column to give 155 g. (51.5% yield) of 2-methyl-2,3-dihydrobenzofuran, b.p. 197° (740 mm.), *n*_D²⁰ 1.5284.¹ Triplicate runs gave the same yields.

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A New Short-Lived Isotope of Cesium¹

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A search has been made for neutron-deficient Cs¹²⁸, previously expected to be quite short-lived.² Spectroscopically pure cesium chloride was bombarded for 2 hours with 96 Mev. protons in the Rochester 130-inch cyclotron in order to prepare 2.4 day Ba¹²⁸, originally reported^{3,4} to decay by emission of 3.0 Mev. positrons through hitherto unknown Cs¹²⁸ to stable Xe¹²⁸.

Radioactive barium was isolated from the CsCl target by a chemical procedure involving three precipitations of barium chromate from a buffered ammonium acetate-acetic acid solution with extensive washings with 1% ammonium acetate to free it from target CsCl. The BaCrO₄ was next dissolved in 2 *M* hydrochloric acid, and the chromate reduced by boiling with excess hydroxylamine hydrochloride. This was followed by precipitation of chromic hydroxide with ammonium hydroxide. Finally, the barium chloride solution was evaporated and ignited lightly to expel ammonium salts and the barium activities taken up in a drop of HCl. About 200 milligrams of BaCl₂ crystals was then added and periodically the cesium daughters growing in were extracted with concentrated HCl from the solid BaCl₂. Over a period of 9 days after bombardment, a total of 16 such parent-daughter separations were performed.

A typical decay curve of the cesium daughters so isolated is shown in Fig. 1, where the half-lives observed were 10 day Cs¹³¹, daughter⁵ of 12 day Ba¹³¹; 31 hour Cs¹²⁹, daughter of 2.0 hour Ba¹²⁹; and new Cs¹²⁸, daughter of 2.4 day Ba¹²⁸. The best value for the half-life of Cs¹²⁸ is an average of 11 different determinations and is 3.13 ± 0.2 minutes. The time lapse between cessation of bombardment and completion of chemical separa-

(1) This work was performed under contract with the U. S. Atomic Energy Commission.

(2) R. W. Fink, F. L. Reynolds and D. H. Templeton, *Phys. Rev.*, **77**, 614 (1950).

(3) C. C. Thomas, Jr., and E. O. Wiig, *THIS JOURNAL*, **72**, 2818 (1950).

(4) R. W. Fink and D. H. Templeton, *ibid.*, **72**, 2818 (1950).

(5) K. Way, *et al.*, *Nuclear Data*, National Bureau of Standards Circular 499 (1950).

(1) D. S. Tarbell, "Organic Reaction," edited by R. Adams, Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1944, pp. 22–26.