suction filtration and crystallized once with 25 cc. of 2:1 benzene–alcohol; 1.4 g. of faintly yellow crystals melting 174–176°, 52% of the theoretical.

Anal. Calcd. for $C_{20}H_{12}O_6Cl_2N_2$: Cl, 15.8; mol. wt., 447.2. Found: Cl, 15.4; mol. wt., 425, 487.

When the carbinol and 3,5-dinitrobenzoyl chloride were allowed to react without pyridine as a solvent, di-(*p,p'*-dichlorobenzohydryl) ether, m. p. 120–123°, was obtained.[2]

(2) Grummitt and Buck, THIS JOURNAL, **67**, 693 (1945).

Determination of the molecular weight of the 3,5-dinitrobenzoate ebullioscopically in benzene consistently gave values ranging from 1.5–2 times the theoretical, indicating that association probably had occurred. The reported values were obtained with acetone as the solvent.

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RECEIVED JANUARY 20, 1945

COMMUNICATIONS TO THE EDITOR

FURAN AND TETRAHYDROFURAN DERIVATIVES. IV. THE SYNTHESIS OF HEXAHYDRO-2-OXO-1-FURO[3,4]IMIDAZOLE DERIVATIVES

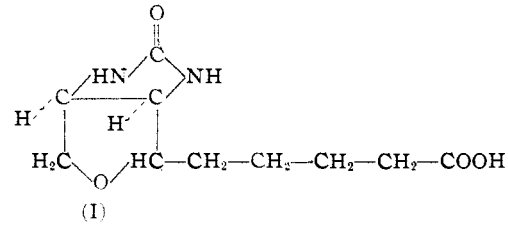
Sir:

In a search for convenient methods to synthesize biotin-like compounds, a number of 3,4-diaminocarbethoxyfurans have recently been prepared.[1] It has now been observed that low pressure hydrogenation converts these substances into the corresponding tetrahydro derivatives. Thus, catalytic hydrogenation of 3,4-diaminocarbethoxy-2-methylfuran under conditions which favor *cis* addition of hydrogen gave a *cis* 3,4-diaminocarbethoxy-2-methyltetrahydrofuran isomer,[2] m. p. 95–96°. (*Anal.* Calcd. for $C_{11}H_{20}O_5N_2$: C, 50.75; H, 7.74; N, 10.75; OC_2H_5, 34.62. Found: C, 50.80; H, 7.79; N, 10.68; OC_2H_5, 34.87.) Mild hydrolysis of this substance with dilute barium hydroxide resulted in a ring closure and a hexahydro-2-oxo-4-methyl-1-furo[3,4]imidazole isomer, m. p. 234–236°, was obtained. (*Anal.* Calcd. for $C_6H_{10}O_2N_2$: C, 50.71; H, 7.09; N, 19.70. Found: C, 51.11; H, 7.26; N, 19.38.)

The structure of this compound was established by drastic hydrolysis with barium hydroxide at 130–140° for twenty hours, which opened the urea ring with the formation of the corresponding *cis*-3,4-diamino-2-methyltetrahydrofuran, isolated as the crystalline sulfate, m. p. 270–275°. (*Anal.* Calcd. for $C_5H_{14}O_5N_2S$: C, 28.03; H, 6.59; N, 13.07; S, 14.97. Found: C, 27.82; H, 6.38; N, 12.89; S, 15.35.) Treatment of the above diamine sulfate with phosgene in sodium bicarbonate solution gave a hexahydro-2-oxo-4-methyl-1-furo[3,4]imidazole, m. p. 234–236°. (*Anal.* Calcd. for $C_6H_{10}O_2N_2$: N, 19.70. Found: N, 20.03), which by mixed melting point was found to be identical with the starting material.

The above described reactions demonstrate that *cis*-3,4-diaminocarbethoxy-2 substituted tetrahydrofurans on treatment with dilute barium hydroxide undergo ring closure to form hexahydro-2-oxo-1-furo[3,4]imidazoles. This novel procedure represents a convenient way to prepare derivatives of this new class of compounds.

Hydrogenation of 3,4-diaminocarbethoxy-2-furanpentanol[1] followed by mild alkaline treatment gave a hexahydro-2-oxo-1-furo[3,4]-imidazole-4-pentanol isomer, m. p. 152–153°. (*Anal.* Calcd. for $C_{10}H_{18}O_3N_2$: C, 56.07; H, 8.47; N, 13.07. Found: C, 56.06; H, 8.18; N, 12.90), which on oxidation was converted into the corresponding hexahydro-2-oxo-1-furo[3,4]imidazole-4-valeric acid (I), m. p. 208–210°. (*Anal.* Calcd. for $C_{10}H_{16}O_4N_2$: C, 52.63; H, 7.07; N, 12.27. Found: C, 52.67; H, 7.43; N, 12.12.) Compound (I) represents one of the oxygen analogs of biotin. Further work which is at present under way will demonstrate the stereochemical relationships between this new compound and biotin.



(I)

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(1) Hofmann and Bridgwater, THIS JOURNAL, in press.

(2) The designation *cis* indicates the position of the amino groups. The steric position of the side chain is not established.

STUDIES IN THE TERPENE SERIES. II.[1] HYDROGEN DISPROPORTIONATION OF LIMONENE

Sir:

During the study of bromination of terpenic hydrocarbons it was noticed that the unreacted terpenes contained a large amount of aromatic hydrocarbons. On further investigation it was found that a small amount of organic bromides admixed with *d*-limonene caused hydrogen transfer to occur during the process of distillation,

(1) For Paper I of this series, see V. N. Ipatieff and H. Pines, THIS JOURNAL, **66**, 1120 (1944).

resulting in the formation of cyclic monoölefins and *p*-cymene

$$2C_{10}H_{16} \xrightarrow{\text{catalyst}} C_{10}H_{18} + C_{10}H_{14}$$

Preliminary experiments made using limonene and halogen-containing catalysts showed that the degree of hydrogen disproportionation depended upon the type of catalysts used. Not all of the tested catalysts were equally suited for the hydrogen transfer reaction; some caused pronounced polymerization. The experimental results are given in Table I. All experiments were conducted at 174–178° and were of four hours duration. The reactions were carried out in a flask connected to a reflux condenser; in the case of monochloroacetic acid catalyst, a sealed tube was used. Iodine and hydrogen bromide, in the form of 1,8-dibromo-*p*-menthane, were the most effective catalysts; the latter, however, is preferred since it is a weaker polymerizing catalyst causing the formation of only six per cent. of higher boiling hydrocarbons. The chloro derivatives of acetic acid caused both hydrogen transfer and polymerization to occur.

TABLE I

Catalyst used: Kind	Moles/100 moles of limonene	Mole % of limonene converted to: Aromatics	Higher boiling compounds
Dibromolimonene[a]	3	45	6
Iodine	2	48	12
Monochloroacetic acid	6	3	5
Dichloroacetic acid	4	9	6
Trichloroacetic acid	4	25	24

[a] 1,8-Dibromo-*p*-menthane prepared by the action of hydrogen bromide on limonene in acetic acid solution.

In view of the ease with which hydrogen transfer occurs, we intend to reinvestigate the dehydration of alcohols and glycols to hydrocarbons in the presence of acidic type of dehydrating catalysts.

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RECEIVED FEBRUARY 26, 1945

NEW BOOKS

Lead Poisoning. By ABRAHAM CANTAROW, M.D., Associate Professor of Medicine, Jefferson Medical College, Assistant Physician, Jefferson Hospital, Biochemist, Jefferson Hospital, Philadelphia, Pa., and MAX TRUMPER, Ph.D., Lt. Commander, H-V(S), U.S.N.R., Naval Medical Research Institute, Bethesda, Md., formerly Lecturer in Toxicology, Jefferson Medical College, Consultant in Industrial Toxicology, Cynwyd, Pa. The Williams and Wilkins Company, Baltimore 2, Md., 1944. xiii + 264 pp. 15.5 × 23.5 cm. Price, $3.00.

This publication presents an extensive review of lead poisoning, particularly of material published within recent years. The authors discuss the absorption, transportation, deposition and excretion of lead, the pathology and pathological physiology and clinical manifestations of lead poisoning, treatment, chronic lead poisoning, lead in blood, body fluids and excretions, normal intake of lead, lead products in industry and the biochemical analysis of lead.

The literature has been ably summarized, although the review is largely non-critical. In spite of the great number of papers that have been published on the subject of plumbism, one is amazed that so little real progress has been made in our knowledge of the fundamental physiology and pathology of lead poisoning. It is quite possible that this is due to inherent difficulties of tracing the effects of exceedingly minute amounts of lead and it reflects the need for exacting physico-chemical study in this field. The tendency to place more emphasis on the interpretation rather than the critical evaluation of data probably accounts for much of the voluminous literature of lead poisoning.

The authors have made a signal contribution in bringing the literature of lead poisoning up to date. The work has been well planned and successfully carried through. A few typographical errors were noted. Among these were (p. 51) prophyrinuria, (p. 89) Stanzi, (p. 90) hemorrage, (p. 140) Leivy, (p. 141) Luthje, (p. 141) p. 000, (p. 160) Bradham and (p. 206) nickle. The maximum permissible concentration of lead in potable water used by interstate carriers (p. 170) was set by the U. S. Public Health Service, not the American Public Health Association. The reference to Sawyer, Wagoner, and Erickson (p. 253) is incorrect.

Although the quality of the paper reflects the difficulties of the times, the printing conforms to the usual high standard of the publishers. The book may well be recommended to all who are interested in lead.

L. T. FAIRHALL

The Washington Scientist. A Magazine for the Scientists of Washington. WARE CATTELL, Editor. Published by the Science Press of Washington. Volume I, No. 1, February, 1945. 23 pp. Subscription, $3.00; single copies, $0.30.

In announcing the first issue of *The Washington Scientist*, its Editor, Ware Cattell, writes in part as follows:

"The scientific men and women of Washington belong to many groups and occupations. They are the backbone of our Government research agencies; they serve on the faculties of our local schools, colleges, and universities; they administer great foundations for the advancement of human knowledge; they represent scientific societies whose headquarters are in Washington. Some of them are professional engineers, physicians, chemists; others are searching for truth for its own sake, believing only in that ancient fundament that to know the truth will make me free. Still others may be the indispensable handmaidens of science—the technicians, the computers, the apprentices. All, however, belong to the same fraternity, whose objective is to discover and to understand.