formation of this substance into salicylic acid was 70% efficient.

DEPARTMENT OF CHEMISTRY
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## Preparation of Tetramethylene Bromide

BY SHERMAN FRIED AND RICHARD D. KLEENE

Tetramethylene bromide has previously been available by the reaction between N-benzoylpyrrolidine and phosphorus pentabromide.[1] We have now found that the cleavage of tetrahydrofuran by hydrogen bromide, which is analogous to the reaction previously applied by Starr and Hixon,[2] for the preparation of the corresponding chlorohydrin, is much simpler and gives a comparable yield from more readily available starting materials.

Furan was prepared by the decarboxylation of furoic acid in the presence of copper oxide and quinoline,[3] using, however, a Dewar jacketed trap cooled with dry-ice to prevent entrainment of the furan by escaping carbon dioxide. The furan was readily hydrogenated in 95% yield to tetrahydrofuran using palladium–palladium oxide as a catalyst.[4]

Dry hydrogen bromide was passed into the tetrahydrofuran in a flask fitted with a side-tube, reflux condenser and thermometer, until the temperature reached 150°, when the theoretical quantity of hydrogen bromide had been added. The resulting black tarry product was washed thoroughly with water and then with sodium bicarbonate solution until it was free of hydrobromic acid. It was then taken up with ether and dried over anhydrous copper sulfate. The product was fractionated under diminished pressure and 134 g. (yield, 70%) of tetramethylene bromide (b. p. 198° at normal pressure) was collected.

The authors gratefully acknowledge the assistance of Professor W. G. Brown, who designed the apparatus used in the preparation of the furan.

(1) Von Braun and Muller, *Ber.*, **39**, 4124 (1906).
(2) Starr and Hixon, THIS JOURNAL, **56**, 1595 (1934).
(3) Wagner and Simmons, *J. Chem. Ed.*, **13**, 270 (1936).
(4) Shriner and Adams, THIS JOURNAL, **46**, 1683 (1924).

DEPARTMENT OF CHEMISTRY
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## The Isolation of Eriodictyol and Homoeriodictyol. An Improved Procedure

BY T. A. GEISSMAN

The question of the existence of a substance having vitamin-like properties in its effect upon capillary permeability, and called "Vitamin P" by Szent-Györgyi[1] remains unanswered. So far practically all the studies that have been made upon the putative vitamin have been made upon "citrin," a crude flavanone fraction isolated from lemon peel. Reports of experiments in which this material has been used in clinical studies and in animal (guinea pig) tests are conflicting[2] and allow no definite conclusion to be drawn as to the physiological action of "citrin," although the balance of the evidence seems to support the belief that it does contain some substance which exerts a vitamin-like action, when acting in conjunction with vitamin C, in certain types of hemorrhagic diathesis.

Szent-Györgyi has attributed the vitamin activity of "citrin" to the presence of an eriodictyol glycoside[3] although definite experimental proof of its presence in "citrin" is lacking. It is probable, however, that "citrin" does contain eriodictyol (as a glycoside?) since certain color tests (ferric chloride, hot aqueous alkali) shown by "citrin" are also given by pure eriodictyol. It is clear that even if it can be shown that "citrin" contains eriodictyol this will not constitute proof that eriodictyol is "vitamin P." Indeed, Scarborough's results[2c] indicate that hesperidin (also a constituent of "citrin") has "citrin" activity in certain types of purpura.

In an approach to the problem through studies on pure flavanones which are known to be or suspected of being present in "citrin" the preparation of pure eriodictyol has been undertaken.

Eriodictyol (5,7,3′,4′-tetrahydroxyflavanone) has been isolated from *Eriodictyon californicum* by Power and Tutin[4] and from *Lespedeza cyrtobotrya* by Ohira.[5] It has been isolated, along with homoeriodictyol, for the present study from *Eriodictyon californicum* by a procedure which combines the best features of the method of Power and Tutin with those of the method used by Mossler[6] in isolating homoeriodictyol from the same plant. The yields of both eriodictyol and homoeriodictyol were about double those obtained by Power and Tutin but this improvement may not be inherent in the present method since studies now in progress in this Laboratory have indicated that considerable variation can occur

(1) Szent-Györgyi, *et al.*, *Nature*, **138**, 27, 798 (1936); *Z. physiol. Chem.*, **255**, 216 (1938).
(2) (a) Szent-Györgyi, *et al.*, *Nature*, **139**, 326 (1937); **140**, 426 (1938); (b) Zilva, *Biochem. J.*, **31**, 915, 1488 (1937); (c) Scarborough, *ibid.*, **33**, 1400 (1939).
(3) Bruckner and Szent-Györgyi, *Nature*, **138**, 1057 (1936).
(4) Power and Tutin, *Pharm. Rev.*, **24**, 301 (1907); *Pharm. J.*, **77**, 381 (1906); Tutin and Clewer, *J. Chem. Soc.*, **95**, 81 (1909).
(5) T. Ohira, *J. Agr. Chem. Soc. Japan*, **9**, 448 (1933).
(6) Mossler, *Ann.*, **351**, 223 (1907).