Iodine as a Carrier in the Bromination of Fluorene in the Dark.—A saturated solution of iodine in carbon tetrachloride was found to contain 0.29 g. in 10 cc. Dilutions of this, 1 to 10, and 1 to 100 were made. Fluorene, 0.01 mole, was added to 10 cc. of the carbon tetrachloride solution and then 10 cc. of a 1 molar solution of bromine in the same solvent was added quickly. The reaction was stopped by the addition of potassium iodide solution as usual. The results are in Table I.

TABLE I

BROMINATION OF FLUORENE IN THE DARK WITH IODINE AS CARRIER

Time, min.	0.29 g.	0.029 g.	0.0029 g.
0.5	79%	..	8
0.5	80	..	..
3.0	89	20	..
3.0	89	19	..

For comparison the same concentration of toluene and bromine in the same solvent were tried with 0.29 g. of iodine. There was no bromination in three minutes.

The bromine used was freed from traces of iodine by prolonged shaking with concentrated sulfuric acid, washing with water, drying over phosphorus pentoxide and fractionally distilling.

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The Optical Rotatory Power of *epi*-Ergostanol

BY KARL J. SAX, LOUIS DORFMAN[1] AND SEYMOUR BERNSTEIN

In their development of a theory on the relationship between optical rotatory power and constitution of the steroids, Bernstein, Kauzmann and Wallis[2] noted a number of compounds for which large discrepancies existed between observed and calculated values of the optical rotation. For *epi*-ergostanol it was stated that the observed value for this compound was in error by at least 10°.[3] Also it has been pointed out[4] that the C_3-diastereomers, ergostanol and *epi*-ergostanol, do not conform to the rule that the C_3 α-form of any steroid will have a higher positive rotatory power than the corresponding β-form.

Accordingly it was of interest to redetermine the optical rotations of ergostanol and *epi*-ergostanol for evaluating the above discrepancies. The rotation of ergostanol was found to be +15.3° which is in excellent agreement with the recorded values of +15.3°[5] and +15.9°.[6] However, for *epi*-ergostanol we have found the rotation to be +16.9° which is higher than the recorded values of +13.5°[7] and +14.6°.[8]

These results show that the diastereomers, ergostanol and *epi*-ergostanol, do not constitute an exception to the above stated rule. Also it may be assumed that the value (+2300) for the constant, E_l^2, derived from *epi*-cholestanol, and used in the calculation of the rotation of *epi*-ergostanol, is incorrect. Use of *epi*-stigmastanol, $[\alpha]_D$ + 25,[9] as the standard substance, gave a E_l value of 0. Recalculation of the rotation of *epi*-ergostanol with this revised value gave +19.1°, which is in good agreement with the observed rotation of +16.9°.

(1) Present address, William R. Warner and Company, Inc., New York.

(2) Bernstein, Kauzmann and Wallis, *J. Org. Chem.*, **6**, 319 (1941).

(3) All rotations are for sodium D light and chloroform solution.

(4) Bernstein, Hicks, Clark and Wallis, *J. Org. Chem.*, **11**, 646 (1946).

(5) Windaus and Brunken, *Ann.*, **460**, 225 (1928).

(6) Reindel, Walter and Rauch, *Ann.*, **452**, 34 (1927).

(7) Reindel and Detzel, *Ann.*, **475**, 78 (1929).

(8) Windaus, *et al.*, *Ann.*, **477**, 268 (1930).

(9) Dalmer, *et al.*, *Ber.*, **68**, 1814 (1935).

LEDERLE LABORATORIES DIVISION
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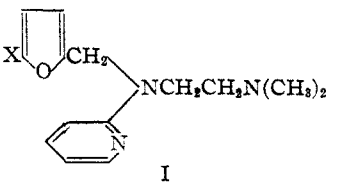
Antihistamine Agents. II. Furan Derivatives

BY J. R. VAUGHAN, JR., AND G. W. ANDERSON

In a continuation of our investigation on the effect of substituting various heterocyclic systems into compounds of known antihistamine activity,[1] we have prepared and tested N,N-dimethyl-N′-(2-pyridyl)-N′-furfurylethylenediamine (I, X = H) and N,N-dimethyl-N′-(2-pyridyl)-N′-(5-bromofurfuryl)-ethylenediamine (I, X = Br). The first of these (I, X = H) has been reported by Viaud to be an active antihistaminic.[2] The compounds may be considered as oxygen analogs of the thiophene substituted ethylenediamines previously reported in which the furan nucleus replaces the thiophene group.



I

They were synthesized by an initial reaction of furfuryl alcohol, or 5-bromofurfuryl alcohol, with thionyl chloride in toluene solution at −30 to −40°. The intermediate furfuryl chlorides obtained are extremely unstable[3] and were not isolated but were treated directly with the sodium salt of N,N-dimethyl-N′-(2-pyridyl)-ethylenediamine, also in toluene solution, at low tempera-

(1) Clapp, Clark, Vaughan, English and Anderson, THIS JOURNAL, **69**, 1549 (1947).

(2) Viaud, *Technologie Produits Pharmaceutiques*, **2**, 53 (1947); *Drug Trade News*, **22** [9], 63 (1947). We have been unable to obtain the original article but have been advised that the name "methylfurfuryl" used by the *Drug Trade News* is intended to mean "furylmethyl" or furfuryl.

(3) Gilman and Vernon, THIS JOURNAL, **46**, 2576 (1924).