LETTERS TO THE EDITORS

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Synthesis of a Substance Containing the Chelerythrine-Sanguinarine Skeleton (C, N, O)

The skeleton indicated is (I) and we first attempted an extension of the method whereby (II) had been synthesized in 1937 ¹.

However, cyclization of 1-formamido-6:7-dimethoxy- $2\cdot(2'$ -bromo-4':5'-dimethoxy)phenyl-1:2:3:4-tetrahydronaphthalene could not be effected. R. D. Haworth² was also interested in this problem and prepared 6:7-dimethoxy- $3\cdot(3':4'$ -dimethoxy)phenacylphthalide (III), but was unable to add the elements of hydrocyanic acid to this substance.

In ignorance of Haworth's work, for the first months, we adopted the same route and were fortunate to find appropriate conditions for the reaction of III with potassium cyanide. The product (IV), m.p. $148-152^{\circ}$ (found: C, $63\cdot2$; H, $5\cdot5$ per cent), gives, on heating, a neutral, yellow compound, m.p. $234-35^{\circ}$ (found: C, $65\cdot8$; H, $5\cdot0$; N, $3\cdot2$ per cent), and a molecule of water.

$$\begin{array}{c} \text{CH(CN)} \cdot \text{CH}_2 \cdot \text{CO} & \text{OMe} \\ \text{OMe} & \text{OMe} \\ \text{MeO} & \text{IV} \\ \text{CH}_2 - \text{CH}_2 \\ \text{CH} & \text{OMe} \\ \text{MeO} & \text{OMe} \\ \\ \text{MeO} & \text{OMe} \\ \end{array}$$

v

Hydrolysis of this yellow compound by means of boiling 2N sodium hydroxide, or of the nitrile-acid (IV) by means of hydrogen peroxide followed by 2N sodium hydroxide, afforded the keto-dibasic acid related to IV; m.p. $195-97^{\circ}$ (decomp.) (found: C, $60\cdot1$; H, $5\cdot3$ per cent; equiv. by titration, 218).

Potentiometric titration showed two carboxyl groups to be present, and the anhydride had m.p. 164-66° (found: C, 63·1; H, 4·9 per cent) and the

properties of a homophthalic anhydride.

Reduction of the keto-dibasic acid (IV, CN \rightarrow CO₂H) by Clemmensen's method gave a dibasic acid (CO \rightarrow CH₂), C₂₁H₂₄O₈, m.p. 171–72° (decomp.) (found: C, 62·4; 62·6; H, 6·0, 5·8 per cent; M, 417) (two carboxyls by potentiometric titration and anhydride, m.p. 155–56°).

On heating the ammonium salt, the imide (V), namely, $4\cdot[\beta\cdot(3':4'\text{-dimethoxyphenyl})\text{ethyl}]-7:8$ -dimethoxyhomophthalimide, was obtained; m.p. $127-127\cdot5^\circ$ (found: C, $65\cdot7$; H, $6\cdot1$; N, $3\cdot3$ per cent). On cyclization by means of phosphoric anhydride in glacial phosphoric acid solution, 7:8:4':5'-tetramethoxy-3:4-dihydro-1:2-benzphenanthridone (V1), m.p. $259-61^\circ$ (found: C, $68\cdot4$, $68\cdot8$; H, $5\cdot8$, $5\cdot8$; N, $3\cdot8$ per cent) was obtained in good yield.

$$\begin{array}{c} \operatorname{CH}_2 \\ \operatorname{CH}_2 \\ \operatorname{OMe} \\ \operatorname{OMe} \\ \\ \operatorname{NH} \\ \operatorname{CO} \\ \text{VI} \end{array}$$

The further steps necessary to develop this method so as to effect the syntheses of chelerythrine and sanguinarine, also of their dihydro derivatives, are obvious, and unlikely to cause serious difficulty. Experiments on the synthesis of chelidonine and α -homochelidonine are also in progress.

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 $^{\rm l}$ Richardson, Robinson and Seijo, J. Chem. Soc., 835 (1937). $^{\rm 2}$ Haworth, R. D., J. Chem. Soc., 1312 (1937).

Formation of a Tetrahydrofuran Derivative from Diallyl

When diallyl (hexa-1:5-diene) is treated with perbenzoic acid, 1:2-5:6-diepoxyhexane (I) is produced in good yield (80 per cent).