## Dibenzo [b,f] azepin-2-one

By K. E. HAQUE and G. R. PROCTOR\*

(Department of Pure and Applied Chemistry, The University of Strathclyde, Glasgow, C.1)

The only known heterocyclic derivatives of the novel system (I; R = H, OH, and OMe)<sup>1,2</sup> are the compounds (II; R = H and Ac):<sup>3</sup> we have now prepared the parent, dibenzo[b,f]azepin-2-one (III). When iminostilbene† (IV) was treated with Fremy's salt<sup>4</sup> in acetone buffered with disodium hydrogen phosphate, two products‡ were obtained and separated by chromatography from unchanged starting material (5%).

(IV)

The first product  $C_{14}H_9NO$  (37%) was yellow: we regard this as acridine-9-carbaldehyde (V) from the following data: m.p. 141—142° (lit., 5 139—140°), 2,4-dinitrophenylhydrazone, m.p. 232—235° (lit., 6 235°); the n.m.r. spectrum showed a signal at  $\tau$  —1·4 which was not affected by addition of  $D_2O$ ,  $v_{max}$  (CCl<sub>4</sub>) 5·9  $\mu$  and the mass spectrum contained

a strong M — CO peak. The sequence from (IV) to the aldehyde (V) is formally similar to a ring contraction which N-acetyliminostibene is reported to undergo under acidic conditions, but we cannot suggest a simple mechanism to explain it.

$$(VI)$$

$$VI)$$

$$VII)$$

$$VIII)$$

The second product (53%) from the Fremy's salt reaction was a deep red crystalline material  $(C_{14}H_9NO)$ , m.p.  $135-136^\circ$ : to this we allocate structure (III) on the following grounds. The n.m.r. spectrum consisted only of a series of multiplets between  $\tau$  1·8 and 3·6, in the mass spectrum the base peak was the M — CO ion² (its stability may be connected with the derived aza-azulene structure), the i.r. spectrum (Nujol) contained 3 strong bands at 6·1, 6·3, and 6·46  $\mu$  [the imino-ketone<sup>7,8</sup> (VI) has bands at 6·05, 6·14, and 6·33  $\mu$ ], and catalytic hydrogenation of (III) yielded a substance (VII) identical to that obtained from the imino-ketone (VI).

The stability of (III) is a little surprising since the unsubstituted bicyclic analogue (I; R = H) could not be isolated without decomposition<sup>1</sup> and carbotricyclic analogues of (III) have so far

(V)

<sup>†</sup> Aldrich Chemical Company, Milwaukee, Wisconsin, U.S.A.

<sup>‡</sup> Satisfactory analytical and spectral data were obtained for all new compounds.

proved elusive.9 Our data are too meagre to allow comment on the aromaticity of the system (III).

We thank the S.R.C. for a fellowship (to K. E. H.).

(Received, September 11th, 1968; Com. 1238.)

- <sup>1</sup> A. M. Khan, G. R. Proctor, and L. Rees, J. Chem. Soc. (C), 1966, 990.

  <sup>2</sup> G. R. Proctor and A. H. Renfrew, J. Chem. Soc. (C), 1968, 1187.

  <sup>3</sup> A. Butenandt, E. Biekert and G. Neubert, Annalen, 1957, 603, 200.

  <sup>4</sup> R. P. Singh, Canad. J. Chem., 1966, 44, 1994.

  <sup>5</sup> I. Heilbron and H. M. Burnbury, "Dictionary of Organic Compounds", Eyre and Spottiswoode, 1964, vol. 35.

  <sup>6</sup> A. E. Porai-Koshits and A. A. Kharkharov, Bull. Acad. Sci., U.S.S.R., 1944, 79.

  <sup>7</sup> W. Schindler and H. Blattner, Helv. Chim. Acta, 1961, 44, 753.

  <sup>8</sup> W. Schindler, Helv. Chim. Acta, 1960, 43, 35.

  <sup>9</sup> G. R. Proctor, H. Renfrew, and J. Savage, unpublished data.