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A 1,2-DIAZETIDINE INTERMEDIATE

FROM PHOTOCYCLIZATION OF A SCHIFF BASE Scott Searles, Jr., and Robert A. Clasen Department of Chemistry, Kansas State University Manhattan, Kansas 66504

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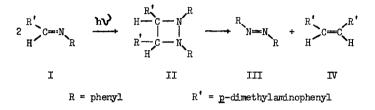
There have been two recent reports of the photocyclization of Schiff bases to phenanthridines,^{1,2} as well as 5,6-benzoquinolines.³ We also had independently observed phenanthridine formation on irradiation of several other Schiff bases, and have now isolated products from the irradiation of $\underline{N-p}$ -dimethylaminobenzylideneaniline (I) which indicate the intermediacy of the substituted 1,2-diazetidine (II).

Irradiation of a dilute, ethereal solution of (I) for sixty hours with a Hanovia "S" quartz mercury lamp gave <u>trans</u>-azobenzene (III) and <u>cis-</u> $h_{2}h_{1}^{*}$ -bis(dimethylamino)stilbene (IV), isolated in 35 and 25% yields, respectively. Also obtained was a 15% yield of a material, m.p. <u>ca</u>. 180° (subl.), tentatively identified as 9-dimethylaminophenanthridine on the basis of its elemental analysis, infrared and n.m.r. spectra; and 10% of (I) was recovered. The irradiation was carried out without exclusion of air.

Azobenzene was identified by its melting point (m.p. 66-7°, m.m.p. 66-7°, reported 66°⁴) and the identity of its infrared spectrum with that of an authentic sample. The l_1, l_1 -bis(dimethylamino)stilbene was identified by its melting point of 257-8° (reported 253- $l_1^{\circ 5}$) and by its infrared and n.m.r. spectra. This was confirmed by its identity with a sample of (IV) prepared by another, previously reported method.⁵

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While details concerning the mechanism of this process are still lacking, it seems reasonable to postulate cycloaddition of a photochemically excited molecule of (I) to another molecule of (I), in either an excited or ground state, in a head to head fashion to give (II) in an excited state. Such a molecule of (II) could then collapse to the observed products by a simple electron redistribution process.⁶ Further work on this and other photochemical reactions of Schiff bases is in progress.



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