The Synthesis of 6,11-Diphenyldibenzo[*b*,*f*][1,4]diazocine, a New Heterocyclic System

By HOWARD D. PERLMUTTER

(*Department of Chemical Engineering and Chemistry, Newark College of Engineering, Newark, New Jersey* 07102)

SINCE the reported[1] physiological activity of a dichloro-derivative of (II) might also apply to derivatives of 6,11-diphenyldibenzo[*b*,*f*][1,4]-diazocine (III) we attempted the synthesis of (III), the first 1,4-diazocine of its kind.† Previous attempts[2] to prepare 1,4-diazocines related to (I) had proved fruitless.

The synthetic procedure followed was that reported[3] for the preparation of (II). $AlCl_3$ (403 mg.) was mixed with a solution of *o*-phenylenediamine (287 mg.) and *o*-dibenzoylbenzene (712 mg.) in chlorobenzene (13 ml.), and the resulting mixture heated under reflux for 10 hours. The cooled mixture was diluted with benzene and extracted with dilute aqueous alkali. Evaporation of the organic solvents yielded 695 mg. of amber oil. This material, when chromatographed on silica gel, gave two substances. Elution with 1 : 1 hexane–CCl_4 (v/v) gave a pale yellow solid (A) (53 mg). Further elution with CCl_4–benzene 1 : 1 (v/v) afforded a yellow solid (B) (287 mg).

Substance (B), m.p. 174—178°, was shown to be homogeneous by t.l.c. Its spectral characteristics are similar to those of (II)[3] and (IV)[4]: strong i.r. absorption at 6·08 μ (conjugated C=N) and u.v. λ_{max} 252 (ϵ 38,710) and 330 mμ (ϵ 3300) (propan-2-ol). The n.m.r. spectrum consisted of multiplets centred at τ 2·2 and 2·7 and a singlet at τ 3·1. By hydrolyzing (B) under acidic conditions, the starting diketone, *o*-dibenzoylbenzene, was isolated.

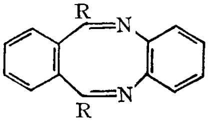
---

† For a description of 6,7-diphenyldibenzo[*e*,*g*][1,4]diazocine, a 2,2′-bridged biphenyl, see N. L. Allinger and G. A. Youngdale, *J. Org. Chem.*, 1959, **24**, 306.

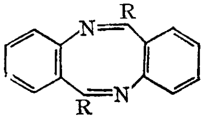
Substance (A), a fluorescent material, m.p. 248—253°, was also shown to be homogeneous by t.l.c. Its i.r. spectrum lacked the 6·08 μ peak and was quite different from that of either (B) or (II), although its n.m.r. spectrum resembled that of (B), showing a broad multiplet in the range τ 2—3.

The mode of synthesis, as well as the spectroscopic and chemical properties of (B) are all consistent with its formulation as (III).‡

We thank the Foundation for the Advancement of Graduate Study in Engineering of Newark College of Engineering for encouragement and financial support of this work.



(I) R=H
(III) R=Ph



(II) R=Ph
(IV) R=H

(*Received, July* 11*th,* 1968; *Com.* 935.)

---

‡ Compound (III) gave satisfactory elemental and molecular weight analyses.

[1] W. Metlesics, R. Tavares, and L. H. Sternbach, *J. Org. Chem.*, 1966, **31**, 3356, and references cited therein.

[2] H. D. Perlmutter and P. S. Knapp, *J. Org. Chem.*, 1967, **32**, 2350.

[3] W. Metlesics, T. Resnick, G. Silverman, R. Tavares, and L. H. Sternbach, *J. Medicin. Chem.*, 1966, **9**, 633.

[4] W. W. Paudler and A. G. Zeiler, *Chem. Comm.*, 1967, 1077.