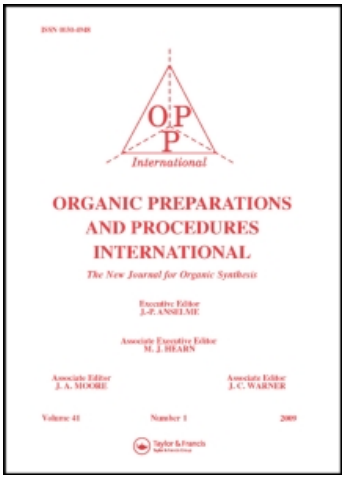

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SYNTHESIS OF NEW PHENYLQUINOXALINES

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2. L. S. Harris and A. K. Pierson, J. Pharmacol. Exptl. Therap., 143, 141 (1964).

3. S. Archer, N. F. Albertson, L. S. Harris, A. K. Pierson and J. G. Bind, J. Med. Chem., 7, 123 (1964).

4. R. Grewe and A. Mondon, Chem. Ber., 81, 279 (1948).

5. E. L. May and E. M. Fry, J. Org. Chem., 22, 1366 (1957).

6. N. F. Albertson and W. F. Wetterau, J. Med. Chem., 13, 302 (1970).

7. T. Kametani, K. Kigasawa, M. Hiiragi, T. Hayasaka, N. Wagatsuma and K. Wakisaka, J. Heterocyclic Chem., 6, 43 (1969).

8. M. G. Van Campen, D. F. Meisner and S. M. Parmerter, J. Am. Chem. Soc., 70, 2296 (1948).

9. R. C. Elderfield and V. B. Meyer, ibid., 76, 1886 (1954).

SYNTHESIS OF NEW PHENYLQUINOXALINES

Submitted by (4/5/76) J. A. Harvey and M. A. Ogliaruso*

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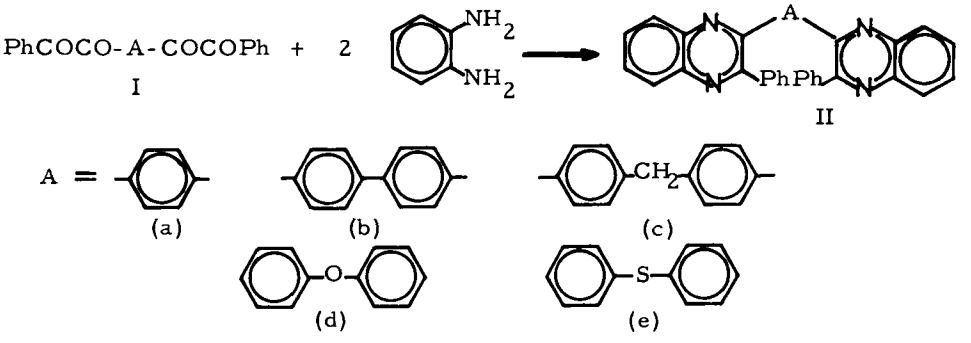
The unusual solubility characteristics of phenylated quinoxaline polymers,[1-3] has prompted the synthesis of several model compounds.



EXPERIMENTAL

General Procedure. - A 125 ml Erlenmeyer flask containing 5.0 mmole of 4-phenylglyoxalylbenzil (Ia),[4] 4,4'-diphenylglyoxalylbiphenyl (Ib),[5] 4,4'-diphenylglyoxalyldiphenyl methane (Ic),[5] 4-phenylglyoxalylphenyl ether (Id),[5] or 4-phenylglyoxalylphenyl sulfide (Ie)[5] and 1.62 g (15.0 mmole) of o-phenylenediamine was placed in a Wood's Metal bath and heated at 200° for 15 min. with occasional swirling. The flask was then removed from the bath, cooled and the precipitated brown solid was recrystallized three times from 25-50 ml portions of a 2:1 benzene-pentane mixture and afforded in each case, pure off-white crystals of product.

Table I. Physical Data for Phenylquinoxalines

Phenylquinoxalines	Yield %	mp, °C	Formula	Analysis % Calcd. (Found) C	H	N
1,4-bis[2-(3-phenylquinoxalyl)]benzene (IIa)	52	266-268[a]	$C_{34}H_{22}N_4$	83.93 (83.68	4.56 4.71	11.51 11.22)
4,4'-bis[2-(3-phenylquinoxalyl)]biphenyl (IIb)	48	254-255	$C_{40}H_{26}N_4$	85.39 (85.64	4.66 4.83	9.96 10.21)
4,4'-bis[2-(3-phenylquinoxalyl)]diphenyl methane (IIc)	82	197-198	$C_{41}H_{28}N_4$	85.39 (85.16	4.89 4.94	9.72 9.38)
4,4'-bis[2-(3-phenylquinoxalyl)]diphenyl ether (IId)	92[b]	225-226	$C_{40}H_{26}N_4O$	83.03 (83.01	4.53 4.48	9.67 9.57)
4,4'-bis[2-(3-phenylquinoxalyl)]diphenyl sulfide (IIe)	70	206-208	$C_{40}H_{26}N_4S$	80.78 (80.92	4.41 4.62	9.42 9.15)

a) Reported[1] 226-227°, this article also has the incorrect analysis reported for this compound, p. 1457.

b) Previously reported[4] but the analysis was incorrect.

REFERENCES

1. P. M. Hergenrother and H. H. Levine, J. Polym. Sci., A-1, 5, 1453 (1967).
2. P. M. Hergenrother, ibid., A-1, 6, 3170 (1968).
3. W. Wrasidlo and J. M. Augl, ibid., A-1, 7, 3393 (1969).
4. M. A. Ogliaruso, L. A. Shadoff and E. I. Becker, J. Org. Chem., 28, 2725 (1963).
5. M. A. Ogliaruso and E. I. Becker, ibid., 30, 3354 (1965).

DIETHYL 2,5-DI [BENZYLOXY] TEREPHTHALATE

Submitted by (6/1/76) S. Wawzonek* and J. E. Durham

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Repeated fractional crystallization of the crude reaction mixture (9.1 g) from the benzylation of the disodium derivative of 2,5-dicarbethoxycyclohexane-1,4-dione [D. Y. Chang and M. C. Chan, J. Org. Chem., 39, 976 (1974)] gave 2.6 g of pure trans-2,5-dibenzyl-2,5-dicarbethoxycyclohexane-1,4-dione and 2.7 g of the pure cis-isomer, both known compounds. Preparative layer chromatography of the combined mother liquors on silica gel with methylene chloride as the developing solvent, yielded 0.09 g of diethyl 2,5-di[benzyloxy]terephthalate, mp. 95-96°; IR(Nujol) 5.95(CO); NMR($CDCl_3$) δ1.30 (t, 6H, $2CH_2\underline{CH_3}$), 4.32 (q, 4H, $2\underline{CH_2}CH_3$), 5.10 (s, 4H, $2OCH_2$), 7.10-7.50 (m, $2C_6H_5$ and 2CH); m/e 434.

Anal. Calcd. for $C_{26}H_{26}O_6$: C, 71.86; H, 6.04.
Found: C, 72.21; H, 5.95.