

This article was downloaded by:

On: 27 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

### SYNTHESIS OF NEW PHENYLQUINOXALINES

J. A. Harvey<sup>a</sup>; M. A. Ogliaruso<sup>a</sup>

<sup>a</sup> Department of Chemistry, Virginia Polytechnic Institute and State University, Blacksburg, Virginia

**To cite this Article** Harvey, J. A. and Ogliaruso, M. A. (1976) 'SYNTHESIS OF NEW PHENYLQUINOXALINES', *Organic Preparations and Procedures International*, 8: 3, 146 – 148

**To link to this Article:** DOI: 10.1080/00304947609355609

**URL:** <http://dx.doi.org/10.1080/00304947609355609>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

OPPI BRIEFS

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).

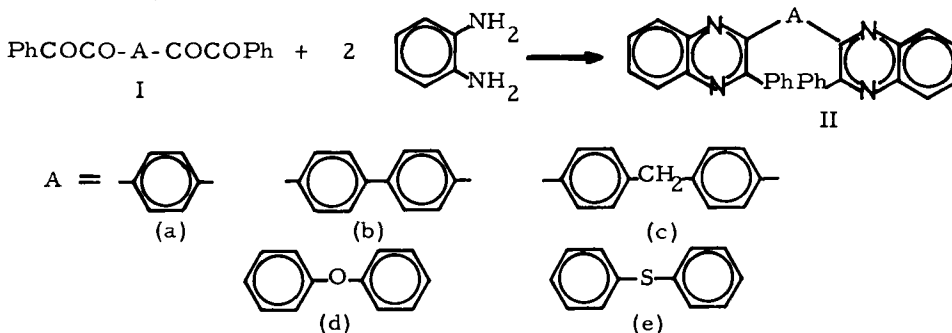
SYNTHESIS OF NEW PHENYLQUINOXALINES

Submitted by J. A. Harvey and M. A. Ogliaruso\*

(4/5/76)

Department of Chemistry  
Virginia Polytechnic Institute  
and State University  
Blacksburg, Virginia 24061

The unusual solubility characteristics of phenylated quinoxaline polymers,<sup>1-3</sup> has prompted the synthesis of several model compounds.



## EXPERIMENTAL

General Procedure. - A 125 ml Erlenmeyer flask containing 5.0 mmole of 4-phenylglyoxalylbenzil (Ia),<sup>4</sup> 4,4'-diphenylglyoxalylbiphenyl (Ib),<sup>5</sup> 4,4'-diphenylglyoxalyl-diphenyl methane (Ic),<sup>5</sup> 4-phenylglyoxalylphenyl ether (Id),<sup>5</sup> or 4-phenylglyoxalylphenyl sulfide (Ie)<sup>5</sup> 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 <sup>a</sup>	C <sub>34</sub> H <sub>22</sub> N <sub>4</sub>	83.93 (83.68)	4.56 4.71	11.51 11.22)
4,4'-bis[2-(3-phenylquinoxalyl)]biphenyl (IIb)	48	254-255	C <sub>40</sub> H <sub>26</sub> N <sub>4</sub>	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 <sub>41</sub> H <sub>28</sub> N <sub>4</sub>	85.39 (85.16)	4.89 4.94	9.72 9.38)
4,4'-bis[2-(3-phenylquinoxalyl)]diphenyl ether (IIId)	92 <sup>b</sup>	225-226	C <sub>40</sub> H <sub>26</sub> N <sub>4</sub> O	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 <sub>40</sub> H <sub>26</sub> N <sub>4</sub> S	80.78 (80.92)	4.41 4.62	9.42 9.15)

a) Reported<sup>1</sup> 226-227°, this article also has the incorrect analysis reported for this compound, p. 1457.

b) Previously reported<sup>4</sup> but the analysis was incorrect.

OPPI BRIEFS

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 S. Wawzonek\* and J. E. Durham  
(6/1/76) Department of Chemistry  
University of Iowa  
Iowa City, Iowa 52242

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<sub>3</sub>) δ1.30 (t, 6H, 2CH<sub>2</sub>CH<sub>3</sub>), 4.32 (q, 4H, 2CH<sub>2</sub>CH<sub>3</sub>), 5.10 (s, 4H, 2OCH<sub>2</sub>), 7.10-7.50 (m, 2C<sub>6</sub>H<sub>5</sub> and 2CH); m/e 434.

Anal. Calcd. for C<sub>26</sub>H<sub>26</sub>O<sub>6</sub>: C, 71.86; H, 6.04.

Found: C, 72.21; H, 5.95.