

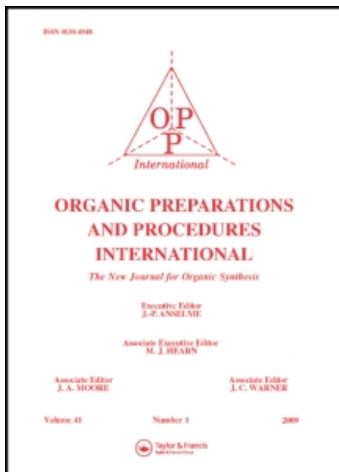
This article was downloaded by:

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>

A CONVENIENT PREPARATION OF DIPHENYLISOBENZOFURAN AND DIPHENYLISOBENZOTHIOPHENE

K. T. Potts^a; A. J. Elliott^a

^a Department of Chemistry, Rensselaer, Polytechnic Institute, Troy, New York

To cite this Article Potts, K. T. and Elliott, A. J.(1972) 'A CONVENIENT PREPARATION OF DIPHENYLISOBENZOFURAN AND DIPHENYLISOBENZOTHIOPHENE', *Organic Preparations and Procedures International*, 4: 6, 269 – 272

To link to this Article: DOI: 10.1080/00304947209458276

URL: <http://dx.doi.org/10.1080/00304947209458276>

PLEASE SCROLL DOWN FOR ARTICLE

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

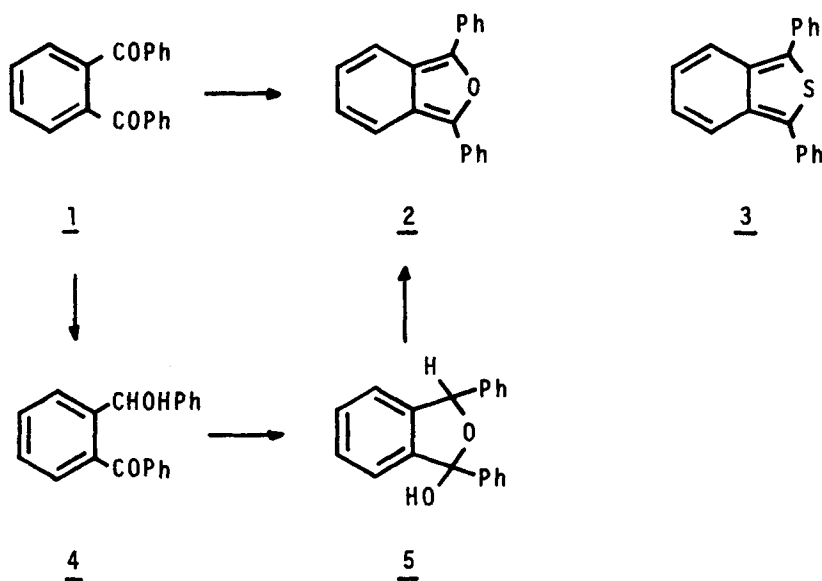
This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

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.

A CONVENIENT PREPARATION OF DIPHENYLISOBENZOFURAN
AND DIPHENYLISOBENZOTHIOPHENE¹

K.T. Potts* and A.J. Elliott

Department of Chemistry, Rensselaer
Polytechnic Institute, Troy, New York 12181



Diphenylisobenzofuran (2), a highly reactive trapping agent for olefinic and acetylenic dipolarophiles,² has been prepared³ from o-dibenzoylbenzene (1) by repetitive potassium borohydride reduction followed by treatment of the reduced product with acid. Oxidation of 2 + 1 is a troublesome feature associated with the use of 2 and can only be

avoided by rigorous exclusion of air, especially if prolonged reaction times are involved.⁴

We have found the following procedure to be both economical and useful for obtaining 2 free from 1, an impurity often present in many preparations. Reduction of 1 in methanol with an excess of sodium borohydride, followed by removal of the solvent and treatment of the residue with hot acetic anhydride provides 2 in yields of the order of 70%. Isolation of the presumed intermediate alcohol 4 is not necessary nor advisable, since attempted purification of this product leads either to oxidation to 1 or dehydration to 2; it is possible that 5 is the intermediate involved.³

Diphenylisobenzothiophene (3) though less reactive than 2, does form adducts with maleic anhydride and benzyne⁵ and has been prepared⁶ by the action of P₂S₅ and 2. The conversion of 2 into 3 does not go to completion, the separation of 2 and 3 being possible by the preferential reaction of 2 with N-phenylmaleimide.⁷ An alternative preparation of 3 from o-dibenzoylbenzene (1) by successive treatment with PCl₅ and potassium ethyl xanthate has also been reported.⁸ We have found that the direct action of P₂S₅/pyridine⁹ on 1 results in excellent yields of diphenylisobenzothiophene (3).

EXPERIMENTAL¹⁰

Diphenylisobenzofuran (1).- o-Dibenzoylbenzene (2.9 g, 0.01 mole) in refluxing methanol (130 ml) was treated with sodium borohydride (0.4 g, 0.012 mole) added portionwise over 10 min. After 15 min. of further reflux, the solvent

A CONVENIENT PREPARATION OF DIPHENYLISOBENZOFURAN

was evaporated and acetic anhydride (75 ml) was added to the residue. The reaction mixture was heated to reflux (15 min.) and then cooled. The orange-yellow needles of 1 which separated were collected, washed well with cold water and dried to give 1.9 g (70%) of 1, mp. 129-130°, lit.³ mp 130-131°.

Diphenylisobenzothiophene (3).- o-Dibenzoylbenzene (2.9 g, 0.01 mole), P₂S₅ (3.3 g, 0.015 mole), and dry pyridine (100 ml) were heated together under reflux for 3 hr, the reaction mixture cooled, and then poured into 1 L of water. Acetic acid (10 ml) was added to coagulate the precipitate which was collected, washed well with water and dried. It crystallized¹¹ from cyclohexane as bright yellow needles, 2.1 g (74%), mp. 120-121°, lit.³ mp. 119-120°.

REFERENCES

- (1) Support of this work by U.S. Public Health Service Research Grant HE-09991, National Heart and Lung Institute, is gratefully acknowledged.
- (2) L. F. Fieser and M. Fieser, "Reagents for Organic Synthesis," John Wiley and Sons, Inc., New York, N. Y., 1972, p. 342.
- (3) M. P. Cava, M. J. Mitchell and A. A. Deana, J. Org. Chem., 25, 1481 (1960).
- (4) Diphenylisobenzofuran is available commercially. However, this present procedure provides an economic recycling process for the laboratory.
- (5) B. Iddon, Advances in Heterocycl. Chem., 14, 359 (1972).
- (6) Ref. 5, p. 357.
- (7) C. Dufraisse and D. Daniel, Bull. soc. chim. Fr., 4, 2063 (1937).
- (8) A. Schonberg and E. Frese, Chem. Ber., 101, 701 (1968).

K. T. POTTS AND A. J. ELLIOTT

- (9) Many reports on the use of this reagent have appeared. See e.g., E. Klingsberg and D. Pappa, J. Amer. Chem. Soc., 73, 4988 (1951); E. A. Falco, E. Pappas and G. H. Hitchings, ibid., 78, 1938 (1956); J. J. Fox, I. Wempen, A. Hampton and I. L. Doerr, ibid., 80, 1669 (1958).
- (10) All evaporations were done under vacuum using a rotatory evaporator. Mps. were taken in capillaries.
- (11) The purity of the pyridine, as well as that of the starting material, has an appreciable effect on the quality of the crude product. In unfavorable cases, chromatography on silica gel (60 g) using benzene as eluent was found to be satisfactory for purification of product.

(Received December 6, 1972)