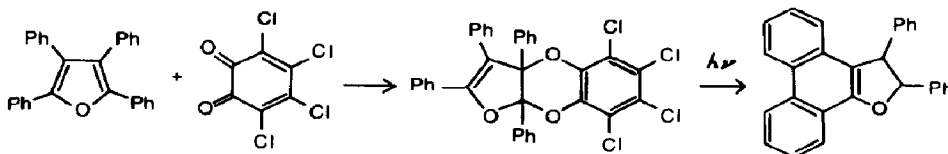


## Organic Photochemical Synthesis

2,3-Diphenylphenanthro[9,10-*b*]furan

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## 1. Procedure

1.1. 5,6,7,8-Tetrachloro-3a,9a-dihydro-2,3,3a,9a-tetraphenylfuro[2,3-*b*][1,4]benzodioxin

Tetraphenylfuran (3.72 g, 0.01 mol) (note 1) and tetrachloro-1,2-benzoquinone (2.46 g, 0.01 mol) (note 2) are heated at reflux in dry benzene (50 ml). Removal of the solvent by rotary evaporation gives a pale yellow solid, which is recrystallized from benzene-(petroleum ether) (boiling point, 60 - 80 °C) to give the dioxin (4.3 g, 70%) (melting point, 202 - 203 °C;  $\nu_{\max}$ (Nujol), 1130, 1060, 1040 and 1010  $\text{cm}^{-1}$ ).

1.2. 2,3-Diphenylphenanthro[9,10-*b*]furan

The dioxin (0.62 g, 0.001 mol) is suspended in dry propan-2-ol (500 ml), and the mixture is irradiated in a Pyrex immersion apparatus for 8 h under an atmosphere of nitrogen (notes 3 and 4). The propan-2-ol is removed by rotary evaporation, and the resultant material is chromatographed on alumina (20 g). Benzene elutes a solid product (0.29 g, 79%), which is recrystallized from either benzene-(petroleum ether) (boiling point, 60 - 80 °C) or cyclohexane to yield 2,3-diphenylphenanthro[9,10-*b*]furan (0.18 g, 48%) (melting point, 202.5 - 203 °C) (note 5).

## 2. Notes

(1) Tetraphenylfuran is prepared by the self-condensation of benzoin using hydrochloric acid [1].

(2) Tetrachloro-1,2-benzoquinone can be purchased from Aldrich.

(3) The photolysis apparatus used is a conventional immersion apparatus with a Pyrex immersion well surrounding a 450 W medium pressure mercury arc lamp. The suspension of the dioxin in propan-2-ol is flushed with nitrogen prior to and during the photolysis. The flow of nitrogen, passed into the solution by means of a long aspirating needle, is also used to ensure adequate mixing of the suspended solid.

(4) The progress of the reaction can be monitored by thin layer chromatography on alumina using benzene-(petroleum ether) as eluent.

(5) Previously [2], the melting point of the furan had been reported as 194 °C.

### 3. Merits of the preparation

The free tetraphenylfuran does not undergo photocyclization to give the phenanthrofuran, but the benzodioxin acts as an activating group. This method for the preparation of 2,3-diphenylphenanthro[9,10-*b*]furan is simple and does not involve many steps or tedious chromatographic separations. Furthermore it lends itself to the synthesis of previously unknown phenanthrofurans such as 2-phenylphenanthro[9,10-*b*]furan, which can be prepared by the use of 2,3,5-triphenylfuran in place of tetraphenylfuran [3].

- 1 S. Patai, Y. Halpern and M. Michman, *U.S. Patent 3,359,284*, December 19, 1967; *Chem. Abstracts*, 69 (1968), no. 10347.
- 2 W. W. Sullivan, D. Ullman and H. Shechter, *Tetrahedron Lett.*, (1969) 2101.
- 3 D. T. Anderson and W. M. Horspool, *J. Chem. Soc., Perkin Trans. I*, (1972) 532, 536.

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