

3,4-Diphenylfuran *N*-Oxide

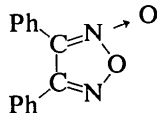
BY ALAN K. SILLITOE AND MARJORIE M. HARDING

Chemistry Department, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

(Received 12 January 1978; accepted 28 January 1978)

Abstract. C₁₄H₁₀N₂O₂, monoclinic, $P2_1/c$, $a = 15.151(5)$, $b = 6.130(9)$, $c = 12.659(3)$ Å, $\beta = 94.36(3)^\circ$, $Z = 4$, $D_m = 1.35$, $D_c = 1.35$ g cm⁻³; Final $R = 0.049$ for 1212 observed reflections. The phenyl rings are at angles of 17 and 59° to the five-membered furazan *N*-oxide ring. There is some disorder in the position of the exocyclic O atom; in 16% of molecules it is bonded to the N atom adjacent to the (nearly) coplanar phenyl ring while in 84% it is bonded to the more distant N atom.

Introduction. We are making a comparative study of bond lengths and stereochemistry in relation to chemical structure and thermal stability in furazans and furazan *N*-oxides (furoxans). The title compound, otherwise known as 3,4-diphenylfuroxan,



prepared by the method of Boyer, Reinisch, Danzig, Stoner & Sahhar (1955), was provided by Mr J. F. Barnes and Dr R. M. Paton and recrystallized from ethanol/petroleum ether. Intensities were recorded on a Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å), an ω - 2θ scan, and $\theta_{\max} = 25^\circ$. Lp corrections were made, but no absorption corrections were applied. Phase solution by manual symbolic addition used triple-phase relationships for 160 reflections with $E > 1.4$, and eighteen atoms were located in the first E map. After isotropic then anisotropic least-squares refinement (to $R = 0.11$) and inclusion of H atoms, a difference synthesis indicated disorder of the furoxan ring, affecting primarily the exocyclic O(2). A second exocyclic O atom, O(22), was introduced, attached to N(1), and the population parameters of these two O atoms refined to 0.84 and 0.16. Subsequent least-squares refinement, with the population parameters of these O atoms fixed, their thermal-vibration parameters and those of H treated as isotropic, and a weighting function $w = 1/(1 - 0.01F + 0.0002F^2)$, led to a final R of 0.049 for 1212 observed reflections [*i.e.* those with $F > 3\sigma(F)$]. All

computer calculations were done with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).*

Discussion. Atom parameters are given in Table 1 and important bond lengths and angles in Fig. 1. The bond lengths in the furoxan ring are in agreement with those in other furoxans, *e.g.* acenaphthofuroxan (Calleri,

* Lists of structure factors, thermal parameters, bond lengths, bond angles and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33373 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Positional parameters ($\times 10^4$, for H $\times 10^3$) and their *e.s.d.*'s

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	6864 (2)	6566 (6)	4879 (3)
N(2)	7977 (2)	4731 (5)	4221 (3)
O(1)	7406 (2)	4773 (5)	5054 (2)
O(2)*	8497 (2)	3283 (6)	4257 (3)
O(22)*	6470 (11)	6380 (27)	5461 (13)
C(1)	7098 (2)	7534 (6)	4015 (3)
C(2)	6615 (2)	9465 (6)	3620 (3)
C(3)	6662 (3)	10153 (7)	2589 (3)
C(4)	6207 (3)	11986 (9)	2231 (5)
C(5)	5690 (3)	13106 (9)	2865 (6)
C(6)	5620 (4)	12425 (10)	3874 (6)
C(7)	6078 (3)	10613 (9)	4259 (4)
C(8)	7808 (2)	6416 (5)	3599 (3)
C(9)	8344 (2)	6816 (5)	2689 (2)
C(10)	8383 (3)	5282 (7)	1900 (3)
C(11)	8891 (3)	5676 (8)	1061 (3)
C(12)	9350 (3)	7579 (8)	1012 (3)
C(13)	9319 (2)	9107 (7)	1788 (3)
C(14)	8810 (2)	8739 (6)	2631 (3)
H(3)	705 (2)	934 (6)	211 (3)
H(4)	620 (3)	1234 (9)	151 (4)
H(5)	535 (3)	1441 (8)	259 (4)
H(6)	531 (3)	1317 (8)	438 (4)
H(7)	606 (3)	1008 (7)	494 (3)
H(10)	890 (2)	398 (6)	195 (3)
H(11)	890 (2)	464 (6)	50 (3)
H(12)	968 (2)	781 (6)	41 (3)
H(13)	963 (2)	1046 (6)	176 (3)
H(14)	879 (2)	977 (5)	321 (2)

* Population parameters: O(2) 0.86, O(22) 0.14.

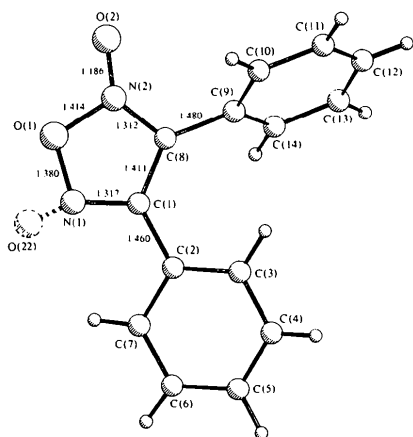


Fig. 1. One molecule of diphenylfuroxan. The bond lengths shown have e.s.d.'s of 0.005 Å. O(22) is the alternative position found for $\frac{1}{3}$ of the O(2) atoms.

Chiari & Viterbo, 1973), and two forms of methyl-*p*-bromophenylfuroxan (Calleri, Ferraris & Viterbo, 1969*a,b*). The atoms of the furoxan ring [N(1),N(2),O(1),O(2),C(1),C(8)] together with C(2) and C(9) are coplanar within 0.03 Å but the phenyl rings are twisted out of this plane; ring (I) is 17° from the furoxan plane and ring (II) 59°. Repulsion of non-bonded atoms would be expected to prevent the two rings being coplanar (as in stilbene or hexaphenylbenzene). In this crystal environment one phenyl ring is twisted much further out of plane than the other; thus

the alternative position, O(22), for the exocyclic O(2) corresponds to a stereochemically different isomer. Rapid interchange between isomers should be possible in solution. The conformation is similar to that of diphenylfuroxan (Mellini & Merlino, 1976) where the phenyl groups are twisted 19 and 61° from the furazan plane.

We thank the Science Research Council for a research studentship to AKS, the Edinburgh Regional Computing Centre for their cooperation, and Mr S. Motherwell for the program *PLUTO* used to prepare the illustration.

References

- BOYER, J. H., REINISCH, R. F., DANZIG, M. J., STONER, G. A. & SAHAR, F. (1955). *J. Am. Chem. Soc.* **77**, 5688–5690.
- CALLERI, M., CHIARI, G. & VITERBO, D. (1973). *Cryst. Struct. Commun.* **2**, 335–338.
- CALLERI, M., FERRARIS, G. & VITERBO, D. (1969*a*). *Acta Cryst.* **B25**, 1126–1133.
- CALLERI, M., FERRARIS, G. & VITERBO, D. (1969*b*). *Acta Cryst.* **B25**, 1133–1139.
- MELLINI, M. & MERLINO, S. (1976). *Acta Cryst.* **B32**, 1079–1082.
- STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland; implemented for the ICL 4/75 computer at Edinburgh Regional Computing Centre.

Acta Cryst. (1978). **B34**, 2022–2025

(3*aRS*,6*RS*,6*aRS*,7*RS*,9*aSR*)-7-Benzyl-5,6-dimethyl-3,3*a*,6,6*a*,7,8-hexahydrofuro-[3,4-*h*]isoindole-1,9(9*aH*)-dione

BY PETER G. JONES AND OLGA KENNARD*

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

(Received 13 January 1978; accepted 28 January 1978)

Abstract. C₁₉H₂₁NO₃, *M_r* = 311.15, triclinic, *P* $\bar{1}$, *a* = 11.128 (6), *b* = 12.016 (8), *c* = 13.722 (7) Å, α = 113.37 (5), β = 94.45 (5), γ = 95.68 (6)°, *U* = 1622 Å³, *Z* = 4, *D_x* = 1.243 g cm⁻³, μ (Mo *K*α) = 0.5 cm⁻¹. There are two independent molecules, linked by hydrogen bonds, in the asymmetric unit. The structure was refined to *R* = 0.061 for 4209 independent reflexions. The relative configurations at the five asym-

metric centres have been established to be those given in the title.

Introduction. The title compound (Fig. 1) was prepared as an intermediate during studies towards the synthesis of cytochalasan (Owens & Raphael, 1978). The structure determination was undertaken to establish the relative configurations at the five asymmetric centres. A related compound, obtained in the same synthetic pathway, has also been investigated crystallo-

* External Staff, Medical Research Council.