The seventh oxygen O(20) is 2·85 (1) Å above the basal hexagon, *i.e.* in an 'apical' position; this results from a change of C(15)—C(16)—O(17)—C(18) from the usual *anti* conformation to −86·4 (5)° and from the O(17)—C(18)—C(19)—O(20) unit displaying an $ag^{-}a$ conformation.

The same sign of the *gauche* angles about C(15)—C(16) and C(18)—C(19) is indicative of a helical structure, as found in crystalline poly(ethylene oxide) (Dale, 1980). C(1)—O(2)—C(3)—C(4) = −100·8 (4)° is probably a consequence of steric hindrance.

The observed conformation of the polyether is such as to allow (i) for all its O atoms Ba···O contacts only 0·1–0·2 Å longer than the sum of the respective radii and (ii) additional interactions between Ba^{2+} and the anions (Table 2).

There are no obvious linkages between the complex units.

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References

CLEGG, W. (1981). *Acta Cryst.* A**37**, 22–28.
DALE, J. (1980). *Isr. J. Chem.* **20**, 3–11.
HILGENFELD, R., HIRAYAMA, F., BHAGWAT, V., SAENGER, W., WEBER, G., SHELDRICK, G. M., SIEGER, H. & VÖGTLE, F. (1984). *Isr. J. Chem.* In the press.
HILGENFELD, R. & SAENGER, W. (1982). *Top. Curr. Chem.* **101**, 1–82.
HODGSON, K. O. & RAYMOND, K. N. (1972). *Inorg. Chem.* **11**, 3030–3035.
HUGHES, D. L., MORTIMER, C. L. & TRUTER, M. R. (1978). *Inorg. Chim. Acta*, **28**, 83–89.
HUGHES, D. L. & WINGFIELD, J. N. (1978). *J. Chem. Soc. Chem. Commun.* pp. 1001–1003.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press.
IWAMOTO, R. (1973). *Bull. Chem. Soc. Jpn*, **46**, 1114–1127.
IWAMOTO, R. & WAKANO, H. (1976). *J. Am. Chem. Soc.* **98**, 3764–3768.
MAVERICK, E., SEILER, P., SCHWEIZER, W. B. & DUNITZ, J. D. (1980). *Acta Cryst.* B**36**, 615–620.
PARSONS, D. G., TRUTER, M. R. & WINGFIELD, J. N. (1981). *Inorg. Chim. Acta*, **51**, 93–97.
SAENGER, W., SUH, I.-H. & WEBER, G. (1979). *Isr. J. Chem.* **18**, 253–258.
SIEGER, H. & VÖGTLE, F. (1978). *Angew. Chem.* **90**, 212–213.
SUH, I.-H., NAMGUNG, H., KU YOON, Y., SAENGER, W. & VÖGTLE, F. (1984). *J. Incl. Phenom.* In the press.
TÜMMLER, B., MAASS, G., VÖGTLE, F., SIEGER, H., HEIMANN, U. & WEBER, E. (1979). *J. Am. Chem. Soc.* **101**, 2588–2598.
VÖGTLE, F. & SIEGER, H. (1977). *Angew. Chem.* **89**, 410–412.
VÖGTLE, F. & WEBER, E. (1979). *Angew. Chem.* **91**, 813–837.
YAMAGUCHI, I., MIKI, K., YASUOKA, N. & KASAI, N. (1982). *Bull. Chem. Soc. Jpn*, **55**, 1372–1375.

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Order and Disorder in the Structure of Dibenzofuran, $C_{12}H_8O$

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Abstract. $M_r = 168{\cdot}20$, orthorhombic, *Pnma*, $a = 7{\cdot}5154\ (8)$, $b = 19{\cdot}098\ (2)$, $c = 5{\cdot}7739\ (6)$ Å, $V = 828{\cdot}7\ (2)$ Å^3, $Z = 4$, $F(000) = 352$, $D_x = 1{\cdot}348$ g cm^{-3}, $T = 169$ K, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}789$ cm^{-1}, $R = 0{\cdot}039$, $wR = 0{\cdot}040$ for 1254 reflections with $I > 3\sigma_I$. The dibenzofuran molecule has mirror symmetry, with the reflection plane passing through the O atom and bisecting the furan ring. The five- and six-membered rings are planar, and have a dihedral angle of 1·5°. The average C—C bond length in the six-membered ring is 1·392 Å. The structure is disordered, with 9% of the molecules adopting an alternative orientation. The disordered molecules are related to the ordered ones by a 180° rotation about an axis which passes through the center of mass and is perpendicular to the furan ring. Reanalysis of the data provided by previous studies of dibenzofuran at room temperature shows that disorder similar in both kind and degree was present, but was not reported. Accounting for the disorder brings the C—O bond length into agreement with values from similar compounds, thus removing an earlier discrepancy.

Introduction. The structure of dibenzofuran at room temperature has been reported by Dideberg, Dupont & André (1972) and by Banerjee (1973). The present determination of the structure at 169 K was undertaken in conjunction with low-temperature EPR experiments on the lowest triplet state of biphenyl dilutely oriented in dibenzofuran host crystals. Since certain aspects of the observed low-temperature magnetic resonance spectra could be reasonably ascribed to the

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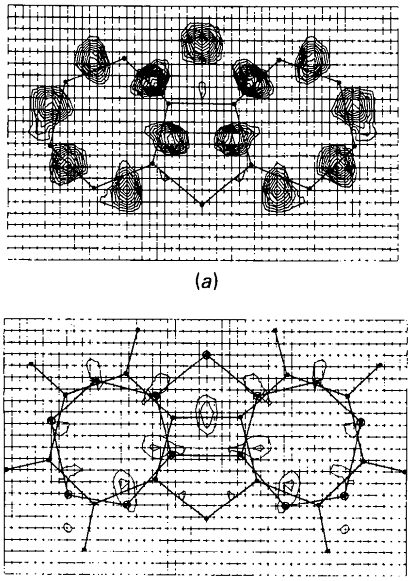
occurrence of a symmetry lower than that reported for the room-temperature structure, it was of interest to determine whether dibenzofuran indeed undergoes a phase transition below room temperature. The present study was directed toward resolving this question for the range from room temperature to 169 K.

Experimental. Dibenzofuran obtained from Aldrich (#13,568-2) was purified by vertical, multipass zone refining (630 passes at $2{\cdot}5\ \mathrm{cm^{-1}h^{-1}}$). After the final refinement cycle, a single-crystal specimen was obtained from a crystalline region near the center of the ingot. The clear, irregularly shaped crystal was cut into a parallelepiped ($0{\cdot}46 \times 0{\cdot}48 \times 0{\cdot}58$ mm), mounted on a glass rod, and cooled to 169 K on a Syntex $P\bar{1}$ automated four-circle diffractometer equipped with an LT-1 low-temperature attachment. Temperature measurements were made at the cold-stream exit nozzle during data collection and at the crystal site after data collection with a Fluke 2100A digital thermometer (Type K thermocouple). The temperature measured at the site of the crystal was 169 K, with an estimated uncertainty of ± 2 K.

Unit-cell parameters from a least-squares fit of the diffractometer setting angles for 25 centered reflections ($20° \leq 2\theta \leq 30°$). 2304 unique reflections, $4° \leq 2\theta \leq 70°$ ($+h$, $+k$, $+l$), ω–2θ scan technique. Uncertainties assigned to each reflection using the formula $\sigma_I^2 = R^2\ (C + 4B) + (0{\cdot}02I)^2$, where R is the variable scan rate, C the total number of counts, B the total number of background counts, and I the intensity. Six standard reflections (121, 153, 241, 222, 311, 480): intensity variations of about 4% throughout data collection. The seven strongest reflections (020, 040, 101, 111, 200, 210, 211) saturated the detector and were not used in the least-squares refinement. Data corrected for Lorentz and polarization effects; analytical absorption correction applied using the program of Alcock (1970), transmission factors 0·96–0·98. The systematic absences as well as the results from the zero-moment test of Howells, Phillips & Rogers (1950) suggested that the space group had not changed from that of the room-temperature structure, *Pnma* (D_{2h}^{16}). This space group was adopted, and the deletion of the systematic absences left 2033 reflections, 1254 of which had $I > 3\sigma_I$.

Positions of O and C atoms from an E map generated by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement (*SHELX*76, Sheldrick, 1976) for reflections with $I > 3\sigma_I$; $\sum \sigma_F^{-2}(|F_o| - |F_c|)^2$ minimized. After anisotropic thermal parameters were introduced, the refinement converged to $R = 0{\cdot}11$. H-atom positions determined from an electron density difference map, and further refinement including isotropic H atoms converged to $R = 0{\cdot}092$ and $wR = 0{\cdot}095$; $w = 1/\sigma_F^2$. The values of these agreement indices were much larger than expected, and the appearance of the electron density difference map (Fig. 1*a*) indicated that some aspects of the structure were not accounted for by our model. The arrangement of the 13 largest peaks suggested the presence of a dibenzofuran molecule in an alternative orientation. The height of the largest peak was $1{\cdot}24\ \mathrm{e\ Å^{-3}}$, indicating that the space-group assignment was probably correct, but that a small degree of disorder was present in the crystal.

As can be seen from Fig. 1(*a*), the C atoms of the disordered molecule are located near the centers of the C—C bonds of the ordered molecule. In an attempt to separate the electron density of the disordered molecule from the bonding electron density of the ordered molecule, a high-angle refinement was performed on the 616 reflections satisfying the conditions $(\sin\theta/\lambda) \geq 0{\cdot}6\ \mathrm{Å^{-1}}$ and $I > 3\sigma_I$. The resultant positional and thermal parameters for the disordered molecule, as well as the occupancy factors for all the atoms, were then held constant in a refinement of the ordered molecule using all of the data. The final refinement cycle converged with $R = 0{\cdot}039$, $wR = 0{\cdot}040$, $S = 1{\cdot}8$, and $(\Delta/\sigma)_{\max} = 0{\cdot}04$. The final electron density difference map (Fig. 1*b*) had max. and min. peak heights of 0·31 and $-0{\cdot}28\ \mathrm{e\ Å^{-3}}$. Scattering factors for C and O from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965).


(*a*)

(*b*)

Fig. 1. (*a*) Electron density difference map (based on the ordered molecule only) showing the presence of disorder in the dibenzofuran crystal structure. Contours start at $0{\cdot}2\ \mathrm{e\ Å^{-3}}$ and are drawn in steps of $0{\cdot}1\ \mathrm{e\ Å^{-3}}$. (*b*) Electron density difference map calculated after the final refinement cycle. Contours start at $0{\cdot}1\ \mathrm{e\ Å^{-3}}$ and are drawn in steps of $0{\cdot}1\ \mathrm{e\ Å^{-3}}$. Atoms of the disordered molecule are denoted by the symbol ⊙.

Discussion. The final atomic coordinates and temperature factors are given in Table 1.*

The asymmetric unit consists of one half of the dibenzofuran molecule, the other half of the molecule being related by a mirror plane which passes through the O atom and bisects the furan ring. The individual five- and six-membered rings are planar, but not coplanar, having a dihedral angle of 1·5° between the benzene and furan rings. This gives the dibenzofuran molecule a slight 'boat' conformation.

The least-squares planes for the furan rings of the corresponding ordered and disordered molecules coincide, as do the centers of mass (within one standard deviation). This indicates that the ordered and disordered dibenzofuran molecules are related by a 180° rotation about an axis which passes through the centers of mass and is perpendicular to the furan rings.

Bond lengths and angles for the ordered dibenzofuran molecule, calculated using the *XRAY* system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972), are shown in Fig. 2(*a*). The average C—C bond length in the benzene ring is 1·392 Å, a typical value for aromatic C—C bonds. The O—C(1) and C(6)—C(6')† bond lengths are 1·384 (1) and 1·438 (1) Å, respectively, which are each considerably shorter than the corresponding values of 1·418 (6) and 1·480 (6) Å found for the bond lengths by Banerjee, and the values of 1·404 (3) and 1·481 (3) Å found by Dideberg *et al.* The bond lengths and angles calculated for the disordered molecule are given in Fig. 2(*b*). Although corresponding angles agree fairly well for the ordered and disordered molecules, there is only fair agreement between the corresponding bond lengths. We attribute these differences to the relatively small population of the disordered molecule and to the closeness of the disordered C atoms to the high bonding electron density regions of the ordered molecule, both of which limit the accuracy to which the atomic coordinates of the disordered molecule can be determined.

A stereoview of the dibenzofuran unit cell is shown in Fig. 3. The smallest intermolecular contact distance, 2·6 Å, occurs between H atoms, and compares favorably with the expected van der Waals separation of 2·4 Å. The contact distances between the ordered and disordered molecules are similar to those between two ordered molecules. Since it was not possible to locate the H atoms of the disordered molecule, further comparison cannot be made.

Table 1. *Atomic coordinates and thermal parameters* ($Å^2 \times 10^4$)

Atoms in the disordered positions are denoted by the letter *D*. *k* is the occupancy factor. $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ [for O to C(6)].

	x	*y*	*z*	*k*	U_{eq}/U_{iso}
O	0·7516 (1)	0·25	0·7713 (2)	0·454 (7)	301 (3)
C(1)	0·7024 (1)	0·30769 (5)	0·6412 (2)	0·908 (14)	255 (2)
C(2)	0·7328 (1)	0·37636 (6)	0·7040 (2)	0·908 (14)	313 (3)
C(3)	0·6764 (1)	0·42709 (6)	0·5482 (2)	0·908 (14)	331 (3)
C(4)	0·5935 (1)	0·40912 (6)	0·3395 (2)	0·908 (14)	312 (3)
C(5)	0·5653 (1)	0·33965 (5)	0·2793 (2)	0·908 (14)	271 (2)
C(6)	0·6216 (1)	0·28766 (5)	0·4332 (2)	0·908 (14)	231 (2)
H(2)	0·793 (2)	0·3884 (7)	0·850 (2)	0·908 (14)	400 (34)
H(3)	0·690 (1)	0·4751 (7)	0·588 (2)	0·908 (14)	409 (34)
H(4)	0·555 (2)	0·4464 (7)	0·237 (2)	0·908 (14)	431 (35)
H(5)	0·510 (2)	0·3275 (6)	0·133 (2)	0·908 (14)	314 (31)
O(*D*)	0·541 (2)	0·25	0·237 (3)	0·046 (7)	277 (49)
C(1*D*)	0·593 (2)	0·3076 (8)	0·363 (3)	0·092 (14)	203 (45)
C(2*D*)	0·572 (2)	0·375 (1)	0·296 (3)	0·092 (14)	320 (50)
C(3*D*)	0·627 (3)	0·425 (1)	0·438 (4)	0·092 (14)	330 (49)
C(4*D*)	0·721 (3)	0·407 (2)	0·656 (4)	0·092 (14)	396 (54)
C(5*D*)	0·735 (3)	0·339 (2)	0·717 (4)	0·092 (14)	375 (54)
C(6*D*)	0·668 (2)	0·2891 (8)	0·564 (3)	0·092 (14)	247 (45)

It was thought initially that the disorder characterized in this study might be the result of our crystal-growth method, since Banerjee grew his dibenzofuran crystals by slow evaporation from an alcohol solution at ambient temperature, and Dideberg *et al.* grew theirs by the Bridgman–Stockbarger method, and neither report made mention of disorder. In view of this speculation, it was of interest to generate final electron density difference maps from the earlier sets of published data. These difference maps, shown in Fig. 4, clearly indicate the presence of disorder in each of those structures. Inclusion in the least-squares refinement of an isotropic O atom at the disordered position produced a decrease in the *R* factor from 0·086 to 0·079 for Banerjee's data and from 0·082 to 0·072 for the data of Dideberg *et al.*, with 8 (2) and 11 (1)% of the molecules being disordered, respectively. These results indicate that the disorder in our crystal did not arise solely from the particular crystal-growth method, but that it is a rather common property exhibited by dibenzofuran crystals grown at or above room temperature.

The differences between the bond lengths and angles found in this study of dibenzofuran and those found in the previous studies can now be rationalized. Neglect of the disordered molecule in the refinement causes the C atoms of the ordered molecule to converge to positions which are slightly different from their 'true' positions, due to the nearby small electron density not accounted for by the model. The presence or absence of the disordered molecule has little effect on the position of the O atom of the ordered molecule, since there is no other electron density nearby. When the disordered molecule is included in the refinement, the C atoms of the ordered molecule can converge to their 'true' positions. The result in the present case is longer C—C bond lengths in the benzene rings, but shorter O—C(1) and C(6)—C(6') bond lengths in the furan ring, than when disorder was neglected in the refinement.

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* Lists of structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39284 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Primed atoms are related to the corresponding unprimed atoms by the mirror plane.

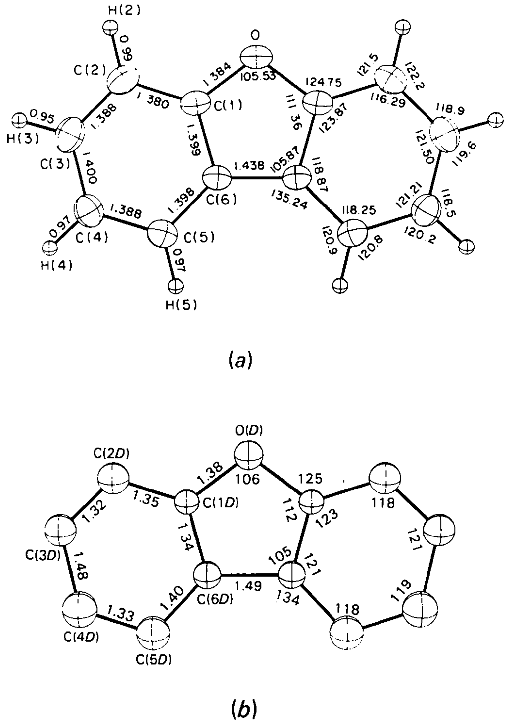

Fig. 2. (*a*) Bond lengths (Å) and angles (°) for (*a*) the ordered dibenzofuran molecule (uncertainties in bond lengths and angles between nonhydrogen atoms are less than 0·002 Å and 0·11°, respectively; for bonds involving H atoms, the uncertainties are less than 0·01 Å and 0·7°), and (*b*) the disordered dibenzofuran molecule (uncertainties in the bond lengths and angles are less than 0·04 Å and 2°, respectively). Thermal ellipsoids are drawn at 50% probability for all atoms except H, for which they have been set artificially small.

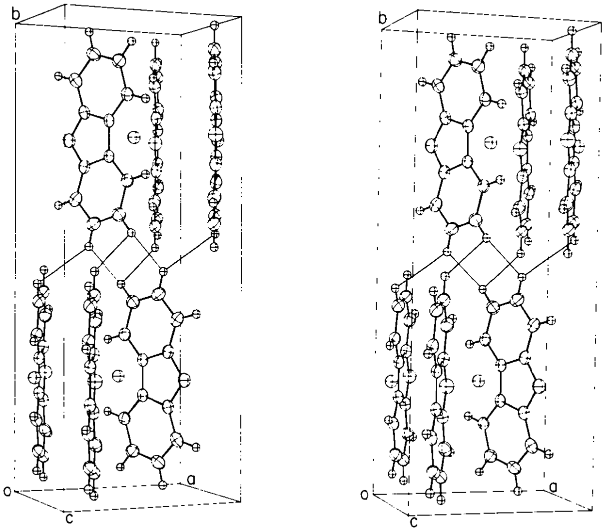

Fig. 3. Stereoview of the dibenzofuran unit cell, drawn using *ORTEP*II (Johnson, 1971). Thermal ellipsoids are drawn at 50% probability for all atoms except H, for which they have been set artificially small. Also shown are the disordered O atoms and the closest contact distance between molecules.

The decreased length of the O—C(1) bond is such that the present value is in good agreement with values from dibenzo-*p*-dioxin and related compounds as reported by Goldberg & Shmueli (1973).

Conclusion. While the appearance of a small but significant degree of disorder in the dibenzofuran structure was unexpected, it is not unprecedented. Hanson (1965) has reported the structure of an azulene:*s*-trinitrobenzene complex, in which 7% of the azulene molecules adopt an alternative orientation. On the other hand, Stezowski, Binder & Karl (1982) have reported the structure of a 1:1 charge-transfer complex between pyromellitic dianhydride and carbazole in which the carbazole molecules randomly adopt one of two orientations, producing a situation similar to that shown in Fig. 1(*a*).

The presence of disorder in dibenzofuran appears to be unrelated to the method of crystal growth (at or above room temperature) and the degree of disorder appears, perhaps fortuitously, to have a nearly constant value. While more highly ordered dibenzofuran crystals might be obtained by slow growth from solution at temperatures below 298 K, we have no present plans for further investigation of this matter.

The structures of dibenzothiophene (Schaffrin & Trotter, 1970), dibenzoselenophene (Hope, Knobler & McCullough, 1970), and dibenzotellurophene (McCullough, 1975) have been reported with no mention of any disorder. We are currently investigating the crystal structures of fluorene and carbazole to determine whether disorder similar to that found in dibenzofuran is present.

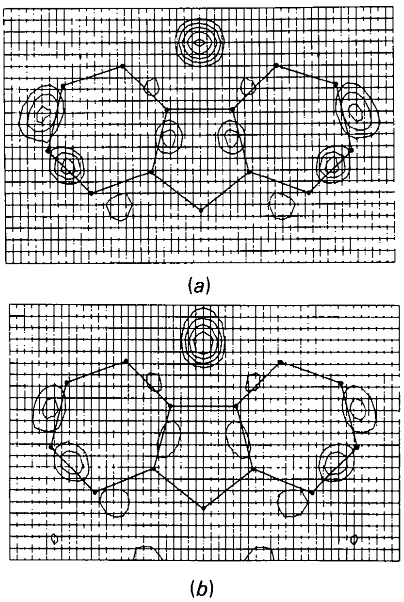

Fig. 4. Electron density difference maps calculated using the results presented by (*a*) Dideberg, Dupont & André (1972), and (*b*) Banerjee (1973). Contours start at 0·1 e Å^{-3} and are drawn in steps of 0·1 e Å^{-3}.

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References

ALCOCK, N. W. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 271–278. Copenhagen: Munksgaard.
BANERJEE, A. (1973). *Acta Cryst.* B**29**, 2070–2074.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
DIDEBERG, O., DUPONT, L. & ANDRÉ, J. M. (1972). *Acta Cryst.* B**28**, 1002–1007.
GOLDBERG, I. & SHMUELI, U. (1973). *Acta Cryst.* B**29**, 432–440.
HANSON, A. W. (1965). *Acta Cryst.* **19**, 19–26.
HOPE, H., KNOBLER, C. & MCCULLOUGH, J. D. (1970). *Acta Cryst.* B**26**, 625–640.
HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* **3**, 210–214.
JOHNSON, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee.
MCCULLOUGH, J. D. (1975). *Inorg. Chem.* **14**, 2639–2643.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
SCHAFFRIN, R. M. & TROTTER, J. (1970). *J. Chem. Soc. A*, pp. 1561–1565.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. W. & HALL, S. R. (1972). The *XRAY* system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
STEZOWSKI, J. J., BINDER, W. & KARL, N. (1982). *Acta Cryst.* B**38**, 2912–2914.

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1-Phenyl-1,2-dihydro-2-quinolinone, $C_{15}H_{11}NO$

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Abstract. $M_r = 221{\cdot}26$, $P2_1/c$, $a = 5{\cdot}621\,(1)$, $b = 16{\cdot}098\,(5)$, $c = 12{\cdot}368\,(3)$ Å, $\beta = 98{\cdot}74\,(2)^\circ$, $V = 1106{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}329$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}70926$ Å, $\mu = 0{\cdot}909$ cm^{-1}, $F(000) = 464$, 298 K, $R_1 = 0{\cdot}042$ based on 1595 observed reflections. The phenyl ring attached to N(1) is rotated to make a dihedral angle of $71{\cdot}9\,(1)^\circ$ with the plane of the quinoline ring, orienting the ring electrons to account for the abnormal chemical shifts in the proton NMR spectrum. The C(3)—C(4) distance of $1{\cdot}333\,(3)$ Å indicates the localization of a double bond at this position.

Introduction. The crystal structure analysis of 1-phenyl-1,2-dihydro-2-quinolinone was undertaken primarily to verify the position of attachment of the phenyl group (both N and O sites are feasible) and to help to understand an unusual chemical shift in its proton NMR spectrum.

Experimental. The crystal was grown from ethanol solution. Intensity data collected with Picker FACS-I four-circle diffractometer, graphite-monochromatized Mo $K\alpha_1$ radiation, stepped 2θ scan (basic step size $= 0{\cdot}10^\circ$ 2θ); $h \pm 6$, $k \pm 19$, $l \pm 14$ (Baenziger, Foster, Howells, Howells, Vander Valk & Burton, 1977); total reflections measured = 11 050, $\sin\theta/\lambda_{\max} = 0{\cdot}59$ Å^{-1}, 1959 unique reflections, 1595 greater than 3σ. No absorption correction [$\mu = 0{\cdot}91$ cm^{-1} and crystal dimensions $\{011\}$ 0·22, (100) 1·3 mm (φ axis approximately parallel to $\mathbf{a}^*$) small]. $R_{\rm int} = 0{\cdot}045$ based on F^2; 3 standard reflections varied < 3%. Cell dimensions determined from 12 carefully centered reflections in 2θ range 36 to 47°. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971) and electron density map methods. All hydrogen atoms located in electron density difference maps. Atom scattering factors for O, N and C from *International Tables for X-ray Crystallography* (1974); for H from Stewart, Davidson & Simpson (1965). Max. and min. electron densities in final difference map = 0·1 e Å^{-3}. Full-matrix least-squares refinement (local adaption of programs from Lawrence Radiation Laboratories, Berkeley) including hydrogen atoms (isotropic) and extinction parameter gave discrepancy factors $R_1 = 0{\cdot}042$, $R_2 = 0{\cdot}070$, $S = 1{\cdot}80$ based on 1595 reflections and 199 parameters. $w = 1/(s^2_F + 0{\cdot}06\,F^2)$, where s_F is the larger of the two estimates of standard deviation in F obtained from counting statistics and agreement among equivalent reflections. Max. $\Delta/\sigma = 0{\cdot}013$ for nonhydrogen atoms and 0·066 for H atoms in the last cycle.

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