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# Structure of 2,8-Dimethoxydibenzofuran*

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**Abstract.** $C_{14}H_{12}O_3$, orthorhombic, $P2_12_12_1$, $a$ = 21·230 (5), $b$ = 11·112 (2), $c$ = 4·805 (2) Å, $Z$ = 4, $V$ = 1133·5 Å$^3$, $D_x$ = 1·34 g cm$^{-3}$. The final residual $R$ is 0·04 for 824 observed reflexions. The molecule adopts a slight boat conformation with the two benzene planes making an angle with the furan plane. The individual rings are not quite planar, and there is a dihedral angle of 4·0 (2·2)° between the planes. The torsion angles calculated around the inner core of the molecule, *i.e.* the bonds C(1)–C(9b), C(9b)–C(9a) and C(9a)–C(9), are 176·5 (0·6), 0·7 (0·7) and −177·1 (0·6)° respectively.

**Introduction.** The crystal structure of the title compound has been investigated in a research program concerning X-ray structure analyses of cyclo-oligomerization products of quinones. Quinones frequently undergo condensation reactions under the influence of strong acids, with the formation of complex dibenzofuran derivatives. 1,4-Naphthoquinone and a number of 2,3-dialkylquinones have been found to give dimers, trimers and tetramers containing dibenzofuran elements. The structures of these dibenzofuranoid products have been elucidated by chemical analyses (Högberg, 1973). X-ray structure analyses, however, were needed to determine the molecular geometries (Berg, Erdtman, Högberg, Karlsson, Pilotti & Söderholm, 1977).

Unit-cell dimensions and intensity data were measured on a Philips PW 1100 diffractometer with Cu $K\alpha$ radiation and the $\theta$–$2\theta$ scan technique. Unit-cell parameters were refined by least squares from the observed $2\theta$ values of 25 reflexions. Of the 1216 independent reflexions with $2\theta \leq 140°$, 824 had intensities greater than $4\sigma(I)$, where $\sigma(I)$ is based on counting statistics. Lorentz and polarization corrections were applied and the structure amplitudes derived. The crystal volume was approximately 0·007 mm$^3$. No absorption correction was applied.

The structure was solved by direct methods with the multisolution procedure (Germain, Main & Woolfson, 1971) and refined by the full-matrix least-squares method with the weighting scheme of Hughes (1941). Table 1 lists the final coordinates of the C and O atoms. The positions of the H atoms are given in Table 2. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The isotropic temperature factor of each H atom was constrained to have the same value as that of the C atom to which it is bonded. For the H atoms, only the positional parameters were refined, resulting in an $R$ value of 0·04 for all 824 observed reflexions.†

Table 1. *Positional parameters* (×10$^4$) *of the non-hydrogen atoms with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| C(1) | 5058(2) | −1062(3) | 11420(7) |
| C(2) | 5634(2) | −493(3) | 11682(8) |
| C(3) | 5768(2) | 567(4) | 10207(10) |
| C(4) | 5344(2) | 1058(4) | 8391(10) |
| C(4A) | 4779(2) | 458(3) | 8051(8) |
| O(5) | 4310(1) | 803(2) | 6249(6) |
| C(5A) | 3841(2) | −57(3) | 6553(8) |
| C(6) | 3292(2) | −77(4) | 5050(9) |
| C(7) | 2885(2) | −1012(4) | 5585(8) |
| C(8) | 3024(2) | −1885(4) | 7604(8) |
| C(9) | 3578(2) | −1846(3) | 9105(8) |
| C(9A) | 3998(2) | −913(3) | 8537(7) |
| C(9B) | 4623(2) | −577(3) | 9522(7) |
| O(10) | 6115(1) | −875(3) | 13366(7) |
| C(11) | 6027(2) | −1959(4) | 14881(11) |
| O(12) | 2566(1) | −2747(3) | 7878(7) |
| C(13) | 2651(2) | −3655(5) | 9917(12) |

Table 2. *Positional* (×10$^3$) *and isotropic thermal* (Å$^2$ × 10$^2$) *parameters of the hydrogen atoms with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | $B$ |
|---|---|---|---|---|
| H(C1) | 497(2) | −176(4) | 1250(10) | 405 |
| H(C3) | 615(2) | 93(4) | 1046(10) | 499 |
| H(C4) | 541(2) | 180(4) | 718(10) | 543 |
| H(C6) | 320(2) | 61(4) | 378(10) | 491 |
| H(C7) | 248(3) | −113(4) | 458(11) | 529 |
| H(C9) | 362(2) | −242(4) | 1049(10) | 424 |
| H1(C11) | 595(2) | −268(5) | 1367(11) | 566 |
| H2(C11) | 567(2) | −198(4) | 1618(11) | 566 |
| H3(C11) | 646(3) | −210(4) | 1595(11) | 566 |
| H1(C13) | 230(3) | −414(5) | 996(12) | 626 |
| H2(C13) | 306(3) | −416(5) | 967(11) | 626 |
| H3(C13) | 274(3) | −332(5) | 1192(13) | 626 |

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* Quinone Oligomerization. I.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33392 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

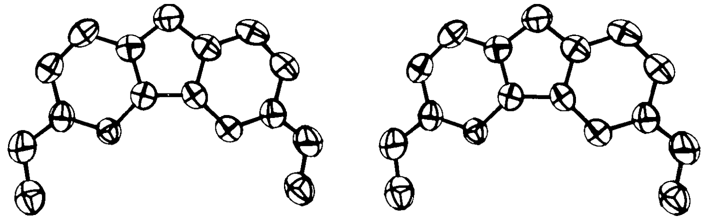

Fig. 1. A stereoscopic view of the molecule.

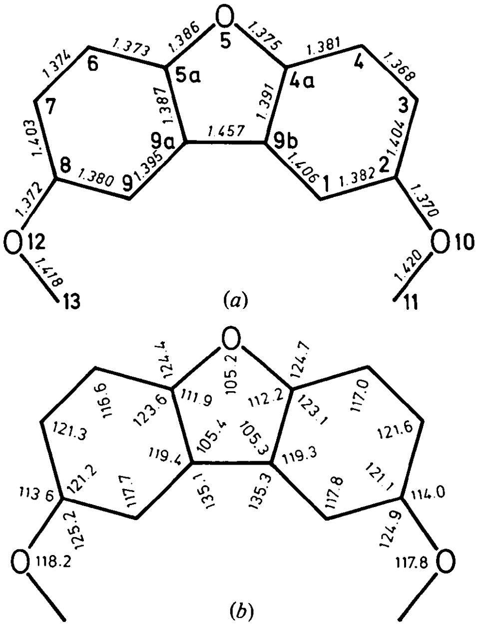

Fig. 2. (*a*) Bond distances (Å) and numbering of atoms. (*b*) Bond angles (°).

**Discussion.** Fig. 1 shows the molecular configuration. Bond lengths and angles, and the numbering of the atoms are given in Fig. 2. Standard deviations are estimated to be 0·005 Å and 0·3° respectively.

The molecule, as a whole, appears to be slightly but significantly boat-shaped. A least-squares plane fitted to all non-hydrogen atoms shows that there is a maximum deviation from planarity of 0·09 Å (see Table 3). The angle between the two benzene rings is 4·0° with an e.s.d. of 2·2°; this is because the individual rings are not strictly planar (see Table 3). The extent of the bowing is indicated by the torsion angles calculated around the inner core of the molecule [*i.e.* the bonds C(1)—C(9b), C(9b)—C(9a) and C(9a)—C(9)]; these are 176·5 (0·6), 0·7 (0·7) and −177·1 (0·6)° respectively. This is in conformity with crystal structure studies of dibenzofuran (Dideberg, Dupont & André, 1972; Banerjee, 1973), ruscodibenzofuran (ElSohly *et al.*, 1977) and related compounds, for example dibenzoselenophene (Hope, Knobler & McCullough, 1970).

Intermolecular distances correspond to van der Waals contacts, and no strong intermolecular interactions are apparent.

Table 3. *Deviations* (Å) *from least-squares planes calculated for the whole molecule and the three separate rings*

| Plane 1 | | | | | |
|---|---|---|---|---|---|
| C(1) | −0·0650 | C(5a) | −0·0012 | C(9b) | −0·0621 |
| C(2) | −0·0007 | C(6) | 0·0666 | O(10) | 0·0497 |
| C(3) | −0·0001 | C(7) | 0·0870 | C(11) | 0·0886 |
| C(4) | −0·0202 | C(8) | 0·0177 | O(12) | 0·0418 |
| C(4a) | −0·0281 | C(9) | −0·0564 | C(13) | −0·0596 |
| O(5) | −0·0048 | C(9a) | −0·0530 | | |
| Plane 2 | | Plane 3 | | Plane 4 | |
| C(1) | −0·0150 | C(5a) | −0·0022 | C(4a) | 0·0034 |
| C(2) | 0·0165 | C(6) | −0·0050 | O(5) | −0·0067 |
| C(3) | −0·0032 | C(7) | 0·0070 | C(5a) | 0·0075 |
| C(4) | −0·0116 | C(8) | −0·0018 | C(9a) | −0·0052 |
| C(4a) | 0·0132 | C(9) | −0·0053 | C(9b) | 0·0010 |
| C(9b) | 0·0002 | C(9a) | 0·0072 | | |

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