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# 1,4-Dimethoxybenzocycloheptene-5,9(6*H*,8*H*)-dione

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**Abstract.** $C_{13}H_{14}O_4$, orthorhombic, *Pnam* (non-standard setting), $a = 8{\cdot}903\ (1)$, $b = 8{\cdot}077\ (2)$, $c = 16{\cdot}952\ (3)$ Å, $Z = 4$, $\mu r = 0{\cdot}02$. Diffractometer data collected with monochromatic Mo $K\alpha$ radiation consisted of 968 independent reflections with $I > 2\sigma(I)$. The structure was solved by direct methods and refined by a full-matrix least-squares procedure to a final $R$ value of 0·069. The molecule lies in a special position, the benzene ring is planar and the cycloheptene ring is in the boat conformation.

**Introduction.** Small transparent crystals of the title compound were obtained from methanol. The average size of the crystals was 0·2–0·3 mm. The absorption coefficient was neglected because of the small value of $\mu r$(Mo $K\alpha$) = 0·02. 2168 independent reflections were measured by the $\omega$–$2\theta$ step-scan technique (Mo $K\alpha$ radiation) with a Philips diffractometer. The maximum value of $\sin\theta/\lambda = 0{\cdot}7456$ Å$^{-1}$.

The cell constants were determined with a Philips diffractometer by the least-squares method. 968 reflections have been treated as observed [$I > 2\sigma(I)$] and were used to obtain the $E$ map (*MULTAN*, Main, Woolfson, Lessinger, Germain & Declercq, 1976). All non-hydrogen atoms were found on the $E$ map and the $R$ factor calculated for this model was 0·4. After five isotropic cycles, the $R$ factor became 0·17 and the H atoms were found on a $\Delta F$ map. The largest peak on the final difference electron density map was 0·4 e Å$^{-3}$. Three reflections (002, 209, 211) were removed from the calculations because of a large disagreement between $F_c$ and $F_o$. The $\theta$ angles were close to 10° and therefore the extinction factor could be large. Six anisotropic cycles of refinement were performed (isotropic for H atoms). The starting set of isotropic thermal parameters for the H atoms was taken from the C atoms to which they were bonded. The final three cycles of refinement were performed with Cruickshank's weighting scheme. The *ORFLS* program (Busing, Martin & Levy, 1962) was applied. The $R$ factor was 0·069 and the weighted $R$ 0·040.* The final positional parameters are listed in Table 1 along with the standard deviations. Interatomic distances and angles (uncorrected for thermal motion) are given in Table 2.

Table 1. *Final positional parameters* (×10$^4$), *with standard deviations, and* $B_{iso}$ $\{B_{iso} = \frac{1}{3}[(4\beta_{11}/a^{*2}) + (4\beta_{22}/b^{*2}) + (4\beta_{33}/c^{*2})]\}$

| | $x$ | $y$ | $z$ | $B_{iso}$ (Å$^2$) |
|---|---|---|---|---|
| C(1) | 10264 (3) | 7130 (3) | 2907 (1) | 4·52 |
| C(2) | 9387 (3) | 6030 (3) | 3322 (2) | 4·18 |
| C(3) | 8528 (3) | 4869 (2) | 2909 (1) | 3·47 |
| C(4) | 7538 (3) | 3688 (3) | 3349 (2) | 3·98 |
| C(5) | 7800 (4) | 1856 (3) | 3247 (2) | 5·57 |
| C(6) | 8582 (5) | 1345 (4) | 2500 | 5·17 |
| C(7) | 10106 (7) | 7155 (5) | 4568 (3) | 8·46 |
| O(1) | 9323 (3) | 5936 (2) | 4121 (1) | 6·34 |
| O(2) | 6560 (2) | 4191 (2) | 3778 (1) | 5·56 |
| H(11) | 10849 (26) | 7942 (27) | 3158 (14) | 1·97 |
| H(15) | 6877 (42) | 1287 (36) | 3306 (18) | 4·94 |
| H(25) | 8447 (37) | 1533 (41) | 3705 (18) | 5·39 |
| H(16) | 8672 (46) | 61 (53) | 2500 | 4·38 |
| H(26) | 9592 (43) | 1662 (39) | 2500 | 1·99 |
| H(17) | 9880 (43) | 6919 (43) | 5092 (22) | 7·40 |
| H(27) | 11230 (41) | 6986 (46) | 4375 (22) | 6·73 |
| H(37) | 9803 (47) | 8343 (51) | 4401 (23) | 8·38 |

Table 2. *Distances* (Å) *and angles* (°) *with estimated standard deviations*

| | | | |
|---|---|---|---|
| C(1)–C(1) | 1·380 (5) | C(1)–C(2) | 1·376 (3) |
| C(1)–H(11) | 0·94 (2) | C(2)–C(3) | 1·393 (3) |
| C(2)–O(1) | 1·358 (3) | C(3)–C(3) | 1·388 (4) |
| C(3)–C(4) | 1·498 (3) | C(4)–C(5) | 1·508 (3) |
| C(4)–O(2) | 1·205 (3) | C(5)–C(6) | 1·504 (5) |
| C(5)–H(15) | 0·95 (4) | C(5)–H(25) | 1·00 (3) |
| C(6)–H(16) | 1·04 (4) | C(6)–H(26) | 0·93 (4) |
| C(7)–O(1) | 1·424 (5) | C(7)–H(17) | 0·93 (4) |
| C(7)–H(27) | 1·06 (4) | C(7)–H(37) | 1·04 (4) |
| H(11)–C(1)–C(2) | 122 (1) | H(11)–C(1)–C(1) | 116 (1) |
| C(1)–C(1)–C(2) | 120·8 (1) | C(1)–C(2)–C(3) | 119·2 (2) |
| C(1)–C(2)–O(1) | 116·0 (2) | C(2)–C(3)–C(3) | 120·0 (1) |
| C(3)–C(3)–C(4) | 119·8 (1) | C(2)–C(3)–C(4) | 120·0 (2) |
| C(3)–C(4)–C(5) | 118·5 (2) | C(3)–C(4)–O(2) | 120·7 (2) |
| C(5)–C(4)–O(2) | 120·7 (2) | C(4)–C(5)–C(6) | 115·9 (3) |
| C(4)–C(5)–H(15) | 109 (2) | C(4)–C(5)–H(25) | 105 (2) |
| C(6)–C(5)–H(15) | 111 (2) | C(6)–C(5)–H(25) | 108 (2) |
| H(15)–C(5)–H(25) | 107 (3) | C(5)–C(6)–C(5) | 114·8 (4) |
| C(5)–C(6)–H(16) | 108 (1) | C(5)–C(6)–H(26) | 111·7 (9) |
| H(16)–C(6)–H(26) | 101 (3) | C(2)–O(1)–C(7) | 118·2 (2) |
| O(1)–C(7)–H(17) | 105 (2) | O(1)–C(7)–H(27) | 102 (2) |
| O(1)–C(7)–H(37) | 112 (2) | H(17)–C(7)–H(27) | 118 (3) |
| H(17)–C(7)–H(37) | 113 (3) | H(27)–C(7)–H(37) | 106 (3) |
| C(3)–C(3)–C(4)–C(5) | 62·5 (3) | C(4)–C(5)–C(6)–C(5) | −58·3 (5) |
| C(3)–C(4)–C(5)–C(6) | −23·3 (4) | | |

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35399 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

**Discussion.** The molecule lies in a special position [C(6) on the mirror plane]. The benzene ring is planar. Atoms H(11), atoms O(1) and atoms C(4) are coplanar with the benzene ring, and the methoxy carbon atoms C(7) lie below the benzene plane (0·113 Å). The cycloheptene ring is in the boat conformation in the crystal. Atoms O(2) lie below the benzene plane (0·977 Å) and the angle formed between the C(4)–O(2) bond and the benzene ring is 54·16° (Fig. 1).

There are no intermolecular interactions other than van der Waals.

The torsion angles are given in Table 2.

A full discussion of the conformation of the cycloheptene ring system and a comparison with other such systems will be presented when a study on a group of disubstituted benzocycloheptenediones has been completed.

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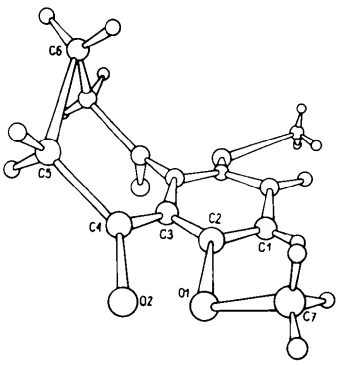


Fig. 1. The observed conformation of the title compound.

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# 1,4-Dihydroxybenzocycloheptene-5,9(6*H*,8*H*)-dione

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**Abstract.** $C_{11}H_{10}O_4$, monoclinic, $C2/c$, $a = 10{\cdot}637$ (2), $b = 13{\cdot}200$ (4), $c = 6{\cdot}731$ (2) Å, $\beta = 102{\cdot}55$ (2)°, $Z = 4$, $V = 922{\cdot}49$ Å$^3$, $\mu r = 0{\cdot}1$. Diffractometer data collected with monochromatic Cu $K\alpha$ radiation consisted of 867 independent reflections with $I > 2\sigma(I)$. The structure was solved by direct methods and refined by a full-matrix least-squares procedure to a final $R$ value of 0·049. The molecule lies in a special position, the benzene ring is planar and the cycloheptene ring is twisted.

**Introduction.** Crystals of the title compound were obtained from methanol solution. The average size of the crystals was 0·2–0·3 mm. The absorption coefficient has been neglected because of the small value of $\mu r$(Cu $K\alpha$) = 0·1. 931 independent reflections were measured by the $\omega$–$2\theta$ step-scan technique with a CAD-4 diffractometer and a maximum value of sin $\theta/\lambda$ of 0·6095 Å$^{-1}$.

The cell constants were determined with a CAD-4 diffractometer by the least-squares method. 867 reflections were treated as observed [$I > 2\sigma(I)$] and used to obtain the $E$ map (*MULTAN*, Main, Woolfson, Lessinger, Germain & Declercq, 1976). The parameters of 1,4-dimethoxybenzocycloheptene-5,9(6*H*,8*H*)-dione (Olszak, Stępień, Wajsman & Grabowski, 1980) for the non-hydrogen atoms were used to calculate the Debye curve (program *NORMAL*). All non-hydrogen atoms were found on the $E$ map and the $R$ factor calculated for this model was 0·43. After four isotropic cycles $R$ reduced to 0·14. The value of $R$ after the next three anisotropic cycles was 0·099. The $\Delta F$ map calculated at this stage revealed all the H atoms. These were given isotropic thermal parameters taken from the

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