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.

The authors are grateful to Dr Z. Madeja-Kotkowska (Hahn & Madeja-Kotkowska, 1963) for supplying the crystals, to Dr Keulen for the data collection, and to the Polish Academy of Sciences for financial support.

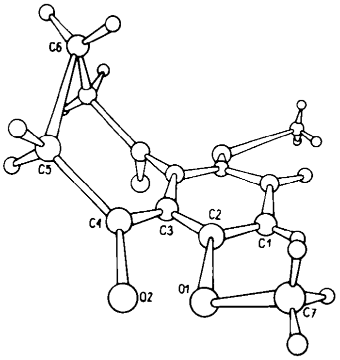

Fig. 1. The observed conformation of the title compound.

## References

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

Hahn, W. E. & Madeja-Kotkowska, Z. (1963). *Rocz. Chem.* **37**, 1447–1456.

*International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1976). *MULTAN 76. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

---

*Acta Cryst.* (1980). B**36**, 2850–2851

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

By Tomasz A. Olszak, A. Stępień, E. Wajsman and M. J. Grabowski

*Department of Crystallography, Institute of Chemistry, University of Łódź,* 91–416 *Łódź, Nowotki* 18, *Poland*

(*Received* 9 *May* 1979; *accepted* 5 *June* 1980)

**Abstract.** $C_{11}H_{10}O_4$, monoclinic, $C2/c$, $a$ = 10·637 (2), $b$ = 13·200 (4), $c$ = 6·731 (2) Å, $\beta$ = 102·55 (2)°, $Z$ = 4, $V$ = 922·49 Å$^3$, $\mu r$ = 0·1. 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

0567-7408/80/112850-02$01.00

atoms to which they were bonded. Anisotropic thermal parameters were introduced for non-hydrogen atoms and six cycles of refinement were performed. The *ORFLS* program (Busing, Martin & Levy, 1962) was applied. The final *R* factor was 0·049.* The largest peak on the final difference electron density map was 0·4 e Å$^{-3}$. 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. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

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

| | *x* | *y* | *z* | $B_{iso}/B$ (Å$^2$) |
|---|---|---|---|---|
| C(1) | 5653 (2) | 10373 (1) | 7614 (3) | 3·73 (10) |
| C(2) | 6335 (2) | 9450 (1) | 7711 (3) | 3·23 (8) |
| C(3) | 5683 (2) | 8529 (1) | 7555 (3) | 2·83 (8) |
| C(4) | 6386 (2) | 7614 (1) | 7169 (3) | 3·10 (8) |
| C(5) | 5638 (2) | 6732 (1) | 6095 (3) | 3·59 (9) |
| C(6) | 5000 | 6090 (2) | 7500 | 4·06 (13) |
| O(1) | 7618 (1) | 9517 (1) | 7861 (3) | 4·41 (8) |
| O(2) | 7576 (1) | 7595 (1) | 7567 (3) | 4·18 (7) |
| H\|C(1)\| | 6122 (25) | 11043 (18) | 7711 (44) | 2·56 (53) |
| H\|O(1)\| | 7935 (32) | 8880 (27) | 8037 (58) | 4·55 (76) |
| H\|1C(5)\| | 4919 (23) | 6989 (15) | 4998 (38) | 1·06 (36) |
| H\|2C(5)\| | 6282 (24) | 6309 (17) | 5535 (41) | 2·26 (46) |
| H\|C(6)\| | 5693 (22) | 5624 (17) | 8314 (42) | 2·19 (45) |

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

| | | | |
|---|---|---|---|
| C(1)–C(1*) | 1·365 (4) | C(1)–C(2) | 1·411 (3) |
| C(1)–H\|C(1)\| | 1·01 (2) | C(2)–C(3) | 1·393 (3) |
| C(2)–O(1) | 1·349 (3) | C(3)–C(3*) | 1·439 (4) |
| C(3)–C(4) | 1·474 (3) | C(4)–C(5) | 1·505 (3) |
| C(4)–O(2) | 1·236 (2) | C(5)–C(6) | 1·534 (3) |
| C(5)–H\|1C(5)\| | 1·00 (2) | C(5)–H\|2C(5)\| | 1·02 (3) |
| C(6)–H\|C(6)\| | 1·02 (2) | O(1)–H\|O(1)\| | 0·90 (4) |
| O(2)–H\|O(1)\| | 1·75 (4) | | |
| C(1*)–C(1)–C(2) | 120·4 (1) | C(4)–C(5)–H\|1C(5)\| | 109 (1) |
| C(1*)–C(1)–H\|C(1)\| | 119 (1) | C(4)–C(5)–H\|2C(5)\| | 106 (1) |
| C(2)–C(1)–H\|C(1)\| | 121 (1) | H\|1C(5)\|–C(5)–H\|2C(5)\| | 112 (2) |
| C(1)–C(2)–C(3) | 120·5 (2) | C(6)–C(5)–H\|1C(5)\| | 106 (2) |
| C(1)–C(2)–O(1) | 116·7 (2) | C(6)–C(5)–H\|2C(5)\| | 110 (1) |
| C(3)–C(2)–O(1) | 122·8 (2) | C(5)–C(6)–C(5*) | 112·9 (2) |
| C(3*)–C(3)–C(2) | 119·0 (1) | C(5)–C(6)–H\|C(6)\| | 107 (2) |
| C(3*)–C(3)–C(4) | 122·5 (1) | C(5)–C(6)–H\|C(6)*\| | 111 (2) |
| C(2)–C(3)–C(4) | 117·7 (2) | H\|C(6)\|–C(6)–H\|C(6)*\| | 106 (3) |
| C(3)–C(4)–C(5) | 118·9 (1) | C(2)–O(1)–H\|O(1)\| | 107 (2) |
| C(3)–C(4)–O(2) | 120·9 (2) | C(4)–O(2)–H\|O(1)\| | 101 (1) |
| C(5)–C(4)–O(2) | 119·9 (2) | O(1)–H\|O(1)\|–O(2) | 145 (3) |
| C(4)–C(5)–C(6) | 113·0 (2) | | |
| C(1*)–C(1)–C(2)–C(3) | 0·6 (4) | C(3)–C(4)–C(5)–C(6) | 79·0 (2) |
| C(1)–C(2)–C(3)–C(3*) | 4·5 (3) | C(4)–C(5)–C(6)–C(5*) | 43·3 (1) |
| C(2)–C(3)–C(3*)–C(2*) | 6·4 (4) | C(5)–C(6)–C(5*)–C(4*) | 43·3 (1) |
| C(3)–C(3*)–C(2*)–C(1*) | 4·5 (3) | C(6)–C(5*)–C(4*)–C(3*) | 79·0 (2) |
| C(3*)–C(2*)–C(1*)–C(1) | 0·6 (4) | C(5*)–C(4*)–C(3*)–C(3) | 16·7 (3) |
| C(2*)–C(1*)–C(1)–C(2) | 1·4 (5) | C(4*)–C(3*)–C(3)–C(4) | 27·4 (4) |
| C(1*)–C(1)–C(2)–O(1) | 177·6 (3) | C(3*)–C(3)–C(4)–C(5) | 16·7 (3) |
| C(3*)–C(3)–C(4)–O(2) | −169·1 (2) | O(1)–C(2)–C(3)–C(4) | 11·3 (3) |
| C(6)–C(5)–C(4)–O(2) | 106·7 (2) | C(2)–C(3)–C(4)–O(2) | 21·2 (3) |

* Atoms related by the twofold axis: $x, y, \frac{1}{2} - z$.

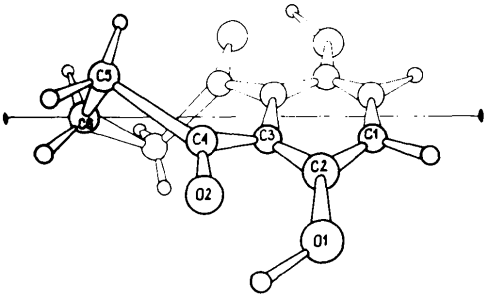

Fig. 1. Observed conformation of the title compound.

**Discussion.** The molecule of the title compound lies in a special position – C(6) is on the twofold axis.

The benzene ring is almost planar. The best plane taken through the benzene C atoms is given by $\pi \equiv -0{\cdot}982x + 6{\cdot}678z = 4{\cdot}517$ ($x$ and $z$ are the positional parameters given in Table 1). The average distance of the benzene C atoms from the best plane is ~0·03 Å. The torsion angles in the molecule are given in Table 2. There are strong O(1)–H|O(1)|···O(2) hydrogen bonds in the molecule. C(2), C(3), C(4), O(2), O(1) and H|O(1)| form rings which are almost planar. The equation of the best plane taken through C(2), C(3), C(4), O(1), O(2) is $\pi_k \equiv -0{\cdot}786x - 1{\cdot}470y + 6{\cdot}619z = 3{\cdot}229$ ($x$, $y$ and $z$ are the positional parameters given in Table 1). The hydrogen atom H|O(1)| has not been included in the calculation because of the lower precision of its positional parameters. The average distance of the atoms from the best plane $\pi_k$ is ~0·1 Å. H|O(1)| lies 1·6 Å out of the $\pi_k$ plane. The value of the dihedral angle formed by the planes $\pi$ and $\pi_k$ is 152·8°. The cycloheptene ring is in the twisted conformation (Fig. 1).

A full discussion of the conformation of the cycloheptene ring system and a comparison with other such systems will be presented later.

The authors are grateful to Dr Z. Madeja-Kotkowska (Hahn & Madeja-Kotkowska, 1963) for supplying the crystals, and the Polish Academy of Sciences for financial support.

## References

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

Hahn, W. E. & Madeja-Kotkowska, Z. (1963). *Rocz. Chem.* **37**, 1447–1456.

*International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1976). *MULTAN 76. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Olszak, T. A., Stępień, A., Wajsman, E. & Grabowski, M. J. (1980). *Acta Cryst.* **B36**, 2849–2850.

---

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35400 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.