Fig. 2. View of the molecules in the crystallographic unit cell.

and, consequently, the two oxygens, each occupying an axial position, can influence the ring conformation.

The O(1)—S—O(2) plane is almost perpendicular to the C(9)—S—O(3) plane [87·0 (3)°] and some angles around the sulphur show a significant deviation from the tetrahedral value [O(1)—S—O(2) = 119·2 (3); O(1)—S—O(3) = 102·9 (7); O(3)—S—C(9) = 104·4 (4)°]. The same effect has been observed in other sulphones (Andreetti, Bocelli & Sgarabotto, 1973; Yasuoka, Kasai, Tanaka, Nagai & Tokura, 1972; Bordner, Levine, Mazur & Morrow, 1973). The sulphur—carbon and sulphur—oxygen bond lengths are as expected.

The packing scheme is illustrated in Fig. 2. There are no unusually short intermolecular contacts.

References

ANDREETTI, G. D., BOCELLI, G. & SGARABOTTO, P. (1973) J. Chem. Soc. Perkin Trans. 2, pp. 1189–1196.

BELLETTI, D., UGOZZOLI, F., CANTONI, A. & PASQUINELLI, G. (1979). Gestione on Line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo 220. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma. Internal Report I-79.

BOCELLI, G. & GRENIER-LOUSTALOT, M. F. (1981). Acta Cryst. B37, 1302–1304.

BOCELLI, G. & GRENIER-LOUSTALOT, M. F. (1982). Cryst. Struct. Commun. 11, 85–88.

BORDNER, J., LEVINE, S. G., MAZUR, Y. & MORROW, L. R. (1973). Cryst. Struct. Commun. 2, 59–62.

BRAUN, P. B., HORNSTRA, J., KNOBLER, C., RUTTEN, E. W. M. & ROMERS, C. (1973). Acta Cryst. B29, 463–469.

CHADWICK, D. J. & DUNITZ, J. D. (1978). Acta Cryst. B34, 965–968.

CHADWICK, D. J., DUNITZ, J. D. & SCHWEIZER, W. B. (1977). Acta Cryst. B33, 1643–1645.

CREMER, D. & POPLE, J. A. (1975). J. Am. Chem. Soc. 97, 1354–1358.

DUNITZ, J. D. (1968). Perspectives in Structural Chemistry, Vol. II, edited by J. D. DUNITZ & J. A. IBERS, p. 10. New York: John Wiley.

HAMILTON, W. C. (1959). Acta Cryst. 12, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

KARLE, I. L. (1975). Acta Cryst. B31, 1519–1522.

LEHMANN, M. S. & LARSEN, F. K. (1974). Acta Cryst. A30, 580–589.

NARDELLI, M. (1982). Comput. Chem. Submitted.

SHELDRICK, G. M. (1976). SHELX. Program for crystal structure determination. Univ. of Cambridge, England.

YASUOKA, N., KASAI, N., TANAKA, M., NAGAI, T. & TOKURA, N. (1972). Acta Cryst. B28, 3393–3399.

Structure of 3-Phenyl-1-oxacyclohexane-2,6-dione*

BY GABRIELE BOCELLI

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Istituti di Chimica Generale e di Strutturistica Chimica, Università degli Studi di Parma, Via M. D'Azeglio 85, 43100 Parma, Italy

AND MARIE-FLORENCE GRENIER-LOUSTALOT

Institut Universitaire de Recherche Scientifique, Avenue Philippon, 64000 Pau, France

Abstract. $C_{11}H_{10}O_3$, $M_r = 190·2$, monoclinic, $P2_1/n$, $a = 10·990$ (3), $b = 10·115$ (3), $c = 8·662$ (2) Å, $\beta = 107·1$ (3)°, $V = 920·3$ (2) Å^3, $Z = 4$, $D_x = 1·37$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·79 mm^{-1}. Final $R = 0·085$ for 760 observed reflections. The structure was solved by direct methods. The non-aromatic ring shows a half-boat conformation and the two carbonyl oxygens are both equatorial.

* Stereochemistry of Rings VIII. Cyclohexane Derivatives. 3. Part VII and Part 2: Bocelli, Grenier-Loustalot & Iratçabal (1982).

Introduction. A precise knowledge of the conformation of cyclohexane derivatives having one or more sp^2 hybridized carbon atoms is of great importance from both spectroscopic and crystallographic points of view. Cyclohexane derivatives normally present the chair conformation, nevertheless some alkyl derivatives do show a twisted boat conformation (Robinson & Thiobald, 1967). Moreover, it was found that 1,4-cyclohexanedione shows the boat conformation both in solution and in the solid state (Mossel & Romers, 1964; Allinger, Blatter, Freiberg & Karkowski, 1966), while 1,3-cyclohexanedione derivatives exist in the boat conformation in solution (Kwart, Rock, Sanchez-Obregon & Walls, 1972), in the envelope conformation (Semmingsen, 1974; Singh & Calvo, 1975) or in the boat conformation in the solid (Rafferty, Benjamin, Raaen, Thiessen & Johnson, 1979).

The analyses of conformational changes in 1,3-cyclohexanedione derivatives by both X-ray diffractometry and NMR spectroscopy becomes particularly interesting when the carbon atom between the two carboxylic functions is substituted by a heteroatom. The structure determination of one of these compounds, with an oxygen as substituent in the ring, is reported in this paper.

Crystals of the title compound were grown from petroleum ether. The crystal used for data collection was a white prism of $0.8 \times 0.4 \times 0.2$ mm. Cell parameters and intensities were obtained on a Siemens

AED single-crystal diffractometer employing programs written by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979) using Ni-filtered Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å). The specimen showed a great sensitivity to air and in order to speed up the data collection we used a method which measured a 'flash' of the intensity of any reflection. In this manner the outline of the peak was collected only for the reflections present. Following this procedure 1901 reflections were collected and only 760 of these having $I > 1.0\sigma(I)$ were used in the refinement. The poor quality of the specimen and the measurement technique adopted resulted in the small number of observed reflections. A check reflection, monitored periodically, showed a specimen decomposition of about 10%. No absorption correction was made.

The structure was solved by direct methods with *SHELX* (Sheldrick, 1976) and refined by full-matrix least squares to $R = 0.085$. The H atoms, clearly localized in a difference synthesis, were refined isotropically, while all other atoms were refined anisotropically. The systematic extinctions ($0k0$: $k \neq 2n$ and $h0l$: $h + l \neq 2n$) uniquely define the space group $P2_1/n$ which is a non-standard orientation for the C_{2h}^5 space group (No. 14).

A final difference map showed no peaks >0.3 e Å^{-3}. The function minimized was $\sum w|\Delta F|^2$ with unit weights. The scattering curves for the atoms are those quoted in *SHELX*.

Table 1 lists the final parameters with their e.s.d.'s.*

All the calculations were performed on the General Automation SPC/16 computer using a local set of programs and on the CDC Cyber 76 computer of the Centro di Calcolo Interuniversitario dell'Italia Nord-Orientale, Casalecchio, Bologna, and with financial support of the University of Parma.

Discussion. Bond distances and angles are listed in Table 2; Fig. 1 shows a perspective drawing of the

Table 1. *Atomic coordinates* ($\times 10^4$ *for* O *and* C, $\times 10^3$ *for* H *atoms*), B_{eq} (Å^2) *for heavy atoms and* U_{iso} *for* H (Å$^2 \times 10^3$)

	x	y	z	$B_{eq}*/U_{iso}$
O(1)	2421 (4)	3725 (4)	8526 (6)	6·30 (8)
O(2)	4442 (5)	3247 (5)	9124 (8)	8·17 (8)
O(3)	421 (5)	4299 (5)	7958 (7)	8·13 (8)
C(1)	3389 (7)	2829 (8)	8948 (9)	6·32 (9)
C(2)	3093 (9)	1415 (9)	9175 (18)	6·78 (9)
C(3)	1678 (8)	1110 (9)	8598 (15)	7·68 (11)
C(4)	942 (8)	2132 (7)	9146 (14)	7·20 (10)
C(5)	1167 (7)	3455 (7)	8462 (10)	6·40 (8)
C(6)	−492 (7)	1895 (7)	8822 (10)	5·67 (8)
C(7)	−1163 (7)	923 (7)	7824 (10)	5·97 (8)
C(8)	−2480 (8)	759 (8)	7641 (11)	6·25 (9)
C(9)	−3059 (8)	1553 (8)	8486 (10)	6·14 (9)
C(10)	−2386 (8)	2509 (8)	9489 (11)	8·17 (8)
C(11)	−1092 (7)	2654 (7)	9692 (11)	8·13 (8)
H(1)	356 (7)	116 (8)	1005 (10)	101 (38)
H(2)	334 (9)	97 (9)	838 (11)	143 (43)
H(3)	138 (8)	113 (9)	728 (10)	140 (36)
H(4)	151 (5)	31 (7)	908 (7)	71 (21)
H(5)	146 (7)	198 (8)	998 (10)	119 (28)
H(6)	−87 (6)	39 (7)	702 (8)	99 (25)
H(7)	−293 (6)	14 (6)	689 (8)	71 (22)
H(8)	−410 (8)	129 (8)	827 (9)	129 (28)
H(9)	−284 (7)	313 (8)	1011 (10)	119 (29)
H(10)	−62 (5)	332 (5)	1063 (7)	50 (15)

* Defined according to Hamilton (1959).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36748 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
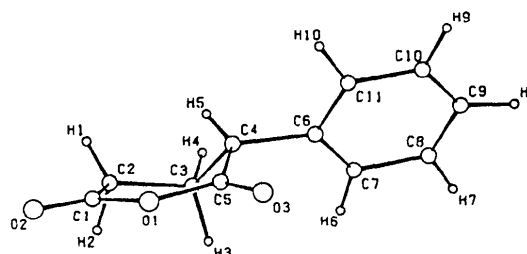

Fig. 1. Projection of the molecule.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

O(1)–C(1)	1·363 (9)	C(2)–H(1)	0·82 (8)
O(1)–C(5)	1·390 (9)	C(2)–H(2)	0·93 (10)
O(2)–C(1)	1·199 (10)	C(3)–H(3)	1·09 (8)
O(3)–C(5)	1·175 (9)	C(3)–H(4)	0·95 (7)
C(1)–C(2)	1·493 (13)	C(4)–H(5)	0·79 (7)
C(2)–C(3)	1·519 (13)	C(7)–H(6)	1·01 (8)
C(3)–C(4)	1·475 (14)	C(8)–H(7)	0·93 (6)
C(4)–C(5)	1·513 (12)	C(9)–H(8)	1·14 (9)
C(4)–C(6)	1·536 (12)	C(10)–H(9)	1·04 (9)
C(6)–C(7)	1·372 (10)	C(11)–H(10)	1·07 (5)
C(6)–C(11)	1·373 (13)		
C(7)–C(8)	1·419 (12)		
C(8)–C(9)	1·364 (14)		
C(9)–C(10)	1·364 (11)		
C(10)–C(11)	1·388 (11)		
C(1)–O(1)–C(5)	124·5 (6)	O(1)–C(5)–O(3)	116·6 (7)
O(1)–C(1)–O(2)	116·6 (7)	O(1)–C(5)–C(4)	115·6 (7)
O(1)–C(1)–C(2)	119·3 (8)	O(3)–C(5)–C(4)	127·7 (8)
O(2)–C(1)–C(2)	124·1 (8)	C(4)–C(6)–C(7)	124·0 (8)
C(1)–C(2)–C(3)	113·2 (8)	C(4)–C(6)–C(11)	116·4 (7)
C(2)–C(3)–C(4)	110·9 (8)	C(7)–C(6)–C(11)	119·4 (8)
C(3)–C(4)–C(5)	109·0 (8)	C(6)–C(7)–C(8)	119·7 (7)
C(3)–C(4)–C(6)	117·5 (7)	C(7)–C(8)–C(9)	119·5 (8)
C(5)–C(4)–C(6)	110·1 (6)	C(8)–C(9)–C(10)	120·6 (9)
		C(9)–C(10)–C(11)	119·9 (8)
		C(6)–C(11)–C(10)	120·7 (8)
C(6)–C(4)–C(3)–C(2)	173·5 (8)	O(2)–C(1)–O(1)–C(5)	171·8 (7)
C(6)–C(4)–C(5)–O(3)	10·5 (13)	C(5)–O(1)–C(1)–C(2)	7·7 (12)
C(6)–C(4)–C(5)–O(1)	–173·5 (7)	O(1)–C(1)–C(2)–C(3)	10·5 (13)
C(1)–O(1)–C(5)–C(4)	9·1 (11)	C(1)–C(2)–C(3)–C(4)	–44·9 (12)
C(1)–O(1)–C(5)–O(3)	–174·3 (7)	O(2)–C(1)–C(2)–C(3)	–170·0 (9)

molecule with the arbitrary numbering scheme used in the analysis. As expected, the molecule as a whole is not planar. The angle formed by the two mean ring planes is 50·9 (3)°. The phenyl ring is planar within the errors and the bond distances and angles are normal.

The cyclohexane ring shows a half-boat conformation. Atom C(4) is out of the plane formed by the other ring atoms by 0·57 (8) Å. The ring-puckering parameters calculated following Cremer & Pople (1975) are $Q = 0·49$ Å, $\theta = 56·0$ and $\varphi = 208·6°$.

Both carbonyl oxygens occupy an equatorial position and the angles formed by the two C=O bonds with the normal to the mean ring plane are 96·2 (3) and 100·7 (4)°.

None of the intermolecular contacts are shorter than 3·41 Å; consequently the molecules are held in the crystal by van der Waals forces.

References

ALLINGER, N. L., BLATTER, H. M., FREIBERG, L. A. & KARKOWSKI, R. M. (1966). *J. Am. Chem. Soc.* **88**, 2999–3011.
BELLETTI, D., UGOZZOLI, F., CANTONI, A. & PASQUINELLI, G. (1979). *Gestione on Line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo 220.* Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma. Internal Report I-79.
BOCELLI, G., GRENIER-LOUSTALOT, M.-F. & IRATÇABAL, P. (1982). *Acta Cryst.* B38, 2068–2070.
CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
KWART, H., ROCK, M. C., SANCHEZ-OBREGON, R. & WALLS, F. (1972). *J. Am. Chem. Soc.* **94**, 1759–1760.
MOSSEL, A. & ROMERS, C. (1964). *Acta Cryst.* **17**, 1217–1223.
RAFFERTY, P., BENJAMIN, B. M., RAAEN, V. F., THIESSEN, W. E. & JOHNSON, C. K. (1979). *Acta Cryst.* B35, 2067–2071.
ROBINSON, C. L. & THIOBALD, D. W. (1967). *Q. Rev. Chem. Soc.* **21**, 314–318.
SEMMINGSEN, D. (1974). *Acta Chem. Scand. Ser. B.* **28**, 169–174.
SHELDRICK, G. M. (1976). *SHELX. Program for crystal structure determination.* Univ. of Cambridge, England.
SINGH, I. & CALVO, C. (1975). *Can. J. Chem.* **53**, 1046–1050.

Acta Cryst. (1982). B38, 2072–2075

Structure of 1,4-Butanediyl Dibenzoate*

BY GABRIELE BOCELLI

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Istituti di Chimica Generale e di Strutturistica Chimica, Università degli Studi di Parma, Via M. D'Azeglio 85, 43100 Parma, Italy

AND MARIE-FLORENCE GRENIER-LOUSTALOT

Institut Universitaire de Recherche Scientifique, Avenue Philippon, 64000 Pau, France

Abstract. $C_{18}H_{18}O_4$, $M_r = 298·3$, triclinic, $P\bar{1}$, $a = 12·591$ (3), $b = 8·555$ (2), $c = 7·854$ (3) Å, $\alpha = 115·3$ (2), $\beta = 90·2$ (2), $\gamma = 83·7$ (1)°, $V = 759·4$ (2) Å^3, $D_x = 1·30$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·71 mm^{-1}. The structure was solved by direct methods and refined to $R = 0·057$ for 2725 collected reflections. A

* Stereochemical Studies of Oligomers. 1.