reflections ($\bar{2}20$ and $\bar{2}02$) showed decreases in intensity of 5 and 3%, respectively, but no corrections were made for this effect. Intensities were corrected for Lp and absorption and the structure was solved by direct methods. The H atoms were placed 1.0 Å from the N atoms and were not refined. The K, Se and O atoms were refined by anisotropic block-diagonal least squares on F_o, $w = 1/[\sigma^2(F_o) + (0.04\,F_o)^2]$; anomalous dispersion was taken into account for the K and Se atoms.

*SHELXS*86 (Sheldrick, 1986) was used for the structure solution and a PC version (Kowalski, 1988) of the *XTL* program system was used for the refinement (including atomic scattering factors for neutral atoms) and the molecular geometry calculations. The figures were drawn using *ORTEP* (Johnson, 1965). The calculations were performed on an Acer 1100/25 computer.

Fig. 1 shows a unit cell. The maximum residual electron density of 2.1 e Å^{-3} is associated with the Se atom and can be explained by the use of a neutral-atom scattering factor for Se.

Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55360 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1009]

References

Dostál, K. (1977). *Folia Fac. Sci. Natl Univ. Purkyn. Brun.* **18**(5), 1–63.

Dostál, K. & Zbořilová, L. (1962). *Z. Anorg. Allg. Chem.* **316**, 335–346.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Kocman, V. & Rucklidge, J. (1974). *Acta Cryst.* B**30**, 6–12.

Kocman, V. & Szymanski, J. T. (1990). *Can. J. Chem.* **68**, 1606–1610.

Kowalski, A. (1988). PC version of *XTL*. Univ. of Wrocław, Poland.

Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Syntex (1971). *XTL Structure Determination System*. Syntex Analytical Instruments, Cupertino, California, USA.

Žák, Z., Růžička, A. & Glowiak, T. (1991). *Can. J. Chem.* **69**, 1080–1083.

Acta Cryst. (1992). C**48**, 2255–2257

Structure of 4,4′-[2,6-Naphthylenebis(oxycarbonyl)]dibutyric Acid

R. Centore, M. R. Ciajolo and A. Tuzi

Dipartimento di Chimica, Università di Napoli 'Federico II' Via Mezzocannone 4, 80134, *Napoli, Italy*

(*Received* 6 *April* 1992; *accepted* 17 *July* 1992)

Abstract

The molecules of the title compound are bonded along extended rows through hydrogen bonds between carboxylic groups. The rows are parallel to the $(3\mathbf{a} + \mathbf{c})$ direction and belong to the t_i line repetition group (*i.e.* molecules along the rows are repeated by inversion centers). The packing of the rows shows a lateral matching of aromatic with aliphatic molecular sections.

Comment

The present study was undertaken as part of research into the synthesis and crystal structure analysis of α,ω-dicarboxylic acids containing a mesogenic core. These compounds may be considered as models for the crystal packing of semi-flexible mesogenic polymers (Centore, Roviello & Sirigu, 1989; Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1990; Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1992). The title compound is a model for semi-flexible polyesters based on 2,6-dihydroxynaphthalene and aliphatic dicarboxylic acids containing an even number of C atoms. The synthesis and phase characterization of these polymers has been reported by Watanabe, Ikeda & Krigbaum (1987). Unlike the analogous polyesters based on 4,4′-dihydroxybiphenyl which are smectogenic (Krigbaum, Watanabe & Ishikawa, 1983), the compounds containing a 2,6-naphthylenedioxy group are not mesogenic; only a monotropic liquid-crystal phase has been reported for one term of the series. On the other hand, random copolyesters in which only a small proportion of the 2,6-naphthylenedioxy groups have been replaced by 4,4′-biphenylenedioxy groups are mesogenic (Watanabe, Ikeda & Krigbaum, 1987). The crystal structure of a dicarboxylic acid (which is the model for semi-flexible polyesters based on 4,4′-dihydroxybiphenyl) has previously been reported (Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1992). A complete structural analysis of the title compound was therefore undertaken in order to detect any significant differences in the crystal packing, in particular the lateral packing of aromatic moieties. The title compound was prepared by esterification of 2,6-dihydroxynaphthalene with glutaric anhydride. 4.000 g of 2,6-dihydroxynaphthalene, 11.410 g of glutaric anhydride and 27.600 g of potassium carbonate were refluxed in 200 ml of dioxane for 2 h. The reaction mixture was then poured into a large excess of water which was acidified with acetic acid. The solid residue was filtered, washed with water and crystallized once from ethanol/water and twice from ethanol giving pure crystals of the title compound (m.p. 465 K, yield 48%).

The conformation of the aliphatic chain is substantially *trans* planar with the largest deviation from planarity being observed for the torsion angle around the C4—C5 bond [C3—C4—C5—O3 = 21.5 (4)°]. The value of the torsion angle around the

0108-2701/92/122255-03$06.00

O4—C6 bond [C5—O4—C6—C7 = −63.9 (3)°] is close to that expected from theoretical calculations (Hummel & Flory, 1980). The naphthalene ring is planar to within 0.003 Å. The C—C bonds in the ring are not of equal length, C6—C7 and C9—C10 being shorter than the others. This is in agreement with previous crystal structure reports for compounds containing naphthalene (White, Carnduff, Guy & Bovill, 1977; Trotter, 1986) and with simple quantum mechanical treatments (Cotton, 1971). Molecules are bonded along extended rows through hydrogen bonds between carboxylic groups. The intermolecular distances between hydrogen bonded O atoms [O1···O2^{i} = 2.661 (1) Å, (i) = 3 − *x*, −*y*, 1 − *z*] are in good agreement with values reported by Leiserowitz (1976). The angle O1—HO1—O2^{i} is 172 (2)°. The cyclic hydrogen-bonded dimer (C1, O1, O2, C1^{i}, O1^{i}, O2^{i}) is planar to within 0.006 Å. Molecular rows are parallel to the (3**a** + **c**) direction and belong to the t_i line repetition group, *i.e.* the molecules along the row are repeated by inversion centers. Laterally adjacent rows are shifted along the (3**a** + **c**) direction, producing a highly intercalated packing in which aromatic groups are laterally packed with aliphatic groups. This is substantially different from the results for other dicarboxylic acids containing a mesogenic core (Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1990; Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1992) which found a lateral correspondence between chemically homologous molecular sections (aromatic–aromatic, aliphatic–aliphatic) in the packing of the rows.

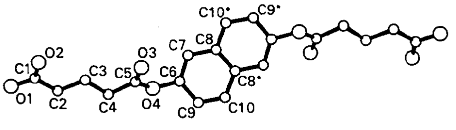

Fig. 1. Molecular drawing of title compound showing the labeling of non-H atoms.

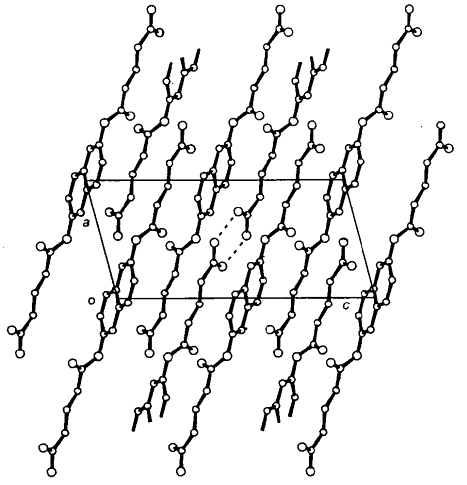

Fig. 2. Crystal packing viewed down [010].

Experimental

Crystal data

$C_{20}H_{20}O_8$
M_r = 388.38
Monoclinic
$P2_1/c$
a = 7.002 (1) Å
b = 9.253 (2) Å
c = 14.869 (3) Å
β = 104.83 (1)°
V = 931 (2) Å^3
Z = 2
D_x = 1.385 Mg m^{-3}

Cu *Kα* radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 15–28°
μ = 0.8678 mm^{-1}
T = 293 K
Prism
0.44 × 0.25 × 0.12 mm
Colorless

Data collection

Enraf-Nonius diffractometer
ω/θ scans
Absorption correction: none
2042 measured reflections
1760 independent reflections
1423 observed reflections [$I > 3\sigma(I)$]

θ_{max} = 69.93°
h = 0 → 8
k = 0 → 11
l = −18 → 18
2 standard reflections
frequency: 60 min
intensity variation: ≤0.5%

Refinement

Refinement on *F*
Final *R* = 0.054
wR = 0.065
S = 2.904
1423 reflections
167 parameters
All H-atom parameters refined

$w = 4F_o^2/[\sigma^2(F_o^2)+(0.02F_o^2)^2]$
$(\Delta/\sigma)_{max}$ = 0.03
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
O1	1.4706 (2)	0.1670 (2)	0.4339 (1)	0.0750 (6)
O2	1.2692 (2)	−0.0131 (2)	0.4408 (1)	0.0784 (6)
O3	0.5545 (3)	0.0035 (2)	0.2411 (2)	0.1058 (6)
O4	0.4951 (2)	0.1762 (2)	0.1335 (1)	0.0641 (5)
C1	1.2985 (3)	0.1026 (2)	0.4097 (2)	0.0551 (6)
C2	1.1436 (3)	0.1869 (3)	0.3421 (2)	0.0587 (6)
C3	0.9495 (3)	0.1064 (3)	0.3118 (2)	0.0673 (9)
C4	0.7886 (3)	0.1967 (3)	0.2497 (2)	0.0612 (6)
C5	0.6036 (3)	0.1124 (3)	0.2107 (2)	0.0592 (6)
C6	0.3054 (3)	0.1246 (3)	0.0899 (2)	0.0525 (6)
C7	0.2736 (3)	−0.0085 (3)	0.0517 (2)	0.0534 (6)
C8	0.0787 (3)	−0.0495 (2)	0.0035 (1)	0.0476 (6)
C9	0.1523 (3)	0.2242 (3)	0.0840 (2)	0.0582 (6)
C10	−0.0362 (3)	0.1872 (3)	0.0377 (2)	0.0570 (6)

Table 2. *Geometric parameters* (Å, °)

O1—C1	1.309 (2)	C4—C5	1.496 (2)
O2—C1	1.205 (2)	C6—C7	1.350 (2)
O3—C5	1.190 (2)	C6—C9	1.399 (2)
O4—C5	1.340 (2)	C7—C8	1.421 (2)
O4—C6	1.404 (1)	C8—C8*	1.417 (2)
C1—C2	1.495 (2)	C8*—C10	1.411 (2)
C2—C3	1.514 (2)	C9—C10	1.366 (2)
C3—C4	1.512 (2)		
C5—O4—C6	120.5 (1)	O4—C5—C4	110.3 (1)
O1—C1—O2	122.3 (1)	O4—C6—C7	122.6 (1)
O1—C1—C2	113.9 (1)	O4—C6—C9	115.1 (1)
O2—C1—C2	123.8 (1)	C7—C6—C9	122.2 (1)
C1—C2—C3	112.7 (1)	C6—C7—C8	119.3 (1)
C2—C3—C4	112.4 (1)	C8*—C8—C7	119.5 (1)
C3—C4—C5	112.6 (1)	C8—C8*—C10	118.6 (1)
O3—C5—O4	122.9 (1)	C6—C9—C10	119.5 (1)
O3—C5—C4	126.8 (1)	C8*—C10—C9	120.9 (1)
C6—O4—C5—O3	6.9 (4)	C2—C3—C4—C5	173.4 (2)
C6—O4—C5—C4	−173.6 (2)	C3—C4—C5—O3	21.5 (4)
C5—O4—C6—C7	−63.9 (3)	C3—C4—C5—O4	−158.0 (2)
C5—O4—C6—C9	119.6 (2)	O4—C6—C7—C8	−175.9 (2)
O1—C1—C2—C3	177.1 (2)	C9—C6—C7—C8	0.3 (3)
O2—C1—C2—C3	−4.0 (3)	O4—C6—C9—C10	176.0 (4)
C1—C2—C3—C4	175.3 (2)	C7—C6—C9—C10	−0.5 (4)

Programs used to solve the structure: *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989). Program used to refine the structure: *SDP* (Enraf-Nonius, 1985). Refinement by full-matrix least-squares methods. All programs were run on a MicroVAX computer.

Financial support from the Ministero dell'Università e della Ricerca Scientifica e Tecnologica is gratefully acknowledged.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55353 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1008]

References

Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389–393.

Centore, R., Ciajolo, M. R., Roviello, A., Sirigu, A. & Tuzi, A. (1990). *Mol. Cryst. Liq. Cryst.* **185**, 99–108.

Centore, R., Ciajolo, M. R., Roviello, A., Sirigu, A. & Tuzi, A. (1992). *Mol. Cryst. Liq. Cryst.* **214**, 105–115.

Centore, R., Roviello, A. & Sirigu, A. (1989). *Liq. Cryst.* **6**, 175–183.

Cotton, F. A. (1971). *Chemical Application of Group Theory*, 2nd ed., pp. 161–165. New York: Wiley-Interscience.

Enraf-Nonius (1985). *Structure Determination Package.* Enraf-Nonius, Delft, The Netherlands.

Hummel, J. P. & Flory, P. J. (1980). *Macromolecules*, **13**, 479–484.

Krigbaum, W. R., Watanabe, J. & Ishikawa, T. (1983). *Macromolecules*, **16**, 1271–1279.

Leiserowitz, L. (1976). *Acta Cryst.* **B32**, 775–802.

Trotter, J. (1986). *Acta Cryst.* **C42**, 351–353.

Watanabe, J., Ikeda, K. & Krigbaum, W. R. (1987). *J. Polym. Sci. Polym. Phys. Ed.* **25**, 19–30.

White, D. N. J., Carnduff, J., Guy, M. H. P. & Bovill, M. J. (1977). *Acta Cryst.* **B33**, 2982–2984.

0108-2701/92/122257-02$06.00

Acta Cryst. (1992). C**48**, 2257–2258

Structure of 4-[(4-Methoxyphenylimino)methyl]phenol

Guan-Yeow Yeap

School of Chemical Sciences, Universiti Sains Malaysia, Minden, 11800 *Penang, Malaysia*

Hoong-Kun Fun

School of Physics, Universiti Sains Malaysia, Minden, 11800 *Penang, Malaysia*

Siang-Guan Teoh* and Soon-Beng Teo

School of Chemical Sciences, Universiti Sains Malaysia, Minden, 11800 *Penang, Malaysia*

K. Chinnakali† and Boon-Chuan Yip

School of Physics, Universiti Sains Malaysia, Minden, 11800 *Penang, Malaysia*

(*Received* 25 *March* 1992; *accepted* 22 *July* 1992)

Abstract

The benzylideneaniline moiety adopts a non-planar conformation with a dihedral angle of 51.0 (1)° between the two phenyl rings. The methoxy substituent is twisted from the aniline ring by 4.1 (5)°.

Comment

Benzylideneaniline normally adopts a non-planar conformation minimizing steric hindrance (Bürgi & Dunitz, 1970; Bernstein, Engel & Hagler, 1981) but disorder can give rise to a nominally planar conformation (Bar & Bernstein, 1983). An almost planar conformation, stabilized by an intramolecular hydrogen bond, is observed in some of its derivatives (Yeap, Fun, Teo & Teoh, 1992). The title compound, which was prepared according to Srivastava & Chauhan (1977), exhibits a non-planar conformation [C(7)—N—C(8)—C(9) −147.4 (4), C(8)—N—C(7)—C(1) −170.5 (4) and N—C(7)—C(1)—C(2) 14.7 (4)°]. The methoxy substitutent is nearly coplanar with the phenyl ring having a torsion angle of 4.1 (5)° about O(2)—C(11). The widening of the angle O(2)—C(11)—C(10) [124.6 (3)°] and the narrowing of O(2)—C(11)—C(12) [115.4 (3)°] are due to steric interactions between C(10) and C(14).

---

† Post Doctoral Research Fellow. Permanent address: Physics Division, Department of Chemical Engineering, AC Tech. Campus, Anna University, Madras 600025, India.