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n-Butyl hydrogen phthalate

Masood Parvez,[a]* Sadiq-ur-Rahman,[b] Saqib Ali[b] and Muhammad Mazhar[b]

[a]Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4, and [b]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan

Correspondence e-mail: parvez@ucalgary.ca

The molecules of the title compound, $C_{12}H_{14}O_4$, form dimeric pairs about inversion centers through hydrogen-bonding interactions between carboxylic acid groups. These hydrogen-bonding interactions can be described in terms of the graph-set notation $R_2^2(8)$.

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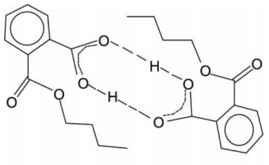
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.119
Data-to-parameter ratio = 17.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Phthalic anhydride shows high reactivity towards nucleophiles, *e.g.* amines, alcohols *etc.* It is used for the synthesis of alkyl and phenyl hydrogenphthalates that can then be employed for syntheses of corresponding phthalamates (Leung & Frechet, 1993).



(I)

The molecules of (I) form dimeric pairs (Fig. 1) about inversion centers through hydrogen-bonding interactions between carboxylic acid groups. These hydrogen-bonding interactions can be described in terms of the graph-set notation $R_2^2(8)$ (Bernstein *et al.*, 1994); details of the hydrogen-bonding geometry are in Table 2. The bond distances C7—O1 and C7—O2 [1.267 (2) and 1.273 (2) Å, respectively] are essentially identical within experimental error and lie between those of a single and a double C—O bond, with H2 weakly bonded to O2, forming a delocalized eight-membered ring system. Atom H2 lies between O2 and O1^{i} (symmetry code as in Table 2), with O—H distances of 1.24 (2) and 1.39 (2) Å,

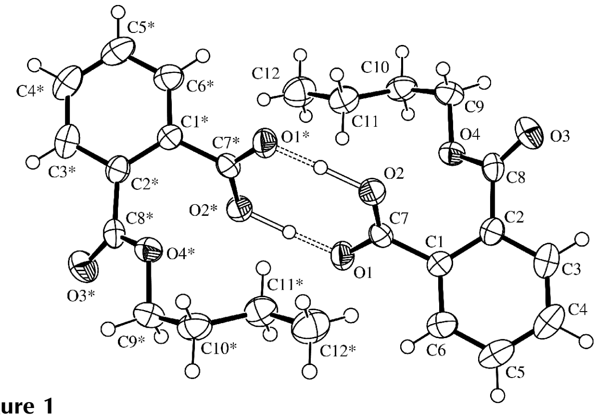


Figure 1
*ORTEP*II (Johnson, 1976) drawing of the hydrogen-bonded dimer of (I), with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (*) $1 - x, -y, 2 - z$.]

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respectively. The remainder of the molecular dimensions are unexceptional. The mean plane of the carboxylic acid group (C1/C7/O1/O2) is inclined at an angle of 50.14 (7)° to the benzene ring, while the alkoxycarbonyl group (C2/C8/O3/O4) is inclined at 35.76 (7)° to the benzene ring. The *n*-butyl chain is fully extended, with a torsion angle C9—C10—C11—C12 of 177.50 (14)°.

Experimental

A mixture of phthalic anhydride (2.5 g, 0.017 mol) and 1-butanol (dry and in excess) (Armarego & Perrin, 1997) was refluxed for 4 h. The reaction mixture was cooled to room temperature, washed with water and extracted with $CHCl_3$. After drying over anhydrous Na_2SO_4, it was filtered and evaporated to dryness, affording a colorless solid in 80% yield (Leung & Frechet, 1993); m.p. 345–347 K. Crystals suitable for X-ray crystallographic study were grown from a concentrated solution of (I) in 1-butanol, to which a few drops of *n*-hexane were added. Slow evaporation of the solvent mixture at room temperature over several days yielded fine crystals that were subsequently washed with *n*-hexane.

Crystal data

$C_{12}H_{14}O_4$
$M_r = 222.23$
Monoclinic, $P2_1/c$
$a = 10.832$ (4) Å
$b = 8.845$ (3) Å
$c = 12.922$ (4) Å
$\beta = 111.22$ (2)°
$V = 1154.1$ (7) Å^3
$Z = 4$

$D_x = 1.279$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4888 reflections
$\theta = 2.9$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 173$ (2) K
Prism, colorless
0.22 × 0.20 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: none
4888 measured reflections
2619 independent reflections
1629 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.042$
$\theta_{\rm max} = 27.5°$
$h = -14 \rightarrow 14$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.119$
$S = 1.04$
2619 reflections
149 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.057P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.063 (16)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.267 (2)	O4—C8	1.334 (2)
O2—C7	1.273 (2)	O4—C9	1.460 (2)
O3—C8	1.204 (2)		
C8—O4—C9	116.14 (12)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1$^{\rm i}$	1.24 (2)	1.39 (2)	2.626 (2)	175.4 (14)

Symmetry code: (i) $1 - x, -y, 2 - z$.

H atoms were located in a difference Fourier synthesis and were included in the refinement at idealized positions (C—H = 0.95–0.99 Å), with isotropic displacement parameters equal to 1.5 (methyl) and 1.2 (others) times $U_{\rm eq}$ of their parent atoms, with the exception of the carboxylic acid H atom, which was refined freely.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

References

Armarego, W. L. F. & Perrin, D. D. (1997). *Purification of Laboratory Chemicals*, 4th ed., pp. 121–130. Oxford: Pergamon.

Bernstein, J., Etter, M. C. & Leiserowitz, L. (1994). *Structure Correlation*, Vol. 2, edited by H.-B. Bürgi &J. D. Dunitz, pp. 431–507. New York: VCH Publishers.

Fan, H.-F. (1991). *SAPI*91. Rigaku Corporation, Tokyo, Japan.

Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Leung, M. K. & Frechet, J. M. J. (1993). *J. Chem. Soc. Perkin Trans.* 2, pp. 2329–2335.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.