organic compounds

Diisopropyl terephthalate

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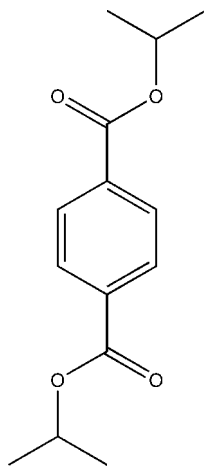Correspondence e-mail: kaitner@chem.pmf.hr

The title compound, $C_{14}H_{18}O_4$, comprises discrete centrosymmetric molecules. The crystal structure is stabilized by intermolecular contacts of the type C—H···O.

Related literature

For related structures, see: dimethyl terephthalate (Brisse & Pérez, 1976), diethyl terephthalate (Bailey, 1949; Hašek *et al.*, 1982) and di-*p*-tolyl terephthalate (Ciajolo *et al.*, 1991).

Experimental

Crystal data

$C_{14}H_{18}O_4$	V = 708.2 (6) Å^3
M_r = 250.28	Z = 2
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 9.208 (3) Å	μ = 0.09 mm^{-1}
b = 9.718 (3) Å	T = 298 (2) K
c = 15.844 (5) Å	$0.75 \times 0.49 \times 0.42$ mm
β = 150.03 (2)°	

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	1521 independent reflections
	1267 reflections with $I > 2\sigma(I)$
Absorption correction: none	R_{int} = 0.055
3803 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056	84 parameters
$wR(F^2)$ = 0.17	H-atom parameters constrained
S = 1.07	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
1521 reflections	$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2202).

References

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supplementary materials

Diisopropyl terephthalate

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Comment

Molecules of (I) are centrosymmetric being disposed about a centre of inversion, as is the case in the crystal structures of related compounds (Brisse & Pérez, 1976; Bailey, 1949; Hašek *et al.*, 1982 & Ciajolo *et al.*, 1991). Similarly, all bond lengths and angles have normal values. The C1/C2/C4/O2 dihedral angle of 161.2 (2)° indicates a deviation from planarity.

The structure comprises essentially discrete molecules, with the closest intermolecular contacts being of the type C—H···O: C3—H···O1 = 3.53 Å (−1 + x, 1/2 − y, −1/2 + z), C1—H···O1 = 3.55 Å (−1 + x, 1/2 − y, −1/2 + z), and C7—H···O1 = 3.560 Å (−2 + x, 1 − y, −3/2 + z). The molecules have an elongated shape and their orientation is almost parallel with the 2**a** + **c** vector, being inclined at an angle of 7.3°. Such a tendency for parallel arrangement is also seen in the structure of the di-*p*-tolyl terephtalate (Ciajolo *et al.*, 1991) derivative, but not in the structures of dimethyl (Brisse & Pérez, 1976) and diethyl (Bailey, 1949; Hašek *et al.*, 1982) esters.

Experimental

Compound (I) was prepared unintentionally during an attempt to isolate a triketone derived from terephtalic acid. In an isooctane (20 ml) solution of sodium 2,2,6,6-tetramethylheptane-3,5-dionate (0.21 g), terephtaloyl dichloride (0.20 g) was added and the mixture heated for 15 min. The NaCl precipitate was filtered off, the filtrate evaporated to approximately 5 ml, 2-propanol (10 ml) added and the mixture left for 24 h, after which crystallized large colourless plates of (I).

Refinement

All H atoms were placed geometrically and included in the refinement in the riding-model approximation, with C—H = 0.93, and with $U_{iso} = 1.2U_{eq}(C)$.
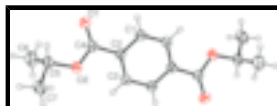
Figures

Fig. 1. View of (I) with the atom labeling scheme. Displacement ellipsoids of are shown at 30% probability.
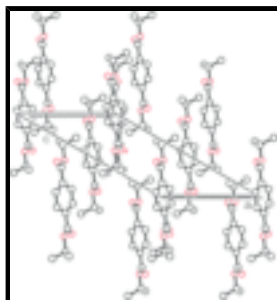

Fig. 2. Crystal packing of (I) viewed along the *y* axis. Hydrogen atoms have been omitted for clarity.

Diisopropyl terephthalate

Crystal data

$C_{14}H_{18}O_4$	$F_{000} = 268$
$M_r = 250.28$	$D_x = 1.174$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1344 reflections
$a = 9.208$ (3) Å	$\theta = 4.6$–$52.0°$
$b = 9.718$ (3) Å	$\mu = 0.09$ mm^{-1}
$c = 15.844$ (5) Å	$T = 298$ (2) K
$\beta = 150.03$ (2)°	Prismatic, colourless
$V = 708.2$ (6) Å^3	$0.75 \times 0.49 \times 0.42$ mm
$Z = 2$	

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	1267 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.055$
Monochromator: graphite	$\theta_{max} = 27.0°$
$T = 295(2)$ K	$\theta_{min} = 4.9°$
ω scans	$h = -11\rightarrow11$
Absorption correction: none	$k = -7\rightarrow12$
3803 measured reflections	$l = -20\rightarrow19$
1521 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0956P)^2 + 0.1119P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.17$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.31$ e Å^{-3}
1521 reflections	Extinction correction: none
84 parameters	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
O2	0.6575 (2)	0.08936 (11)	0.44294 (13)	0.0556 (4)
C2	0.2478 (3)	−0.01186 (14)	0.17469 (18)	0.0427 (4)
C3	0.2212 (3)	0.09717 (15)	0.10444 (19)	0.0475 (4)
H3	0.369	0.162	0.1743	0.057*
C1	0.0255 (3)	−0.10864 (15)	0.06930 (19)	0.0485 (4)
H1	0.0429	−0.1813	0.1159	0.058*
C4	0.5081 (3)	−0.02752 (14)	0.36151 (18)	0.0458 (4)
O1	0.5706 (3)	−0.13377 (13)	0.42786 (15)	0.0704 (4)
C5	0.9128 (3)	0.09072 (19)	0.62716 (19)	0.0574 (5)
H5	0.8796	0.0288	0.6583	0.069*
C7	0.9347 (4)	0.2370 (2)	0.6695 (3)	0.0728 (6)
H7A	0.9438	0.2986	0.6268	0.109*
H7B	1.1071	0.247	0.7899	0.109*
H7C	0.7671	0.2584	0.6188	0.109*
C6	1.1736 (4)	0.0427 (3)	0.7094 (3)	0.0848 (7)
H6A	1.1394	−0.0487	0.6715	0.127*
H6B	1.3413	0.0425	0.8299	0.127*
H6C	1.2065	0.1037	0.6792	0.127*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0551 (7)	0.0525 (7)	0.0425 (6)	−0.0100 (5)	0.0399 (6)	−0.0063 (4)
C2	0.0446 (7)	0.0404 (7)	0.0419 (8)	0.0014 (5)	0.0373 (7)	0.0001 (5)
C3	0.0472 (8)	0.0431 (8)	0.0458 (8)	−0.0068 (6)	0.0393 (7)	−0.0040 (6)
C1	0.0539 (8)	0.0425 (7)	0.0474 (8)	−0.0043 (6)	0.0437 (8)	−0.0004 (6)
C4	0.0456 (7)	0.0447 (8)	0.0427 (8)	−0.0007 (6)	0.0377 (7)	−0.0006 (6)
O1	0.0657 (8)	0.0545 (7)	0.0485 (7)	−0.0020 (5)	0.0434 (7)	0.0057 (5)
C5	0.0510 (9)	0.0657 (11)	0.0421 (9)	−0.0100 (7)	0.0384 (8)	−0.0054 (7)
C7	0.0743 (12)	0.0767 (13)	0.0617 (11)	−0.0183 (10)	0.0581 (11)	−0.0197 (9)
C6	0.0573 (11)	0.0934 (16)	0.0656 (12)	0.0003 (10)	0.0478 (11)	−0.0053 (11)

Geometric parameters (Å, °)

O2—C4	1.3311 (18)	C5—C7	1.509 (3)
O2—C5	1.469 (2)	C5—H5	0.98
C2—C1	1.393 (2)	C7—H7A	0.96
C2—C3	1.396 (2)	C7—H7B	0.96
C2—C4	1.496 (2)	C7—H7C	0.96
C3—H3	0.93	C6—H6A	0.96
C1—H1	0.93	C6—H6B	0.96
C4—O1	1.2058 (19)	C6—H6C	0.96
C5—C6	1.504 (3)		
C4—O2—C5	117.70 (12)	O2—C5—H5	109.5

C1—C2—C3	119.73 (14)	C6—C5—H5	109.5
C1—C2—C4	118.34 (13)	C7—C5—H5	109.5
C3—C2—C4	121.92 (13)	C5—C7—H7A	109.5
C1^{i}—C3—C2	119.93 (13)	C5—C7—H7B	109.5
C1^{i}—C3—H3	120	H7A—C7—H7B	109.5
C2—C3—H3	120	C5—C7—H7C	109.5
C3^{i}—C1—C2	120.34 (14)	H7A—C7—H7C	109.5
C3^{i}—C1—H1	119.8	H7B—C7—H7C	109.5
C2—C1—H1	119.8	C5—C6—H6A	109.5
O1—C4—O2	125.00 (15)	C5—C6—H6B	109.5
O1—C4—C2	123.28 (13)	H6A—C6—H6B	109.5
O2—C4—C2	111.72 (12)	C5—C6—H6C	109.5
O2—C5—C6	108.93 (16)	H6A—C6—H6C	109.5
O2—C5—C7	105.04 (14)	H6B—C6—H6C	109.5
C6—C5—C7	114.16 (17)		

Symmetry codes: (i) −x, −y, −z.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{ii}	0.93	2.90	3.532 (4)	126
C1—H1···O1^{iii}	0.93	2.93	3.546 (3)	125
C7—H7B···O1^{iv}	0.96	2.67	3.560 (4)	153

Symmetry codes: (ii) −x+1, y+1/2, −z+1/2; (iii) x−1, −y−1/2, z−1/2; (iv) −x+2, y+1/2, −z+3/2.

Fig. 1

Fig. 2