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tert-Butylglycolic acid

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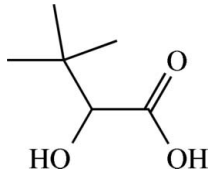
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.142; data-to-parameter ratio = 16.0.

In the title compound, $C_6H_{12}O_3$, which has a sterically demanding *tert*-butyl group attached to a hydroxyacetic acid residue, centrosymmetric hydrogen-bonded dimers are formed; the hydroxy OH group functions as the donor and the double-bonded O atom of the carboxyl group functions as the acceptor. The dimer engages in interdimer bonding through four shorter hydrogen bonds involving two donors (the carboxyl OH) and two acceptors (the hydroxyl O atom). A three-dimensional system of hydrogen bonds is established that has channels for the hydrophobic butyl groups along (0, 0, z) and ($\frac{1}{2}$, $\frac{1}{2}$, z). There are two independent molecules in the asymmetric unit.

Related literature

For synthesis of the title compound, see Reetz & Heimbach (1983). For the crystal structures of 1-hydroxy-1-carboxylic acids with hydrophobic residues of similar size, see Betz & Klüfers (2007*a*,*b*,*c*).



Experimental

Crystal data

$C_6H_{12}O_3$
M_r = 132.16
Monoclinic, $C2/c$
a = 21.236 (2) Å
b = 13.4486 (13) Å
c = 11.4351 (13) Å
β = 111.537 (10)°
V = 3037.8 (6) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 200 (2) K
0.44 × 0.36 × 0.16 mm

Data collection

Oxford Diffraction XCalibur diffractometer
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
T_{min} = 0.972, T_{max} = 0.988
7814 measured reflections
3003 independent reflections
2266 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.10
3003 reflections
188 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O12—H12···O111^{i}	0.81 (2)	2.08 (2)	2.8456 (17)	159 (2)
O22—H22···O211^{ii}	0.80 (2)	2.15 (2)	2.9308 (18)	163.4 (19)
O112—H112···O22	0.82 (2)	1.81 (3)	2.6317 (18)	177 (2)
O212—H212···O12^{iii}	0.87 (3)	1.82 (3)	2.6883 (18)	170 (2)

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $-x, -y+1, -z$; (iii) $x, -y+1, z-\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2330).

References

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  doi:10.1107/S1600536807046156

supplementary materials

Acta Cryst. (2007). E**63**, o4144 [**doi:10.1107/S1600536807046156**]

tert-Butylglycolic acid

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Comment

tert-Butylglycolic acid was prepared as a chelating molecule bearing the sterically demanding *tert*-butyl group as a substituent. In order to estimate the influence of chelation on structural parameters of the chelating molecule, the structure of the parent compound was investigated.

The molecular structure of (I) comprises a *tert*-butyl group attached to hydroxy acetic acid (glycolic acid). The two molecules present in the asymmetric unit differ only slightly from each other by the arrangement of the *tert*-butyl group relative to the carboxyl group.

Hydrogen bonds between hydroxyl donors and carboxyl-O acceptors connect pairs of molecules to centrosymmetric dimers. Each dimer is incorporated in three-dimensional network by means of two carboxyl-donor and two hydroxyl-acceptor sites. Figure 2 shows hydrophobic channels along 0,0,*z* and 1/2,1/2,*z* running through the hydrogen-bonded network of the hydrophilic functions.

Experimental

The title compound was prepared according to a published procedure (Reetz & Heimbach, 1983) upon addition of *tert*-butyl chloride to tris(trimethylsilyloxyethene). Crystals suitable for X-ray analysis were directly obtained from the crystallized reaction product.

Refinement

All H atoms were located in a difference map. Methyl H atoms were refined as riding with one common isotropic temperature parameter. Individual isotropic temperature parameters were refined for the other H atoms. The positional parameters of O-bonded H atoms were refined freely.

Figures

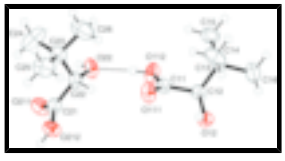

Fig. 1. The structure of the molecules of (I) in the asymmetric unit, with atom labels and anisotropic displacement ellipsoids (drawn at the 50% probability level) for non-H atoms.

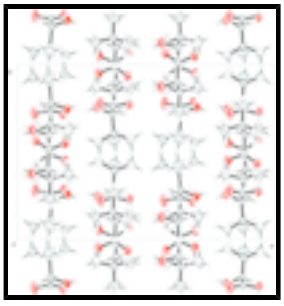

Fig. 2. The molecular packing of (I) viewed along [0 0 1].

tert-Butylglycolic acid

Crystal data

$C_6H_{12}O_3$	$Z = 16$
$M_r = 132.16$	$F_{000} = 1152$
Monoclinic, $C2/c$	$D_x = 1.156$ Mg m^{-3}
Hall symbol: -C 2yc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 21.236$ (2) Å	$\theta = 3.9$–26.0º
$b = 13.4486$ (13) Å	$\mu = 0.09$ mm^{-1}
$c = 11.4351$ (13) Å	$T = 200$ (2) K
$\beta = 111.537$ (10)º	Block, colourless
$V = 3037.8$ (6) Å^3	0.44 × 0.36 × 0.16 mm

Data collection

Oxford Diffraction XCalibur diffractometer	$R_{int} = 0.025$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0$º
Monochromator: graphite	$\theta_{min} = 4.5$º
$T = 200$(2) K	$h = -19 \rightarrow 26$
ω scans	$k = -16 \rightarrow 12$
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	$l = -14 \rightarrow 14$
$T_{min} = 0.972$, $T_{max} = 0.988$	Standard reflections: ?;
7814 measured reflections	every ? reflections
3003 independent reflections	intensity decay: ?
2266 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.142$	$w = 1/[\sigma^2(F_o^2) + (0.0759P)^2 + 0.3594P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
3003 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
188 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

Refinement. Treatment of H atoms: *GEOM* for methyl Hs, 1 common isotropic U. *GEOM* for methylidine Hs, individual isotropic Us All H-atom parameters refined for O-bonded Hs

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O12	0.19291 (6)	0.25485 (9)	0.62987 (11)	0.0395 (3)
H12	0.2237 (12)	0.2390 (17)	0.608 (2)	0.073 (7)*
O22	0.05822 (6)	0.37302 (10)	0.12597 (12)	0.0476 (3)
H22	0.0265 (11)	0.4000 (15)	0.074 (2)	0.051 (6)*
O111	0.17753 (5)	0.27864 (10)	0.38322 (12)	0.0534 (4)
O112	0.06607 (5)	0.27901 (10)	0.33272 (12)	0.0508 (4)
H112	0.0644 (11)	0.3065 (18)	0.267 (2)	0.072 (7)*
O211	0.07378 (6)	0.56407 (9)	0.05469 (13)	0.0541 (4)
O212	0.18526 (5)	0.54759 (10)	0.15797 (12)	0.0487 (4)
H212	0.1857 (13)	0.612 (2)	0.156 (2)	0.090 (8)*
C11	0.12818 (7)	0.26207 (11)	0.40870 (15)	0.0358 (4)
C12	0.13211 (7)	0.21920 (11)	0.53441 (14)	0.0347 (4)
H121	0.0932	0.2472	0.5531	0.035 (4)*
C13	0.12590 (8)	0.10491 (12)	0.53469 (16)	0.0400 (4)
C14	0.18576 (10)	0.05486 (14)	0.5147 (2)	0.0636 (6)
H141	0.2279	0.0739	0.5828	0.0897 (19)*
H142	0.1875	0.0761	0.4340	0.0897 (19)*
H143	0.1803	−0.0175	0.5146	0.0897 (19)*
C15	0.06016 (9)	0.07141 (15)	0.4313 (2)	0.0570 (5)
H151	0.0548	−0.0005	0.4374	0.0897 (19)*
H152	0.0618	0.0875	0.3489	0.0897 (19)*
H153	0.0218	0.1058	0.4413	0.0897 (19)*
C16	0.12455 (12)	0.07342 (17)	0.6625 (2)	0.0714 (7)
H161	0.0866	0.1060	0.6762	0.0897 (19)*
H162	0.1671	0.0930	0.7292	0.0897 (19)*
H163	0.1192	0.0011	0.6640	0.0897 (19)*
C21	0.12311 (8)	0.51224 (12)	0.10414 (15)	0.0387 (4)
C22	0.11975 (7)	0.40017 (12)	0.11120 (15)	0.0380 (4)
H221	0.1578	0.3786	0.1889	0.031 (4)*

C23	0.12843 (9)	0.34628 (13)	−0.00079 (17)	0.0464 (4)
C24	0.07322 (13)	0.3743 (2)	−0.1234 (2)	0.0892 (8)
H241	0.0290	0.3595	−0.1188	0.0897 (19)*
H242	0.0787	0.3360	−0.1920	0.0897 (19)*
H243	0.0761	0.4455	−0.1390	0.0897 (19)*
C25	0.19713 (11)	0.37017 (18)	−0.0065 (2)	0.0713 (6)
H251	0.1996	0.4414	−0.0226	0.0897 (19)*
H252	0.2033	0.3319	−0.0744	0.0897 (19)*
H253	0.2328	0.3527	0.0737	0.0897 (19)*
C26	0.12598 (15)	0.23381 (17)	0.0209 (3)	0.0888 (8)
H261	0.1634	0.2152	0.0983	0.0897 (19)*
H262	0.1302	0.1978	−0.0504	0.0897 (19)*
H263	0.0828	0.2167	0.0286	0.0897 (19)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O12	0.0292 (6)	0.0446 (7)	0.0392 (7)	0.0007 (5)	0.0061 (5)	−0.0006 (5)
O22	0.0284 (6)	0.0597 (8)	0.0526 (8)	0.0021 (5)	0.0122 (6)	0.0249 (6)
O111	0.0270 (6)	0.0774 (9)	0.0543 (8)	0.0029 (5)	0.0130 (6)	0.0266 (6)
O112	0.0255 (6)	0.0817 (10)	0.0420 (7)	0.0054 (5)	0.0087 (6)	0.0214 (6)
O211	0.0310 (6)	0.0446 (7)	0.0766 (9)	0.0029 (5)	0.0080 (6)	0.0077 (6)
O212	0.0310 (6)	0.0433 (8)	0.0611 (8)	−0.0029 (5)	0.0044 (6)	0.0050 (6)
C11	0.0262 (8)	0.0383 (9)	0.0396 (9)	0.0010 (6)	0.0082 (7)	0.0058 (6)
C12	0.0254 (7)	0.0407 (9)	0.0357 (8)	0.0006 (6)	0.0085 (7)	0.0019 (6)
C13	0.0348 (8)	0.0396 (9)	0.0437 (9)	−0.0042 (7)	0.0123 (8)	0.0049 (7)
C14	0.0508 (11)	0.0393 (10)	0.1000 (17)	0.0027 (8)	0.0269 (12)	−0.0026 (10)
C15	0.0463 (10)	0.0557 (12)	0.0621 (12)	−0.0151 (8)	0.0116 (10)	−0.0018 (9)
C16	0.0837 (16)	0.0688 (14)	0.0574 (13)	−0.0183 (11)	0.0210 (12)	0.0173 (10)
C21	0.0301 (8)	0.0464 (10)	0.0381 (9)	−0.0002 (7)	0.0107 (7)	0.0042 (7)
C22	0.0261 (8)	0.0447 (9)	0.0401 (9)	−0.0005 (6)	0.0085 (7)	0.0124 (7)
C23	0.0378 (9)	0.0447 (10)	0.0552 (11)	0.0003 (7)	0.0153 (8)	0.0006 (8)
C24	0.0748 (16)	0.135 (2)	0.0494 (13)	0.0289 (15)	0.0132 (12)	−0.0139 (13)
C25	0.0602 (13)	0.0745 (15)	0.0925 (17)	−0.0056 (10)	0.0437 (13)	−0.0153 (12)
C26	0.106 (2)	0.0496 (13)	0.126 (2)	−0.0121 (12)	0.0607 (19)	−0.0130 (13)

Geometric parameters (Å, °)

O12—C12	1.4331 (18)	C15—H152	0.9800
O12—H12	0.81 (2)	C15—H153	0.9800
O22—C22	1.4253 (18)	C16—H161	0.9800
O22—H22	0.80 (2)	C16—H162	0.9800
O111—C11	1.2071 (18)	C16—H163	0.9800
O112—C11	1.3058 (18)	C21—C22	1.512 (2)
O112—H112	0.82 (2)	C22—C23	1.540 (2)
O211—C21	1.2116 (19)	C22—H221	1.0000
O212—C21	1.3230 (19)	C23—C24	1.508 (3)
O212—H212	0.87 (3)	C23—C25	1.519 (3)
C11—C12	1.522 (2)	C23—C26	1.537 (3)

C12—C13	1.543 (2)	C24—H241	0.9800
C12—H121	1.0000	C24—H242	0.9800
C13—C14	1.528 (2)	C24—H243	0.9800
C13—C15	1.529 (2)	C25—H251	0.9800
C13—C16	1.532 (3)	C25—H252	0.9800
C14—H141	0.9800	C25—H253	0.9800
C14—H142	0.9800	C26—H261	0.9800
C14—H143	0.9800	C26—H262	0.9800
C15—H151	0.9800	C26—H263	0.9800
C12—O12—H12	106.5 (17)	H161—C16—H163	109.5
C22—O22—H22	110.7 (14)	H162—C16—H163	109.5
C11—O112—H112	112.3 (16)	O211—C21—O212	123.66 (16)
C21—O212—H212	111.4 (17)	O211—C21—C22	123.27 (14)
O111—C11—O112	124.16 (14)	O212—C21—C22	113.06 (13)
O111—C11—C12	123.12 (14)	O22—C22—C21	108.95 (12)
O112—C11—C12	112.72 (13)	O22—C22—C23	111.89 (14)
O12—C12—C11	108.32 (12)	C21—C22—C23	113.71 (13)
O12—C12—C13	112.68 (12)	O22—C22—H221	107.3
C11—C12—C13	113.84 (13)	C21—C22—H221	107.3
O12—C12—H121	107.2	C23—C22—H221	107.3
C11—C12—H121	107.2	C24—C23—C25	109.8 (2)
C13—C12—H121	107.2	C24—C23—C26	109.5 (2)
C14—C13—C15	109.16 (16)	C25—C23—C26	107.77 (18)
C14—C13—C16	109.19 (17)	C24—C23—C22	111.46 (15)
C15—C13—C16	108.95 (15)	C25—C23—C22	110.27 (15)
C14—C13—C12	111.24 (13)	C26—C23—C22	107.92 (17)
C15—C13—C12	110.27 (14)	C23—C24—H241	109.5
C16—C13—C12	107.99 (15)	C23—C24—H242	109.5
C13—C14—H141	109.5	H241—C24—H242	109.5
C13—C14—H142	109.5	C23—C24—H243	109.5
H141—C14—H142	109.5	H241—C24—H243	109.5
C13—C14—H143	109.5	H242—C24—H243	109.5
H141—C14—H143	109.5	C23—C25—H251	109.5
H142—C14—H143	109.5	C23—C25—H252	109.5
C13—C15—H151	109.5	H251—C25—H252	109.5
C13—C15—H152	109.5	C23—C25—H253	109.5
H151—C15—H152	109.5	H251—C25—H253	109.5
C13—C15—H153	109.5	H252—C25—H253	109.5
H151—C15—H153	109.5	C23—C26—H261	109.5
H152—C15—H153	109.5	C23—C26—H262	109.5
C13—C16—H161	109.5	H261—C26—H262	109.5
C13—C16—H162	109.5	C23—C26—H263	109.5
H161—C16—H162	109.5	H261—C26—H263	109.5
C13—C16—H163	109.5	H262—C26—H263	109.5
O111—C11—C12—O12	32.7 (2)	O211—C21—C22—O22	−34.9 (2)
O112—C11—C12—O12	−147.03 (13)	O212—C21—C22—O22	144.80 (14)
O111—C11—C12—C13	−93.44 (19)	O211—C21—C22—C23	90.67 (19)
O112—C11—C12—C13	86.80 (16)	O212—C21—C22—C23	−89.66 (17)

O12—C12—C13—C14	−58.85 (19)	O22—C22—C23—C24	62.7 (2)
C11—C12—C13—C14	64.99 (18)	C21—C22—C23—C24	−61.3 (2)
O12—C12—C13—C15	179.89 (14)	O22—C22—C23—C25	−175.10 (15)
C11—C12—C13—C15	−56.26 (18)	C21—C22—C23—C25	60.93 (19)
O12—C12—C13—C16	60.95 (18)	O22—C22—C23—C26	−57.62 (19)
C11—C12—C13—C16	−175.21 (15)	C21—C22—C23—C26	178.40 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O111^{i}	0.81 (2)	2.08 (2)	2.8456 (17)	159 (2)
O22—H22···O211^{ii}	0.80 (2)	2.15 (2)	2.9308 (18)	163.4 (19)
O112—H112···O22	0.82 (2)	1.81 (3)	2.6317 (18)	177 (2)
O212—H212···O12^{iii}	0.87 (3)	1.82 (3)	2.6883 (18)	170 (2)

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

Fig. 1

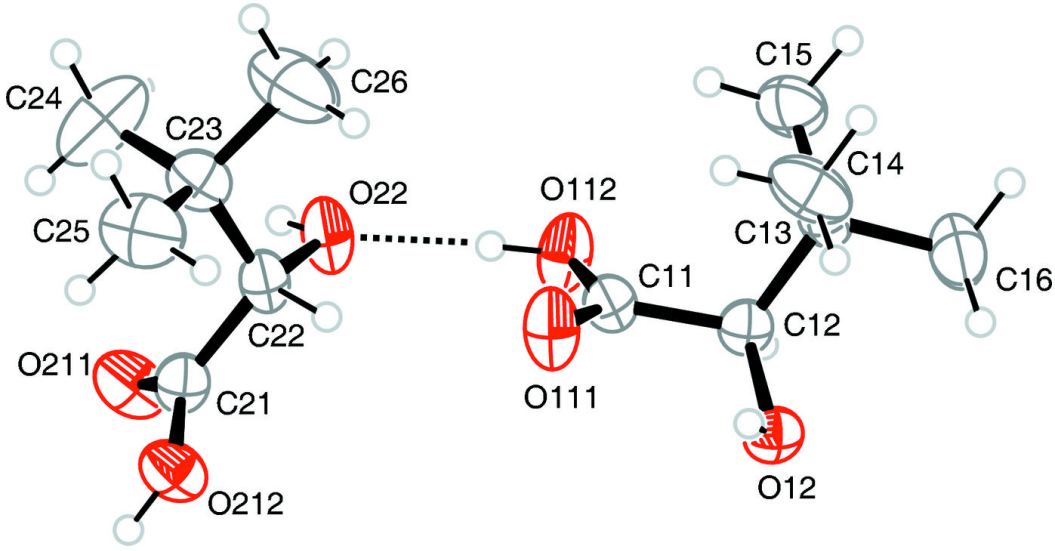
C24
C23
C26
C25
O22
O112
C15
C14
C13
C16
C11
C12
C22
O211
C21
O111
O12
O212

Fig. 2