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5-*tert*-Butylbenzene-1,3-dicarboxylic acid

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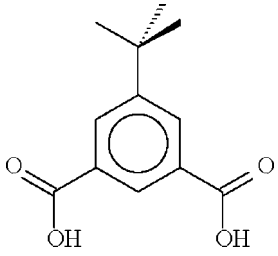
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.059; wR factor = 0.171; data-to-parameter ratio = 13.2.

In the crystal structure of the title compound, $C_{12}H_{14}O_4$, the carboxy groups are linked across centers of inversion by O—H···O hydrogen bonds; the resulting hydrogen-bonded chain adopts a zigzag motif.

Related literature

For the microporous nickel(II) derivative of 5-*tert*-butyl-1,3-benzenedicarboxylic acid, see Ma *et al.* (2007), and for the microporous copper(I,II) derivative, see Pan *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{14}O_4$
M_r = 222.23
Monoclinic, $P2_1/c$
a = 6.2917 (5) Å
b = 10.5847 (9) Å
c = 17.838 (2) Å
β = 97.137 (1)°
V = 1178.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.40 × 0.34 × 0.20 mm

Data collection

Bruker APEX diffractometer
Absorption correction: none
5746 measured reflections
2055 independent reflections
1711 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.171
S = 1.05
2055 reflections
156 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O2^{i}	0.86 (1)	1.75 (1)	2.606 (2)	178 (4)
O3—H3O···O4^{ii}	0.86 (1)	1.73 (1)	2.588 (2)	176 (5)

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+2, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2322).

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supplementary materials

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5-*tert*-Butylbenzene-1,3-dicarboxylic acid

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Comment

The nickel (Ma *et al.*, 2007) and zinc (Pan *et al.*, 2006) derivatives of 5-tertbutyl-1,3-benzenedicarboxylic acid have a microporous framework and can be used for gas separation. Attempt to synthesize a similar copper derivative returned the starting reactants. In the crystal structure, the carboxy $–CO_2H$ groups are linked across different centers-of-inversion by O–H···O hydrogen bonds. Because the groups are at the 1,3-positions, the resulting chain adopts a zigzag motif.

Experimental

The compound was returned unchanged in an attempt to synthesize the copper derivative. 5-*tert*-Butyl-benzene-1,3-dicarboxylic acid (0.5 mmol) was dissolved in DMF (5 ml); the solution was layered over silica gel (5 ml) containing copper nitrate (0.5 mmol). Colorless crystals were found at the interface between the solution and the gel after several days.

Refinement

H atoms were placed in calculated positions (C–H 0.93 – 0.96 Å) and were included in the refinement, with U_{iso}(H) set to 1.2 − 1.5 U_{eq}(C). The H atoms of the carboxylate OH groups were located in a difference Fourier map, and were refined with a distance restraint of O–H 0.85±0.01 Å; their temperature factors were freely refined.

Figures

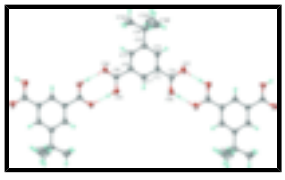

Fig. 1. **Figure 1**. Thermal ellipsoid plot of the hydrogen-bonded chain structure. The hydrogen bonds are shown as dashed lines. Ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radius.

5-*tert*-Butylbenzene-1,3-dicarboxylic acid

Crystal data

$C_{12}H_{14}O_4$	F_{000} = 472
M_r = 222.23	D_x = 1.252 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1901 reflections
a = 6.2917 (5) Å	θ = 2.4–25.5º
b = 10.5847 (9) Å	μ = 0.09 mm^{-1}
c = 17.838 (2) Å	T = 293 (2) K

β = 97.137 (1)°	Block, colorless
V = 1178.7 (2) Å^3	0.40 × 0.34 × 0.20 mm
Z = 4	

Data collection

Bruker APEX diffractometer	1711 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.019
Monochromator: graphite	θ_{max} = 25.0°
T = 293(2) K	θ_{min} = 2.2°
φ and ω scans	h = −7→7
Absorption correction: None	k = −9→12
5746 measured reflections	l = −20→21
2055 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.171	$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 0.4202P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
2055 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
156 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.1901 (3)	0.11648 (18)	0.51399 (10)	0.0684 (6)
H1O	0.089 (5)	0.084 (4)	0.535 (2)	0.152 (17)*
O2	0.1249 (3)	−0.02278 (17)	0.42164 (9)	0.0663 (5)
O3	0.7687 (3)	0.41928 (19)	0.51110 (10)	0.0748 (6)
H3O	0.842 (7)	0.482 (3)	0.530 (3)	0.18 (2)*
O4	0.9974 (3)	0.39797 (17)	0.42778 (9)	0.0648 (5)
C1	0.2288 (3)	0.0675 (2)	0.45294 (12)	0.0473 (5)
C2	0.4090 (3)	0.1196 (2)	0.41733 (11)	0.0447 (5)
C3	0.4595 (3)	0.0712 (2)	0.35009 (12)	0.0470 (5)
H3	0.3767	0.0058	0.3272	0.056*
C4	0.6285 (3)	0.1162 (2)	0.31544 (11)	0.0456 (5)
C5	0.7481 (3)	0.2123 (2)	0.35179 (11)	0.0441 (5)
H5	0.8647	0.2441	0.3305	0.053*

C6	0.6994 (3)	0.26268 (19)	0.41898 (11)	0.0423 (5)
C7	0.5287 (3)	0.2176 (2)	0.45191 (11)	0.0453 (5)
H7	0.4942	0.2525	0.4967	0.054*
C8	0.8324 (3)	0.3665 (2)	0.45451 (11)	0.0466 (5)
C9	0.6793 (4)	0.0612 (2)	0.24053 (13)	0.0573 (7)
C10	0.7336 (7)	−0.0771 (3)	0.2514 (2)	0.1065 (12)
H10A	0.7691	−0.1118	0.2049	0.160*
H10B	0.6125	−0.1212	0.2665	0.160*
H10C	0.8536	−0.0861	0.2899	0.160*
C11	0.4867 (5)	0.0735 (4)	0.18159 (16)	0.0911 (10)
H11A	0.5203	0.0400	0.1345	0.137*
H11B	0.4484	0.1610	0.1753	0.137*
H11C	0.3688	0.0274	0.1975	0.137*
C12	0.8677 (6)	0.1269 (4)	0.2130 (2)	0.1218 (17)
H12A	0.8957	0.0899	0.1661	0.183*
H12B	0.9916	0.1175	0.2498	0.183*
H12C	0.8358	0.2150	0.2056	0.183*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0706 (12)	0.0808 (13)	0.0595 (10)	−0.0314 (10)	0.0312 (9)	−0.0120 (9)
O2	0.0623 (10)	0.0750 (12)	0.0641 (10)	−0.0342 (9)	0.0181 (8)	−0.0107 (9)
O3	0.0834 (13)	0.0877 (14)	0.0602 (11)	−0.0446 (11)	0.0361 (9)	−0.0355 (10)
O4	0.0670 (11)	0.0749 (12)	0.0576 (10)	−0.0362 (9)	0.0280 (8)	−0.0205 (8)
C1	0.0475 (12)	0.0528 (13)	0.0425 (11)	−0.0120 (10)	0.0092 (9)	−0.0007 (10)
C2	0.0432 (11)	0.0475 (12)	0.0444 (11)	−0.0096 (9)	0.0094 (9)	0.0012 (9)
C3	0.0449 (11)	0.0465 (12)	0.0500 (12)	−0.0112 (9)	0.0082 (9)	−0.0087 (9)
C4	0.0446 (11)	0.0498 (13)	0.0435 (11)	−0.0068 (9)	0.0096 (9)	−0.0086 (9)
C5	0.0419 (11)	0.0494 (12)	0.0427 (11)	−0.0090 (9)	0.0117 (8)	−0.0036 (9)
C6	0.0456 (11)	0.0442 (11)	0.0381 (10)	−0.0093 (9)	0.0092 (8)	−0.0015 (9)
C7	0.0484 (11)	0.0491 (13)	0.0402 (10)	−0.0092 (9)	0.0123 (9)	−0.0041 (9)
C8	0.0536 (12)	0.0514 (13)	0.0369 (10)	−0.0163 (10)	0.0140 (9)	−0.0044 (9)
C9	0.0530 (13)	0.0671 (16)	0.0542 (13)	−0.0117 (11)	0.0161 (10)	−0.0250 (11)
C10	0.132 (3)	0.092 (2)	0.098 (2)	0.028 (2)	0.023 (2)	−0.036 (2)
C11	0.087 (2)	0.132 (3)	0.0554 (16)	0.0004 (19)	0.0119 (14)	−0.0316 (18)
C12	0.106 (2)	0.179 (4)	0.093 (2)	−0.068 (3)	0.066 (2)	−0.080 (3)

Geometric parameters (Å, °)

O1—C1	1.257 (3)	C6—C7	1.372 (3)
O1—H1O	0.855 (10)	C6—C8	1.476 (3)
O2—C1	1.250 (3)	C7—H7	0.9300
O3—C8	1.261 (3)	C9—C12	1.508 (4)
O3—H3O	0.859 (10)	C9—C11	1.508 (4)
O4—C8	1.241 (2)	C9—C10	1.510 (4)
C1—C2	1.474 (3)	C10—H10A	0.9600
C2—C3	1.377 (3)	C10—H10B	0.9600
C2—C7	1.382 (3)	C10—H10C	0.9600

C3—C4	1.379 (3)	C11—H11A	0.9600
C3—H3	0.9300	C11—H11B	0.9600
C4—C5	1.379 (3)	C11—H11C	0.9600
C4—C9	1.527 (3)	C12—H12A	0.9600
C5—C6	1.380 (3)	C12—H12B	0.9600
C5—H5	0.9300	C12—H12C	0.9600
C1—O1—H1O	117 (3)	O3—C8—C6	116.99 (18)
C8—O3—H3O	117 (3)	C12—C9—C11	109.2 (3)
O2—C1—O1	123.7 (2)	C12—C9—C10	108.3 (3)
O2—C1—C2	118.8 (2)	C11—C9—C10	108.8 (3)
O1—C1—C2	117.47 (19)	C12—C9—C4	111.58 (19)
C3—C2—C7	119.74 (19)	C11—C9—C4	109.9 (2)
C3—C2—C1	120.62 (19)	C10—C9—C4	109.0 (2)
C7—C2—C1	119.63 (19)	C9—C10—H10A	109.5
C2—C3—C4	122.41 (19)	C9—C10—H10B	109.5
C2—C3—H3	118.8	H10A—C10—H10B	109.5
C4—C3—H3	118.8	C9—C10—H10C	109.5
C5—C4—C3	116.75 (19)	H10A—C10—H10C	109.5
C5—C4—C9	122.10 (19)	H10B—C10—H10C	109.5
C3—C4—C9	121.15 (19)	C9—C11—H11A	109.5
C4—C5—C6	121.75 (18)	C9—C11—H11B	109.5
C4—C5—H5	119.1	H11A—C11—H11B	109.5
C6—C5—H5	119.1	C9—C11—H11C	109.5
C7—C6—C5	120.50 (18)	H11A—C11—H11C	109.5
C7—C6—C8	120.47 (18)	H11B—C11—H11C	109.5
C5—C6—C8	119.02 (17)	C9—C12—H12A	109.5
C6—C7—C2	118.82 (19)	C9—C12—H12B	109.5
C6—C7—H7	120.6	H12A—C12—H12B	109.5
C2—C7—H7	120.6	C9—C12—H12C	109.5
O4—C8—O3	123.57 (19)	H12A—C12—H12C	109.5
O4—C8—C6	119.44 (18)	H12B—C12—H12C	109.5
O2—C1—C2—C3	1.1 (3)	C8—C6—C7—C2	−179.47 (19)
O1—C1—C2—C3	−179.5 (2)	C3—C2—C7—C6	−1.5 (3)
O2—C1—C2—C7	−178.6 (2)	C1—C2—C7—C6	178.25 (19)
O1—C1—C2—C7	0.8 (3)	C7—C6—C8—O4	173.3 (2)
C7—C2—C3—C4	0.6 (3)	C5—C6—C8—O4	−7.3 (3)
C1—C2—C3—C4	−179.1 (2)	C7—C6—C8—O3	−7.1 (3)
C2—C3—C4—C5	0.6 (3)	C5—C6—C8—O3	172.3 (2)
C2—C3—C4—C9	−179.5 (2)	C5—C4—C9—C12	−1.2 (4)
C3—C4—C5—C6	−1.0 (3)	C3—C4—C9—C12	178.9 (3)
C9—C4—C5—C6	179.1 (2)	C5—C4—C9—C11	−122.5 (3)
C4—C5—C6—C7	0.1 (3)	C3—C4—C9—C11	57.6 (3)
C4—C5—C6—C8	−179.27 (19)	C5—C4—C9—C10	118.4 (3)
C5—C6—C7—C2	1.1 (3)	C3—C4—C9—C10	−61.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O1—H1O···O2^{i}	0.86 (1)	1.75 (1)	2.606 (2)	178 (4)
O3—H3O···O4^{ii}	0.86 (1)	1.73 (1)	2.588 (2)	176 (5)

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

Fig. 1