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3-Bromo-5-*tert*-butyl-2-hydroxybenzaldehyde

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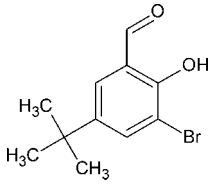
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.056; wR factor = 0.162; data-to-parameter ratio = 21.4.

The molecular conformation of the title compound, $C_{11}H_{13}BrO_2$, is stabilized by an intramolecular O—H···O hydrogen bond. All non-H atoms except the methyl groups lie approximately in a common plane (r.m.s. deviation = 0.011 Å).

Related literature

For the biological activity of substituted salicylaldehyde and its derivatives, see: Mounika *et al.* (2010); Dueke-Eze *et al.* (2010); Jesmin *et al.* (2010). For a related structure, see: Wang *et al.* (2010).



Experimental

Crystal data

$C_{11}H_{13}BrO_2$
M_r = 257.11
Orthorhombic, *Pbca*
a = 9.9727 (19) Å
b = 12.174 (2) Å
c = 18.558 (3) Å
V = 2253.0 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.62 mm^{-1}
T = 293 K
0.2 × 0.2 × 0.2 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
11555 measured reflections
2808 independent reflections
1442 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.162
S = 1.02
2808 reflections
131 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1	0.82	1.93	2.650 (6)	145

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5688).

References

Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dueke-Eze, C. U., Fasina, T. M. & Idika, N. (2010). *Afr. J. Pure Appl. Chem.* **5**, 13–18.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Jesmin, M., Ali, M. M. & Khanam, J. A. (2010). *Thai J. Pharm. Sci.* **34**, 20–31.
Mounika, K., Anupama, B., Pragathi, J. & Gyanakumari, C. (2010). *J. Sci. Res.* **2**, 513-524.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148-155.
Wang, Y., Qiu, Z. & Liang, H. (2010). *Acta Cryst.* E**66**, o2218.

supporting information

Acta Cryst. (2011). E**67**, o3375 [https://doi.org/10.1107/S1600536811048847]

3-Bromo-5-*tert*-butyl-2-hydroxybenzaldehyde

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S1. Comment

The crystal structure determination of the title compound was undertaken as a part of the synthesis, structure and properties of new substituted salicylaldehyde derivatives.

In the title compound, the substituted aldehyde group, hydroxy group and and bromine are essentially coplanar with the benzene ring with a plane mean deviation of 0.025 (6)°, -0.029 (4)°, -0.015 (1)° and -0.015 (1)°, respectively. An intramolecular O2—H2A···O(1) hydrogen bonding observed between the oxygen atoms of the hydroxy group and the aldehyde group stabilizes the molecular structure.

S2. Experimental

The synthesis of the title compound follows the modified Riemmer-Tiemann reaction, in which the substituted salicyl-aldehydes were synthesized from substituted phenols. To 80 mL of water 60g of sodium hydroxide was added and dissolved completely. Then 15g of 4-*tert*-butyl phenol was added and heated to 60-65°C. 30 mL chloroform was added step by step to the mixture. The resulting reaction mixture was heated for one hour, until the formation of precipitate. The liquid layer containing 5-*tert*-butyl-2-hydroxy benzaldehyde as the product was separated through suction pump. It was then brominated using liquid bromine and acetic acid. The final product 3-bromo-5-*tert*-butyl-2-hydroxy benzaldehyde with a maximum yield of 83% was checked for purity using TLC.

S3. Refinement

Hydrogen atoms were placed in calculated positions with O—H = 0.82Å, $C_{aromatic}$—H = 0.93Å, C_{methyl}—H = 0.96Å and refined using a riding model with fixed isotropic displacement parameters U_{iso}(H) = 1.5 U_{eq}(C_{methyl}, O) or U_{iso}(H) = 1.2 U_{eq}($C_{aromatic}$) or.

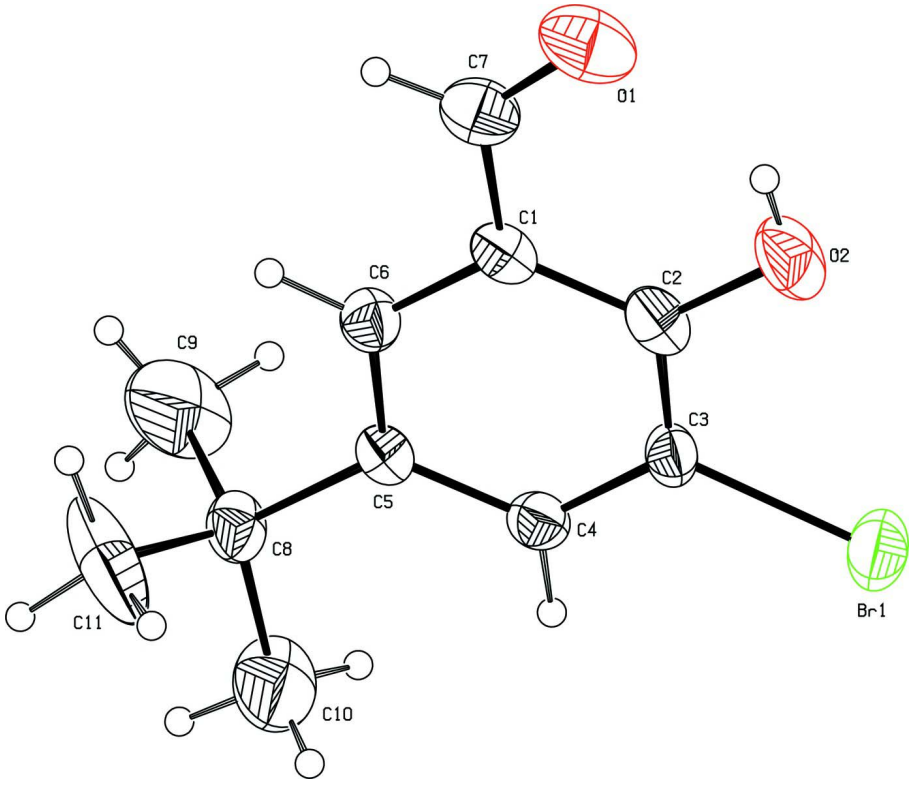

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

3-Bromo-5-*tert*-butyl-2-hydroxybenzaldehyde

Crystal data

$C_{11}H_{13}BrO_2$
$M_r = 257.11$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 9.9727\ (19)$ Å
$b = 12.174\ (2)$ Å
$c = 18.558\ (3)$ Å
$V = 2253.0\ (7)$ Å^3
$Z = 8$
$F(000) = 1040$
$D_x = 1.516$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2808 reflections
$\theta = 2.2–28.3°$
$\mu = 3.62$ mm^{-1}
$T = 293$ K
Block, red
0.2 × 0.2 × 0.2 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
11555 measured reflections
2808 independent reflections
1442 reflections with $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -13\rightarrow13$
$k = -16\rightarrow16$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.162$
$S = 1.02$
2808 reflections
131 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 1.3566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.59$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0228 (17)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2770 (5)	0.1032 (4)	0.2246 (2)	0.0474 (11)
C2	0.3680 (5)	0.0161 (4)	0.2178 (2)	0.0507 (12)
C3	0.4296 (4)	−0.0219 (3)	0.2794 (2)	0.0454 (10)
C4	0.4025 (4)	0.0242 (3)	0.3456 (2)	0.0462 (10)
H4	0.4468	−0.0027	0.3860	0.055*
C5	0.3112 (4)	0.1097 (3)	0.3540 (2)	0.0422 (10)
C6	0.2493 (5)	0.1482 (3)	0.2924 (2)	0.0473 (11)
H2	0.1878	0.2054	0.2960	0.057*
C7	0.2094 (6)	0.1487 (4)	0.1612 (3)	0.0656 (14)
H7	0.1509	0.2073	0.1680	0.079*
C8	0.2802 (5)	0.1613 (4)	0.4275 (2)	0.0526 (12)
C9	0.1367 (8)	0.1334 (7)	0.4478 (4)	0.136 (3)
H9A	0.1272	0.1358	0.4992	0.204*
H9B	0.1153	0.0610	0.4308	0.204*
H9C	0.0770	0.1857	0.4262	0.204*
C10	0.3682 (8)	0.1149 (6)	0.4877 (3)	0.104 (2)
H10A	0.4604	0.1319	0.4780	0.156*
H10B	0.3570	0.0367	0.4901	0.156*
H10C	0.3423	0.1471	0.5328	0.156*
C11	0.3014 (10)	0.2826 (5)	0.4242 (3)	0.131 (4)
H11A	0.3899	0.2977	0.4063	0.196*
H11B	0.2919	0.3133	0.4716	0.196*
H11C	0.2362	0.3149	0.3926	0.196*

O1	0.2253 (5)	0.1147 (3)	0.1006 (2)	0.0919 (14)
O2	0.3953 (4)	−0.0316 (3)	0.15377 (18)	0.0749 (10)
H2A	0.3523	−0.0009	0.1219	0.112*
Br1	0.55350 (5)	−0.13980 (4)	0.27226 (4)	0.0729 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.050 (3)	0.052 (2)	0.040 (2)	−0.012 (2)	0.001 (2)	−0.0048 (19)
C2	0.050 (3)	0.056 (2)	0.047 (3)	−0.017 (2)	0.013 (2)	−0.012 (2)
C3	0.041 (2)	0.0376 (19)	0.057 (3)	−0.0019 (17)	0.005 (2)	−0.0125 (18)
C4	0.046 (2)	0.045 (2)	0.047 (3)	−0.004 (2)	−0.005 (2)	−0.0026 (19)
C5	0.047 (3)	0.042 (2)	0.038 (2)	−0.0035 (18)	0.001 (2)	−0.0045 (16)
C6	0.052 (3)	0.043 (2)	0.047 (3)	0.001 (2)	0.000 (2)	−0.0012 (17)
C7	0.073 (4)	0.076 (3)	0.048 (3)	−0.003 (3)	−0.010 (3)	0.002 (2)
C8	0.065 (3)	0.057 (3)	0.036 (2)	0.010 (2)	0.007 (3)	−0.0029 (19)
C9	0.095 (5)	0.231 (10)	0.081 (5)	−0.019 (6)	0.037 (5)	−0.059 (5)
C10	0.142 (7)	0.119 (5)	0.052 (3)	0.040 (5)	−0.007 (4)	−0.004 (3)
C11	0.276 (12)	0.059 (3)	0.057 (3)	0.006 (5)	−0.001 (5)	−0.017 (3)
O1	0.111 (4)	0.119 (3)	0.046 (2)	−0.009 (3)	−0.015 (2)	−0.001 (2)
O2	0.080 (2)	0.092 (2)	0.053 (2)	−0.007 (2)	0.019 (2)	−0.0289 (18)
Br1	0.0566 (4)	0.0588 (4)	0.1033 (6)	0.0078 (2)	0.0097 (3)	−0.0204 (3)

Geometric parameters (Å, º)

C1—C6	1.400 (6)	C8—C11	1.494 (7)
C1—C2	1.401 (7)	C8—C9	1.518 (9)
C1—C7	1.465 (7)	C8—C10	1.528 (8)
C2—O2	1.350 (5)	C9—H9A	0.9600
C2—C3	1.377 (6)	C9—H9B	0.9600
C3—C4	1.377 (6)	C9—H9C	0.9600
C3—Br1	1.899 (4)	C10—H10A	0.9600
C4—C5	1.392 (6)	C10—H10B	0.9600
C4—H4	0.9300	C10—H10C	0.9600
C5—C6	1.381 (6)	C11—H11A	0.9600
C5—C8	1.533 (6)	C11—H11B	0.9600
C6—H2	0.9300	C11—H11C	0.9600
C7—O1	1.209 (6)	O2—H2A	0.8200
C7—H7	0.9300		
C6—C1—C2	120.3 (4)	C9—C8—C10	106.1 (5)
C6—C1—C7	118.9 (4)	C11—C8—C5	109.8 (4)
C2—C1—C7	120.8 (4)	C9—C8—C5	108.6 (5)
O2—C2—C3	119.8 (4)	C10—C8—C5	112.6 (4)
O2—C2—C1	122.3 (4)	C8—C9—H9A	109.5
C3—C2—C1	117.9 (4)	C8—C9—H9B	109.5
C4—C3—C2	121.1 (4)	H9A—C9—H9B	109.5
C4—C3—Br1	119.8 (4)	C8—C9—H9C	109.5

C2—C3—Br1	119.1 (3)	H9A—C9—H9C	109.5
C3—C4—C5	122.2 (4)	H9B—C9—H9C	109.5
C3—C4—H4	118.9	C8—C10—H10A	109.5
C5—C4—H4	118.9	C8—C10—H10B	109.5
C6—C5—C4	116.9 (4)	H10A—C10—H10B	109.5
C6—C5—C8	120.5 (4)	C8—C10—H10C	109.5
C4—C5—C8	122.5 (4)	H10A—C10—H10C	109.5
C5—C6—C1	121.6 (4)	H10B—C10—H10C	109.5
C5—C6—H2	119.2	C8—C11—H11A	109.5
C1—C6—H2	119.2	C8—C11—H11B	109.5
O1—C7—C1	123.9 (5)	H11A—C11—H11B	109.5
O1—C7—H7	118.1	C8—C11—H11C	109.5
C1—C7—H7	118.1	H11A—C11—H11C	109.5
C11—C8—C9	111.4 (6)	H11B—C11—H11C	109.5
C11—C8—C10	108.3 (5)	C2—O2—H2A	109.5

C6—C1—C2—O2	178.4 (4)	C4—C5—C6—C1	0.1 (6)
C7—C1—C2—O2	−1.7 (7)	C8—C5—C6—C1	179.4 (4)
C6—C1—C2—C3	−0.8 (6)	C2—C1—C6—C5	0.8 (7)
C7—C1—C2—C3	179.0 (4)	C7—C1—C6—C5	−179.1 (4)
O2—C2—C3—C4	−179.2 (4)	C6—C1—C7—O1	−179.1 (5)
C1—C2—C3—C4	0.0 (6)	C2—C1—C7—O1	1.0 (8)
O2—C2—C3—Br1	0.7 (6)	C6—C5—C8—C11	−53.8 (7)
C1—C2—C3—Br1	180.0 (3)	C4—C5—C8—C11	125.4 (6)
C2—C3—C4—C5	0.9 (7)	C6—C5—C8—C9	68.3 (6)
Br1—C3—C4—C5	−179.1 (3)	C4—C5—C8—C9	−112.5 (6)
C3—C4—C5—C6	−0.9 (6)	C6—C5—C8—C10	−174.5 (5)
C3—C4—C5—C8	179.8 (4)	C4—C5—C8—C10	4.7 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O1	0.82	1.93	2.650 (6)	145