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2-Hydroxy-3-nitrobenzaldehyde

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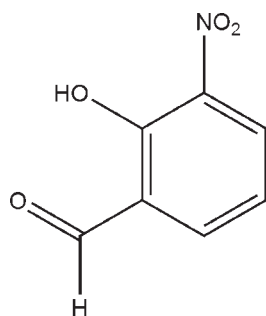
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.119; data-to-parameter ratio = 11.3.

The title compound, $\text{C}_7\text{H}_5\text{NO}_4$, isolated from the leaves of *Actephila merrilliana*, is essentially planar (r.m.s. deviation = 0.026 Å). The conformation is supported by an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond, which generates an $S(6)$ ring. In the crystal, $\text{C}-\text{H}\cdots\text{O}$ interactions and aromatic $\pi-\pi$ stacking [centroid-centroid distance = 3.754 (4) Å] help to establish the packing.

Related literature

For medicinal background, see: Ovenden *et al.* (2001); Song *et al.* (2007). For related structures, see: Rizal *et al.* (2008); Garden *et al.* (2004).



Experimental

Crystal data

$\text{C}_7\text{H}_5\text{NO}_4$

$M_r = 167.12$

Monoclinic, $P2_1/n$
 $a = 8.8276$ (7) Å
 $b = 8.7296$ (8) Å
 $c = 9.011$ (9) Å
 $\beta = 90.124$ (1)°
 $V = 694.4$ (7) Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 298$ K
 $0.48 \times 0.48 \times 0.42$ mm

Data collection

Bruker SMART CCD diffractometer
3289 measured reflections

1230 independent reflections
929 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.119$
 $S = 1.07$
1230 reflections

109 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2}\cdots\text{O1}$	0.82	1.86	2.597 (3)	148
$\text{C5}-\text{H5}\cdots\text{O2}^i$	0.93	2.51	3.422 (4)	168

Symmetry code: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5495).

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supplementary materials

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2-Hydroxy-3-nitrobenzaldehyde

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Comment

Although chemical investigations into specimens of the plant family Euphorbiaceae are very common, there have been no related reports on chemical constituents of *Actephila merrilliana* (Ovenden *et al.*, 2001). The plants in this family were used in folk medicine such as, for the treatment of hemorrhoids and as anti-inflammatory agents (Song *et al.*, 2007). The title compound was isolated from the 75% EtOH extract of the leaves of *Actephila merrilliana* which were collected from Sanya City, Hainan Province, P. R. China. We have undertaken the X-ray crystal structure analysis of the title compound in order to establish its molecular structure and relative stereochemistry. In the title compound, the bond lengths and angles in (I) have normal values, and are comparable with those in the related structures (Rizal *et al.*, 2008; Garden *et al.*, 2004). An intramolecular O—H \cdots O hydrogen bond helps to establish an essentially planar conformation for the molecule (r.m.s. deviation = 0.0258 Å).

In the crystal, molecules are linked by intermolecular C—H \cdots O hydrogen bonds into chains (Fig.2). The hydrogen bonds and angles are listed in Table 1.

Experimental

Air-dried leaves of *Actephila merrilliana* (14.0 kg) were ground and percolated (3 \times 3 h) with 75% EtOH at 60°C, which was suspended in 6 L water and then partitioned with petroleum ether, chloroform, ethyl acetate and n-BuOH, successively, yielding a petroleum ether extract, a chloroform extract, an ethyl acetate extract and a n-BuOH extract, respectively. The chloroform extract was subjected to a silica gel CC column using petroleum ether as first eluent and then increasing the polarity with EtOAc, to afford 23 fractions (A—W). Fraction M was further separated by column chromatography with a gradient of petroleum ether-EtOAc to give the title compound. The crude product was dissolved in a small amount of chloroform to obtain colourless blocks of (I) by slow evaporation of a chloroform solution at 298 K.

Refinement

H atoms bonded to C atoms were placed in geometrically calculated position and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. H atoms attached to O atoms were found in a difference Fourier synthesis and were refined using a riding model, with the O—H distances fixed as initially found and with $U_{\text{iso}}(\text{H})$ values set at $1.5 U_{\text{eq}}(\text{O})$.

Figures

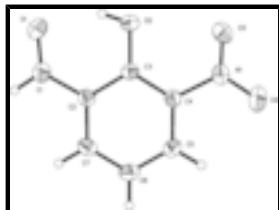


Fig. 1. View of (I) with displacement ellipsoids drawn at the 30% probability level.

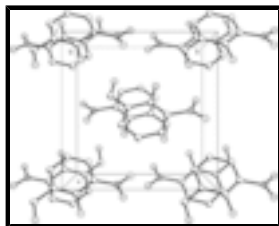


Fig. 2. A view of the molecular packing.

2-hydroxy-3-nitrobenzaldehyde

Crystal data

$C_7H_5NO_4$

$M_r = 167.12$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.8276$ (7) Å

$b = 8.7296$ (8) Å

$c = 9.011$ (9) Å

$\beta = 90.124$ (1)°

$V = 694.4$ (7) Å³

$Z = 4$

$F(000) = 344$

$D_x = 1.599$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1493 reflections

$\theta = 2.3$ – 27.7 °

$\mu = 0.13$ mm⁻¹

$T = 298$ K

Block, colourless

$0.48 \times 0.48 \times 0.42$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

ϕ and ω scans

3289 measured reflections

1230 independent reflections

929 reflections with $I > 2\sigma(I)$

$R_{int} = 0.026$

$\theta_{max} = 25.0$ °, $\theta_{min} = 3.2$ °

$h = -6 \rightarrow 10$

$k = -9 \rightarrow 10$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

$wR(F^2) = 0.119$	H-atom parameters constrained
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 0.1126P]$
1230 reflections	where $P = (F_o^2 + 2F_c^2)/3$
109 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$

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. 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.50055 (17)	0.84855 (17)	0.75856 (18)	0.0503 (5)
O1	0.29576 (16)	0.32129 (17)	0.56300 (17)	0.0660 (5)
O2	0.33112 (14)	0.61257 (16)	0.61024 (14)	0.0548 (4)
H2	0.2948	0.5346	0.5739	0.082*
O3	0.4312 (2)	0.89120 (17)	0.6494 (2)	0.0793 (5)
O4	0.55192 (19)	0.93655 (17)	0.85100 (18)	0.0769 (5)
C1	0.3983 (2)	0.2987 (2)	0.6511 (2)	0.0519 (5)
H1	0.4261	0.1978	0.6698	0.062*
C2	0.48004 (18)	0.4189 (2)	0.72888 (18)	0.0391 (4)
C3	0.44390 (18)	0.5747 (2)	0.70290 (18)	0.0376 (4)
C4	0.52863 (19)	0.68465 (19)	0.77951 (19)	0.0390 (4)
C5	0.64236 (19)	0.6430 (2)	0.8776 (2)	0.0440 (5)
H5	0.6965	0.7184	0.9277	0.053*
C6	0.67596 (19)	0.4909 (2)	0.9015 (2)	0.0490 (5)
H6	0.7529	0.4634	0.9669	0.059*
C7	0.5946 (2)	0.3804 (2)	0.8277 (2)	0.0451 (5)
H7	0.6168	0.2776	0.8444	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0559 (10)	0.0375 (9)	0.0574 (11)	-0.0026 (7)	-0.0013 (8)	-0.0013 (8)
O1	0.0698 (10)	0.0569 (9)	0.0712 (10)	-0.0092 (7)	-0.0219 (8)	-0.0111 (7)
O2	0.0597 (8)	0.0466 (8)	0.0580 (8)	0.0000 (6)	-0.0271 (7)	0.0031 (6)
O3	0.1053 (13)	0.0440 (9)	0.0886 (12)	0.0024 (8)	-0.0348 (10)	0.0140 (8)

supplementary materials

O4	0.1027 (13)	0.0452 (9)	0.0828 (11)	-0.0027 (8)	-0.0148 (9)	-0.0167 (8)
C1	0.0595 (12)	0.0415 (10)	0.0546 (12)	-0.0029 (9)	-0.0042 (10)	-0.0039 (9)
C2	0.0439 (10)	0.0373 (10)	0.0362 (9)	0.0015 (7)	0.0008 (7)	0.0003 (7)
C3	0.0403 (9)	0.0396 (9)	0.0329 (9)	0.0007 (7)	-0.0037 (7)	0.0014 (7)
C4	0.0433 (9)	0.0344 (9)	0.0392 (9)	-0.0012 (7)	-0.0007 (7)	0.0004 (7)
C5	0.0413 (10)	0.0477 (11)	0.0429 (10)	-0.0058 (8)	-0.0039 (8)	-0.0032 (8)
C6	0.0440 (10)	0.0569 (12)	0.0461 (11)	0.0047 (8)	-0.0091 (8)	0.0014 (9)
C7	0.0486 (10)	0.0429 (10)	0.0438 (10)	0.0094 (8)	-0.0007 (8)	0.0036 (8)

Geometric parameters (Å, °)

N1—O3	1.216 (2)	C2—C3	1.416 (2)
N1—O4	1.220 (2)	C3—C4	1.399 (3)
N1—C4	1.464 (2)	C4—C5	1.384 (2)
O1—C1	1.219 (2)	C5—C6	1.377 (3)
O2—C3	1.340 (2)	C5—H5	0.9300
O2—H2	0.8200	C6—C7	1.374 (3)
C1—C2	1.452 (3)	C6—H6	0.9300
C1—H1	0.9300	C7—H7	0.9300
C2—C7	1.388 (2)		
O3—N1—O4	123.06 (18)	C5—C4—C3	121.42 (17)
O3—N1—C4	119.23 (15)	C5—C4—N1	117.45 (15)
O4—N1—C4	117.69 (17)	C3—C4—N1	121.13 (16)
C3—O2—H2	109.5	C6—C5—C4	120.55 (16)
O1—C1—C2	124.42 (19)	C6—C5—H5	119.7
O1—C1—H1	117.8	C4—C5—H5	119.7
C2—C1—H1	117.8	C7—C6—C5	119.31 (17)
C7—C2—C3	120.15 (16)	C7—C6—H6	120.3
C7—C2—C1	119.71 (18)	C5—C6—H6	120.3
C3—C2—C1	120.14 (17)	C6—C7—C2	121.32 (18)
O2—C3—C4	122.28 (16)	C6—C7—H7	119.3
O2—C3—C2	120.47 (16)	C2—C7—H7	119.3
C4—C3—C2	117.24 (16)		
O1—C1—C2—C7	179.64 (18)	O3—N1—C4—C5	161.56 (17)
O1—C1—C2—C3	-0.9 (3)	O4—N1—C4—C5	-16.7 (2)
C7—C2—C3—O2	-178.44 (15)	O3—N1—C4—C3	-18.3 (3)
C1—C2—C3—O2	2.1 (2)	O4—N1—C4—C3	163.53 (16)
C7—C2—C3—C4	0.5 (2)	C3—C4—C5—C6	0.5 (3)
C1—C2—C3—C4	-178.95 (15)	N1—C4—C5—C6	-179.32 (16)
O2—C3—C4—C5	178.41 (15)	C4—C5—C6—C7	-0.5 (3)
C2—C3—C4—C5	-0.5 (2)	C5—C6—C7—C2	0.5 (3)
O2—C3—C4—N1	-1.8 (3)	C3—C2—C7—C6	-0.5 (3)
C2—C3—C4—N1	179.32 (14)	C1—C2—C7—C6	178.94 (17)

Hydrogen-bond geometry (Å, °)

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
O2—H2 \cdots O1	0.82	1.86	2.597 (3)	148
C5—H5 \cdots O2 ⁱ	0.93	2.51	3.422 (4)	168

Symmetry codes: (i) $x+1/2, -y+3/2, z+1/2$.

Fig. 1

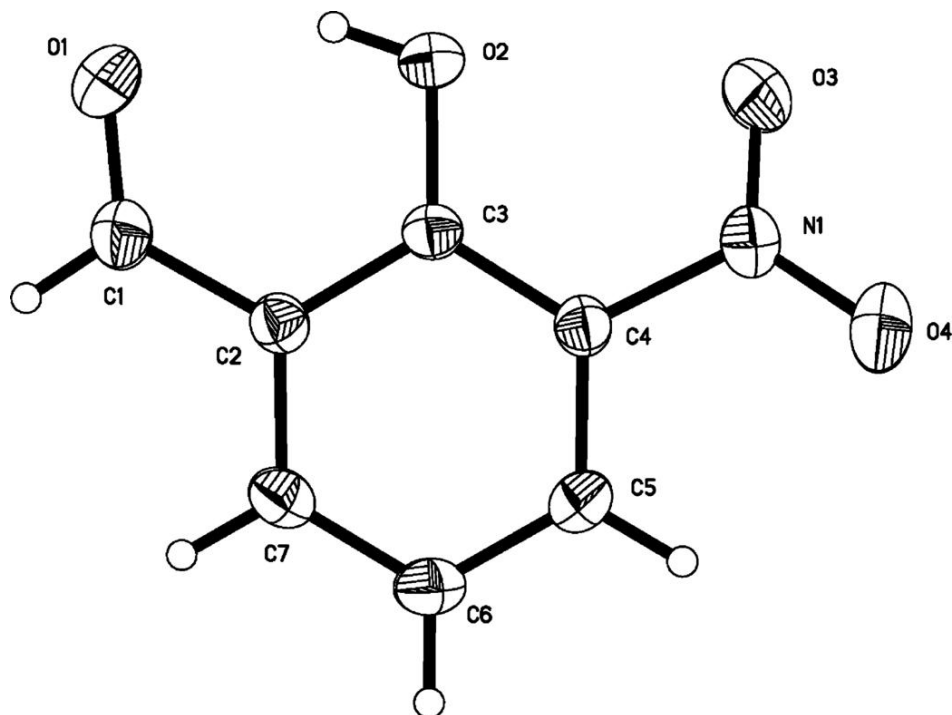


Fig. 2

