Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Hydroxy-4-nitrophenyl acetate

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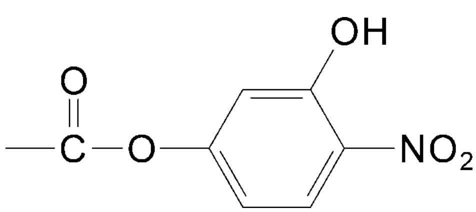
Received 21 November 2008; accepted 4 December 2008

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.083; data-to-parameter ratio = 11.2.

In the molecule of the title compound, $C_8H_7NO_5$, the acetate group is oriented with respect to the aromatic ring at a dihedral angle of 85.30 (3)°. An intramolecular O—H···O hydrogen bond results in the formation of a non-planar six-membered ring, adopting an envelope conformation. In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules.

Related literature

For general background to phenolic esters as intermediates in organic synthesis, see: Trollsås *et al.* (1996); Svensson *et al.* (1998); Atkinson *et al.* (2005); Hu *et al.* (2001). For a related structure, see: Ji *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_7NO_5$
M_r = 197.15
Monoclinic, $P2_1/n$
a = 10.881 (2) Å
b = 5.0543 (10) Å
c = 15.318 (3) Å
β = 93.75 (3)°
V = 840.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 153 (2) K
0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.969, T_{max} = 0.979
5058 measured reflections
1449 independent reflections
1232 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.083
S = 1.12
1449 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4	0.82	1.91	2.605 (2)	142
C5—H5···O1^{i}	0.93	2.58	3.229 (2)	127
C8—H8···O3^{ii}	0.93	2.56	3.481 (2)	170

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2582).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Atkinson, P. J., Bromidge, S. M., Duxon, M. S., Gaster, L. M., Hadley, M. S., Hammond, B., Johnson, C. N., Middlemiss, D. N., North, S. E., Price, G. W., Rami, H. K., Riley, G. J., Scott, C. M., Shaw, T. E., Starr, K. R., Stemp, G., Thewlis, K. M., Thomas, D. R., Thompson, M., Vong, A. K. K. & Watson, J. M. (2005). *Bioorg. Med. Chem. Lett.* **15**, 737–741.
Bruker (1997). *SMART* and *SAINT* . Bruker AXS Inc., Madison, Wisconsin, USA.
Hu, B., Ellingboe, J., Gunawan, I., Han, S., Largis, E., Li, Z., Malamas, M., Mulvey, R., Oliphant, A., Sum, F.-W., Tillett, J. & Wong, V. (2001). *Bioorg. Med. Chem. Lett.* **11**, 757–760.
Ji, X. & Li, C. (2006). *Synthesis*, **15**, 2478–2482.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Svensson, M., Helgee, B., Skarp, K. & Andersson, G. (1998). *J. Mater. Chem.* **8**, 353–362.
Trollsås, M., Orrenius, C., Sahlén, F., Gedde, U. W., Norin, T., Hult, A., Hermann, D., Rudquist, P., Komitov, L., Lagerwall, S. T. & Lindström, J. (1996). *J. Am. Chem. Soc.* **118**, 8542–8548.

supporting information

Acta Cryst. (2009). E**65**, o46 [doi:10.1107/S1600536808040981]

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Chao Liu, Chunqing Cheng and Xiujie Ji

S1. Comment

Phenolic esters are useful intermediates in organic synthesis (Trollsås *et al.*, 1996; Svensson *et al.*, 1998; Atkinson *et al.*, 2005; Hu *et al.*, 2001). We have developed a new method for the syntheses of some phenolic esters (Ji *et al.*, 2006). The title compound is one of the products, and we report herein its crystal structure.

In the molecule of the title compound (Fig. 1) the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The acetate group is oriented with respect to the aromatic ring at a dihedral angle of 85.30 (3)°. The intramolecular O-H···O hydrogen bond (Table 1) results in the formation of a nonplanar six-membered ring (N1/O3/O4/C6/C7/H3), adopting envelope conformation.

In the crystal structure, intermolecular C-H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

S2. Experimental

For the preparation of the title compound, 2-nitroresorcin acetate (239 mg, 1.0 mmol) was dissolved in chloroform (20 ml). At 273-278 K, anhydrous $AlCl_3$ (133.5 mg, 1 mmol) was added to this solution, the reaction was stirred at room temperature for 1 h, and then hydrochloric acid (5 ml, 10%) was added. The reaction mixture was extracted with chloroform and dried with anhydrous sodium sulfate. After concentration, the residue was separated by flash column chromatography and purified by recrystallization from chloroform (yield; 144 mg, 73%, m.p. 360 K). Spectroscopic analysis: IR (KBr, *v*, cm^{-1}): 3253, 3083, 2946, 1758, 1530, 1204, 1138, 978, 847. Analysis required for $C_8H_7NO_5$: C 48.74; H 3.58; N 7.10%. Found: C 48.80; H 3.61; N 7.08%.

S3. Refinement

H atoms were positioned geometrically, with O-H = 0.82 Å (for OH) and C-H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = xU_{eq}(C,O)$, where x = 1.2 for aromatic H and x = 1.5 for all other H atoms.

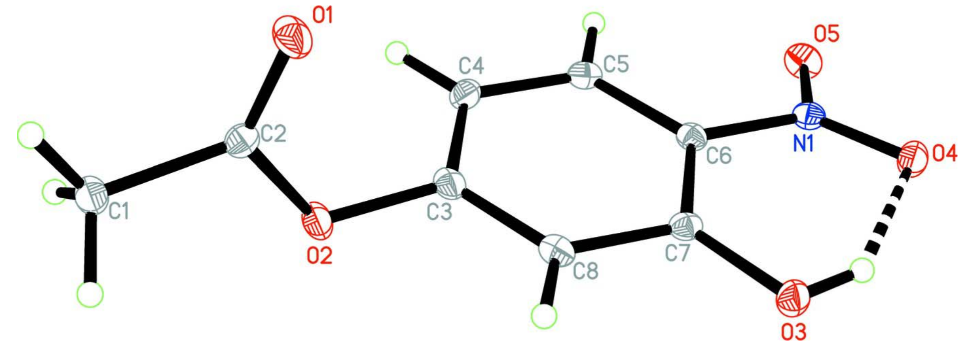

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bond is shown as ashed line.

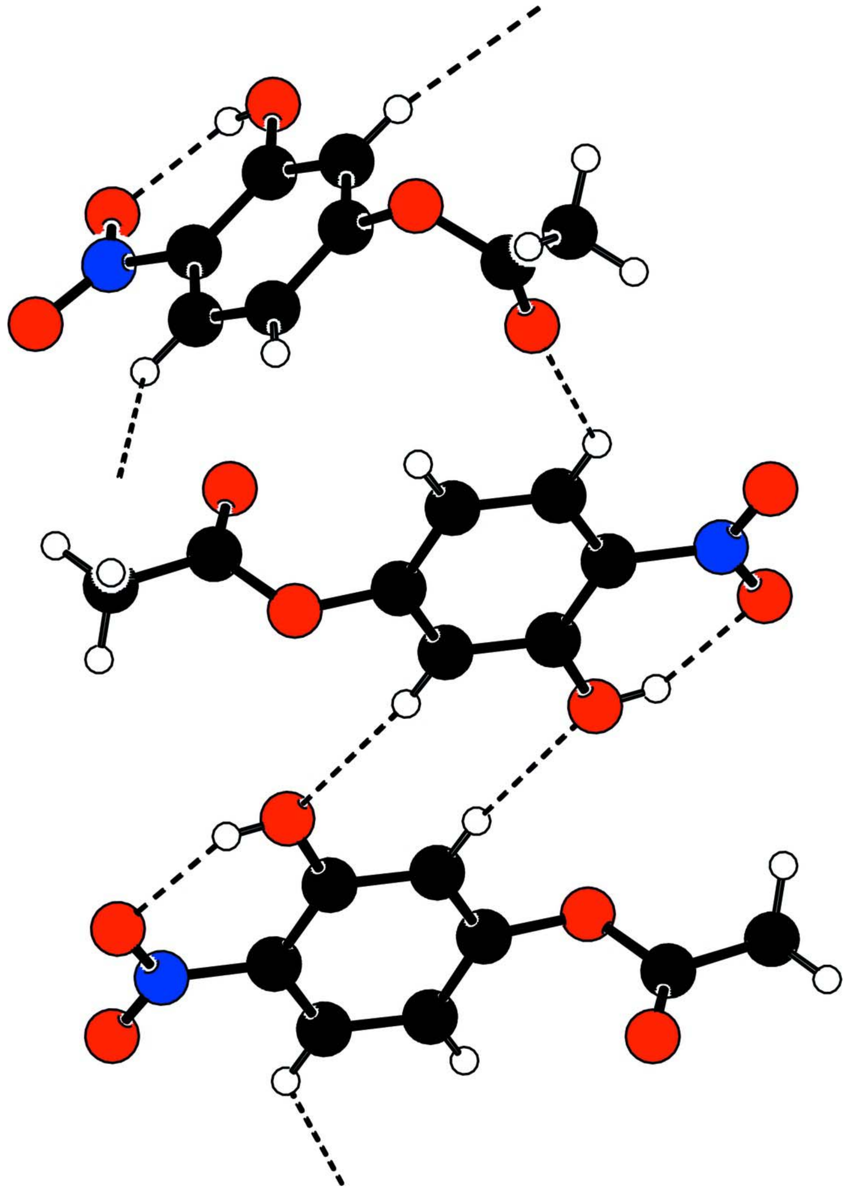

Figure 2

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

3-Hydroxy-4-nitrophenyl acetate

Crystal data

$C_8H_7NO_5$
M_r = 197.15
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 10.881 (2) Å
b = 5.0543 (10) Å
c = 15.318 (3) Å
β = 93.75 (3)°
V = 840.6 (3) Å^3
Z = 4
F(000) = 408
D_x = 1.558 Mg m^{-3}
Melting point: 360 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2650 reflections
θ = 2.2–27.5°
μ = 0.13 mm^{-1}
T = 153 K
Block, colorless
0.24 × 0.20 × 0.16 mm

Data collection

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.969$, $T_{max} = 0.979$
5058 measured reflections
1449 independent reflections
1232 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -12\rightarrow12$
$k = -6\rightarrow5$
$l = -18\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.083$
$S = 1.12$
1449 reflections
129 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
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.1223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.14195 (8)	0.58036 (19)	0.89666 (6)	0.0279 (3)
O2	0.31702 (7)	0.35401 (18)	0.93249 (5)	0.0244 (3)
O3	0.59843 (8)	1.04495 (18)	0.88687 (5)	0.0223 (2)
H3	0.6350	1.1192	0.8486	0.033*
O4	0.67226 (8)	1.08418 (17)	0.72904 (5)	0.0230 (2)
O5	0.63078 (8)	0.77288 (18)	0.63478 (5)	0.0247 (2)
N1	0.61633 (9)	0.8778 (2)	0.70551 (6)	0.0179 (3)
C1	0.13908 (11)	0.2225 (3)	1.00085 (8)	0.0213 (3)
H1A	0.0532	0.2620	1.0041	0.032*
H1B	0.1807	0.2425	1.0577	0.032*
H1C	0.1482	0.0438	0.9810	0.032*
C2	0.19373 (11)	0.4079 (2)	0.93821 (7)	0.0179 (3)
C3	0.38267 (10)	0.5030 (3)	0.87381 (8)	0.0195 (3)
C4	0.38272 (11)	0.4184 (3)	0.78716 (8)	0.0207 (3)
H4	0.3323	0.2804	0.7666	0.025*
C5	0.46001 (11)	0.5463 (2)	0.73292 (8)	0.0194 (3)

H5	0.4633	0.4919	0.6751	0.023*
C6	0.53337 (10)	0.7573 (2)	0.76450 (7)	0.0164 (3)
C7	0.53043 (10)	0.8455 (2)	0.85126 (7)	0.0168 (3)
C8	0.45238 (10)	0.7116 (2)	0.90598 (7)	0.0189 (3)
H8	0.4480	0.7643	0.9639	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0222 (5)	0.0277 (6)	0.0342 (5)	0.0061 (4)	0.0054 (4)	0.0129 (4)
O2	0.0180 (5)	0.0266 (5)	0.0290 (5)	0.0010 (4)	0.0041 (3)	0.0131 (4)
O3	0.0269 (5)	0.0225 (5)	0.0177 (4)	−0.0062 (4)	0.0019 (4)	−0.0023 (3)
O4	0.0233 (5)	0.0224 (5)	0.0232 (5)	−0.0057 (4)	0.0018 (3)	−0.0005 (4)
O5	0.0302 (5)	0.0282 (5)	0.0164 (4)	0.0030 (4)	0.0064 (4)	−0.0034 (4)
N1	0.0165 (5)	0.0200 (6)	0.0171 (5)	0.0032 (4)	0.0004 (4)	0.0005 (4)
C1	0.0219 (6)	0.0208 (7)	0.0216 (6)	−0.0008 (5)	0.0033 (5)	0.0030 (5)
C2	0.0176 (6)	0.0169 (6)	0.0190 (6)	−0.0003 (5)	0.0003 (5)	−0.0026 (5)
C3	0.0143 (6)	0.0208 (7)	0.0235 (6)	0.0042 (5)	0.0021 (5)	0.0071 (5)
C4	0.0177 (6)	0.0182 (7)	0.0255 (7)	−0.0008 (5)	−0.0033 (5)	0.0010 (5)
C5	0.0204 (6)	0.0191 (7)	0.0180 (6)	0.0029 (5)	−0.0022 (5)	−0.0012 (5)
C6	0.0150 (6)	0.0177 (6)	0.0166 (6)	0.0033 (5)	0.0012 (4)	0.0018 (4)
C7	0.0162 (6)	0.0158 (6)	0.0182 (6)	0.0035 (5)	−0.0016 (4)	−0.0003 (5)
C8	0.0197 (6)	0.0211 (7)	0.0159 (6)	0.0043 (5)	0.0022 (5)	0.0017 (5)

Geometric parameters (Å, º)

O1—C2	1.1982 (15)	C1—H1C	0.9600
O2—C2	1.3773 (15)	C3—C8	1.3716 (18)
O2—C3	1.4032 (14)	C3—C4	1.3944 (17)
O3—C7	1.3447 (15)	C4—C5	1.3807 (17)
O3—H3	0.8200	C4—H4	0.9300
O4—N1	1.2489 (13)	C5—C6	1.3994 (17)
O5—N1	1.2255 (12)	C5—H5	0.9300
N1—C6	1.4521 (15)	C6—C7	1.4041 (16)
C1—C2	1.4923 (16)	C7—C8	1.4054 (17)
C1—H1A	0.9600	C8—H8	0.9300
C1—H1B	0.9600		
C2—O2—C3	118.26 (9)	C4—C3—O2	118.51 (11)
C7—O3—H3	109.5	C5—C4—C3	117.87 (12)
O5—N1—O4	121.88 (10)	C5—C4—H4	121.1
O5—N1—C6	119.32 (10)	C3—C4—H4	121.1
O4—N1—C6	118.79 (9)	C4—C5—C6	120.33 (11)
C2—C1—H1A	109.5	C4—C5—H5	119.8
C2—C1—H1B	109.5	C6—C5—H5	119.8
H1A—C1—H1B	109.5	C5—C6—C7	121.42 (11)
C2—C1—H1C	109.5	C5—C6—N1	117.92 (10)
H1A—C1—H1C	109.5	C7—C6—N1	120.63 (11)

H1B—C1—H1C	109.5	O3—C7—C6	125.16 (10)
O1—C2—O2	122.43 (11)	O3—C7—C8	117.20 (10)
O1—C2—C1	127.24 (11)	C6—C7—C8	117.62 (11)
O2—C2—C1	110.33 (10)	C3—C8—C7	119.85 (11)
C8—C3—C4	122.88 (11)	C3—C8—H8	120.1
C8—C3—O2	118.37 (10)	C7—C8—H8	120.1

C3—O2—C2—O1	−1.83 (17)	O5—N1—C6—C7	169.13 (10)
C3—O2—C2—C1	177.59 (10)	O4—N1—C6—C7	−9.92 (16)
C2—O2—C3—C8	98.78 (13)	C5—C6—C7—O3	179.46 (10)
C2—O2—C3—C4	−86.75 (14)	N1—C6—C7—O3	1.57 (18)
C8—C3—C4—C5	2.32 (18)	C5—C6—C7—C8	0.95 (17)
O2—C3—C4—C5	−171.88 (10)	N1—C6—C7—C8	−176.94 (10)
C3—C4—C5—C6	−1.34 (18)	C4—C3—C8—C7	−1.64 (18)
C4—C5—C6—C7	−0.25 (18)	O2—C3—C8—C7	172.57 (10)
C4—C5—C6—N1	177.70 (10)	O3—C7—C8—C3	−178.67 (10)
O5—N1—C6—C5	−8.83 (15)	C6—C7—C8—C3	−0.03 (17)
O4—N1—C6—C5	172.12 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4	0.82	1.91	2.605 (2)	142
C5—H5···O1^{i}	0.93	2.58	3.229 (2)	127
C8—H8···O3^{ii}	0.93	2.56	3.481 (2)	170

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