Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Hydroxy-3,5-dimethoxybenzonitrile at 125 K

Xiang-Wei Cheng

Zhejiang Police College Laboratory Center, Zhejiang Police College, Hangzhou 310053, People's Republic of China
Correspondence e-mail: zpccxw@126.com

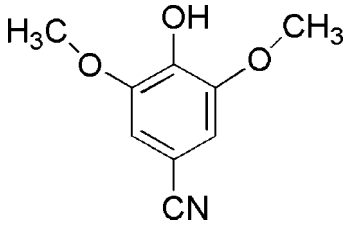
Received 7 August 2007; accepted 8 August 2007

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.113; data-to-parameter ratio = 17.3.

In the title compound, $C_9H_9NO_3$, all non-H atoms are coplanar. An O—H···O hydrogen bond is observed in the molecular structure and intermolecular O—H···N hydrogen bonds link the molecules into a chain along the a axis.

Related literature

For synthesis, see: Van Es (1965). For general background, see: Chihiro *et al.* (1995); Diana *et al.* (1993). For a related structure, see: Zabinski *et al.* (2007).



Experimental

Crystal data

$C_9H_9NO_3$
M_r = 179.17
Orthorhombic, *Pbca*
a = 8.9624 (12) Å
b = 12.5413 (16) Å
c = 15.881 (2) Å
V = 1785.1 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 123 (2) K
0.50 × 0.50 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.848, T_{max} = 0.874
(expected range = 0.932–0.960)
17694 measured reflections
2036 independent reflections
1469 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.113
S = 1.05
2036 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.84	2.22	2.6710 (14)	114
O3—H3···N1^{i}	0.84	2.15	2.8286 (17)	138

Symmetry code: (i) $x+1, y, z$.

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: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

The author acknowledges financial support from Zhejiang Police College, People's Republic of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2440).

References

Bruker (2002). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chihiro, M., Nagamoto, H., Takemura, I., Kitano, K., Komatsu, H., Sekiguchi, K., Tabusa, F., Mori, T., Tominnaga, M. & Yabuuchi, Y. (1995). *J. Med. Chem.* **38**, 353–358.
Diana, G. D., Dutcliffe, D., Volkots, D. L., Mallamo, J. P., Bailet, T. R., Vescio, N., Oglesby, R. C., Nitz, T. J., Wetzel, J., Ciranda, V., Pevear, D. C. & Dutko, F. J. (1993). *J. Med. Chem.* **36**, 3240–3250.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Van Es, T. (1965). *J. Chem. Soc.* pp. 1564–1564.
Zabinski, J., Wolska, I. & Maciejewska, D. (2007). *J. Mol. Struct.* **833**, 74–81.

 doi:10.1107/S1600536807039256

supplementary materials

Acta Cryst. (2007). E**63**, o3797 [**doi:10.1107/S1600536807039256**]

4-Hydroxy-3,5-dimethoxybenzonitrile at 125 K

X.-W. Cheng

Comment

Nitriles, important reagents for organic synthesis, have been known to chemists for a long time. Nitriles are used in the synthesis of thiazole derivatives which act as inhibitors of superoxide produced by neutrophils (Chihiro *et al.*, 1995). They are also used in the preparation of antipicornavirus tetrazole anologues (Diana *et al.*, 1993). The title compound is an important intermediate leading to bis-amidine which exhibits antimicrobial activity against a widespread range of microorganisms (Zabinski *et al.*, 2007). We report here the crystal structure of the title compound.

The non-hydrogen atoms in the molecule of the title compound are coplanar, with atoms N1, C7 and C8 deviating by 0.025 (2), 0.066 (2) and 0.042 (2) Å, respectively. The C≡N distance (1.1373 (17) Å) shows normal value (1.136 (2) Å; Zabinski *et al.*, 2007).

An intramolecular O—H···N hydrogen bond is observed. Intermolecular O—H···O hydrogen bonds link the molecules into chains along the *a* axis (Fig.2 and Table 1).

Experimental

The title compound was prepared according to the literature method of Van Es (1965). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an isoproanol solution at 295 K (m.p. 459–461 K).

Refinement

H atoms were positioned geometrically (O—H = 0.84 Å and C—H = 0.95 or 0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(O or C_{methyl}) or 1.2U_{eq}(C).

Figures

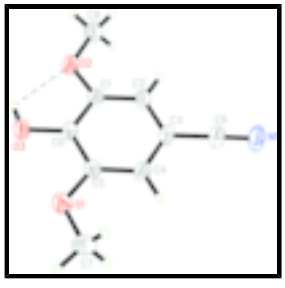


Fig. 1. Molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atomic numbering. The dashed line indicates a hydrogen bond.

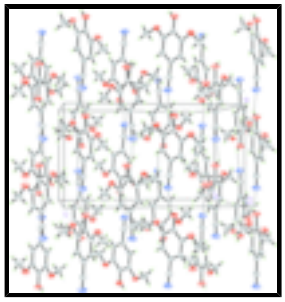


Fig. 2. The crystal packing of the title compound, viewed approximately down the *b* axis. Dashed lines indicate intermolecular hydrogen bonds.

4-Hydroxy-3,5-dimethoxybenzonitrile

Crystal data

$C_9H_9NO_3$	F_{000} = 752
M_r = 179.17	D_x = 1.333 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2036 reflections
a = 8.9624 (12) Å	θ = 2.6–27.5º
b = 12.5413 (16) Å	μ = 0.10 mm^{-1}
c = 15.881 (2) Å	*T* = 123 (2) K
V = 1785.1 (4) Å^3	Block, colourless
Z = 8	0.50 × 0.50 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2036 independent reflections
Radiation source: fine-focus sealed tube	1469 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 123(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	*h* = −11→10
T_{min} = 0.848, T_{max} = 0.874	*k* = −15→16
17694 measured reflections	*l* = −19→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.041	H-atom parameters constrained
$wR(F^2)$ = 0.113	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.2676P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
2036 reflections	$\Delta\rho_{max}$ = 0.12 e Å^{-3}

118 parameters	$\Delta\rho_{min} = -0.18$ 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. 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}
O2	0.31350 (10)	0.37213 (8)	0.04912 (7)	0.0553 (3)
O1	0.20865 (11)	0.07072 (9)	0.20812 (7)	0.0630 (3)
O3	0.39718 (10)	0.19656 (9)	0.13177 (7)	0.0603 (3)
H3	0.4478	0.2415	0.1049	0.090*
C4	−0.00241 (15)	0.17879 (11)	0.16337 (8)	0.0464 (3)
H4	−0.0729	0.1337	0.1904	0.056*
C5	0.14822 (15)	0.15641 (11)	0.16763 (8)	0.0450 (3)
C6	0.25106 (14)	0.22342 (11)	0.12798 (8)	0.0429 (3)
N1	−0.33080 (14)	0.30710 (12)	0.10859 (10)	0.0687 (4)
C3	−0.04928 (14)	0.26813 (11)	0.11906 (8)	0.0451 (3)
C2	0.05144 (15)	0.33650 (11)	0.07979 (9)	0.0462 (3)
H2	0.0174	0.3976	0.0501	0.055*
C1	0.20164 (14)	0.31359 (10)	0.08489 (9)	0.0427 (3)
C9	−0.20643 (15)	0.28999 (12)	0.11296 (10)	0.0520 (4)
C8	0.27231 (18)	0.46331 (13)	0.00121 (11)	0.0626 (4)
H8A	0.3623	0.4980	−0.0207	0.094*
H8B	0.2177	0.5134	0.0372	0.094*
H8C	0.2085	0.4414	−0.0458	0.094*
C7	0.1083 (2)	−0.00458 (15)	0.24377 (13)	0.0779 (5)
H7A	0.1651	−0.0620	0.2707	0.117*
H7B	0.0450	−0.0345	0.1993	0.117*
H7C	0.0456	0.0308	0.2859	0.117*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0366 (5)	0.0562 (6)	0.0730 (7)	−0.0017 (4)	0.0037 (4)	0.0177 (5)
O1	0.0479 (6)	0.0646 (7)	0.0765 (7)	−0.0023 (5)	−0.0043 (5)	0.0267 (5)
O3	0.0323 (5)	0.0663 (7)	0.0823 (8)	0.0024 (4)	−0.0006 (5)	0.0222 (5)

C4	0.0360 (7)	0.0545 (8)	0.0486 (7)	−0.0071 (6)	0.0021 (6)	0.0001 (6)
C5	0.0399 (7)	0.0495 (8)	0.0457 (7)	−0.0016 (6)	−0.0029 (6)	0.0047 (6)
C6	0.0301 (6)	0.0507 (8)	0.0477 (7)	−0.0009 (5)	−0.0022 (5)	0.0019 (6)
N1	0.0345 (7)	0.0733 (10)	0.0982 (11)	−0.0005 (6)	0.0002 (7)	0.0032 (8)
C3	0.0312 (6)	0.0511 (8)	0.0530 (8)	−0.0004 (6)	0.0002 (6)	−0.0065 (6)
C2	0.0361 (7)	0.0470 (7)	0.0554 (8)	0.0031 (6)	−0.0024 (6)	0.0023 (6)
C1	0.0351 (7)	0.0452 (7)	0.0477 (7)	−0.0027 (5)	0.0014 (5)	0.0008 (6)
C9	0.0360 (8)	0.0532 (8)	0.0668 (9)	−0.0028 (6)	0.0003 (6)	−0.0027 (7)
C8	0.0546 (9)	0.0538 (9)	0.0793 (11)	−0.0029 (7)	0.0051 (8)	0.0182 (8)
C7	0.0715 (11)	0.0710 (11)	0.0914 (13)	−0.0040 (9)	0.0082 (10)	0.0354 (10)

Geometric parameters (Å, °)

O2—C1	1.3663 (15)	N1—C9	1.1373 (17)
O2—C8	1.4223 (18)	C3—C2	1.3924 (19)
O1—C5	1.3645 (17)	C3—C9	1.4381 (18)
O1—C7	1.4217 (19)	C2—C1	1.3788 (18)
O3—C6	1.3536 (16)	C2—H2	0.95
O3—H3	0.84	C8—H8A	0.98
C4—C5	1.3806 (19)	C8—H8B	0.98
C4—C3	1.3883 (19)	C8—H8C	0.98
C4—H4	0.95	C7—H7A	0.98
C5—C6	1.3972 (19)	C7—H7B	0.98
C6—C1	1.3941 (19)	C7—H7C	0.98
C1—O2—C8	117.64 (11)	C3—C2—H2	120.7
C5—O1—C7	117.37 (12)	O2—C1—C2	125.45 (12)
C6—O3—H3	109.5	O2—C1—C6	114.01 (11)
C5—C4—C3	119.00 (13)	C2—C1—C6	120.53 (12)
C5—C4—H4	120.5	N1—C9—C3	179.62 (19)
C3—C4—H4	120.5	O2—C8—H8A	109.5
O1—C5—C4	124.84 (12)	O2—C8—H8B	109.5
O1—C5—C6	115.10 (12)	H8A—C8—H8B	109.5
C4—C5—C6	120.05 (13)	O2—C8—H8C	109.5
O3—C6—C1	122.08 (12)	H8A—C8—H8C	109.5
O3—C6—C5	117.94 (12)	H8B—C8—H8C	109.5
C1—C6—C5	119.97 (12)	O1—C7—H7A	109.5
C4—C3—C2	121.86 (12)	O1—C7—H7B	109.5
C4—C3—C9	118.96 (13)	H7A—C7—H7B	109.5
C2—C3—C9	119.17 (13)	O1—C7—H7C	109.5
C1—C2—C3	118.58 (13)	H7A—C7—H7C	109.5
C1—C2—H2	120.7	H7B—C7—H7C	109.5
C7—O1—C5—C4	4.3 (2)	C4—C3—C2—C1	0.5 (2)
C7—O1—C5—C6	−175.11 (14)	C9—C3—C2—C1	−178.88 (13)
C3—C4—C5—O1	−179.15 (12)	C8—O2—C1—C2	−1.2 (2)
C3—C4—C5—C6	0.2 (2)	C8—O2—C1—C6	177.66 (13)
O1—C5—C6—O3	1.36 (18)	C3—C2—C1—O2	179.30 (13)
C4—C5—C6—O3	−178.10 (13)	C3—C2—C1—C6	0.5 (2)
O1—C5—C6—C1	−179.83 (12)	O3—C6—C1—O2	−1.29 (19)
C4—C5—C6—C1	0.7 (2)	C5—C6—C1—O2	179.95 (12)

C5—C4—C3—C2	−0.9 (2)	O3—C6—C1—C2	177.69 (13)
C5—C4—C3—C9	178.54 (13)	C5—C6—C1—C2	−1.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	0.84	2.22	2.6710 (14)	114
O3—H3···N1^{i}	0.84	2.15	2.8286 (17)	138

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

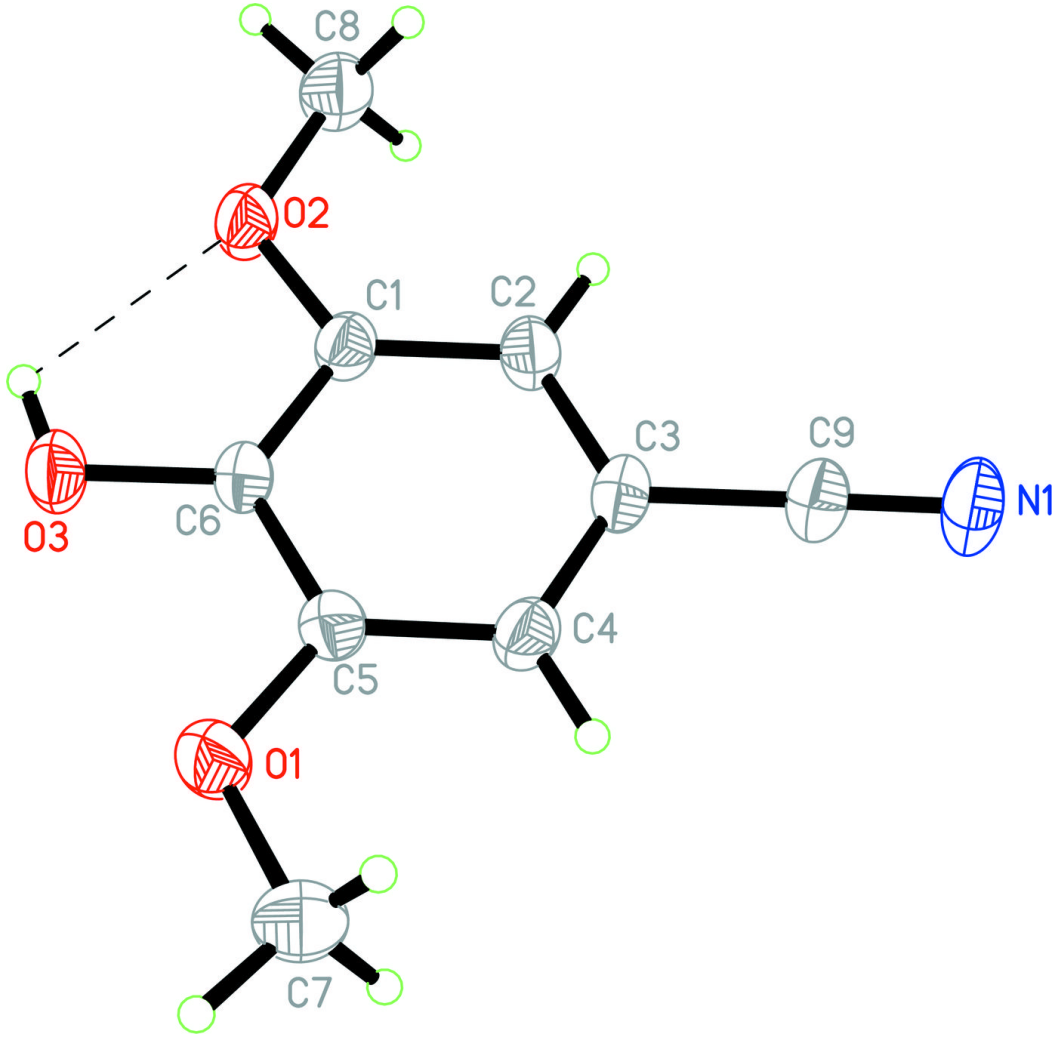


Fig. 2