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Dimethyl 3,4-dihydroxy-1*H*-pyrrole-2,5-dicarboxylate

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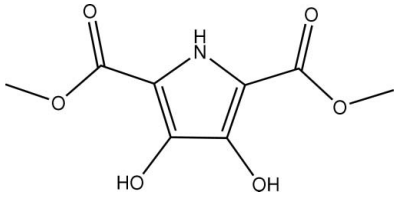
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.108; data-to-parameter ratio = 11.3.

The molecule of the title compound, $C_8H_9NO_6$, is nearly planar (r.m.s. deviation for all non-H atoms = 0.024 Å). The carbonyl O atoms and the pyrrole N atom are in a synperiplanar conformation. Torsion angles of −177.2 (2) and −178.0 (2)° are formed between the methyl C atoms and the pivot C atoms of the pyrrole ring. A bifurcated hydrogen bond of one of the hydroxyl H atoms is observed. The crystal packing is characterized by planes of molecules parallel to (102).

Related literature

For related structures, see: Furstner *et al.* (2002); Tomilov *et al.* (2006).



Experimental

Crystal data

$C_8H_9NO_6$
M_r = 215.16
Monoclinic, $P2_1/c$
a = 10.1782 (10) Å
b = 12.4456 (8) Å
c = 7.1922 (8) Å
β = 96.080 (9)°
V = 905.94 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 173 (2) K
0.54 × 0.42 × 0.40 mm

Data collection

STOE IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.929, T_{max} = 0.947
12133 measured reflections
1697 independent reflections
1438 reflections with $I > 2\sigma(I)$
R_{int} = 0.100

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.108
S = 1.02
1697 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O41^{i}	0.91 (2)	1.90 (3)	2.8019 (18)	170 (2)
O31—H31···O51^{ii}	0.95 (3)	1.91 (3)	2.7815 (17)	150 (3)
O41—H41···O52	0.90 (3)	2.07 (3)	2.7641 (16)	134 (2)
O41—H41···O21^{ii}	0.90 (3)	2.17 (3)	2.8672 (16)	134 (2)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

We thank Professor Dr E. Egert for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2487).

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supplementary materials

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Dimethyl 3,4-dihydroxy-1*H*-pyrrole-2,5-dicarboxylate

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Comment

Dimethyl 3,4-dihydroxy-1*H*-pyrrole-2,5-dicarboxylate is a nearly planar molecule (r.m.s. deviation for all non-H atoms 0.024 Å). The carbonyl O atoms and the pyrrole N atom are in a synperiplanar conformation [torsion angles: N1—C2—C21—O21: 0.8 (2)°, N1—C5—C51—O51: 3.1 (3)°]. Torsion angles of −177.2 (2)° and −178.0 (2)° are formed between the methyl C atoms and the pivot C atoms of the pyrrole ring. The H41 atom is involved in a bifurcated hydrogen bond: intramolecular to the ether O atom O52 and intermolecular to the carbonyl O atom O21. Another O—H···O and N—H···O hydrogen bonds stabilize the catemeric structure. The crystal packing is characterized by planes of molecules parallel to (102).

Experimental

Single crystals of title compound were obtained by recrystallization of the commercially available dimethyl 3,4-dihydroxy-1*H*-pyrrole-2,5-dicarboxylate from dimethylsulfoxide at 323 K.

Refinement

All H atoms were initially located by difference Fourier synthesis. Subsequently the positions of those bonded to C atoms were refined with fixed individual displacement parameters [U(H) = 1.5 U_{eq}] using a riding model with C—H= 0.98 Å. The H atoms bonded to N and O were refined isotropically.

Figures

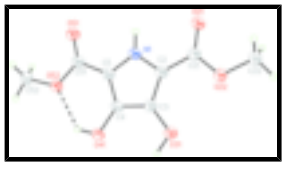

Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. The hydrogen bond is indicated by a dashed line.

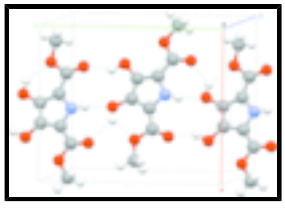

Fig. 2. Partial packing diagram of the title compound. Hydrogen bonds shown as dashed lines.

Dimethyl 3,4-dihydroxy-1*H*-pyrrole-2,5-dicarboxylate

Crystal data

$C_8H_9NO_6$	F_{000} = 448
M_r = 215.16	D_x = 1.578 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 11682 reflections
a = 10.1782 (10) Å	θ = 3.3–25.9º
b = 12.4456 (8) Å	μ = 0.14 mm^{-1}
c = 7.1922 (8) Å	T = 173 (2) K
β = 96.080 (9)º	Block, yellow
V = 905.94 (15) Å^3	0.54 × 0.42 × 0.40 mm
Z = 4	

Data collection

STOE IPDS II two-circle-diffractometer	1697 independent reflections
Radiation source: fine-focus sealed tube	1438 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.100
T = 173(2) K	θ_{max} = 25.6º
ω scans	θ_{min} = 3.3º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −12→12
T_{min} = 0.929, T_{max} = 0.947	k = −14→15
12133 measured reflections	l = −8→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.0213P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
1697 reflections	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
150 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. ;

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}
N1	0.45309 (13)	0.36410 (11)	0.28286 (19)	0.0289 (3)
H1	0.468 (2)	0.292 (2)	0.283 (3)	0.049 (6)*
C2	0.34066 (15)	0.41261 (12)	0.3319 (2)	0.0266 (4)
C3	0.35706 (15)	0.52401 (12)	0.3153 (2)	0.0267 (3)
C4	0.48165 (15)	0.54066 (12)	0.2528 (2)	0.0263 (4)
C5	0.53931 (15)	0.44033 (13)	0.2341 (2)	0.0271 (4)
C21	0.23610 (15)	0.34484 (12)	0.3917 (2)	0.0265 (3)
C22	0.02535 (17)	0.33844 (15)	0.5018 (3)	0.0363 (4)
H22A	−0.0112	0.2885	0.4044	0.054*
H22B	−0.0445	0.3868	0.5351	0.054*
H22C	0.0609	0.2978	0.6126	0.054*
O21	0.24307 (11)	0.24696 (8)	0.40263 (18)	0.0342 (3)
O22	0.13041 (11)	0.40103 (9)	0.43276 (17)	0.0321 (3)
O31	0.26669 (11)	0.59955 (9)	0.35335 (18)	0.0339 (3)
H31	0.303 (3)	0.669 (2)	0.335 (4)	0.074 (8)*
O41	0.53217 (12)	0.63930 (9)	0.21922 (18)	0.0335 (3)
H41	0.614 (3)	0.633 (2)	0.185 (4)	0.060 (7)*
C51	0.66822 (16)	0.41271 (12)	0.1787 (2)	0.0279 (4)
C52	0.86421 (16)	0.49092 (15)	0.0833 (3)	0.0353 (4)
H52A	0.9264	0.4646	0.1866	0.053*
H52B	0.8945	0.5606	0.0408	0.053*
H52C	0.8595	0.4394	−0.0203	0.053*
O51	0.71173 (12)	0.32209 (10)	0.16192 (19)	0.0381 (3)
O52	0.73483 (10)	0.50292 (9)	0.14626 (16)	0.0313 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0264 (7)	0.0172 (7)	0.0447 (8)	−0.0005 (5)	0.0106 (6)	−0.0007 (5)
C2	0.0250 (7)	0.0200 (8)	0.0357 (8)	0.0001 (6)	0.0074 (6)	−0.0008 (6)
C3	0.0258 (7)	0.0190 (7)	0.0359 (8)	0.0012 (6)	0.0064 (6)	−0.0003 (6)
C4	0.0280 (8)	0.0175 (8)	0.0342 (8)	−0.0016 (5)	0.0068 (6)	0.0005 (6)
C5	0.0262 (7)	0.0199 (7)	0.0364 (8)	−0.0007 (6)	0.0085 (6)	−0.0006 (6)
C21	0.0247 (8)	0.0227 (8)	0.0325 (8)	0.0001 (6)	0.0052 (6)	−0.0010 (6)
C22	0.0284 (8)	0.0327 (9)	0.0504 (10)	−0.0007 (7)	0.0157 (7)	0.0042 (7)

O21	0.0311 (6)	0.0195 (6)	0.0541 (7)	−0.0007 (5)	0.0139 (5)	0.0007 (5)
O22	0.0262 (6)	0.0235 (6)	0.0485 (7)	0.0004 (4)	0.0131 (5)	0.0019 (5)
O31	0.0286 (6)	0.0186 (6)	0.0568 (8)	0.0014 (4)	0.0152 (5)	−0.0005 (5)
O41	0.0296 (6)	0.0168 (6)	0.0566 (7)	−0.0015 (4)	0.0165 (5)	0.0012 (5)
C51	0.0275 (7)	0.0199 (8)	0.0372 (8)	−0.0003 (6)	0.0070 (6)	−0.0010 (6)
C52	0.0239 (8)	0.0329 (9)	0.0509 (10)	−0.0009 (6)	0.0121 (7)	0.0023 (7)
O51	0.0334 (6)	0.0219 (6)	0.0615 (8)	0.0030 (5)	0.0161 (5)	−0.0013 (5)
O52	0.0257 (6)	0.0216 (6)	0.0483 (7)	−0.0008 (4)	0.0126 (5)	0.0009 (4)

Geometric parameters (Å, °)

N1—C5	1.363 (2)	C22—O22	1.4523 (19)
N1—C2	1.373 (2)	C22—H22A	0.9800
N1—H1	0.91 (2)	C22—H22B	0.9800
C2—C3	1.403 (2)	C22—H22C	0.9800
C2—C21	1.458 (2)	O31—H31	0.95 (3)
C3—O31	1.3632 (19)	O41—H41	0.90 (3)
C3—C4	1.405 (2)	C51—O51	1.222 (2)
C4—O41	1.3623 (18)	C51—O52	1.3446 (19)
C4—C5	1.392 (2)	C52—O52	1.4450 (19)
C5—C51	1.452 (2)	C52—H52A	0.9800
C21—O21	1.2222 (19)	C52—H52B	0.9800
C21—O22	1.3419 (19)	C52—H52C	0.9800
C5—N1—C2	109.69 (13)	O22—C22—H22B	109.5
C5—N1—H1	124.8 (16)	H22A—C22—H22B	109.5
C2—N1—H1	125.5 (16)	O22—C22—H22C	109.5
N1—C2—C3	107.62 (13)	H22A—C22—H22C	109.5
N1—C2—C21	118.41 (14)	H22B—C22—H22C	109.5
C3—C2—C21	133.94 (14)	C21—O22—C22	115.60 (13)
O31—C3—C2	125.13 (14)	C3—O31—H31	108.3 (19)
O31—C3—C4	127.85 (14)	C4—O41—H41	110.2 (17)
C2—C3—C4	107.02 (13)	O51—C51—O52	123.96 (15)
O41—C4—C5	128.33 (14)	O51—C51—C5	126.35 (14)
O41—C4—C3	124.06 (13)	O52—C51—C5	109.68 (13)
C5—C4—C3	107.61 (13)	O52—C52—H52A	109.5
N1—C5—C4	108.06 (14)	O52—C52—H52B	109.5
N1—C5—C51	122.06 (14)	H52A—C52—H52B	109.5
C4—C5—C51	129.87 (14)	O52—C52—H52C	109.5
O21—C21—O22	123.21 (14)	H52A—C52—H52C	109.5
O21—C21—C2	123.80 (14)	H52B—C52—H52C	109.5
O22—C21—C2	112.99 (13)	C51—O52—C52	117.46 (12)
O22—C22—H22A	109.5		
C5—N1—C2—C3	−0.61 (18)	O41—C4—C5—C51	1.5 (3)
C5—N1—C2—C21	−179.10 (14)	C3—C4—C5—C51	−178.30 (16)
N1—C2—C3—O31	−179.49 (14)	N1—C2—C21—O21	0.8 (2)
C21—C2—C3—O31	−1.3 (3)	C3—C2—C21—O21	−177.24 (17)
N1—C2—C3—C4	0.72 (18)	N1—C2—C21—O22	−178.86 (13)
C21—C2—C3—C4	178.87 (17)	C3—C2—C21—O22	3.1 (3)
O31—C3—C4—O41	−0.2 (3)	O21—C21—O22—C22	3.2 (2)

C2—C3—C4—O41	179.63 (14)	C2—C21—O22—C22	−177.18 (14)
O31—C3—C4—C5	179.65 (15)	N1—C5—C51—O51	3.1 (3)
C2—C3—C4—C5	−0.56 (18)	C4—C5—C51—O51	−178.56 (17)
C2—N1—C5—C4	0.26 (18)	N1—C5—C51—O52	−177.28 (14)
C2—N1—C5—C51	178.89 (15)	C4—C5—C51—O52	1.0 (2)
O41—C4—C5—N1	180.00 (15)	O51—C51—O52—C52	1.6 (2)
C3—C4—C5—N1	0.19 (18)	C5—C51—O52—C52	−177.98 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O41^{i}	0.91 (2)	1.90 (3)	2.8019 (18)	170 (2)
O31—H31···O51^{ii}	0.95 (3)	1.91 (3)	2.7815 (17)	150 (3)
O41—H41···O52	0.90 (3)	2.07 (3)	2.7641 (16)	134 (2)
O41—H41···O21^{ii}	0.90 (3)	2.17 (3)	2.8672 (16)	134 (2)

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

Fig. 1

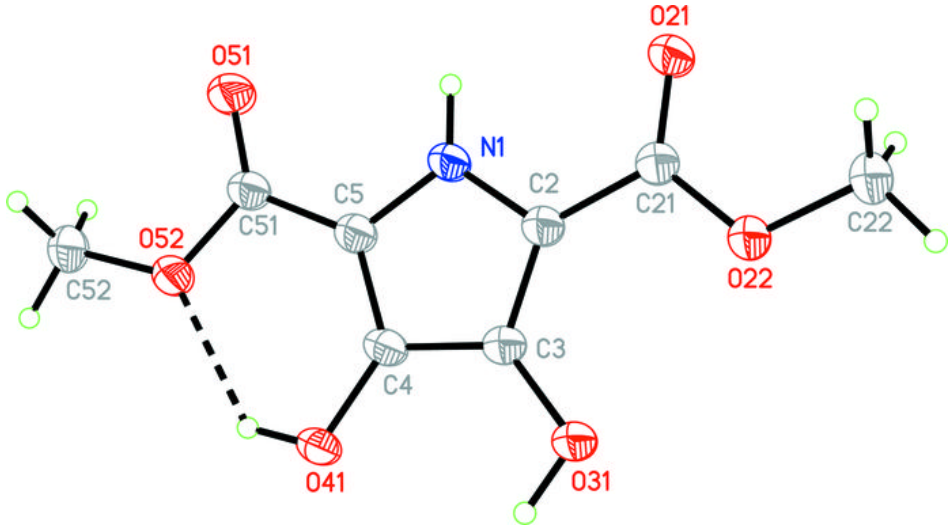

Fig. 2