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2-(2,4-Dinitrophenyl)-1-(pyridin-4-yl)ethanol monohydrate

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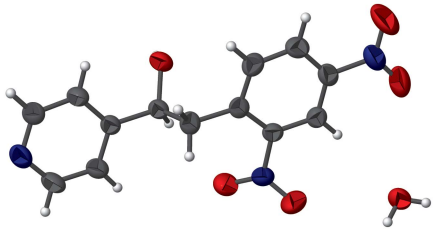
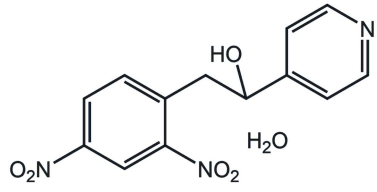
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{11}N_3O_5\cdot H_2O$, the dihedral angle between the aromatic rings is 9.60 (7)° and the chain linking the rings has an *anti* conformation with a torsion angle of −178.28 (12)°. In the crystal, the components are linked by O—H···O and O—H···N hydrogen bonds, generating (010) sheets.

3D view



Chemical scheme



Structure description

Pyridine derivatives have been widely used in biochemistry. Pyridine salts, for example, are well known for their photoactivity and exhibit potential for targetting mitochondria and in the photodynamic therapy of diseases (Wang *et al.*, 2020; Li *et al.*,2017). The Knoevenagel reaction is one of the most efficient methods of constructing pyridine-containing organic semiconductors though a two-step process: (1) format the hydroxyl-containing intermediates; (2) obtain products by a dehydration reaction. We report here the of the title compound, $C_{13}H_{11}N_3O_5\cdot H_2O$ (Fig. 1), a hydroxyl-containing intermediate. The dihedral angle between the benzene and pyridine rings is 9.60 (7)° and the chain linking the rings has an *anti* conformation with a C5—C11—C18—C6 torsion angle of −178.28 (12)°. In the crystal, the components are linked by O—H···O and O—H···N hydrogen bonds (Table 1), generating (010) sheets.

Synthesis and crystallization

1-Methyl-2,4-dinitro-benzene (0.546 g, 3.00 mmol) and pyridine-4-carbaldehyde (0.321 g, 3.00 mmol) were dissolved in dimethyl sulfoxide (50 ml). The mixture was heated to 80°C for 4 h, then cooled to room temperature and poured into water. The precipitate was collected by filtration and dried to obtain a yellow solid (0.712 g, 2.4 mol). Yellow crystals suitable for X-ray analysis were obtained by recrystallization from ethanol solution.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.80 (2)	2.01 (2)	2.801 (3)	173 (2)
O1—H1B···N10^{ii}	0.86 (3)	2.00 (3)	2.841 (3)	166.1 (19)
O2—H2···O1^{iii}	0.82	1.85	2.666 (3)	175
C17—H17···O20^{iv}	0.93	2.53	3.226 (4)	131

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

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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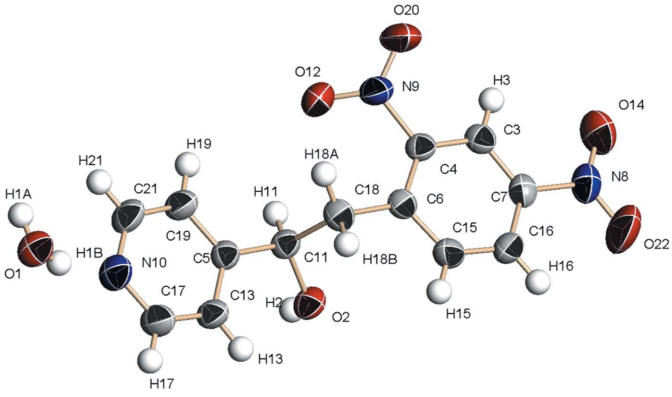

Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{11}N_3O_5·H_2O$
M_r	307.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	8.639 (8), 20.045 (12), 8.116 (5)
β (°)	101.326 (5)
V (Å^3)	1378.2 (17)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.12
Crystal size (mm)	0.19 × 0.18 × 0.17
Data collection	
Diffractometer	Bruker SMART CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.616, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10488, 2889, 2531
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.645
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.125, 1.03
No. of reflections	2889
No. of parameters	208
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.24

Computer programs: *SMART* and *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015) and *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

IUCrData (2021). **6**, x201640 [https://doi.org/10.1107/S2414314620016405]

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Crystal data

$C_{13}H_{11}N_3O_5·H_2O$
M_r = 307.26
Monoclinic, $P2_1/c$
a = 8.639 (8) Å
b = 20.045 (12) Å
c = 8.116 (5) Å
β = 101.326 (5)°
V = 1378.2 (17) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.481 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6924 reflections
θ = 2.6–27.1°
μ = 0.12 mm^{-1}
T = 296 K
Block, yellow
0.19 × 0.18 × 0.17 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.616, T_{max} = 0.746
10488 measured reflections
2889 independent reflections
2531 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.3°, θ_{min} = 2.0°
h = −11→11
k = −22→25
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.03
2889 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.4306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.11854 (12)	0.30340 (6)	0.04733 (14)	0.0475 (3)
H2	0.0856	0.3009	−0.0544	0.071*
C3	−0.03252 (17)	0.51894 (7)	0.25550 (18)	0.0418 (3)
H3	−0.0432	0.5644	0.2332	0.050*
C4	0.10032 (16)	0.48431 (7)	0.23286 (18)	0.0389 (3)
C5	0.40549 (16)	0.30336 (7)	0.08026 (17)	0.0383 (3)
C6	0.12367 (17)	0.41637 (7)	0.26901 (17)	0.0393 (3)
C7	−0.14831 (17)	0.48357 (8)	0.31235 (18)	0.0435 (3)
N8	−0.29271 (16)	0.51917 (8)	0.33273 (18)	0.0563 (4)
N9	0.21854 (15)	0.52431 (6)	0.16714 (18)	0.0500 (3)
N10	0.68428 (15)	0.23063 (7)	0.09129 (19)	0.0534 (4)
C11	0.25687 (16)	0.34380 (7)	0.07980 (18)	0.0394 (3)
H11	0.2482	0.3787	−0.0060	0.047*
O12	0.30318 (16)	0.49757 (7)	0.08429 (19)	0.0669 (4)
C13	0.41333 (17)	0.23595 (7)	0.1180 (2)	0.0451 (3)
H13	0.3254	0.2137	0.1408	0.054*
O14	−0.30398 (18)	0.57810 (8)	0.2980 (2)	0.0795 (4)
C15	0.0034 (2)	0.38446 (8)	0.3309 (2)	0.0488 (4)
H15	0.0154	0.3396	0.3594	0.059*
C16	−0.1328 (2)	0.41660 (8)	0.3516 (2)	0.0504 (4)
H16	−0.2119	0.3937	0.3910	0.060*
C17	0.55312 (19)	0.20209 (8)	0.1213 (2)	0.0529 (4)
H17	0.5560	0.1567	0.1459	0.064*
C18	0.26799 (18)	0.37632 (7)	0.25304 (19)	0.0438 (3)
H18A	0.2839	0.3417	0.3382	0.053*
H18B	0.3596	0.4054	0.2747	0.053*
C19	0.54071 (18)	0.33341 (8)	0.0472 (2)	0.0499 (4)
H19	0.5407	0.3785	0.0205	0.060*
O20	0.2244 (2)	0.58322 (7)	0.1986 (3)	0.1047 (7)
C21	0.67505 (19)	0.29564 (9)	0.0543 (2)	0.0547 (4)
H21	0.7647	0.3166	0.0320	0.066*
O22	−0.3925 (2)	0.48738 (9)	0.3855 (3)	0.1002 (6)
O1	−0.00579 (15)	0.69790 (7)	0.28295 (16)	0.0537 (3)
H1A	−0.044 (3)	0.7282 (11)	0.324 (3)	0.072 (7)*
H1B	0.094 (3)	0.7007 (10)	0.319 (3)	0.072 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0343 (5)	0.0568 (6)	0.0508 (6)	−0.0054 (4)	0.0066 (4)	−0.0012 (5)
C3	0.0400 (7)	0.0391 (7)	0.0445 (7)	0.0017 (6)	0.0040 (6)	−0.0014 (6)
C4	0.0356 (7)	0.0387 (7)	0.0413 (7)	−0.0043 (5)	0.0053 (5)	−0.0016 (6)
C5	0.0340 (7)	0.0411 (7)	0.0395 (7)	−0.0008 (5)	0.0064 (5)	−0.0012 (5)
C6	0.0403 (7)	0.0395 (7)	0.0377 (7)	0.0010 (6)	0.0062 (5)	−0.0019 (5)
C7	0.0380 (7)	0.0513 (8)	0.0412 (7)	0.0024 (6)	0.0075 (6)	−0.0072 (6)

N8	0.0428 (7)	0.0717 (10)	0.0559 (8)	0.0063 (7)	0.0134 (6)	−0.0074 (7)
N9	0.0424 (7)	0.0421 (7)	0.0661 (9)	−0.0068 (5)	0.0121 (6)	−0.0001 (6)
N10	0.0366 (7)	0.0576 (8)	0.0659 (9)	0.0065 (6)	0.0094 (6)	0.0050 (7)
C11	0.0337 (7)	0.0408 (7)	0.0433 (7)	0.0001 (5)	0.0065 (5)	0.0032 (6)
O12	0.0645 (8)	0.0615 (8)	0.0841 (9)	−0.0096 (6)	0.0378 (7)	−0.0020 (7)
C13	0.0352 (7)	0.0427 (8)	0.0577 (9)	−0.0029 (6)	0.0095 (6)	0.0028 (6)
O14	0.0669 (9)	0.0698 (9)	0.1059 (12)	0.0257 (7)	0.0271 (8)	0.0022 (8)
C15	0.0599 (9)	0.0382 (7)	0.0521 (8)	−0.0003 (7)	0.0200 (7)	0.0022 (6)
C16	0.0502 (9)	0.0520 (9)	0.0534 (9)	−0.0089 (7)	0.0212 (7)	−0.0031 (7)
C17	0.0424 (8)	0.0441 (8)	0.0718 (11)	0.0034 (6)	0.0100 (7)	0.0057 (7)
C18	0.0423 (8)	0.0433 (8)	0.0442 (8)	0.0061 (6)	0.0049 (6)	0.0004 (6)
C19	0.0407 (8)	0.0444 (8)	0.0661 (10)	−0.0025 (6)	0.0144 (7)	0.0063 (7)
O20	0.0977 (12)	0.0435 (7)	0.193 (2)	−0.0191 (7)	0.0773 (13)	−0.0134 (10)
C21	0.0345 (8)	0.0607 (10)	0.0708 (11)	−0.0036 (7)	0.0149 (7)	0.0058 (8)
O22	0.0645 (9)	0.1094 (13)	0.1428 (17)	0.0092 (9)	0.0599 (11)	0.0136 (12)
O1	0.0379 (6)	0.0607 (8)	0.0601 (7)	−0.0024 (5)	0.0034 (5)	−0.0086 (6)

Geometric parameters (Å, º)

O2—C11	1.4248 (19)	N10—C17	1.334 (2)
O2—H2	0.8200	N10—C21	1.336 (2)
C3—C7	1.378 (2)	C11—C18	1.536 (2)
C3—C4	1.384 (2)	C11—H11	0.9800
C3—H3	0.9300	C13—C17	1.381 (2)
C4—C6	1.400 (2)	C13—H13	0.9300
C4—N9	1.478 (2)	C15—C16	1.380 (2)
C5—C13	1.384 (2)	C15—H15	0.9300
C5—C19	1.387 (2)	C16—H16	0.9300
C5—C11	1.518 (2)	C17—H17	0.9300
C6—C15	1.395 (2)	C18—H18A	0.9700
C6—C18	1.510 (2)	C18—H18B	0.9700
C7—C16	1.380 (2)	C19—C21	1.377 (2)
C7—N8	1.474 (2)	C19—H19	0.9300
N8—O14	1.214 (2)	C21—H21	0.9300
N8—O22	1.216 (2)	O1—H1A	0.80 (2)
N9—O20	1.207 (2)	O1—H1B	0.86 (3)
N9—O12	1.2113 (19)		
C11—O2—H2	109.5	C5—C11—H11	109.3
C7—C3—C4	117.52 (14)	C18—C11—H11	109.3
C7—C3—H3	121.2	C17—C13—C5	119.26 (14)
C4—C3—H3	121.2	C17—C13—H13	120.4
C3—C4—C6	123.32 (13)	C5—C13—H13	120.4
C3—C4—N9	115.20 (13)	C16—C15—C6	122.89 (15)
C6—C4—N9	121.48 (13)	C16—C15—H15	118.6
C13—C5—C19	117.41 (14)	C6—C15—H15	118.6
C13—C5—C11	121.77 (13)	C15—C16—C7	118.20 (14)
C19—C5—C11	120.79 (14)	C15—C16—H16	120.9

C15—C6—C4	115.76 (13)	C7—C16—H16	120.9
C15—C6—C18	118.25 (14)	N10—C17—C13	123.82 (16)
C4—C6—C18	125.94 (13)	N10—C17—H17	118.1
C3—C7—C16	122.26 (14)	C13—C17—H17	118.1
C3—C7—N8	118.34 (15)	C6—C18—C11	113.58 (12)
C16—C7—N8	119.40 (14)	C6—C18—H18A	108.8
O14—N8—O22	124.18 (16)	C11—C18—H18A	108.8
O14—N8—C7	118.42 (15)	C6—C18—H18B	108.8
O22—N8—C7	117.39 (17)	C11—C18—H18B	108.8
O20—N9—O12	123.07 (15)	H18A—C18—H18B	107.7
O20—N9—C4	117.22 (14)	C21—C19—C5	119.22 (15)
O12—N9—C4	119.71 (13)	C21—C19—H19	120.4
C17—N10—C21	116.36 (14)	C5—C19—H19	120.4
O2—C11—C5	112.04 (13)	N10—C21—C19	123.92 (15)
O2—C11—C18	108.06 (12)	N10—C21—H21	118.0
C5—C11—C18	108.88 (11)	C19—C21—H21	118.0
O2—C11—H11	109.3	H1A—O1—H1B	106 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.80 (2)	2.01 (2)	2.801 (3)	173 (2)
O1—H1*B*···N10^{ii}	0.86 (3)	2.00 (3)	2.841 (3)	166.1 (19)
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