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(*E*)-4-Methoxy-3,5-dimethyl-2-[(3-nitrophenyl)-ethenyl]pyridine

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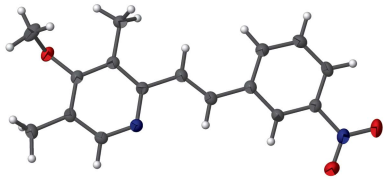
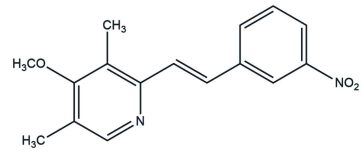
In the crystal of the title compound, $C_{16}H_{16}N_2O_3$, weak C—H···O hydrogen bonds involving the nitro group as acceptor form chains extending in the *b*-axis direction. The chains are arranged into layers by π–π stacking interactions along the *c*-axis direction between the substituted pyridine rings, separated by 3.624 (1) Å.

3D view



Chemical scheme



Structure description

Pyridine derivatives form one of the most important classes of heterocyclic compounds and their prevalence in natural products and pharmaceuticals as well as their potent bioactivity have created significant interest in academia and the pharmaceutical industry (Daly *et al.*, 1999). Indeed, pyridines have been studied for over a century as a result of their wide range of applications in many branches of chemistry, such as catalysis, drug design, molecular recognition, and materials science. Notably, many pyridine derivatives exhibit remarkable medicinal properties, including hypnotic and sedative, HIV antiviral (Harrison & Scott, 2005), or cholesterol and triglyceride regulator (Watts & Chan, 2008). Pyridines also form integral parts of more complex natural products, such as diploclidine and nakinadine (Kubota *et al.*, 2007).

In the crystal of the title compound (Fig. 1), C6—H6···O2(x, 1 + y, z) weak hydrogen bonds form chains extending in the *b*-axis direction (Table 1 and Fig. 2). These chains are arranged into layers (Fig. 3) by π–π-stacking interactions between the substituted pyridine rings [Fig. 4, centroid–centroid distance = 3.624 (1) Å, dihedral angle between rings = 6.73 (6)°].

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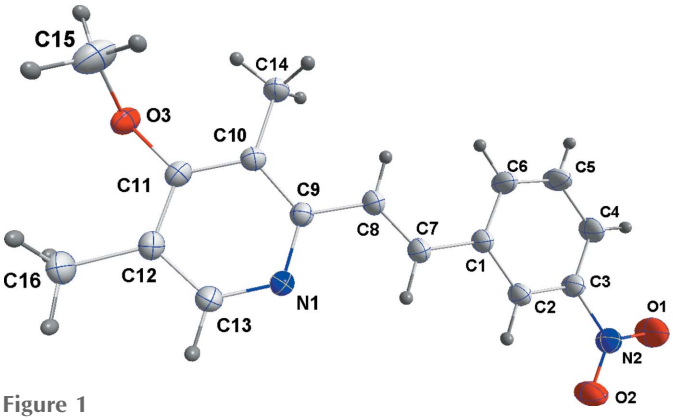

Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids for non-H atoms.

Synthesis and crystallization

To a solution of 5-methoxy-2-[((4-methoxy-3,5-dimethyl-pyridin-2-yl)methyl)sulfinyl]-1*H*-benzo[*d*]imidazole (0.5 g, 1.45 mmol), was added sodium methanolate (0.06 g, 1.45 mmol), and 3-nitrobenzaldehyde (0.44 g, 2.9 mmol). The mixture was refluxed in 15 ml of *N*,*N*-dimethylformamide for 48 h. The solution was then concentrated to dryness under reduced pressure and the obtained residue was chromatographed on a silica gel column with a mixture of ethyl acetate/hexane (90/100) as eluent. (*E*)-4-Methoxy-3,5-dimethyl-2-[(3-nitrophenyl)ethenyl]pyridine was obtained and recrystallized from ethanol solution, to afford the compound as crystals, with a yield of 40%.

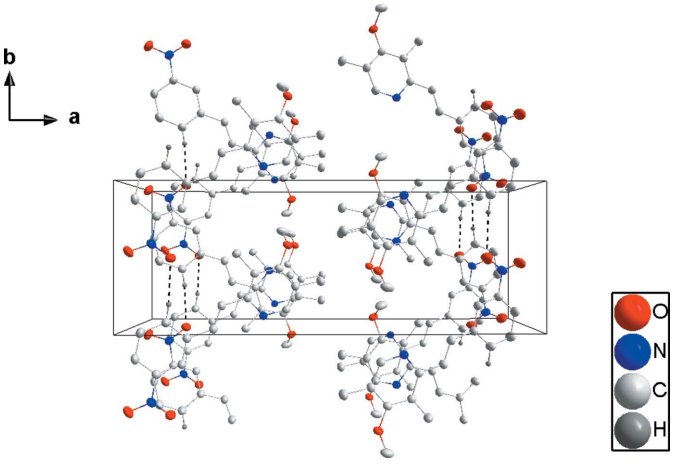

Figure 2
Packing viewed along the *c* axis with C—H···O hydrogen bonds shown as dotted lines.

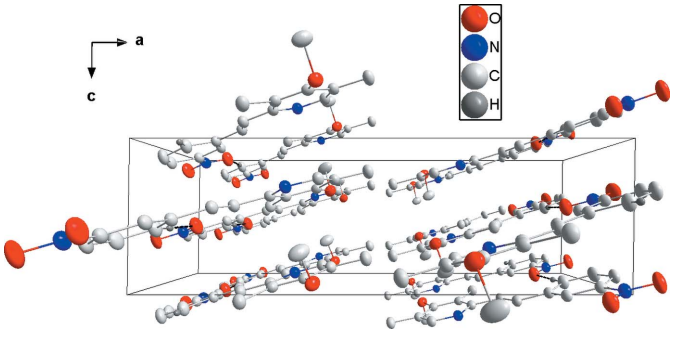

Figure 3
Packing viewed along the *b* axis emphasizing the layer structure.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.97 (2)	2.45 (2)	3.414 (2)	174 (1)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{16}N_2O_3$
M_r	284.31
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	23.3174 (7), 8.2979 (2), 7.2260 (2)
β (°)	90.899 (1)
V (Å^3)	1397.95 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.78
Crystal size (mm)	0.25 × 0.16 × 0.01
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.87, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10335, 2764, 2395
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.621
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.104, 1.05
No. of reflections	2764
No. of parameters	255
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.20

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

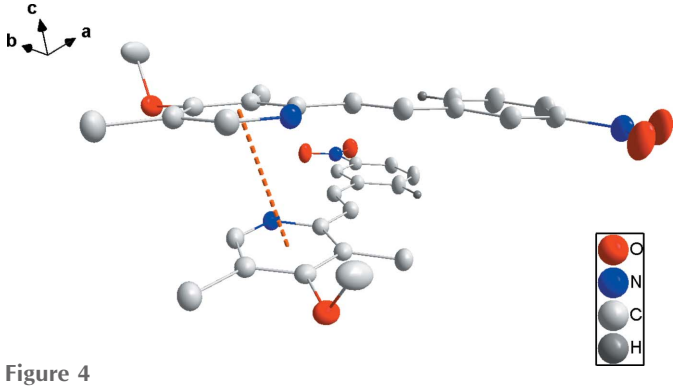

Figure 4
Detail of the π–π stacking between substituted pyridine rings at (x, y, z) (top) and (x, $\frac{3}{2}-y$, $-\frac{1}{2}+z$) (bottom).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The positions and isotropic factors for all H atoms were refined, since diffraction data were collected at low temperature.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161966 [https://doi.org/10.1107/S2414314616019660]

(*E*)-4-Methoxy-3,5-dimethyl-2-[(3-nitrophenyl)ethenyl]pyridine

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Crystal data

$C_{16}H_{16}N_2O_3$
M_r = 284.31
Monoclinic, $P2_1/c$
a = 23.3174 (7) Å
b = 8.2979 (2) Å
c = 7.2260 (2) Å
β = 90.899 (1)°
V = 1397.95 (7) Å^3
Z = 4
$F(000)$ = 600
D_x = 1.351 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7563 reflections
θ = 3.8–73.2°
μ = 0.78 mm^{-1}
T = 150 K
Plate, colourless
0.25 × 0.16 × 0.01 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.87, T_{max} = 0.99
10335 measured reflections
2764 independent reflections
2395 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 73.3°, θ_{min} = 3.8°
h = −28→28
k = −10→9
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.104
S = 1.05
2764 reflections
255 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.417P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0024 (3)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.95105 (5)	0.03936 (13)	0.34676 (19)	0.0474 (3)
O2	0.86494 (5)	−0.00204 (12)	0.44015 (18)	0.0428 (3)
O3	0.60432 (4)	1.08094 (11)	0.66857 (13)	0.0278 (2)
N1	0.66199 (4)	0.60746 (13)	0.65809 (15)	0.0247 (2)
N2	0.90441 (5)	0.08754 (14)	0.39837 (17)	0.0308 (3)
C1	0.83106 (5)	0.48038 (15)	0.48252 (18)	0.0247 (3)
C2	0.84183 (5)	0.31514 (15)	0.46961 (18)	0.0246 (3)
H2	0.8130 (7)	0.236 (2)	0.502 (2)	0.031 (4)*
C3	0.89481 (5)	0.26256 (15)	0.41017 (18)	0.0254 (3)
C4	0.93826 (6)	0.36645 (18)	0.3615 (2)	0.0316 (3)
H4	0.9740 (7)	0.326 (2)	0.324 (2)	0.036 (4)*
C5	0.92717 (6)	0.53084 (18)	0.3736 (2)	0.0366 (4)
H5	0.9565 (8)	0.605 (2)	0.343 (2)	0.044 (5)*
C6	0.87477 (6)	0.58745 (17)	0.4331 (2)	0.0327 (3)
H6	0.8689 (8)	0.703 (2)	0.434 (2)	0.043 (5)*
C7	0.77435 (5)	0.53257 (16)	0.54498 (19)	0.0260 (3)
H7	0.7471 (7)	0.4469 (19)	0.576 (2)	0.030 (4)*
C8	0.75595 (5)	0.68419 (16)	0.55851 (18)	0.0256 (3)
H8	0.7825 (7)	0.773 (2)	0.531 (2)	0.040 (5)*
C9	0.69782 (5)	0.72919 (15)	0.61500 (17)	0.0222 (3)
C10	0.68107 (5)	0.89220 (15)	0.61999 (17)	0.0226 (3)
C11	0.62485 (5)	0.92452 (15)	0.67111 (17)	0.0228 (3)
C12	0.58704 (5)	0.80139 (15)	0.71531 (17)	0.0240 (3)
C13	0.60879 (5)	0.64531 (15)	0.70639 (19)	0.0253 (3)
H13	0.5831 (6)	0.5569 (19)	0.736 (2)	0.024 (4)*
C14	0.72122 (6)	1.02562 (16)	0.5674 (2)	0.0282 (3)
H14A	0.7019 (11)	1.132 (3)	0.571 (3)	0.076 (7)*
H14B	0.7544 (8)	1.032 (2)	0.652 (3)	0.048 (5)*
H14C	0.7363 (9)	1.009 (2)	0.444 (3)	0.055 (6)*
C15	0.61979 (8)	1.17496 (18)	0.8279 (2)	0.0386 (4)
H15A	0.6201 (11)	1.282 (3)	0.789 (4)	0.084 (8)*
H15B	0.5878 (11)	1.164 (3)	0.920 (4)	0.084 (8)*
H15C	0.6548 (10)	1.142 (3)	0.881 (3)	0.060 (6)*
C16	0.52598 (6)	0.83394 (18)	0.7665 (2)	0.0319 (3)
H16A	0.5035 (8)	0.882 (2)	0.661 (3)	0.048 (5)*
H16B	0.5240 (8)	0.909 (2)	0.874 (3)	0.053 (5)*
H16C	0.5066 (8)	0.732 (2)	0.800 (2)	0.045 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0351 (6)	0.0367 (6)	0.0708 (8)	0.0121 (5)	0.0170 (5)	−0.0044 (6)
O2	0.0385 (6)	0.0243 (5)	0.0660 (8)	−0.0005 (4)	0.0125 (5)	0.0002 (5)
O3	0.0305 (5)	0.0214 (4)	0.0314 (5)	0.0054 (4)	−0.0024 (4)	−0.0019 (4)
N1	0.0239 (5)	0.0224 (5)	0.0279 (6)	0.0002 (4)	0.0024 (4)	−0.0011 (4)

N2	0.0292 (6)	0.0270 (6)	0.0362 (6)	0.0054 (5)	0.0045 (5)	−0.0013 (5)
C1	0.0226 (6)	0.0245 (6)	0.0271 (6)	0.0007 (5)	0.0026 (5)	−0.0006 (5)
C2	0.0220 (6)	0.0242 (6)	0.0278 (6)	−0.0008 (5)	0.0039 (5)	0.0001 (5)
C3	0.0249 (6)	0.0230 (6)	0.0283 (6)	0.0019 (5)	0.0031 (5)	−0.0014 (5)
C4	0.0216 (6)	0.0335 (7)	0.0399 (8)	0.0014 (5)	0.0082 (5)	−0.0031 (6)
C5	0.0274 (7)	0.0300 (7)	0.0528 (9)	−0.0049 (6)	0.0114 (6)	−0.0008 (7)
C6	0.0284 (6)	0.0241 (6)	0.0457 (8)	−0.0006 (5)	0.0080 (6)	−0.0004 (6)
C7	0.0227 (6)	0.0261 (6)	0.0292 (7)	0.0002 (5)	0.0031 (5)	−0.0005 (5)
C8	0.0225 (6)	0.0258 (6)	0.0285 (7)	0.0004 (5)	0.0032 (5)	−0.0004 (5)
C9	0.0225 (6)	0.0221 (6)	0.0221 (6)	0.0001 (5)	0.0009 (5)	−0.0005 (5)
C10	0.0238 (6)	0.0224 (6)	0.0217 (6)	−0.0005 (5)	−0.0005 (5)	−0.0001 (5)
C11	0.0252 (6)	0.0213 (6)	0.0218 (6)	0.0029 (5)	−0.0013 (5)	−0.0010 (5)
C12	0.0226 (6)	0.0256 (6)	0.0237 (6)	0.0023 (5)	0.0018 (5)	−0.0008 (5)
C13	0.0233 (6)	0.0237 (6)	0.0291 (7)	−0.0019 (5)	0.0024 (5)	−0.0003 (5)
C14	0.0263 (6)	0.0232 (6)	0.0352 (7)	−0.0017 (5)	0.0020 (6)	0.0023 (6)
C15	0.0518 (9)	0.0255 (7)	0.0382 (8)	0.0090 (6)	−0.0106 (7)	−0.0078 (6)
C16	0.0242 (6)	0.0324 (7)	0.0393 (8)	0.0024 (5)	0.0059 (6)	−0.0007 (6)

Geometric parameters (Å, º)

O1—N2	1.2224 (15)	C7—H7	0.982 (16)
O2—N2	1.2246 (16)	C8—C9	1.4699 (17)
O3—C11	1.3835 (14)	C8—H8	0.982 (18)
O3—C15	1.4320 (17)	C9—C10	1.4084 (17)
N1—C13	1.3316 (16)	C10—C11	1.3936 (17)
N1—C9	1.3503 (16)	C10—C14	1.5026 (17)
N2—C3	1.4722 (17)	C11—C12	1.3900 (18)
C1—C2	1.3973 (17)	C12—C13	1.3928 (17)
C1—C6	1.4025 (18)	C12—C16	1.5011 (17)
C1—C7	1.4693 (17)	C13—H13	0.972 (15)
C2—C3	1.3848 (17)	C14—H14A	0.99 (2)
C2—H2	0.968 (17)	C14—H14B	0.98 (2)
C3—C4	1.3802 (19)	C14—H14C	0.97 (2)
C4—C5	1.391 (2)	C15—H15A	0.93 (3)
C4—H4	0.942 (17)	C15—H15B	1.01 (3)
C5—C6	1.3838 (19)	C15—H15C	0.94 (2)
C5—H5	0.949 (19)	C16—H16A	1.00 (2)
C6—H6	0.965 (19)	C16—H16B	1.00 (2)
C7—C8	1.3333 (18)	C16—H16C	0.99 (2)
C11—O3—C15	114.69 (10)	C10—C9—C8	120.55 (11)
C13—N1—C9	117.79 (11)	C11—C10—C9	117.00 (11)
O1—N2—O2	123.54 (12)	C11—C10—C14	121.17 (11)
O1—N2—C3	118.50 (11)	C9—C10—C14	121.81 (11)
O2—N2—C3	117.96 (11)	O3—C11—C12	118.16 (11)
C2—C1—C6	118.20 (12)	O3—C11—C10	120.26 (11)
C2—C1—C7	118.25 (11)	C12—C11—C10	121.48 (11)
C6—C1—C7	123.55 (12)	C11—C12—C13	116.11 (11)

C3—C2—C1	119.47 (12)	C11—C12—C16	122.13 (11)
C3—C2—H2	119.2 (9)	C13—C12—C16	121.75 (12)
C1—C2—H2	121.4 (9)	N1—C13—C12	124.95 (12)
C4—C3—C2	122.98 (12)	N1—C13—H13	117.3 (9)
C4—C3—N2	119.24 (11)	C12—C13—H13	117.7 (9)
C2—C3—N2	117.78 (11)	C10—C14—H14A	111.2 (14)
C3—C4—C5	117.28 (12)	C10—C14—H14B	111.7 (11)
C3—C4—H4	120.6 (10)	H14A—C14—H14B	107.0 (18)
C5—C4—H4	122.1 (10)	C10—C14—H14C	111.5 (12)
C6—C5—C4	121.22 (13)	H14A—C14—H14C	108.9 (18)
C6—C5—H5	119.8 (11)	H14B—C14—H14C	106.4 (16)
C4—C5—H5	118.9 (11)	O3—C15—H15A	106.2 (16)
C5—C6—C1	120.85 (13)	O3—C15—H15B	107.5 (15)
C5—C6—H6	117.7 (10)	H15A—C15—H15B	107 (2)
C1—C6—H6	121.4 (10)	O3—C15—H15C	112.3 (13)
C8—C7—C1	126.36 (12)	H15A—C15—H15C	113 (2)
C8—C7—H7	117.1 (9)	H15B—C15—H15C	110.7 (19)
C1—C7—H7	116.5 (9)	C12—C16—H16A	111.7 (11)
C7—C8—C9	124.01 (12)	C12—C16—H16B	111.1 (11)
C7—C8—H8	119.0 (10)	H16A—C16—H16B	108.4 (16)
C9—C8—H8	117.0 (10)	C12—C16—H16C	110.3 (11)
N1—C9—C10	122.67 (11)	H16A—C16—H16C	107.0 (15)
N1—C9—C8	116.77 (11)	H16B—C16—H16C	108.2 (15)

C6—C1—C2—C3	0.4 (2)	C7—C8—C9—N1	1.2 (2)
C7—C1—C2—C3	179.66 (12)	C7—C8—C9—C10	−177.88 (13)
C1—C2—C3—C4	−0.3 (2)	N1—C9—C10—C11	−0.35 (18)
C1—C2—C3—N2	−179.96 (12)	C8—C9—C10—C11	178.63 (11)
O1—N2—C3—C4	0.5 (2)	N1—C9—C10—C14	−178.60 (12)
O2—N2—C3—C4	−179.11 (13)	C8—C9—C10—C14	0.38 (19)
O1—N2—C3—C2	−179.74 (13)	C15—O3—C11—C12	102.35 (15)
O2—N2—C3—C2	0.61 (19)	C15—O3—C11—C10	−81.30 (16)
C2—C3—C4—C5	−0.1 (2)	C9—C10—C11—O3	−176.17 (11)
N2—C3—C4—C5	179.58 (13)	C14—C10—C11—O3	2.08 (18)
C3—C4—C5—C6	0.4 (2)	C9—C10—C11—C12	0.05 (18)
C4—C5—C6—C1	−0.2 (3)	C14—C10—C11—C12	178.30 (12)
C2—C1—C6—C5	−0.2 (2)	O3—C11—C12—C13	176.66 (11)
C7—C1—C6—C5	−179.39 (14)	C10—C11—C12—C13	0.36 (18)
C2—C1—C7—C8	−177.22 (14)	O3—C11—C12—C16	−2.31 (18)
C6—C1—C7—C8	2.0 (2)	C10—C11—C12—C16	−178.61 (12)
C1—C7—C8—C9	177.47 (12)	C9—N1—C13—C12	0.3 (2)
C13—N1—C9—C10	0.21 (19)	C11—C12—C13—N1	−0.5 (2)
C13—N1—C9—C8	−178.80 (11)	C16—C12—C13—N1	178.44 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C6—H6···O2^{i}	0.97 (2)	2.45 (2)	3.414 (2)	174 (1)

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