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(*Z*)-*N*-(2,6-Dimethylphenyl)-1-[(2-methoxyphenyl)-amino]methanimine oxide methanol monosolvate

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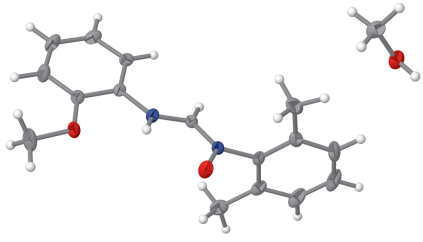
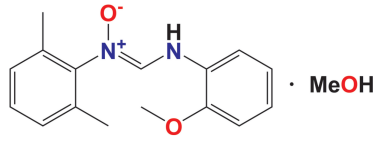
In the title solvate, $C_{16}H_{18}N_2O_2\cdot CH_4O$, the dihedral angles between the formamidine backbone and the pendant 2-methoxyphenyl and 2,6-dimethylphenyl groups are 14.84 (11) and 81.61 (12)°, respectively. In the crystal, the components are linked by C—H···O, O—H···O and C—H··· π hydrogen bonds, generating a supramolecular chain that extends along the crystallographic *a*-axis direction.

3D view



Chemical scheme



Structure description

The title compound is categorized in the class of formamidines (Cibian *et al.*, 2011, Zamisa *et al.*, 2021). The formamidine backbone features two nitrogen atoms that provide bidentate coordination sites, making them effective ligands in coordination chemistry (Oshovsky & Pinchuk, 2000). These metal complexes have demonstrated biological activities such as antioxidant (Oladipo *et al.*, 2020) and antibacterial, and significant catalytic activities in the microwave-assisted Suzuki–Miyaura cross-coupling of aryl bromides (Khormi *et al.*, 2019) and ring-opening polymerization reactions (Akpan *et al.*, 2016). As part of our studies in this area, we synthesized the title compound, $C_{16}H_{18}N_2O_2\cdot CH_4O$, (I), and determined its crystal structure.

The asymmetric unit of (I) consists of one substituted formamidine molecule and one methanol solvent molecule as illustrated in Fig. 1. The molecular structure reveals a non-coplanar arrangement between the formamidine backbone and its pendant phenyl rings with a dihedral angle of 14.84 (11)° between the plane of the C3/C4/C11–C14 2-methoxyphenyl group and the C6/N1/N2/O1 formamidine backbone. In contrast, the dihedral angle between the C7–C10/C15/C17 2,6-dimethylphenyl group and the backbone is 81.61 (12)°. The aromatic rings are nearly orthogonal, exhibiting a dihedral angle of 89.25 (5)°.

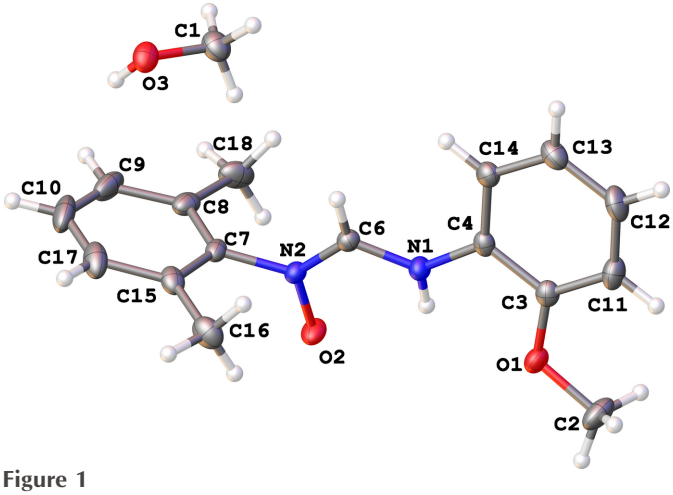


Figure 1
The molecular structure of (I) showing displacement ellipsoid at the 50% probability level.

In the extended structure of (I), C6—H6···O3 and O3—H3···O2 hydrogen bonds (Table 1) occur as depicted in Fig. 2. The former interaction involves the solvent O atom as acceptor. The latter hydrogen bond involves the methanol OH group as donor and the formamidine O atom as acceptor. Finally, a C—H···π interaction exists between the a methyl H atom of the solvent molecule and the centre of gravity of the dimethylphenyl ring (π_{DMP}). Together, these generate a one-dimensional supramolecular structure that extends along the crystallographic *a*-axis direction as shown in Fig. 2.

Synthesis and crystallization

The title compound was synthesized following the literature procedure (Munzeiwa *et al.*, 2018). The crude solid was then recrystallized from methanol solution to produce colourless blocks of (I) suitable for X-ray diffraction.

Refinement

Crystallographic data and structure refinement details are summarized in Table 2.

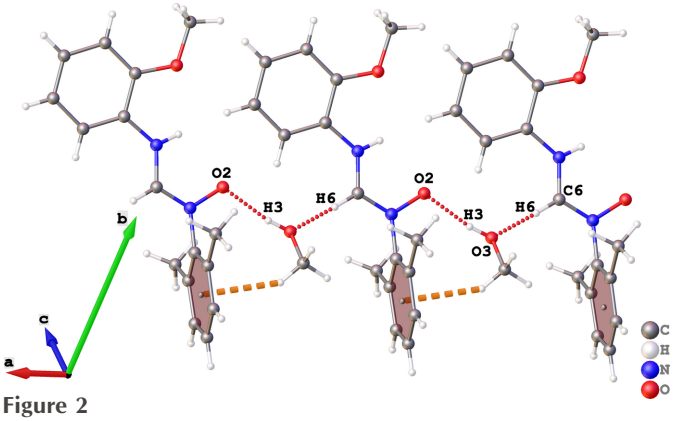


Figure 2
Representation of hydrogen bonds (dotted lines) in the crystal packing of (I).

Table 2
Hydrogen-bond geometry (Å, °).

π is the centroid of the C7–C10/C15/C17 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3^{i}	0.95	2.20	3.1134 (14)	160
O3—H3···O2^{ii}	0.84	1.85	2.6887 (12)	173
C1—H1C···π^{i}	0.98	2.66	3.5182 (16)	148

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

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{18}N_2O_2{\cdot}CH_4O$
M_r	302.36
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	7.3519 (5), 28.4173 (19), 7.8982 (5)
β (°)	94.481 (2)
V (Å^3)	1645.06 (19)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.28 × 0.23 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9891, 3421, 2988
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.635
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.095, 1.04
No. of reflections	3421
No. of parameters	204
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXL2018/3* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2024). **9**, x240989 [https://doi.org/10.1107/S2414314624009891]

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Crystal data

$C_{16}H_{18}N_2O_2·CH_4O$
M_r = 302.36
Monoclinic, $P2_1/n$
a = 7.3519 (5) Å
b = 28.4173 (19) Å
c = 7.8982 (5) Å
β = 94.481 (2)°
V = 1645.06 (19) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.221 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5449 reflections
θ = 2.9–26.8°
μ = 0.08 mm^{-1}
T = 296 K
Block, colourless
0.28 × 0.23 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
Graphite monochromator
φ and ω scans
9891 measured reflections
3421 independent reflections
2988 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.8°, θ_{min} = 2.7°
h = −5→9
k = −35→29
l = −10→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.095
S = 1.04
3421 reflections
204 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.7025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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}
O1	0.95025 (12)	0.23202 (3)	0.86095 (11)	0.0234 (2)
O2	0.68659 (11)	0.13040 (3)	0.54807 (11)	0.0253 (2)
N1	0.99859 (13)	0.15522 (4)	0.69356 (13)	0.0196 (2)
H1	0.891940	0.165508	0.721129	0.023*
N2	0.83888 (13)	0.11068 (3)	0.49709 (12)	0.0172 (2)
C2	0.9131 (2)	0.27445 (5)	0.9498 (2)	0.0360 (4)
H2A	0.929940	0.268838	1.072488	0.054*
H2B	0.787040	0.284311	0.919371	0.054*
H2C	0.996864	0.299228	0.918477	0.054*
C3	1.12632 (17)	0.21578 (4)	0.87530 (15)	0.0204 (3)
C4	1.15367 (16)	0.17426 (4)	0.78411 (14)	0.0183 (2)
C6	0.99662 (16)	0.12294 (4)	0.56934 (15)	0.0177 (2)
H6	1.106504	0.109551	0.535504	0.021*
C7	0.82105 (15)	0.07864 (4)	0.35527 (15)	0.0190 (3)
C8	0.77144 (16)	0.03222 (5)	0.38559 (18)	0.0252 (3)
C9	0.74859 (19)	0.00278 (5)	0.2439 (2)	0.0362 (4)
H9	0.714657	−0.029146	0.258361	0.043*
C10	0.7747 (2)	0.01942 (6)	0.0827 (2)	0.0407 (4)
H10	0.759648	−0.001348	−0.011605	0.049*
C11	1.27136 (19)	0.23666 (5)	0.96940 (17)	0.0273 (3)
H11	1.253247	0.264546	1.032260	0.033*
C12	1.44422 (19)	0.21639 (5)	0.97109 (17)	0.0299 (3)
H12	1.543994	0.230569	1.035715	0.036*
C13	1.47188 (17)	0.17590 (5)	0.87973 (16)	0.0265 (3)
H13	1.590666	0.162711	0.880300	0.032*
C14	1.32620 (17)	0.15440 (5)	0.78691 (15)	0.0214 (3)
H14	1.344791	0.126243	0.725748	0.026*
C15	0.84516 (16)	0.09678 (5)	0.19363 (16)	0.0241 (3)
C16	0.89151 (19)	0.14775 (5)	0.16957 (17)	0.0309 (3)
H16A	0.884152	0.155090	0.047995	0.046*
H16B	1.015574	0.153864	0.219298	0.046*
H16C	0.805035	0.167507	0.225731	0.046*
C17	0.82203 (19)	0.06565 (6)	0.05721 (18)	0.0347 (3)
H17	0.839093	0.076404	−0.054328	0.042*
C18	0.7448 (2)	0.01537 (5)	0.5626 (2)	0.0330 (3)
H18A	0.695056	−0.016641	0.557656	0.050*
H18B	0.659657	0.036362	0.615160	0.050*
H18C	0.862325	0.015410	0.630341	0.050*
O3	0.63925 (12)	−0.10123 (3)	0.59154 (11)	0.0256 (2)
H3	0.533296	−0.108674	0.553427	0.038*
C1	0.64434 (18)	−0.09642 (5)	0.76969 (17)	0.0300 (3)
H1A	0.765090	−0.085163	0.813127	0.045*
H1B	0.620195	−0.126986	0.820810	0.045*
H1C	0.551275	−0.073758	0.798853	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0260 (5)	0.0184 (5)	0.0257 (5)	0.0002 (4)	0.0019 (4)	−0.0049 (4)
O2	0.0162 (4)	0.0293 (5)	0.0304 (5)	0.0031 (4)	0.0026 (4)	−0.0106 (4)
N1	0.0170 (5)	0.0210 (5)	0.0205 (5)	−0.0002 (4)	0.0003 (4)	−0.0037 (4)
N2	0.0166 (5)	0.0168 (5)	0.0185 (5)	0.0007 (4)	0.0021 (4)	−0.0021 (4)
C2	0.0344 (8)	0.0243 (7)	0.0503 (9)	−0.0006 (6)	0.0086 (7)	−0.0147 (7)
C3	0.0235 (6)	0.0200 (6)	0.0180 (6)	−0.0033 (5)	0.0027 (5)	0.0017 (5)
C4	0.0216 (6)	0.0189 (6)	0.0144 (5)	−0.0040 (5)	0.0008 (4)	0.0021 (4)
C6	0.0183 (5)	0.0165 (6)	0.0185 (6)	−0.0002 (5)	0.0019 (4)	0.0003 (4)
C7	0.0127 (5)	0.0213 (6)	0.0229 (6)	0.0011 (4)	−0.0001 (4)	−0.0066 (5)
C8	0.0168 (6)	0.0213 (6)	0.0369 (7)	0.0019 (5)	−0.0015 (5)	−0.0044 (5)
C9	0.0250 (7)	0.0239 (7)	0.0588 (10)	0.0003 (6)	−0.0032 (6)	−0.0180 (7)
C10	0.0286 (7)	0.0509 (10)	0.0420 (9)	0.0018 (7)	−0.0005 (6)	−0.0304 (8)
C11	0.0319 (7)	0.0255 (7)	0.0246 (7)	−0.0082 (6)	0.0031 (5)	−0.0062 (5)
C12	0.0258 (7)	0.0395 (8)	0.0238 (7)	−0.0119 (6)	−0.0026 (5)	−0.0037 (6)
C13	0.0208 (6)	0.0369 (8)	0.0215 (6)	−0.0015 (5)	−0.0001 (5)	0.0021 (5)
C14	0.0231 (6)	0.0235 (6)	0.0173 (6)	−0.0001 (5)	0.0007 (5)	0.0010 (5)
C15	0.0148 (6)	0.0353 (7)	0.0220 (6)	−0.0001 (5)	0.0007 (5)	−0.0051 (5)
C16	0.0290 (7)	0.0396 (8)	0.0241 (7)	−0.0030 (6)	0.0024 (5)	0.0067 (6)
C17	0.0240 (7)	0.0541 (10)	0.0262 (7)	−0.0006 (6)	0.0030 (5)	−0.0156 (7)
C18	0.0295 (7)	0.0216 (7)	0.0472 (9)	−0.0033 (6)	−0.0022 (6)	0.0078 (6)
O3	0.0190 (4)	0.0304 (5)	0.0278 (5)	−0.0047 (4)	0.0041 (4)	−0.0061 (4)
C1	0.0239 (6)	0.0385 (8)	0.0273 (7)	−0.0006 (6)	0.0001 (5)	0.0000 (6)

Geometric parameters (Å, º)

O1—C2	1.4323 (15)	C10—C17	1.378 (2)
O1—C3	1.3706 (15)	C11—H11	0.9500
O2—N2	1.3417 (13)	C11—C12	1.394 (2)
N1—H1	0.8800	C12—H12	0.9500
N1—C4	1.4061 (15)	C12—C13	1.382 (2)
N1—C6	1.3423 (15)	C13—H13	0.9500
N2—C6	1.2995 (15)	C13—C14	1.3911 (18)
N2—C7	1.4415 (15)	C14—H14	0.9500
C2—H2A	0.9800	C15—C16	1.503 (2)
C2—H2B	0.9800	C15—C17	1.3943 (19)
C2—H2C	0.9800	C16—H16A	0.9800
C3—C4	1.4049 (17)	C16—H16B	0.9800
C3—C11	1.3847 (18)	C16—H16C	0.9800
C4—C14	1.3870 (17)	C17—H17	0.9500
C6—H6	0.9500	C18—H18A	0.9800
C7—C8	1.3941 (18)	C18—H18B	0.9800
C7—C15	1.4008 (17)	C18—H18C	0.9800
C8—C9	1.397 (2)	O3—H3	0.8400
C8—C18	1.505 (2)	O3—C1	1.4113 (16)
C9—H9	0.9500	C1—H1A	0.9800

C9—C10	1.386 (2)	C1—H1B	0.9800
C10—H10	0.9500	C1—H1C	0.9800
C3—O1—C2	117.25 (10)	C12—C11—H11	120.3
C4—N1—H1	116.7	C11—C12—H12	119.7
C6—N1—H1	116.7	C13—C12—C11	120.68 (12)
C6—N1—C4	126.64 (10)	C13—C12—H12	119.7
O2—N2—C7	118.01 (9)	C12—C13—H13	119.9
C6—N2—O2	119.55 (10)	C12—C13—C14	120.15 (12)
C6—N2—C7	122.31 (10)	C14—C13—H13	119.9
O1—C2—H2A	109.5	C4—C14—C13	119.68 (12)
O1—C2—H2B	109.5	C4—C14—H14	120.2
O1—C2—H2C	109.5	C13—C14—H14	120.2
H2A—C2—H2B	109.5	C7—C15—C16	121.06 (11)
H2A—C2—H2C	109.5	C17—C15—C7	117.02 (13)
H2B—C2—H2C	109.5	C17—C15—C16	121.92 (13)
O1—C3—C4	114.35 (10)	C15—C16—H16A	109.5
O1—C3—C11	125.65 (11)	C15—C16—H16B	109.5
C11—C3—C4	120.00 (12)	C15—C16—H16C	109.5
C3—C4—N1	116.18 (11)	H16A—C16—H16B	109.5
C14—C4—N1	123.75 (11)	H16A—C16—H16C	109.5
C14—C4—C3	120.07 (11)	H16B—C16—H16C	109.5
N1—C6—H6	121.2	C10—C17—C15	120.59 (14)
N2—C6—N1	117.55 (11)	C10—C17—H17	119.7
N2—C6—H6	121.2	C15—C17—H17	119.7
C8—C7—N2	118.24 (11)	C8—C18—H18A	109.5
C8—C7—C15	123.93 (12)	C8—C18—H18B	109.5
C15—C7—N2	117.74 (11)	C8—C18—H18C	109.5
C7—C8—C9	116.49 (13)	H18A—C18—H18B	109.5
C7—C8—C18	121.02 (12)	H18A—C18—H18C	109.5
C9—C8—C18	122.49 (13)	H18B—C18—H18C	109.5
C8—C9—H9	119.5	C1—O3—H3	109.5
C10—C9—C8	121.00 (14)	O3—C1—H1A	109.5
C10—C9—H9	119.5	O3—C1—H1B	109.5
C9—C10—H10	119.5	O3—C1—H1C	109.5
C17—C10—C9	120.96 (13)	H1A—C1—H1B	109.5
C17—C10—H10	119.5	H1A—C1—H1C	109.5
C3—C11—H11	120.3	H1B—C1—H1C	109.5
C3—C11—C12	119.41 (12)		
O1—C3—C4—N1	−0.57 (15)	C6—N1—C4—C14	−16.25 (19)
O1—C3—C4—C14	179.46 (10)	C6—N2—C7—C8	102.55 (13)
O1—C3—C11—C12	−179.40 (12)	C6—N2—C7—C15	−80.82 (14)
O2—N2—C6—N1	0.11 (16)	C7—N2—C6—N1	175.94 (10)
O2—N2—C7—C8	−81.55 (14)	C7—C8—C9—C10	0.08 (19)
O2—N2—C7—C15	95.08 (13)	C7—C15—C17—C10	1.02 (19)
N1—C4—C14—C13	179.74 (11)	C8—C7—C15—C16	177.54 (12)
N2—C7—C8—C9	177.55 (11)	C8—C7—C15—C17	−1.69 (18)

N2—C7—C8—C18	−2.53 (17)	C8—C9—C10—C17	−0.7 (2)
N2—C7—C15—C16	1.12 (17)	C9—C10—C17—C15	0.1 (2)
N2—C7—C15—C17	−178.12 (11)	C11—C3—C4—N1	179.35 (11)
C2—O1—C3—C4	−179.56 (11)	C11—C3—C4—C14	−0.62 (18)
C2—O1—C3—C11	0.52 (18)	C11—C12—C13—C14	−1.1 (2)
C3—C4—C14—C13	−0.30 (18)	C12—C13—C14—C4	1.14 (19)
C3—C11—C12—C13	0.2 (2)	C15—C7—C8—C9	1.15 (18)
C4—N1—C6—N2	−178.00 (11)	C15—C7—C8—C18	−178.94 (12)
C4—C3—C11—C12	0.69 (19)	C16—C15—C17—C10	−178.21 (13)
C6—N1—C4—C3	163.79 (11)	C18—C8—C9—C10	−179.84 (13)

Hydrogen-bond geometry (Å, º)

π is the centroid of the C7–C10/C15/C17 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O3^{i}	0.95	2.20	3.1134 (14)	160
O3—H3···O2^{ii}	0.84	1.85	2.6887 (12)	173
C1—H1*C*···π^{i}	0.98	2.66	3.5182 (16)	148

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