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4-Methyl-*N*-(4-methylpyridin-2-yl)-*N*-(3,4,5,6-tetrahydro-2*H*-pyran-4-yl)pyridin-2-amine

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Keywords: crystal structure; pyridinamine; tertiary amine; palladium-catalysed aryl-amination.

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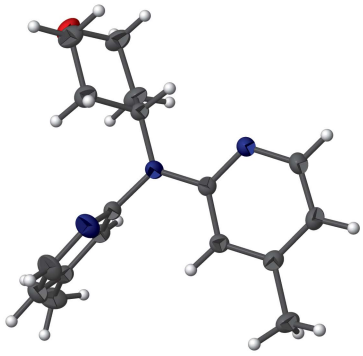
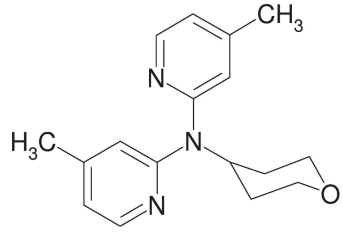
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{21}N_3O$, the pyridine rings make a dihedral angle of 84.44 (5)°. In the crystal, a C—H···N interaction forms a chain of molecules propagating along the twofold screw axis.

3D view



Chemical scheme



Structure description

The title compound was obtained as a side product in the palladium-catalysed reaction of 2-bromo-4-methylpyridine and tetrahydro-2*H*-pyran-4-amine hydrochloride (Laufer & Koch, 2008; Koch *et al.*, 2008). The title compound is shown in Fig. 1. One pyridine ring is nearly perpendicular to the other pyridine ring, the dihedral angle between them being 84.44 (5)°. The molecular conformation features a short intramolecular C—H···N contact (Table 1). In the crystal, an intermolecular interaction between H14*B* and N16 forms a chain of molecules along the twofold screw axis.

Synthesis and crystallization

2-Bromo-4-methylpyridine (0.20 g, 1.2 mmol), tetrahydro-2*H*-pyran-4-amine hydrochloride (0.19 g, 1.4 mmol), *t*-BuONa (0.27 g, 2.8 mmol), $Pd_2(dba)_3$ (21 mg, 0.023 mmol) and BINAP (29 mg, 0.046 mmol) were dissolved in dry toluene (30 ml) under an argon atmosphere. The mixture was stirred 2 h at 343 K. The mixture was allowed to cool to 298 K before *n*-hexane was added. The formed precipitate was filtered off and the filtrate concentrated to dryness. Once again, *n*-hexane was added to the residue and the precipitate was filtered off. The filtrate was concentrated *in vacuo* and the crude product was purified by flash chromatography (SiO_2, from *n*-hexane/EtOAc 1:1 to EtOAc 100%) to afford 20 mg of the title compound as a colorless solid. Suitable crystals for X-ray

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···N9	1.00	2.33	2.7852 (13)	107
C14—H14B···N16^{i}	0.98	2.61	3.4881 (16)	149

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

diffraction were obtained by slow evaporation at 298 K of a solution of *n*-hexane–ethyl acetate (1:1).

Refinement

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

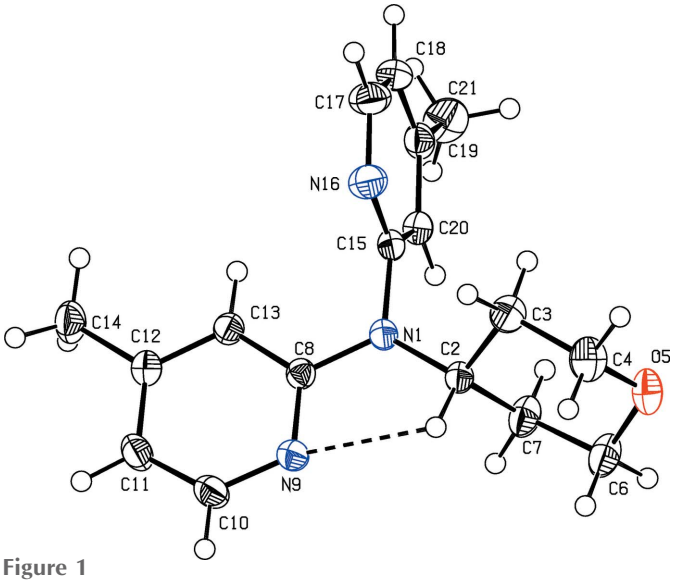


Figure 1
Molecular structure of the title compound with atom labelling and displacement ellipsoids drawn at the 50% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{21}N_3O$
M_r	283.37
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, b, c (Å)	14.5487 (9), 10.1675 (6), 10.4269 (6)
β (°)	100.1035 (17)
V (Å^3)	1518.47 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.50 × 0.40 × 0.24
Data collection	
Diffractometer	Bruker SMART CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	65777, 3668, 3310
R_{int}	0.024
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.660
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.122, 1.05
No. of reflections	3668
No. of parameters	192
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.25

Computer programs: *APEX2* (Bruker, 2006), *SIR97* (Altomare *et al.*, 1999), *SHELXL2014* and *XP* (Sheldrick, 2015).

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full crystallographic data

IUCrData (2016). **1**, x160804 [doi:10.1107/S241431461600804X]

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4-Methyl-*N*-(4-methylpyridin-2-yl)-*N*-(3,4,5,6-tetrahydro-2*H*-pyran-4-yl)pyridin-2-amine

Crystal data

$C_{17}H_{21}N_3O$
$M_r = 283.37$
Monoclinic, $P2_1/c$
$a = 14.5487$ (9) Å
$b = 10.1675$ (6) Å
$c = 10.4269$ (6) Å
$\beta = 100.1035$ (17)°
$V = 1518.47$ (16) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 9759 reflections
$\theta = 2.2$–$36.2°$
$\mu = 0.08$ mm^{-1}
$T = 173$ K
Block, colorless
0.50 × 0.40 × 0.24 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: sealed Tube
Graphite monochromator
CCD scan
65777 measured reflections
3668 independent reflections
3310 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.5°$
$h = -19 \rightarrow 19$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.122$
$S = 1.05$
3668 reflections
192 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.4545P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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}
N1	0.24545 (6)	0.35573 (9)	0.11326 (9)	0.0270 (2)
C2	0.18456 (7)	0.35008 (10)	−0.01542 (9)	0.0240 (2)
H2	0.2066	0.2757	−0.0650	0.029*
C3	0.19032 (8)	0.47553 (11)	−0.09352 (11)	0.0311 (2)
H3A	0.1747	0.5522	−0.0429	0.037*
H3B	0.2548	0.4872	−0.1100	0.037*
C4	0.12302 (9)	0.46855 (13)	−0.22235 (11)	0.0363 (3)
H4A	0.1263	0.5517	−0.2707	0.044*
H4B	0.1421	0.3961	−0.2755	0.044*
O5	0.02931 (6)	0.44727 (9)	−0.20449 (8)	0.0362 (2)
C6	0.02138 (8)	0.32609 (13)	−0.13960 (12)	0.0370 (3)
H6A	0.0397	0.2532	−0.1928	0.044*
H6B	−0.0445	0.3122	−0.1305	0.044*
C7	0.08279 (7)	0.32332 (12)	−0.00489 (11)	0.0323 (2)
H7A	0.0778	0.2362	0.0357	0.039*
H7B	0.0609	0.3908	0.0513	0.039*
C8	0.30513 (6)	0.25363 (9)	0.15970 (9)	0.0215 (2)
N9	0.30153 (6)	0.14462 (8)	0.08667 (8)	0.02608 (19)
C10	0.36135 (8)	0.04730 (10)	0.13208 (11)	0.0319 (2)
H10	0.3605	−0.0296	0.0802	0.038*
C11	0.42372 (8)	0.05082 (11)	0.24818 (11)	0.0319 (2)
H11	0.4648	−0.0207	0.2742	0.038*
C12	0.42478 (7)	0.16324 (10)	0.32680 (10)	0.0258 (2)
C13	0.36555 (7)	0.26533 (10)	0.28109 (9)	0.0238 (2)
H13	0.3654	0.3434	0.3311	0.029*
C14	0.48623 (8)	0.17290 (13)	0.45881 (11)	0.0359 (3)
H14A	0.5002	0.2655	0.4798	0.054*
H14B	0.5445	0.1250	0.4577	0.054*
H14C	0.4539	0.1345	0.5247	0.054*
C15	0.25355 (7)	0.47598 (10)	0.18586 (10)	0.0244 (2)
N16	0.32140 (7)	0.55840 (10)	0.16619 (10)	0.0332 (2)
C17	0.33256 (8)	0.66720 (12)	0.23981 (13)	0.0370 (3)
H17	0.3802	0.7272	0.2273	0.044*
C18	0.27875 (8)	0.69742 (11)	0.33315 (12)	0.0331 (2)
H18	0.2903	0.7755	0.3835	0.040*
C19	0.20782 (7)	0.61220 (11)	0.35218 (10)	0.0283 (2)
C20	0.19537 (7)	0.49906 (10)	0.27543 (10)	0.0257 (2)
H20	0.1474	0.4383	0.2845	0.031*
C21	0.14761 (10)	0.63831 (15)	0.45276 (12)	0.0435 (3)
H21A	0.1839	0.6861	0.5263	0.065*
H21B	0.1261	0.5546	0.4835	0.065*
H21C	0.0935	0.6913	0.4140	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0261 (4)	0.0235 (4)	0.0280 (4)	0.0056 (3)	−0.0045 (3)	−0.0052 (3)
C2	0.0229 (4)	0.0240 (5)	0.0235 (5)	0.0033 (3)	−0.0005 (3)	−0.0016 (3)
C3	0.0308 (5)	0.0327 (5)	0.0297 (5)	−0.0025 (4)	0.0051 (4)	0.0041 (4)
C4	0.0387 (6)	0.0449 (7)	0.0254 (5)	0.0034 (5)	0.0064 (4)	0.0071 (4)
O5	0.0315 (4)	0.0450 (5)	0.0302 (4)	0.0100 (3)	−0.0001 (3)	0.0081 (3)
C6	0.0265 (5)	0.0471 (7)	0.0341 (6)	−0.0024 (5)	−0.0043 (4)	0.0072 (5)
C7	0.0255 (5)	0.0410 (6)	0.0284 (5)	−0.0032 (4)	−0.0007 (4)	0.0091 (4)
C8	0.0192 (4)	0.0224 (4)	0.0232 (4)	0.0003 (3)	0.0042 (3)	0.0007 (3)
N9	0.0285 (4)	0.0227 (4)	0.0257 (4)	0.0018 (3)	0.0014 (3)	−0.0017 (3)
C10	0.0390 (6)	0.0234 (5)	0.0316 (5)	0.0059 (4)	0.0016 (4)	−0.0031 (4)
C11	0.0356 (6)	0.0265 (5)	0.0316 (5)	0.0094 (4)	0.0009 (4)	0.0021 (4)
C12	0.0238 (5)	0.0289 (5)	0.0242 (5)	0.0023 (4)	0.0033 (4)	0.0022 (4)
C13	0.0225 (4)	0.0251 (5)	0.0233 (4)	0.0013 (4)	0.0029 (3)	−0.0020 (3)
C14	0.0350 (6)	0.0417 (6)	0.0275 (5)	0.0114 (5)	−0.0039 (4)	−0.0008 (4)
C15	0.0226 (4)	0.0222 (5)	0.0261 (5)	0.0044 (3)	−0.0017 (3)	−0.0020 (3)
N16	0.0286 (4)	0.0319 (5)	0.0398 (5)	−0.0025 (4)	0.0078 (4)	−0.0081 (4)
C17	0.0331 (6)	0.0301 (6)	0.0482 (7)	−0.0065 (4)	0.0087 (5)	−0.0080 (5)
C18	0.0349 (6)	0.0254 (5)	0.0368 (6)	0.0023 (4)	0.0003 (4)	−0.0087 (4)
C19	0.0290 (5)	0.0304 (5)	0.0239 (5)	0.0072 (4)	0.0001 (4)	−0.0021 (4)
C20	0.0250 (5)	0.0257 (5)	0.0247 (5)	0.0013 (4)	0.0001 (4)	0.0011 (4)
C21	0.0454 (7)	0.0548 (8)	0.0320 (6)	0.0017 (6)	0.0115 (5)	−0.0107 (5)

Geometric parameters (Å, º)

N1—C8	1.3850 (12)	C10—H10	0.9500
N1—C15	1.4319 (12)	C11—C12	1.4051 (15)
N1—C2	1.4736 (12)	C11—H11	0.9500
C2—C3	1.5236 (14)	C12—C13	1.3796 (14)
C2—C7	1.5281 (14)	C12—C14	1.5072 (14)
C2—H2	1.0000	C13—H13	0.9500
C3—C4	1.5188 (15)	C14—H14A	0.9800
C3—H3A	0.9900	C14—H14B	0.9800
C3—H3B	0.9900	C14—H14C	0.9800
C4—O5	1.4245 (15)	C15—N16	1.3379 (14)
C4—H4A	0.9900	C15—C20	1.3861 (14)
C4—H4B	0.9900	N16—C17	1.3400 (15)
O5—C6	1.4201 (15)	C17—C18	1.3863 (17)
C6—C7	1.5269 (15)	C17—H17	0.9500
C6—H6A	0.9900	C18—C19	1.3882 (16)
C6—H6B	0.9900	C18—H18	0.9500
C7—H7A	0.9900	C19—C20	1.3948 (14)
C7—H7B	0.9900	C19—C21	1.5034 (15)
C8—N9	1.3406 (12)	C20—H20	0.9500
C8—C13	1.4137 (13)	C21—H21A	0.9800
N9—C10	1.3476 (14)	C21—H21B	0.9800

C10—C11	1.3801 (15)	C21—H21C	0.9800
C8—N1—C15	117.82 (8)	N9—C10—H10	117.5
C8—N1—C2	121.94 (8)	C11—C10—H10	117.5
C15—N1—C2	119.68 (8)	C10—C11—C12	118.11 (9)
N1—C2—C3	111.92 (8)	C10—C11—H11	120.9
N1—C2—C7	112.14 (8)	C12—C11—H11	120.9
C3—C2—C7	109.41 (8)	C13—C12—C11	117.92 (9)
N1—C2—H2	107.7	C13—C12—C14	120.06 (9)
C3—C2—H2	107.7	C11—C12—C14	122.00 (9)
C7—C2—H2	107.7	C12—C13—C8	119.91 (9)
C4—C3—C2	110.25 (9)	C12—C13—H13	120.0
C4—C3—H3A	109.6	C8—C13—H13	120.0
C2—C3—H3A	109.6	C12—C14—H14A	109.5
C4—C3—H3B	109.6	C12—C14—H14B	109.5
C2—C3—H3B	109.6	H14A—C14—H14B	109.5
H3A—C3—H3B	108.1	C12—C14—H14C	109.5
O5—C4—C3	112.01 (9)	H14A—C14—H14C	109.5
O5—C4—H4A	109.2	H14B—C14—H14C	109.5
C3—C4—H4A	109.2	N16—C15—C20	123.47 (9)
O5—C4—H4B	109.2	N16—C15—N1	116.64 (9)
C3—C4—H4B	109.2	C20—C15—N1	119.83 (9)
H4A—C4—H4B	107.9	C15—N16—C17	116.60 (10)
C6—O5—C4	110.71 (9)	N16—C17—C18	123.96 (11)
O5—C6—C7	111.78 (10)	N16—C17—H17	118.0
O5—C6—H6A	109.3	C18—C17—H17	118.0
C7—C6—H6A	109.3	C17—C18—C19	119.18 (10)
O5—C6—H6B	109.3	C17—C18—H18	120.4
C7—C6—H6B	109.3	C19—C18—H18	120.4
H6A—C6—H6B	107.9	C18—C19—C20	117.24 (10)
C6—C7—C2	110.40 (9)	C18—C19—C21	121.89 (10)
C6—C7—H7A	109.6	C20—C19—C21	120.86 (10)
C2—C7—H7A	109.6	C15—C20—C19	119.54 (10)
C6—C7—H7B	109.6	C15—C20—H20	120.2
C2—C7—H7B	109.6	C19—C20—H20	120.2
H7A—C7—H7B	108.1	C19—C21—H21A	109.5
N9—C8—N1	117.48 (8)	C19—C21—H21B	109.5
N9—C8—C13	122.30 (9)	H21A—C21—H21B	109.5
N1—C8—C13	120.21 (9)	C19—C21—H21C	109.5
C8—N9—C10	116.68 (9)	H21A—C21—H21C	109.5
N9—C10—C11	125.02 (10)	H21B—C21—H21C	109.5
C8—N1—C2—C3	−132.02 (10)	C10—C11—C12—C13	−2.15 (16)
C15—N1—C2—C3	39.22 (13)	C10—C11—C12—C14	176.26 (11)
C8—N1—C2—C7	104.59 (11)	C11—C12—C13—C8	1.15 (15)
C15—N1—C2—C7	−84.18 (12)	C14—C12—C13—C8	−177.30 (9)
N1—C2—C3—C4	−177.13 (8)	N9—C8—C13—C12	1.28 (15)
C7—C2—C3—C4	−52.21 (12)	N1—C8—C13—C12	179.97 (9)

C2—C3—C4—O5	57.26 (13)	C8—N1—C15—N16	81.25 (12)
C3—C4—O5—C6	−61.05 (13)	C2—N1—C15—N16	−90.35 (11)
C4—O5—C6—C7	60.54 (13)	C8—N1—C15—C20	−95.89 (11)
O5—C6—C7—C2	−56.63 (13)	C2—N1—C15—C20	92.52 (12)
N1—C2—C7—C6	176.76 (9)	C20—C15—N16—C17	0.90 (16)
C3—C2—C7—C6	51.96 (12)	N1—C15—N16—C17	−176.12 (10)
C15—N1—C8—N9	−175.55 (9)	C15—N16—C17—C18	0.18 (18)
C2—N1—C8—N9	−4.16 (14)	N16—C17—C18—C19	−0.85 (19)
C15—N1—C8—C13	5.70 (14)	C17—C18—C19—C20	0.44 (16)
C2—N1—C8—C13	177.09 (9)	C17—C18—C19—C21	179.31 (11)
N1—C8—N9—C10	178.73 (9)	N16—C15—C20—C19	−1.29 (15)
C13—C8—N9—C10	−2.55 (14)	N1—C15—C20—C19	175.64 (9)
C8—N9—C10—C11	1.48 (17)	C18—C19—C20—C15	0.55 (14)
N9—C10—C11—C12	0.88 (18)	C21—C19—C20—C15	−178.33 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N9	1.00	2.33	2.7852 (13)	107
C14—H14*B*···N16^{i}	0.98	2.61	3.4881 (16)	149

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