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4-{2-[Methyl(2-pyridyl)amino]ethoxy}-benzaldehyde

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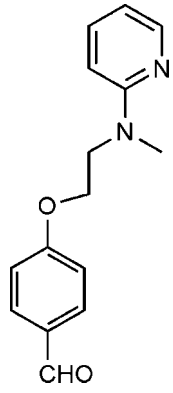
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.035; wR factor = 0.101; data-to-parameter ratio = 8.5.

In the title compound, $C_{15}H_{16}N_2O_2$, the dihedral angle between the aromatic ring planes is 81.37 (13)°. In the crystal structure, π–π stacking [centroid–centroid separation = 3.890 (2) Å] helps to establish the packing.

Related literature

For the synthesis, see: Cantello *et al.* (1994).



Experimental

Crystal data

$C_{15}H_{16}N_2O_2$
M_r = 256.30
Monoclinic, $P2_1$
a = 6.1995 (19) Å
b = 9.416 (3) Å
c = 11.714 (3) Å
β = 98.328 (5)°
V = 676.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.10 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.980, T_{max} = 0.992
3897 measured reflections
1477 independent reflections
980 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.101
S = 0.98
1477 reflections
174 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2594).

References

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supplementary materials

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4-{2-[Methyl(2-pyridyl)amino]ethoxy}benzaldehyde

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Comment

The title compound, (I), (Fig. 1) is an intermediate in the synthesis of the anti-diabetic drug rosiglitazone.

The dihedral angle between the N1/C1—C5 pyridine ring and C9—C14 phenyl ring is 81.37 (13)°. In the crystal (Fig. 2), weak aromatic π-π stacking helps to establish the packing with a centroid-centroid separation of 3.890 (2)Å for the N1/C1—C5 and C9^{i}—C14^{i} (i = 1 − *x*, 1/2 + *y*, 1 − *z*) rings.

Experimental

The title compound was prepared according to the method of Cantello *et al.* (1994). Colourless blocks of (I) were obtained by recrystallization from dichromethane.

Refinement

Anomalous dispersion was negligible and Friedel pairs were merged before refinement.

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

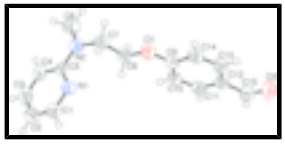

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

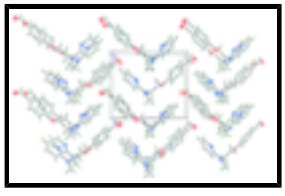

Fig. 2. The crystal packing for (I).

4-{2-[Methyl(2-pyridyl)amino]ethoxy}benzaldehyde

Crystal data

$C_{15}H_{16}N_2O_2$	F_{000} = 272
M_r = 256.30	D_x = 1.258 Mg m^{-3}

Monoclinic, $P2_1$	Melting point: 338-339 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.1995 (19) Å	Cell parameters from 1123 reflections
b = 9.416 (3) Å	θ = 2.8–23.7º
c = 11.714 (3) Å	μ = 0.09 mm^{-1}
β = 98.328 (5)º	T = 294 (2) K
V = 676.6 (3) $Å^3$	Block, colorless
Z = 2	0.24 × 0.22 × 0.10 mm

Data collection

Bruker SMART 1000 CCD diffractometer	1477 independent reflections
Radiation source: fine-focus sealed tube	980 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
T = 294(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 7$
T_{min} = 0.980, T_{max} = 0.992	$k = -6 \rightarrow 11$
3897 measured reflections	$l = -11 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H-atom parameters constrained
$wR(F^2)$ = 0.101	$w = 1/[\sigma^2(F_o^2) + (0.058P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.98	$(\Delta/\sigma)_{max} < 0.001$
1477 reflections	$\Delta\rho_{max}$ = 0.11 e $Å^{-3}$
174 parameters	$\Delta\rho_{min}$ = −0.11 e $Å^{-3}$
1 restraint	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.144 (13)

Special details

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 calculat-

ing *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}
O1	0.4167 (3)	0.5026 (2)	0.66454 (15)	0.0590 (5)
O2	0.1555 (4)	0.1192 (3)	1.0759 (2)	0.0905 (8)
N1	0.6212 (3)	0.4415 (2)	0.33536 (18)	0.0537 (6)
N2	0.7653 (3)	0.6252 (2)	0.45508 (18)	0.0554 (6)
C1	0.6382 (5)	0.3521 (3)	0.2477 (2)	0.0672 (8)
H1	0.5234	0.2897	0.2258	0.081*
C2	0.8123 (6)	0.3465 (4)	0.1885 (3)	0.0751 (10)
H2	0.8161	0.2825	0.1284	0.090*
C3	0.9817 (5)	0.4388 (4)	0.2208 (3)	0.0731 (9)
H3	1.1034	0.4373	0.1829	0.088*
C4	0.9714 (4)	0.5337 (3)	0.3093 (2)	0.0571 (8)
H4	1.0853	0.5968	0.3312	0.068*
C5	0.7866 (4)	0.5338 (3)	0.36600 (19)	0.0437 (6)
C6	0.9392 (5)	0.7225 (4)	0.4982 (3)	0.0798 (10)
H6A	1.0639	0.6696	0.5332	0.120*
H6B	0.8910	0.7843	0.5546	0.120*
H6C	0.9780	0.7780	0.4355	0.120*
C7	0.5769 (4)	0.6150 (3)	0.5166 (2)	0.0552 (7)
H7A	0.4488	0.5907	0.4624	0.066*
H7B	0.5512	0.7067	0.5499	0.066*
C8	0.6100 (4)	0.5051 (3)	0.6109 (2)	0.0530 (7)
H8A	0.6349	0.4125	0.5788	0.064*
H8B	0.7354	0.5293	0.6670	0.064*
C9	0.4029 (4)	0.4081 (3)	0.7518 (2)	0.0467 (6)
C10	0.5581 (4)	0.3040 (3)	0.7876 (2)	0.0524 (7)
H10	0.6831	0.2961	0.7527	0.063*
C11	0.5236 (4)	0.2129 (3)	0.8756 (2)	0.0533 (7)
H11	0.6271	0.1434	0.8992	0.064*
C12	0.3387 (4)	0.2219 (3)	0.9301 (2)	0.0492 (6)
C13	0.1869 (4)	0.3291 (3)	0.8948 (2)	0.0554 (7)
H13	0.0641	0.3386	0.9312	0.066*
C14	0.2173 (4)	0.4204 (3)	0.8069 (2)	0.0527 (7)
H14	0.1146	0.4906	0.7839	0.063*
C15	0.3057 (5)	0.1201 (4)	1.0211 (2)	0.0661 (8)
H15	0.4108	0.0497	1.0380	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0572 (11)	0.0594 (12)	0.0629 (11)	0.0153 (9)	0.0170 (8)	0.0084 (11)
O2	0.0982 (16)	0.0866 (17)	0.0965 (16)	0.0028 (16)	0.0467 (14)	0.0149 (16)
N1	0.0592 (14)	0.0494 (13)	0.0526 (13)	−0.0098 (12)	0.0081 (10)	−0.0044 (12)

N2	0.0621 (13)	0.0538 (14)	0.0506 (12)	−0.0115 (13)	0.0093 (10)	−0.0076 (13)
C1	0.089 (2)	0.0523 (18)	0.0591 (18)	−0.0121 (17)	0.0078 (16)	−0.0050 (16)
C2	0.104 (3)	0.064 (2)	0.0608 (19)	0.013 (2)	0.0237 (18)	−0.0032 (17)
C3	0.075 (2)	0.090 (2)	0.0601 (18)	0.022 (2)	0.0290 (16)	0.018 (2)
C4	0.0484 (14)	0.0660 (19)	0.0570 (17)	−0.0023 (14)	0.0086 (12)	0.0161 (16)
C5	0.0480 (13)	0.0414 (14)	0.0402 (13)	−0.0013 (12)	0.0018 (10)	0.0092 (13)
C6	0.088 (2)	0.074 (2)	0.0747 (18)	−0.027 (2)	0.0024 (17)	−0.0139 (19)
C7	0.0631 (16)	0.0519 (16)	0.0516 (14)	0.0118 (15)	0.0113 (12)	0.0012 (14)
C8	0.0527 (14)	0.0530 (16)	0.0548 (15)	0.0069 (13)	0.0128 (11)	−0.0026 (15)
C9	0.0500 (14)	0.0436 (15)	0.0460 (14)	0.0049 (12)	0.0053 (11)	−0.0079 (13)
C10	0.0495 (14)	0.0536 (16)	0.0566 (16)	0.0088 (13)	0.0161 (12)	−0.0047 (14)
C11	0.0547 (15)	0.0506 (16)	0.0534 (15)	0.0110 (14)	0.0032 (12)	−0.0025 (15)
C12	0.0504 (14)	0.0507 (15)	0.0461 (14)	−0.0032 (14)	0.0053 (11)	−0.0066 (14)
C13	0.0473 (15)	0.0605 (18)	0.0593 (17)	−0.0024 (14)	0.0110 (12)	−0.0060 (15)
C14	0.0425 (14)	0.0538 (18)	0.0614 (17)	0.0072 (13)	0.0065 (13)	−0.0025 (15)
C15	0.0736 (19)	0.0639 (19)	0.0610 (17)	−0.0011 (17)	0.0107 (15)	0.0025 (18)

Geometric parameters (Å, °)

O1—C9	1.367 (3)	C6—H6C	0.9600
O1—C8	1.431 (3)	C7—C8	1.506 (4)
O2—C15	1.205 (3)	C7—H7A	0.9700
N1—C1	1.343 (3)	C7—H7B	0.9700
N1—C5	1.353 (3)	C8—H8A	0.9700
N2—C5	1.373 (3)	C8—H8B	0.9700
N2—C6	1.449 (4)	C9—C10	1.394 (3)
N2—C7	1.462 (3)	C9—C14	1.403 (4)
C1—C2	1.366 (4)	C10—C11	1.382 (4)
C1—H1	0.9300	C10—H10	0.9300
C2—C3	1.373 (5)	C11—C12	1.393 (3)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.376 (4)	C12—C13	1.401 (4)
C3—H3	0.9300	C12—C15	1.470 (4)
C4—C5	1.405 (3)	C13—C14	1.375 (4)
C4—H4	0.9300	C13—H13	0.9300
C6—H6A	0.9600	C14—H14	0.9300
C6—H6B	0.9600	C15—H15	0.9300
C9—O1—C8	119.0 (2)	C8—C7—H7B	109.2
C1—N1—C5	117.7 (2)	H7A—C7—H7B	107.9
C5—N2—C6	120.8 (2)	O1—C8—C7	107.4 (2)
C5—N2—C7	120.3 (2)	O1—C8—H8A	110.2
C6—N2—C7	118.5 (2)	C7—C8—H8A	110.2
N1—C1—C2	124.6 (3)	O1—C8—H8B	110.2
N1—C1—H1	117.7	C7—C8—H8B	110.2
C2—C1—H1	117.7	H8A—C8—H8B	108.5
C1—C2—C3	117.7 (3)	O1—C9—C10	124.5 (2)
C1—C2—H2	121.2	O1—C9—C14	115.7 (2)
C3—C2—H2	121.2	C10—C9—C14	119.7 (2)
C2—C3—C4	120.1 (3)	C11—C10—C9	119.1 (2)

C2—C3—H3	119.9	C11—C10—H10	120.5
C4—C3—H3	119.9	C9—C10—H10	120.5
C3—C4—C5	119.1 (3)	C10—C11—C12	122.1 (2)
C3—C4—H4	120.4	C10—C11—H11	119.0
C5—C4—H4	120.4	C12—C11—H11	119.0
N1—C5—N2	116.9 (2)	C11—C12—C13	118.1 (2)
N1—C5—C4	120.8 (2)	C11—C12—C15	120.2 (3)
N2—C5—C4	122.3 (2)	C13—C12—C15	121.8 (2)
N2—C6—H6A	109.5	C14—C13—C12	120.8 (2)
N2—C6—H6B	109.5	C14—C13—H13	119.6
H6A—C6—H6B	109.5	C12—C13—H13	119.6
N2—C6—H6C	109.5	C13—C14—C9	120.2 (2)
H6A—C6—H6C	109.5	C13—C14—H14	119.9
H6B—C6—H6C	109.5	C9—C14—H14	119.9
N2—C7—C8	111.9 (2)	O2—C15—C12	126.2 (3)
N2—C7—H7A	109.2	O2—C15—H15	116.9
C8—C7—H7A	109.2	C12—C15—H15	116.9
N2—C7—H7B	109.2		
C5—N1—C1—C2	1.1 (4)	N2—C7—C8—O1	−179.4 (2)
N1—C1—C2—C3	−0.1 (5)	C8—O1—C9—C10	−5.3 (4)
C1—C2—C3—C4	−0.7 (5)	C8—O1—C9—C14	174.6 (2)
C2—C3—C4—C5	0.4 (4)	O1—C9—C10—C11	−178.9 (2)
C1—N1—C5—N2	179.4 (2)	C14—C9—C10—C11	1.3 (4)
C1—N1—C5—C4	−1.3 (3)	C9—C10—C11—C12	−0.2 (4)
C6—N2—C5—N1	176.5 (3)	C10—C11—C12—C13	−1.2 (4)
C7—N2—C5—N1	3.4 (3)	C10—C11—C12—C15	178.2 (3)
C6—N2—C5—C4	−2.7 (4)	C11—C12—C13—C14	1.6 (4)
C7—N2—C5—C4	−175.8 (2)	C15—C12—C13—C14	−177.8 (2)
C3—C4—C5—N1	0.7 (4)	C12—C13—C14—C9	−0.5 (4)
C3—C4—C5—N2	179.9 (2)	O1—C9—C14—C13	179.2 (2)
C5—N2—C7—C8	84.3 (3)	C10—C9—C14—C13	−0.9 (4)
C6—N2—C7—C8	−89.0 (3)	C11—C12—C15—O2	178.3 (3)
C9—O1—C8—C7	178.3 (2)	C13—C12—C15—O2	−2.3 (4)

Fig. 1

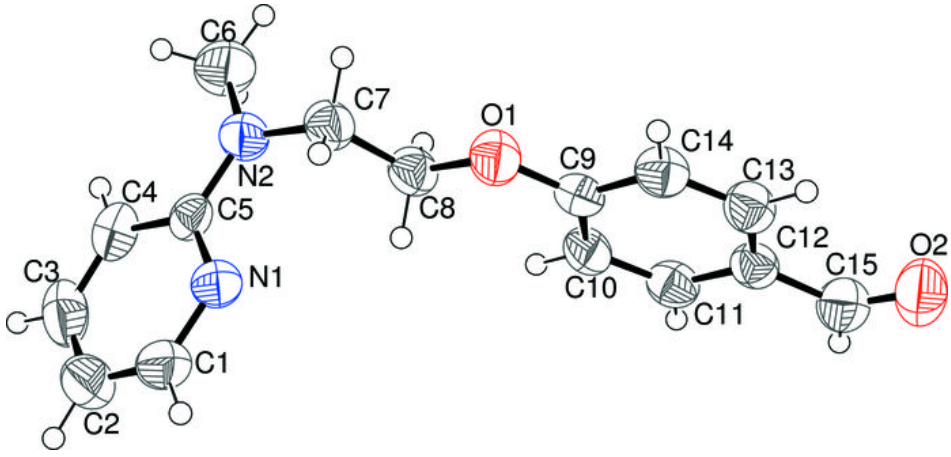

Fig. 2