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4-Methoxy-*N*-(3,4-methylenedioxybenzyl)aniline

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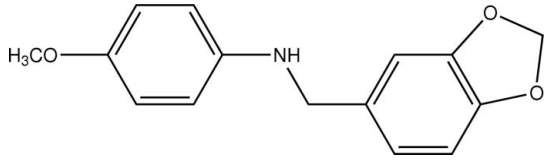
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; disorder in main residue; R factor = 0.094; wR factor = 0.214; data-to-parameter ratio = 12.7.

In the crystal structure of the title compound, $C_{15}H_{15}NO_3$, there are no classical hydrogen bonds. The molecules are connected by four weak C—H$\cdots\pi$ interactions, resulting in a three-dimensional structure. The N-bound H atom is disordered equally over two sites.

Related literature

For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{15}H_{15}NO_3$
M_r = 257.28
Orthorhombic, *Pbcn*
a = 54.050 (3) Å
b = 7.353 (1) Å
c = 6.4830 (9) Å
V = 2576.5 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.52 × 0.49 × 0.45 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.953, T_{max} = 0.959
9965 measured reflections
2201 independent reflections
1478 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.094
$wR(F^2)$ = 0.214
S = 1.07
2201 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the rings C2–C7 and C9–C14, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C3—H3$\cdots$*Cg*1^{i}	0.93	2.82	3.64 (2)	148
C6—H6$\cdots$*Cg*1^{ii}	0.93	2.75	3.55 (2)	145
C11—H11$\cdots$*Cg*2^{iii}	0.93	2.80	3.63 (2)	150
C14—H14$\cdots$*Cg*2^{iv}	0.93	2.76	3.59 (2)	149

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2401).

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supplementary materials

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4-Methoxy-*N*-(3,4-methylenedioxybenzyl)aniline

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Comment

The title compound, (I), was obtained by the reductive reaction of the schiff base, 4-methoxy-*N*-(3,4-methylenedioxybenzylidene)-aniline. we report here the crystal structure of (I).

The compounds (I) crystallizes in the orthorhombic space group *Pbcn* with $Z = 8$. A s a result, the H1A and H1B bonded to N1 atom are disordered, each with 0.5 site occupancy (Fig. 1), the dihedral angles between the two benzene rings are 68.7 (2)°. Geometric parameters are normal (Allen *et al.*, 1987) in (I).

In the crystal structure of (I), there are no classic hydrogen bonds. The molecules are connected by four weak C—H···π interactions with the distances in the range 3.55 (2) to 3.64 (2) Å, resulting in a three-dimensional structure (Table 1).

Experimental

To a solution containing 4-methoxy-*N*-(3,4-methylenedioxybenzylidene)-aniline (1.14 g, 5 mmol) and ethanol (20 ml), the borohydride sodium (0.76 g, 20 mmol) was added and stirred for 4 h (at 333–343 K), then acetone (10 ml) and water (20 ml) were added in turn, and the reaction mixture was cooled and the crude products were filtered off, washed with ethanol and dried. Colourless crystals of (I) suitable for X-ray structure analysis were obtained by recrystallizing the crude product from ethanol [m.p. 339–341 K].

Refinement

H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic), 0.97 Å (methylene) and 0.96 Å (methyl), N—H = 0.90 Å (amino), and refined in riding mode with $U_{iso}(H) = 1.5U_{eq}(C)$ (methyl) and $U_{iso}(H) = 1.2U_{eq}(C, N)$ (aromatic, methylene and amino). H atom on the amino group is disorder, which was identified from a difference Fourier map, the site occupancies were fixed at 0.5. The disordered H atoms were refined isotropically with riding mode.

Figures

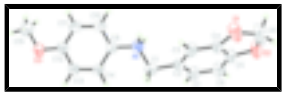

Fig. 1. A view of the title compund (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

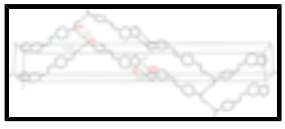

Fig. 2. Packing diagram.

4-Methoxy-*N*-(3,4-methylenedioxybenzyl)aniline

Crystal data

$C_{15}H_{15}NO_3$	$D_x = 1.327$ Mg m^{-3}
$M_r = 257.28$	Melting point: 339 K
Orthorhombic, *Pbcn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2n 2ab	Cell parameters from 2390 reflections
a = 54.050 (3) Å	θ = 2.8–21.7º
b = 7.3530 (10) Å	μ = 0.09 mm^{-1}
c = 6.4830 (9) Å	*T* = 298 (2) K
V = 2576.5 (5) Å^3	Block, colourless
Z = 8	0.52 × 0.49 × 0.45 mm
F_{000} = 1088	

Data collection

Bruker SMART CCD area-detector diffractometer	2201 independent reflections
Radiation source: fine-focus sealed tube	1478 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.061$
T = 298(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.5$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −64→41
$T_{min} = 0.953$, $T_{max} = 0.959$	*k* = −8→8
9965 measured reflections	*l* = −6→7

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.094$	H-atom parameters constrained
$wR(F^2) = 0.214$	$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 7.8609P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
2201 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 calculating *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}	Occ. (<1)
N1	0.37308 (6)	0.2577 (5)	0.7958 (6)	0.0443 (10)	
H1A	0.3836	0.2220	0.8954	0.053*	0.50
H1B	0.3765	0.3751	0.7687	0.053*	0.50
O1	0.47087 (6)	0.1691 (6)	0.6437 (6)	0.0680 (12)	
O2	0.47446 (6)	0.3279 (5)	0.3376 (6)	0.0621 (11)	
O3	0.27794 (6)	0.2373 (5)	1.1237 (6)	0.0570 (10)	
C1	0.37902 (8)	0.1547 (6)	0.6126 (7)	0.0457 (12)	
H1C	0.3668	0.1788	0.5067	0.055*	
H1D	0.3786	0.0257	0.6440	0.055*	
C2	0.40408 (8)	0.2046 (6)	0.5346 (7)	0.0366 (10)	
C3	0.42537 (8)	0.1544 (6)	0.6460 (7)	0.0436 (11)	
H3	0.4242	0.0916	0.7701	0.052*	
C4	0.44761 (8)	0.2016 (6)	0.5649 (7)	0.0396 (11)	
C5	0.45004 (8)	0.2954 (6)	0.3819 (7)	0.0419 (11)	
C6	0.42980 (9)	0.3458 (7)	0.2706 (8)	0.0507 (13)	
H6	0.4312	0.4090	0.1469	0.061*	
C7	0.40707 (8)	0.2980 (6)	0.3510 (8)	0.0456 (12)	
H7	0.3930	0.3305	0.2776	0.055*	
C8	0.48742 (10)	0.2496 (9)	0.5030 (10)	0.0771 (19)	
H8A	0.4970	0.3425	0.5727	0.093*	
H8B	0.4988	0.1582	0.4512	0.093*	
C9	0.34927 (7)	0.2513 (6)	0.8788 (7)	0.0344 (10)	
C10	0.34322 (8)	0.3562 (6)	1.0500 (7)	0.0383 (11)	
H10	0.3553	0.4287	1.1099	0.046*	
C11	0.31982 (8)	0.3557 (6)	1.1336 (7)	0.0421 (11)	
H11	0.3163	0.4286	1.2472	0.051*	
C12	0.30174 (8)	0.2492 (6)	1.0509 (7)	0.0396 (11)	
C13	0.30695 (8)	0.1451 (6)	0.8808 (7)	0.0429 (11)	
H13	0.2945	0.0744	0.8220	0.051*	
C14	0.33044 (8)	0.1440 (6)	0.7956 (7)	0.0402 (11)	
H14	0.3337	0.0711	0.6816	0.048*	
C15	0.27188 (10)	0.3404 (9)	1.3020 (9)	0.0772 (19)	
H15A	0.2819	0.3011	1.4157	0.116*	

H15B	0.2547	0.3230	1.3352	0.116*
H15C	0.2749	0.4670	1.2755	0.116*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.034 (2)	0.052 (2)	0.047 (2)	−0.0017 (17)	−0.0002 (18)	−0.011 (2)
O1	0.0409 (19)	0.098 (3)	0.065 (2)	−0.0062 (19)	−0.0082 (19)	0.034 (2)
O2	0.0409 (19)	0.087 (3)	0.058 (2)	−0.0027 (18)	0.0128 (18)	0.022 (2)
O3	0.0356 (18)	0.075 (2)	0.060 (2)	−0.0044 (16)	0.0113 (16)	−0.013 (2)
C1	0.048 (3)	0.046 (3)	0.043 (3)	−0.003 (2)	−0.003 (2)	−0.007 (2)
C2	0.038 (2)	0.034 (2)	0.038 (2)	−0.0043 (19)	0.000 (2)	−0.003 (2)
C3	0.048 (3)	0.048 (3)	0.034 (2)	0.000 (2)	0.004 (2)	0.010 (2)
C4	0.037 (2)	0.044 (3)	0.038 (3)	0.002 (2)	0.0003 (19)	0.003 (2)
C5	0.040 (3)	0.043 (3)	0.043 (3)	0.000 (2)	0.005 (2)	0.006 (2)
C6	0.053 (3)	0.051 (3)	0.048 (3)	0.001 (2)	0.002 (2)	0.016 (3)
C7	0.039 (2)	0.047 (3)	0.050 (3)	0.007 (2)	−0.003 (2)	0.003 (2)
C8	0.042 (3)	0.107 (5)	0.082 (4)	−0.008 (3)	0.005 (3)	0.037 (4)
C9	0.034 (2)	0.034 (2)	0.035 (2)	0.0020 (18)	−0.002 (2)	0.002 (2)
C10	0.042 (3)	0.036 (2)	0.037 (2)	−0.002 (2)	−0.008 (2)	−0.005 (2)
C11	0.042 (3)	0.047 (3)	0.037 (2)	0.006 (2)	−0.004 (2)	−0.006 (2)
C12	0.034 (2)	0.041 (3)	0.043 (3)	0.002 (2)	−0.001 (2)	−0.003 (2)
C13	0.034 (2)	0.048 (3)	0.046 (3)	−0.005 (2)	−0.002 (2)	−0.009 (2)
C14	0.039 (2)	0.044 (3)	0.037 (3)	0.000 (2)	0.003 (2)	−0.015 (2)
C15	0.056 (3)	0.098 (5)	0.077 (4)	−0.002 (3)	0.026 (3)	−0.030 (4)

Geometric parameters (Å, °)

N1—C9	1.396 (5)	C6—H6	0.9300
N1—C1	1.445 (6)	C7—H7	0.9300
N1—H1A	0.9000	C8—O2^{i}	3.074 (7)
N1—H1B	0.9000	C8—O1^{ii}	3.267 (7)
O1—C4	1.378 (5)	C8—H8A	0.9700
O1—C8	1.408 (6)	C8—H8B	0.9700
O2—C5	1.371 (5)	C9—C10	1.391 (6)
O2—C8	1.405 (6)	C9—C14	1.396 (6)
O3—C12	1.373 (5)	C10—C11	1.376 (6)
O3—C15	1.420 (6)	C10—H10	0.9300
C1—C2	1.491 (6)	C11—C12	1.362 (6)
C1—H1C	0.9700	C11—H11	0.9300
C1—H1D	0.9700	C12—C13	1.371 (6)
C2—C7	1.384 (6)	C13—C14	1.385 (6)
C2—C3	1.408 (6)	C13—H13	0.9300
C3—C4	1.357 (6)	C14—H14	0.9300
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.379 (6)	C15—H15B	0.9600
C5—C6	1.362 (6)	C15—H15C	0.9600
C6—C7	1.380 (6)		

C9—N1—C1	120.3 (4)	O2—C8—O1^{ii}	162.5 (4)
C9—N1—H1A	107.3	O1—C8—O1^{ii}	84.7 (3)
C1—N1—H1A	107.3	O2^{i}—C8—O1^{ii}	94.30 (17)
C9—N1—H1B	107.3	O2—C8—H8A	109.6
C1—N1—H1B	107.3	O1—C8—H8A	109.6
H1A—N1—H1B	106.9	O2^{i}—C8—H8A	81.0
C4—O1—C8	105.5 (4)	O1^{ii}—C8—H8A	55.3
C5—O2—C8	104.4 (4)	O2—C8—H8B	109.6
C12—O3—C15	117.5 (4)	O1—C8—H8B	109.6
N1—C1—C2	110.6 (4)	O2^{i}—C8—H8B	57.1
N1—C1—H1C	109.5	O1^{ii}—C8—H8B	71.4
C2—C1—H1C	109.5	H8A—C8—H8B	108.1
N1—C1—H1D	109.5	C10—C9—N1	120.4 (4)
C2—C1—H1D	109.5	C10—C9—C14	116.8 (4)
H1C—C1—H1D	108.1	N1—C9—C14	122.8 (4)
C7—C2—C3	118.4 (4)	C11—C10—C9	122.0 (4)
C7—C2—C1	121.3 (4)	C11—C10—H10	119.0
C3—C2—C1	120.2 (4)	C9—C10—H10	119.0
C4—C3—C2	117.3 (4)	C12—C11—C10	120.4 (4)
C4—C3—H3	121.4	C12—C11—H11	119.8
C2—C3—H3	121.4	C10—C11—H11	119.8
C3—C4—O1	128.4 (4)	C11—C12—C13	119.4 (4)
C3—C4—C5	123.1 (4)	C11—C12—O3	125.0 (4)
O1—C4—C5	108.6 (4)	C13—C12—O3	115.7 (4)
C6—C5—O2	127.9 (4)	C12—C13—C14	120.8 (4)
C6—C5—C4	121.0 (4)	C12—C13—H13	119.6
O2—C5—C4	111.1 (4)	C14—C13—H13	119.6
C5—C6—C7	116.5 (4)	C13—C14—C9	120.7 (4)
C5—C6—H6	121.8	C13—C14—H14	119.6
C7—C6—H6	121.8	C9—C14—H14	119.6
C6—C7—C2	123.7 (4)	O3—C15—H15A	109.5
C6—C7—H7	118.1	O3—C15—H15B	109.5
C2—C7—H7	118.1	H15A—C15—H15B	109.5
O2—C8—O1	110.5 (4)	O3—C15—H15C	109.5
O2—C8—O2^{i}	73.1 (3)	H15A—C15—H15C	109.5
O1—C8—O2^{i}	165.9 (5)	H15B—C15—H15C	109.5
C9—N1—C1—C2	171.2 (4)	C5—O2—C8—O1	0.7 (7)
N1—C1—C2—C7	−111.3 (5)	C5—O2—C8—O2^{i}	166.3 (3)
N1—C1—C2—C3	69.7 (5)	C5—O2—C8—O1^{ii}	−148.4 (13)
C7—C2—C3—C4	0.0 (6)	C4—O1—C8—O2	−1.0 (7)
C1—C2—C3—C4	179.0 (4)	C4—O1—C8—O2^{i}	−103.3 (15)
C2—C3—C4—O1	179.8 (5)	C4—O1—C8—O1^{ii}	170.0 (3)
C2—C3—C4—C5	0.3 (7)	C1—N1—C9—C10	−177.3 (4)
C8—O1—C4—C3	−178.6 (5)	C1—N1—C9—C14	1.8 (7)
C8—O1—C4—C5	0.9 (6)	N1—C9—C10—C11	178.8 (4)
C8—O2—C5—C6	179.2 (5)	C14—C9—C10—C11	−0.4 (7)

C8—O2—C5—C4	−0.1 (6)	C9—C10—C11—C12	0.8 (7)
C3—C4—C5—C6	−0.4 (7)	C10—C11—C12—C13	−1.3 (7)
O1—C4—C5—C6	−179.9 (5)	C10—C11—C12—O3	179.2 (4)
C3—C4—C5—O2	179.0 (4)	C15—O3—C12—C11	−1.7 (7)
O1—C4—C5—O2	−0.5 (5)	C15—O3—C12—C13	178.9 (5)
O2—C5—C6—C7	−179.2 (5)	C11—C12—C13—C14	1.5 (7)
C4—C5—C6—C7	0.1 (7)	O3—C12—C13—C14	−179.0 (4)
C5—C6—C7—C2	0.2 (7)	C12—C13—C14—C9	−1.1 (7)
C3—C2—C7—C6	−0.2 (7)	C10—C9—C14—C13	0.5 (7)
C1—C2—C7—C6	−179.3 (4)	N1—C9—C14—C13	−178.6 (4)

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2; (ii) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15B···O3^{iii}	0.96	2.61	3.454 (6)	147
C3—H3···Cg1^{iv}	0.93	2.82	3.64 (2)	148
C6—H6···Cg1^{v}	0.93	2.75	3.55 (2)	145
C11—H11···Cg2^{vi}	0.93	2.80	3.63 (2)	150
C14—H14···Cg2^{vii}	0.93	2.76	3.59 (2)	149

Symmetry codes: (iii) −*x*+1/2, −*y*+1/2, *z*+1/2; (iv) *x*, −*y*, *z*+1/2; (v) *x*, −*y*+1, *z*−1/2; (vi) *x*, −*y*+1, *z*+1/2; (vii) *x*, −*y*, *z*−1/2.

Fig. 1

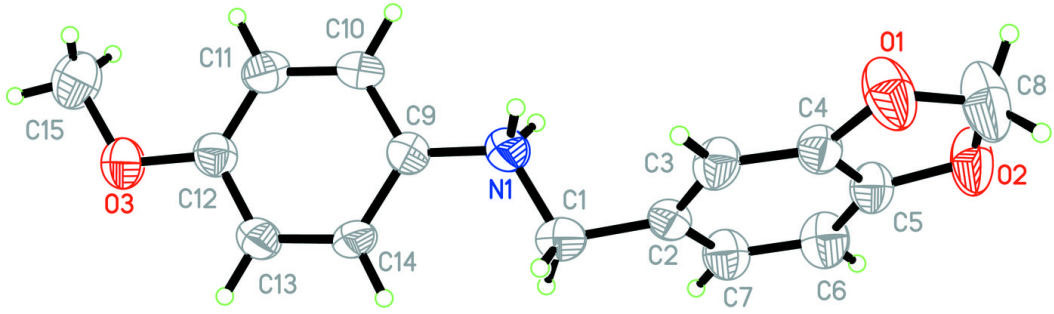

Fig. 2