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2-Methoxybenzaldehyde azine

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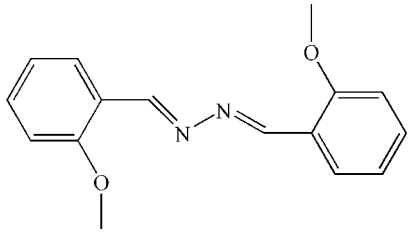
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.166; data-to-parameter ratio = 18.0.

The molecule of the title compound, $C_{16}H_{16}N_2O_2$, is located on a centre of inversion (at the midpoint of the N—N bond), so that only one half of the molecule is crystallographically independent, and it adopts a *syn* structure with respect to the methoxy group and the aldehyde H atom. The benzene ring and adjacent N atom are coplanar (r.m.s. deviation of 0.017 Å for the non-H atoms). The methoxy group deviates from the benzene plane by 0.167 (4) Å for the methyl C atom.

Related literature

For related literature, see: Allen *et al.* (1987); Amadei *et al.* (1998); Glaser *et al.* (1995); Hsu *et al.* (1993); Xu *et al.* (1994).



Experimental

Crystal data

$C_{16}H_{16}N_2O_2$
M_r = 268.31
Monoclinic, $P2_1/c$
a = 7.755 (5) Å
b = 14.757 (12) Å
c = 6.889 (5) Å
β = 112.64 (3)°
V = 727.6 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.26 × 0.21 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: none
6946 measured reflections
1659 independent reflections
1203 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.166
S = 1.01
1659 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2015).

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supplementary materials

Acta Cryst. (2007). E**63**, o2993 [**doi:10.1107/S1600536807024865**]

2-Methoxybenzaldehyde azine

Z.-W. Fu

Comment

Azines represent a well known class of organic compounds, obtained from the condensation of an aldehyde or ketone with hydrazine (Glaser *et al.*, 1995; Amadei *et al.*, 1998). As a extension of work on the structure characterization of azines, we report here the crystal structure of compound, 2,2'-di(*N*)-methoxybenzaldehyde azine (I).

The molecule geometry of (I) is illustrated is Fig. 1. A l l bond lengths are within in normal ranges (Allen *et al.*, 1987). The molecule is plased on the centre of inversion (the middle N1—N1A bond) so that only half of the molecule of crystallographically independent. The methoxy group is *syn* to the H atom on C7, so the molecule adopts a *syn* configuration in solid state (Xu *et al.*, 1994; Hsu & Nordman, 1993). The N1—C7 (1.269 (2) Å) and N1—N1A (1.403 (2) Å) distances indicate these correspond to double and single bonds, respectively. The torsion angle N1—C7—C1—C2 (−176.4 (1) °) indicates the molecule is practically planar. The maximum deviation from the mean phenyl plane is 0.017 Å for non–hydrogen atoms. The atom H7 and O1 atom of methoxy group form contact 2.401 Å and atom C8 of methoxy group displaced out of the phenyl plane on −0.167 (4) Å. The packing diagram of (I) is showed in Fig. 2.

Experimental

The reagents are commercial products and used without further purification. 2-methoxybenzaldehyde (0.2 mmol, 28.4 mg) and the hydrazine (0.1 mmol, about 5.0 mg) were dissolved in ethanol (99%, 15 ml). The reaction mixture was stirring for 1 h to give a clear solution at room temperature. After allowing the solution to stand at room temperature in air for a week, large yellow crystals were isolated. The crystals were flitrated, washed three times with ethanol and dried in air, yield 82.4%.

Refinement

The methyl H atoms were positioned geometrically and treated as riding (C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)); other H atoms were positioned geometrically and treated as riding (C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C)).

Figures

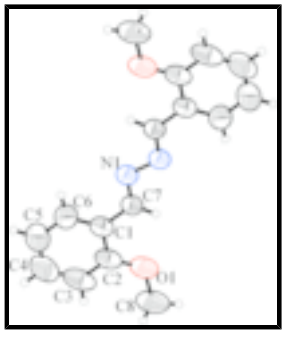

Fig. 1. The structure (I) showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability lavel. The H atoms are drawn as sphere with arbitrary radius.

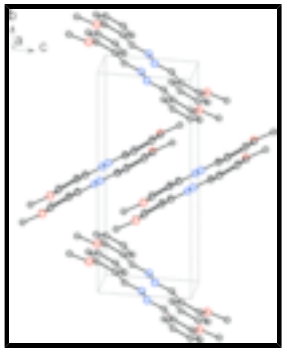

Fig. 2. Packing diagram of (I). The H atoms have been omitted for clarity.

2-Methoxybenzaldehyde azine

Crystal data

$C_{16}H_{16}N_2O_2$	F_{000} = 284
M_r = 268.31	D_x = 1.225 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5435 reflections
a = 7.755 (5) Å	θ = 3.2–27.5º
b = 14.757 (12) Å	μ = 0.08 mm^{-1}
c = 6.889 (5) Å	T = 293 (2) K
β = 112.64 (3)º	Block, yellow
V = 727.6 (9) Å^3	0.26 × 0.21 × 0.18 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID diffractometer	1203 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.035
Monochromator: graphite	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 3.2º
oscillation scans	h = −10→9
Absorption correction: none	k = −19→19
6946 measured reflections	l = −8→8
1659 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.046	$w = 1/[\sigma^2(F_o^2) + (0.1031P)^2 + 0.0369P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.166	$(\Delta/\sigma)_{max} < 0.001$

S = 1.01	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
1659 reflections	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
92 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001 \times Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.23 (3)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\sigma(F^2)$ 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}
C1	0.1164 (2)	0.08853 (9)	0.1805 (2)	0.0613 (4)
C2	0.0362 (2)	0.13640 (9)	−0.0083 (2)	0.0678 (4)
C3	0.1480 (4)	0.16846 (13)	−0.1093 (3)	0.0966 (7)
H3A	0.0958	0.2016	−0.2332	0.116*
C4	0.3364 (4)	0.1510 (2)	−0.0251 (4)	0.1257 (10)
H4A	0.4111	0.1711	−0.0944	0.151*
C5	0.4147 (3)	0.1046 (2)	0.1579 (4)	0.1267 (9)
H5A	0.5427	0.0938	0.2141	0.152*
C6	0.3058 (2)	0.07374 (15)	0.2604 (3)	0.0903 (6)
H6A	0.3612	0.0422	0.3862	0.108*
C7	−0.00032 (18)	0.05390 (9)	0.28700 (19)	0.0577 (4)
H7A	−0.1292	0.0612	0.2234	0.069*
C8	−0.2450 (4)	0.18658 (18)	−0.2825 (3)	0.1183 (8)
H8A	−0.3763	0.1911	−0.3125	0.177*
H8B	−0.2260	0.1487	−0.3858	0.177*
H8C	−0.1954	0.2459	−0.2863	0.177*
N1	0.06864 (15)	0.01420 (8)	0.46363 (16)	0.0628 (4)
O1	−0.15225 (19)	0.14829 (8)	−0.07995 (17)	0.0871 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0803 (9)	0.0508 (7)	0.0577 (7)	−0.0112 (5)	0.0321 (6)	−0.0054 (5)
C2	0.1069 (11)	0.0475 (7)	0.0580 (7)	−0.0068 (6)	0.0416 (7)	−0.0048 (5)
C3	0.1614 (19)	0.0734 (10)	0.0744 (10)	−0.0385 (11)	0.0670 (11)	−0.0075 (8)

C4	0.1358 (19)	0.161 (2)	0.1059 (16)	−0.0805 (17)	0.0745 (15)	−0.0260 (15)
C5	0.0896 (13)	0.185 (2)	0.1142 (17)	−0.0513 (14)	0.0486 (12)	−0.0095 (16)
C6	0.0744 (10)	0.1140 (14)	0.0814 (11)	−0.0237 (9)	0.0288 (8)	0.0008 (9)
C7	0.0667 (7)	0.0543 (7)	0.0536 (7)	0.0026 (5)	0.0248 (6)	0.0042 (5)
C8	0.1629 (19)	0.1248 (17)	0.0738 (11)	0.0578 (15)	0.0529 (11)	0.0415 (11)
N1	0.0672 (7)	0.0695 (7)	0.0532 (6)	−0.0008 (5)	0.0249 (5)	0.0069 (5)
O1	0.1201 (10)	0.0851 (8)	0.0633 (7)	0.0299 (6)	0.0433 (6)	0.0244 (5)

Geometric parameters (Å, °)

C1—C6	1.373 (2)	C5—H5A	0.9300
C1—C2	1.398 (2)	C6—H6A	0.9300
C1—C7	1.4590 (19)	C7—N1	1.2684 (17)
C2—O1	1.362 (2)	C7—H7A	0.9300
C2—C3	1.388 (2)	C8—O1	1.418 (2)
C3—C4	1.373 (4)	C8—H8A	0.9600
C3—H3A	0.9300	C8—H8B	0.9600
C4—C5	1.355 (4)	C8—H8C	0.9600
C4—H4A	0.9300	N1—N1^{i}	1.404 (2)
C5—C6	1.370 (3)		
C6—C1—C2	118.49 (14)	C5—C6—C1	121.1 (2)
C6—C1—C7	121.18 (14)	C5—C6—H6A	119.4
C2—C1—C7	120.32 (14)	C1—C6—H6A	119.4
O1—C2—C3	124.40 (16)	N1—C7—C1	122.00 (13)
O1—C2—C1	115.69 (13)	N1—C7—H7A	119.0
C3—C2—C1	119.92 (17)	C1—C7—H7A	119.0
C4—C3—C2	119.54 (19)	O1—C8—H8A	109.5
C4—C3—H3A	120.2	O1—C8—H8B	109.5
C2—C3—H3A	120.2	H8A—C8—H8B	109.5
C5—C4—C3	120.68 (18)	O1—C8—H8C	109.5
C5—C4—H4A	119.7	H8A—C8—H8C	109.5
C3—C4—H4A	119.7	H8B—C8—H8C	109.5
C4—C5—C6	120.2 (2)	C7—N1—N1^{i}	112.50 (13)
C4—C5—H5A	119.9	C2—O1—C8	118.37 (15)
C6—C5—H5A	119.9		
C6—C1—C2—O1	179.56 (13)	C4—C5—C6—C1	0.2 (4)
C7—C1—C2—O1	0.69 (18)	C2—C1—C6—C5	−0.3 (3)
C6—C1—C2—C3	−0.6 (2)	C7—C1—C6—C5	178.59 (19)
C7—C1—C2—C3	−179.48 (13)	C6—C1—C7—N1	4.8 (2)
O1—C2—C3—C4	−178.68 (17)	C2—C1—C7—N1	−176.39 (12)
C1—C2—C3—C4	1.5 (3)	C1—C7—N1—N1^{i}	−179.52 (12)
C2—C3—C4—C5	−1.5 (3)	C3—C2—O1—C8	7.6 (2)
C3—C4—C5—C6	0.7 (4)	C1—C2—O1—C8	−172.55 (16)

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

Fig. 1

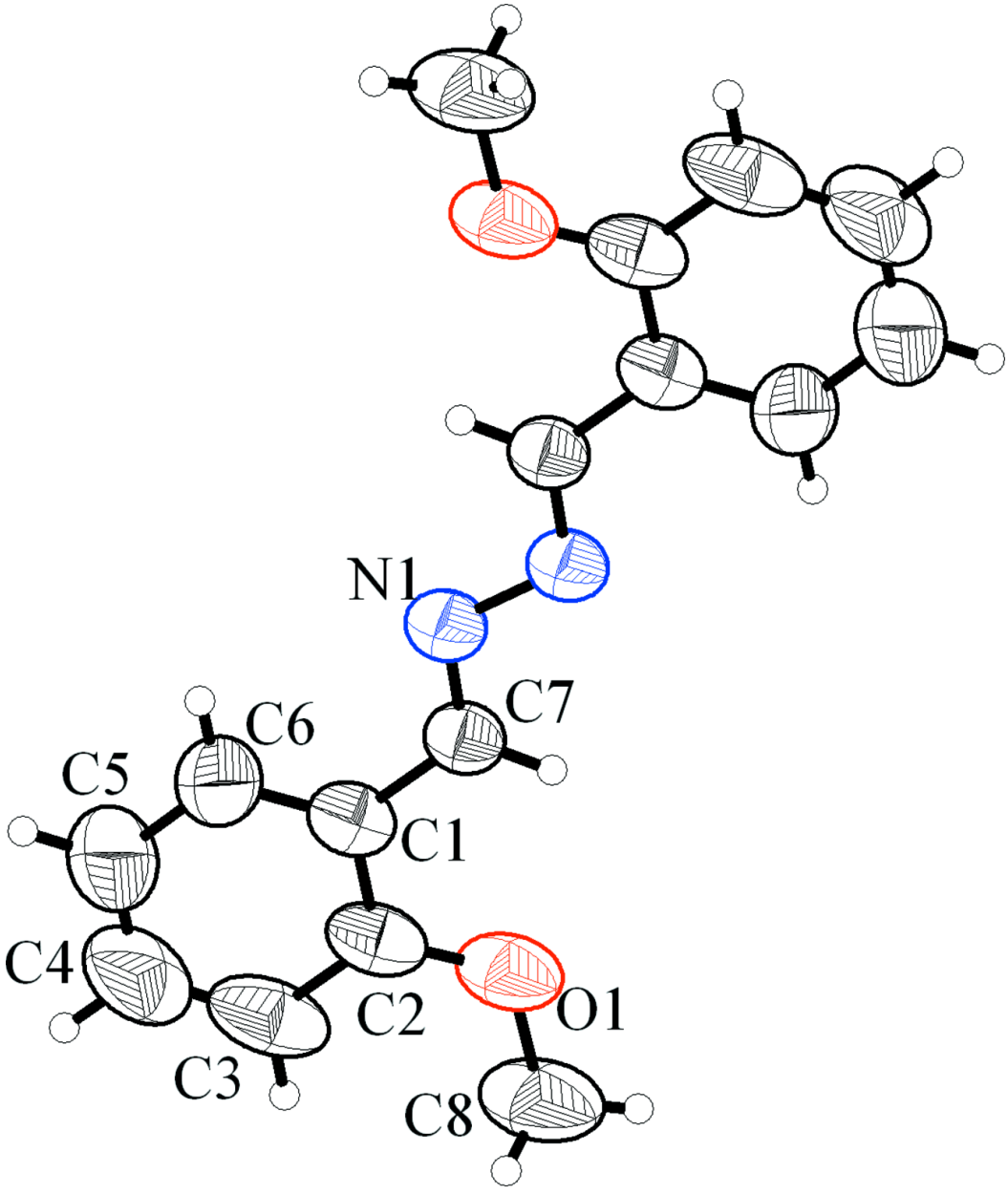

Fig. 2