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2,4-Dimethoxybenzaldehyde azine

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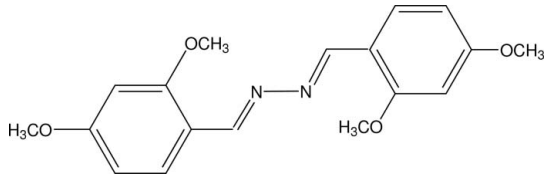
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.121; data-to-parameter ratio = 11.8.

The title molecule, $C_{18}H_{20}N_2O_4$, is located on a crystallographic centre of symmetry. The methoxy groups are coplanar with the benzene ring [interplanar angles of 14.4 (2) and 3.1 (3)°], indicating a conjugation effect.

Related literature

For the structures of related compounds, see: Narayana *et al.* (2007); Liu *et al.* (2007); Takakashi *et al.* (2006). For a statistical study of methoxy groups bound to phenyl rings, see: Hummel *et al.* (1988).



Experimental

Crystal data

$C_{18}H_{20}N_2O_4$
M_r = 328.36
Monoclinic, $P2_1/c$
a = 6.775 (2) Å
b = 9.014 (3) Å
c = 14.081 (3) Å
β = 100.42 (2)°
V = 845.7 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.45 × 0.30 × 0.12 mm

Data collection

Enraf–Nonius DIP1030 image-plate diffractometer
Absorption correction: none
3535 measured reflections
1293 independent reflections
719 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 24.1°

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.121
S = 0.84
1293 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

Data collection: *XPRESS* (MacScience, 2002; cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2359).

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supporting information

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2,4-Dimethoxybenzaldehyde azine

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S1. Comment

The molecule of the title compound (Fig. 1) possesses a crystallographically imposed inversion centre at the midpoint of the N—N bond. Thus the conformation about the N1—N1^{i} azine bridge (symmetry code: (i) -*x* - 1, -*y* + 1, -*z* + 1) is *trans*, with a torsion angle C1—N1—N1′—C1′ of 180°. The molecule has coplanar non hydrogen atoms as found in other similar structures (Narayana *et al.*, 2007; Takakashi *et al.*, 2006). The coplanarity of methoxy groups with the benzene ring suggests the presence of a conjugation effect (Hummel *et al.*, 1988). The bond distance N1—N1^{i} of 1.414 (4) Å, is comparable to those measured in the structure of methoxybenzaldehyde azine and *N*,*N*′-bis(4-hydroxybenzylidene)-hydrazine, which are 1.418 and 1.415 Å, respectively (Narayana *et al.*, 2007; Liu *et al.*, 2007). The crystal packing does not show any π-π stacking interactions between aromatic rings.

S2. Experimental

Hydrazine hydrate (99.99%) (0.13 g, 2.5 mmol) was added to a solution of 2,4-dimethoxybenzaldehyde (0.83 g, 5 mmol) in methanol (10 ml) with constant stirring for 5 min, at room temperature. A light yellow precipitate was formed, which was separated by filtration, washed with methanol and dried at 70°C for 1 h and then preserved *in vacuo* over anhydrous $CaCl_2$ (0.43 g, 44.79%, m.p. 473 K). The compound was soluble in chloroform, benzene and toluene whereas insoluble in acetonitrile, methanol and ethanol. The compound (0.37 g) was dissolved in chloroform (60 ml) at boiling temperature and allowed to stand at room temperature (20–25°C). Bright large yellow crystals, suitable for X-ray single-crystal analysis, were obtained after 7 days. The crystals were collected, washed with absolute ethanol and dried *in vacuo* over anhydrous $CaCl_2$.

S3. Refinement

All H atoms were located geometrically and treated as riding atoms, with C—H = 0.93–0.96 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms.

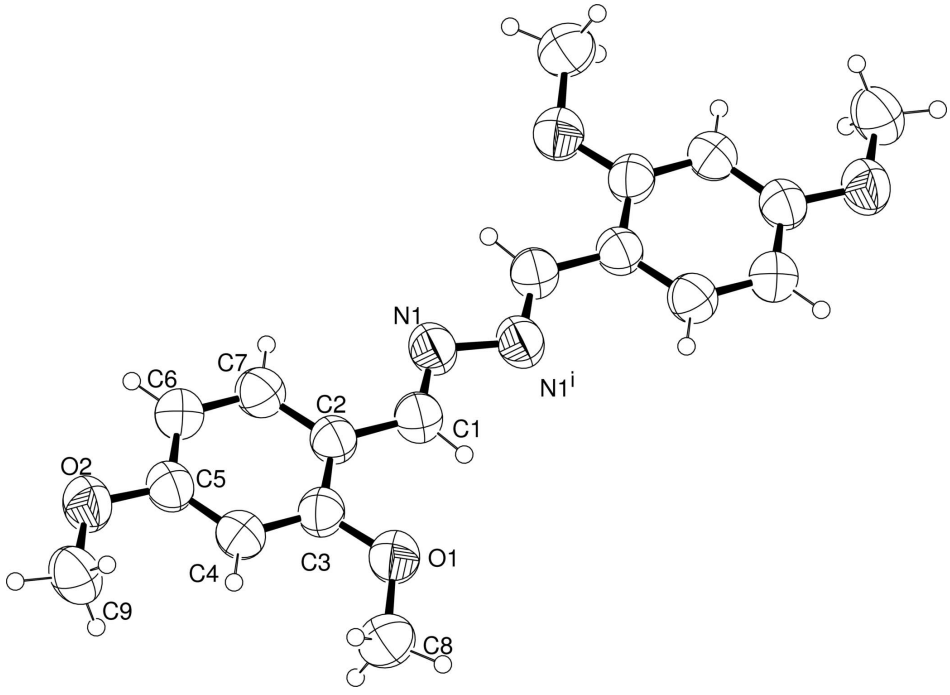

Figure 1

ORTEP drawing (ellipsoids at the 40% probability level) of the title compound with atom labelling scheme. Primed atom at -*x* - 1, -*y* + 1, -*z* + 1.

2,4-Dimethoxybenzaldehyde azine

Crystal data

$C_{18}H_{20}N_2O_4$
M_r = 328.36
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 6.775 (2) Å
b = 9.014 (3) Å
c = 14.081 (3) Å
β = 100.42 (2)°
V = 845.7 (4) Å^3
Z = 2
$F(000)$ = 348
D_x = 1.289 Mg m^{-3}
Melting point: 475 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 482 reflections
θ = 3.6–20.7°
μ = 0.09 mm^{-1}
T = 293 K
Plate, yellow
0.45 × 0.30 × 0.12 mm

Data collection

Enraf–Nonius DIP1030 image-plate diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ–scans with narrow frames
3535 measured reflections
1293 independent reflections
719 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 24.1°, θ_{min} = 2.9°
h = −7→7
k = −10→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.121$
$S = 0.84$
1293 reflections
110 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.10$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.070 (13)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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}
N1	−0.4202 (3)	0.4904 (2)	0.53948 (13)	0.0857 (6)
O1	0.0297 (2)	0.77203 (18)	0.52320 (12)	0.0960 (6)
O2	0.4398 (2)	0.55904 (18)	0.80720 (11)	0.0952 (6)
C1	−0.2709 (4)	0.5738 (3)	0.53259 (16)	0.0833 (7)
H1	−0.2800	0.6376	0.4800	0.100*
C2	−0.0880 (3)	0.5722 (2)	0.60384 (15)	0.0745 (6)
C3	0.0667 (3)	0.6732 (2)	0.59790 (16)	0.0768 (6)
C4	0.2433 (3)	0.6732 (2)	0.66487 (16)	0.0785 (7)
H4	0.3431	0.7425	0.6606	0.094*
C5	0.2699 (3)	0.5695 (3)	0.73791 (16)	0.0787 (6)
C6	0.1196 (4)	0.4673 (3)	0.74624 (17)	0.0850 (7)
H6	0.1372	0.3984	0.7962	0.102*
C7	−0.0547 (3)	0.4704 (3)	0.67925 (18)	0.0830 (7)
H7	−0.1546	0.4017	0.6845	0.100*
C8	0.1918 (4)	0.8551 (3)	0.49880 (19)	0.1041 (9)
H8A	0.1429	0.9185	0.4449	0.156*
H8B	0.2905	0.7885	0.4820	0.156*
H8C	0.2514	0.9144	0.5531	0.156*
C9	0.5936 (3)	0.6675 (3)	0.80431 (19)	0.1042 (9)
H9A	0.7046	0.6488	0.8557	0.156*
H9B	0.5410	0.7648	0.8118	0.156*
H9C	0.6381	0.6614	0.7435	0.156*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0716 (13)	0.0972 (13)	0.0854 (14)	0.0055 (12)	0.0066 (9)	−0.0007 (11)
O1	0.0858 (11)	0.1004 (12)	0.0995 (12)	−0.0022 (9)	0.0106 (9)	0.0169 (10)
O2	0.0820 (11)	0.1060 (13)	0.0915 (12)	−0.0027 (9)	−0.0012 (9)	0.0038 (9)
C1	0.0778 (16)	0.0904 (16)	0.0803 (15)	0.0037 (13)	0.0101 (13)	0.0021 (12)
C2	0.0693 (14)	0.0799 (14)	0.0732 (14)	0.0017 (12)	0.0099 (12)	−0.0017 (12)
C3	0.0782 (15)	0.0787 (15)	0.0728 (14)	0.0082 (13)	0.0118 (13)	0.0026 (12)
C4	0.0734 (15)	0.0799 (15)	0.0818 (15)	−0.0017 (12)	0.0132 (13)	−0.0029 (13)
C5	0.0722 (15)	0.0864 (15)	0.0755 (15)	0.0053 (13)	0.0084 (13)	−0.0065 (13)
C6	0.0806 (16)	0.0919 (16)	0.0815 (16)	0.0009 (13)	0.0114 (14)	0.0056 (13)
C7	0.0772 (16)	0.0870 (16)	0.0841 (16)	−0.0032 (12)	0.0126 (13)	−0.0010 (13)
C8	0.1061 (19)	0.0956 (17)	0.113 (2)	−0.0067 (16)	0.0255 (16)	0.0164 (15)
C9	0.0775 (16)	0.115 (2)	0.114 (2)	−0.0073 (15)	0.0006 (15)	−0.0033 (16)

Geometric parameters (Å, º)

N1—C1	1.278 (3)	C4—H4	0.9300
N1—N1^{i}	1.414 (4)	C5—C6	1.393 (3)
O1—C3	1.366 (2)	C6—C7	1.372 (3)
O1—C8	1.422 (3)	C6—H6	0.9300
O2—C5	1.371 (3)	C7—H7	0.9300
O2—C9	1.435 (3)	C8—H8A	0.9600
C1—C2	1.446 (3)	C8—H8B	0.9600
C1—H1	0.9300	C8—H8C	0.9600
C2—C7	1.391 (3)	C9—H9A	0.9600
C2—C3	1.402 (3)	C9—H9B	0.9600
C3—C4	1.383 (3)	C9—H9C	0.9600
C4—C5	1.378 (3)		
C1—N1—N1^{i}	111.8 (2)	C7—C6—C5	118.7 (2)
C3—O1—C8	119.13 (18)	C7—C6—H6	120.7
C5—O2—C9	116.98 (19)	C5—C6—H6	120.7
N1—C1—C2	122.2 (2)	C6—C7—C2	122.6 (2)
N1—C1—H1	118.9	C6—C7—H7	118.7
C2—C1—H1	118.9	C2—C7—H7	118.7
C7—C2—C3	117.0 (2)	O1—C8—H8A	109.5
C7—C2—C1	122.4 (2)	O1—C8—H8B	109.5
C3—C2—C1	120.5 (2)	H8A—C8—H8B	109.5
O1—C3—C4	122.7 (2)	O1—C8—H8C	109.5
O1—C3—C2	115.7 (2)	H8A—C8—H8C	109.5
C4—C3—C2	121.5 (2)	H8B—C8—H8C	109.5
C5—C4—C3	119.3 (2)	O2—C9—H9A	109.5
C5—C4—H4	120.4	O2—C9—H9B	109.5
C3—C4—H4	120.4	H9A—C9—H9B	109.5
O2—C5—C4	123.9 (2)	O2—C9—H9C	109.5
O2—C5—C6	115.3 (2)	H9A—C9—H9C	109.5

C4—C5—C6	120.8 (2)	H9B—C9—H9C	109.5
N1^{i}—N1—C1—C2	−178.8 (2)	C2—C3—C4—C5	1.5 (3)
N1—C1—C2—C7	6.4 (3)	C9—O2—C5—C4	2.6 (3)
N1—C1—C2—C3	−175.1 (2)	C9—O2—C5—C6	−176.66 (18)
C8—O1—C3—C4	15.1 (3)	C3—C4—C5—O2	179.32 (18)
C8—O1—C3—C2	−166.0 (2)	C3—C4—C5—C6	−1.5 (3)
C7—C2—C3—O1	−179.90 (18)	O2—C5—C6—C7	−179.73 (19)
C1—C2—C3—O1	1.5 (3)	C4—C5—C6—C7	1.0 (3)
C7—C2—C3—C4	−1.0 (3)	C5—C6—C7—C2	−0.5 (3)
C1—C2—C3—C4	−179.62 (19)	C3—C2—C7—C6	0.5 (3)
O1—C3—C4—C5	−179.68 (18)	C1—C2—C7—C6	179.1 (2)

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