(E)-4-Bromo-N-(2,4-dimethoxybenzyl-idene)aniline

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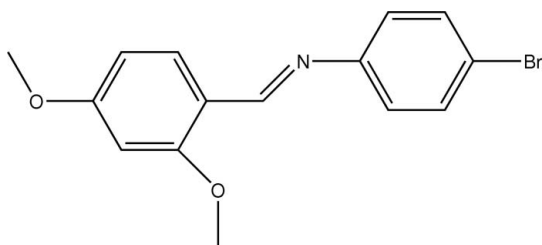Correspondence e-mail: jsimpson@alkali.otago.ac.nz

The title Schiff base compound, $C_{15}H_{14}BrNO_2$, adopts an E configuration with respect to the C=N bond. The C and O atoms of the two methoxy substituents lie very close to the dimethoxyphenyl ring plane [maximum deviation = 0.17 (1) Å]. The dihedral angle between the two aromatic rings is 43.69 (16)°, while the plane through the central C—C=N—C system is inclined at 10.6 (6)° to the dimethoxyphenyl ring and 34.6 (3)° to the bromophenyl ring. In the crystal structure, each molecule is involved in the formation of two inversion-related dimers through weak C—H⋯N and C—H⋯O interactions, respectively. These contacts link the molecules into independent rows parallel to the b axis.

Related literature

For related structures, see: Khalaji et al. (2007); Khalaji & Harrison (2008); Khalaji & Simpson (2009). For reference structural data, see: Allen et al. (1987). For graph-set motifs, see: Bernstein et al. (1995).

Experimental

Crystal data

$C_{15}H_{14}BrNO_2$	$a = 4.1323$ (6) Å
$M_r = 320.18$	$b = 10.7406$ (14) Å
Monoclinic, $P2_1/c$	$c = 29.911$ (4) Å
$\beta = 90.992$ (8)°	$\mu = 3.09$ mm^{-1}
$V = 1327.4$ (3) Å^3	$T = 89$ K
$Z = 4$	$0.25 \times 0.10 \times 0.02$ mm
Mo $K\alpha$ radiation	

Data collection

Bruker APEXII CCD area-detector diffractometer	13728 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2006)	2390 independent reflections
$T_{min} = 0.570$, $T_{max} = 0.940$	1664 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.106$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$	174 parameters
$wR(F^2) = 0.118$	H-atom parameters constrained
$S = 1.21$	$\Delta\rho_{max} = 0.83$ e Å^{-3}
2390 reflections	$\Delta\rho_{min} = -0.82$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7A⋯N1[i]	0.98	2.74	3.667 (7)	159
C4—H4C⋯O2[ii]	0.98	2.54	3.398 (6)	145

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 1, -y + 2, -z + 1$.

Data collection: APEX2 (Bruker, 2006); cell refinement: APEX2 and SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and TITAN2000 (Hunter & Simpson, 1999); molecular graphics: SHELXTL (Sheldrick, 2008) and Mercury (Macrae et al., 2006); software used to prepare material for publication: SHELXL97, enCIFer (Allen et al., 2004), PLATON (Spek, 2009) and publCIF (Westrip, 2009).

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supporting information

(*E*)-4-Bromo-*N*-(2,4-dimethoxybenzylidene)aniline

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S1. Comment

As a continuation of our work on the synthesis and structural characterization of Schiff-base compounds (Khalaji *et al.*, 2007; Khalaji & Harrison, 2008; Khalaji & Simpson, 2009), we report here the structure of the title compound, $C_{15}H_{14}BrNO_2$, (I), Fig 1.

The compound adopts an *E* configuration with respect to the C1=N1 bond. The C4, O1 and C7 O2 methoxy substituents lie close to the plane of the C2···C9 ring (maximum deviation 0.17 (1) Å for C7. Bond lengths in the molecule are normal (Allen, *et al.*, 1987) and similar to those found in related compounds (Khalaji *et al.*, 2007; Khalaji & Harrison, 2008; Khalaji & Simpson 2009). The dihedral angle between the two aromatic rings is 43.69 (16) ° while the plane through the central C2—C2?N1—C10 system is inclined at 10.6 (6)° to the dimethoxyphenyl ring and 34.6 (3)° to the bromobenzene ring.

In the crystal structure, each molecule is involved in the formation of two inversion related dimers with $R^2_2(18)$ and $R^2_2(14)$ ring motifs (Bernstein *et al.* 1995) through weak C7—H7A···N1 and C4—H4···O2 interactions respectively, Table 1. These contacts link the molecules into independent rows parallel to the *b* axis, Fig. 2.

S2. Experimental

To a solution of 2,4-Dimethoxy benzaldehyde (332 mg, 0.2 mmol) in methanol (5 ml), cooled in an ice bath, a solution of 4-bromoaniline (344 mg, 0.2 mmol) in methanol (5 ml) was added slowly dropwise with constant stirring (1 h) at 298 k in the presence of molecular sieves. The mixture was filtered and the solution cooled to 273 K to give the compound in about 85% yield. Pale yellow crystals were grown from methanol.

S3. Refinement

H-atoms were refined using a riding model with d(C—H) = 0.95 Å, U_{iso}= 1.2U_{eq} (C) for aromatic and 0.98 Å, U_{iso} = 1.5U_{eq} (C) for CH$_3$ H atoms.
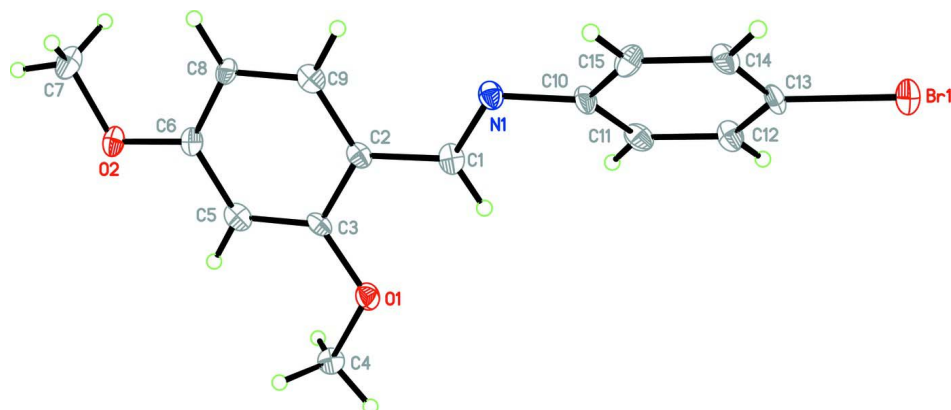
Figure 1

The structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level.
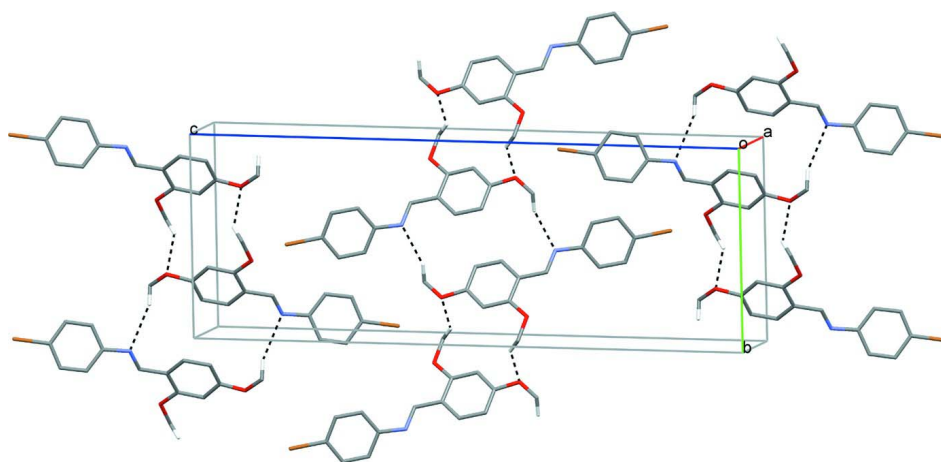
Figure 2

Crystal packing of (I) viewed down the *a* axis with hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

(*E*)-4-Bromo-*N*-(2,4-dimethoxybenzylidene)aniline

Crystal data

$C_{15}H_{14}BrNO_2$	$F(000) = 648$
$M_r = 320.18$	$D_x = 1.602$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2351 reflections
$a = 4.1323$ (6) Å	$\theta = 2.7–23.6°$
$b = 10.7406$ (14) Å	$\mu = 3.09$ mm^{-1}
$c = 29.911$ (4) Å	$T = 89$ K
$\beta = 90.992$ (8)°	Rectangular plate, pale yellow
$V = 1327.4$ (3) Å^3	$0.25 \times 0.10 \times 0.02$ mm
$Z = 4$	

Data collection

Bruker APEXII CCD area-detector diffractometer	13728 measured reflections
Radiation source: fine-focus sealed tube	2390 independent reflections
Graphite monochromator	1664 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.106$
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)	$\theta_{max} = 25.3°$, $\theta_{min} = 2.0°$
	$h = -3 \rightarrow 4$
$T_{min} = 0.570$, $T_{max} = 0.940$	$k = -12 \rightarrow 12$
	$l = -35 \rightarrow 35$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.056$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.118$	
$S = 1.21$	H-atom parameters constrained
2390 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0212P)^2 + 3P]$
174 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.83$ e Å⁻³
	$\Delta\rho_{min} = -0.82$ e Å⁻³

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
N1	0.8952 (10)	0.5770 (4)	0.37825 (15)	0.0157 (10)
C1	0.7282 (12)	0.6745 (5)	0.38778 (18)	0.0154 (12)
H1	0.6759	0.7321	0.3647	0.018*
C2	0.6171 (12)	0.6989 (5)	0.43302 (17)	0.0138 (12)
C3	0.4148 (12)	0.8016 (5)	0.44237 (18)	0.0133 (12)
O1	0.3071 (9)	0.8689 (3)	0.40592 (12)	0.0174 (9)
C4	0.1171 (13)	0.9771 (4)	0.41475 (18)	0.0159 (12)
H4A	−0.0826	0.9525	0.4296	0.024*
H4B	0.0632	1.0192	0.3865	0.024*
H4C	0.2406	1.0339	0.4342	0.024*
C5	0.3327 (12)	0.8293 (5)	0.48586 (18)	0.0155 (12)
H5	0.2003	0.8994	0.4919	0.019*
C6	0.4459 (12)	0.7535 (5)	0.52094 (17)	0.0138 (12)
O2	0.3510 (9)	0.7888 (3)	0.56290 (11)	0.0167 (9)
C7	0.4941 (14)	0.7232 (5)	0.59995 (18)	0.0222 (13)
H7A	0.4270	0.6357	0.5989	0.033*

H7B	0.4228	0.7607	0.6280	0.033*
H7C	0.7304	0.7283	0.5984	0.033*
C8	0.6428 (12)	0.6515 (5)	0.51264 (17)	0.0153 (12)
H8	0.7184	0.5999	0.5364	0.018*
C9	0.7263 (12)	0.6267 (5)	0.46874 (18)	0.0160 (12)
H9	0.8634	0.5578	0.4629	0.019*
C10	1.0177 (13)	0.5654 (6)	0.33449 (18)	0.0169 (13)
C11	1.1201 (12)	0.6657 (5)	0.30882 (18)	0.0181 (13)
H11	1.0997	0.7481	0.3200	0.022*
C12	1.2516 (13)	0.6466 (5)	0.26700 (18)	0.0179 (12)
H12	1.3267	0.7153	0.2501	0.022*
C13	1.2726 (12)	0.5278 (5)	0.25015 (18)	0.0164 (13)
Br1	1.44896 (13)	0.49899 (7)	0.192736 (17)	0.02466 (19)
C14	1.1702 (12)	0.4254 (5)	0.27488 (19)	0.0172 (12)
H14	1.1866	0.3432	0.2633	0.021*
C15	1.0447 (13)	0.4469 (6)	0.31650 (18)	0.0176 (13)
H15	0.9738	0.3779	0.3336	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.018 (2)	0.013 (3)	0.017 (2)	0.000 (2)	0.004 (2)	−0.0016 (19)
C1	0.018 (3)	0.011 (3)	0.017 (3)	−0.005 (2)	0.002 (2)	−0.003 (2)
C2	0.014 (3)	0.009 (3)	0.018 (3)	−0.001 (2)	0.003 (2)	−0.002 (2)
C3	0.008 (3)	0.013 (3)	0.019 (3)	0.000 (2)	0.001 (2)	−0.003 (2)
O1	0.020 (2)	0.016 (2)	0.016 (2)	0.0038 (17)	0.0036 (16)	−0.0012 (16)
C4	0.022 (3)	0.006 (3)	0.021 (3)	0.001 (2)	0.002 (2)	0.002 (2)
C5	0.015 (3)	0.008 (3)	0.024 (3)	−0.003 (2)	0.003 (2)	−0.003 (2)
C6	0.016 (3)	0.012 (3)	0.014 (3)	−0.006 (2)	0.002 (2)	−0.002 (2)
O2	0.023 (2)	0.015 (2)	0.012 (2)	−0.0021 (17)	0.0038 (16)	−0.0013 (16)
C7	0.036 (3)	0.015 (3)	0.015 (3)	−0.001 (3)	0.004 (3)	0.000 (2)
C8	0.017 (3)	0.016 (3)	0.013 (3)	−0.002 (2)	0.003 (2)	0.006 (2)
C9	0.014 (3)	0.012 (3)	0.022 (3)	−0.003 (2)	0.001 (2)	−0.003 (2)
C10	0.017 (3)	0.020 (3)	0.014 (3)	0.003 (2)	0.003 (2)	−0.005 (2)
C11	0.018 (3)	0.012 (3)	0.024 (3)	0.002 (2)	0.003 (2)	−0.002 (2)
C12	0.018 (3)	0.014 (3)	0.022 (3)	−0.003 (2)	0.007 (2)	0.002 (2)
C13	0.014 (3)	0.020 (4)	0.016 (3)	0.002 (2)	0.006 (2)	−0.005 (2)
Br1	0.0271 (3)	0.0313 (3)	0.0159 (3)	0.0036 (3)	0.0069 (2)	−0.0002 (3)
C14	0.020 (3)	0.009 (3)	0.022 (3)	0.002 (2)	0.005 (2)	−0.002 (2)
C15	0.022 (3)	0.016 (3)	0.014 (3)	−0.005 (2)	0.000 (3)	0.003 (2)

Geometric parameters (Å, º)

N1—C1	1.288 (7)	C7—H7A	0.9800
N1—C10	1.417 (7)	C7—H7B	0.9800
C1—C2	1.460 (7)	C7—H7C	0.9800
C1—H1	0.9500	C8—C9	1.389 (7)
C2—C9	1.389 (7)	C8—H8	0.9500

C2—C3	1.415 (7)	C9—H9	0.9500
C3—O1	1.376 (6)	C10—C15	1.387 (8)
C3—C5	1.383 (7)	C10—C11	1.393 (8)
O1—C4	1.430 (6)	C11—C12	1.388 (7)
C4—H4A	0.9800	C11—H11	0.9500
C4—H4B	0.9800	C12—C13	1.375 (7)
C4—H4C	0.9800	C12—H12	0.9500
C5—C6	1.402 (7)	C13—C14	1.396 (7)
C5—H5	0.9500	C13—Br1	1.902 (5)
C6—O2	1.375 (6)	C14—C15	1.376 (8)
C6—C8	1.390 (7)	C14—H14	0.9500
O2—C7	1.432 (6)	C15—H15	0.9500
C1—N1—C10	118.5 (5)	O2—C7—H7C	109.5
N1—C1—C2	122.0 (5)	H7A—C7—H7C	109.5
N1—C1—H1	119.0	H7B—C7—H7C	109.5
C2—C1—H1	119.0	C6—C8—C9	118.4 (5)
C9—C2—C3	117.9 (5)	C6—C8—H8	120.8
C9—C2—C1	120.6 (5)	C9—C8—H8	120.8
C3—C2—C1	121.3 (5)	C2—C9—C8	122.5 (5)
O1—C3—C5	123.4 (5)	C2—C9—H9	118.7
O1—C3—C2	115.9 (4)	C8—C9—H9	118.7
C5—C3—C2	120.6 (5)	C15—C10—C11	117.9 (5)
C3—O1—C4	116.9 (4)	C15—C10—N1	118.1 (5)
O1—C4—H4A	109.5	C11—C10—N1	123.9 (5)
O1—C4—H4B	109.5	C12—C11—C10	120.6 (5)
H4A—C4—H4B	109.5	C12—C11—H11	119.7
O1—C4—H4C	109.5	C10—C11—H11	119.7
H4A—C4—H4C	109.5	C13—C12—C11	119.7 (5)
H4B—C4—H4C	109.5	C13—C12—H12	120.1
C3—C5—C6	119.7 (5)	C11—C12—H12	120.1
C3—C5—H5	120.2	C12—C13—C14	121.0 (5)
C6—C5—H5	120.2	C12—C13—Br1	120.6 (4)
O2—C6—C8	123.9 (5)	C14—C13—Br1	118.3 (4)
O2—C6—C5	115.2 (5)	C15—C14—C13	118.0 (5)
C8—C6—C5	120.9 (5)	C15—C14—H14	121.0
C6—O2—C7	116.8 (4)	C13—C14—H14	121.0
O2—C7—H7A	109.5	C14—C15—C10	122.6 (5)
O2—C7—H7B	109.5	C14—C15—H15	118.7
H7A—C7—H7B	109.5	C10—C15—H15	118.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···N1[i]	0.98	2.74	3.667 (7)	159
C4—H4*C*···O2[ii]	0.98	2.54	3.398 (6)	145

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