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Crystal structure of (*E*)-*N*-(3,4-dimethoxybenzylidene)morpholin-4-amine. Corrigendum

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The list of authors in the paper by Çelikesir *et al.* [*Acta Cryst.* (2014), E**70**, o935] is corrected.

In the paper by Çelikesir *et al.* (2014), the list of authors was incorrect. The correct list is given above.

References

Çelikesir, S. T., Akkurt, M., Jarrahpour, A. & Büyükgüngör, O. (2014). *Acta Cryst.* E**70**, o935.

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Crystal structure of (*E*)-*N*-(3,4-dimethoxybenzylidene)morpholin-4-amine

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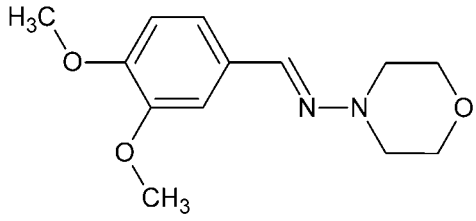
In the title compound, $C_{13}H_{18}N_2O_3$, the benzene ring makes a dihedral angle of 17.19 (11)° with the least-squares plane formed by the four C atoms of the morpholine ring, which adopts a chair conformation. In the crystal, C—H···N hydrogen bonds link the molecules into supramolecular chains running along a 2_1 screw axis parallel to the *b*-axis direction. Weak C—H···π interactions are also observed.

Keywords: crystal structure; hydrogen bonding; C—H···π interactions; Schiff bases; morpholin-4-amine.

CCDC reference: 1015028

1. Related literature

For the structures of related compounds, see: Akkurt *et al.* (2013, 2014). For ring-puckering parameters, see: Cremer & Pople (1975).



2. Experimental

2.1. Crystal data

$C_{13}H_{18}N_2O_3$
M_r = 250.29
Monoclinic, $P2_1$
a = 9.1644 (6) Å
b = 6.0277 (6) Å
c = 13.1327 (9) Å
β = 109.989 (5)°
V = 681.75 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.58 × 0.42 × 0.24 mm

2.2. Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.962, T_{max} = 0.983
8493 measured reflections
3219 independent reflections
2071 reflections with $I > 2\sigma(I)$
R_{int} = 0.229

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.106
S = 1.00
3219 reflections
164 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1353 Friedel pairs
Absolute structure parameter: −0.4 (19)

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

*Cg*1 is the centroid of the C6–C11 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···N1^{i}	0.97	2.61	3.542 (3)	161
C8—H8···*Cg*1^{ii}	0.93	2.87	3.576 (3)	134

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012), *PARST* (Nardelli, 1983) and *PLATON* (Spek, 2009).

Acknowledgements

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS 2 diffractometer (purchased under grant F.279 of the University Research Fund).

Supporting information for this paper is available from the IUCr electronic archives (Reference: RZ5130).

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supporting information

Acta Cryst. (2014). E**70**, o935 [doi:10.1107/S160053681401678X]

Crystal structure of (*E*)-*N*-(3,4-dimethoxybenzylidene)morpholin-4-amine

Sevim Türktekin Çelikesir, Mehmet Akkurt, Aliasghar Jarrahpour and Orhan Büyükgüngör

S1. Comment

As part of our continuing interest in the design and chemistry of Schiff bases containing a morpholine moiety, the title compound has been synthesized and its crystal structure is reported herein. In the title compound (Fig. 1), the benzene ring (C6–C11) makes a dihedral angle of 17.19 (11)° with the least-squares plane formed by the four C atoms of the morpholine ring (C1–C4/N1/O1), which adopts a chair conformation [the puckering parameters (Cremer & Pople, 1975) are Q_T = 0.557 (3) Å, θ = 177.2 (3)°, φ = 177 (7)°]. The N1–N2–C5–C6, C10–C9–O2–C12 and C9–C10–O3–C13 torsion angles are -173.7 (2), 178.2 (3) and -178.9 (3)°, respectively. The bond lengths and bond angles are normal and comparable with those reported for related compounds (Akkurt *et al.* 2013, 2014). In the crystal structure, molecules are linked by intermolecular C—H···N hydrogen bonds forming supramolecular chains running along a 2_1 screw axis parallel to the [010] direction (Table 1, Fig. 2). In addition, weak C—H. . .π interactions also occur (Table 1).

S2. Experimental

Reaction of 3,4-dimethoxybenzaldehyde (1.0 mmol) with morpholin-4-amine (1.0 mmol) in refluxing ethanol gave the title compound. Recrystallization from ethanol gave colourless crystals in 85 % yield. M.p.: 345-347 K. IR (KBr) cm^{-1}:1604 (C=N). ^{1}H-NMR (250 MHz, $CDCl_3$), δ (ppm): 3.06 (CH_2-N, t, 4H, J=5 Hz), 3.79 (CH_2-O, t, 4H, J=5 Hz), 3.83 (2OMe, s, 6H), 6.73 (aromatic H, d, 1H, J=7.5 Hz), 6.93 (aromatic H, d, 1H, J=7.5 Hz), 7.49 (aromatic H, s, 1H), 7.81 (HC=N, s, 1H). ^{13}CNMR (62.9 MHz, $CDCl_3$), δ (p.p.m): 52.1 (CH_2-N), 55.8 (2OMe), 66.4 (CH_2-O), 107.4-137.0 (aromatic carbons), 149.6 (C═N).

S3. Refinement

All H atoms were located geometrically with C—H = 0.93-0.97 Å, and refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms. Owing to the poor quality of the crystal, the data obtained were rather poor and the value of R_{int} remained high (0.229).

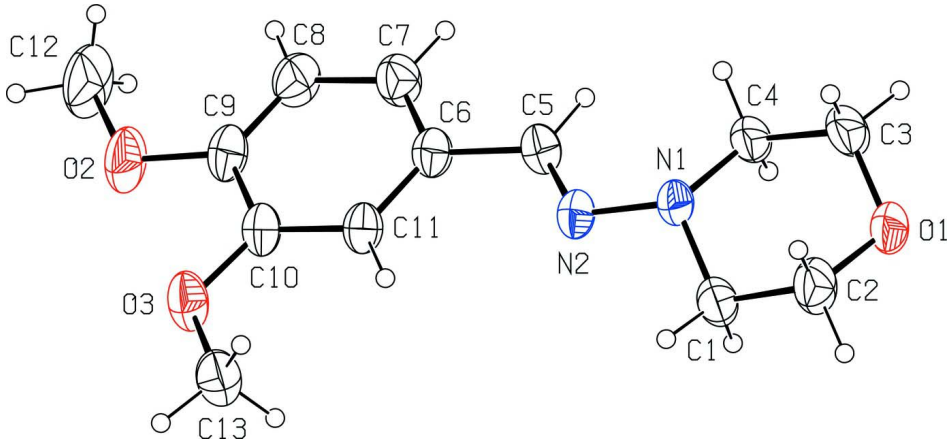

Figure 1

The molecular structure of the title compound with 30% probability displacement ellipsoids.

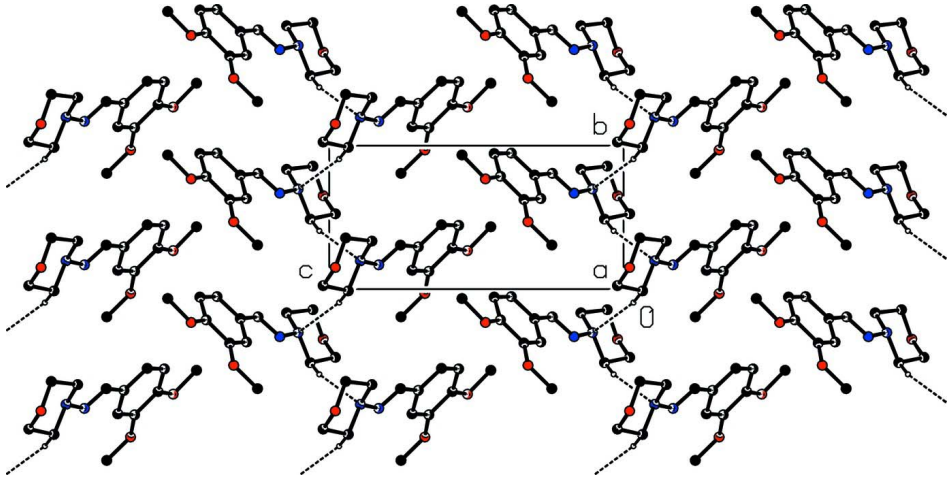

Figure 2

Packing diagram of the title compound viewed down the *a* axis. Hydrogen bonds are indicated by dashed lines. For clarity, H atoms not participating in hydrogen bonding are omitted.

(*E*)-*N*-(3,4-Dimethoxybenzylidene)morpholin-4-amine

Crystal data

$C_{13}H_{18}N_2O_3$
M_r = 250.29
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 9.1644 (6) Å
b = 6.0277 (6) Å
c = 13.1327 (9) Å
β = 109.989 (5)°
V = 681.75 (10) Å^3
Z = 2
$F(000)$ = 268
D_x = 1.219 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8791 reflections
θ = 3.3–28.7°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.58 × 0.42 × 0.24 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.962, T_{max} = 0.983
8493 measured reflections
3219 independent reflections
2071 reflections with $I > 2\sigma(I)$
R_{int} = 0.229
θ_{max} = 28.4°, θ_{min} = 3.3°
h = −12→12
k = −8→7
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.106
S = 1.00
3219 reflections
164 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1353 Friedel pairs
Absolute structure parameter: −0.4 (19)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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	0.87001 (19)	0.1514 (4)	0.97941 (17)	0.0790 (8)
O2	−0.3171 (2)	0.2682 (4)	0.53060 (17)	0.0904 (9)
O3	−0.19462 (19)	−0.0304 (4)	0.67524 (15)	0.0760 (7)
N1	0.5456 (2)	0.1976 (4)	0.89414 (16)	0.0551 (7)
N2	0.3907 (2)	0.1691 (4)	0.83192 (16)	0.0559 (7)
C1	0.6164 (3)	−0.0123 (5)	0.9354 (2)	0.0690 (10)
C2	0.7736 (3)	0.0250 (7)	1.0207 (3)	0.0825 (13)
C3	0.8006 (3)	0.3569 (6)	0.9444 (3)	0.0961 (13)
C4	0.6442 (3)	0.3349 (5)	0.8551 (3)	0.0741 (10)
C5	0.3240 (3)	0.3067 (5)	0.7579 (2)	0.0618 (9)
C6	0.1559 (3)	0.2948 (5)	0.6976 (2)	0.0577 (9)
C7	0.0901 (3)	0.4480 (6)	0.6185 (2)	0.0717 (10)
C8	−0.0684 (3)	0.4447 (6)	0.5604 (2)	0.0749 (10)
C9	−0.1606 (3)	0.2862 (6)	0.5817 (2)	0.0677 (9)
C10	−0.0941 (3)	0.1245 (5)	0.66164 (19)	0.0581 (9)
C11	0.0628 (3)	0.1315 (5)	0.71871 (18)	0.0564 (9)

C12	−0.3889 (4)	0.4346 (9)	0.4511 (4)	0.1281 (18)
C13	−0.1323 (3)	−0.1930 (6)	0.7562 (3)	0.0776 (10)
H1A	0.62780	−0.09990	0.87660	0.0830*
H1B	0.55040	−0.09360	0.96640	0.0830*
H2A	0.76090	0.10140	1.08210	0.0990*
H2B	0.82230	−0.11720	1.04580	0.0990*
H3A	0.86890	0.44530	0.91820	0.1150*
H3B	0.78720	0.43450	1.00540	0.1150*
H4A	0.59760	0.48020	0.83530	0.0890*
H4B	0.65700	0.26800	0.79150	0.0890*
H5	0.38240	0.41810	0.74130	0.0740*
H7	0.15210	0.55610	0.60340	0.0860*
H8	−0.11160	0.55040	0.50700	0.0900*
H11	0.10700	0.02590	0.77200	0.0680*
H12A	−0.49830	0.40600	0.42040	0.1920*
H12B	−0.34400	0.43100	0.39490	0.1920*
H12C	−0.37230	0.57810	0.48490	0.1920*
H13A	−0.21360	−0.29130	0.75850	0.1160*
H13B	−0.08730	−0.12190	0.82530	0.1160*
H13C	−0.05390	−0.27630	0.73970	0.1160*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0392 (9)	0.0779 (15)	0.1082 (16)	0.0027 (11)	0.0100 (9)	0.0184 (13)
O2	0.0436 (10)	0.127 (2)	0.0832 (13)	0.0098 (12)	−0.0007 (9)	0.0218 (14)
O3	0.0453 (9)	0.1042 (16)	0.0703 (11)	−0.0062 (12)	0.0093 (8)	0.0151 (13)
N1	0.0362 (9)	0.0605 (14)	0.0612 (11)	−0.0022 (10)	0.0070 (9)	−0.0039 (11)
N2	0.0393 (9)	0.0702 (15)	0.0539 (11)	0.0014 (11)	0.0105 (8)	−0.0019 (12)
C1	0.0479 (13)	0.070 (2)	0.0838 (18)	−0.0075 (15)	0.0156 (12)	0.0121 (18)
C2	0.0496 (14)	0.093 (3)	0.091 (2)	−0.0002 (17)	0.0060 (14)	0.023 (2)
C3	0.0467 (15)	0.074 (2)	0.138 (3)	−0.0134 (15)	−0.0066 (17)	0.020 (2)
C4	0.0451 (14)	0.0609 (18)	0.102 (2)	−0.0048 (14)	0.0068 (14)	0.0169 (18)
C5	0.0459 (12)	0.0722 (19)	0.0632 (15)	−0.0077 (14)	0.0133 (12)	−0.0020 (16)
C6	0.0446 (12)	0.071 (2)	0.0521 (13)	0.0005 (14)	0.0094 (10)	−0.0019 (14)
C7	0.0562 (14)	0.079 (2)	0.0709 (16)	−0.0042 (16)	0.0100 (13)	0.0092 (17)
C8	0.0605 (15)	0.084 (2)	0.0666 (16)	0.0101 (18)	0.0042 (13)	0.0193 (17)
C9	0.0428 (13)	0.094 (2)	0.0570 (15)	0.0066 (14)	0.0050 (11)	−0.0003 (16)
C10	0.0416 (11)	0.081 (2)	0.0488 (12)	−0.0002 (14)	0.0119 (10)	−0.0001 (15)
C11	0.0436 (11)	0.075 (2)	0.0456 (12)	0.0016 (14)	0.0087 (10)	0.0017 (14)
C12	0.0602 (18)	0.159 (4)	0.130 (3)	0.022 (3)	−0.013 (2)	0.050 (3)
C13	0.0599 (16)	0.092 (2)	0.0796 (18)	−0.0077 (17)	0.0223 (14)	0.0102 (18)

Geometric parameters (Å, º)

O1—C2	1.408 (4)	C1—H1A	0.9700
O1—C3	1.397 (4)	C1—H1B	0.9700
O2—C9	1.364 (3)	C2—H2A	0.9700

O2—C12	1.435 (6)	C2—H2B	0.9700
O3—C10	1.366 (4)	C3—H3A	0.9700
O3—C13	1.415 (4)	C3—H3B	0.9700
N1—N2	1.385 (3)	C4—H4A	0.9700
N1—C1	1.441 (4)	C4—H4B	0.9700
N1—C4	1.443 (4)	C5—H5	0.9300
N2—C5	1.265 (3)	C7—H7	0.9300
C1—C2	1.509 (4)	C8—H8	0.9300
C3—C4	1.516 (5)	C11—H11	0.9300
C5—C6	1.474 (4)	C12—H12A	0.9600
C6—C7	1.367 (4)	C12—H12B	0.9600
C6—C11	1.391 (4)	C12—H12C	0.9600
C7—C8	1.391 (4)	C13—H13A	0.9600
C8—C9	1.366 (5)	C13—H13B	0.9600
C9—C10	1.409 (4)	C13—H13C	0.9600
C10—C11	1.376 (4)		

C2—O1—C3	109.4 (2)	C1—C2—H2B	109.00
C9—O2—C12	116.5 (3)	H2A—C2—H2B	108.00
C10—O3—C13	117.1 (2)	O1—C3—H3A	109.00
N2—N1—C1	110.6 (2)	O1—C3—H3B	109.00
N2—N1—C4	120.1 (2)	C4—C3—H3A	109.00
C1—N1—C4	112.4 (2)	C4—C3—H3B	109.00
N1—N2—C5	120.0 (2)	H3A—C3—H3B	108.00
N1—C1—C2	110.0 (3)	N1—C4—H4A	110.00
O1—C2—C1	111.1 (3)	N1—C4—H4B	110.00
O1—C3—C4	112.4 (3)	C3—C4—H4A	110.00
N1—C4—C3	108.5 (3)	C3—C4—H4B	110.00
N2—C5—C6	121.5 (3)	H4A—C4—H4B	108.00
C5—C6—C7	119.0 (3)	N2—C5—H5	119.00
C5—C6—C11	121.8 (2)	C6—C5—H5	119.00
C7—C6—C11	119.2 (3)	C6—C7—H7	120.00
C6—C7—C8	120.9 (3)	C8—C7—H7	120.00
C7—C8—C9	120.0 (3)	C7—C8—H8	120.00
O2—C9—C8	125.1 (3)	C9—C8—H8	120.00
O2—C9—C10	115.2 (3)	C6—C11—H11	120.00
C8—C9—C10	119.7 (3)	C10—C11—H11	120.00
O3—C10—C9	115.5 (2)	O2—C12—H12A	109.00
O3—C10—C11	125.1 (2)	O2—C12—H12B	109.00
C9—C10—C11	119.3 (3)	O2—C12—H12C	109.00
C6—C11—C10	120.7 (3)	H12A—C12—H12B	109.00
N1—C1—H1A	110.00	H12A—C12—H12C	109.00
N1—C1—H1B	110.00	H12B—C12—H12C	110.00
C2—C1—H1A	110.00	O3—C13—H13A	109.00
C2—C1—H1B	110.00	O3—C13—H13B	109.00
H1A—C1—H1B	108.00	O3—C13—H13C	109.00
O1—C2—H2A	109.00	H13A—C13—H13B	109.00
O1—C2—H2B	109.00	H13A—C13—H13C	109.00

C1—C2—H2A	109.00	H13B—C13—H13C	110.00
C2—O1—C3—C4	−60.7 (3)	N2—C5—C6—C7	180.0 (3)
C3—O1—C2—C1	59.7 (4)	N2—C5—C6—C11	−0.3 (4)
C12—O2—C9—C8	−2.2 (5)	C5—C6—C11—C10	179.7 (3)
C12—O2—C9—C10	178.2 (3)	C11—C6—C7—C8	1.0 (4)
C13—O3—C10—C9	−178.9 (3)	C7—C6—C11—C10	−0.6 (4)
C13—O3—C10—C11	1.5 (4)	C5—C6—C7—C8	−179.2 (3)
N2—N1—C1—C2	−168.4 (2)	C6—C7—C8—C9	−0.3 (5)
C4—N1—N2—C5	−20.1 (4)	C7—C8—C9—C10	−0.9 (4)
N2—N1—C4—C3	173.6 (2)	C7—C8—C9—O2	179.4 (3)
C4—N1—C1—C2	54.3 (3)	O2—C9—C10—O3	1.4 (4)
C1—N1—N2—C5	−153.6 (2)	C8—C9—C10—C11	1.3 (4)
C1—N1—C4—C3	−53.6 (3)	O2—C9—C10—C11	−179.0 (2)
N1—N2—C5—C6	−173.7 (2)	C8—C9—C10—O3	−178.3 (3)
N1—C1—C2—O1	−56.7 (3)	O3—C10—C11—C6	179.1 (3)
O1—C3—C4—N1	57.1 (3)	C9—C10—C11—C6	−0.6 (4)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the C6–C11 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···N1^{i}	0.97	2.61	3.542 (3)	161
C8—H8···*Cg*1^{ii}	0.93	2.87	3.576 (3)	134

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