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4-Chloro-*N*-(3,4,5-trimethoxybenzylidene)aniline

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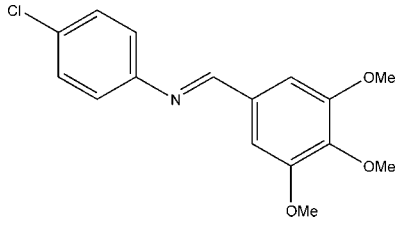
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.109; data-to-parameter ratio = 11.8.

The title compound, $C_{16}H_{16}ClNO_3$, is a Schiff base displaying a *trans* configuration of the C=N double bond. In the crystal structure, intermolecular C—H···N and bifurcated C—H···(O,O) hydrogen bonds are observed.

Related literature

For backgroud and related structures, see: Khalaji *et al.* (2008); Khalaji & Harrison (2008); Khalaji *et al.* (2007); Zhang (2008); Akkurt *et al.* (2008); Kashmiri *et al.* (2008); Ren & Jian (2008). For the synthesis of the title compound, see: Khalaji & Ng (2008).



Experimental

Crystal data

$C_{16}H_{16}ClNO_3$
M_r = 305.75
Monoclinic, $P2_1$
a = 7.2012 (2) Å
b = 8.18700 (10) Å
c = 12.9734 (3) Å
β = 105.050 (2)°
V = 738.63 (3) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 2.37 mm^{-1}
T = 120 K
0.22 × 0.20 × 0.11 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: numerical [Clark & Reid (1995) in *CrysAlis RED* (Oxford Diffraction, 2008)]
T_{min} = 0.680, T_{max} = 0.809
5670 measured reflections
2225 independent reflections
2039 reflections with $I > 3\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.109
S = 1.93
2225 reflections
189 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 915 Friedel pairs
Flack parameter: 0.06 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^{i}	0.96	2.59	3.177 (4)	119
C7—H7···O2^{i}	0.96	2.51	3.471 (4)	178
C12—H12···N1^{ii}	0.96	2.61	3.545 (5)	164

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2007); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2006*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2844).

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supplementary materials

Acta Cryst. (2009). E**65**, o253 [**doi:10.1107/S1600536809000300**]

4-Chloro-*N*-(3,4,5-trimethoxybenzylidene)aniline

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Comment

Studies on the Schiff-base compounds, products of condensation between aldehydes (or ketones) and amines, have received a lot of attention in recent years, (Khalaji *et al.*, 2008; Khalaji & Harrison, 2008; Khalaji *et al.*,2007; Zhang, 2008; Akkurt *et al.*, 2008; Kashmiri *et al.*, 2008; Ren & Jian, 2008). As a continuation of these studies we present the crystal structure of $C_{16}H_{16}ClNO_3$.

The molecular structure of the title compound is shown in Fig. 1. Bond lengths and angles are comparable with those observed in similar compounds (Khalaji *et al.*, 2008; Khalaji & Harrison, 2008; Khalaji *et al.*,2007; Zhang, 2008; Akkurt *et al.*, 2008; Kashmiri *et al.*, 2008; Ren & Jian, 2008). In the crystal structure, intermolecular C—H···N and C—H···O hydrogen bonds are observed.

Experimental

The title compound was synthesized using a method analogous to the literature procedure of Khalaji and Ng (2008). Crystals appropriate for data collection were obtained by slow evaporation from methanol-chloroform (1:5 *v/v*) at a room temperature (yield 83%).

Refinement

All the H atoms were found in difference Fourier maps, but they were constrained to ideal positions. The isotropic atomic displacement parameters of hydrogen atoms were set to $1.2U_{eq}$ of the parent atom.

Figures

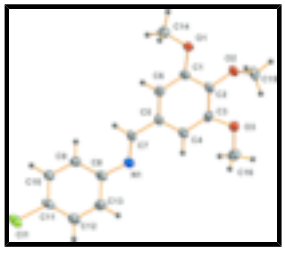

Fig. 1. The molecular structure of the title compound with atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

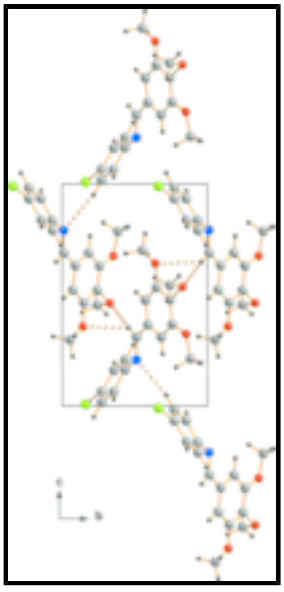

Fig. 2. The packing of (I) viewed along *a*, with hydrogen bonds shown as dashed lines.

4-Chloro-*N*-(3,4,5-trimethoxybenzylidene)aniline

Crystal data

$C_{16}H_{16}Cl_1N_1O_3$	F_{000} = 320
M_r = 305.75	D_x = 1.379 Mg m^{-3}
Monoclinic, $P2_1$	Cu $K\alpha$ radiation λ = 1.54184 Å
Hall symbol: P 2yb	Cell parameters from 4239 reflections
a = 7.2012 (2) Å	θ = 3.5–62.6º
b = 8.18700 (10) Å	μ = 2.37 mm^{-1}
c = 12.9734 (3) Å	*T* = 120 K
β = 105.050 (2)º	Prism, colorless
V = 738.63 (3) Å^3	0.22 × 0.20 × 0.11 mm
Z = 2	

Data collection

Oxford Diffraction Gemini diffractometer with Xcalibur goniometer, Atlas detector and Gemini ultra Cu source	2225 independent reflections
Radiation source: X-ray tube	2039 reflections with $I > 3\sigma(I)$
Monochromator: mirror	R_{int} = 0.048
Detector resolution: 20.7567 pixels mm^{-1}	θ_{max} = 62.7º
T = 120 K	θ_{min} = 3.5º
rotation method data acquisition using ω scans	*h* = −8→7
Absorption correction: numerical [based on the crystal shape, using the method implemented in CrysAlis RED (Oxford Diffraction, 2008) according to Clark & Reid (1995)]	*k* = −9→9
T_{min} = 0.680, T_{max} = 0.809	*l* = −14→14

5670 measured reflections

Refinement

Refinement on F^2	Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0016I^2]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$(\Delta/\sigma)_{max} = 0.013$
$wR(F^2) = 0.109$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
$S = 1.93$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
2225 reflections	Extinction correction: none
189 parameters	Absolute structure: Flack (1983), 915 Friedel pairs
65 constraints	Flack parameter: 0.06 (2)
H-atom parameters constrained	

Special details

Refinement. The refinement was carried out against all reflections. The conventional *R*-factor is always based on *F*. The goodness of fit as well as the weighted *R*-factor are based on *F* and F^2 for refinement carried out on *F* and F^2, respectively. The threshold expression is used only for calculating *R*-factors *etc*. and it is not relevant to the choice of reflections for refinement.

The program used for refinement, Jana2006, uses the weighting scheme based on the experimental expectations, see _refine_ls_weighting_details, that does not force *S* to be one. Therefore the values of *S* are usually larger than the ones from the *SHELX* program.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.00790 (13)	0.16442 (1)	0.00937 (6)	0.0366 (3)
O1	1.2823 (3)	0.6425 (3)	0.64185 (15)	0.0252 (7)
O2	1.4726 (3)	0.8288 (3)	0.53638 (16)	0.0255 (7)
O3	1.3717 (3)	0.8527 (3)	0.32476 (16)	0.0298 (8)
N1	0.7360 (4)	0.5116 (4)	0.2085 (2)	0.0261 (10)
C1	1.2081 (4)	0.6506 (4)	0.5342 (2)	0.0199 (9)
C2	1.3141 (5)	0.7435 (4)	0.4792 (2)	0.0220 (10)
C3	1.2528 (5)	0.7593 (4)	0.3685 (3)	0.0226 (11)
C4	1.0855 (4)	0.6828 (4)	0.3111 (2)	0.0239 (10)
C5	0.9789 (5)	0.5918 (4)	0.3668 (2)	0.0212 (10)
C6	1.0392 (5)	0.5747 (4)	0.4767 (2)	0.0226 (11)
C7	0.7985 (5)	0.5127 (4)	0.3104 (2)	0.0226 (10)
C8	0.5578 (5)	0.4334 (4)	0.1638 (2)	0.0240 (10)
C9	0.4039 (4)	0.4350 (4)	0.2092 (2)	0.0250 (11)
C10	0.2330 (5)	0.3568 (4)	0.1612 (2)	0.0263 (11)
C11	0.2161 (5)	0.2724 (4)	0.0662 (2)	0.0273 (11)
C12	0.3676 (5)	0.2727 (4)	0.0172 (3)	0.0324 (12)
C13	0.5356 (5)	0.3535 (5)	0.0654 (2)	0.0315 (12)
C14	1.1767 (5)	0.5531 (5)	0.7017 (3)	0.0370 (13)
C15	1.6479 (5)	0.7425 (5)	0.5441 (3)	0.0319 (12)
C16	1.3098 (5)	0.8853 (5)	0.2132 (3)	0.0366 (14)

H4	1.043968	0.692286	0.234845	0.0286*
H6	0.964788	0.510597	0.51341	0.0271*
H9	0.415976	0.491463	0.275474	0.03*
H10	0.126966	0.360818	0.193233	0.0316*
H12	0.354653	0.217079	−0.049398	0.0389*
H13	0.638975	0.355215	0.031235	0.0378*
H14a	1.230813	0.572264	0.776497	0.0444*
H14b	1.044923	0.588152	0.682085	0.0444*
H14c	1.182975	0.43867	0.686918	0.0444*
H15a	1.753739	0.805308	0.585471	0.0382*
H15b	1.643203	0.638974	0.578074	0.0382*
H15c	1.664554	0.725127	0.473844	0.0382*
H16a	1.401652	0.954863	0.192956	0.0439*
H16b	1.299241	0.784387	0.174383	0.0439*
H16c	1.186828	0.938464	0.197126	0.0439*
H7	0.723679	0.459098	0.351803	0.0271*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0321 (4)	0.0363 (4)	0.0337 (4)	−0.0062 (4)	−0.0049 (3)	−0.0031 (4)
O1	0.0277 (12)	0.0274 (12)	0.0184 (10)	−0.0043 (10)	0.0021 (8)	−0.0007 (9)
O2	0.0210 (12)	0.0256 (12)	0.0276 (11)	−0.0031 (9)	0.0023 (9)	−0.0061 (9)
O3	0.0298 (13)	0.0353 (12)	0.0235 (11)	−0.0092 (11)	0.0054 (10)	0.0014 (10)
N1	0.0248 (16)	0.0292 (15)	0.0227 (15)	−0.0020 (12)	0.0032 (12)	−0.0012 (11)
C1	0.0216 (16)	0.0189 (14)	0.0184 (14)	0.0012 (14)	0.0039 (11)	−0.0004 (13)
C2	0.0190 (16)	0.0203 (15)	0.0239 (17)	−0.0012 (13)	0.0007 (13)	−0.0033 (12)
C3	0.0235 (18)	0.0192 (17)	0.0280 (17)	−0.0008 (13)	0.0115 (14)	0.0002 (12)
C4	0.0229 (17)	0.0235 (17)	0.0235 (15)	0.0008 (14)	0.0030 (12)	0.0005 (13)
C5	0.0182 (18)	0.0199 (15)	0.0251 (17)	0.0011 (12)	0.0046 (14)	−0.0007 (12)
C6	0.0239 (19)	0.0209 (17)	0.0227 (17)	0.0018 (13)	0.0057 (13)	0.0015 (12)
C7	0.0211 (17)	0.0200 (16)	0.0251 (17)	−0.0003 (13)	0.0033 (13)	−0.0005 (13)
C8	0.0263 (17)	0.0220 (16)	0.0206 (16)	−0.0015 (15)	0.0007 (13)	−0.0004 (13)
C9	0.0252 (18)	0.0281 (18)	0.0201 (16)	0.0027 (15)	0.0032 (13)	0.0004 (13)
C10	0.0254 (19)	0.0296 (18)	0.0219 (17)	0.0010 (14)	0.0024 (13)	0.0003 (14)
C11	0.034 (2)	0.0230 (16)	0.0191 (16)	0.0008 (14)	−0.0037 (14)	0.0007 (12)
C12	0.032 (2)	0.0353 (19)	0.0270 (18)	0.0025 (16)	0.0020 (15)	−0.0050 (14)
C13	0.032 (2)	0.040 (2)	0.0226 (17)	−0.0018 (16)	0.0080 (14)	−0.0030 (15)
C14	0.035 (2)	0.048 (2)	0.0262 (19)	−0.0060 (18)	0.0048 (15)	0.0083 (16)
C15	0.0185 (18)	0.0334 (18)	0.040 (2)	0.0021 (15)	0.0018 (15)	0.0018 (15)
C16	0.042 (2)	0.043 (2)	0.0260 (18)	−0.0086 (17)	0.0097 (16)	0.0074 (15)

Geometric parameters (Å, °)

Cl1—C10	2.708 (3)	C7—H7	0.96
Cl1—C11	1.732 (3)	C8—C9	1.384 (5)
Cl1—C12	2.714 (4)	C8—C13	1.406 (5)
O1—C1	1.362 (3)	C9—C10	1.383 (4)
O1—C14	1.423 (5)	C9—H9	0.96

O2—C2	1.379 (4)	C10—C11	1.390 (5)
O2—C15	1.427 (4)	C10—H10	0.96
O3—C3	1.375 (4)	C11—C12	1.398 (6)
O3—C16	1.424 (4)	C12—C13	1.378 (5)
N1—C7	1.282 (4)	C12—H12	0.96
N1—C8	1.417 (4)	C13—H13	0.96
C1—C2	1.397 (5)	C14—H14a	0.96
C1—C6	1.397 (4)	C14—H14b	0.96
C2—C3	1.395 (4)	C14—H14c	0.96
C3—C4	1.390 (4)	C15—H15a	0.96
C4—C5	1.398 (5)	C15—H15b	0.96
C4—H4	0.96	C15—H15c	0.96
C5—C6	1.385 (4)	C16—H16a	0.96
C5—C7	1.466 (4)	C16—H16b	0.96
C6—H6	0.96	C16—H16c	0.96
C10—Cl1—C12	52.95 (11)	C8—C9—H9	119.2976
C1—O1—C14	117.5 (2)	C10—C9—H9	119.2983
C2—O2—C15	112.4 (3)	C9—C10—C11	119.5 (3)
C3—O3—C16	117.3 (2)	C9—C10—H10	120.2657
C7—N1—C8	117.6 (3)	C11—C10—H10	120.265
O1—C1—C2	115.4 (3)	C10—C11—C12	120.2 (3)
O1—C1—C6	125.5 (3)	C11—C12—C13	119.5 (3)
C2—C1—C6	119.0 (3)	C11—C12—H12	120.2721
O2—C2—C1	119.1 (3)	C13—C12—H12	120.2719
O2—C2—C3	120.3 (3)	C8—C13—C12	120.9 (4)
C1—C2—C3	120.5 (3)	C8—C13—H13	119.5347
O3—C3—C2	114.3 (3)	C12—C13—H13	119.5353
O3—C3—C4	125.1 (3)	O1—C14—H14a	109.4713
C2—C3—C4	120.5 (3)	O1—C14—H14b	109.4711
C3—C4—C5	118.6 (3)	O1—C14—H14c	109.4713
C3—C4—H4	120.6757	H14a—C14—H14b	109.4712
C5—C4—H4	120.6756	H14a—C14—H14c	109.4714
C4—C5—C6	121.2 (3)	H14b—C14—H14c	109.471
C4—C5—C7	120.8 (3)	O2—C15—H15a	109.4714
C6—C5—C7	118.0 (3)	O2—C15—H15b	109.4711
C1—C6—C5	120.1 (3)	O2—C15—H15c	109.4715
C1—C6—H6	119.9695	H15a—C15—H15b	109.4705
C5—C6—H6	119.9698	H15a—C15—H15c	109.4714
N1—C7—C5	123.1 (3)	H15b—C15—H15c	109.4714
N1—C7—H7	118.4336	O3—C16—H16a	109.4709
C5—C7—H7	118.4335	O3—C16—H16b	109.4714
N1—C8—C9	124.2 (3)	O3—C16—H16c	109.4711
N1—C8—C13	117.3 (3)	H16a—C16—H16b	109.4712
C9—C8—C13	118.4 (3)	H16a—C16—H16c	109.4712
C8—C9—C10	121.4 (3)	H16b—C16—H16c	109.4715

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C7—H7···O1^{i}	0.96	2.59	3.177 (4)	119
C7—H7···O2^{i}	0.96	2.51	3.471 (4)	178
C12—H12···N1^{ii}	0.96	2.61	3.545 (5)	164

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

Fig. 1

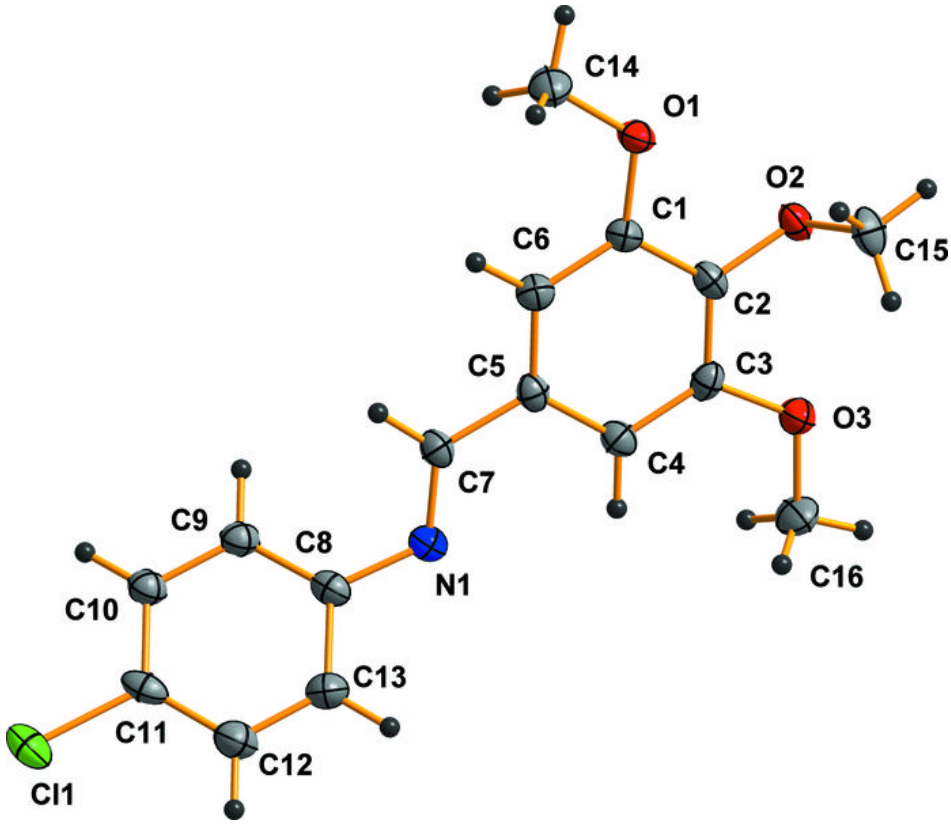
C14
O1
O2
C1
C6
C15
C2
C5
C3
O3
C4
C7
C9
C8
N1
C16
C10
C13
C11
C12
Cl1

Fig. 2