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2-[(2-Chloro-3,4-dimethoxybenzylidene)amino]-adamantane

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.125
Data-to-parameter ratio = 16.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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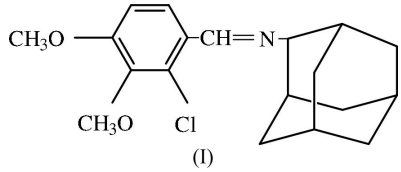
The title compound, $C_{19}H_{24}ClNO_2$, is isomeric with the 1-[(2-chloro-3,4-dimethoxybenzylidene)amino]adamantane structure reported in the previous paper [Işık, Köysal, Septioğlu & Çalış (2005). *Acta Cryst.* E**61**, o1851–o1852].

Comment

A discussion of the chemical importance of this class of compounds is presented in the previous paper (Işık *et al.*, 2005).



(I)

The structure of the title compound, (I) (Fig. 1), differs from that reported for the 1-(2-chloro)-isomer, (II) (Işık *et al.*, 2005), only in the position of the adamantyl group in relation to the rest of the molecule. Both compounds exhibit weak, but slightly different, intermolecular attractions. In (I), there are C—H···Cl and C—H···π interactions (Table 2), while in (II), the interactions are C—H···O and C—H···π. The packing for (I) is shown in Fig. 2.

Experimental

The title compound was synthesized using the same procedure as in the previous paper (Işık *et al.*, 2005). A solution of 2-adamantanamine (0.1 mol) in ethanol (30 ml, 99.9%) was refluxed with an equimolar amount of 2-chloro-3,4-dimethoxybenzaldehyde. The reaction time was 12 h. The solvent was removed *in vacuo* and the residue was recrystallized from ethanol. The IR and ^{1}H NMR spectroscopic data for (I) were found to be the same as given in the literature (Çalış *et al.*, 2002), as shown below. Spectroscopic analysis

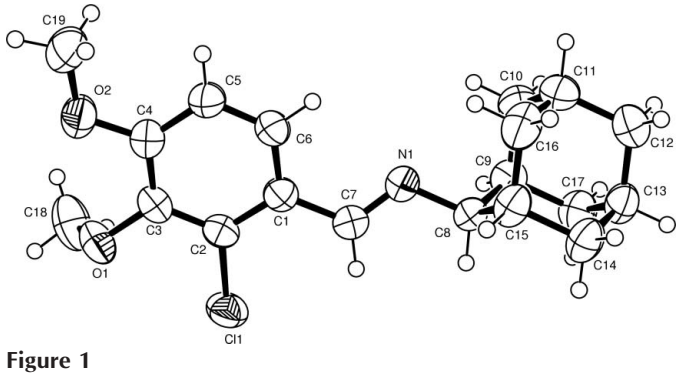


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

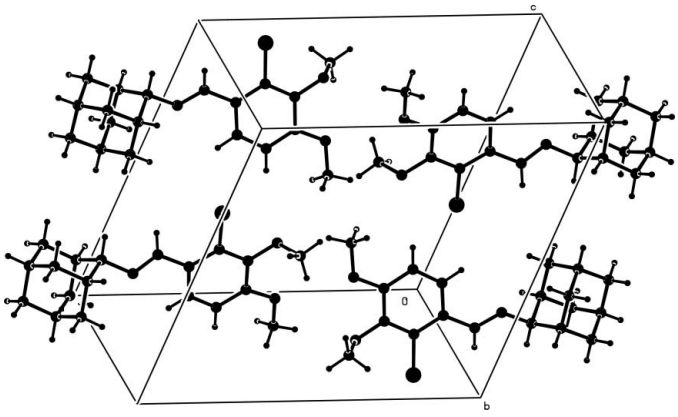


Figure 2
A packing diagram for (I).

for (I), 2-[(2-chloro-3,4-dimethoxybenzylidene)amino]adamantane: IR (KBr, cm^{-1}): 1639 (C═N); ^1H NMR ($CDCl_3$, δ, p.p.m., 303 K): 1.60–2.00 (10H, *m*, CH_2-Ad), 2.15 (5H, *bs*, CH-Ab), 3.80 (3H, *s*, CH_3O), 4.00 (3H, *s*, CH_3O), 6.80–7.50 (2H, *m*, H-Ar), 8.20 (1H, *s*, CH═N).

Crystal data

$C_{19}H_{24}ClNO_2$
M_r = 333.86
Monoclinic, $P2_1/c$
a = 14.2591 (11) Å
b = 9.9334 (5) Å
c = 12.7325 (10) Å
β = 107.610 (6)°
V = 1718.9 (2) Å^3
Z = 4
D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 884 reflections
θ = 1.5–27.5°
μ = 0.23 mm^{-1}
T = 293 (2) K
Prism, colourless
0.80 × 0.42 × 0.12 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.898, T_{max} = 0.964
11 934 measured reflections
3371 independent reflections
2604 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 26.0°
$h = -17 \rightarrow 16$
$k = -11 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.125
S = 1.05
3371 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2 + 0.0027P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—Cl1	1.7310 (15)	C7—N1	1.252 (2)
C3—O1	1.3759 (18)	C8—N1	1.458 (2)
C4—O2	1.365 (2)		
C7—N1—C8	119.22 (14)	C4—O2—C19	117.28 (14)
C3—O1—C18	113.47 (14)		
C6—C1—C7—N1	−15.2 (2)	C1—C7—N1—C8	173.44 (14)
C2—C1—C7—N1	167.95 (15)		

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C1–C6 ring

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*A*···Cl1^{i}	0.97	2.94	3.6784 (19)	134
C14—H14*A*···*Cg*1^{ii}	0.97	2.93	3.7274 (19)	141

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

H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å, the $C—H_2$ distances at 0.97 Å and the $C—H_3$ distances at 0.96 Å. $U_{iso}(H)$ = $1.2U_{eq}$ or $1.5U_{eq}$(parent C atom).

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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