Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[(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) = 0.003 Å
R factor = 0.045
wR factor = 0.122
Data-to-parameter ratio = 17.8

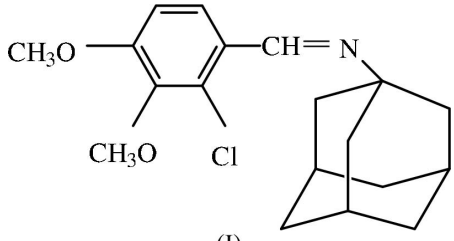
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 5 April 2005
Accepted 17 May 2005
Online 21 May 2005

The title compound, $C_{19}H_{24}ClNO_2$, was synthesized and characterized by single-crystal X-ray diffraction. The adamantyl and benzene groups are bridged by an imine group, with a C—C═N—C torsion angle of −178.2 (2)°. Weak C—H···O and C—H···π interactions are found in the crystal packing.

Comment

Compounds containing an adamantanyl subunit have long been of interest to chemists as a result of the rigid structure and well defined substitution chemistry of these compounds. Adamantanes in general are of biological and medicinal interest because of their antifungal, antiviral, antimicrobial, antibacterial, antitumour and anti-HIV activities (Orzeszko *et al.*, 2000; Kolocouris *et al.*, 1996; Ferle-Vidovic *et al.*, 1993; Monforte *et al.*, 1981; Da Settimo *et al.*, 1995). The synthesis, structural characterization and antimicrobial activity evaluation of adamantane derivatives, including the title compound, have recently been published (Çalış *et al.*, 2002). In an earlier study, we presented the crystal structure of the related compound 2-[(3,4-dimethoxybenzylidene)amino]adamantane (Çoruh *et al.*, 2002). In this paper, we report the crystal structure of the title compound, (I), and in the following paper (Işık *et al.*, 2005), we report the structure of the 2-(2-chloro)-isomer of (I).



(I)

The rings in the adamantane moiety in (I) have normal chair conformations. The bonding along the C—C═N—C chain indicates that this system is conjugated (Table 1), which is consistent with the situation in a related structure (Fernandez-G *et al.*, 2001).

Although the title compound has no classical hydrogen bonds, it does exhibit weak C—H···O and C—H···π interactions, namely C11—H11*B*···O2(x, $-y+\frac{3}{2}$, $z+\frac{1}{2}$) and C16—H16*A*···*Cg*1 (*Cg*1 is the centroid of the C1–C6 ring) (Table 2).

Experimental

The title compound was synthesized according to the well known procedures for preparing Schiff bases by the reaction of aldehydes

and amines (Amal *et al.*, 1976; Chimirri *et al.*, 1994; Çalış *et al.*, 2002). A solution of 1-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 8 h. The solvent was removed *in vacuo* and the residue was recrystallized from ethanol. The IR and ^{1}H NMR spectroscopic data of the title compound were found to be the same as those in the literature (Çalış *et al.*, 2002). IR (KBr, cm^{-1}): 1628 (C═N). ^{1}H NMR ($CDCl_3$, p.p.m., 303 K): 1.25–2.00 (12H, *m*, CH_2—Ad), 2.15 (3H, *bs*, CH—Ab), 3.80 (3H, *s*, CH_3O), 3.90 (3H, *s*, CH_3O), 6.70–7.85 (2H, *m*, H—Ar), 8.60 (1H, *s*, CH═N).

Crystal data

$C_{19}H_{24}ClNO_2$	D_x = 1.299 Mg m^{-3}
M_r = 333.84	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 9696 reflections
a = 13.939 (5) Å	θ = 1.6–27.2°
b = 6.878 (5) Å	μ = 0.23 mm^{-1}
c = 19.533 (4) Å	T = 293 (2) K
β = 114.279 (17)°	Prism, colourless
V = 1707.1 (14) Å^3	0.80 × 0.43 × 0.24 mm
Z = 4	

Data collection

Stoe IPDS-2 diffractometer	2738 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.060
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)	θ_{max} = 27.2°
T_{min} = 0.879, T_{max} = 0.965	$h = -17 \rightarrow 17$
13 143 measured reflections	$k = -8 \rightarrow 8$
3730 independent reflections	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.3578P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.122	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
3730 reflections	Extinction correction: *SHELXL97*
209 parameters	Extinction coefficient: 0.011 (3)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.734 (2)	N1—C8	1.481 (2)
O1—C3	1.372 (2)	C4—O2	1.360 (2)
N1—C7	1.261 (2)		
C3—O1—C18	115.73 (15)	C4—O2—C19	118.38 (16)
C7—N1—C8	119.27 (16)		
C8—N1—C7—C1	−178.20 (15)	N1—C7—C1—C2	170.16 (17)
N1—C7—C1—C6	−6.7 (3)		

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*
C11—H11*B*···O2^{i}	0.97	2.60	3.537 (3)	163
C16—H16*A*···*Cg*1^{ii}	0.97	2.73	3.629 (3)	154

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x, y-1, z$.

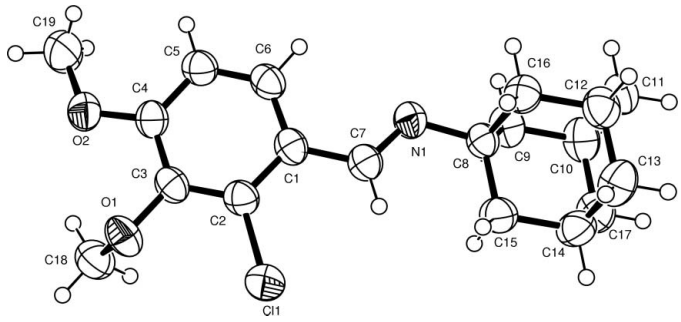


Figure 1
The structure of the title compound, (I), with 50% probability displacement ellipsoids and the atom-numbering scheme.

H atoms were positioned geometrically and refined using a riding model, fixing the C—H distances at 0.93 (aromatic), 0.97 (CH_2), 0.96 (CH_3) and 0.98 Å (other CH), and with 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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