

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U^{ij} a^i a^j$$

	x	y	z	U _{eq}
I1	1/2	0.60730 (5)	3/4	0.04532 (9)
I2	1/2	1	1/2	0.04959 (9)
C11	0.60788 (4)	0.59938 (15)	0.77674 (5)	0.0572 (2)
C12	0.49036 (3)	0.74997 (16)	0.39624 (5)	0.0578 (2)
C13	0.23404 (4)	0.47104 (15)	0.18731 (4)	0.0581 (2)
N1	0.17955 (10)	0.4378 (4)	0.29888 (13)	0.0405 (5)
N2	0.30368 (10)	0.4784 (4)	0.35927 (13)	0.0390 (5)
C1	0.12287 (14)	0.4092 (6)	0.27063 (19)	0.0521 (8)
C2	0.09313 (14)	0.4071 (6)	0.3173 (2)	0.0540 (8)
C3	0.12298 (14)	0.4363 (5)	0.3927 (2)	0.0470 (7)
C4	0.18333 (12)	0.4647 (4)	0.42346 (15)	0.0350 (5)
C5	0.21622 (14)	0.4921 (4)	0.50262 (16)	0.0395 (6)
C6	0.27449 (14)	0.5114 (4)	0.53120 (15)	0.0407 (6)
C7	0.30476 (12)	0.5066 (4)	0.48267 (15)	0.0355 (6)
C8	0.36529 (14)	0.5224 (5)	0.51043 (18)	0.0479 (7)
C9	0.39313 (14)	0.5155 (6)	0.4631 (2)	0.0551 (9)
C10	0.36088 (14)	0.4924 (5)	0.38637 (19)	0.0498 (8)
C11	0.27358 (11)	0.4841 (4)	0.40443 (14)	0.0325 (5)
C12	0.21184 (11)	0.4638 (4)	0.37421 (14)	0.0329 (5)

Table 2. Selected geometric parameters (Å, °)

I1—C11	2.5522 (8)	C4—C12	1.405 (4)
I2—C12	2.5464 (9)	C4—C5	1.428 (4)
N1—C1	1.320 (4)	C5—C6	1.349 (4)
N1—C12	1.362 (3)	C6—C7	1.428 (4)
N2—C10	1.322 (4)	C7—C8	1.400 (4)
N2—C11	1.368 (3)	C7—C11	1.403 (3)
C1—C2	1.386 (5)	C8—C9	1.359 (5)
C2—C3	1.358 (5)	C9—C10	1.382 (5)
C3—C4	1.403 (4)	C11—C12	1.429 (4)
C11 ⁱ —I1—C11	177.62 (5)	C1—N1—C12	122.7 (3)
C12 ⁱⁱ —I2—C12	180.0	C10—N2—C11	122.7 (3)

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

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...C13	1.00 (4)	2.00 (4)	2.995 (3)	176 (4)
N2—H10...C13	0.99 (4)	2.08 (4)	3.069 (3)	171 (3)

All H atoms were located by difference Fourier synthesis and were refined isotropically. C—H distances are in the range 0.86 (4)–1.04 (3) Å.

Data collection: CAD-4 ARGUS Software (Nonius, 1996). Cell refinement: CAD-4 ARGUS Software. Data reduction: MolEN (Fair, 1990). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997b). Molecular graphics: SHELXTL/PC (Sheldrick, 1990). Software used to prepare material for publication: SHELXL97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1249). Services for accessing these data are described at the back of the journal.

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3,9-Dimethyl-1,11-diphenyl-4,8-diazaundecane-1,11-dione dihydrate

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Abstract

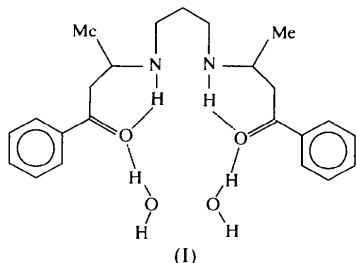
The title compound, C₂₃H₂₆N₂O₂·2H₂O, displays strong intramolecular N—H...O [2.637 (2) Å] and intermolecular O—H...O [2.763 (4) and 2.842 (3) Å] hydrogen bonds within each salicylideneimine unit and water molecule. The Schiff base has a twofold axis passing

through the central C atom and therefore adopts a *cis* conformation.

Comment

Schiff bases and their biochemically active complexes have been studied extensively over the past decade. Although numerous transition metal complexes of Schiff bases have been structurally investigated, relatively few free Schiff bases have been similarly characterized (Garnovskii *et al.*, 1993). *N*-Salicylideneaniline and its derivatives show photochromism and thermochromism in the solid state (Cohen *et al.*, 1964). These effects are produced by intramolecular proton transfer associated with a change in the π -electron configuration (Hadjoudis *et al.*, 1987).

In the course of a structural investigation of Schiff bases (Elerman *et al.*, 1991, 1992, 1994, 1995, 1997, 1998; Elmali *et al.*, 1995, 1999; Elmali & Elerman, 1997, 1998), the structure of the tetradentate Schiff base ligand (I) was determined.



Several non-planar tetradentate Schiff bases similar to (I) have been reported (Pahor *et al.*, 1976, 1978; Subrahmanyam *et al.*, 1982; Cimernan *et al.*, 1992; Senn & Nowacki, 1977; Elerman *et al.*, 1991, 1994; Corden *et al.*, 1996). The title molecule has site symmetry 2.

The torsion angle C3—N1—C2—C1 [175.1 (3) $^\circ$] and the dihedral angle between the two moieties [15.93 (6) $^\circ$] indicate that the molecule is not planar. The conformation of the free ligand in the solid state is of particular interest in relation to that in a metal complex (Corden *et al.*, 1996).

Intramolecular hydrogen bonding occurs between O1 and N1, and intermolecular hydrogen bonds are formed between O1 and O2, and between O2 and O2(1 - x, 1 - y, 1 - z) [distances of 2.637 (2), 2.842 (3) and 2.763 (4) Å, respectively]. Two types of intramolecular hydrogen bonds (either N—H \cdots O or N \cdots H—O) can exist in Schiff bases (Garnovskii *et al.*, 1993). The Schiff bases derived from salicylaldehyde always form the N \cdots H—O type of hydrogen bonding, regardless of the nature of the N-substituted group (alkyl and aryl) (Gavranic *et al.*, 1996, and references therein).

Experimental

Ethanol solutions of benzylacetone (2.768 g, 0.008 mol) and 1,3-diaminopropane (2.965 g, 0.004 mol) were mixed and the resulting solution was refluxed at room temperature for 2 h. Colorless single crystals were obtained by slow evaporation.

Crystal data

C₂₃H₂₆N₂O₂·2H₂O

M_r = 398.50

Orthorhombic

Pbcn

a = 24.830 (2) Å

b = 9.642 (2) Å

c = 9.176 (6) Å

V = 2197 (1) Å³

Z = 4

D_x = 1.205 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 25 reflections

θ = 10.1–13.0 $^\circ$

μ = 0.082 mm⁻¹

T = 293.2 K

Hexagonal

0.80 × 0.40 × 0.10 mm

Colorless

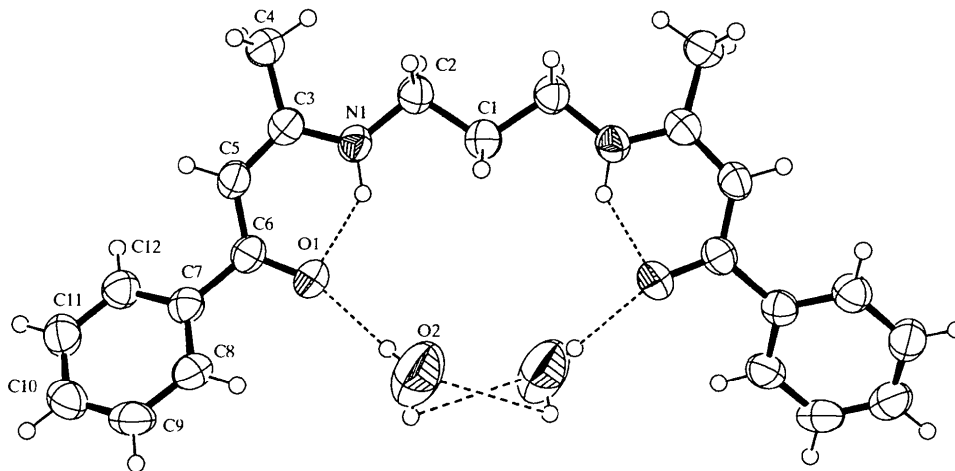


Fig. 1. The molecular structure and atomic labeling scheme of the title compound. Displacement ellipsoids are plotted at the 50% probability level (ORTEP-3; Farrugia, 1997).

Data collection

Rigaku AFC-7S diffractometer	1344 reflections with $I > 2\sigma(I)$
ω - 2θ scans	$\theta_{\max} = 29^\circ$
Absorption correction:	$h = 0 \rightarrow 33$
ψ scan (North <i>et al.</i> , 1968)	$k = 0 \rightarrow 13$
$T_{\min} = 0.928$, $T_{\max} = 1.000$	$l = 0 \rightarrow 12$
2192 measured reflections	3 standard reflections every 150 reflections
2192 independent reflections	intensity decay: 4.98%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$\Delta\rho_{\max} = 0.275 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.160$	$\Delta\rho_{\min} = -0.240 \text{ e } \text{\AA}^{-3}$
$S = 0.960$	Extinction correction: (Zachariasen, 1967)
2192 reflections	Extinction coefficient: 0.0010 (14)
133 parameters	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
H atoms treated by a mixture of independent and constrained refinement	
$w = 1/[\sigma^2(F_o^2) + (0.0851P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{eq}
O1	0.60215 (6)	0.22699 (15)	0.46385 (17)	0.0622 (5)
O2	0.51504 (9)	0.4061 (3)	0.3938 (3)	0.1292 (10)
N1	0.57601 (6)	-0.03338 (18)	0.41178 (18)	0.0471 (4)
C1	1/2	-0.0415 (3)	1/4	0.0494 (7)
C2	0.53884 (8)	-0.1268 (2)	0.3395 (2)	0.0500 (5)
C3	0.61389 (8)	-0.0656 (2)	0.5085 (2)	0.0450 (5)
C4	0.62112 (11)	-0.2156 (2)	0.5487 (3)	0.0642 (7)
C5	0.64453 (8)	0.0364 (2)	0.5739 (2)	0.0469 (5)
C6	0.63609 (8)	0.1794 (2)	0.5535 (2)	0.0461 (5)
C7	0.66623 (8)	0.2793 (2)	0.6484 (2)	0.0458 (5)
C8	0.64200 (9)	0.4033 (2)	0.6889 (3)	0.0556 (6)
C9	0.66663 (10)	0.4911 (2)	0.7880 (3)	0.0636 (6)
C10	0.71634 (11)	0.4581 (3)	0.8437 (3)	0.0628 (7)
C11	0.74202 (9)	0.3386 (3)	0.8004 (3)	0.0616 (6)
C12	0.71715 (9)	0.2495 (2)	0.7033 (2)	0.0551 (6)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O1—C6	1.264 (2)	C6—C7	1.499 (3)
N1—C3	1.330 (3)	C7—C8	1.389 (3)
N1—C2	1.450 (3)	C7—C12	1.391 (3)
C1—C2	1.510 (3)	C8—C9	1.385 (3)
C3—C5	1.381 (3)	C9—C10	1.373 (3)
C3—C4	1.503 (3)	C10—C11	1.376 (3)
C5—C6	1.407 (3)	C11—C12	1.384 (3)
C3—N1—C2	127.59 (17)	N1—C3—C5	120.89 (18)
C2 ¹ —C1—C2	114.0 (3)	N1—C3—C4	118.25 (19)
N1—C2—C1	108.46 (19)	C5—C3—C4	120.83 (19)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...O1	0.93	1.86	2.637 (2)	140
O2—H2...O2 ¹	0.80	2.42	2.763 (4)	108
O2—H3...O1	0.81	2.04	2.842 (3)	172

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

All H atoms were placed geometrically on their corresponding C atoms, except for the H1, H2 and H3 atoms, which

were located from the difference Fourier synthesis map. The H2—O2 and H3—O2 bond distances were restrained during the refinement process in order to prevent anomalous bond distances.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993) and *DIRDIF94* (Beurskens *et al.*, 1994). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP-3* (Farrugia, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1327). Services for accessing these data are described at the back of the journal.

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3,3-Dichloro-1,4-diphenylazetid-2-one

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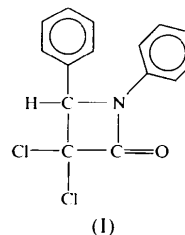
(Received 13 January 1999; accepted 29 April 1999)

Abstract

In the crystal structure of the title compound, C₁₅H₁₁Cl₂NO, the Cl—C—Cl plane is nearly perpendicular to the four-membered β -lactam ring [89.0(2)°] and the C—C bond distances in this group are 1.571(6) and 1.543(7) Å. The most out-of-plane atom from the best plane of the lactam ring is the carbonyl C atom [−0.029(5) Å]. The dihedral angle between the best planes of the phenyl rings is 77.4(2)°.

Comment

Recent developments in the field of β -lactams have shown that the essential feature of the antibacterial activity of these compounds is the presence of the β -lactam ring (azetid-2-one) (Brady & Gu, 1989; Takasuka *et al.*, 1982; Manhas *et al.*, 1988). The selectivity or activity can be decisively influenced by novel ring substituents (Sharma *et al.*, 1994; Kumar *et al.*, 1993). The biological activity of β -lactams has been studied by Chambers & Doedens (1980) and structural studies have been performed by Ercan *et al.* (1996a,b), Ülkü *et al.* (1997) and Paulus *et al.* (1969). The four atoms of the β -lactam ring are coplanar within experimental error. This paper describes the structure of (I), a molecule containing a β -lactam ring.



The four-membered β -lactam ring of (I) is nearly planar and the maximum deviation [−0.029(5) Å] is assumed by atom C7. The bond lengths in the lactam ring in (I) are comparable with the previous work on monocyclic azetid-2-ones (Ercan *et al.*, 1996a, b and references therein). The N1—C7 bond length of 1.362(6) Å, conjugated with the carbonyl group, is shorter than the N1—C9 and N1—C6 bond lengths [1.469(5) and 1.417(5) Å, respectively] and these results show a fair agreement with those of the previous studies (Ercan *et al.*, 1996a,b; Ülkü *et al.*, 1997). However, the O1=C7 bond length of 1.186(6) Å is slightly shorter than those found in the literature [1.198(12) Å; Allen *et al.*, 1987].

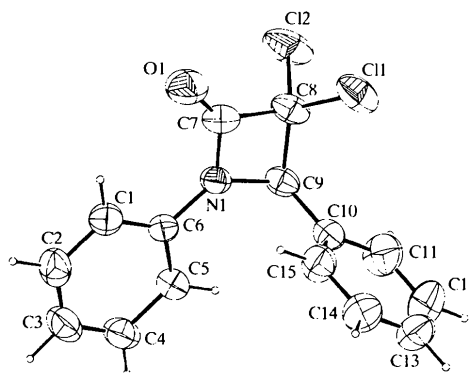


Fig. 1. PLATON (Spek, 1996) drawing of the title molecule, showing the molecular structure and atomic labelling scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

In the present work, the C7—C8 and C8—C9 bond lengths [1.543(7) and 1.571(6) Å, respectively] deviate slightly from those reported by Ercan *et al.* (1996a,b) and Ülkü *et al.* (1997) [1.536(5), 1.55(2), 1.535(5), 1.558(4), 1.60(2) and 1.566(5) Å]. The bond angle at C8 (C7—C8—C9) is 86.1(3)°, which is almost equal to those reported in previous work [86.3(2), 87.0(3) and 87.1(7)°; Ercan *et al.* (1996a,b); Ülkü *et al.* (1997)]. The dihedral angle formed by the phenyl rings is 77.4(2)°. The C1—C6 phenyl ring is nearly coplanar with the β -lactam moiety [6.0(2)°] and atom C7 deviates from the plane of the aromatic ring by 0.090(5) Å. In contrast, the second phenyl ring, C10—C15, makes a dihedral angle of 71.9(2)° with the best