

S = 0.932
 4385 reflections
 438 parameters
 H atoms treated by a
 mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0917P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction:
 SHELXL97 (Sheldrick,
 1997)
 Extinction coefficient:
 0.0069 (5)
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

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Table 1. Selected geometric parameters (Å, °)

C11A—C2A	1.735 (3)	C11B—C2B	1.738 (3)
C8A—O9A	1.214 (3)	C8B—O9B	1.223 (3)
C8A—C10A	1.521 (4)	C8B—C10B	1.510 (4)
C10A—N11A	1.446 (3)	C10B—N11B	1.441 (4)
N11A—C12A	1.377 (3)	N11B—C12B	1.377 (3)
N11A—C1A	1.455 (4)	N11B—C18B	1.448 (4)
C7A—C2A—C11A	119.1 (2)	C7B—C2B—C11B	119.5 (3)
C3A—C2A—C11A	119.2 (2)	C3B—C2B—C11B	119.5 (2)
C4A—C5A—C8A	122.6 (2)	C4B—C5B—C8B	119.2 (2)
C6A—C5A—C8A	118.7 (2)	C6B—C5B—C8B	122.1 (2)
O9A—C8A—C5A	120.6 (2)	O9B—C8B—C5B	119.9 (2)
O9A—C8A—C10A	120.0 (3)	O9B—C8B—C10B	120.6 (2)
C5A—C8A—C10A	119.4 (2)	C5B—C8B—C10B	119.6 (2)
N11A—C10A—C8A	114.1 (2)	N11B—C10B—C8B	113.7 (2)
C12A—N11A—C10A	120.0 (2)	C12B—N11B—C10B	119.6 (2)
C12A—N11A—C18A	119.0 (3)	C12B—N11B—C18B	120.2 (3)
C10A—N11A—C18A	116.8 (3)	C10B—N11B—C18B	117.9 (3)
N11A—C12A—C13A	121.8 (3)	N11B—C12B—C17B	122.1 (3)
N11A—C12A—C17A	121.9 (2)	N11B—C12B—C13B	121.0 (3)

Scattering factors, dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography*, Vol. C, Tables 6.1.1.4, 4.2.6.8 and 4.2.4.2, respectively.

Data collection: Seifert diffractometer software. Cell refinement: LSUCRE (Appleman, 1971). Data reduction: XRAY80 (Stewart *et al.*, 1980). Program(s) used to solve structure: MULTAN80 (Main *et al.*, 1980). Program(s) used to refine structure: XRAY80; SHELXL97 (Sheldrick, 1997). Molecular graphics: Xtal3.0 (Hall & Stewart, 1990). Software used to prepare material for publication: SHELXL97.

This work was supported by the DGICYT PB96-0001.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1278). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 258–260

2-Salicylideneamino-4,5-bis(2-furyl)furan-3-carbonitrile

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(Received 27 July 1998; accepted 25 September 1998)

Abstract

There are two independent molecules in the asymmetric unit of the title compound, C₂₀H₁₂N₂O₄. The main difference between the independent molecules is that while one of them is nearly planar, the other has the furyl substituents at 24.1 (1) and 16.1 (1)° to the central furan ring. The near planarity of the molecules introduces steric strain and distortions of the exocyclic bond angles around the central furan ring. Each molecule contains a strong intramolecular O—H...N hydrogen bond.

Comment

Although Schiff bases have been widely used as ligands in the formation of transition metal complexes and structurally characterized, a relatively small number of the free Schiff bases have been similarly characterized (Calligaris & Randaccio, 1987). Schiff base compounds show photochromism and thermochromism in the solid state by proton transfer from the hydroxyl O atom to the imine N atom (Hadjoudis *et al.*, 1987).

The title compound, (I), has two crystallographically independent molecules in the asymmetric unit. The extent of the planarity differs; in one the angles between the planar moiety A (O1, O2, N1, N2, C1–C9, C19, C20) and the planar moieties B (O3, C11–C13) and C (O4, C15–C18) are 2.3 (1) and 7.1 (1)°, respectively, whereas in the other molecule they are 21.4 (1) and 16.1 (1)°. The near planarity of the molecules re-

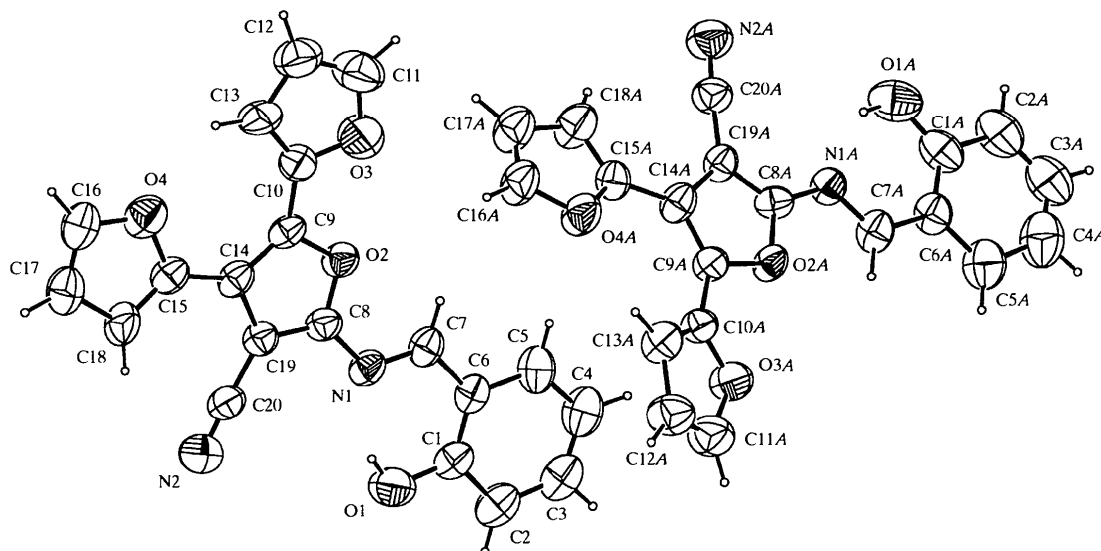
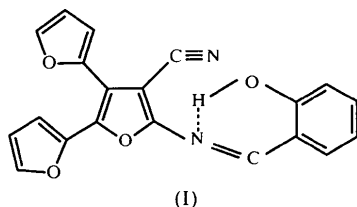


Fig. 1. The molecular structure and atomic labelling scheme of the title compound. Displacement ellipsoids are plotted at the 50% probability level for non-H atoms. H atoms are shown as spheres of an arbitrary radius.

sults in large differences in the exocyclic angles at C9, C10, C14, C15 and C19 of 22.4 (2), 15.8 (2), 7.8 (2), 8.8 (2) and 6.6 (2)°, respectively, due to the close intramolecular contacts between O4···H13 [2.257 (2) Å] and H18···C20 [2.526 (3) Å]. There is also a similar effect in the other molecule where the exocyclic angles at C9A, C10A, C14A, C15A and C19A differ by 20.9 (2), 18.6 (2), 6.6 (2), 8.3 (2) and 5.1 (2)°, respectively, and the corresponding intramolecular contacts are O4A···H13A [2.389 (2) Å] and H18A···C20A [2.563 (2) Å].



Strong intramolecular hydrogen bonds occur between O1 and N1 and between O1A and N1A (Table 2). The sum of the van der Waals radii of O and N (3.07 Å; Bondi, 1964) is significantly longer than the O···N hydrogen-bond length, which is similar to the corresponding bond lengths in *N*-(2-hydroxyphenyl)-salicylaldehyde [2.625 (7) Å; Elerman *et al.*, 1995], 2,2'-azinodimethyldiphenol [2.611 (6) Å; Xu *et al.*, 1994] and 2-salicylideneamino-4,5,6,7-tetrahydrobenzo[*b*]thiophene-3-carbonitrile [2.626 (2) Å; Elerman & Elmali, 1998].

Apart from the intramolecular hydrogen bonds, there are also significant intra- and intermolecular C—H···O and C—H···N contacts (Taylor & Kennard, 1982; Berkovitch-Yellin & Leiserowitz, 1984; Sharma & Desiraju, 1994), which are listed in Table 2.

Experimental

The title compound was obtained from the reaction of 2-amino-4,5-bis(2-furyl)furan-3-carbonitrile (0.001 mol) in 40 ml of hot ethanol with a solution of salicylaldehyde (0.001 mol) in 30 ml of hot ethanol. The precipitate was recrystallized for 2 to 3 d using 1.5 g of Schiff base in 100 ml of chloroform solution.

Crystal data

C₂₀H₁₂N₂O₄
M_r = 344.32
 Triclinic
P $\bar{1}$
a = 9.024 (3) Å
b = 11.442 (2) Å
c = 17.506 (3) Å
 α = 86.12 (1)°
 β = 79.10 (2)°
 γ = 68.33 (2)°
V = 1649.5 (7) Å³
Z = 4
D_x = 1.386 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 6–15°
 μ = 0.099 mm⁻¹
T = 293 (2) K
 Needle
 0.45 × 0.15 × 0.10 mm
 Red

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 6634 measured reflections
 5744 independent reflections
 3792 reflections with $I > 2\sigma(I)$

*R*_{int} = 0.045
 θ_{\max} = 25.99°
h = -10 → 11
k = -14 → 14
l = 0 → 21
 3 standard reflections
 frequency: 120 min
 intensity decay: 3.4%

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.162$

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.31P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.092$
5744 reflections
475 parameters
H-atom parameters
constrained

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.186 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.188 \text{ e } \text{Å}^{-3}$
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

This work was supported by grant TBAG-1597 of the Scientific and Technical Research Council of Turkey.

Table 1. Selected geometric parameters (Å, °)

C1—O1	1.361 (3)	C1A—O1A	1.349 (3)
C7—N1	1.283 (3)	C7A—N1A	1.298 (3)
C8—O2	1.334 (2)	C8A—O2A	1.341 (2)
C8—N1	1.384 (2)	C8A—N1A	1.374 (2)
C9—C14	1.378 (3)	C9A—C14A	1.370 (3)
C9—O2	1.386 (2)	C9A—O2A	1.391 (2)
C9—C10	1.419 (3)	C9A—C10A	1.430 (3)
C10—O3	1.346 (3)	C10A—C13A	1.349 (3)
C10—C13	1.350 (3)	C10A—O3A	1.349 (2)
C11—C12	1.314 (4)	C11A—C12A	1.313 (4)
C11—O3	1.362 (3)	C11A—O3A	1.389 (3)
C12—C13	1.406 (4)	C12A—C13A	1.451 (4)
C14—C19	1.420 (3)	C14A—C19A	1.434 (3)
C14—C15	1.438 (3)	C14A—C15A	1.445 (3)
C15—C18	1.335 (3)	C15A—O4A	1.343 (3)
C15—O4	1.338 (3)	C15A—C18A	1.343 (3)
C16—C17	1.313 (4)	C16A—C17A	1.317 (4)
C16—O4	1.376 (3)	C16A—O4A	1.383 (3)
C17—C18	1.458 (3)	C17A—C18A	1.463 (3)
O1—C1—C6	122.1 (2)	O1A—C1A—C6A	121.6 (2)
O1—C1—C2	115.9 (2)	O1A—C1A—C2A	114.9 (2)
O2—C8—N1	122.2 (2)	O2A—C8A—N1A	121.3 (2)
C19—C8—N1	127.8 (2)	C19A—C8A—N1A	128.5 (2)
C14—C9—C10	136.7 (2)	C14A—C9A—C10A	135.8 (2)
O2—C9—C10	114.3 (2)	O2A—C9A—C10A	114.9 (2)
O3—C10—C9	117.3 (2)	C13A—C10A—C9A	134.1 (2)
C13—C10—C9	133.1 (2)	O3A—C10A—C9A	115.5 (2)
C9—C14—C15	131.3 (2)	C9A—C14A—C15A	130.5 (2)
C19—C14—C15	123.5 (2)	C19A—C14A—C15A	123.9 (2)
C18—C15—C14	129.9 (2)	O4A—C15A—C14A	120.8 (2)
O4—C15—C14	121.1 (2)	C18A—C15A—C14A	129.1 (2)
C8—C19—C20	122.6 (2)	C8A—C19A—C20A	123.7 (2)
C14—C19—C20	129.2 (2)	C20A—C19A—C14A	128.8 (2)
N2—C20—C19	177.6 (2)	N2A—C20A—C19A	175.9 (2)

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

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N1	0.82 (4)	1.88 (4)	2.596 (2)	147 (4)
O1A—H1A...N1A	0.81 (4)	1.91 (3)	2.605 (3)	142 (4)
C3A—H3A...N2 ⁱ	0.96	2.53	3.385 (4)	149
C12—H12...N2 ⁱⁱ	0.96	2.41	3.341 (4)	163
C13—H13...O4	0.96	2.26	2.962 (3)	130
C13A—H13A...O4A	0.96	2.39	2.993 (3)	121
C4—H4...O3A	0.96	2.83	3.406 (4)	120
C16A—H16A...O3	0.96	2.74	3.337 (4)	121
C17A—H17A...O3	0.96	2.88	3.399 (4)	115
C16—H16...N2A ⁱⁱⁱ	0.96	2.68	3.354 (4)	128

Symmetry codes: (i) $x-1, 1+y, z-1$; (ii) $x, 1+y, z$; (iii) $1-x, 1-y, 1-z$.

The structure was solved by direct-phase determination. The parameters of the complete structure could be refined by full-matrix least-squares methods including anisotropic displacement parameters for non-H atoms. H atoms bonded to C atoms were refined using a riding model and H-atom displacement parameters were restricted to be $1.2U_{\text{eq}}$ of the parent atom. The hydroxyl H atoms were located in a difference electron-density map and their positions were allowed to refine, but their displacement parameters were also constrained to be $1.2U_{\text{eq}}$ of the parent atom.

Data collection: *Nonius Diffractometer Control Software* (Nonius GmbH, 1993). Cell refinement: *Nonius Diffractometer Control Software*. Data reduction: *REDU4* (Stoe & Cie, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1070). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 260–262

Ethyl mustard oil sulfide hydrochloride

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(Received 16 February 1998; accepted 21 August 1998)

Abstract

In the title compound, *N*-(4-ethyl-5-thioxo-1,2,4-dithiazolidin-3-ylidene)ethylammonium chloride, C₆H₁₁N₂S₃·Cl[−], the five-membered C₂NS₂ ring is essentially