Refinement

Refinement on F^2
$R(F) = 0.0828$
$wR(F^2) = 0.2541$
$S = 0.993$
1848 reflections
152 parameters
H atoms: riding model

$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} = -0.16$
$\Delta\rho_{\text{max}} = 0.28$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.21$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

It was not possible to locate the eight water protons and thus unambiguously identify the fluoride ion. We therefore performed five separate refinements with the fluoride in each of the five possible positions. The final model was chosen because it had the lowest s.u.'s (both in the bond lengths and intermolecular contacts) and R value. In addition, the chosen model placed the fluoride such that it made four contacts with solvent molecules at distances of 2.63–2.68 Å; in the four other models, the fluoride made only three contacts with distances of 2.63–2.83 Å. Furthermore, in the alternative refinements, some distances contracted giving O···O distances which were shorter than the F···O distances. It should be noted that as the data set was collected at room temperature and the crystal diffracted weakly, the proportion of observed data was rather low. This has probably contributed to the problems in locating the water H atoms.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1992). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

The authors would like to thank Edward Croft (University of York) for help with the figures and Madeleine Moore (University of York) for helpful discussions. LC and DJN would like to thank the EPSRC for funding and JHC would like to thank the RAEng–EPSRC for a Clean Technology Fellowship.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BM1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 791–793

2-[(2-Iodophenyl)iminomethyl]phenol

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(Received 23 October 1996; accepted 16 January 1997)

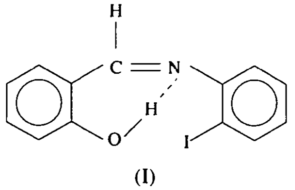
Abstract

Molecules of the title compound, $C_{13}H_{10}INO$, are non-planar and contain an intramolecular O—H···N hydrogen bond.

Comment

Schiff bases have been widely used as ligands in the formation of transition metal complexes (Calligaris & Randaccio, 1987). N-Substituted salicylaldimines are also of interest because of their thermochromism and photochromism in the solid state which may involve reversible proton transfer from the hydroxyl O to the imine N atom (Moustakali, Mavridis & Hadjoudis, 1978; Hadjoudis, Vitterakis & Mavridis, 1987; Xu, You, Sun, Wang & Liu, 1994) and charge transport occurs through intermolecular overlap between π orbitals.

Our structural investigations of Schiff bases (Elerman, Svoboda & Fuess, 1991; Elerman, Paulus, Svoboda & Fuess, 1992; Elerman, Elmali, Kabak, Aydin & Peder, 1994; Elerman, Elmali & Svoboda, 1995; Elmali, Özbey, Kendi, Kabak & Elerman, 1995; Kevran, Elmali & Elerman, 1996) have led us to examine the title compound, (I) (Fig. 1). The space group and lattice constants of the title compound were determined previously (Bernstein, 1967) and are consistent with the corresponding values presented here. It has been proposed that Schiff base molecules exhibiting thermochromy are planar, while those exhibiting photochromy are non-planar (Moustakali *et al.*, 1978). In agreement with this, the title molecule is non-planar and photochromic (Bernstein, 1967); moieties *A* (O1, C1–C7) and *B* (N1, C8–C13, I1) [both planar with a maximum deviation of 0.025 (4) Å] are inclined at an angle of 45.7 (1)° reflecting mainly the twist about N1—C8 [C7—N1—C8—C9 43.8 (6)°]. Clearly this conformation is not suitable for direct coordination to a metal ion.



(I)

Acta Crystallographica Section C
ISSN 0108-2701 © 1997

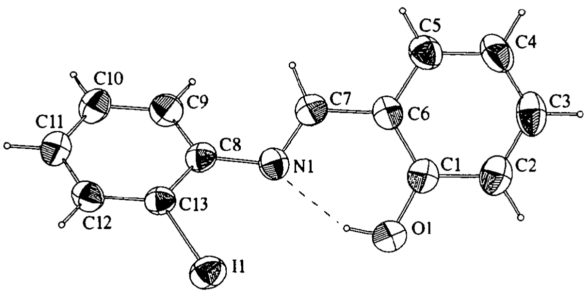


Fig. 1. The molecular structure of the title compound (*ORTEP*II; Johnson, 1976). Displacement ellipsoids are plotted at the 50% probability level.

In compound (I), a strong intramolecular hydrogen bond occurs between the O1 and N1 atoms [2.624 (5) Å], the H atom being bonded to the O1 atom. This intramolecular O1···N1 distance is comparable to those observed for *N*-(2-hydroxyphenyl)-salicylaldimine [2.625 (7) Å; Elerman *et al.*, 1995], 2,2′-azinodimethyldiphenol [2.611 (6) Å; Xu *et al.*, 1994] and *N*,*N*′-bis(*p*-chlorosalicylideneamine)-1,2-diaminobenzene [2.615 (6) Å; Elerman *et al.*, 1994]. The N1—C7 [1.267 (5) Å] and O1—C1 [1.339 (5) Å] distances are consistent with those found in similar compounds (Elerman *et al.*, 1991, 1992, 1994, 1995; Elmali *et al.*, 1995; Kevran *et al.*, 1996), as is the C13—I1 bond length [2.088 (4) Å] [*cf.* values quoted by Balasubramanian & Muthiah (1996), Loll, Gravito, Carrell & Carrell (1996) and Prince, Fronczek & Gandour (1996)].

Experimental

The title compound was obtained from the reaction of 2-iodoaniline (0.01 mol) with salicylaldehyde (0.01 mol) in 50 ml ethanol. The precipitate which separated from the solution was recrystallized from dioxane solution.

Crystal data

$C_{13}H_{10}INO$
$M_r = 323.12$
Orthorhombic
$P2_12_12_1$
$a = 7.4538 (6)$ Å
$b = 12.563 (2)$ Å
$c = 12.9830 (10)$ Å
$V = 1215.8 (2)$ Å^3
$Z = 4$
$D_x = 1.765$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 3.24$–$12.13°$
$\mu = 2.61$ mm^{-1}
$T = 297 (2)$ K
Prism
0.45 × 0.20 × 0.20 mm
Light beige

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (*SDP*; Frenz, 1985)
$T_{\min} = 0.503$, $T_{\max} = 0.593$
2251 measured reflections
1695 independent reflections
1634 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.0122$
$\theta_{\max} = 23°$
$h = -8 \rightarrow 8$
$k = -1 \rightarrow 13$
$l = -1 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: 0.1%

Refinement

Refinement on F^2
$R(F) = 0.0217$
$wR(F^2) = 0.0589$
$S = 1.093$
1695 reflections
154 parameters
H atoms: riding with fixed U's, except for those bonded to C7 and O1, which were freely refined
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2 + 0.5571P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.007$
$\Delta\rho_{\max} = 0.63$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = −0.04 (3)

Table 1. *Selected geometric parameters (Å, °)*

C1—O1	1.339 (5)	C8—N1	1.404 (5)
C6—C7	1.466 (6)	C13—I1	2.088 (4)
C7—N1	1.267 (5)		
O1—C1—C6	122.0 (4)	C13—C8—N1	120.3 (4)
O1—C1—C2	118.8 (4)	C12—C13—I1	118.8 (3)
N1—C7—C6	122.1 (4)	C8—C13—I1	120.0 (3)
C9—C8—N1	122.3 (4)	C7—N1—C8	119.2 (4)

Values of distances and angles in the rings show no significant differences from those of an ideal benzene ring.

Data collection: *SDP* (Frenz, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); direct methods. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: MU1303). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 793–794

3-Phenylsulfonyl-3-(2-propenyl)chroman-4-one

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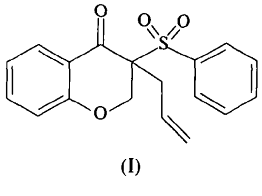
(*Received 2 September 1996; accepted 24 January 1997*)

Abstract

In the title compound, $C_{18}H_{16}O_4S$, the pyran ring adopts a sofa conformation. The bond angles around the two planar C atoms in the ring deviate from their ideal values.

Comment

Isoflavonoids, which are built upon a 3-phenylchroman skeleton, represent an important and distinctive subclass of flavonoids. They are found in plants belonging to the subfamily Papilionoidae of the Leguminosae and are known to possess antifungal and antibacterial properties (Dewick, 1988). The title compound, (I), is a key intermediate in the synthesis of isoflavonones.



(I)

The bond angles around the C3 and C9 atoms deviate considerably from the ideal value of 120°. The shortening of the double bond between the C17 and C18 atoms may be due to the large thermal motion of the C18 atom. The dihedral angle between the two phenyl rings is 33.6 (1)°. The pyran ring adopts a sofa conformation, with C1 displaced by 0.532 Å from the mean plane formed by the other atoms in the ring (C2, C3, C8, C9 and O1), instead of the normal half-chair conformation (Alex, Srinivasan, Krishnasamy, Suresh, Iyer & Iyer, 1993). The crystal structure is stabilized by intermolecular van der Waals contacts.

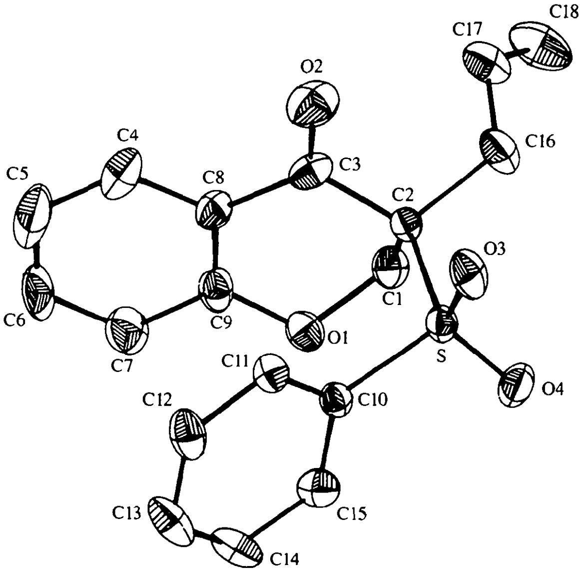


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecular structure and atom numbering of (I). The displacement ellipsoids are drawn at the 50% probability level.

Experimental

3-Phenylsulfonylchroman-4-one in DMF was added to sodium hydride also in DMF. To this solution was added an excess of 3-bromopropene and the mixture was kept in an ice bath and stirred for 2 h. The reaction mixture was purified by column chromatography to yield the propenylated sulfone as a yellow solid. The compound was characterized by IR, NMR and mass spectral studies (Santhosh, 1994).

Crystal data

$C_{18}H_{16}O_4S$
$M_r = 328.37$
Monoclinic
$P2_1/c$
$a = 9.890\,(3)$ Å
$b = 14.122\,(3)$ Å
$c = 11.856\,(3)$ Å
$\beta = 98.04\,(2)°$
$V = 1639.5\,(7)$ Å^3
$Z = 4$
$D_x = 1.330$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8–14°$
$\mu = 0.214$ mm^{-1}
$T = 293\,(2)$ K
Cylindrical
0.40 × 0.25 × 0.22 mm
Yellow

Acta Crystallographica Section C
ISSN 0108-2701 © 1997