C1-C10	1.401 (3)	C12-C13	1.526 (4)			
C2-C3	1.387 (4)	C13-C14	1.547 (3)			
C3—C4	1.373 (3)	C13-C17	1.531 (3)			
C4—C5	1.397 (3)	C13C18	1.537 (4)			
C5C6	1.516 (4)	C14—C15	1.531 (3)			
C5-C10	1.389 (4)	C15-C16	1.520 (4)			
C6C7	1.517 (4)	C16C17	1.545 (4)			
C7—C8	1.524 (3)	$O2 \cdot \cdot \cdot F$	2.613 (3)			
C3-01-C19	116.6 (2)	C5-C10-C9	121.0 (2)			
C2-C1-C10	121.7 (2)	C9-C11-C12	112.9 (2)			
C1-C2-C3	120.0 (2)	C11C12C13	111.4 (2)			
01C3C2	115.6 (2)	C12-C13-C14	108.3 (2)			
O1C3C4	124.9 (2)	C12-C13-C17	116.6 (2)			
C2-C3-C4	119.4 (2)	C12-C13-C18	110.9 (2)			
C3-C4-C5	120.7 (2)	C14C13C17	100.5 (2)			
C4C5C6	117.4 (2)	C14—C13—C18	113.2 (2)			
C4-C5-C10	120.7 (2)	C17C13C18	107.0 (2)			
C6C5C10	122.0 (2)	C8-C14-C13	112.2 (2)			
C5-C6-C7	113.2 (2)	C8-C14-C15	120.7 (2)			
C6C7C8	110.0 (2)	C13-C14-C15	104.1 (2)			
C7-C8-C9	108.9 (2)	C14-C15-C16	104.9 (2)			
C7-C8-C14	113.5 (2)	F-C16-C15	112.0 (2)			
C9C8C14	107.9 (2)	FC16C17	109.4 (2)			
C8-C9-C10	111.5 (2)	C15-C16-C17	107.1 (2)			
C8-C9-C11	112.1 (2)	O2-C17-C13	110.0 (2)			
C10C9C11	113.4 (2)	O2-C17-C16	110.9 (2)			
C1-C10-C5	117.6 (2)	C13-C17-C16	102.8 (2)			
C1-C10-C9	121.3 (2)	O2—H25···F	114.2 (3)			
Symmetry code: (i) $2 - x, y - 0.5, 2.5 - z$; (ii) $x, 0.5 - y, 0.5 + z$;						

(iii) x, -0.5 - y, 0.5 + z.

ortho-Fluorophenylglycine (Aldrich Chemical Co.) was recrystallized from H₂O/ethanol. Methyl 4-(fluorocarbonyl)benzoate was synthesized from terephthalic acid monomethyl ether by treatment with SF₄ (Hasek, Smith & Englehardt, 1960) and recrystallized from benzene/hexane. The 16 α -fluoro- α -estradiol 3-methyl ether was prepared from estrone 3-methyl ether (Sigma Chemical Co.) by fluorination of the trimethylsilyl derivative of the enol ether with fluoropyridinium trifluoromethanesulfonate (Umemoto, Tomita & Kawada, 1990), followed by reduction of the α -fluoroketone with NaBH₄ in methanol. Crystals were obtained from hexane. Calculations were performed with the *Enraf–Nonius Structure Determination Package* (Frenz, 1983). The structure was solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier maps.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, as well as a stero diagram showing the hydrogen-bond network of *o*-fluorophenylglycine, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71016 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1019]

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Structure of 4-[(3-Methoxyphenylimino)methyl]phenol

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Abstract

3-Methoxy-N-(4-hydroxybenzylidene)aniline exhibits a non-planar conformation with a dihedral angle C7—N—C8—C13 of 45.9° between the planes of the two phenyl rings. The methoxy group is twisted from the phenyl ring by 3.0°.

Comment

The interaction of Schiff bases having a 2-hydroxy substituent on the benzylidene fragment with several metal ions has been described (Bullock, Ladd, Povey & Tajmir-Riahi, 1979; Kamwaya & Khoo, 1985; Fun, Teo, Teoh & Yeap, 1991). As part of our study on the coordination behaviour of a ligand having a 4-hydroxy substituent on the benzylidene fragment, X-ray structural analysis of the title compound was carried out; the results are reported herein. The molecule exists in the solid state in a non-planar conformation arising mainly from the rotation of the benzylidene moiety about the N-C8 bond with a C13-C8-N1-C7 torsion angle of 45.9°. The methoxy group is twisted from the aniline ring by 3.0° in order to minimize the interaction between the methyl protons and H9. For the same reason, the O2-C10-C9 angle $[124.3 (2)^{\circ}]$ is larger than the O2-C10-C11 angle $[115.2 (2)^{\circ}]$.



Fig. 1. A perspective view of the molecule showing the labelling of the non-H atoms.

Experimental

Crystal data

C14H13NO2 $M_r = 227.26$ Monoclinic P2/aa = 12.248 (2) Å b = 8.637 (1) Åc = 10.934 (3) Å $\beta = 92.25 (1)^{\circ}$ V = 1155.8 (7) Å³ Z = 4 $D_x = 1.306 \text{ Mg m}^{-3}$ $D_m = 1.309 \text{ Mg m}^{-3}$

Data collection

Syntex P21 diffractometer $2\theta/\theta$ scans, speed 5.0-29.3 min⁻¹ Absorption correction: empirical $T_{\min} = 0.8847, T_{\max} =$ 0.9762 2630 measured reflections 2356 independent reflections 1892 observed reflections $[I > 2.5\sigma(I)]$

Mo $K\alpha$ radiation $\lambda = 0.71069 \text{ Å}$ Cell parameters from 30 reflections $\theta = 17.5 - 22.5^{\circ}$ $\mu = 0.08 \text{ mm}^{-1}$ T = 298 KNeedle $0.3 \times 0.2 \times 0.2$ mm Yellow Crystal source: prepared according to Srivastava & Chauhan (1977)

 $R_{\rm int} = 0.1941$ $\theta_{\rm max}$ = 26.1° $h = 0 \rightarrow 14$ $k = 0 \rightarrow 10$ $l = -12 \rightarrow 12$ 1 standard reflection monitored every 50 reflections intensity variation: none

Refinement

Cl C_2

C3 C4 C5

C6 01

C7 Ν

C8 C9 C10 C11

C12

C13 02

C14

 $w = 5.0845/[\sigma^2(F)]$ Refinement on F $+ 0.000264F^{2}$] Final R = 0.0526wR = 0.0566 $(\Delta/\sigma)_{\rm max} = 0.003$ $\Delta \rho_{\rm max} = 0.206 \ {\rm e} \ {\rm \AA}^{-3}$ S = 1.321892 reflections $\Delta \rho_{\rm min} = -0.201 \ {\rm e} \ {\rm \AA}^{-3}$ 197 parameters Atomic scattering factors from SHELX76 Only H-atom U's refined; riding model (C-H (Sheldrick, 1976) 0.96 Å)

Program used to solve structure: SHELXS86 (Sheldrick, 1986). Program used to refine structure: SHELX76 (Sheldrick, 1976). Program used for absorption correction: ABSORB (Ugozzoli, 1987). Program used for geometrical calculations: XANADU (Roberts & Sheldrick, 1975). Program used for molecular drawing: ORTEP (Johnson, 1965).

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters $(Å^2)$

$B_{\rm eq} = \frac{8\pi^2}{3}(U_{11} + U_{22} + U_{33}).$				
	x	у	z	Beq
	0.5972 (2)	0.3449 (2)	0.3986 (2)	2.91 (8)
	0.7003 (2)	0.3102 (3)	0.4507 (2)	3.32 (9)
	0.7120 (2)	0.2167 (3)	0.5516 (2)	3.26 (8)
	0.6203 (2)	0.1528 (2)	0.6042 (2)	2.86 (8)
	0.5172 (2)	0.1859 (3)	0.5533 (2)	3.62 (9)
	0.5064 (2)	0.2801 (3)	0.4527 (2)	3.48 (9)
	0.6273 (1)	0.0592 (2)	0.7030(1)	3.78 (7)
	0.5798 (2)	0.4399 (2)	0.2903 (2)	3.12 (8)
	0.6553 (1)	0.5021 (2)	0.2306(1)	2.99 (7)
	0.6236 (2)	0.5928 (2)	0.1257 (2)	2.98 (8)
	0.6818 (2)	0.5727 (2)	0.0203 (2)	3.24 (8)
	0.6531 (2)	0.6588 (3)	-0.0828 (2)	3.47 (9)
	0.5714 (2)	0.7693 (3)	-0.0789 (2)	3.91 (10)
	0.5154 (2)	0.7887 (3)	0.0261 (2)	4.15 (11)
	0.5402 (2)	0.7002 (3)	0.1294 (2)	3.71 (10)
	0.6997 (1)	0.6424 (2)	-0.1935 (1)	4.78 (8)
	0.7792 (2)	0.5256 (4)	-0.2056 (2)	5.72 (14)

Table 2. Geometric parameters (Å, °)

C2-C1	1.397 (3)	C8N	1.430 (2)
C6C1	1.398 (3)	C9C8	1.389 (3)
C7-C1	1.450 (3)	C13—C8	1.382 (3)
C3-C2	1.370 (3)	C10—C9	1384 (3)
C4C3	1.396 (3)	C11-C10	1.385 (3)
C5—C4	1.390 (3)	O2-C10	1.367 (2)
01-C4	1.349 (2)	C12C11	1.370 (3)
C6-C5	1.371 (3)	C13-C12	1.388 (3)
NC7	1.272 (2)	C14—O2	1.411 (3)
C6C1C2	117.6 (2)	C9—C8—N	117.7 (2)
C7-C1-C2	123.8 (2)	C13C8N	121.5 (2)
C7-C1-C6	118.6 (2)	C13-C8-C9	120.8 (2)
C3-C2-C1	121.3 (2)	C10-C9-C8	119.1 (2)
C4-C3-C2	120.3 (2)	C11-C10-C9	120.4 (2)
C5-C4-C3	119.2 (2)	O2-C10-C9	124.3 (2)
O1-C4-C3	122.7 (2)	O2-C10-C11	115.2 (2)
01C4C5	118.1 (2)	C12-C11-C10	119.7 (2)
C6-C5-C4	120.0 (2)	C13-C12-C11	120.9 (2)
C5-C6-C1	121.6 (2)	C12-C13-C8	119.0 (2)
N-C7-C1	124.8 (2)	C14	118.1 (2)
C8-N-C7	117.5 (2)		

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71028 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1028]

Comment

Biisoquinolinium salt (I) and polymethylene-bridged analogs (III) and (IV) are reported to produce blue chemiluminescence in hydroxylic solvents by addition of base (Mason & Roberts, 1967). Heller, Henry

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Structure of Diiodide Salts of *N*-Heteroaromatic Dications

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Abstract

The structures of 2,2'-dimethyl-1,1'-biisoquinolinium diiodide monohydrate (I) and 1,1'-dimethyl-4,4'biquinolinium diiodide (II) have been determined. Both biisoquinolinium and biquinolinium dications have a twofold rotation axis. The two Nheteroaromatic planes in (I) are twisted from each other to a greater extent than those in (II); this can be explained by steric repulsion of the N-methyl groups in (I). In both crystals each I⁻ anion is in a relatively close contact with one of the positively charged N atoms of the dications. These interionic interactions seem to be responsible for the deep red color of the crystals.

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& Fritsch (1973) proposed that the chemiluminescence reaction involved two-electron reduction of the biisoquinolinium dications and subsequent oxygenation and then cleavage to yield excited carbonyl compounds. Recently, with interest in the relationship between structure and chemiluminescence efficiency and electronic redox property, we determined crystal structures of (III) and (IV) (Mori, Matsuyama, Yamada & Maeda, 1992). In these crystals the biisoquinolinium dications adopt a C_2 conformation. Unlike (I), (III) or (IV), biquinolinium salt (II) produced no chemiluminescence in the same alkaline solution. (II) showed a charge-transfer (CT) absorption band around 460 nm in dimethylformamide (DMF) (Ishii, 1986), while (I) showed a weak CT absorption band around 440 nm in DMF and the intensity of the CT band increased in less polar media such as DMF-dichloromethane (3:1 v/v)mixed solvent. These spectral properties indicate that the organic dication and I^- form a CT complex in the ground state in both (I) and (II). The characteristic red color of the crystal may be a result of the CT interaction. It has been reported, however, that in the crystal of 1-ethyl-2-methylquinolinium iodide, interionic interaction is weak and an ion-pair does not exist (Sakanoue, Kai, Yasuoka, Kasai & Kakudo, 1970). In the present study, structures of (I) and (II) have been determined to investigate conformational features and interaction between the ions.

As shown in Fig. 1, both (I) and (II) show C_2 symmetry and the molecular axes coincide with the crystallographic twofold axes. In (I), a water molecule also is located on the C_2 axis. Most of the bond distances and angles in (I) are similar to the corresponding ones in (III) and (IV) (Mori, Matsuyama, Yamada & Maeda, 1992). The C(1)—N(2)—C(11) angle is somewhat enlarged, while C(3)—N(2)—C(11) is significantly narrower than 120°. The isoquinoline ring of (I) is almost planar with maximum deviation of 0.022 Å at C(8); the *N*-methyl C atom C(11) deviates by only 0.054 Å from the plane. The two N atoms N(2) and N(2') are located on opposite