| 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) | C13-C18 | 1.537 (4) |
| C5-C6 | 1.516 (4) | C14-C15 | 1.531 (3) |
| C5-C10 | 1.389 (4) | C15-C16 | 1.520 (4) |
| C6-C7 | 1.517 (4) | C16-C17 | 1.545 (4) |
| C7-C8 | 1.524 (3) | O2..F | 2.613 (3) |
| C3-O1-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) | C11-C12-C13 | 111.4 (2) |
| O1-C3-C2 | 115.6 (2) | C12-C13-C14 | 108.3 (2) |
| O1-C3-C4 | 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) | C14-C13-C17 | 100.5 (2) |
| C4-C5-C6 | 117.4 (2) | C14-C13-C18 | 113.2 (2) |
| C4-C5-C10 | 120.7 (2) | C17-C13-C18 | 107.0 (2) |
| C6-C5-C10 | 122.0 (2) | C8-C14-C13 | 112.2 (2) |
| C5-C6-C7 | 113.2 (2) | C8-C14-C15 | 120.7 (2) |
| C6-C7-C8 | 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) |
| C9-C8-C14 | 107.9 (2) | F-C16-C17 | 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) |
| C10-C9-C11 | 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) $x, 0.5-y, 0.5+z$;

$$
\text { (iii) } x,-0.5-y, 0.5+z
$$

ortho-Fluorophenylglycine (Aldrich Chemical Co.) was recrystallized from $\mathrm{H}_{2} \mathrm{O}$ /ethanol. Methyl 4 -(fluorocarbonyl)benzoate was synthesized from terephthalic acid monomethyl ether by treatment with $\mathrm{SF}_{4}$ (Hasek, Smith \& Englehardt, 1960) and recrystallized from benzene/hexane. The $16 \alpha$-fluoro- $\alpha$-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 $\alpha$-fluoroketone with $\mathrm{NaBH}_{4}$ in methanol. Crystals were obtained from hexane. Calculations were performed with the Enraf-Nonius Structure Determination Package (Frenz, 1983). The structure was solved by MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq \& Woolfson, 1982) and difference Fourier maps.

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## Structure of 4-[(3-Methoxyphenylimino)methyllphenol

Guan-Yeow Yeap and Soon-Beng Teo
School of Chemical Sciences,
Universiti Sains Malaysdia, Minden, 11800 Penang, Malaysia

## Hoong-Kun Fun and Siang-Guan Teoh*

School of Physics, Universiti Sains Malaysia, Minden, 11800 Penang, Malaysia
(Received 5 June 1992; accepted 18 January 1993)


#### Abstract

3-Methoxy- $N$-(4-hydroxybenzylidene)aniline exhibits a non-planar conformation with a dihedral angle $\mathrm{C} 7-\mathrm{N}-\mathrm{C} 8-\mathrm{C} 13$ of $45.9^{\circ}$ between the planes of the two phenyl rings. The methoxy group is twisted from the phenyl ring by $3.0^{\circ}$.


## 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 $\mathrm{N}-\mathrm{C} 8$ bond with a $\mathrm{Cl} 3-\mathrm{C} 8-\mathrm{N} 1-\mathrm{C} 7$ torsion angle of $45.9^{\circ}$. The methoxy group is twisted from the aniline ring by $3.0^{\circ}$ in order to minimize the interaction between the methyl protons and H9. For the same reason, the $\mathrm{O} 2-\mathrm{Cl0}-\mathrm{C} 9$ angle $\left[124.3(2)^{\circ}\right.$ ] is larger than the $\mathrm{O} 2-\mathrm{Cl} 0-\mathrm{Cll}$ angle $\left[115.2(2)^{\circ}\right]$.


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

## Experimental

Crystal data
$\mathrm{C}_{14} \mathrm{H}_{13} \mathrm{NO}_{2}$
$M_{r}=227.26$
Monoclinic
P2/a
$a=12.248$ (2) $\AA$
$b=8.637$ (1)
$c=10.934(3) \AA$
$\beta=92.25(1)^{\circ}$
$V=1155.8(7) \AA^{3}$
$Z=4$
$D_{x}=1.306 \mathrm{Mg} \mathrm{m}^{-3}$
$D_{m}=1.309 \mathrm{Mg} \mathrm{m}^{-3}$

Data collection
Syntex $P 2_{1}$ diffractometer
$2 \theta 1 \theta$ scans, speed 5.0-29.3 $\min ^{-1}$
Absorption correction: empirical
$T_{\text {min }}=0.8847, T_{\text {max }}=$ 0.9762

2630 measured reflections 2356 independent reflections 1892 observed reflections
[ $I>2.5 \sigma(I)$ ]

Refinement
Refinement on $\boldsymbol{F}$
Final $R=0.0526$
$w R=0.0566$
$S=1.32$
1892 reflections
197 parameters
Only H-atom $U$ 's refined; riding model ( $\mathrm{C}-\mathrm{H}$

$$
\begin{aligned}
& w=5.0845 /\left[\sigma^{2}(F)\right. \\
& \left.\quad+0.000264 F^{2}\right] \\
& (\Delta / \sigma)_{\max }=0.003 \\
& \Delta \rho_{\max }=0.206 \mathrm{e}^{-3} \AA^{-3} \\
& \Delta \rho_{\min }=-0.201 \mathrm{e}^{-3}
\end{aligned}
$$

Atomic scattering fac-
tors from SHELX76
(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 ( $\AA^{2}$ )

| $B_{\text {eq }}=\frac{8 \pi^{2}}{3}\left(U_{11}+U_{22}+U_{33}\right)$ |  |  |  |  |
| :--- | :---: | :---: | ---: | :---: |
|  | $\boldsymbol{x}$ | $y$ | $z$ | $B_{\text {eq }}$ |
| C1 | $0.5972(2)$ | $0.3449(2)$ | $0.3986(2)$ | $2.91(8)$ |
| C2 | $0.7003(2)$ | $0.3102(3)$ | $0.4507(2)$ | $3.32(9)$ |
| C3 | $0.7120(2)$ | $0.2167(3)$ | $0.5516(2)$ | $3.26(8)$ |
| C4 | $0.6203(2)$ | $0.1528(2)$ | $0.6042(2)$ | $2.86(8)$ |
| C5 | $0.5172(2)$ | $0.1859(3)$ | $0.5533(2)$ | $3.62(9)$ |
| C6 | $0.5064(2)$ | $0.2801(3)$ | $0.4527(2)$ | $3.48(9)$ |
| O1 | $0.6273(1)$ | $0.0592(2)$ | $0.7030(1)$ | $3.78(7)$ |
| C7 | $0.5798(2)$ | $0.4399(2)$ | $0.2903(2)$ | $3.12(8)$ |
| N | $0.6553(1)$ | $0.5021(2)$ | $0.2306(1)$ | $2.99(7)$ |
| C8 | $0.6236(2)$ | $0.5928(2)$ | $0.1257(2)$ | $2.98(8)$ |
| C9 | $0.6818(2)$ | $0.5727(2)$ | $0.0203(2)$ | $3.24(8)$ |
| C10 | $0.6531(2)$ | $0.6588(3)$ | $-0.0828(2)$ | $3.47(9)$ |
| C11 | $0.5714(2)$ | $0.7693(3)$ | $-0.0789(2)$ | $3.91(10)$ |
| C12 | $0.5154(2)$ | $0.7887(3)$ | $0.0261(2)$ | $4.15(11)$ |
| C13 | $0.5402(2)$ | $0.7002(3)$ | $0.1294(2)$ | $3.71(10)$ |
| O2 | $0.6997(1)$ | $0.6424(2)$ | $-0.1935(1)$ | $4.78(8)$ |
| C14 | $0.7792(2)$ | $0.5256(4)$ | $-0.2056(2)$ | $5.72(14)$ |

Table 2. Geometric parameters ( $\AA,{ }^{\circ}$ )

| C2-C1 | 1.397 (3) | C8-N | 1.430 (2) |
| :---: | :---: | :---: | :---: |
| C6-C1 | 1.398 (3) | C9-C8 | 1.389 (3) |
| C7-C1 | 1.450 (3) | C13-C8 | 1.382 (3) |
| C3-C2 | 1.370 (3) | C10-C9 | 1384 (3) |
| C4-C3 | 1.396 (3) | C11-C10 | 1.385 (3) |
| C5-C4 | 1.390 (3) | O2-C10 | 1.367 (2) |
| O1-C4 | 1.349 (2) | C12-C11 | 1.370 (3) |
| C6-C5 | 1.371 (3) | C13-C12 | 1.388 (3) |
| $\mathrm{N}-\mathrm{C} 7$ | 1.272 (2) | C14-O2 | 1.411 (3) |
| C6-C1-C2 | 117.6 (2) | $\mathrm{C} 9-\mathrm{C8}-\mathrm{N}$ | 117.7 (2) |
| C7-C1-C2 | 123.8 (2) | C13-C8-N | 121.5 (2) |
| C7-C1-C6 | 118.6 (2) | C13-C8-C9 | 120.8 (2) |
| C3-C2-Cl | 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) |
| O1-C4-C5 | 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) |
| $\mathrm{N}-\mathrm{C7}-\mathrm{Cl}$ | 124.8 (2) | C14-O2-Cl0 | 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 CHl 2HU, England. [CIF reference: HH1028]

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## Structure of Diiodide Salts of $\boldsymbol{N}$-Heteroaromatic Dications

Yukie Mori, Yoko Matsuyama, Junko Suzuki, Yukiko Ishi, Sachiko Yamada and Koko Maeda*<br>Department of Chemistry, Faculty of Science, Ochanomizu University, Otsuka, Bunkyo-ku, Tokyo 112, Japan

(Received 25 September 1992; accepted 17 January 1993)


#### Abstract

The structures of $2,2^{\prime}$-dimethyl-1, $1^{\prime}$-biisoquinolinium diiodide monohydrate (I) and $1,1^{\prime}$-dimethyl-4,4'biquinolinium diiodide (II) have been determined. Both biisoquinolinium and biquinolinium dications have a twofold rotation axis. The two N heteroaromatic 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 $\mathrm{I}^{-}$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.


## 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

(I)

(II)

(III) $n=2$
(IV) $n=3$
\& 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 ( $3: 1 \mathrm{v} / \mathrm{v}$ ) mixed solvent. These spectral properties indicate that the organic dication and $\mathrm{I}^{-}$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 $\mathrm{C}(1)-\mathrm{N}(2)-\mathrm{C}(11)$ angle is somewhat enlarged, while $\mathrm{C}(3)-\mathrm{N}(2)-$ $\mathrm{C}(11)$ is significantly narrower than $120^{\circ}$. The isoquinoline ring of (I) is almost planar with maximum deviation of $0.022 \AA$ at $\mathrm{C}(8)$; the $N$-methyl C atom $\mathrm{C}(11)$ deviates by only $0.054 \AA$ from the plane. The two N atoms $\mathrm{N}(2)$ and $\mathrm{N}\left(2^{\prime}\right)$ are located on opposite


[^0]:    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]

