

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) $x, 0.5 - y, 0.5 + z$;
(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 *MULTAN11/82* (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 stereo 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]

References

- Bailey, M. & Brown, C. J. (1967). *Acta Cryst.* **22**, 387–391.
 Brisse, F. & Pérez, S. (1976). *Acta Cryst.* **B32**, 2110–2115.
- Busetta, B., Courseille, C., Geoffre, S. & Hospital, M. (1972). *Acta Cryst.* **B28**, 1349–1351.
 Busetta, B. & Hospital, M. (1972). *Acta Cryst.* **B28**, 560–567.
 Duax, W. L. (1972). *Acta Cryst.* **B28**, 1864–1871.
 Frenz, B. A. (1983). *Enraf-Nonius Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
 Go, K. & Kartha, G. (1981). *Am. Crystallogr. Assoc. Abstr. Ser.* **2**, 9, 33.
 Hagaman, E. W. & Burns, J. H. (1993). *Fuel*. In the press.
 Hagaman, E. W., Ho, P. C., Brown, L. L., Schell, F. M. & Woody, M. C. (1990). *J. Am. Chem. Soc.* **112**, 7445–7450.
 Hasek, W. R., Smith, W. C. & Englehardt, V. A. (1960). *J. Am. Chem. Soc.* **82**, 543–551.
 Jönsson, P.-G. & Kvik, Å. (1972). *Acta Cryst.* **B28**, 1827–1833.
 Kashino, S. & Haisa, M. (1975). *Acta Cryst.* **B31**, 1819–1822.
 Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 Marsh, R. E. (1958). *Acta Cryst.* **11**, 654–663.
 Norton, D. A., Kartha, G. & Lu, C. T. (1964). *Acta Cryst.* **17**, 77–82.
 Umemoto, T., Tomita, K. & Kawada, K. (1990). *Org. Synth.* **69**, 129–143.
- Acta Cryst.* (1993). **C49**, 1396–1398

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;

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]

References

- Bullock, J. I., Ladd, M. F. C., Povey, D. C. & Tajmir-Riahi, H. A. (1979). *Acta Cryst.* **B35**, 2013–2020.
 Fun, H.-K., Teo, S.-B., Teoh, S.-G. & Yeap, G.-Y. (1991). *Acta Cryst.* **C47**, 1824–1826.
 Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 Kamwaya, M. E. & Khoo, L. E. (1985). *Acta Cryst.* **C41**, 1027–1029.
 Roberts, P. & Sheldrick, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
 Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
 Srivastava, T. N. & Chauhan, A. K. S. (1977). *J. Inorg. Nucl. Chem.* **39**, 371–373.
 Ugozzoli, G. (1987). *Comput. Chem.* **11**, 109–120.

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Structure of Diiodide Salts of *N*-Heteroaromatic Dications

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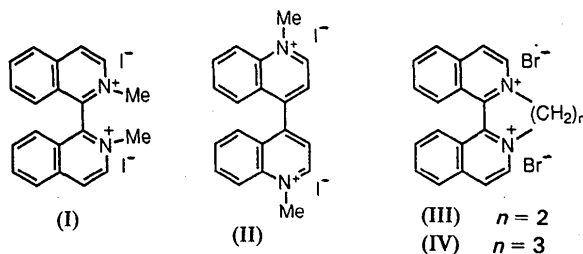
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Abstract

The structures of 2,2'-dimethyl-1,1'-bisoquinolinium diiodide monohydrate (I) and 1,1'-dimethyl-4,4'-biquinolinium diiodide (II) have been determined. Both bisoquinolinium 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 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

Bissoquinolinium 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



& Fritsch (1973) proposed that the chemiluminescence reaction involved two-electron reduction of the bisoquinolinium 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 bisoquinolinium 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