Acta Cryst. (1997). **C53**, 1649–1650

The Hydrated Tosylate of Dimeric 2′-Hydroxy-4-*N*-methylstilbazolium†

De-Chun Zhang,[a] Tian-Zhu Zhang,[a] Yan-Qiu Zhang,[a] Zheng-Hao Fei[a] and Kai-bei Yu[b]

[a]*Department of Chemistry, Suzhou University, Suzhou 215006, People's Republic of China, and* [b]*Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, Chengdu 610041, People's Republic of China. E-mail: yjhx@nsad.suda.edu.cn*

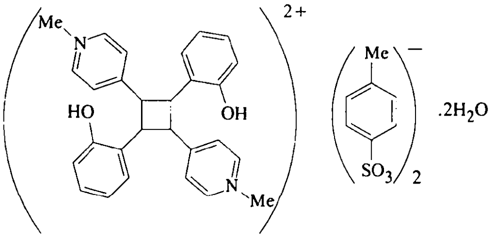
(*Received 19 November 1996; accepted 6 June 1997*)

Abstract

The centrosymmetric dimeric cation in the title compound, bis(2′-hydroxy-4-*N*-methylstilbazolium) ditoluenesulfonate dihydrate, $C_{28}H_{28}N_2O_2^{2+}.2C_7H_7SO_3^-.2H_2O$ is propeller-like, and the planar central four-membered ring makes dihedral angles of 85.0 (2) and 63.1 (2)° with the mean planes of the pyridinium and phenyl rings, respectively. The cations, anions and water molecules are connected by hydrogen bonding.

Comment

During our systematic search for organic salts with non-linear optical properties, we isolated the title compound, (I).



(I)

The cation consists of five rings; the four substituent rings have a propeller-like arrangement around the central ring (Fig. 1). Bond lengths, angles and torsion angles are all normal. The hydroxy group is nearly in the plane of its aromatic ring [torsion angle 1.5 (3)°] and is located almost equally close to the central C(15) and C(14) atoms. The exactly planar four-membered ring makes dihedral angles of 63.1 (2)° with the hydroxyphenyl substituent ring and an angle of 85.0 (2)° with the pyridinium ring.

The negatively charged sulfonate groups in the anions are near the positively charged N atoms of the cation (Fig. 1). The cations, anions and water molecules are connected by O—H···O hydrogen bonds (Table 2). In addition, there are some rather long intermolecular contacts which may be weak C—H···O hydrogen bonds (Taylor & Kennard, 1982; Berkovitch-Yellin & Leiserowitz, 1984; Krishnamohan Sharma & Desiraju, 1994).

The structure is centrosymmetric, similar to that of the related compound 2′-methoxy-2-*N*-methylstilbazolium trifluoromethanesulfonate (Marder, Perry & Tiemann, 1990), although that compound is not a dimer. Hence, these materials show no non-linear optical properties. On the other hand, another simi-

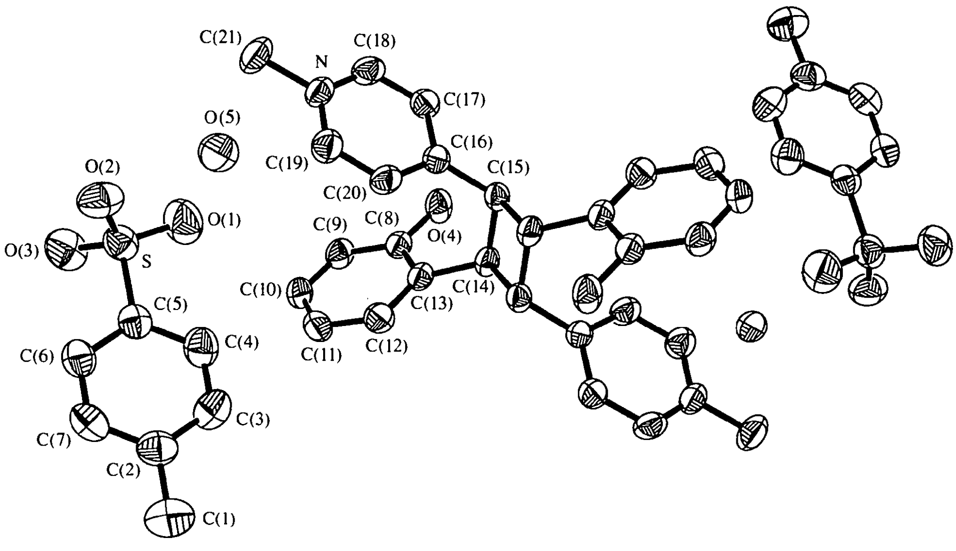


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

---

† Full systematic name: 4,4′-[2,4-bis(2-hydroxyphenyl)cyclobutane-1,3-diyl]bis(1-methylpyridinium) bis(*p*-toluenesulfonate) dihydrate.

lar compound, 1-methyl-4-[2-(4-hydroxyphenyl)vinyl]-pyridinium 4-toluenesulfonate (Okada *et al.*, 1990), crystallizing in space group $P1$, is non-linear optical; it differs from the monomer of the title compound only in the position of the hydroxy substituent, indicating the important role of the group *ortho* to the central vinyl group in the monomeric cation. We are currently studying the actual mechanism for the photochemical dimerization.

Experimental

1,4-Dimethylpyridinium iodide (7.05 g, 30 mmol) (prepared from CH_3I and 4-methylpyridine) and 5.2 ml (49 mmol) of 2-hydroxybenzaldehyde in methanol (10 ml) were heated to 353 K for 12 h. The product was recrystallized twice from water, dissolved in water again (0.68 g in 100 ml) and treated with a saturated solution of silver *p*-tolylsulfonate added drop-wise with stirring at 363 K over 20 min. The title compound was separated, recrystallized twice and finally crystals suitable for X-ray analysis were grown from methanol–water (10:1) by slow evaporation.

Crystal data

$C_{28}H_{28}N_2O_2^{2+}$.-
$2C_7H_7SO_3^-.2H_2O$
$M_r = 802.92$
Triclinic
$P\bar{1}$
$a = 8.704\ (1)$ Å
$b = 9.116\ (1)$ Å
$c = 12.968\ (2)$ Å
$\alpha = 84.69\ (1)^\circ$
$\beta = 76.63\ (1)^\circ$
$\gamma = 80.35^\circ$
$V = 985.3\ (2)$ Å^3
$Z = 1$
$D_x = 1.353$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 27 reflections
$\theta = 3.06$–15.88°
$\mu = 0.197$ mm^{-1}
$T = 295\ (2)$ K
Block
0.46 × 0.46 × 0.37 mm
Pale yellow

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
3723 measured reflections
3475 independent reflections
2531 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.010$

$\theta_{\rm max} = 24.99^\circ$
$h = 0 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 3.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.112$
$S = 0.981$
3474 reflections
345 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.230$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.344$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C(14)—C(15$^{\rm i}$)	1.553 (2)	C(14)—C(15)	1.583 (2)
C(13)—C(14)—C(15$^{\rm i}$)	120.52 (15)	C(16)—C(15)—C(14$^{\rm i}$)	117.21 (15)
C(13)—C(14)—C(15)	116.49 (14)	C(16)—C(15)—C(14)	116.16 (14)
C(15$^{\rm i}$)—C(14)—C(15)	90.43 (13)	C(14$^{\rm i}$)—C(15)—C(14)	89.57 (13)

Symmetry code: (i) $-x, -y, 1 - z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O(4)—H(4O)···O(5$^{\rm i}$)	0.81 (3)	1.85 (3)	2.653 (2)	171 (3)
O(5)—H(5OB)···O(2$^{\rm ii}$)	0.88 (3)	1.86 (3)	2.733 (2)	171 (3)
O(5)—H(5OA)···O(2)	0.79 (3)	2.01 (3)	2.802 (3)	177 (3)

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x, 1 - y, 2 - z$.

Data collection: *XSCANS* (Siemens, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1158). Services for accessing these data are described at the back of the journal.

References

Berkovitch-Yellin, Z. & Leiserowitz, L. (1984). *Acta Cryst.* B**40**, 159–165.
Krishnamohan Sharma, C. V. & Desiraju, G. R. (1994). *J. Chem. Soc. Perkin Trans.* 2, pp. 2345–2352.
Marder, S. R., Perry, J. W. & Tiemann, B. G. (1990). *Chem. Mater.* **2**, 685–690.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Okada, S., Masaki, A., Matsuda, H., Nakanishi, H., Kato, M. & Muramatsu, R. (1990). *Jpn J. Appl. Phys.* **29**, 1112–1115.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Acta Cryst. (1997). C**53**, 1650–1653

8-Amino-7-(4-morpholinobutyl)theophylline

Zbigniew Karczmarzyk^a and Maciej Pawłowski^b

^{a}*Department of Chemistry, Agricultural and Teachers University, ul. 3 Maja 54, PL-08 110 Siedlce, Poland, and* ^{b}*Department of Pharmaceutical Chemistry, Jagiellonian University, Collegium Medicum, ul. Medyczna 9, PL-30 688 Kraków, Poland. E-mail: kar@wsrp.siedlce.pl*

(*Received 25 March 1997; accepted 28 April 1997*)

Abstract

The crystal structure of the title compound, 8-amino-1,3-dimethyl-7-(4-morpholinobutyl)-3,7-dihydro-1*H*-purine-2,6-dione, $C_{15}H_{24}N_6O_3$, (III), is described and com-

Acta Crystallographica Section C
ISSN 0108-2701 © 1997