the DMF molecules in (I*a*) and the DMSO(1) guest in (II*b*) had to be refined isotropically with bond-length constraints in order to yield acceptable geometry. Two major disorder sites were modelled for the S atom in DMSO(1). The refined site-occupation factors are 0.65 (1) and 0.35 (1) for S11*D* and S12*D*, respectively. Rather high residual electron density (0.960 e Å^{-3}) was observed in the proximity of the S atom in DMSO(2) indicating possible disorder. However, it could not be modelled in a chemically reasonable way. The H-atom positions were either calculated assuming ideal geometry with C—H = 1.00 Å (C-bonded atoms) or were derived from $\Delta\rho$ maps (N- or O-bonded atoms). They were held riding on their carrier atoms during subsequent calculations. Their $U_{\rm iso}$ values were either kept riding on the respective carrier atoms [DMF and C-bonded H atoms of cyclohexylamine guests in (I*a*); H_2O and DMSO(1) guests in (II*b*)] or were refined isotropically (all other H atoms). Since both compounds contain the respective host molecule in stereochemically pure form, the final refinement calculations were carried out twice assuming either *S*,*S* or *R*,*R* configuration for the chiral centres at C11 and C12, and the η parameter (Rogers, 1981) was refined. The *R* and *wR* values calculated for the 11*S*,12*S* and the 11*R*,12*R* models of (I*a*) are indistinguishable (R = 0.0839 and wR = 0.0950). For (II*b*), the final agreement factors for the 11*S*,12*S* configuration are slightly lower (R = 0.0880 and wR = 0.1076) than for the 11*R*,12*R* (R = 0.0887 and wR = 0.1094). Large e.s.d. values of the η parameter, which refined to 0.4 (4) for (I*a*) and 1.0 (6) for (II*b*), assuming the 11*S*,12*S* configuration, do not allow us to support the absolute configuration assignment based on chemical correlation *via* synthesis. The observed disorder, together with the modest data quality, might be the reason that the refinement of both inclusion compounds (I*a*) and (II*b*) ended with relatively high crystallographic *R* values.

Data collection: *DIF*4 (Stoe & Cie, 1988*a*) for (I*a*); *XSCANS* (Siemens, 1994) for (II*b*). Cell refinement: *DIF*4 for (I*a*); *XSCANS* for (II*b*). Data reduction: *REDU*4 (Stoe & Cie, 1988*b*) for (I*a*); *XSCANS* for (II*b*). For both compounds, program(s) used to solve structures: *MULTAN*80 (Main *et al.*, 1980); program(s) used to refine structures: *SHELXTL/PC* (Sheldrick, 1990); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *PLATON* (Spek, 1990).

OH and IC thank Professor Rolf Norrestam (Stockholm University) for kind interest and helpful assistance. Financial support from the Swedish Natural Science Research Council (NFR) to IC, and from the Deutsche Forschungsgemeinschaft (GRK 208/1) and the Fonds der Chemischen Industrie to EW is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1479). Services for accessing these data are described at the back of the journal.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Csöregh, I., Czugler, M., Ertan, A., Weber, E. & Ahrendt, J. (1990). *J. Incl. Phenom.* **8**, 275–287.

Csöregh, I., Finge, S. & Weber, E. (1991). *Bull. Chem. Soc. Jpn*, **64**, 1971–1975.

Csöregh, I., Gallardo, O., Weber, E., Finge, S. & Reutel, C. (1992). *Tetrahedron Asymm.* **3**, 1555–1562.

Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.

Etter, M. C. (1982). *J. Am. Chem. Soc.* **104**, 1095–1096.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Etter, M. C. (1991). *J. Phys. Chem.* **95**, 4601–4610.

Jeffrey, G. A. (1995). *Cryst. Rev.* **4**, 213–259.

MacDonald, J. C. & Whitesides, G. M. (1994). *Chem. Rev.* **94**, 2383–2420.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.

Rogers, D. (1981). *Acta Cryst.* A**37**, 734–741.

Sheldrick, G. M. (1990). *SHELXTL/PC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Stoe & Cie (1988*a*). *DIF*4. *Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1988*b*). *REDU*4. *Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Weber, E. (1989). *J. Mol. Graph.* **7**, 12–27.

Weber, E., Csöregh, I., Ahrendt, J., Finge, S. & Czugler, M. (1988). *J. Org. Chem.* **53**, 5831–5839.

Acta Cryst. (1997). C**53**, 1895–1897

2′-Hydroxy-4-*N*-methylstilbazonium Iodide Methanol Solvate,† $C_{14}H_{14}NO^+.I^-.CH_3OH$

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 8 April 1997; accepted 21 July 1997*)

Abstract

The cation in the title compound has a planar structure [the maximum deviation from the best plane through the non-H atoms is 0.043 (3) Å]. The dihedral angle between the two rings is 0.9 (1)°. Cations are connected through I^- anions and weak O···H—O hydrogen bonds, and are packed in an anti-parallel fashion through $\pi\cdots\pi$ interactions along the *a* axis. Methanol molecules, which

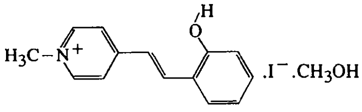
---

† Alternative name: 4-[2-(2-hydroxyphenyl)ethenyl]-*N*-methylpyridinium iodide methanol solvate.

are co-crystallized, play an important role in stablizing the structure, and the O···O distance between the OH group of the cation and the solvent molecule is 2.677 (3) Å.

Comment

During our systematic search of organic salts (Marder & Perry, 1993; Marder, Perry, Tiemann, Marsh & Schaefer, 1990) with non-linear optical (NLO) properties, we isolated the title compound. A number of similar compounds have NLO properties (Marder, Perry & Yakymyshyn, 1994), for example, 4-*N*-methylstilbazolium toluene-*p*-sulfonate salts (Yakymyshyn *et al.*, 1991). However, the present compound, (I), has no second harmonic generation properties.



(I)

In the crystal structure of (I), every cation consists of two planar rings. The $I^- \cdots N^+$ distance is 4.285 (2) Å. Significant overlap between cations related by a symmetry center was found. The short contacts between parallel cations [$C12\cdots C6^i$ and $C4\cdots C14^i$ are 3.407 (6) and 3.358 (5) Å, respectively; symmetry code: (i) $-1-x$, $1-y$, $-z$] show the existence of strong $\pi\cdots\pi$ interactions. The shortest distances between the mean planes (Nardelli, 1983) and between the center of the cations (related by $-1-x$, $1-y$, $-z$) are 3.37 (1) and 3.68 (1) Å, respectively. The next shortest distances between the mean planes and between the center of the cations [related by $-x$, $1-y$, $-z$, *i.e.* (iii)] are 3.40 (1) and 4.91 (1) Å, respectively; the short contacts $C1\cdots C14^{iii}$, $C4\cdots C4^{iii}$ and $C6\cdots C8^{iii}$ are 3.473 (3), 3.423 (2) and 3.420 (2) Å, respectively. Neighbouring cations related by translation vector **c** are connected by methanol molecules and I^- anions through hydrogen bonds (Table 2).

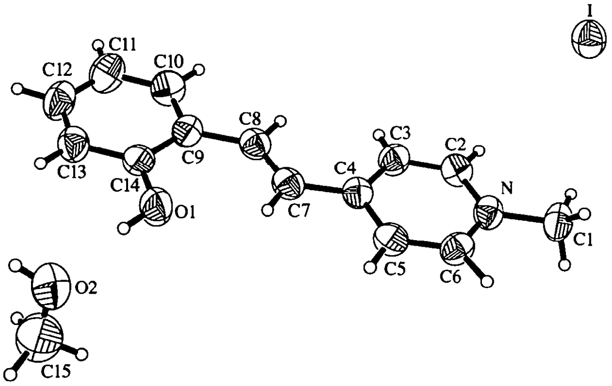


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

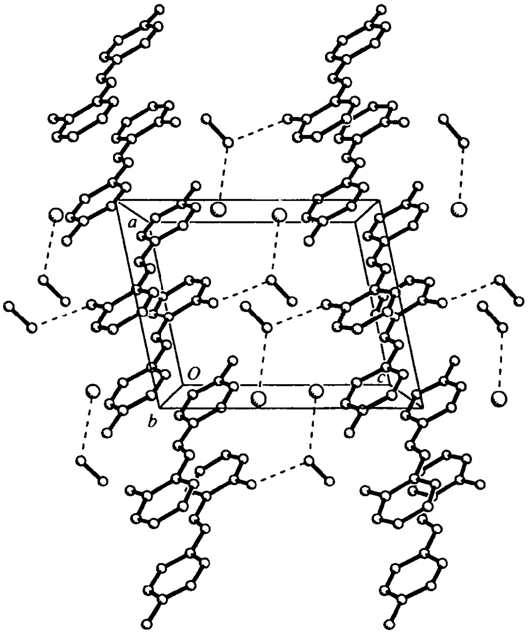


Fig. 2. Packing diagram viewed down the *b* axis.

Experimental

1,4-Dimethylpyridinium iodide (7.05 g, 30 mmol), prepared from CH_3I and γ-methylpyridine, and 2-hydroxybenzaldehyde (6.07 g, 49 mmol) in 10 ml methanol were heated to 353 K for 12 h. The title compound was separated, recrystallized twice from water, and then crystals were grown from a methanol–dichloromethane (1:1) solution by slow evaporation.

Crystal data

$C_{14}H_{14}NO^+.I^-.CH_4O$
$M_r = 371.20$
Monoclinic
$P2_1/c$
$a = 7.9960\,(10)$ Å
$b = 20.776\,(2)$ Å
$c = 9.7610\,(10)$ Å
$\beta = 101.320\,(10)^\circ$
$V = 1590.0\,(3)$ Å^3
$Z = 4$
$D_x = 1.551$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 3.17$–19.42°
$\mu = 2.012$ mm^{-1}
$T = 291\,(2)$ K
Elongated plate
0.50 × 0.50 × 0.32 mm
Brown-yellow

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: semi-empirical from ψ scans (Sheldrick, 1993)
$T_{\min} = 0.398$, $T_{\max} = 0.525$
3602 measured reflections
3112 independent reflections
2222 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.014$
$\theta_{\max} = 26.01^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 25$
$l = -12 \rightarrow 11$
3 standard reflections every 97 reflections intensity decay: 12.70%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.065$
$S = 0.989$
3112 reflections
239 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.077$
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0039 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C14	1.356 (4)	C4—C7	1.460 (4)
N—C6	1.331 (4)	C7—C8	1.325 (4)
N—C2	1.341 (4)	C8—C9	1.462 (4)
N—C1	1.477 (4)		
C5—C4—C3	116.0 (3)	C14—C9—C10	116.9 (3)
C3—C4—C7	124.0 (3)	C14—C9—C8	125.5 (3)
C8—C7—C4	124.6 (3)	O1—C14—C13	120.9 (3)
C7—C8—C9	129.6 (3)	O1—C14—C9	118.2 (3)
C5—C4—C7—C8	−178.9 (3)	C7—C8—C9—C10	−179.3 (3)
C4—C7—C8—C9	179.5 (3)		

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

D—H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O2	2.677 (3)	172.5 (6)
O2—H2O···I^{i}	3.425 (5)	160.8 (3)
O1—H1O···$O2^{ii}$	3.520 (6)	100.9 (5)

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

The title structure was solved by direct methods. Except for the H atoms on C1 and C15, which were placed in calculated positions and not refined, all other H atoms were located from difference maps and their displacement parameters refined isotropically. During refinement, the C—H distances and H—C—H angles were restrained to be 0.96 Å and 109.5°, respectively. $U_{\rm iso}$ values were in the range 0.049 (8)–0.17 (3) Å^2.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *SHELXTL* (Siemens, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1059). Services for accessing these data are described at the back of the journal.

References

Marder, S. R. & Perry, J. W. (1993). *Adv. Mater.* **5**, 804–815.
Marder, S. R., Perry, J. W., Tiemann, B. G., Marsh, R. E. & Schaefer, W. P. (1990). *Chem. Mater.* **2**, 685–690.
Marder, S. R., Perry, J. W. & Yakymyshyn, C. P. (1994). *Chem. Mater.* **6**, 1137–1147.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
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.
Siemens (1995). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc.. Madison, Wisconsin, USA.
Yakymyshyn, C. P., Stewart, K. R., Boden, E. P., Marder, S. R., Perry, J. W. & Schaefer, W. P. (1991). In *Organic Materials for Non-Linear Optics* II, edited by R. A. Hann & D. Bloor. Cambridge University Press.

Acta Cryst. (1997). C**53**, 1897–1899

Polysulfonylamines. XCVIII.† 1,2-Benzenedisulfonylamine Acetonitrile Solvate (1/1)

Peter G. Jones*, Andreas Wirth, Oliver Moers and Armand Blaschette

Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany. E-mail: jones@xray36.anchem.nat.tu-bs.de

(*Received 22 July 1997; accepted 15 September 1997*)

Abstract

In the 1:1 acetonitrile solvate of 1,3,2-benzodithiazole-1,1,3,3-tetrone, $C_6H_5NO_4S_2.C_2H_3N$, the nitrile molecule is linked to the NH function by a slightly bent N—H···N hydrogen bond of medium strength [H···N 1.99 (3) and N···N 2.834 (3) Å]. One methyl H atom forms a three-centre C—H···O bond to different symmetry-related formula units. The five-membered heterocycle possesses an envelope conformation, the N atom deviating by 0.358 (2) Å from the mean plane of the C_6S_2 moiety.

Comment

Molecules containing the $(SO_2)_2NH$ moiety are strong NH acids (*e.g.* Dauphin & Kergomard, 1961; Ali *et al.*, 1980; Foropoulos & DesMarteau, 1984). With appropriate bases *B*, they either form onium salts $BH^+.(XSO_2)_2N^-$ (Henschel, 1996, and references therein) or uncharged hydrogen-bonded complexes $(XSO_2)_2N$—H···*B*. Among the latter class of compounds, crystal structures presently known involve a variety of O-atom acceptors; see, for example, *B* = H_2O (Attig & Mootz, 1975), Ph_3PO (Blaschette *et al.*, 1990), Me_2SO (Schomburg, Blaschette & Schulte-Körne, 1990; Blaschette *et al.*, 1993) and 18-crown-6 (Blaschette *et al.*, 1994; Henschel *et al.*, 1995). We now report the first structure of such a complex, (1), in which the N atom of a nitrile function acts as the hydrogen-bond acceptor.

---

† Part XCVII: Henschel, Wijaya, Moers, Blaschette & Jones (1997).