$S = 1.054$
4120 reflections
394 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1171P)^2 + 0.9778P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0023 (5)
Scattering factors from *SHELXL*93

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

Cl1—C12′	1.804 (4)	O2*A*—C11	1.253 (12)
Cl2—C13′	1.816 (4)	O3*A*—C11	1.235 (13)
O2—C11	1.232 (4)	O3*A*—C13	1.601 (14)
O3—C11	1.333 (4)	C1—C1′	1.504 (3)
O3—C13	1.432 (4)	C3—C11	1.490 (3)
C11—O3—C13	116.0 (3)	O2*A*—C11—C3	114.1 (6)
C11—O3*A*—C13	110.9 (9)	O3—C11—C3	113.2 (2)
O2—C11—O3	121.3 (3)	C3′—C12′—Cl1	111.9 (2)
O2—C11—C3	125.1 (3)	C6′—C13′—Cl2	111.3 (2)
O3*A*—C11—C3	110.6 (7)		
C12—O1—C2—C1	−95.0 (3)	C2—C1—C1′—C2′	−85.5 (3)
C13—O3*A*—C11—O2*A*	−14.8 (19)	C11′—O4—C2′—C1′	−92.1 (3)
C13—O3—C11—O2	−2.0 (6)	C4′—C3′—C12′—Cl1	−100.3 (3)
C4—C3—C11—O2	152.0 (3)	C5′—C6′—C13′—Cl2	96.9 (3)
C4—C3—C11—O2*A*	−135.3 (8)		

All H-atom parameters were refined except those of H135 where only the H-atom *U* value was refined. C—H distances are in the range 0.78 (3)–1.02 (4) Å and U_{iso} values are in the range 0.044 (7)–0.15 (2) Å^2 for the refined H atoms. Atoms O2/O2*A* and O3/O3*A* have occupancies of 0.801 (9)/0.199 (9).

Data collection: *SDP* (B. A. Frenz & Associates Inc., 1982). Cell refinement: *SDP*. Data reduction: *SDP*. 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: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1060). Services for accessing these data are described at the back of the journal.

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A 2:1 Co-Crystal of Hydroquinone and 3,5-Bis(2-pyridyl)-1,2,4-triazole

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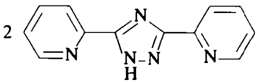
Abstract

The title compound, $2C_{12}H_9N_5.C_6H_6O_2$, exhibits a three-dimensional hydrogen-bonded network of N—H···N, C—H···N, O—H···N, C—H···O, C—H···π and π···π interactions.

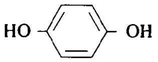
Comment

Recently, the synthesis and physical properties of a series of ruthenium(II)–polypyridyl complexes containing triazole ligands with pendant phenol (Hage, Haasnoot, Reedijk *et al.*, 1990) and hydroquinone (Wang *et al.*, 1993) moieties have been reported. In these studies, crystallographic, spectroscopic and electrochemical evidence was obtained for the presence of intermolecular hydrogen bonding in these complexes involving the hydroxy groups and the free N atoms of the triazole rings. On the basis of these results, we believe that a systematic investigation of the hydroquinone–pyridyl-triazole solid-state systems will lead to the development of new materials with interesting electron- and proton-transfer properties.

The bond lengths and angles for the bis(pyridyl)-triazole (Hbpt) moieties are consistent with those found for the coordinated species in the mono- and binuclear ruthenium complexes $[Ru(bpy)_2(bpt)]PF_6.0.5H_2O$ and $[Ru(bpy)_2(bpt)](CF_3SO_3).4H_2O$ (Hage *et al.*, 1989; Hage, Haasnoot, Nieuwenhuis *et al.*, 1990).



Hbpt



Hydroquinone

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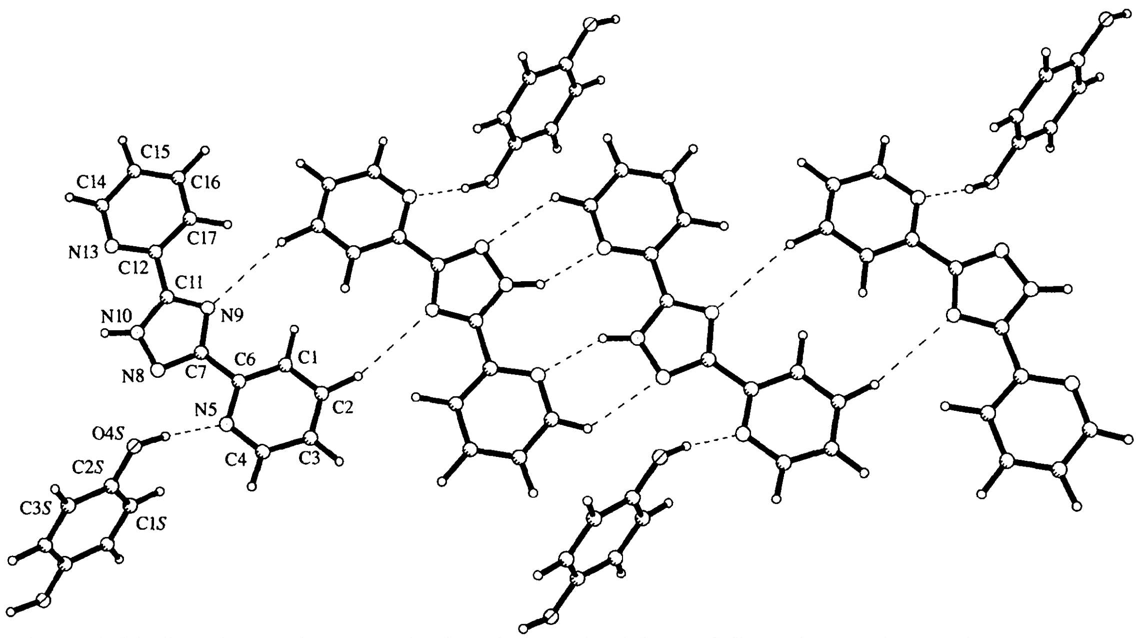

Fig. 1. The atomic labelling scheme and geometry showing a hydrogen-bonded tape of Hbpt and hydroquinone molecules. The hydroquinone molecules are located about inversion centres and provide a link between hydrogen-bonded tapes forming a three-dimensional hydrogen-bonded network. The dashed lines indicate hydrogen bonds and neighbouring tapes have been omitted for clarity.

The asymmetric unit contains one Hbpt molecule and half a hydroquinone situated about an inversion centre. The Hbpt molecules form dimers *via* C—H···N (pyridyl-to-triazole) and N—H···N (triazole-to-pyridyl) hydrogen bonds. The dimers are then extended into chains in the [101] direction by another C—H···N hydrogen bond from a pyridyl moiety to the triazole moiety. This second C—H···N (C2—H2···N9) interaction is much weaker than the other and is hindered by the presence of two pyridyl H atoms, H1 and H17, causing the pyridyl rings to be twisted out of the plane of the triazole ring. The degree of twist is different for the two pyridyl rings, being 12.0 (1)° for the C1 ring and 4.9 (1)° for the C12 ring (Fig. 1) and is a result of the asymmetric nature of the H···H repulsions; *cf.* C1···$C2^{i}$ 4.087 (3) and C17···$C2^{i}$ 3.715 (3) Å [symmetry code: (i) $-x, 1-y, 1-z$].

The chains of Hbpt molecules are stacked in the [100] direction and are involved in $\pi\cdots\pi$ interactions (3.5–3.9 Å) with adjacent hydrogen-bonded chains (Table 1). The hydroquinone molecules bridge between these chains *via* O—H···N and C—H···N hydrogen bonds to the pyridyl N atom not involved in the dimer formation. They are also linked to the Hbpt molecules *via* C—H···O and C—H···π interactions from the pyridyl moiety (Table 1). This creates a three-dimensional hydrogen-bonded network with the hydroquinone molecules acting as links between adjacent chains of Hbpt molecules. It is notable that there are no interactions between the hydroquinone molecules and that they form a herring-bone arrangement with respect to the Hbpt molecules.

Experimental

1,4-Dihydroxybenzene (Aldrich) was purified by recrystallization from ethanol and Hbpt was prepared as reported in the literature (Hage *et al.*, 1988). Co-crystals were obtained by refluxing Hbpt and hydroquinone in a 2:1 ratio in an ethanol–water solution. Upon cooling of the solution, tan-coloured rod-like crystals were obtained (m.p. 471.5–473.5 K).

Crystal data

$2C_{12}H_9N_5.C_6H_6O_2$
$M_r = 556.59$
Triclinic
$P\overline{1}$
$a = 7.3223\,(7)$ Å
$b = 7.6708\,(6)$ Å
$c = 13.2160\,(10)$ Å
$\alpha = 105.062\,(9)°$
$\beta = 92.139\,(8)°$
$\gamma = 110.002\,(9)°$
$V = 667.03\,(14)$ Å^3
$Z = 1$
$D_x = 1.386$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 5.0$–$12.5°$
$\mu = 0.093$ mm^{-1}
$T = 120\,(2)$ K
Needle
0.65 × 0.34 × 0.19 mm
Light tan

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
2513 measured reflections
2312 independent reflections
1642 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$

$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -15 \rightarrow 15$
3 standard reflections every 97 reflections intensity variation: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.107$
$S = 1.083$
2312 reflections
190 parameters
H atoms were located from ΔF synthesis and refined using a riding model

$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.1659P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O4S—H4S···N5	1.05	1.816 (3)	2.844 (3)	163.77 (8)
N10—H10···N13$^{\text{i}}$	0.98	2.067 (3)	2.953 (3)	149.56 (7)
C14—H14···N8$^{\text{i}}$	1.04	2.520 (3)	3.301 (3)	131.57 (7)
C2—H2···N9$^{\text{ii}}$	1.14	2.852 (3)	3.931 (3)	156.94 (6)
C16—H16···O4S^{iii}	1.18	2.646 (3)	3.379 (3)	118.80 (7)
C3—H3···O4S^{iv}	1.11	2.535 (3)	3.521 (3)	147.69 (7)
C15—H15···C1S^{v}	1.06	2.836 (3)	3.769 (3)	146.99 (7)
C15—H15···C2S^{v}	1.06	2.704 (3)	3.759 (3)	173.83 (7)
C15—H15···C3S^{v}	1.06	2.841 (3)	3.788 (3)	148.82 (7)

Symmetry codes: (i) $1 - x, 3 - y, 1 - z$; (ii) $-x, 1 - y, 1 - z$; (iii) $-x, 2 - y, 1 - z$; (iv) $x, y - 1, z$; (v) $x, 1 + y, 1 + z$.

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

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1161). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1875–1877

Polysulfonylamines. XCI.† Ethylammonium Di(benzenesulfonyl)amide 1,4,7,10,13,16-Hexaoxacyclooctadecane (1/1/1)

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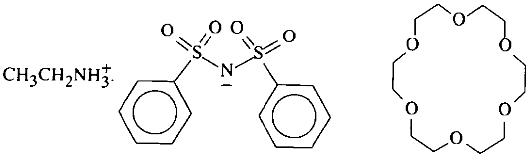
Abstract

In the structure of the title compound, $C_2H_8N^+.C_{12}H_{10}NO_4S_2^-.C_{12}H_{24}O_6$, the non-coordinating $(C_6H_5SO_2)_2N^-$ anion adopts an extended conformation with the phenyl groups lying on opposite sides of the S—N—S plane. The conformation of the macrocycle and the hydrogen-bond geometry in the complex cation closely resemble those in related adducts between 18-crown-6 and primary alkylammonium salts.

Comment

We are interested in the structures of polysulfonylamines in general and in the conformation of di(benzenesulfonyl)amine in particular. Di(benzenesulfonyl)amine, $HN(SO_2C_6H_5)_2$, adopts an extended conformation in the solid state, the $N(SO_2C)_2$ moiety approximating to C_2 symmetry, whereas in the corresponding sodium salt the conjugate anion has a folded pseudo-C_s symmetry that favours close contacts between Na$^+$ and the electronegative N and O atoms (Cotton & Stokely, 1970).



(1)

In contrast to the Na salt, the title compound, (1), consists of a large organic cation, with effectively shielded charge, and a non-coordinating $(C_6H_5SO_2)_2N^-$ anion (Fig. 1). The closest cation–anion contact is observed between a methylene H and a sulfonyl O atom [H40D···O9 2.80, C40···O9 3.314 (4) Å and C40—H40D···O9 113°]. In this environment, the anion adopts an extended conformation resembling that of its parent acid; however, the deviation of the $N(SO_2C)_2$ group

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