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(*E*)-4-{[2-(Methylsulfanyl)phenyl]-diazenyl}phenol

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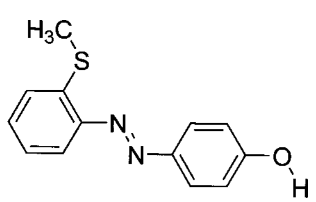
Key indicators: single-crystal X-ray study; *T* = 273 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.037; *wR* factor = 0.110; data-to-parameter ratio = 19.6.

The title compound, $C_{13}H_{12}N_2OS$, contains a diazene group (—N═N—), and the configuration around the —N═N— double bond is *trans*. The dihedral angle between the benzene rings is 33.88 (6)°. The molecular units are linked into chains by intermolecular O—H···N hydrogen bonds.

Related literature

For related literature, see: Antonov *et al.* (1998), (1999); Betteridge & John (1973); Burawoy *et al.* (1954); Das *et al.* (2006); Ersanlı *et al.* (2005); Karadayı *et al.* (2006); Liu *et al.* (2005); Li *et al.* (2004); Moggach *et al.* (2005); Pollard *et al.* (1959); Portilla *et al.* (2007); Şahin *et al.* (2005).



Experimental

Crystal data

$C_{13}H_{12}N_2OS$
M_r = 244.31
Monoclinic, $P2_1/c$
a = 11.8379 (2) Å
b = 8.6159 (2) Å
c = 12.5056 (2) Å
β = 106.029 (1)°
V = 1225.91 (4) Å^3
Z = 4
Mo *K*α radiation
μ = 0.25 mm^{-1}
T = 273 (2) K
0.32 × 0.12 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.963, T_{max} = 0.974
15771 measured reflections
3053 independent reflections
2508 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.110
S = 1.03
3053 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.82	2.04	2.8596 (16)	176

Symmetry code: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2317).

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supplementary materials

Acta Cryst. (2007). E**63**, o3114 [**doi:10.1107/S1600536807026839**]

(*E*)-4-{[2-(Methylsulfanyl)phenyl]diazenyl}phenol

A. N. Biswas, P. Das, U. S. Agarwalla, A. Saha and P. Bandyopadhyay

Comment

Organic molecules containing the diazene moiety are among the largest group of dyes. The extensive application of azo dyes in industry and analytical chemistry have attracted attention for decades. Some arylazo compounds derived from resorcinol or *β*-naphthol have been widely used in the spectrophotometric determination of traces of metals (Betteridge & John, 1973; Pollard *et al.*, 1959). Optically active azobenzene polymers are very important functional materials because of their photoresponsive properties. The position of azo and hydroxyl groups in arylazo compounds brings into play the azo-hydrazo equilibrium, which has been the subject of intensive investigation in recent years (Antonov *et al.*, 1998, 1999). Generally arylazonaphthalenes have been found to exist in the hydrazo-keto form in the solid state (Liu *et al.*, 2005). Here in, we report the crystal structure of (*E*)-1-[2-(methylsulfanyl)phenyldiazenyl]-4-hydroxybenzene where the azo-enol form has been found to be retained in the solid state.

The asymmetric unit of the title compound, (I), is shown in Fig. 1, with the atom-numbering scheme. Phenyl rings of the molecule adopt a *trans* configuration about the azo functional group. Three planar fragments in the molecular structure of (I) may be identified: the phenyl ring (C1–C6) connected to N1 (*A*), azo group along with C1 and C7 (*B*) and the benzene ring (C7–C12) connected to N2 (*C*). The dihedral angles between the planes *A*/*B*, *B*/*C* and *A*/*C* are 8.61 (14), 25.95 (10) and 33.88 (06)°, respectively.

The molecular arrangement of (I) has been shown in Fig. 2. The N1\═N2 bond length, 1.2569 (17) Å of the title compound is slightly smaller than other *trans* azo compounds (Ersanlı *et al.*, 2005; Das *et al.*, 2006). Both the C—N bonds distances of the title compound are almost equal; the values are typical of *trans* azo compounds (Karadayı *et al.*, 2006). The S—C bond distances are in good agreement with the reported S—C distances under similar hybridization schemes of the bonded carbon atom (Li *et al.*, 2004; Moggach *et al.*, 2005). The O—C distance of the hydroxy group is in good agreement with the literature values (Şahin *et al.*, 2005).

The H···N seperation of 2.04 Å implies a strong interaction (Portilla *et al.*, 2007). The supramolecular structure of compound (I) is simple. A chain structure results by the intermolecular hydrogen-bonds where O1 atom in the molecule at ($-x$, $1/2+y$, $1/2-z$) acts as a hydrogen-bond donor, *via* H1, to the N1atom in the molecule at (x,$1/2-y$,$1/2+z$) (Table 1) (Fig. 3).

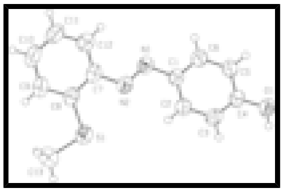
Experimental

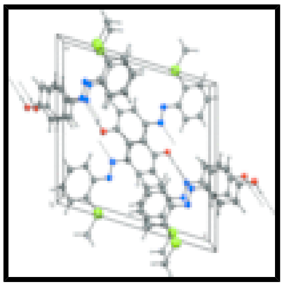
1-[2-(Methylsulfanyl)phenyldiazenyl]-4-hydroxybenzene was prepared according to the literature method (Burawoy *et al.*, 1954), using Phenol and 2-methylthioaniline as starting materials. The product was crystallized from ethanol (Yield: 67%; m.p. 360 K). Suitable crystals of (I) were obtained by slow diffusion of a dichloromethane solution into *n*-hexane.

Refinement

H atoms were included at calculated positions as riding atoms with C–H set to 0.93 Å for (aromatic) and 0.96 Å for (CH_3) H atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ ($1.5U_{eq}$ for methyl group).

Figures





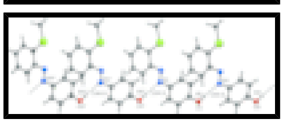


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level.
Figure 2
The molecular arrangement of (I) in the *ac* plane (dashed lines indicate the hydrogen bonds).
Figure 3
Linear chain like association of the molecular units of (I) through intermolecular hydrogen bonds [shown by dashed lines; symmetry codes: (i) *x*, 1/2 − *y*, 1/2 + *z*; (ii) *x*, 1/2 − *y*, −1/2 + *z* and (iii) −*x*, 1/2 + *y*, −1/2 − *z*].

(*E*)-4-{[2-(Methylsulfanyl)phenyl]diazenyl}phenol

Crystal data

$C_{13}H_{12}N_2OS$	$F_{000} = 512$
$M_r = 244.31$	$D_x = 1.324$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3053 reflections
$a = 11.8379$ (2) Å	θ = 2.9–28.3º
$b = 8.6159$ (2) Å	$\mu = 0.25$ mm^{-1}
$c = 12.5056$ (2) Å	$T = 273$ (2) K
β = 106.0290 (10)º	Block, orange
$V = 1225.91$ (4) Å^3	0.32 × 0.12 × 0.11 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3053 independent reflections
Radiation source: fine-focus sealed tube	2508 reflections with $I > 2\sigma(I)$

Monochromator: graphite	R_{int} = 0.018
T = 273(2) K	θ_{max} = 28.3°
φ and ω scans	θ_{min} = 2.9°
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -15 \rightarrow 15$
T_{min} = 0.963, T_{max} = 0.974	$k = -11 \rightarrow 11$
15771 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.2258P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
3053 reflections	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
156 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
S1	−0.01841 (4)	0.01491 (5)	0.74707 (4)	0.06431 (15)
C1	0.36949 (11)	0.13649 (15)	0.94129 (9)	0.0450 (3)
C2	0.30360 (12)	0.15560 (17)	1.01740 (11)	0.0517 (3)
H2	0.2325	0.1036	1.0071	0.062*
C4	0.45018 (12)	0.33051 (16)	1.12358 (10)	0.0499 (3)
C3	0.34444 (13)	0.25175 (17)	1.10772 (11)	0.0550 (3)
H3	0.3008	0.2641	1.1586	0.066*
C6	0.47552 (13)	0.21233 (19)	0.95873 (12)	0.0565 (3)
H6	0.5200	0.1984	0.9087	0.068*
C5	0.51672 (13)	0.3089 (2)	1.04954 (12)	0.0586 (4)
H5	0.5887	0.3589	1.0608	0.070*

C8	0.06544 (12)	−0.09020 (15)	0.67607 (11)	0.0504 (3)
C7	0.18786 (13)	−0.08959 (15)	0.72203 (11)	0.0511 (3)
C9	0.01938 (15)	−0.17087 (18)	0.57666 (12)	0.0610 (4)
H9	−0.0612	−0.1703	0.5434	0.073*
C10	0.09249 (17)	−0.25136 (19)	0.52751 (13)	0.0707 (5)
H10	0.0605	−0.3049	0.4615	0.085*
C12	0.26046 (15)	−0.17292 (19)	0.67215 (14)	0.0646 (4)
H12	0.3413	−0.1743	0.7042	0.078*
C11	0.21193 (18)	−0.2535 (2)	0.57485 (16)	0.0739 (5)
H11	0.2602	−0.3092	0.5413	0.089*
N1	0.33188 (10)	0.04703 (13)	0.84242 (9)	0.0483 (3)
N2	0.22843 (11)	−0.00323 (13)	0.82199 (9)	0.0517 (3)
O1	0.49329 (10)	0.42891 (14)	1.21012 (8)	0.0643 (3)
H1	0.4451	0.4387	1.2458	0.096*
C13	−0.16635 (16)	−0.0417 (3)	0.67795 (19)	0.0875 (6)
H13A	−0.1858	−0.0089	0.6017	0.131*
H13B	−0.2192	0.0061	0.7141	0.131*
H13C	−0.1732	−0.1525	0.6811	0.131*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0637 (3)	0.0630 (2)	0.0638 (2)	0.00088 (17)	0.01359 (18)	−0.01475 (17)
C1	0.0502 (7)	0.0448 (6)	0.0377 (6)	0.0045 (5)	0.0082 (5)	0.0034 (5)
C2	0.0536 (7)	0.0536 (7)	0.0485 (7)	−0.0055 (6)	0.0153 (5)	−0.0003 (6)
C4	0.0561 (7)	0.0516 (7)	0.0388 (6)	0.0002 (6)	0.0078 (5)	0.0013 (5)
C3	0.0617 (8)	0.0616 (8)	0.0454 (7)	−0.0038 (6)	0.0208 (6)	−0.0030 (6)
C6	0.0537 (7)	0.0706 (9)	0.0472 (7)	−0.0022 (7)	0.0173 (6)	−0.0053 (6)
C5	0.0512 (7)	0.0727 (9)	0.0516 (7)	−0.0108 (7)	0.0137 (6)	−0.0069 (7)
C8	0.0632 (8)	0.0423 (6)	0.0444 (6)	−0.0055 (6)	0.0125 (6)	−0.0002 (5)
C7	0.0631 (8)	0.0440 (6)	0.0449 (6)	−0.0023 (6)	0.0127 (6)	−0.0024 (5)
C9	0.0725 (9)	0.0567 (8)	0.0501 (7)	−0.0122 (7)	0.0111 (7)	−0.0052 (6)
C10	0.0986 (13)	0.0625 (9)	0.0532 (8)	−0.0193 (9)	0.0245 (8)	−0.0184 (7)
C12	0.0686 (9)	0.0593 (9)	0.0679 (9)	−0.0008 (7)	0.0220 (7)	−0.0117 (7)
C11	0.0890 (12)	0.0652 (10)	0.0748 (10)	−0.0068 (9)	0.0347 (9)	−0.0235 (8)
N1	0.0547 (6)	0.0469 (6)	0.0414 (5)	0.0032 (5)	0.0100 (4)	0.0020 (4)
N2	0.0562 (7)	0.0517 (6)	0.0448 (6)	−0.0003 (5)	0.0099 (5)	−0.0039 (5)
O1	0.0679 (7)	0.0755 (7)	0.0488 (5)	−0.0126 (6)	0.0153 (5)	−0.0142 (5)
C13	0.0623 (10)	0.1049 (15)	0.0930 (13)	−0.0035 (10)	0.0177 (9)	−0.0171 (12)

Geometric parameters (Å, °)

S1—C8	1.7551 (14)	C8—C7	1.404 (2)
S1—C13	1.793 (2)	C7—C12	1.393 (2)
C1—C6	1.378 (2)	C7—N2	1.4197 (17)
C1—C2	1.3969 (18)	C9—C10	1.379 (2)
C1—N1	1.4202 (16)	C9—H9	0.9300
C2—C3	1.3763 (19)	C10—C11	1.374 (3)
C2—H2	0.9300	C10—H10	0.9300

C4—O1	1.3584 (16)	C12—C11	1.381 (2)
C4—C5	1.384 (2)	C12—H12	0.9300
C4—C3	1.389 (2)	C11—H11	0.9300
C3—H3	0.9300	N1—N2	1.2569 (17)
C6—C5	1.383 (2)	O1—H1	0.8200
C6—H6	0.9300	C13—H13A	0.9600
C5—H5	0.9300	C13—H13B	0.9600
C8—C9	1.3970 (19)	C13—H13C	0.9600

C8—S1—C13	103.26 (8)	C12—C7—N2	124.33 (14)
C6—C1—C2	119.46 (12)	C8—C7—N2	114.99 (12)
C6—C1—N1	116.62 (11)	C10—C9—C8	120.53 (15)
C2—C1—N1	123.87 (12)	C10—C9—H9	119.7
C3—C2—C1	119.63 (13)	C8—C9—H9	119.7
C3—C2—H2	120.2	C11—C10—C9	120.83 (15)
C1—C2—H2	120.2	C11—C10—H10	119.6
O1—C4—C5	117.33 (13)	C9—C10—H10	119.6
O1—C4—C3	122.96 (12)	C11—C12—C7	119.72 (16)
C5—C4—C3	119.71 (12)	C11—C12—H12	120.1
C2—C3—C4	120.64 (13)	C7—C12—H12	120.1
C2—C3—H3	119.7	C10—C11—C12	120.09 (16)
C4—C3—H3	119.7	C10—C11—H11	120.0
C1—C6—C5	120.95 (13)	C12—C11—H11	120.0
C1—C6—H6	119.5	N2—N1—C1	114.68 (11)
C5—C6—H6	119.5	N1—N2—C7	115.68 (12)
C6—C5—C4	119.57 (13)	C4—O1—H1	109.5
C6—C5—H5	120.2	S1—C13—H13A	109.5
C4—C5—H5	120.2	S1—C13—H13B	109.5
C9—C8—C7	118.13 (13)	H13A—C13—H13B	109.5
C9—C8—S1	124.79 (12)	S1—C13—H13C	109.5
C7—C8—S1	117.07 (10)	H13A—C13—H13C	109.5
C12—C7—C8	120.65 (13)	H13B—C13—H13C	109.5

C6—C1—C2—C3	1.1 (2)	C9—C8—C7—N2	179.00 (12)
N1—C1—C2—C3	−176.31 (12)	S1—C8—C7—N2	0.34 (16)
C1—C2—C3—C4	0.3 (2)	C7—C8—C9—C10	2.0 (2)
O1—C4—C3—C2	178.74 (13)	S1—C8—C9—C10	−179.45 (12)
C5—C4—C3—C2	−1.8 (2)	C8—C9—C10—C11	−0.3 (3)
C2—C1—C6—C5	−1.0 (2)	C8—C7—C12—C11	1.8 (2)
N1—C1—C6—C5	176.56 (14)	N2—C7—C12—C11	179.87 (15)
C1—C6—C5—C4	−0.4 (2)	C9—C10—C11—C12	−0.8 (3)
O1—C4—C5—C6	−178.66 (14)	C7—C12—C11—C10	0.0 (3)
C3—C4—C5—C6	1.8 (2)	C6—C1—N1—N2	−170.97 (12)
C13—S1—C8—C9	11.12 (16)	C2—C1—N1—N2	6.47 (18)
C13—S1—C8—C7	−170.32 (12)	C1—N1—N2—C7	178.42 (10)
C9—C8—C7—C12	−2.8 (2)	C12—C7—N2—N1	27.0 (2)
S1—C8—C7—C12	178.57 (12)	C8—C7—N2—N1	−154.82 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O1—H1···N1[i]	0.82	2.04	2.8596 (16)	176

Symmetry codes: (i) x, $-y+1/2$, $z+1/2$.

Fig. 1

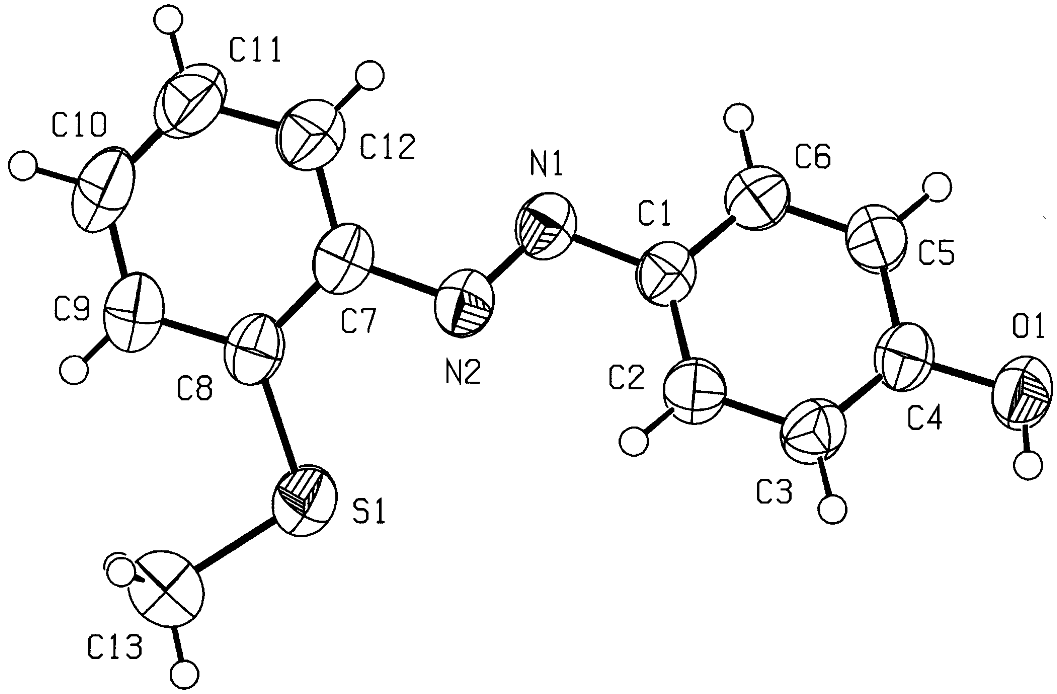


Fig. 2

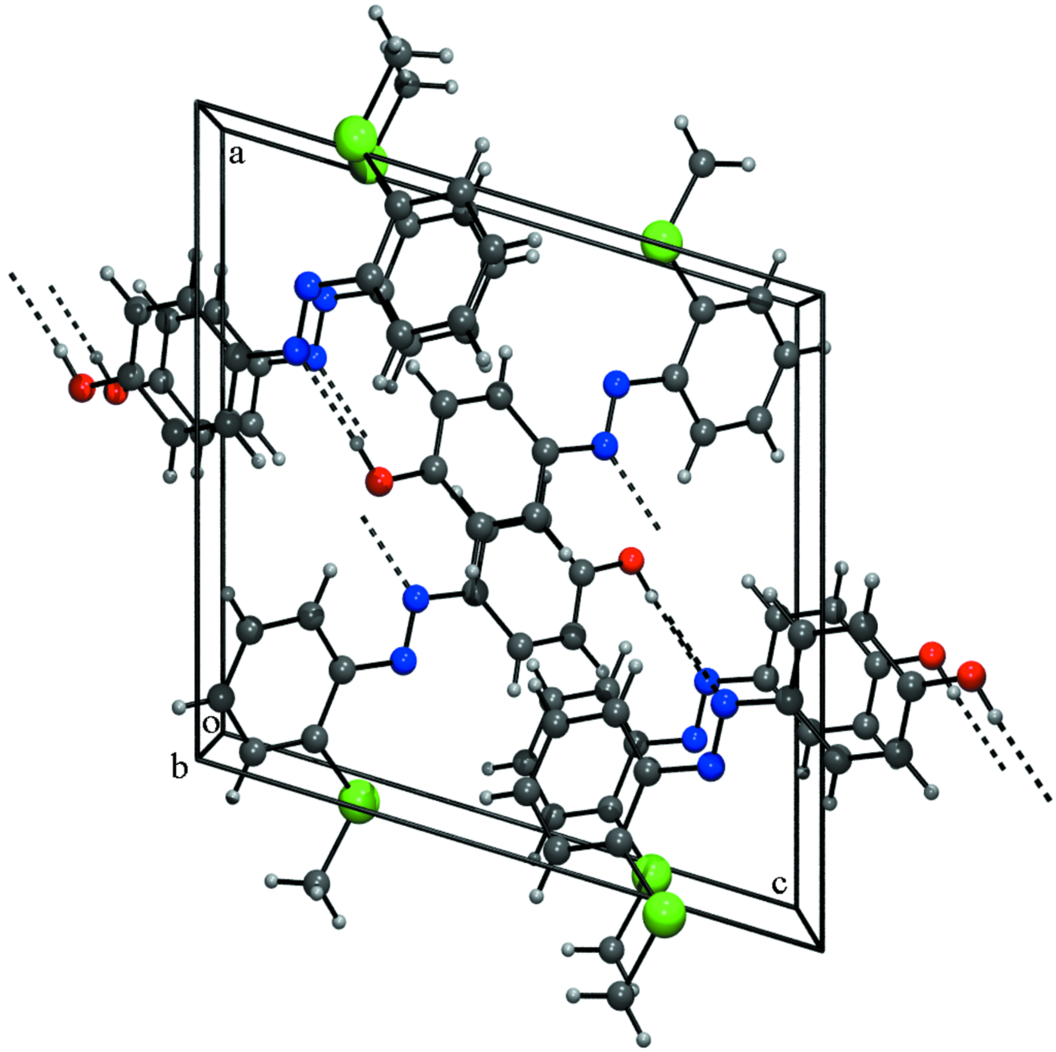
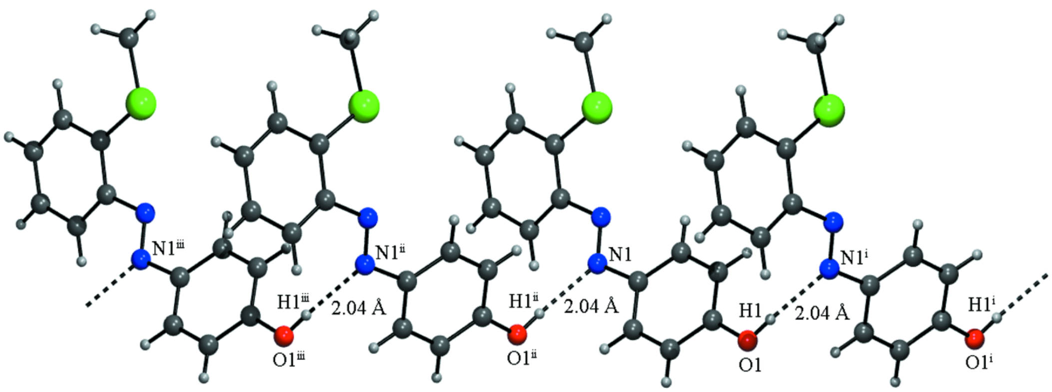


Fig. 3


N1iii
N1ii
N1
N1i
H1iii
2.04 Å
H1ii
2.04 Å
H1
2.04 Å
H1i
O1iii
O1ii
O1
O1i