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Section A: Chemical Sciences

Research Article

Synthesis and crystal structure of 4-((2-aphthalen-1-yl)-diazenyl) benzenesulfonamide

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ABSTRACT

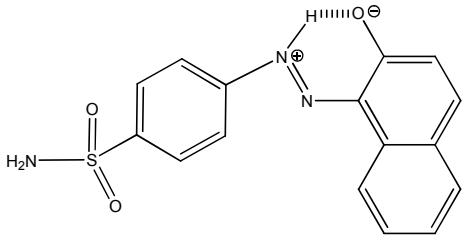
The unsymmetrical azo-compound namely 4-((2-hydroxynaphthalen-1-yl) diazenyl) benzeneesulfonamide was synthesized and isolated as crystal reddish-orange needles. Crystal structure determination was occurred by X-ray diffraction. Resolution of structure shows a dimer of formula $C_{32}H_{26}N_6O_6S_2$ which crystallizes in space group $P2_1$ / a, monoclinic system with: a = 7.4033Å, b = 15.1317Å c = 26.2888Å; α =90.00°, β = 95.1787 °, γ =90.00°.

Keywords: Dazotization, azo-coupling, single-crystal,X-ray study, azo-dyes.

INTRODUCTION

Azo dyes are the most common types of synthetic dyes and constitute the largest class of dyes used commercially[1]. Synthetic azo dyes are used extensively as dyes for textiles, food and cosmetics. Most of them, which are released into the environment, originate from the textile industry and the dyestuff manufacturing industry[2, 3]. It well known that azo dyes are a group of compounds characterized by the presence of one or more azo bonds (-N=N-) in association with one or more aromatic systems[4].This makes them relatively resistant to biological and chemical degradations. However, several studies have shown azo dyes to be toxic and/or carcinogenic[5-14]. They may be converted into potentially carcinogenic and/or mutagenic amines. As a consequence of π-delocalization, aryl azo compounds have vivid colors, especially reds, oranges and yellows. Aryl azo compounds are usually stable, crystalline species. It exists mainly as the *trans*isomer, but upon photolysis, converts to the *cis* isomer. Aromatic azo compounds can be synthesized by using a classical azo coupling reaction, which entails an electrophilic substitution reaction where an aryl diazonium -cation attacks another aryl ring, since diazonium salts are often unstable near room temperature, the azo coupling reactions are typically conducted near ice temperatures[15-19]. The pH of solution is quite important; it must be mildly acidic or neutral, since no reaction takes place if the pH is too low[20-23].

The X-ray diffraction of our structure **(scheme1)** achieves not only the size of the mesh, but the nature of chemical bonds and form molecules. All this information is of fundamental importance for the study of material properties that depend either on their atomic structure or defects of this structure



Scheme-1: **4-((2-hydroxynaphthalen-1-yl) diazenyl) benzenesulfonamide**

EXPERIMENTAL

The title compound 4-((2-hydroxynaphthalen-1-yl) diazenyl)benzenesulfonamidewas prepared following the classical method of synthesis of an azo dye[15](diazo coupling reaction on4-aminobenzenesulonamide).This gives a reddish-orange powder which was recrystallized from pentane leading to crystals in the form of reddish-orange needles, stable in air.

RESULTS AND DISCUSSION

Refinement: Resolution of our XRD structure shows a dimer of formula $C_{32}H_{26}N_6O_6S_2$ which crystallizes in space group $P2_1$ / a, monoclinic system[24-28].

All H atoms, except those of the NH_2 group, have been placed in geometrically idealized positions and refined as riding, with C—H = 0.93Å, and refined in a riding model with Uiso(H) = 0.05Ueq(C). The H atom of amino-group was refined freely.

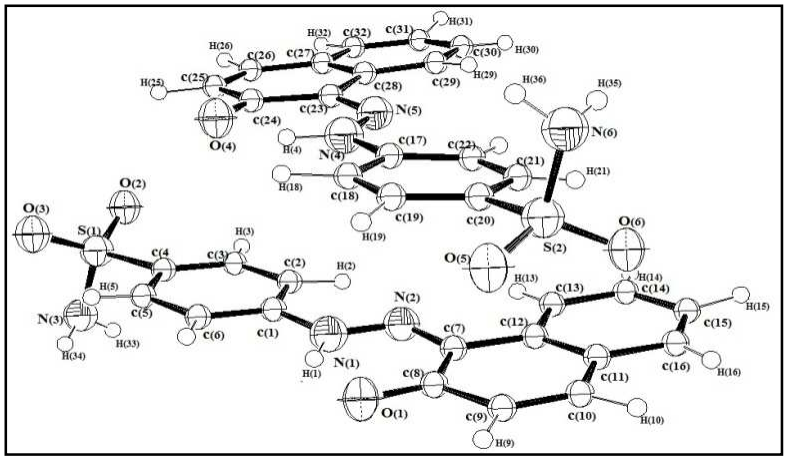


Fig.1:4-((2-hydroxynaphthalen-1-yl) diazenyl)benzenesulfonamide

This molecule consists of a benzene ring linked to the first nitrogen atom of the N = N chromophore and two aromatic rings of the core β-naphthol. H atoms are presented as small spheres.

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. Crystal Data of 4-((2-hydroxynaphthalen-1-yl) diazenyl)benzenesulfonamide is given in **Table-1**and **Table-2.**

Table -1:Crystal Data of 4-((2-hydroxynaphthalen-1-yl) diazenyl)benzenesulfonamide

Crystal data	
$C_{32}H_{26}N_6O_6S_2$	Z = 4
Mr= 654.7	F(000) = 1359.7
a = 7.4033Å α =90.00° b = 15.1317Å β = 95.1787 ° c = 26.2888Å γ =90.00°	Monoclinique $P2_1/a$
V= 2932.969	D_x= 1.48 g/cm^3
Mo Kα radiation, λ = 0.71073 Å	θ = 2.91à 25.35°
μ= 0.240 mm^{-1}	T = 293 K
0.01× 0.02× 0.01 mm	
Data collection	
Collect (Bruker AXS BV, 1997 2004)'diffractometer	
Absorption correction: multi-scan HKL Scalepack (Otwinowski& Minor 1997)'	
T_{min} = 2.910	T_{max} = 25.350
8928 measured reflections5368independent reflections 3587 reflections with I > 2θ(I)	R int = 0.0315
-8 ≤ h ≤ +8 -31 ≤ l ≤ +31	-15 ≤ k ≤ +18
Refinement	
R[F^2> 2 θ(F^2)] = 0,049wR(F_2) = 0.125 S = 1.06	
5368 reflections	525 parameters
Primary atom site location: structure-invariant direct methods Secondary atom site location: difference Fourier mapHydrogen site location: inferred from neigh bouring sitesw = 1/[σ^2(F_o^2)] where P = (F_o^2 + 2F_c^2)/3	
(Δ/σ)max = 0.003 6 restraintsΔρmin = −0.34 e Å^{-3}	Δρmax = 0.23 e Å^{-3}

REFINEMENT

Refinement of F2 against ALL reflections: The weighted R-factor wR and goodness of fit S are based on F2, conventional R-factors R are based on F, with F set to zero for negative F2. The threshold expression of F2 > σ(F2) is used only for calculating R- factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.47975 (9)	−0.13841 (4)	0.52672 (3)	0.0426 (2)
O1	0.9633 (3)	−0.14156 (13)	0.81398 (8)	0.0614 (8)
O2	0.4289 (3)	−0.22979 (12)	0.52403 (8)	0.0576 (7)
O3	0.3486 (2)	−0.07261 (13)	0.50989 (7)	0.0520 (7)
N1	0.7903 (3)	−0.07008 (16)	0.73799 (9)	0.0455 (8)
N2	0.7818 (3)	0.00480 (15)	0.76295 (8)	0.0409 (8)
N3	0.6471 (3)	−0.12704 (16)	0.49192 (9)	0.0460 (8)
C1	0.7083 (4)	−0.08096 (18)	0.68832 (10)	0.0410 (9)
C2	0.6299 (4)	−0.01278 (17)	0.65909 (10)	0.0416 (9)
C3	0.5570 (4)	−0.03002 (17)	0.60995 (10)	0.0421 (9)
C4	0.5608 (4)	−0.11518 (17)	0.59032 (10)	0.0394 (9)
C5	0.6383 (4)	−0.18318 (19)	0.61963 (11)	0.0542 (10)
C6	0.7132 (4)	−0.16610 (19)	0.66842 (11)	0.0553 (10)
C7	0.8590 (3)	0.00706 (18)	0.81068 (10)	0.0401 (9)
C8	0.9462 (4)	−0.0691 (2)	0.83657 (11)	0.0467 (10)
C9	1.0143 (4)	−0.0587 (2)	0.88915 (11)	0.0537 (11)
C10	0.9982 (4)	0.0178 (2)	0.91381 (11)	0.0540 (10)
C11	0.9151 (4)	0.0944 (2)	0.88999 (10)	0.0464 (10)
C12	0.8460 (3)	0.09042 (19)	0.83820 (10)	0.0430 (9)
C13	0.7641 (4)	0.1659 (2)	0.81555 (11)	0.0503 (10)
C14	0.7496 (4)	0.2420 (2)	0.84369 (13)	0.0592 (11)
C15	0.8172 (4)	0.2463 (2)	0.89422 (13)	0.0644 (12)
C16	0.8993 (4)	0.1738 (2)	0.91726 (12)	0.0573 (11)
S2	0.61219 (9)	−0.11986 (4)	0.96326 (3)	0.0431 (2)
O4	0.2013 (3)	−0.12663 (13)	0.66723 (8)	0.0611 (8)
O5	0.6942 (2)	−0.04127 (12)	0.98563 (7)	0.0503 (7)
O6	0.7176 (3)	−0.19920 (13)	0.96295 (8)	0.0618 (8)

N4	0.2963 (3)	−0.05476 (16)	0.75206 (9)	0.0474 (8)
N5	0.2236 (3)	0.02051 (14)	0.73576 (8)	0.0428 (8)
N6	0.4410 (3)	−0.14094 (17)	0.99494 (9)	0.0490 (9)
C17	0.3786 (3)	−0.06436 (18)	0.80157 (10)	0.0406 (9)
C18	0.4023 (3)	0.00471 (18)	0.83629 (10)	0.0430 (9)
C19	0.4784 (3)	−0.01124 (17)	0.88531 (10)	0.0416 (9)
C20	0.5319 (3)	−0.09629 (17)	0.89968 (10)	0.0393 (8)
C21	0.5132 (4)	−0.16453 (19)	0.86484 (11)	0.0546 (10)
C22	0.4370 (4)	−0.14848 (19)	0.81612 (11)	0.0558 (10)
C23	0.1450 (3)	0.02280 (17)	0.68820 (10)	0.0392 (9)
C24	0.1398 (4)	−0.0523 (2)	0.65301 (11)	0.0462 (10)
C25	0.0648 (4)	−0.0383 (2)	0.60132 (11)	0.0523 (11)
C26	−0.0009 (4)	0.0409 (2)	0.58602 (11)	0.0524 (10)
C27	−0.0044 (3)	0.11561 (19)	0.61934 (10)	0.0450 (9)
C28	0.0630 (3)	0.10719 (18)	0.67105 (10)	0.0414 (9)
C29	0.0506 (4)	0.1792 (2)	0.70359 (11)	0.0499 (10)
C30	−0.0190 (4)	0.2587 (2)	0.68506 (12)	0.0578 (11)
C31	−0.0782 (4)	0.2679 (2)	0.63398 (14)	0.0629 (11)
C32	−0.0733 (4)	0.1976 (2)	0.60167 (12)	0.0568 (11)
H1	0.849 (4)	−0.1135 (16)	0.7557 (12)	0.083 (12)*
H2	0.62630	0.04410	0.67240	0.0500*
H3	0.50520	0.01550	0.58990	0.0510*
H5	0.63990	−0.24020	0.60650	0.0650*
H6	0.76720	−0.21150	0.68820	0.0660*
H9	1.07090	−0.10620	0.90640	0.0640*
H10	1.04310	0.02130	0.94800	0.0650*
H13	0.71930	0.16470	0.78140	0.0600*
H14	0.69320	0.29140	0.82830	0.0710*
H15	0.80700	0.29830	0.91260	0.0770*
H16	0.94530	0.17690	0.95130	0.0690*
H33	0.740 (3)	−0.1664 (16)	0.4998 (11)	0.064 (10)*
H34	0.697 (4)	−0.0717 (11)	0.4895 (12)	0.075 (11)*
H4	0.289 (5)	−0.0995 (16)	0.7290 (10)	0.084 (12)*
H18	0.36700	0.06170	0.82650	0.0520*
H19	0.49380	0.03490	0.90870	0.0500*
H21	0.55190	−0.22110	0.87430	0.0660*

H22	0.42450	−0.19450	0.79260	0.0670*
H25	0.06180	−0.08480	0.57810	0.0630*
H26	−0.04650	0.04760	0.55210	0.0630*
H29	0.08940	0.17360	0.73810	0.0600*
H30	−0.02600	0.30640	0.70710	0.0690*
H31	−0.12150	0.32210	0.62160	0.0750*
H32	−0.11600	0.20410	0.56750	0.0680*
H35	0.374 (4)	−0.0908 (14)	1.0007 (12)	0.078 (11)*
H36	0.372 (4)	−0.1896 (14)	0.9847 (12)	0.074 (10)*

Atomicdisplacementparameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0502 (4)	0.0394 (4)	0.0367 (4)	−0.0035 (3)	−0.0046 (3)	−0.0035 (3)
O1	0.0788 (15)	0.0467 (13)	0.0556 (14)	0.0043 (11)	−0.0111 (11)	0.0017 (11)
O2	0.0771 (14)	0.0417 (11)	0.0519 (13)	−0.0182 (10)	−0.0054 (10)	−0.0071 (9)
O3	0.0487 (11)	0.0552 (12)	0.0497 (12)	0.0076 (9)	−0.0085 (9)	−0.0022 (10)
N1	0.0558 (15)	0.0432 (14)	0.0360 (14)	−0.0002 (12)	−0.0032 (11)	−0.0036 (11)
N2	0.0448 (13)	0.0427 (13)	0.0352 (13)	−0.0062 (10)	0.0032 (10)	−0.0040 (11)
N3	0.0525 (15)	0.0448 (15)	0.0404 (14)	0.0055 (13)	0.0031 (11)	−0.0014 (12)
C1	0.0459 (16)	0.0430 (16)	0.0334 (15)	−0.0016 (13)	−0.0003 (12)	−0.0004 (12)
C2	0.0514 (16)	0.0358 (15)	0.0370 (16)	0.0012 (13)	0.0015 (12)	−0.0069 (12)
C3	0.0499 (16)	0.0370 (15)	0.0385 (16)	0.0024 (13)	−0.0004 (12)	0.0014 (12)
C4	0.0461 (15)	0.0370 (15)	0.0343 (15)	−0.0016 (12)	−0.0010 (11)	−0.0010 (12)
C5	0.081 (2)	0.0352 (16)	0.0442 (18)	0.0015 (15)	−0.0060 (15)	−0.0061 (13)
C6	0.083 (2)	0.0373 (16)	0.0429 (18)	0.0063 (15)	−0.0091 (15)	0.0005 (14)
C7	0.0377 (14)	0.0481 (16)	0.0339 (15)	−0.0052 (13)	0.0006 (11)	−0.0003 (13)
C8	0.0454 (16)	0.0494 (18)	0.0444 (18)	−0.0077 (14)	−0.0003 (13)	0.0021 (14)
C9	0.0530 (18)	0.061 (2)	0.0453 (18)	−0.0048 (15)	−0.0059 (14)	0.0084 (16)
C10	0.0456 (17)	0.080 (2)	0.0345 (16)	−0.0126 (16)	−0.0070 (13)	0.0032 (16)
C11	0.0391 (15)	0.0647 (19)	0.0355 (16)	−0.0129 (14)	0.0033 (12)	−0.0062 (14)
C12	0.0381 (15)	0.0528 (17)	0.0382 (16)	−0.0087 (13)	0.0045 (12)	−0.0064 (13)
C13	0.0540 (18)	0.0535 (19)	0.0421 (17)	−0.0011 (15)	−0.0027 (13)	−0.0084 (14)
C14	0.0597 (19)	0.0538 (19)	0.063 (2)	0.0015 (15)	0.0004 (16)	−0.0130 (16)
C15	0.057 (2)	0.070 (2)	0.066 (2)	−0.0023 (17)	0.0044 (17)	−0.0299 (19)
C16	0.0478 (18)	0.082 (2)	0.0419 (18)	−0.0109 (17)	0.0036 (14)	−0.0184 (17)

S2	0.0487 (4)	0.0396 (4)	0.0389 (4)	0.0020 (3)	−0.0072 (3)	0.0031 (3)
O4	0.0828 (16)	0.0439 (12)	0.0544 (13)	0.0049 (11)	−0.0066 (11)	−0.0028 (10)
O5	0.0525 (11)	0.0499 (12)	0.0460 (12)	−0.0092 (9)	−0.0088 (9)	−0.0026 (9)
O6	0.0738 (14)	0.0515 (13)	0.0573 (14)	0.0244 (11)	−0.0088 (11)	0.0057 (10)
N4	0.0616 (15)	0.0418 (14)	0.0370 (14)	0.0038 (12)	−0.0052 (11)	0.0021 (12)
N5	0.0451 (13)	0.0435 (14)	0.0391 (14)	−0.0009 (11)	−0.0006 (10)	0.0053 (11)
N6	0.0605 (16)	0.0428 (15)	0.0433 (15)	−0.0127 (13)	0.0030 (12)	0.0030 (12)
C17	0.0456 (15)	0.0412 (16)	0.0339 (15)	−0.0015 (12)	−0.0017 (12)	0.0020 (12)
C18	0.0485 (16)	0.0358 (15)	0.0438 (17)	0.0037 (12)	−0.0014 (12)	0.0071 (13)
C19	0.0480 (16)	0.0355 (15)	0.0405 (16)	−0.0002 (12)	−0.0006 (12)	−0.0037 (12)
C20	0.0423 (15)	0.0352 (14)	0.0389 (15)	0.0019 (12)	−0.0043 (11)	0.0003 (12)
C21	0.079 (2)	0.0338 (16)	0.0476 (18)	0.0085 (15)	−0.0131 (15)	−0.0005 (13)
C22	0.084 (2)	0.0373 (16)	0.0428 (18)	0.0054 (15)	−0.0115 (15)	−0.0048 (13)
C23	0.0401 (14)	0.0440 (16)	0.0330 (15)	−0.0022 (12)	0.0000 (11)	0.0060 (12)
C24	0.0470 (16)	0.0488 (18)	0.0422 (17)	−0.0037 (14)	0.0013 (13)	−0.0011 (14)
C25	0.0547 (18)	0.062 (2)	0.0390 (17)	−0.0022 (16)	−0.0017 (13)	−0.0061 (15)
C26	0.0484 (17)	0.072 (2)	0.0352 (16)	−0.0029 (16)	−0.0042 (13)	0.0020 (15)
C27	0.0377 (15)	0.0565 (18)	0.0399 (16)	−0.0018 (13)	−0.0009 (12)	0.0071 (14)
C28	0.0363 (14)	0.0492 (17)	0.0390 (16)	−0.0017 (12)	0.0044 (11)	0.0052 (13)
C29	0.0495 (17)	0.0549 (19)	0.0446 (17)	0.0026 (14)	0.0010 (13)	0.0027 (15)
C30	0.0583 (19)	0.0516 (19)	0.063 (2)	0.0093 (15)	0.0031 (16)	0.0012 (16)
C31	0.0560 (19)	0.060 (2)	0.072 (2)	0.0147 (16)	0.0016 (17)	0.0183 (19)
C32	0.0501 (18)	0.069 (2)	0.0492 (19)	0.0081 (16)	−0.0066 (14)	0.0166 (17)

Geometricparameters (Å, °)

S1—O2	1.433 (2)	O1—C8	1.259 (4)
S1—O3	1.433 (2)	O4—C24	1.258 (4)
S1—N3	1.614 (3)	N1—C1	1.399 (4)
S1—C4	1.760 (3)	N1—N2	1.314 (3)
S2—O5	1.437 (2)	N2—C7	1.332 (3)
S2—O6	1.432 (2)	N4—N5	1.315 (3)
S2—N6	1.610 (3)	N4—C17	1.394 (4)
S2—C20	1.759 (3)	N5—C23	1.331 (3)

O2—S1—O3	119.19 (12)	N1—C1—C6	115.8 (2)
O2—S1—N3	106.65 (13)	N1—C1—C2	123.8 (2)

O2—S1—C4	107.76 (13)	S1—C4—C5	118.1 (2)
O3—S1—N3	106.52 (12)	S1—C4—C3	121.4 (2)
O3—S1—C4	108.54 (12)	N2—C7—C12	116.5 (2)
N3—S1—C4	107.69 (13)	N2—C7—C8	123.6 (2)
N6—S2—C20	108.51 (12)	O1—C8—C7	121.8 (3)
O5—S2—C20	108.37 (12)	O1—C8—C9	120.5 (3)
O5—S2—O6	119.07 (12)	N4—C17—C22	116.9 (2)
O5—S2—N6	105.99 (12)	N4—C17—C18	123.7 (2)
O6—S2—N6	106.95 (13)	S2—C20—C19	120.7 (2)
O6—S2—C20	107.58 (12)	S2—C20—C21	119.0 (2)
N2—N1—C1	122.1 (2)	N5—C23—C28	116.4 (2)
N1—N2—C7	117.2 (2)	N5—C23—C24	123.9 (2)
N5—N4—C17	121.7 (2)	O4—C24—C23	121.2 (3)
N4—N5—C23	117.4 (2)	O4—C24—C25	120.8 (3)

Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles.The dimeric structure of the compound in the crystal is given in **Fig. 2.**

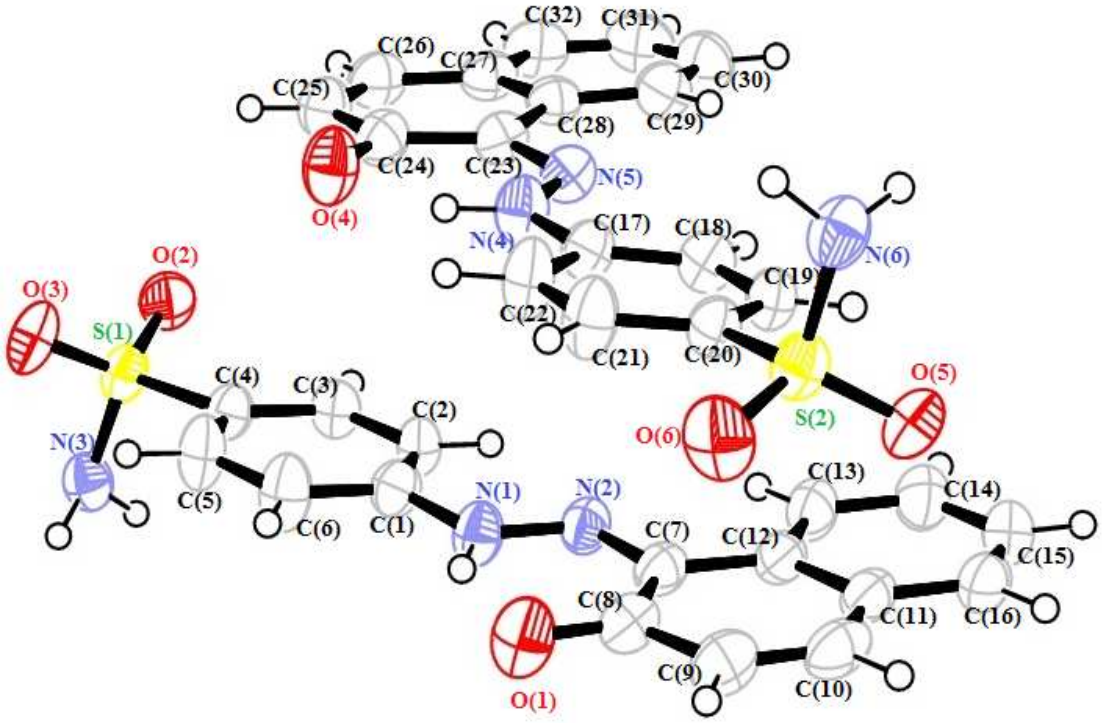


Figure 2: Thedimeric structure of the compound in the crystal.

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