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N-[7-Ethoxy-2-(prop-2-en-1-yl)-2H-indazol-6-yl]-4-methylbenzene-sulfonamide

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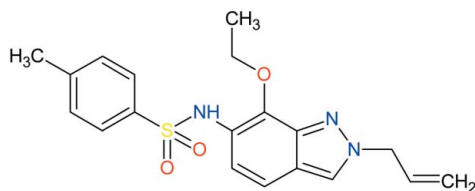
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Key indicators: single-crystal X-ray study; $T = 296$ K, $P = 0.0$ kPa; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 16.9.

In the title compound, $\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_3\text{S}$, the $\text{C}-\text{SO}_2-\text{NH}-\text{C}$ torsion angle is 66.20 (9)°. The dihedral angle between the benzene ring and the essentially planar indazole ring system [r.m.s. deviation = 0.0361 (1) Å] is 72.97 (6)°. The S atom has a distorted tetrahedral geometry [maximum deviation = $\text{O}-\text{S}-\text{O} = 119.30$ (6)°]. The crystal structure features inversion-related dimers linked by pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. In addition, weak $\text{C}-\text{H}\cdots\text{O}$ interactions may stabilize the crystal packing.

Related literature

For related structures, see: Abbassi *et al.* (2011*a,b*). For the biological activity of sulfonamides, see: Soledade *et al.* (2006); Lee & Lee (2002). For the synthesis of 7-ethoxy-*N*-alkyl-indazole derivatives, see: Abbassi *et al.* (2011*c*).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_3\text{S}$
 $M_r = 371.45$
 Monoclinic, $P2_1/c$
 $a = 10.1459$ (7) Å
 $b = 9.9506$ (7) Å
 $c = 18.3720$ (13) Å
 $\beta = 95.097$ (3)°
 $V = 1847.5$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.20$ mm⁻¹
 $T = 296$ K
 $0.32 \times 0.31 \times 0.24$ mm

Data collection

Bruker APEXII CCD detector
 diffractometer
 20488 measured reflections
 4034 independent reflections
 3693 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.100$
 $S = 1.06$
 4034 reflections
 238 parameters
 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.69$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O3}^i$	0.86	2.21	3.0199 (15)	159
$\text{C8}-\text{H8}\cdots\text{O2}^{ii}$	0.93	2.44	3.1270 (17)	131

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: publCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5700).

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supplementary materials

Acta Cryst. (2011). E67, o3211 [doi:10.1107/S1600536811045855]

N-[7-Ethoxy-2-(prop-2-en-1-yl)-2*H*-indazol-6-yl]-4-methylbenzenesulfonamide

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Comment

Various sulfonamides are widely used as anti-hypertensive (Soledade *et al.*, 2006; Lee & Lee, 2002). In former papers, we reported the crystal structures of *N*-(7-ethoxy-1*H*-indazol-4-yl)-4-methylbenzenesulfonamide (Abbassi *et al.*, 2011*a*) and *N*-[7-ethoxy-1-(prop-2-en-1-yl)-1*H*-indazol-4-yl]-4-methylbenzenesulfonamide (Abbassi *et al.*, 2011*b*). In this communication, the crystal structure of *N*-[7-ethoxy-2-(prop-2-en-1-yl)-2*H*-indazol-6-yl]-4-methylbenzenesulfonamide is reported.

In the title compound, C₁₉H₂₁N₃O₃S, the C—SO₂—NH—C torsion angle is 66.20 (9)°. The dihedral angle between the aromatic ring and the indazol system is 72.97 (6)°. The S atom has a distorted tetrahedral geometry [maximum deviation: O—S—O = 119.3 (6)°] (Fig. 1).

Two neighbouring molecules generate a hydrogen-bonded dimer about a center of inversion through a pair of intermolecular N—H···O interactions (Figs. 2 and 3).

Experimental

A mixture of 2-allyl-6-nitro-2*H*-indazole [Abbassi *et al.*, 2011*c*] (1.22 mmol) and anhydrous SnCl₂ (1.1 g, 6.1 mmol) in 25 mL of absolute ethanol was heated at 60 °C for 2 h. After reduction, the starting material disappeared, and the solution was allowed to cool down. The pH was made slightly basic (pH 7–8) by addition of 5% aqueous potassium bicarbonate before extraction with ethyl acetate. The organic phase was washed with brine and dried over magnesium sulfate. The solvent was removed to afford the amine, which was immediately dissolved in pyridine (5 ml) and then reacted with 4-methylbenzenesulfonyl chloride (0.26 g, 1.25 mmol) at room temperature for 24 h. After the reaction mixture was concentrated *in vacuo*, the resulting residue was purified by flash chromatography (eluted with Ethyl acetate: Hexane 1:9).

Refinement

The H atoms bound to C were positioned geometrically and constrained to ride on their parent atoms [C—H distances are 0.93 Å for CH groups with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$, and 0.97 Å for CH₃ groups, and the coordinates for the H atom bonded to N were taken from a difference map, and the atom was refined using a riding model.

Figures

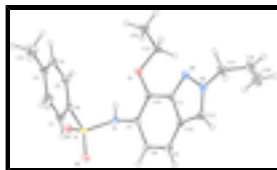


Fig. 1. Molecular view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

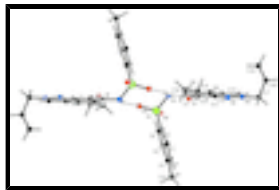


Fig. 2. View of the N—H···O bonded dimers of the title compound.

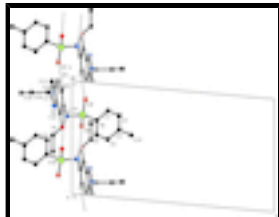


Fig. 3. Partial packing view showing N—H···O and C—H···O hydrogen bonds. H atoms not involved in hydrogen bonds have been omitted for clarity.

***N*-[7-Ethoxy-2-(prop-2-en-1-yl)-2*H*-indazol-6-yl]-4- methylbenzenesulfonamide**

Crystal data

C₁₉H₂₁N₃O₃S

M_r = 371.45

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 10.1459 (7) Å

b = 9.9506 (7) Å

c = 18.3720 (13) Å

β = 95.097 (3)°

V = 1847.5 (2) Å³

Z = 4

F(000) = 784

D_x = 1.335 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 243 reflections

θ = 2.3–27.2°

μ = 0.20 mm⁻¹

T = 296 K

Prism, colourless

0.32 × 0.31 × 0.24 mm

Data collection

Bruker APEXII CCD detector
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω and φ scans

20488 measured reflections

4034 independent reflections

3693 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.031

θ_{max} = 27.0°, θ_{min} = 2.0°

h = -12→12

k = -12→12

l = -22→23

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.036

wR(*F*²) = 0.100

S = 1.06

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0522*P*)² + 0.8814*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

4034 reflections	$\Delta\rho_{\max} = 0.69 \text{ e } \text{\AA}^{-3}$
238 parameters	$\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0206 (15)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.76485 (15)	0.66788 (16)	0.17902 (8)	0.0312 (3)
C10	0.93118 (13)	0.91852 (15)	-0.08185 (7)	0.0252 (3)
C11	0.79381 (13)	0.94222 (14)	-0.07596 (7)	0.0216 (3)
C12	0.71089 (12)	0.83505 (13)	-0.05612 (7)	0.0207 (3)
C13	0.98070 (14)	1.04447 (16)	-0.09800 (8)	0.0296 (3)
C14	0.5674 (2)	0.8996 (2)	0.29302 (10)	0.0556 (6)
C15	0.50380 (15)	0.94048 (18)	-0.09413 (10)	0.0373 (4)
C16	0.36049 (15)	0.90887 (17)	-0.09383 (10)	0.0354 (4)
C17	0.88027 (17)	1.27272 (16)	-0.11789 (10)	0.0375 (4)
C18	0.86633 (18)	1.2985 (2)	-0.19860 (12)	0.0523 (5)
C19	0.8539 (2)	1.2068 (3)	-0.24945 (12)	0.0708 (7)
C2	0.73094 (17)	0.75204 (19)	0.23458 (9)	0.0389 (4)
C3	0.60421 (17)	0.80532 (16)	0.23408 (8)	0.0335 (3)
C4	0.51129 (15)	0.77163 (14)	0.17715 (8)	0.0283 (3)
C5	0.54257 (13)	0.68865 (14)	0.12097 (8)	0.0244 (3)
C6	0.67012 (13)	0.63684 (13)	0.12255 (7)	0.0211 (3)
C7	0.76898 (13)	0.71167 (13)	-0.04258 (7)	0.0208 (3)
C8	0.90547 (13)	0.68843 (14)	-0.05067 (8)	0.0256 (3)
C9	0.98638 (14)	0.78904 (15)	-0.06954 (8)	0.0284 (3)
H1	0.8499	0.6328	0.1797	0.037*
H13	1.0678	1.0651	-0.1057	0.036*
H14A	0.5216	0.9759	0.2711	0.083*
H14B	0.6461	0.9291	0.3214	0.083*
H14C	0.5109	0.8538	0.3242	0.083*
H15A	0.5298	0.9353	-0.1436	0.045*
H15B	0.5208	1.0310	-0.0761	0.045*

supplementary materials

H16A	0.3429	0.8224	-0.1158	0.053*
H16B	0.3094	0.9761	-0.1211	0.053*
H16C	0.3368	0.9077	-0.0444	0.053*
H17A	0.9628	1.3116	-0.0969	0.045*
H17B	0.8085	1.3171	-0.0958	0.045*
H18	0.8666	1.3877	-0.2137	0.063*
H19A	0.8532	1.1163	-0.2367	0.085*
H19B	0.8458	1.2318	-0.2984	0.085*
H1N	0.6049	0.6137	-0.0394	0.026*
H2	0.7940	0.7731	0.2728	0.047*
H4	0.4259	0.8057	0.1768	0.034*
H5	0.4795	0.6678	0.0828	0.029*
H8	0.9400	0.6025	-0.0429	0.031*
H9	1.0756	0.7732	-0.0742	0.034*
N1	0.68677 (11)	0.60110 (11)	-0.02548 (6)	0.0216 (2)
N2	0.76199 (11)	1.07276 (12)	-0.08760 (7)	0.0254 (3)
N3	0.87862 (12)	1.13021 (12)	-0.10026 (7)	0.0275 (3)
O1	0.57901 (9)	0.84573 (10)	-0.04824 (5)	0.0253 (2)
O2	0.84526 (10)	0.49024 (11)	0.06679 (6)	0.0292 (2)
O3	0.61092 (10)	0.41953 (10)	0.04911 (6)	0.0286 (2)
S1	0.70831 (3)	0.52451 (3)	0.053468 (17)	0.02072 (11)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0241 (7)	0.0381 (8)	0.0310 (7)	0.0053 (6)	-0.0010 (6)	-0.0003 (6)
C10	0.0198 (6)	0.0297 (7)	0.0264 (6)	-0.0003 (5)	0.0042 (5)	0.0030 (5)
C11	0.0198 (6)	0.0234 (6)	0.0218 (6)	0.0011 (5)	0.0027 (5)	0.0019 (5)
C12	0.0169 (6)	0.0233 (6)	0.0221 (6)	0.0018 (5)	0.0038 (5)	0.0004 (5)
C13	0.0210 (7)	0.0338 (8)	0.0343 (7)	-0.0038 (6)	0.0038 (5)	0.0060 (6)
C14	0.0765 (14)	0.0595 (12)	0.0305 (8)	0.0257 (11)	0.0018 (9)	-0.0124 (8)
C15	0.0246 (7)	0.0411 (9)	0.0460 (9)	0.0066 (6)	0.0024 (6)	0.0175 (7)
C16	0.0217 (7)	0.0376 (8)	0.0463 (9)	0.0036 (6)	-0.0010 (6)	0.0049 (7)
C17	0.0338 (8)	0.0267 (8)	0.0519 (10)	-0.0066 (6)	0.0024 (7)	0.0104 (7)
C18	0.0383 (10)	0.0547 (12)	0.0637 (13)	-0.0004 (8)	0.0034 (9)	0.0353 (10)
C19	0.0733 (16)	0.099 (2)	0.0401 (11)	0.0078 (14)	0.0022 (10)	0.0231 (12)
C2	0.0410 (9)	0.0466 (10)	0.0272 (7)	0.0060 (7)	-0.0072 (6)	-0.0061 (7)
C3	0.0465 (9)	0.0310 (8)	0.0236 (7)	0.0083 (7)	0.0064 (6)	0.0012 (6)
C4	0.0294 (7)	0.0253 (7)	0.0312 (7)	0.0078 (6)	0.0079 (6)	0.0042 (6)
C5	0.0220 (6)	0.0225 (6)	0.0284 (7)	0.0020 (5)	0.0016 (5)	0.0012 (5)
C6	0.0222 (6)	0.0191 (6)	0.0225 (6)	0.0017 (5)	0.0044 (5)	0.0023 (5)
C7	0.0204 (6)	0.0214 (6)	0.0210 (6)	0.0013 (5)	0.0044 (5)	0.0006 (5)
C8	0.0223 (7)	0.0264 (7)	0.0287 (7)	0.0071 (5)	0.0064 (5)	0.0026 (5)
C9	0.0189 (6)	0.0338 (8)	0.0333 (7)	0.0045 (5)	0.0071 (5)	0.0046 (6)
N1	0.0201 (5)	0.0204 (5)	0.0242 (5)	0.0014 (4)	0.0022 (4)	0.0021 (4)
N2	0.0225 (6)	0.0233 (6)	0.0306 (6)	-0.0027 (4)	0.0036 (4)	0.0047 (5)
N3	0.0249 (6)	0.0261 (6)	0.0317 (6)	-0.0052 (5)	0.0024 (5)	0.0063 (5)
O1	0.0169 (5)	0.0249 (5)	0.0346 (5)	0.0035 (4)	0.0052 (4)	0.0079 (4)

O2	0.0237 (5)	0.0299 (5)	0.0345 (5)	0.0111 (4)	0.0053 (4)	0.0055 (4)
O3	0.0317 (5)	0.0183 (5)	0.0362 (5)	-0.0026 (4)	0.0045 (4)	0.0027 (4)
S1	0.02049 (18)	0.01647 (17)	0.02557 (18)	0.00367 (11)	0.00417 (12)	0.00238 (11)

Geometric parameters (Å, °)

C1—H1	0.9300	C19—H19B	0.9300
C1—C2	1.387 (2)	C19—H19A	0.9300
C10—C9	1.415 (2)	C19—C18	1.304 (4)
C10—C13	1.392 (2)	C2—H2	0.9300
C11—C10	1.4271 (18)	C3—C14	1.505 (2)
C11—C12	1.4257 (18)	C3—C2	1.390 (2)
C11—N2	1.3509 (18)	C4—H4	0.9300
C12—O1	1.3626 (15)	C4—C3	1.386 (2)
C13—H13	0.9300	C5—H5	0.9300
C13—N3	1.340 (2)	C5—C4	1.381 (2)
C14—H14C	0.9600	C6—C5	1.3910 (18)
C14—H14B	0.9600	C6—C1	1.385 (2)
C14—H14A	0.9600	C7—N1	1.4323 (17)
C15—H15B	0.9700	C7—C8	1.4247 (18)
C15—H15A	0.9700	C7—C12	1.3749 (18)
C15—C16	1.488 (2)	C8—H8	0.9300
C15—O1	1.4380 (17)	C8—C9	1.359 (2)
C16—H16C	0.9600	C9—H9	0.9300
C16—H16B	0.9600	N1—H1N	0.8562
C16—H16A	0.9600	N2—N3	1.3528 (16)
C17—H17B	0.9700	S1—C6	1.7600 (13)
C17—H17A	0.9700	S1—N1	1.6358 (11)
C17—C18	1.499 (3)	S1—O3	1.4352 (10)
C17—N3	1.4549 (19)	S1—O2	1.4305 (10)
C18—H18	0.9300		
C2—C1—H1	120.5	H19A—C19—H19B	120.0
C6—C1—H1	120.5	C18—C19—H19B	120.0
C6—C1—C2	119.01 (14)	C18—C19—H19A	120.0
C9—C10—C11	120.91 (13)	C3—C2—H2	119.5
C13—C10—C11	103.93 (12)	C1—C2—H2	119.5
C13—C10—C9	135.12 (13)	C1—C2—C3	121.00 (14)
C12—C11—C10	119.89 (12)	C2—C3—C14	121.45 (16)
N2—C11—C10	111.60 (12)	C4—C3—C14	119.87 (15)
N2—C11—C12	128.43 (12)	C4—C3—C2	118.67 (14)
C7—C12—C11	117.44 (12)	C3—C4—H4	119.2
O1—C12—C11	125.18 (12)	C5—C4—H4	119.2
O1—C12—C7	117.36 (12)	C5—C4—C3	121.52 (13)
C10—C13—H13	126.6	C6—C5—H5	120.6
N3—C13—H13	126.6	C4—C5—H5	120.6
N3—C13—C10	106.70 (12)	C4—C5—C6	118.77 (13)
H14B—C14—H14C	109.5	C5—C6—S1	118.97 (10)
H14A—C14—H14C	109.5	C1—C6—S1	119.95 (11)
C3—C14—H14C	109.5	C1—C6—C5	121.02 (13)

supplementary materials

H14A—C14—H14B	109.5	C8—C7—N1	119.24 (12)
C3—C14—H14B	109.5	C12—C7—N1	118.49 (11)
C3—C14—H14A	109.5	C12—C7—C8	122.08 (12)
H15A—C15—H15B	108.3	C7—C8—H8	119.3
C16—C15—H15B	109.9	C9—C8—H8	119.3
O1—C15—H15B	109.9	C9—C8—C7	121.45 (13)
C16—C15—H15A	109.9	C10—C9—H9	120.9
O1—C15—H15A	109.9	C8—C9—H9	120.9
O1—C15—C16	109.09 (13)	C8—C9—C10	118.16 (12)
H16B—C16—H16C	109.5	S1—N1—H1N	112.5
H16A—C16—H16C	109.5	C7—N1—H1N	112.9
C15—C16—H16C	109.5	C7—N1—S1	121.19 (9)
H16A—C16—H16B	109.5	C11—N2—N3	103.48 (11)
C15—C16—H16B	109.5	N2—N3—C17	118.53 (12)
C15—C16—H16A	109.5	C13—N3—C17	127.12 (13)
H17A—C17—H17B	107.8	C13—N3—N2	114.27 (12)
C18—C17—H17B	109.1	C12—O1—C15	117.63 (11)
N3—C17—H17B	109.1	N1—S1—C6	108.84 (6)
C18—C17—H17A	109.1	O3—S1—C6	107.93 (6)
N3—C17—H17A	109.1	O2—S1—C6	107.50 (6)
N3—C17—C18	112.65 (15)	O3—S1—N1	104.63 (6)
C17—C18—H18	117.2	O2—S1—N1	108.30 (6)
C19—C18—H18	117.2	O2—S1—O3	119.30 (6)
C19—C18—C17	125.68 (19)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O3 ⁱ	0.86	2.21	3.0199 (15)	159.
C8—H8 \cdots O2 ⁱⁱ	0.93	2.44	3.1270 (17)	131.

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

Fig. 1

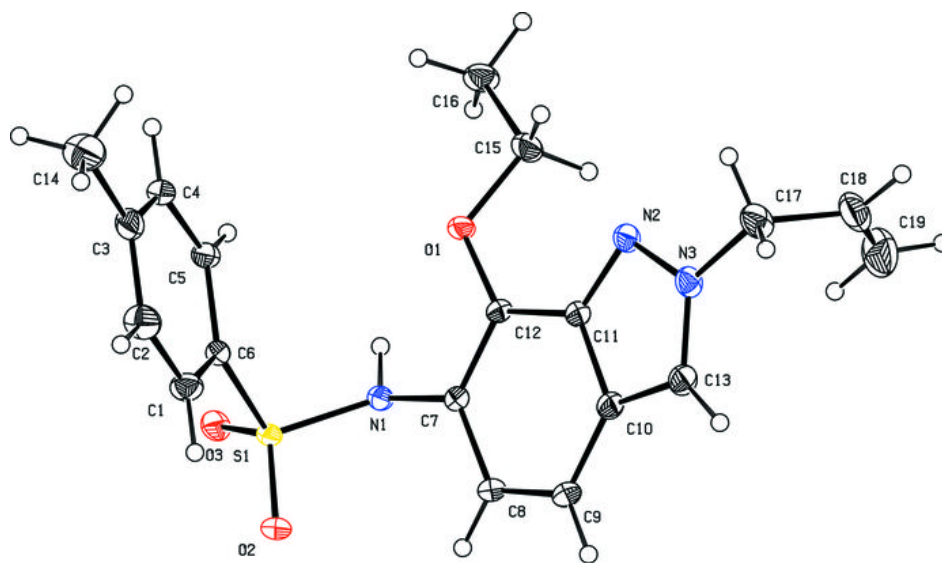


Fig. 2

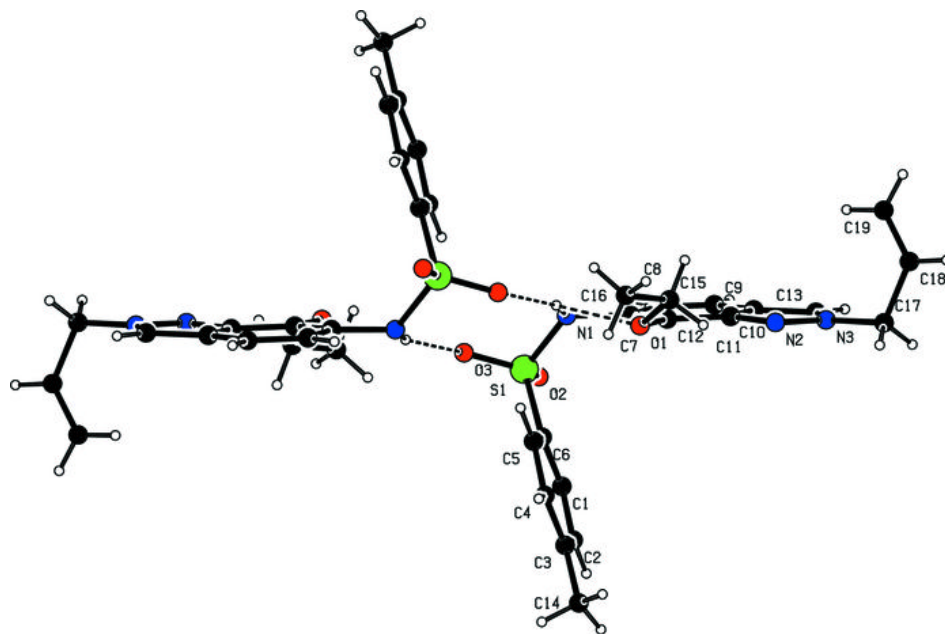


Fig. 3

