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N-[3-Cyano-2-oxo-5,6,7,8-tetrahydroquinoline-1(2*H*)-yl]-4-methylbenzenesulfonamide

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Key indicators

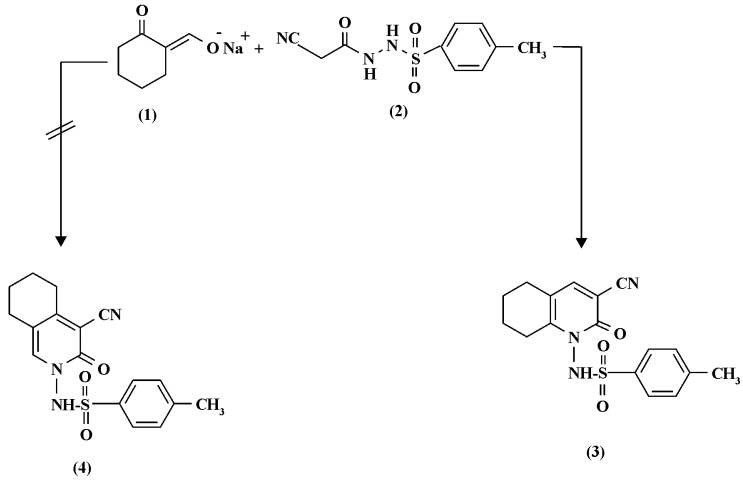
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.089
Data-to-parameter ratio = 11.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{17}H_{17}N_3O_3S$, key bond lengths are N—N 1.408 (2) and N—S 1.7023 (15) Å. The relative orientation of the two ring systems is defined by the torsion angles between them, namely C—N—N—S 94.99 (15), N—N—S—C 123.90 (12) and N—S—C—C 107.86 (15)°. The molecules are linked in inversion-symmetric pairs by classical hydrogen bonds N—H···O=C. All four CH_2 groups of the non-aromatic ring are disordered over two positions, corresponding to two alternative conformations.

Comment

Recently, antimetabolites have attracted much interest in organic chemistry, because of their biological activity and unique chemical properties. We have studied the chemistry of antimetabolites and developed some new routes for the synthesis of novel nonclassical antimetabolites (Elgemeie *et al.*, 1997, 1999). Recently, arylsulfonylhydrazones of pyridines have been shown to inhibit thymidine and uridine incorporation into DNA and RNA; thus, they have useful properties as antimetabolites in biochemical reactions (Bernstein *et al.*, 1995). Since these sulfonylhydrazones of pyridines appear to constitute a new class of compounds with potent antineoplastic properties, it was of interest to evaluate relevant syntheses.



The present investigation reports the synthesis of a novel *N*-arylsulfonylamino derivative of 2-pyridone; as far as we know, this is the first such derivative to be reported for pyridones. 2-(Hydroxymethylene)-1-cyclohexanone (1) reacts with cyanoacetyl-*N*-(*p*-tolyl)sulfonylhydrazide (2) in piperidine acetate, to afford a product of molecular formula $C_{17}H_{17}N_3O_3S$ for which two isomeric structures, (3) and (4), seemed possible. Spectroscopic data cannot differentiate between these structures. The X-ray structure determination

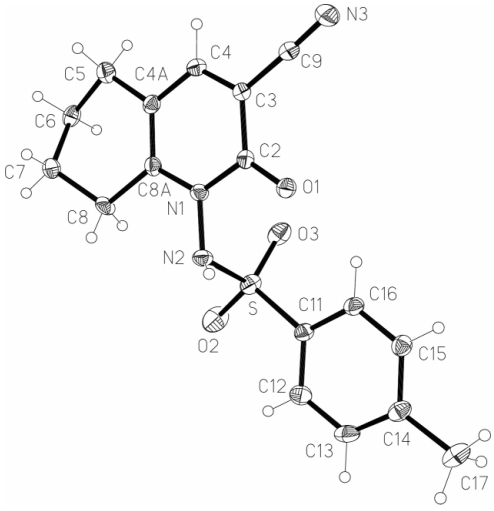


Figure 1
The molecule of compound (3) in the crystal. Ellipsoids are drawn at the 30% probability level. H-atom radii are arbitrary. Only the major disorder component is shown (see *Experimental*).

indicated form (3) for the product in the solid state. The formation of (3) from the reaction of (1) and (2) is assumed to proceed *via* initial addition of the active methylene-C atom of (2) to the formyl group of (1) to give the favoured, kinetically controlled product (3).

The structure of (3) is shown in Fig. 1. Molecular dimensions, *e.g.* bond lengths N1—N2 1.408 (2) and N2—S 1.7023 (15) Å, may be considered normal. The molecular conformation is defined by the torsion angles given in Table 1. The molecules are linked by the classical hydrogen bond N2—H0···O1 to form inversion-symmetric dimers.

The non-aromatic ring is disordered, showing two alternative conformations (see *Experimental*).

Experimental

A solution of the sodium salt of 2-(hydroxymethylene)-1-cyclohexanone [(1); 1.47 g, 0.01 mol], cyanoaceto-*N*-(*p*-tolyl)sulfonylhydrazide [(2); 3.4 g, 0.01 mol] , and piperidine acetate (1 ml) in water (30 ml) and ethanol (30 ml) was refluxed for 10 min. Acetic acid (1.5 ml) was added to the hot solution. The precipitate was collected by filtration and crystallized from ethanol in 88% yield (m.p. 513 K).

Crystal data

$C_{17}H_{17}N_3O_3S$
M_r = 343.40
Monoclinic, $P2_1/n$
a = 11.3275 (12) Å
b = 12.0995 (10) Å
c = 11.8282 (10) Å
β = 92.377 (9)°
V = 1619.7 (3) Å^3
Z = 4
D_x = 1.408 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 73 reflections
θ = 4.5–12.5°
μ = 0.22 mm^{-1}
T = 173 (2) K
Prism, colourless
0.45 × 0.40 × 0.40 mm

Data collection

Siemens *P*4 diffractometer
ω- scans
3893 measured reflections
2846 independent reflections
2195 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°
$h = 0 \rightarrow 13$
$k = -14 \rightarrow 3$
$l = -14 \rightarrow 14$
3 standard reflections
every 247 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 1.01
2846 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S—O2	1.4215 (13)	N1—C8*A*	1.387 (2)
S—O3	1.4341 (14)	N1—C2	1.398 (2)
S—N2	1.7023 (15)	N1—N2	1.408 (2)
S—C11	1.7586 (17)	N3—C9	1.144 (2)
O1—C2	1.2318 (19)		
O2—S—O3	118.57 (9)	C8*A*—N1—N2	117.35 (13)
O2—S—N2	106.22 (8)	C2—N1—N2	117.21 (13)
O3—S—N2	106.77 (8)	N1—N2—S	113.63 (11)
O2—S—C11	108.51 (9)	O1—C2—N1	120.70 (15)
O3—S—C11	110.41 (8)	O1—C2—C3	125.86 (15)
N2—S—C11	105.51 (8)	N1—C2—C3	113.43 (14)
C8*A*—N1—C2	125.44 (14)		
C8*A*—N1—N2—S	94.99 (15)	N2—S—C11—C12	107.86 (15)
C11—S—N2—N1	123.90 (12)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H0···O1^{i}	0.820 (18)	2.089 (19)	2.8884 (19)	164.8 (18)

Symmetry code: (i) $1-x, 1-y, 1-z$.

The hydrogen at N2 was refined freely. Methyl H atoms were located in difference syntheses, idealized (C—H 0.98 Å, H—C—H 109.5°) and refined on the basis of rigid groups allowed to rotate but not tip. Other H atoms were included using a riding model with fixed C—H bond lengths (aromatic 0.95, methylene 0.99 Å); U(H) values were fixed at $1.2U_{eq}$ of the parent atom. The atoms C5–C8 are disordered over two positions with occupancies 0.611:0.389 (7); to improve stability of refinement, appropriate similarity restraints were employed (the final instruction file is included in the deposited material).

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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