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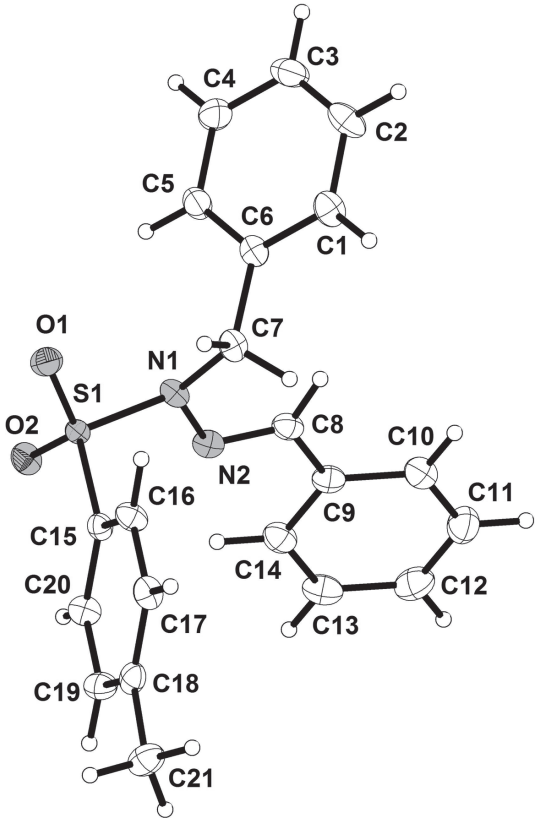
The crystal structure of (*E*)-*N*-benzyl-*N*′-benzylidene-4-methylbenzenesulfonohydrazide, $C_{21}H_{20}N_2O_2S$



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Abstract

$C_{21}H_{20}N_2O_2S$, monoclinic, $P2_1/c$ (no. 14), $a = 19.436(3)$ Å, $b = 6.1396(6)$ Å, $c = 16.362(3)$ Å, $\beta = 108.100(16)°$, $V = 1855.8(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0502$, $wR_{ref}(F^2) = 0.1224$, $T = 100$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Needle, colorless
Size:	0.56 × 0.11 × 0.1 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.19 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
$2\theta_{max}$, completeness:	26.4°, >89%; (> 99% up to 25.2θ)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14722, 3797, 0.047
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3283
$N(param)_{refined}$:	236
Programs:	CrysAlis^{PRO} [1], SHELX [2]

Source of material

The title compound was prepared by the following method: *N*′-benzylidene-4-methylbenzenesulfonohydrazide (0.823 g, 3 mmol) and NaOMe (0.243 g, 4.5 mol) in anhydrous methanol 20 mL were mixed. The reaction mixture was refluxed for 24 h in 323 K, then washed in portions with water and extracted by dichloromethane. The organic layer was evaporated and purified by column chromatography using ethyl acetate and petroleum ether. Crystallization from ethyl acetate/petroleum ether gives a colorless solid; Yield 81%.

Experimental details

All hydrogen atoms were placed geometrically on calculated positions using a riding model with the help of the SHELX program [2].

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.72510(8)	0.4340(3)	0.62858(11)	0.0161(4)
N2	0.78332(9)	0.3428(3)	0.69111(11)	0.0169(4)
O1	0.59676(7)	0.4039(2)	0.54563(9)	0.0206(3)
O2	0.65873(7)	0.0892(2)	0.63330(9)	0.0201(3)
S1	0.64591(3)	0.31857(8)	0.62333(3)	0.01553(15)
C1	0.77937(11)	0.9122(3)	0.51902(15)	0.0217(5)
H1	0.7802	1.0241	0.5594	0.026*
C2	0.80290(12)	0.9551(4)	0.44847(15)	0.0268(5)
H2	0.8198	1.0967	0.4411	0.032*
C3	0.80201(12)	0.7936(4)	0.38892(15)	0.0271(5)
H3	0.8180	0.8242	0.3408	0.032*
C4	0.77757(12)	0.5869(4)	0.40020(15)	0.0260(5)
H4	0.7770	0.4753	0.3597	0.031*
C5	0.75394(11)	0.5422(3)	0.47056(14)	0.0216(5)
H5	0.7373	0.4003	0.4779	0.026*
C6	0.75453(10)	0.7048(3)	0.53034(13)	0.0170(4)
C7	0.72448(11)	0.6648(3)	0.60403(14)	0.0180(4)
H7A	0.7533	0.7495	0.6546	0.022*
H7B	0.6741	0.7193	0.5876	0.022*
C8	0.84486(11)	0.4391(3)	0.70750(13)	0.0182(4)
H8	0.8493	0.5652	0.6759	0.022*
C9	0.90824(11)	0.3552(3)	0.77473(13)	0.0186(4)
C10	0.96876(11)	0.4903(4)	0.80476(14)	0.0224(5)
H10	0.9689	0.6295	0.7795	0.027*
C11	1.02865(12)	0.4225(4)	0.87122(15)	0.0275(5)
H11	1.0694	0.5157	0.8914	0.033*
C12	1.02924(12)	0.2187(4)	0.90842(15)	0.0282(5)
H12	1.0702	0.1730	0.9542	0.034*
C13	0.96972(12)	0.0819(4)	0.87847(15)	0.0265(5)
H13	0.9701	−0.0577	0.9037	0.032*
C14	0.90971(11)	0.1488(3)	0.81186(14)	0.0221(5)
H14	0.8694	0.0541	0.7913	0.027*
C15	0.62046(10)	0.4122(3)	0.71154(13)	0.0146(4)
C16	0.58931(11)	0.6184(3)	0.70862(14)	0.0186(4)
H16	0.5821	0.7090	0.6596	0.022*
C17	0.56909(11)	0.6885(3)	0.77841(14)	0.0189(4)
H17	0.5487	0.8296	0.7772	0.023*
C18	0.57813(10)	0.5556(3)	0.85067(13)	0.0185(4)
C19	0.60883(11)	0.3500(3)	0.85148(14)	0.0200(4)
H19	0.6151	0.2577	0.8998	0.024*
C20	0.63040(11)	0.2781(3)	0.78261(14)	0.0188(4)
H20	0.6518	0.1384	0.7842	0.023*
C21	0.55247(11)	0.6285(4)	0.92408(14)	0.0251(5)
H21A	0.5538	0.7878	0.9276	0.038*
H21B	0.5841	0.5671	0.9780	0.038*
H21C	0.5028	0.5779	0.9145	0.038*

Discussion

Tosylhydrazones have versatile applications converting into various pharmaceutical and biological medicine [3–7]. It is well known that tosylhydrazones transform into diazo compounds under basic environment *via* a Bamford-Stevens reaction [8]. In addition, *N*-alkylation of tosylhydrazones *via* a metal-free reductive coupling procedure, are reported [9] while palladium catalysts are used in previous cases [10]. The product of the *N*-alkylation of tosylhydrazones are important synthetic intermediates as well [11, 12].

The geometry and labeling for the crystal structure of the title complex is depicted in the figure. Geometric parameters are in the expected ranges.

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