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N,*N*-Dibenzyltriphenylmethanesulfenamide

Iván Brito,[a]* Danitza Vargas,[a] Yasna León,[a] Alejandro Cárdenas,[b] Matías López-Rodríguez[c] and Oscar Wittke[d]

[a]Department of Chemistry, University of Antofagasta, Antofagasta Casilla 170, Antofagasta, Chile, [b]Department of Physics, University of Antofagasta, Antofagasta Casilla 170, Antofagasta, Chile, [c]Centro de Productos Naturales Orgánicos 'Antonio González', Instituto Universitario de Bio-Orgánica, Universidad de La Laguna, Astrofisico Francisco Sánchez No. 2, La Laguna, Tenerife, Spain, and [d]Department of Physics, Facultad de Ciencias Fisicas y Matemáticas, University of Chile, Casilla 487-3, Santiago, Chile

Correspondence e-mail: ibrito@uantof.cl

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The title compound, $C_{33}H_{29}NS$, is the result of the condensation reaction of $(C_6H_5)_3CSCl$ and $HN(CH_2C_6H_5)_2$. In its crystalline state, the C—S—N plane forms a dihedral angle of 88.5 (1)% with the C—N—C plane, in good agreement with the value of 90.0° for the torsional fundamental state of the molecule.

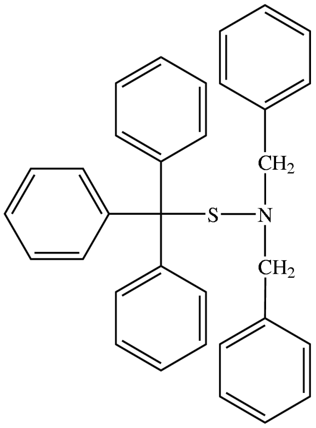
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.122
Data-to-parameter ratio = 15.1

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

Comment

Sulfenamides are important intermediates in organic synthesis. They have been useful in investigations of lone-pair interactions, bond-polarization effects and $(p\text{-}d)_\pi$ conjugation (Craine & Raban, 1989; Makosza & Bialecki, 1988; Blanca *et al.*, 1997). Sulfenamides also have important industrial applications, such as additives in the rubber industry, and in the agrochemical industry as insecticides, fungicides, and ovicides. They have been found to be potentially useful as growth regulators in plants, and some have potential medicinal value (Kuhle, 1973). Bond polarization in sulfenamides derivatives, resulting from the difference in electronegativity between sulfur and nitrogen, activates the S—N bond for attack by both nucleophiles and electrophiles and appears to be the factor primarily responsible for the chemistry of these compounds.



(I)

A view of (I) with the atom-numbering scheme is given in Fig. 1 and selected geometric parameters are given in Table 2. The C1—C*n* bonds [n = 7, 8, 14; mean 1.533 (3) Å] are all well above the upper quartile value of 1.521 Å for C1(sp^3)—C(aryl) bonds (Allen *et al.*, 1987), but still longer than the mean value of 1.513 (3) Å for C20(sp^3)—C(aryl) and C21(sp^3)—C(aryl) bonds. The mean of the 30 phenyl C—C distances is 1.379 (4) Å. The S—C bond distance of 1.894 (2) Å is comparable to those found in the literature

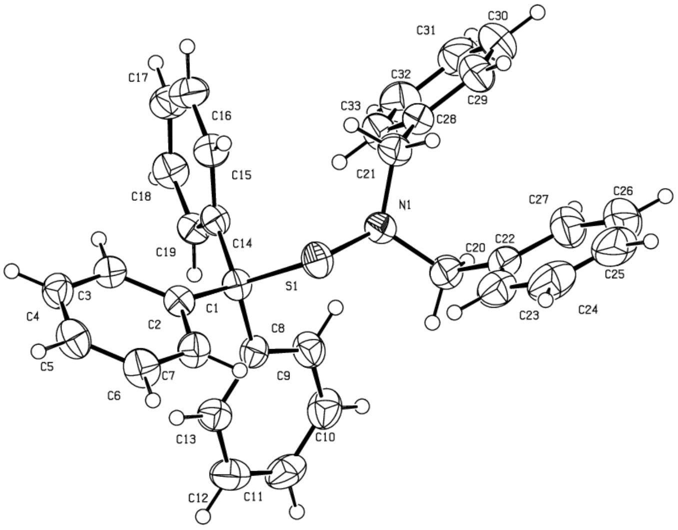


Figure 1
View of (I) (50% probability displacement ellipsoids).

(Glidewell & Ferguson, 1994, and references therein). The S—N bond length of 1.685 (2) Å is shorter than similar bonds in the above related compounds [1.695 (4)-1.713 (4) Å], but is consistent with previously observed S—N single bonds (1.63–1.68 Å) exhibiting π character (Brito *et al.*, 2000, 2002, 2003; Iwasaki & Masuko, 1986). The N—C distances are typical of such bonds in cases where the three covalent N-atom links are nearly planar rather than pyramidal, as found here. The distance of atom N1 from the plane C20—S1—C21 is 0.322 (2) Å (Fig. 1) and the sum of the three bond angles with their vertex at N1 is 347.26°.

The structure of (I) contains two C—H···N and C—H···S intramolecular bonds. The intramolecular hydrogen bonds are formed between the C-bound H9 atom and the N atom of the amine group and between the C-bound H15 atom and the S atom. The dihedral angles between phenyl rings (Fig. 1) C2–C7/C8–C13, C2–C7/C14–C19, C8–C13/C14–C19 and C22–C27/C28–C33 are 70.15 (7), 77.10 (8), 66.39 (6) and 13.62 (8)°, respectively.

Experimental

The title compound was synthesized from sulfenyl chloride triphenylmethane and *N*,*N*-dibenzylamine (molar ratio 1:1.5) in *n*-hexane over a period of 2 h at 261 K. The product was separated by extraction into diethyl ether from the dibenzylammonium hydrochloride by-product. After removing the solvent *in vacuo*, the residue was recrystallized from diethyl ether at room temperature.

Crystal data

$C_{33}H_{29}NS$
M_r = 471.63
Triclinic, $P\bar{1}$
a = 10.009 (2) Å
b = 10.519 (3) Å
c = 13.311 (1) Å
α = 106.822 (13)°
β = 99.037 (13)°
γ = 100.245 (19)°
V = 1287.3 (5) $Å^3$

Z = 2
D_x = 1.217 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 12.0–18.4°
μ = 1.26 mm^{-1}
T = 293 (2) K
Prism, colourless
0.24 × 0.24 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.716, T_{max} = 0.827
5442 measured reflections
5207 independent reflections
3437 reflections with $I > 2\sigma(I)$

R_{int} = 0.024
θ_{max} = 73.9°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 13$
$l = -16 \rightarrow 0$
3 standard reflections every 120 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.122$
S = 1.07
5207 reflections
345 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.217P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.20 e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.6852 (18)	C1—C2	1.533 (3)
S1—C1	1.8941 (19)	C1—C8	1.537 (3)
N1—C21	1.469 (3)	C20—C22	1.513 (3)
N1—C20	1.480 (3)	C21—C28	1.514 (3)
C1—C14	1.529 (3)		
N1—S1—C1	105.61 (8)	C14—C1—C8	112.01 (14)
C21—N1—C20	113.52 (16)	C2—C1—C8	111.58 (15)
C21—N1—S1	116.97 (14)	C14—C1—S1	109.78 (12)
C20—N1—S1	116.77 (14)	C2—C1—S1	101.39 (12)
C14—C1—C2	111.42 (15)	C8—C1—S1	110.15 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···N1	0.93	2.57	3.185 (3)	124
C15—H15···S1	0.93	2.81	3.148 (3)	103

H atoms attached to C atoms were placed in calculated positions, with C—H distances of 0.93 or 0.97 Å, and were included in the final cycles of refinement as riding, with U_{iso} values equal to 1.2U_{eq} of the carrier atoms. Atoms C25 and C26 show a large thermal motion, as indicated by their elongated displacement ellipsoids (Fig. 1). Split peaks for these atoms were not observed and consequently a disorder model was not used.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

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