Crystal structure of 2-*S*-benzyl-5-phenyl-1-(1-thia-3,4-diazacyclopenta-2,4-diene), $C_{15}H_{12}N_2S_2$

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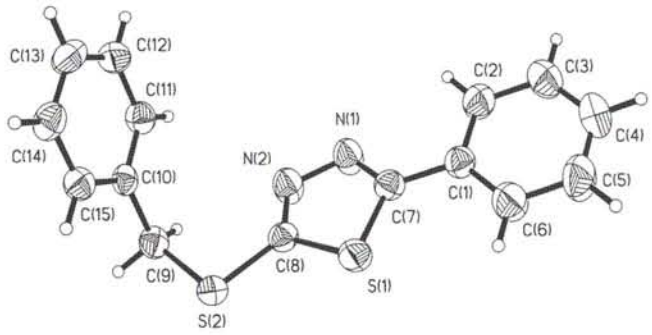

Abstract

$C_{15}H_{12}N_2S_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 5.6723(1)$ Å, $b = 30.5425(6)$ Å, $c = 7.9336(2)$ Å, $\beta = 100.782(1)°$, $V = 1350.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.138$, $T = 293$ K.

Source of material

S-benzyldithiocarbazate (SBDTC) was prepared as previously described [1]. Clear solutions of SBDTC (3.96 g, 0.02 mol) and benzoyl chloride (2.81 g, 0.02 mol) in hot absolute ethanol (50 ml) were mixed and heated for 15 minutes. The reaction mixture was then concentrated, cooled to room temperature and allowed to stand overnight. The white solid formed was filtered, washed with ethanol and then recrystallized from ethanol. The recrystallized product, which formed monoclinic crystals, was dried in vacuo (yield, 4.55 g, 80%, mp = 381 K).

The IR absorption at 1600 cm^{-1} shows the presence of the C=N Schiff base functional group, however, ν(C=S) was not observed. This indicated that the product was a cyclized compound rather than the expected linear condensation product. CHN analysis (C 63.25, H 4.45, N 10.01) is in agreement with values calculated on the basis of crystal structure investigation (C 63.34, H 4.22, N 9.85) for 2-*S*-benzyl-5-phenyl-1-(1-thia-3,4-diaza-cyclopenta-2,4-diene).

Experimental details

The data collection covered over a hemisphere of reciprocal space by acombination of three sets of exposures; each set had a different φ angle (0.88° and 180° for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was –35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating fifty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

Table 1. Data collection and handling.

Crystal:	colourless slab, size 0.24 × 0.38 × 0.46 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.80 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω scans
$2\theta_{max}$:	56.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9506, 3296
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2551
$N(param)_{refined}$:	172
Programs:	PARST [2], SADABS [3], SHELXTL [4], PLATON [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	4*e*	–0.3642	–0.0105	0.6270	0.067
H(3A)	4*e*	–0.3171	–0.0841	0.6889	0.078
H(4A)	4*e*	0.0449	–0.1109	0.8288	0.075
H(5A)	4*e*	0.3574	–0.0637	0.9183	0.089
H(6A)	4*e*	0.3161	0.0103	0.8573	0.076
H(9A)	4*e*	–0.3410	0.2024	0.5696	0.049
H(9B)	4*e*	–0.1913	0.2425	0.5262	0.049
H(11A)	4*e*	–0.5798	0.1673	0.3356	0.049
H(12A)	4*e*	–0.6730	0.1495	0.0471	0.057
H(13A)	4*e*	–0.4223	0.1705	–0.1355	0.058
H(14A)	4*e*	–0.0840	0.2118	–0.0330	0.056
H(15B)	4*e*	0.0118	0.2295	0.2553	0.050

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*e*	0.17724(9)	0.09121(2)	0.71770(8)	0.0339(3)	0.0432(3)	0.0610(4)	–0.0002(2)	–0.0023(2)	0.0003(2)
S(2)	4*e*	0.06464(9)	0.18510(2)	0.62661(6)	0.0483(3)	0.0418(3)	0.0381(3)	–0.0074(2)	–0.0005(2)	0.0011(2)
N(1)	4*e*	–0.2674(3)	0.07270(6)	0.6361(3)	0.0364(9)	0.0403(9)	0.073(1)	0.0011(7)	0.0043(8)	0.0094(8)
N(2)	4*e*	–0.2516(3)	0.11732(6)	0.6065(3)	0.0367(9)	0.0421(9)	0.069(1)	0.0027(7)	0.0060(8)	0.0086(8)
C(1)	4*e*	–0.0292(4)	0.00787(6)	0.7351(2)	0.041(1)	0.039(1)	0.041(1)	0.0033(8)	0.0070(8)	0.0000(8)
C(2)	4*e*	–0.2174(4)	–0.02089(8)	0.6855(3)	0.049(1)	0.051(1)	0.063(1)	–0.004(1)	–0.006(1)	0.010(1)
C(3)	4*e*	–0.1888(5)	–0.06512(8)	0.7222(4)	0.068(2)	0.046(1)	0.077(2)	–0.012(1)	0.004(1)	0.006(1)
C(4)	4*e*	0.0259(5)	–0.08110(8)	0.8067(3)	0.073(2)	0.040(1)	0.076(2)	0.007(1)	0.020(1)	0.008(1)
C(5)	4*e*	0.2122(5)	–0.05300(8)	0.8584(4)	0.058(2)	0.054(1)	0.105(2)	0.017(1)	–0.002(2)	0.012(1)
C(6)	4*e*	0.1872(4)	–0.00852(8)	0.8226(4)	0.046(1)	0.048(1)	0.090(2)	0.003(1)	–0.005(1)	0.003(1)
C(7)	4*e*	–0.0608(3)	0.05478(6)	0.6950(2)	0.0338(9)	0.0391(9)	0.040(1)	0.0009(7)	0.0056(8)	–0.0008(8)
C(8)	4*e*	–0.0311(3)	0.13101(6)	0.6435(2)	0.0384(9)	0.0388(9)	0.0329(9)	0.0015(7)	0.0056(7)	–0.0016(7)
C(9)	4*e*	–0.2092(4)	0.21105(6)	0.5146(2)	0.051(1)	0.0346(9)	0.039(1)	0.0052(8)	0.0113(9)	–0.0022(7)
C(10)	4*e*	–0.2727(3)	0.19982(5)	0.3268(2)	0.0380(9)	0.0276(8)	0.0360(9)	0.0060(7)	0.0078(7)	0.0022(7)
C(11)	4*e*	–0.4785(4)	0.17609(6)	0.2623(3)	0.037(1)	0.042(1)	0.045(1)	–0.0017(8)	0.0109(8)	0.0035(8)
C(12)	4*e*	–0.5346(4)	0.16536(7)	0.0891(3)	0.043(1)	0.047(1)	0.049(1)	–0.0045(9)	–0.0027(9)	0.0005(9)
C(13)	4*e*	–0.3860(4)	0.17820(7)	–0.0201(3)	0.053(1)	0.055(1)	0.034(1)	0.008(1)	–0.0004(9)	0.0000(8)
C(14)	4*e*	–0.1825(4)	0.20260(7)	0.0415(3)	0.047(1)	0.057(1)	0.039(1)	0.0002(9)	0.0122(9)	0.0076(9)
C(15)	4*e*	–0.1256(4)	0.21326(6)	0.2143(3)	0.040(1)	0.042(1)	0.043(1)	–0.0043(8)	0.0076(8)	0.0033(8)

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