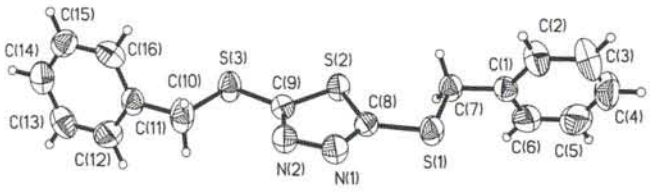
Crystal structure of 2,5-di-*S*-benzyl(1-thia-3,4-diazacyclopenta-2,5-diene), $C_{16}H_{14}N_2S_3$

M. T. H. Tarafder*[I], N. Saravanan[I], K. A. Crouse[I], B. M. Yamin[II], S. S. S. Raj[III], I. A. Razak[III] and H.-K. Fun[III]

[I] Universiti Putra Malaysia, Department of Chemistry, 43400 Serdang, Selangor, Malaysia
[II] Universiti Kebangsaan Malaysia, School of Chemical Sciences and Food Technology, 43600 Bangi, Selangor, Malaysia
[III] Universiti Sains Malaysia, School of Physics, X-ray Crystallography Unit, 11800 USM, Penang, Malaysia

Received December 21, 1999, CCDC-No. 1267/375



Abstract

$C_{16}H_{14}N_2S_3$, orthorhombic, *Pbca* (No. 61), $a = 8.7643(1)$ Å, $b = 10.3305(2)$ Å, $c = 36.0581(1)$ Å, $V = 3264.7$ Å^3, $Z = 8$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.125$, $T = 293$ K.

Source of material

S-benzyldithiocarbazate (SBDTC) (0.01 mole) which was prepared as previously described [1] was dissolved in absolute ethanol (35 ml). To this, ethyl benzoyl acetate (0.01 mole) was added and the mixture was refluxed for 24 hours. The crude product was then recrystallized three times from absolute ethanol. Spectral evidence and elemental analyses showed this was the expected product, *S*-benzyl-β-*N*-(phenyl-methylaceto)-methylenedithiocarbazate. A small quantity of this product was redissolved in absolute ethanol to grow single crystals. The clear crystals that appeared after two months were collected and dried in vacuo over P_2O_5 (mp = 363 K). The diagnostic ν(C=O), ν(C=S) and ν(N—H) bands present the IR spectrum of the initial product disappeared in the IR spectrum of the crystals indicating that all these bonds were involved in the formation of the final product, 2,5-di-*S*-benzyl-(1-thia-3,4-diazacyclopenta-2,4-diene). Its IR spectrum also shows a weak band at 1602 cm^{-1} indicating the presence of C=N. The results of elemental analysis (%C = 57.93, %H = 4.14, %N = 8.76) are in good agreement with the values calculated from crystal structure investigation (58.18, 4.24, 8.48). For data collection see [2].

Table 1. Data collection and handling.

Crystal:	colourless slab, size 0.30 × 0.38 × 0.46 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.48 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω scans
$2\theta_{max}$:	56.54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21531, 4029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2677
$N(param)_{refined}$:	190
Programs:	PARST [2], SADABS [4], SHELXTL [5], PLATON [6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	8*c*	–0.1758	0.9718	0.2982	0.083
H(3A)	8*c*	–0.2495	0.8867	0.2420	0.113
H(4A)	8*c*	–0.1640	0.6864	0.2242	0.117
H(5A)	8*c*	–0.0115	0.5671	0.2631	0.114
H(6A)	8*c*	0.0599	0.6512	0.3202	0.084
H(7A)	8*c*	0.0258	0.8061	0.3684	0.059
H(7B)	8*c*	–0.0839	0.9247	0.3623	0.059
H(10A)	8*c*	0.4424	1.1740	0.5048	0.084
H(10B)	8*c*	0.3202	1.2839	0.4997	0.084
H(12A)	8*c*	0.5251	1.1174	0.5659	0.076
H(13A)	8*c*	0.5313	1.1613	0.6284	0.086
H(14A)	8*c*	0.3545	1.2974	0.6546	0.075
H(15A)	8*c*	0.1689	1.3894	0.6180	0.072
H(16A)	8*c*	0.1602	1.3455	0.5554	0.066

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)	8*c*	0.16327(7)	0.98015(7)	0.34428(2)	0.0541(4)	0.0740(5)	0.0366(3)	–0.0167(3)	0.0041(3)	0.0023(3)
S(2)	8*c*	0.09961(7)	0.97803(6)	0.42795(2)	0.0492(3)	0.0545(4)	0.0379(3)	–0.0161(3)	0.0040(2)	0.0015(2)
S(3)	8*c*	0.19956(8)	1.08320(7)	0.50183(2)	0.0640(4)	0.0601(4)	0.0404(3)	–0.0197(3)	0.0026(3)	–0.0016(3)
N(1)	8*c*	0.3103(2)	1.1106(2)	0.39693(6)	0.054(1)	0.065(1)	0.044(1)	–0.019(1)	0.0064(9)	0.0033(9)
N(2)	8*c*	0.3226(2)	1.1368(2)	0.43466(5)	0.053(1)	0.056(1)	0.044(1)	–0.0136(9)	0.0026(9)	–0.0001(9)

* Correspondence author (e-mail: tofazzal@fsas.upm.edu.my)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*c*	–0.0484(3)	0.8207(2)	0.31472(6)	0.042(1)	0.052(1)	0.046(1)	–0.009(1)	–0.000(1)	0.001(1)
C(2)	8*c*	–0.1420(3)	0.8900(3)	0.29124(8)	0.085(2)	0.062(2)	0.059(2)	0.000(2)	–0.021(2)	0.001(1)
C(3)	8*c*	–0.1859(4)	0.8395(4)	0.2576(1)	0.108(3)	0.104(3)	0.070(2)	–0.014(2)	–0.042(2)	0.001(2)
C(4)	8*c*	–0.1359(4)	0.7199(4)	0.2472(1)	0.096(3)	0.132(3)	0.065(2)	–0.027(2)	–0.008(2)	–0.037(2)
C(5)	8*c*	–0.0446(4)	0.6490(4)	0.2702(1)	0.078(2)	0.098(3)	0.109(3)	0.000(2)	0.003(2)	–0.048(2)
C(6)	8*c*	–0.0013(3)	0.6997(3)	0.30433(8)	0.060(2)	0.073(2)	0.076(2)	0.007(1)	–0.003(1)	–0.014(2)
C(7)	8*c*	–0.0006(3)	0.8756(2)	0.35150(6)	0.050(1)	0.057(2)	0.041(1)	–0.009(1)	0.001(1)	0.001(1)
C(8)	8*c*	0.2007(2)	1.0303(2)	0.38948(6)	0.041(1)	0.046(1)	0.038(1)	–0.005(1)	0.0046(9)	0.0055(9)
C(9)	8*c*	0.2202(2)	1.0752(2)	0.45375(6)	0.043(1)	0.038(1)	0.043(1)	–0.0016(9)	–0.0014(9)	0.0017(9)
C(10)	8*c*	0.3428(3)	1.2041(3)	0.51274(8)	0.079(2)	0.078(2)	0.053(2)	–0.035(2)	0.009(1)	–0.013(1)
C(11)	8*c*	0.3435(3)	1.2281(2)	0.55378(7)	0.049(1)	0.051(1)	0.047(1)	–0.016(1)	0.002(1)	–0.005(1)
C(12)	8*c*	0.4529(3)	1.1729(3)	0.57618(8)	0.059(2)	0.057(2)	0.076(2)	0.008(1)	–0.001(1)	–0.010(1)
C(13)	8*c*	0.4565(4)	1.1990(3)	0.61364(8)	0.069(2)	0.074(2)	0.072(2)	–0.002(2)	–0.024(2)	0.005(2)
C(14)	8*c*	0.3513(3)	1.2797(3)	0.62928(8)	0.073(2)	0.067(2)	0.047(2)	–0.022(1)	0.000(1)	–0.004(1)
C(15)	8*c*	0.2410(3)	1.3344(3)	0.60751(8)	0.055(2)	0.058(2)	0.066(2)	–0.008(1)	0.015(1)	–0.007(1)
C(16)	8*c*	0.2362(3)	1.3085(2)	0.56994(7)	0.045(1)	0.059(2)	0.061(2)	–0.006(1)	–0.005(1)	0.008(1)

Acknowledgments. The authors would like to thank the Malaysian Government, Universiti Putra Malaysia, Universiti Kebangsaan Malaysia and Universiti Sains Malaysia for research grant R&D Nos.IRPA 09-04-0083,03-02-02-005 and 305/pfizik/622004 respectively. S. S .S. R. thanks the Universiti Sains Malaysia for a Visiting Post-Doctoral Fellowship.

References

1. Ali, M. A.; Tarafder, M. T. H.: Metal complexes of sulphur and nitrogen containing ligands: Complexes of *S*-benzyldithiocarbazate and a Schiff base formed by its condensation with pyridine-2-carboxyldehyde. J. Inorg. Nucl. Chem. **39** (1977) 1785-1781.
2. Tarafder, M. T. H.; Azahari, K,; Crouce, K. A.; Yamin, B. M.; Raj, S. S. S.; Razak, I. A.; Fun, H.-K.: Crystal structure of 2-S-benzyl-5-phenyl-1-(1-thia-3,4-diazacyclopenta-2,4-diene), $C_{15}H_{12}N_2S_2$. Z. Kristallogr. NCS **215** (2000) 487-488.
3. Nardelli, M.:PARST95 - an update to PARST: A system of Fortran routines for calculating molecular structure parameters from the results of the crystal structure analysis. J. Appl. Crystallogr. **28** (1995) 659.
4. Sheldrick, G. M.: SADABS. Program for Empirical Absorption Correction of Area Detector Data, University of Göttingen, Germany 1996.
5. Sheldrick, G. M.: SHELXTL V5.1 Software Reference Manual, Bruker AXS, Inc., Madison, Wisconsin, USA 1997.
6. Spek, A. L.: PLATON, an integrated tool for the analysis of the results of a single crystal structure determination. Acta Crystallogr. **46** (1996) C-34.