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X-ray crystal structural analysis of dicyclohexylthiocarbamide

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ABSTRACT

The title compound dicyclohexylthiocarbamide has been determined by single crystal X-ray crystal diffraction analysis. The crystals are monoclinic, space group P2(1)/c with a = *12.5908(9)*, b = *11.2158(9)*, c = *10.4255(8)* Å, $\alpha = 90$, $\beta = 110.7360(10)$, $\gamma = 90°$, $V = 1376.88(18)$ Å^3, $Z = 4$, $F(000) = 528$, $D_c = 1.160$ g/cm^3, $\mu = 0.214$ mm^{-1}, *the final* $R = 0.0381$ *and* $wR = 0.1030$. A total of 6836 reflections were collected, of which 2423 were independent (R_{int} = 0.0154). In the crystal packing diagram, intermolecular N–H···S hydrogen bonds stabilize the solid state of the title compound.

KEYWORDS

Cyclohexyl; thiocarbamide; crystal structure

Introduction

As a continuation of our studies regarding simple organic compounds [1–4], we obtained the single crystals of the title compound which is available for X-ray diffraction analysis. In this paper, we wish to report the X-ray crystal structural analysis of the title compound dicyclohexylthiocarbamide.

Experimental

Crystal structure determination

The crystal of the title compound with dimensions of 0.24 × 0.23 × 0.12 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite-monochromated MoKα radiation ($\lambda = 0.71073$Å) by using a phi and scan modes at 296(2) K in the range of $1.73° \leq \theta \leq 25.00°$. The crystal belongs to monoclinic system with space group P2(1)/*c* and crystal parameters of $a = 12.5908(9)$ Å, $b = 11.2158(9)$ Å, $c = 10.4255(8)$ Å, $\alpha = 90°$, $\beta = 110.7360(10)°$, $\gamma = 90°$, $V = 1376.88(18)$ A^3, $D_c = 1.160$ g/cm^3, The absorption coefficient $\mu = 0.214$ mm^{-1}, and $Z = 4$. A summary of crystal data is presented in Table 1.

The structure was solved by direct methods with SHELXS-97 [5] and refined by the full-matrix least-squares method on F^2 data using SHELXL-97 [6]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference Fourier map and were refined with the restraint Uiso(H) = 1.2Ueq(N). Other H atoms were positioned geometrically and refined using a riding model, with d(C–H) = 0.93–0.97 Å

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Table 1. Crystal data and structure refinement.

Empirical formula	$C_{13}H_{24}N_2S$
Formula weight	240.40
Crystal system	Monoclinic
Unit cell dimensions	
a (Å)	12.5908(9)
b (Å)	11.2158(9)
c (Å)	10.4255(8)
Unit cell angles (°)	
α	90
β	110.7360(10)
γ	90
Volume (Å^3)	1376.88(18)
Z	4
Temperature (K)	296(2)
Space group	P2(1)/*c*
Wavelength (Å)	0.71073
Calculated density (g/cm^3)	1.160
Absorption coefficient (mm^{-1})	0.214
F(000)	528
Crystal size (mm)	0.24 × 0.23 × 0.12
Theta range for data collection (°)	1.73–25.00
Reflections collected	6836
Independent reflections	2423 [$R_{(int)} = 0.0154$]
Final *R* indices [I>2σ(*I*)]	$R_1 = 0.0381$, w$R_2 = 0.1030$

Table 2. Selected bond lengths (Å) and bond angles (°).

Bond lengths			
S(1)–C(7)	1.7021(18)	N(1)–C(7)	1.336(2)
N(1)–C(1)	1.460(2)	N(2)–C(8)	1.290(11)
N(2)–C(7)	1.338(2)	C(1)–C(6)	1.516(2)
C(1)–C(2)	1.522(3)	C(2)–C(3)	1.515(3)
C(3)–C(4)	1.514(3)	C(4)–C(5)	1.513(3)
C(5)–C(6)	1.523(3)	C(8)–C(9)	1.445(8)
C(8)–C(13)	1.448(8)	C(9)–C(10)	1.519(7)
C(10)–C(11)	1.475(9)	C(11)–C(12)	1.498(9)
Bond angles			
C(7)–N(1)–C(1)	127.13(15)	C(8)–N(2)–C(7)	119.4(5)
N(1)–C(1)–C(6)	108.97(14)	N(1)–C(1)–C(2)	111.44(15)
C(6)–C(1)–C(2)	111.08(15)	C(3)–C(2)–C(1)	110.88(18)
C(4)–C(3)–C(2)	111.26(18)	C(5)–C(4)–C(3)	110.55(18)
C(4)–C(5)–C(6)	111.82(18)	C(1)–C(6)–C(5)	111.25(16)
N(1)–C(7)–N(2)	117.26(16)	N(1)–C(7)–S(1)	119.55(13)
N(2)–C(7)–S(1)	123.18(14)	N(2)–C(8)–C(9)	121.3(8)
N(2)–C(8)–C(13)	124.7(8)	C(9)–C(8)–C(13)	106.9(7)

and Uiso(H) = 1.2Ueq(C) or 1.5Ueq(Cmethyl). The final full-matrix least-squares refinement gave $R = 0.0381$ and $wR = 0.1030$.

Results and discussion

The title compound dicyclohexylthiocarbamide was confirmed by single crystal X-ray diffraction analysis. The selected bond lengths and bond angles are listed in Table 2. The structure was solved by direct methods. Anisotropic displacement parameters were applied to all non-hydrogen atoms in full-matrix least-square refinements based on F^2. The hydrogen atoms were set in calculated positions with a common fixed isotropic thermal parameter.

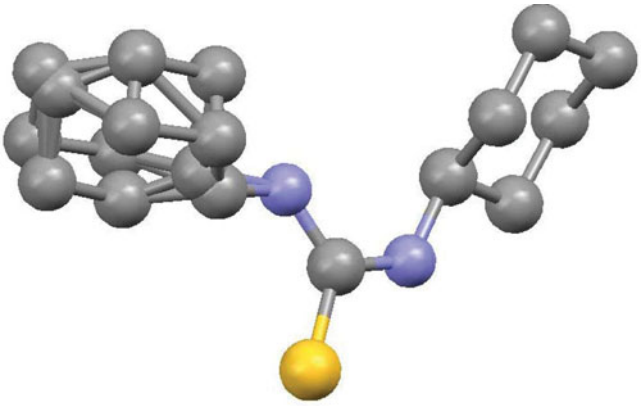

Figure 1. Molecular structure of the title compound.

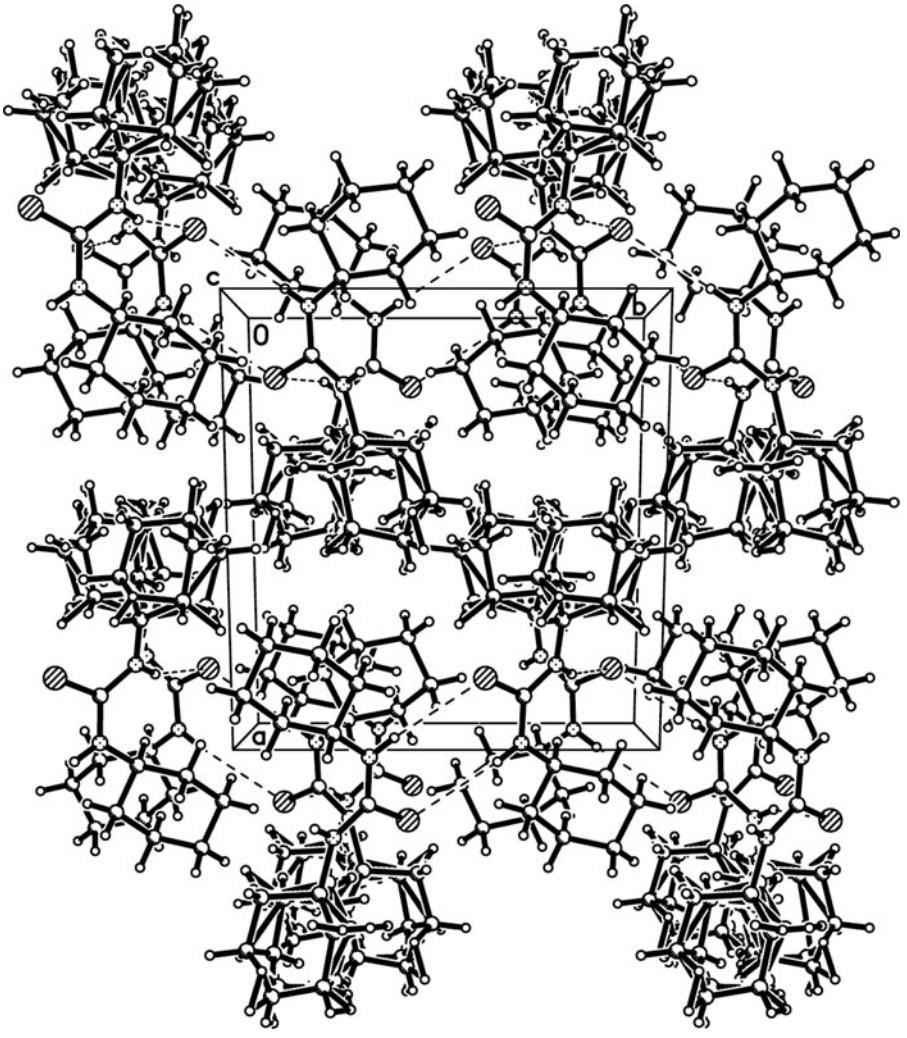

Figure 2. The crystal packing view of the title compound.

The molecular structure and packing views of the title compound are displayed in Figs. 1 and 2, respectively. The title compound crystallizes in monoclinic space group P2(1)/*c* with four molecules in the unit cell and one molecule in the asymmetric unit. As shown in Fig. 1, the molecular structure of the title compound contains two cyclohexyl groups linking together through thiocarbamide group. One cyclohexyl group is in disordered state whereas the other cyclohexyl group is in ordered state. The orientation of the cyclohexyl groups occupies the chair conformation. The bond distances [S(1)–C(7) = 1.7021(18) Å, N(1)–C(7) = 1.336(2) Å, N(1)–C(1) = 1.460(2) Å, N(2)–C(8) = 1.290(11) Å, N(2)–C(7) = 1.338(2) Å, C(1)–C(6) = 1.516(2) Å, C(1)–C(2) = 1.522(3) Å, C(2)–C(3) = 1.515(3) Å, C(3)–C(4) = 1.514(3) Å, C(4)–C(5) = 1.513(3) Å and C(5)–C(6) = 1.523(3) Å] and bond angles [C(7)–N(1)–C(1) = 127.13(15)°, C(8)–N(2)–C(7) = 119.4(5)°, N(1)–C(1)–C(6) = 108.97(14)°, N(1)–C(1)–C(2) = 111.44(15)°, C(6)–C(1)–C(2) = 111.08(15)°, C(3)–C(2)–C(1) = 110.88(18)°, C(4)–C(3)–C(2) = 111.26(18)° and C(5)–C(4)–C(3) = 110.55(18)°] are similar to other compound [7–29].

As shown in Fig. 2, the crystal packing diagram of the title compound displays that intermolecular N–H···S hydrogen bonds existing to stabilize the solid state.

Conclusions

In summary, the title compound dicyclohexylthiocarbamide has been characterized by X-ray diffraction analysis.

Funding

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Supplementary Data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 1036538 for the title compound. Copies of the data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk.

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