Crystal Structure of 3-Pentanone Semicarbazone

Mehmet AKKURT,* Sema ÖZTÜRK,*† and Semra İDE**

**Department of Physics, Faculty of Arts and Sciences, Erciyes University, Kayseri-38039, Turkey*
***Department of Physics Engineering, Hacettepe University, Beytepe, Ankara-06532, Turkey*

(Received October 8, 1999; Accepted March 1, 2000)

A great number of studies have been devoted to the search for derivatives of semicarbazides and thiosemicarbadizes. These materials have been used as drugs whose action is attributed to their ability to form metal complexes.[1] Several ligands and metal complexes of semicarbazide and thiosemicarbazide have been the subject of chemical and structural studies.[2] Especially, semicarbazones and thiosemicarbazones as *anti*-cancer and *anti*-viral agents have been known for many years.[3,4] In order to elucidate the stereochemistry of the title compound, we carried out the crystal structure determination.

A summary of the key crystallographic information is given in Table 1. The atomic coordinates and equivalent isotropic displacement parameters with estimated standard deviations for atoms except H are listed in Table 2, and the bond lengths and bond angles are given in Table 3.

The conformation of the title compound as found in the crystal structure is shown in Fig. 2. The structure showed *E*-configuration. The plane formed by N1,C1,O1,N2,N3 atoms is almostly planar; the maximum deviation from this plane is 0.02(1)Å for N2. The C2=N3, C1–N2 and C1–N1 bond distances of the title compound are comparable with C6=N2 [1.27(3)Å], C7–N3 [1.35(3)Å] and C7–N4 [1.33(3)Å] bond

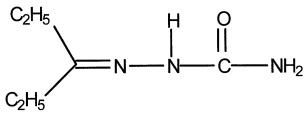

Fig. 1 Chemical structure.

Table 1 Crystal and experimental data

Formula: $C_6H_{13}N_3O$	
Formula weight = 143.19	
Crystal system: triclinic	
Space group: $P\bar{1}$ (No. 2); Z = 2	
a = 6.361(1)Å	α = 66.70(1)°
b = 7.319(1)Å	β = 84.33(1)°
c = 9.569(1)Å	γ = 83.40(1)°
V = 405.7(1)Å^3	
D_x = 1.172 g/cm^3	
μ(Mo K$_\alpha$) = 0.83 cm^{-1}	
T = 295 K	
F(0 0 0) = 156	
Crystal size = 0.40 × 0.30 × 0.25 mm	
Radiation: Mo K$_\alpha$	
R = 0.053	
Rw = 0.124	
No. of unique data measured = 578	
No. of observed data with [$I \geq 2\sigma(I)$] = 376	
No. of parameters = 89	
Goodness-of-fit = 1.991	
$(\Delta/\sigma)_{max}$ = 0.00	
$(\Delta\rho)_{max}$ = 0.19 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.18 eÅ^{-3}	
Measurements: Enraf Nonius CAD-4 diffractometer	
Program system: CAD-4 EXPRESS Software	
Structure determination: SIR92	
Treatment of hydrogen atoms: geometric calculation	
Refinement: full matrix least-squares SHELXL93	

Table 2 Final atomic coordinates and equivalent isotropic thermal displacement parameters for non-hydrogen atoms

Atom	x	y	z	U_{eq}
O1	0.484(2)	0.277(1)	0.974(1)	0.093(6)
N1	0.743(2)	0.030(2)	1.064(2)	0.102(8)
N2	0.742(2)	0.319(2)	1.103(2)	0.082(6)
N3	0.922(2)	0.237(2)	1.181(2)	0.079(6)
C1	0.648(3)	0.211(2)	1.044(2)	0.076(9)
C2	1.011(3)	0.338(2)	1.237(2)	0.075(8)
C3	1.200(3)	0.241(2)	1.325(2)	0.091(9)
C4	1.259(2)	0.026(2)	1.349(2)	0.12(1)
C5	0.930(2)	0.541(2)	1.230(2)	0.09(1)
C6	0.757(3)	0.531(2)	1.353(2)	0.11(1)

$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

Table 3 Bond lengths (Å) and bond and torsion angles (°)

O1 - C1	1.24(2)	C2 - C3	1.49(2)
N1 - C1	1.34(2)	C2 - C5	1.49(2)
N2 - C1	1.35(2)	C3 - C4	1.50(2)
N2 - N3	1.38(2)	C5 - C6	1.52(2)
N3 - C2	1.27(2)		
N3 - N2 - C1	119(1)	N3 - C2 - C3	117(2)
N2 - N3 - C2	119(1)	C3 - C2 - C5	118(1)
O1 - C1 - N1	122(2)	N3 - C2 - C5	125(2)
N1 - C1 - N2	116(2)	C2 - C3 - C4	116(1)
O1 - C1 - N2	122(2)	C2 - C5 - C6	112(1)
C1 - N2 - N3 - C2	180(1)	N3 - C2 - C3 - C4	-4(1)
N3 - N2 - C1 - O1	179(1)	C5 - C2 - C3 - C4	171(1)
N3 - N2 - C1 - N1	-1(1)	N3 - C2 - C5 - C6	81(1)
N2 - N3 - C2 - C3	178(1)	C3 - C2 - C5 - C6	-94(1)
N2 - N3 - C2 - C5	3(1)		

† To whom correspondence should be addressed.
E-mail: ozturk@erciyes.edu.tr

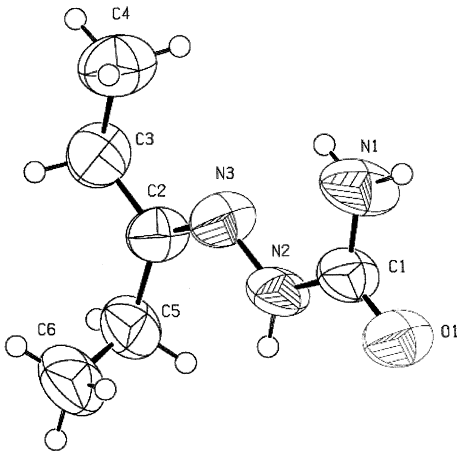

Fig. 2 Molecular structure of 3-pentanone semicarbazone. Thermal ellipsoids are drawn at the 50% probability level.

distances of 4-formylpyridine thiosemicarbazone.[5] C2=N3 [1.27(2)Å] is a double bond and the distances C1–N2 and C1–N1 possess partial double-bond character. The N2–N3 distance is close to the N–N distance of 1.376(6)Å in 2-keto-3-ethoxybutyrlaldehyde thiosemicarbazone[6] and may be due to an extensively delocalized group attached to the N atom.

Hydrogen bond geometries are shown in Table 4. The conformation of the semicarbazone fragment orients the imino N atom in such a way that an intramolecular hydrogen bond is formed between N3 and the NH_2 group, resulting a five-membered ring. Two N–H··O and one C–H··O intermolecular hydrogen bonds stabilize the crystal structure.

Table 4 Possible hydrogen bond lengths (Å) and angles (°)

D–H (Å)		D···A (Å)		H···A (Å)		D–H···A (°)
N1–H1B	0.86(2)	N1...N3	2.62(2)	H1B...N3	2.26(2)	105(2)
N1–H1A	0.86(2)	N1...O1	2.96(2)	H1A...O1^{i}	2.14(2)	158(2)
N2–H2	0.86(2)	N2...O1	2.98(2)	H2...O1^{ii}	2.14(2)	167(2)
C5–H5A	0.97(2)	C5...O1	3.27(2)	H5A...O1^{ii}	2.50(2)	136(1)

Equivalent positions: [*i* = 1–*x*,–*y*,–*z*; *ii* = 1–*x*,1–*y*,–*z*].

References

1. H. G. Petering and G. J. Van Giessen, *Biochem*. *Copper Proc*. *Symp*., **1965**, New York, Harriman, 197.
2. M. Sorina-Garcia, J. Valdes-Martinez, R. A. Toscano, J. Gomez-Lara, and M. Villalobos-Penaloza, *Acta Crystallogr*., **1986**, *C42*, 623.
3. D. X. West, A. E. Liberta, S. B. Padhye, R. C. Chikate, P. B. Sonawane, A. S. Kumbhar, and R. G. Yerande, *Coord*. *Chem*. *Rev*., **1993**, *123*, 49.
4. S. Padhye and G. B. Kauffman, *Coord*. *Chem*. *Rev*., **1985**, *63*, 127.
5. R. Restino and G. J. Palenik, *Acta Crystallogr*., **1970**, *B26*, 1397.
6. E. J. Gabe, M. R. Taylor, J. P. Glusker, J. A Minkin, and A. L. Patterson, *Acta Crystallogr*., **1969**, *B25*, 1620.