

КРАТКИЕ СООБЩЕНИЯ

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CRYSTAL STRUCTURE OF *N'*-PIVALOYL-*N*-PHENYLPIVALOHYDRAZIDEA. Saeed¹, I. Arshad¹, U. Flörke²¹Department of Chemistry, Quaid-I-Azam University, Islamabad, Pakistan²Department of Inorganic and Analytical Chemistry, Universität Paderborn, Paderborn, Germany

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A novel hydrazide compound (C₁₆H₂₄N₂O₂) is synthesized in a single step by the reaction of pivaloyl chloride with phenylhydrazine in a 2:1 molar ratio in dry DCM. The molecular structure is determined by the single crystal X-ray analysis. The compound crystallized in the monoclinic system, space group *P*2₁/*c* with *a* = 15.4638(17) Å, *b* = 9.9481(11) Å, *c* = 10.5276(12) Å, β = 90.194(3)°, *Z* = 4, *V* = 1619.5(3) Å³.

Keywords: *N'*-pivaloyl-*N*-phenylpivalohydrazide, crystal structure.

The diacylhydrazine derivatives are a promising class of chemically and mechanistically novel insect control agents that were serendipitously discovered at Rohm and Haas Company [1]. The diacylhydrazine insecticide family shows efficacy against lepidopteran larvae in the field and laboratory [2]. Tebufenozide and its methoxylated derivative methoxyfenozide presents intrepid as agonist or mimic insect molting hormone 20-hydroxyecdysone by inducing premature, incomplete ecdysis resulting in death of the exposed insects [3–7]. Halofenozide and chromafenozide are novel members of the diacylhydrazine insecticide belonging to the class of non-steroidal ecdysone agonists. Tebufenozide and its analogue methoxyfenozide, halofenozide, and chromafenozide have been used extensively in crops such as apple, grape, and cruciferous vegetables in order to obtain good yields in different countries [8–10]. Thus, it becomes important to determine these insecticides in crop to ensure food safety.

With extensive industrial applications and pharmacological importance, we herein report an efficient one-pot synthesis and the crystal structure of *N'*-pivaloyl-*N*-phenylpivalohydrazide (**1**) obtained by the efficient method from acid chlorides in dichloromethane as a solvent in a short time with high yield.

Experimental. *N'*-pivaloyl-*N*-phenylpivalohydrazide (**1**) was synthesized as follows: to the magnetically stirred solution of phenyl hydrazine (3 mmol) a solution of pivaloyl chloride (6 mmol) in 10 ml of dichloromethane was added dropwise. The mixture solution was heated under reflux for about 1 h. The resulting solution was allowed to cool at room temperature. After cooling, the solution

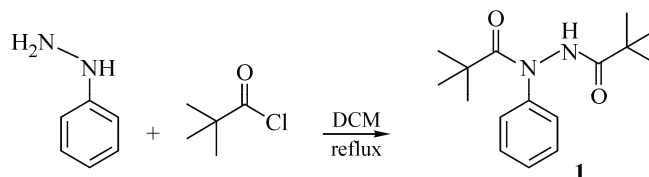
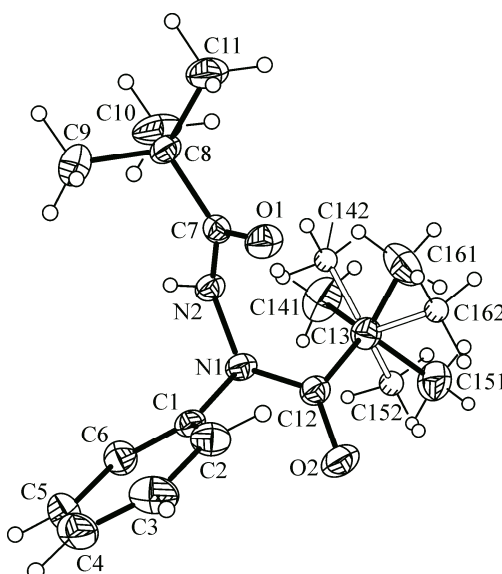
Scheme 1. *N'*-pivaloyl-*N*-phenylpivalohydrazide **1**

Fig. 1. Molecular structure of **1**.

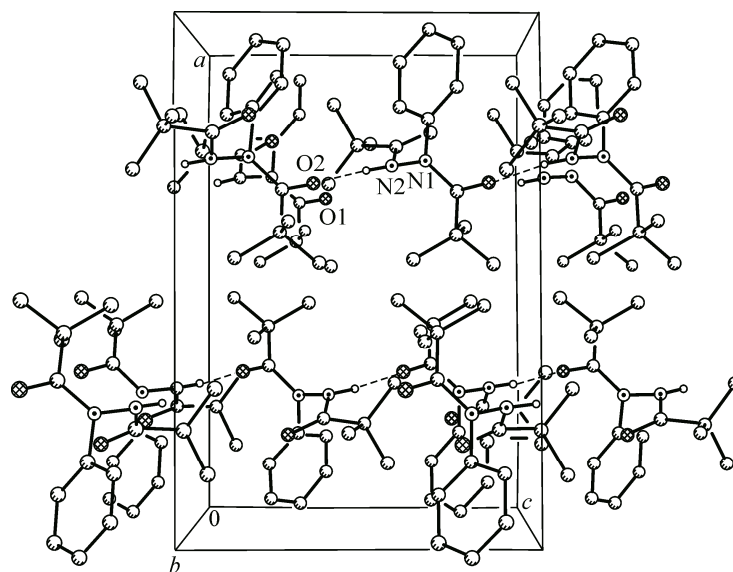
The tBu group at C13 is disordered over two sites with occupation factors of 0.877(4)/0.123(4). Anisotropic displacement ellipsoids are drawn at a 50 % probability level. H atoms are shown as spheres of arbitrary radii



was evaporated in ambient environment. Two days later, white crystals suitable for X-ray analysis were collected, and washed with a small amount of methanol. Yield: 96 %. The chemical diagram of compound **1** is shown in Scheme 1. CIF-file containing complete information on the studied structure was deposited with CCDC, deposition number 951829 and is freely available upon request from the following website: www.ccdc.cam.ac.uk/data_request/cif.

X-ray crystallography. $C_{16}H_{24}N_2O_2$, $M_r = 276.4$, crystal size $0.48 \times 0.21 \times 0.20 \text{ mm}^3$, monoclinic, space group $P2_1/c$, $a = 15.4638(17) \text{ \AA}$, $b = 9.9481(11) \text{ \AA}$, $c = 10.5276(12) \text{ \AA}$, $\beta = 90.194(3)^\circ$, $Z = 4$, $V = 1619.5(3) \text{ \AA}^3$, $\rho_{\text{calc}} = 1.133 \text{ g/cm}^3$, $\mu = 0.075 \text{ mm}^{-1}$, $F(000) = 600$. Data were collected at $130(2) \text{ K}$ on a Bruker AXS SMART APEX CCD diffractometer using $\text{MoK}\alpha$ radiation. 15056 intensities $2.4 \leq \Theta \leq 27.9^\circ$. The structure was solved by direct methods [11], the full-matrix least-squares refinement [11] on F^2 was performed using 198 parameters for 3847 unique intensities ($R_{\text{int}} = 0.035$), all but H atoms and the minor occupied ${}^t\text{Bu}$ site (C142, C152, C162) were refined anisotropically. All H atom positions were clearly derived from difference Fourier maps and refined at idealized positions with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C/N})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$; C—H distances of 0.95—0.98 \AA . H(N) was refined freely. H(C_{methyl}) were allowed to rotate but not to tip. The refinement converged at $R1(I > 2\sigma(I)) = 0.048$, $wR2(\text{all data}) = 0.124$, $\text{GOOF} = 1.02$, $\text{min/max } \Delta F = -0.18/0.34 \text{ e/\AA}^3$. The ${}^t\text{Bu}$ group at C13 is disor-

Fig. 2. Crystal packing of **1** viewed along the b axis with intermolecular hydrogen bond interactions drawn as dotted lines. H atoms not involved are omitted. Only the major orientation of the disordered tBu group is shown



dered over two sites with occupation factors of 0.877(4) (C141—C161) and 0.123(4) (C142—C162), respectively.

Results and discussion. The molecular structure of title compound **1** is closely related to that of 1,2-dibenzoyl-1-phenylhydrazine [12] with two phenyl groups replaced by ^tBu each. The conformation of **1** is determined by torsion angles O1—C7—N2—N1 of 13.0(2)° and N2—N1—C12—O2 175.1(1)°. In the crystal packing strong intermolecular N(2)—H...O2 (*x*, $-y+0.5$, $z+0.5$) hydrogen bonds with N—H of 0.88(2) Å, H...O of 2.07(2) Å and N—H...O of 165.3(15)° link molecules into endless chains along the [001] direction (Fig. 2).

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