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SYNTHESIS AND STRUCTURE OF ALLYL 3-OXO-2-(2-PHENYLHYDRAZONE) BUTANOATE

Abstract: *Biologically active allyl 3-oxo-2-(2-phenylhydrazone) butanoate was synthesized in the presence of allyl 3-oxo-butanate and aromatic amine and structure was approved by the X-Ray method.*

Key words: *β-diketones, hydrazones, allyl 3-oxo-2-(2-phenylhydrazone) butanoate, X-Ray.*

Language: *English*

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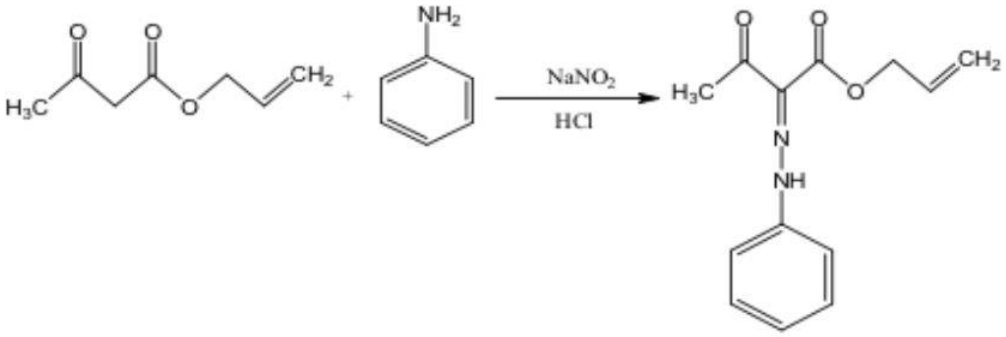
Introduction

Now a day the field of β-diketones is a very exciting area of research because of biological activity [1-3]. β-Diketones are a group of commonly applied spectrophotometric reagents in analytical chemistry because of their capability to form complexes[4-9]. Furthermore, derivatives of these compounds are widely used in the treatment of antioxidant, antiviral centered inflammatory diseases [10-12].

The purpose of the research is to study the structure of the from the mixture of aromatic amine and β-diketone through diazotization reaction. The diazotization reaction between allyl 3-oxo butanoate (I) and aniline led to allyl 3-oxo-2- (2-phenylhydrazone) butanoate (II).



(I) (II)

The reaction was controlled by thin-layer chromatography (Sorbfil). The molecular structure of the obtained compound was determined by the X-ray method and deposited in the Cambridge Crystallographic Data Centre (CCDC 1537217). The compound ($C_{13}H_{14}N_2O_3$, M_r = 146.26), which was studied by the X-ray method, is a yellow crystalline compound with sizes 0.2*0.2*0.2 mm. The dimensions of the monocrystalline structure lattice units are a = 8.7022 (12)Å, b = 8.7472 (12)Å, c = 9.3558 (13)Å, α = 107.090 (2)0, β = 109.747 (2)0, γ = 94.640 (3)0, V = 627.70 (15) Å3, z = 2, Dx = 1.205 mg /cm^3, μ = 0.08 m^{-1}, θ = 2.2–29.2°.

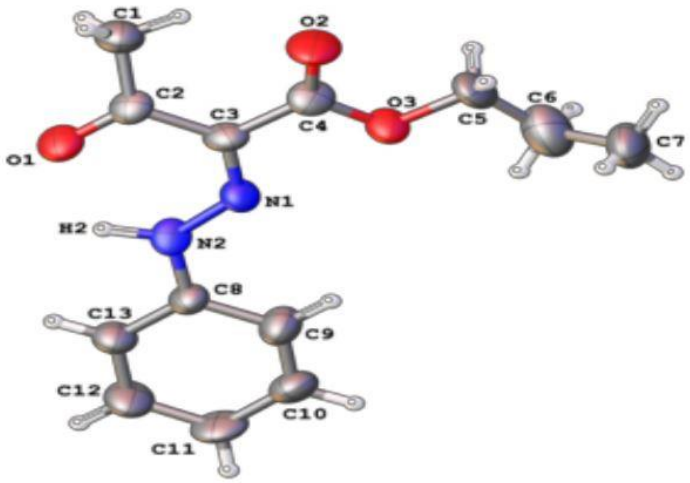


Figure 1. View of the crystal lattice of allyl-3-oxo-2-(2-phenylhydrazone) butanoate molecule.

The reaction and the purity of synthesized compounds were monitored by TLC. The structure of the compound was studied in the "Bruker APEX II CCD" diffractometre (T = 100K, λMoKα-radiation, graphite monochromator, ϕ- and w-scanning, 20max = 560).

General method for the synthesis of allyl-3-oxo-2-(2-phenylhydrazone) butane. 0.0625 mol Aniline and 0.35 g KOH is dissolved 10 ml distilled water and placed in the three-neck flask. Amine was formed an alkaline mixture after completely dissolve. The solution of 0.0625 mol $NaNO_2$ in 2 ml distilled water was added to the mixture and left to stir. 2 ml HCL was added drop by drop to the mixture by the controlling temperature (0°C). If the temperature rises above 0°C the ice is added to the mixture. The reaction is carried out at 0°C for 30 min. Then 0.0625 mol β-diketone and 0.5125 g CH_3COONa were dissolved 10 ml CH_3OH. The temperature of the mixture was cooled to 0°C and was added drop by drop to the previous mixture. The reaction is carried out at 0°C for 1 hour. The product was filtered and recrystallized in ethanol.

References:

1. Vaidya, S. R., Shelke., V. A, Jadhav, S. M., Shankarwar, S. G., & Chondhekar, T. K. (2012). Synthesis and characterization of β-diketones ligands and their antimicrobal activity. *Scholars Research Library, 4(4) 18*, 39-43.
2. Annamalai, S., Balasubramaniyam, A. P., Sabeta, K., & Rajnikant, V. (2011). Synthesis, spectral and RAHB studies on some arylhydrazones of β- diketones: crystal and molecular structure of 2-(2-(3-pyridyl)hidrazono)-5,5-dimethylcyclohexane-1,3-dione and 2-(2-(2-methoxyphenyl)hydrazono)-5,5 dimethylcyclohexane-1,3-dione. *Stuc. Chem., 22*, 23-33.
3. Jadhav, S. M., Shelke, V. A., Munde, A. S., Shankarwar, S. G., Patharkar, V. R., & Chondhekar, T. K. (2010). Synthesis, characterization, potontiometry and antimicrobal studies of transition metal complexes of a tridentate ligand. *J.Coord.Chem., 23*, 41-53.
4. Karvembu, R., Chinnasamy Jayabalakrishanan, & Natarajan, K. (2002). Thiobis (β-diketonato)-bridged binuclear ruthenium(III) complexes containing triphenilphosphine or triphenilarsine. *Trans. Met.Chem., 27*, 574.
5. Ryszard, G., Erkki, K., Henryk, J., Reijo, K., Maija, N., & Borys, O. (2001). Premidonance of 2-arylhydrazones of 1,3-diphenylpropane-1,2,3-trione over its proton transfer products. *J.Phys.Org.Chem., 14*: 797-803.
6. Lasri, J., et al. (2012). Solvent-dependent reactivates of acyclic nitrones with β-diketones: catalyst-free syntheses of endiones and enones. *Tetrahedron, 68, № 35*, pp. 7019-7027.
7. Kumar, C. U., Sethukumar, A., & Prakasam, B. A. (2013). Synthesis and spectral studies of some 4H-pyran derivatives: Crystal and molecular structure of isobutyl 6-amono-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate. *Journal of Molecular Structure, 1036 (2013)*, 257-266.
8. Lingaiah, B. P. V., et al. (2008). Efficient and convenient method for the synthesis of poly functionalized 4H-pyrans. *Synth.Commun., 34*, 4431-4437.
9. Gou, S., Wang, S., & Li, J. (2007). D,L-Proline-catalyzed one-pot synthesis of pyrans and pyrano[2,3-c]pyrazole derivatives by a grinding method under solvent-free condi-tions. *Synth. Commun. 2007, 37*, 2111-2120.
10. John, V. D., & Krishanankutty, K. (2006). Antitumour activity er complexes. *Apply. Organoment. Chem., 20*, 477-486.
11. Kalevi, P., Antti, T., & Ryszard, G. (2003). Henryof synthetic curcuminoid analogues (1,7-diaryl-1,6- heptadiene-3,5-diones) and their coppk J. Behaviour of 1,3-diphenyl-2-arylhydrazono-1,3-propanediones under electron ionisation. *Rapid Commun. Mass Spectrom., 17*,104-106.
12. Maurya, R. C., & Rajput, S. (2004). Encapsulation of N,N- ethylenenis(salisylamide) metal complexes in fly ash based zeolite, characterization and catalytic activity. *J.Mol. Struct., 687*, 35-44.