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**SYNTHESIS OF NEW TRIAZOLE DERIVATIVE FROM THE
REACTION OF ACYL AZIDE WITH BENZYLIDINE ACETOPHENONE**

Annotation: The 1,2,3 – triazole (5) has been synthesized from the reaction of acyl azide(3) with benzylidene acetophenone(4) with a high yield 71%.

Key words: triazole, acyl azide, Chalcone.

**СИНТЕЗ НОВОГО ПРОИЗВОДНОГО ТРИАЗОЛА ЧЕРЕЗ
РЕАКЦИЮ АЦИЛАЗИДА С БЕНЗИЛИДИНА АЦЕТОФЕНОНОМ**

Аннотация: 1,2,3 - триазол (5) синтезирован по реакции ацилазида (3) с бензилидин ацетофеноном (4) с высоким выходом 71%.

Ключевые слова: триазол, ацилазид, халкон.

1.Introduction:

The 1,2,3-triazoles are very important heterocyclic not only for their biological properties. but also for their importance in the synthesis , They rapidly extended to other areas of the fine chemical industries, comprising dyes, agrochemicals, corrosion inhibitors and photostabilizers [1,c.2]. They can be synthesized by many methodology, the new approach to generating functionalized triazoles compounds is by copper-catalyzed Huisgen 1,3-dipolar cycloaddition of azides and alkynes. This conception called Click chemistry which used in first time in 2001 by Sharpless and co-workers.

Acyl azides have widespread utility as highly reactive reagent in organic chemistry. They are extremely useful in the preparation of amides and heterocyclic chemistry. The well known Curtius rearrangement of acyl azides under thermal condition lead to isocyanates, which in turn undergo easy conversion into amines, carbodiimides, urease, urethanes, thiourethanes and other derivatives [2,c.1]. The reaction of alkynes and azides is the most commonly described 1,3-dipolar cycloaddition, and it is also the most used synthetic route for 1,2,3-triazoles.1a-c The importance of this synthetic route increased when Sharpless and Meldal introduced copper as the catalyst in this reaction, which made it possible to obtain 1,4-disubstituted 1,2,3-triazoles with high regioselectivity.1d Heterocycles containing a triazole skeleton have become important pharmacophores for the development of drugs, mainly because they are stable compounds that can mimic peptide bonds [3,c.2].

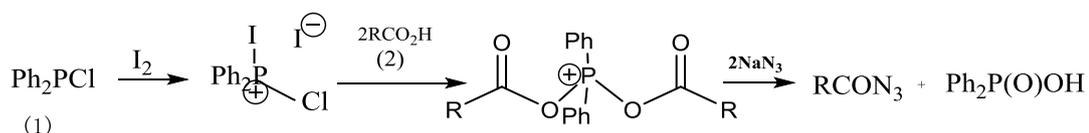
In 2014, It developed an efficient 1,3-dipolar cycloaddition of nitroolefins and inorganic NaN_3 for the synthesis of 4-aryl-NH-1,2,3-triazoles. To suppress the undesired cyclotrimerization of nitroolefins, the reaction was conducted under p-TsOH mediated acidic conditions. The reaction was one of the most effective methods for the synthesis of 4-aryl-NH-1,2,3-triazoles . However, the explosive and toxic hydrazoic acid was released inevitably under the acidic conditions [4,c.1].

By cycloaddition reaction of sodium azide with chalcone in the presence of CuO as a catalyst in DMF a 1,2,3-triazole are prepared in reaction with pentafluoropyridine

to give 2-(tetrafluoropyridin-4-yl)-1,2,3-triazole derivatives in good yields and high regioselectivity [5,c.1961].

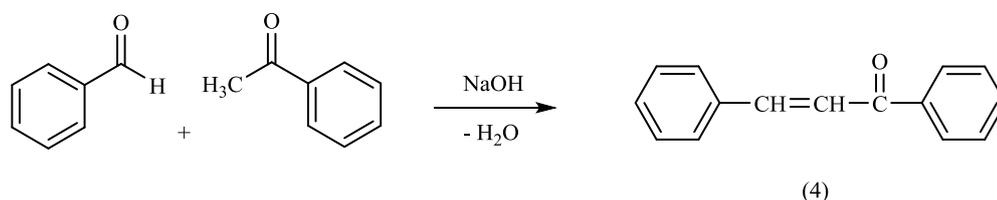
2: Synthesis of Acyl azides:

To a flask containing a stirring mixture of Ph_2PCl (1.0 mmol, 0.18 mL) and I_2 (1.0 mmol, 0.253 g) in dichloromethane (5 mL), was added benzoic acid (2.0 mmol, 0.244 g) at room temperature. Sodium azide (2.1 mmol, 0.136 g) was then added to the reaction mixture. After 90 min, the organic layer was washed successively with saturated aqueous sodium carbonate (3_ 5 mL), aqueous sodium thiosulfate (2_ 5 mL) and water (5 mL). The organic layer was dried with anhydrous Na_2SO_4 and concentrated. Purification by a short silica gel column chromatography using n-hexane afforded the pure benzoyl azide in 80% yield [2,c.445].



3: Synthesis of Benzylidene acetophenone:

Add a conical flask in a 20 ° C water bath with sodium hydroxide solution (3.2g, 0.08mol) and 25 ml water, then add 8ml ethanol and add with stirring with a magnetic motor, acetophenone (6g, 0.05mol) and benzaldehyde (5.3g, 0.05mol) Stirring for three hours with the same degree and then leaving the reaction mixture for several days, where the product (3) is collected by filtration and purified by re-crystallization of the ethanol. Melting point 53-56 ° C [6.c.273].



4: Synthesis of 3,2,1-triazole with acyl azide reaction with acetofinone benzylidene:

Add to a spherical flask with a condenser for (21mmole) of the replaced acyl azide, benzylidene acetone phenon (15mmole) and a DMSO Solvent) In the presence of the catalyst CuO . The mixture is left for a period of time (time required for the

formation of triazole) at the boiling point of the solvent (the flow of the reaction was followed by thin layer chromatography), giving the compound.

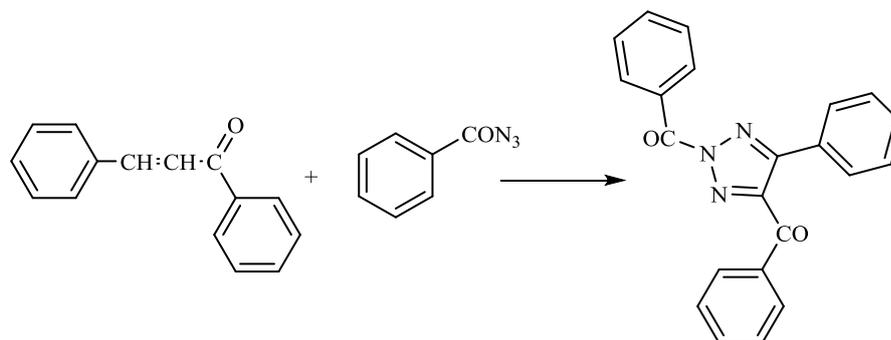


Table 1.

General formulas - Conditions of reaction - Re-crystalline solvent - Melting point and yield of the component:

Molecular Formula	Reflux Time [h]	Yield [%]	m.p [°C]	Crystallization Solvent
C ₂₂ H ₁₇ N ₃ O ₃	35	71	165-166	THF: EtOH 2:1

4: Result and discussion:

The acyl azide reaction with benzylidene acetone was reactivated by 35 hours in the DMSO solvent where the output was separated by evaporating the solvent, then purified by crystallization and then determined by molecular structure using infrared spectra and nuclear magnetic resonance of hydrogen and carbon 13. The spectrum of mass. The infrared spectra of the compound shows the shape absorption band at 1740 cm⁻¹ which belongs to C = O, and other absorption bands at 3090cm⁻¹, 1563cm⁻¹, 1480cm⁻¹, 1149cm⁻¹, 924cm⁻¹, 750cm⁻¹ dating to CH (Stretch aromatic), C=C (Aromatic), N=N, C-N, C-H (Bend aromatic, mono and bis substituted 1,2), respectively.

The ¹H-NMR spectrum of the resulting compound shows diatoms at displacement (δH=7.84ppm, d, 2H) and HH = 7.82ppm, d, 2H) Are associated with carbonates associated with carbonates C-21,25 and carbonates C-15,19, whereas phenyl-proton protons appear in a complex spectrum in the field (δH=7.40-7.05 ppm, m, 10H) .

Table 2.

Descriptive description: IR, ¹H-NMR, ¹³C-NMR and MS for the composite

Product	IR KBr,cm-1	¹ H-NMR δ,ppm	¹³ C-NMR δ,ppm	NMR Solvent	MS Peaks Z / e
C ₂₂ H ₁₇ N ₃ O ₂	3090, 1740,1563, 1480, 1147,924, 750	7.05 - 7.40(m,10H) 7.82(d, 2H) 7.84(d, 2H)	188.65,166.65 126.37- 140.15(Ar)	CDCl ₃	M+(353) 327,296 250, 222 208, 165 105,77 28

The ¹³C-NMR spectra also shows uptake at 188.64ppm due to the carbonyl group. (166.65 ppm and 140.15ppm) that return to C-4 and C-5 in the triazole ring, while benzyl and other benzene carbonate are absorbed in the field 126.37-136.32ppm.

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