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SYNTHESIS OF (3-(5-METHYL-1-PHENYL)-1H-1,2,3-TRIAZOL-4-YL)-4,5-DIHYDRO-1H-PYRAZOL-1-YL) PHENYL METHANONE AND ITS DERIVATIVES FROM SUBSTITUTED ANILINES

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ABSTRACT:
Triazoles, a five-membered cyclic skeleton containing three nitrogens, have been identified as useful building blocks for the synthesis of a large number of organic molecules. Triazoles and their derivatives have attracted considerable attention in the last few decades due to their chemotherapeutic value. It has been considered as a functional core that has almost all types of biological activities, mainly antibiotic, antimicrobial and antifungal.

KEY WORDS: Anilines, Acetyl Acetone, Benzaldehydes, sodium azide, sodium nitrate, sodium methoxide, hydrazine hydrate, benzoic acid.

INTRODUCTION:

Among infectious diseases, tuberculosis (TB) is considered one of the most dangerous chronic communicable diseases with more than 2 million victims per year in the world[i]. At the same time, the emergence of AIDS, which reduced the emphasis on tuberculosis control programs, contributed to the resurgence of the disease in industrialized countries[ii]. Resistance of Mycobacterium tuberculosis (MTB) strains to antimycobacterial agents is also a growing problem worldwide[iii-v]. However, the key problem with tuberculosis chemotherapy is not that no new effective TB drugs with a new mechanism of action have been developed in recent years. Therefore, the use of new anti-TB drugs that are effective against persistent MTB infection is urgently desired. A literature review shows that pyrazoline derivatives have various biological activities such as antibacterial and antifungal[vi], antidiabetic[vii], anti-inflammatory[viii] and are also active against many mycobacterial[ix-xiii]. Therefore, we synthesized some new (3-(5-methyl-1-phenyl-1H-1,2,3-thiazol-4-yl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl) Methanone and its derivatives. All structures are determined by MS, IR, CHN, 1H-NMR and 13C-NMR spectral data.

EXPERIMENTAL SECTION:

All melting points were determined on a Kofler XT4-100x melting point apparatus and are uncorrected. Mass were performed on an HP-5988A spectrometer (EI at 70 eV). 1 H-NMR

spectroscopy ($CDCl_3$) was recorded on NMR 400MHz (JEOL) instrument with TMS as internal standard. Elemental analysis was performed on a Yanaco CHN Corder MT-3 analyzer.

General method for the synthesis of 1-Azido substituted benzene 2a-b:
Compound **2a-b** were prepared by the reaction of substituted anilines **1a-b** with sodium nitrate and sodium azide. In 80ml of water, charged conc, H_2SO_4 (0.45 moles) slowly below 10°C. charged substituted aniline (0.15 moles) in it at same temperature. Cooled this reaction mass to 0°C. charged NaNO2 solution (0.15 moles in 30ml of water) in it at 0°C. stirred this reaction mass for 20mins. Charged NaN3 solution (0.15 moles in 30ml of water) in it below 5°C. Stirred it at RT for 2hrs. After completion of reaction extract it with MDC. Removed MDC under vacuum.

1-Azido-4-nitro benzene 2a
Yellow color solid. Yield 96%. MP 78°C-80°C. 1H NMR (400MHz, $CDCl_3$): δ 7.5 (d, 2H), 8.1(d, 2H); Anal. Data for $C_6H_4N_4O_2$ (164.12): Calcd C 43.91, H 2.46, N 34.14, O 19.5, Found C 43.99, H 2.41, N 34.1, O 19.5.

1-Azido-4-chloro benzene 2b
Light yellow liquid. Yield 66%. BP 95°C-96°C. 1H NMR (400MHz, $CDCl_3$): δ 7.18 (d, 2H), 7.37(d, 2H); Anal. Data for $C_6H_4N_3Cl$ (153.57): Calcd C 46.93, H 2.63, N 27.36, Cl 23.08. Found C 36.39, H 2.04, N 21.22, Br 40.35.

General method for the synthesis of 1-(5-Methyl-1-(Substituted phenyl)-1H-1,2,3-triazol-4-yl)-ethan-1-one 3a-b:
Compound **3a-b** were prepared by the reaction of 1-azido-substituted benzene **2a-b** with acetyl acetone. A cold solution of sodium methoxide (0.23 mole, in 120 mL absolute methanol) was added to the mixture of acetyl acetone (17 mL) and 1-azido-substituted benzene (about 0.15 mole) and stirred for 1 h at 0°–5°C. Then the mixture was heated under reflux on an oil-bath for 4 h. Finally, the mixture was acidified with concentrated hydrochloric acid. Compound 4 was separated and crystallized from methanol.

1-(5-Methyl-1-(4-nitro phenyl)-1H-1,2,3-triazol-4-yl)-ethan-1-one 3a:
Yellow color solid. Yield 92%. MP 106°C-108°C. H-NMR (400 MHz, $CDCl_3$) δ 2.1 (s, 3H, CH_3), δ 2.42 (s, 3H, CH_3), δ 8.11 (d, 2H, Ar), δ 8.47 (d, 2H, Ar). Anal. Data for $C_{11}H_{10}N_4O_3$ (246.23): Calcd C 53.66, H 4.09, N 22.75, O 19.49, Found C 53.33, H 3.89, N 23.05, O 19.72.

1-(5-Methyl-1-(4-chloro phenyl)-1H-1,2,3-triazol-4-yl)-ethan-1-one 3b:
Yellow color solid. Yield 83%. MP 102°C-104°C. H-NMR (400 MHz, $CDCl_3$) δ 2.1 (s, 3H, CH_3), δ 2.42 (s, 3H, CH_3), δ 7.35 (d, 2H, Ar), δ 7.55 (d, 2H, Ar). Anal. Data for $C_{11}H_{10}ClN_3O$ (235.67): Calcd C 56.06, H 4.28, Cl 15.04, N 17.18, O 5.71, Found C 46.55, H 3.12, Br 28.32, N 14.71, O 7.3.

General method for the synthesis of (E)-3-aryl-1-(5-methyl-1-(substituted phenyl)-1H-1,2,3-triazol-4-yl)-prop-2-en-1-one 4a-f:
A mixture of the aromatic aldehyde (0.012 mole) and compound **3a-b** (0.01 mole) dissolved in ethanol (20 mL) was added slowly to an aqueous solution of potassium hydroxide (0.0128 mole) in water (3 mL). The reaction mixture was stirred in crushed-ice bath for 2 h, stirred at 20–25°C for 4 h. The mixture was filtered and the solid was washed with cold water and cold ethanol. The product was crystallized from ethanol to give 4a–d. All the products were

characterized by IR, 1 H-NMR, and mass spectral data.

(E)-1-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-3-(3-nitrophenyl) prop-2-en-1-one 4a:
Yellow color solid. Yield 92%. MP 188°C-189°C. H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), δ 6.95 (d, 1H, CH), δ 7.81 (d, 1H, CH), δ 7.69 (dd, 1H, Ar_1), δ 7.99 (d, 1H, Ar_1), δ 8.14 (d, 1H, Ar_1), δ 8.31 (d, 1H, Ar_1), δ 8.11 (d, 2H, Ar_2), δ 8.47 (d, 2H, Ar_2), Anal. Data for $C_{18}H_{13}N_5O_5$ (379.33): Calcd C 56.99, H 3.45, N 18.46, O 21.09, Found C 56.54, H 3.89, N 17.26, O 22.3.

(E)-1-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-3-(2-chlorophenyl) prop-2-en-1-one 4b:
Off white color solid. Yield 92%. MP 183°C-185°C. H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), δ 6.53 (d, 1H, CH), δ 7.97 (d, 1H, CH), δ 7.08 (dd, 1H, Ar_1), δ 7.25 (dd, 1H, Ar_1), δ 7.28 (d, 1H, Ar_1), δ 7.53 (d, 1H, Ar_1), δ 8.11 (d, 2H, Ar_2), δ 8.47 (d, 2H, Ar_2), Anal. Data for $C_{18}H_{13}ClN_4O_3$ (368.78): Calcd C 58.63, H 3.55, Cl 9.61, N 15.19, O 13.02, Found C 58.5, H 3.67, Cl 9.4 N 16.6, O 11.83.

(E)-1-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-3-(4-chlorophenyl) prop-2-en-1-one 4c:
Off white color solid. Yield 92%. MP 183°C-185°C. H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), δ 6.7 (d, 1H, CH), δ 7.7 (d, 1H, CH), δ 7.62 (d, 2H, Ar_1), δ 7.68 (d, 2H, Ar_1), δ 8.11 (d, 2H, Ar_2), δ 8.47 (d, 2H, Ar_2), Anal. Data for $C_{18}H_{13}ClN_4O_3$ (368.78): Calcd C 58.63, H 3.55, Cl 9.61, N 15.19, O 13.02, Found C 58.5, H 3.67, Cl 9.4 N 16.6, O 11.83.

(E)-1-(5-methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)-3-(3-nitrophenyl) prop-2-en-1-one 4d:
Off white color solid. Yield 92%. MP 183°C-185°C. H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), δ 6.95 (d, 1H, CH), δ 7.81 (d, 1H, CH), δ 7.69 (dd, 1H, Ar_1), δ 7.99 (d, 1H, Ar_1), δ 8.14 (d, 1H, Ar_1), δ 8.31 (d, 1H, Ar_1), δ 7.35 (d, 2H, Ar_2), δ 7.55 (d, 2H, Ar_2), Anal. Data for $C_{18}H_{13}ClN_4O_3$ (368.78): Calcd C 58.63, H 3.55, Cl 9.61, N 15.19, O 13.02, Found C 58.5, H 3.5, Cl 9.6, N 15.1, O 13.3.

(E)-1-(5-methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)-3-(2-chlorophenyl) prop-2-en-1-one 4e:
Off white color solid. Yield 92%. MP 155°C-156°C. H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), δ 6.53 (d, 1H, CH), δ 7.97 (d, 1H, CH), δ 7.08 (dd, 1H, Ar_1), δ 7.25 (dd, 1H, Ar_1), δ 7.28 (d, 1H, Ar_1), δ 7.53 (d, 1H, Ar_1), δ 7.35 (d, 2H, Ar_2), δ 7.55 (d, 2H, Ar_2), Anal. Data for $C_{18}H_{13}Cl_2N_3O$ (358.22): Calcd C 60.35, H 3.66, Cl 19.79, N 11.73, O 4.47, Found C 60.3, H 3.60, Cl 19.7, N 12.8, O 3.60.

(E)-1-(5-methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)-3-(4-chlorophenyl) prop-2-en-1-one 4f:
White color solid. Yield 92%. MP 152°C-154°C. H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), δ 6.7 (d, 1H, CH), δ 7.7 (d, 1H, CH), δ 7.62 (d, 2H, Ar_1), δ 7.68 (d, 2H, Ar_1), δ 7.35 (d, 2H, Ar_2), δ 7.55 (d, 2H, Ar_2), Anal. Data for $C_{18}H_{13}Cl_2N_3O$ (358.22). Calcd C 60.35, H 3.66, Cl 19.79, N 11.73, O 4.47, Found C 60.3, H 3.60, Cl 19.7, N 12.8, O 3.60.

General method for the synthesis of (5-(4-substituted phenyl)-3-(5-methyl-1-(4-substituted phenyl-1H-1,2,3-triazol-4-yl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)methanone-5a-f:

A mixture of compound **4a-f** (0.01 mole), hydrazine hydrate (0.03 mole) and benzoic acid (0.01mole) in ethanol was refluxed for 3 h, then poured into crushed-ice. The precipitate was separated by filtration, washed with water and the crude products **5a–f** was obtained, which were crystallized from ethanol.

(3-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-5-(3-nitrophenyl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)Methanone 5a:

Yellow color solid. Yield 75%. MP 180°C-181°C. H-NMR (400 MHz, $CDCl_3$) δ 2.72-2.79 (s, 3H, CH_3), δ 3.29-3.32 (dd, 1H, CH), δ 3.83-3.89 (dd, 1H, CH), δ 5.04-5.09 (dd, 1H, CH), δ 7.58-8.48 (m, 13H, Ar_1-H, Ar_2-H, Ar_3-H), Anal. Data for $C_{25}H_{19}N_7O_5$ (497.14): Calcd C 60.36, H 3.85, N 19.71, O 16.08, Found C 60.20, H 3.65, N 20.1, O 16.05.

(3-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-5-(2-chlorophenyl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)Methanone 5b:

Yellow color solid. Yield 45%. MP 116°C-118°C. H-NMR (400 MHz, $CDCl_3$) δ 2.7-2.79 (s, 3H, CH_3), δ 3.32 (dd, 1H, CH), δ 3.86-3.92 (dd, 1H, CH), δ 5.34-5.40 (dd, 1H, CH), δ 7.27-8.51 (m, 13H, Ar_1-H, Ar_2-H, Ar_3-H), Anal. Data for $C_{25}H_{19}ClN_6O_3$ (486.12): Calcd C 61.67, H 3.93, Cl 7.28, N 17.16, O 9.68, Found C 61.58, H 3.90, Cl 7.42 N 17.10, O 9.72.

(3-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-5-(4-chlorophenyl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)Methanone 5c:

Off white color solid. Yield 56%. MP 116°C-118°C. H-NMR (400 MHz, $CDCl_3$) δ 2.68-2.77 (s, 3H, CH_3), δ 3.29-3.32 (dd, 1H, CH), δ 3.72-3.79 (dd, 1H, CH), δ 4.89-4.95 (dd, 1H, CH), δ 7.34-8.43 (m, 13H, Ar_1-H, Ar_2-H, Ar_3-H), Anal. Data for $C_{25}H_{19}ClN_6O_3$ (486.12): Calcd C 61.67, H 3.93, Cl 7.28, N 17.16, O 9.68, Found C 61.58, H 3.90, Cl 7.42 N 17.10, O 9.72.

(3-(5-methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)-5-(3-nitrophenyl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)Methanone 5d:

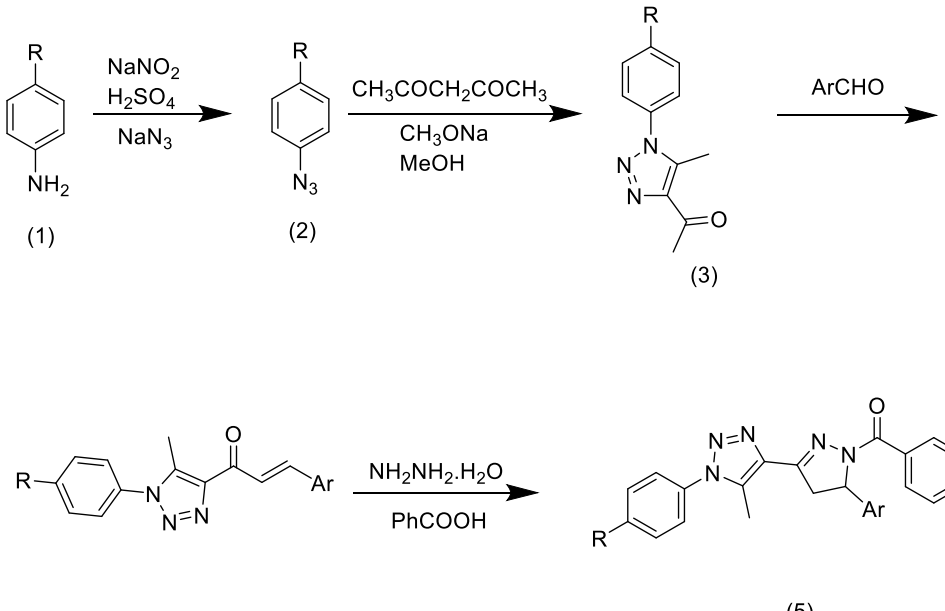
Off white color solid. Yield 45%. MP 162°C-163°C. H-NMR (400 MHz, $CDCl_3$) δ 2.63 (s, 3H, CH_3), δ 3.56-3.61 (dd, 1H, CH), δ 3.77 (dd, 1H, CH), δ 5.45-5.48 (dd, 1H, CH), δ 7.36-8.67 (m, 14H, Ar_1-H, Ar_2-H, Ar_3-H), Anal. Data $C_{25}H_{19}ClN_6O_3$ (486.12): Calcd C 61.67, H 3.93, Cl 7.28, N 17.16, O 9.68, Found C 61.58, H 3.90, Cl 7.42 N 17.10, O 9.72.

(3-(5-methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)-5-(2-chlorophenyl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)Methanone 5e:

Off white color solid. Yield 65%. MP 115°C-116°C. H-NMR (400 MHz, $CDCl_3$) δ 2.70-2.79 (s, 3H, CH_3), δ 3.23 (dd, 1H, CH), δ 3.86-3.92 (dd, 1H, CH), δ 5.34-5.40 (dd, 1H, CH), δ 7.27-7.77 (m, 13H, Ar_1-H, Ar_2-H, Ar_3-H), Anal. Data for $C_{25}H_{19}Cl_2N_5O$ (475.1): Calcd C 63.04, H 4.02, Cl 14.88, N 14.70, O 3.36, Found C 62.85, H 4.01, Cl 15.5, N 13.9, O 3.74.

(3-(5-methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)-5-(4-chlorophenyl)-4,5-dihydro-1H-pyrazol-1-yl) (phenyl)Methanone 5f:

Off white color solid. Yield 53%. MP 112°C-114°C. H-NMR (400 MHz, $CDCl_3$) δ 2.68-2.77 (s, 3H, CH_3), δ 3.29-3.32 (dd, 1H, CH), δ 3.72-3.79 (dd, 1H, CH), δ 4.90-4.95 (dd, 1H, CH), δ 7.34-7.83 (m, 12H, Ar_1-H, Ar_2-H, Ar_3-H), Anal. Data for $C_{25}H_{19}Cl_2N_5O$ (475.1): Calcd C 63.04, H 4.02, Cl 14.88, N 14.70, O 3.36, Found C 62.85, H 4.01, Cl 15.5, N 13.9, O 3.74.



(1) (2) (3) (4) (5)

R	**Ar**
NO_2 (p), Cl(p)	o-ClC_6H_4, p-ClC_6H_4, m-$NO_2C_6H_4$

Scheme 1

RESULT AND DISCUSSION:

Compounds (E)-3-aryl-1-(5-methyl-1-(substituted phenyl)-1H-1,2,3-triazol-4-yl)-prop-2-en-1-one 4a–f were prepared by the Claisen-Schmidt condensation reaction of 1-(5-methyl-1-(substituted phenyl)-1H-1,2,3-triazol-4-yl)-ethan-1-one 3a-b and some aromatic aldehydes. Target compounds (5-(4-substituted phenyl)-3-(5-methyl-1-(4-substituted phenyl)-1H-1,2,3-triazol-4-yl)-4,5-dihydro-1H -pyrazol-1-yl) phenyl Methanone 5a–f was prepared from 4a–f, hydrazine hydrate, and benzoic acid by sequential reactions involving intermolecular conjugate addition, hydrazone formation.

In summary, a number of pyrazoline derivatives have been conveniently prepared by a simple synthetic method from an aromatic amine. Its structure was characterized by MS, CHN, 1H-NMR spectral data.

CONCLUSION:

This literature reveals various diverse biological activities such as antimicrobial, antibacterial, antifungal, anticonvulsant, antitubercular and anti-inflammatory properties of (3-(5-methyl-1-phenyl)-1H-1,2,3-triazol-4-yl)- 4,5-dihydro-1H-pyrazol-1-yl) (phenyl) Methanone and its derivatives. Various drugs on the market today contain a triazole group. It can therefore be concluded that the derivatives (3-(5-methyl-1-phenyl)-1H-1,2,3-triazol-4-yl)-4,5-dihydro-1H-pyrazol-1-yl) phenyl Methanone has great potential as a bioactive molecule.

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