Synthesis and characterization of some 3-(2-(5-phenyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-ones, using Click reaction.

3.1) Introduction

3.1.1) Introduction of Triazole

1,2,3-triazoles are an important class of five-membered nitrogen-containing aromatic compounds with high chemical stability to oxidation, reduction and hydrolysis. 1,2,3-triazoles are commonly employed as a powerful tool in many fields. Triazoles are known to have two structural isomeric forms, 1, 2, 3-triazole and 1, 2, 4-triazole. Each exists in two dissimilar tautomeric forms. The different isomers are characterised by the position of the nascent hydrogen. Thus 1, 2, 3-triazoles are existed in two forms i.e. 1H and 2H.

They are target materials for drug discovery since they show a wide range of biological properties such as antibacterial, antiviral, anticancer and anti-allergic. In addition, they have found applications in optical brighteners, Agrochemicals, dyes, anti-HIV therapy, fluorescence chemosensors, etc. and also are interesting materials in organic synthesis, coordination chemistry and N-heterocyclic chemistry. Consequently, the development of simple, efficient and practical methods for their synthesis in a single-step operation has attracted much attention in recent years. Huisgen’s 1,3-dipolar cycloaddition of terminal alkynes with organic azides is the most popular method for the construction of 1,2,3-triazoles. This reaction also is one of the well-known, highly useful and premier reactions in ‘click chemistry’. Recently, the catalytic preparation of 1,2,3-triazoles in the presence of transition metal ions such as Zn, Cu, Ru, Pd, Ce, etc. have been developed. Among these, the Cu-catalyzed Azide-Alkyne Cycloaddition (CuAAC) reaction has attracted much attention due to exclusive region selectivity, wide substrate scope, mild reaction conditions and very high yields. CuAAC reaction is the most reliable ‘click reaction’ for the practical and efficient preparation of 1,4-disubstituted 1,2,3-triazoles from a wide range of substrates which cannot be prepared via traditional Huisgen uncatalysed thermal approaches. In recent years, a variety of Cu(I) salts or coordination complexes have been employed as homogeneous and heterogeneous catalysts for the azide-alkyne cycloaddition. Nevertheless, the Cu(I) species in these catalysts are unstable and can be
easily oxidised to Cu(II) or disproportionate to Cu(0) and Cu(II), which limited their utilisation. Although organic azides are generally stable, preparation, isolation or purification of some organic azides can be problematic processes. Thus, a procedure that avoids the isolation of organic azides like in situ generation of organic azides is desirable. In situ preparing of organic azides minimises hazards derived from their isolation or handling and avoids the time-consuming and waste generation of additional synthetic steps. In recent years, the copper-catalyzed multicomponent syntheses of 1,2,3-triazoles from the reaction of terminal alkynes with in situ generated organic azides by the reaction of sodium azide with organic halides or by the reaction of sodium azide with epoxides have been reported in the literature. Acidolysis of epoxides forms azidoalcohols which further cycloaddition with alkynes results in hydroxyethyl-1,2,3-triazoles found in peptide surrogates of HIV-1 protease inhibitors. Therefore, it is not surprising that the development of newer catalysts, methods and strategies for the synthesis of 1,2,3-triazoles has remained a highly attractive proposition. To our knowledge, the Cu(II) complexes with Schiff base ligands have seldom been used as the catalysts for the azide-alkyne coupling reaction and there is not any report for the use of Cu(II)-azide complexes as a catalyst for such reactions. Therefore, we reasonably assumed that azide coordinated Cu(II) complexes might be a class of effective copper catalysts and also azide sources for the preparation of 1,2,3-triazoles. Herein, we wish to report our results in using of a Cu(II)-Azide complex as a new catalytic system for preparing 1,2,3-triazoles and also introduce a new function for Cu(II)-azide complexes. The reported Cu(II)-Azide complex in this study is stable in air and moisture and was synthesised from the cheap starting materials with a satisfactory yield. In addition, considering the requirement of green chemistry, 1,2,3-triazoles has been widely explored using click chemistry approach due to its complete specificity, efficiency, simple reaction workup procedure, and quantitative reaction yield of the products.

3.1.2) Introduction of Coumarin

Coumarin and their derivatives have attracted considerable attention due to their extensively biological activities such as antibacterial, antifungal, antiviral, anti-tubercular, anti-malarial, anti-coagulant, anti-inflammatory, anti-cancer, antioxidant properties and so on. Numerous efforts, including the separation and purification of naturally occurring coumarins from a variety of plants as well as their artificial synthesis of coumarin compounds with novel structures and properties, have been focusing on the research and development of coumarins as potential drugs. So far some coumarins, for example, Warfarin, Acenocoumarol, Armillarisin A, Hymecromone and Carbochromen have been approved for therapeutic purposes in the clinic. More importantly, an increasing number of coumarin compounds have displayed great potency in the treatment of various types of diseases. Coumarin compounds, containing 1,2-benzopyrone skeleton structurally similar to clinical anti-infective quinolone drugs with benzopyrone backbone, as a new type of antibiotics has received specific interest along with the dramatically rising prevalence of multi-drug resistant microbial infections. It is well known that the quinolone anti-infective are of wholly synthetic origin and not modelled knowingly after any natural antibiotics of all the totally synthetic
antimicrobial agents, the quinolones have proved to be most successful economically and clinically. As predominant antibacterial drugs, quinolones have been widely used in the clinic with orally and parentally active properties, broad antimicrobial spectrum including many frequently encountered pathogens, and bactericidal behaviour in clinically achievable doses. Clearly, quinolone anti-infective have been playing important roles in the past fifty years in the unending struggles against morbidity and mortality caused by microbial pathogens. However, the prevalently clinical use of this class of anti-infectives has led to increasing worrisome resistance at a disturbing rate, which has posed serious problems in the treatment of infectious diseases. This trend has highlighted the urgent need for designing and developing powerful antimicrobial compounds, especially for the exploration of new quinolone-like compounds. Reasonably, a great deal of work has recently directed towards coumarin compounds which have asimilar structural framework to quinolones. Some naturally occurring coumarins such as Novobiocin, Coumermycin and Clorobiocin have been found to be an unprecedented class of antibiotics, but they are not used in clinic owing to their relatively weak activity towards Gram-negative bacteria, poor water solubility and side effects. However, wholly synthetic coumarin compounds have drawn renewed attention because of their prominent antibacterial properties, specifically against methicillin-resistant Staphylococcus aureus (MRSA). In recent years, some works have manifested that coumarin backbone in combination with some nitrogen-containing heterocyclic moieties such as azetidine, thiazolidine, thiazole and so on could significantly increase the antimicrobial efficiency and broaden their antimicrobial spectrum. In this work, we would like to introduce 1,2,4-triazolyl and 1,2,3-triazolyling into coumarin. As important aromatic nitrogen-containing heterocycle, 1,2,4-triazole compounds have aroused special interest due to their excellent pharmacokinetic characteristics, favourable safety profile, as well as the latent ability for the formation of hydrogen bonds. With other active molecules. A number of triazole drugs including Fluconazole, Itraconazole and Voriconazole have been prevalently used in the anti-infective therapy. Recently, some triazole derivatives have been reported to exhibit good anti-MRSA potency. Although the extensively clinical use of triazole anti-infective agents has ever revolutionised the treatment of many infectious diseases, some of them are still limited by poor activities towards intractable fungi, high frequency of renal toxicity and several adverse effects. Therefore, these situations have been served as the impetus for developing new triazole antimicrobial agents. Prompted by above observations and in continuation of our ongoing interest in the development of new antimicrobial agents, herein we designed and synthesised a class of new coumarintriazole compounds and evaluated their antibacterial and antifungal activities in vitro. In order to investigate the effect of heterocyclic triazole moiety on antimicrobial activity, coumarinbis-triazoles, mono-triazoles as well as on-triazolebis-coumarins and coumarin bromides were prepared. A lot of researches have provided evidence that the linkers could modulate the physic-chemical properties of the whole molecule and thereby affect biological activities. Based on this, and with the aim of better understanding of structure–activity relationship, various types of linkers including alkyl and aralkyl ones, as well as the spacers with different lengths of aliphatic chains were introduced into the target compounds. Recent studies also found that the transformation of azoles into their corresponding salts could enhance antimicrobial efficacy remarkably
due to the improvement of water solubility and membrane permeability.\textsuperscript{35} Thus, some representative coumarin mono-triazoles and as well as bistriazoles were converted into the corresponding hydrochlorides. The desired coumarin compounds were prepared from commercially available phenols and the coupling of coumarin halides with hydroxycoumarin generated bis-coumarins with good yields.

Triazole, a heterocyclic nucleus has attracted a wide attention of the medicinal chemist in search for the new therapeutic molecules. Now a day’s research is focused towards the introduction of new and safe therapeutic agents of clinical importance. The nitrogen-containing heterocycles are found in abundance in most of the pharmaceutically active compounds. Triazoles are well known five-membered heterocyclic compounds belong to one of the most widely used classes of antifungal drugs known as azoles.\textsuperscript{36}

3.2) Synthesis Aspect

Shabnam Shaabani\textsuperscript{37} and Co-workers have reported one pot synthesis six component Coumarin-3-carboxamides containing a Triazole Ring via an Isocyanide-Based via salicylaldehyde and Meldrum’s acid in EtOH was stirred for 10 hr. Then, amine, proparglyoxy aldehyde, and isocyanide were added and the mixture stirred at ambient temperature for 8 hr. After completion of the reaction, as indicated by TLC, aryl azide, Cu(OAc), and sodium ascorbate were added. Then, the resulting mixture was stirred for 6 hr at room temperature, gave good isolated yield. (Scheme 3.1)

Yoshinori Tominaga\textsuperscript{38} and coworkers reported direct synthesis of 1,2,4-triazolo[1,5-\textit{a}]pyrimidine derivatives via condensation of 5-amino-1,2,4-triazole with ketene dithioacetals by heating at 150°C for 3 hrs in 55% yield. (Scheme 3.2)

Dalip Kumar\textsuperscript{39} et al was synthesis 1,4-disubstituted 1,2,3-triazoles were synthesised
using the one-pot reaction of α-tosyloxy ketones/α-halo ketones, sodium azide, and terminal alkynes in the presence of aq PEG using the click chemistry approach and evaluated for SRC kinase inhibitory activity. Eco-friendly reaction process, having good isolated yield. (Scheme 3.3)

Scheme 3.3

Xian-Wei Ye et al. designed two-step novel route synthesis of coumarin-1,2,3-triazole-dithiocarbamate hybrid from phenols and ethyl 4-chloroacetoacetate by using Pechman condensation conditions. Condensation of substituted salicylaldehyde with propanoic anhydride in refluxing propionic anhydride gave coumarin, displayed excellent selectivity against lysine-specific demethylase without inhibition against monoamine oxidases (MAOs). Further investigation revealed that compound was active in both recombinant and cells level by up-regulating the expression of H3K4me1, H3K4me2 and H3K9me2. Then second step click reaction condition, it was good route design and synthesis, excellent isolated yield. (Scheme 3.4)

Scheme 3.4

H. J. Carlsen and coworkers have reported the synthesis of sterically hindered 4-
alkyl-3,5-diphenyl-4H-1,2,4-triazoles by heating bis(α-chlorobenzylidene) hydrazine with alkylamines either neat or in different solvents, were transformed into the triazole analogous in high yields. (Scheme 3.5)

Scheme 3.5

Xu Y. A.\textsuperscript{42} and co-workers reported the synthesis of series of new 1,5-disubstituted-1,2,4-triazole from oxamide-derived amidine and various hydrazine hydrochloride salts in excellent yield with minimal purification. Both aromatic and aliphatic hydrazines readily react with the amidine reagents under very mild reaction conditions. (Scheme 3.6)

Scheme 3.6

K. Easwaramoorth\textsuperscript{43} and co-workers have reported the synthesis of 1,4-Disubstituted 1,2,3-Triazolo-Bosentan Derivatives from Bosentan were accomplished in good yields by sequential chlorination, azidation followed by Cu(I) catalysed 1,3-dipolar cycloaddition. All obtained compounds were evaluated for their antimicrobial, in vitro anticancer activities and in silico docking studies. Among all tested compounds and have shown better antimicrobial activities against different strains of bacteria and fungi. (Scheme 3.7)

Scheme 3.7

Rodrigo González-Olvera\textsuperscript{44} \textit{et al.} have synthesis series of new mono-1,2,3-triazole
derivatives of pyrimidine nucleobases were synthesised by one-pot copper(I)-catalyzed 1,3-dipolar cycloaddition reactions between N-1-propargyluracil and thymine, sodium azide and several benzyl halides. The desired heterocyclic compounds were obtained in good yields. (Scheme 3.8)

Nazariy Pokhodylo et al. have reported some 5-methyl-1-phenyl-1H-1,2,3-triazole-4-carboxylic acid from ethyl 3-oxobutanoate compound with azidobenzene, and it was observed that some compounds showed slight anticancer activity. One of them possessed a moderate activity against melanoma, colon, and breast cancer. Compounds containing triazole and flavonoid fragments in one molecule were prepared by a one-step synthetic route and good isolation yield. (Scheme 3.9)

Eun Jeong Yoo and co-workers carry out the synthesis of N-sulfonyltriazoles arising from the reaction of sulfonylazides with those acetylene compounds in the present of CuAAC as acatalyst, can undergo rearrangement process leading to a mixture of triazoles and the ring-opened tautomers, a-diazoiminospecies. The reversibility of the ring-chain tautomerism, known as the Dimroth rearrangement. It novel Controlling Selectivity target. Good isolation yield and easy work-up process. (Scheme 3.10)

Alina K. Feldman et al. have reported 1,4-Disubstituted 1,2,3-Triazoles in situ
generate Azides from Iodobenzene was mixed with 1-chloro-4-prop-2-ynyloxy-benzene in a 20 mL scintillation vial. and CuSO₄·5H₂O, Sodium ascorbate and second route of the catalystare L-Proline and Na₂CO₃. Both route routeis a success but more covenant yield gives by CuSO₄·5H₂O, Sodium ascorbate, and simple work up the process and excellent isolation yield. (Scheme 3.11)

![Scheme 3.11](image)

Prasad Appukuttan and co-workers have asynthesis of 1,4-Disubstituted 1,2,3-Triazoles, from azide and alkyne were used with the halide in water and t-BuOH, Cu turnings, and 1M of CuSO₄ Solution in water, this three-component reaction by microwave-assisted, its eco-friendly reaction, and time consumable technique, easy work-up process, and excellent isolation yield. (Scheme 3.12)

![Scheme 3.12](image)

### 3.3) Current Work

**Aim of current work:**

The biological importance of 3-(2-(1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one is well documented over the last few decades. A variety of substituted derivatives of 3-(2-(1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one moiety have shown usefulness against a range of biological target molecules. For example, they have possessed activity against Antitubercular, malaria, coronary vasodilators, antihypertensive agents, antibiotics, adenosine A2A receptor antagonists, antitumor agents as well as fungicides.

Various methodologies have been described for the synthesis of 3-(2-(1H-1,2,3-triazol-}
1-yl)acetyl)-2H-chromen-2-one During the course of our ongoing interest in synthesis of various heterocyclic compounds using active methylene compound, we observed that active methylenes are versatile intermediate for the synthesis of triazole. Thus, to synthesized target molecules, the various acetylene and 3-(2-azidoacetyl)-2H-chromen-2-one were condensed in the presence of sodium ascorbate (20 mmol%) and 1 M CuSO₄ to afford 3-(2-(1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one. The newly synthesised compounds were characterised by IR, Mass, ¹H NMR, ¹³C NMR spectroscopy and elemental analyses.

3.4) Reaction scheme

3.4.1) Experimental procedure:

**Step01:** Preparation of 3-acetyl-2H-chromen-2-one

**Step02:** Preparation of 3-(2-bromoacetyl)-2H-chromen-2-one

**Step03:** Preparation of 3-(2-azidoacetyl)-2H-chromen-2-one
Step 04: Preparation of 3-(2-(1H-1,2,3-triazol-1-yl)ethyl)-2H-chromen-2-one

3.5) Experimental section

Melting points were determined in open capillary tubes and are uncorrected. Formation of the compounds was checked by TLC on silica gel-G plates of 0.5 mm thickness and spots were located by iodine and UV light. IR spectra were recorded on SHIMADZU FT-IR-Affinity-1S. Mass spectra were recorded on Shimadzu GC-MS-QP-2010 model using direct inlet probe technique. $^{1}$H NMR and $^{13}$C NMR was determined in DMSO-$d_6$ solution on a Bruker Ac 400 MHz spectrometer. The purity of the synthesised compounds was checked by HPLC Shimadzu-10AT. Elemental analysis of the all the synthesised compounds was carried out on Euro EA 3000 elemental analyser and the results are in agreements with the structures assigned.

Step 01: Synthesis of 3-Acetyl-2H-chromene-2-one

In RBF, cold mixture of salicylaldehyde (1.0 mmol), ethyl acetoacetate (1.0 mmol), ACN (10 ml) and catalytic amount of piperidine was added and thereaction mixture was stirred for 30 min at room temperature. The solid separated was filtered and washed with ethanol. Recrystallization of the solid from chloroform afforded 3-acetyl-2H-chromene-2-one. Yield (93%): m.p.: 119–121 °C (20% EtOAc/hexane).

Step 02: Synthesis of 3-(2-bromoacetyl)-2H-chromen-2-one

In RBF, 3-Acetyl-2H-chromene-2-one (1.0 mmol) was dissolved in dry chloroform and a solution of Br$_2$ (1.0 mmol) in chloroform was added dropwise with continuous stirring and mixture was heated at reflux for 6–7 hr. The progress of the reaction was
monitored by thin layer chromatography (TLC). After completion of the reaction, the mixture was concentrated under reduced pressure, and washed with diethyl ether and recrystallized (70% CHCl₃/EtOH) to afford 3-(2-bromoacetyl)-2H-chromen-2-one. Yield: 69%, m.p.: 161–163°C.⁴⁹

Step03:- Synthesis of 3-(2-azidoacetyl)-2H-chromen-2-one

In RBF, mixture of 3-(2-bromoacetyl)-2H-chromen-2-one (1.0 mmol), DMF (2 ml), NaN₃ (1.1 mmol) slowly addition at 0°C, then RT stir for 1 hr. The progress of the reaction was monitored by thin layer chromatography (TLC). Pour into ice, solid fallout, filter and dry it. Yield of 3-(2-(5-phenyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one, yield 90 %, Dark green color.

Step04:- Synthesis of 3-(2-(5-phenyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one

In RBF, mixture of 3-(2-azidoacetyl)-2H-chromen-2-one, acetylene (1.0 mmol) in aq DMF: t. Butenol : Water (1:1:1, 5 mL) was added sodium ascorbate (20 mmol%) and 1 M CuSO₄ (5 mmol%) solution. The reaction mixture was allowed to stir at room temperature for 3 hr. After the reaction was complete, as indicated by TLC, the reaction mixture was poured in ice then stir 30 min at RT solid fallout, then filter and wash with Methanol, The afforded crude product was recrystallized from Acetone. M.P.- 275-277°C. Elemental Analysis: Calculated: C (68.88%), H (3.95%), N (12.72%), Found: C (68.70 %), H (3.81 %), N (12.68 %), Yield 85 %

Similarly, other compounds (EJ-226 to EJ-248) were synthesised by the above-mentioned process [Step 4] from Scaffold. The physical data are recorded in Table-II.

Table.1 Optimization of the reaction conditions 3-(2-azidoacetyl)-2H-chromen-2-one and acetylene derivative.
aReaction condition: 3-(2-azidoacetyl)-2H-chromen-2-one (1 mmol), acetylene (1 mmol), catalyst, and solvent (5 mL).

bIsolated yield

**Table 2** Synthesis of various derivatives of 3-(2-(1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one.

<table>
<thead>
<tr>
<th>Code-No</th>
<th>R₁</th>
<th>R₂</th>
<th>Yield %</th>
<th>mp°C</th>
</tr>
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<tbody>
<tr>
<td>EJ-229</td>
<td>H</td>
<td>CycloHexane</td>
<td>87%</td>
<td>252-254</td>
</tr>
<tr>
<td>EJ-230</td>
<td>H</td>
<td>4-F-Ph</td>
<td>91%</td>
<td>264-266</td>
</tr>
<tr>
<td>EJ-231</td>
<td>H</td>
<td>Cyclopentan</td>
<td>85%</td>
<td>277-279</td>
</tr>
<tr>
<td>EJ-232</td>
<td>H</td>
<td>4-ter.But.-Ph</td>
<td>92%</td>
<td>290-292</td>
</tr>
<tr>
<td>EJ-237</td>
<td>Br</td>
<td>Ph</td>
<td>79%</td>
<td>264-266</td>
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<tr>
<td>EJ-238</td>
<td>Br</td>
<td>CycloHexane</td>
<td>82%</td>
<td>254-256</td>
</tr>
<tr>
<td>EJ-239</td>
<td>Br</td>
<td>4-Ethyl-Ph</td>
<td>75%</td>
<td>266-268</td>
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<tr>
<td>EJ-240</td>
<td>Br</td>
<td>Cyclopentan</td>
<td>79%</td>
<td>261-263</td>
</tr>
<tr>
<td>EJ-241</td>
<td>Br</td>
<td>4-ter. But.-Ph</td>
<td>85%</td>
<td>280-282</td>
</tr>
<tr>
<td>EJ-242</td>
<td>Br</td>
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<tr>
<td>EJ-243</td>
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<td>4-CH₃-Ph</td>
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<td>EJ-247</td>
<td>H</td>
<td>4-Propyl-Ph</td>
<td>92%</td>
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<td>EJ-248</td>
<td>H</td>
<td>Cyclopropyl</td>
<td>89%</td>
<td>252-254</td>
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aReaction condition: 3-(2-azidoacetyl)-2H-chromen-2-one (1 mmol), acetylene (1 mmol), sodium ascorbate (20 mmol%), 1 M CuSO₄ (5 mmol%) solution, and solvent (5 mL).

bIsolated yield
### 3.6) Spectral and Physical Characterization

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<thead>
<tr>
<th>EJ-229</th>
<th>3-(2-(4-cyclohexyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
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<tr>
<td>Molecular Formula</td>
<td>C₁₉H₁₉N₃O₃</td>
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<tr>
<td>Obs</td>
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<tr>
<td>ℏ¹H NMR δ ppm (DMSO-d₆)</td>
<td>1.26-1.30 (m, 2H, CH₂), 1.44-1.49 (m, 6H, CH₂), 1.88-1.97 (m, 2H, CH₂), 2.95-3.03 (m, 1H, CH*), 6.02 (s, 2H, CH₂), 7.18-7.24 (m, 2H, ArH), 7.27 (s, 1H, ArH), 7.42-7.46 (t, 1H, ArH), 7.61 (d, 1H, J=6.4Hz, ArH), 8.2 (s, 1H, ArH)</td>
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<td>ℏ¹³C NMR δ ppm (DMSO-d₆)</td>
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<td>FT—IR νmax cm⁻¹</td>
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<table>
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<tr>
<th>EJ-230</th>
<th>3-(2-(4-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
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<td>ℏ¹³C NMR δ ppm (DMSO-d₆)</td>
<td>58.4, 115.7, 115.9, 116.2, 118.0, 122.1, 122.9, 125.2, 127.1, 127.2, 131.2, 135.2, 145.3, 148.6, 154.7, 158.6, 160.5, 162.9, 189.4</td>
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### EJ-231

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<th>Molecular Formula</th>
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<th>3-(2-(4-cyclopentyl-1<strong>H</strong>-1,2,3-triazol-1-yl)acetyl)-2<strong>H</strong>-chromen-2-one</th>
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<tr>
<td>( ^1)H NMR δ ppm (DMSO-d\textsubscript{6})</td>
<td>0.73-0.74 (m, 4H, CH\textsubscript{2}), 0.91-0.93(m, 4H, CH\textsubscript{2}), 2.01 (s,1H, CH\textsubscript{*}), 5.93(s, 2H,CH\textsubscript{2}), 7.44-7.48(t,1H, ArH), 7.53(d, 1H, J=8.4Hz, ArH), 7.78-7.83(m, 2H, ArH), 8.04(d, 1H, j=7.2Hz, ArH) 8.84(s, 1H, ArH)</td>
<td></td>
</tr>
<tr>
<td>( ^{13})C NMR δ ppm (DMSO-d\textsubscript{6})</td>
<td>6.5, 7.6, 38.8, 39.0, 39.2, 39.4, 39.6, 39.9, 40.1, 58.1, 116.2, 118.0, 122.2, 122.4, 125.2, 131.1, 135.2, 148.5, 154.6, 158.5, 189.6.</td>
<td></td>
</tr>
<tr>
<td>FT—IR ν\textsubscript{max} cm\textsuperscript{-1}</td>
<td>3045, 3020, 2974, 1748,1700, 1480, 1398, 1024,759.</td>
<td></td>
</tr>
</tbody>
</table>

### EJ-232

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th><strong>C</strong>\textsubscript{23}<strong>H</strong>\textsubscript{21}<strong>N</strong>\textsubscript{3}<strong>O</strong>\textsubscript{3}</th>
<th>3-(2-(4-(4-(tert-butyl)phenyl)-1<strong>H</strong>-1,2,3-triazol-1-yl)acetyl)-2<strong>H</strong>-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.P.</td>
<td>290-292 °C</td>
<td><img src="image" alt="Molecular Structure" /></td>
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<tr>
<td>Mol. wt.</td>
<td>387.43</td>
<td></td>
</tr>
<tr>
<td>Elemental Analysis</td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>Cal</td>
<td>71.30</td>
<td>5.46</td>
</tr>
<tr>
<td>Obs</td>
<td>71.21</td>
<td>5.49</td>
</tr>
<tr>
<td>( ^1)H NMR δ ppm (DMSO-d\textsubscript{6})</td>
<td>1.25(s, 9H, CH\textsubscript{3}), 6.07 (s, 2H, CH\textsubscript{2}), 7.45-7.50 (m, 3H, ArH), 7.54(d, 1H, j=7.6Hz, ArH), 7.78-7.82(m, 3H, ArH), 8.46(s, 1H, ArH),8.03(d, j=7.6Hz, ArH), 8.8(s, 1H, ArH)</td>
<td></td>
</tr>
<tr>
<td>( ^{13})C NMR δ ppm (DMSO-d\textsubscript{6})</td>
<td>31.0, 34.3, 58.3, 116.2, 118.0, 122.1, 122.6, 124.9, 125.2, 125.4, 125.6, 127.9, 131.2, 135.2, 146.1, 148.6, 150.3, 154.7, 158.6, 189.5.</td>
<td></td>
</tr>
<tr>
<td>FT—IR ν\textsubscript{max} cm\textsuperscript{-1}</td>
<td>3298, 3021, 2850, 1740, 1680, 1542, 1352, 1074.</td>
<td></td>
</tr>
</tbody>
</table>
### EJ-237

<table>
<thead>
<tr>
<th>6-bromo-3-(2-(4-phenyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
</tr>
<tr>
<td><strong>Mol. wt.</strong></td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
</tr>
<tr>
<td>Cal</td>
</tr>
<tr>
<td>Obs</td>
</tr>
<tr>
<td><strong>\textsuperscript{1}H NMR δ ppm (DMSO-d\textsubscript{6})</strong></td>
</tr>
<tr>
<td><strong>\textsuperscript{13}C NMR δ ppm (DMSO-d\textsubscript{6})</strong></td>
</tr>
<tr>
<td><strong>FT—IR \textnu\textsubscript{max} cm\textsuperscript{-1}</strong></td>
</tr>
</tbody>
</table>

### EJ-238

<table>
<thead>
<tr>
<th>6-bromo-3-(2-(4-cyclohexyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
</tr>
<tr>
<td><strong>Mol. wt.</strong></td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
</tr>
<tr>
<td>Cal</td>
</tr>
<tr>
<td>Obs</td>
</tr>
<tr>
<td><strong>\textsuperscript{1}H NMR δ ppm (DMSO-d\textsubscript{6})</strong></td>
</tr>
<tr>
<td><strong>\textsuperscript{13}C NMR δ ppm (DMSO-d\textsubscript{6})</strong></td>
</tr>
<tr>
<td><strong>FT—IR \textnu\textsubscript{max} cm\textsuperscript{-1}</strong></td>
</tr>
<tr>
<td><strong>EJ-239</strong></td>
</tr>
<tr>
<td>---</td>
</tr>
<tr>
<td><strong>Molecular Formula</strong></td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
</tr>
<tr>
<td><strong>Mol. wt.</strong></td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
</tr>
<tr>
<td><strong>Cal</strong></td>
</tr>
<tr>
<td><strong>Obs</strong></td>
</tr>
<tr>
<td><strong>^1H NMR δ ppm</strong> (DMSO-d&lt;sub&gt;6&lt;/sub&gt;)</td>
</tr>
<tr>
<td><strong>^13C NMR δ ppm</strong> (DMSO-d&lt;sub&gt;6&lt;/sub&gt;)</td>
</tr>
<tr>
<td><strong>FT—IR ν&lt;sub&gt;max&lt;/sub&gt; cm&lt;sup&gt;-1&lt;/sup&gt;</strong></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>EJ-240</strong></th>
<th><strong>6-bromo-3-(2-(4-cyclopentyl-1H,1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
<td>C_{18}H_{16}BrN_{3}O_{3}</td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
<td>261-263 °C</td>
</tr>
<tr>
<td><strong>Mol. wt.</strong></td>
<td>402.24</td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
<td>C</td>
</tr>
<tr>
<td><strong>Cal</strong></td>
<td>53.75</td>
</tr>
<tr>
<td><strong>Obs</strong></td>
<td>53.87</td>
</tr>
<tr>
<td><strong>^1H NMR δ ppm</strong> (DMSO-d&lt;sub&gt;6&lt;/sub&gt;)</td>
<td>1.57-1.67(m, 6H, CH&lt;sub&gt;3&lt;/sub&gt;), 1.94(d, 2H, CH&lt;sub&gt;2&lt;/sub&gt;),2.74- 2.83(m, 1H, CH*), 5.90(d, 2H, CH&lt;sub&gt;2&lt;/sub&gt;), 7.07-7.12(t, 2H, ArH), 7.60-7.62(d, 1H, J=8.8Hz, ArH), 7.71(s, 1H, ArH), 8.06(s, 1H, ArH)</td>
</tr>
<tr>
<td><strong>^13C NMR δ ppm</strong> (DMSO-d&lt;sub&gt;6&lt;/sub&gt;)</td>
<td>26.1, 26.2, 34.0, 34.7, 36.7, 48.8, 118.9, 120.1, 121.3, 122.4, 124.2, 131.1, 135.2, 135.6, 136.6, 154.1, 160.9, 180.5</td>
</tr>
<tr>
<td><strong>FT—IR ν&lt;sub&gt;max&lt;/sub&gt; cm&lt;sup&gt;-1&lt;/sup&gt;</strong></td>
<td>3055, 3000, 2964, 1746, 1738,1690, 1481, 1398, 1054,759, 597.</td>
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</tbody>
</table>
### EJ-241

<table>
<thead>
<tr>
<th>EJ-241</th>
<th>6-bromo-3-(2-(4-(4-(tert-butyl)phenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
<td>C_{23}H_{20}BrN_{3}O_{3}</td>
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<tr>
<td><strong>M.P.</strong></td>
<td>280-282 °C</td>
</tr>
<tr>
<td><strong>Mol. wt.</strong></td>
<td>466.33</td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
<td></td>
</tr>
<tr>
<td>Cal</td>
<td>59.24</td>
</tr>
<tr>
<td>Obs</td>
<td>59.17</td>
</tr>
</tbody>
</table>

**{^1}H NMR δ ppm** (DMSO-d<sub>6</sub>)

1.12 (s, 9H, CH<sub>3</sub>), 6.05 (s, 2H, CH<sub>2</sub>), 7.09 (d, 1H, J=7.6Hz, ArH), 7.37 (d, 2H, J=8.0Hz, ArH), 7.52 (d, 2H, J=6.4Hz, ArH), 7.60 (d, 1H, J=7.2Hz, ArH), 7.73 (d, 1H, J=8.8Hz, ArH), 7.90 (s, 1H, ArH), 8.36 (s, 1H, ArH)

**{^{13}}C NMR δ ppm** (DMSO-d<sub>6</sub>)

29.2, 29.5, 29.7, 34.3, 50.3, 118.9, 120.1, 121.3, 122.4, 124.0, 124.2, 125.9, 127.0, 127.6, 131.1, 132.0, 135.6, 147.7, 152.5, 154.1, 160.9, 180.5.

**FT—IR ν<sub>max</sub> cm<sup>-1</sup>**

3290, 3021, 2850, 1740, 1680, 1542, 1352, 1074, 526

### EJ-242

<table>
<thead>
<tr>
<th>EJ-242</th>
<th>6-bromo-3-(2-(4-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
<td>C_{19}H_{11}BrFN_{3}O_{3}</td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
<td>261-263 °C</td>
</tr>
<tr>
<td><strong>Mol. wt.</strong></td>
<td>428.21</td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
<td></td>
</tr>
<tr>
<td>Cal</td>
<td>53.29</td>
</tr>
<tr>
<td>Obs</td>
<td>53.11</td>
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</tbody>
</table>

**{^1}H NMR δ ppm** (DMSO-d<sub>6</sub>)

5.92 (s, 2H, CH<sub>2</sub>), 7.09-7.13 (t, 3H, ArH), 7.56-7.62 (m, 3H, ArH), 7.71 (s, 1H, ArH), 7.81 (s, 1H, ArH), 8.31 (s, 1H, ArH)

**{^{13}}C NMR δ ppm** (DMSO-d<sub>6</sub>)

50.3, 115.9, 118.9, 120.1, 121.3, 122.4, 124.2, 128.5, 129.8, 131.1, 132.0, 135.6, 147.7, 154.1, 160.9, 162.3, 180.5.

**FT―IR ν<sub>max</sub> cm<sup>-1</sup>**

3027, 2935, 1729, 1681, 1651, 1429, 1188.1056, 567.
<table>
<thead>
<tr>
<th><strong>EJ-243</strong></th>
<th>6-bromo-3-(2-(4-(p-tolyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
<td>C₂₉H₁₄BrN₃O₃</td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
<td>271-273 °C</td>
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<tr>
<td><strong>Mol. wt.</strong></td>
<td>424.25</td>
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<td><strong>Elemental Analysis</strong></td>
<td>C</td>
</tr>
<tr>
<td><strong>Cal</strong></td>
<td>56.62</td>
</tr>
<tr>
<td><strong>Obs</strong></td>
<td>56.48</td>
</tr>
<tr>
<td><strong>¹H NMR δ ppm (DMSO-d₆)</strong></td>
<td>2.24(s, 3H, CH₃), 6.01(s, 2H, CH₂), 7.12(s, 1H, ArH), 7.29(d, 2H, J=7.6Hz, ArH), 7.45(s, 1H, ArH), 7.52(d, 2H, J=10.4Hz), 7.62(d, 1H, J=8.4Hz), 7.77(s, 1H, ArH), 8.28(s, 1H, ArH)</td>
</tr>
<tr>
<td><strong>¹³C NMR δ ppm (DMSO-d₆)</strong></td>
<td>21.1, 50.3, 118.9, 120.1, 121.3, 122.4, 124.2, 127.8, 129.0, 129.1, 131.1, 131.5, 132.0, 135.6, 135.8, 147.7, 154.1, 160.9, 180.5</td>
</tr>
<tr>
<td><strong>FT—IR νₘₐₓ cm⁻¹</strong></td>
<td>3312, 3042, 2925, 1748, 1675, 1364, 1140, 531</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>EJ-246</strong></th>
<th>3-(2-(4-(p-tolyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
<td>C₂₀H₁₅N₃O₃</td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
<td>276-278 °C</td>
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<tr>
<td><strong>Mol. wt.</strong></td>
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<tr>
<td><strong>Elemental Analysis</strong></td>
<td>C</td>
</tr>
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<td><strong>Cal</strong></td>
<td>69.56</td>
</tr>
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<td><strong>Obs</strong></td>
<td>69.45</td>
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<tr>
<td><strong>¹H NMR δ ppm (DMSO-d₆)</strong></td>
<td>2.31(s, 3H, CH₃), 6.07(s, 2H, CH₂), 7.26(d, 2H, J=7.6Hz, ArH), 7.45-7.48(t, 1H, ArH), 7.53(d,1H, J=8.4Hz, ArH), 7.75(d,2H, J=7.6Hz, ArH), 7.80-7.84(t, 1H, ArH), 8.03(d, 1H, J=6.8Hz, ArH), 8.5(s, 1H, ArH), 9.0(s,1H, ArH).</td>
</tr>
<tr>
<td><strong>¹³C NMR δ ppm (DMSO-d₆)</strong></td>
<td>180.5, 160.9, 154.1, 147.7, 135.8, 132.8, 132.0, 131.5, 129.5, 129.1, 129.1, 127.8, 127.8, 125.0, 123.9, 122.1, 120.6, 117.1, 50.3, 21.1</td>
</tr>
<tr>
<td><strong>FT—IR νₘₐₓ cm⁻¹</strong></td>
<td>3300, 3010, 2866, 1796, 1556, 1395, 1240</td>
</tr>
</tbody>
</table>
### EJ-247

3-(2-(4-(4-propylphenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>C$<em>{22}$H$</em>{19}$N$_3$O$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.P.</td>
<td>271-273 °C</td>
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<td>Mol. wt.</td>
<td>373.40</td>
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<td>Elemental Analysis</td>
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<td>Cal</td>
<td>70.76 5.13 11.25</td>
</tr>
<tr>
<td>Obs</td>
<td>70.70 5.08 11.17</td>
</tr>
<tr>
<td>$^1$H NMR δ ppm (DMSO-d$_6$)</td>
<td>0.87-0.93(m, 3H, CH$_3$), 1.61-1.63(m, 2H, CH$_2$), 2.57-2.60(m, 2H, CH$_2$), 6.07(s, 2H, CH$_2$), 7.27(d, 2H, J=8.4Hz, ArH), 7.45-7.49(m, 1H, ArH), 7.54(d, 2H, J=8.0Hz, ArH), 7.77-7.84(m, 2H, ArH), 8.03(d, 1H, J=6.4Hz, ArH), 8.45(s, 1H, ArH), 8.88(s, 1H, ArH).</td>
</tr>
<tr>
<td>$^{13}$C NMR δ ppm (DMSO-d$_6$)</td>
<td>12.9, 24.4, 38.1, 50.3, 117.1, 120.6, 122.1, 123.9, 125.0, 125.6, 127.5, 129.5, 132.0, 132.3, 132.8, 142.5, 147.7, 154.1, 160.9, 180.5.</td>
</tr>
<tr>
<td>FT-IR ν$_{max}$ cm$^{-1}$</td>
<td>3321, 3030, 2924, 1735, 1670, 1425, 1342, 1002.</td>
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</tbody>
</table>

### EJ-248

3-(2-(4-(cyclopropyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>C$<em>{16}$H$</em>{13}$N$_3$O$_3$</th>
</tr>
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<td>M.P.</td>
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<td>Mol. wt.</td>
<td>295.29</td>
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<td>Elemental Analysis</td>
<td></td>
</tr>
<tr>
<td>Cal</td>
<td>65.08 4.44 14.23</td>
</tr>
<tr>
<td>Obs</td>
<td>65.00 4.37 14.18</td>
</tr>
<tr>
<td>$^1$H NMR δ ppm (DMSO-d$_6$)</td>
<td>0.57-0.64(m, 2H, CH$_2$), 0.76-0.84(m, 2H, CH$_2$), 1.21-1.31(m, 1H, CH*), 5.69(s, 2H, CH$_2$), 6.80(s, 1H, ArH), 7.17 -7.41(m, 2H, ArH), 7.42-7.46(m, 1H, ArH), 7.58-7.61(m, 1H, ArH), 8.06(s, 1H, ArH).</td>
</tr>
<tr>
<td>$^{13}$C NMR δ ppm (DMSO-d$_6$)</td>
<td>7.3, 7.4, 48.8, 117.1, 120.6, 122.1, 123.9, 125.0, 129.5, 132.7, 132.8, 136.0, 154.1, 160.9, 180.5.</td>
</tr>
<tr>
<td>FT-IR ν$_{max}$ cm$^{-1}$</td>
<td>3312, 3020, 2937, 1852, 1738, 1696, 1485, 1398, 1054,</td>
</tr>
</tbody>
</table>
3.7) Spectral data

Mass spectroscopy of 3-(2-(5-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-230)

IR spectroscopy of 3-(2-(5-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-230)
$^{1}H$ NMR spectroscopy of 3-(2-(5-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-230)

$^{13}C$ NMR spectroscopy of 3-(2-(5-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-230)
$^1$H NMR spectroscopy of 3-(2-(5-cyclopentyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ231)

$^{13}$C NMR spectroscopy of 3-(2-(5-cyclopentyl-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ231)
\(^1\)H NMR spectroscopy of 3-(2-(5-(4-(tert-butyl)phenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-232)

IR spectroscopy of 6-bromo-3-(2-(5-phenyl-1H-1,2,3-triazol-1-yl)acetyl) - 2H-chromen-2-one (EJ-237)
Chapter 3

$^1$H NMR Spectroscopy of $3$-(2-(5-(p-tolyl)-1$H$-1,2,3-triazol-1-yl)acetyl)-$2H$-chromen-2-one (EJ-246)

$^{13}$C NMR Spectroscopy of $3$-(2-(5-(p-tolyl)-1$H$-1,2,3-triazol-1-yl)acetyl)-$2H$-chromen-2-one (EJ-246)
$^1$H NMR spectroscopy of 3-(2-(5-(4-propylphenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-247)

$^{13}$C NMR spectroscopy of 3-(2-(5-(4-propylphenyl)-1H-1,2,3-triazol-1-yl)acetyl)-2H-chromen-2-one (EJ-247)
3.8) Conclusion

In this chapter, New triazole derivatives have been synthesized 1,3-dipolar cycloaddition reaction between the acetylene group of and azide takes place to produce the bifunctional coumarin triazole products by reported convention method no more further purification required, as the desired product were isolated in 95% yield. The structures of products were derived from their IR, $^1$H NMR, $^{13}$C NMR, mass spectra, and elemental analysis data. Some of the compounds, thus synthesized were screened for their antitubercular activity. Detailed discussion is given in Chapter -6.
Chapter 3

3.9) Reference


47. Feldman, Alina K., BenoitColasson, and Valery V. Fokin. "One-pot synthesis of 1, 4-disubstituted 1, 2, 3-triazoles from in situ generated azides." Organic letters 6.22 (2004): 3897-3899.
