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1,2,4-Triazolyl thioacetamides: Design, synthesis, and photoelectrochemical properties

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Attempts to synthesize *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide was unsuccessful *via* cycloaddition reaction between the *N*-phenyl-2-thiocyanatoacetamide and benzohydrazide in the presence of Lewis acid. During this procedure, a side product, 2-(phenylamino)thiazol-4(5*H*)-one is obtained through intramolecular cyclization. However, nucleophilic substitution reactions performed between 5-phenyl-4*H*-1,2,4-triazole-3-thiol and 2-chloro-*N*-arylacetamides in the presence of K_2CO_3 yielded a series of 4*H*-1,2,4-triazole-tethered acetamides in aqueous medium. The procedure affords excellent yields of desired products containing electron-withdrawing and electron-donating groups on the aromatic rings in a short reaction time with ease of operation. The oxidation potential and photocurrent measurements of the targets indicated that the presence of the electron-withdrawing substituents afforded better results with a lower oxidation potential and higher photocurrent measurements.

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1. Introduction

Triazoles are among the most stable azole derivatives. Within this class, 1,2,4-triazoles have recently attracted renewed interest in both organic and medicinal chemistry. This structural motif serves as a fundamental component in numerous pharmaceuticals used in therapy (**Fig. 1**). The 1,2,4-triazole core and their fused heterocyclic derivatives display a wide range of biological activities, including neuroprotective,[1] antioxidant,[2] antimalarial,[3] anti-leishmanial,[4] and anti-urease effects.[5] Furthermore, the versatility of 1,2,4-triazole frameworks has expanded into various fields owing to their structural attribute such as rigidity, hydrogen-bonding capacity, solubility, and dipole moment that facilitate strong interactions with biological and material targets.[6-9] These features underscore 1,2,4-triazoles as key intermediates in both biological and material applications.[10] Extensive efforts have been made to synthesize 1,2,4-triazoles, with a focus on developing methodologies for introducing various functional groups. Typically, 1,2,4-triazoles can be efficiently synthesized by reacting formamide with hydrazine under microwave irradiation, in the absence of a catalyst.[11] A protocol involving the combination of amidines and trialkyl amines in the presence of K_3PO_4 as a base and copper (II) catalyst has been employed to produce 1,3-disubstituted 1,2,4-triazoles.[12] Another environment friendly approach to obtain 1,2,4-triazoles involves the I_2-mediated formation of N–S and C–N bonds from isothiocyanates in water.[13] Furthermore, 1,2,4-triazoles have been synthesized from aliphatic amines and hydrazones in a process catalysed by iodine.[14]

The optical properties of 1,2,4-triazoles can be modulated by introducing different substituents into the triazole core. For instance, the photophysical analysis revealed that the 4*H*-1,2,4-triazole derivative (A) (**Fig. 2**) shows exceptional luminescence with a high quantum yield of emitted photons.[15] The symmetrical 3,6-bis(4-(5-(4-methoxyphenyl)-4-phenyl-4*H*-1,2,4-triazol-3-yl)phenyl)-1,2,4,5-tetrazine (B) displayed intense, broad luminescence with near-unity quantum yield, highlighting the potential of 4*H*-triazole-based compounds in optoelectronic applications.[16] 3-Hydroxypyridinyl-substituted-1,2,4-triazoles (C) exhibited significant fluorescence enhancement through an excited-state intramolecular

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proton transfer (ESIPT) mechanism, making them promising scaffolds for applications in materials science.[17] Monometallic copper (I) complexes with functionalized 3-(2′-pyridyl)-1,2,4-triazole ligands demonstrated luminescent properties, providing insights into designing blue phosphorescent materials with distinct emission characteristics.[18] Beyond their photophysical properties, the 5-aryl-3-phenylamino-1,2,4-triazole (D) showed notable anticancer activity in XTT assays across various cancer cell lines, indicating anticancer property.[19] Fluorescence spectroscopy studies on the nucleoside 1-[(2-hydroxyethoxy)methyl]-5-(*p*-tolylthio)-1*H*-1,2,4-triazole-3-carboxamide (E) demonstrated its potential as a fluorescent chemosensor, showing high selectivity for Hg^{2+} and Pb^{2+} ions through fluorescence quenching.[20,21] The characteristics of 4-[5-(4-chlorophenyl)-4-(4-fluorophenyl)-4*H*-triazol-3-ylsulfanyl]phthalonitrile (F), indicated its potential as photosensitizer for photodynamic therapy.[22] Moreover, specially designed fused triazolo[1,5-*a*] pyridine derivatives[23] and 1,2,4-triazole-based triphenylamine compounds[24] have shown promise as efficient materials for blue fluorescence OLEDs and phosphorescent OLEDs, respectively.

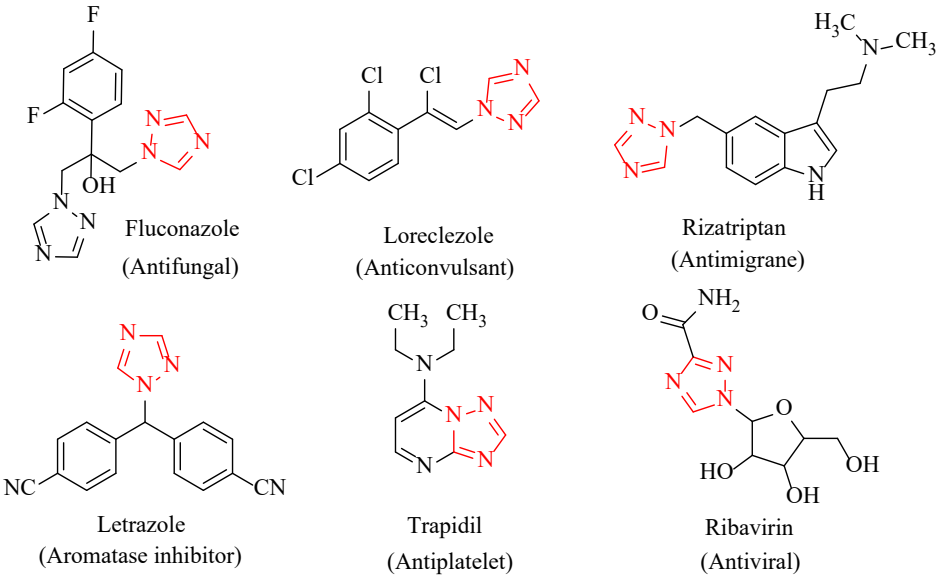


Fig. 1. Drugs with a 1,2,4-triazole scaffold

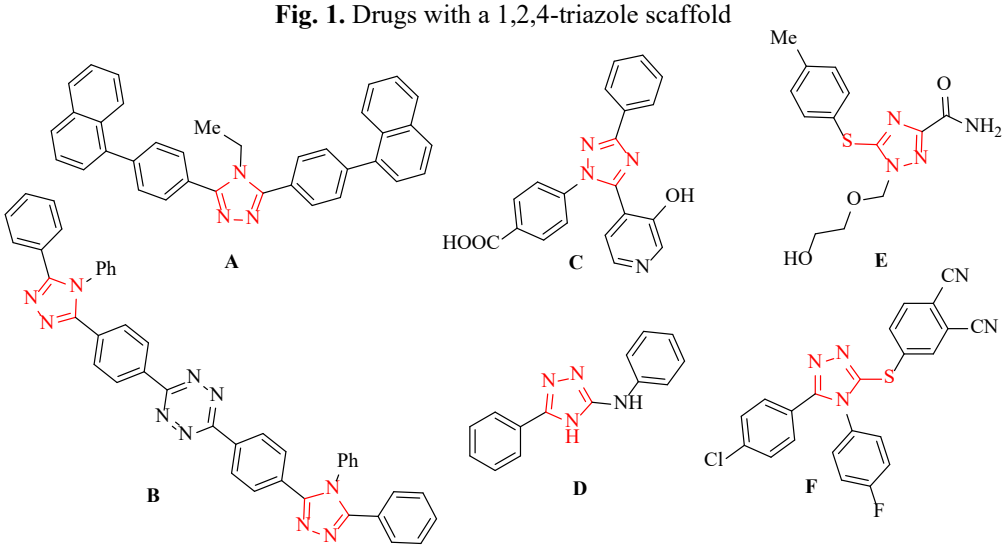


Fig. 2. A few 1,2,4-triazoles having applications

Based on our interests in heterocyclic compounds[25-27] and their photoelectronic properties, the present objective was to modulate the electron density of the isomeric 4*H*-1,2,4-triazole ring by introducing a heteroatom linked at the C3 position and a phenyl group at the C5 position. We hypothesized that the attachment of an *N*-aryl thioacetamide[28] would enable us to control the electronic effects while preserving conformational flexibility (**Fig. 3**). Guided by this instinct, we expanded the scope of this privileged heterocycle by synthesizing several *N*-aryl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamides. These scaffolds were successfully accessed via an eco-friendly, convenient, cost-effective synthetic route, and their preliminary photoelectrochemical properties are discussed.

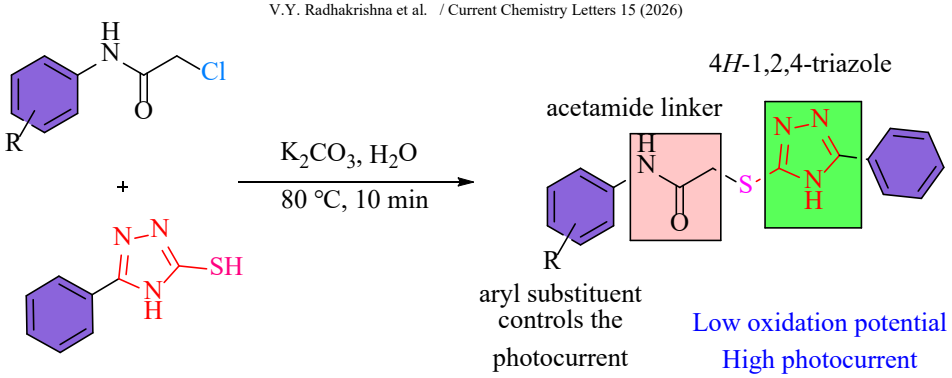
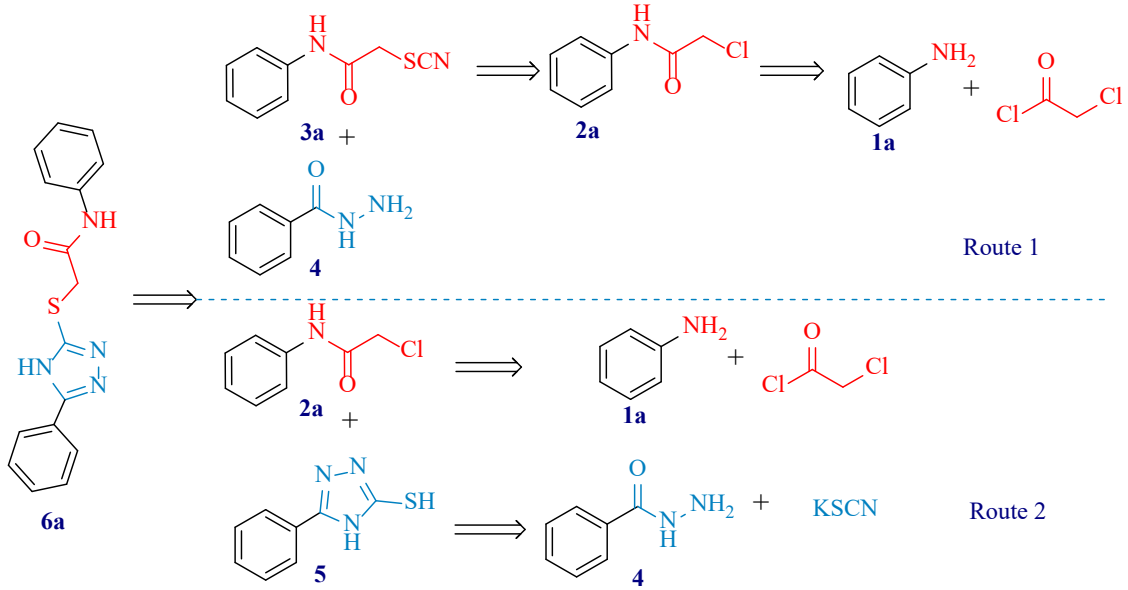


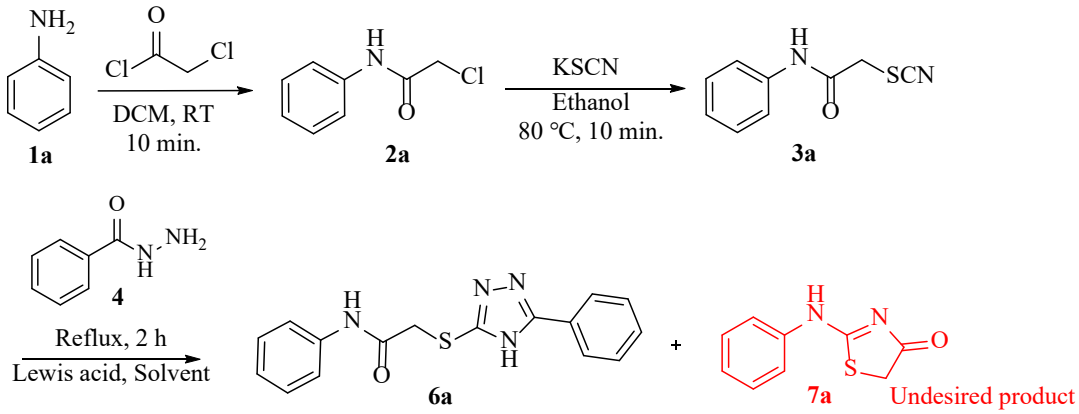
Fig. 3. Rational design of thioacetamide derivatives of 4*H*-1,2,4-triazoles

2. Results and Discussion

The synthesis of *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6a) was devised through retrosynthetic analysis (**Scheme 1**). An important intermediate in this pathway is *N*-phenyl-2-thiocyanatoacetamide (3a), a compound that would serve as a suitable synthetic precursor. This intermediate can be efficiently prepared from 2-chloro-*N*-phenylacetamide (2a), which is obtained by reacting 2-chloroacetyl chloride with aniline (1a)[29,30] followed by nucleophilic substitution with potassium thiocyanate.[31] The formation of the 1,2,4-triazole ring involves a [3+2] cycloaddition between *N*-phenyl-2-thiocyanatoacetamide (3a) and benzohydrazide (4). This 1,3-dipolar cycloaddition would result in the formation of the target compound, *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6a).



Scheme 1. Retrosynthetic analysis of *N*-phenyl thioacetamide linked 1,2,4-triazole

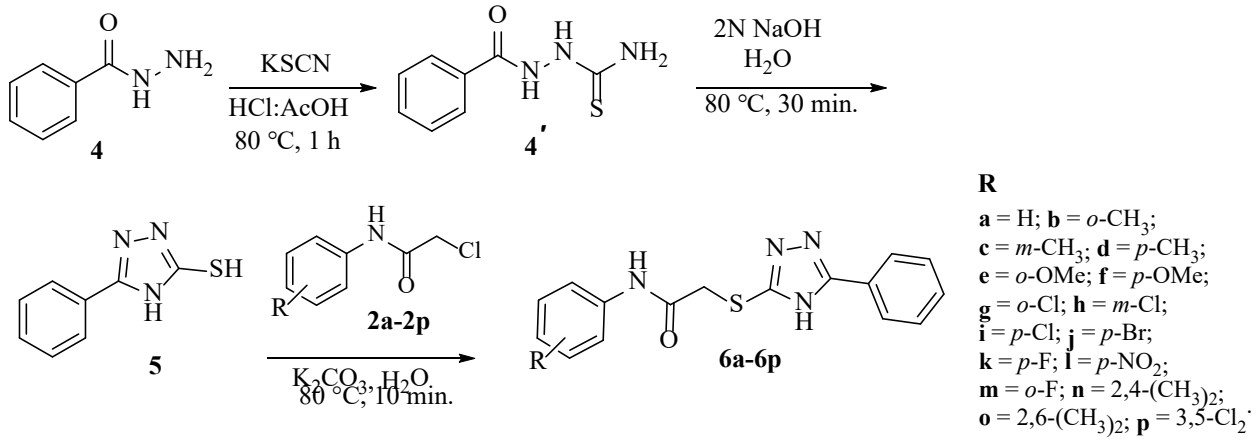


Scheme 2. Strategy 1: Synthesis of *N*-aryl thioacetamide linked 1,2,4-triazoles

Consistent with the retrosynthetic approach outlined in route 1, the synthesis of the target molecule (6a) commenced with a nucleophilic substitution of potassium thiocyanate on 2-chloro-*N*-phenylacetamide (2a), followed by a cycloaddition

reaction (**Scheme 2**). This cycloaddition was performed in the presence of various Lewis acids such as $AlCl_3$, $CuCl_2$, $BF_3{\cdot}OEt_2$, and $ZnCl_2$, using different solvents at reflux temperature for a duration of 2 hours. The dipolar cycloaddition yielded only moderate conversions, with yields ranging from 30% to 55%. During this process, intramolecular cyclization also occurred, producing 2-(phenylamino)thiazol-4(5*H*)-one (7a). Thus, the method for synthesizing the 1,2,4-triazole faced limitations, including lengthy reaction times, formation of significant amounts of side product which restricted substrate scope, and low isolated yield. An alternative retrosynthetic strategy for synthesizing *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6a) was therefore envisaged.

Route 2 signifies the intermediate 5-phenyl-4*H*-1,2,4-triazole-3-thiol (5), a compound well-known for its application in the synthesis of organic materials. This intermediate can be efficiently synthesized via a cycloaddition reaction between benzohydrazide (4) and potassium thiocyanate. The other intermediate required would be 2-chloro-*N*-phenylacetamide (2a). We therefore decided to utilize these two versatile intermediates to construct the target molecule. Benzohydrazide (4) and potassium thiocyanate were reacted in acetic acid with conc. HCl at 80 °C, yielding 2-benzoylhydrazine-1-carbothioamide (4′) for subsequent cyclization (**Scheme 3**). This intermediate was then refluxed in aqueous 2N NaOH for one hour, followed by acidification with conc. HCl, which produced the desired 5-phenyl-4*H*-1,2,4-triazole-3-thiol (5) in excellent yield. Finally, nucleophilic substitution of this thiol intermediate with various 2-chloro-*N*-phenylacetamide (2a-2p) under mild, environment friendly conditions resulted in the formation of the target compounds, *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6a-6p). The nucleophilic substitution reaction was explored using various conditions to expand the substrate scope.



Scheme 3. Strategy 2: Synthesis of *N*-aryl thioacetamide linked 1,2,4-triazoles

The role of the base was examined in terms of its ability to generate the necessary thiolate intermediate for synthesizing the acetamide-functionalized 1,2,4-triazole via nucleophilic substitution.

Table 1. Conditions for the synthesis of *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamides

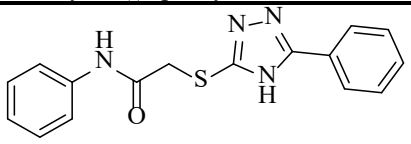
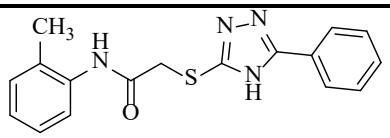
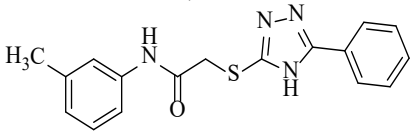
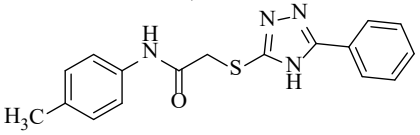
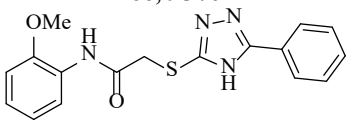
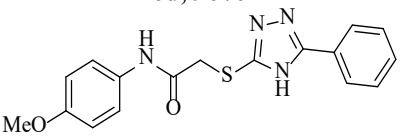
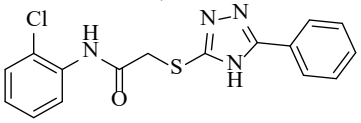
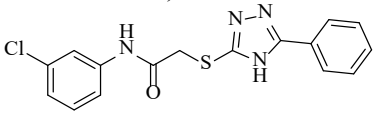
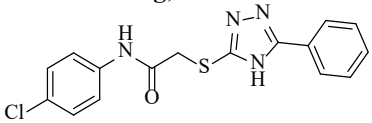
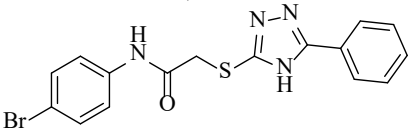
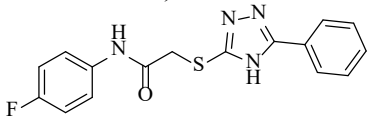
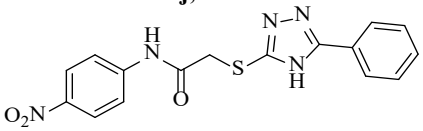
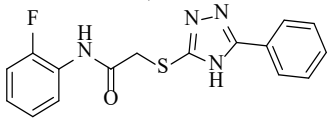
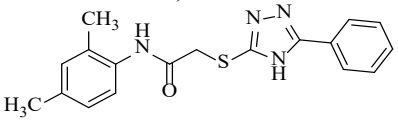
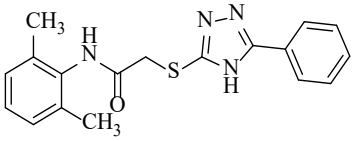
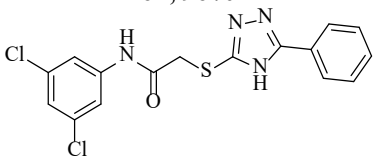
Entry	Base	Solvent	Temp (°C)	Time (min)	Yield (%)
1	TEA	DCM	40	45	65
2	DIPEA	EtOH	80	30	68
3	Pyridine	EtOH	80	25	74
4	NaH	THF	0-RT	35	35
5	NaOMe	MeOH	60	20	72
6	K_2CO_3	EtOH	80	10	96
7	Na_2CO_3	EtOH	80	10	88
8	$NaHCO_3$	EtOH	80	15	82
9	K_2CO_3	Toluene	100	45	60
10	K_2CO_3	THF/Dioxane	60	30	72
11	K_2CO_3	Acetonitrile	80	20	84
12	K_2CO_3	DMSO	100	10	87
13	K_2CO_3	DMF	100	10	85
14	K_2CO_3	Water	80/100	10	97
15	K_2CO_3	Water	RT	60	40
16	K_2CO_3	Water	50	40	82

5-Phenyl-4*H*-1,2,4-triazole-3-thiol (1.0 mmol), 2-chloro-*N*-phenylacetamide (1.0 mmol), base (1.1 mmol), solvent (10 mL)

Screening of bases was carried out at different temperatures (**Table 1, Entries 1–9**), and in all the cases moderate to good results were obtained. Inorganic bases surpassed organic bases in performance. Using triethylamine (TEA) resulted in a 65% yield in 45 minutes, whereas diisopropylethylamine (DIPEA) shortened the reaction time, in ethanol at 80 °C. The heterocyclic aromatic base pyridine provided even higher yields with shorter reaction times compared to TEA and DIPEA. Conversely, the non-nucleophilic base NaH gave unsatisfactory results; however, significant improvement was observed with the nucleophilic base NaOMe in methanol, prompting further experiments with inorganic bases. Notably, the bases $NaHCO_3$ and Na_2CO_3 enhanced reaction efficiency, achieving yields of 82% and 88% respectively, within 10 to 15 minutes. In ethanol, K_2CO_3 demonstrated a superior ionic character, affording 96% yield in 10 minutes. Based on this optimal performance, K_2CO_3 in ethanol medium was selected for the reactions between 5-phenyl-4*H*-1,2,4-triazole-3-thiol (5) and 2-chloro-*N*-arylacetamides 2(a-p).

Polar solvents are expected to enhance the rate of the nucleophilic substitution reaction between 5-phenyl-4*H*-1,2,4-triazole-3-thiolate (5′) and 2-chloro-*N*-phenylacetamide (2a). Consequently, solvent screening was performed using K_2CO_3 as the base (**Table 1, Entries 9–14**). Initial reaction with the non-polar solvent toluene at 100 °C showed moderate progress while the ether solvents such as THF and 1,4-dioxane produced improved results within 30 minutes.

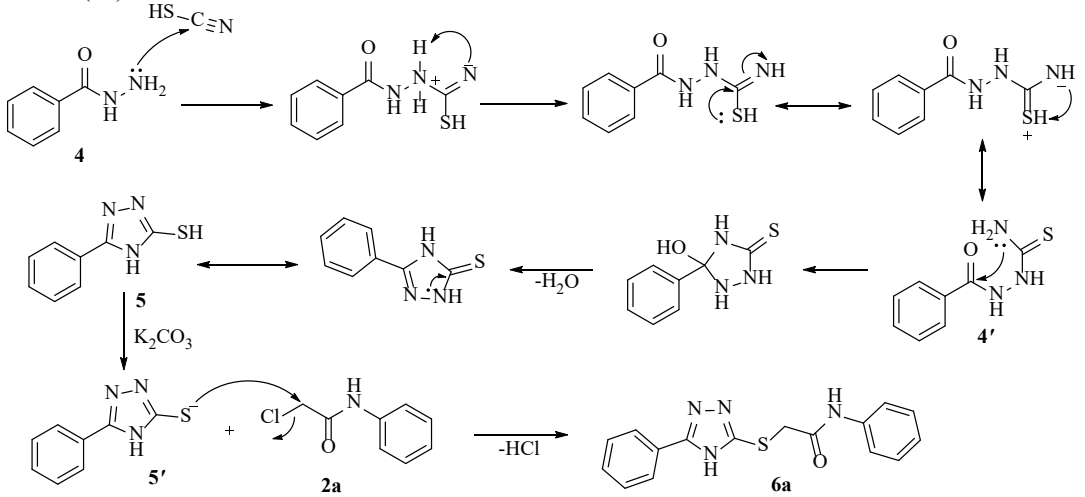
Table 2. Synthesised *N*-aryl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamides

1		**2**	
	6a, 97%		**6b, 95%**
3		**4**	
	6c, 95%		**6d, 96%**
5		**6**	
	6e, 95%		**6f, 96%**
7		**8**	
	6g, 95%		**6h, 94%**
9		**10**	
	6i, 95%		**6j, 93%**
11		**12**	
	6k, 93%		**6l, 90%**
13		**14**	
	6m, 92%		**6n, 96%**
15		**16**	
	6o, 97%		**6p, 94%**

Notable yields were achieved with acetonitrile, while DMSO and DMF delivered good yields within just 10 minutes. When conducted in aqueous media, the reaction proceeded smoothly and provided high yields in a short time, comparable to that in ethanol. As a greener, polar protic solvent, water's capacity to engage in hydrogen bonding and facilitate strong solute-solvent interactions contribute to additional stabilization of the reaction intermediate. Based on these solvent studies, water was identified as the most suitable medium for promoting the nucleophilic substitution, achieving a 97% overall yield in only 10 minutes.

At room temperature, the reaction proceeded slowly, reaching only 40% yield after one hour. Increasing the temperature to 50 °C significantly improved the yield and decreased the reaction time. Elevated temperatures facilitate a greater number of reactant molecules to overcome the activation energy barrier, allowing for the rapid formation of the desired product with excellent yield when water is used as the solvent. Under the optimized conditions, various 2-chloro-*N*-arylacetamides (2a–p) were reacted with 5-phenyl-4*H*-1,2,4-triazole-3-thiol (5). Substituents on the aryl ring had minimal impact on the reaction rate; both electron-donating and electron-withdrawing groups on 2-chloro-*N*-arylacetamides were well tolerated, yielding excellent results (**Table 2**). Scalability was demonstrated by reacting one gram of 2-chloro-*N*-phenylacetamide (2a) with stoichiometrically equivalent amount of 5-phenyl-4*H*-1,2,4-triazole-3-thiol (5). The products were characterized by ^{1}H NMR, ^{13}C NMR, and HRMS spectral analyses.

Based on the experimental findings, a plausible reaction mechanism is proposed (**Scheme 4**). Initially, potassium thiocyanate reacts with conc. HCl in acetic acid to generate thiocyanic acid, which facilitates the nucleophilic attack by benzohydrazide, leading to the formation of 2-benzoylhydrazine-1-carbothioamide (4′) through tautomerization of 2-benzoylhydrazine-1-carbimidothioic acid. Following this, intramolecular cyclization occurs via the attack of the lone pair electrons on the terminal nitrogen of the thioamide to the carbonyl carbon of the benzamide, resulting in the formation of 5-phenyl-4*H*-1,2,4-triazole-3-thiol (5) through dehydration. Thereafter, potassium carbonate abstracts a proton from the compound (5), generating the nucleophilic species 5-phenyl-4*H*-1,2,4-triazole-3-thiolate (5′), which subsequently attacks the 2-chloro-*N*-phenylacetamide (2a), yielding the target compound *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6a).



Scheme 4. A plausible mechanism for the formation of 1,2,4-triazole tethered *N*-phenylacetamide

Photoactive organic compounds have garnered considerable interest due to their diverse applications.[32-35] Both molecular and polymeric photoactive organic materials can serve as electron acceptors, enabling efficient photocurrent generation and offering significant potential for the development of monochromatic light-to-current molecular converters.[36-38] For instance, triphenylamine-thienothiophene derivatives have been studied as high-performance charge transport materials in hybrid perovskite solar cells.[39] Inspired by these prospects, we investigated the electrooxidation potentials of *N*-aryl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide. Linear sweep voltammograms (LSVs) were recorded for these substrates in dry acetonitrile containing tetrabutylammonium hexafluorophosphate as the supporting electrolyte, utilizing a three-electrode configuration comprising a glassy carbon disc as the working electrode, a silver wire as the quasi-reference electrode, and platinum as the counter electrode.[40] **Fig. 4(i)** presents the LSVs of the regioisomeric methyl-substituted derivatives 6b, 6c, and 6d, during the anodic sweep. Notable steep increases in current and elevated current densities were observed, indicating the oxidation of the compounds. The ortho-substituted electron-donating methyl and methoxy derivatives, 6b and 6e, showed lower onset oxidation potentials and enhanced electrochemical activity compared to the meta- and para-substituted derivatives 6c, 6d, and 6f (**Fig. 4(i,ii)**). Conversely, the *N*-phenyl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide 6a exhibited a markedly higher onset oxidation potential (exceeding 1.6 V) coupled with reduced current density. As shown in **Fig. 4(iii)**, the regioisomeric chloro-substituted derivatives 6g, 6h, and 6i demonstrated distinct oxidation behaviors, with the *o*-chlorophenyl derivative 6g exhibiting a more anodic oxidation potential compared to the *m*-chlorophenyl (6h) and *p*-chlorophenyl (6i) analogues. Similarly, among the halogen derivatives 6i, 6j, and 6k, the *p*-bromo compound 6j displayed

the lowest oxidation potential (**Fig. 4(iv)**). All these compounds underwent irreversible oxidation, as no Faradaic current peaks were observed during cathodic polarization in cyclic voltammetry. **Figs. 4(v,vi)** present the cyclic voltammograms of 6d and 6i over 1-5 cycles, confirming the absence of reduction peaks during reverse scans. This irreversible behavior aligns with the typical oxidation pathway of amines, which involves radical cation formation and leads to irreversibly oxidized products.[41] Overall, the results indicate that electronic effects of substituents significantly impact both the oxidation potential[42] and current density, reflecting changes in the reaction kinetics.

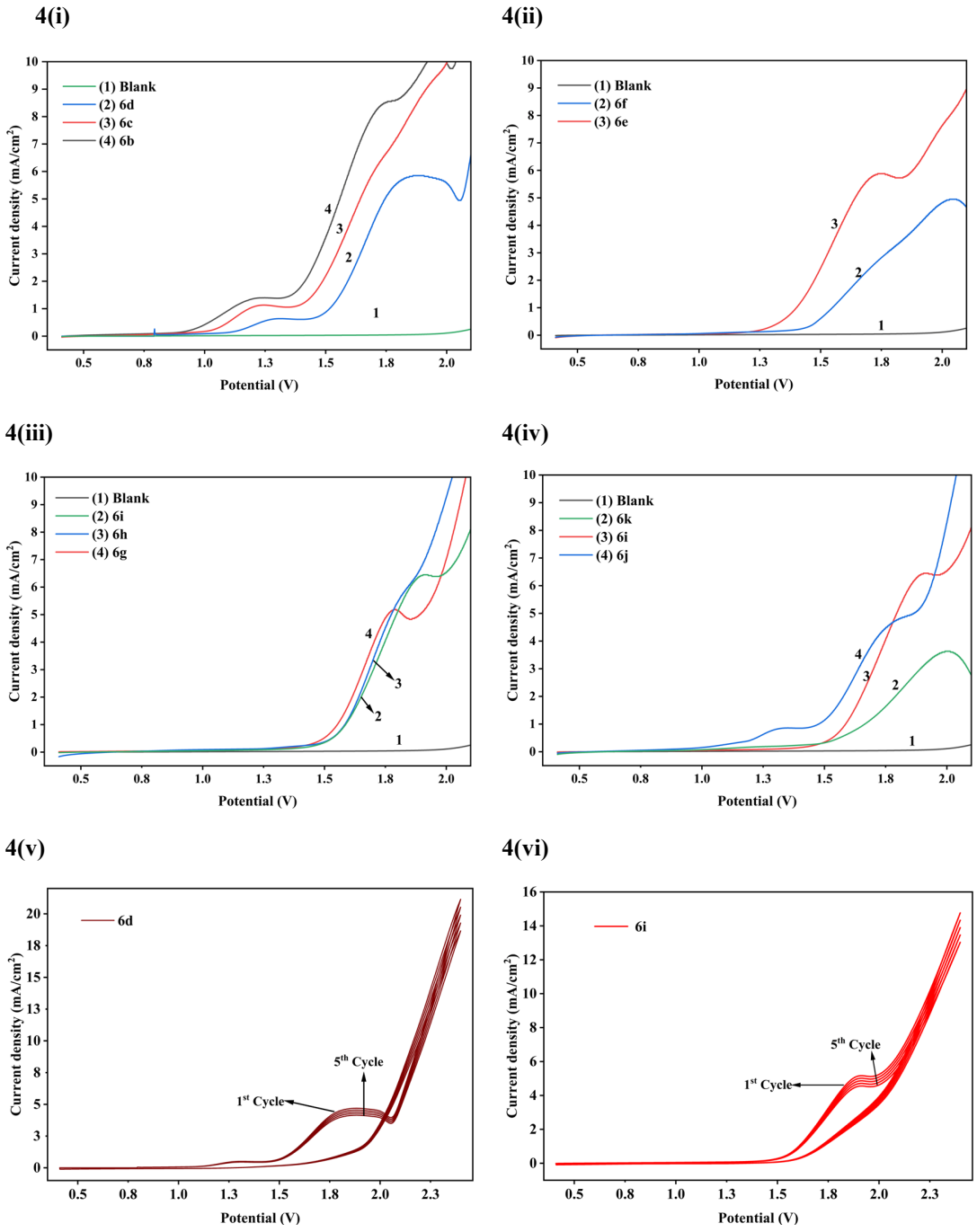


Fig. 4. (i-iv) Linear sweep voltammograms of respective compounds in acetonitrile containing 0.1 M tetrabutylammonium hexafluorophosphate at the scan rate of 20 mV sec^{-1}, potential (V) versus Ag/AgCl,
(v) Cyclic voltammogram of 6d from 1 to 5 cycles, (vi) Cyclic voltammogram of 6i from 1 to 5 cycles.

The photoelectrochemical properties of the 1,2,4-triazole derivatives were assessed using a three-electrode setup. The photoelectrodes were prepared by coating the respective compounds, dispersed with a Nafion binder, onto transparent fluorine-doped tin oxide (FTO) substrates. Back-side illumination was carried out using a xenon lamp source with an intensity of 100 mW/cm².[43] Photocurrent and photovoltage measurements revealed the p-type semiconducting nature of the materials at the aqueous interface. **Fig. 5** illustrates the photocurrent responses obtained under a constant applied bias during repeated light on-off cycles. Among the examined derivatives, the unsubstituted compound 6a exhibited the lowest photocurrent (~210 nA/cm²), similar to that of the electron-donating *p*-methylphenyl derivative 6d. Substituents on the aryl

ring notably affected the response: the *p*-bromophenyl derivative 6j produced a substantially higher photocurrent (~900 nA/cm²) compared to the *p*-chlorophenyl analogue 6i. Conversely, the *p*-methoxyphenyl derivative 6f, being electron-donating, showed a reduced photocurrent relative to the other compounds. These findings highlight the significant influence of aryl substituents on modulating the photoelectrochemical performance of the materials. Our ongoing research efforts are focused on customizing the triazole core with various chromophoric groups to enhance photoactivity, aiming for potential applications in organic photoconversion devices.

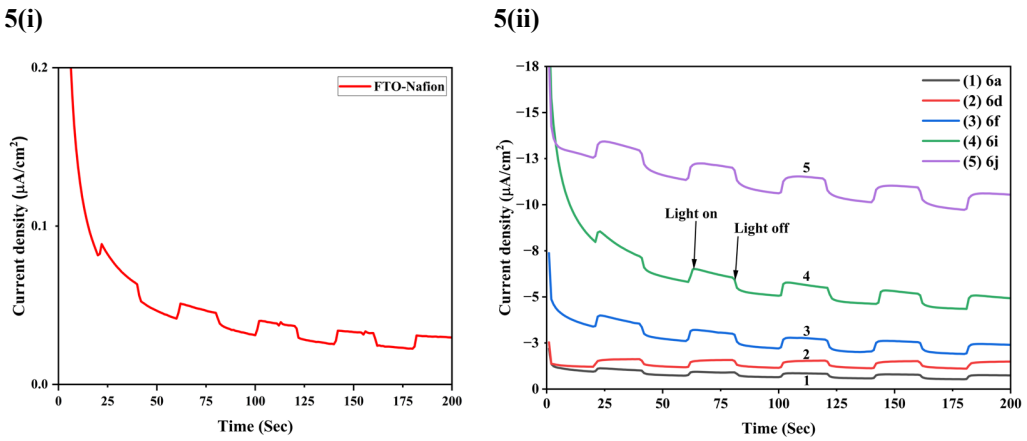


Fig. 5. (i) Photocurrent responses of Nafion on FTO substrate.
(ii) Photocurrent responses of 6a, 6d, 6f, 6i, 6j

3. Conclusions

An efficient and eco-friendly synthetic route was developed for the preparation of substituted *N*-aryl-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamides from 5-phenyl-4*H*-1,2,4-triazole-3-thiol and 2-chloro-*N*-arylacetamides *via* K_2CO_3 mediated nucleophilic substitution in water. The reaction proceeds smoothly with both electron-donating and electron-withdrawing substituents on the aryl ring, delivering excellent yields within short reaction times. This method utilizes inexpensive reagents under environmentally benign conditions, making it attractive for practical applications and scalable synthesis. Electrooxidation and photocurrent studies of the 1,2,4-triazole derivatives revealed that para-substituted electron-withdrawing groups lower the onset oxidation potential while enhancing photocurrent response. Further structural modifications are underway to improve both biological and photophysical properties.

Acknowledgment

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Supporting Information

^{1}H & ^{13}C NMR Spectra and High-Resolution Mass Spectra of all the synthesized compounds can be found in the Electronic Supplementary Content of this article.

Conflict of Interest

The authors declare no conflict of interest either of a financial or personal nature.

4. Experimental

4.1. Materials and Methods

All chemicals were obtained from commercial suppliers and used without further purification. Reactions were conducted in oven-dried glassware under appropriate atmospheric conditions. The progress of reactions was monitored by thin-layer chromatography (TLC) on 0.25 mm Merck Silica Gel 60 F254 plates, with visualization under UV light. Column chromatography was performed on silica gel (60-120 mesh) using hexane and ethyl acetate as eluents. NMR spectra were recorded on a Jeol ECZ-400R spectrometer (^{1}H at 400 MHz and ^{13}C at 100 MHz) in DMSO-*d6*, with tetramethylsilane (TMS) as the internal standard. Chemical shifts (δ) are reported relative to residual solvent peaks (δ = 2.5 ppm for ^{1}H NMR; δ = 39.5 ppm for ^{13}C NMR, triplet). High-resolution mass spectrometry (HRMS) analyses were carried out using an electrospray ionization (ESI) quadrupole time-of-flight (QTOF) mass spectrometer (Agilent).

4.2. General procedure

Synthesis of 5-phenyl-4H-1,2,4-triazole-3-thiol (5)

To a solution of benzohydrazide **4** (0.14 g, 1.0 mmol) in a conc. HCl: acetic acid mixture (1:2), potassium thiocyanate (0.12 g, 1.2 mmol) was added. The reaction mixture was stirred in an oil bath at 80 ℃ for 1 hour. Upon completion, as monitored by thin-layer chromatography (TLC), the mixture was cooled to room temperature and poured into ice-cold water, resulting in precipitation of the product. The solid was collected by filtration and washed thoroughly with ice-cold water. The crude product was then refluxed in 2N NaOH solution for 30 minutes. After completion, as confirmed by TLC, the mixture was cooled to room temperature and neutralized with ice-cold 2N HCl. The precipitated solid was filtered, washed with cold water, and dried. The resulting crude product was used directly in subsequent synthesis without further purification.

Synthesis of N-aryl-2-((5-phenyl-4H-1,2,4-triazol-3-yl)thio)acetamide (6a-p)

To a solution of 5-phenyl-4*H*-1,2,4-triazole-3-thiol **5** (0.18 g, 1.0 mmol) in water (10 mL), 2-chloro-*N*-arylacetamide **2(a-p)** (1.0 mmol) and K_2CO_3 (0.15 g, 1.1 mmol) were added. The reaction mixture was stirred in an oil bath at 80 ℃ for 10 minutes. After completion, as confirmed by TLC, the mixture was cooled to room temperature, and the precipitated product was collected by filtration, washed thoroughly with ice-cold water, and purified by column chromatography on silica gel (60-120 mesh) using ethyl acetate-hexane (20:80) as the eluent, yielding the desired product.

4.3. Physical and Spectral Data

_N_-phenyl-2-((5-phenyl-4_H_-1,2,4-triazol-3-yl)thio)acetamide (6a)

White solid; Yield: 0.30 g, 0.96 mmol, 97%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.51 (s, 1H, NH), 10.28 (s, 1H, NH), 7.95(d, J = 8.0 Hz, 2H, Ar), 7.59 (d, J = 8.1 Hz, 2H, Ar), 7.52 (d, J = 6.9 Hz, 2H, Ar), 7.31 (t, J = 7.4 Hz, 3H, Ar), 7.06 (t, J = 7.4 Hz, 1H, Ar), 4.10 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.38, 159.33, 155.24, 138.99, 130.43, 129.12, 128.79, 126.70, 126.05, 123.41, 119.09, 36.23; **HRMS (ESI):** $C_{16}H_{14}N_4OS$ $[M+H]^+$; calculated: 311.0967; found: 311.0975.

2-((5-phenyl-4_H_-1,2,4-triazol-3-yl)thio)-_N_-(_o_-tolyl)acetamide (6b)

White solid; Yield: 0.30 g, 0.94 mmol, 95%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.53 (s, 1H, NH), 9.57 (s, 1H, NH), 7.98 (d, J = 6.4 Hz, 2H, Ar), 7.53 (d, J = 6.4 Hz, 2H, Ar), 7.43 (d, J = 7.4 Hz, 2H, Ar), 7.18 (t, J = 5.9 Hz, 2H, Ar), 7.08 (d, J = 7.4 Hz, 1H, Ar), 4.12 (s, 2H, CH_2), 2.16 (s, 3H, CH_3); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.52, 159.31, 155.31, 136.11, 131.35, 130.45, 130.31, 129.12, 126.70, 126.09, 125.98, 125.22, 124.55, 35.72, 17.69; **HRMS (ESI):** $C_{17}H_{16}N_4OS$ $[M+H]^+$; calculated: 325.1123; found: 325.1145.

2-((5-phenyl-4_H_-1,2,4-triazol-3-yl)thio)-_N_-(_m_-tolyl)acetamide (6c)

White solid; Yield: 0.30 g, 0.94 mmol, 95%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.48 (s, 1H, NH), 10.22 (s, 1H, NH), 7.96 (d, J = 6.7 Hz, 2H, Ar), 7.46 (d, J = 6.7 Hz, 4H, Ar), 7.34 - 7.38 (d, 1H, Ar), 7.18 (t, J = 7.8 Hz, 1H, Ar), 6.87 (d, J = 7.6 Hz, 1H, Ar), 4.11 (s, 2H, CH_2), 2.26 (s, 3H, CH_3); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.17, 159.22, 154.99, 138.86, 137.98, 130.29, 130.09, 129.00, 128.62, 125.96, 124.15, 119.65, 116.32, 36.42, 21.18; **HRMS (ESI):** $C_{17}H_{16}N_4OS$ $[M+H]^+$; calculated: 325.1123; found: 325.1144.

2-((5-phenyl-4_H_-1,2,4-triazol-3-yl)thio)-_N_-(_p_-tolyl)acetamide (6d)

White solid; Yield: 0.31 g, 0.95 mmol, 96%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.41 (s, 1H, NH), 10.20 (s, 1H, NH), 7.95 (d, J = 6.4 Hz, 2H, Ar), 7.49 (d, J = 7.3 Hz, 3H, Ar), 7.46 (s, 2H, Ar), 7.11 (d, J = 6.3 Hz, 2H, Ar), 4.11 (s, 2H, CH_2), 2.24 (s, 3H, CH_3); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.17, 159.22, 154.99, 138.86, 137.98, 129.00, 128.62, 125.96, 124.15, 119.65, 116.32, 36.42, 21.18; **HRMS (ESI):** $C_{17}H_{16}N_4OS$ $[M+H]^+$; calculated: 325.1123; found: 325.1130.

_N_-(2-methoxyphenyl)-2-((5-phenyl-4_H_-1,2,4-triazol-3-yl)thio)acetamide (6e)

White solid; Yield: 0.32 g, 0.94 mmol, 95%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.58 (s, 1H, NH), 9.50 (s, 1H, NH), 7.99 (d, J = 7.3 Hz, 2H, Ar), 7.52 (d, J = 4.2 Hz, 2H, Ar), 6.99 (m, J = 7.3 Hz, 3H, Ar), 6.90 (t, J = 7.4 Hz, 2H, Ar), 4.12 (s, 2H, CH_2), 3.70 (s, 3H, OCH_3); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.82, 159.20, 155.37, 148.86, 130.51, 129.09, 128.76, 127.19, 126.05, 124.27, 120.56, 120.33, 111.04, 55.63, 35.83; **HRMS (ESI):** $C_{17}H_{16}N_4O_2S$ $[M+H]^+$; calculated: 341.1072; found: 341.2044.

_N_-(4-methoxyphenyl)-2-((5-phenyl-4_H_-1,2,4-triazol-3-yl)thio)acetamide (6f)

White solid; Yield: 0.32 g, 0.95 mmol, 96%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.50 (s, 1H, NH), 10.13 (s, 1H, NH), 7.95 (t, J = 7.3 Hz, 2H, Ar), 7.53 (m, J = 6.4 Hz, 3H, Ar), 7.47 (d, J = 5.3 Hz, 2H, Ar), 6.88 (t, J = 7.3 Hz, 2H, Ar), 4.06 (s, 2H,

CH_2), 3.71 (s, 3H, OCH_3); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 165.82, 159.40, 155.33, 132.14, 130.44, 129.13, 128.70, 126.71, 126.05, 120.63, 113.91, 55.17, 36.12; **HRMS (ESI):** $C_{17}H_{16}N_4O_2S$ $[M+H]^+$; calculated: 341.1072; found: 341.1085.

***N*-(2-chlorophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6g)**

White solid; Yield: 0.32 g, 0.94 mmol, 95%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 9.79 (s, 1H, NH), 7.96 (d, J = 7.1 Hz, 2H, Ar), 7.48 (dd, J = 7.4 Hz, 3H, Ar), 7.37 (t, J = 2.3 Hz, 2H, Ar), 7.14 - 7.18 (t, J = 2.2 Hz, 2H, Ar), 4.17 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 167.04, 160.46, 156.44, 147.75, 134.72, 130.25, 129.50, 129.03, 127.56, 126.17, 126.06, 125.40, 124.85, 35.81; **HRMS (ESI):** $C_{16}H_{13}ClN_4OS$ $[M+H]^+$; calculated: 345.0577; found: 345.0594.

***N*-(3-chlorophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide 6(h)**

White solid; Yield: 0.32 g, 0.93 mmol, 94%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.51 (s, 1H, NH), 10.49 (s, 1H, NH), 7.95 (d, J = 8.0 Hz, 2H, Ar), 7.80 (s, 1H, Ar), 7.51 (t, J = 8.1 Hz, 2H, Ar), 7.34 (t, J = 8.0 Hz, 2H, Ar), 7.12 (d, J = 7.9 Hz, 2H, Ar), 4.12 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 167.32, 159.69, 155.73, 140.82, 133.60, 130.98, 130.89, 129.51, 126.44, 123.63, 119.15, 119.06, 117.97, 36.53; **HRMS (ESI):** $C_{16}H_{13}ClN_4OS$ $[M+H]^+$; calculated: 345.0577; found: 345.0592.

***N*-(4-chlorophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6i)**

White solid; Yield: 0.32 g, 0.94 mmol, 95%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.50 (s, 1H, NH), 10.42 (s, 1H, NH), 7.94 (dd, J = 7.4 Hz, 2H, Ar), 7.63 (m, J = 7.6 Hz, 2H, Ar), 7.52 (d, J = 7.6 Hz, 2H, Ar), 7.38 (t, J = 7.8 Hz, 1H, Ar), 7.32 (d, J = 7.6 Hz, 2H, Ar), 4.09 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.58, 159.24, 155.25, 137.94, 130.45, 129.13, 128.71, 126.68, 126.04, 125.69, 120.62, 36.17; **HRMS (ESI):** $C_{16}H_{13}ClN_4OS$ $[M+H]^+$; calculated: 345.0577; found: 345.0581.

***N*-(4-bromophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6j)**

White solid; Yield: 0.36 g, 0.92 mmol, 93%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.50 (s, 1H, NH), 10.41 (s, 1H, NH), 7.93 (d, J = 7.8 Hz, 3H, Ar), 7.57 (d, J = 7.9 Hz, 3H, Ar), 7.50 (t, J = 2.6 Hz, 2H, Ar), 7.48 (t, J = 2.2 Hz, 1H, Ar), 4.09 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.60, 159.24, 155.25, 138.35, 131.62, 130.44, 129.12, 126.67, 126.03, 121.01, 114.96, 36.20; **HRMS (ESI):** $C_{16}H_{13}BrN_4OS$ $[M+H]^+$; calculated: 389.0071; found: 391.0062.

***N*-(4-fluorophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6k)**

White solid; Yield: 0.30 g, 0.92 mmol, 93%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.50 (s, 1H, NH), 10.33 (s, 1H, NH), 7.95 (t, J = 7.0 Hz, 2H, Ar), 7.61 (dd, J = 8.1, 5.0 Hz, 2H, Ar), 7.49 - 7.52 (t, J = 7.1 Hz, 2H, Ar), 7.17 (s, 1H, Ar), 7.14 (d, J = 8.9 Hz, 2H, Ar), 4.08 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.31, 159.28, 155.24, 135.40, 130.44, 129.13, 126.69, 126.04, 120.87, 115.48, 115.26, 36.12; **HRMS (ESI):** $C_{16}H_{13}FN_4OS$ $[M+H]^+$; calculated: 329.0872; found: 329.0891.

***N*-(2-fluorophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6l)**

White solid; Yield: 0.30 g, 0.91 mmol, 92%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.54 (s, 1H, NH), 10.07 (s, 1H, NH), 7.97 (dd, J = 7.8, 1.4 Hz, 2H, Ar), 7.51 (t, J = 4.3 Hz, 2H, Ar), 7.25 (m, J = 4.4 Hz, 2H, Ar), 7.16 (d, J = 2.7 Hz, 2H, Ar), 7.12 (d, J = 2.8 Hz, 1H, Ar), 4.17 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 167.00, 159.28, 154.50, 152.07, 130.43, 129.05, 126.00, 125.29, 124.42, 124.39, 123.58, 115.57, 115.37, 35.74; **HRMS (ESI):** $C_{16}H_{13}FN_4OS$ $[M+H]^+$; calculated: 329.0872; found: 329.1147.

***N*-(4-nitrophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6m)**

White solid; Yield: 0.32 g, 0.89 mmol, 90%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.53 (s, 1H, NH), 10.91 (s, 1H, NH), 8.22 (d, J = 6.6 Hz, 3H, Ar), 7.92 (d, J = 7.8 Hz, 2H, Ar), 7.84 (d, J = 8.1 Hz, 2H, Ar), 7.50 (d, J = 6.4 Hz, 2H, Ar), 4.16 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 167.60, 159.19, 155.38, 145.13, 142.40, 130.51, 129.18, 126.70, 126.08, 125.13, 118.88, 36.34; **HRMS (ESI):** $C_{16}H_{13}N_5O_3S$ $[M+H]^+$; calculated: 356.0817; found: 356.0833.

***N*-(2,4-dimethylphenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6n)**

White solid; Yield: 0.32 g, 0.95 mmol, 96%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.53 (s, 1H, NH), 9.50 (s, 1H, NH), 7.97 (d, J = 7.6 Hz, 2H, Ar), 7.52 (t, J = 4.8 Hz, 2H, Ar), 7.28 (dd, J = 8.1, 3.9 Hz, 2H, Ar), 6.98 (t, J = 4.9 Hz, 2H, Ar), 4.09 (s, 2H, CH_2), 2.22 (s, 3H, CH_3), 2.10 (s, 3H, CH_3); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.48, 159.38, 155.36, 134.37, 133.50, 131.46, 130.84, 130.46, 129.13, 126.49, 126.09, 124.76, 124.69, 35.61, 20.49, 17.62; **HRMS (ESI):** $C_{18}H_{18}N_4OS$ $[M+H]^+$; calculated: 339.1279; found: 339.1296.

***N*-(2,6-dimethylphenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6o)**

White solid; Yield: 0.32 g, 0.96 mmol, 97%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 9.57 (s, 1H, NH), 7.98 (d, J = 7.7 Hz, 2H, Ar), 7.51 (t, J = 7.5 Hz, 2H, Ar), 7.03 (m, J = 5.2 Hz, 4H, Ar), 4.12 (s, 2H, CH_2), 2.08 (s, 6H, $2CH_3$); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 165.98, 159.20, 155.33, 135.26, 134.86, 129.11, 129.04, 127.75, 127.68, 126.57, 126.02, 35.37, 17.99; **HRMS (ESI):** $C_{18}H_{18}N_4OS$ $[M+H]^+$; calculated: 339.1279; found: 339.1290.

***N*-(3,5-dichlorophenyl)-2-((5-phenyl-4*H*-1,2,4-triazol-3-yl)thio)acetamide (6p)**

White solid; Yield: 0.35 g, 0.93 mmol, 94%; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 10.66 (s, 1H, NH), 7.93 (m, J = 6.6 Hz, 2H, Ar), 7.65 (s, 3H, Ar), 7.49 (m, J = 6.5 Hz, 2H, Ar), 7.29 (s, 1H, Ar), 4.11 (s, 2H, CH_2); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 167.22, 159.27, 155.41, 145.72, 141.25, 134.22, 130.23, 129.05, 125.98, 122.75, 117.28, 36.40; **HRMS (ESI):** $C_{16}H_{12}Cl_2N_4OS$ $[M+H]^+$; calculated: 379.0187; found: 379.0192.

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