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RESEARCH ARTICLE

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Synthesis and crystal structures of novel (4-phenylthiazol-2(3H)-ylidene) benzamide and ((benzoylimino)-3-(9,10-dioxo-9,10-dihydroanthracen-1-yl)-4-oxothiazolidin-5-ylidene)acetate derivatives

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Abstract

A new approach to the synthesis of new heterocyclic compounds with benzoylisothiocyanate, amines, and 2-bromoacetophenone (phenacyl bromide) fragments in one molecule has been developed. The synthesis method comprises condensation reaction for the preparation of novel (4-phenylthiazol-2(3*H*)-ylidene) benzamide derivatives. These new compounds were synthesized via one-pot and multicomponent reaction of 2-furanmethylamine, 5-chloro-2-phenoxyaniline, and 1-naphthylamine with phenacyl bromide and substituted benzoylisothiocyanates. Moreover, new series of anthraquinone-thiazole hybrids have been synthesized. These compounds were prepared from unsymmetrical thiourea containing anthraquinone and dialkyl acetylenedicarboxylate. The nature of the compounds was confirmed by IR, ¹HNMR, ¹³CNMR, mass spectrometry, and CHNS elemental analysis. The structural elucidation was accomplished by single-crystal X-ray diffraction methods.

1 | **INTRODUCTION**

Heterocyclic chemistry has constituted one of the largest areas of research in organic chemistry. A large number of natural products, such as vitamins, hormones, antibiotics, alkaloids, are constituted of heterocyclic compounds. Furthermore, most drugs are based on heterocyclic molecules. They have also contributed to industrial development and to the improvement in the quality of life through their applications as herbicide, agrochemicals, dyes, plastics, food additives, cosmetics, and so on.^[1]

Heterocyclic compounds containing nitrogen and sulfur specifically thiazoles and their derivatives are core structure in synthetic intermediates and common substructure in numerous biologically active compounds that are known to exhibit significant biological activities such as anticancer,^[2] antifungal,^[3] anti-inflammatory,^[4] antimicrobial,^[5] antidiabetic,^[6] inhibiting neuraminidase of influenza H1N1 virus,^[7]

and anti-HIV^[8] antischistosomal^[9] activity. Various methods for the synthesis of thiazole have been developed. A general synthetic strategy for the preparation of these heterocyclic compounds is the cyclization of thiourea derivatives with acetylenedicarboxylates,^[10,11] β -nitroacrylates,^[12] α -haloketone,^[13] 3-aryl-2-chloropropanals,^[14] and α -halocarboxylic acids.^[15,16] Other methods include reaction of suitable amine with benzaldehyde in the presence of an excess of mercaptoacetic acid^[17,18] and with chloroacetylchloride in the presence of ammonium thiocyanate.^[19] Venkata et al^[20] reported multicomponent synthesis using phenacyl bromide, thiosemicarbazide, ethylacetoacetate, and aryl aldehydes for synthesis of thiazole derivatives.

Anthraquinone and its derivatives are an important class of heterocycles. They are found in plants such as aloe latex, senna and rhubarb, fungi, and lichens and also in some insects. These compounds attract dye molecules because of their important role in various photochemical and colorimetric



sensor systems. On the other hand, anthraquinones reveal a wide range of application in synthetic and medicinal chemistry.^[21,22] Anthraquinones are essential structural motifs in a great number natural products such as emodin, aloe-emodin, and rhein with cytotoxic properties; also, there are several anthraquinone-based drugs such as mitoxantrone, ametantrone, and doxorubicin (Figure 1) used for treatment of various cancer in clinic.^[23]

More recently, new series of heterocycles and carbocycles-thiazole hybrids as important core structures in many biologically active natural products and pharmaceuticals have been designed. In this regard, a series of coumarin derivatives containing 2-methylbenzothiazoline motif and related compounds were synthesized and evaluated their radical scavenging activities.^[24] Also, new combinations of quinazolinone derivatives by incorporating the thiazole are synthesized. In most of these compounds, analgesic activity, anti-antibacterial, and anti-inflammatory properties have been evaluated and proven. The problem of multidrug resistance toward several anticancer compounds has also become important, and much effort has been

directed toward combination of a five- or six-membered heterocyclic ring such as thiazole in the anthracenedione moiety.[25,26]

In view of the various activities of thiazole and anthraquinones, we have developed a research program focused on the synthesis of new series of thiazole derivative and novel anthraquinones-thiazole hybrids.

2 **RESULTS AND DISCUSSION**

Synthesis of novel (4-phenylthiazol-2.1 2(3H)-vlidene) benzamide

One-pot, multicomponent synthesis of novel (4-phenylthiazol-2(3H)-ylidene) benzamide derivatives have been performed using benzoylisothiocyanate derivatives (1), various amine (2), and phenacyl bromide (3) in the presence of triethylamine as a catalyst and hydrobromic acid scavenger in reflux conditions (Scheme 1).

We started our investigation on the reaction in room temperature conditions for 8 hours, but no suitable progress



of the reaction was observed. To improve the reaction efficiency, the reaction was carried out under reflux conditions. The reaction products were isolated and were identified by FTIR, ¹HNMR, ¹³CNMR spectra, mass spectrometry, and CHNS elemental analysis. For compounds **4a** and **5b**, single crystals of good quality adapted for X-ray diffraction analysis were obtained, and the crystal structure of those compounds is discussed later in the text. Various amines, including substituted 2-furanmethylamine (a), 5-chloro-2-phenoxyaniline (b), and 1-naphthylamine (c), were used to give new thiazole derivatives.

For instance, in the FTIR spectrum of (Z)-*N*-(3-(furan-2-ylmethyl)-4-phenylthiazol-<math>2(3H)-ylidene) benzamide (**4a**), C–H aliphatic and aromatic absorptions were observed at

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3056, 3025, and 2965 cm⁻¹, respectively. Also absorption at 1620 cm⁻¹ corresponds to carbonyl group, and C=C bond in thiazole ring exhibits absorption at 1566 cm⁻¹. The ¹H NMR spectrum showed the presence of 2 aliphatic protons at 5.45 ppm (CH₂ – C_{furfurylamine ring}), CH of thiazole at 6.57 ppm, and aromatic protons of phenyl and furfurylamine

TABLE 1	Selected bond distances (Å) and angles (°) of
molecular struc	ture of compound 4a

Bond	Bond lengths (Å)	Bond	Bond lengths (Å)
C1–C2	1.335 (3)	C3–S	1.7464 (19)
C1–S	1.730 (2)	C401	1.230 (2)
C2-N2	1.401 (2)	C4-N1	1.372 (2)
C2-C16	1.481 (3)	C4–C5	1.496 (3)
C3-N1	1.305 (2)	C1-N2	1.476 (2)
C3-N2	1.361 (2)		
Bond	Bond angles (°)	Bond	Bond angles (°)
C2C1S	113 37 (15)	01 C4 C5	100.00 (17)
	113.37 (13)	01 - 04 - 03	120.88 (17)
C1C2N2	111.41 (17)	N1-C4-C5	114.28 (17)
C1C2N2 C1C2C16	111.41 (17) 126.14 (17)	N1-C4-C5 C3-N1-C4	120.88 (17) 114.28 (16) 117.01 (16)
C1-C2-N2 C1-C2-C16 N2-C2-C16	111.41 (17) 126.14 (17) 122.44 (15)	N1-C4-C5 C3-N1-C4 C3-N2-C2	120.88 (17) 114.28 (16) 117.01 (16) 115.18 (14)
C1-C2-N2 C1-C2-C16 N2-C2-C16 N1-C3-N2	111.41 (17) 126.14 (17) 122.44 (15) 121.06 (16)	N1-C4-C5 C3-N1-C4 C3-N2-C2 C3-N2-C11	120.88 (17) 114.28 (16) 117.01 (16) 115.18 (14) 119.54 (15)
C1-C2-N2 C1-C2-C16 N2-C2-C16 N1-C3-N2 N1-C3-S	111.41 (17) 126.14 (17) 122.44 (15) 121.06 (16) 129.30 (14)	N1-C4-C5 C3-N1-C4 C3-N2-C2 C3-N2-C11 C2-N2-C11	120.88 (17) 114.28 (16) 117.01 (16) 115.18 (14) 119.54 (15) 125.20 (14)
C1-C2-N2 C1-C2-C16 N2-C2-C16 N1-C3-N2 N1-C3-S N2-C3-S	111.41 (17) 126.14 (17) 122.44 (15) 121.06 (16) 129.30 (14) 109.57 (13)	N1-C4-C5 C3-N1-C4 C3-N2-C2 C3-N2-C11 C2-N2-C11 C3-S-C1	120.88 (17) 114.28 (16) 117.01 (16) 115.18 (14) 119.54 (15) 125.20 (14) 90.44 (10)

rings at 8.40-6.24 ppm. The ¹³C NMR spectrum of this compound showed carbonyl groups and carbon in thiazole ring at 174.22 and 168.80 ppm, respectively.



FIGURE 4 Partial crystal packing view of compound **5b** evidencing the distance in Å for pi-pi stacking of two adjacent A molecules

TABLE 2 Selected bond distances (Å) and angles (°) of molecular structure of compound 5b

Bond	Bond lengths (Å) ^a	Bond	Bond lengths $(\text{\AA})^{a}$
C1-N1	1.308 (3) [1.311(3)]	C3–S1	1.720 (3) [1.718(3)]
C1-N2	1.373 (3) [1.369(3)]	C4–O1	1.240 (3) [1.238(3)]
C1-S1	1.741 (3) [1.741(3)]	C4-N1	1.367 (3) [1.373(3)]
C2–C3	1.333 (4) [1.333(3)]	C4–C5	1.485 (4) [1.481(4)]
C2-N2	1.407 (4) [1.414(3)]	C12-N2	1.436 (3) [1.443(3)]
C2–C24	1.470 (4) [1.466(4)]		
Bond	Bond angles (°) ^a	Bond	Bond angles (°) ^a
N1-C1-N2	120.9 (3) [121.8(2)]	01–C4–C5	120.8 (3) [119.7(3)]
N1-C1-S1	129.6 (2) [128.3(2)]	N1-C4-C5	115.2 (3) [116.1(2)]
N2-C1-S1	109.5 (2) [109.85(19)]	C1-N1-C4	115.7 (3) [115.7(2)]
C3-C2-N2	111.2 (3) [111.0(2)]	C1-N2-C2	114.6 (2) [114.4(2)]
C3-C2-C24	126.0 (3) [125.5(2)]	C1-N2-C12	120.6 (3) [121.9(2)]
N2-C2-C24	122.3 (3) [123.3(2)]	C2-N2-C12	123.4 (2) [123.5(2)]
C2-C3-S1	113.9 (3) [114.1(2)]	C3-S1-C1	90.68 (16) [90.58(13)]
O1-C4-N1	124.0 (3) [124.1(3)]		

^aData in parenthesis refer to molecule B in Figure 2.

The mass spectrometry and CHNS elemental analysis confirmed the proposed structure. The molecular structure of compounds **4a** and **5b** was established unequivocally by single-crystal X-ray diffraction analysis. The ORTEP views of molecular structure of compounds **4a** and **5b** are reported in Figures 2 and 3, respectively, together with the atomic numbering scheme.

Tables 1 and 2 show a list of the most important bond and angles. In the crystals of compound **5b**, two crystallographic independent but similar molecules (molecule A and B) are



SCHEME 2 Proposed mechanism for the synthesis of (4-phenylthiazol-2(3H)-ylidene) benzamide derivatives

present, and in Table 2, the geometrical parameters of molecule B are reported in the parenthesis.

The most important difference between the two molecules A and B of compound **5b** regards the dihedral angle between the mean plane of the system defined by atoms C1, C2, C3, C4, O1, N1, N2 and S and that of the *p*-tolyl substituent. In fact, in molecule A, the dihedral angle is of 3.85° while in molecule B is of 26.47° . Considering the crystal packing, the better overall planarity of molecule A in compound **5b** allows a stacking of molecules at a distance of 3.368 Å as evidenced in Figure 4.

In the molecular structure of compounds **4a** and **5b**, the moiety defined by atoms C1, C2, C3, C4, O1, N1, N2 and S is almost planar with maximum deviation for atom O1 of 0.064 Å for **4a** and 0.021 Å [0.016 Å for molecule B] for **5b**. Moreover, the substituted phenyl ring forms a dihedral angle of 24.72° and 39.52° [42.52° for molecule B] in compounds **4a** and **5b**, respectively.

A proposed mechanism for the reaction is given in Scheme 2. (4-Phenylthiazol-2(3H)-ylidene) benzamide derivatives are synthesized via one-pot, two-step cyclocondensation. In the first step, benzoylisothiocyanate (1) reacts with heterocyclic aliphatic or aromatic amines (2) to produce N-(carbamothioyl) benzamide as an unsymmetrical



SCHEME 3 Illustrative synthesis of novel (benzoylimino)-3-((9,10-dioxo-9, 10-dihydroanthracen-1-yl)-4-oxothiazolidin-5-ylidene) acetate derivative

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thiourea. In the next step, the condensation and cyclization reaction is carried out through a nucleophilic attack on phenacyl bromide (3). Finally, the water is removed to produce the final product.

2.2 | Synthesis of novel (benzoylimino)-3-((9,10-dioxo-9,10-dihydroanthracen-1-yl)-4oxothiazolidin-5-ylidene)acetate

Synthesis of novel hybrid structures that include both anthraquinone and thiazole ring has been described in the literature. The reaction have been performed using N,N'-disubstituted asymmetric thioureas with dialkyl acetylenedicarboxylate (Scheme 3).

For our initial investigation, N-(9,10-dioxo-4a,9,9a,10tetrahydroanthracen-1-ylcarbamothioyl) benzamide with DMAD was taken as the model reaction. We began this study by performing the reaction in ethanol, acetonitrile, dimethylformamide, and acetic acid at room temperature as well as at reflux temperature. The best reaction conditions were optimized in acetic acid at reflux temperature. Illustrative mechanism for the generation of compounds is shown in Scheme 4. In the first step, aminoanthraquinone (6) reacts with benzoylisothiocyanate (7) to produce N-(9,10-dioxo-4a,9,9a,10tetrahydroanthracen-1-ylcarbamothioyl)-4-methylbenzamide derivative (8) as an unsymmetrical thiourea. In the next step, cyclocondensation reaction and cyclization via by DMAD (9)



FIGURE 5 ORTEP view of compound **11a**, evidencing the formation of dimers between molecules of acetic acid. Ellipsoids are depicted at their 30% probability level. Symmetry code = -x, -y, -z

and unsymmetrical thiourea. Finally, the alcohol is removed to produce the final product.

The FTIR spectrum of these compounds was observed C–H aliphatic and aromatic absorptions. Also absorption at 1737-1733 cm⁻¹ corresponds to carbonyl ester group. The ¹H NMR spectra of product in Scheme 2 contained vinyl proton



 R_1 = H, Cl, CH₃ R_2 = CH₃,CH₂CH₃

SCHEME 4 Proposed mechanism for the synthesis of ((benzoylimino)-3-(9,10-dioxo-9,10-dihydroanthracen-1-yl)-4-oxothiazolidin-5-ylidene) acetate



FIGURE 6 ORTEP view of compound **11b**. Ellipsoids are depicted at their 30% probability level

singlet at 7.15 ppm and aromatic protons of phenyl and anthraquinone rings at 8.67-7.27 ppm also showed the presence of methoxy in compounds (**10a-12a**) at 3.97 ppm and moreover methyl proton singlet at 3.96 ppm for **12a**. Aliphatic protons for **10b** and **11b** (CH₂CH₃) are seen at 1.44 and 2.13 ppm. The formation of a thiazole ring was also clearly confirmed by the ¹³C NMR spectra. The mass spectrometry and CHNS elemental analysis confirmed the proposed structure.

The exact nature of compounds 11a and 11b was established unequivocally by single-crystal X-ray diffraction analysis. The molecular structure of the complexes is very similar but in the crystals of 11a, acetic acid molecules are also present. The molecules of acetic acid are involved in a strong hydrogen bond forming dimers. [O7...O8' 2.673(2) Å, H7...O8' 1.884(3) Å, O7-H7...O8'161.24(3)°]. The ORTEP view of compound 11a and 11b is reported in Figures 5 and 6 respectively, together with the atomic numbering scheme; a list of the most important bond distances and angles is reported in Tables 3 and 4, respectively, for compound 11a and 11b. The two molecules are very similar, and the only difference is the presence of an ethyl moiety in 11b instead of a methyl group in 11a in the esoteric part of the compound. Analyzing the bond distances and angles in both compounds, they are very similar and in agreement with those observed in the literature for relating fragments in compounds reported. If we exclude the anthraquinone ring connected to the N1 atom, the molecule is planar in 11a and also in 11b. The dihedral angle between the mean plane of the molecule, excepted the anthraquinone moiety,

TARLE 3	Selected bond distances ()	$(\check{\Delta})$ and angles (°)) of molecular structure	of compound 119
IADLE J	Selected bolid distances (F	(A) and angles () of molecular structure	or compound 11a

Bond	Bond lengths (Å)	Bond	Bond lengths (Å)
C1-N1	1.445 (3)	C18–O4	1.222 (3)
C15-N1	1.386 (3)	C18-N2	1.397 (3)
C15-C16	1.488 (4)	C18–C19	1.486 (4)
C16-C25	1.333 (4)	C25-C26	1.463 (4)
C16–S1	1.750 (3)	C26–O5	1.205 (3)
C17-N2	1.282 (3)	C26–O6	1.337 (3)
C17-N1	1.382 (3)	C27–O6	1.453 (3). ?
C17–S1	1.756 (3)		
Bond	Bond angles (°)	Bond	Bond angles (°)
C2-C1-C6	121.02 (16)	O4-C18-C19	122.21 (16)
C2-C1-N1	117.99 (16)	N2-C18-C19	114.18 (15)
C6-C1-N1	120.96 (15)	C16-C25-C26	120.27 (19)
O3-C15-N1	124.59 (17)	O5-C26-O6	124.5 (2)
O3-C15-C16	126.82 (18)	O5-C26-C25	124.0 (2)
N1-C15-C16	108.59 (15)	O6-C26-C25	111.49 (19)
C25-C16-C15	121.54 (18)	C28-C27-O6	107.3 (2)
C25-C16-S1	126.62 (15)	C17-N1-C15	116.97 (14)
C15-C16-S1	111.83 (14)	C17-N1-C1	120.72 (15)
N2-C17-N1	119.20 (15)	C15-N1-C1	121.94 (14)
N2-C17-S1	128.97 (13)	C17-N2-C18	118.17 (15)
N1-C17-S1	111.82 (13)	C26–O6–C27	115.68 (19)
O4-C18-N2	123.59 (19)	C16-S1-C17	90.67 (9)

TABLE 4 Selected bond distances (Å) and angles (°) of molecular structure of compound 11b

Bond	Bond lengths (Å)	Bond	Bond lengths (Å)
C1-N1	1.439 (2)	C18–O4	1.222 (2)
C15–O3	1.204 (2)	C18–N2	1.399 (2)
C15-N1	1.388 (2)	C18–C19	1.474 (3)
C15-C16	1.496 (2)	C25–C26	1.480 (3)
C16-C25	1.335 (3)	C26–O5	1.199 (3)
C16–S1	1.750 (2)	C26–O6	1.325 (3)
C17-N2	1.285 (2)	C27–C28	1.459 (4)
C17–N1	1.377 (2)	C27–O6	1.462 (3)
C17-S1?	1.7612 (18)		
Bond	Bond angles (°)	Bond	Bond angles (°)
C2C1C6	121.02 (16)	O4-C18-C19	122.21 (16)
C2C1N1	117.99 (16)	N2-C18-C19	114.18 (15)
C6-C1-N1	120.96 (15)	C16-C25-C26	120.27 (19)
O3-C15-N1	124.59 (17)	O5-C26-O6	124.5 (2)
O3-C15-C16	126.82 (18)	O5-C26-C25	124.0 (2)
N1-C15-C16	108.59 (15)	O6-C26-C25	111.49 (19)
C25-C16-C15	121.54 (18)	C28-C27-O6	107.3 (2)
C25-C16-S1	126.62 (15)	C17–N1–C15	116.97 (14)
C15-C16-S1	111.83 (14)	C17–N1–C1	120.72 (15)
N2-C17-N1	119.20 (15)	C15–N1–C1	121.94 (14)
N2-C17-S1	128.97 (13)	C17–N2–C18	118.17 (15)
N1-C17-S1	111.82 (13)	C26-O6-C27	115.68 (19)
O4-C18-N2	123.59 (19)	C16-S1-C17	90.67 (9)

and the mean plane of the anthraquinone ring is $80.66(2)^{\circ}$ and $86.47(3)^{\circ}$ for **11a** and **11b**, respectively.

The presence of the acetic acid in the crystals of **11a** affects the crystal packing of the compounds. In fact, the molecule, been formed by extended planar systems, can produce $\pi \cdots \pi$ stacking in the crystal packing. This is evident in the case of **11b**, in which interplanar distance of 3.318(2) Å is present between the anthraquinone rings as shown in Figure 7 (ii). The planar system of the rest of the molecule is stacked in the crystal packing at a distance of 3.362(2) Å, Figure 7 (i). In the case of the crystal packing of compound **11a**, the presence of the dimers of acetic acid in the unit cell prevents the stacking of the molecules as shown in Figure 7 (ii).

3 | EXPERIMENTAL

3.1 | General

The reagents and solvents were purchased from Merck and Aldrich Chemical companies. The products were analyzed by FTIR spectroscopy (JASCO FT/IR-460 plus spectrometer), and melting points were measured by Electrothermal 9100 apparatus. ¹HNMR and ¹³CNMR spectra of compounds were recorded on a Bruker DRX-400 Avance instrument in CDCl₃. The mass spectra were recorded on an Agilent Technology HP 5973 MSD mass spectrometer operating at an ionization potential of 70 eV. Elemental analyses were performed using a Heraeus CHNS Rapid analyzer.

3.2 | General procedure for synthesis of novel (4-phenylthiazol-2(3H)-ylidene) benzamide

At first, the benzoyl isothiocyanate derivative was prepared by adding benzoyl chloride derivative (1 mmol) to ammonium thiocyanate (1 mmol) in 5 mL of acetonitrile and refluxed for 20 minutes. The reaction mixture was filtered, and the solution was added to the appropriate amine (1.0 mmol) and phenacyl bromide (1.1 mmol) in acetonitrile (5 mL) and stirred at reflux conditions in the presence of triethylamine (1.0 mmol, 0.10 g) for a period of time (specified hour or minutes). The progress of the reaction was monitored by thin-layer chromatography (TLC). After completion of the reaction, the mixture was filtered. The





residue was washed with acetonitrile and recrystallized from ethanol for further purification. The isolated compounds were confirmed by FTIR, ¹HNMR, ¹³CNMR, and mass spectroscopy.

3.3 | General procedure for synthesis of N,N'-disubstituted asymmetric thioureas (8a-e)

Benzoyl isothiocyanate derivative was prepared by adding benzoyl chloride derivative (1 mmol) to ammonium thiocyanate (1 mmol) in 5 mL of acetonitrile and refluxed for 20 minutes. The reaction mixture was filtered, and the solution was added to the 1-aminoanthraquinone (1.0 mmol, 0.223 g) and 20 mL of acetonitrile in the reflux conditions. The progress of reaction was followed by TLC. The products obtained were collected by filtration and washed with acetonitrile several times.

3.4 | General procedure for synthesis of novel (benzoylimino)-3-(9,10-dioxo-9,10dihydroanthracen-1-yl)-4-oxothiazolidin-5ylidene)acetate

Into a 100-cm³ two-necked round bottom flask containing a solution of **8a-e** (1 mmol) in glacial acetic acid (50-80 mL), (1 mmol) dimethyl acetylenedicarboxylate (DMAD) or diethyl acetylenedicarboxylate (DEAD) was added in a dropwise fashion with stirring. The mixture was stirred for 30 minutes

in room temperature and then gently refluxed. (The reaction was monitored by TLC analyses.) After completion of the reaction, the products obtained were collected by filtration, and the residue was washed and recrystallized from acetonitrile for further purification. The isolated compounds were confirmed by FTIR, ¹HNMR, ¹³CNMR, and mass spectroscopy.

3.4.1 | Spectral data

(*Z*)-*N*-(*3*-(*furan*-2-*ylmethyl*)-4-*phenylthiazol*-2 (*3H*)-*ylidene*) *benzamide* (4*a*): light yellow crystals, m.p. = 149-150°C IR (KBr) ν/cm^{-1} 3056, 3025 (C–H aromatic), 2965 (CH aromatic), 1620 (C=O), 1601 &1472 (C=C, aromatic), 1566 (C=C thiazole), 1493; ¹HNMR (400 MHz, CDCl₃) δ 8.40 (2H, d, *J* 6.8 Hz CH), 7.44-7.57 (5H, m CH), 7.44-7.42 (3H, m CH), 7.30 (1H, d, *J* 7.6 Hz CH), 6.57 (1H, s CH thiazole), 6.28 (1H, d CH furan), 6.24 (1H, d CH furan), 5.45 (2H, s CH₂); ¹³C NMR (100 MHz, CDCl₃) δ 174.22, 168.80, 149.01, 142.17, 139.11, 136.93, 131.56, 130.43, 129.83, 129.78, 129.37, 128.83, 128.11, 110.64, 109.32, 107.24, 43.61; MS (EI, 70 eV) *m*/*z* (%): 360 (*M*+), 255 (105), 279 (81), Anal. Calcd. for C₂₁H₁₆N₂O₂S: C: 70.00, H: 4.44, N: 7.77, S: 8.90; found: C: 70.09, H: 4.41, N: 7.69, S: 8.92.

(*Z*)-*N*-(3-(5-chloro-2-phenoxyphenyl)-4-phenylthiazol-2(3*H*)-ylidene) benzamide (4b): white crystals, m.p. = 199-200°C. IR (KBr) ν/cm^{-1} 3051 (C–H aromatic), 1688 (C=O), 1600 & 1455 (C=C, aromatic), 1567 (C=C thiazole), 1491; ¹HNMR (400 MHz, CDCl₃) δ 8.17 (2H, d, CH benzene), 7.62

TABLE 5 Details for the X-ray data collection for compounds 4a, 5c, 11a, and 11b

Compound	4a	5c	11a	11b
Formula	$C_{21}H_{16}N_2O_2S$	C ₂₉ H ₂₁ ClN ₂ O ₂ S	C29H19ClN2O8S	C ₂₈ H ₁₇ ClN ₂ O ₆ S
Molecular weight	360.42	496.99	590.97	544.94
Crystal system	Monoclinic	Triclinic	Triclinic	Triclinic
Space group	P2 ₁ /n	P - 1	P - 1	P - 1
a/Å	13.064 (8)	12.506 (2)	8.1996 (12)	10.187 (3)
b/Å	9.962 (6)	14.034 (4)	11.5559 (16)	11.411 (4)
c/Å	14.058 (9)	15.414 (5)	15.985 (2)	12.051 (4)
α/°	90	77.961 (3)	69.586 (2)	113.341 (5)
β / °	102.540 (10)	72.726 (2)	78.862 (3)	98.690 (5)
γ/°	90	77.687 (3)	71.784 (2)	99.822 (5)
Volume (Å ³)	1785.9 (19)	2493.1 (11)	1342.3 (3)	1229.8 (7)
Ζ	4	4	2	2
$D_{\rm calc}/{\rm g~cm}^{-3}$	1.340	1.324	1.462	1.472
F(000)	752	1032	608.0	560.0
μ (Mo-K α)/mm ⁻¹	0.199	0.266	0.276	0.289
Reflections collected	24 397	27 583	11 672	20 584
Unique reflections	4427	8786	3518	7665
Observed reflections $[I > 2\sigma(I)]$	3151 [$R_{int} = 0.0273$]	4822 [$R_{\rm int} = 0.0630$]	2691 [$R_{\rm int} = 0.0299$]	4943 [$R_{\rm int} = 0.0357$]
$R, wR [I > 2\sigma((I)]$	R = 0.0481 wR = 0.1340	R = 0.0478 wR = 0.0989	R = 0.0408 wR = 0.0981	R = 0.0501 wR = 0.1669
R, wR [all data]	R = 0.0694 wR = 0.1507	R = 0.1052 wR = 0.1218	R = 0.0579 wR = 0.1084	R = 0.0814 wR = 0.2032

 $R = \Sigma |Fo - Fc| / \Sigma |Fo|; wR = [\Sigma [w(Fo^2 - Fc^2)^2] / \Sigma [w(Fo^2)^2]]^{1/2}.$

(1H, d, CH) 7.50 (1H, d, CH), 7.48-7.37 (3H, m, CH), 7.35-7.28 (5H, m, CH), 7.16 (2H, d, CH) 7.07 (1H, t, CH), 6.74 (1H, d CH), 6.69 (1H, s, CH thiazole), 6.60 (2H, d, CH)); ¹³CNMR (100 MHz, CDCl₃) δ 174.49, 170.06, 154.95, 151.78, 139.02, 136.61, 131.64, 130.85, 130.47, 130.41, 129.71, 129.40, 129.12, 128.17, 128.61, 128.55, 128.06, 127.38, 124.53, 119.49, 118.96, 107.00; MS (EI, 70 eV) *m*/*z* (%): 482 (*M*+), 484 (*M* + 2), 405 (77), 203, 105; MS (EI, 70 eV) *m*/*z* (%): Anal. Calcd. for C₂₈H₁₉ClN₂O₂S: C: 69.70, H: 3.94, N: 5.80, S: 6.65; found: C: 69.73, H: 3.91, N: 5.78, S: 6.63.

(*Z*)-*N*-(*3*-(*5*-*chloro*-2-*phenoxyphenyl*)-*4*-*phenylthiazol*-2(*3H*)-*ylidene*)-*4*-*methylbenzamide* (*5b*): light green crystals, m.p. = 176-177°C. IR (KBr) ν /cm⁻¹ 3058, 3033 (C–H aromatic), 1600 & 1454 (C=C, aromatic) 1562 (C=C thiazole), 1461; ¹HNMR (400 MHz, CDCl₃) δ 8.084 (2H, d, *J* 8.00 Hz CH benzene), 7.62 (1H d), 7.38 (1H, d), 7.37-7.38 (5H, m), 7.24 (2H, d, CH), 7.27 (2H, t, *J* 8.00 Hz CH benzene), 7.05 (1H, t CH), 6.73 (1H, d CH), 6.67 (1H, s CH thiazole), 6.60 (2H, d CH) 2.42 (3H, s CH₃); ¹³C NMR (100 MHz, CDCl₃) δ 174.50, 169.86, 154.97, 151.75, 142.07, 138.93, 133.98, 130.92, 130.47, 130.42, 129.71, 129.47, 129.08, 128.43, 128.58, 128.54, 128.05, 127.37, 124.51, 119.47, 118.97, 106.93, 21.96 ppm. MS (EI, 70 eV) *m/z* (%):496 (*M*+), 498 (M + 2), 377 (119), 77; MS (EI, 70 eV) m/z (%): Anal. Calcd. for C₂₉H₂₁ClN₂OS, C: 70.16, H: 4.23, N: 5.64, S: 6.46; C: 70.18, H: 4.20, N: 5.61, S: 6.43.

(*Z*)-*N*-(*3*-(*naphthalen*-*1*-*yl*)-*4*-*phenylthiazol*-2(*3H*)*ylidene*) *benzamide* (*4c*): white powder, m.p. = 209-210°C, IR (KBr) ν/cm^{-1} 3051 (C–H aromatic), 1683 (C=O), 1592 & 1464 (C=C, aromatic), 1562 (C=C thiazole), 1510 (C=N); ¹HNMR (400 MHz, CDCl₃) δ 7.96 (2H, d, *J* 8.0 Hz CH benzene), 7.83 (2H, d, *J* 7.2 Hz CH naphthyl), 7.58 (1H, t CH), 7.5-7.48 (2H, dd, CH), 7.36 (2H, t), 7.26-7.17 (4H, m), 7.11 (4H, d), 6.86 (1H, s, CH thiazole); ¹³CNMR (100 MHz, CDCl₃) δ 174.69, 169.97, 139.97, 136.60, 134.59, 134.04, 131.38, 130.41, 130.15, 129.68, 129.25, 128.94, 128.50, 128.41, 128.24, 127.80, 127.40, 126.93, 126.59, 125.11, 123.00, 107.24; MS (EI, 70 eV) *m*/*z* (%): 406 (*M*+), 329 (77), 301 (105), 127; Anal. Calcd. for: C₂₆H₁₈N₂OS: C: 76.84, H: 4.43, N: 6.89, S: 7.89, found: C: 76.80, H: 4.45, N: 6.90, S7.79.

(*Z*)-*N*-(3-(*naphthalen-1-yl*)-4-*phenylthiazol-2*(3*H*)ylidene) benzamide (5*c*): white powder, m.p. = 197-198°C, IR (KBr) ν /cm⁻¹ 3051 (C–H aromatic), 2913 (C–H aliphatic), 1694 (C=O), 1594 & 1470 (C=C, aromatic), 1556 (C=C thiazole), 1511 (C=N); ¹H NMR (400 MHz, CDCl₃) δ 7.95 (2H, d, *J* 8.0 Hz CH benzene), 7.72 (2H, d, CH naphthyl), 7.60-7.46 (4H, m), 7.36 (2H, d), 7.18 (1H, dd), 7.11(4H, d), 7.03 (2H, d, *J* 8.0 Hz CH benzene), 6.84 (1H, s CH thiazole), 2.29 (3H, s CH₃); ¹³CNMR (100 MHz, CDCl₃) δ 174.69, 169.75, 141.78, 139.89, 134.64, 134.04, 133.96, 130.46, 130.18, 129.64, 129.32, 128.91, 128.57, 128.49, 128.39, 128.23, 127.36, 129.97, 126.56, 125.12, 123.06, 107.19, 21.56; MS (EI, 70 eV) *m*/*z* (%): MS (EI, 70 eV) *m*/*z* (%): 420 (*M*+), 301 (119), 105, 91; Anal. Calcd. for: C₂₇H₂₀N₂OS, C: 77.14, H: 4.76, N: 6.66, S: 7.63, found: C: 77.17, H: 4.73, N: 6.67, S: 7.61.

(*E*)-*Methyl* 2-((*Z*)-2-(*benzoylimino*)-3-(9,10-*dioxo*-9,10*dihydroanthracen*-1-*yl*)-4-*oxothiazolidin*-5-*ylidene*)*acetate* (10*a*): Slime green powder, m.p. = 277°C, IR (KBr) ν /cm⁻¹: 3065, 2946, 1735, 1697, 1671, 1642, 1613, 1580. ¹HNMR (400 MHz, CDCl₃) δ : 8.66 (1H, d*J* = 8), 8.34 (1H, d*J* = 5.6), 8.16 (1H, d, *J* = 5.6), 8.061 (1H, t*J* = 8), 7.88 (2H, d), 7.82 (2H, m), 7.47 (1H, t), 7.31 (3H, m), 7.15 (1H, s), 3.97 (3H, s). ¹³C NMR (100 MHz, CDCl₃) δ ; 182.18, 182, 176.75, 166.70, 165.53, 165.16, 141.15, 136.38, 135.51, 134.60, 134.47, 134.44, 133.79, 133.62, 133.47, 132.59, 130.14, 130.06, 129.72, 128.60, 128.31, 127.71, 127.64, 127.12, 120.84, 52.81. MS (EI, 70 eV) *m*/*z* (%): 496 (*M*+), 391 (105), 275, 105, 77. Anal. Calcd. For: C₂₇H₁₆N₂O₆S, C: 65.34, H: 3.22, N: 5.64, S: 6.46, found: C: 65.36, H: 3.24, O: 19.30, N: 5.61, S: 6.48.

(*E*)-*Methyl* 2-((*Z*)-2-(4-chlorobenzoylimino)-3-(9,10-dioxo-9,10-dihydroanthracen-1-yl)-4-oxothiazolidin-5-ylidene) acetate (11a): Slime green powder, m.p. = 275°C, IR (KBr) ν/cm^{-1} : 3061, 2927, 1735, 1709, 1673, 1646, 1624, 1587, ¹HNMR (400 MHz, CDCl₃) δ ; 8.66 (1H, d *J* = 8), 8.33 (1H, d *J* = 7.2), 8.16 (1H, d *J* = 7.2), 8.06 (1H, t *J* = 8), 7.87-7.77 (4H, m), 7.29 (1H, d), 7.25 (2H, d), 7.15 (1H, s), 3.97 (3H, s). ¹³CNMR (100 MHz, CDCl₃) δ ; 182.10, 82.01, 177.15, 167.48, 165.52, 165.06, 140.95, 139.93, 136.29, 135.52, 134.62, 134.50, 133.74, 133.51, 133, 132.58, 131.48, 131.40, 129.78, 128.65, 128.56, 127.61, 127.16, 121.10, 52.84. MS (EI, 70 eV) *m/z* (%): 530 (*M*+), 511 (*M* + 1), 471 (59), 391 (139), 275, 111. Anal. Calcd. For: C₂₇H₁₆ClN₂ O₆S, C: 61.10, H: 2.82, N: 5.27, S: 6.02, found: C: 61.11, H: 2.80, N: 5.23, S: 6.03.

(*E*)-*Methyl* 2-((*Z*)-3-(9,10-dioxo-9,10-dihydroanthracen-1-yl)-2-(4-methylbenzoylimino)-4-oxothiazolidin-5-ylidene) acetate (12a): Slime green crystal, m.p. = 271°C, IR (KBr) ν/cm^{-1} : 3067, 2956, 1737, 1690, 1679, 1649, 1605, 1588. ¹HNMR (400 MHz, CDCl₃) δ ; 8.66 (1H, d *J* = 8), 8.33 (1H, d *J* = 5.6), 8.16 (1H, d *J* = 5.6), 8.05 (1H, t *J* = 8), 7.86-7.76 (5H, m), 7.14 (1H, s), 7.10 (2H, d), 3.96 (3H, s), 2.34 (3H, s). ¹³CNMR (100 MHz, CDCl₃) δ ; 128.20, 181.97, 176.63, 166.13, 165.54, 165.18, 144.43, 141.27, 136.41, 135.49, 134.57, 134.41, 133.80, 133.67, 132.58, 131.92, 130.25, 130.16, 129.67, 129.07, 128.61, 127.65, 127.10, 120.66, 52.79, 21.73. MS (EI, 70 eV) *m/z* (%): 510 (*M*+), 511 (*M* + 1), 451 (59), 391 (119), 275, 91. Anal. Calcd. For: $C_{30}H_{18}N_2O_6S$, C: 67.43, H: 3.37, N: 5.24, S: 6.00, found: C: 67.46, H: 3.35, N: 5.23, S: 6.01.

(*E*)-*Ethyl* 2-((*Z*)-2-(*benzoylimino*)-3-(9,10-*dioxo*-9,10*dihydroanthracen*-1-*yl*)-4-*oxothiazolidin*-5-*ylidene*)*acetate* (10b): Brown crystal, m.p. = 272°C, *IR* (*KBr*) ν/cm^{-1} : 3027, 2979, 1734, 1717, 1691, 1672, 1654. ¹HNMR (400 MHz, CDCl₃) δ ; 8.67 (1H, d *J* = 8), 8.34 (1H, d *J* = 5.6), 8.16 (1H, d, *J* = 5.6), 8.059 (1H, t *J* = 8), 7.87-7.75 (5H, m), 7.28 (2H, d), 7.15 (1H, s), 4.42 (2H, q), 1.44 (3H, t). ¹³CNMR (100 MHz, CDCl₃) δ ; 182.11, 182.02, 175.79, 167.61, 165.14, 165.09, 140.64, 136.30, 135.52, 134.62, 134.50, 134.43, 133.75, 133.54, 133.04, 132.58, 131.48, 131.39, 129.77, 128.65, 128.57, 127.66, 127.62, 127.16, 121.64, 62.22, 14.26. MS (EI, 70 eV) *m/z* (%): 510 (*M*+) 405 (105), 275, 77. Anal. Calcd. For: C₂₈H₁₈N₂O₆S, C: 65.90, H: 3.52, N: 5.48, S: 6.28, found: C: 65.92, H: 3.51, N: 5.46, S: 6.07.

(E)-Ethyl 2-((Z)-2-(4-chlorobenzoylimino)-3-(9,10-dioxo-9,10-dihydroanthracen-1-yl)-4-oxothiazolidin-5-ylidene) acetate (11b): Mustard crystal, m.p. = 273° C, IR (KBr) ν / cm⁻¹: 3064, 2982, 1733, 1692, 1671, 1643, 1613, 1579. $H^{1}NMR$ (400 MHz, CDCl₃) δ ; 8.66 (1H, d J = 8), 8.33 (1H, d J = 7.2, 8.15 (1H, d J = 7.2), 8.05 (1H, t J = 8), 7.88 (2H, d), 7.87-7.76 (3H, m), 7.31 (2H, d), 7.29 (1H, d), 7.14 (1H, s), 4.41 (2H, q), 1.43 (3H, t). C¹³NMR (100 MHz, CDCl₃) δ; 182.19, 182.01, 176.73, 166.82, 165.21, 165.10, 140.85, 136.39, 135.50, 134.59, 134.51, 134.43, 134.35, 133.80, 133.64, 133.44, 132.59, 130.13, 130.05, 129.70, 128.62, 128.30, 127.68, 127.63, 127.11, 126.47, 121.36, 62.05, 14.27. MS (EI, 70 eV) m/z (%): 544 (M+), 545 (M + 1), 510, 471, 391 (139), 275, 105, 77. Anal. Calcd. For: C₂₈H₁₇ClN₂O₆S, C: 61.61, H: 3.29, N: 5.13, S: 5.78, found: C: 61.60, H: 3.28, N: 5.16, S: 5.77.

3.5 | Experimental details of X-ray diffraction analysis for compounds 4a, 5b, 11a, and 11b

The crystallographic data for the complexes were collected at room temperature on a Bruker APEX II single-crystal diffractometer, working with Mo-K α graphite monochromatic radiator ($\lambda = 0.71073$ Å) and equipped with an area detector. The raw frame data (20 seconds per frame scan time for a sphere of diffraction data) were processed using SAINT software [SAINT, Version 7.06a; Bruker AXS Inc.: Madison, Wisconsin, USA]; a correction for absorption was made using SCALE program [G. M. Sheldrick, SADABS 2000 Version 2.01, Bruker AXS Inc., Madison, Wisconsin, USA] to yield the reflection data file. The structures were solved by direct methods with SHELXS-97 and refined against F² with SHELXL-2014/7^[27] using anisotropic thermal parameters for all nonhydrogen atoms. The hydrogen atoms were placed in the ideal geometrical positions. Details for the X-ray data collection are reported in Table 5. Crystallographic data for the complexes have been deposited with the Cambridge Crystallographic Data Centre as supplementary publications CCDC.

4 | CONCLUSION

In conclusion, we have introduced an efficient and facile approach for the synthesis of novel thiazole derivatives. In the first approach, synthesis of some novel (4-phenylthiazol-2(3H)-ylidene) benzamide derivatives via one-pot and multicomponent reaction between benzoylisothiocyanate, various amines, and phenacyl bromide was performed under reflux conditions. The reaction is mainly condensation followed by cyclization. Initially, unsymmetrical thiourea was synthesized then cyclocondensation reaction and cyclization via by attacked nucleophile to phenacyl bromide. In the second method, new anthraquinone-thiazole hybrids were synthesized from condensation of the unsymmetrical thiourea with anthraguinone and DMAD or DEAD. These new compounds were fully characterized, and the single-crystal X-ray diffraction analysis reveals clearly the exact identity of the compounds. The benefits of the presented method are one-pot, excellent yields, simple work-up, and the lack of need for column chromatography.

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