# Synthesis and characterization of new heterocycles containing the 1,2,3-triazole ring system

Bakr F. Abdel-Wahab [a], Benson M. Kariuki [b], Hanan A. Mohamed [a], Gamal A. El-Hiti [c,*]

[a] *Applied Organic Chemistry Department, Chemical Industries Research Institute, National Research Centre, Dokki, Giza 12622, Egypt*
[b] *School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK*
[c] *Department of Optometry, College of Applied Medical Sciences, King Saud University, Riyadh 11433, Saudi Arabia*

ARTICLE INFO

*Keywords:*
1,2,3-triazole
2-butene-1,4-dione
Benzimidazole
2,3-dibromoketone
2-bromo-3-ethoxyketone
Hydrazonoyl chloride

ABSTRACT

The unexpected (*E*)-1,4-*bis*(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)but-2-ene-1,4-dione was produced in 89 % yield from the reaction of 2-bromo-1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethan-1-one and sodium benzenesulfinate dihydrate in boiling ethanol. The reaction of the synthesized symmetrical 2-butene-1,4-dione and 1,2-phenylenediamine in acetic acid afforded 2-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[*d*]imidazole in 68 % yield. 2,3-Dibromopropan-1-one was synthesized in 82 % from bromination of the corresponding chalcone under acidic conditions. Treatment of dibromoketone and benzylamine or phos- phonic dihydrazide in ethanol in the presence of a basic catalyst unexpectedly afforded 2-bromo-3-ethoxyketone in reasonable yield. The reactivity of hydrazonoyl chloride towards 5-methyl- and 5-chloro-1,2,3-triazol-4-carbohydrazides was also investigated to afford the corresponding bis-hydrazone and the unanticipated *tris*- hydrazone.

## 1. Introduction

1,4-Diketones are convenient starting materials for the synthesis of a range of heterocycles. The but-2-ene-1,4-dione system and its functionalized derivatives are potentially even more valuable. Their use has, however, been limited because of their relatively complex synthesis [1–4]. The standard synthesis of core 1,4-enedione includes manganese oxide ($KMn_8O_{16}$) catalyzed reaction of β-ketoesters and α-iodoacetophenones [5], the direct nucleophilic substitution of α-haloacetophenones and sodium *p*-toluenesulfinate [6], iodine/copper complex or hypervalent iodine mediated oxidative self- or cross-coupling of methyl ketones [7–9].

Chalcones present a wide variety of cytoprotective and modulatory properties and have therapeutic potential for multiple diseases. They act as antiviral, antimalarial, anti-inflammatory, and antioxidant agents [10–13]. The standard methods for the synthesis of chalcones are Claisen-Schmidt's condensation, coupling reaction, and continuous-flow deuteration reaction [14]. α,β-Dibbromodihydrochalcones and α-bromo-β-methoxydihydrochalcones are precursors for the production of many heterocycles, including natural products [15–17]. The synthesis of α,β-dibromodihydrochalcones involves a bromination process using either bromine or *N*-bromosuccinimide in a polar aprotic solvent (e.g., chlorinated solvent). α-Bromo-β-methoxydihydrochalcones are produced through bromination of chalcones in polar solvent (e.g., alcohol) [18,19].

Acid carbohydrazides and their hydrazone-containing heterocycles are adaptable classes of compounds with various applications. They exhibit significant biological activities, including acting as antibacterial, anticancer, anti-fungal, and anti-inflammatory agents [20,21]. Acid hydrazides can be obtained from the reaction of hydrazine with aldehydes, esters, anhydrides, and acyl halides [22,23]. Hydrazides have been used as precursors in the synthesis of various heterocycles [24]. In addition, reactions of acid hydrazides and carbonyl compounds have led to the production of the corresponding hydrazones [25–27]. Hydrazonoyl chlorides are useful building blocks for the synthesis of many heterocycles [28,29].

1,2,3-Triazole-containing heterocycles show beneficial biological activities and act as antibacterial and antiviral agents [30–32]. The production of 1,2,3-triazoles has gained significant interest because of their significance in synthetic and medicinal chemistry [33]. The most common method for the production of 1,2,3-triazoles involves 1, 3-dipolar cycloaddition of terminal alkynes and azides [34]. The pre- sent study deals with the synthesis of new 1,2,3-triazoles, which is related to previous work [35–38].

* Corresponding author at: Department of Optometry, College of Applied Medical Sciences, King Saud University, Riyadh 11433, Saudi Arabia.
*E-mail address:* gelhiti@ksu.edu.sa (G.A. El-Hiti).

**Table 1**
Crystal and structure refinement data.

| Compound | **4** | **8** | **16** | **20** | **21** |
|---|---|---|---|---|---|
| Formula | $C_{22}H_{16}N_8O_6$ | $C_{16}H_{12}N_6O_2$ | $C_{21}H_{22}BrN_3O_3$ | $C_{25}H_{23}ClN_9O_3S$, $C_3H_7NO$ | $C_{34}H_{30}Cl_2N_{14}O4S$, $C_3H_7NO$ |
| Formula weight | 488.43 | 320.32 | 444.32 | 638.13 | 874.77 |
| Temperature (K) | 293(2) | 293(2) | 296(2) | 298(2) | 293(2) |
| Wavelength (Å) | 0.71073 | 1.54184 | 1.54184 | 0.71073 | 1.54184 |
| Crystal system | Monoclinic | Triclinic | Monoclinic | Triclinic | Triclinic |
| Space group | $P2_1/c$ | $P\bar{1}$ | $P2_1/c$ | $P\bar{1}$ | $P\bar{1}$ |
| a (Å) | 9.5457(11) | 6.8316(3) | 21.4890(7) | 8.3050(4) | 8.4323(6) |
| b (Å) | 7.8998(6) | 8.0830(5) | 7.8695(2) | 10.0464(5) | 14.8519(12) |
| c (Å) | 15.1330(12) | 14.4920(7) | 12.5833(4) | 19.2729(9) | 17.5530(9) |
| α (°) | 90 | 93.498(4) | 90 | 99.776(4) | 104.183(5) |
| β (°) | 101.815(10) | 96.825(4) | 101.517(3). | 92.819(4) | 99.382(5) |
| γ (°) | 90 | 113.936(5) | 90. | 91.852(4) | 97.861(6) |
| Volume ($Å^3$) | 1116.99(18) | 720.96(7) | 2085.09(11) | 1581.42(13) | 2066.6(3) |
| Z | 2 | 2 | 4 | 2 | 2 |
| Density (calc) ($Mg/m^3$) | 1.452 | 1.476 | 1.415 | 1.342 | 1.406 |
| Absorp. Coeff. ($mm^{-1}$) | 0.110 | 0.858 | 2.903 | 0.237 | 2.411 |
| F(000) | 504 | 332 | 912 | 668 | 908 |
| Crystal size ($mm^3$) | 0.73 × 0.26 × 0.04 | 0.40 × 0.30 × 0.06 | 0.33 × 0.24 × 0.09 | 0.456 × 0.152 × 0.057 | 0.32 × 0.11 × 0.06 |
| Reflections collected | 9474 | 10,841 | 16,355 | 15,323 | 14,569 |
| Independent refls | 2795 | 2809 | 4124 | 7605 | 7604 |
| R(int) | 0.0385 | 0.0282 | 0.0316 | 0.0327 | 0.0371 |
| Parameters | 205 | 218 | 257 | 503 | 590 |
| Goodness-of-fit on $F^2$ | 1.067 | 1.061 | 1.030 | 1.038 | 1.030 |
| R1 [$I > 2\sigma(I)$] | 0.0650 | 0.0483 | 0.0400 | 0.0683 | 0.0792, |
| wR2 [$I > 2\sigma(I)$] | 0.1770 | 0.1229 | 0.1110 | 0.1690 | 0.2239 |
| Extinction coeff. | — | — | 0.00150(19) | — | — |
| Largest diff. peak/hole ($e.Å^{-3}$) | 0.189/–0.174 | 0.170/–0.263 | 0.428/–0.498 | 0.422/–0.462 | 0.621/–0.268 |

## 2. Experimental

### *2.1. General*

The melting points of synthesized heterocycles were determined using an electrothermal melting point apparatus. A JEOLNMR 500 MHz spectrometer was used to record the NMR spectra. A Bruker Vertex 80 ATR-FTIR spectrometer was used to record the FTIR spectra. NMR spectra at 500 MHz for $^1H$ and 125 MHz for $^{13}C$ were obtained in deuterated dimethyl sulfoxide (DMSO-$d_6$). The chemical shift (δ) and coupling constant (*J*) were reported in ppm and Hz, respectively. Compounds **1** [39], **10** [40], **12** [41], **17** [42], **18** [43] and **19a,b** [44] were produced based on literature procedures.

### *2.2. (E)-1,4-Bis(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) but-2-ene-1,4-dione (**4**)*

A mixture of **1** (1.62 g, 5 mmol) and **2** (2.40 g, 12 mmol) in EtOH (20 mL) was refluxed for 8 h. The solid was filtered from the mixture after cooling to room temperature and then poured into ice water (150 mL). The solid was dried and crystallized using dimethylformamide (DMF) to give **4**. Yield 89 %, mp > 300 °C. FTIR (KBr): 3406, 3093, 1678, 1612, 1370, 1288 $cm^{-1}$. $^1H$ NMR (*E*-isomer; major): 2.67 (s, 6H, 2 Me), 8.01 (d, 9.1 Hz, 4H, Ar), 8.28 (s, 2H, 2 CH), 8.48 (d, 9.1 Hz, 4H, Ar). $^{13}C$ NMR: 9.8, 125.4, 126.9, 139.1, 139.8, 142.4, 148.3, 149.0, 192.3. Anal. Calcd. for $C_{22}H_{16}N_8O_6$ (488.12): C, 54.10; H, 3.30; N, 22.94. Found: C, 54.21; H, 3.39; N, 23.11 %.

### *2.3. 2-(5-Methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-1H-benzo[d] imidazole (**8**)*

A mixture of **4** (0.97 g, 2 mmol) and **6** (2.16 g, 2 mmol) was refluxed in AcOH (15 mL) for 6 h. The solid formed on cooling was filtered, dried, and then subjected to crystallization from DMF, yielding **8**. Yield 68 %, mp 293–295 °C. FTIR (KBr): 3118, 3097, 1611, 1587 $cm^{-1}$. $^1H$ NMR: 2.83 (s, 3H, Me), 7.18–7.24 (m, 2H, Ar), 7.52 (app t, 8.1 Hz, 1H, Ar), 7.86 (app t, 8.1 Hz, 1H, Ar), 8.05 (d, 8.6 Hz, 2H, Ar), 8.47 (d, 8.6 Hz, 2H, Ar), 13.16 (s, exch., 1H, NH). $^{13}C$ NMR: 10.5, 112.1, 119.4, 122.2, 123.2, 125.6, 126.7, 134.6, 135.1, 137.4, 140.9, 144.3, 145.3, 148.3. Anal. Calcd. for $C_{16}H_{12}N_6O_2$ (320.10): C, 60.00; H, 3.78; N, 26.24; Found: C, 60.13; H, 3.88; N, 26.32 %.

### *2.4. 2,3-Dibromo-3-(4-methoxyphenyl)-1-(5-methyl-1-phenyl-1H-1,2,3-triazol-4-yl)propan-1-one (**13**)*

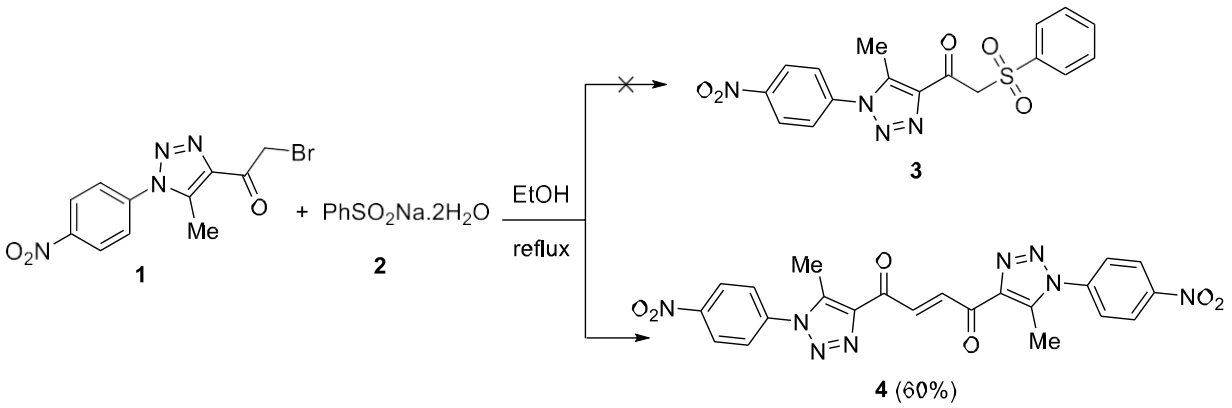
A mixture of chalcone **12** (1.60 g, 5 mmol) and $Br_2$ (0.8 g, 5 mmol) in AcOH (15 mL) was stirred at 20 °C for 12 h. The mixture was added to ice water (150 mL). The solid that formed was filtered, dried, and subsequently crystallized from DMF to afford **13**. Yield 82 %, mp 158–160 °C. FTIR (KBr): 3060, 2972, 1763, 1660, 1581, 1363, 1276 $cm^{-1}$. $^1H$ NMR: 2.58 (s, 3H, Me), 3.82 (s, 3H, OMe), 6.99–7.07 (m, 2H, 2CH), 7.63–7.84 (m, 9H, Ar). Anal. Calcd. for $C_{19}H_{17}Br_2N_3O_2$ (476.96): C, 47.63; H, 3.58; N, 8.77; Found: C, 47.77; H, 3.69; N, 8.86 %.

### *2.5. 2-Bromo-3-ethoxy-3-(4-methoxyphenyl)-1-(5-methyl-1-phenyl-1H-1,2,3-triazol-4-yl)propan-1-one (**16**)*

A mixture of **3** (0.95 g, 2 mmol) and **14** or **17** (2 mmol) in dry EtOH (15 mL) and $Et_3N$ (0.2 mL) was refluxed for 14 h. The mixture was cooled and added to ice water (150 mL). The resulting solid was collected, dried, and crystallized from DMF to give **16**. Yield 45 % (in case of **14**) or 90 % (in case of **17**), mp 152–153 °C. FTIR (KBr): 3060, 2950, 1772, 1612, 1589, 1372, 1278 $cm^{-1}$. $^1H$ NMR (major component): 0.89 (t, 7.2 Hz, 3H, Me), 2.57 (s, 3H, Me), 3.80 (s, 3H, OMe), 4.89 (q, 7.2 Hz, 2H, $CH_2$), 5.59 (d, 10.0 Hz, 1H, CH), 5.84 (d, 10.0 Hz, 1H, CH), 6.96 (d, 8.6 Hz, 2H, Ar), 7.40 (d, 8.6 Hz, 2H, Ar), 7.58–7.68 (m, 5H, Ar). $^{13}C$ NMR: 10.4, 15.4, 49.5, 55.6, 64.8, 80.5, 114.3, 126.0, 126.0, 129.8, 129.9, 130.1, 130.3, 130.9, 159.8, 160.0, 188.3. Anal. Calcd. for $C_{21}H_{22}BrN_3O_3$ (443.08): C, 56.77; H, 4.99; N, 9.46; Found: C, 56.83; H, 512; N, 9.62 %.

### *2.6. Synthesis of **20** and **21***

A mixture of **18** (0.71 g, 2 mmol) and **19a** or **19b** (2 mmol) in dry EtOH (15 mL) was refluxed for 4 h. The solid obtained on cooling was filtered, dried, and crystallized from DMF to give either **20** or **21**,




**Scheme 1.** Synthetic route to symmetrical 1,4-disubstituted 2-butene-1,4-dione **4**.

respectively.

*2.6.1. 2-(2-(5-Methyl-1-(4-methylphenyl)-1H-1,2,3-triazole-4-carbonyl) hydrazineylidene)-N-(4-(N-(pyridin-2-yl)sulfamoyl)phenyl) propanehydrazonoyl chloride (**20**)*

Yield 88 %, mp 248–250 ˚C. FTIR (KBr): 3348, 2987, 1702, 1621, 1592, 1384, 1272 $cm^{-1}$. $^1H$ NMR: 2.39 (s, 3H, Me), 2.47 (s, 3H, Me), 2.69 (s, 3H, Me), 6.71 (d, 8.6 Hz, 1H, Ar), 6.86 (m, 1H, Ar), 7.06 (d, 8.6 Hz, 1H, Ar), 7.42–7.49 (m, 7H, Ar), 7.30 (d, 8.6 Hz, 2H, Ar), 10.22 (s, exch., 1H, NH), 10.55 (s, exch., 1H, NH), 10.95 (s, exch., 1H, NH). $^{13}C$ NMR: 9.9, 13.7, 21.3, 111.5, 114.0, 115.1, 116.9, 125.7, 129.0, 130.7, 133.3, 133.5, 140.0, 140.3, 140.5, 147.2, 153.0, 153.2, 162.9. Anal. Calcd. for $C_{25}H_{24}ClN_9O_3S$ (566.03): C, 53.05; H, 4.27; N, 22.27; Found: C, 53.16; H, 4.32; N, 22.38 %.

*2.6.2. 4-(2-(2-(2-((1-(4-Chlorophenyl)-5-methyl-1H-1,2,3-triazol-4-yl) (2-(1-(4-chlorophenyl)-5-methyl-1H-1,2,3-triazole-4-carbonyl) hydrazineylidene)methyl)hydrazineylidene)propanoyl)hydrazineyl)-N-(pyridin-2-yl)benzenesulfonamide (**21**)*

Yield 72 %, mp 235–237 ˚C. FTIR (KBr): 3349, 3228, 3052, 1680, 1629, 1569, 1385, 1272 $cm^{-1}$. $^1H$ NMR: 2.17 (s, 3H, Me), 2.47 (s, 3H, Me), 2.69 (s, 3H, Me), 6.70–6.85 (m, 4H, Ar), 7.63–8.03 (m, 12H, Ar), 10.13 (s, exch., 1H, NH), 10.56 (s, exch., 1H, NH), 10.58 (s, exch., 1H, NH), 10.87 (s, exch., 1H, NH), 10.93 (s, exch., 1H, NH). $^{13}C$ NMR: 10.0, 12.5, 13.8, 111.4, 113.2, 114.0, 115.1, 127.8, 129.0, 130.4, 134.5, 135.4, 138.8, 140.2, 145.3, 146.0, 152.9, 162.8, 164.8. Anal. Calcd. for

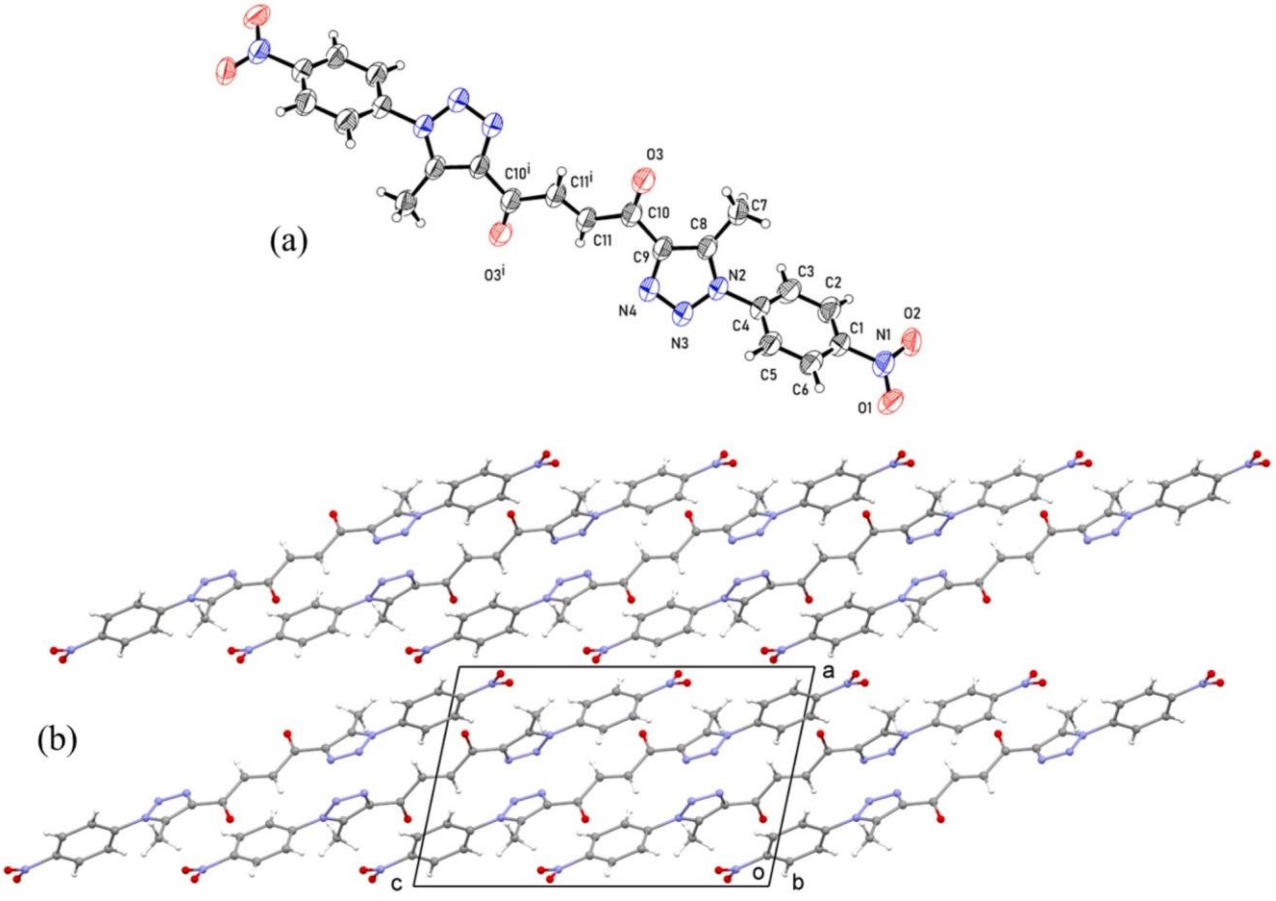
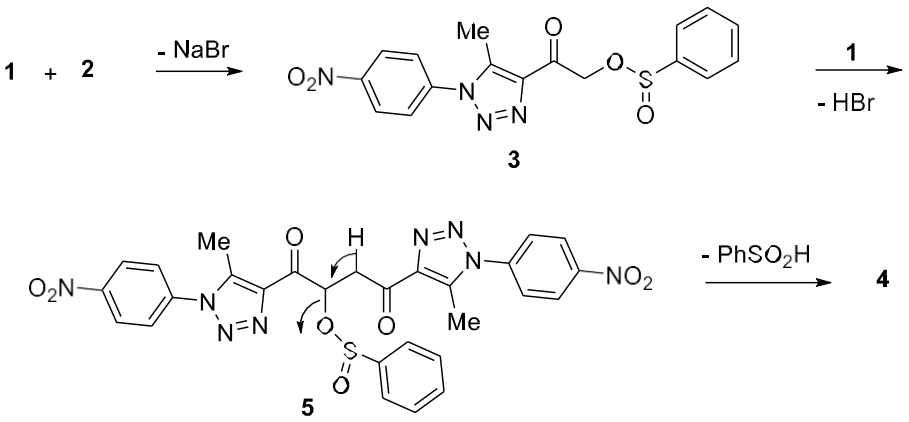



**Fig. 1.** (a) An ortep representation of **4** showing atomic displacement ellipsoids at 50 % probability and (b) crystal packing viewed along the *b* axis.




**Scheme 2.** A proposed mechanism for the synthesis of **4**.

$C_{34}H_{30}Cl_2N_{14}O_4S$ (801.67): C, 50.94; H, 3.77; N, 24.46; Found: C, 51.02; H, 3.88; N, 24.59 %.

### *2.7. Crystal structure determination*

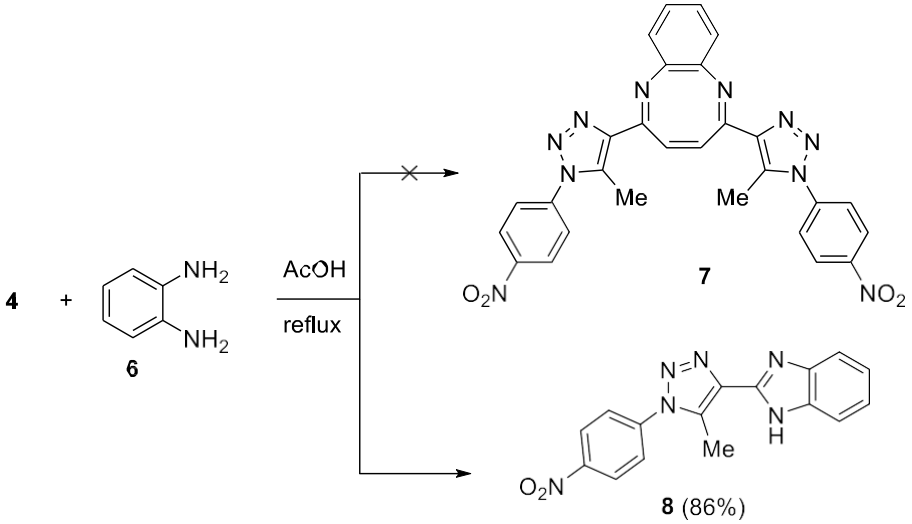
The Agilent SuperNova Dual Atlas single-crystal diffractometer with a mirror monochromator was used to record data, utilizing either Mo or Cu radiation. The structures were solved using SHELXS [45] and refinement by SHELXL [46]. Atoms other than hydrogens were refined using anisotropic displacement parameters. Idealized geometry was used for hydrogen atoms in a riding model with *U*isoset to 1.2 or 1.5 times *Ueq* for the atom they are bonded to. For **20**, the methylphenyl ring is disordered, and the DMF solvent is disordered in the structures of both **20** and **21**. For **4**, the butenedioyl and the nitro groups are also disordered. In all cases, the disordered groups were modeled with two components. Crystal and structure refinement data are shown in Table 1. The crystal structures of **4, 8, 16, 20,** and **21** have been deposited in the CSD with reference numbers CCDC 2312429, 2312431, 2312430, 2172180, and 2172181, respectively.

## 3. Results and discussion

The synthesis of symmetric and unsymmetric (*E*)-1,4-diaryl-2-butene-1,4-diones by reaction of α-bromoacetophenones through a sodium sulfinate mediated reaction in the presence of potassium carbonate ($K_2CO_3$) has been reported [47]. The symmetrical 1,4-disubstituted 2-butene-1,4-dione (**4**) was obtained in 45 % yield via the reaction of 2-bromo-1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethan-1-one (**1**) and sodium benzenesulfinate dihydrate ($PhSO_2Na{\cdot}2H_2O$; **2**) in boiling ethanol (EtOH). The expected product, 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-2-(phenylsulfonyl)ethan-1-one (**3**; Scheme 1), was not obtained. The use of an excess of **1** (2.4 mole equivalents) led to a high yield of product **4** (89 %).

Various spectral analyses have been used to establish the structure of **4.** For example, the FTIR spectrum of **4** showed the C–H stretching bands at 3406 and 3093 $cm^{-1}$. Additionally, the carbonyl and the C–C groups appeared at 1678 and 1612 $cm^{-1}$, respectively. The $^1H$ NMR spectrum of **4** showed the presence of the *E* and *Z*-isomers in unequal proportion. For the *E*-isomer (major), the protons of the two methyl groups appeared as a singlet at 2.67 ppm in the $^1H$ NMR spectrum. The aryl protons (8 protons) appeared as two doublets ($J$ = 9.1 Hz) at 8.01 and 8.48 ppm. In addition, the two CH protons of the double bond appeared as a singlet at 8.28 ppm. In comparison, the $^{13}C$ NMR spectrum of the *E*-isomer of **4** showed nine signals that represented twenty-two carbons due to the symmetry within the compound. For example, the two CH (CH–CH) carbons appeared downfield at 142.4 ppm and the carbonyl carbon at 192.3 ppm. Moreover, the carbon of the two methyl groups appeared as a sharp signal at an upfield at 9.8 ppm.

The structure of **4** was confirmed further using single-crystal X-ray diffraction. The molecule consists of nitrophenyl (**nphen**, C1–C6, N1, O1, O2), methyltriazolyl (**mtria**, C7–C9, N2–N4) and butenedioyl (**bdio**, C10, C11, C11′, C10′, O3, O3′) groups (Fig. 1a). It is symmetrical with




**Scheme 3.** Synthetic route to 2-(1,2,3-triazol-4-yl)benzimidazole **8**.

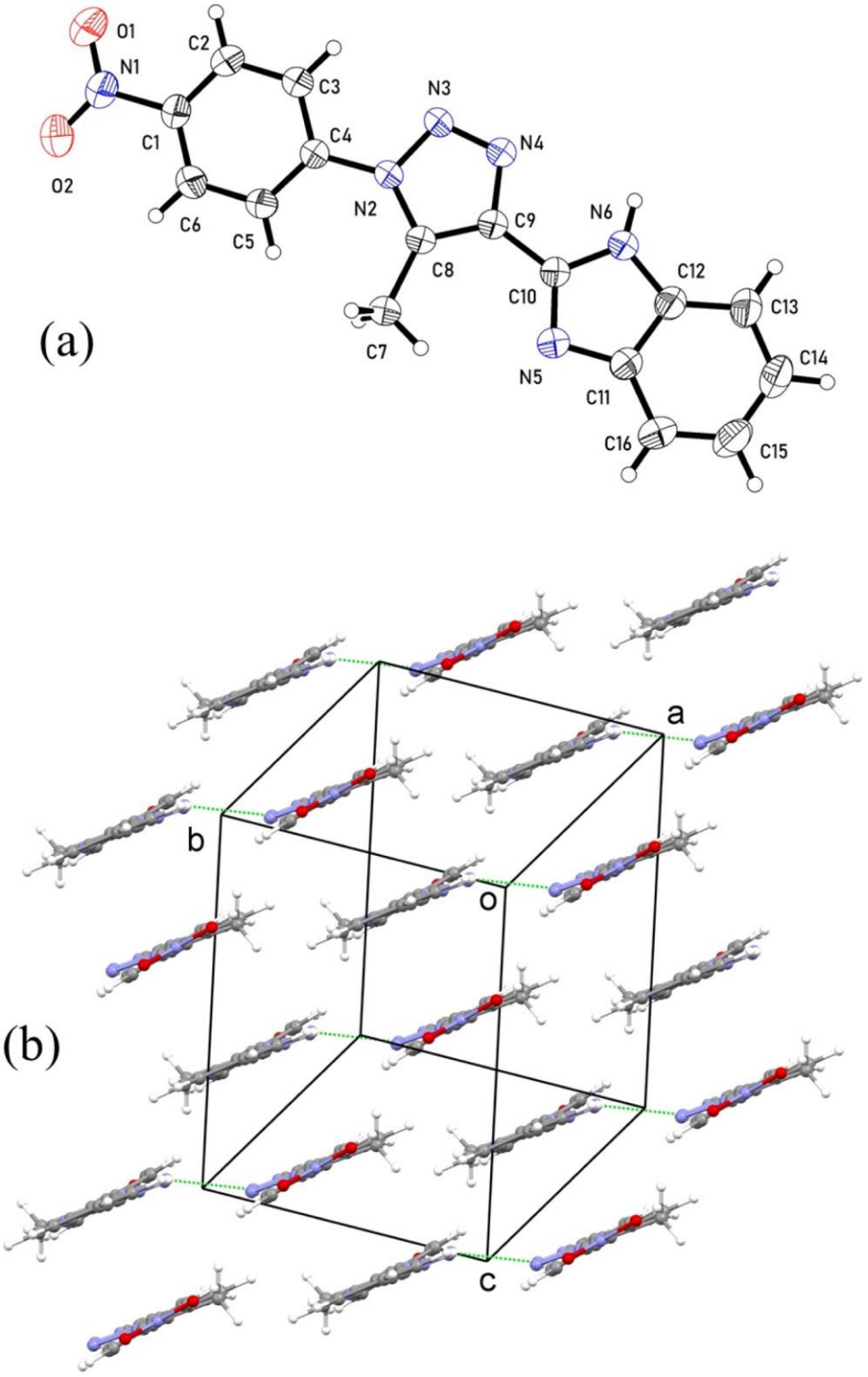
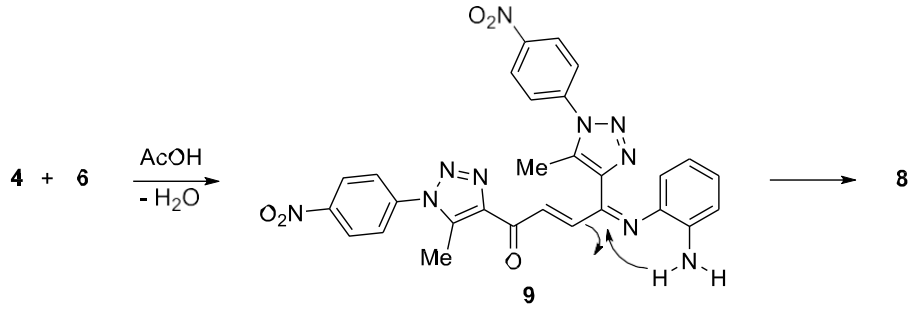



**Fig. 2.** (a) An ortep representation of **8** showing atomic displacement ellipsoids at 50 % probability and (b) crystal packing viewed along the molecular axis with N–H···H contacts shown as dotted lines.

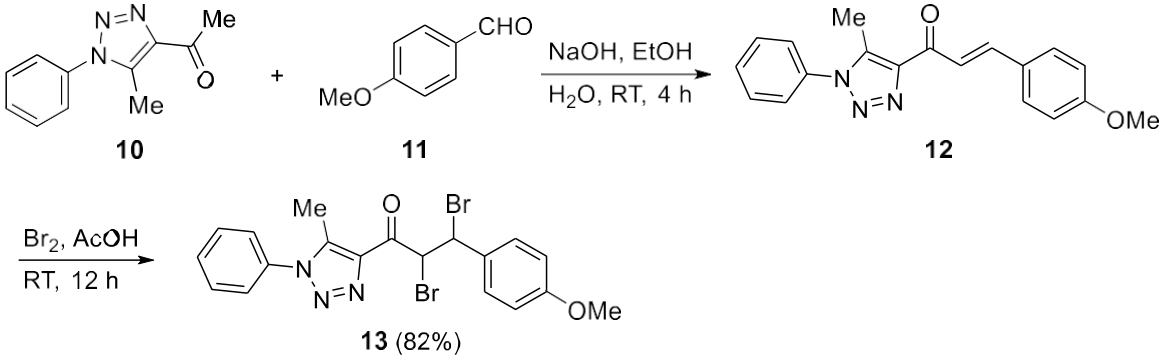
inversion symmetry in the middle of the butenedioyl group. The butenedioyl and methyltrazolyl groups are almost coplanar in the molecule. The twist angles between the planes of the groups, **nphen**/**mtria** and **mtria**/**bdio**, are 55.172(103)˚ and 19.23˚, respectively.

The molecules pack in the crystal structure with the molecular axis aligned to the [−102] direction (Fig. 1b). Close intermolecular C–H···O interactions form a network in the crystal structure. In the interactions, the C2–H2···O3, C6–H6···O1, and C7–H7A···O2 angles are 127.96˚, 140.29˚, and 171.21,˚ respectively and the corresponding C2···O3, C6···O1, and C7···O2 distances are 3.126 Å, 3.375 Å, and 3.593 Å, respectively.

A proposed mechanism for the production of **4** involves the formation of **3** as an intermediate from the reaction of **1** with **2**. The reaction between intermediate **3** and a second mole of **1** liberates HBr to produce




**Scheme 4.** A proposed mechanism for the formation of **8**.




**Scheme 5.** Synthetic route to 2,3-dibromoketone **13**.

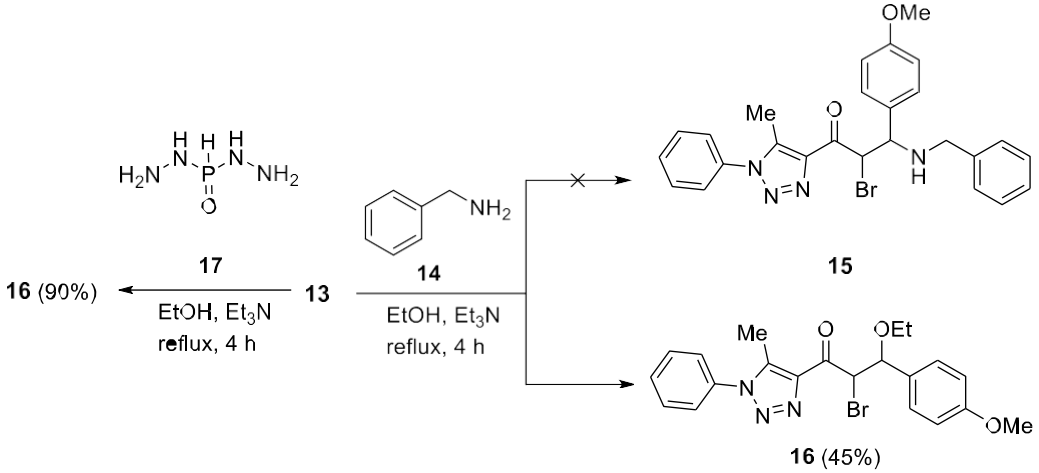
**5.** The elimination of benzenesulfinic acid ($PhSO_2H$) from **5** furnishes the desired product **4** (Scheme 2).

The reaction of **4** and 1,2-phenylenediamine (**6**) in refluxing EtOH containing acetic acid (AcOH) for 6 h afforded 2-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[d]imidazole (**8**) in 68 % yield. Under the conditions employed, the expected product, 2,5-bis(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)benzo[*b*][1,4]diazocine (**7**) was not produced (Scheme 3).

The FTIR spectrum of **8** showed strong absorption bands at 1611 to 1586 $cm^{-1}$ due to the C–C and N–N groups, respectively. The stretching vibrations of the N–H and C–H bonds appeared at 3118 and 3097 $cm^{-1}$, respectively, as broad bands. The $^1$H NMR spectrum of **8** showed a characteristic exchangeable singlet at 13.16 ppm corresponding to the NH proton. The methyl protons appeared as a singlet at 2.83 ppm. In addition, the four protons corresponding to the aryl ring appeared as two doublets (two protons each; $J$ = 8.6 Hz) at 8.05 and 8.47 ppm. The $^{13}$C NMR spectrum of **8** showed 14 signals that represented 16 carbons. For example, the methyl carbon appeared at a high field at 10.5 ppm. On the other hand, the C2 of the imidazole ring appeared at 148.3 ppm (at a low field). Additionally, the C-1 and C-4 of the aryl ring (4-nitrophenyl) appeared at 140.9 and 145.3 ppm, respectively.

Compound **8** was also structurally characterized using single-crystal X-ray diffraction, and the asymmetric unit is one molecule (Fig 2a). The molecule comprises nitrophenyl (**nphen**, C1–C6, N1, O1, O2), methyltriazolyl (**mtria**, C7–C9, N2–N4), and benzimidazolyl (**bimid**, C10–C16, N5, N6) groups (Fig. 2). The molecule has a nearly flat structure, with the twist angles between the planes through the groups being 10.57(12)˚ and 3.34(11)˚, respectively, for **nphen/mtria** and **mtria/bimid**.

In the crystal, the molecules are oriented parallel to the (13–5) plane (Fig. 2b) with neighboring molecules related by inversion symmetry and separated by 3.49 Å are involved in π···π interactions and stacked in the [110] direction. Adjacent stacks are linked by N–H···N interactions with N6–H6A···N4 angles of 149.9˚ and N6···N4 distances of 3.120(2)Å.




**Scheme 6.** Synthetic routes to 2-bromo-3-ethoxyketone **16**.

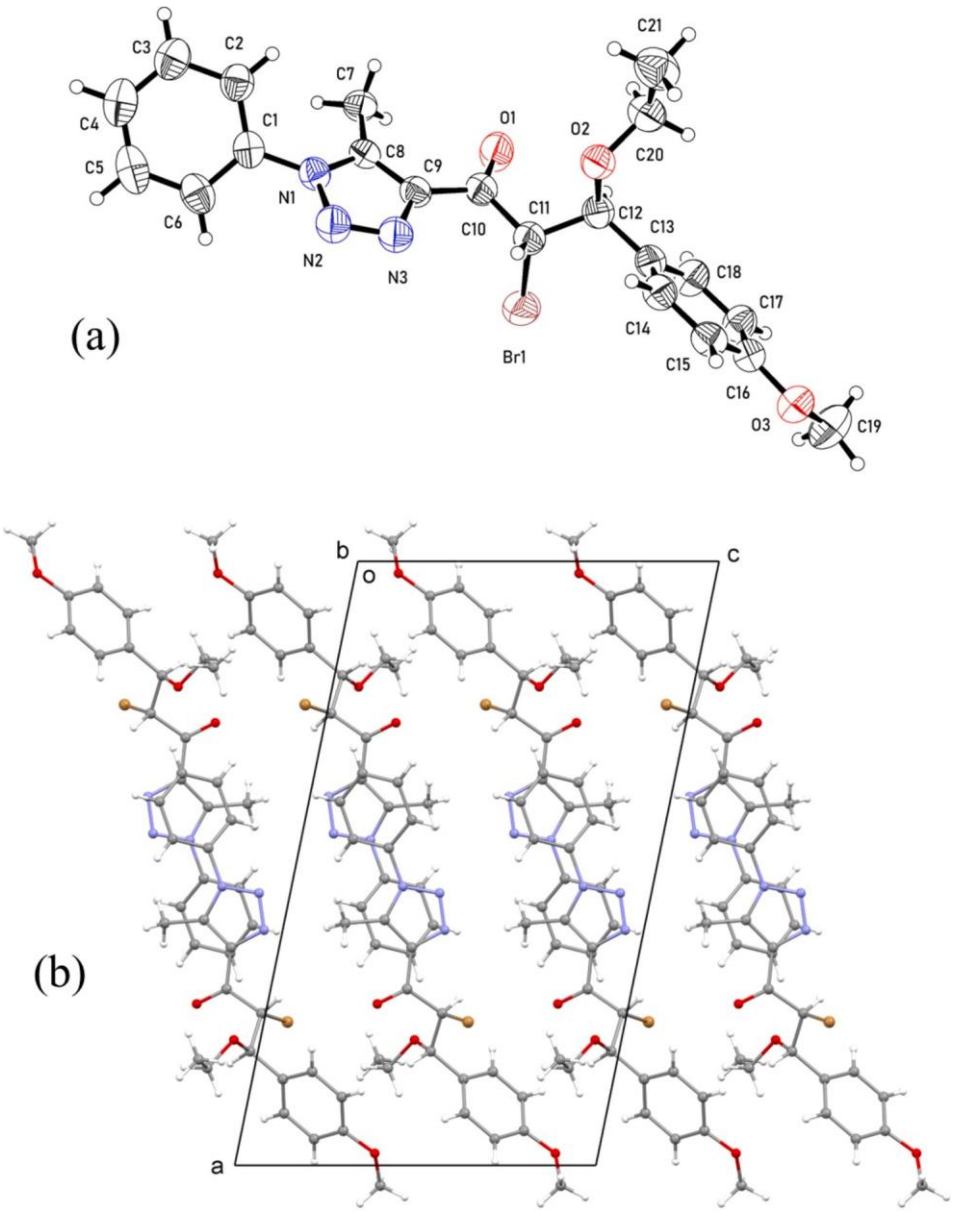



**Fig. 3.** (a) An ortep representation of **16** showing atomic displacement ellipsoids at 50 % probability and (b) crystal packing viewed down the *b* axis.

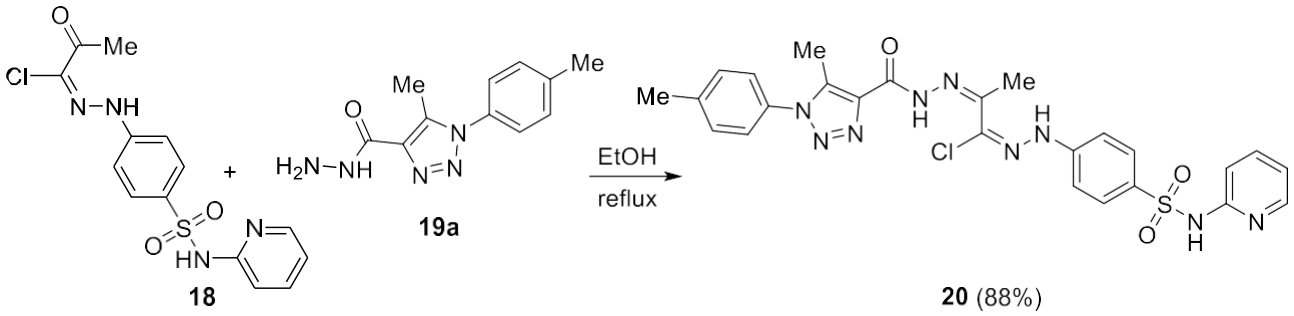
The suggested mechanism for the formation of **8** involves a dehydration reaction between **4** and **6** to produce an imine-amine intermediate **9**. The intramolecular cyclization of **9** leads to **8** (Scheme 4).

Stirring 4-acetyl-5-methyl-1-phenyl-1,2,3-triazole (**10**) and 4-anisaldehyde (**11**) in alcoholic sodium ethoxide (EtONa) afforded the reported chalcone **12** [41]. The addition of bromine to **12** in AcOH at room temperature afforded the 2,3-dibromo-3-(4-methoxyphenyl)-1-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)propan-1-one (**13**) in 82 % yield (Scheme 5).

The FTIR spectrum of **13** showed absorption bands at 3060 and 2972 $cm^{-1}$ due to the stretching vibration of the C–H bond. Also, the absorption bands corresponding to C–O, C–C, and N=N groups appeared at 1763, 1660, and 1581 $cm^{-1}$, respectively. The $^{1}H$ NMR spectrum of **13** showed the methyl and methoxy protons as singlet peaks at 2.58 and 3.82 ppm, respectively. The two CH protons appeared as a multiplet at 6.99–7.07 ppm. Additionally, the nine aromatic protons (phenyl and 4-methoxyphenyl rings) appeared as multiplets at the 7.63–7.84 ppm region. The solubility of **13** in DMSO-$d_6$ was poor, and the $^{13}C$ NMR spectrum was not recorded.

Treatment of **13** with benzylamine in boiling EtOH containing triethyl amine ($Et_3N$) as a catalyst afforded 2-bromo-3-ethoxy-3-(4-methoxyphenyl)-1-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)propan-1-one (**16**) in 45 % yield. There was no evidence for the formation of the expected 3-(benzylamino)-2-bromo-3-(4-methoxyphenyl)-1-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)propan-1-one (**15**) under the conditions used. When the reaction was carried out using phosphonic dihydrazide (**17**) instead of **14** under the same conditions, it afforded **16** in 90 % yield (Scheme 6).

The FTIR spectrum of **16** showed absorption bands at 3060 to 2950 $cm^{-1}$ due to the C–H bond stretching vibration. In addition, it showed strong absorption bands at 1772, 1612, and 1589 $cm^{-1}$ that attributed to the presence of the C–O, C–C, and N=N groups, respectively. Heterocycles **16** is highly insoluble in organic solvents. The sample was dissolved in boiling DMSO-$d_6$ to record the NMR spectra. The NMR spectra of **16** exhibited a mixture of two components in unequal proportion, but the reason for that is unclear. For the major isomer, the $^{1}H$ NMR spectrum of **16** showed the methyl and methylene protons of the ethyl group as a triplet and a quartet at 0.89 and 4.89 ppm, respectively.




**Scheme 7.** Synthesis of *bis*-hydrazone **20**.

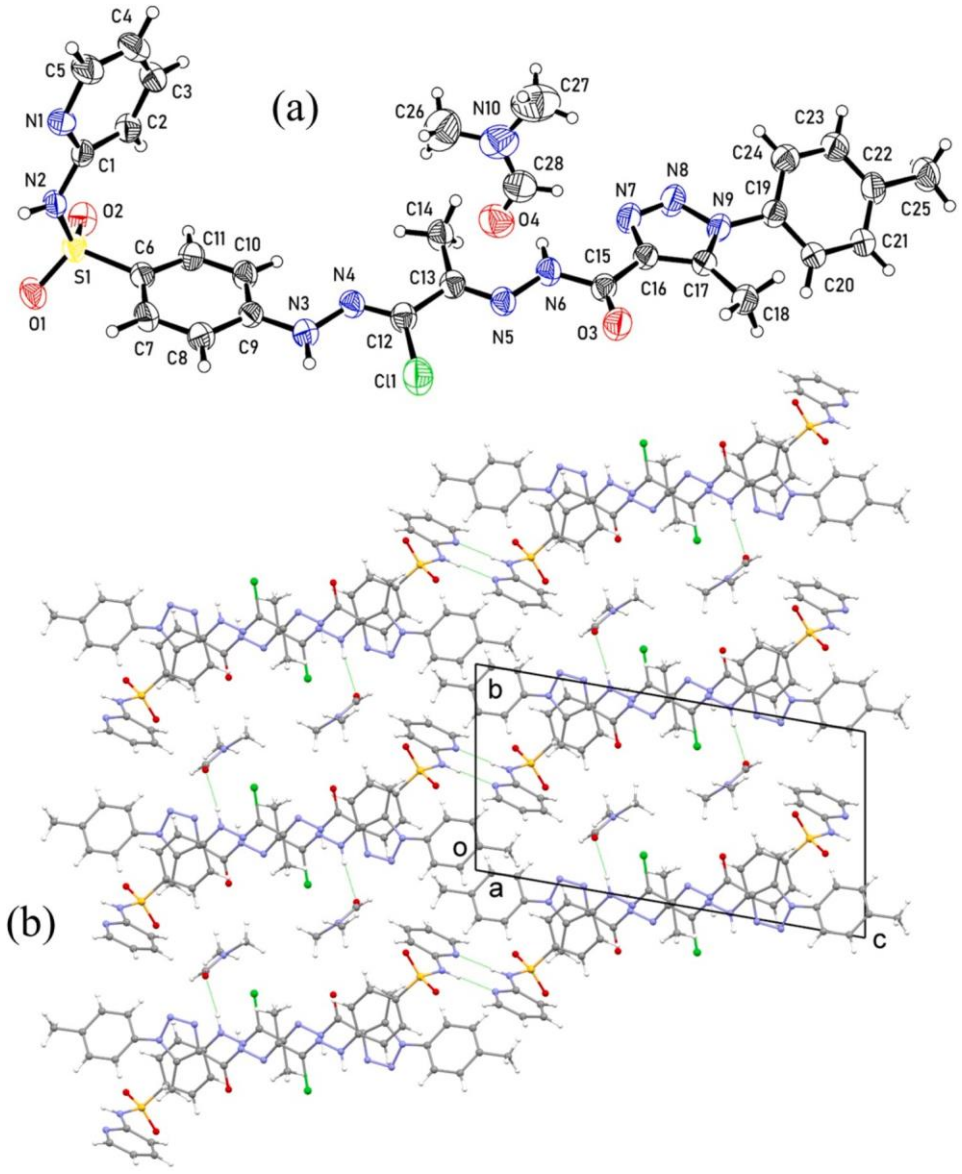
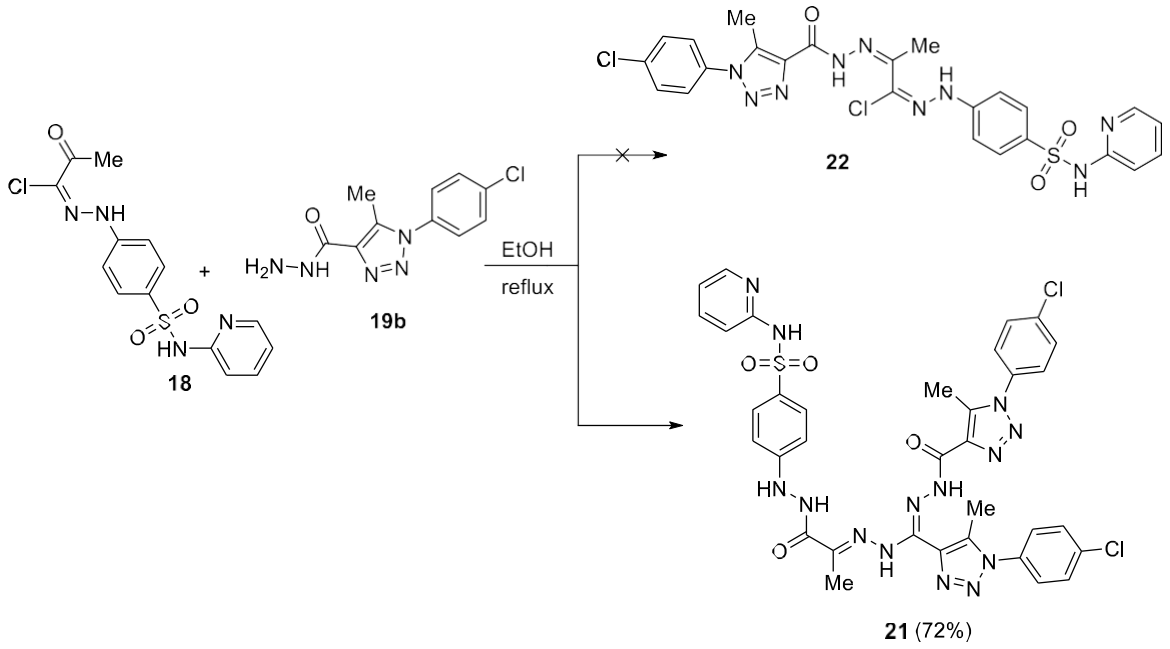



**Fig. 4.** (a) The asymmetric unit of **20** showing atomic displacement ellipsoids at 50 % probability and (b) crystal packing viewed along the *a* axis with N–H···O and N–H···N contacts presented as a green dotted line.

In comparison, the methyl and methoxy protons appeared as singlets at 2.57 and 3.80 ppm, respectively. In addition, the two CH protons appeared as two doublets at 5.59 and 5.84 ppm. In the $^{13}$C NMR spectrum of **16**, the C–O carbon appeared at 188.3 ppm (downfield), and the carbon of the methoxy group was observed at 55.6 ppm (upfield). The two CH carbons appeared at 64.8 and 80.5 ppm, and the two methyl carbons appeared at 10.4 and 15.4 ppm. The C-4 carbon of the 4-methoxyphenyl ring appeared at 160.0 ppm.

A crystal from a sample of compound **16** was analyzed using single-crystal X-ray diffraction and showed the presence of a single component.




**Scheme 8.** Synthesis of *tris*-hydrazone **21**.

The molecule consists of phenyl (**phen**, C1–C6), methyltriazolyl (**mtria**, C7–C9, N2–N4), carbonyl (C10, O1), bromoethoxyethane (**breth**, C11, C12, C20, C21, Br1, O2) and methoxyphenyl (**mphen**, C13–C20, O3) groups (Fig. 3a). In the molecule, the carbonyl and methyltriazolyl groups are coplanar. The twist angles between the planes through the rings, **phen**/**mtria** and **mtria/mphen,** are 46.60(6)˚ and 30.89(10)˚, respectively. The bromoethoxyethane group (**breth**) is in approximately all-trans conformation, with torsion angles: Br-C11–C12–O2 = 176.7 (2)˚, C11–C12–O2–C20 = 155.0(2)˚ and C12–02–C20–C21 = 166.7(2)˚.

In the crystal, the molecules are arranged in layers that are parallel to the bc plane (Fig. 3b). In one layer, phenyl and triazole rings of neighboring rings of molecules related by 2-fold screw symmetry are involved in π···π interactions to form stacks parallel to the *b* axis. The centroid-to-centroid distances between the triazole and phenyl rings of neighboring molecules in the stack are 4.21 Å and 3.77 Å. An intramolecular N–H···N contact with a N2–H2···N1 angle of 115.80˚ and N2···N1 distance of 2.654 Å occurs. C–H···N contact with a C7–H7A···N3 angle of 162.16˚ and C···N3 distance of 3.555 Å as well as C–H···Br contact with a C21–H21C···Br1 angle of 115.36˚ and C21···Br1 distance of 3.603 Å is also observed.

It has been reported that the reaction of 1,4-dihydro-1-phenylindeno [1,2-*c*]pyrazole-3-carbohydrazide with hydrazonoyl chlorides gave the corresponding *bis*-hydrazono compounds [48]. Under similar conditions, the reaction of (*E*)-2-oxo-*N*-(4-(*N*-(pyridin-2-yl)sulfamoyl)phenyl) propanehydrazonoyl chloride (**18**) and 5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbohydrazide (**19a**) was attempted. The product obtained, after four hours of reaction in dry EtOH under reflux, was 2-(2-(5-Methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbonyl)hydrazineylidene)-*N*-(4-(*N*-(pyridin-2-yl)sulfamoyl) phenyl)propanehydrazonoyl chloride (**20**) in 88 % yield (Scheme 7).

The IR spectrum of **20** showed absorption bands at 3348 and 2987 $cm^{-1}$ due to the N–H and C–H bond stretching vibrations, respectively. It showed a strong absorption band at 1702 $cm^{-1}$ due to the C–O group. In addition, the absorption bands corresponding to the C–C and C–N groups appeared at 1612 and 1592 $cm^{-1}$, respectively. The chemical structure of **20** was established by NMR spectroscopy and single-crystal X-ray diffraction (Fig. 4). The $^{1}$H NMR spectrum of **20** showed the presence of three exchangeable singlet signals that appeared in the 10.22–10.95 ppm region, corresponding to the three NH protons. The four aromatic protons due to the 4-methylphenyl ring appeared as two doublets (two protons each; *J* = 8.6 Hz) at 7.06 and 7.30 ppm. The signals for the protons of the three methyl groups appeared as singlets at 2.39, 2.47, and 2.52 ppm. There were three signals in the $^{13}$C NMR spectra, which appeared at 9.9, 13.7, and 21.3 ppm, and they corresponded to the carbons of the methyl groups. The carbonyl carbon signal appeared downfield at 162.9 ppm. Additionally, the C–Cl carbon appeared at 153.2 ppm, while the C-2 and C-6 of the pyridinyl ring appeared at 153.0 and 147.2 ppm, respectively.

The crystal structure consists of **20** and DMF solvent molecules (Fig. 4a). The central part of the molecule of **20** comprises phenyl (**phen**, C6–C11) and methyl-triazolyl (**tria**, C16, C17, N7–N9) rings as well as a diimine linking group (**diim**, N3–N6, C12–C15, O3, Cl1). In the crystal, the maximum deviation by atoms from the least-squares plane of the **diim** group is 0.07 Å. This part of the molecule is approximately planar, as indicated by the twist angles of 13.3 (1)˚ and 10.3 (1)˚ between groups **phen**/**diim** and **diim**/**tria,** respectively. An intramolecular C–H···O contact [C2–H2···O2 =120.3˚, C2···O2 = 3.061(4) Å] occurs between the pyridyl and sulfonyl groups.

Crystal packing is shown in Fig. 4b. The pyridinamine groups (C1–C5, N1, N2) of neighboring molecules of **20** in the structure interact through two equivalent N–H···N hydrogen bonds (with a N2–H2a···N1 angle of 163.5˚ and N2···N1 distance of 2.927(3) Å)) to form a ring with graph set geometry $R(8)^{2}_{2}$. The molecules of **20** are stacked along the *a* axis forming uninterrupted channels which accommodate the DMF solvent molecules. Hydrogen bonding of type N–H···O occurs between a molecule of **20** and DMF with a N3–H3A···O4 angle of 153.2˚ and N3···O4 distance of 2.992(6) Å.

The reaction of hydrazonoyl chloride (**18**) and 1-(4-chlorophenyl)-5-methyl-1*H*-1,2,3-triazole-4-carbohydrazide (**19b**) in boiling EtOH under the same conditions as for the synthesis of **20**, afforded the unexpected *tris*-hydrozone **21** in 72 % yield rather than the expected product **22** (Scheme 8).

The IR spectrum of **21** showed an absorption band at 3349 due to the vibration of the N–H group. The C–H stretching vibration bands appeared at 3228 and 3052 $cm^{-1}$. In addition, the C–O, C–C, and C–N absorption bands appeared at 1680, 1629, and 1593 $cm^{-1}$, respectively. The structure of **21** was confirmed by NMR spectroscopy and X-ray diffraction (Fig. 5). The $^{1}$H NMR spectrum of **21** showed the presence of five exchangeable singlets that appeared in the 10.13–10.93 ppm region and corresponded to the five NH protons. It also showed the protons

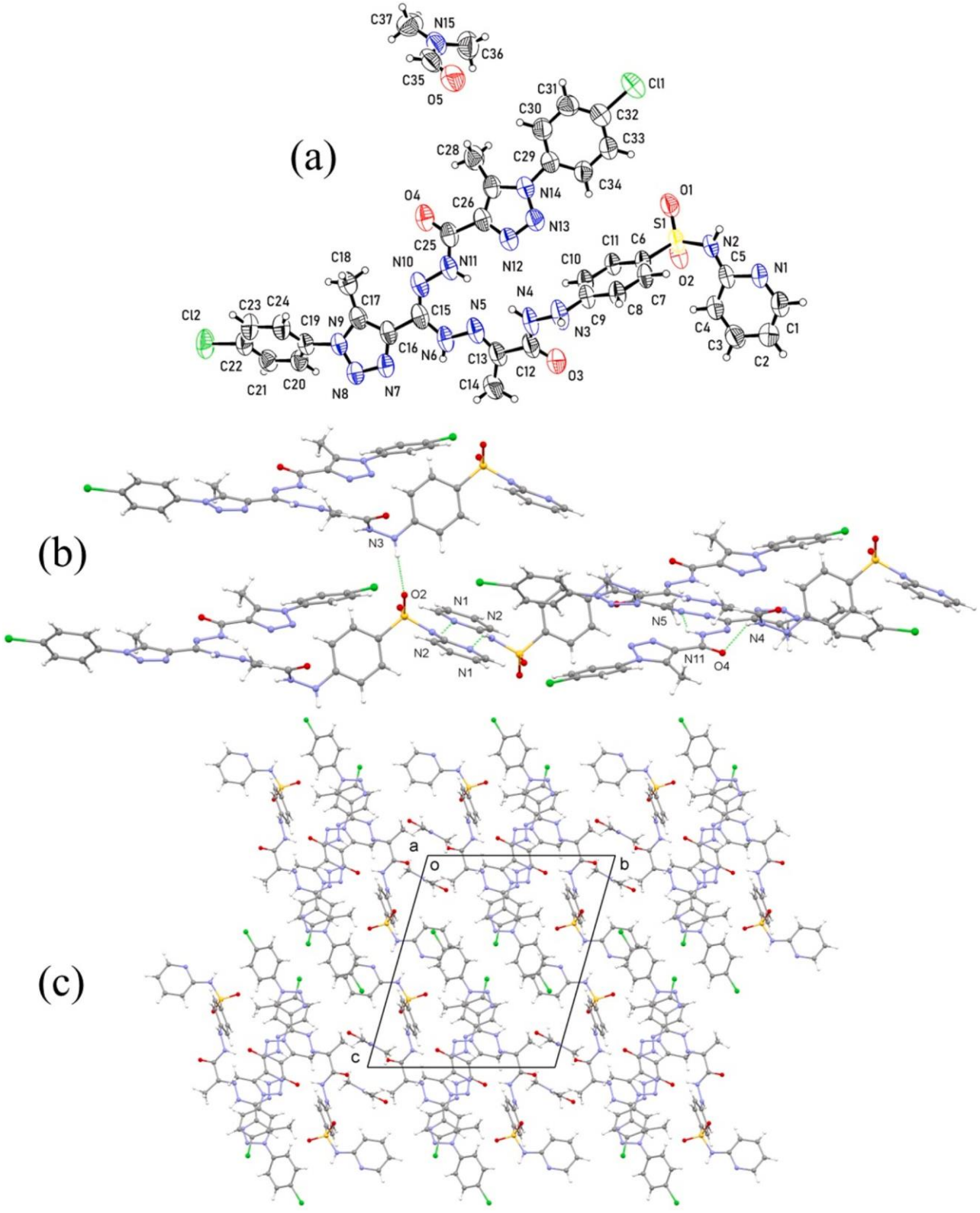
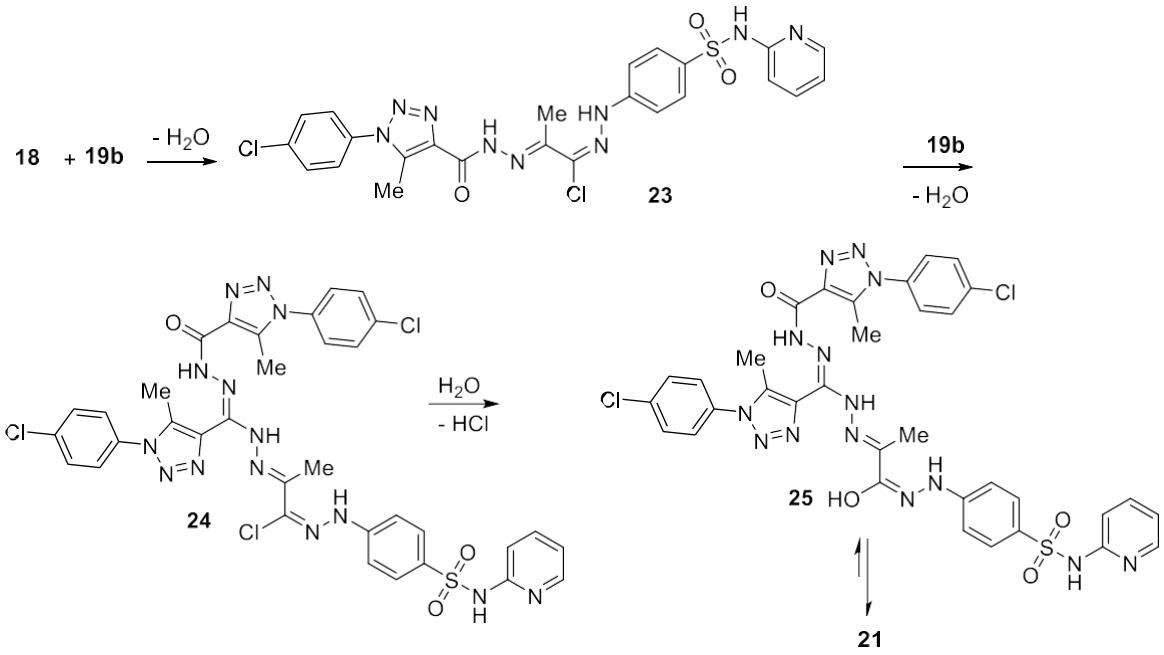



**Fig. 5.** (a) The asymmetric unit of **21** showing atomic displacement ellipsoids at 50 % probability, (b) a segment of the structure showing hydrogen bonding contacts as green dotted lines and (c) crtystal packing viewedalong the *a* axis.

corresponding to the three methyl groups at 2.17, 2.47, and 2.69 ppm. The aromatic protons appeared as multiplets at 6.70–6.85 (four protons) and 7.63–8.03 ppm (12 protons) regions. Three methyl carbons were detected in the $^{13}$C NMR spectrum, with chemical shifts of 10.0, 12.5, and 13.8 ppm. The two carbonyl carbons were observed at higher chemical shifts of 162.8 and 164.8 ppm.

In the molecule of compound **21**, the two triazolyl groups (**tria1**, [C16, C17, N7–N9] and **tria2**, [C26, C27, N12 –N14]) are linked by a formohydrazide group (**hydra**, C15, C25, N6, N10, N11, O4). The atoms of the **hydra** group have a maximum deviation of 0.075(2) Å from the



**Scheme 9.** Proposed mechanism for the formation of **21**.

least-squares plane. This group is essentially coplanar with the two triazolyl groups, as indicated by the twist angles **tria1**/**hydra** and **hydra**/**tria2** of 5.1(3)˚ and 7.7(3)˚, respectively. An intramolecular N–H···N contact (N11–H11A···N5 =130.5˚, N11···N5 = 2.755(7) Å) occurs in the molecule (Fig 5b).

Extended π···π interactions form columns of molecules along the *a* axis (Fig. 5c). The molecules are linked by a network of intermolecular N–H···N hydrogen bonds (with a N2–H2A···N1 angle of 148.8˚ and N2···N1 distance of 2.987(5) Å) and N–H···O bonds (with N3–H3A···O2 and N4–H4A···O4 angles of 170.5˚ and 140.3˚ respectively and N3···O2 and N4···O4 distances of 2.903(5) Å and 2.755(7) Å respectively). Hydrogen bonding involving the pyridinamine groups (C1–C5, N1, N2) of neighboring molecules of **21** forms rings with graph set geometry $R_2^2(8)$. Molecules of the DMF solvent occupy channels oriented parallel to the *a* axis in the structure and accept a C–H···O contact with a C14–H14A···O5 angle of 171.2˚ and C14···O5 distance of 3.245(17) Å.

A proposed mechanism for the formation of **21** involves the dehydration of **18** and **19b** to afford the intermediate **23**. A second mole of **19b** then reacts with **23** to give **24**. Hydrolysis of **24** and elimination of HCl affords **25**, which then tautomerizes to give product **21** (Scheme 9).

## 4. Conclusions

Novel heterocycles containing the 1*H*-1,2,3-triazole moiety have been synthesized in good yields using simple procedures. Many of the newly synthesized heterocycles were unexpectedly obtained under the conditions used. The structures of the new heterocycles have been established using various spectroscopic techniques and single-crystal X-ray diffraction.

## CRediT authorship contribution statement

**Bakr F. Abdel-Wahab:** Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Benson M. Kariuki:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Hanan A. Mohamed:** Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation. **Gamal A. El-Hiti:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

## Declaration of competing interest

There are no conflicts of interest to declare.

## Data availability

Data will be made available on request.

## Acknowledgments

We thank the National Research Centre and Cardiff University for technical support. Gamal A. El-Hiti acknowledges the support received from the Researchers Supporting Project (number RSP2024R404), King Saud University, Riyadh, Saudi Arabia.

## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2024.138404.

## References

[1] C. Paradisi, M. Prato, U. Quintily, G. Scorrano, Synthesis, structure, and reactivity of 1,4-diaryl-2-(arylamino)but-2-ene-1,4-diones, J. Org. Chem. 46 (1981) 5156–5159, https://doi.org/10.1021/jo00338a018.

[2] G. Yin, Z. Wang, A. Chen, M. Gao, A. Wu, Y. Pan, A new facile approach to the synthesis of 3-methylthio-substituted furans, pyrroles, thiophenes, and related derivatives, J. Org. Chem. 73 (2008) 3377–3383, https://doi.org/10.1021/jo702585s.

[3] M. Shindo, Y. Yoshimura, M. Hayashi, H. Soejima, T. Yoshikawa, K. Matsumoto, K. Shishido, Synthesis of multisubstituted furans, pyrroles, and thiophenes via ynolates, Org. Lett. 9 (2007) 1963–1966, https://doi.org/10.1021/ol0705200.

[4] H.S.P. Rao, S. Jothilingam, One-pot synthesis of pyrrole derivatives from (*E*)-1,4-diaryl-2-butene-1,4-diones, Tetrahedron Lett 42 (2001) 6595–6597, https://doi.org/10.1016/S0040-4039(01)01334-X.

[5] X. Meng, J. Zhang, G. Chen, B. Chen, P. Zhao, Heterogeneous synthesis of 1,4-enediones and 1,4-diketones with manganese oxide molecular sieves OMS-2 as a

recyclable catalyst, Catal. Commun. 69 (2015) 239–242, https://doi.org/10.1016/j.catcom.2015.07.003.
[6] S.-Y. Li, X.-B. Wang, N. Jiang, L.-Y. Kong, Synthesis of (*E*)-1,4-enediones from α-halo ketones through a sodium sulfinate mediated reaction, Eur. J. Org. Chem. 2014 (2014) 8035–8039, https://doi.org/10.1002/ejoc.201403236.
[7] M. Gao, Y. Yang, Y.-D. Wu, C. Deng, L.-P. Cao, X.-G. Meng, Z.-X. Wu, Formation of unsymmetrical 1,4-enediones via a focusing domino strategy: cross-coupling of 1,3-dicarbonyl compounds and methyl ketones or terminal aryl alkenes, Org. Lett. 12 (2010) 1856–1859, https://doi.org/10.1021/ol100473f.
[8] G. Yin, B. Zhou, X. Meng, Z.-X. Wu, Y. Pan, Efficient C—C double-bond formation reaction via a new synthetic strategy: A self-sorting tandem reaction, Org. Lett. 8 (2006) 2245–2248, https://doi.org/10.1021/ol060541e.
[9] B. Crone, S.F. Kirsch, A new method for the synthesis of *Z*-enediones via IBX-mediated oxidative rearrangement of 2-alkynyl alcohol systems, Chem. Commun. (2006) 764–766, https://doi.org/10.1039/B515838A.
[10] B. Salehi, C. Quispe, I. Chamkhi, N. El Omari, A. Balahbib, J. Sharifi-Rad, A. Bouyahya, M. Akram, M. Iqbal, A.O. Docea, C. Caruntu, G. Leyva-Go´mez, A. Dey, M. Martorell, D. Calina, V. Lo´pez, F. Les, Pharmacological properties of chalcones: a review of preclinical including molecular mechanisms and clinical evidence, Front. Pharmacol. 11 (2021) 592654, https://doi.org/10.3389/fphar.2020.592654.
[11] C. Zhuang, W. Zhang, C. Sheng, W. Zhang, C. Xing, Z. Miao, Chalcone: a privileged structure in medicinal chemistry, Chem. Rev. 117 (2017) 7762–7810, https://doi.org/10.1021/acs.chemrev.7b00020.
[12] N.A.A. Elkanzi, H. Hrichi, R.A. Alolayan, W. Derafa, F.M. Zahou, R.B. Bakr, Synthesis of chalcones derivatives and their biological activities: a review, ACS Omega 7 (2022) 27769–27786, https://doi.org/10.1021/acsomega.2c01779.
[13] H.A. Jasim, L. Nahar, M.A. Jasim, S.A. Moore, K.J. Ritchie, S.D. Sarker, Chalcones: synthetic chemistry follows where nature leads, Biomolecules 11 (2021) 1203, https://doi.org/10.3390/biom11081203.
[14] S. Mukhtar, N. Morsy, A. Hassan, T. Hafez, H. Hassaneen, F. Saleh, A review of chalcones: synthesis, reactions, and biological importance, Egypt. J. Chem. 65 (2022) 379–395, https://doi.org/10.21608/ejchem.2022.112735.5125.
[15] Y. Ma, X. Zheng, H. Gao, C. Wan, G. Rao, Z. Mao, Design, synthesis, and biological evaluation of novel benzofuran derivatives bearing N-aryl piperazine moiety, Molecules 21 (2016) 1684, https://doi.org/10.3390/molecules21121684.
[16] S. Samshuddin, B. Narayana, B.K. Sarojini, R. Srinivasan, M. Vinayachandra, K. R. Chandrashekar, Synthesis, characterization and biological evaluation of some pyrazoles derived from α,β-dibromo-4,4′-difluoro chalcone, Der Pharma Chem 4 (2012) 587–592.
[17] R. Kataria, S. Yadav, D. Pathak, Synthesis, spectral studies and biological profile of some new substituted diphenyl isoxazole derivatives, Res. J. Pharm. Biol. Chem. Sci. 4 (2013) 389–394.
[18] K. Jakhar, J.K. Makrandi, Ecofriendly bromination of chalcones and synthesis of flavones using grinding technique, Indian J. Chem. 52B (2013) 141–145.
[19] J. Berthelot, Y. Benammar, B. Desmazieres, Action of tetrabutylammonium tribromide with parasubstituted chalcones in protic and aprotic media, Can. J. Chem. 73 (1995) 1526–1530, https://doi.org/10.1139/v95-189.
[20] G. Verma, A. Marella, M. Shaquiquzzaman, M. Akhtar, M.R. Ali, M.M. Alam, A review exploring biological activities of hydrazones, J. Pharm. Bioallied Sci. 6 (2014) 69–80, https://doi.org/10.4103/0975-7406.129170.
[21] S.N. Mali, B.R. Thorat, D.R. Gupta, A. Pandey, Mini-review of the importance of hydrazides and their derivatives—Synthesis and biological activity, Process. Eng. 11 (2021) 21, https://doi.org/10.3390/ASEC2021-11157.
[22] A. Shamsabadi, V. Chudasama, An overview of the synthesis of acyl hydrazides from aldehydes and reactions of the products thereof, Org. Biomol. Chem. 15 (2017) 17–33, https://doi.org/10.1039/C6OB02099B.
[23] P. Majumdar, A. Pati, M. Patra, R.K. Behera, A.K. Behera, Acid hydrazides, potent reagents for synthesis of oxygen-, nitrogen-, and/or sulfur-containing heterocyclic rings, Chem. Rev. 114 (2014) 2942–2977, https://doi.org/10.1021/cr300122t.
[24] A.A. Hassan, A.M. Shawky, Chemistry and heterocyclization of carbohydrazides, J. Heterocycl. Chem. 47 (2010) 745–763, https://doi.org/10.1002/jhet.405.
[25] S. Rollas, S¸.G. Küçükgüzel, Biological activities of hydrazone derivatives, Molecules 12 (2007) 1910–1939, https://doi.org/10.3390/12081910.
[26] M.S. Ordon˜ez, A. Garcia de Torres, J.M. Cano Pavon, Di-2-pyridyl ketone 2-furoylhydrazone as a reagent for the fluorimetric determination of low concentrations of aluminium, Talanta 32 (1985) 887–891, https://doi.org/10.1016/0039-9140(85)80202-2.
[27] J. Pisk, I. Đilovi´c, T. Hrenar, D. Cvijanovi´c, G. Pavlovi´cc, V. Vrdoljak, Effective methods for the synthesis of hydrazones, quinazolines, and Schiff bases: reaction monitoring using a chemometric approach, RSC Adv 10 (2020) 38566–38577, https://doi.org/10.1039/D0RA06845D.
[28] A.S. Shawali, M.A. Abdallah, The chemistry of heterocyclic hydrazonoyl halides, Adv. Heterocycl. Chem. 80 (2001) 277–338, https://doi.org/10.1016/S0065-2725(08)60474-2.
[29] A.S. Shawali, M.M. Edrees, Reactions of nitrilimines with heterocyclic amines and enamines. Convenient methodology for synthesis and annulation of heterocycles, ARKIVOC 2006 (2006) 292–365, https://doi.org/10.1002/chin.200725234.
[30] C.P. Kaushik, J. Sangwan, R. Luxmi, K. Kumar, A. Pahwa, Synthetic routes for 1,4-disubstituted 1,2,3-triazoles: a review, Curr. Org. Chem. 23 (2019) 860–900, https://doi.org/10.2174/1385272823666190514074146.
[31] B.F. Abdel-Wahab, E. Abdel-Latif, H.A. Mohamed, G.E. Awad, Design and synthesis of new 4-pyrazolin-3-yl-1,2,3-triazoles and 1,2,3-triazol-4-yl-pyrazolin-1-ylthiazoles as potential antimicrobial agents, Eur. J. Med. Chem. 52 (2012) 263–268, https://doi.org/10.1016/j.ejmech.2012.03.023.
[32] R. Varala, H.B. Bollikolla, C.M. Kurmarayuni, Synthesis of pharmacological relevant 1,2,3-triazole and its analogues - A review, Curr. Org. Synth. 18 (2021) 101–124, https://doi.org/10.2174/1570179417666200914142229.
[33] J. Dai, S. Tian, X. Yang, Z. Liu, Synthesis methods of 1,2,3-/1,2,4-triazoles: a review, Front. Chem. 10 (2022) 891484, https://doi.org/10.3389/fchem.2022.891484.
[34] M. Di Antonio, G. Biffi, A. Mariani, E.A. Raiber, R. Rodriguez, S. Balasubramanian, Selective RNA versus DNA G-quadruplex targeting by in situ click chemistry, Angew. Chem., Int. Ed. Engl. 51 (2012) 11235–11240, https://doi.org/10.1002/ange.201206281.
[35] B.M. Kariuki, B.F. Abdel-Wahab, G.A. El-Hiti, Synthesis and structural characterization of isostructural 4-(4-aryl)-2-(5-(4-fluorophenyl)-3-(1-(4-fluorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl)-4,5-dihydro-1*H*-pyrazol-1-yl) thiazoles, Crystals 11 (2021) 795, https://doi.org/10.3390/cryst11070795.
[36] Y. Sert, G.A. El-Hiti, H. Go¨kce, F. Ucun, B.F. Abdel-Wahab, B.M. Kariuki, DFT, Molecular docking and experimental FT-IR, laser-Raman, NMR and UV investigations on a potential anticancer agent containing triazole ring system, J. Mol. Struct. 1211 (2020) 128077, https://doi.org/10.1016/j.molstruc.2020.128077.
[37] B.F. Abdel-Wahab, B.M. Kariuki, H.A. Mohamed, M.S. Bekheit, G.A. El-Hiti, Synthesis of new derivatives of the 1*H*-1,2,3-triazole ring using 1-aryl-5-phenyl-1*H*-1,2,3-triazole-4-carbohydrazides as precursors, Arkivoc 2023 (2023) 202312020, https://doi.org/10.24820/ark.5550190.p012.020.
[38] B.M. Kariuki, B.F. Abdel-Wahab, H.A. Mohamed, M.S. Bekheit, G.A. El-Hiti, Synthesis and characterization of novel 2-(1,2,3-triazol-4-yl)-4,5-dihydro-1*H*-pyrazol-1-yl)thiazoles and 2-(4,5-dihydro-1*H*-pyrazol-1-yl)-4-(1*H*-1,2,3-triazol-4-yl)thiazoles, Molecules 27 (2022) 8904, https://doi.org/10.3390/molecules27248904.
[39] A.S. Bunev, Y.O. Trushkova, G.I. Ostapenko, V.E. Statsyuk, A.S. Peregudov, Synthesis of 4-(1*H*-1,2,3-triazol-4-yl)-1,3-thiazole-2-amine derivatives, Chem. Heterocycl. Compd. 50 (2014) 1027–1031, https://doi.org/10.1007/s10593-014-1559-3.
[40] V.R. Kamalraj, S. Senthil, P. Kannan, One-pot synthesis and the fluorescent behavior of 4-acetyl-5-methyl-1,2,3-triazole regioisomers, J. Mol. Struct. 892 (2008) 210–215, https://doi.org/10.1016/j.molstruc.2008.05.028.
[41] S.V. Rodríguez-Guti´errez, O. Barreiro-Costa, C.D.A. Leo´n, J. Heredia-Moya, Synthesis and leishmanicidal activity of molecular hybrids 1,2,3-triazole-chalcones, Chem. Proc. 3 (2021) 55, https://doi.org/10.3390/ecsoc-24-08356.
[42] J.S. Shukla, M.G.H. Zaidi, Synthesis of some newer polyhydrazides containing hydrazinophosphite linkage, Asian J. Chem. 5 (1993) 253–258.
[43] B.F. Abdel-Wahab, H.E. Gaffer, M.M.G. Fouda, E.M. Osman, H.M. Fahmy, Synthesis of some new 2-[(2,3-dihydroinden-1-ylidene)hydrazinyl]-4-methylthiazole derivatives for simultaneous dyeing and finishing for UV protective cotton fabrics, J. Appl. Polym. Sci. 112 (2009) 2221–2228, https://doi.org/10.1002/APP.29768.
[44] Y. Zhang, R.-Z. Qiao, P.-F. Xu, Z.-Y. Zhang, Q. Wang, L.-M. Mao, K.-B. Yu, Synthesis and antibacterial activities of 2-(1-aryl-5-methyl-1,2,3-triazol-4-yl)-1,3,4-oxadiazole derivatives, J. Chin. Chem. Soc. 49 (2002) 369–373, https://doi.org/10.1002/jccs.200200057.
[45] G.M. Sheldrick, A short history of SHELX, Acta Crystallogr A64 (2008) 112–122, https://doi.org/10.1107/S0108767307043930.
[46] G.M. Sheldrick, Crystal structure refinement with SHELXL, Acta Crystallogr C71 (2008) 3–8, https://doi.org/10.1107/S2053229614024218.
[47] C.-K. Chan, M.-Y. Chang, Synthesis of (E)-1,4-diaryl-2-butene-1,4-diones, ARKIVOC 2016 (2016) 390–405, https://doi.org/10.24820/ark.5550190.p009.878.
[48] B. Hegazi, H.A. Mohamed, K.M. Dawood, F.A. Badria, Cytotoxicity and utility of 1-indanone in the synthesis of some new heterocycles, Chem. Pharm. Bull. 58 (2010) 479–483, https://doi.org/10.1248/cpb.58.479.